Synthesis and Evaluation of Tetrahydropyrazino Diisoquinolines as SARS-CoV-2 Entry Inhibitors and Development of Novel Protocols for the Synthesis of α-Chloroenaminones, 1,4-Benzodiazepines and 1*H*-Indenes

四氫吡嗪二異喹啉作為SARS-CoV-2進入抑制劑的合成與評估，以及α-氯烯胺酮、1,4-苯二氮䓬和1H-茚類化合物合成新方法的開發

A Dissertation submitted to the National Taiwan Normal University

for the Degree of Doctor of Philosophy in Chemistry

Submitted by

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CERTIFICATE

This is to certify that the work incorporated in the thesis entitled **"Synthesis and Evaluation of Tetrahydropyrazino Diisoquinolines as SARS-CoV-2 Entry Inhibitors and Development of Novel Protocols for the Synthesis of α-Chloroenaminones, 1,4-Benzodiazepines and 1*H*-Indenes"** submitted by Sowndarya Palla was carried out by her under my supervision at the Department of Chemistry, National Taiwan Normal University, Taipei, Taiwan.

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CANDIDATE'S DECLARATION

I hereby declare that the work presented in the dissertation entitled **"Synthesis and Evaluation of Tetrahydropyrazino Diisoquinolines as SARS-CoV-2 Entry Inhibitors and Development of Novel Protocols for the Synthesis of α-Chloroenaminones, 1,4-Benzodiazepines and 1*H*-Indenes"** submitted for Ph.D. degree to National Taiwan Normal University, Taipei, Taiwan. The work has been carried out by myself at the Department of Chemistry, National Taiwan Normal University, Taipei, Taiwan, R.O.C., under the supervision of Prof. Ching-Fa Yao. The work is original and any of the part of this work was not submitted by me for another degree or diploma to this or any other university. Any inadvertent omissions that might have occurred, due to oversight or error in judgment are regretted.

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Date: January 2025
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Dedicated to

My Father: Mr. Swamy Naidu Bongu

My Mother: Mrs. Gowramma Bongu

My Beloved Brothers: Mr. Koteswara Rao Bongu

Mr. Rajesh Kumar (Satish) Bongu

My Husband: Dr. Srinivasa Rao Palla Ph.D.

Gorgeous Daughters: Miss: Hanshvi Naidu Palla

Miss: Hanvika Naidu Palla

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LIST OF ABBREVIATION

°C	:	Degrees Celsius
$[\alpha]_D$	:	Specific rotation at the sodium D line (589 nm)
aq	:	Aqueous
Ar	:	Aryl
Bn	:	Benzyl
n-Bu	:	normal-Butyl
t-Bu	:	tertiary-Butyl
Bz	:	Benzoyl
cat.	:	Catalyst/Catalytic
d	:	Doublet in NMR spectroscopy
dd	:	Doublet of doublets in NMR spectroscopy
ddd	:	Doublet of double doublets in NMR spectroscopy
dt	:	Doublet of triplets in NMR spectroscopy
DABCO	:	1,4-Diazabicyclo[2.2.2]octane
DCM	:	Dichloromethane
1,2-DCE	:	1,2-Dichloroethane
DIPEA	:	*N*,*N*-Diisopropylethylamine
DMAP	:	4-Dimethylaminopyridine
DMSO	:	Dimethylsulfoxide
DMF	:	*N*,*N*-dimethylformamide
d.r.	:	Diastereomeric ratio
de	:	Diastereomeric excess
E	:	Entgegen (trans)
ent	:	Enantiomer
ee	:	Enantiomeric excess
e.r.	:	Enantiomeric ratio
ESI	:	Electro Spray Ionization
Et	:	Ethyl
equiv	:	Equivalent(s)
g	:	Gram(s)
h	:	Hours
HPLC	:	High performance liquid chromatography
HRMS	:	High resolution mass spectrometry
Hz	:	Hertz (cycles per second)

IR	:	Infra-red
J	:	Coupling constant (NMR)
λ	:	Wavelength
m	:	Multiplet, in NMR spectroscopy
Me	:	Methyl
mL	:	Milliliter(s)
mmol	:	Millimole
mol	:	Mole(s)
mp	:	Melting point
4Å MS	:	4-Angstrom molecular sieves
NMR	:	Nuclear magnetic resonance
o-	:	*ortho* position
m-	:	Meta position
p-	:	*para* position
Ph	:	Phenyl
^{1}H NMR	:	Proton nuclear magnetic resonance
ppm	:	Parts per million
i-Pr	:	Isopropyl
q	:	Quartet, in NMR spectroscopy
quin	:	Quintet, in NMR spectroscopy
R	:	Rectus (configurational)
rt	:	Room temperature
s	:	Singlet, in NMR spectroscopy
S	:	Sinister (configurational)
t	:	Triplet, in NMR spectroscopy
TEA	:	Triethyl amine
TFA	:	Trifluoroacetic acid
TFAA	:	Trifluoroacetic anhydride
THF	:	Tetrahydrofuran
TLC	:	Thin layer chromatography
TMS	:	Trimethylsilyl
Z	:	Zusammen (cis)
ACN	:	acetonitrile
Bn	:	Benzyl group
DIPEA	:	N,N-Diisopropylethylamine

DMAP	:	4-Dimethylaminopyridine
DBU	:	1,8-Diazabicyclo(5.4.0)undec-7-ene
DABCO	:	1,4-diazabicyclo[2.2.2]octane
$EtPh_2P$	:	ethlydiphenylphosphine
Et_2PhP	:	diethlyphenylphosphine
EtOAc	:	ethyl acetate
Et_3N	:	triethylamine
EDG	:	electron donating group
EWG	:	electron withdrawing group
$MePh_2P$	:	methlydiphenylphosphine
Me_2PhP	:	dimethlyphenylphosphine
PBu_3	:	tributylphosphine
PPh_3	:	triphenylphosphine

Part I

Green Synthesis and Evaluation of Tetrahydropyrazino[2,1-*a*:5,4-*a'*]diisoquinolines as SARS-CoV-2 Entry Inhibitors.

摘要

一類四氫吡嗪并[2,1-a:5,4-a']二異喹啉衍生物在以水作為溶劑的環保條件下被成功合成。部分合成化合物的三維結構通過X射線繞射確定。由於天然存在的異喹啉生物鹼對包括冠狀病毒在內的多種病毒具有顯著的抗病毒活性，對所合成的化合物進行了針對SARS-CoV-2的抑制活性測試。結果顯示，活性化合物50和96分別通過抑制SARS-CoV-2刺突蛋白受體結合域（RBD）與人類受體血管緊張素轉化酶2（ACE2）之間的相互作用，阻止Delta SARS-CoV-2進入VeroE6細胞，表現出EC50值分別為26.5 ± 6.9和17.0 ± 3.7 μM，且CC50均大於100 μM。本研究為異喹啉的抗病毒或其他應用提供了一種綠色合成方法。

Keywords: SARS CoV 2：嚴重急性呼吸系統綜合症

RBD：滾輪驅動

NSP：國際衛生戰略

Abstract

A class of tetrahydropyrazino[2,1-a:5,4-a']diisoquinoline derivatives were synthesized under an environmentally friendly condition using water as a solvent. The 3-D structures of some synthesized compounds were determined by X-ray diffraction. Since naturally occurring isoquinoline alkaloids have significant antiviral activities against a wide range of viruses, including coronaviruses, the synthesized compounds were assayed for their inhibitory activities against SARS-CoV-2. The results showed that the active compounds **50** and **96** blocked the delta SARS-CoV-2 entry into VeroE6 cells to display EC_{50} of 26.5 ± 6.9 and 17.0 ± 3.7 μM, respectively, by inhibiting the interaction between SARS-CoV-2 Spike's receptor binding domain (RBD) and human receptor angiotensin converting enzyme 2 (ACE2), and CC_{50} greater than 100 μM. This study provides a green synthesis method of isoquinolines for antiviral or other applications.

Keywords: SARS-CoV-2: Severe acute respiratory syndrome coronavirus 2

RBD: Receptor-Binding Domain

NSP: non-structural proteins

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Part I

1. Introduction

Since its emergence in December 2019, coronavirus disease 2019 (COVID-19) has caused a global pandemic with an estimated case mortality rate of approximately 1%. (1–3) The causative agent, severe acute respiratory syndrome coronavirus 2 (SARS-CoV-2), shares 79.6% of its genome with SARS-CoV, the virus responsible for the 2002–2003 SARS outbreak. (4,5) Unlike Middle East respiratory syndrome coronavirus (MERS-CoV), which uses dipeptidyl peptidase-4 (DPP4) as its receptor, both SARS-CoV and SARS-CoV-2 utilize the human angiotensin-converting enzyme 2 (ACE2) receptor. This interaction is mediated through the receptor-binding domain (RBD) of the virus's Spike glycoprotein. (6)

Following RBD-ACE2 binding, the Spike protein is cleaved at the S1/S2 and S2’ sites by two human proteases: Furin and transmembrane protease serine 2 (TMPRSS2). This cleavage primes the Spike protein, exposing the membrane fusion peptide and facilitating the fusion of the viral and host cell membranes, allowing viral RNA to enter the host cell. (7) TMPRSS2 can function independently of Furin, and its inhibition by serine protease inhibitors such as Camostat and Nafamost has shown potential to counteract SARS-CoV-2 as an off-target effect. (8,9) An alternative entry mechanism involves the human protease Cathepsin L, which facilitates endosomal entry of the virus. This pathway is particularly prominent in certain variants, such as the Omicron strain of SARS-CoV-2. (10)

After entering the host cell, the 29.7-kb positive-sense single-stranded RNA genome of SARS-CoV-2 is translated by the host's cellular machinery into two polyproteins, pp1a and pp1ab. These polyproteins are subsequently cleaved into 16 functional non-structural proteins (NSPs) by two viral proteases: 3C-like protease (3CLpro), which is homologous to the picornavirus 3C protease, and papain-like protease (PLpro). (11–13) Mature NSPs, such as NSP7, NSP8, NSP10, NSP12,

and NSP13, assemble into a replication-transcription complex. This complex, incorporating RNA-dependent RNA polymerase (RdRp), helicase, exonuclease, and other associated enzymes, synthesizes sub-genomic RNAs encoding the structural proteins Envelope (E), Nucleocapsid (N), Membrane (M), and Spike (S). These structural proteins are then assembled with the viral RNA to form new virus particles. (14)

Given their critical roles in viral replication, 3CLpro, PLpro, and RdRp are key therapeutic targets, and numerous covalent and non-covalent inhibitors have been developed. For example, Pfizer's Paxlovid, which combines the nitrile-based covalent 3CLpro inhibitor nirmatrelvir with ritonavir (a CYP3A4 inhibitor), has been approved for clinical use. (15) Additionally, the non-covalent 3CLpro inhibitor ensitrelvir has been approved in Japan, while China has granted conditional approvals for leritrelvir (17) and simnotrelvir (18,19). In terms of RdRp inhibitors, Gilead's Remdesivir, a nucleotide analog prodrug, has demonstrated efficacy in clinical trials and received approval for COVID-19 treatment. (20–23) Similarly, molnupiravir, a prodrug of the nucleoside analog N4-hydroxycytidine developed by Merck, has also been approved for its antiviral activity against SARS-CoV-2. (24,25) To prevent viral entry, several antibody-based therapies have been approved; however, no small-molecule inhibitors targeting viral entry have yet received approval. While small molecules that inhibit the interaction between the receptor-binding domain (RBD) of the Spike protein and human ACE2 or target human proteases have been identified, none have advanced to clinical use. (26–32) Despite these advancements, resistance to various anti-COVID-19 therapies has emerged, highlighting the ongoing need for the discovery and development of new drug candidates. (33–37)

Isoquinoline alkaloids are a class of naturally occurring compounds with notable antiviral activities against a wide range of viruses, including coronaviruses (CoVs). (38) Chemically,

isoquinoline features a unique structure formed by the fusion of benzene and pyridine rings, where the nitrogen atom of the pyridine ring is positioned away from the benzene ring. Isoquinoline alkaloids encompass various analogs, such as 1,2,3,4-tetrahydroisoquinoline derivatives, which contain a saturated pyridine ring with a secondary amine. Examples include tylophorine, dicentrine, corydaline, discretine, lycorine, emetine, and argemonine. Notably, chemical structures of emetine and cephaline, modified to reduce cytotoxicity by converting methoxy (OCH_3) groups to hydroxyl (OH) groups, are presented in Figure 1.

Another significant subclass of isoquinoline alkaloids features interconnected rings where the nitrogen atom forms a quaternary cation. Examples include palmatine and berberine, whose structures are also illustrated in Figure 1. Compounds like emetine, palmatine, and berberine have demonstrated the ability to inhibit SARS-CoV-2 through multi-target interactions with viral proteins, such as the Spike (S) protein, RNA-dependent RNA polymerase (RdRp), eIF4A, Nucleocapsid (N) protein, and open reading frame 6 (orf6) protein. (39) Additionally, cyclic bis-benzylisoquinoline alkaloids, such as berbamine, have been found to inhibit SARS-CoV-2 by binding to the post-fusion core of the S2 subunit of the Spike protein (Figure 1). (40) Screening a library of 188 natural products identified cepharanthine, another cyclic bis-benzylisoquinoline alkaloid, as a SARS-CoV-2 entry inhibitor. (41)

Inspired by these findings, our in silico studies suggested that extended diisoquinoline derivatives might block the interaction between the receptor-binding domain (RBD) of the Spike protein and human ACE2, thereby preventing viral entry. To explore this hypothesis, we developed an environmentally friendly synthesis protocol for tetrahydropyrazino[2,1-a:5,4-a']diisoquinoline derivatives. Using water as the solvent, isoquinoline or bromoisoquinoline was reacted with various phenacyl bromides, and reaction conditions were optimized to enhance yields. The use of

water as a solvent aligns with increasingly stringent regulations regarding organic solvents, emphasizing the need for greener and safer alternatives. As a non-toxic, cost-effective, and environmentally benign medium, water offers a sustainable option for organic transformations. (42) The synthesized tetrahydropyrazinodiisoquinoline compounds were characterized by X-ray diffraction to confirm their three-dimensional structures and evaluated for their ability to antagonize SARS-CoV-2 through inhibition of the RBD:ACE2 interaction. The green synthetic methodology described here provides a sustainable approach for producing tetrahydropyrazinodiisoquinoline derivatives for antiviral and other potential applications.

Figure 1

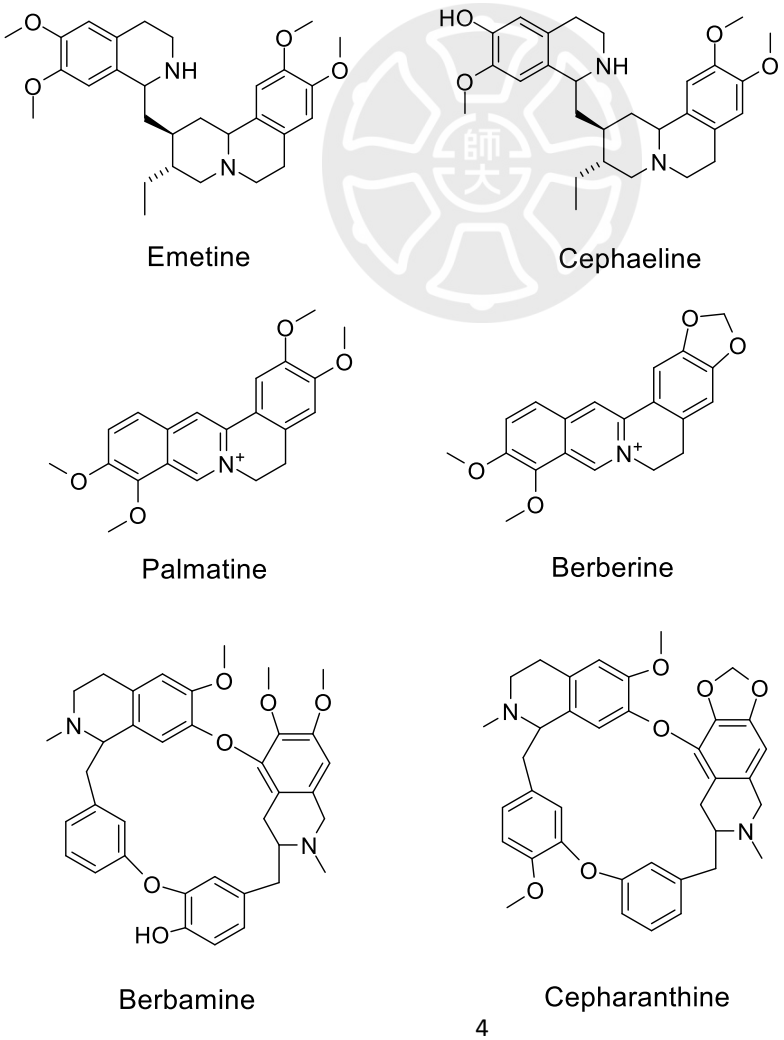


Figure 1. Chemical structures of select biologically active isoquinoline derivatives. These include emetine and its modified analog cephaline, where the OCH_3 group is replaced with an OH group to reduce toxicity; palmatine and berberine, both featuring a nitrogen quaternary cation; and berbamine and cepharanthine, which possess cyclic configurations.

2. Experimental Methods

2.1. General methods

The progress of compound synthesis was monitored using thin-layer chromatography (TLC) on silica gel. For purification, flash chromatography was conducted on silica gel with a particle size of 60–200 μm. Yields were calculated for compounds confirmed to be spectroscopically pure. Melting points were determined using a Fargo MP-1D Melting Point Apparatus.

NMR spectra were recorded on a Bruker AVIIIHD 400 MHz FT-NMR spectrometer at the Department of Chemistry, National Taiwan Normal University. Chemical shifts (δ) are reported relative to tetramethylsilane (TMS), and coupling constants (J) are provided in hertz (Hz). The internal standards used were $CDCl_3$ (δH = 7.24) or DMSO-d_6 (δH = 2.49) for ^{1}H-NMR spectra, and $CDCl_3$ (δC = 77.0) or DMSO-d_6 (δC = 39.5) for ^{13}C-NMR spectra. Splitting patterns are denoted as s (singlet), d (doublet), t (triplet), q (quartet), m (multiplet), br (broad), and dd (double of doublets). High-resolution mass spectra were obtained using a Bruker UPLC-MS at the TechComm core facility, Department of Chemistry, National Taiwan Normal University.

2.2. Synthesis of tetrahydropyrazino[2,1-a:5,4-a']diisoquinoline derivatives

Isoquinoline 1 (1 mmol), 2-bromoacetophenone 2 (1 mmol), K_2CO_3 (1 mmol), and H_2O (0.5 mL) were combined and stirred at 70 °C for 24 hours. The reaction progress was monitored via TLC, and upon complete consumption of isoquinoline 1, the mixture was allowed to cool to room temperature. The reaction mixture was then filtered, concentrated, and purified using silica gel flash chromatography (Hexane/Ethyl Acetate) to yield compound 3a. Other derivatives were synthesized similarly, using isoquinoline or bromoisoquinoline with various phenacyl bromides

under identical conditions.

Compound 3a (50)

Light yellow solid. 86% yield. mp: 178–179 ℃. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.75 (dd, *J* = 6.84 Hz, 4H), 7.47–7.42 (m, 2H), 7.35–7.31 (m, 4H), 7.06–7.01 (m, 2H), 6.90–6.83 (m, 6H), 6.37 (dd, *J* = 7.56 Hz, 2H), 5.59 (d, *J* = 7.28 Hz, 2H), 5.43 (d, *J* = 7.44 Hz, 2H), 5.01 (d, *J* = 8.9 Hz, 2H), ^{13}C NMR (100 MHz, $CDCl_3$): δ 194.6, 138.1, 136.2, 133.2, 131.9, 131.6, 128.6, 128.4, 128.3, 127.3, 126.2, 125.6, 123.2, 101.2, 65.1, 50.2. HRMS (ESI): m/z calculated for $C_{34}H_{27}N_2O_2^+$ $[M+H]^+$: 495.2067, found 495.2051.

Compound 3b (105)

Yellow solid. 53% yield. mp: 136–137 ℃. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.29 (d, *J* = 6.84 Hz, 2H), 7.25 (s, 5H), 7.20–7.16 (m, 2H), 7.13 (dd, *J* = 6.24 Hz, 1H), 7.09 (dd, *J* = 4.72 Hz, 2H), 6.99 (td, *J* = 7.44 Hz, 2H), 6.89 (d, *J* = 7.56 Hz, 2H), 6.01 (d, *J* = 7.28 Hz, 2H), 5.42 (d, *J* = 7.44 Hz, 2H), 5.26 (d, *J* = 8.88 Hz, 2H), 4.81 (d, *J* = 8.84 Hz, 2H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 198.1, 152.5, 138.2, 137.8, 131.8, 131.6, 130.9, 130.2, 129.3, 128.1, 126.7, 125.7, 123.5, 120.5, 100.9, 68.7, 49.9. HRMS (ESI): m/z calculated for $C_{34}H_{24}Cl_2{}^{(35)}N_2O_2^+$ $[M]^+$: 564.1180, found 564.1101 and for $C_{34}H_{24}Cl_2{}^{(37)}N_2O_2^+$ $[M]^+$: 566.1150, found 566.1160.

Compound 3c (110)

Light yellow solid. 61% yield. mp: 172–173 ℃. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.62 (dd, *J* = 5.96 Hz, 3H), 7.37–7.33 (m, 2H), 7.08–7.04 (m, 2H), 6.96–6.84 (m, 8H), 6.75 (d, *J* = 8.36 Hz, 2H), 6.19 (d, *J* = 7.48 Hz, 2H), 5.44 (d, *J* = 7.44 Hz, 2H), 5.30 (d, *J* =9.24 Hz, 2H), 5.16 (d, *J* = 8.92 Hz, 2H), 3.44 (s, 6H). ^{13}C NMR (100 MHz, DMSO-d_6) δ 199.0, 158.0, 139.0, 133.5, 132.4, 130.7, 127.5, 127.0, 123.0, 122.9, 120.8, 111.3, 99.2, 68.9, 55.2, 51.1. HRMS (ESI): m/z calculated for $C_{36}H_{31}N_2O_4$ $[M+H]^+$: 555.2278, found 555.2269.

Compound 3d (103)

Light orange solid. 53% yield. mp: 177–178 ℃. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.23 (d, *J* = 1.28 Hz, 1H), 7.16–7.06 (m, 9H), 6.96 -6.89 (m, 6H), 6.12 (t, *J* = 7.52 Hz, 2H), 5.45 (d, *J* = 7.56 Hz, 2H), 5.43 (d, *J* = 7.56 Hz, 2H), 4.79 (d, *J* = 9.08 Hz, 2H), 2.38 (s, 6H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 198.4, 138.5, 138.1, 137.6, 132.0, 131.7, 131.0, 128.1, 127.7, 127.5, 126.6, 125.5, 125.3,123.5, 100.6, 67.4, 49.9, 20.7. MALDI-TOF MS: m/z calculated for $C_{36}H_{30}N_2O_2^+$ $[M]^+$: 522.2307, found 522.2301.

Compound 3e (74)

Whitish yellow solid. 60% yield. mp: 140–141 ℃. ^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.79 (dd, *J* = 7.76 Hz, 4H), 7.10–6.86 (m, 12H), 6.37 (d, *J* = 7.44 Hz, 2H), 5.62 (d, *J* = 7.4 Hz, 2H), 5.40 (d, *J* = 8.8 Hz, 2H), 4.96 (d, *J* = 8.84 Hz, 2H). ^{13}C NMR (100 MHz, DMSO-d_6): δ = 194.4, 166.9, 164.4, 138.6, 132.5, 132.4, 132.3, 128.4, 127.5, 126.3, 125.9, 123.8, 116.3, 116.1, 101.0, 64.3, 50.2. HRMS (ESI): m/z calculated for $C_{34}H_{25}F_2N_2O_2^+$ $[M+H]^+$: 531.1879, found 531.1885.

Compound 3f (96)

Yellow solid. 54% yield. mp: 160–161 ℃. 1H NMR (400 MHz, $CDCl_3$): δ 7.68–7.66 (m, 4H), 7.32–7.28 (m, 4H), 7.09–7.05 (m, 2H), 6.93 (d, *J* = 7.48 Hz, 2H), 6.89–6.84 (m, 4H), 6.34 (dd, *J* = 7.48 Hz, 2H), 5.62 (d, *J* = 7.48 Hz, 2H), 5.36 (dd, *J* = 8.8 Hz, 2H), 4.92 (d, *J* = 8.8 Hz, 2H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 193.7, 139.7, 137.7, 134.4, 131.7, 130.9, 130.0, 128.8, 128.1, 127.6, 127.2, 126.7, 125.8, 123.7, 101.5, 65.2, 50.2. HRMS (ESI): m/z calculated for $C_{17}H_{13}ClNO^{2+}$ $[M/2 + H]^{2+}$: 282.0680, found 282.0686.

Compound 3g (68)

Light yellow solid. 30% yield. mp: 173–174 ℃. ^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.66 (d, *J* = 7.80 Hz, 4H), 7.14 (d, *J* = 7.84 Hz, 4H), 7.05 (t, *J* = 6.68 Hz, 2H), 6.91–6.81 (m, 6H), 6.38 (d, *J* =

7.36 Hz, 2H), 5.58 (d, *J* = 7.36 Hz, 2H), 5.44 (d, *J* = 8.84 Hz, 2H), 4.99 (d, *J* = 8.84 Hz, 2H), 2.32 (s, 6H). ^{13}C NMR (100 MHz, $CDCl_3$): δ = 194.3, 144.0 138.1, 133.7, 131.9, 129.1, 128.8, 128.2, 127.9, 127.8, 127.6, 126.8, 125.5, 123.4, 100.8, 65.0, 50.2, 21.5. HRMS (ESI): m/z calculated for $C_{36}H_{31}N_2O_2^+$ $[M+H]^+$: 523.2380, found 523.2383.

Compound 3h (72)

Light orange solid. 21% yield. mp: 152–153 ℃. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.74 (d, *J* = 8.84 Hz, 4H), 7.06 –7.02 (m, 2H) 6.91 (d, *J* = 7.4 Hz, 2H), 6.86–6.79 (m, 8H), 6.38 (d, *J* = 7.4 Hz, 2H), 5.57 (d, *J* = 7.44 Hz, 2H), 5.47 (d, *J* = 8.9 Hz, 2H), 4.98 (d, *J* = 8.9 Hz, 2H), 3.79 (s, 6H). ^{13}C (100 MHz, $CDCl_3$): δ 193.4, 163.5, 138.1, 132.0, 131.0, 129.2, 127.9, 127.8, 126.8, 125.5, 123.3, 113.7, 100.6, 64.9, 60.3, 55.4, 50.4, 14.1. HRMS (ESI): m/z calculated for $C_{36}H_{31}N_2O_4^+$ $[M+H]^+$: 555.2278, found 555.2279.

Compound 3i (115)

Light yellow solid. 57% yield. mp: 163–164 ℃. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.88 (d, *J* = 8.4 Hz, 3H), 7.60-7.47 (m, 8H), 7.47–7.39 (m, 8H), 7.11 (td, *J* = 7.16 Hz, 2H), 6.97–6.87 (m, 5H), 6.47 (d, *J* = 7.5 Hz, 2H), 5.66 (d, *J* = 7.44 Hz, 2H), 5.51 (d, *J* = 8.9 Hz, 2H), 5.08 (d, *J* = 8.9 Hz, 2H). ^{13}C NMR (100 MHz, DMSO-d_6) δ 194.2, 145.9, 139.7, 138.1, 131.9, 130.4, 129.3, 129.0, 128.9, 128.2, 128.0, 127.8, 127.6, 127.2, 127.1, 126.8, 126.6, 126.4, 125.7, 123.5, 101.2, 65.2, 50.2. HRMS (ESI): m/z calculated for $C_{46}H_{35}N_2O_2^+$ $[M+H]^+$: 647.2693, found 647.2660.

Compound 3j (106)

Light orange solid. 60% yield. mp: 167–168 ℃. ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.10 (s, 2H), 7.87–7.80 (m, 2H), 7.81–7.80 (m, 5H), 7.56–7.51 (m, 2H), 7.49–7.46 (m, 3H), 7.05–6.99 (m, 2H), 6.94 (d, *J* = 6.84 Hz, 2H), 6.88 (d, *J* = 7.12 Hz, 2H), 6.79 (td, *J* = 6.32 Hz, 2H), 6.48 (d, *J* = 7.52 Hz, 2H), 5.65 (d, *J* = 7.44 Hz, 2H), 5.57 (d, *J* = 8.9 Hz, 2H), 5.22 (d, *J* = 8.9 Hz, 2H). ^{13}C NMR (100

MHz, $CDCl_3$): 195.1, 152.4, 142.8, 138.0, 135.5, 133.6, 132.2, 132.0, 130.6, 129.6, 127.6, 127.6, 126.9, 126.6, 125.6, 124.2, 123.5, 120.5, 101.0, 65.3, 50.8. HRMS (ESI): m/z calculated for $C_{21}H_{16}NO^{2+}$ $[M/2+H]^{2+}$: 298.1226, found 298.1232.

Compound 3k (111)

Light orange solid. 70% yield. mp: 174–175 °C. ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.37 (d, *J* = 8.08 Hz, 2H), 7.89 (d, *J* = 8.08 Hz, 2H), 7.82 (d, *J* = 7.4 Hz, 3H), 7.54–7.46 (m, 7H), 7.34–7.28 (m, 4H), 7.10 (td, *J* = 6.32 Hz, 2H), 7.01 (t, *J* = 6.96 Hz, 2H), 6.91-6.85 (m, 3H), 6.12 (t, *J* = 6.68 Hz, 2H), 5.71 (d, *J* = 9.4 Hz, 2H), 5.44 (d, *J* = 7.48 Hz, 2H), 5.04 (d, *J* = 9.2 Hz, 2H). ^{13}C NMR (100 MHz, $CDCl_3$); δ 198.8, 138.1, 135.4, 133.8, 132.7, 132.1, 130.5, 128.4, 128.2, 128.0, 127.6, 127.4, 126.8, 126.5, 125.6, 125.6, 124.2, 123.6, 100.7, 67.8, 50.7. HRMS (ESI): m/z calculated for $C_{42}H_{31}N_2O_2^+$ $[M+H]^+$: 595.2380, found 595.2386.

Compound 3l (88)

Yellow solid. 22% yield. mp: 148–149 °C. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.41 (d, *J* = 1.92 Hz, 2H), 7.26–7.23 (m, 2H), 7.07–7.03 (m, 2H) 6.92 (d, *J* = 7.5 Hz, 2H), 6.85 (d, *J* = 4.40 Hz, 4H), 6.72 (d, *J* = 8.48 Hz, 2H), 6.37 (d, *J* = 6.92 Hz, 2H), 5.57 (d, *J* = 7.44 Hz, 2H), 5.48 (d, *J* = 8.84 Hz, 2H), 4.99 (d, *J* = 8.96 Hz, 2H), 3.86 (s, 12H). ^{13}C (100 MHz, $CDCl_3$): δ 193.5, 153.4, 149.0, 138.0, 131.9, 129.3, 127.9, 127.8, 126.9, 125.5, 123.4, 123.2, 111.0, 109.9, 100.6, 64.9, 56.0, 55.8, 50.7. HRMS (ESI): m/z calculated for $C_{38}H_{35}N_2O_6^+$ $[M+H]^+$: 615.2490, found 615.2497.

Compound 3m (98)

Yellow solid. 78% yield. mp: 176–177 °C. ^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.27–7.24 (m, 3H), 7.07–7.02 (m, 2H), 6.92 (d, *J* = 7.48 Hz, 2H), 6.86–6.82 (m, 5H), 6.69 (d, *J* = 8.0 Hz, 2H), 6.34 (d, *J* = 7.52 Hz, 2H), 5.97–5.96 (m, 4H), 5.57 (d, *J* = 7.48 Hz, 2H), 5.43 (d, *J* = 8.92 Hz, 2H), 4.92 (d, *J* = 8.96 Hz, 2H). ^{13}C NMR (100 MHz, $CDCl_3$): δ = 192.9, 151.9, 148.1, 137.9, 131.9, 131.0,

127.9, 127.7, 126.8, 125.5, 125.0, 123.4, 108.4, 107.7, 101.8, 100.8, 64.9, 50.5. HRMS (ESI): m/z calculated for $C_{36}H_{27}N_2O_6^+$ $[M+H]^+$: 583.1864, found 583.1867.

Compound 3n (114)

Light orange solid. 30% yield. mp: 153–154 ℃. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.64–7.61 (m, 4H), 7.51–7.49 (m, 4H), 7.12–7.08 (m, 2H), 6.96 (d, *J* = 7.48 Hz, 2H), 6.92–6.87 (m, 3H), 6.37 (d, *J* = 7.52 Hz, 1H), 5.65 (d, *J* = 7.48 Hz, 2H), 5.37 (t, *J* = 8.76 Hz, 2H), 4.94 (d, *J* = 8.84 Hz, 2H). ^{13}C NMR (100 MHz, $CDCl_3$); δ 193.8, 152.3, 142.6, 137.7, 135.8, 134.8, 132.2, 131.8, 130.1, 129.4, 126.5, 125.8, 123.7, 120.5, 101.6, 65.2, 50.1. HRMS (EI): m/z calculated for $C_{17}H_{11}Br_2NOK^+$ $[M/2 + K]^+$: 441.8833, found 441.0895.

Compound 3o (122)

Light orange solid. 74% yield. mp: 148–149 ℃. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.58–7.57 (m, 2H), 7.54–7.53 (m, 2H), 7.13–7.09 (m, 2H), 7.02–6.91 (m, 8H), 6.38 (d, *J* = 7.24 Hz, 2H), 5.66 (d, *J* = 7.48 Hz, 2H), 5.40 (d, *J* = 8.72 Hz, 2H), 4.74 (d, *J* = 8.8 Hz, 2H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 188.3, 152.4, 142.6, 138.0, 134.3, 132.9, 131.7 , 128.0, 127.1, 125.7, 123.5, 101.1, 67.6, 50.7, 29.7. HRMS (ESI): m/z calculated for $C_{30}H_{23}N_2O_2S_2^+$ $[M+H]^+$: 507.1195, found 507.1201.

Compound 3p (50Br)

Light yellow solid. 94% yield. mp: 115–116 ℃. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.82 (d, *J* = 7.52 Hz, 4H), 7.55 (t, *J* = 7.52 Hz, 2H), 7.42 (t, *J* = 7.64 Hz, 4H), 7.32 (d, *J* = 7.88 Hz, 2H), 6.85 (d, *J* = 7.36 Hz, 2H), 6.74 (t, *J* = 7.76 Hz, 2H), 6.55 (d, *J* = 7.56 Hz, 2H), 5.95 (d, *J* = 7.64 Hz, 2H), 5.44 (d, *J* = 9 Hz, 2H), 5.07 (d, *J* = 8.8 Hz, 2H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 193.9, 189.5, 172.1, 139.6, 135.8, 133.5, 132.1, 131.4, 129.1, 128.7, 128.7, 126.6, 126.0, 119.3, 100.0, 64.6, 50.1. MALDI-TOF MS: m/z calculated for $C_{34}H_{24}Br_2N_2O_2$ $[M]^+$: 650.0205, found 650.0285.

Compound 3q (103Br)

Yellow solid. 34% yield. mp: 177–178 ℃. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.37–7.29 (m, 4H), 7.21–7.18 (m, 4H), 7.15 (t, *J* = 7.2 Hz, 2H), 6.92 (d, *J* = 7.3 Hz, 2H), 6.82–6.77 (m, 2H), 6.29 (d, *J* = 8.0 Hz, 2H), 5.81 (d, *J* = 7.6 Hz, 2H), 5.45 (m, *J* = 8.0 Hz, 2H) 4.87 (d, *J* = 9 Hz, 2H), 2.43 (s, 6H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 197.7, 139.8, 138.7, 137.1, 132.2, 131.9, 131.6, 131.4, 128.8, 127.8, 126.5, 125.8, 125.5, 119.3, 99.5, 66.9, 50.1, 20.8. MALDI-TOF MS: m/z calculated for $C_{36}H_{28}Br_2N_2O_2^+$ $[M]^+$: 678.0518, found 678.0512.

Compound 3r (91Br)

Yellow solid. 21% yield. mp: 145–146 ℃. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.38 (s, 2H), 7.32 (t, *J* = 7.4 Hz, 6H), 7.08–7.05 (m, 2H), 6.84 (d, *J* = 7.36 Hz, 2H), 6.75 (t, *J* = 7.76 Hz, 2H), 6.53 (d, *J* = 7.72 Hz, 2H), 5.94 (d, *J* = 7.64 Hz, 2H), 5.43 (d, *J* = 8.84 Hz, 2H), 5.05 (d, *J* = 8.92 Hz, 2H), 3.83 (s, 6H). ^{13}C NMR (100 MHz, $CDCl_3$); δ 193.7, 159.9, 139.6, 137.1, 132.1, 129.6, 126.6, 126.0, 121.2, 120.2, 112.9, 100.0, 64.7, 58.4, 55.4, 50.2, 30.9,18.4. HRMS (ESI): m/z calculated for $C_{36}H_{29}Br_2N_2O_4^+$ $[M+H]^+$: 711.0489, found 711.0494.

Compound 3s (68Br)

Yellow solid. 60% yield. mp: 169–170 ℃. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.71 (d, *J* = 8.16 Hz, 3H), 7.31 (d, *J* = 8.0 Hz, 3H), 7.20 (d, *J* = 8.1 Hz, 4H), 6.84 (d, *J* = 7.44 Hz, 2H), 6.73 (t, *J* = 7.7 Hz, 2H), 6.54 (d, *J* = 7.6 Hz, 2H), 5.92 (d, *J* = 7.6 Hz, 2H), 5.44 (d, *J* = 8.8 Hz, 2H), 5.04 (d, *J* = 8.8 Hz, 2H), 2.36 (s, 6H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 193.5, 144.5, 139.7, 133.2, 132.0, 131.4, 129.3, 129.2, 128.8, 126.5, 126.0, 119.2, 99.8, 64.5, 50.1, 21.6. HRMS (ESI): m/z calculated for $C_{36}H_{29}Br_2N_2O_2^+$ $[M+H]^+$: 679.0590, found 679.0596.

Compound 3t (70Br)

Pale yellow solid. 44% yield. mp: 181–182 ℃. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.79 (d, *J* = 8.8 Hz,

4H), 7.31 (d, *J* = 8 Hz, 2H), 6.87–6.81 (m, 6H) 6.73 (t, *J* = 7.7 Hz, 2H), 6.53 (d, *J* = 7.68 Hz, 2H), 5.91 (d, *J* = 7.56 Hz, 2H), 5.46 (d, *J* = 8.92 Hz, 2H), 5.03 (d, *J* = 8.8 Hz, 2H), 3.83 (s, 6H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 192.5, 163.8, 148.0, 139.8, 132.0, 131.5, 131.1, 129.2, 128.8, 126.5, 126.1, 119.1, 113.8, 99.0, 64.5, 55.5, 50.4. HRMS (ESI): m/z calculated for $C_{18}H_{15}BrNO_2^{2+}$ [M/2 + H]$^{2+}$: 356.0275, found 356.0294.

Compound 3u (122Br)

Light yellow solid. 44% yield. mp: 176–177 °C. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.62 (d, *J* = 4.8 Hz, 2H), 7.58 (d, *J* = 3.8 Hz, 2H), 7.35 (d, *J* = 7.9 Hz, 2H) 7.06 (t, *J* = 4.4 Hz, 2H), 6.96 (d, *J* = 7.4 Hz, 2H), 6.80 (t, *J* = 7.7 Hz, 2H), 6.50 (d, *J* = 7.6 Hz, 2H), 5.98 (d, *J* = 7.6 Hz, 2H), 5.39 (d, *J* = 8.7 Hz, 2H), 4.78 (d, *J* = 8.7 Hz, 2H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 187.5, 142.2, 139.6, 134.7, 133.2, 132.2, 131.3, 129.0, 128.2, 126.7, 126.3, 119.3, 100.1, 67.1, 50.7. MALDI-TOF MS: m/z calculated for $C_{30}H_{20}Br_2N_2O_2S_2^+$ [M]$^+$: 661.9333, found 661.9306.

Compound 3v (115Br)

Light orange solid. 56% yield. mp: 156–157 °C. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.91 (d, *J* = 8.3 Hz, 3H), 7.63 (d, *J* = 8.4 Hz, 4H), 7.59 (t, *J* = 7.1 Hz, 4H), 7.48- 7.38 (m, 7H), 7.33 (t, *J* = 7.3 Hz, 2H), 6.88 (d, *J* = 7.4 Hz, 2H), 6.76 (t, *J* = 7.7 Hz, 2H), 6.60 (d, *J* = 7.6 Hz, 2H), 5.98 (d, *J* = 7.6 Hz, 2H), 5.48 (d, *J* = 8.8 Hz, 2H), 5.11 (d, *J* = 8.8 Hz, 2H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 193.4, 146.3. 139.7, 139.6, 134.4, 132.1, 131.4, 130.5, 129.3, 129.1, 128.9, 128.3, 128.0, 127.3, 127.2, 127.0, 126.7, 126.1, 119.3, 100.1, 64.8, 50.2. HRMS (EI): m/z calculated for $C_{46}H_{33}Br_2N_2O_2^+$ [M+H]$^+$: 803.0898, found 803.0909.

2.3. X-ray analysis of the synthesized compounds

Single-crystal X-ray data were obtained using the slow evaporation method with dichloromethane and ethyl acetate as the solvent system at room temperature. Diffraction measurements were

performed at 200 K on the resulting single crystals, employing Mo Kα radiation (λ = 0.71073 Å) with a Bruker Kappa APEX-II diffractometer. Crystal structures were solved and refined using the SHELX-97 software package. Detailed information about data collection and refinement parameters is provided in the supporting information, Tables S2−S6.

2.4.Antivirus EC_{50} and cytotoxicity CC_{50} measurements

The antiviral activity (EC_{50} values) of the synthesized compounds against SARS-CoV-2 was assessed using the plaque reduction assay as described previously. (26,43) In this assay, Vero E6 cells were cultured in a 24-well plate with Dulbecco's Modified Eagle Medium (DMEM) supplemented with 10% Fetal Bovine Serum (FBS) and antibiotics, seeded one day prior to infection. The cells were then infected with SARS-CoV-2 delta variant (NTU92) at 50−100 plaque-forming units (pfu) for 1 hour at 37 °C. Following infection, the inoculum was removed, the cells were washed with PBS, and overlaid with 1 mL of medium containing 1% methylcellulose. After 5 days of incubation at 37 °C, the cells were fixed overnight with 10% formalin. Post-fixation, the overlay medium was removed, and the cells were stained with 0.5% crystal violet to visualize and count plaques. The percentage of inhibition was calculated using the formula: $[1-(V_D/V_C)]\times 100\%$ where VD and VC represent virus titers in the presence and absence of inhibitors, respectively. EC_{50} values were determined by regression analysis of dose-response curves. Cytotoxicity of the inhibitors was evaluated using the acid phosphatase assay. Briefly, Vero E6 cells were seeded in a 96-well plate at a density of 2×10^4 cells per well. The following day, the medium was removed, and the cells were washed with PBS before adding DMEM containing 2% FBS and various concentrations of the inhibitors. After 1 hour of incubation at 37 °C in the presence of DMEM with 2 μg/mL TPCK-trypsin, the medium was removed, cells were washed with PBS, and fresh DMEM with 2% FBS and the inhibitors was added. After 3 days of

incubation at 37 °C, the medium was discarded, and cells were washed with PBS. Subsequently, a buffer containing 0.1 M sodium acetate (pH 5.0), 0.1% Triton X-100, and 5 mM p-nitrophenyl phosphate was added and incubated at 37 °C for 2 hours. The reaction was terminated with 1 N NaOH, and absorbance was measured at 405 nm using an ELISA reader (VERSAmax, Molecular Devices, Sunnyvale, CA). Cytotoxicity was calculated as: {Cytotoxicity %} = [1-(At/As) × 100] % where At and As denote the absorbance of the test substance and solvent control, respectively. CC_{50} values, indicating the concentration reducing 50% cell viability, were calculated. All measurements were performed in triplicate, and results were reported as mean values ± standard deviation.

2.5. Test of synthesized compounds on inhibiting RBD:ACE2

The active antiviral compounds were evaluated for their ability to inhibit the RBD:ACE2 interaction using a previously established cell-based assay. (26) The assessment employed the NanoBiT technology commercial kit (Promega, WI, USA) to measure inhibition of RBD:ACE2 binding. The IC50 values of the synthesized compounds were calculated from the concentration-dependent inhibition curves, which were fitted to the equation: $A(I) = A(0) \times \{1-[I/(I + IC_{50})]\}$ using GraphPad Prism software (v.9.4.0).

2.6. Molecular docking

Molecular docking studies were conducted using iGEMDOCK software available at (http://gemdock.life.nctu.edu.tw/dock/igemdock.php) (44) to predict the binding interactions of compound 50 with the receptor-binding domain (RBD) of the SARS-CoV-2 delta variant Spike protein. The three-dimensional (3D) structure of the RBD (PDB ID: 7w92) (45) was obtained from the RCSB Protein Data Bank (PDB). Prior to docking, water molecules and bound ligands were removed, and the RBD domain (residues 319 to 541) was isolated from the open state of the SARS-

CoV-2 delta variant Spike protein. The binding site was prepared by assigning residue atom types and charges using iGEMDOCK. The 3D structure of compound 50 was generated using the Molview website (https://molview.org/), and its structural data were converted into mol2 format with Open Babel GUI software. (46)

For the docking simulations, genetic algorithm (GA) settings included a population size of 800, 80 generations, and 10 solutions. iGEMDOCK calculated profiles of protein-ligand interactions based on electrostatic (E), hydrogen-bonding (H), and van der Waals (V) forces. Post-docking, all poses were analyzed and ranked by iGEMDOCK based on their estimated binding energies. The binding energy scores were determined as the sum of the interaction energies, with the pose exhibiting the lowest energy considered the optimal fit for compound 50 within the RBD binding site.

2.7. Drug likeness analysis

The 3D structure of compound 50 was converted to SMILES format using Open Babel GUI software. To assess the drug-likeness of compound 50, the Lipinski's rule of five was evaluated using an online tool (http://www.scfbio-iitd.res.in/software/drugdesign/lipinski.jsp). (47,48). The ADMET (Absorption, Distribution, Metabolism, Excretion, and Toxicity) profile of compound 50 was predicted using the pkCSM website http://biosig.unimelb.edu.au/pkcsm/). (49).

3. RESULTS

3.1. Synthesis of tetrahydropyrazino[2,1-a:5,4-a']diisoquinoline derivatives from isoquinoline and various phenacyl bromides

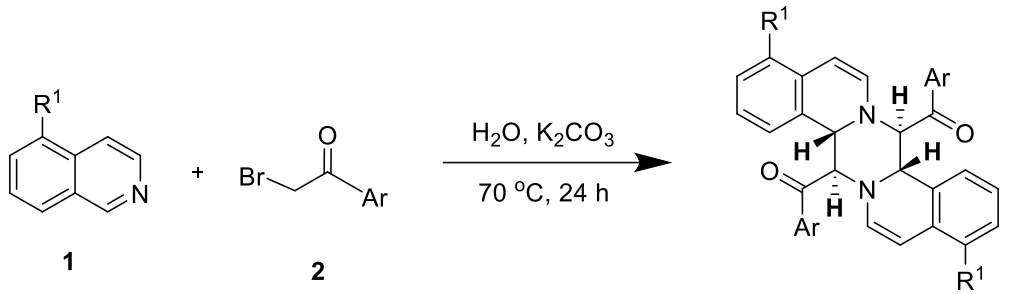
To synthesize the target molecules, we initially explored various solvents, bases, and temperatures. Using ethanol as the solvent, K_2CO_3 as the base, and a temperature of 40°C, we obtained an 86% yield for compound 3a. Further optimization revealed that using K_2CO_3 as the base and water as the solvent at 70°C resulted in a higher yield of 96% (Table 1). Based on this, the general synthetic procedure (Scheme 1) involved mixing isoquinoline 1 (1 mmol) with an R^1 substituent (H or Br),

phenacyl bromide 2 (1 mmol), K_2CO_3 (1 mmol), and water (0.5 mL), stirring at 70°C for 24 hours. Once the isoquinoline 1 was consumed (monitored by TLC), the reaction mixture was cooled to room temperature, filtered, concentrated, and purified by silica gel flash chromatography (Hex/EtOAc) to yield compounds 3a–3v as single diastereomers.

Table 1. Optimization of the synthesis of 3a using the reaction conditions of 1 (1 mmol), 2 (1 mmol), base (1 mmol), and solvent (0.5 mL), at various temperatures

Entry	Solvent	Base	TEMP (^{o}C)	t (h)	Yield (%)
1	H_2O	K_2CO_3	Sonication	2	20
2	H_2O	K_2CO_3	rt	12	30
3	H_2O	K_2CO_3	40 ^{o}C	28	42
4	H_2O	K_2CO_3	50 ^{o}C	28	58
5	H_2O	K_2CO_3	70 ^{o}C	24	96
6	H_2O	K_2CO_3	90 ^{o}C	24	96
7	H_2O	K_2CO_3	100 ^{o}C	24	96

Scheme 1. Synthesis of tetrahydropyrazino[2,1-a:5,4-a']diisoquinoline derivatives from isoquinoline or bromoisoquinoline and various phenacyl bromides



R^1 = H (**3a-3o**) or Br (**3p-3v**)

The chemical structures of the synthesized compounds were confirmed through NMR and MS spectral data. NMR assignments were made based on the additivity rules of substituents, spectral characteristics of structurally related compounds, signal intensities, and multiplicities. The 13C NMR spectra were particularly useful for confirming the interpretation of the carbon resonances. The 1H and 13C NMR spectra used for structural characterization are presented in Figure S1. The 3D structures of five selected compounds, determined by X-ray diffraction, are shown in Figure 2, with the corresponding statistical data summarized in Tables S2−S6.

Figure 2

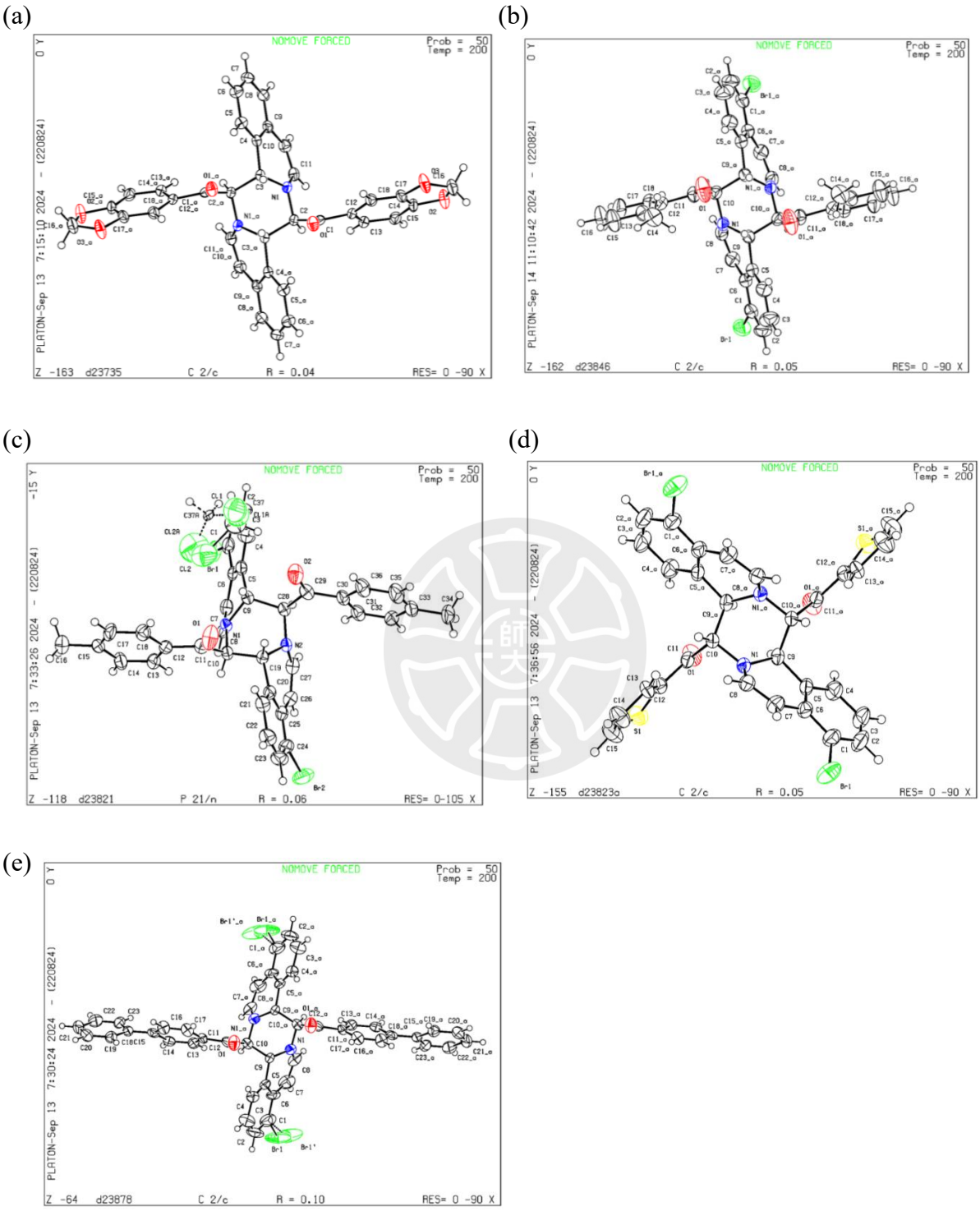
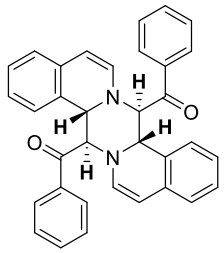
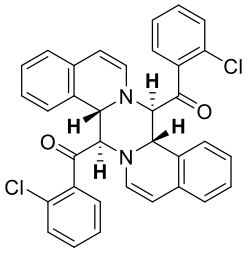
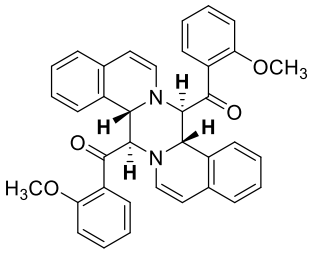
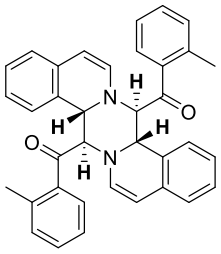
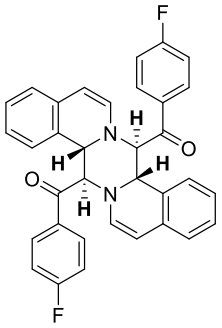
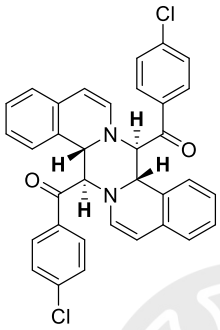
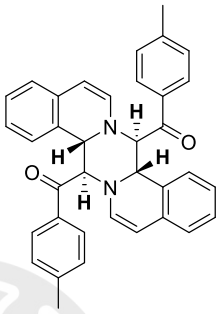
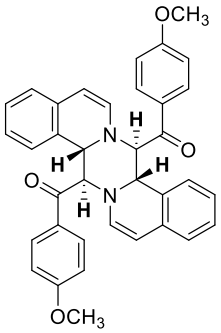
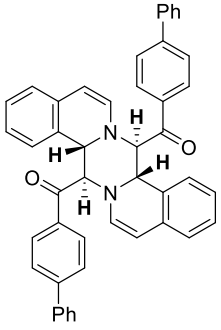
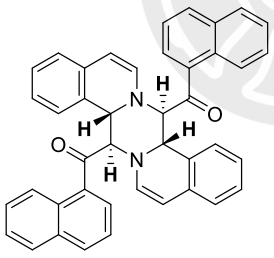
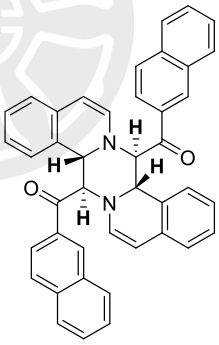
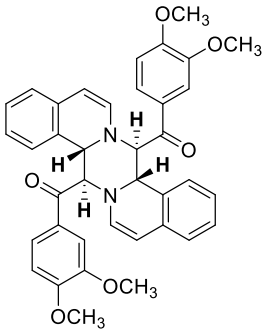
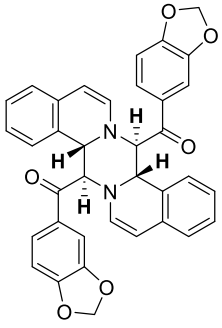
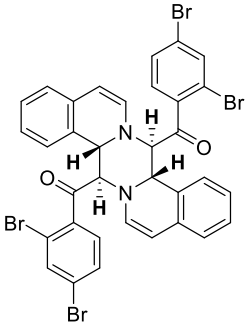
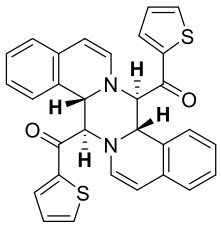


Figure 2. The 3D structures of compounds (A) 98, (B) 103Br, (C) 68Br, (D) 122Br, and (E) 115Br, as determined from the diffraction data presented in Tables S2−S6. Their corresponding chemical structures can be found in Tables 2 and 3.

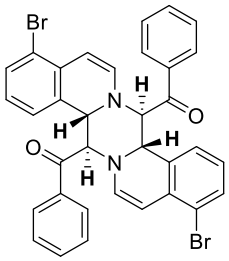
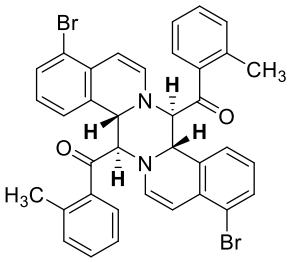
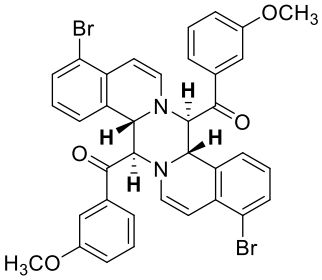
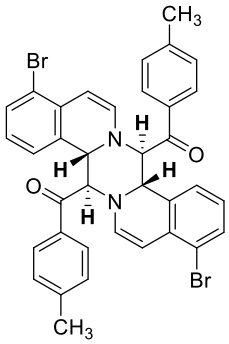
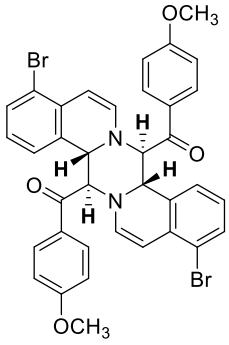
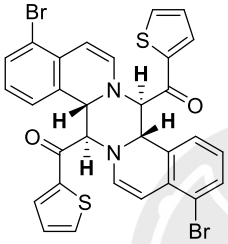
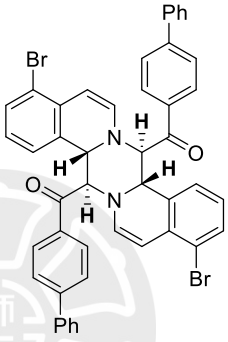
The chemical structures and yields of the synthesized compounds are summarized in Tables 2 and 3. The reaction with unsubstituted phenacyl bromide yielded the product (3a) in an excellent 96% yield. However, ortho- or para-substituted phenacyl bromides containing halogen or methyl groups produced the desired products in moderate yields (50−68%). Substrates with bulky diphenyl or naphthyl groups resulted in lower yields (40−50%) of the corresponding tetrahydropyrazino[2,1-a:5,4-a']diisoquinoline derivatives. In contrast, the substrate with a methylenedioxy group gave an excellent yield of product 3m (98%). The reaction of phenacyl bromides with a thiophene group produced the desired product 3o in a good yield (74%). Furthermore, the method was extended by replacing isoquinoline with bromoisoquinoline and various phenacyl bromides, yielding the corresponding tetrahydropyrazino[2,1-a:5,4-a']diisoquinoline derivatives (3p−3v) in good to moderate yields. Substrates without a substituent or with a phenyl group on the benzene ring gave the best yield (94%).

Table 2. Structures and yields of tetrahydropyrazino[2,1-a:5,4-a']diisoquinoline derivatives synthesized from isoquinoline and various phenacyl bromides[a,b]

 **3a** (**50**) (96%)	 **3b** (**105**) (50%)	 **3c** (**110**) (64%)	 **3d** (**103**) (56%)
 **3e** (**74**) (64%)	 **3f** (**96**) (68%)	 **3g** (**68**) (68%)	 **3h** (**70**) (42%)
 **3i** (**115**) (44%)	 **3j** (**106**) (48%)	 **3k** (**111**) (50%)	 **3l** (**88**) (40%)
 **3m** (**98**) (80%)	 **3n** (**114**) (64%)	 **3o** (**122**) (74%)	

[a]Reactions performed in 1 mmol scale. [b] Isolated yields

Table 3. Structures and yields of tetrahydropyrazino[2,1-a:5,4-a']dibromoisoquinoline derivatives synthesized from bromoisoquinoline and various phenacyl bromides[a,b]

 **3p** (**50Br**) (94%)	 **3q** (**103Br**) (58%)	 **3r** (**91Br**) (56%)	 **3s** (**68Br**) (66%)
 **3t** (**70Br**) (58%)	 **3u** (**122Br**) (66%)	 **3v** (**115Br**) (94%)	

[a]Reactions performed in 1 mmol scale. [b] Isolated yields

3.2. Antiviral activities of some synthesized compounds against SARS-CoV-2

The synthesized compounds were screened for their ability to inhibit infection by the SARS-CoV-2 delta variant in VeroE6 host cells. Due to their weak anti-SARS-CoV-2 activities, EC_{50} values could not be determined for some compounds. However, compounds 50 and 96 exhibited consistent activity during the entry phase (preincubation with the virus). Their dose-dependent inhibition profiles, shown in Figure 3, resulted in EC_{50} values of 26.5 ± 6.9 µM and 17.0 ± 3.7 µM, respectively, with no observed cytotoxicity (CC_{50} >100 µM). Neither compound was active when added to VeroE6 cells after infection (post-entry treatment), suggesting that their inhibitory action occurs outside the cells, targeting the virus entry process.

Figure 3

(a) (b)

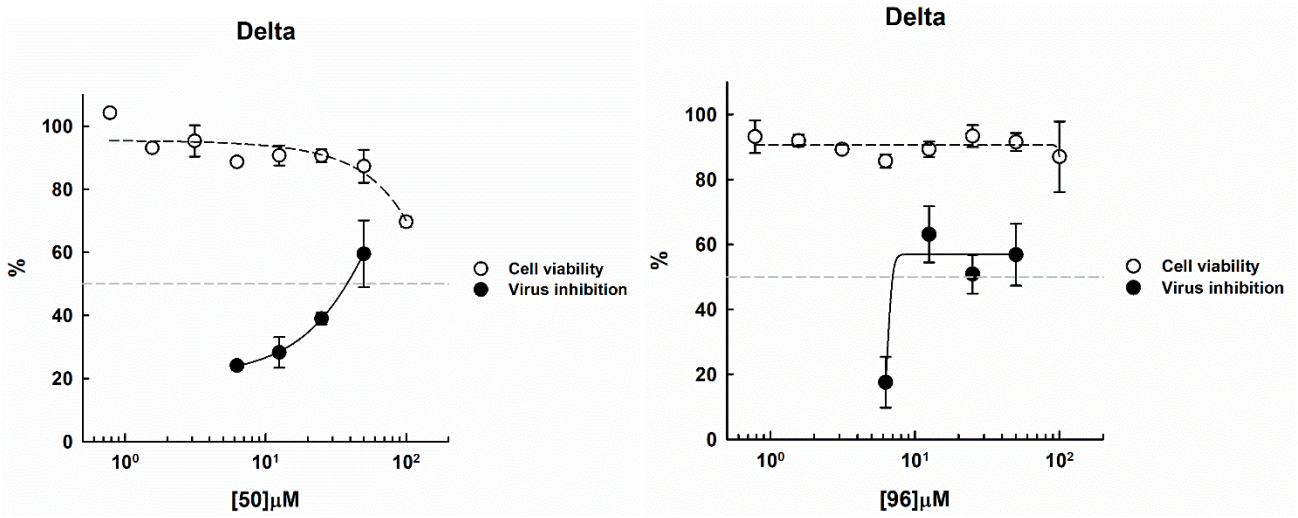


Figure 3. EC_{50} Measurements of Antiviral Activity for Active Compounds. (A, B) Dose-dependent inhibition curves for compounds 50 and 96, showing their EC_{50} values of 26.5 ± 6.9 µM and 17.0 ± 3.7 µM, respectively, against the delta variant of SARS-CoV-2 in VeroE6 cells. These curves were generated from plaque reduction assay data. The calculated CC_{50} values for both compounds were >100 µM. All measurements were performed in triplicate, with averaged EC_{50} values and standard deviations.

3.3. Evaluation of the active antivirals against RBD:ACE2 involved in virus entry

To identify the targets of the active antivirals, RBD:ACE2 interaction assays were conducted. Compounds 50 and 96 exhibited IC50 values of 32.7 ± 8.9 µM and 10.4 ± 2.1 µM, respectively, as shown in the dose-dependent inhibition profiles in Figure 4. These compounds did not inhibit the human proteases Furin, TMPRSS2, or Cathepsin L, which are also involved in virus entry (data not shown).

Figure 4

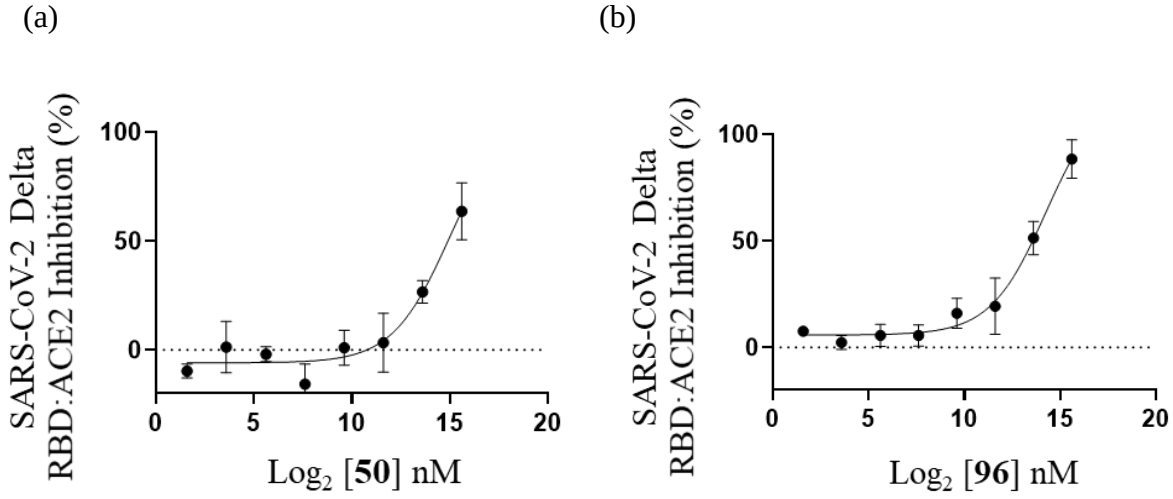


Figure 4. Inhibition of RBD:ACE2 Binding by the Compounds. (A,B) The percentages of RBD:ACE2 binding inhibition were measured at increasing concentrations of compounds, using RBD from the delta SARS-CoV-2 variant. The IC_{50} values obtained were 32.7 ± 8.9 μM for compound 50 and 10.4 ± 2.1 μM for compound 96. All measurements were performed in triplicate to calculate the averaged IC_{50} values and standard deviations.

3.4. Binding mode of the inhibitor with RBD

To investigate the inhibition mechanism of compound 50 in viral entry, a docking study was performed. Compound 50 was docked into the RBD of the SARS-CoV-2 delta variant Spike protein (PDB: 7w92), as illustrated in Figure 5. The binding energy of compound 50 with the RBD of the delta strain Spike protein was estimated to be -93.3 kcal/mol. The analysis showed that compound 50 forms van der Waals interactions with key residues, including Tyr448, Tyr453, Gln493, and Tyr505 of the RBD. In the delta RBD:ACE2 complex, Tyr453, Gln493, and Tyr505 from the RBD interact with His34, Lys31, and Glu37 of ACE2, respectively, through hydrogen bonding. Furthermore, an oxygen atom from one of the acetophenone groups in compound 50 forms a hydrogen bond with Gly496 of the RBD, which also forms a hydrogen bond with Lys353 of ACE2 in the delta RBD:ACE2 complex. Through these interactions with critical residues of the RBD, compound 50 may effectively disrupt the RBD:ACE2 binding.

Figure 5

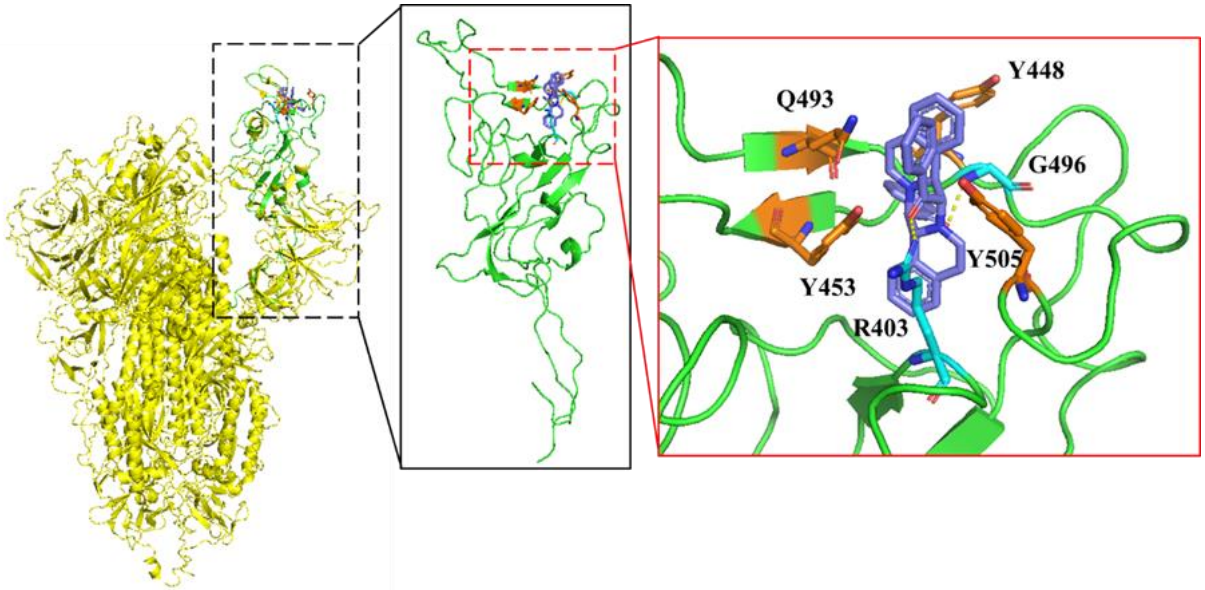


Figure 5. Binding interaction of active antiviral compound 50 with RBD. Compound 50 was docked into the RBD of the delta-strain Spike protein (PDB: 7w92). The compound is shown in purple, with residues involved in hydrogen bonding highlighted in cyan and those participating in van der Waals interactions depicted in orange. The trimeric Spike protein is represented in yellow, and the RBD is shown in green. In this model, compound 50 interacts with key residues at the RBD:ACE2 interface, suggesting its potential to interfere with the binding of RBD to ACE2.

3.5. Drug-likeness of the inhibitor as judged from Lipinski rule of five and ADMET properties

According to the Lipinski rule of five, compound 50 meets several important criteria: its logP value is 5, the number of hydrogen bond acceptors is 4 (less than 10), and the number of hydrogen bond donors is 0 (less than 5). The molecular weight of compound 50 is 494 Daltons, which is below the ideal limit of 500 Daltons. However, its molar refractivity of 146.6 exceeds the standard range (40–130), suggesting that compound 50 may have limitations in some drug-like properties. These parameters indicate that compound 50 has potential as a promising drug candidate.

Furthermore, in silico predictions of ADMET (Absorption, Distribution, Metabolism, Excretion, and Toxicity) properties offer useful insights into its therapeutic potential. The absorption properties suggest good permeability through the Caco-2 cell line and high human intestinal absorption (HIA), indicating efficient absorption in the human intestine. In terms of distribution,

compound 50 is predicted to cross the blood-brain barrier (BBB) and penetrate the central nervous system (CNS), with a higher volume of distribution at steady state (VDss) indicating extensive tissue distribution. Regarding metabolism, compound 50 is predicted to be a substrate for CYP2D6 and CYP3A4, but it is not expected to inhibit CYP1A2, CYP2C19, CYP2C9, CYP2D6, or CYP3A4. For excretion, compound 50 is unlikely to be a substrate for the renal organic cation transporter-2 (OCT2), reducing the likelihood of renal transport-related contraindications. Toxicity predictions show that compound 50 is not mutagenic, does not inhibit the hERG I channel, and does not exhibit skin sensitization potential. The predicted maximum recommended tolerated dose (MRTD) of compound 50 exceeds standard values, suggesting a favorable safety profile with minimal risk of adverse effects. However, a potential concern for hepatotoxicity should be considered.

4. DISCUSSION

Quinoline is a crucial building block in biologically active natural products, pharmaceuticals, and materials science, prompting significant efforts to develop environmentally friendly synthetic methods for quinoline derivatives. The synthesis of tetrahydropyrazino[2,1-a:5,4-a']diisoquinoline was first reported in 1969, where it was observed as a side product (50). Later, Kutsuma et al. synthesized this compound using isoquinoline and phenacyl bromide in the presence of trimethylamine, producing a single compound (51). More recently, Cui et al. reported the synthesis of hydrogenated pyrazino[2,1-a]isoquinoline derivatives from dihydroisoquinolines and phenacyl bromides in acetonitrile with base (52). However, to our knowledge, no green synthetic methodologies have been developed for these derivatives, and their bioactivities remain largely unexplored. In this study, we developed a green, water-based synthetic protocol for tetrahydropyrazino[2,1-a:5,4-a']diisoquinoline derivatives from isoquinoline and phenacyl

bromides. Water, an inexpensive, abundant, and non-toxic solvent, offers an environmentally friendly medium for organic reactions. This approach also allows for easy product isolation via simple filtration, avoiding complex purification processes. Initial studies focused on optimizing reaction conditions to improve yields. After identifying the optimal conditions, we synthesized a range of tetrahydropyrazino[2,1-a:5,4-a']diisoquinoline derivatives using various phenacyl bromides and isoquinoline. We then explored this method by substituting isoquinoline with bromoisoquinoline, obtaining the corresponding derivatives. In all reactions, the trans-diastereomer was the major product, with trace amounts of the other isomer, which was isolated as a pure trans-isomer through column chromatography. This protocol offers several advantages, including simplicity, broad substrate compatibility, good functional group tolerance, and easy product isolation. The synthesized compounds were screened for anti-SARS-CoV-2 activity, targeting the RBD:ACE2 interaction, as predicted by computational modeling. Our results indicate that compounds 50 and 96 inhibit the RBD:ACE2 interaction but do not affect human proteases, showing antiviral activity during entry treatment (when the compound is pre-incubated with the virus before infection). However, they were not effective in post-entry treatments (after the host cell was infected). This suggests that these compounds block virus entry by targeting the RBD:ACE2 interaction. Compared to berbamine hydrochloride, a cyclic bis-benzylisoquinoline alkaloid that binds to the post-fusion core of the SARS-CoV-2 S2 subunit, our compounds 50 and 96 show lower efficacy (EC50 = 26.5 and 17.0 μM, respectively) but better safety (CC50 > 100 μM). Isoquinoline and dihydroisoquinoline derivatives have distinct reactivities and bioactivities, and dihydroisoquinolines may be explored in future studies for the synthesis of natural product-like compounds. To understand the binding mechanism of these compounds at the RBD:ACE2 interface, we utilized computational modeling, which plays a key role in identifying how drug

candidates interact with target proteins. Understanding these interactions accelerates the drug discovery process, highlighting candidates with higher efficacy and fewer side effects. Computational studies of compound 50, for example, revealed its interaction with the Spike RBD of the SARS-CoV-2 delta strain. X-ray analysis shows that all synthesized compounds feature a central tetrahydropyrazino ring with four extended edges bearing different substituents, which helps block the RBD:ACE2 interface. Molecular docking, facilitated by iGEMDOCK software, was essential in identifying the binding modes and pharmacological interactions of the inhibitors. This tool is particularly useful for post-screening analysis and understanding inhibitor activity. Additionally, compound 50 adheres to the Lipinski rule of five and shows favorable ADMET profiles. However, further in vitro and in vivo studies are required to confirm its effectiveness and safety.

Keywords: SARS-CoV-2: Severe acute respiratory syndrome coronavirus 2
RBD: Receptor-Binding Domain
NSP: non-structural proteins

Keywords: SARS CoV 2：嚴重急性呼吸系統綜合症
RBD：滾輪驅動
NSP：國際衛生戰略

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6. Supplementary data

Figure S1. NMR spectra (^{1}H top and ^{13}C bottom) of the synthesized compounds.

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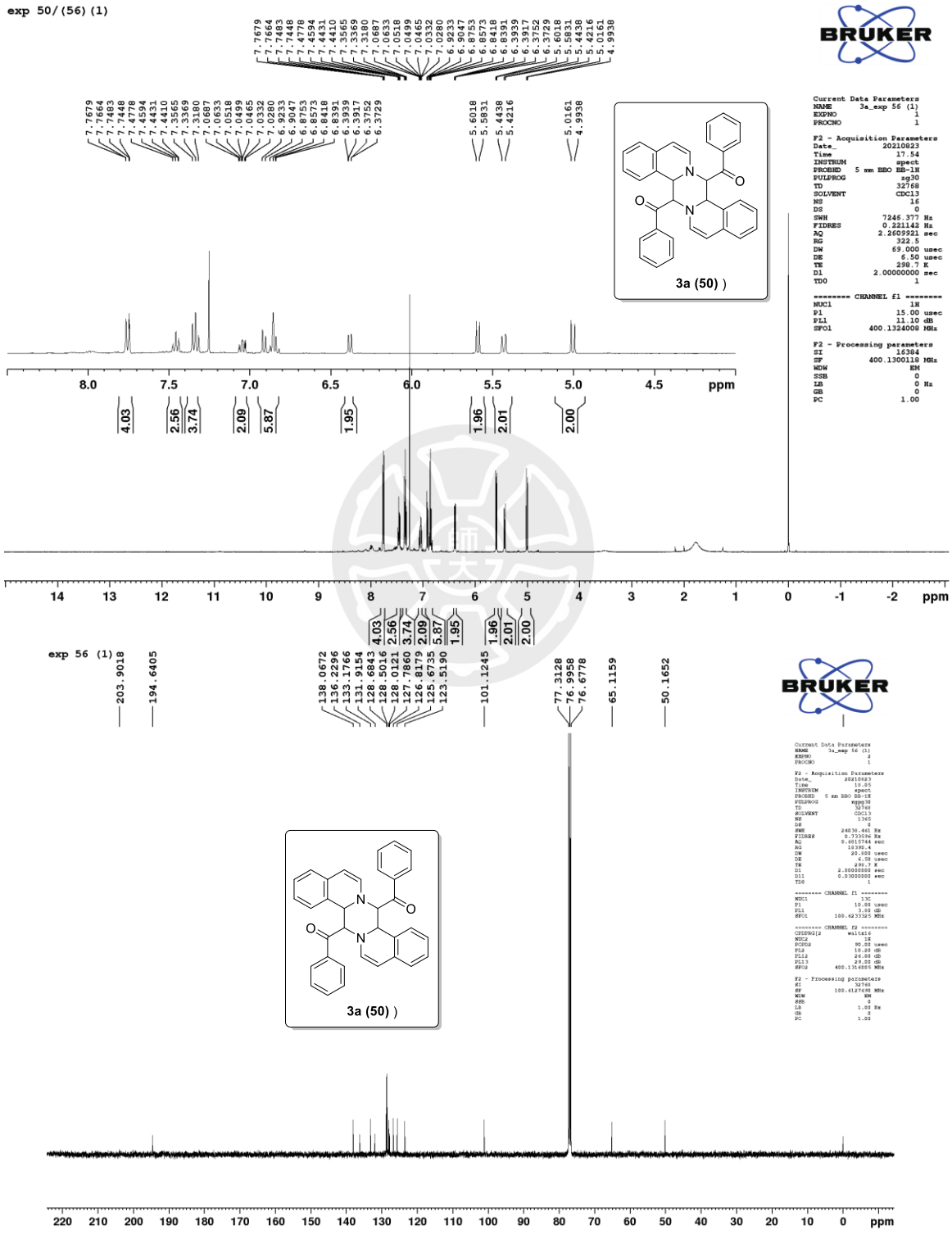

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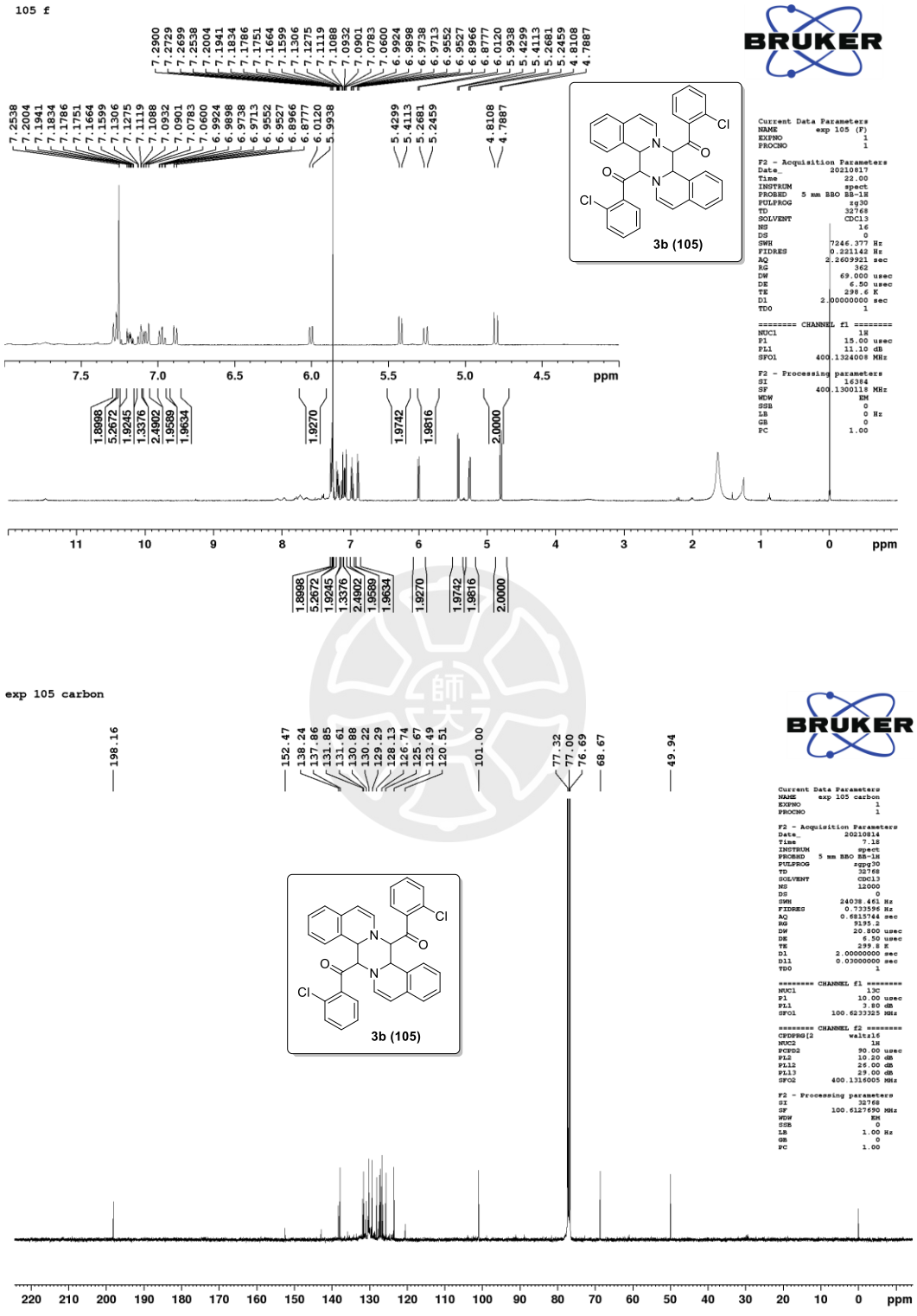
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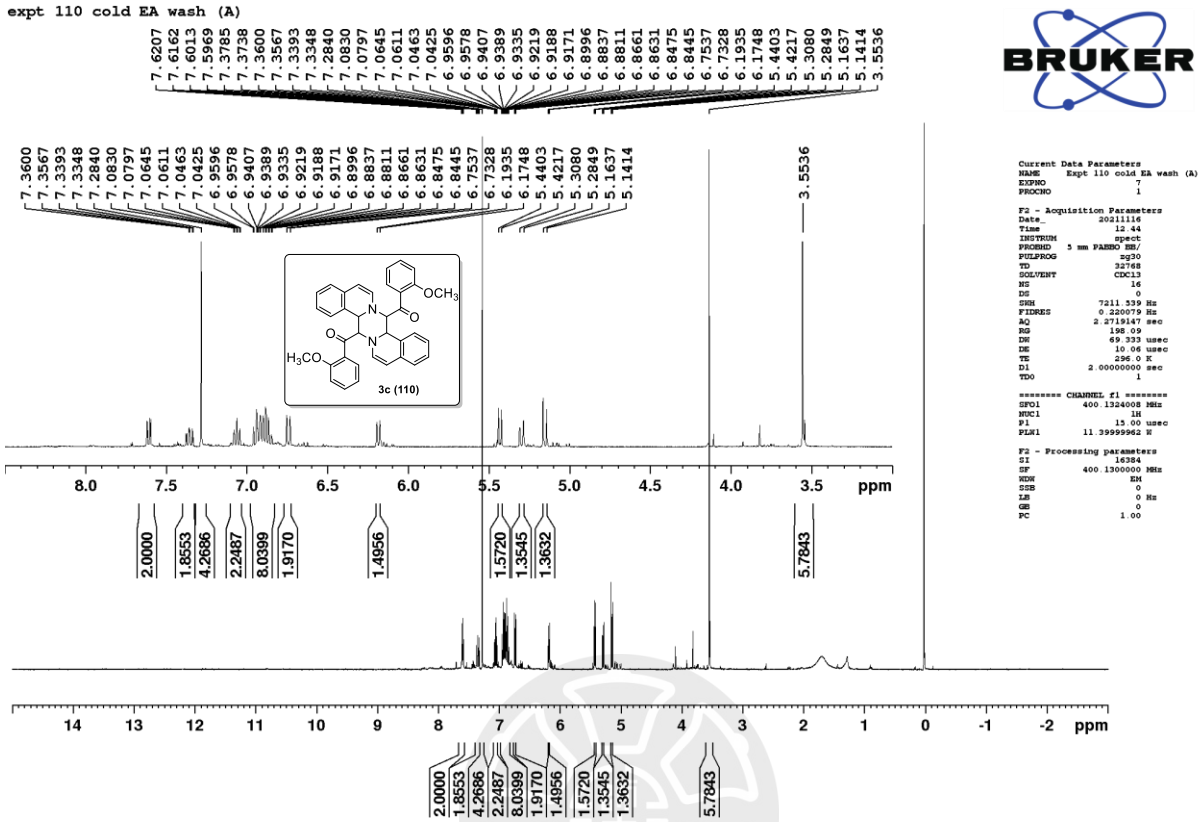
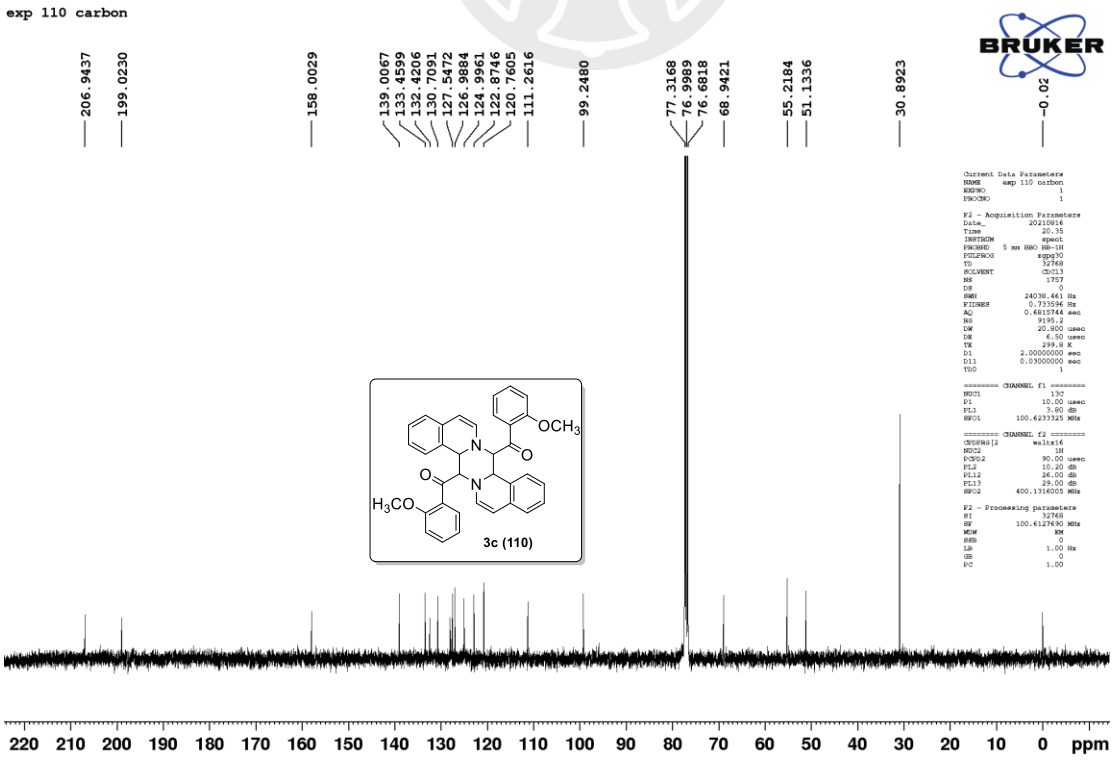
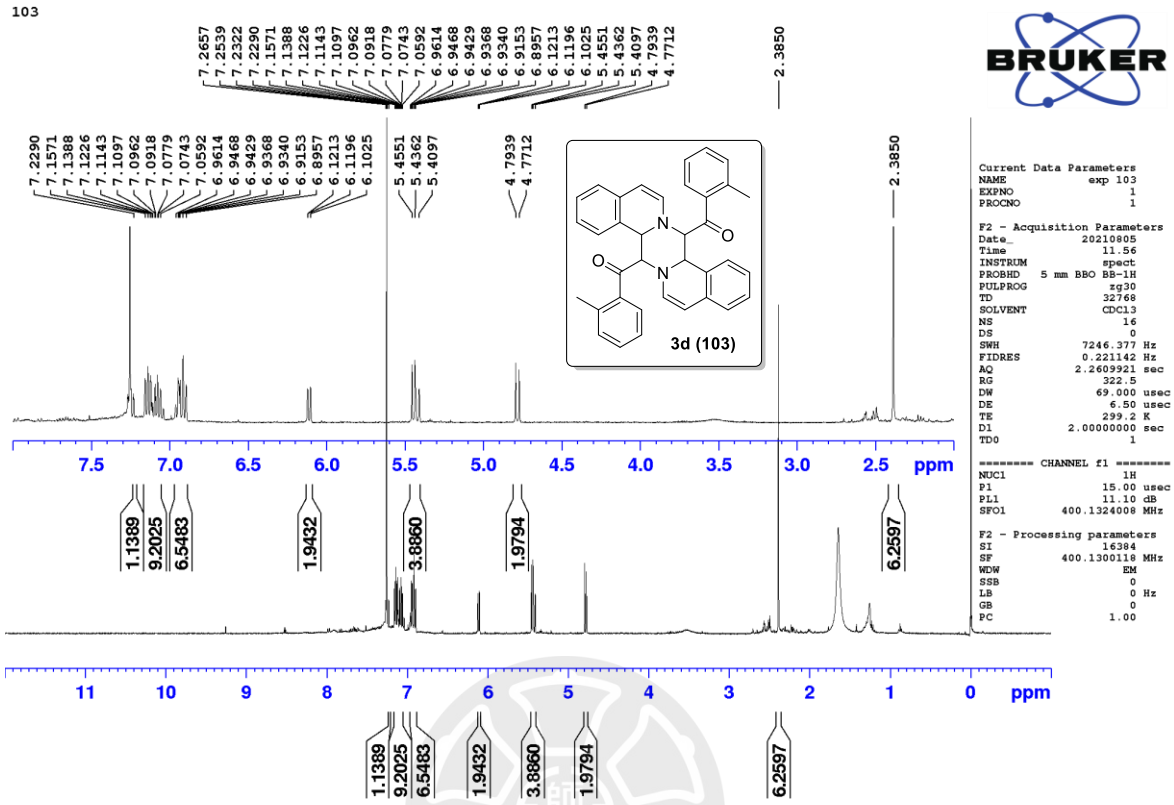
LB 1.00 Hz
GB 0
PC 1.00
ppm

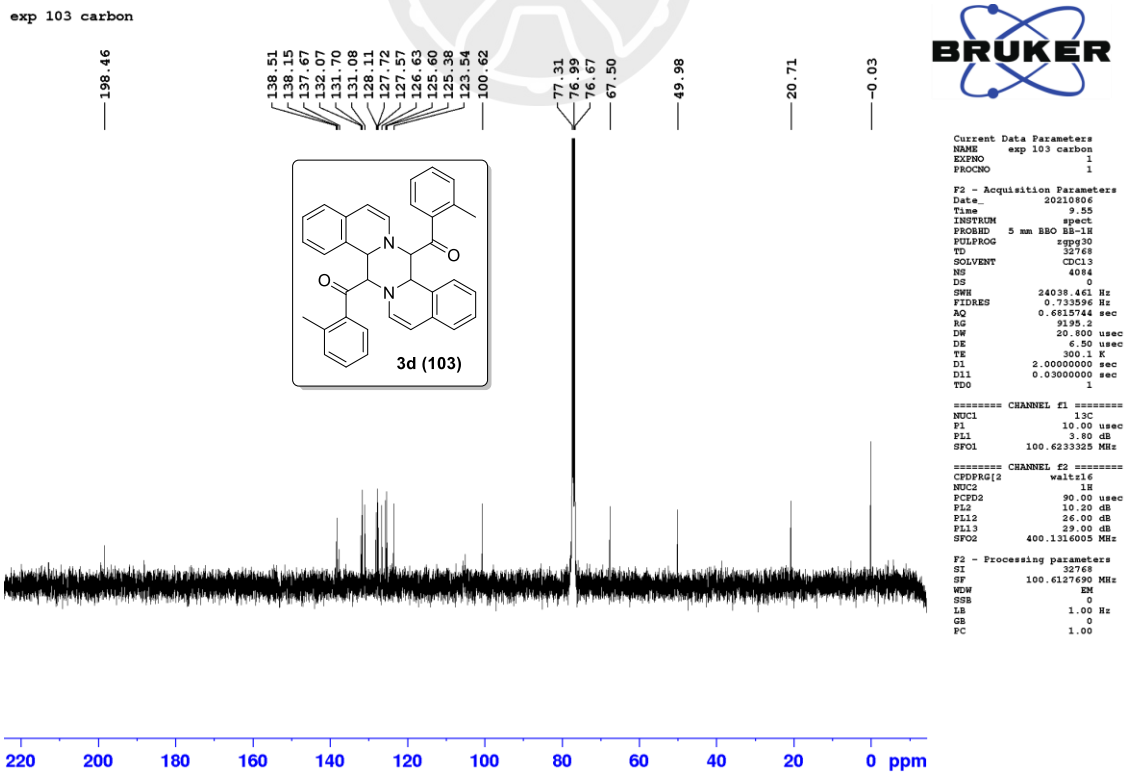
3C (110)



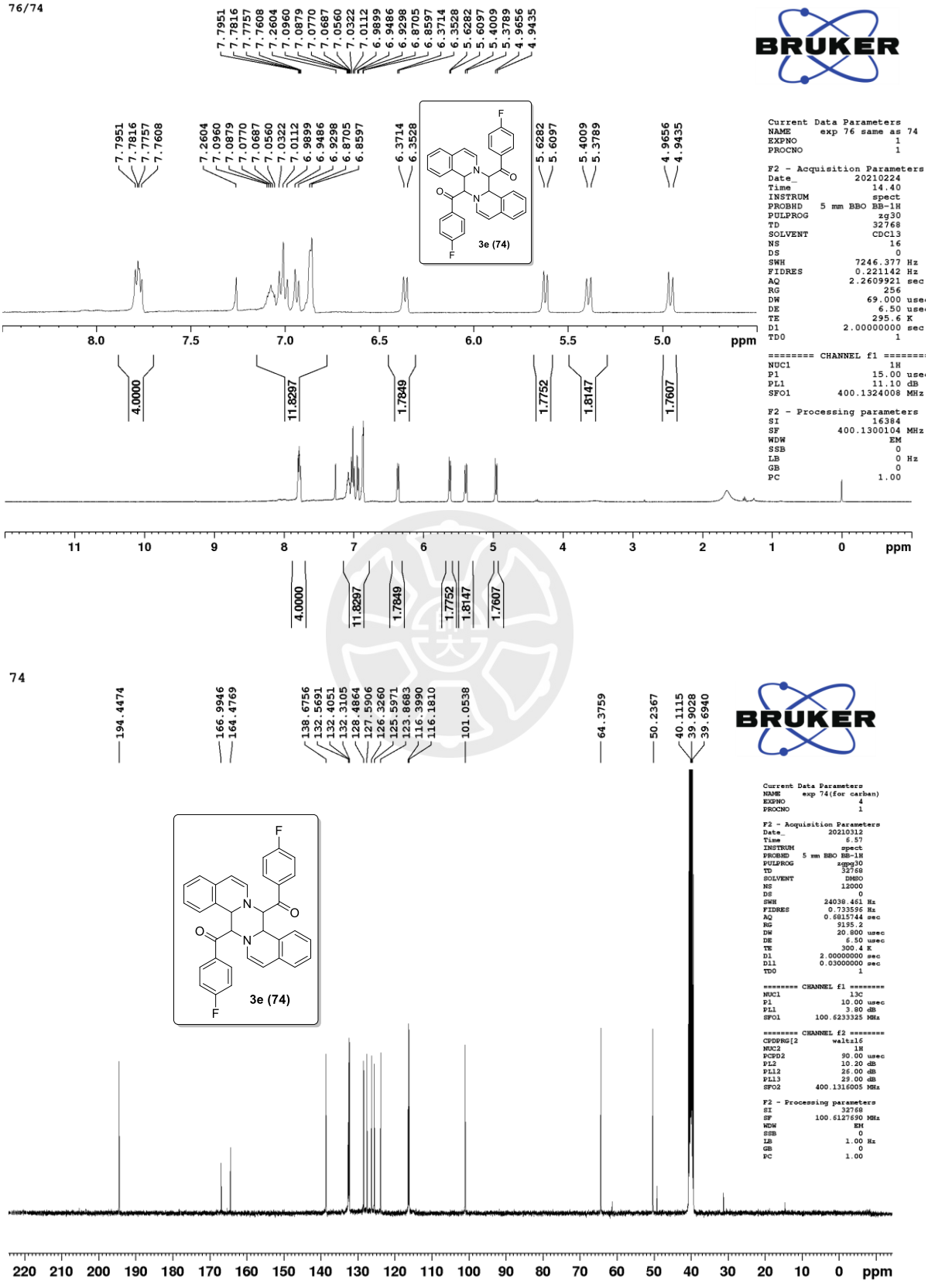


3d (103)

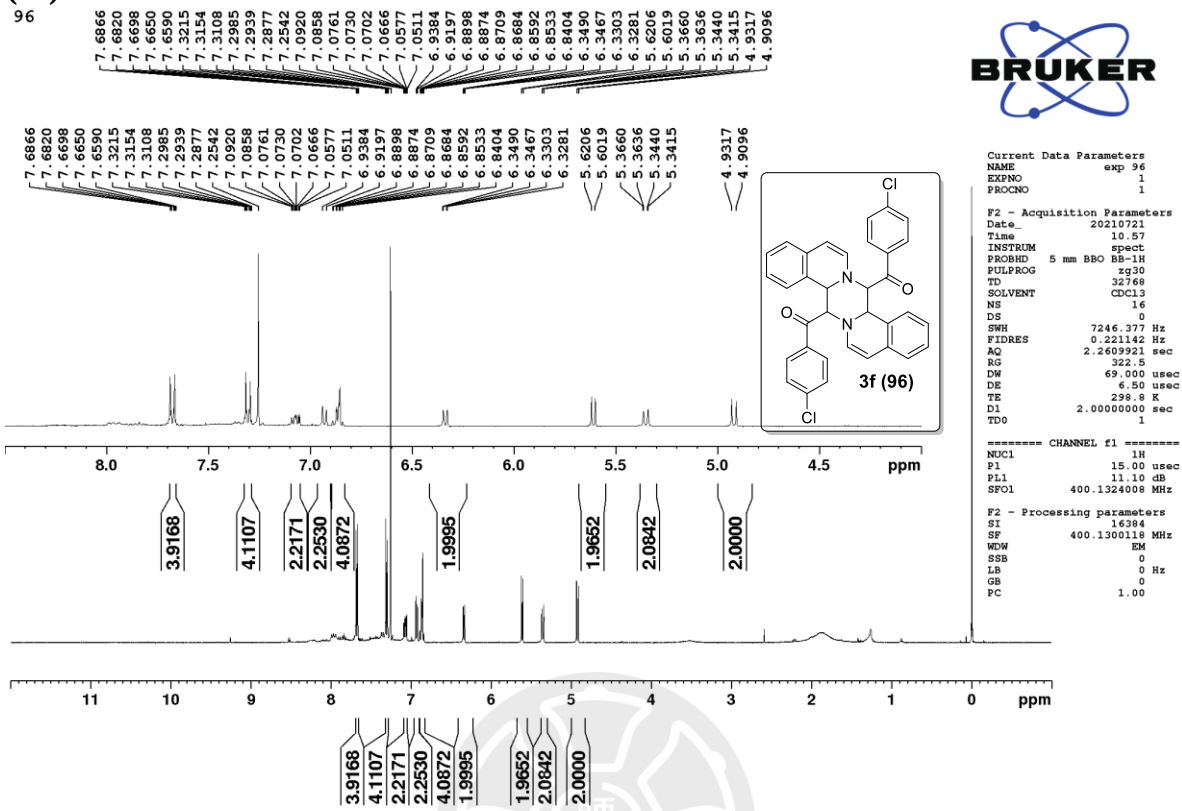

103
7.2657
7.2539
7.2322
7.2290
7.1571
7.1388
7.1226
7.1143
7.1097
7.0962
7.0918
7.0779
7.0743
7.0592
6.9614
6.9468
6.9429
6.9368
6.9340
6.9153
6.8957
6.1213
6.1196
6.1025
5.4551
5.4362
5.4097
4.7939
4.7712
2.3850
3d (103)
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
ppm
1.1389
9.2025
6.5483
1.9432
3.8860
1.9794
6.2597
11
10
9
8
7
6
5
4
3
2
1
0
ppm
BRUKER
Current Data Parameters
NAME exp 103
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20210805
Time 11.56
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 0
SWH 7246.377 Hz
FIDRES 0.221142 Hz
AQ 2.2609921 sec
RG 322.5
DW 69.000 usec
DE 6.50 usec
TE 299.2 K
D1 2.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 15.00 usec
PL1 11.10 dB
SFO1 400.1324008 MHz
F2 - Processing parameters
SI 16384
SF 400.1300118 MHz
WDW EM
SSB 0
LB 0 Hz
GB 0
PC 1.00


exp 103 carbon
198.46
138.51
138.15
137.67
132.07
131.70
131.08
128.11
127.72
127.57
126.63
125.60
125.38
123.54
100.62
77.31
76.99
76.67
67.50
49.98
20.71
-0.03
3d (103)
220
200
180
160
140
120
100
80
60
40
20
0
ppm
BRUKER
Current Data Parameters
NAME exp 103 carbon
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20210806
Time 9.55
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 4084
DS 0
SWH 24038.461 Hz
FIDRES 0.733596 Hz
AQ 0.6815744 sec
RG 9195.2
DW 20.800 usec
DE 6.50 usec
TE 300.1 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 10.00 usec
PL1 3.80 dB
SFO1 100.6233325 MHz
======== CHANNEL f2 ========
CPDPRG[2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 10.20 dB
PL12 26.00 dB
PL13 29.00 dB
SFO2 400.1316005 MHz
F2 - Processing parameters
SI 32768
SF 100.6127690 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.00

3e (74)


76/74
7.7951
7.7816
7.7757
7.7608
7.2604
7.0960
7.0879
7.0770
7.0687
7.0560
7.0322
7.0112
6.9899
6.9486
6.9298
6.8705
6.8597
6.3714
6.3528
5.6282
5.6097
5.4009
5.3789
4.9656
4.9435
3e (74)
Current Data Parameters
NAME exp 76 same as 74
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20210224
Time 14.40
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 0
SWH 7246.377 Hz
FIDRES 0.221142 Hz
AQ 2.2609921 sec
RG 256
DW 69.000 use
DE 6.50 use
TE 295.6 K
D1 2.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 15.00 use
PL1 11.10 dB
SFO1 400.1324008 MHz
F2 - Processing parameters
SI 16384
SF 400.1300104 MHz
WDW EM
SSB 0
LB 0 Hz
GB 0
PC 1.00
4.0000
11.8297
1.7849
1.7752
1.8147
1.7607
ppm
74
194.4474
166.9946
164.4769
138.6756
132.5691
132.4051
132.3105
128.4864
127.5906
126.3260
125.5971
123.8683
116.3990
116.1810
101.0538
64.3759
50.2367
40.1115
39.9028
39.6940
3e (74)
Current Data Parameters
NAME exp 74(for carban)
EXPNO 4
PROCNO 1
F2 - Acquisition Parameters
Date_ 20210312
Time 6.57
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zgpg30
TD 32768
SOLVENT DMSO
NS 12000
DS 0
SWH 24038.461 Hz
FIDRES 0.733596 Hz
AQ 0.6815744 sec
RG 9195.2
DW 20.800 usec
DE 6.50 usec
TE 300.4 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 10.00 usec
PL1 3.80 dB
SFO1 100.6233325 MHz
======== CHANNEL f2 ========
CPDPRG[2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 10.20 dB
PL12 26.00 dB
PL13 29.00 dB
SFO2 400.1316005 MHz
F2 - Processing parameters
SI 32768
SF 100.6127690 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.00
ppm

3f (96)

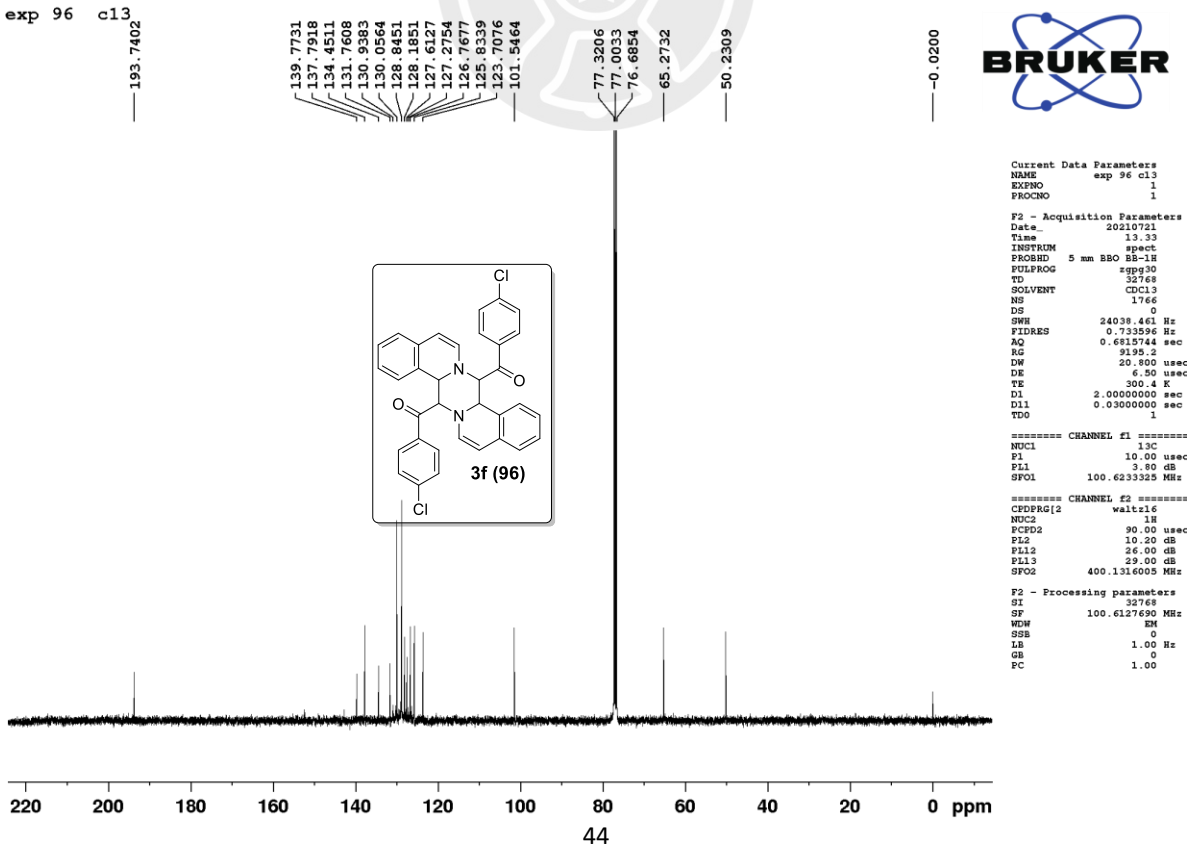



```
Current Data Parameters
NAME                  exp 96
EXPNO                      1
PROCNO                     1

F2 - Acquisition Parameters
Date_              20210721
Time                  10.57
INSTRUM               spect
PROBHD    5 mm BBO BB-1H
PULPROG                zg30
TD                    32768
SOLVENT               CDCl3
NS                       16
DS                        0
SWH                7246.377 Hz
FIDRES             0.221142 Hz
AQ                 2.2609921 sec
RG                     322.5
DW                    69.000 usec
DE                     6.50 usec
TE                    298.8 K
D1                 2.00000000 sec
TD0                        1

======== CHANNEL f1 ========
NUC1                      1H
P1                     15.00 usec
PL1                    11.10 dB
SFO1              400.1324008 MHz

F2 - Processing parameters
SI                        16384
SF                400.1300118 MHz
WDW                        EM
SSB                         0
LB                          0 Hz
GB                          0
PC                       1.00
```




```
Current Data Parameters
NAME              exp 96 c13
EXPNO                      1
PROCNO                     1

F2 - Acquisition Parameters
Date_              20210721
Time                  13.33
INSTRUM               spect
PROBHD    5 mm BBO BB-1H
PULPROG             zgpg30
TD                    32768
SOLVENT               CDCl3
NS                     1766
DS                        0
SWH               24038.461 Hz
FIDRES             0.733596 Hz
AQ                 0.6815744 sec
RG                    9195.2
DW                    20.800 usec
DE                     6.50 usec
TE                    300.4 K
D1                 2.00000000 sec
D11                0.03000000 sec
TD0                        1

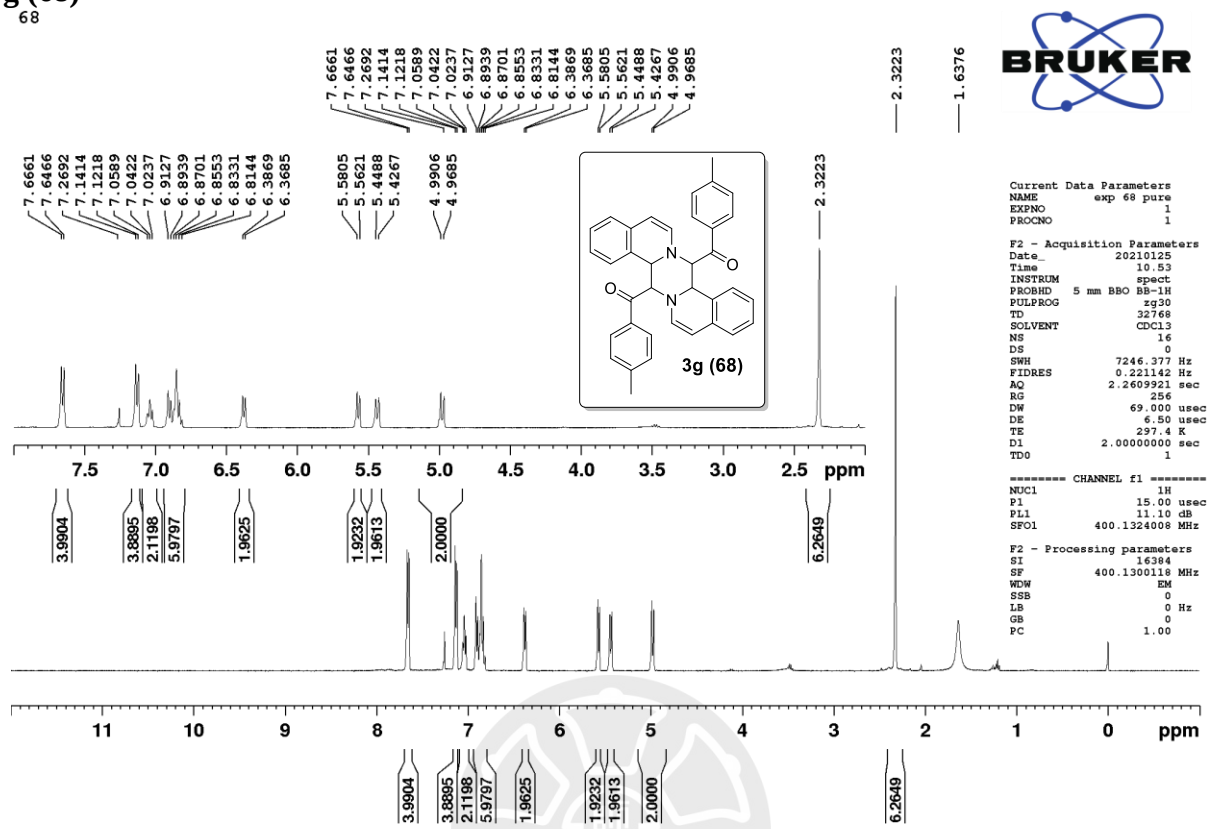
======== CHANNEL f1 ========
NUC1                      13C
P1                     10.00 usec
PL1                     3.80 dB
SFO1              100.6233325 MHz

======== CHANNEL f2 ========
CPDPRG[2          waltz16
NUC2                      1H
PCPD2                  90.00 usec
PL2                    10.20 dB
PL12                   26.00 dB
PL13                   29.00 dB
SFO2              400.1316005 MHz

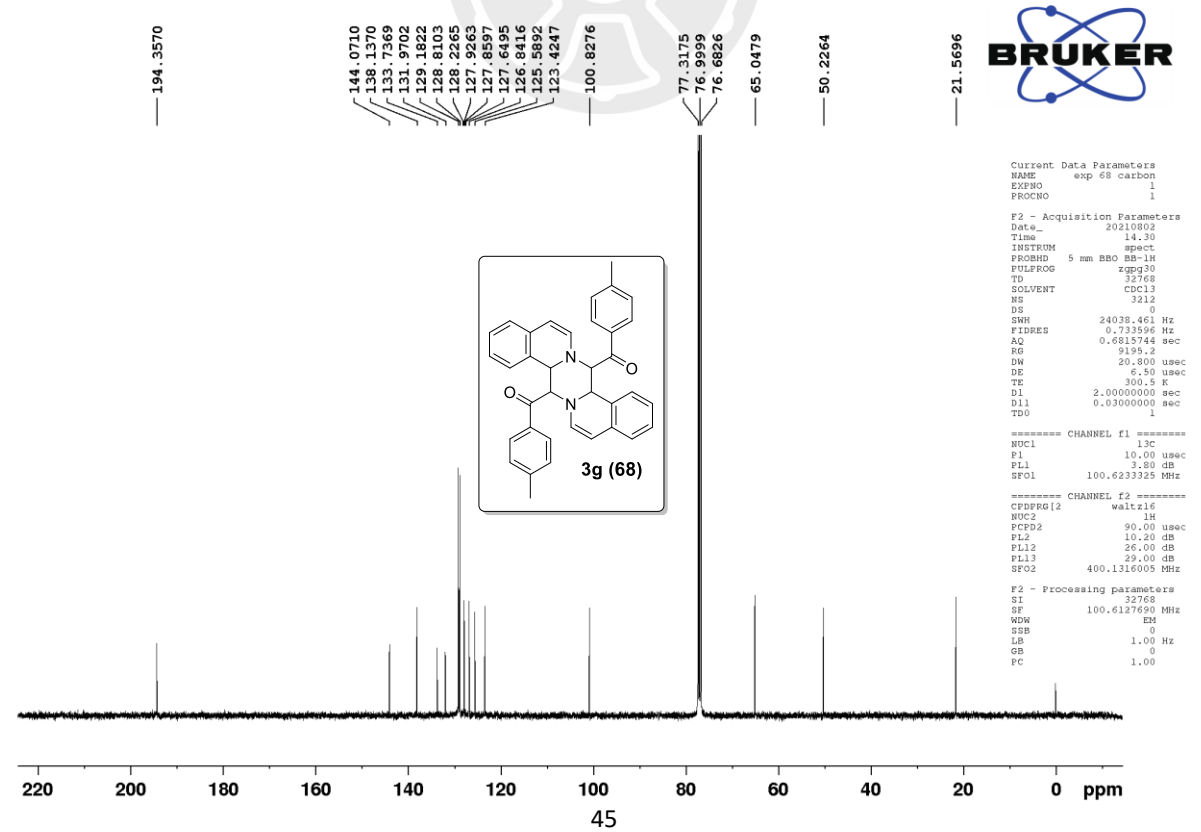
F2 - Processing parameters
SI                        32768
SF                100.6127690 MHz
WDW                        EM
SSB                         0
LB                       1.00 Hz
GB                          0
PC                       1.00
```

3g (68)

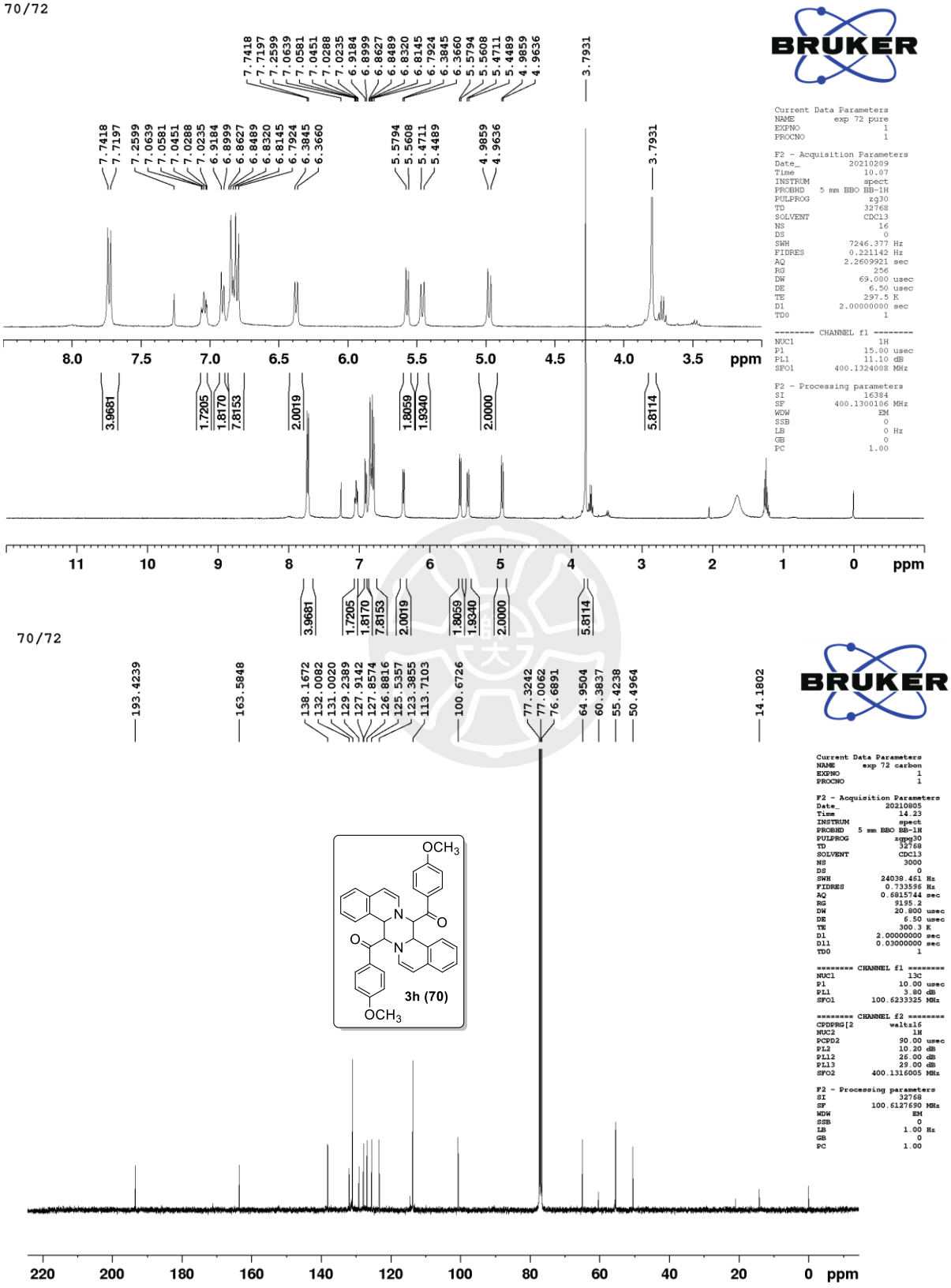
68


7.6661
7.6466
7.2692
7.1414
7.1218
7.0589
7.0422
7.0237
6.9127
6.8939
6.8701
6.8553
6.8331
6.8144
6.3869
6.3685
5.5805
5.5621
5.4488
5.4267
4.9906
4.9685
2.3223
1.6376
3g (68)
Current Data Parameters
NAME exp 68 pure
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20210125
Time 10.53
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 0
SWH 7246.377 Hz
FIDRES 0.221142 Hz
AQ 2.2609921 sec
RG 256
DW 69.000 usec
DE 6.50 usec
TE 297.4 K
D1 2.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 15.00 usec
PL1 11.10 dB
SFO1 400.1324008 MHz
F2 - Processing parameters
SI 16384
SF 400.1300118 MHz
WDW EM
SSB 0
LB 0 Hz
GB 0
PC 1.00
ppm
3.9904
3.8895
2.1198
5.9797
1.9625
1.9232
1.9613
2.0000
6.2649

68


194.3570
144.0710
138.1370
133.7369
131.9702
129.1822
128.8103
128.2265
127.9263
127.8597
127.6495
126.8416
125.5892
123.4247
100.8276
77.3175
76.9999
76.6826
65.0479
50.2264
21.5696
3g (68)
Current Data Parameters
NAME exp 68 carbon
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20210802
Time 14.30
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 3212
DS 0
SWH 24038.461 Hz
FIDRES 0.733596 Hz
AQ 0.6815744 sec
RG 9195.2
DW 20.800 usec
DE 6.50 usec
TE 300.5 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 10.00 usec
PL1 3.80 dB
SFO1 100.6233325 MHz
======== CHANNEL f2 ========
CPDPRG[2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 10.20 dB
PL12 26.00 dB
PL13 29.00 dB
SFO2 400.1316005 MHz
F2 - Processing parameters
SI 32768
SF 100.6127690 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.00
ppm

3h (70)



Current Data Parameters
NAME exp 72 pure
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20210209
Time 10.07
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 0
SWH 7246.377 Hz
FIDRES 0.221142 Hz
AQ 2.2609921 sec
RG 256
DW 69.000 usec
DE 6.50 usec
TE 297.5 K
D1 2.00000000 sec
TD0 1

-------- CHANNEL f1 --------
NUC1 1H
P1 15.00 usec
PL1 11.10 dB
SFO1 400.1324008 MHz

F2 - Processing parameters
SI 16384
SF 400.1300106 MHz
WDW EM
SSB 0
LB 0 Hz
GB 0
PC 1.00

Current Data Parameters
NAME exp 72 carbon
EXPNO 1
PROCNO 1

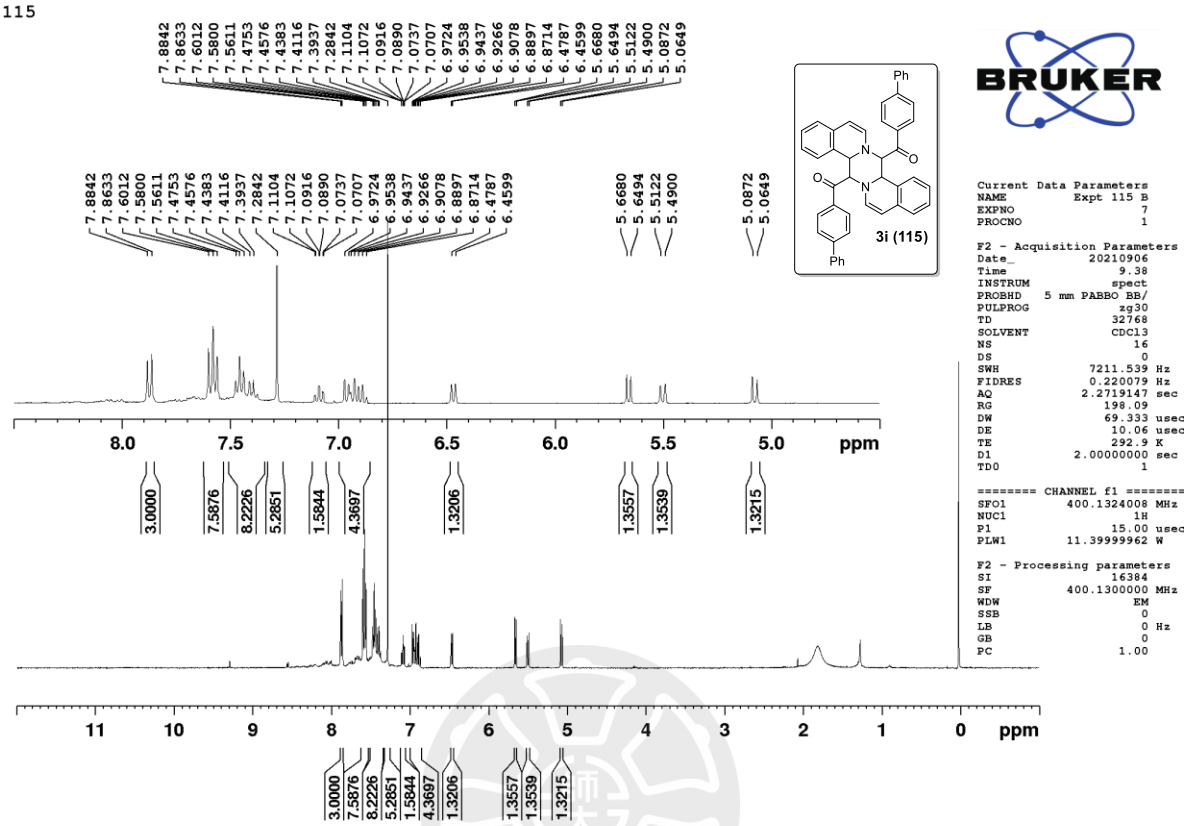
F2 - Acquisition Parameters
Date_ 20210805
Time 14.23
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 3000
DS 0
SWH 24038.461 Hz
FIDRES 0.733596 Hz
AQ 0.6815744 sec
RG 9195.2
DW 20.800 usec
DE 6.50 usec
TE 300.3 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

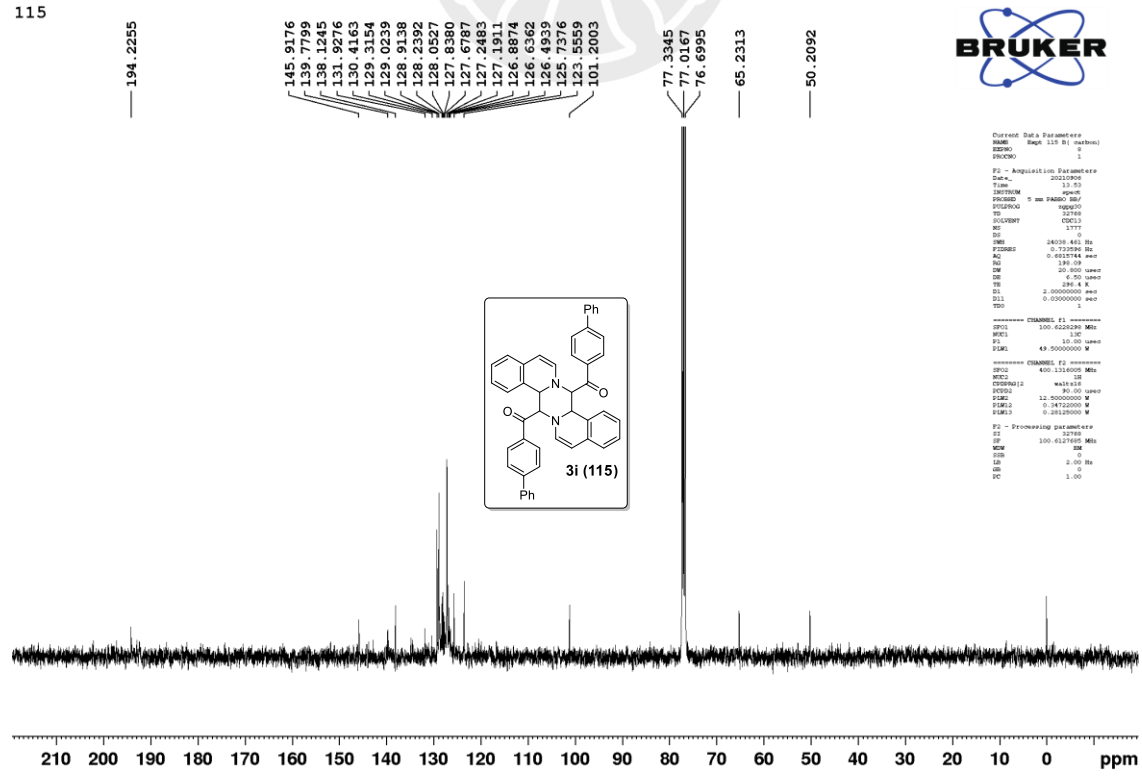
======== CHANNEL f1 ========
NUC1 13C
P1 10.00 usec
PL1 3.80 dB
SFO1 100.6233325 MHz

======== CHANNEL f2 ========
CPDPRG[2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 10.20 dB
PL12 26.00 dB
PL13 29.00 dB
SFO2 400.1316005 MHz

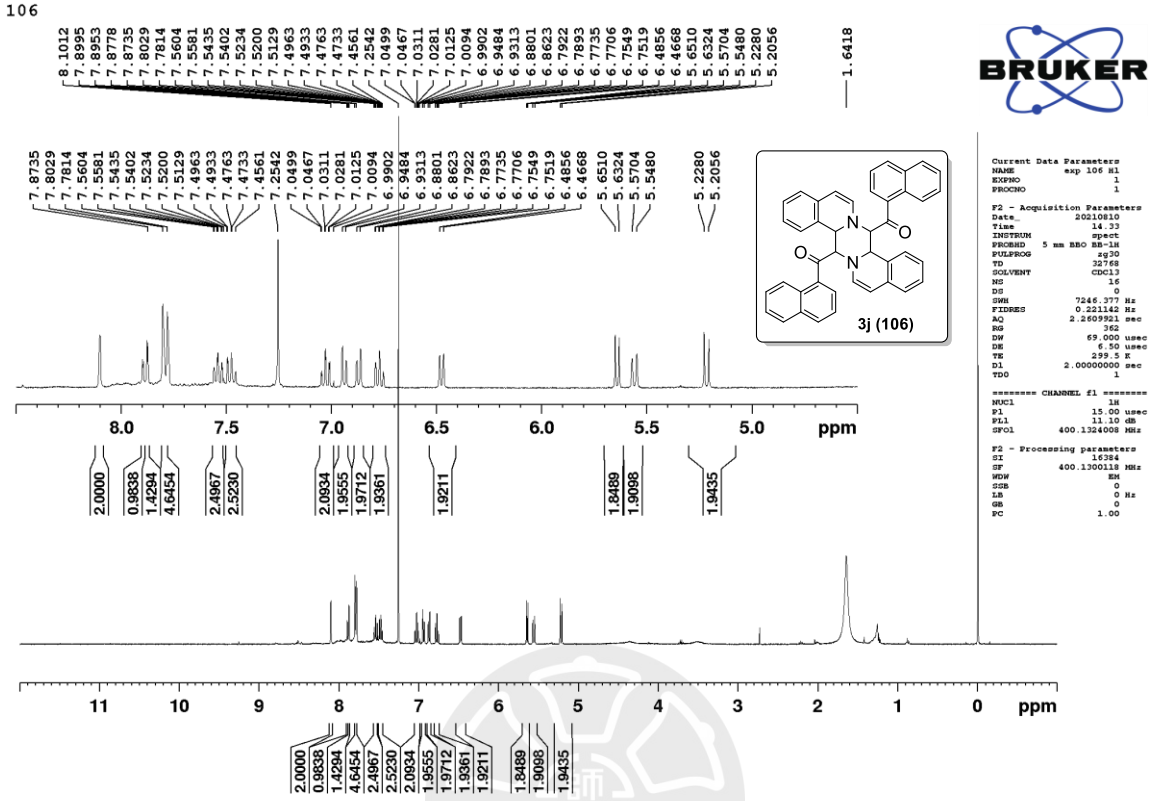
F2 - Processing parameters
SI 32768
SF 100.6127690 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.00

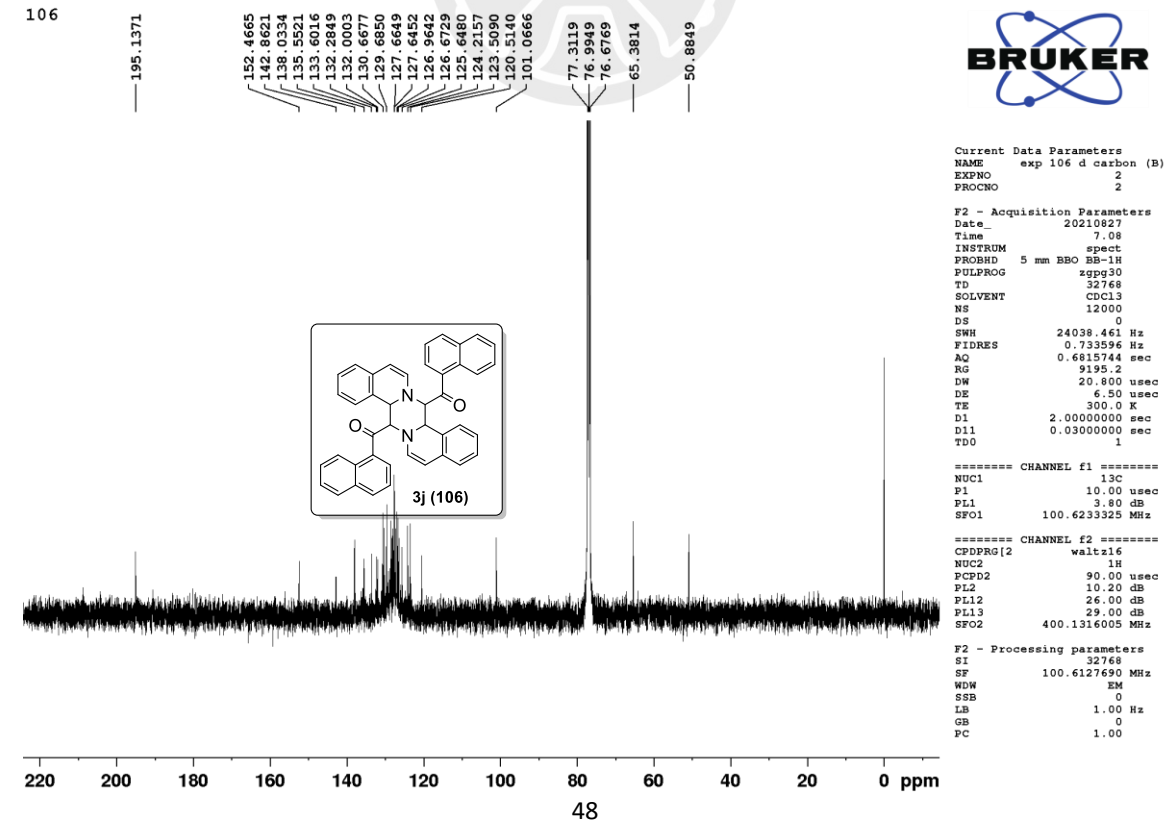
3i (115)


115
7.8842
7.8633
7.6012
7.5800
7.5611
7.4753
7.4576
7.4383
7.4116
7.3937
7.2842
7.1104
7.1072
7.0916
7.0890
7.0737
7.0707
6.9724
6.9538
6.9437
6.9266
6.9078
6.8897
6.8714
6.4787
6.4599
5.6680
5.6494
5.5122
5.4900
5.0872
5.0649
3i (115)
Current Data Parameters
NAME Expt 115 B
EXPNO 7
PROCNO 1
F2 - Acquisition Parameters
Date_ 20210906
Time 9.38
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 0
SWH 7211.539 Hz
FIDRES 0.220079 Hz
AQ 2.2719147 sec
RG 198.09
DW 69.333 usec
DE 10.06 usec
TE 292.9 K
D1 2.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 400.1324008 MHz
NUC1 1H
P1 15.00 usec
PLW1 11.39999962 W
F2 - Processing parameters
SI 16384
SF 400.1300000 MHz
WDW EM
SSB 0
LB 0 Hz
GB 0
PC 1.00
ppm
3.0000
7.5876
8.2226
5.2851
1.5844
4.3697
1.3206
1.3557
1.3539
1.3215

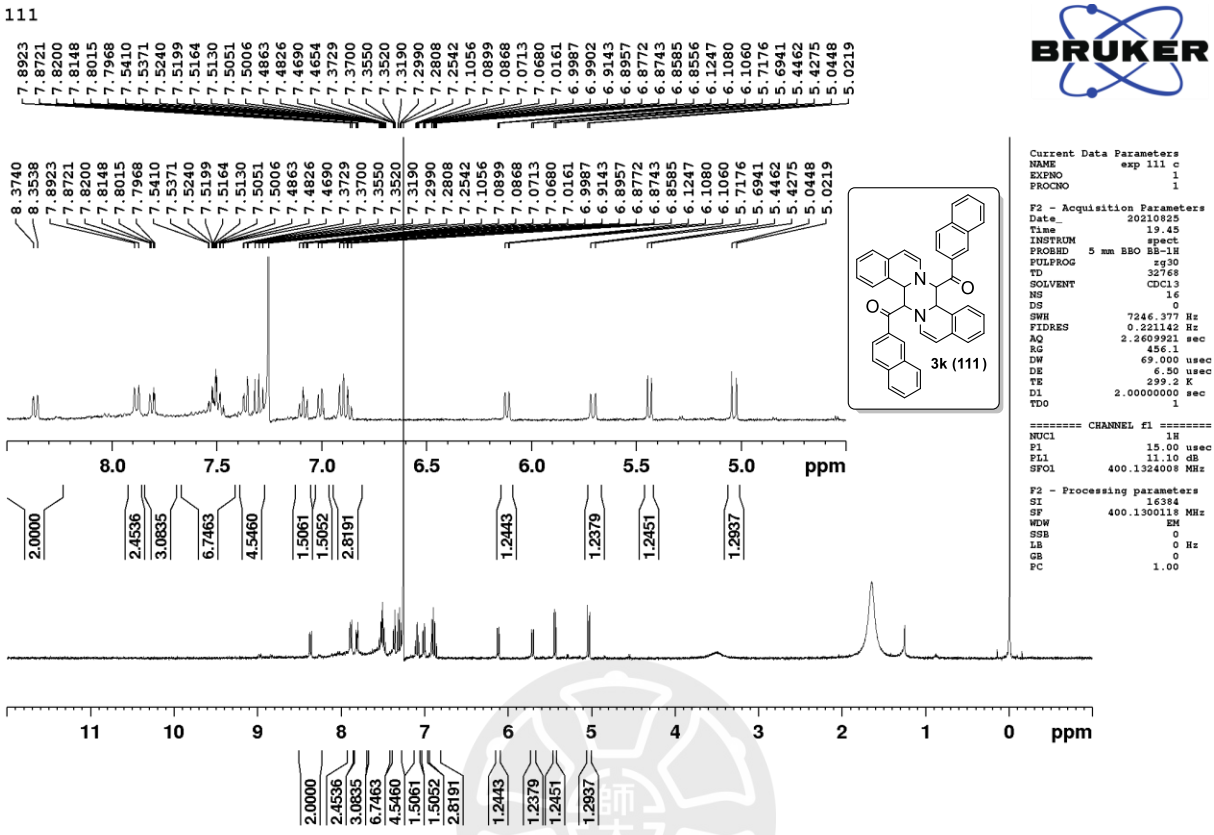

115
194.2255
145.9176
139.7799
138.1245
131.9276
130.4163
129.3154
129.0239
128.9138
128.2392
128.0527
127.8380
127.6787
127.2483
127.1911
126.8874
126.6362
126.4939
125.7376
123.5559
101.2003
77.3345
77.0167
76.6995
65.2313
50.2092
3i (115)
ppm

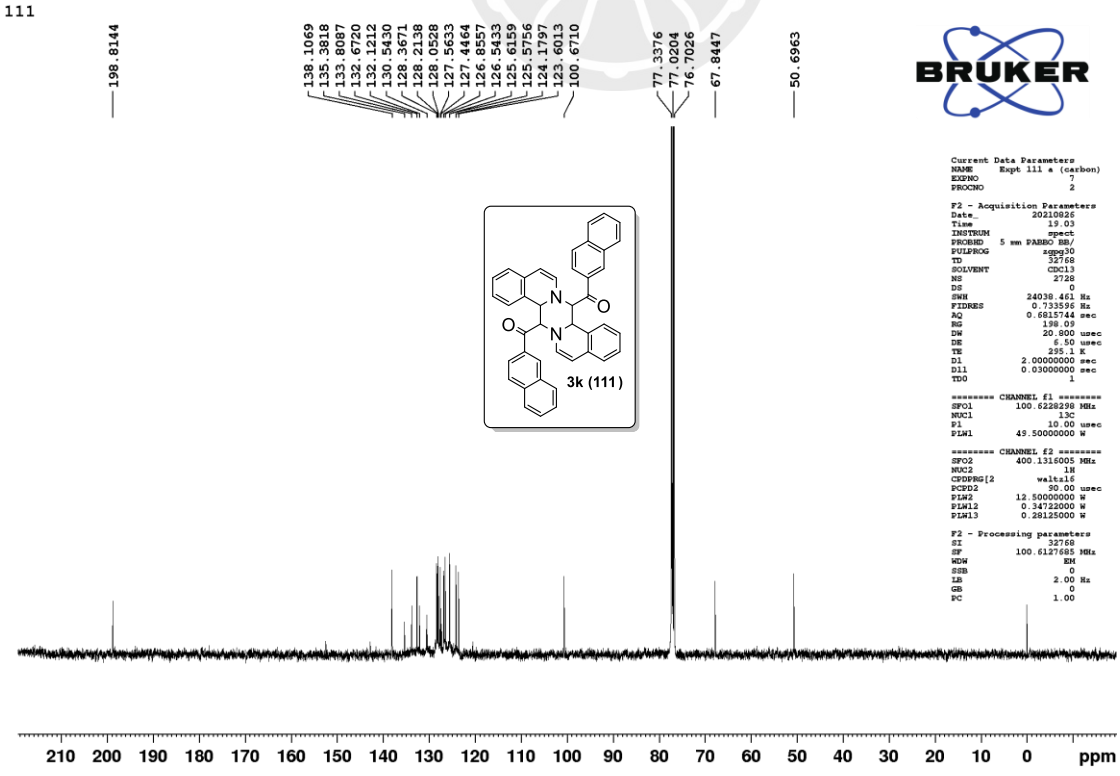
3j (106)


106
3j (106)
Current Data Parameters
NAME exp 106 H1
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20210810
Time 14.33
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 0
SWH 7246.377 Hz
FIDRES 0.221142 Hz
AQ 2.2609921 sec
RG 362
DW 69.000 usec
DE 6.50 usec
TE 299.5 K
D1 2.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 15.00 usec
PL1 11.10 dB
SFO1 400.1324008 MHz
F2 - Processing parameters
SI 16384
SF 400.1300118 MHz
WDW EM
SSB 0
LB 0 Hz
GB 0
PC 1.00

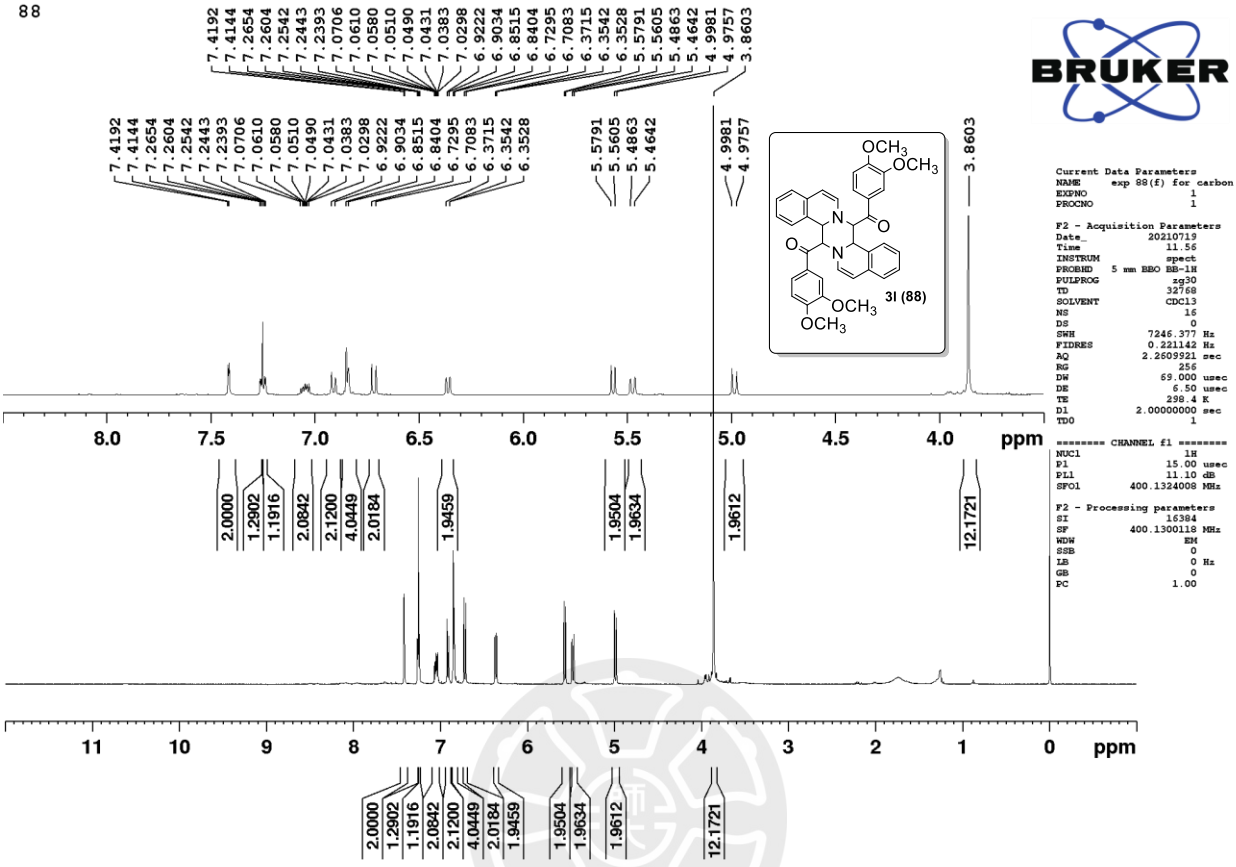

106
3j (106)
Current Data Parameters
NAME exp 106 d carbon (B)
EXPNO 2
PROCNO 2
F2 - Acquisition Parameters
Date_ 20210827
Time 7.08
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 12000
DS 0
SWH 24038.461 Hz
FIDRES 0.733596 Hz
AQ 0.6815744 sec
RG 9195.2
DW 20.800 usec
DE 6.50 usec
TE 300.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 10.00 usec
PL1 3.80 dB
SFO1 100.6233325 MHz
======== CHANNEL f2 ========
CPDPRG[2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 10.20 dB
PL12 26.00 dB
PL13 29.00 dB
SFO2 400.1316005 MHz
F2 - Processing parameters
SI 32768
SF 100.6127690 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.00

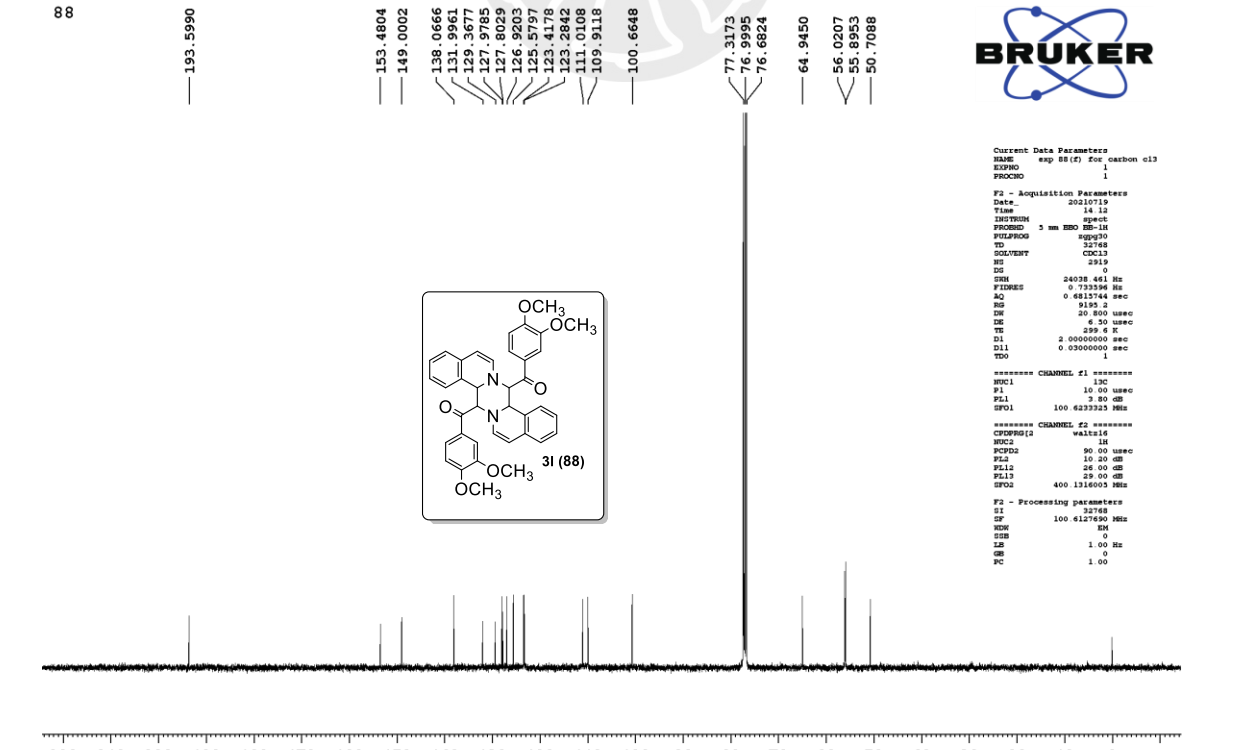
3k (111)



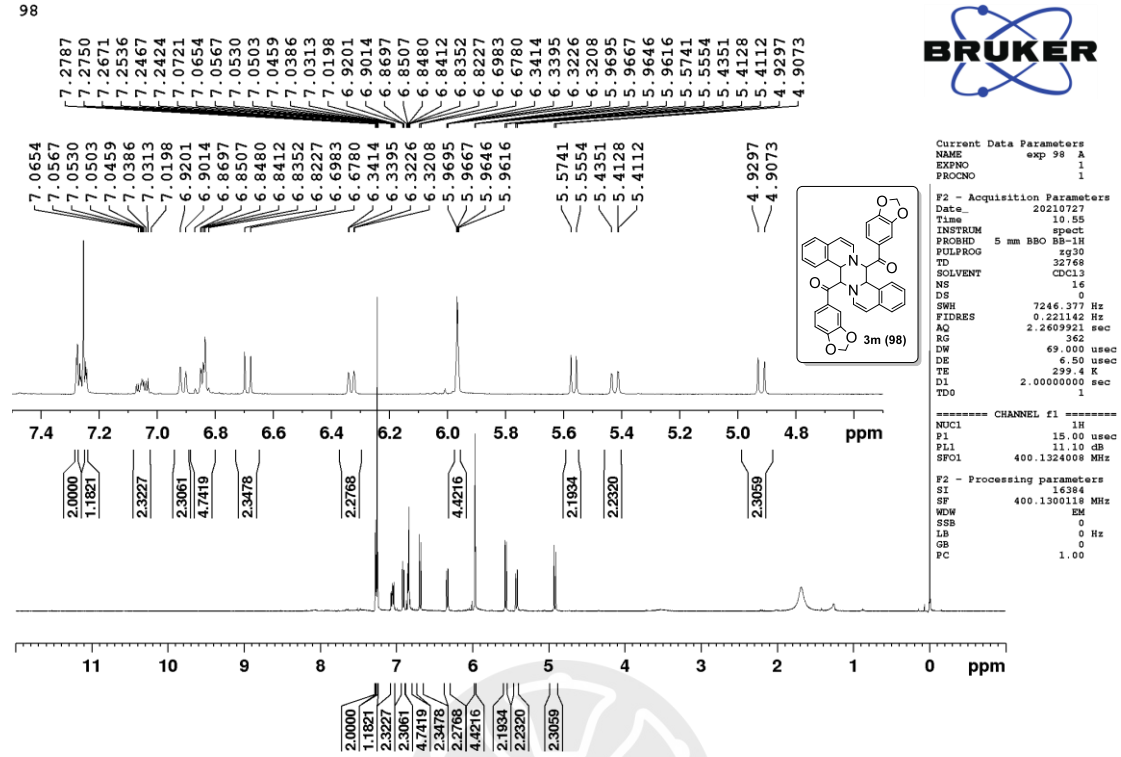


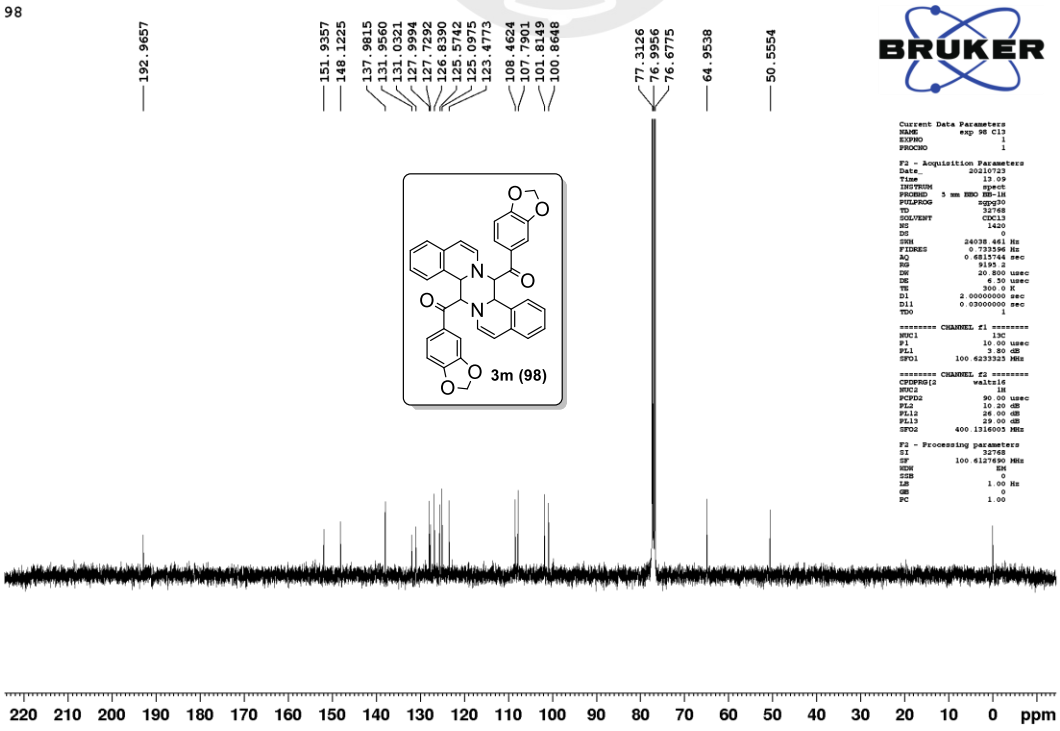
3l (88)


88
7.4192
7.4144
7.2654
7.2604
7.2542
7.2443
7.2393
7.0706
7.0610
7.0580
7.0510
7.0490
7.0431
7.0383
7.0298
6.9222
6.9034
6.8515
6.8404
6.7295
6.7083
6.3715
6.3542
6.3528
5.5791
5.5605
5.4863
5.4642
4.9981
4.9757
3.8603
3l (88)
2.0000
1.2902
1.1916
2.0842
2.1200
4.0449
2.0184
1.9459
1.9504
1.9634
1.9612
12.1721
ppm

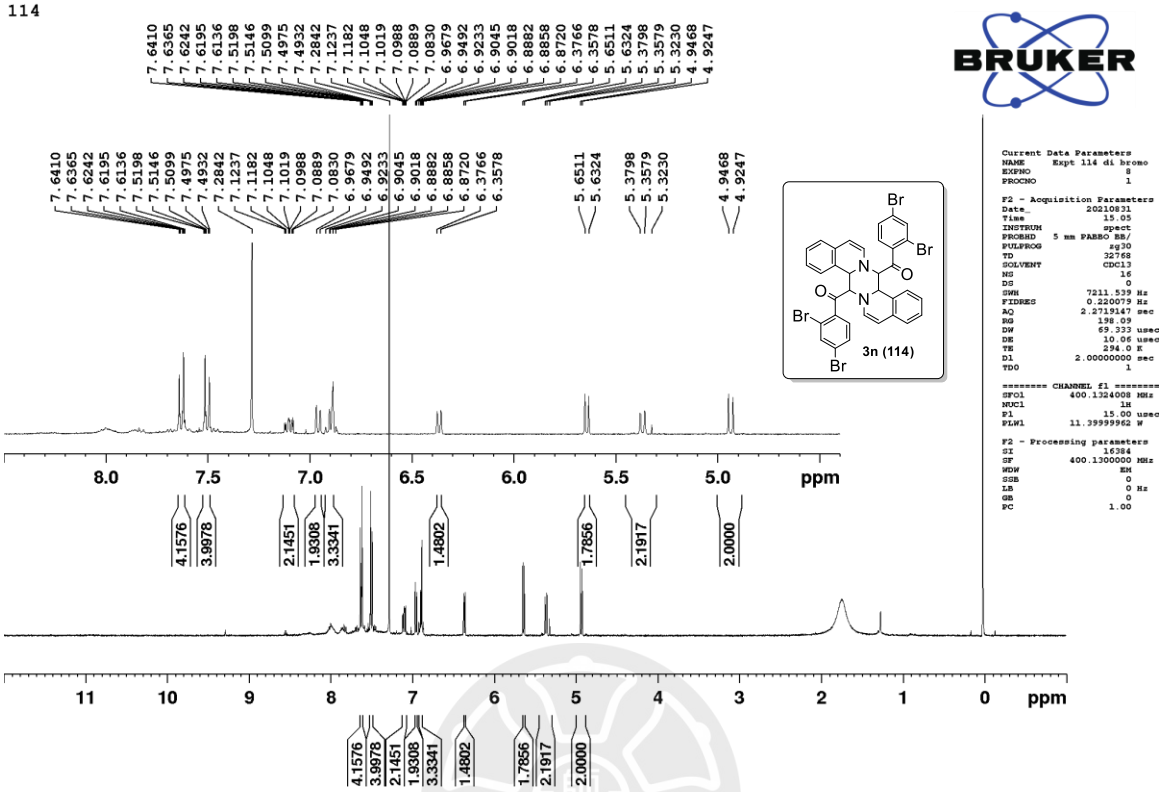

88
193.5990
153.4804
149.0002
138.0666
131.9961
129.3677
127.9785
127.8029
126.9203
125.5797
123.4178
123.2842
111.0108
109.9118
100.6648
77.3173
76.9995
76.6824
64.9450
56.0207
55.8953
50.7088
3l (88)
ppm

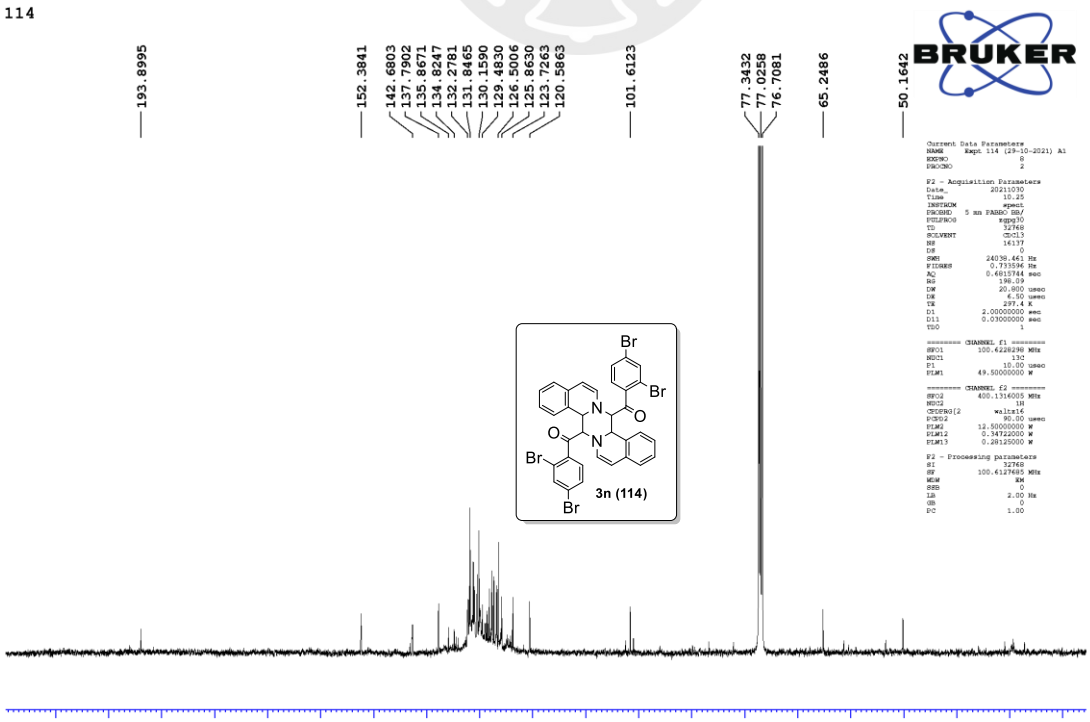
3m (98)


98
7.2787
7.2750
7.2671
7.2536
7.2467
7.2424
7.0721
7.0654
7.0567
7.0530
7.0503
7.0459
7.0386
7.0313
7.0198
6.9201
6.9014
6.8697
6.8507
6.8480
6.8412
6.8352
6.8227
6.6983
6.6780
6.3414
6.3395
6.3226
6.3208
5.9695
5.9667
5.9646
5.9616
5.5741
5.5554
5.4351
5.4128
5.4112
4.9297
4.9073
3m (98)
Current Data Parameters
NAME exp 98 A
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20210727
Time 10.55
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 0
SWH 7246.377 Hz
FIDRES 0.221142 Hz
AQ 2.2609921 sec
RG 362
DW 69.000 usec
DE 6.50 usec
TE 299.4 K
D1 2.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 15.00 usec
PL1 11.10 dB
SFO1 400.1324008 MHz
F2 - Processing parameters
SI 16384
SF 400.1300118 MHz
WDW EM
SSB 0
LB 0 Hz
GB 0
PC 1.00
ppm
2.0000
1.1821
2.3227
2.3061
4.7419
2.3478
2.2768
4.4216
2.1934
2.2320
2.3059

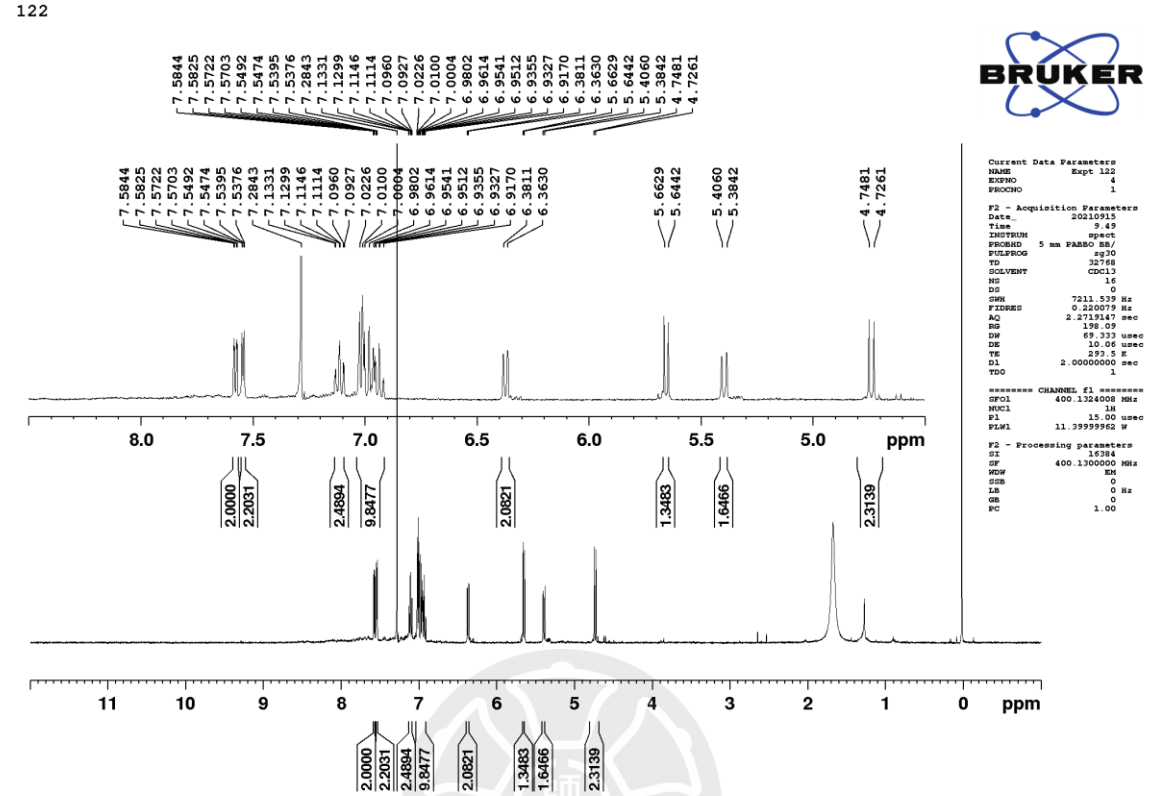

98
192.9657
151.9357
148.1225
137.9815
131.9560
131.0321
127.9994
127.7292
126.8390
125.5742
125.0975
123.4773
108.4624
107.7901
101.8149
100.8648
77.3126
76.9956
76.6775
64.9538
50.5554
3m (98)
Current Data Parameters
NAME exp 98 C13
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20210723
Time 13.09
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 1420
DS 0
SWH 24038.461 Hz
FIDRES 0.733596 Hz
AQ 0.6815744 sec
RG 9195.2
DW 20.800 usec
DE 6.50 usec
TE 300.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 10.00 usec
PL1 3.80 dB
SFO1 100.6233325 MHz
======== CHANNEL f2 ========
CPDPRG[2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 10.20 dB
PL12 26.00 dB
PL13 29.00 dB
SFO2 400.1316005 MHz
F2 - Processing parameters
SI 32768
SF 100.6127690 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.00
ppm

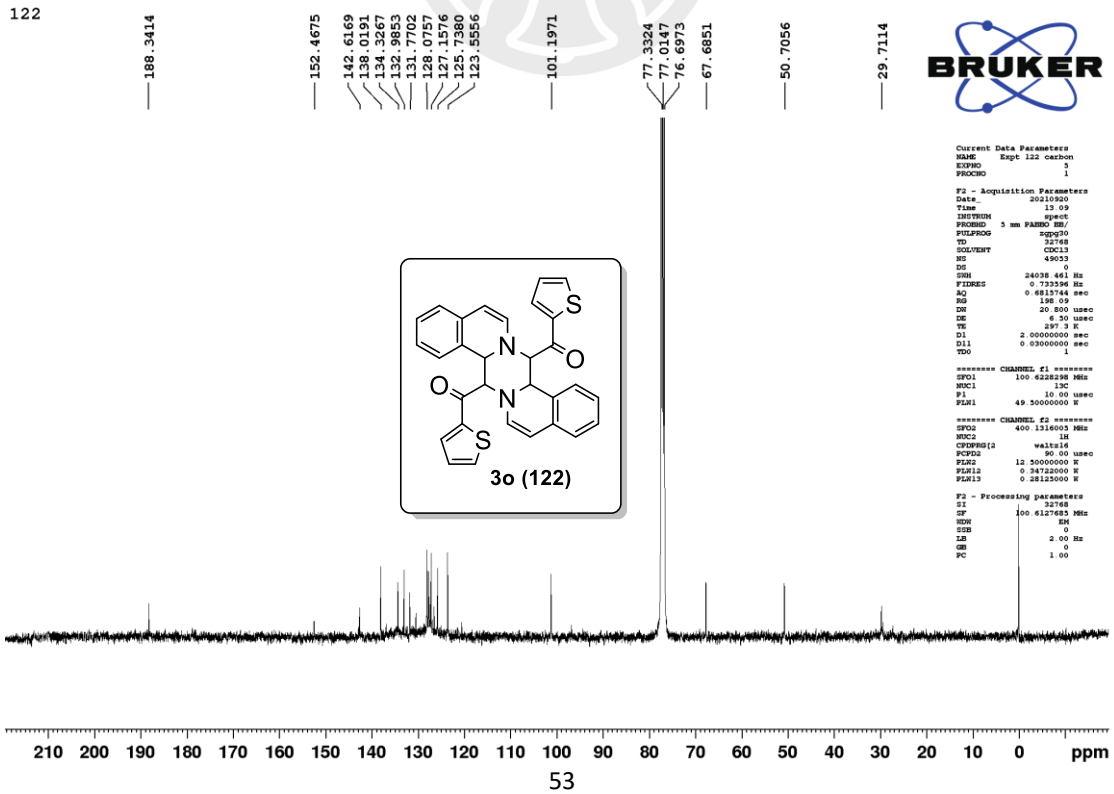
3n (114)


114
7.6410
7.6365
7.6242
7.6195
7.6136
7.5198
7.5146
7.5099
7.4975
7.4932
7.2842
7.1237
7.1182
7.1048
7.1019
7.0988
7.0889
7.0830
6.9679
6.9492
6.9233
6.9045
6.9018
6.8882
6.8858
6.8720
6.3766
6.3578
5.6511
5.6324
5.3798
5.3579
5.3230
4.9468
4.9247
3n (114)
8.0
7.5
7.0
6.5
6.0
5.5
5.0
ppm
4.1576
3.9978
2.1451
1.9308
3.3341
1.4802
1.7856
2.1917
2.0000
11
10
9
8
7
6
5
4
3
2
1
0
ppm

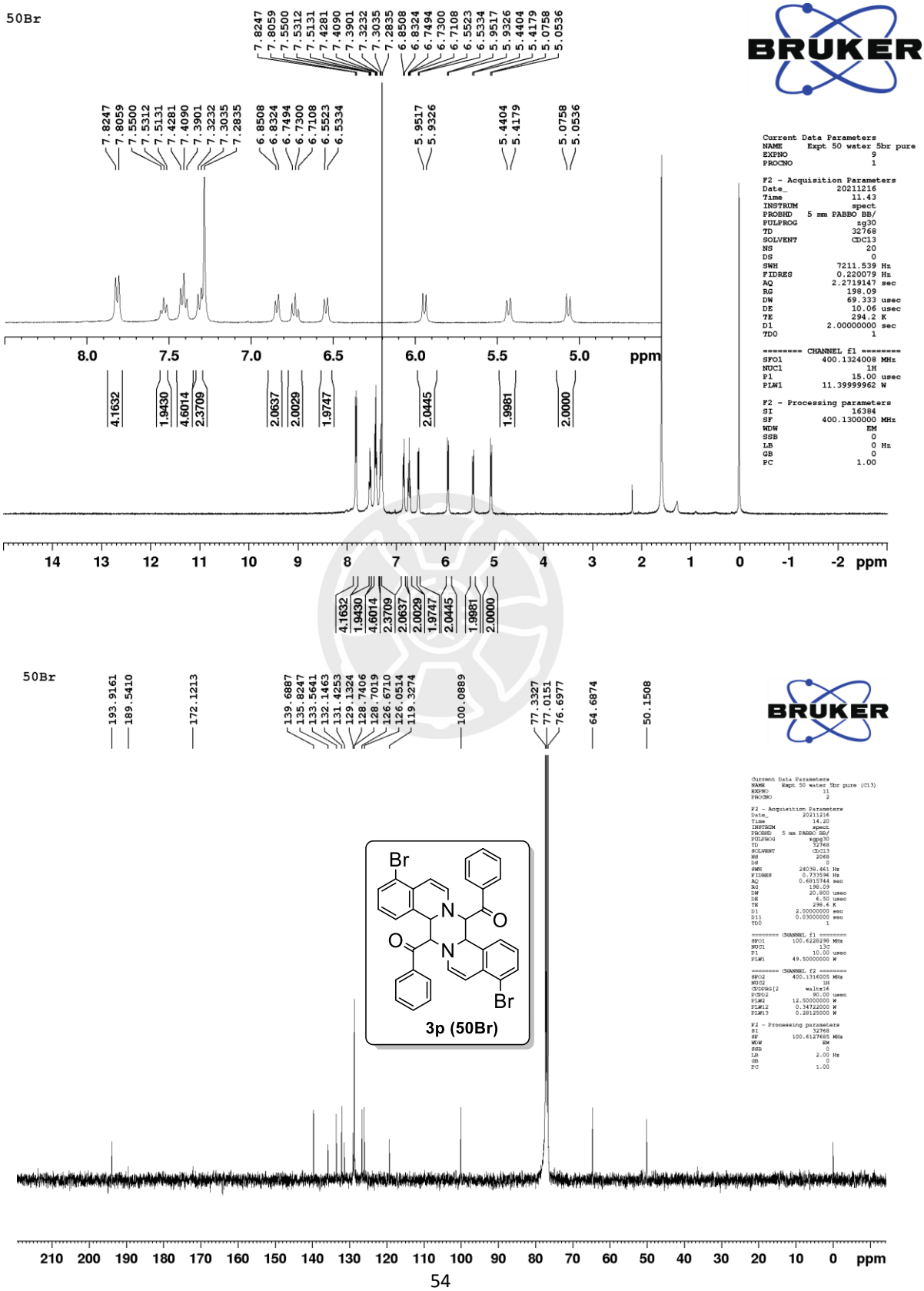

114
193.8995
152.3841
142.6803
137.7902
135.8671
134.8247
132.2781
131.8465
130.1590
129.4830
126.5006
125.8630
123.7263
120.5863
101.6123
77.3432
77.0258
76.7081
65.2486
50.1642
3n (114)
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
ppm

3o (122)

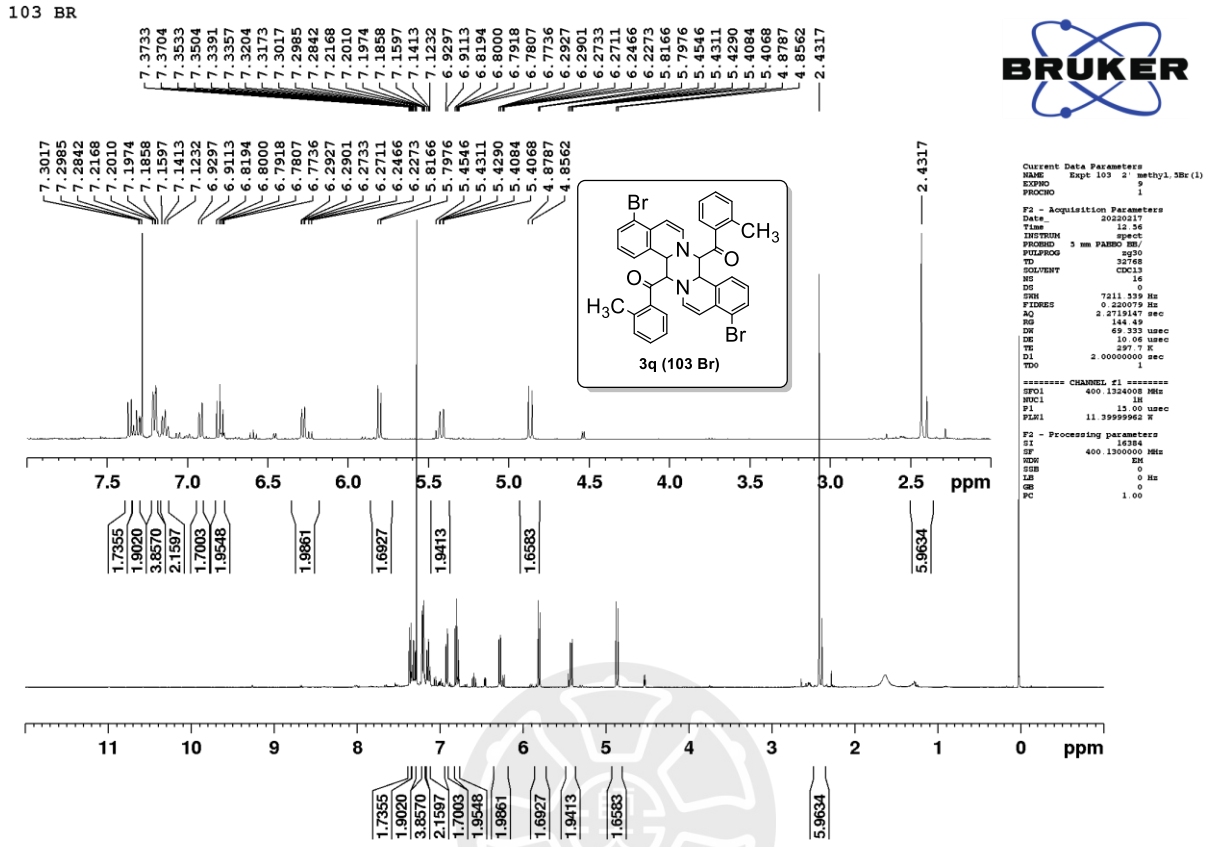

122
7.5844
7.5825
7.5722
7.5703
7.5492
7.5474
7.5395
7.5376
7.2843
7.1331
7.1299
7.1146
7.1114
7.0960
7.0927
7.0226
7.0100
7.0004
6.9802
6.9614
6.9541
6.9512
6.9355
6.9327
6.9170
6.3811
6.3630
5.6629
5.6442
5.4060
5.3842
4.7481
4.7261
2.0000
2.2031
2.4884
9.8477
2.0821
1.3483
1.6466
2.3139
ppm

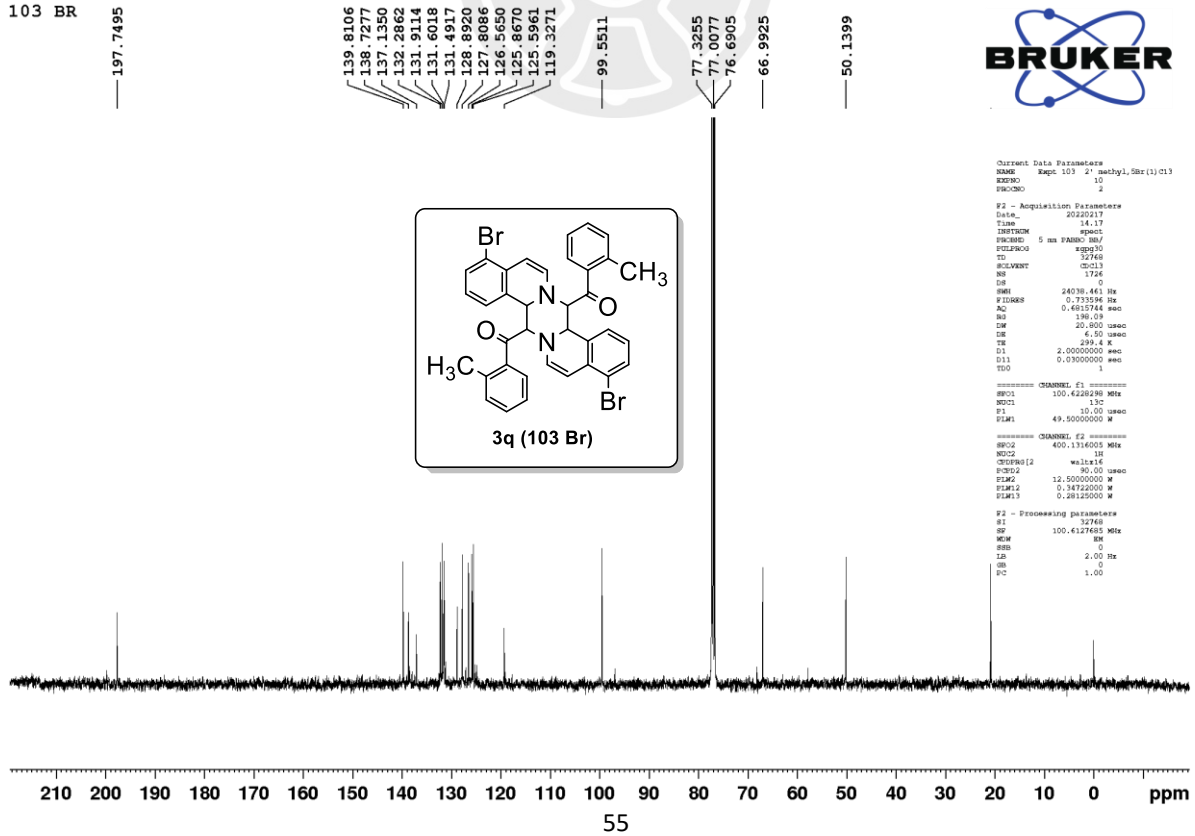

122
188.3414
152.4675
142.6169
138.0191
134.3267
132.9853
131.7702
128.0757
127.1576
125.7380
123.5556
101.1971
77.3324
77.0147
76.6973
67.6851
50.7056
29.7114
3o (122)
ppm

3p (50Br)

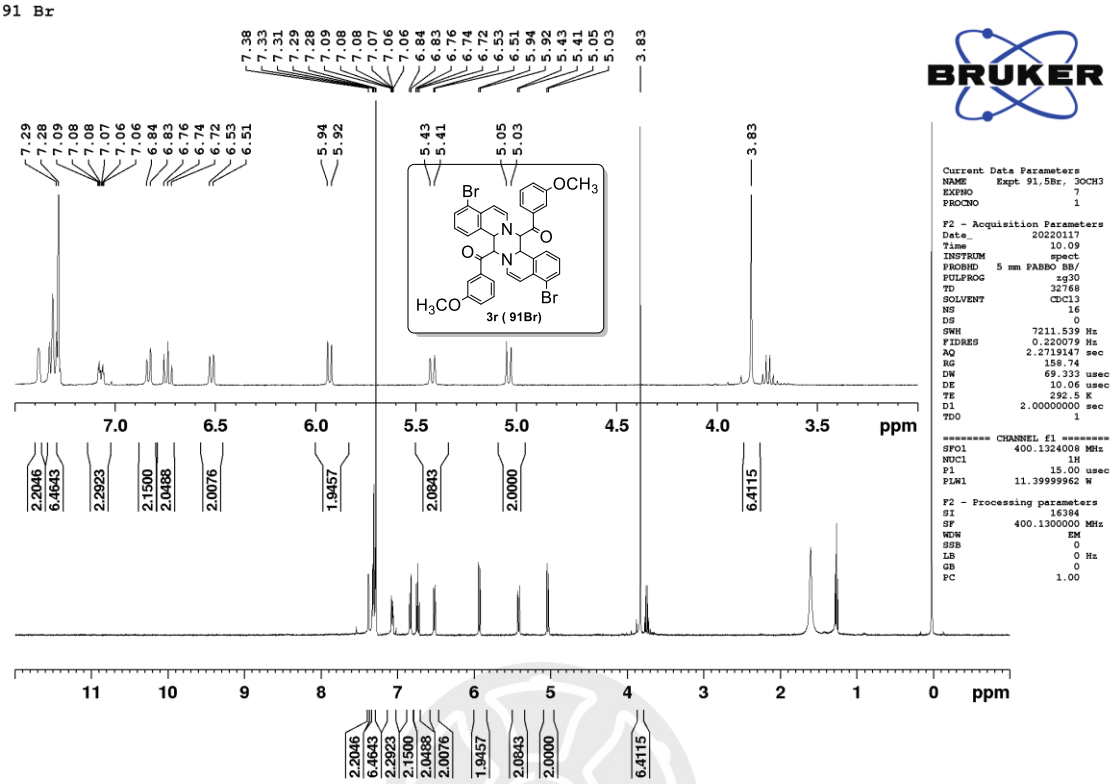

50Br
7.8247
7.8059
7.5500
7.5312
7.5131
7.4281
7.4090
7.3901
7.3232
7.3035
7.2835
6.8508
6.8324
6.7494
6.7300
6.7108
6.5523
6.5334
5.9517
5.9326
5.4404
5.4179
5.0758
5.0536
4.1632
1.9430
4.6014
2.3709
2.0637
2.0029
1.9747
2.0445
1.9981
2.0000
ppm
50Br
193.9161
189.5410
172.1213
139.6887
135.8247
133.5641
132.1463
131.4253
129.1324
128.7406
128.7019
126.6710
126.0514
119.3274
100.0889
77.3327
77.0151
76.6977
64.6874
50.1508
Br
Br
3p (50Br)

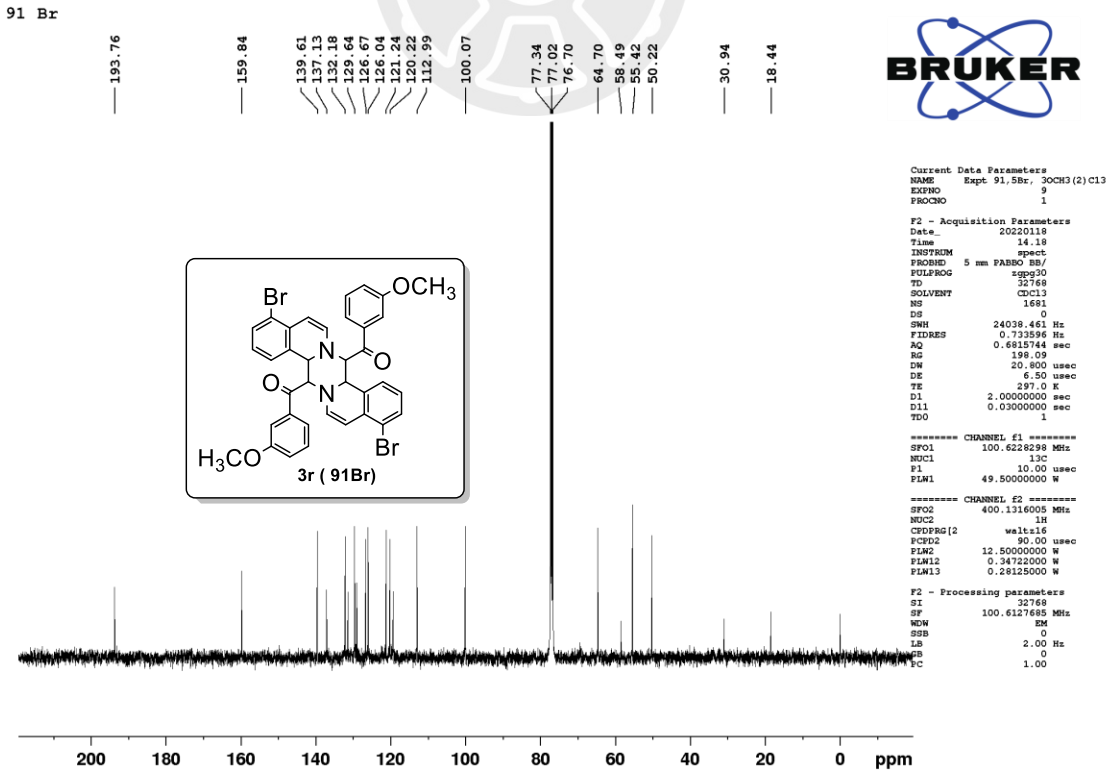
3q (103Br)



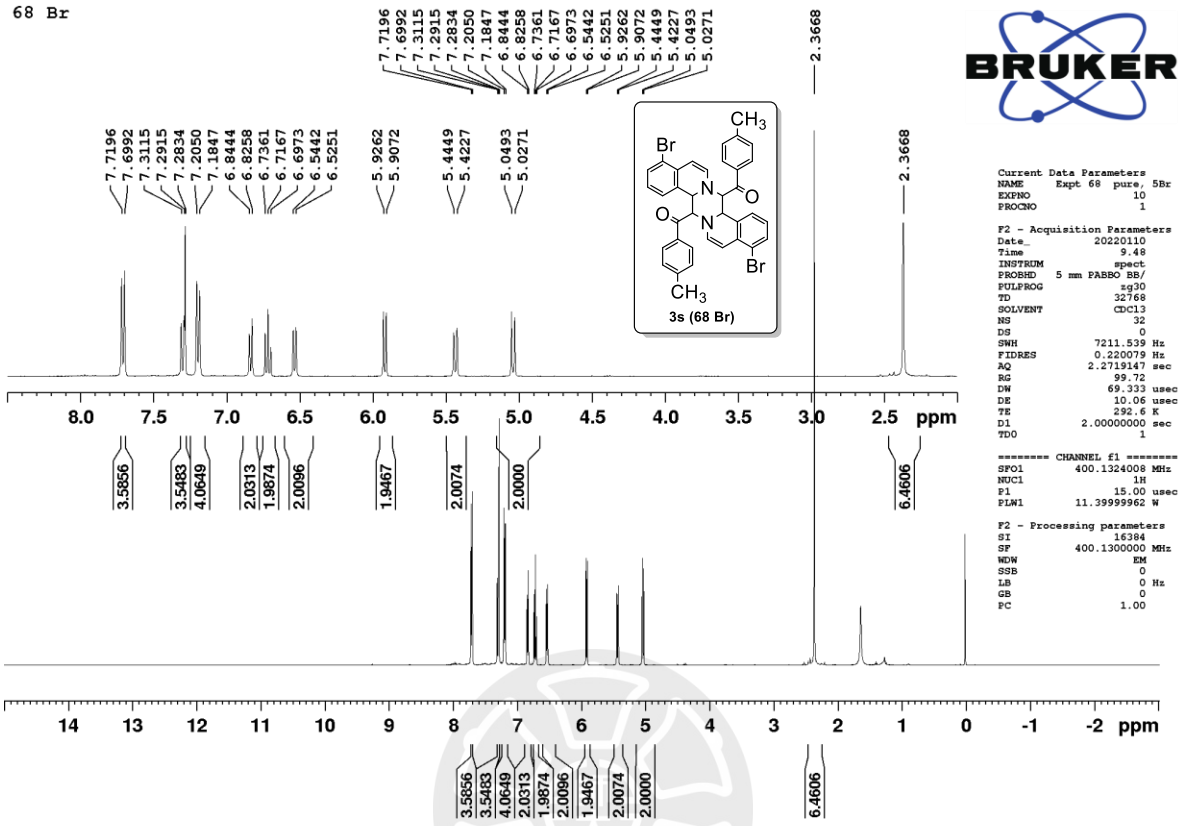


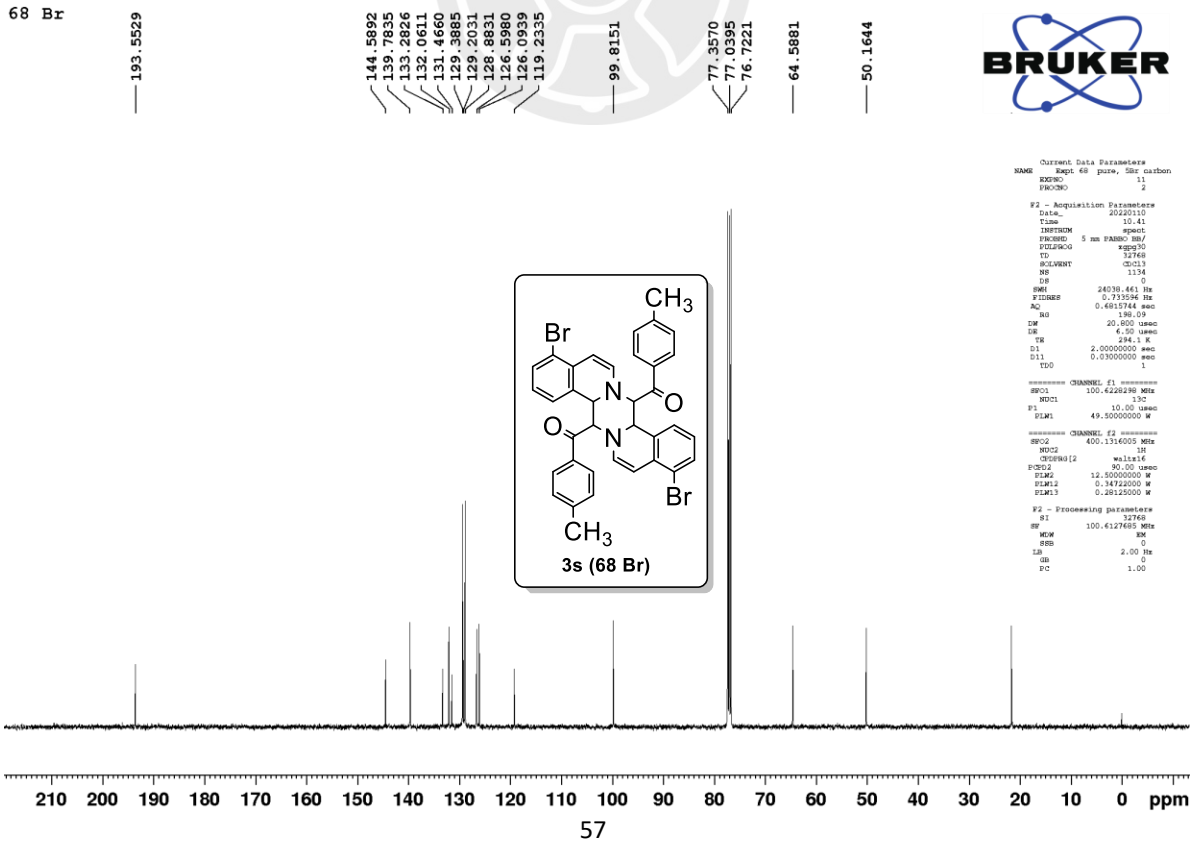
3r (91Br)


91 Br
7.38 7.33 7.31 7.29 7.28 7.09 7.08 7.08 7.07 7.06 7.06 6.84 6.83 6.76 6.74 6.72 6.53 6.51 5.94 5.92 5.43 5.41 5.05 5.03 3.83
Br
OCH3
N
O
O
N
H3CO
Br
3r (91Br)
7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 ppm
2.2046 6.4643 2.2923 2.1500 2.0488 2.0076 1.9457 2.0843 2.0000 6.4115
11 10 9 8 7 6 5 4 3 2 1 0 ppm
BRUKER
Current Data Parameters
NAME Expt 91,5Br, 3OCH3
EXPNO 7
PROCNO 1
F2 - Acquisition Parameters
Date_ 20220117
Time 10.09
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 0
SWH 7211.539 Hz
FIDRES 0.220079 Hz
AQ 2.2719147 sec
RG 158.74
DW 69.333 usec
DE 10.06 usec
TE 292.5 K
D1 2.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 400.1324008 MHz
NUC1 1H
P1 15.00 usec
PLW1 11.39999962 W
F2 - Processing parameters
SI 16384
SF 400.1300000 MHz
WDW EM
SSB 0
LB 0 Hz
GB 0
PC 1.00

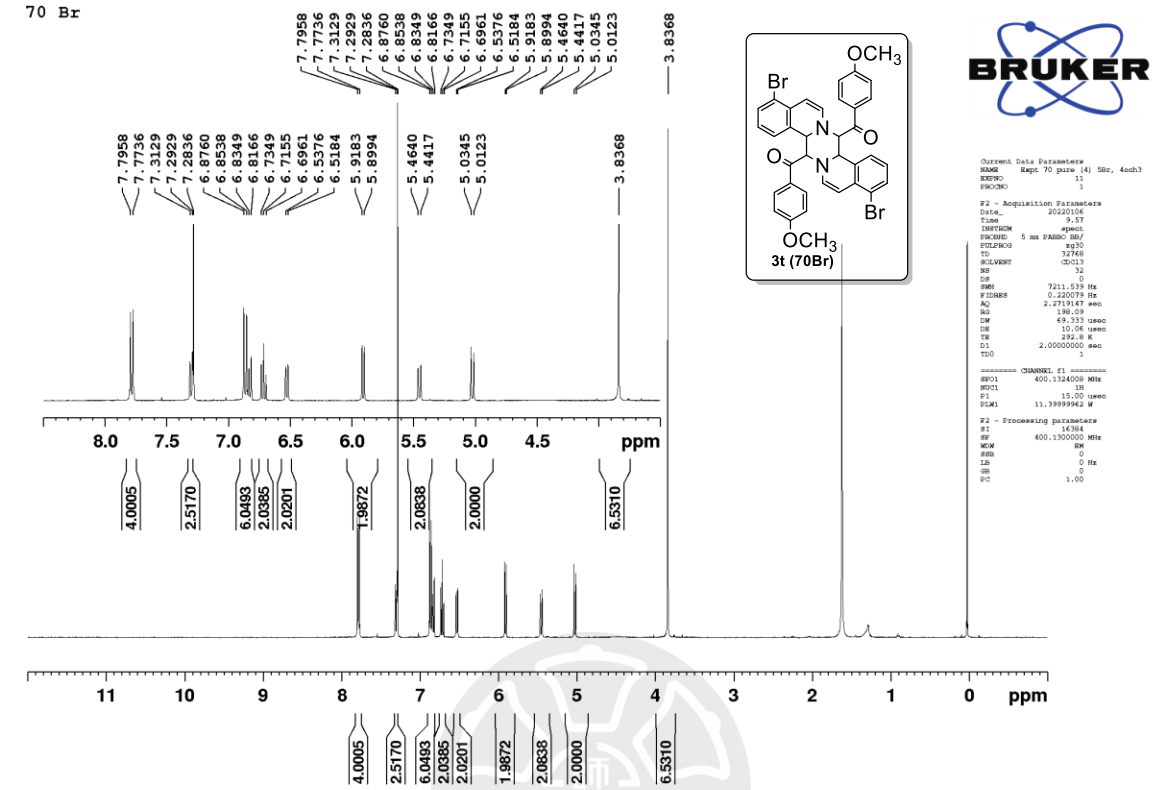

91 Br
193.76 159.84 139.61 137.13 132.18 129.64 126.67 126.04 121.24 120.22 112.99 100.07 77.34 77.02 76.70 64.70 58.49 55.42 50.22 30.94 18.44
Br
OCH3
N
O
O
N
H3CO
Br
3r (91Br)
200 180 160 140 120 100 80 60 40 20 0 ppm
BRUKER
Current Data Parameters
NAME Expt 91,5Br, 3OCH3(2)C13
EXPNO 9
PROCNO 1
F2 - Acquisition Parameters
Date_ 20220118
Time 14.18
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 1681
DS 0
SWH 24038.461 Hz
FIDRES 0.733596 Hz
AQ 0.6815744 sec
RG 198.09
DW 20.800 usec
DE 6.50 usec
TE 297.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 100.6228298 MHz
NUC1 13C
P1 10.00 usec
PLW1 49.50000000 W
======== CHANNEL f2 ========
SFO2 400.1316005 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 90.00 usec
PLW2 12.50000000 W
PLW12 0.34722000 W
PLW13 0.28125000 W
F2 - Processing parameters
SI 32768
SF 100.6127685 MHz
WDW EM
SSB 0
LB 2.00 Hz
GB 0
PC 1.00

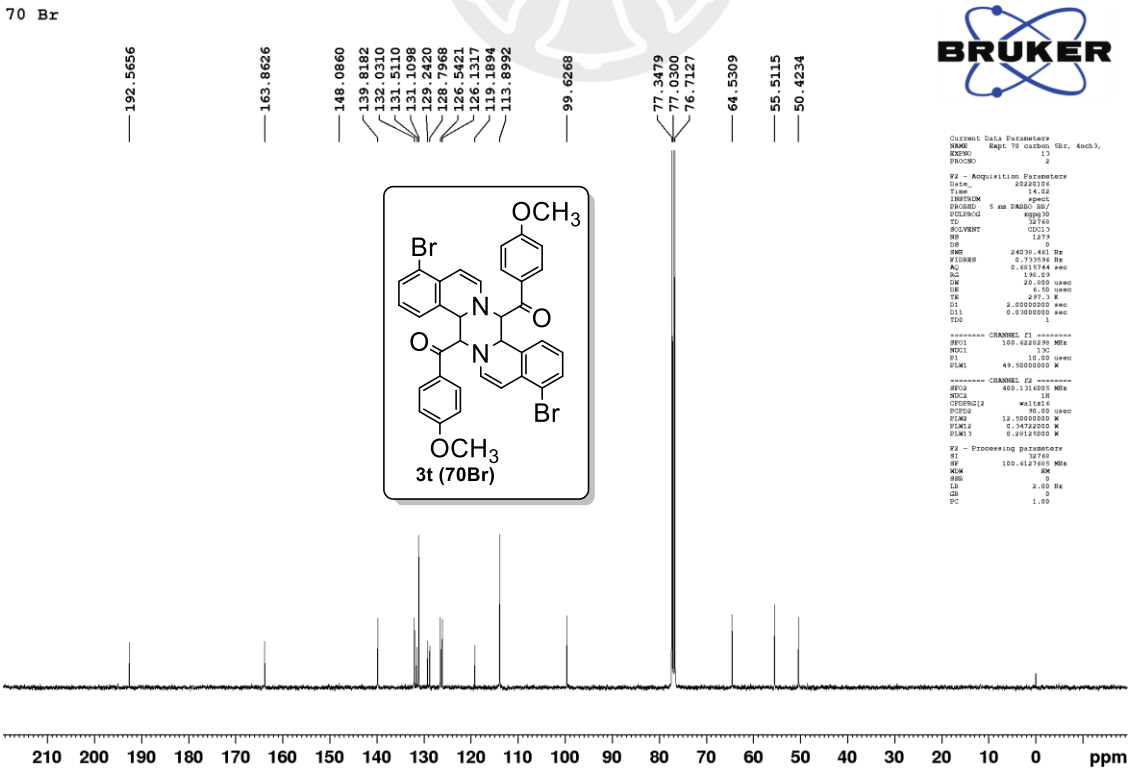
3s (68Br)


68 Br
7.7196
7.6992
7.3115
7.2915
7.2834
7.2050
7.1847
6.8444
6.8258
6.7361
6.7167
6.6973
6.5442
6.5251
5.9262
5.9072
5.4449
5.4227
5.0493
5.0271
2.3668
3s (68 Br)
Current Data Parameters
NAME Expt 68 pure, 5Br
EXPNO 10
PROCNO 1
F2 - Acquisition Parameters
Date_ 20220110
Time 9.48
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 32
DS 0
SWH 7211.539 Hz
FIDRES 0.220079 Hz
AQ 2.2719147 sec
RG 99.72
DW 69.333 usec
DE 10.06 usec
TE 292.6 K
D1 2.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 400.1324008 MHz
NUC1 1H
P1 15.00 usec
PLW1 11.39999962 W
F2 - Processing parameters
SI 16384
SF 400.1300000 MHz
WDW EM
SSB 0
LB 0 Hz
GB 0
PC 1.00
3.5856
3.5483
4.0649
2.0313
1.9874
2.0096
1.9467
2.0074
2.0000
6.4606
ppm

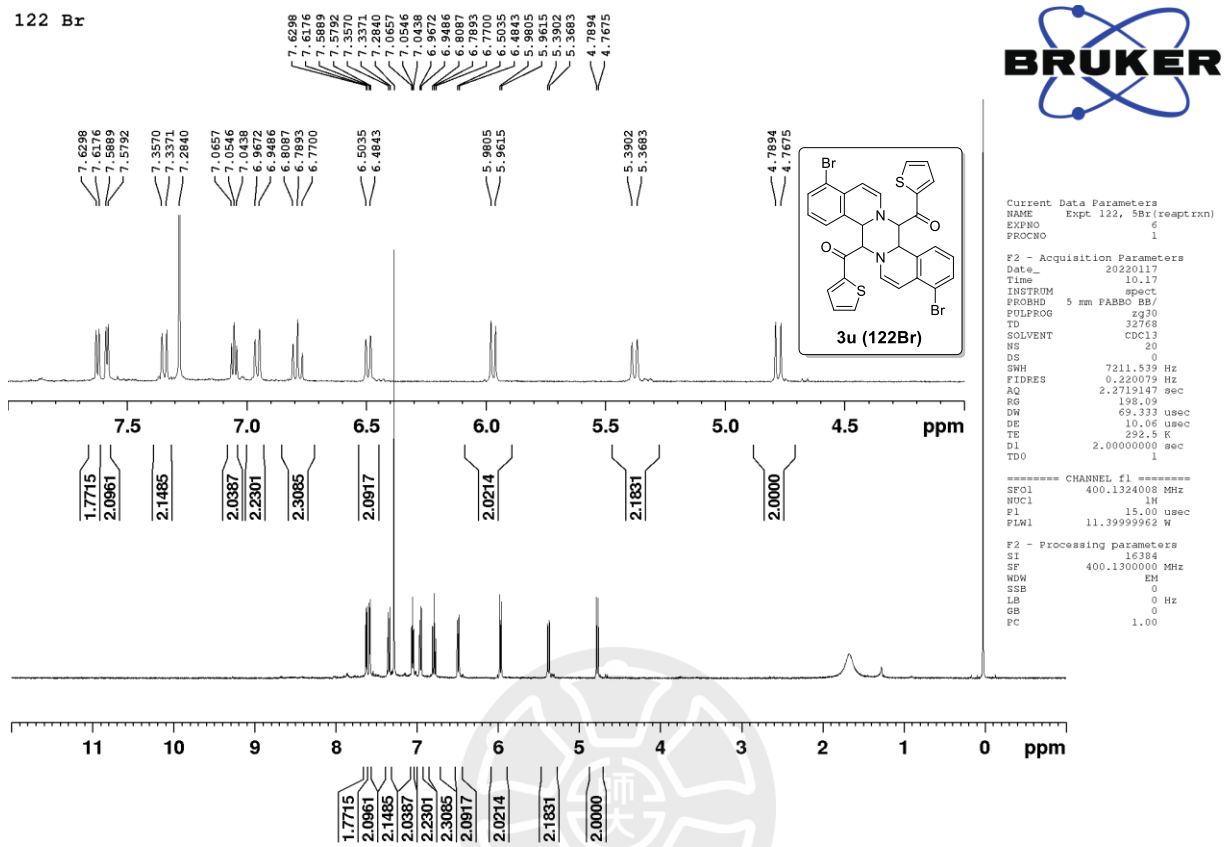

68 Br
193.5529
144.5892
139.7835
133.2826
132.0611
131.4660
129.3885
129.2031
128.8831
126.5980
126.0939
119.2335
99.8151
77.3570
77.0395
76.7221
64.5881
50.1644
3s (68 Br)
Current Data Parameters
NAME Expt 68 pure, 5Br carbon
EXPNO 11
PROCNO 2
F2 - Acquisition Parameters
Date_ 20220110
Time 10.41
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 1134
DS 0
SWH 24038.461 Hz
FIDRES 0.733596 Hz
AQ 0.6815744 sec
RG 198.09
DW 20.800 usec
DE 6.50 usec
TE 294.1 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 100.6228298 MHz
NUC1 13C
P1 10.00 usec
PLW1 49.50000000 W
======== CHANNEL f2 ========
SFO2 400.1316005 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 90.00 usec
PLW2 12.50000000 W
PLW12 0.34722000 W
PLW13 0.28125000 W
F2 - Processing parameters
SI 32768
SF 100.6127685 MHz
WDW EM
SSB 0
LB 2.00 Hz
GB 0
PC 1.00
ppm

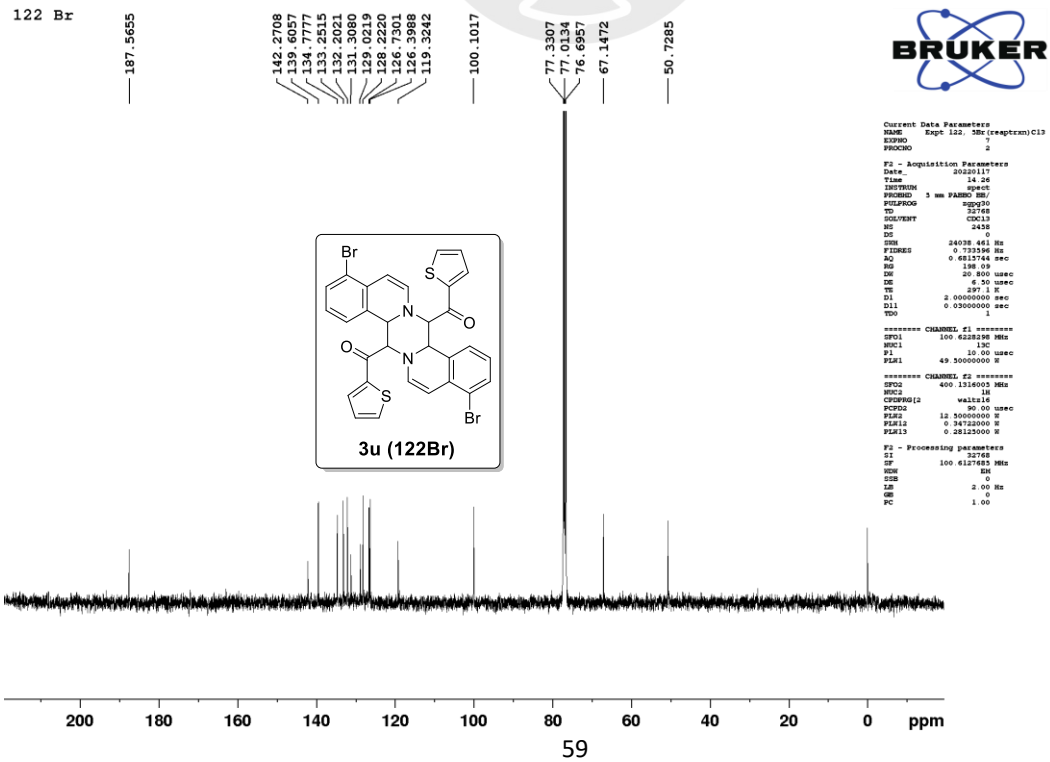
3t (70Br)


70 Br
7.7958
7.7736
7.3129
7.2929
7.2836
6.8760
6.8538
6.8349
6.8166
6.7349
6.7155
6.6961
6.5376
6.5184
5.9183
5.8994
5.4640
5.4417
5.0345
5.0123
3.8368
8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 ppm
4.0005 2.5170 6.0493 2.0385 2.0201 1.9872 2.0638 2.0000 6.5310
11 10 9 8 7 6 5 4 3 2 1 0 ppm
OCH3
Br
N
O
3t (70Br)
BRUKER


70 Br
192.5656
163.8626
148.0860
139.8182
132.0310
131.5110
131.1098
129.2420
128.7968
126.5421
126.1317
119.1894
113.8992
99.6268
77.3479
77.0300
76.7127
64.5309
55.5115
50.4234
OCH3
Br
N
O
3t (70Br)
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm
BRUKER

3u (122Br)


122 Br
7.6298
7.6176
7.5889
7.5792
7.3570
7.3371
7.2840
7.0657
7.0546
7.0438
6.9672
6.9486
6.8087
6.7893
6.7700
6.5035
6.4843
5.9805
5.9615
5.3902
5.3683
4.7894
4.7675
1.7715
2.0961
2.1485
2.0387
2.2301
2.3085
2.0917
2.0214
2.1831
2.0000
3u (122Br)
Current Data Parameters
NAME Expt 122, 5Br(reaptrxn)
EXPNO 6
PROCNO 1
F2 - Acquisition Parameters
Date_ 20220117
Time 10.17
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 20
DS 0
SWH 7211.539 Hz
FIDRES 0.220079 Hz
AQ 2.2719147 sec
RG 198.09
DW 69.333 usec
DE 10.06 usec
TE 292.5 K
D1 2.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 400.1324008 MHz
NUC1 1H
P1 15.00 usec
PLW1 11.39999962 W
F2 - Processing parameters
SI 16384
SF 400.1300000 MHz
WDW EM
SSB 0
LB 0 Hz
GB 0
PC 1.00


122 Br
187.5655
142.2708
139.6057
134.7777
133.2515
132.2021
131.3080
129.0219
128.2220
126.7301
126.3988
119.3242
100.1017
77.3307
77.0134
76.6957
67.1472
50.7285
3u (122Br)
Current Data Parameters
NAME Expt 122, 5Br(reaptrxn)C13
EXPNO 7
PROCNO 2
F2 - Acquisition Parameters
Date_ 20220117
Time 14.26
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 2432
DS 0
SWH 24038.461 Hz
FIDRES 0.733596 Hz
AQ 0.6815744 sec
RG 198.09
DW 20.800 usec
DE 6.50 usec
TE 297.1 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 100.6228298 MHz
NUC1 13C
P1 10.00 usec
PLW1 49.50000000 W
======== CHANNEL f2 ========
SFO2 400.1316005 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 90.00 usec
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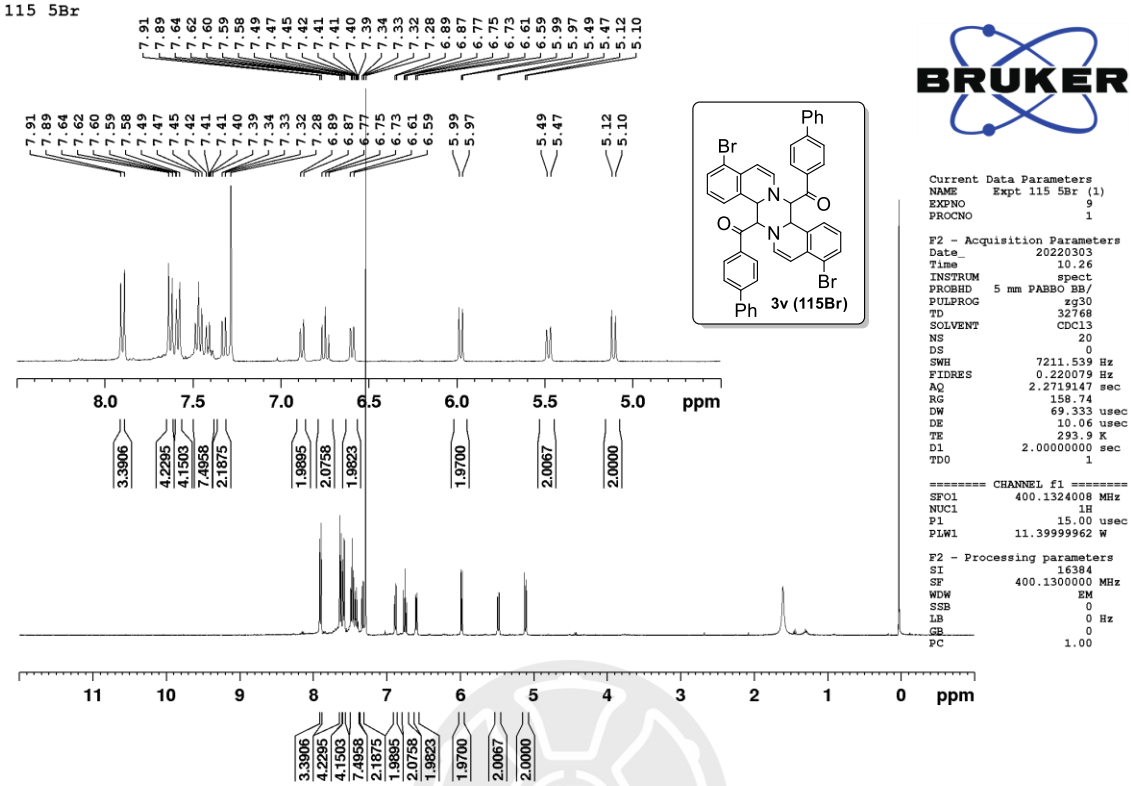
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8.0 7.5 7.0 6.5 6.0 5.5 5.0 ppm
3.3906 4.2295 4.1503 7.4958 2.1875 1.9895 2.0758 1.9823 1.9700 2.0067 2.0000
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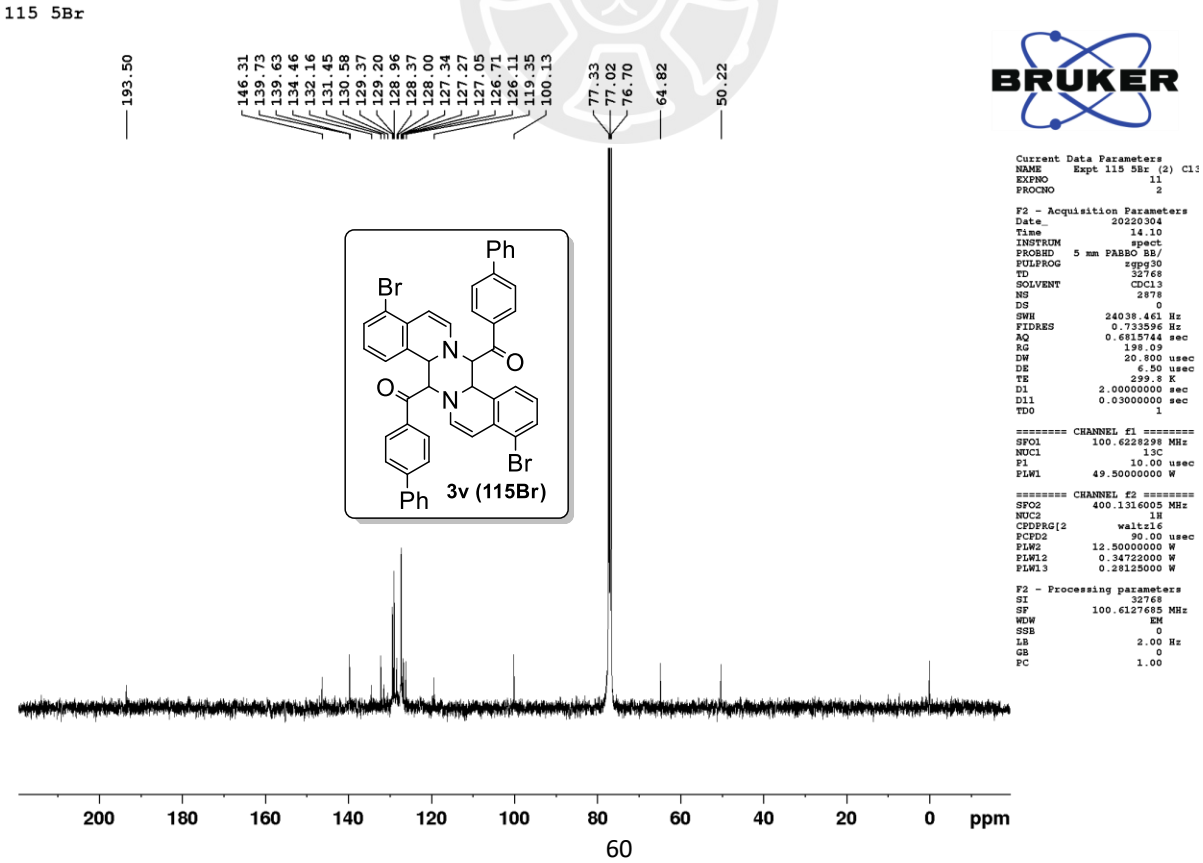
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LB 2.00 Hz
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PC 1.00

Table S1: The ADMET parameters of compound **50**

Properties	Predicted value
Absorption	
Water solubility (log mol/L)	-3.176
Caco2 permeability (log Papp in 10^{-6} cm/s)	1.051
Intestinal absorption (human) (% Absorbed)	95.817
Skin Permeability (log Kp)	-2.735
P-glycoprotein substrate	Yes
P-glycoprotein I inhibitor	Yes
P-glycoprotein II inhibitor	Yes
Distribution	
VDss (human) (log L/kg)	0.475
Fraction unbound (human) (Fu)	0.198
BBB permeability (log BB)	0.099
CNS permeability (log PS)	1.074
Metabolism	
CYP2D6 substrate	Yes
CYP3A4 substrate	Yes
CYP1A2 inhibitior	No
CYP2C19 inhibitior	No
CYP2C9 inhibitior	No
CYP2D6 inhibitior	No
CYP3A4 inhibitior	No
Excretion	
Total Clearance (log ml/min/kg)	0.305
Renal OCT2 substrate	No
Toxicity	
AMES toxicity	No
Max. tolerated dose (human) (log mg/kg/day)	0.787
hERG I inhibitor	No
hERG II inhibitor	Yes
Oral Rat Acute Toxicity (LD_{50}) (mol/kg)	2.474
Oral Rat Chronic Toxicity (LOAEL) (log mg/kg_bw/day)	1.81
Hepatotoxicity	Yes
Skin Sensitization	No
T.Pyriformis toxicity (log ug/L)	0.285
Minnow toxicity (log mM)	-1.532

Table S2. Crystal data and structure refinement for **98**

Identification code	D23735	
Empirical formula	$C_{36}H_{26}N_2O_6$	
Formula weight	582.59	
Temperature	200(2) K	
Wavelength	0.71073 Å	
Crystal system	Monoclinic	
Space group	C 2/c	
Unit cell dimensions	a = 24.4502(16) Å	α= 90°
	b = 5.8771(4) Å	β= 96.850(2)°
	c = 18.9143(14) Å	γ = 90°.
Volume	2698.5(3) Å3	
Z	4	
Density (calculated)	1.434 Mg/m3	
Absorption coefficient	0.098 mm-1	
F(000)	1216	
Crystal size	0.10 x 0.08 x 0.02 mm3	
Theta range for data collection	2.17 to 25.04°	
Index ranges	-28<=h<=26, -6<=k<=6, -22<=l<=22	
Reflections collected	14413	
Independent reflections	2372 [R(int) = 0.0601]	
Completeness to theta = 25.04°	99.7 %	
Absorption correction	multi-scan	
Max. and min. transmission	0.9980 and 0.9902	
Refinement method	Full-matrix least-squares on F2	
Data/restraints/parameters	2372/0/199	
Goodness-of-fit on F2	1.077	
Final R indices [I>2sigma(I)]	R1 = 0.0419, wR2 = 0.0936	
R indices (all data)	R1 = 0.0626, wR2 = 0.1044	
Largest diff. peak and hole	0.164 and -0.232 e.Å-3	

Table S3. Crystal data and structure refinement for**103Br**

Identification code	D23846	
Empirical formula	$C_{36}H_{28}Br_2N_2O_2$	
Formula weight	680.42	
Temperature	200(2) K	
Wavelength	0.71073 Å	
Crystal system	Monoclinic	
Space group	C 2/c	
Unit cell dimensions	a = 20.461(7) Å	α= 90°
	b = 7.501(3) Å	β= 90.528(11)°
	c = 19.144(7) Å	γ = 90°.
Volume	2938.0(8) Å3	
Z	4	
Density (calculated)	1.538 Mg/m3	
Absorption coefficient	2.796 mm-1	
F(000)	1376	
Crystal size	0.07 x 0.03 x 0.02 mm3	
Theta range for data collection	2.89 to 25.31°	
Index ranges	-24<=h<=20, -8<=k<=9, -22<=l<=22	
Reflections collected	10441	
Independent reflections	2636 [R(int) = 0.0810]	
Completeness to theta = 25.04°	98.5 %	
Absorption correction	multi-scan	
Max. and min. transmission	0.9462 and 0.8283	
Refinement method	Full-matrix least-squares on F2	
Data/restraints/parameters	2636/0/191	
Goodness-of-fit on F2	0.958	
Final R indices [I>2sigma(I)]	R1 = 0.0479, wR2 = 0.1080	
R indices (all data)	R1 = 0.1030, wR2 = 0.1408	
Largest diff. peak and hole	0.268 and -0.444 e.Å-3	

Table S4. Crystal data and structure refinement for **68Br**

Identification code	D23821	
Empirical formula	$C_{36}H_{28}Br_2$	
Formula weight	689.29	
Temperature	200(2) K	
Wavelength	0.71073 Å	
Crystal system	Monoclinic	
Space group	P 21/n	
Unit cell dimensions	a = 13.556(2) Å	α= 90°
	b = 13.243(2) Å	β= 94.349(5)°
	c = 18.995(3) Å	γ = 90°.
Volume	3400.1(9) Å3	
Z	4	
Density (calculated)	1.347 Mg/m3	
Absorption coefficient	2.436 mm-1	
F(000)	1393	
Crystal size	0.12 x 0.09 x 0.04 mm3	
Theta range for data collection	2.15 to 25.05°	
Index ranges	-16<=h<=15, -15<=k<=15, -22<=l<=21	
Reflections collected	41666	
Independent reflections	6013 [R(int) = 0.1016]	
Completeness to theta = 25.04°	99.6 %	
Absorption correction	multi-scan	
Max. and min. transmission	0.9089 and 0.7587	
Refinement method	Full-matrix least-squares on F2	
Data/restraints/parameters	6013/2/418	
Goodness-of-fit on F2	1.003	
Final R indices [I>2sigma(I)]	R1 = 0.0609, wR2 = 0.1378	
R indices (all data)	R1 = 0.1074, wR2 = 0.1613	
Largest diff. peak and hole	0.654 and -0.872 e.Å-3	

Table S5. Crystal data and structure refinement for **122Br**

Identification code	D23823a	
Empirical formula	$C_{30}H_{20}Br_2N_2O_2$	
Formula weight	664.42	
Temperature	200(2) K	
Wavelength	0.71073 Å	
Crystal system	Monoclinic	
Space group	C 2/c	
Unit cell dimensions	a = 21.261(13) Å	α= 90°
	b = 6.095(4) Å	β= 98.49(2)°
	c = 20.426(15) Å	γ = 90°.
Volume	2618(3) Å3	
Z	4	
Density (calculated)	1.686 Mg/m3	
Absorption coefficient	3.289 mm-1	
F(000)	1328	
Crystal size	0.22 x 0.10 x 0.01 mm3	
Theta range for data collection	2.58 to 25.22°	
Index ranges	-25<=h<=25, -7<=k<=7, -24<=l<=21	
Reflections collected	10039	
Independent reflections	2355 [R(int) = 0.0584]	
Completeness to theta = 25.04°	99.1 %	
Absorption correction	multi-scan	
Max. and min. transmission	0.9679 and 0.5315	
Refinement method	Full-matrix least-squares on F2	
Data/restraints/parameters	2355/0/172	
Goodness-of-fit on F2	0.981	
Final R indices [I>2sigma(I)]	R1 = 0.0527, wR2 = 0.1405	
R indices (all data)	R1 = 0.0766, wR2 = 0.1577	
Largest diff. peak and hole	1.242 and -0.783 e.Å-3	

Table S6. Crystal data and structure refinement for **115Br**

Identification code	D23878	
Empirical formula	$C_{46}H_{30}Br_2N_2O_2$	
Formula weight	802.54	
Temperature	200(2) K	
Wavelength	0.71073 Å	
Crystal system	Monoclinic	
Space group	C 2/c	
Unit cell dimensions	a = 28.32(3) Å	α= 90°
	b = 6.257(5) Å	β= 111.51(6)°
	c = 25.26(2) Å	γ = 90°.
Volume	4164(7) Å3	
Z	4	
Density (calculated)	1.280 Mg/m3	
Absorption coefficient	1.984 mm-1	
F(000)	1624	
Crystal size	0.26 x 0.04 x 0.01 mm3	
Theta range for data collection	2.71 to 25.08°	
Index ranges	-33<=h<=33, -7<=k<=7, -30<=l<=30	
Reflections collected	24724	
Independent reflections	3669 [R(int) = 0.1449]	
Completeness to theta = 25.04°	98.9 %	
Absorption correction	multi-scan	
Max. and min. transmission	0.9804 and 0.6265	
Refinement method	Full-matrix least-squares on F2	
Data/restraints/parameters	3669/0/245	
Goodness-of-fit on F2	1.060	
Final R indices [I>2sigma(I)]	R1 = 0.1016, wR2 = 0.2146	
R indices (all data)	R1 = 0.1962, wR2 = 0.2578	
Largest diff. peak and hole	0.438 and -0.435 e.Å-3	

Part II

Catalyst-free reactions of anilines with β-chloroenones: synthesis of α-chloroenaminones and 1,4-benzodiazepines

摘要

苯胺與β-氯酮的Michael加成反應通過消除鹽酸（HCl）生成烯胺酮。隨後，這些烯胺酮通過原位的sp^2 C-H官能化轉化為α-氯烯胺酮。苯胺上的電子供體基團能提高其與β-氯酮的反應性，生成對應產物並具有優異的產率。開發了一種高度原子經濟的方法，使用二甲基亞碸（DMSO）作為綠色氧化劑和溶劑。該方法可高效生成α-官能化烯胺酮，具有良好的產率和極高的Z-選擇性。通過多種底物驗證了該反應的普適性，並且放大實驗證明其實際應用價值。此外，還實現了在無催化劑的條件下，β-氯酮與鄰苯二胺的雙環化反應，可在溫和條件下合成1,4-苯二氮䓬衍生物，並取得中等產率。

Abstract

The Michael addition of anilines to β-chloroenones produces enaminones through the elimination of hydrochloric acid (HCl). These enaminones undergo in situ sp^2 C-H functionalization to yield α-chloroenaminones. The reactivity of anilines with β-chloroenones is significantly enhanced by electron-donating groups, enabling the formation of products with excellent yields. A highly atom-efficient method utilizing dimethyl sulfoxide (DMSO) as a green oxidant and solvent has been developed, offering α-functionalized enaminones with high yields and remarkable Z-selectivity. The reaction demonstrates broad substrate compatibility and scalability, highlighting its practical utility. Furthermore, a catalyst-free double annulation of β-chloroenones with o-phenylenediamine has been successfully implemented, facilitating the synthesis of 1,4-benzodiazepine derivatives in moderate yields under mild conditions.

Introduction

Enaminones, characterized by multiple reactive sites, exhibit diverse reactivity, including sp^2 C-H bond functionalization, amino group reactions, and 1,2- and 1,4-addition reactions. Among these, sp^2 C-H bond functionalization has been extensively studied to construct C-C, C-O, C-N, C-X, and C-S bonds. Notably, a highly regioselective, solvent-controlled method was recently developed for site-selective acyloxylation at the α- or β-position of enaminones. In 2016, a metal-free reaction between enaminones and thiophenols was introduced, enabling the synthesis of polyfunctionalized aminothioalkenes using KIO_3. Additionally, various strategies have been established for synthesizing SCN-containing enaminones. Enaminones have also been employed in sp^2 C-H halogenation under both transition-metal-catalyzed and metal-free conditions (Scheme 1a). These versatile compounds, with their dual nucleophilic and electrophilic properties, play a crucial role in generating molecular diversity and complexity. They are vital intermediates in the synthesis of heterocyclic compounds and are present in bioactive molecules with antitumor, anti-inflammatory, antibiotic, and antidepressant activities. While numerous synthetic approaches for enaminones have been documented, methods bypassing the direct use of enaminones to produce α-functionalized derivatives remain relatively scarce. For example, a transition-metal-catalyzed reaction between N-sulfonyl-1,2,3-triazoles and formamides has been reported to yield α-functionalized enaminones directly. More recently, a metal-free, one-pot synthesis of α-bromoenaminones was achieved by reacting 3-bromopropenals with anilines in the presence of catalytic $TsOH \cdot H_2O$ (Scheme 1b). These rare but efficient one-pot methods show significant promise for synthesizing α-functionalized enaminones, which are highly valuable intermediates in synthetic chemistry.

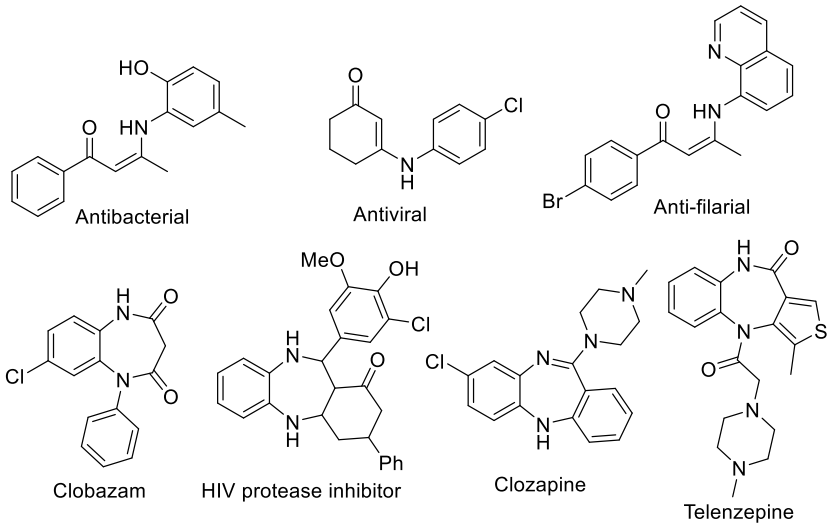
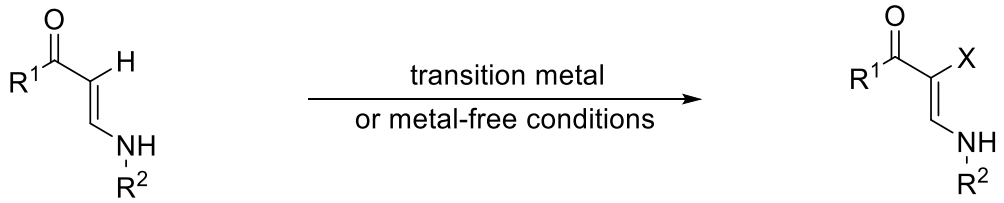
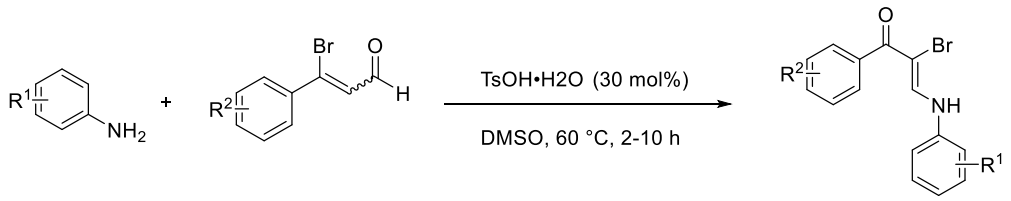


Figure 1. Representative examples of bioactive Enaminones and benzodiazepines

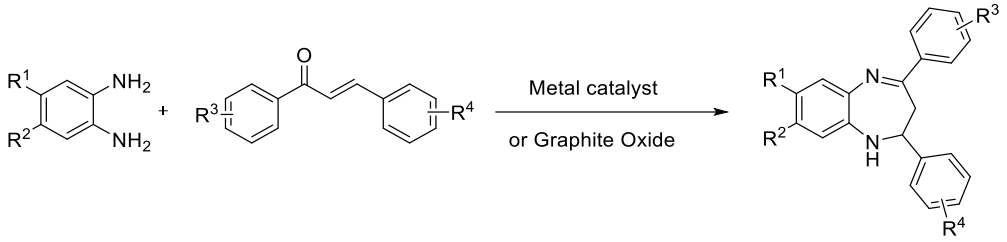
Benzodiazepines are a widely prevalent and essential structural motif found in numerous marketed pharmaceuticals and natural products (Figure 1). Their synthesis is typically achieved through the condensation of o-phenylenediamines with α,β-unsaturated carbonyl compounds under acidic conditions (Scheme 1c). Alternatively, bioactive benzodiazepine derivatives can be prepared by coupling o-phenylenediamines with carbonyl compounds. Interestingly, existing methods do not report the use of β-chloroenones as reaction partners for benzodiazepine synthesis. In this study, we introduce a catalyst-free, divergent strategy for the synthesis of α-chloroenaminones and 1,4-benzodiazepines. This approach leverages dimethyl sulfoxide (DMSO) as both a green solvent and reagent, providing an efficient and sustainable pathway for these valuable transformations (Scheme 1d).



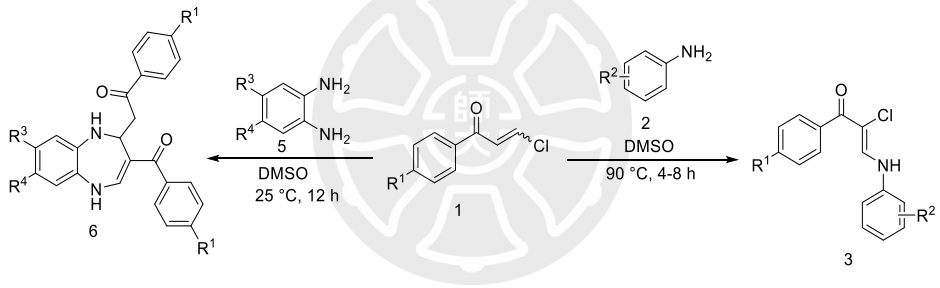
a) Alkenyl sp^2 C-H functionalization of enaminones



b) One-pot synthesis of α-bromoenaminones[12]



c) General approach for the synthesis of 1,5-benzodiazepines[14]



d) This work: Divergent-Synthesis of α-chloroenaminones and 1,4-benzodiazepines

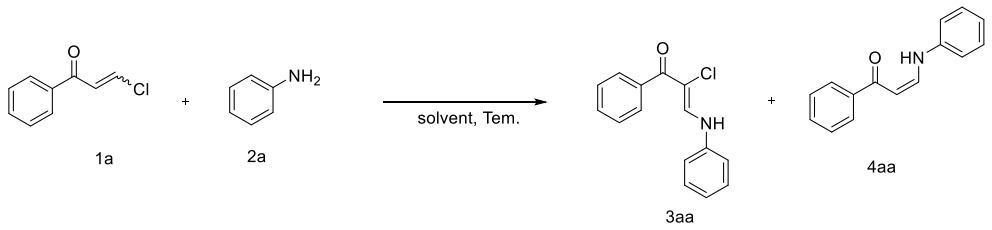
Scheme 1. Synthesis of Enaminones and benzodiazepines

Results and discussion

A sustainable and atom-efficient strategy for synthesizing α-functionalized enaminones was developed by reacting anilines with halopropenones in dimethyl sulfoxide (DMSO). In a preliminary step, β-bromoenone was synthesized by treating phenylpropynone with aluminum chloride ($AlCl_3$) and sodium bromide (NaBr). This β-bromoenone was subsequently employed in a one-pot synthesis of α-functionalized enaminone derivatives, with the results summarized in Table 1. At room temperature, the reaction yielded enaminone 4aa with an isolated yield of 58%

(entry 1). When the reaction temperature was raised to 50 °C, the α-functionalized enaminone 3aa was obtained in a 28% yield, alongside 4aa in a 41% yield (entry 2). Single-crystal analysis of 3aa revealed it to be a (Z)-α-chloroenaminone, rather than the expected bromoenaminone. This finding confirmed that the starting material 1a used in the reaction was β-chloroenone, not β-bromoenone. Further optimization showed that increasing the reaction temperature to 70 °C improved the yield of 3aa to 75% and 84% (entries 3 and 4). The best result was achieved at 90 °C, where (Z)-α-chloroenaminone 3aa was obtained in an excellent 92% yield (entry 5). Solvent screening experiments established that DMSO played a critical dual role as both a green oxidant and a solvent, enabling the in-situ chlorination of enaminone 4aa (entries 6–10). This approach demonstrates a highly efficient and environmentally friendly method for synthesizing α-functionalized enaminones, utilizing DMSO as a sustainable oxidizing agent and solvent.

Table 1. Optimization of reaction conditions[a]



Entry	Solvent	Temp (°C)	Time (h)	Yield **3aa** (%)[b]	Yield **4aa** (%)[b]
1	DMSO	rt	12	-	64 (58)[c]
2	DMSO	50	4	28	41
3	DMSO	70	4	75	6
4	DMSO	70	6	84	5
5	DMSO	90	4	97 (92)[c]	trace
6	DMF	90	4	-	56
7	Toluene	90	4	-	51
8	CH_3CN	90	4	-	49
9	1,4-Dioxane	90	4	-	31
10	DCE	90	4	-	82

[a]All of the reactions were carried out using **1a** (0.3 mmol, 50 mg), **2a** (0.25 mmol, 23 mg), and solvent (2 mL). [b]The yields were determined by ^{1}H NMR analyses of the crude reaction mixtures using CH_2Br_2 as the internal standard. [c]Isolated yield. DMF = Dimethylformamide. DCE = 1,2-Dichloroethane.

To assess the scope of this method, various β-chloroenones and anilines were tested under the optimized conditions that yielded (Z)-3aa with a 92% yield (Table 2). When o-toluidine was reacted with 2a, the enaminone 3ab was obtained in a moderate yield of 68%, likely due to steric hindrance from the ortho-methyl group reducing the reaction rate. In contrast, reactions with meta- and para-toluidines afforded the α-functionalized products 3ac and 3ad in good yields, demonstrating that steric effects are less pronounced in these positions. The reaction of 4-ethylaniline (2e) with 1a proceeded efficiently, yielding 3ae in 80%. Similarly, para-isopropylaniline (2f) produced 3af in an excellent yield of 87%. However, with 2,6-dimethylaniline (2g), the yield of 3ag dropped to 65%. Analysis of the crude mixture revealed a minor side product, 4ag, forming in 15% yield but not isolated in pure form. The reduced yield is

attributed to significant steric hindrance from the two ortho-methyl groups. Interestingly, 2,4-dimethylaniline reacted with 1a to produce 3ah in 80% yield. Anilines with electron-donating groups exhibited higher efficiency, yielding products 3ai and 3aj in excellent yields of 90% and 94%, respectively. Conversely, o-nitroaniline reacted sluggishly, yielding 3ak in only 40% after 8 hours, due to the combined steric and electron-withdrawing effects of the ortho-nitro group. In contrast, meta-nitroaniline produced 3al in a higher yield of 79%, as the meta-position exerted only an inductive electron-withdrawing effect. For para-nitroaniline, the combined inductive and mesomeric effects influenced the reaction efficiency. These results demonstrate that steric hindrance and electron-withdrawing substituents on aniline derivatives reduce the reaction efficiency, while electron-donating groups significantly enhance the yield of the desired products.

Table 2. Atom-economical synthesis of α-chloroenaminones[a,b,c]

3aa, 4 h, 92% | **3ab**, 4 h, 68%[c] | **3ac**, 4 h, 84% | **3ad**, 4 h, 93% | **3ae**, 4 h, 80% | **3af**, 4 h, 87% | **3ag**, 4 h, 65%[c]

3ah, 4 h, 80% | **3ai**, 4 h, 90% | **3aj**, 4 h, 94% | **3ak**, 8 h, 40% | **3al**, 8 h, 79% | **3am**, 8 h, 62% | **3an**, 8 h, 51%

3ao, 8 h, 63% | **3ap**, 8 h, 82% | **3aq**, 8 h, 85% | **3ar**, 8 h, 50% | **3as**, 8 h, 36% | **3ba**, 4 h, 75% | **3bj**, 4 h, 70%

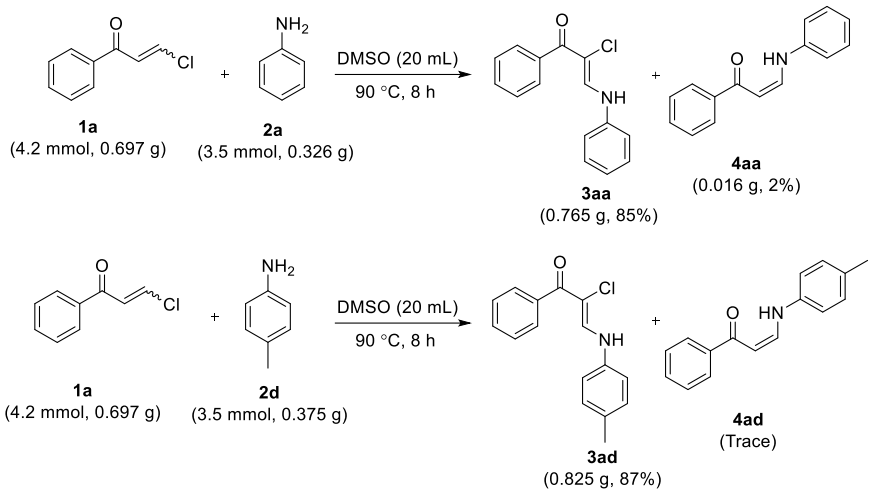
3bl, 8 h, 60% | **3bp**, 8 h, 63% | **3ca**, 4 h, 78% | **3cj**, 4 h, 72% | **3cl**, 8 h, 55% | **3cp**, 8 h, 65% | **3da**, 4 h, 80%

3dj, 4 h, 75% | **3dl**, 8 h, 47% | **3dp**, 8 h, 69% | **3ea**, 4 h, 91% | **3ej**, 4 h, 87% | **3el**, 8 h, 72% | **3ep**, 8 h, 76%

[a]Reactions were performed using **1** (0.3 mmol, 1.2 equiv.), **2** (0.25 mmol), in DMSO (2 mL) at 90 °C. [b]The product **4** was formed in traces. [c]The minor product was formed in 10-15% yield.

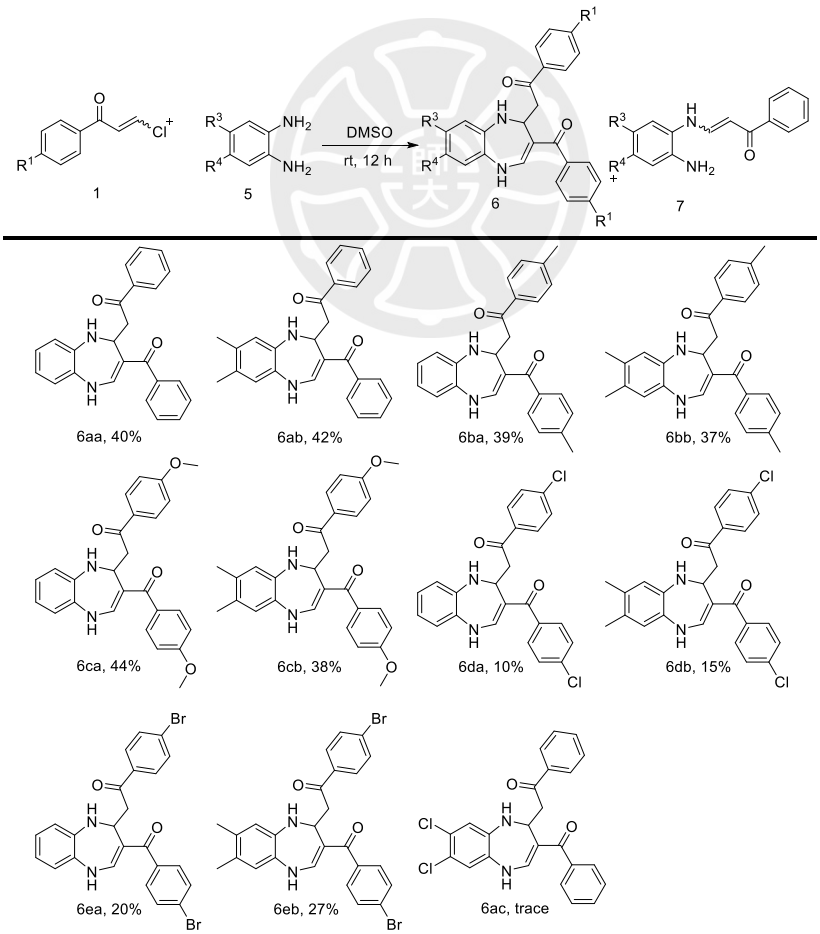
To gain further insights, we investigated the use of halo-substituted aniline derivatives. Reactions with ortho-chloroaniline resulted in enaminone 3an in a 51% yield. Meta- and para-chloroanilines produced the corresponding products 3ao and 3ap in 63% and 82% yields, respectively, highlighting the influence of electronic effects on the reaction outcomes. When para-bromoaniline

was used, the desired product was obtained in an excellent 85% yield. We then tested benzylamine as a coupling partner, yielding product 3ar in 50%. However, using phenylethylamine (2s) under the same conditions resulted in enaminone 3as in only 36%, suggesting that amines with aliphatic units are less effective in this reaction. Attempts to use 5-amino-N-benzylindole as a Michael donor in the reaction produced only a small quantity of inseparable product mixtures. Next, we explored the impact of substituents on the aromatic ring of β-chloroenones. Reactions with β-chloroenone 1b and anilines bearing electron-donating groups confirmed that these anilines react more efficiently than those with electron-withdrawing groups. A similar trend was observed with β-chloroenone 1c, which contains a para-methoxy group as a Michael acceptor, demonstrating consistent reactivity patterns in this atom-economical synthesis. We also examined β-chloroenone 1d, which contains a chloro-substituent, to assess its effect on reaction outcomes. The desired products (3da, 3dj, and 3dp) were obtained in moderate to good yields, though enaminone 3dl, formed from the reaction with meta-nitroaniline (2l), was isolated in only 47% yield. To further evaluate the impact of a bromine substituent, we employed β-chloroenone 1e, which has a para-bromo group. The corresponding α-chloroenaminones (3ea, 3ej, 3el, and 3ep) were produced in good yields under catalyst-free conditions. To demonstrate the practical utility of this strategy, we conducted scale-up reactions under optimal conditions (Scheme 2). A reaction between β-chloroenone 1a and aniline 2a on a 3.5 mmol scale yielded the desired product 3aa in 85% yield. Similarly, using aniline derivative 2d with 1a resulted in the isolation of product 3ad in 87% yield when the reaction was performed at 90 °C for 8 hours.



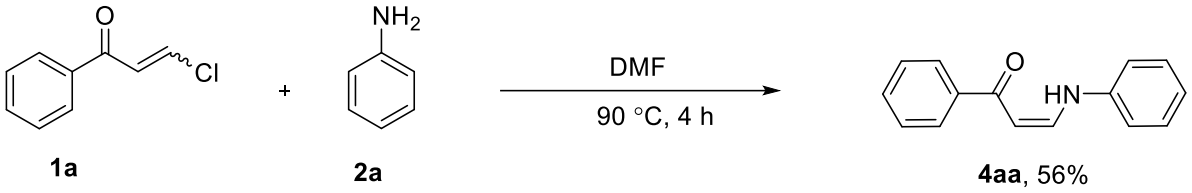
Scheme 2. Scale-up reactions

Table 3. Synthesis of 1,4-benzodiazepines



[a]Reactions were performed using **1** (0.5 mmol), **5** (0.5 mmol), in DMSO (3 mL) at room temperature. [b]The product **7** was formed in traces. [c]Isolated yield based on **1**.

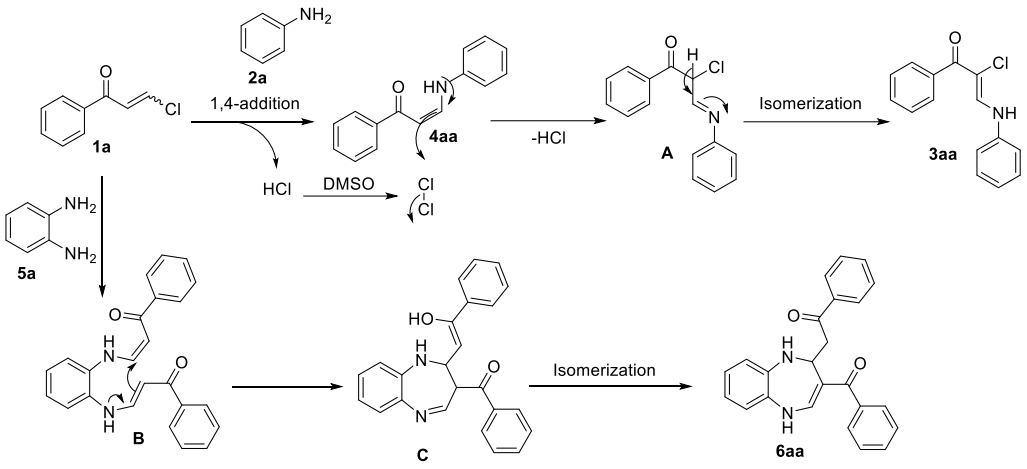
The reaction of β-chloroenone 1a with aniline 2a yielded enaminone 4aa as the product when performed in DMF at 90 °C (Scheme 3). Additionally, reactions conducted in alternative solvent systems consistently produced 4aa as the sole product (Table 1, entries 6–10). These results confirm that the use of DMSO as the solvent system is essential for the successful generation of α-chlorinated enaminone derivatives.



1a **2a** **4aa**, 56%

Scheme 3. Control experiment

A plausible reaction mechanism explaining the atom-economical formation of α-chloroenaminone is outlined in Scheme 4. Initially, the 1,4-addition of aniline 2a to β-chloroenone 1a produces enaminone 4aa, accompanied by the elimination of hydrochloric acid (HCl). The in-situ oxidation of HCl generates chlorine molecules (Cl_2), which react with 4aa to form the imine intermediate A. This imine then undergoes isomerization, yielding the thermodynamically stable product (Z)-3aa under the reaction conditions. In a parallel pathway, o-phenylenediamine 5a reacts with two molecules of 1a to form dienaminone intermediate B. Subsequent intramolecular cyclization of B produces intermediate C, which undergoes further transformations to yield the more stable product 6aa under ambient reaction conditions.



Scheme 4. Proposed reaction mechanism

Conclusions

In summary, we have developed a catalyst-free approach for synthesizing a range of (Z)-α-chloroenaminones. The method delivers excellent yields, particularly when anilines with electron-donating groups are used as reaction partners. High atom economy (>99%) was achieved using DMSO as both a solvent and an oxidizing agent. Anilines with electron-withdrawing groups were found to require longer reaction times to produce the desired products. Additionally, reactions involving o-phenylenediamine as the amine partner yielded benzodiazepine derivatives in moderate yields under catalyst-free conditions. This methodology enables the efficient generation of molecular diversity and holds great potential for applications in synthetic chemistry, given the widespread utility of enaminones as reactive intermediates.

Experimental section

General information

All reagents and solvents used in this study were obtained from commercial suppliers and used as received without further purification. Commercially sourced DMSO was employed directly in the reactions. The 10 mL round-bottom flasks were dried in an oven and cooled to room temperature prior to use. All reactions were conducted under a nitrogen atmosphere. Flash column

chromatography was performed on silica gel (63–200 mesh) using hexane and ethyl acetate as eluents. NMR spectra were recorded on a Bruker Ascend spectrometer at 400 MHz for ^{1}H and 100 MHz for ^{13}C. Chemical shifts are reported in parts per million (ppm) on the δ scale, with $CDCl_3$ as the internal standard. Signal multiplicities are denoted as follows: s = singlet, d = doublet, t = triplet, q = quartet, dd = doublet of doublets, m = multiplet, br = broad signal. Coupling constants are given in Hertz (Hz). High-resolution mass spectra (HRMS) were recorded in ESI-TOF mode. Melting points were determined using an Electro-Thermal FARGO MP-2D capillary melting point apparatus.

General Procedure for the Synthesis of β-chloroenones 1

To a 50 mL reaction flask, dried in an oven and cooled under a nitrogen atmosphere, were added the appropriate propargyl ketone (3 mmol), acetonitrile (20 mL), tert-butanol (2 mL), $AlCl_3$ (4.5 mmol, 599 mg), and NaBr (6 mmol, 617 mg). The reaction mixture was heated at 80°C for 16 hours, then allowed to cool to room temperature and quenched by adding water (5–10 mL). The mixture was then extracted with ethyl acetate (30 mL), and the aqueous phase was re-extracted with ethyl acetate (20 mL). The combined organic phases were washed with brine and dried over $MgSO_4$. After solvent evaporation under reduced pressure, the crude product was purified by column chromatography on silica gel, using a 1:99 ethyl acetate/hexane mixture as the eluent.

3-Chloro-1-phenylprop-2-en-1-one (1a)

The crude product was purified by column chromatography on silica gel using hexane/ethyl acetate = 99:1 (v/v) as eluent. Yield 85% (424 mg); Colorless liquid.^{1}H NMR (400 MHz, $CDCl_3$) δ 7.95-7.92 (m, 2.1H), 7.70 (d, J = 12.0 Hz, 0.08H), 7.62-7.58 (m, 1.1H), 7.52-7.45 (m, 3.1H), 7.32 (d, J = 12.0 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 187.8, 138.2, 136.8,

133.4, 128.8, 128.6, 128.5; HRMS (EI) m/z calcd. for C_9H_7ClO $[M]^+$ 166.0185, found 166.0181.

3-Chloro-1-(*p*-tolyl)prop-2-en-1-one (1b)

The crude product was purified by column chromatography on silica gel using hexane/ethyl acetate = 99:1 (v/v) as eluent. Yield 82% (443 mg); Colorless liquid.^{1}H NMR (400 MHz, $CDCl_3$) δ 7.85-7.82 (m, 2.5H), 7.67 (d, J = 12.0 Hz, 0.17H), 7.57 (d, J = 16.0 Hz, 0.17H), 7.45 (d, J = 12.0 Hz, 1H), 7.33-7.28 (m, 3.6H), 2.43 (s, 3.6H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 187.3, 144.5, 137.7, 134.3, 129.5, 128.8, 128.7, 128.5, 21.7; HRMS (EI) m/z calcd. for $C_{10}H_9ClO$ $[M]^+$ 180.0342, found 180.0338.

3-Chloro-1-(4-methoxyphenyl)prop-2-en-1-one (1c)

The crude product was purified by column chromatography on silica gel using hexane/ethyl acetate = 99:1 (v/v) as eluent. Yield 75% (441 mg); White solid, 53.5-55 °C.^{1}H NMR (400 MHz, $CDCl_3$) δ 7.95-7.91 (m, 2.3H), 7.66 (d, J = 12.0 Hz, 0.14H), 7.57 (d, J = 16.0 Hz, 0.14Hz), 7.43 (d, J = 16.0 Hz, 1H), 7.31 (d, J = 16.0 Hz, 1H), 6.98-6.94 (m, 2.3H), 3.88 (s, 3.4H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 186.1, 163.9, 137.3, 132.5, 131.0, 130.9, 129.7, 128.3, 114.0, 55.5; HRMS (EI) m/z calcd. for $C_{10}H_9ClO_2$ $[M]^+$ 196.0291, found 196.0284.

3-Chloro-1-(4-chlorophenyl)prop-2-en-1-one (1d)

The crude product was purified by column chromatography on silica gel using hexane/ethyl acetate = 99:1 (v/v) as eluent. Yield 52% (312 mg); White solid, melting point: 37.5-39 °C.^{1}H NMR (400 MHz, $CDCl_3$) δ 7.91-7.88 (m, 2.2H), 7.74 (d, J = 16.0 Hz, 0.19H), 7.56 (d, J = 16.0 Hz, 0.19H), 7.52-7.47 (m, 3H), 7.29 (d, J = 12.0 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 186.5, 186.4, 140.0, 138.8, 135.1, 134.8, 132.2, 130.0, 129.9, 128.2, 128.0; HRMS (ESI) m/z calcd. for $C_9H_7Cl_2O$ $[M+H]^+$ 200.9874, found 200.9874.

1-(4-Bromophenyl)-3-chloroprop-2-en-1-one (1e)

The crude product was purified by column chromatography on silica gel using hexane/ethyl acetate = 99:1 (v/v) as eluent. Yield 64% (469 mg); White solid, melting point: 41-42 °C.^{1}H NMR (400 MHz, $CDCl_3$) δ 7.83-7.80 (m, 2.2H), 7.75 (d, J = 12.0 Hz, 0.04H), 7.67-7.64 (m, 2.2H), 7.55 (d, J = 16.0 Hz, 0.06H), 7.50 (d, J = 12.0 Hz, 1H), 7.29 (d, J = 16.0 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 186.6, 138.8, 135.6, 132.2, 130.1, 130.0, 128.7, 128.0; HRMS (EI) m/z calcd. for C_9H_6ClBrO $[M]^+$ 243.9291, found 243.9287.

General Procedure for the Synthesis of α-Chloroenaminones 3

Aniline (0.25 mmol), β-chloroenone (0.3 mmol), and DMSO (2 mL) were added to a 10 mL reaction flask, previously dried in an oven and cooled to room temperature under a nitrogen atmosphere. The reaction mixture was heated at 90°C using an oil bath for 4–8 hours. After completion, the mixture was cooled to room temperature and transferred to a separatory funnel with the addition of ethyl acetate (30 mL). Water (30 mL) was then added, and the organic layer was separated. The aqueous phase was further extracted with ethyl acetate (20 mL). The combined organic layers were washed with brine and dried over $MgSO_4$. The solvent was evaporated under reduced pressure using a rotary evaporator. The crude product was purified by column chromatography on silica gel, using an ethyl acetate/hexane mixture as the eluent.

(*Z*)-2-Chloro-1-phenyl-3-(phenylamino)prop-2-en-1-one (3aa)

The crude product was purified by column chromatography on silica gel using hexane/ethyl acetate = 85:15 (v/v) as eluent. Yield 92% (59 mg); Yellow solid, melting point: 144-145 °C.^{1}H NMR (400 MHz, $CDCl_3$) δ 7.84 (d, J=12 Hz, 1H), 7.63-7.60 (m, 2H), 7.55-7.50 (m, 1H), 7.48-7.44 (m, 2H), 7.33-7.29 (m, 2H), 7.24 (d, J = 12 Hz, 1H), 7.10-7.06 (m, 1H), 6.93-6.90 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 188.1, 141.2, 139.2, 138.8, 131.1, 130.0,

128.5, 128.4, 128.1, 124.1, 116.4, 109.8; HRMS (ESI) m/z calcd. for $C_{15}H_{13}NOCl$ $[M+H]^+$ 258.0686, found 258.0691.

(*Z*)-1-Phenyl-3-(phenylamino)prop-2-en-1-one (4aa)

This product was obtained from a room-temperature reaction (Table 1, entry 1). The crude product was purified by column chromatography on silica gel using hexane/ethyl acetate = 85:15 (v/v) as eluent. Yield 58% (32 mg); Yellow solid, melting point: 138-139 °C.^{1}H NMR (400 MHz, $CDCl_3$) δ 12.18 (d, J = 12 Hz, 1H), 7.98-7.96 (m, 2H), 7.57-7.45 (m, 4H), 7.39-7.34 (m, 2H), 7.14-7.08 (m, 3H), 6.06 (d, J = 8.0 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 191.0, 144.9, 140.2, 139.2, 131.6, 129.7, 128.5, 127.3, 123.7, 116.3, 93.7; HRMS (ESI) m/z calcd. for $C_{15}H_{14}NO$ $[M+H]^+$ 224.1075, found 224.1081.

(*Z*)-2-Chloro-1-phenyl-3-(*o*-tolylamino)prop-2-en-1-one (3ab)

The crude product was purified by column chromatography on silica gel using hexane/ethyl acetate = 90:10 (v/v) as eluent. Yield 68% (46 mg); Yellow solid, melting point: 128-130 °C.^{1}H NMR (400 MHz, $CDCl_3$) δ 7.83 (d, J = 12.0 Hz, 1H), 7.63-7.61 (m, 2H), 7.54-7.50 (m, 1H), 7.47-7.44 (m, 2H), 7.20-7.14 (m, 2H), 7.07 (d, J = 12.0 Hz, 1H), 7.01 (t, J = 8.0 Hz, 1H), 6.84 (d, J = 8.0 Hz, 1H), 2.34 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 187.9, 141.8, 138.8, 137.5, 131.4, 131.0, 128.5, 128.4, 127.5, 126.2, 124.2, 115.2, 110.1, 17.2; HRMS (EI) m/z calcd. for $C_{16}H_{14}ClNO$ $[M]^+$ 271.0764, found 271.0758.

(*Z*)-2-Chloro-1-phenyl-3-(*m*-tolylamino)prop-2-en-1-one (3ac)

The crude product was purified by column chromatography on silica gel using hexane/ethyl acetate = 85:15 (v/v) as eluent. Yield 84% (57 mg); Yellow solid, melting point:104-105.5 °C.^{1}H NMR (400 MHz, $CDCl_3$) δ 7.83 (d, J = 12.0 Hz, 1H), 7.62-7.61 (d, J = 8.0 Hz, 2H), 7.54-7.51 (m, 1H), 7.46 (t, J = 8.0 Hz, 2H), 7.20-7.16 (m, 2H), 6.89 (d, J = 8.0 Hz, 1H);

^{13}C NMR (100 MHz, $CDCl_3$) δ 188.1, 141.3, 140.1, 139.1, 138.8, 131.0, 129.7, 128.5, 128.4, 125.0, 117.2, 113.3, 109.5, 21.4; HRMS (EI) m/z calcd. for $C_{16}H_{14}ClNO$ $[M]^+$ 271.0764, found 271.0780.

(*Z*)-2-Chloro-1-phenyl-3-(*p*-tolylamino)prop-2-en-1-one (3ad)

The crude product was purified by column chromatography on silica gel using hexane/ethyl acetate = 85:15 (v/v) as eluent. Yield 93% (63 mg); Yellow solid, melting point: 161-163 °C.^{1}H NMR (400 MHz, $CDCl_3$) δ 7.81 (d, J = 12.0 Hz, 1H), 7.62-7.59 (m, 2H), 7.53-7.49 (m, 1H), 7.46-7.42 (m, 2H), 7.17 (d, J = 16.0 Hz, 1H), 7.10 (d, J = 8.0 Hz, 2H), 6.83-6.80 (m, 2H), 2.29 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 188.0, 141.7, 138.9, 136.8, 133.9, 130.9, 130.4, 128.5, 128.4, 116.5, 109.3, 20.7; HRMS (EI) m/z calcd. for $C_{16}H_{14}ClNO$ $[M]^+$ 271.0764, found 271.0755.

(*Z*)-2-Chloro-3-((4-ethylphenyl)amino)-1-phenylprop-2-en-1-one (3ae)

The crude product was purified by column chromatography on silica gel using hexane/ethyl acetate = 85:15 (v/v) as eluent. Yield 80% (57 mg); Yellow solid, melting point: 146-147 °C.^{1}H NMR (400 MHz, $CDCl_3$) δ 7.84 (d, J = 12.0 Hz, 1H), 7.65-7.62 (m, 2H), 7.56-7.52 (m, 1H), 7.49-7.45 (m, 2H), 7.20-7.15 (m, 3H), 6.88-6.85 (m, 2H), 2.63 (q, J = 8.0 Hz, 2H), 1.23 (t, J = 8.0 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 188.0, 141.7, 140.4, 138.9, 136.9, 130.9, 129.2, 128.5, 128.4, 116.5, 109.3, 28.1, 15.6; HRMS (EI) m/z calcd. for $C_{17}H_{16}ClNO$ $[M]^+$ 285.0920, found 285.0913.

(*Z*)-2-Chloro-3-((4-isopropylphenyl)amino)-1-phenylprop-2-en-1-one (3af)

The crude product was purified by column chromatography on silica gel using hexane/ethyl acetate = 90:10 (v/v) as eluent. Yield 87% (65 mg); Yellow solid, melting point: 160-161 °C.^{1}H NMR (400 MHz, $CDCl_3$) δ 7.82 (d, J = 12.0 Hz, 1H), 7.62-7.59 (m, 2H), 7.54-7.49

(m, 1H), 7.47-7.42 (m, 2H), 7.19-7.13 (m, 3H), 6.87-6.84 (m, 2H), 2.86 (sep, J = 8.0 Hz, 1H), 1.21 (d, J = 8.0 Hz, 6H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 188.0, 145.1, 141.8, 138.9, 137.0, 130.9, 128.5, 128.4, 127.8, 116.5, 109.3, 33.4, 23.9; HRMS (EI) m/z calcd. for $C_{18}H_{18}ClNO$ $[M]^+$ 299.1077, found 299.1074.

(*Z*)-2-Chloro-3-((2,6-dimethylphenyl)amino)-1-phenylprop-2-en-1-one (3ag)

The crude product was purified by column chromatography on silica gel using hexane/ethyl acetate = 90:10 (v/v) as eluent. Yield 65% (46 mg); Yellow solid, melting point: 153-154 °C.^{1}H NMR (400 MHz, $CDCl_3$) δ 7.56-7.53 (m, 2H), 7.47-7.42 (m, 1H), 7.40-7.36 (m, 2H), 7.31 (d, J = 12.0 Hz, 1H), 7.07 (s, 3H), 6.66 (d, J = 12.0 Hz, 1H), 2.27 (s, 6H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 187.9, 148.2, 139.0, 137.0, 132.7, 130.7, 129.1, 128.3, 126.9, 107.9, 18.5; HRMS (EI) m/z calcd. for $C_{17}H_{16}ClNO$ $[M]^+$ 285.0920, found 285.0910.

(*Z*)-2-Chloro-3-((2,4-dimethylphenyl)amino)-1-phenylprop-2-en-1-one (3ah)

The crude product was purified by column chromatography on silica gel using hexane/ethyl acetate = 90:10 (v/v) as eluent. Yield 80% (57 mg); Yellow solid, melting point: 149-150 °C.^{1}H NMR (400 MHz, $CDCl_3$) δ 7.81 (d, J = 12.0 Hz, 1H), 7.65-7.62 (m, 2H), 7.56-7.51 (m, 1H), 7.49-7.45 (m, 2H), 7.09-6.97 (m, 3H), 6.77 (d, J = 8.0 Hz, 1H), 2.33 (s, 3H), 2.30 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 187.9, 142.4, 138.9, 135.2, 134.1, 132.0, 130.9, 128.5, 128.4, 127.9, 126.4, 115.7, 109.6, 20.6, 17.1; HRMS (EI) m/z calcd. for $C_{17}H_{16}ClNO$ $[M]^+$ 285.0920, found 285.0910.

(*Z*)-2-Chloro-3-((2-methoxyphenyl)amino)-1-phenylprop-2-en-1-one (3ai)

The crude product was purified by column chromatography on silica gel using hexane/ethyl acetate = 80:20 (v/v) as eluent. Yield 90% (65 mg); Yellow solid, melting point: 103-104 °C.^{1}H NMR (400 MHz, $CDCl_3$) δ 7.87 (d, J = 12.0 Hz, 1H), 7.73 (d, J = 12.0 Hz, 1H),

7.62-7.60 (m, 2H), 7.54-7.50 (m, 1H), 7.48-7.44 (m, 2H), 7.04-7.00 (m, 1H), 6.92-6.86 (m, 2H), 6.80 (dd, *J* = 1.2, 8.0 Hz, 1H), 3.92 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 188.1, 148.1, 140.4, 138.9, 130.9, 128.6, 128.5, 128.4, 123.7, 121.2, 113.4, 111.1, 110.1, 55.9; HRMS (ESI) m/z calcd. for $C_{16}H_{15}NO_2Cl$ $[M+H]^+$ 288.0791, found 288.0803.

(*Z*)-2-Chloro-3-((4-methoxyphenyl)amino)-1-phenylprop-2-en-1-one (3aj)

The crude product was purified by column chromatography on silica gel using hexane/ethyl acetate = 80:20 (v/v) as eluent. Yield 94% (68 mg); Yellow solid, melting point: 147-148 °C.^{1}H NMR (400 MHz, $CDCl_3$) δ 7.77 (d, *J* = 12.0 Hz, 1H), 7.63-7.61 (m, 2H), 7.55-7.45 (m, 3H), 7.19 (d, *J* = 12.0 Hz, 1H), 6.92-6.87 (m, 4H), 3.80 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 187.9, 156.6, 142.4, 138.9, 132.7, 130.9, 128.4, 128.4, 118.2, 115.1, 108.8, 55.6; HRMS (ESI) m/z calcd. for $C_{16}H_{15}NO_2Cl$ $[M+H]^+$ 288.0791, found 288.0790.

(*Z*)-2-Chloro-3-((2-nitrophenyl)amino)-1-phenylprop-2-en-1-one (3ak)

The crude product was purified by column chromatography on silica gel using hexane/ethyl acetate = 80:20 (v/v) as eluent. Yield 40% (30 mg); Yellow solid, melting point: 137-138 °C.^{1}H NMR (400 MHz, $CDCl_3$) δ 10.56 (d, *J* = 12.0 Hz, 1H), 8.29 (dd, *J* = 8.0, 1.2 Hz, 1H), 7.98 (d, *J* = 12.0 Hz, 1H), 7.72-7.69 (m, 2H), 7.63-7.57 (m, 2H), 7.53-7.49 (m, 2H)7.16-7.12 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 188.4, 138.1, 137.4, 136.5, 136.3, 135.7, 131.7, 128.7, 128.5, 127.1, 122.2, 115.7, 114.3; HRMS (EI) m/z calcd. for $C_{15}H_{12}N_2O_3Cl$ $[M+H]^+$ 303.0536, found 303.0542.

(*Z*)-2-Chloro-3-((3-nitrophenyl)amino)-1-phenylprop-2-en-1-one (3al)

The crude product was purified by column chromatography on silica gel using hexane/ethyl acetate = 80:20 (v/v) as eluent. Yield 79% (60 mg); Yellow solid, melting point: 188-189 °C.^{1}H NMR (400 MHz, $CDCl_3$) δ 9.90 (d, *J* = 12.0 Hz, 1H), 8.13-8.12 (m, 1H), 7.95 (d, *J* = 12.0 Hz, 1H), 7.87-

7.83 (m, 1H), 7.68-7.65 (m, 2H), 7.61-7.50 (m, 5H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 187.8, 148.9, 142.9, 142.2, 139.1, 131.6, 131.2, 129.0, 128.9, 123.0, 117.8, 112.4, 109.3; HRMS (EI) m/z calcd. for $C_{15}H_{12}N_2O_3Cl$ $[M+H]^+$ 303.0536, found 303.0539.

(*Z*)-2-Chloro-3-((4-nitrophenyl)amino)-1-phenylprop-2-en-1-one (3am)

The crude product was purified by column chromatography on silica gel using hexane/ethyl acetate = 80:20 (v/v) as eluent. Yield 62% (47 mg); Yellow solid, melting point: 255-256 °C.^{1}H NMR (400 MHz, $CDCl_3$) δ 10.04 (brs, 1H), 8.20-8.16 (m, 2H), 7.96 (brs, 1H), 7.69-7.66 (m, 2H), 7.63-7.59 (m, 1H), 7.54-7.51 (m, 2H), 7.41-7.38 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 188.0, 146.9, 142.5, 141.8, 138.8, 131.8, 129.0, 129.0, 126.0, 117.1, 1108; HRMS (EI) m/z calcd. for $C_{15}H_{12}N_2O_3Cl$ $[M+H]^+$ 303.0536, found 303.0541.

(*Z*)-2-Chloro-3-((2-chlorophenyl)amino)-1-phenylprop-2-en-1-one (3an)

The crude product was purified by column chromatography on silica gel using hexane/ethyl acetate = 85:15 (v/v) as eluent. Yield 51% (37 mg); Yellow solid, melting point: 128-129 °C.^{1}H NMR (400 MHz, $CDCl_3$) δ 7.86 (d, J = 12.0 Hz, 1H), 7.71-7.65 (m, 3H), 7.59-7.54 (m, 1H), 7.51-7.47 (m, 2H), 7.42 (dd, J = 8.0, 1.2 Hz, 1H), 7.26-7.21 (m, 1H), 7.02 (dd, J = 8.0 1.2 Hz, 1H), 6.92 (dd, J = 8.0, 1.2 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 188.1, 139.5, 138.5, 135.8, 131.3, 130.2, 128.6, 128.5, 128.3, 124.0, 122.5, 114.8, 111.5; HRMS (EI) m/z calcd. for $C_{15}H_{12}NOCl_2$ $[M+H]^+$ 292.0296, found 292.0284.

(*Z*)-2-Chloro-3-((3-chlorophenyl)amino)-1-phenylprop-2-en-1-one (3ao)

The crude product was purified by column chromatography on silica gel using hexane/ethyl acetate = 85:15 (v/v) as eluent. Yield 63% (46 mg); Yellow solid, melting point: 123-124 °C.^{1}H NMR (400 MHz, $CDCl_3$) δ 7.72 (d, J = 12.0 Hz, 1H), 7.57-7.55 (m, 2H), 7.50-7.46 (m, 1H), 7.43-7.38 (m, 2H), 7.15 (d, J = 8.0 Hz, 1H), 7.06 (d, J = 12.0 Hz, 1H), 6.99-6.96

(m, 1H), 6.85 (t, J = 2.0 Hz, 1H), 6.74-6.71 (m, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 188.1, 140.4, 140.1, 138.4, 135.7, 131.3, 130.9, 128.56, 128.53, 124.0, 116.4, 114.3, 110.7; HRMS (EI) m/z calcd. for $C_{15}H_{12}NOCl_2$ $[M+H]^+$ 292.0296, found 292.0305.

(*Z*)-2-Chloro-3-((4-chlorophenyl)amino)-1-phenylprop-2-en-1-one (3ap)

The crude product was purified by column chromatography on silica gel using hexane/ethyl acetate = 85:15 (v/v) as eluent. Yield 82% (60 mg); Yellow solid, melting point: 179-180 °C.^{1}H NMR (400 MHz, $CDCl_3$) δ 7.79 (d, J = 12.0 Hz, 1H), 7.65-7.62 (m, 2H), 7.58-7.54 (m, 1H), 7.50-7.46 (m, 2H), 7.30-7.24 (m, 3H), 6.90-6.87 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 188.1, 140.7, 138.6, 137.9, 131.2, 129.9, 129.2, 128.53, 128.50, 117.5, 110.2; HRMS (EI) m/z calcd. for $C_{15}H_{12}NOCl_2$ $[M+H]^+$ 292.0296, found 292.0299.

(*Z*)-3-((4-Bromophenyl)amino)-2-chloro-1-phenylprop-2-en-1-one (3aq)

The crude product was purified by column chromatography on silica gel using hexane/ethyl acetate = 85:15 (v/v) as eluent. Yield 85% (71 mg); Yellow solid, melting point: 192-193 °C.^{1}H NMR (400 MHz, $CDCl_3$) δ 7.79 (d, J = 12.0 Hz, 1H), 7.65-7.62 (m, 2H), 7.58-7.54 (m, 1H), 7.50-7.47 (m, 2H), 7.45-7.41 (m, 2H), 7.21 (d, J = 12.0 Hz, 1H), 6.85-6.81 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 188.1, 140.5, 138.5, 138.3, 132.9, 131.2, 128.54, 128.50, 117.8, 116.6, 110.3; HRMS (EI) m/z calcd. for $C_{15}H_{12}NOClBr$ $[M+H]^+$ 335.9791, found 335.9792.

(*Z*)-3-(Benzylamino)-2-chloro-1-phenylprop-2-en-1-one (3ar)

The crude product was purified by column chromatography on silica gel using hexane/ethyl acetate = 90:10 (v/v) as eluent. Yield 50% (34 mg); Yellow solid, melting point: 133-134 °C.^{1}H NMR (400 MHz, $CDCl_3$) δ 7.53-7.50 (m, 2H), 7.49-7.45 (m, 1H), 7.42-7.33 (m, 6H), 7.26-7.23 (m, 2H), 5.71 (t, J = 4.0 Hz, 1H), 4.44 (d, J = 4.0 Hz, 2H); ^{13}C NMR (100 MHz,

$CDCl_3$) δ 187.6, 149.6, 139.2, 136.9, 130.4, 129.1, 128.3, 128.29, 128.26, 127.3, 106.5, 51.9; HRMS (EI) m/z calcd. for $C_{16}H_{15}NOCl$ $[M+H]^+$ 272.0842, found 272.0849.

(*Z*)-2-Chloro-3-(phenethylamino)-1-phenylprop-2-en-1-one (3as)

The crude product was purified by column chromatography on silica gel using hexane/ethyl acetate = 90:10 (v/v) as eluent. Yield 36% (26 mg); Yellow solid, melting point: 169-170 °C.^{1}H NMR (400 MHz, $CDCl_3$) δ 7.42 (t, J = 8.0 Hz, 1H), 7.35-7.25 (m, 6H), 7.14 (d, J = 8.0 Hz, 2H), 7.01 (d, J = 16.0 Hz, 1H), 5.45 (t, J = 8.0 Hz, 1H), 3.50 (q, J = 8.0 Hz, 2H), 2.84 (t, J = 8.0 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 187.4, 150.1, 139.2, 137.4, 130.2, 128.99, 128.95, 128.26, 128.19, 127.0, 105.9, 49.8, 37.6; HRMS (EI) m/z calcd. for $C_{17}H_{16}NOCl$ $[M]^+$ 285.0920, found 285.0912.

(*Z*)-2-Chloro-3-(phenylamino)-1-(*p*-tolyl)prop-2-en-1-one (3ba)

The crude product was purified by column chromatography on silica gel using hexane/ethyl acetate = 85:15 (v/v) as eluent. Yield 75% (51 mg); Yellow solid, melting point: 142-143 °C.^{1}H NMR (400 MHz, $CDCl_3$) δ 7.87 (d, J = 12.0 Hz, 1H), 7.54-7.52 (m, 2H), 7.33-7.31 (m, 2H), 7.29-7.25 (m, 2H), 7.18 (d, J = 12.0 Hz, 1H), 7.10-7.05 (m, 1H), 6.94-6.91 (m, 2H), 2.43 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 188.0, 141.6, 140.8, 139.3, 135.9, 129.9, 129.1, 128.7, 123.9, 116.3, 109.8, 21.5; HRMS (EI) m/z calcd. for $C_{16}H_{15}NOCl$ $[M+H]^+$ 272.0842, found 272.0833.

(*Z*)-2-Chloro-3-((4-methoxyphenyl)amino)-1-(*p*-tolyl)prop-2-en-1-one (3bj)

The crude product was purified by column chromatography on silica gel using hexane/ethyl acetate = 85:15 (v/v) as eluent. Yield 70% (53 mg); Yellow solid, melting point: 160-161 °C.^{1}H NMR (400 MHz, $CDCl_3$) δ 7.77 (d, J = 12.0 Hz, 1H), 7.52-7.50 (m, 2H), 7.26-7.24 (m, 2H), 7.12 (d, J = 12.0 Hz, 1H), 6.91-6.84 (m, 4H), 3.78 (s, 3H), 2.42 (s, 3H); ^{13}C NMR

(100 MHz, $CDCl_3$) δ 187.8, 156.5, 142.0, 141.3, 136.1, 132.8, 129.0, 128.6, 118.1, 115.1, 108.9, 55.6, 21.5; HRMS (EI) m/z calcd. for $C_{17}H_{17}NO_2Cl$ $[M+H]^+$ 302.0948, found 302.0947.

(*Z*)-2-Chloro-3-((3-nitrophenyl)amino)-1-(*p*-tolyl)prop-2-en-1-one (3bl)

The crude product was purified by column chromatography on silica gel using hexane/ethyl acetate = 85:15 (v/v) as eluent. Yield 60% (47 mg); Yellow solid, melting point: 223-224 °C.^{1}H NMR (400 MHz, $CDCl_3$) δ 9.84 (d, J = 12.0 Hz, 1H), 815-8.14 (m, 1H), 7.97 (d, J = 12.0 Hz, 1H), 7.85 (dt, J = 8.0, 4.0 Hz, 1H), 7.61-7.56 (m, 4H), 7.32 (d, J = 8.0 Hz, 2H), 2.40 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 187.6, 148.9, 142.4, 142.3, 141.6, 136.3, 131.3, 129.4, 129.2, 122.9, 117.7, 112.4, 109.4, 21.5; HRMS (EI) m/z calcd. for $C_{16}H_{14}N_2O_3Cl$ $[M+H]^+$ 317.0693, found 317.0696.

(*Z*)-2-Chloro-3-((4-chlorophenyl)amino)-1-(*p*-tolyl)prop-2-en-1-one (3bp)

The crude product was purified by column chromatography on silica gel using hexane/ethyl acetate = 85:15 (v/v) as eluent. Yield 63% (48 mg); Yellow solid, melting point: 189-190 °C.^{1}H NMR (400 MHz, $CDCl_3$) δ 7.89 (d, J = 16.0 Hz, 1H), 7.55-7.52 (m, 2H), 7.28-7.25 (m, 4H), 7.17 (d, J = 12.0 Hz, 1H), 6.89-6.86 (m, 2H), 2.43 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 188.0, 141.8, 140.2, 138.0, 135.7, 129.9, 129.1, 129.0, 128.7, 117.4, 110.3, 21.5; HRMS (EI) m/z calcd. for $C_{16}H_{14}NOCl_2$ $[M+H]^+$ 306.0452, found 306.0450.

(*Z*)-2-Chloro-1-(4-methoxyphenyl)-3-(phenylamino)prop-2-en-1-one (3ca)

The crude product was purified by column chromatography on silica gel using hexane/ethyl acetate = 80:20 (v/v) as eluent. Yield 78% (56 mg); Yellow solid, melting point: 125-127 °C.^{1}H NMR (400 MHz, $CDCl_3$) δ 7.87 (d, J = 12.0 Hz, 1H), 7.66-7.62 (m, 2H), 7.34-7.29 (m, 2H), 7.14 (d, J = 12.0 Hz, 1H), 7.09-7.05 (m, 1H), 6.98-6.92 (m, 4H), 3.88 (s, 3H); ^{13}C

NMR (100 MHz, $CDCl_3$) δ 187.3, 162.1, 140.4, 139.3, 131.0, 130.7, 129.9, 123.9, 116.2, 113.7, 109.6, 55.4; HRMS (EI) m/z calcd. for $C_{16}H_{15}NO_2Cl$ $[M+H]^+$ 288.0791, found 288.0800.

(*Z*)-2-Chloro-1-(4-methoxyphenyl)-3-((4-methoxyphenyl)amino)prop-2-en-1-one (3cj)

The crude product was purified by column chromatography on silica gel using hexane/ethyl acetate = 75:25 (v/v) as eluent. Yield 72% (57 mg); Yellow solid, melting point: 162-163 °C.^{1}H NMR (400 MHz, $CDCl_3$) δ 7.80 (d, J = 12.0 Hz, 1H), 7.66-7.62 (m, 2H), 7.10 (d, J = 12.0 Hz, 1H), 6.99-6.95 (m, 2H), 6.94-6.89 (m, 4H), 3.89 (s, 3H), 3.80 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 187.1, 161.9, 156.5, 141.5, 132.9, 131.3, 130.6, 118.1, 115.1, 113.7, 108.7, 55.6, 55.4; HRMS (EI) m/z calcd. for $C_{17}H_{16}NO_3Cl$ $[M]^+$ 317.0819, found 317.0812.

(*Z*)-2-Chloro-1-(4-methoxyphenyl)-3-((3-nitrophenyl)amino)prop-2-en-1-one (3cl)

The crude product was purified by column chromatography on silica gel using hexane/ethyl acetate = 80:20 (v/v) as eluent. Yield 55% (46 mg); Yellow solid, melting point: 169-170 °C.^{1}H NMR (400 MHz, $CDCl_3$) δ 7.94-7.88 (m, 2H), 7.82 (t. J = 4.0 Hz, 1H), 7.73-7.69 (m, 2H), 7.52 (t, J = 8.0 Hz, 1H), 7.34-7.27 (m, 2H), 7.03-6.99 (m, 2H), 3.91 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 187.3, 162.6, 149.4, 140.8, 138.2, 130.9, 130.8, 130.4, 121.5, 117.9, 113.9, 111.5, 110.7, 55.5; HRMS (EI) m/z calcd. for $C_{16}H_{14}N_2O_4Cl$ $[M+H]^+$ 333.0642, found 333.0645.

(*Z*)-2-Chloro-3-((4-chlorophenyl)amino)-1-(4-methoxyphenyl)prop-2-en-1-one (3cp)

The crude product was purified by column chromatography on silica gel using hexane/ethyl acetate = 80:20 (v/v) as eluent. Yield 65% (52 mg); Yellow solid, melting point: 174-175 °C.^{1}H NMR (400 MHz, $CDCl_3$) δ 7.81 (d, J = 12.0 Hz, 1H), 7.68-7.65 (m, 2H), 7.32-7.28 (m, 2H), 7.12 (d, J = 16.0 Hz, 1H), 7.00-6.96 (m, 2H), 6.92-6.88 (m, 2H), 3.90 (s, 3H); ^{13}C

NMR (100 MHz, $CDCl_3$) δ 187.2, 162.3, 139.5, 138.1, 130.8, 130.7, 129.9, 128.9, 117.3, 113.7, 110.1, 55.4; HRMS (EI) m/z calcd. for $C_{16}H_{13}NO_2Cl_2$ $[M]^+$ 321.0323, found 321.0318.

(*Z*)-2-Chloro-1-(4-chlorophenyl)-3-(phenylamino)prop-2-en-1-one (3da)

The crude product was purified by column chromatography on silica gel using hexane/ethyl acetate = 85:15 (v/v) as eluent. Yield 80% (58 mg); Yellow solid, melting point: 170-171 °C.^{1}H NMR (400 MHz, $CDCl_3$) δ 7.85 (d, J = 12.0 Hz, 1H), 7.61-7.57 (m, 2H), 7.48-7.44 (m, 2H), 7.37-7.30 (m, 3H), 7.15-7.11 (m, 1H), 6.98-6.95 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 186.8, 141.1, 139.0, 137.3, 137.1, 129.99, 129.97, 128.7, 124.3, 116.4, 109.3; HRMS (EI) m/z calcd. for $C_{15}H_{12}NOCl_2$ $[M+H]^+$ 292.0296, found 292.0301.

(*Z*)-2-Chloro-1-(4-chlorophenyl)-3-((4-methoxyphenyl)amino)prop-2-en-1-one (3dj)

The crude product was purified by column chromatography on silica gel using hexane/ethyl acetate = 85:15 (v/v) as eluent. Yield 75% (60 mg); Yellow solid, melting point: 173-174 °C.^{1}H NMR (400 MHz, $CDCl_3$) δ 7.74 (d, J = 16.0 Hz, 1H), 7.59-7.57 (m, 2H), 7.46-7.43 (m, 2H), 7.17 (d, J = 12.0 Hz, 1H), 6.93-6.88 (m, 4H), 3.81 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 186.6, 156.8, 142.3, 137.3, 137.1, 132.5, 129.9, 128.7, 118.3, 115.2, 108.5, 55.6; HRMS (EI) m/z calcd. for $C_{16}H_{14}NO_2Cl_2$ $[M+H]^+$ 322.0402, found 322.0405.

(*Z*)-2-Chloro-1-(4-chlorophenyl)-3-((3-nitrophenyl)amino)prop-2-en-1-one (3dl)

The crude product was purified by column chromatography on silica gel using hexane/ethyl acetate = 85:15 (v/v) as eluent. Yield 47% (39 mg); Yellow solid, melting point: 200-201 °C.^{1}H NMR (400 MHz, $CDCl_3$) δ 9.90 (d, J = 12.0 Hz, 1H), 8.19 (t, J = 4.0 Hz, 1H), 7.88-7.85 (m, 1H), 7.71-7.65 (m, 3H), 7.60-7.55 (m, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 186.6,

148.9, 143.2, 142.1, 137.9, 136.3, 131.2, 130.9, 128.9, 123.2, 117.9, 112.7, 109.1; HRMS (EI) m/z calcd. for $C_{15}H_{11}N_2O_3Cl_2$ $[M+H]^+$ 337.0147, found 337.0147.

(*Z*)-2-Chloro-1-(4-chlorophenyl)-3-((4-chlorophenyl)amino)prop-2-en-1-one (3dp)

The crude product was purified by column chromatography on silica gel using hexane/ethyl acetate = 85:15 (v/v) as eluent. Yield 69% (56 mg); Yellow solid, melting point: 199-200.5 °C.^{1}H NMR (400 MHz, $CDCl_3$) δ 9.69 (d, J = 12.0 Hz, 1H), 7.84 (d, J = 12.0 Hz, 1H), 7.66-7.63 (m, 2H), 7.56-7.53 (m, 2H), 7.38-7.34 (m, 2H), 7.28-7.24 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 186.3, 143.8, 139.8, 138.1, 136.0, 130.8, 129.7, 128.9, 127.8, 119.4, 108.1; HRMS (EI) m/z calcd. for $C_{15}H_{11}NOCl_3$ $[M+H]^+$ 325.9906, found 325.9912.

(*Z*)-1-(4-Bromophenyl)-2-chloro-3-(phenylamino)prop-2-en-1-one (3ea)

The crude product was purified by column chromatography on silica gel using hexane/ethyl acetate = 85:15 (v/v) as eluent. Yield 91% (76 mg); Yellow solid, melting point: 179-180 °C.^{1}H NMR (400 MHz, $CDCl_3$) δ 7.82 (d, J = 12.0 Hz, 1H), 7.62-7.59 (m, 2H), 7.52-7.48 (m, 2H), 7.36-7.32 (m, 2H), 7.23 (d, J = 12.0 Hz, 1H), 6.95-6.93 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 186.9, 141.1, 138.9, 137.5, 131.7, 130.1, 125.7, 124.4, 116.4, 109.4; HRMS (EI) m/z calcd. for $C_{15}H_{11}NOClBr$ $[M]^+$ 334.9713, found 334.9703.

(*Z*)-1-(4-Bromophenyl)-2-chloro-3-((4-methoxyphenyl)amino)prop-2-en-1-one (3ej)

The crude product was purified by column chromatography on silica gel using hexane/ethyl acetate = 80:20 (v/v) as eluent. Yield 87% (79 mg); Yellow solid, melting point: 188-189 °C.^{1}H NMR (400 MHz, $CDCl_3$) δ 7.72 (d, J = 12.0 Hz, 1H), 7.60-7.57 (m, 2H), 7.50-7.46 (m, 2H), 7.18 (d, J = 12.0 Hz, 1H), 6.91-6.85 (m, 4H), 3.78 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 186.7, 156.8, 142.3, 137.7, 132.5, 131.6, 130.1, 125.5, 118.3, 115.2, 108.5, 55.6; HRMS (EI) m/z calcd. for $C_{16}H_{13}NO_2ClBr$ $[M]^+$ 364.9818, found 364.9812.

(*Z*)-1-(4-Bromophenyl)-2-chloro-3-((3-nitrophenyl)amino)prop-2-en-1-one (3el)

The crude product was purified by column chromatography on silica gel using hexane/ethyl acetate = 80:20 (v/v) as eluent. Yield 72% (68 mg); Yellow solid, melting point: 197-198 °C.^{1}H NMR (400 MHz, $CDCl_3$) δ 9.91 (s, 1H), 8.20 (t, J = 2.0 Hz, 1H), 7.99 (s, 1H), 7.88-7.85 (m, 1H), 7.72-7.56 (m, 6H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 186.7, 148.9, 143.2, 142.1, 138.2, 131.9, 131.2, 131.1, 125.2, 123.2, 117.9, 112.7, 109.0; HRMS (EI) m/z calcd. for $C_{15}H_{10}N_2O_3ClBr$ $[M]^+$ 379.9563, found 379.9554.

(*Z*)-1-(4-Bromophenyl)-2-chloro-3-((4-chlorophenyl)amino)prop-2-en-1-one (3ep)

The crude product was purified by column chromatography on silica gel using hexane/ethyl acetate = 85:15 (v/v) as eluent. Yield 76% (70 mg); Yellow solid, melting point: 209-210 °C.^{1}H NMR (400 MHz, $CDCl_3$) δ 9.69 (d, J = 12.0 Hz, 1H), 7.84 (d, J = 16.0 Hz, 1H), 7.70-7.67 (m, 2H), 7.59-7.56 (m, 2H), 7.38-7.34 (m, 2H), 7.28-7.24 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 186.4, 143.7, 139.8, 138.5, 131.9, 130.9, 129..7, 127.8, 124.9, 119.4, 108.1; HRMS (EI) m/z calcd. for $C_{15}H_{11}NOCl_2Br$ $[M+H]^+$ 369.9401, found 369.9404.

General procedure for the scale-up synthesis of 3aa and 3ad

Aniline (2a or 2d, 3.5 mmol), β-chloroenone (1a, 4.2 mmol, 0.697 g), and DMSO (20 mL) were added to a 100 mL reaction flask, which had been dried in an oven and cooled to room temperature under a nitrogen atmosphere. The reaction mixture was heated at 90°C using an oil bath for 8 hours. Upon completion, the mixture was cooled to room temperature and transferred to a separatory funnel with the addition of ethyl acetate (60 mL). Water (50 mL) was added, and the organic layer was separated. The aqueous layer was extracted with ethyl acetate (30 mL). The combined organic layers were washed with brine and dried over $MgSO_4$. The solvent was then removed under reduced pressure using a rotary evaporator. The crude product was purified by

silica gel column chromatography using an ethyl acetate/hexane mixture as the eluent.

General procedure for the Synthesis of 1,4-benzodiazepine derivatives

To a 10 mL reaction flask, oven-dried and cooled under a nitrogen atmosphere, were added o-phenylenediamine (0.5 mmol), β-chloroenone (0.5 mmol), and DMSO (3 mL). The reaction mixture was stirred at room temperature for 12 hours. After completion, the mixture was transferred to a separatory funnel, and ethyl acetate (30 mL) was added. Water (30 mL) was then introduced, and the organic layer was separated. The aqueous phase was extracted with ethyl acetate (20 mL). The combined organic layers were washed with brine and dried over $MgSO_4$. The solvent was removed under reduced pressure using a rotary evaporator, and the crude product was purified by silica gel column chromatography, using an ethyl acetate/hexane mixture as the eluent.

2-(3-Benzoyl-2,5-dihydro-1*H*-benzo[*b*][1,4]diazepin-2-yl)-1-phenylethan-1-one 6aa

The crude product was purified by column chromatography on silica gel using hexane/ethyl acetate = 80:20 (v/v) as eluent. Yield 40% (37 mg); Yellow solid, melting point: 102-103 °C.^{1}H NMR (400 MHz, DMSO-d_6) δ 9.45 (d, J = 8.0 Hz, 1H), 7.94-7.91 (m, 2H), 7.60 (t, J = 8.0 Hz, 1H), 7.50-7.39 (m, 7H), 7.01 (d, J = 8.0 Hz, 1H), 6.89-6.85 (m, 2H), 6.78-6.72 (m, 2H), 6.69-6.66 (m, 2H), 5.72 (d, J = 4.0 Hz, 1H), 5.11-5.06 (m, 1H), 3.11-2.98 (m, 2H); ^{13}C NMR (100 MHz, DMSO-d_6) δ 199.2, 192.4, 146.7, 141.0, 137.7, 137.0, 133.6, 131.4, 129.9, 129.0, 128.5, 128.4, 123.8, 122.1, 121.0, 120.3, 116.0, 51.5, 45.6; HRMS (EI) m/z calcd. for $C_{24}H_{20}N_2O_2$ $[M]^+$ 368.1525, found 368.1537.

2-(3-Benzoyl-7,8-dimethyl-2,5-dihydro-1*H*-benzo[*b*][1,4]diazepin-2-yl)-1-phenylethan-1-one 6ab

The crude product was purified by column chromatography on silica gel using hexane/ethyl acetate = 80:20 (v/v) as eluent. Yield 42% (44 mg); Yellow solid, melting point: 113-114

°C.^{1}H NMR (400 MHz, $CDCl_3$) δ 7.85 (d, J = 4.0 Hz, 2H), 7.50 (t, J = 8.0 Hz, 1H), 7.46-7.34 (m, 6H), 7.30-7.26 (m, 2H), 7.05 (d, J = 8.0 Hz, 1H), 6.45 (s, 1H), 6.37 (s, 1H), 5.17 (dd, J = 8.0, 4.0 Hz, 1H), 4.98 (brs, 1H), 3.32 (dd, J = 16.0, 4.0 Hz, 1H), 2.87 (dd, J = 12.0, 16.0 Hz, 1H), 2.00 (s, 3H), 1.96 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 200.0, 193.9, 146.9, 140.3, 136.8, 134.0, 133.1, 132.9, 130.0, 129.7, 128.5, 128.4, 128.3, 128.0, 123.4, 121.1, 115.7, 52.2, 43.8, 18.8, 18.7; HRMS (EI) m/z calcd. for $C_{26}H_{24}N_2O_2$ $[M]^+$ 396.1838, found 396.1834.

2-(3-(4-Methylbenzoyl)-2,5-dihydro-1*H*-benzo[*b*][1,4]diazepin-2-yl)-1-(*p*-tolyl)ethan-1-one 6ba

The crude product was purified by column chromatography on silica gel using hexane/ethyl acetate = 80:20 (v/v) as eluent. Yield 39% (39 mg); Yellow solid, melting point: 67-68 °C.1H NMR (400 MHz, DMSO-d_6) δ 9.38 (d, J = 8.0 Hz, 1H), 7.81 (d, J = 8.0 Hz, 2H), 7.32-7.23 (m, 6H), 7.02 (d, J = 8.0 Hz, 1H), 6.87-6.85 (m, 2H), 6.76-6.70 (m, 2H), 6.66-6.63 (m, 1H), 5.67 (d, J = 4.0 Hz, 1H), 5.05 (quint, J = 4.0 Hz, 1H), 3.06-2.95 (m, 2H), 2.35 (s, 3H), 2.34 (s, 3H); ^{13}C NMR (100 MHz, DMSO-d_6) δ 198.7, 192.4, 146.4, 143.9, 139.6, 138.2, 137.7, 134.6, 131.5, 129.6, 128.9, 128.6, 123.7, 122.1, 120.9, 120.2, 116.1, 51.5, 45.4, 21.6, 21.4; HRMS (EI) m/z calcd. for $C_{26}H_{24}N_2O_2$ $[M]^+$ 396.1838, found 396.1850.

2-(7,8-Dimethyl-3-(4-methylbenzoyl)-2,5-dihydro-1H-benzo[b][1,4]diazepin-2-yl)-1-(p-tolyl)ethan-1-one 6bb

The crude product was purified by column chromatography on silica gel using hexane/ethyl acetate = 80:20 (v/v) as eluent. Yield 37% (39 mg); Yellow solid, melting point: 108-109 °C.^{1}H NMR (400 MHz, DMSO-d_6) δ 9.23 (d, J = 8.0 Hz, 1H), 7.83-7.80 (m, 2H), 7.28-7.22 (m, 6H), 6.97 (d, J = 8.0 Hz, 1H), 6.61 (s, 1H), 6.41 (s, 1H), 5.41 (brs, 1H), 5.1-4.96

(m, 1H), 3.05-2.90 (m, 2H), 2.36 (s, 6H), 2.05 (s, 3H), 1.97 (s, 3H); ^{13}C NMR (100 MHz, DMSO-d_6) δ 198.9, 192.2, 146.4, 143.8, 139.4, 138.4, 135.2, 134.7, 131.5, 130.1, 129.5, 128.9, 128.6, 128.5, 128.4, 123.1, 121.2, 115.7, 51.6, 45.4, 21.6, 21.4, 19.0, 18.8; HRMS (EI) m/z calcd. for $C_{28}H_{28}N_2O_2$ $[M]^+$ 424.2151, found 424.2136.

2-(3-(4-Methoxybenzoyl)-2,5-dihydro-1H-benzo[b][1,4]diazepin-2-yl)-1-(4-methoxyphenyl)ethan-1-one 6ca

The crude product was purified by column chromatography on silica gel using hexane/ethyl acetate = 60:40 (v/v) as eluent. Yield 41% (44 mg); Yellow solid, melting point: 110-111 °C.^{1}H NMR (400 MHz, $CDCl_3$) δ 7.85-7.83 (m, 2H), 7.65-7.52 (m, 1H), 7.45-7.43 (m, 2H), 7.13 (d, J = 8.0 Hz, 1H), 6.84-6.81 (m, 4H), 6.76-6.69 (m, 3H), 6.62-6.61 (m, 1H), 5.16 (dd, J = 1.7, 12.0 Hz, 1H), 4.98 (brs, 1H), 3.80 (s, 6H), 3.33 (d, J = 12.0 Hz, 1H), 2.82 (dd, J = 12.0, 16.0 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 198.3, 193.4, 163.5, 161.2, 146.0, 136.4, 132.4, 131.2, 130.6, 130.3, 129.8, 124.1, 122.4, 121.7, 119.9, 116.0, 113.6, 113.4, 55.4, 55.3, 52.5, 43.7; HRMS (EI) m/z calcd. for $C_{26}H_{24}N_2O_4$ $[M]^+$ 428.1736, found 428.1731.

2-(3-(4-Methoxybenzoyl)-7,8-dimethyl-2,5-dihydro-1H-benzo[b][1,4]diazepin-2-yl)-1-(4-methoxyphenyl)ethan-1-one 6cb

The crude product was purified by column chromatography on silica gel using hexane/ethyl acetate = 60:40 (v/v) as eluent. Yield 38% (44 mg); Yellow solid, melting point: 115-116 °C.^{1}H NMR (400 MHz, DMSO-d_6) δ 9.23 (d, J = 8.0 Hz, 1H), 7.93-7.89 (m, 2H), 7.42-7.38 (m, 2H), 7.03 (d, J = 8.0 Hz, 1H), 7.00-6.96 (m, 4H), 6.63 (s, 1H), 6.41 (s, 1H), 5.42 (brs, 1H), 4.97 (d, J = 8.0 Hz, 1H), 3.82 (s, 3H), 3.81 (s, 3H), 3.03 (dd, J = 4.0, 16.0 Hz, 1H), 2.89 (dd, J = 12.0, 16.0 Hz, 2.05 (s, 3H), 1.97 (s, 3H); ^{13}C NMR (100 MHz, DMSO-d_6) δ 197.7, 191.6, 163.5, 160.8, 145.9, 135.1, 133.3, 131.3, 130.8, 130.5, 130.1, 129.0,

128.4, 123.1, 121.1, 115.6, 114.2, 113.7, 55.9, 55.7, 45.1, 19.0, 18.8; HRMS (EI) m/z calcd. for $C_{28}H_{28}N_2O_4$ $[M]^+$ 456.2049, found 456.2033.

2-(3-(4-Chlorobenzoyl)-2,5-dihydro-1*H*-benzo[*b*][1,4]diazepin-2-yl)-1-(4-chlorophenyl)ethan-1-one 6da

The crude product was purified by column chromatography on silica gel using hexane/ethyl acetate = 80:20 (v/v) as eluent. Yield 10% (11 mg); Yellow solid, melting point: 151-152 °C.^{1}H NMR (400 MHz, DMSO-d_6) δ 9.51 (d, J = 8.0 Hz, 1H), 7.94-7.91 (m, 2H), 7.56-7.52 (m, 2H), 7.51-7.49 (m, 2H), 7.43-7.40 (m, 2H), 6.97 (d, J = 8.0 Hz, 1H), 6.90-6.86 (m, 1H), 6.78-6.74 (m, 2H), 6.70-6.64 (m, 1H), 5.77 (brs, 1H), 5.02 (t, J = 8.0 Hz, 1H), 3.06-2.98 (m, 2H); ^{13}C NMR (100 MHz, DMSO-d_6) δ 198.1, 190.9, 146.8, 139.7, 138.5, 137.8, 135.7, 134.6, 131.2, 130.4, 130.3, 129.1, 128.5, 123.9, 122.1, 121.0, 120.5, 115.8, 51.5, 45.9; HRMS (EI) m/z calcd. for $C_{24}H_{18}N_2O_2Cl_2$ $[M]^+$ 436.0745, found 436.0736.

2-(3-(4-Chlorobenzoyl)-7,8-dimethyl-2,5-dihydro-1*H*-benzo[*b*][1,4]diazepin-2-yl)-1-(4-chlorophenyl)ethan-1-one 6db

The crude product was purified by column chromatography on silica gel using hexane/ethyl acetate = 80:20 (v/v) as eluent. Yield 15% (18 mg); Yellow solid, melting point: 141-142 °C.^{1}H NMR (400 MHz, DMSO-d_6) δ 9.37 (d, J = 8.0 Hz, 1H), 7.94-7.91 (m, 2H), 7.56-7.52 (m, 2H), 7.51-7.48 (m, 2H), 7.40-7.37 (m, 2H), 6.93 (d, J = 8.0 Hz, 1H), 6.64 (s, 1H), 6.43 (s, 1H), 5.57 (s, 1H), 4.99 (t, J = 8.0 Hz, 1H), 3.05-2.95 (m, 2H), 2.06 (s, 3H), 1.98 (s, 3H); ^{13}C NMR (100 MHz, DMSO-d_6) δ 198.3, 190.8, 146.8, 139.9, 138.4, 135.8, 135.2, 134.5, 131.8, 130.4, 130.2, 129.1, 128.8, 128.6, 128.5, 123.2, 121.4, 115.5, 51.7, 45.8, 19.0, 18.8; HRMS (EI) m/z calcd. for $C_{26}H_{22}N_2O_2Cl_2$ $[M]^+$ 464.1058, found 464.1051.

2-(3-(4-Bromobenzoyl)-2,5-dihydro-1*H*-benzo[*b*][1,4]diazepin-2-yl)-1-(4-bromophenyl)ethan-1-one 6ea

The crude product was purified by column chromatography on silica gel using hexane/ethyl acetate = 85:15 (v/v) as eluent. Yield 20% (26 mg); Yellow solid, melting point: 203-204 °C.^{1}H NMR (400 MHz, DMSO-d_6) δ 9.48 (d, J = 8.0 Hz, 1H), 7.86-7.83 (m, 2H), 7.70-7.66 (m, 2H), 7.65-7.62 (m, 2H), 7.36-7.32 (m, 2H), 6.96 (d, J = 8.0 Hz, 1H), 6.89-6.86 (m, 1H), 6.77-6.73 (m, 2H), 6.69-6.65 (m, 1H), 5.76 (d, J = 4.0 Hz, 1H), 5.05-5.00 (m, 1H), 3.06-2.96 (m, 2H); ^{13}C NMR (100 MHz, DMSO-d_6) δ 198.3, 191.0, 146.8, 140.1, 137.8, 136.1, 132.1, 131.4, 131.2, 130.5, 127.7, 124.0, 123.3, 122.1, 121.0, 120.5, 115.8, 51.5, 45.8; HRMS (EI) m/z calcd. for $C_{24}H_{18}N_2O_2Br_2$ $[M]^+$ 523.9735, found 523.9738.

2-(3-(4-Bromobenzoyl)-7,8-dimethyl-2,5-dihydro-1*H*-benzo[*b*][1,4]diazepin-2-yl)-1-(4-bromophenyl)ethan-1-one 6eb

The crude product was purified by column chromatography on silica gel using hexane/ethyl acetate = 85:15 (v/v) as eluent. Yield 27% (37 mg); Yellow solid, melting point: 167-169 °C.^{1}H NMR (400 MHz, DMSO-d_6) δ 9.35 (d, J = 8.0 Hz, 1H), 7.85-7.82 (m, 2H), 7.68-7.66 (m, 2H), 7.64-7.61 (m, 2H), 7.33-7.30 (m, 2H), 6.93 (d, J = 8.0 Hz, 1H), 6.63 (s, 1H), 6.42 (s, 1H), 5.52 (d, J = 8.0 Hz, 1H), 5.05-4.96 (m, 1H), 3.05-2.95 (m, 2H), 2.05 (s, 3H), 1.98 (s, 3H); ^{13}C NMR (100 MHz, DMSO-d_6) δ 198.5, 190.8, 146.9, 140.2, 136.1, 135.4, 132.0, 131.8, 131.4, 130.5, 130.4, 128.7, 128.5, 127.6, 123.2, 123.1, 121.4, 115.5, 51.6, 45.8, 19.1, 18.8; HRMS (EI) m/z calcd. for $C_{26}H_{22}N_2O_2Br_2$ $[M+2]^+$ 554.0028, found 554.0021.

3-((2-Aminophenyl)amino)-1-phenylprop-2-en-1-one 7aa

The crude product was purified by column chromatography on silica gel using hexane/ethyl acetate = 85:15 (v/v) as eluent. Yield 28% (33 mg); The product was obtained as a mixture

of stereoisomers in a 1:0.7 ratio. Yellow solid, melting point: 145-147 °C.^{1}H NMR (400 MHz, $CDCl_3$) δ 12.20 (d, J = 12.0 Hz, 0.7H), 12.11 (d, J = 12.0 Hz, 1H), 7.95-7.93 (m, 3.3H), 7.49-7.38 (m, 7H), 7.17-6.96 (m, 3.5H), 6.85-6.79 (m, 2H), 6.12 (d, J = 8.0 Hz, 0.7H), 6.05 (d, J = 8.0 Hz, 1H), 3.56 (brs, 2.7H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 191.3, 190.9, 147.3, 146.4, 139.3, 139.2, 137.2, 132.2, 131.5, 131.4, 128.6, 128.5, 128.3, 127.5, 127.3, 125.3, 125.2, 119.9, 119.0, 117.8, 117.3, 95.5, 93.8; HRMS (EI) m/z calcd. for $C_{15}H_{14}N_2O$ $[M]^+$ 238.1106, found 238.1102.

1. Single crystal X-ray analytical data

A solvent mixture of ethyl acetate and dichloromethane was used to obtain crystals of **3aa** by a slow evaporation method at ambient temperature. In the case of **3af**, a mixture of hexanes and dichloromethane was used. The crystals of **6ea** were obtained using ethyl acetate as a solvent system. X-ray reflections were collected using Mo Kα X-radiation (λ = 0.71073 Å) on the single crystals at 200 K using a Bruker Kappa APEX-II diffractometer. All the crystal structures were solved and refined using SHELX-97. The details of crystals data collections and data refinement parameters are given in Table S1, S2 and S3.

Ortep (ellipsoid counter 30% probability**) diagram and crystal data of compound 3aa: CCDC Number 2334143**

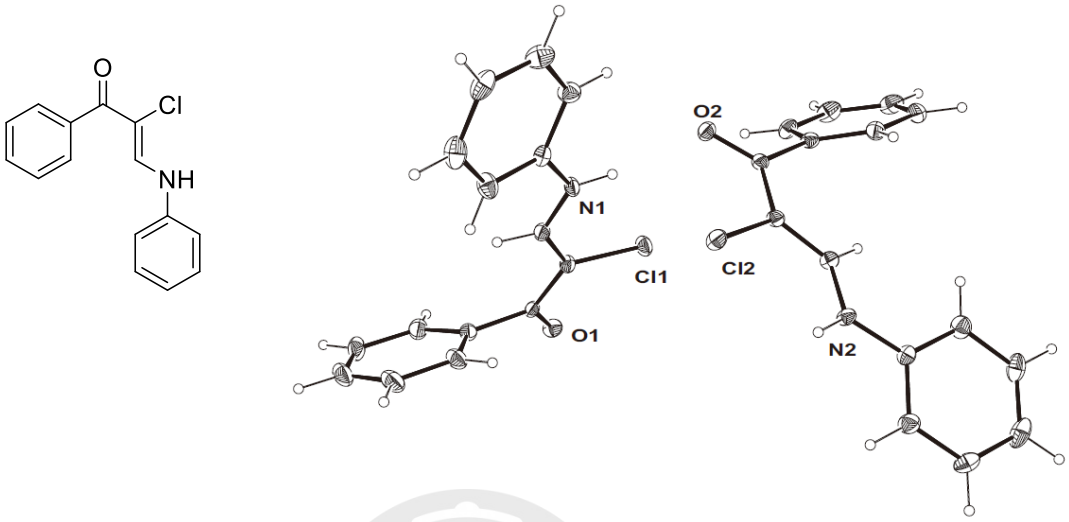


Table S1. Crystal data and structure refinement for d24546 (**3aa**).

Identification code	d24546	
Empirical formula	$C_{15}H_{12}BrClNO$	
Formula weight	257.71	
Temperature	200(2) K	
Wavelength	0.71073 Å	
Crystal system	Orthorhombic	
Space group	P c a 21	
Unit cell dimensions	a = 20.4024(11) Å	α= 90°.
	b = 8.0914(4) Å	β= 90°.
	c = 16.3490(8) Å	γ = 90°.
Volume	2699.0(2) $Å^3$	
Z	8	
Density (calculated)	1.268 Mg/m^3	
Absorption coefficient	0.270 mm^{-1}	
F(000)	1072	
Crystal size	0.52 x 0.48 x 0.30 mm^3	
Theta range for data collection	2.00 to 25.00°.	

Index ranges	-24<=h<=23, -8<=k<=9, -16<=l<=19
Reflections collected	19163
Independent reflections	4314 [R(int) = 0.0842]
Completeness to theta = 25.00°	99.6 %
Absorption correction	multi-scan
Max. and min. transmission	0.9234 and 0.8725
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	4314 / 1 / 325
Goodness-of-fit on F^2	1.002
Final R indices [I>2sigma(I)]	R1 = 0.0435, wR2 = 0.1049
R indices (all data)	R1 = 0.0635, wR2 = 0.1140
Absolute structure parameter	0.06(7)
Largest diff. peak and hole	0.604 and -0.176 e.$Å^{-3}$

Ortep (ellipsoid counter 30% probability**) diagram and crystal data of compound 3af: CCDC Number 2370736**

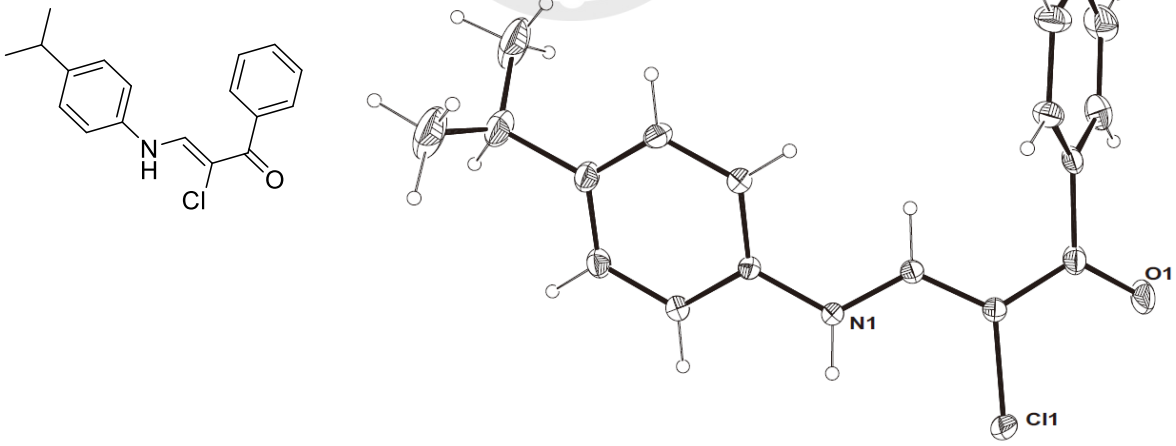


Table S2. Crystal data and structure refinement for 25152 (**3af**).

Identification code	d25152	
Empirical formula	$C_{18}H_{18}ClNO$	
Formula weight	299.78	
Temperature	200(2) K	
Wavelength	0.71073 Å	
Crystal system	Monoclinic	
Space group	P 21/n	
Unit cell dimensions	a = 9.9614(6) Å	α= 90°.
	b = 15.4727(9) Å	β= 109.434(2)°.
	c = 11.3672(7) Å	γ = 90°.
Volume	1652.20(17) $Å^3$	
Z	4	
Density (calculated)	1.205 Mg/m^3	
Absorption coefficient	0.230 mm^{-1}	
F(000)	632	
Crystal size	0.53 x 0.44 x 0.03 mm^3	
Theta range for data collection	2.31 to 25.05°.	
Index ranges	-11<=h<=11, -18<=k<=18, -13<=l<=13	
Reflections collected	34591	
Independent reflections	2917 [R(int) = 0.0954]	
Completeness to theta = 25.05°	99.7 %	
Absorption correction	None	
Max. and min. transmission	0.9931 and 0.8880	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	2917 / 0 / 186	
Goodness-of-fit on F^2	1.149	
Final R indices [I>2sigma(I)]	R1 = 0.0576, wR2 = 0.1394	
R indices (all data)	R1 = 0.0746, wR2 = 0.1516	
Largest diff. peak and hole	0.503 and -0.540 $e.Å^{-3}$	

Ortep (ellipsoid counter 30% probability**) diagram and crystal data of compound 6ea: CCDC Number 2360356**

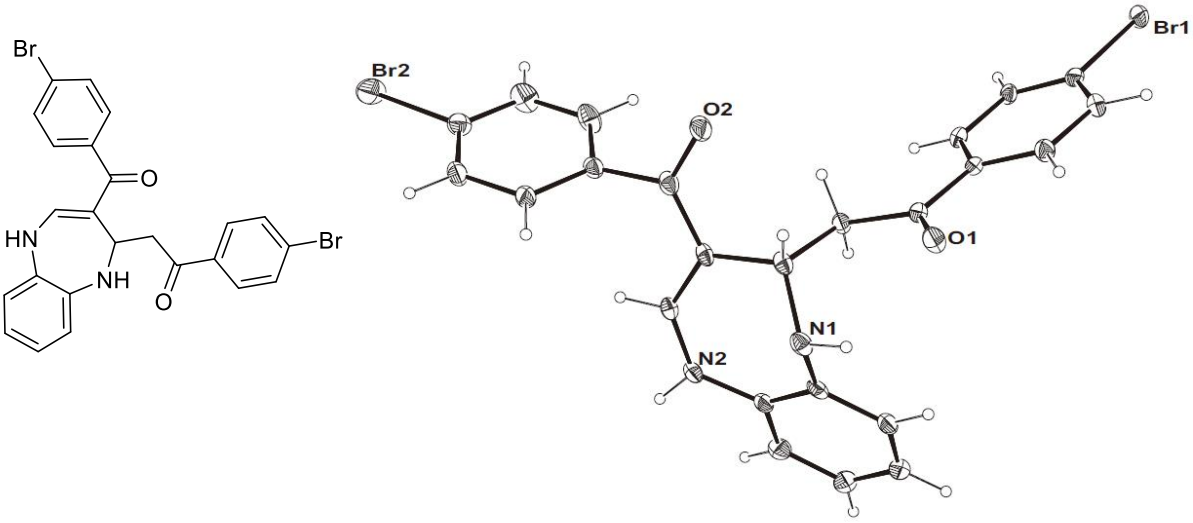


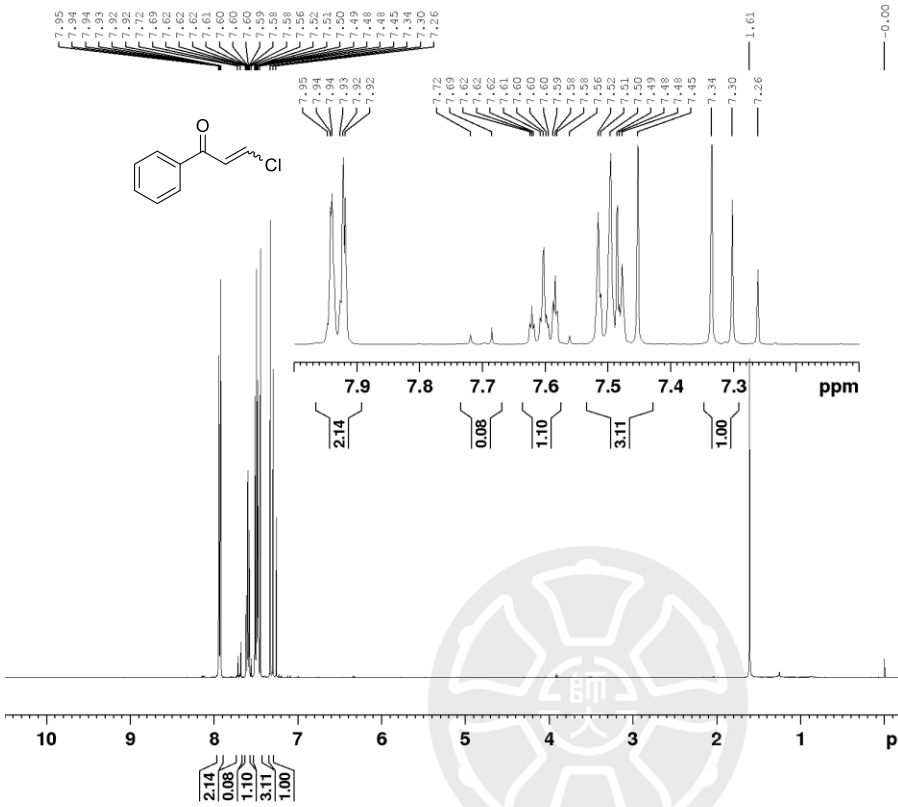
Table S3. Crystal data and structure refinement for d25025 (**6ea**).

Identification code	d25025	
Empirical formula	C_{24} H_{18} Br_2 N_2 O_2	
Formula weight	526.22	
Temperature	200(2) K	
Wavelength	0.71073 Å	
Crystal system	Monoclinic	
Space group	C 2/c	
Unit cell dimensions	a = 32.4571(15) Å	α= 90°.
	b = 13.4666(5) Å	β= 110.33°.
	c = 11.2783(5) Å	γ = 90°.
Volume	4622.4(3) $Å^3$	
Z	8	
Density (calculated)	1.512 Mg/m^3	
Absorption coefficient	3.529 mm^{-1}	
F(000)	2096	
Crystal size	0.21 x 0.10 x 0.03 mm^3	
Theta range for data collection	2.36 to 25.07°.	
Index ranges	-38<=h<=38, -16<=k<=14, -13<=l<=13	
Reflections collected	23074	
Independent reflections	4072 [R(int) = 0.0642]	

Completeness to theta = 25.07°	99.4 %
Absorption correction	None
Max. and min. transmission	0.9015 and 0.5244
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	4072 / 0 / 271
Goodness-of-fit on F^2	0.685
Final R indices [I>2sigma(I)]	R1 = 0.0355, wR2 = 0.0960
R indices (all data)	R1 = 0.0431, wR2 = 0.1038
Largest diff. peak and hole	0.546 and -1.033 e.$Å^{-3}$

2. NMR Spectra copies

3-chloro-1-phenylprop-2-en-1-one (1a)



BRUKER

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PROCNO                  1

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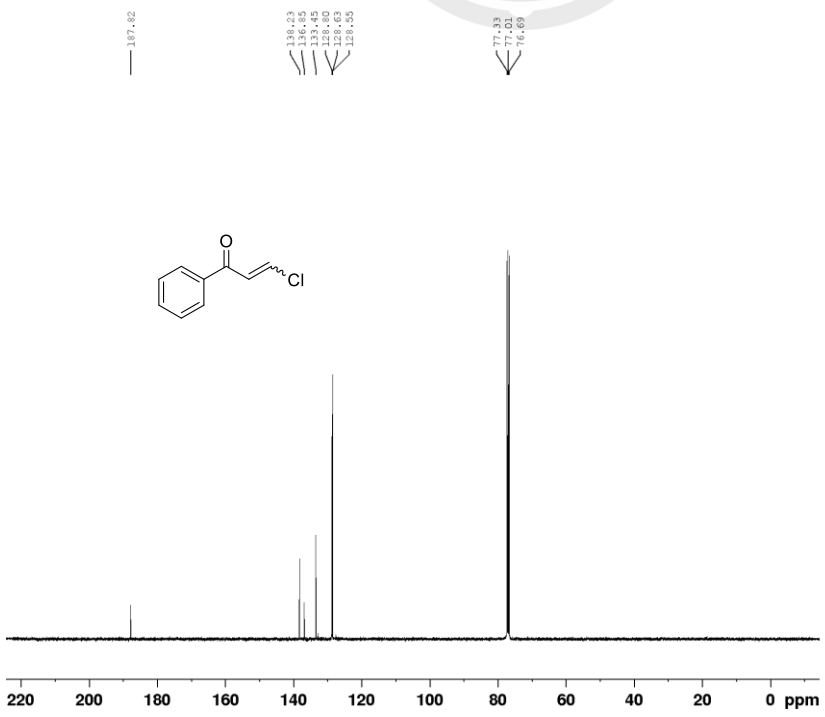

BRUKER

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======== CHANNEL f2 ========
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PCPD2              90.00 usec
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PL12               26.00 dB
PL13               29.00 dB
SFO2          400.1316005 MHz

F2 - Processing parameters
SI                  32768
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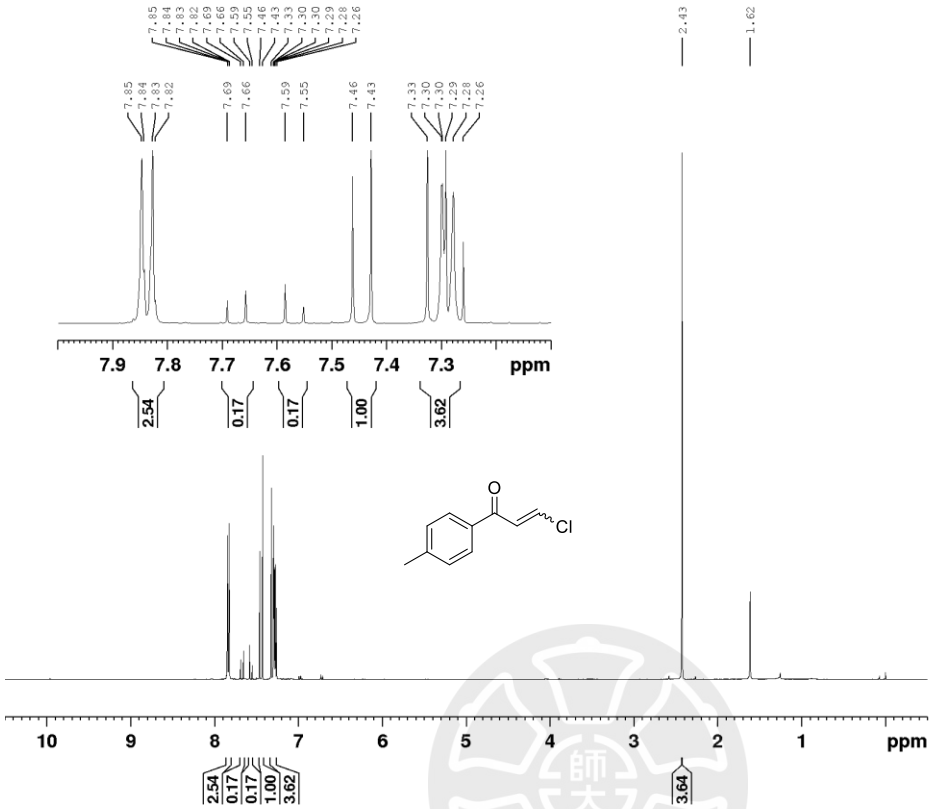
3-chloro-1-(*p*-tolyl)prop-2-en-1-one (1b)



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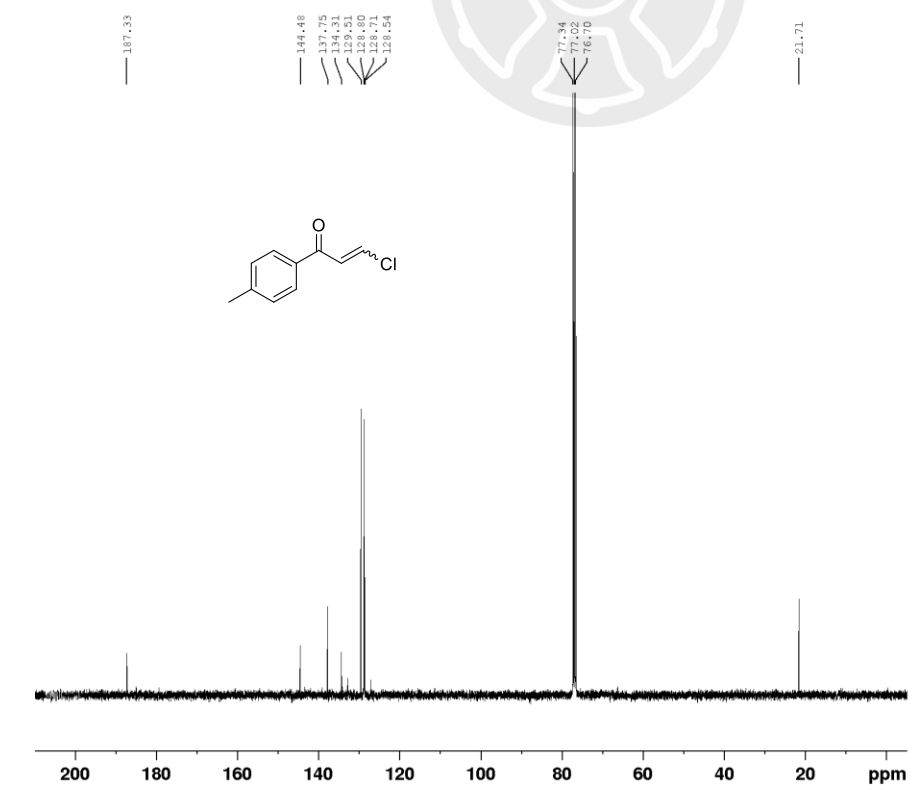

Current Data Parameters
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PROCNO 1

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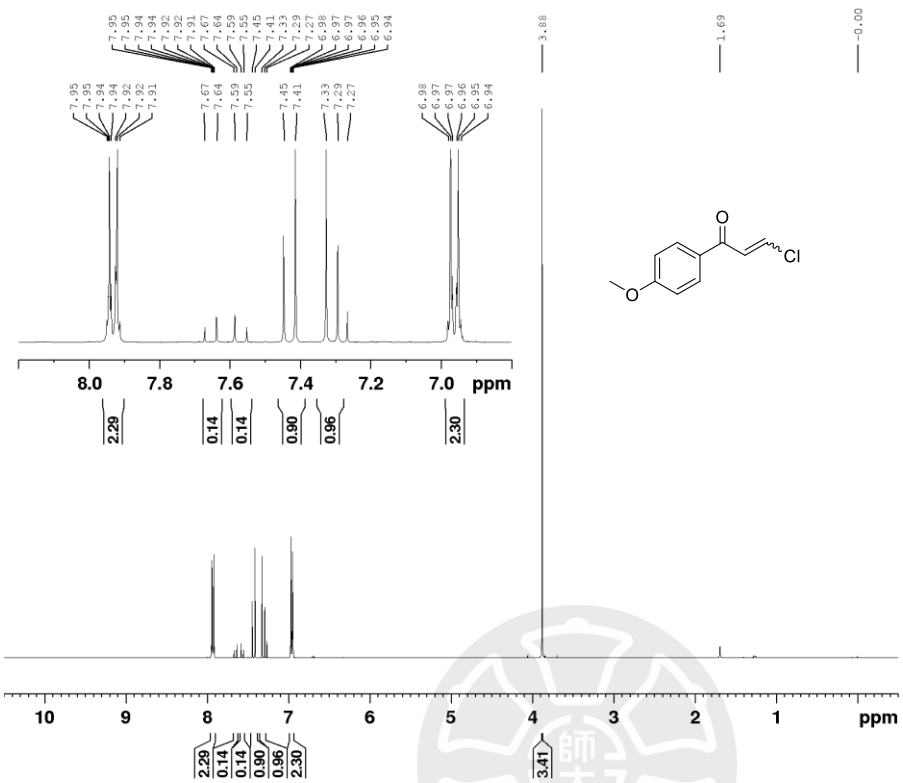
NS 2100
DS 0
SWH 24038.461 Hz
FIDRES 0.733596 Hz
AQ 0.6815744 sec
RG 32768
DW 20.800 usec
DE 6.50 usec
TE 296.8 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

======== CHANNEL f1 ========
NUC1 13C
P1 10.00 usec
PL1 4.20 dB
SFO1 100.6233325 MHz

======== CHANNEL f2 ========
CPDPRG[2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 10.20 dB
PL12 26.00 dB
PL13 29.00 dB
SFO2 400.1316005 MHz

F2 - Processing parameters
SI 32768
SF 100.6127690 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.00

3-chloro-1-(4-methoxyphenyl)prop-2-en-1-one (1c)



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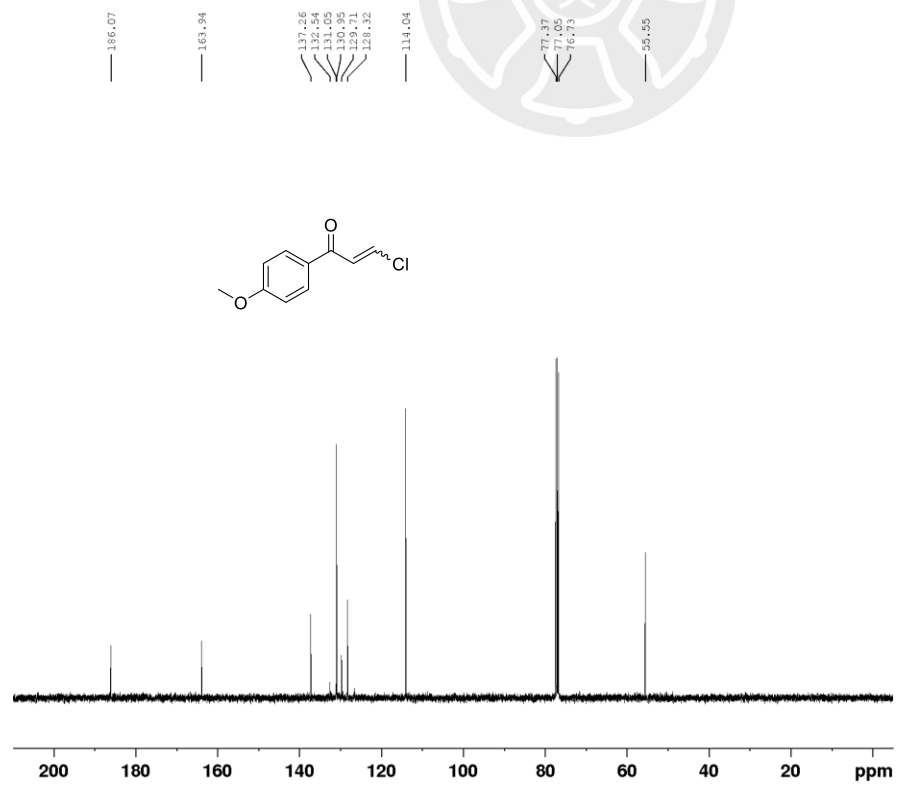
```
Current Data Parameters
NAME          Suresh-735
EXPNO                  2
PROCNO                 1

F2 - Acquisition Parameters
Date_           20231207
Time               21.42
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG             zg30
TD                 32768
SOLVENT            CDCl3
NS                    16
DS                     0
SWH             7211.539 Hz
FIDRES          0.220079 Hz
AQ              2.2719147 sec
RG                 89.08
DW                69.333 usec
DE                10.06 usec
TE                298.3 K
D1             2.00000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1         400.1324008 MHz
NUC1                   1H
P1                  15.00 usec
PLW1          13.50000000 W

F2 - Processing parameters
SI                 16384
SF           400.1300072 MHz
WDW                   EM
SSB                    0
LB                     0 Hz
GB                     0
PC                  1.00
```



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```
Current Data Parameters
NAME             exp 735
EXPNO                  2
PROCNO                 1

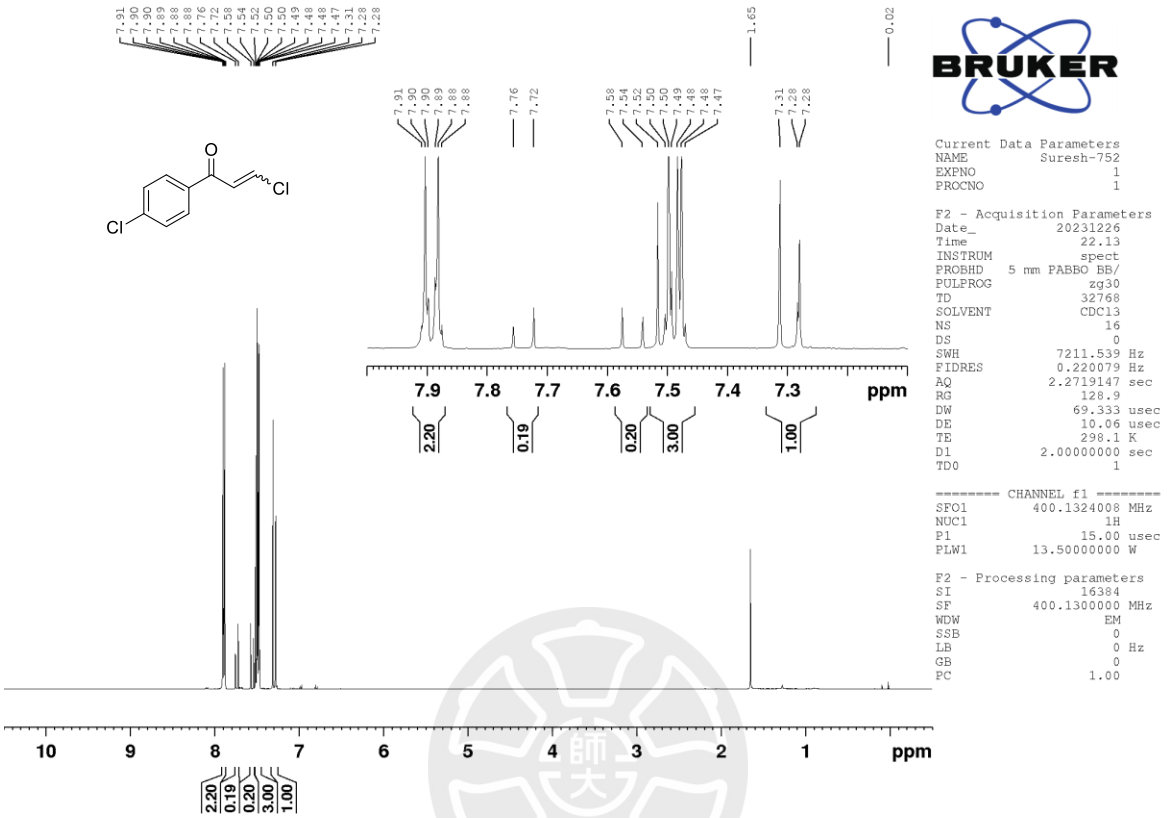
F2 - Acquisition Parameters
Date_           20231207
Time               20.58
INSTRUM            spect
PROBHD   5 mm BBO BB-1H
PULPROG          zgpg30
TD                 32768
SOLVENT            CDCl3
NS                   151
DS                     0
SWH           24038.461 Hz
FIDRES         0.733596 Hz
AQ             0.6815744 sec
RG                 32768
DW                20.800 usec
DE                 6.50 usec
TE                295.3 K
D1             2.00000000 sec
D11            0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  13C
P1                 10.00 usec
PL1                 4.20 dB
SFO1         100.6233325 MHz

======== CHANNEL f2 ========
CPDPRG[2          waltz16
NUC2                   1H
PCPD2              90.00 usec
PL2                10.20 dB
PL12               26.00 dB
PL13               29.00 dB
SFO2         400.1316005 MHz

F2 - Processing parameters
SI                 32768
SF           100.6127690 MHz
WDW                   EM
SSB                    0
LB                  1.00 Hz
GB                     0
PC                  1.00
```

3-chloro-1-(4-chlorophenyl)prop-2-en-1-one (1d)

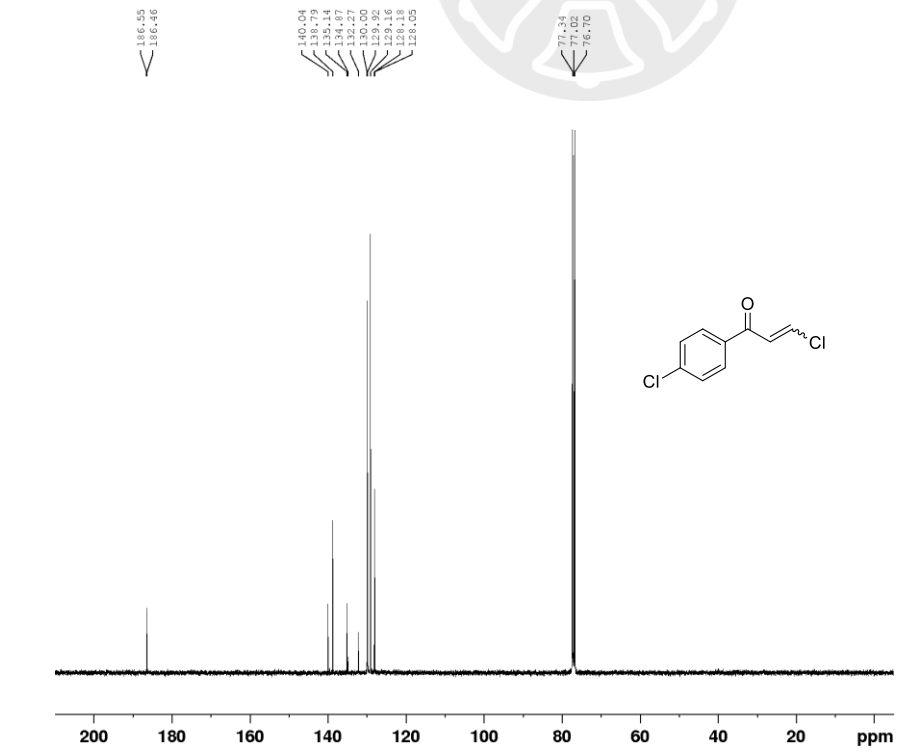



```
Current Data Parameters
NAME                 Suresh-752
EXPNO                         1
PROCNO                        1

F2 - Acquisition Parameters
Date_               20231226
Time                    22.13
INSTRUM                 spect
PROBHD    5 mm PABBO BB/
PULPROG                 zg30
TD                     32768
SOLVENT                CDCl3
NS                        16
DS                         0
SWH                 7211.539 Hz
FIDRES              0.220079 Hz
AQ                  2.2719147 sec
RG                      128.9
DW                     69.333 usec
DE                     10.06 usec
TE                     298.1 K
D1                  2.00000000 sec
TD0                           1

======== CHANNEL f1 ========
SFO1              400.1324008 MHz
NUC1                        1H
P1                       15.00 usec
PLW1             13.50000000 W

F2 - Processing parameters
SI                        16384
SF                400.1300000 MHz
WDW                         EM
SSB                          0
LB                           0 Hz
GB                           0
PC                        1.00
```




```
Current Data Parameters
NAME                   exp 752
EXPNO                        2
PROCNO                       1

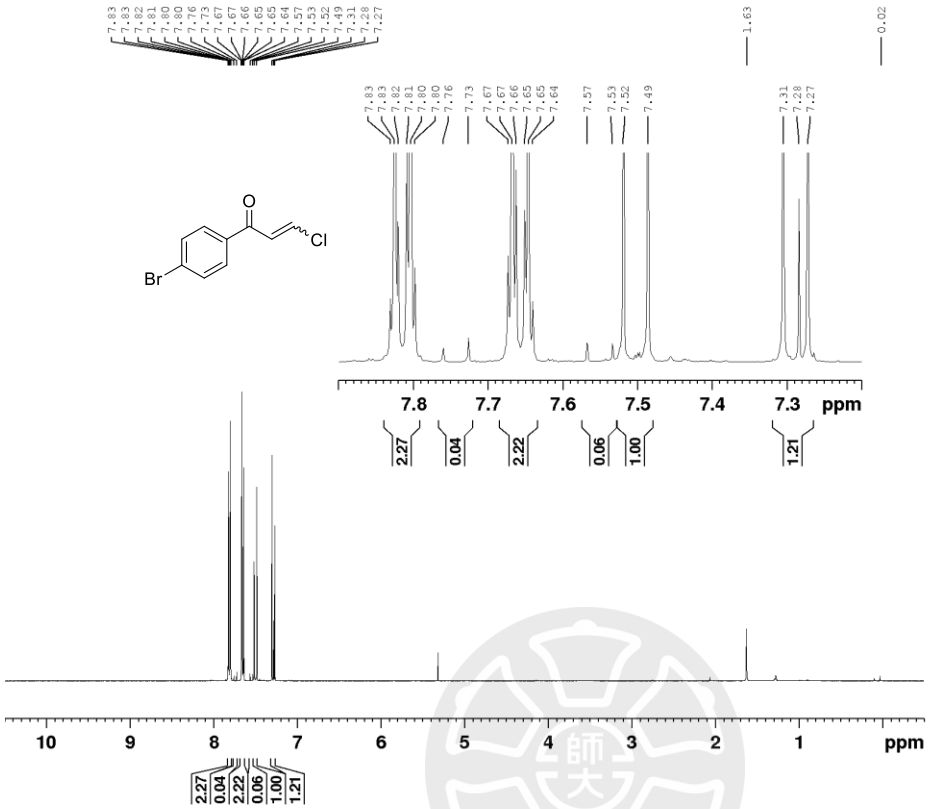
F2 - Acquisition Parameters
Date_               20231227
Time                    12.10
INSTRUM                 spect
PROBHD    5 mm BBO BB-1H
PULPROG                 zgpg30
TD                     32768
SOLVENT                CDCl3
NS                      1392
DS                         0
SWH                24038.461 Hz
FIDRES              0.733596 Hz
AQ                  0.6815744 sec
RG                      32768
DW                     20.800 usec
DE                      6.50 usec
TE                     298.4 K
D1                  2.00000000 sec
D11                 0.03000000 sec
TD0                          1

======== CHANNEL f1 ========
NUC1                        13C
P1                       10.00 usec
PL1                       4.20 dB
SFO1              100.6233325 MHz

======== CHANNEL f2 ========
CPDPRG[2                waltz16
NUC2                        1H
PCPD2                    90.00 usec
PL2                      10.20 dB
PL12                     26.00 dB
PL13                     29.00 dB
SFO2              400.1316005 MHz

F2 - Processing parameters
SI                        32768
SF                100.6127690 MHz
WDW                         EM
SSB                          0
LB                        1.00 Hz
GB                           0
PC                        1.00
```

1-(4-bromophenyl)-3-chloroprop-2-en-1-one (1e)

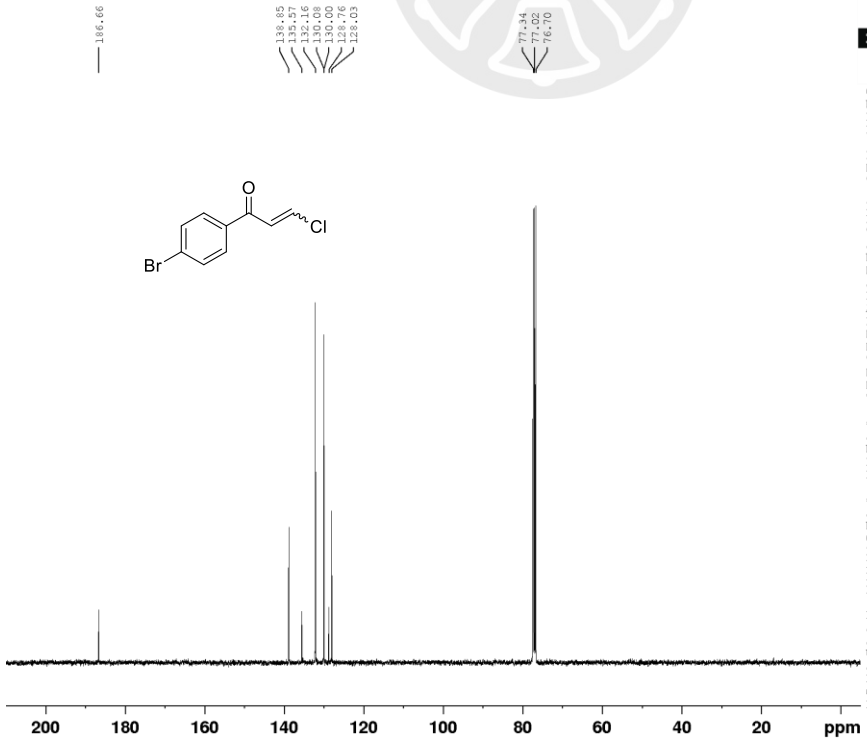



```
Current Data Parameters
NAME          Suresh-740
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20231211
Time               15.11
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG             zg30
TD                 32768
SOLVENT            CDC13
NS                    16
DS                     0
SWH             7211.539 Hz
FIDRES          0.220079 Hz
AQ              2.2719147 sec
RG                 113.31
DW                69.333 usec
DE                 10.06 usec
TE                 298.8 K
D1              2.00000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1          400.1324008 MHz
NUC1                  1H
P1                 15.00 usec
PLW1          13.50000000 W

F2 - Processing parameters
SI                 16384
SF            400.1300000 MHz
WDW                   EM
SSB                    0
LB                  0 Hz
GB                     0
PC                  1.00
```




```
Current Data Parameters
NAME          Suresh-740
EXPNO                  3
PROCNO                 1

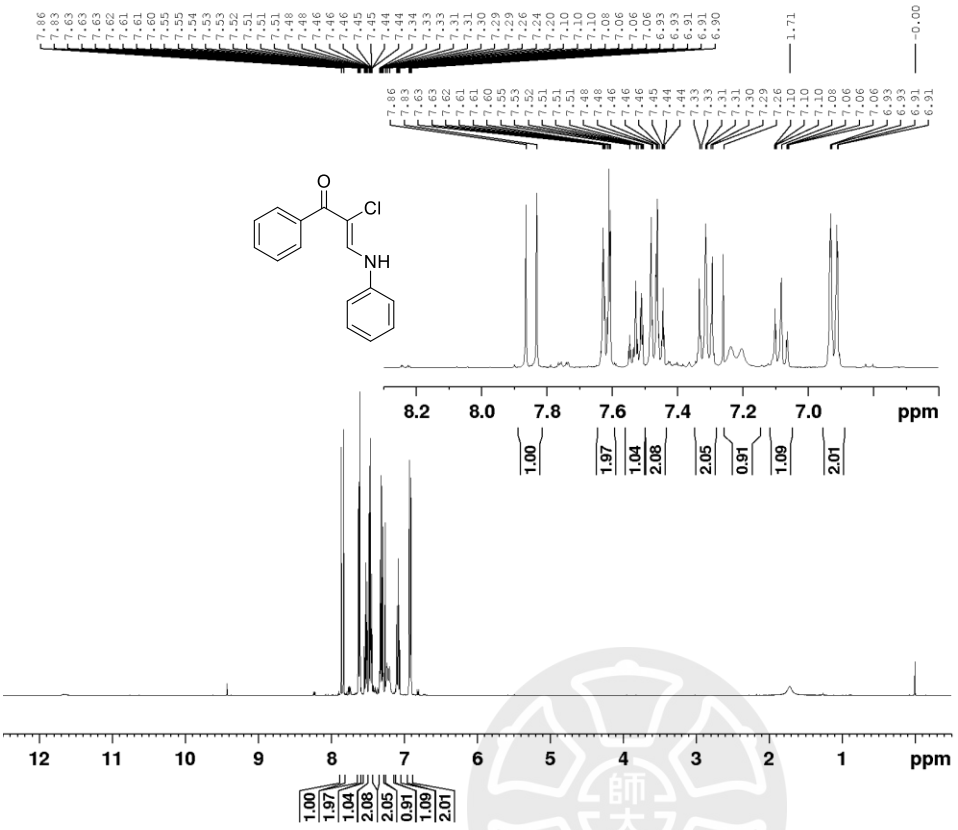
F2 - Acquisition Parameters
Date_           20231211
Time               17.20
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG          zgpg30
TD                 32768
SOLVENT            CDC13
NS                   512
DS                     0
SWH            24038.461 Hz
FIDRES          0.733596 Hz
AQ              0.6815744 sec
RG                 198.09
DW                20.800 usec
DE                 6.50 usec
TE                 298.5 K
D1              2.00000000 sec
D11             0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1          100.6228298 MHz
NUC1                  13C
P1                 10.00 usec
PLW1          49.50000000 W

======== CHANNEL f2 ========
SFO2          400.1316005 MHz
NUC2                  1H
CPDPRG[2          waltz16
PCPD2              90.00 usec
PLW2          12.50000000 W
PLW12          0.34722000 W
PLW13          0.28125000 W

F2 - Processing parameters
SI                 32768
SF            100.6127685 MHz
WDW                   EM
SSB                    0
LB                  2.00 Hz
GB                     0
PC                  1.00
```

(*Z*)-2-chloro-1-phenyl-3-(phenylamino)prop-2-en-1-one (3aa)



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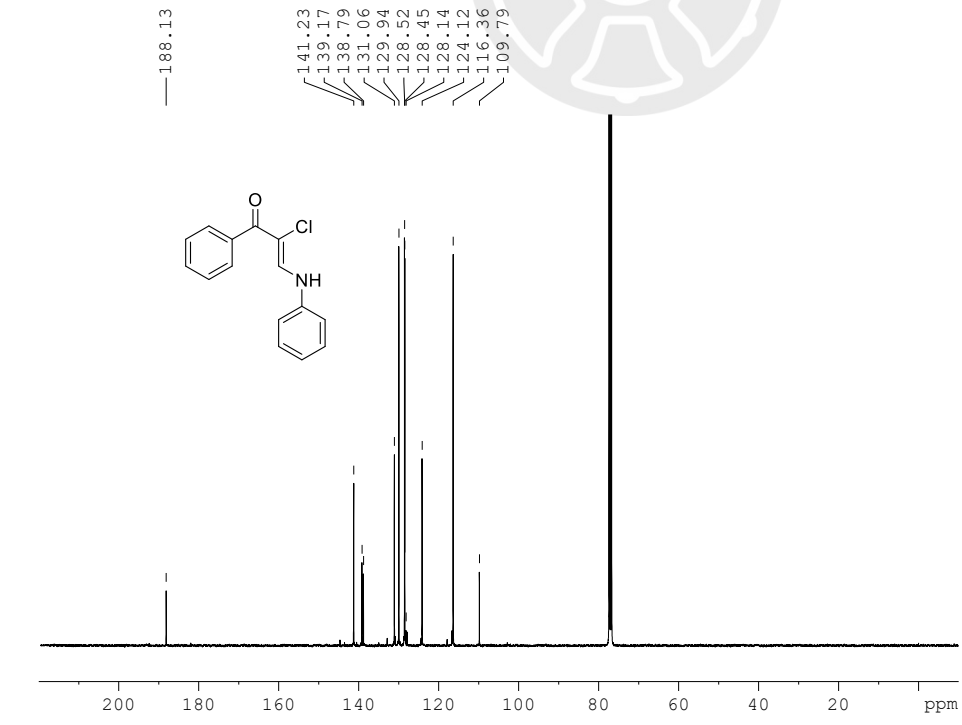
```
Current Data Parameters
NAME          Suresh-514
EXPNO                  4
PROCNO                 1

F2 - Acquisition Parameters
Date_           20221101
Time               23.18
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG             zg30
TD                 32768
SOLVENT            CDCl3
NS                    16
DS                     0
SWH             7211.539 Hz
FIDRES          0.220079 Hz
AQ              2.2719147 sec
RG                113.31
DW                69.333 usec
DE                10.06 usec
TE                299.3 K
D1             2.00000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1         400.1324008 MHz
NUC1                   1H
P1                 15.00 usec
PLW1        13.50000000 W

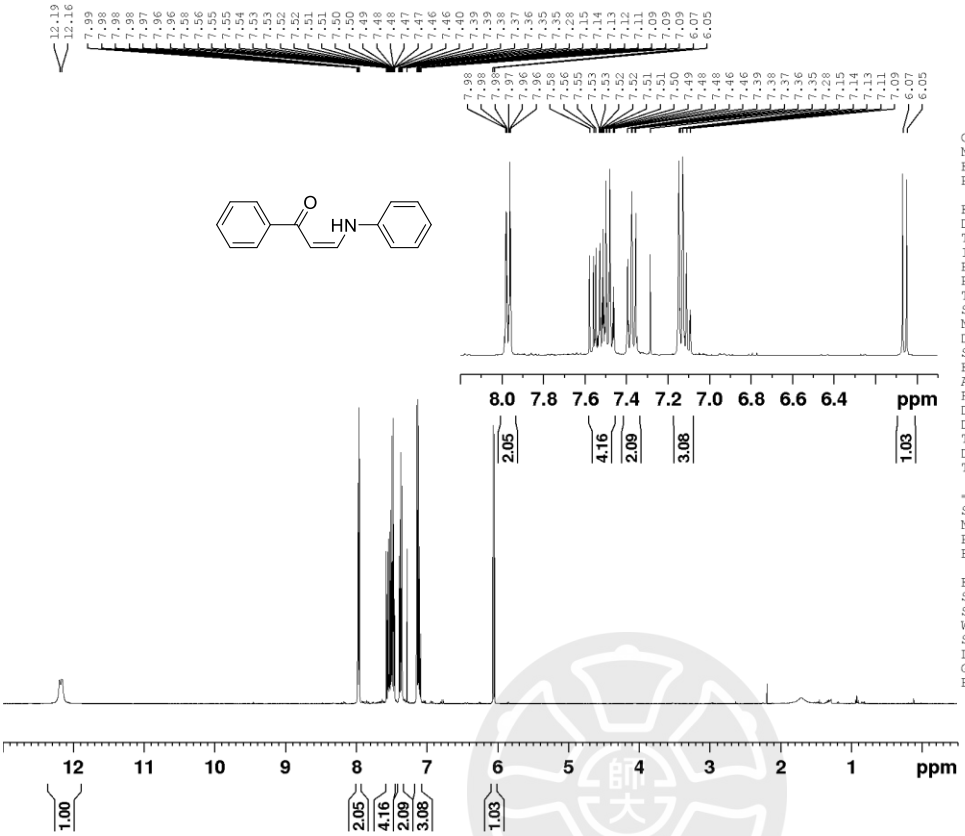
F2 - Processing parameters
SI                 16384
SF           400.1300103 MHz
WDW                   EM
SSB                    0
LB                     0 Hz
GB                     0
PC                  1.00
```




```
NAME          Suresh-514
EXPNO                  5
PROCNO                 1
Date_           20221101
Time               23.20
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG          zgpg30
TD                 32768
SOLVENT            CDCl3
NS                  6000
DS                     0
SWH            24038.461 Hz
FIDRES          0.733596 Hz
AQ              0.6816244 sec
RG                198.09
DW                20.800 usec
DE                 6.50 usec
TE                299.4 K
D1             2.00000000 sec
D11            0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1        100.6228298 MHz
NUC1                   13C
P1                 10.00 usec
SI                 32768
SF          100.6127685 MHz
WDW                   EM
SSB                    0
LB                  2.00 Hz
GB                     0
PC                  1.00
```

(*Z*)-1-phenyl-3-(phenylamino)prop-2-en-1-one (4aa)

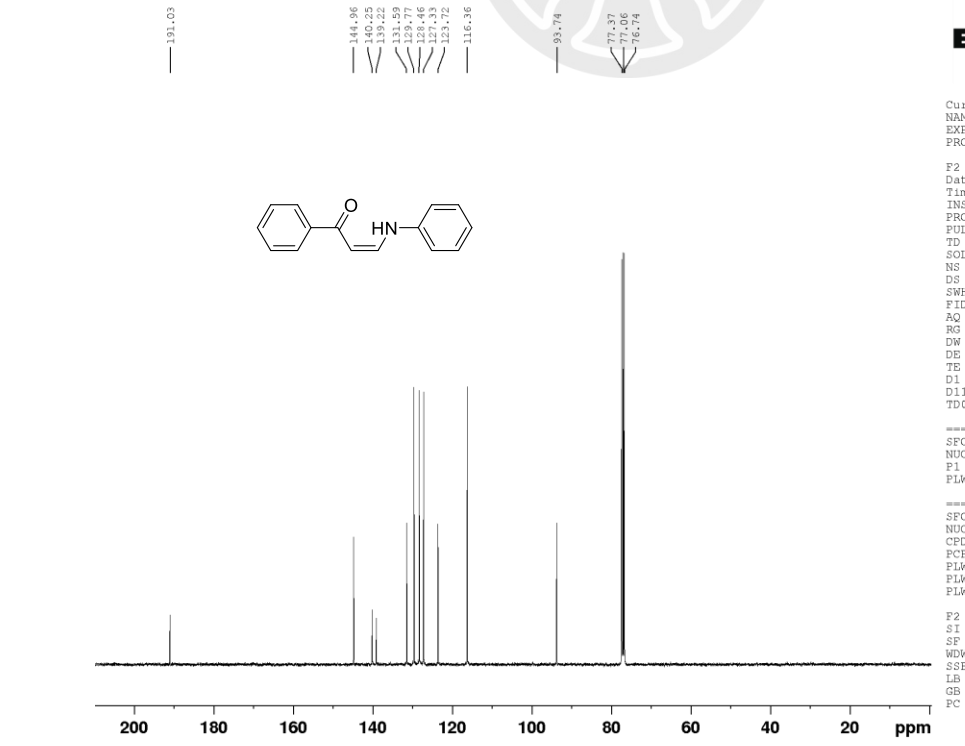


```
Current Data Parameters
NAME          Suresh-515
EXPNO                  4
PROCNO                 1

F2 - Acquisition Parameters
Date_           20221101
Time               22.25
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG             zg30
TD                 32768
SOLVENT            CDC13
NS                    16
DS                     0
SWH             7211.539 Hz
FIDRES          0.220079 Hz
AQ              2.2719147 sec
RG                 89.08
DW                69.333 usec
DE                10.06 usec
TE                299.1 K
D1             2.00000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1         400.1324008 MHz
NUC1                   1H
P1                 15.00 usec
PLW1         13.50000000 W

F2 - Processing parameters
SI                 16384
SF           400.1300000 MHz
WDW                   EM
SSB                    0
LB                     0 Hz
GB                     0
PC                  1.00
```



```
Current Data Parameters
NAME          Suresh-515
EXPNO                  5
PROCNO                 1

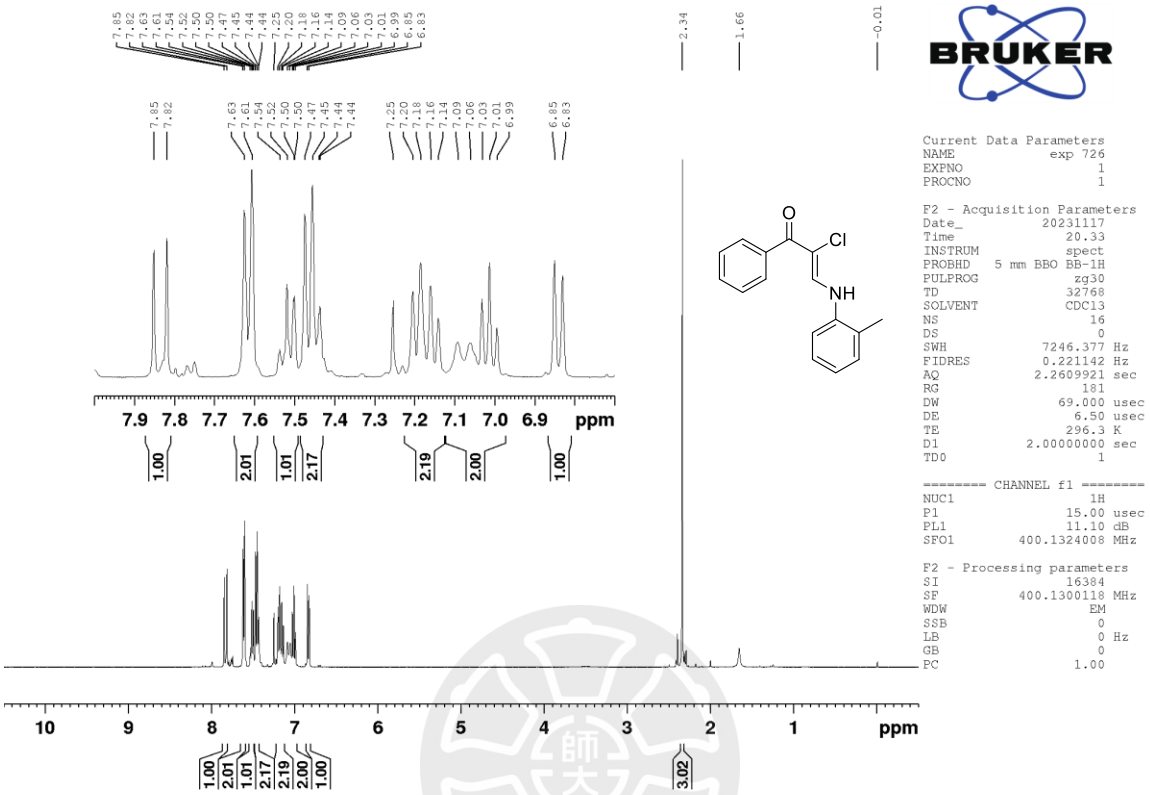
F2 - Acquisition Parameters
Date_           20221101
Time               22.29
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG          zgpg30
TD                 32768
SOLVENT            CDC13
NS                  1024
DS                     0
SWH            24038.461 Hz
FIDRES          0.733596 Hz
AQ              0.6815744 sec
RG                198.09
DW                20.800 usec
DE                 6.50 usec
TE                299.8 K
D1             2.00000000 sec
D11            0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1         100.6228298 MHz
NUC1                  13C
P1                10.00 usec
PLW1         49.50000000 W

======== CHANNEL f2 ========
SFO2         400.1316005 MHz
NUC2                   1H
CPDPRG[2          waltz16
PCPD2             90.00 usec
PLW2         12.50000000 W
PLW12         0.34722000 W
PLW13         0.28125000 W

F2 - Processing parameters
SI                 32768
SF           100.6127685 MHz
WDW                   EM
SSB                    0
LB                  2.00 Hz
GB                     0
PC                  1.00
```

(*Z*)-2-chloro-1-phenyl-3-(*o*-tolylamino)prop-2-en-1-one (3ab)



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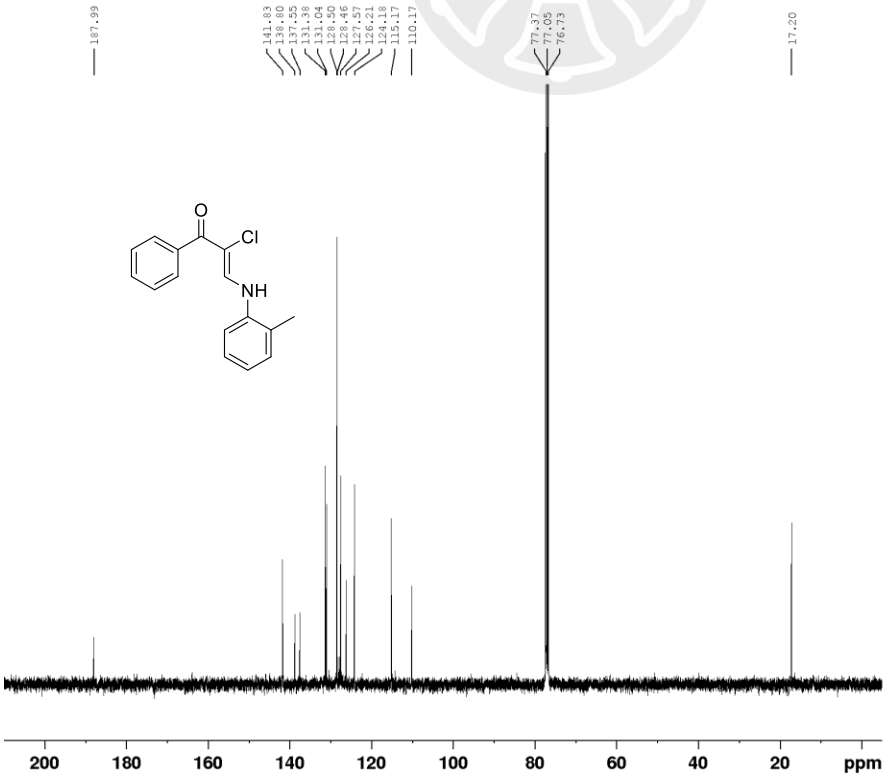
```
Current Data Parameters
NAME                  exp 726
EXPNO                       1
PROCNO                      1

F2 - Acquisition Parameters
Date_               20231117
Time                   20.33
INSTRUM                spect
PROBHD    5 mm BBO BB-1H
PULPROG                zg30
TD                     32768
SOLVENT                CDCl3
NS                        16
DS                         0
SWH                7246.377 Hz
FIDRES             0.221142 Hz
AQ                 2.2609921 sec
RG                       181
DW                  69.000 usec
DE                    6.50 usec
TE                   296.3 K
D1                 2.00000000 sec
TD0                        1

======== CHANNEL f1 ========
NUC1                      1H
P1                     15.00 usec
PL1                    11.10 dB
SFO1           400.1324008 MHz

F2 - Processing parameters
SI                        16384
SF             400.1300118 MHz
WDW                        EM
SSB                         0
LB                          0 Hz
GB                          0
PC                       1.00
```



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```
Current Data Parameters
NAME                  exp 726
EXPNO                       2
PROCNO                      1

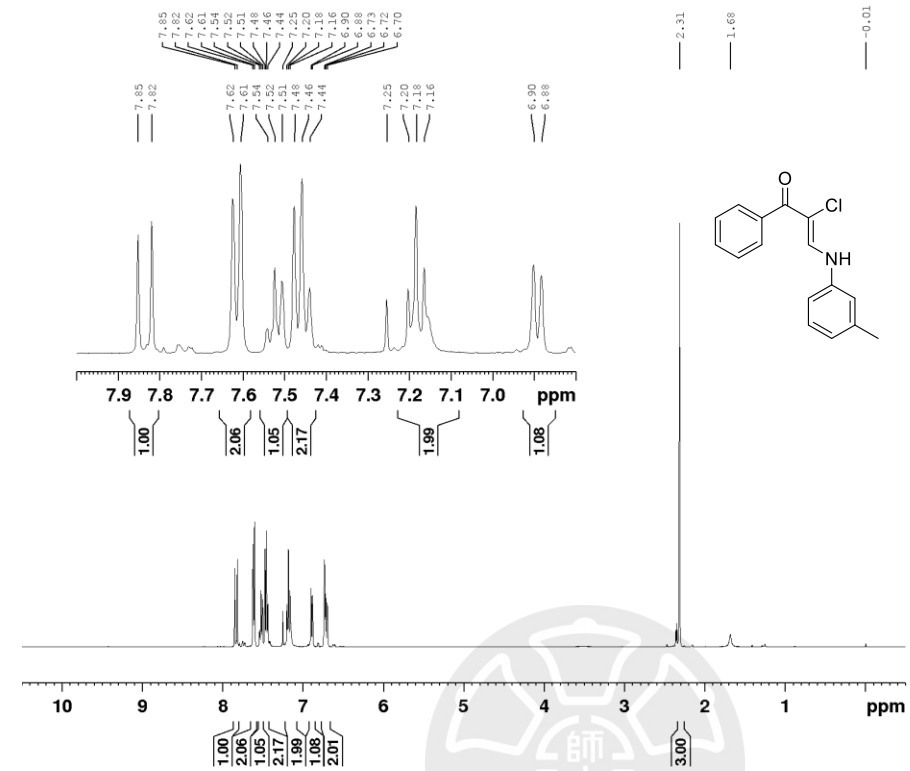
F2 - Acquisition Parameters
Date_               20231117
Time                   20.35
INSTRUM                spect
PROBHD    5 mm BBO BB-1H
PULPROG             zgpg30
TD                     32768
SOLVENT                CDCl3
NS                       513
DS                         0
SWH               24038.461 Hz
FIDRES             0.733596 Hz
AQ                 0.6815744 sec
RG                     32768
DW                  20.800 usec
DE                    6.50 usec
TE                   296.4 K
D1                 2.00000000 sec
D11                0.03000000 sec
TD0                        1

======== CHANNEL f1 ========
NUC1                      13C
P1                     10.00 usec
PL1                     4.20 dB
SFO1           100.6233325 MHz

======== CHANNEL f2 ========
CPDPRG[2              waltz16
NUC2                      1H
PCPD2                  90.00 usec
PL2                    10.20 dB
PL12                   26.00 dB
PL13                   29.00 dB
SFO2           400.1316005 MHz

F2 - Processing parameters
SI                        32768
SF             100.6127690 MHz
WDW                        EM
SSB                         0
LB                       1.00 Hz
GB                          0
PC                       1.00
```

(*Z*)-2-chloro-1-phenyl-3-(*m*-tolylamino)prop-2-en-1-one (3ac)

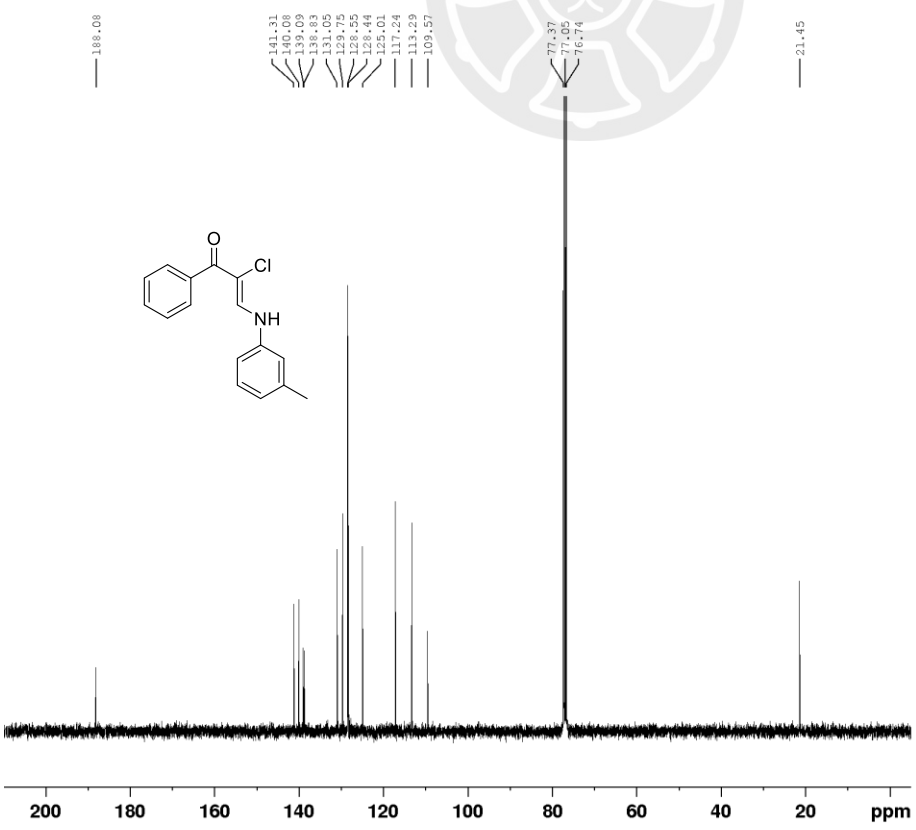



```
Current Data Parameters
NAME                  exp 729
EXPNO                       2
PROCNO                      1

F2 - Acquisition Parameters
Date_               20231120
Time                   17.40
INSTRUM                spect
PROBHD    5 mm BBO BB-1H
PULPROG                zg30
TD                    32768
SOLVENT               CDCl3
NS                       16
DS                        0
SWH                7246.377 Hz
FIDRES            0.221142 Hz
AQ                2.2609921 sec
RG                      181
DW                 69.000 usec
DE                  6.50 usec
TE                  296.2 K
D1               2.00000000 sec
TD0                       1

======== CHANNEL f1 ========
NUC1                      1H
P1                    15.00 usec
PL1                   11.10 dB
SFO1           400.1324008 MHz

F2 - Processing parameters
SI                    16384
SF             400.1300118 MHz
WDW                      EM
SSB                       0
LB                     0 Hz
GB                        0
PC                     1.00
```




```
Current Data Parameters
NAME                  exp 729
EXPNO                       3
PROCNO                      1

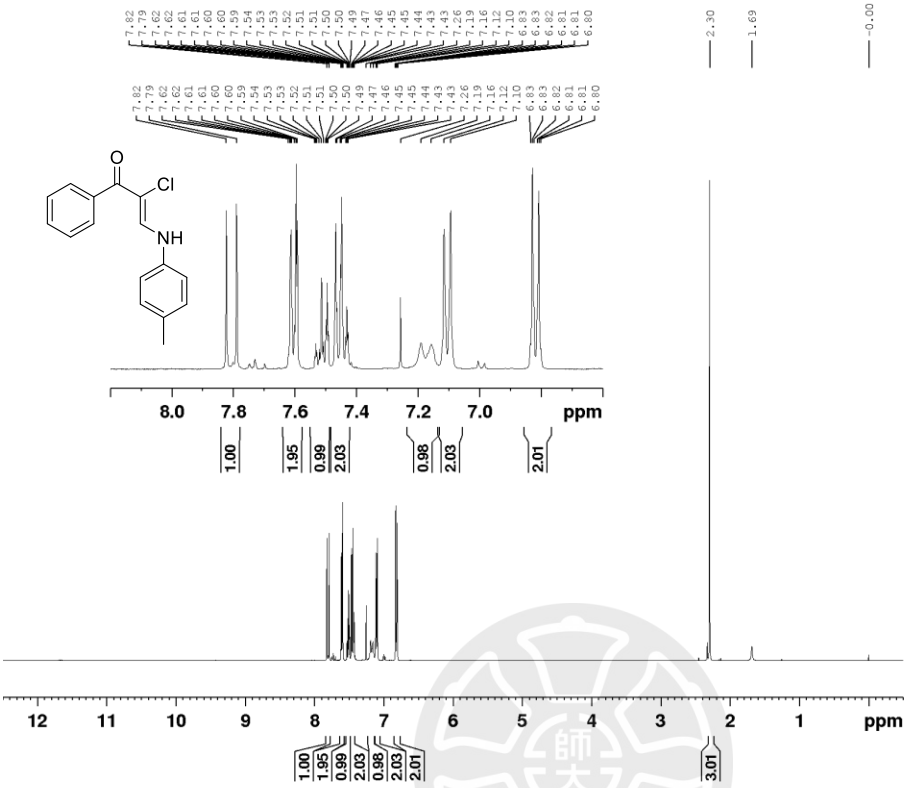
F2 - Acquisition Parameters
Date_               20231120
Time                   17.42
INSTRUM                spect
PROBHD    5 mm BBO BB-1H
PULPROG             zgpg30
TD                    32768
SOLVENT               CDCl3
NS                      412
DS                        0
SWH               24038.461 Hz
FIDRES            0.733596 Hz
AQ                0.6815744 sec
RG                      32768
DW                 20.800 usec
DE                  6.50 usec
TE                  296.2 K
D1               2.00000000 sec
D11              0.03000000 sec
TD0                       1

======== CHANNEL f1 ========
NUC1                     13C
P1                    10.00 usec
PL1                    4.20 dB
SFO1           100.6233325 MHz

======== CHANNEL f2 ========
CPDPRG[2            waltz16
NUC2                      1H
PCPD2                 90.00 usec
PL2                   10.20 dB
PL12                  26.00 dB
PL13                  29.00 dB
SFO2           400.1316005 MHz

F2 - Processing parameters
SI                    32768
SF             100.6127690 MHz
WDW                      EM
SSB                       0
LB                     1.00 Hz
GB                        0
PC                     1.00
```

(*Z*)-2-chloro-1-phenyl-3-(*p*-tolylamino)prop-2-en-1-one (3ad)



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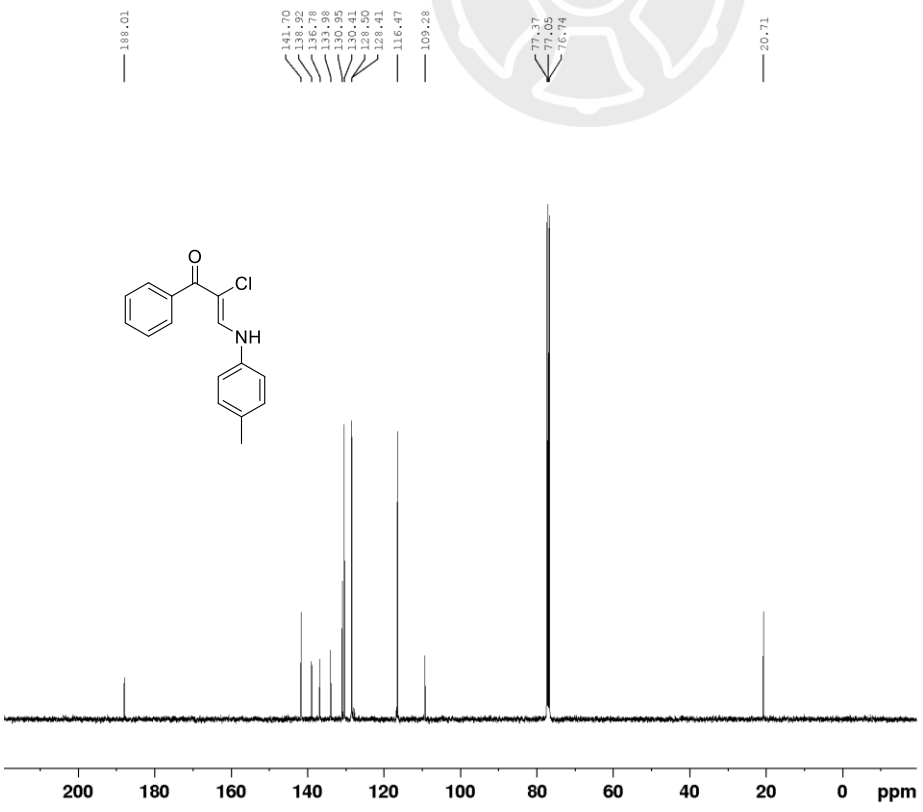
```
Current Data Parameters
NAME          Suresh-730
EXPNO                  2
PROCNO                 1

F2 - Acquisition Parameters
Date_           20231120
Time              12.04
INSTRUM           spect
PROBHD   5 mm PABBO BB/
PULPROG            zg30
TD                32768
SOLVENT           CDCl3
NS                   16
DS                    0
SWH           7211.539 Hz
FIDRES        0.220079 Hz
AQ            2.2719147 sec
RG                89.08
DW               69.333 usec
DE               10.06 usec
TE                299.4 K
D1           2.00000000 sec
TD0                   1

======== CHANNEL f1 ========
SFO1        400.1324008 MHz
NUC1                1H
P1                15.00 usec
PLW1        13.50000000 W

F2 - Processing parameters
SI                16384
SF          400.1300107 MHz
WDW                  EM
SSB                   0
LB                 0 Hz
GB                    0
PC                 1.00
```



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```
Current Data Parameters
NAME          Suresh-730
EXPNO                  3
PROCNO                 1

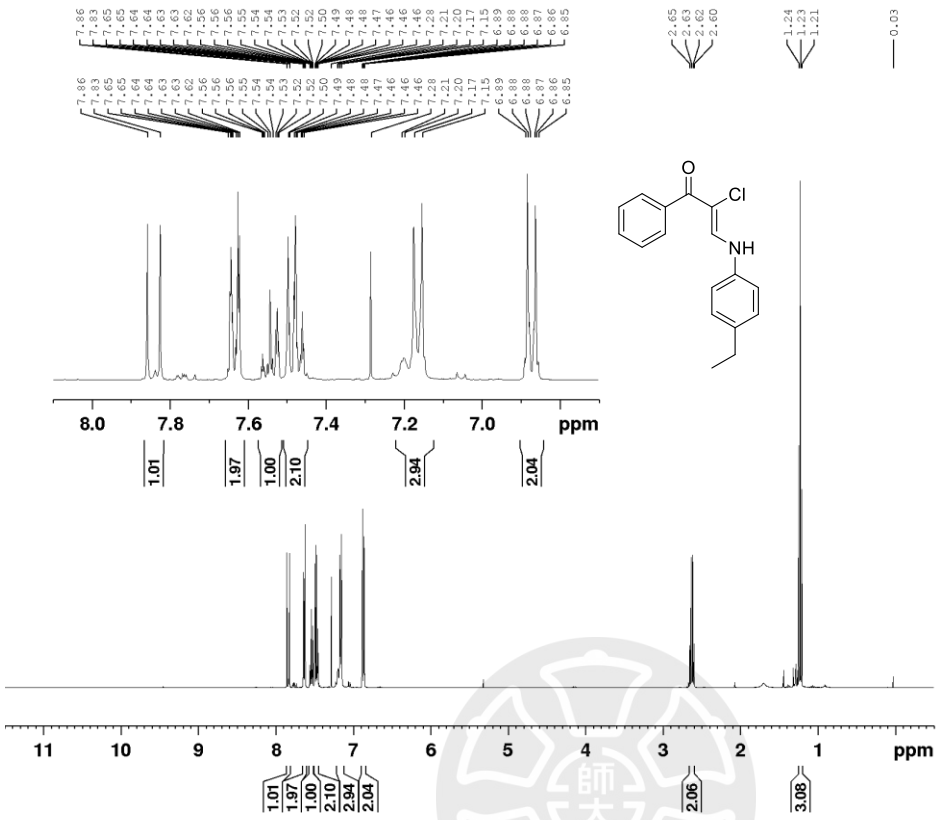
F2 - Acquisition Parameters
Date_           20231120
Time              12.06
INSTRUM           spect
PROBHD   5 mm PABBO BB/
PULPROG          zgpg30
TD                32768
SOLVENT           CDCl3
NS                  400
DS                    0
SWH          24038.461 Hz
FIDRES        0.733596 Hz
AQ            0.6815744 sec
RG                198.09
DW               20.800 usec
DE                6.50 usec
TE                299.4 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
SFO1        100.6228298 MHz
NUC1                13C
P1                10.00 usec
PLW1        49.50000000 W

======== CHANNEL f2 ========
SFO2        400.1316005 MHz
NUC2                1H
CPDPRG[2         waltz16
PCPD2             90.00 usec
PLW2        12.50000000 W
PLW12        0.34722000 W
PLW13        0.28125000 W

F2 - Processing parameters
SI                32768
SF          100.6127685 MHz
WDW                  EM
SSB                   0
LB                 2.00 Hz
GB                    0
PC                 1.00
```

(*Z*)-2-chloro-3-((4-ethylphenyl)amino)-1-phenylprop-2-en-1-one (3ae)

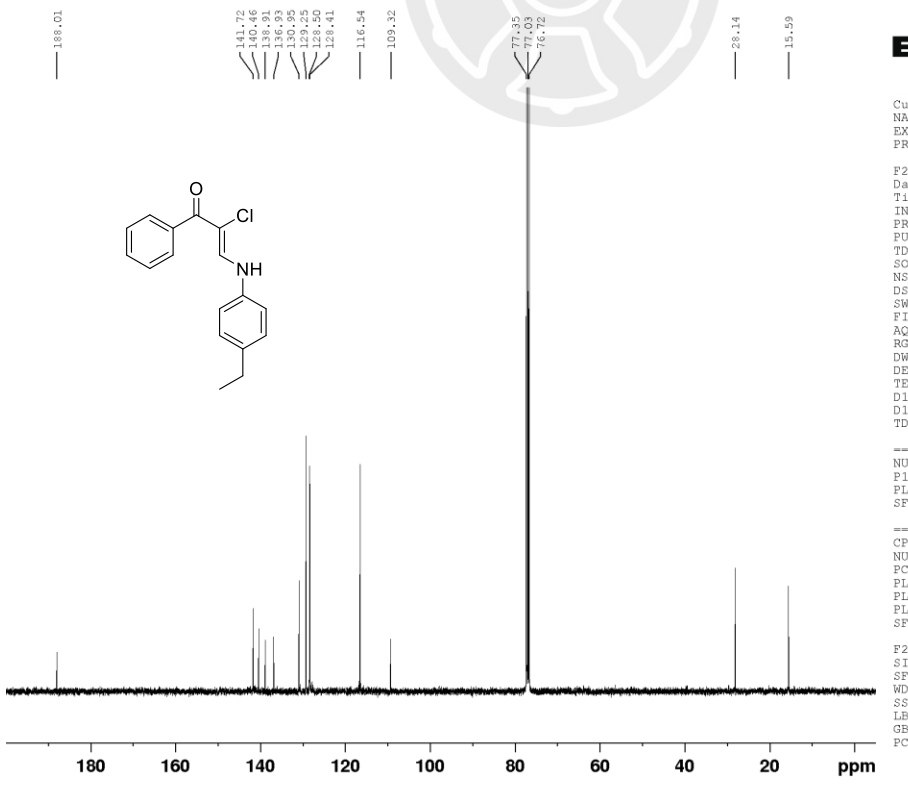


```
Current Data Parameters
NAME                  Enaminone
EXPNO                        22
PROCNO                        1

F2 - Acquisition Parameters
Date_                 20231101
Time                     11.19
INSTRUM                  spect
PROBHD    5 mm PABBO BB/
PULPROG                  zg30
TD                      32768
SOLVENT                 CDC13
NS                         16
DS                          0
SWH                  7211.539 Hz
FIDRES               0.220079 Hz
AQ                   2.2719147 sec
RG                      113.31
DW                      69.333 usec
DE                      10.06 usec
TE                      298.7 K
D1                   2.00000000 sec
TD0                           1

======== CHANNEL f1 ========
SFO1                400.1324008 MHz
NUC1                        1H
P1                       15.00 usec
PLW1                13.50000000 W

F2 - Processing parameters
SI                          16384
SF                  400.1300000 MHz
WDW                            EM
SSB                             0
LB                              0 Hz
GB                              0
PC                           1.00
```



```
Current Data Parameters
NAME                  Enaminone
EXPNO                        11
PROCNO                        1

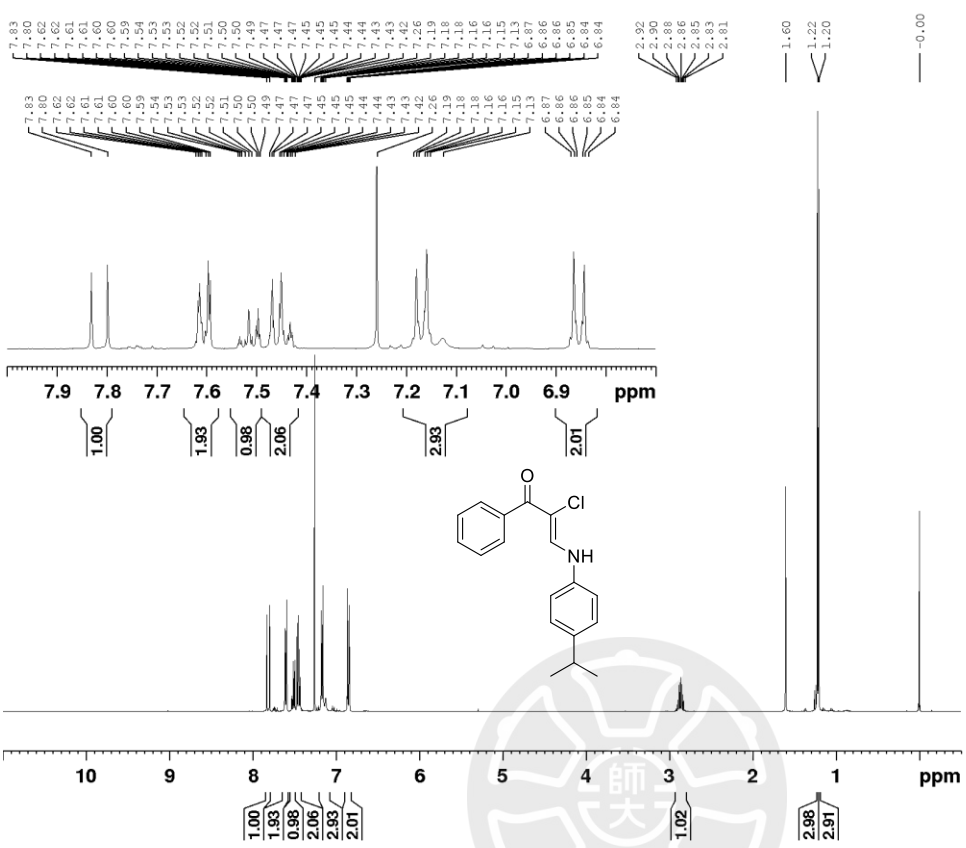
F2 - Acquisition Parameters
Date_                 20231101
Time                     11.57
INSTRUM                  spect
PROBHD    5 mm BBO BB-1H
PULPROG                zgpg30
TD                      32768
SOLVENT                 CDC13
NS                         1024
DS                          0
SWH                 24038.461 Hz
FIDRES               0.733596 Hz
AQ                   0.6815744 sec
RG                      32768
DW                      20.800 usec
DE                       6.50 usec
TE                      295.6 K
D1                   2.00000000 sec
D11                  0.03000000 sec
TD0                           1

======== CHANNEL f1 ========
NUC1                       13C
P1                      10.00 usec
PL1                      4.20 dB
SFO1               100.6233325 MHz

======== CHANNEL f2 ========
CPDPRG[2                waltz16
NUC2                        1H
PCPD2                   90.00 usec
PL2                     10.20 dB
PL12                    26.00 dB
PL13                    29.00 dB
SFO2               400.1316005 MHz

F2 - Processing parameters
SI                          32768
SF                 100.6127690 MHz
WDW                            EM
SSB                             0
LB                           1.00 Hz
GB                              0
PC                           1.00
```

(*Z*)-2-chloro-3-((4-isopropylphenyl)amino)-1-phenylprop-2-en-1-one (3af)

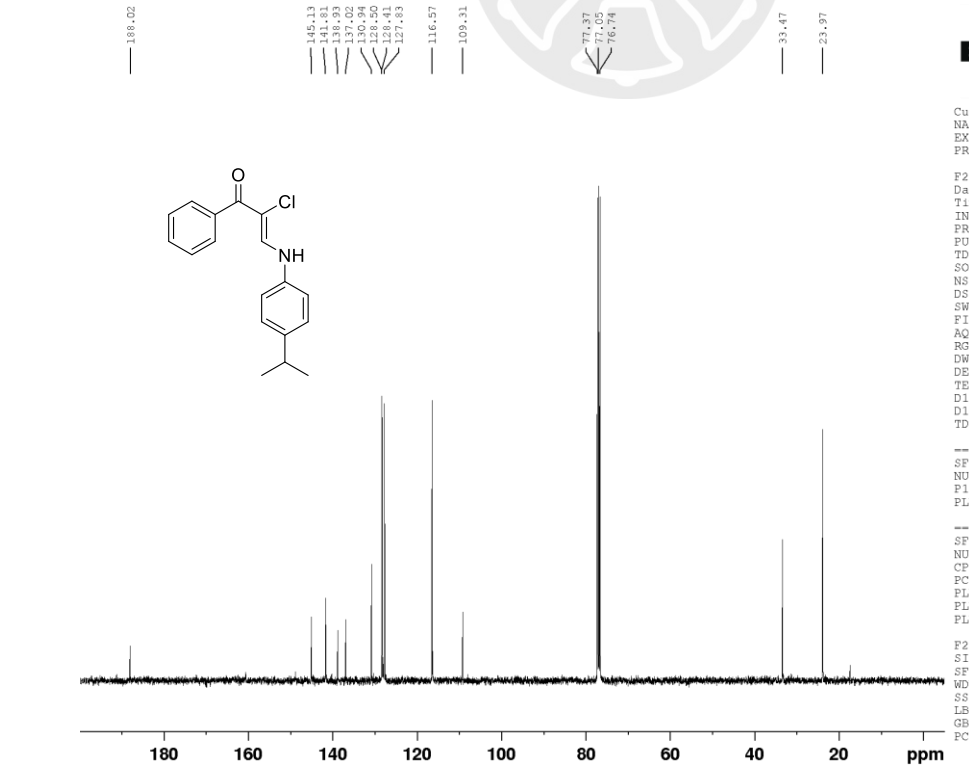


```
Current Data Parameters
NAME           Enaminone
EXPNO                 78
PROCNO                 1

F2 - Acquisition Parameters
Date_           20240705
Time               10.30
INSTRUM            spect
PROBHD    5 mm PABBO BB/
PULPROG            zg30
TD                32768
SOLVENT           CDCl3
NS                   16
DS                    0
SWH            7211.539 Hz
FIDRES        0.220079 Hz
AQ            2.2719147 sec
RG                128.9
DW               69.333 usec
DE                10.06 usec
TE                300.6 K
D1           2.00000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1        400.1324008 MHz
NUC1                   1H
P1                 15.00 usec
PLW1        13.50000000 W

F2 - Processing parameters
SI                 16384
SF          400.1300103 MHz
WDW                   EM
SSB                    0
LB                     0 Hz
GB                     0
PC                  1.00
```



```
Current Data Parameters
NAME          Suresh-701
EXPNO                  3
PROCNO                 1

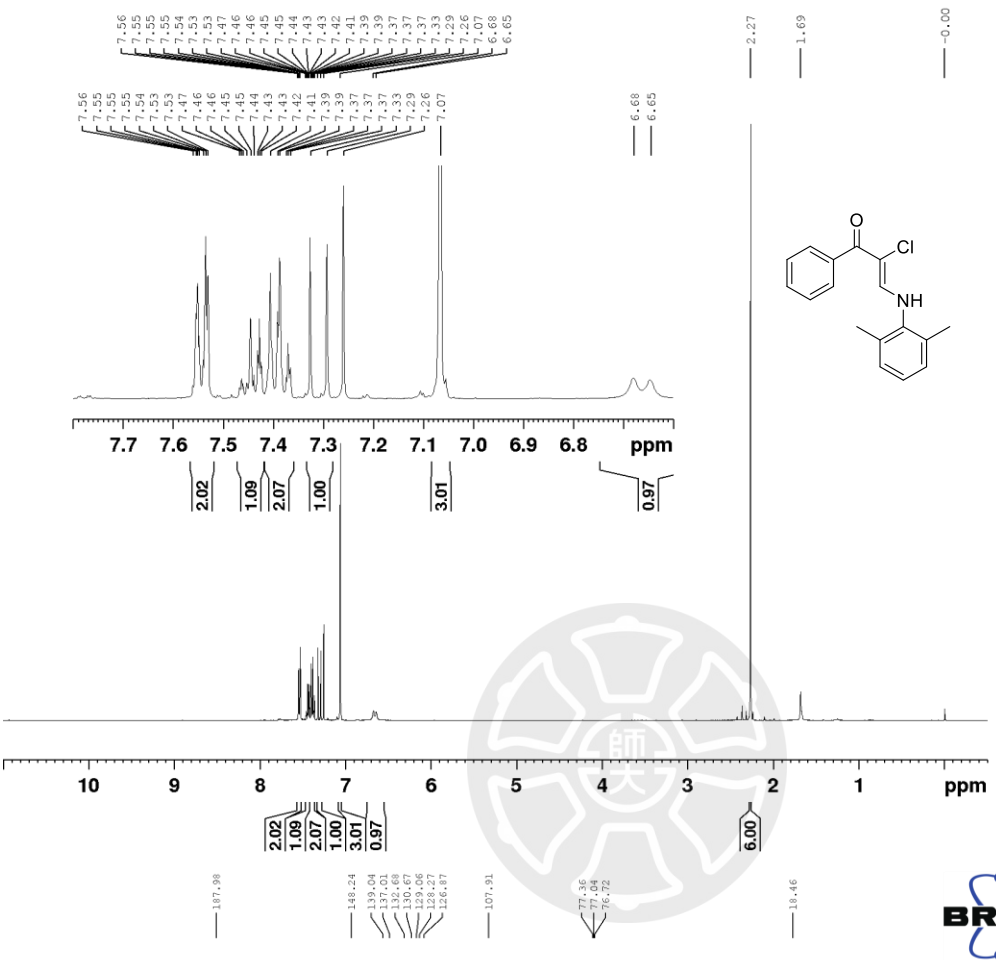
F2 - Acquisition Parameters
Date_           20231008
Time               13.18
INSTRUM            spect
PROBHD    5 mm PABBO BB/
PULPROG          zgpg30
TD                32768
SOLVENT           CDCl3
NS                  256
DS                    0
SWH           24038.461 Hz
FIDRES        0.733596 Hz
AQ            0.6815744 sec
RG                198.09
DW               20.800 usec
DE                 6.50 usec
TE                297.7 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1        100.6228298 MHz
NUC1                  13C
P1                 10.00 usec
PLW1        49.50000000 W

======== CHANNEL f2 ========
SFO2        400.1316005 MHz
NUC2                   1H
CPDPRG[2         waltz16
PCPD2              90.00 usec
PLW2        12.50000000 W
PLW12        0.34722000 W
PLW13        0.28125000 W

F2 - Processing parameters
SI                 32768
SF          100.6127685 MHz
WDW                   EM
SSB                    0
LB                  2.00 Hz
GB                     0
PC                  1.00
```

(*Z*)-2-chloro-3-((2,6-dimethylphenyl)amino)-1-phenylprop-2-en-1-one (3ag)



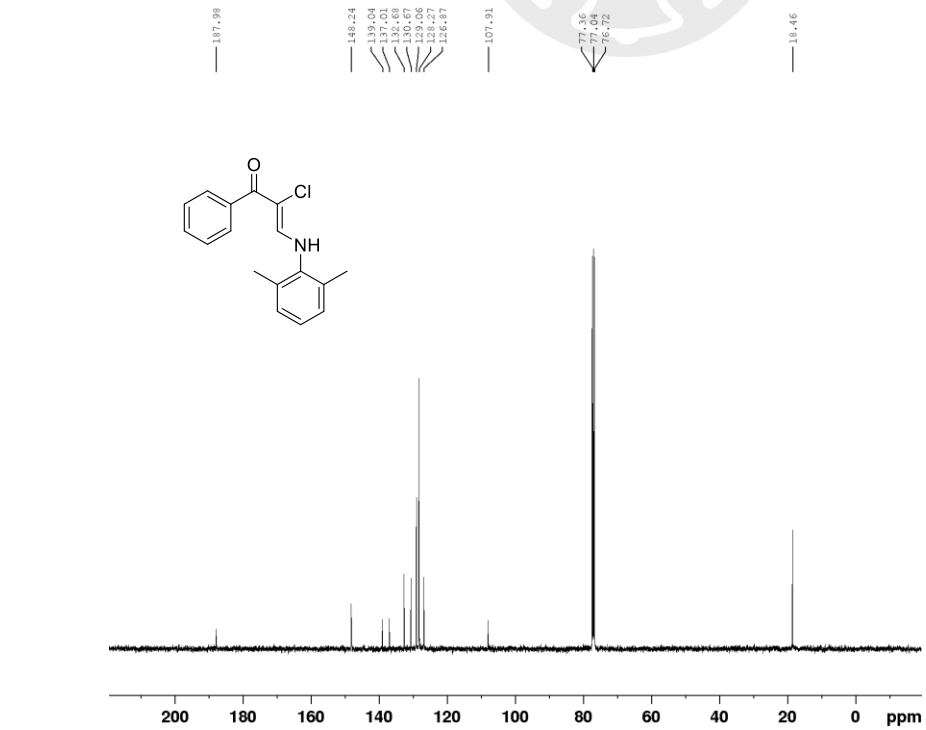
BRUKER

```
Current Data Parameters
NAME            Enaminone
EXPNO                  72
PROCNO                  1

F2 - Acquisition Parameters
Date_           20240702
Time               19.56
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG             zg30
TD                 32768
SOLVENT            CDC13
NS                    16
DS                     0
SWH             7211.539 Hz
FIDRES          0.220079 Hz
AQ              2.2719147 sec
RG                113.31
DW                69.333 usec
DE                10.06 usec
TE                298.2 K
D1             2.00000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1          400.1324008 MHz
NUC1                  1H
P1                 15.00 usec
PLW1          13.50000000 W

F2 - Processing parameters
SI                 16384
SF            400.1300103 MHz
WDW                   EM
SSB                    0
LB                     0 Hz
GB                     0
PC                  1.00
```



BRUKER

```
Current Data Parameters
NAME            Enaminone
EXPNO                  25
PROCNO                  1

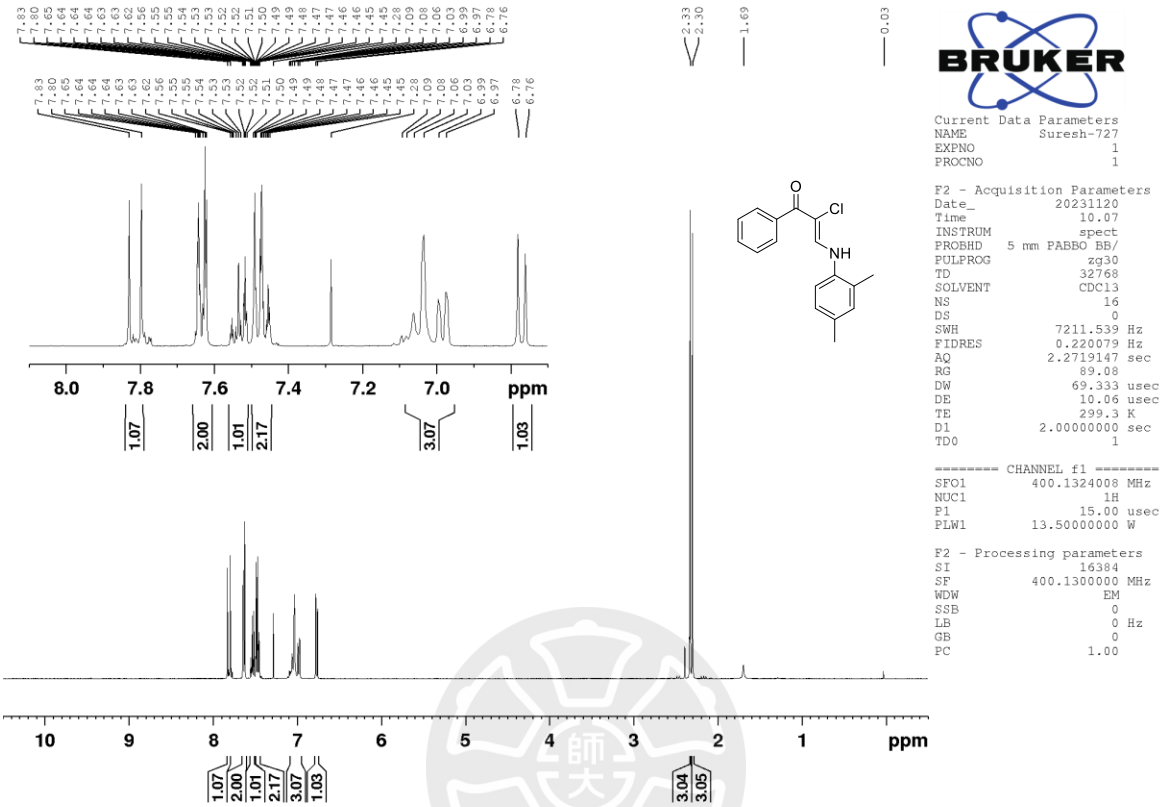
F2 - Acquisition Parameters
Date_           20231102
Time               21.51
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG           zgpg30
TD                 32768
SOLVENT            CDC13
NS                   256
DS                     0
SWH            24038.461 Hz
FIDRES          0.733596 Hz
AQ              0.6815744 sec
RG                198.09
DW                20.800 usec
DE                 6.50 usec
TE                299.2 K
D1             2.00000000 sec
D11            0.03000000 sec
TD0                    1

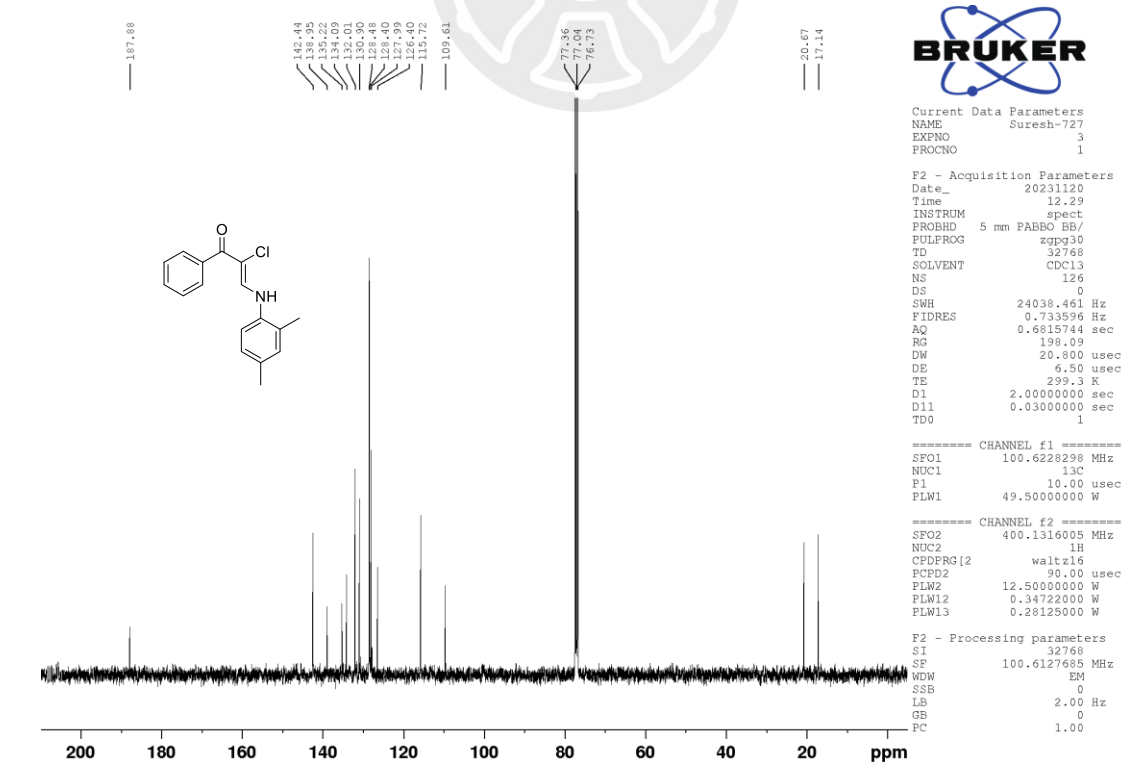
======== CHANNEL f1 ========
SFO1          100.6228298 MHz
NUC1                  13C
P1                 10.00 usec
PLW1          49.50000000 W

======== CHANNEL f2 ========
SFO2          400.1316005 MHz
NUC2                  1H
CPDPRG[2          waltz16
PCPD2              90.00 usec
PLW2          12.50000000 W
PLW12          0.34722000 W
PLW13          0.28125000 W

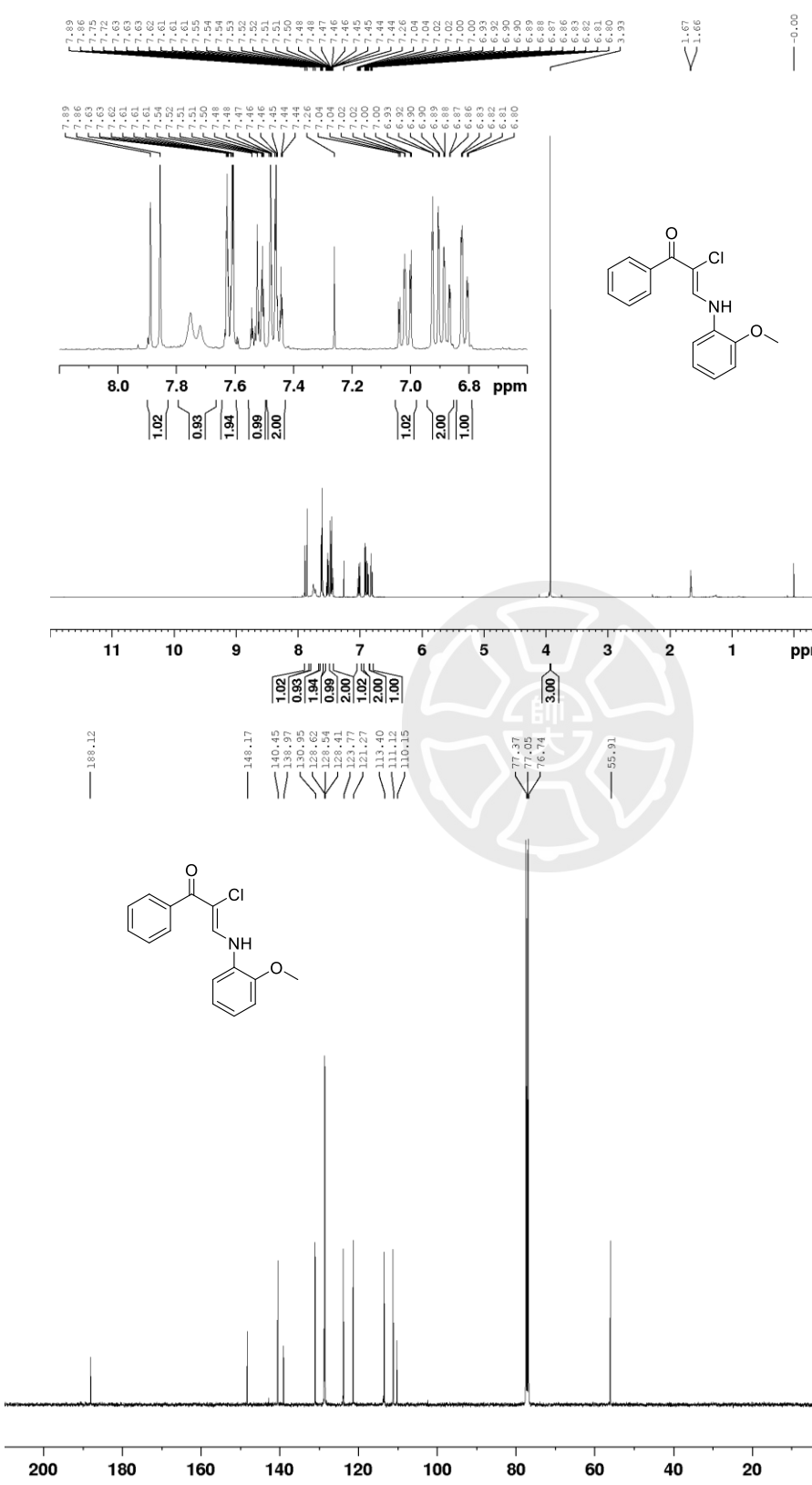
F2 - Processing parameters
SI                 32768
SF            100.6127685 MHz
WDW                   EM
SSB                    0
LB                  2.00 Hz
GB                     0
PC                  1.00
```

(*Z*)-2-chloro-3-((2,4-dimethylphenyl)amino)-1-phenylprop-2-en-1-one (3ah)





(*Z*)-2-chloro-3-((2-methoxyphenyl)amino)-1-phenylprop-2-en-1-one (3ai)



BRUKER

```
Current Data Parameters
NAME          Suresh-512
EXPNO                  2
PROCNO                 1

F2 - Acquisition Parameters
Date_           20221029
Time               15.55
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG            zg30
TD                32768
SOLVENT           CDC13
NS                   16
DS                    0
SWH           7211.539 Hz
FIDRES         0.220079 Hz
AQ            2.2719147 sec
RG                89.08
DW               69.333 usec
DE               10.06 usec
TE               298.2 K
D1            2.00000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1        400.1324008 MHz
NUC1                   1H
P1                 15.00 usec
PLW1        13.50000000 W

F2 - Processing parameters
SI                 16384
SF          400.1300098 MHz
WDW                   EM
SSB                    0
LB                  0 Hz
GB                     0
PC                  1.00
```

BRUKER

```
Current Data Parameters
NAME          Suresh-512
EXPNO                  4
PROCNO                 1

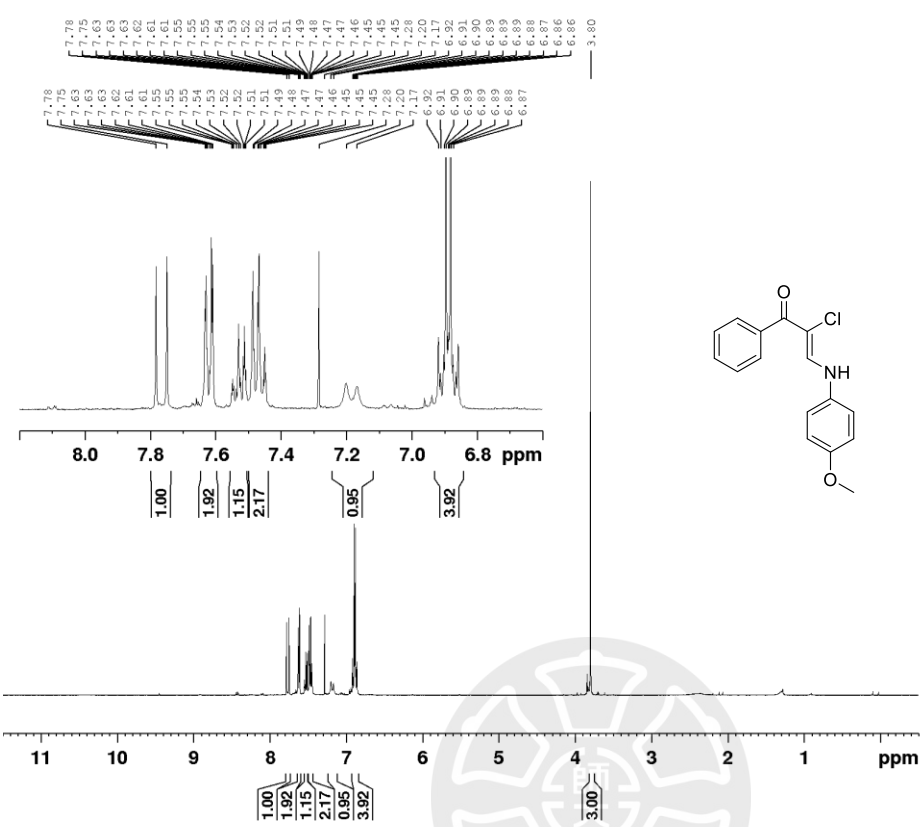
F2 - Acquisition Parameters
Date_           20221029
Time               16.13
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG          zgpg30
TD                32768
SOLVENT           CDC13
NS                 1495
DS                    0
SWH           24038.461 Hz
FIDRES         0.733596 Hz
AQ            0.6815744 sec
RG                198.09
DW               20.800 usec
DE                6.50 usec
TE               298.9 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1        100.6228298 MHz
NUC1                   13C
P1                 10.00 usec
PLW1        49.50000000 W

======== CHANNEL f2 ========
SFO2        400.1316005 MHz
NUC2                   1H
CPDPRG[2         waltz16
PCPD2              90.00 usec
PLW2        12.50000000 W
PLW12        0.34722000 W
PLW13        0.28125000 W

F2 - Processing parameters
SI                 32768
SF          100.6127685 MHz
WDW                   EM
SSB                    0
LB                  2.00 Hz
GB                     0
PC                  1.00
```

(Z)-2-chloro-3-((4-methoxyphenyl)amino)-1-phenylprop-2-en-1-one (3aj)



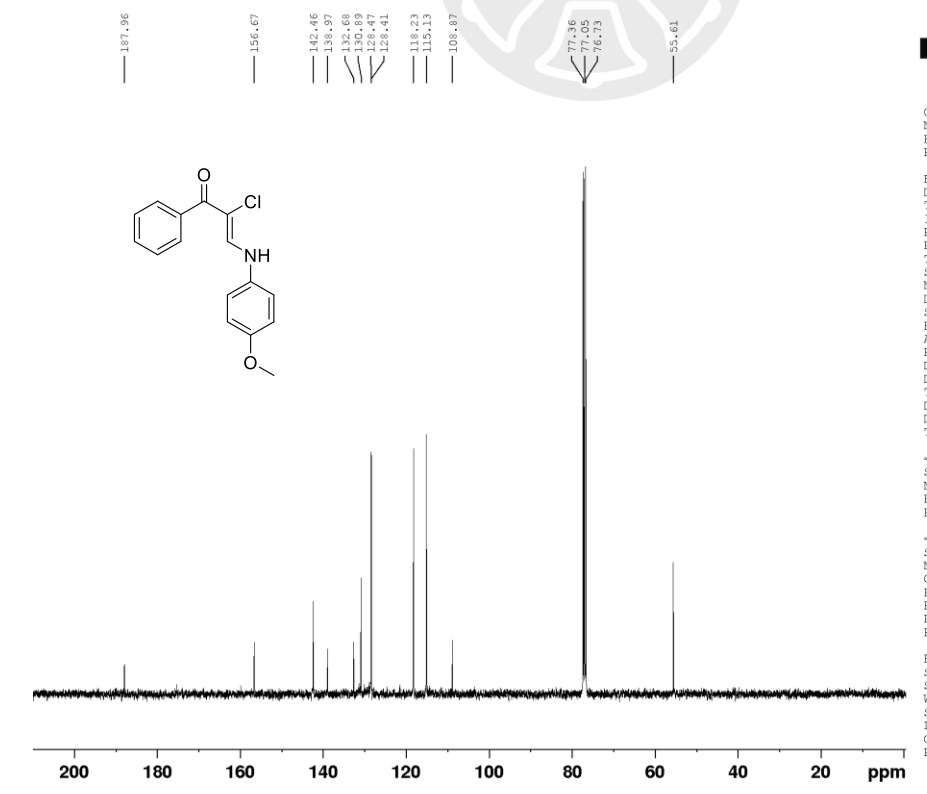
BRUKER

```
Current Data Parameters
NAME                  20230307
EXPNO                        3
PROCNO                       1

F2 - Acquisition Parameters
Date_                 20230307
Time                     13.48
INSTRUM                  spect
PROBHD    5 mm PABBO BB/
PULPROG                  zg30
TD                      32768
SOLVENT                 CDCl3
NS                           1
DS                           0
SWH                  7211.539 Hz
FIDRES               0.220079 Hz
AQ                   2.2719147 sec
RG                      113.31
DW                      69.333 usec
DE                      10.06 usec
TE                      299.2 K
D1                   2.00000000 sec
TD0                          1

======== CHANNEL f1 ========
SFO1                400.1324008 MHz
NUC1                        1H
P1                       15.00 usec
PLW1                13.50000000 W

F2 - Processing parameters
SI                          16384
SF                  400.1300000 MHz
WDW                            EM
SSB                          0
LB                           0 Hz
GB                           0
PC                        1.00
```



BRUKER

```
Current Data Parameters
NAME                  20230307
EXPNO                        4
PROCNO                       1

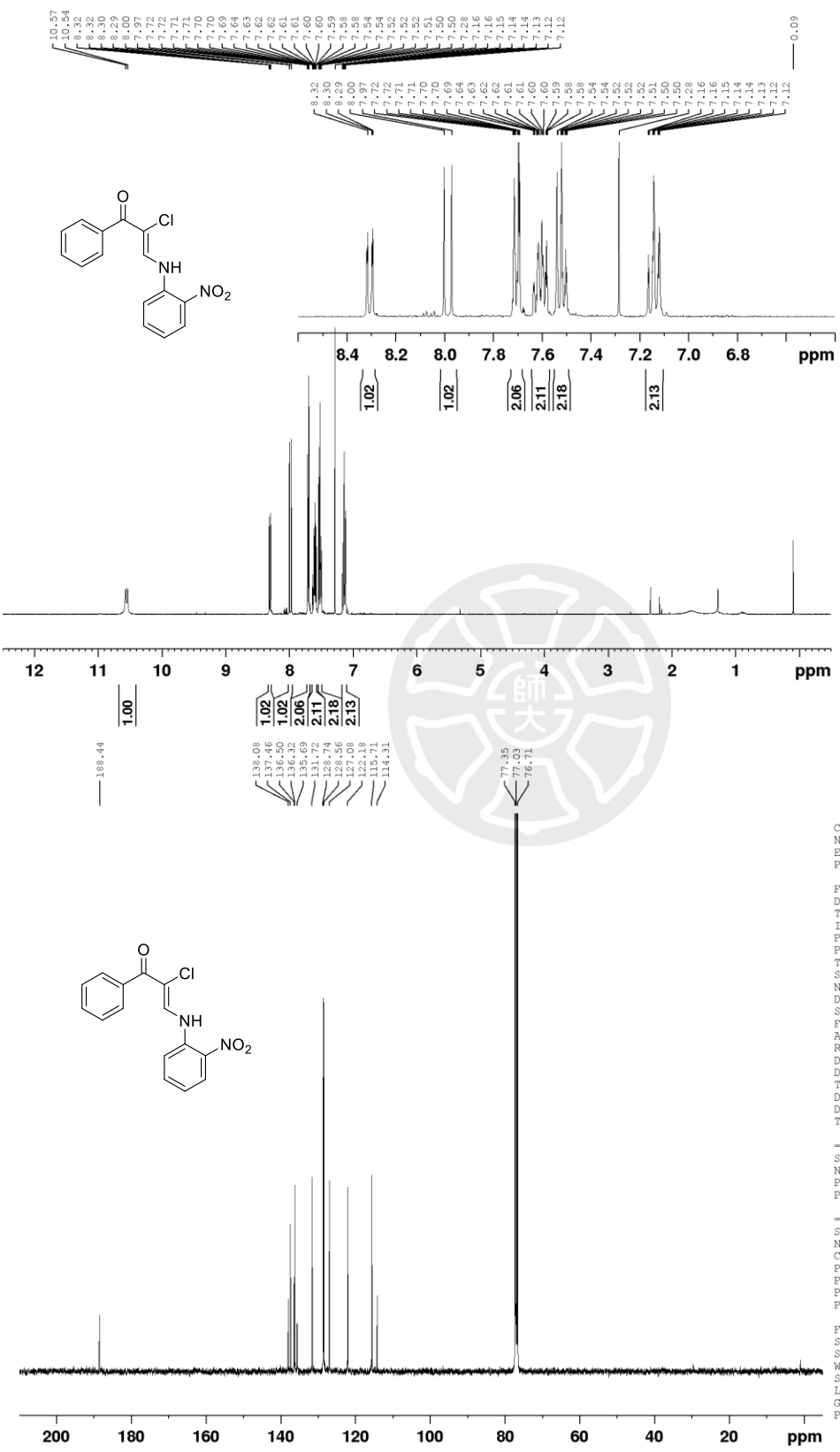
F2 - Acquisition Parameters
Date_                 20230307
Time                     13.51
INSTRUM                  spect
PROBHD    5 mm PABBO BB/
PULPROG                zgpg30
TD                      32768
SOLVENT                 CDCl3
NS                         238
DS                           0
SWH                 24038.461 Hz
FIDRES               0.733596 Hz
AQ                   0.6815744 sec
RG                      198.09
DW                      20.800 usec
DE                       6.50 usec
TE                      299.9 K
D1                   2.00000000 sec
D11                  0.03000000 sec
TD0                          1

======== CHANNEL f1 ========
SFO1                100.6228298 MHz
NUC1                        13C
P1                       10.00 usec
PLW1                49.50000000 W

======== CHANNEL f2 ========
SFO2                400.1316005 MHz
NUC2                        1H
CPDPRG[2                waltz16
PCPD2                    90.00 usec
PLW2                12.50000000 W
PLW12                0.34722000 W
PLW13                0.28125000 W

F2 - Processing parameters
SI                          32768
SF                  100.6127685 MHz
WDW                            EM
SSB                          0
LB                        2.00 Hz
GB                           0
PC                        1.00
```

(*Z*)-2-chloro-3-((2-nitrophenyl)amino)-1-phenylprop-2-en-1-one (3ak)



BRUKER

```
Current Data Parameters
NAME              20230309
EXPNO                    3
PROCNO                   1

F2 - Acquisition Parameters
Date_             20230309
Time                 16.54
INSTRUM              spect
PROBHD    5 mm PABBO BB/
PULPROG               zg30
TD                   32768
SOLVENT              CDC13
NS                       8
DS                       0
SWH               7211.539 Hz
FIDRES            0.220079 Hz
AQ                2.2719147 sec
RG                   198.09
DW                  69.333 usec
DE                  10.06 usec
TE                  299.0 K
D1              2.00000000 sec
TD0                      1

======== CHANNEL f1 ========
SFO1            400.1324008 MHz
NUC1                    1H
P1                   15.00 usec
PLW1           13.50000000 W

F2 - Processing parameters
SI                   16384
SF              400.1300000 MHz
WDW                     EM
SSB                      0
LB                    0 Hz
GB                       0
PC                    1.00
```

BRUKER

```
Current Data Parameters
NAME              20230309
EXPNO                    4
PROCNO                   1

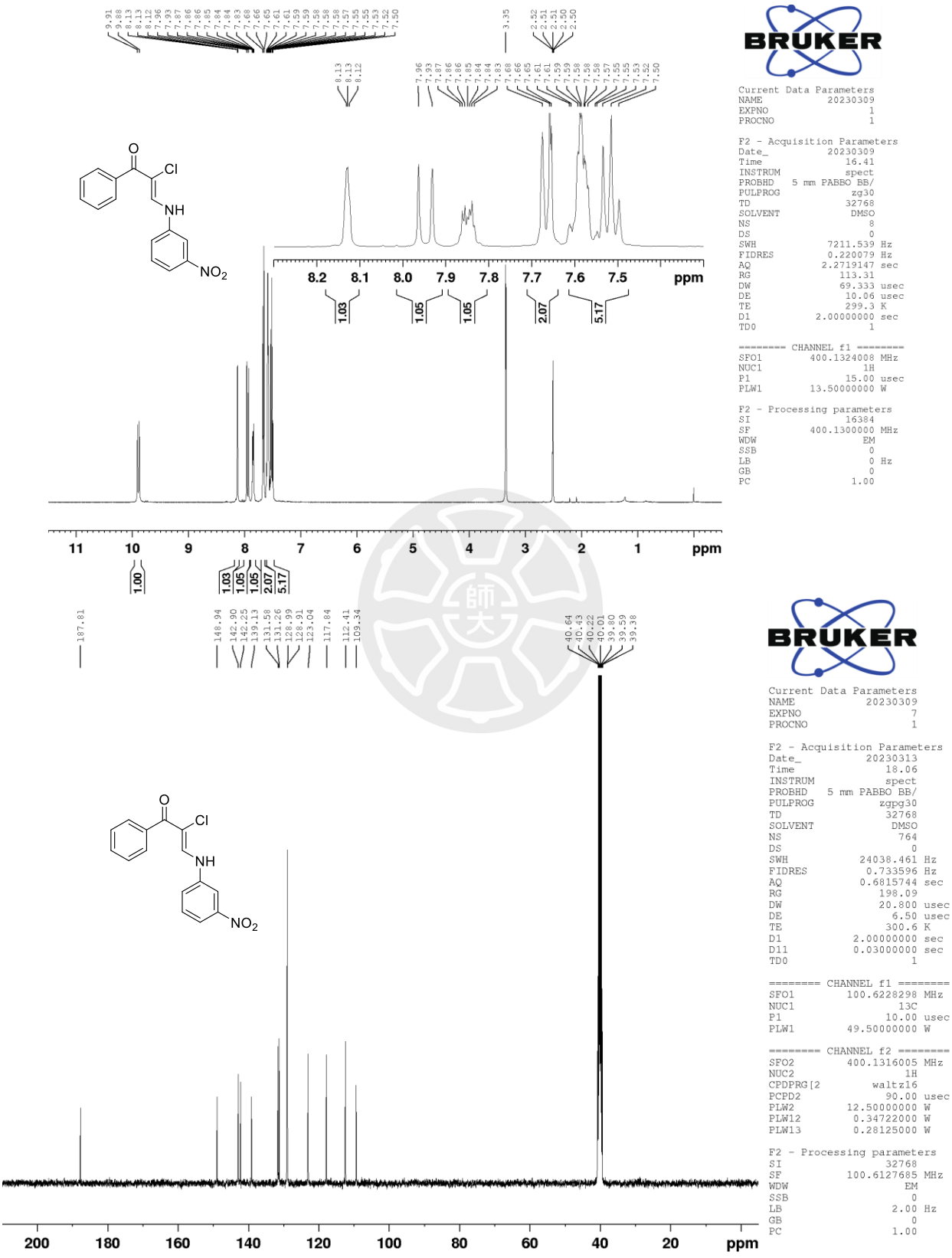
F2 - Acquisition Parameters
Date_             20230313
Time                 18.11
INSTRUM              spect
PROBHD    5 mm PABBO BB/
PULPROG             zgpg30
TD                   32768
SOLVENT              CDC13
NS                    1673
DS                       0
SWH              24038.461 Hz
FIDRES            0.733596 Hz
AQ                0.6815744 sec
RG                   198.09
DW                  20.800 usec
DE                   6.50 usec
TE                  300.0 K
D1              2.00000000 sec
D11             0.03000000 sec
TD0                      1

======== CHANNEL f1 ========
SFO1            100.6228298 MHz
NUC1                    13C
P1                   10.00 usec
PLW1           49.50000000 W

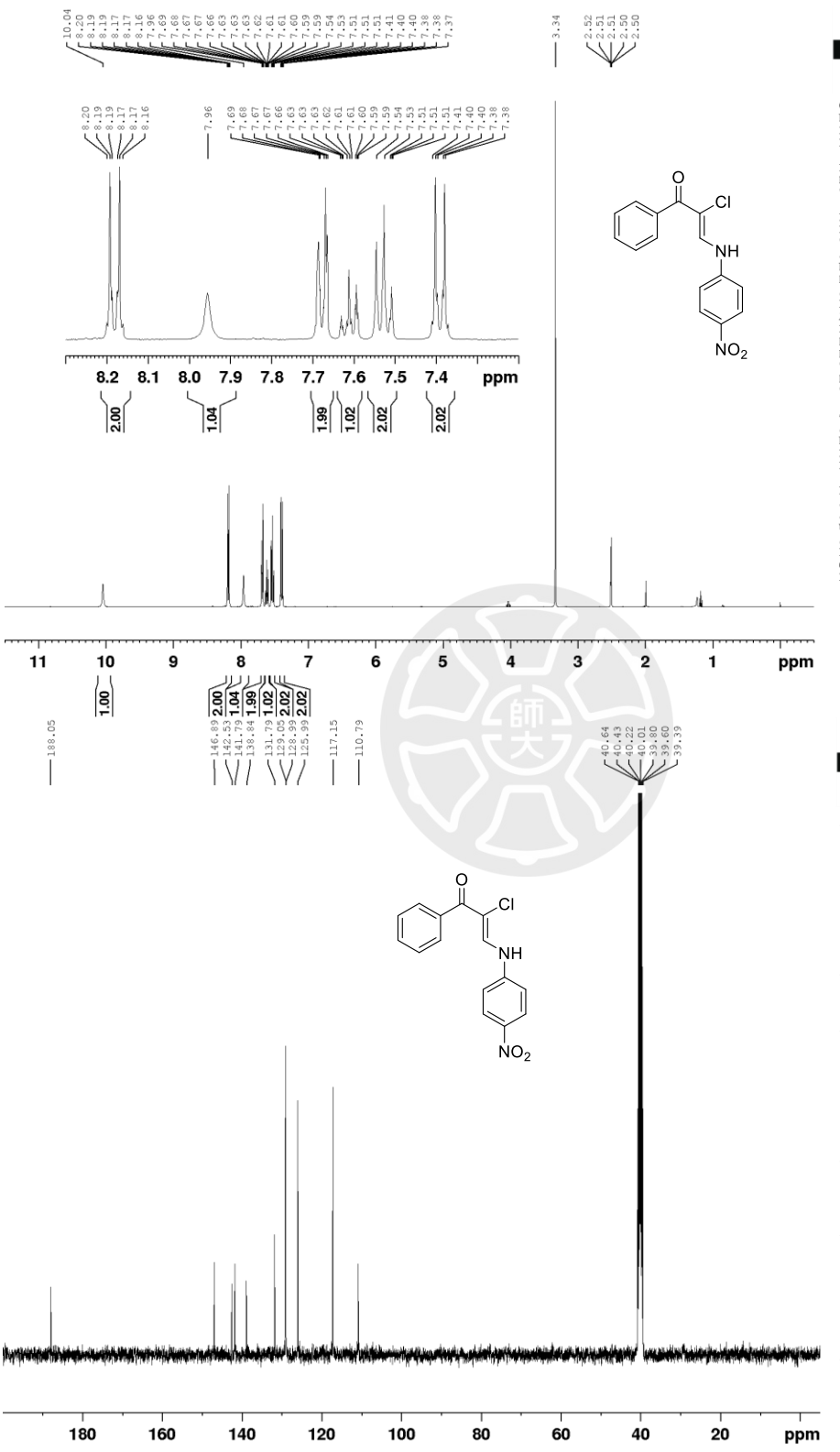
======== CHANNEL f2 ========
SFO2            400.1316005 MHz
NUC2                    1H
CPDPRG[2             waltz16
PCPD2                90.00 usec
PLW2           12.50000000 W
PLW12           0.34722000 W
PLW13           0.28125000 W

F2 - Processing parameters
SI                   32768
SF              100.6127685 MHz
WDW                     EM
SSB                      0
LB                    2.00 Hz
GB                       0
PC                    1.00
```

(*Z*)-2-chloro-3-((3-nitrophenyl)amino)-1-phenylprop-2-en-1-one (3al)



(*Z*)-2-chloro-3-((4-nitrophenyl)amino)-1-phenylprop-2-en-1-one (3am)



```
Current Data Parameters
NAME                 Enaminone
EXPNO                       33
PROCNO                       1

F2 - Acquisition Parameters
Date_               20231107
Time                   15.07
INSTRUM                spect
PROBHD    5 mm PABBO BB/
PULPROG                zg30
TD                    32768
SOLVENT                DMSO
NS                       16
DS                        0
SWH                7211.539 Hz
FIDRES                0.220079 Hz
AQ                 2.2719147 sec
RG                     128.9
DW                    69.333 usec
DE                    10.06 usec
TE                    298.3 K
D1                 2.00000000 sec
TD0                          1

======== CHANNEL f1 ========
SFO1          400.1324008 MHz
NUC1                     1H
P1                    15.00 usec
PLW1          13.50000000 W

F2 - Processing parameters
SI                       16384
SF            400.1300000 MHz
WDW                         EM
SSB                          0
LB                        0 Hz
GB                           0
PC                        1.00
```

```
Current Data Parameters
NAME                 Enaminone
EXPNO                       32
PROCNO                       1

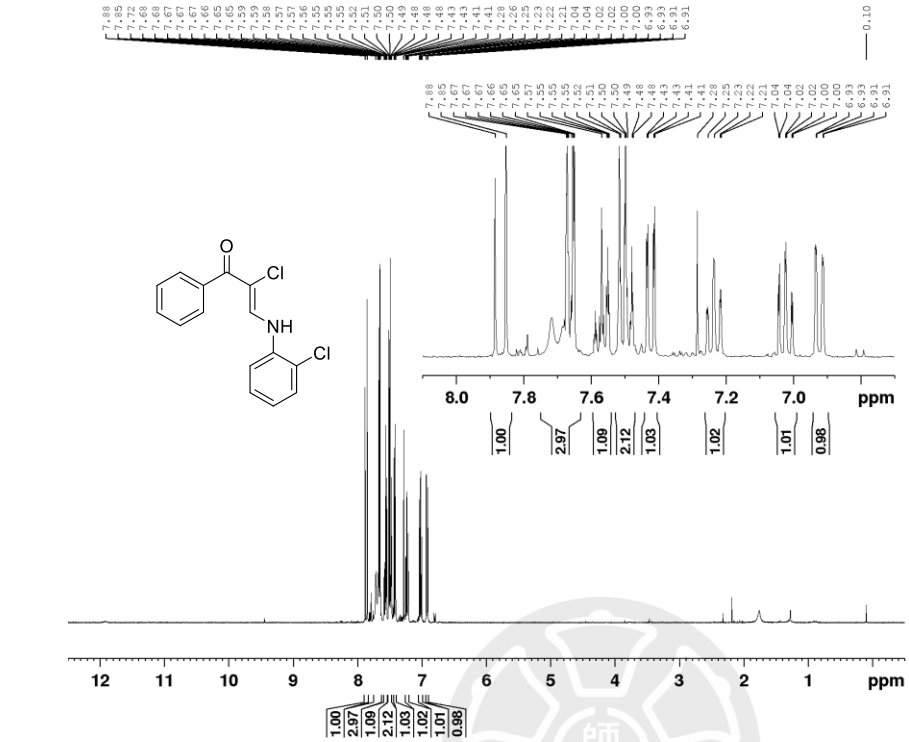
F2 - Acquisition Parameters
Date_               20231107
Time                   14.09
INSTRUM                spect
PROBHD    5 mm PABBO BB/
PULPROG             zgpg30
TD                    32768
SOLVENT                DMSO
NS                      256
DS                        0
SWH            24038.461 Hz
FIDRES             0.733596 Hz
AQ                 0.6815744 sec
RG                     198.09
DW                    20.800 usec
DE                     6.50 usec
TE                    298.8 K
D1                 2.00000000 sec
D11                0.03000000 sec
TD0                          1

======== CHANNEL f1 ========
SFO1          100.6228298 MHz
NUC1                     13C
P1                    10.00 usec
PLW1          49.50000000 W

======== CHANNEL f2 ========
SFO2          400.1316005 MHz
NUC2                     1H
CPDPRG[2          waltz16
PCPD2                 90.00 usec
PLW2          12.50000000 W
PLW12          0.34722000 W
PLW13          0.28125000 W

F2 - Processing parameters
SI                       32768
SF            100.6127685 MHz
WDW                         EM
SSB                          0
LB                    2.00 Hz
GB                           0
PC                        1.00
```

(*Z*)-2-chloro-3-((2-chlorophenyl)amino)-1-phenylprop-2-en-1-one (3an)



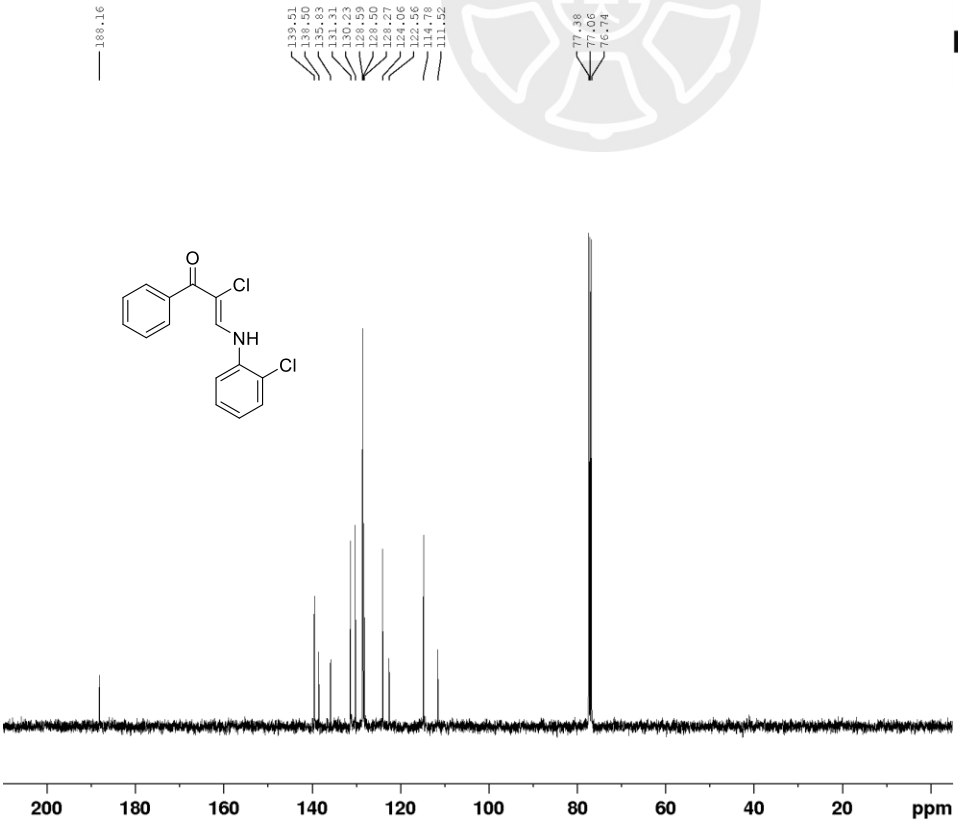
BRUKER

```
Current Data Parameters
NAME                 20230307
EXPNO                       5
PROCNO                      1

F2 - Acquisition Parameters
Date_               20230307
Time                   14.04
INSTRUM                spect
PROBHD    5 mm PABBO BB/
PULPROG                zg30
TD                    32768
SOLVENT               CDC13
NS                        1
DS                        0
SWH                7211.539 Hz
FIDRES            0.220079 Hz
AQ                2.2719147 sec
RG                     89.08
DW                  69.333 usec
DE                  10.06 usec
TE                  299.3 K
D1                2.00000000 sec
TD0                       1

======== CHANNEL f1 ========
SFO1            400.1324008 MHz
NUC1                      1H
P1                    15.00 usec
PLW1           13.50000000 W

F2 - Processing parameters
SI                    16384
SF              400.1300000 MHz
WDW                      EM
SSB                       0
LB                     0 Hz
GB                        0
PC                     1.00
```



BRUKER

```
Current Data Parameters
NAME                 20230307
EXPNO                       6
PROCNO                      1

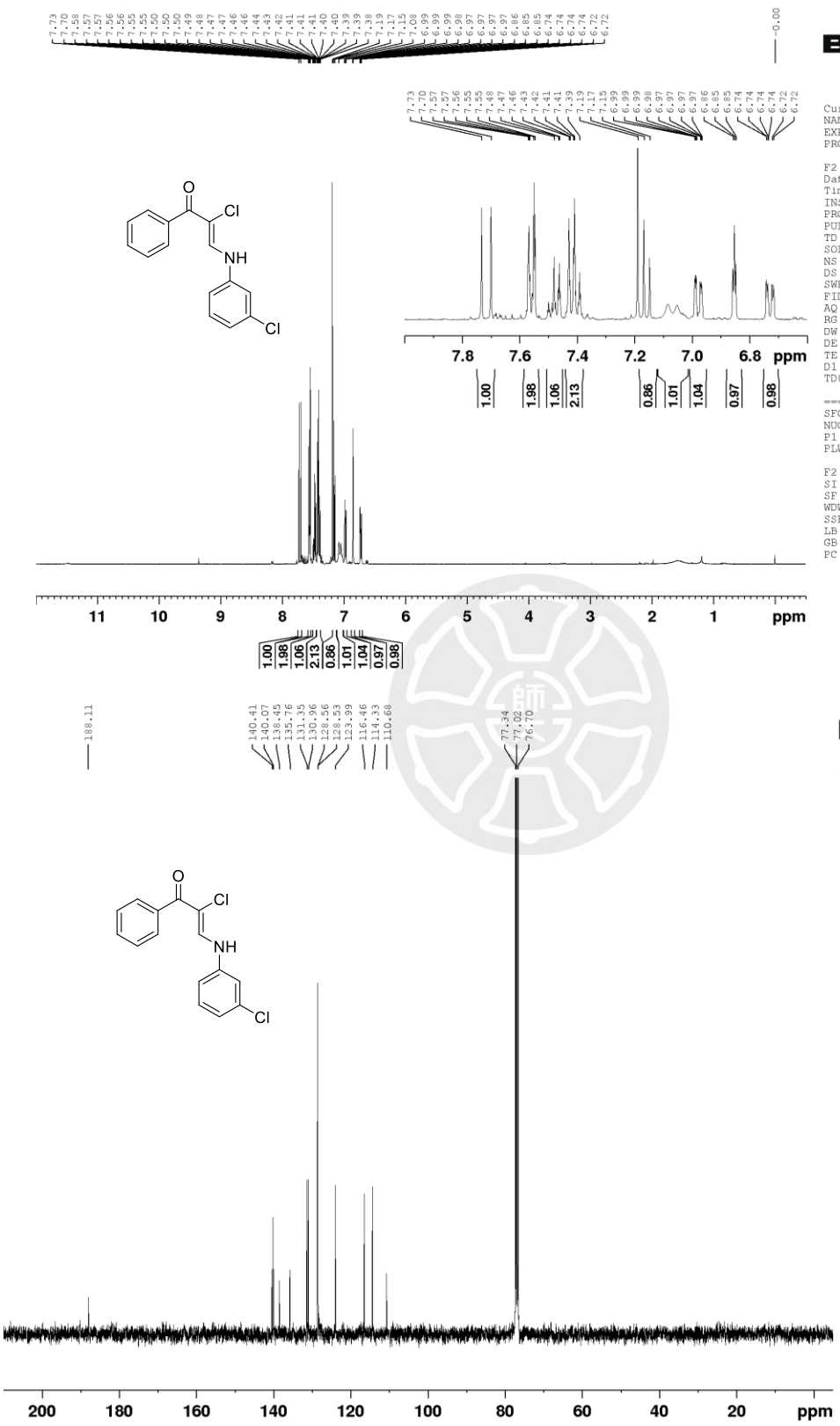
F2 - Acquisition Parameters
Date_               20230307
Time                   14.08
INSTRUM                spect
PROBHD    5 mm PABBO BB/
PULPROG             zgpg30
TD                    32768
SOLVENT               CDC13
NS                       88
DS                        0
SWH               24038.461 Hz
FIDRES            0.733596 Hz
AQ                0.6815744 sec
RG                    198.09
DW                  20.800 usec
DE                   6.50 usec
TE                  300.1 K
D1                2.00000000 sec
D11               0.03000000 sec
TD0                       1

======== CHANNEL f1 ========
SFO1            100.6228298 MHz
NUC1                      13C
P1                    10.00 usec
PLW1           49.50000000 W

======== CHANNEL f2 ========
SFO2            400.1316005 MHz
NUC2                      1H
CPDPRG[2             waltz16
PCPD2                 90.00 usec
PLW2           12.50000000 W
PLW12           0.34722000 W
PLW13           0.28125000 W

F2 - Processing parameters
SI                    32768
SF              100.6127685 MHz
WDW                      EM
SSB                       0
LB                    2.00 Hz
GB                        0
PC                     1.00
```

(*Z*)-2-chloro-3-((3-chlorophenyl)amino)-1-phenylprop-2-en-1-one (3ao)



BRUKER

Current Data Parameters
NAME 20230306
EXPNO 4
PROCNO 1

F2 - Acquisition Parameters
Date_ 20230306
Time 14.46
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 8
DS 0
SWH 7211.539 Hz
FIDRES 0.220079 Hz
AQ 2.2719147 sec
RG 158.74
DW 69.333 usec
DE 10.06 usec
TE 299.3 K
D1 2.00000000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 400.1324008 MHz
NUC1 1H
P1 15.00 usec
PLW1 13.50000000 W

F2 - Processing parameters
SI 16384
SF 400.1300380 MHz
WDW EM
SSB 0
LB 0 Hz
GB 0
PC 1.00

BRUKER

Current Data Parameters
NAME 20230306
EXPNO 5
PROCNO 1

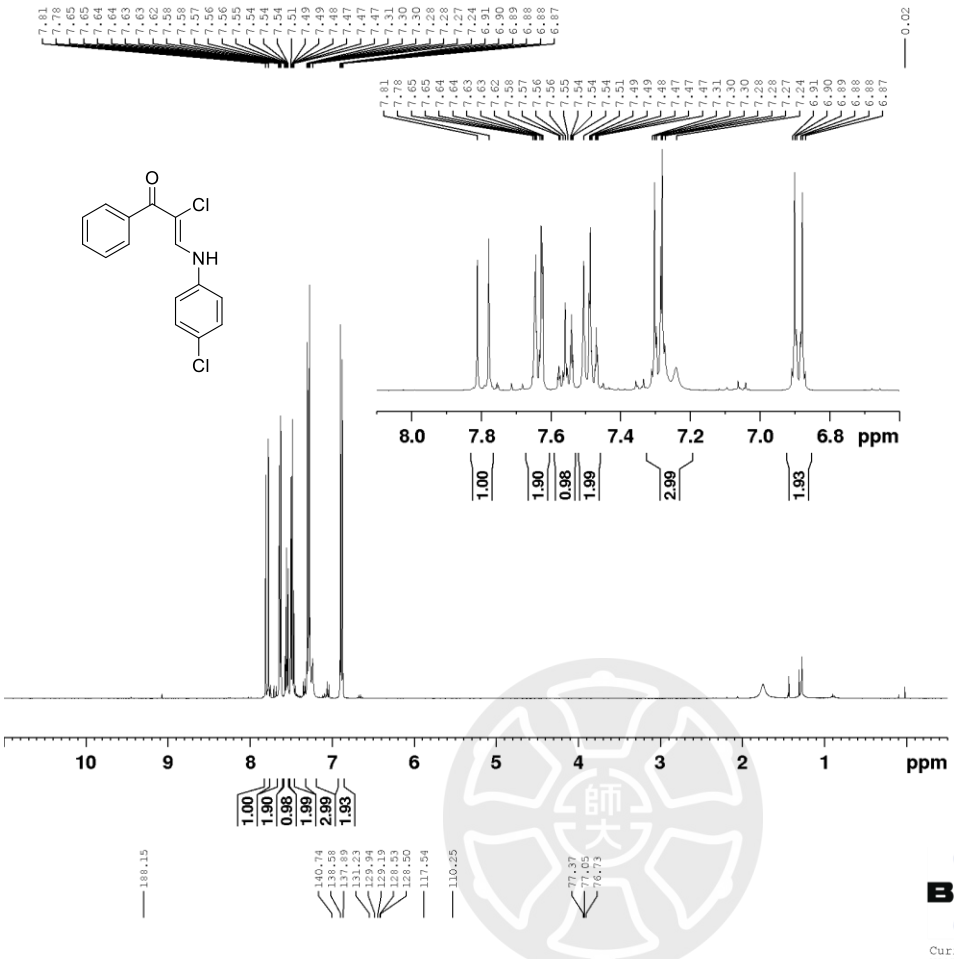
F2 - Acquisition Parameters
Date_ 20230306
Time 14.50
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 430
DS 0
SWH 24038.461 Hz
FIDRES 0.733596 Hz
AQ 0.6815744 sec
RG 198.09
DW 20.800 usec
DE 6.50 usec
TE 299.9 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 100.6228298 MHz
NUC1 13C
P1 10.00 usec
PLW1 49.50000000 W

======== CHANNEL f2 ========
SFO2 400.1316005 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 90.00 usec
PLW2 12.50000000 W
PLW12 0.34722000 W
PLW13 0.28125000 W

F2 - Processing parameters
SI 32768
SF 100.6127685 MHz
WDW EM
SSB 0
LB 2.00 Hz
GB 0
PC 1.00

(*Z*)-2-chloro-3-((4-chlorophenyl)amino)-1-phenylprop-2-en-1-one (3ap)

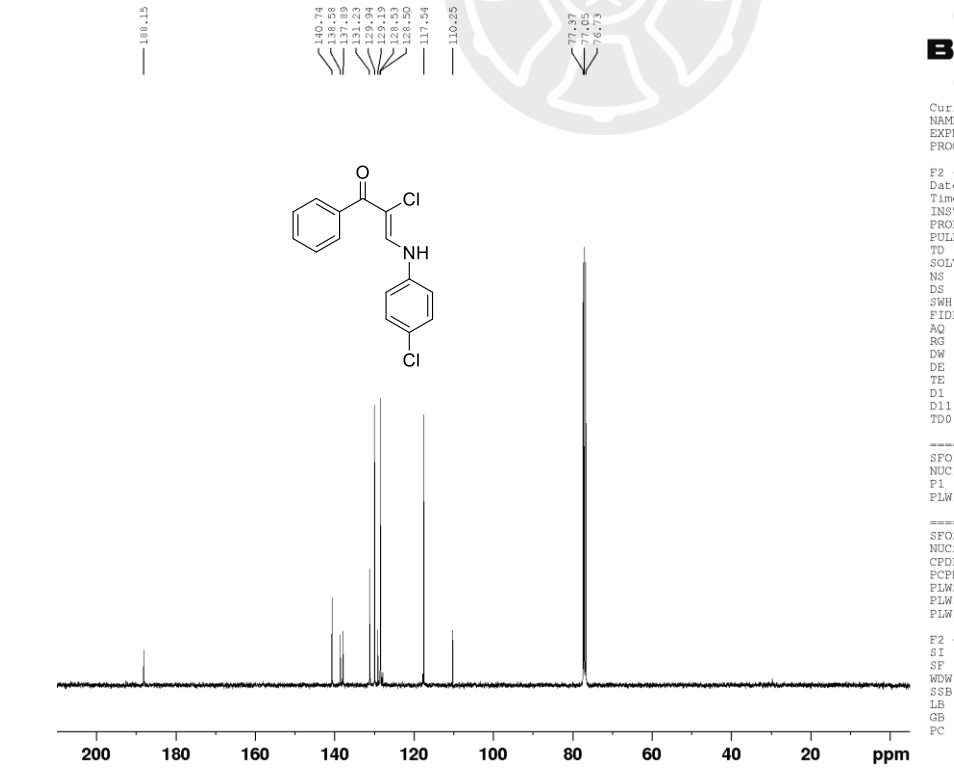



```
Current Data Parameters
NAME          Suresh-722
EXPNO                  4
PROCNO                 1

F2 - Acquisition Parameters
Date_           20231124
Time               21.22
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG            zg30
TD                32768
SOLVENT           CDCl3
NS                   16
DS                    0
SWH           7211.539 Hz
FIDRES        0.220079 Hz
AQ            2.2719147 sec
RG               113.31
DW               69.333 usec
DE               10.06 usec
TE               298.2 K
D1            2.00000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1         400.1324008 MHz
NUC1                   1H
P1                 15.00 usec
PLW1        13.50000000 W

F2 - Processing parameters
SI                 16384
SF            400.1300000 MHz
WDW                   EM
SSB                    0
LB                     0 Hz
GB                     0
PC                  1.00
```




```
Current Data Parameters
NAME          Suresh-722
EXPNO                  5
PROCNO                 1

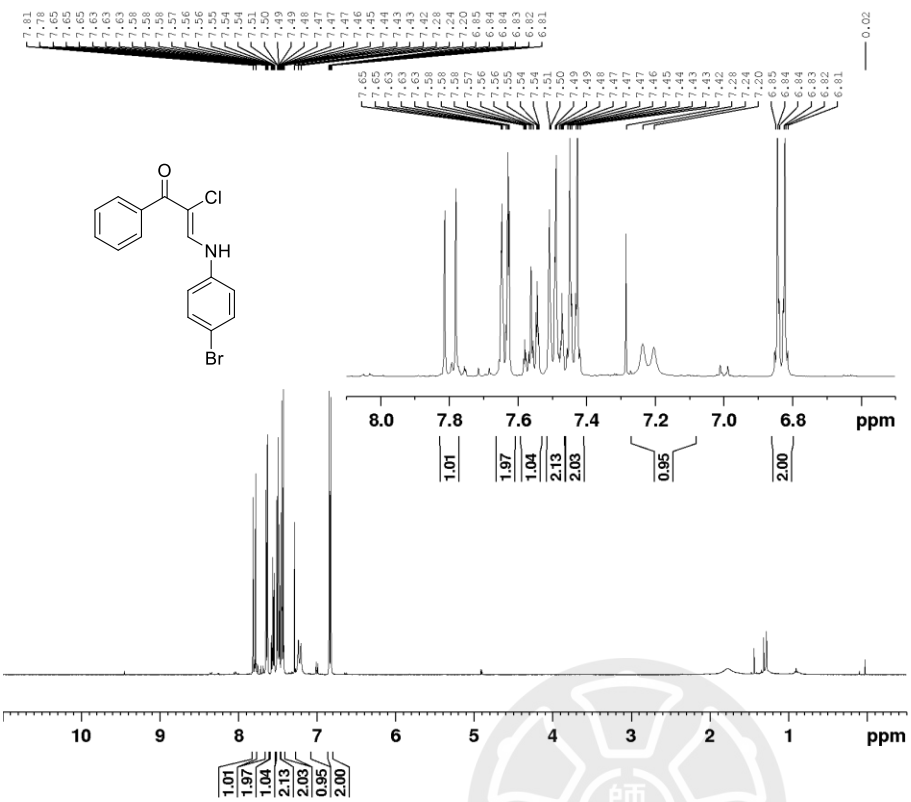
F2 - Acquisition Parameters
Date_           20231124
Time               21.24
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG          zgpg30
TD                32768
SOLVENT           CDCl3
NS                  512
DS                    0
SWH          24038.461 Hz
FIDRES        0.733596 Hz
AQ            0.6815744 sec
RG               198.09
DW               20.800 usec
DE                6.50 usec
TE               298.3 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1         100.6228298 MHz
NUC1                  13C
P1                 10.00 usec
PLW1        49.50000000 W

======== CHANNEL f2 ========
SFO2         400.1316005 MHz
NUC2                   1H
CPDPRG[2         waltz16
PCPD2              90.00 usec
PLW2        12.50000000 W
PLW12        0.34722000 W
PLW13        0.28125000 W

F2 - Processing parameters
SI                 32768
SF            100.6127685 MHz
WDW                   EM
SSB                    0
LB                  2.00 Hz
GB                     0
PC                  1.00
```

(*Z*)-3-((4-bromophenyl)amino)-2-chloro-1-phenylprop-2-en-1-one (3aq)



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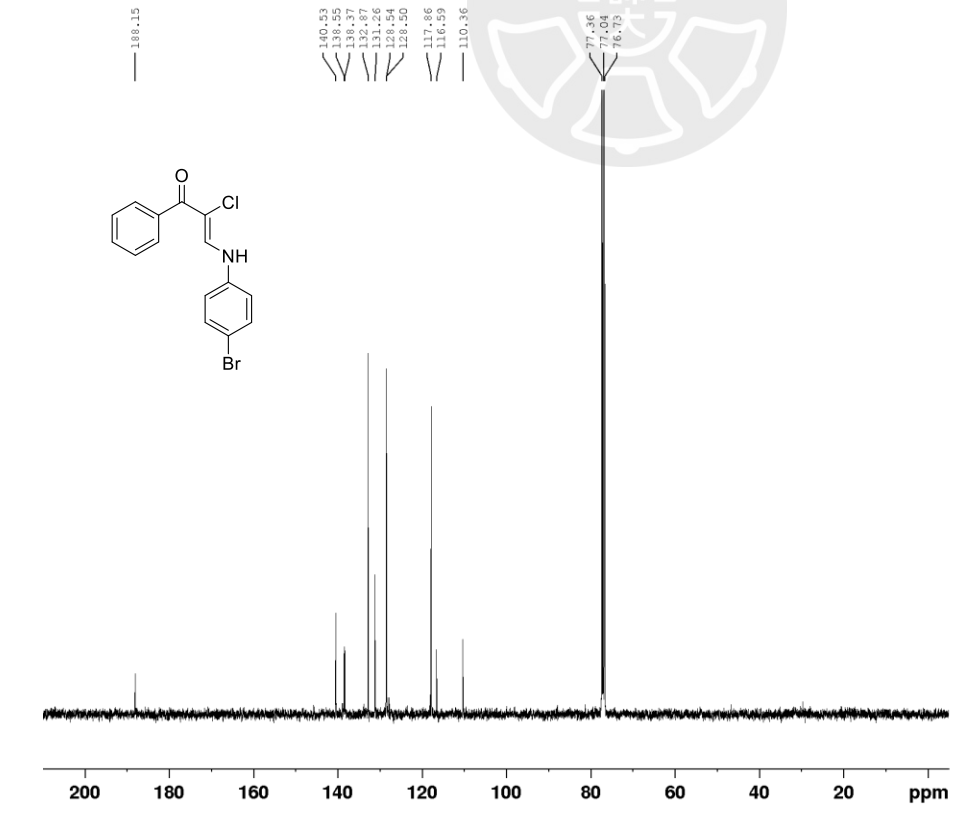
```
Current Data Parameters
NAME          Suresh-734
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20231201
Time               11.02
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG             zg30
TD                 32768
SOLVENT            CDC13
NS                    16
DS                     0
SWH             7211.539 Hz
FIDRES          0.220079 Hz
AQ              2.2719147 sec
RG                 128.9
DW                69.333 usec
DE                10.06 usec
TE                298.3 K
D1            2.00000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1         400.1324008 MHz
NUC1                   1H
P1                 15.00 usec
PLW1         13.50000000 W

F2 - Processing parameters
SI                 16384
SF           400.1300000 MHz
WDW                   EM
SSB                    0
LB                     0 Hz
GB                     0
PC                  1.00
```



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```
Current Data Parameters
NAME          Suresh-734
EXPNO                  4
PROCNO                 1

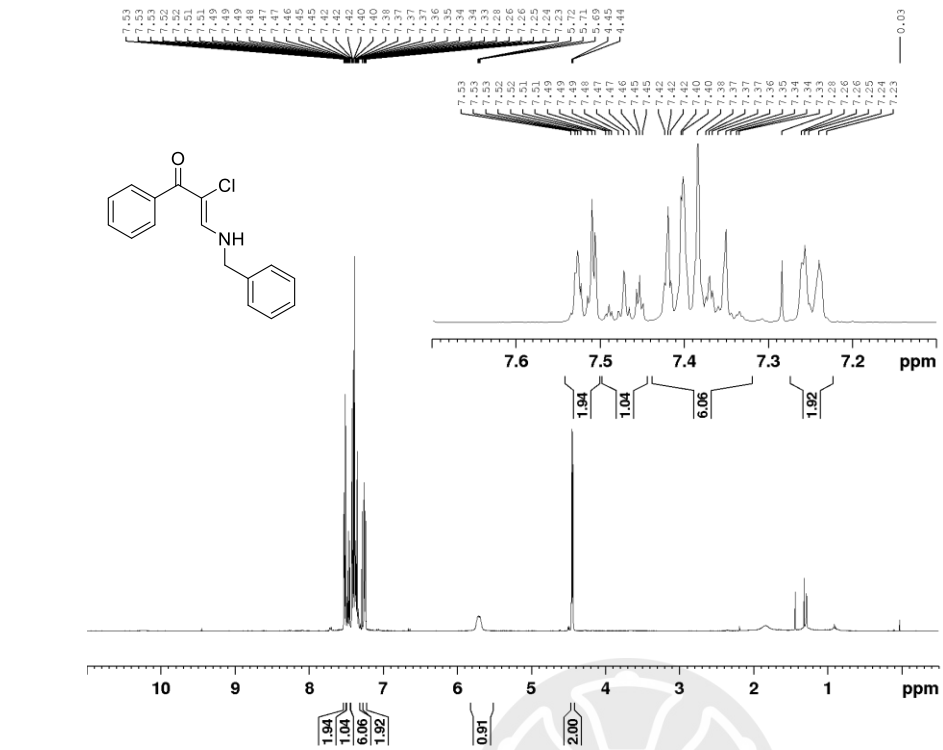
F2 - Acquisition Parameters
Date_           20231204
Time               21.30
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG          zgpg30
TD                 32768
SOLVENT            CDC13
NS                   302
DS                     0
SWH            24038.461 Hz
FIDRES          0.733596 Hz
AQ              0.6815744 sec
RG                 198.09
DW                20.800 usec
DE                 6.50 usec
TE                298.5 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1         100.6228298 MHz
NUC1                  13C
P1                10.00 usec
PLW1         49.50000000 W

======== CHANNEL f2 ========
SFO2         400.1316005 MHz
NUC2                   1H
CPDPRG[2         waltz16
PCPD2             90.00 usec
PLW2         12.50000000 W
PLW12         0.34722000 W
PLW13         0.28125000 W

F2 - Processing parameters
SI                 32768
SF           100.6127685 MHz
WDW                   EM
SSB                    0
LB                  2.00 Hz
GB                     0
PC                  1.00
```

(*Z*)-3-(benzylamino)-2-chloro-1-phenylprop-2-en-1-one (3ar)



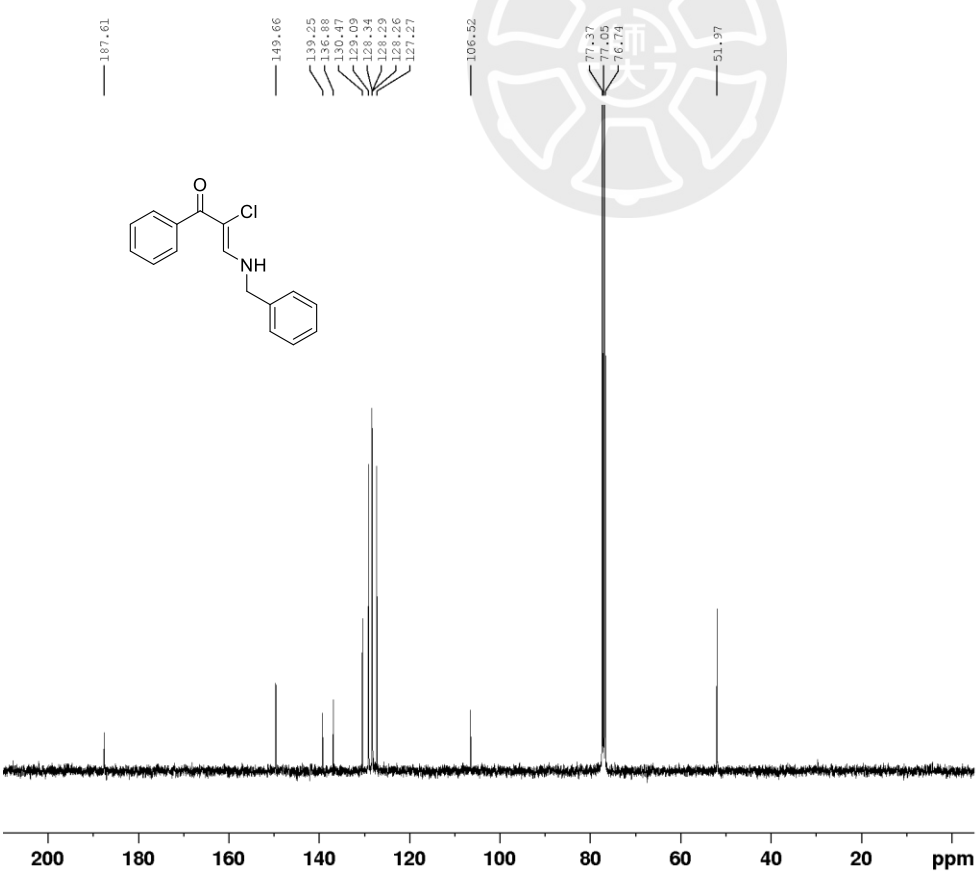
BRUKER

```
Current Data Parameters
NAME          Suresh-733
EXPNO                  3
PROCNO                 1

F2 - Acquisition Parameters
Date_           20231206
Time               14.36
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG             zg30
TD                 32768
SOLVENT            CDC13
NS                    16
DS                     0
SWH             7211.539 Hz
FIDRES          0.220079 Hz
AQ              2.2719147 sec
RG                 99.72
DW                69.333 usec
DE                 10.00 usec
TE                298.5 K
D1              2.00000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1         400.1324008 MHz
NUC1                   1H
P1                  15.00 usec
PLW1         13.50000000 W

F2 - Processing parameters
SI                 16384
SF          400.1300000 MHz
WDW                   EM
SSB                    0
LB                     0 Hz
GB                     0
PC                  1.00
```



BRUKER

```
Current Data Parameters
NAME          Suresh-733
EXPNO                  4
PROCNO                 1

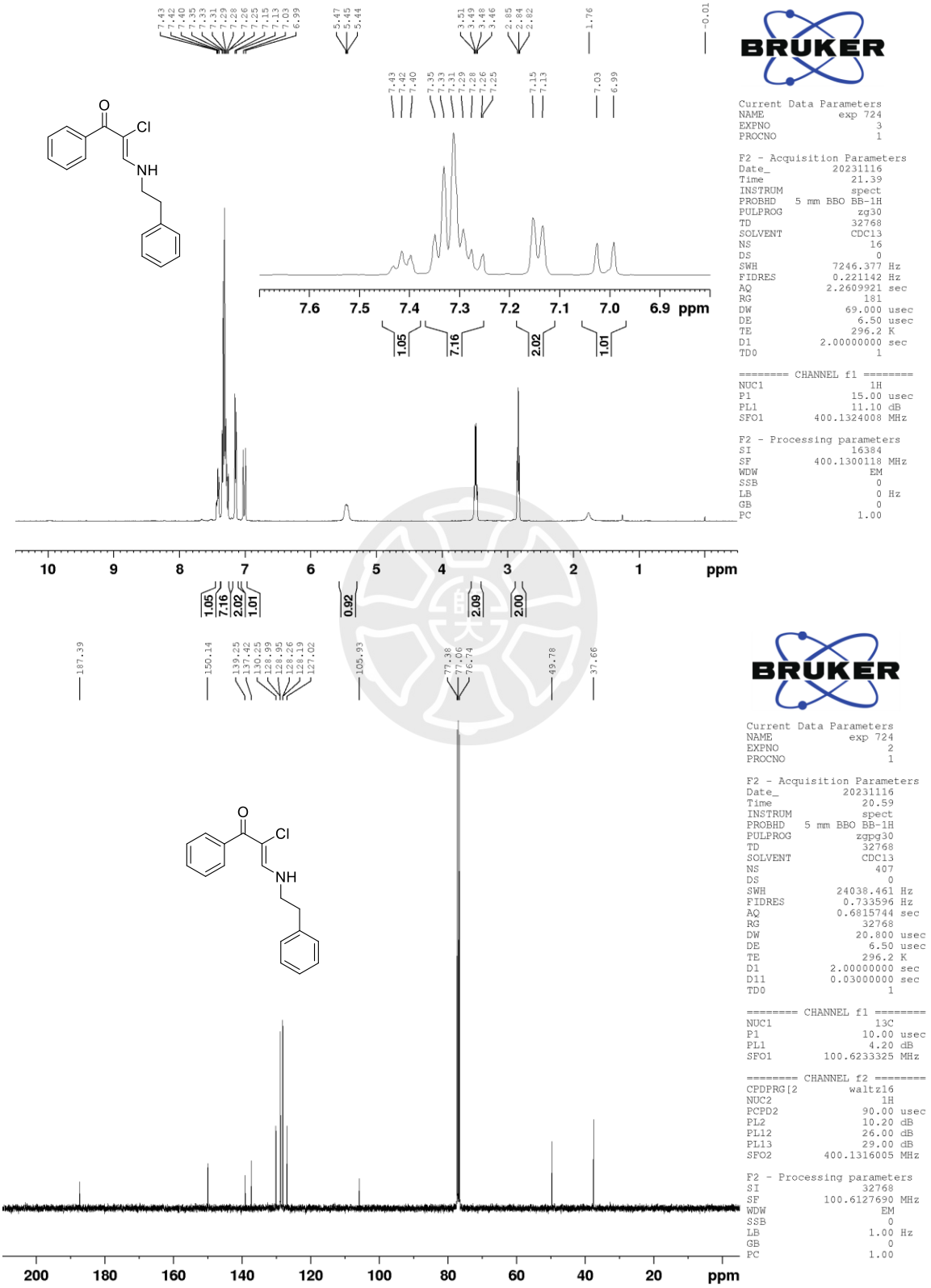
F2 - Acquisition Parameters
Date_           20231206
Time               14.38
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG          zgpg30
TD                 32768
SOLVENT            CDC13
NS                   230
DS                     0
SWH            24038.461 Hz
FIDRES          0.733596 Hz
AQ              0.6815744 sec
RG                198.09
DW                20.800 usec
DE                 6.50 usec
TE                298.6 K
D1              2.00000000 sec
D11             0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1         100.6228298 MHz
NUC1                   13C
P1                  10.00 usec
PLW1         49.50000000 W

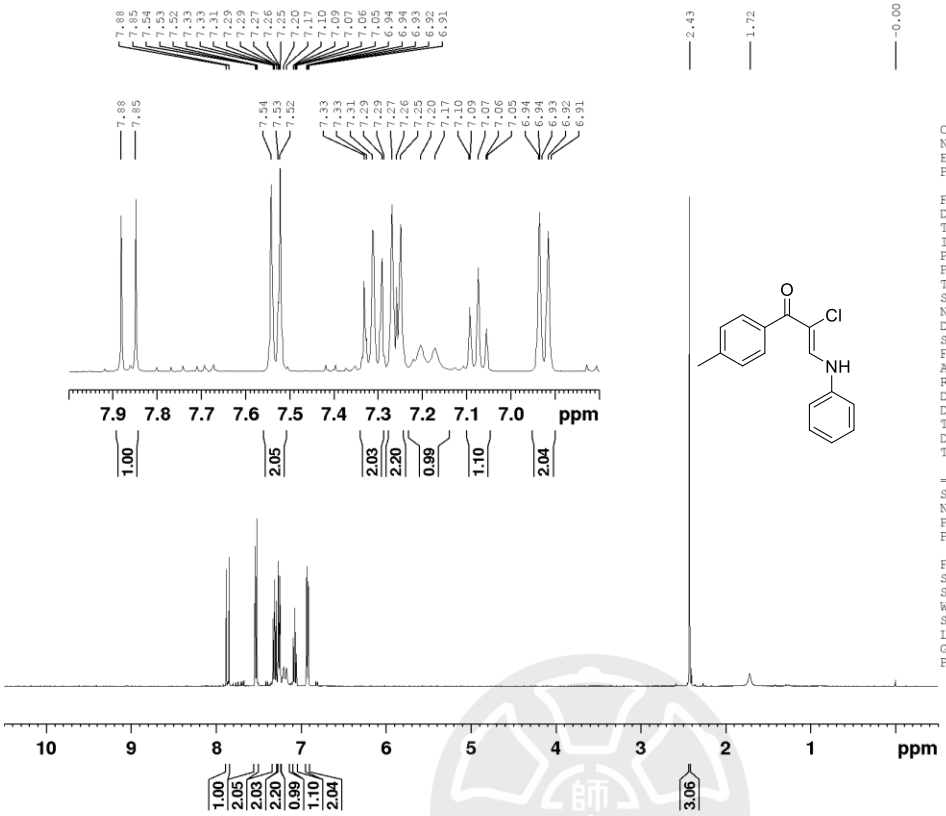
======== CHANNEL f2 ========
SFO2         400.1316005 MHz
NUC2                   1H
CPDPRG[2           waltz16
PCPD2               90.00 usec
PLW2         12.50000000 W
PLW12         0.34722000 W
PLW13         0.28125000 W

F2 - Processing parameters
SI                 32768
SF          100.6127685 MHz
WDW                   EM
SSB                    0
LB                  2.00 Hz
GB                     0
PC                  1.00
```

(*Z*)-2-chloro-3-(phenethylamino)-1-phenylprop-2-en-1-one (3as)



(*Z*)-2-chloro-3-(phenylamino)-1-(*p*-tolyl)prop-2-en-1-one (3ba)

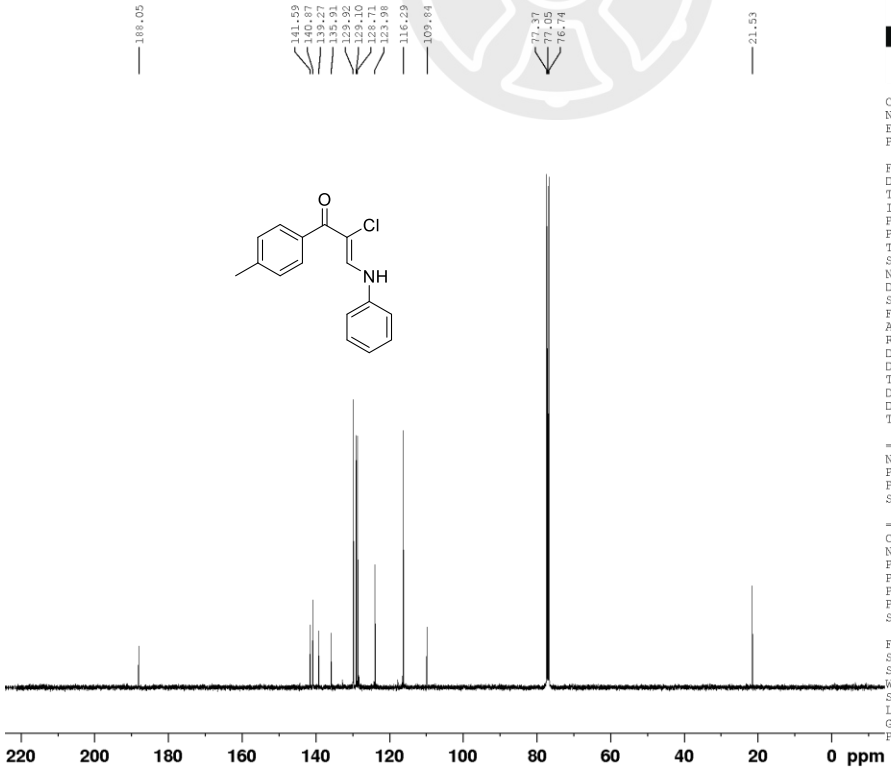



```
Current Data Parameters
NAME          Suresh-747
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20231223
Time               18.17
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG            zg30
TD                32768
SOLVENT           CDC13
NS                   16
DS                    0
SWH             7211.539 Hz
FIDRES          0.220079 Hz
AQ             2.2719147 sec
RG                 99.72
DW                69.333 usec
DE                 10.06 usec
TE                298.2 K
D1            2.00000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1         400.1324008 MHz
NUC1                   1H
P1                 15.00 usec
PLW1         13.50000000 W

F2 - Processing parameters
SI                 16384
SF           400.1300103 MHz
WDW                   EM
SSB                    0
LB                     0 Hz
GB                     0
PC                  1.00
```




```
Current Data Parameters
NAME             exp 747
EXPNO                  2
PROCNO                 1

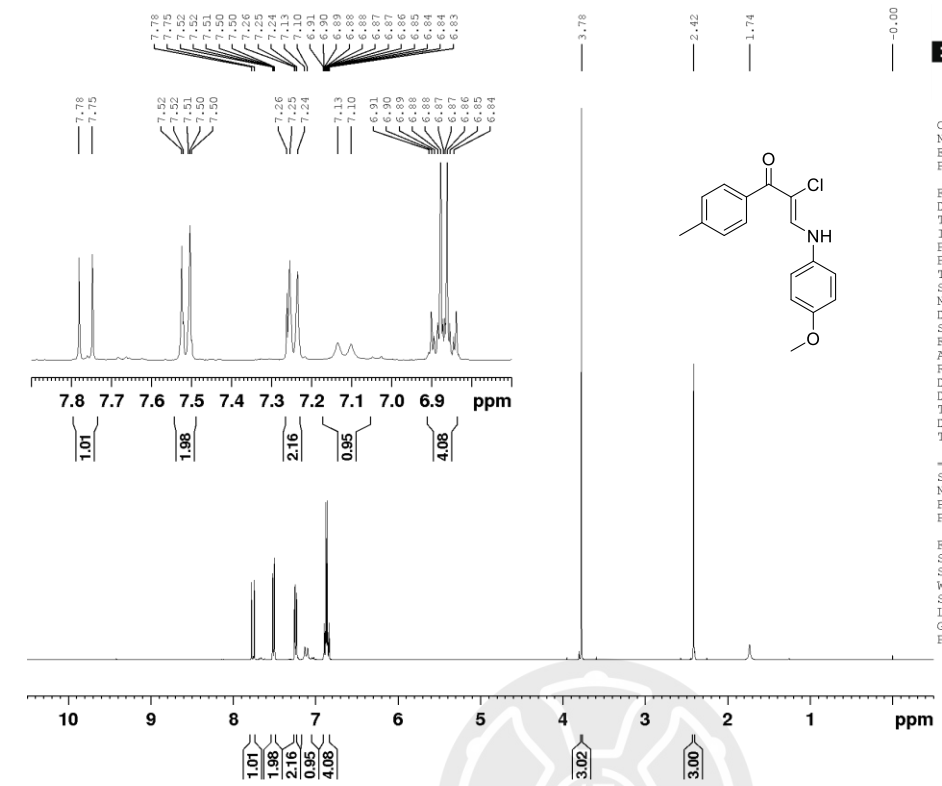
F2 - Acquisition Parameters
Date_           20231223
Time               21.08
INSTRUM            spect
PROBHD   5 mm BBO BB-1H
PULPROG          zgpg30
TD                32768
SOLVENT           CDC13
NS                 1024
DS                    0
SWH            24038.461 Hz
FIDRES          0.733596 Hz
AQ             0.6815744 sec
RG                32768
DW                20.800 usec
DE                 6.50 usec
TE                298.5 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  13C
P1                10.00 usec
PL1                4.20 dB
SFO1        100.6233325 MHz

======== CHANNEL f2 ========
CPDPRG[2         waltz16
NUC2                  1H
PCPD2             90.00 usec
PL2               10.20 dB
PL12              26.00 dB
PL13              29.00 dB
SFO2        400.1316005 MHz

F2 - Processing parameters
SI                32768
SF          100.6127690 MHz
WDW                   EM
SSB                    0
LB                  1.00 Hz
GB                     0
PC                  1.00
```

(*Z*)-2-chloro-3-((4-methoxyphenyl)amino)-1-(*p*-tolyl)prop-2-en-1-one (3bj)

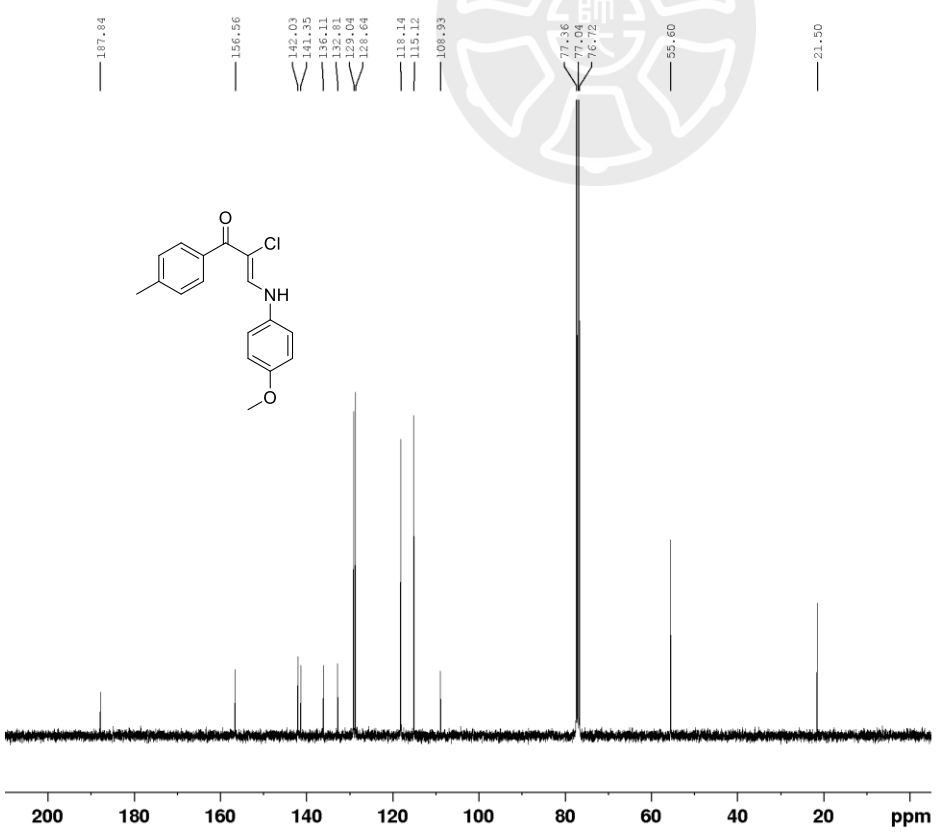



```
Current Data Parameters
NAME                  Suresh-748
EXPNO                          2
PROCNO                         1

F2 - Acquisition Parameters
Date_                   20231223
Time                       17.25
INSTRUM                    spect
PROBHD    5 mm PABBO BB/
PULPROG                    zg30
TD                        32768
SOLVENT                   CDCl3
NS                           16
DS                            0
SWH                    7211.539 Hz
FIDRES                 0.220079 Hz
AQ                     2.2719147 sec
RG                         99.72
DW                        69.333 usec
DE                        10.06 usec
TE                        298.1 K
D1                    2.00000000 sec
TD0                            1

======== CHANNEL f1 ========
SFO1                400.1324008 MHz
NUC1                        1H
P1                        15.00 usec
PLW1                13.50000000 W

F2 - Processing parameters
SI                        16384
SF                  400.1300089 MHz
WDW                          EM
SSB                           0
LB                         0 Hz
GB                            0
PC                         1.00
```




```
Current Data Parameters
NAME                     exp 748
EXPNO                          2
PROCNO                         1

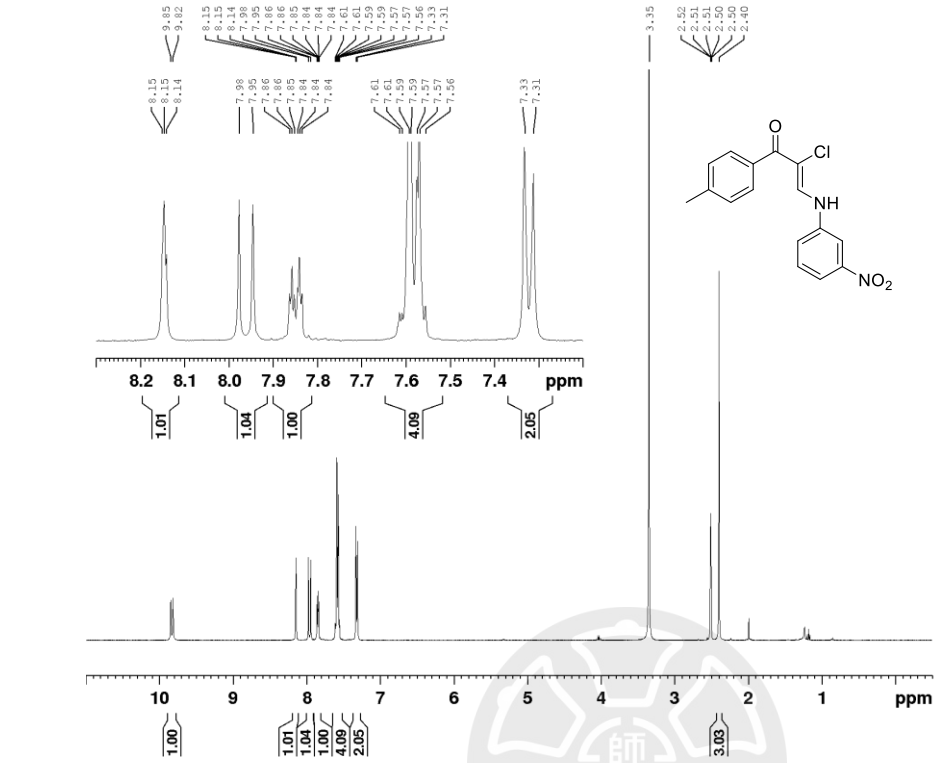
F2 - Acquisition Parameters
Date_                   20231223
Time                       16.46
INSTRUM                    spect
PROBHD    5 mm BBO BB-1H
PULPROG                  zgpg30
TD                        32768
SOLVENT                   CDCl3
NS                           512
DS                            0
SWH                   24038.461 Hz
FIDRES                 0.733596 Hz
AQ                     0.6815744 sec
RG                        32768
DW                        20.800 usec
DE                         6.50 usec
TE                        297.6 K
D1                    2.00000000 sec
D11                   0.03000000 sec
TD0                            1

======== CHANNEL f1 ========
NUC1                         13C
P1                        10.00 usec
PL1                        4.20 dB
SFO1                100.6233325 MHz

======== CHANNEL f2 ========
CPDPRG[2                waltz16
NUC2                         1H
PCPD2                     90.00 usec
PL2                       10.20 dB
PL12                      26.00 dB
PL13                      29.00 dB
SFO2                400.1316005 MHz

F2 - Processing parameters
SI                        32768
SF                  100.6127690 MHz
WDW                          EM
SSB                           0
LB                         1.00 Hz
GB                            0
PC                         1.00
```

(*Z*)-2-chloro-3-((3-nitrophenyl)amino)-1-(*p*-tolyl)prop-2-en-1-one (3bl)

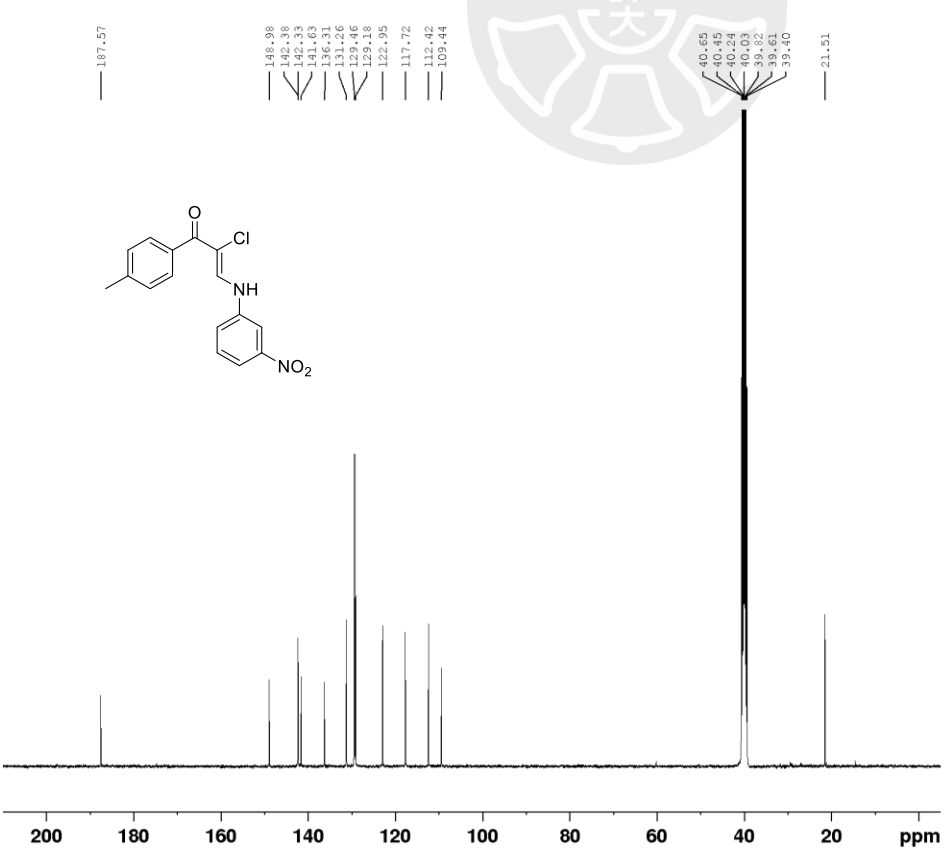



```
Current Data Parameters
NAME          Suresh-749
EXPNO                  4
PROCNO                 1

F2 - Acquisition Parameters
Date_           20240117
Time               20.35
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG             zg30
TD                 32768
SOLVENT             DMSO
NS                    16
DS                     0
SWH            7211.539 Hz
FIDRES        0.220079 Hz
AQ            2.2719147 sec
RG                113.31
DW               69.333 usec
DE                10.06 usec
TE                298.3 K
D1            2.00000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1         400.1324008 MHz
NUC1                   1H
P1                  15.00 usec
PLW1         13.50000000 W

F2 - Processing parameters
SI                 16384
SF           400.1300000 MHz
WDW                   EM
SSB                    0
LB                     0 Hz
GB                     0
PC                  1.00
```




```
Current Data Parameters
NAME          Suresh-749
EXPNO                  6
PROCNO                 1

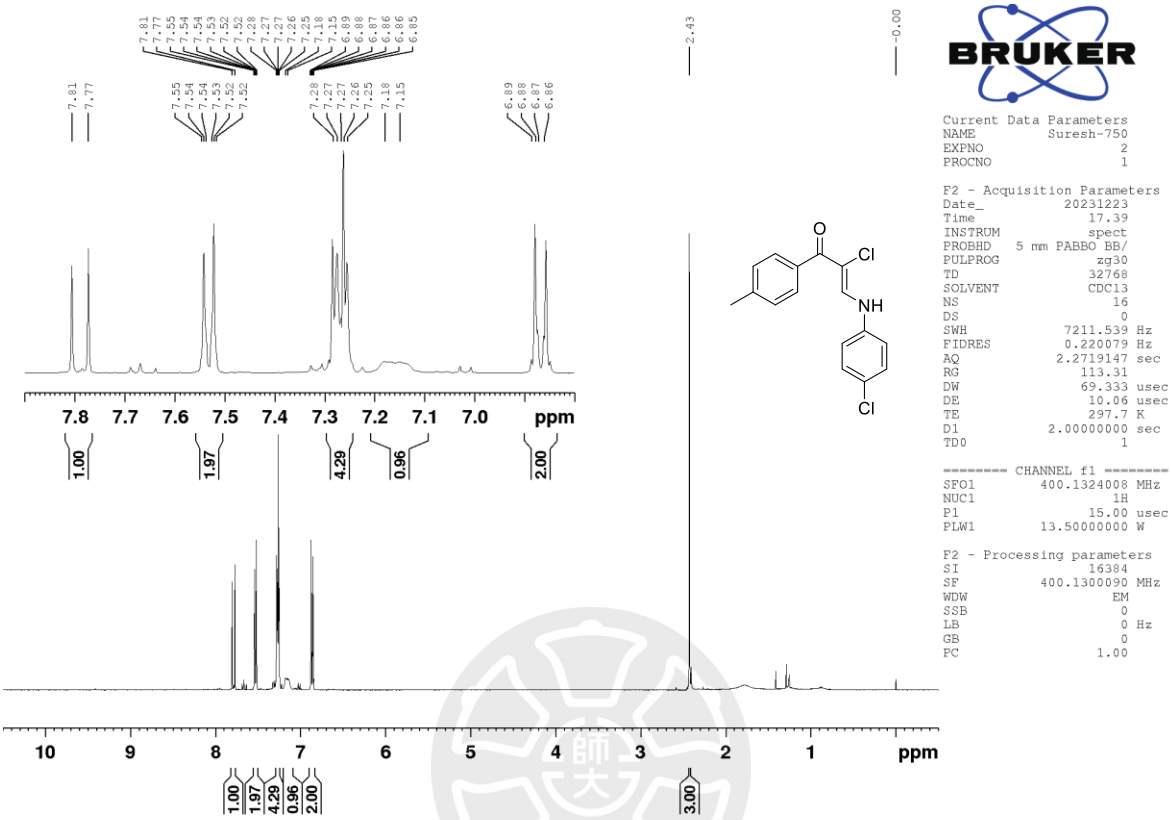
F2 - Acquisition Parameters
Date_           20240117
Time               22.01
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG          zgpg30
TD                 32768
SOLVENT             DMSO
NS                 12096
DS                     0
SWH           24038.461 Hz
FIDRES        0.733596 Hz
AQ            0.6815744 sec
RG                198.09
DW               20.800 usec
DE                 6.50 usec
TE                298.3 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1         100.6228298 MHz
NUC1                  13C
P1                 10.00 usec
PLW1         49.50000000 W

======== CHANNEL f2 ========
SFO2         400.1316005 MHz
NUC2                   1H
CPDPRG[2          waltz16
PCPD2              90.00 usec
PLW2         12.50000000 W
PLW12         0.34722000 W
PLW13         0.28125000 W

F2 - Processing parameters
SI                 32768
SF           100.6127685 MHz
WDW                   EM
SSB                    0
LB                  2.00 Hz
GB                     0
PC                  1.00
```

(*Z*)-2-chloro-3-((4-chlorophenyl)amino)-1-(*p*-tolyl)prop-2-en-1-one (3bp)



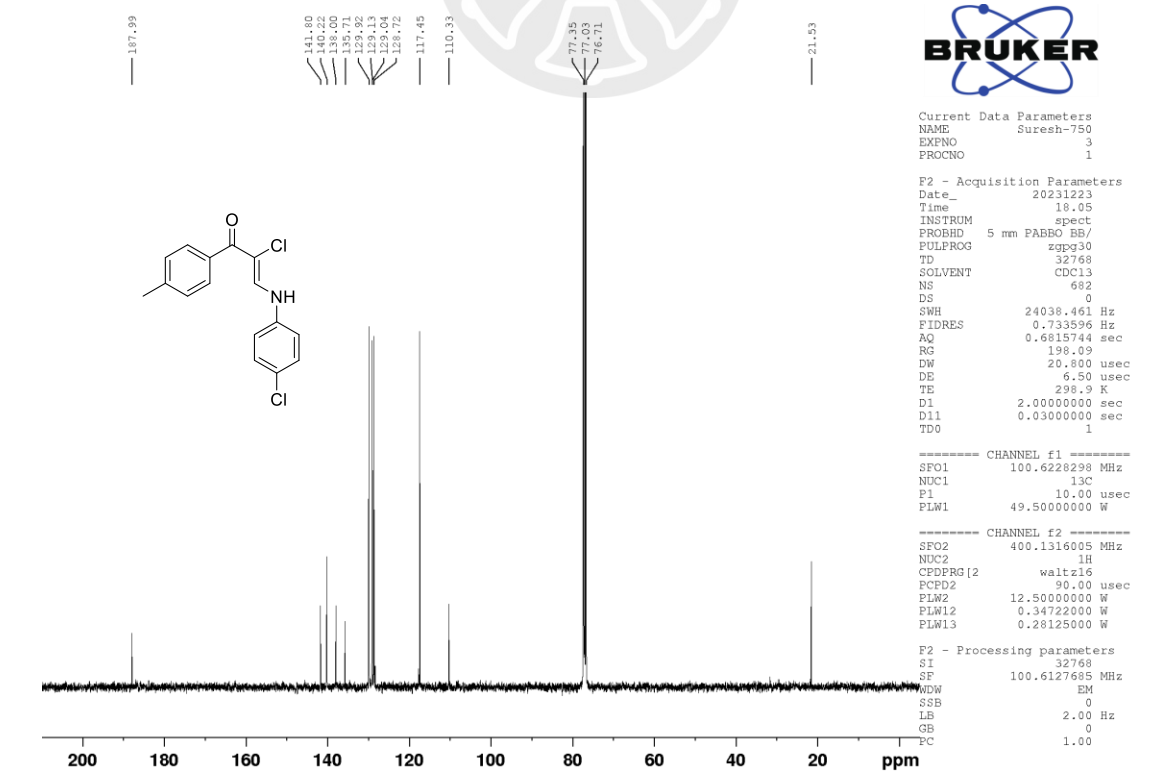
BRUKER

```
Current Data Parameters
NAME          Suresh-750
EXPNO                  2
PROCNO                 1

F2 - Acquisition Parameters
Date_           20231223
Time               17.39
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG            zg30
TD                32768
SOLVENT           CDC13
NS                   16
DS                    0
SWH            7211.539 Hz
FIDRES         0.220079 Hz
AQ             2.2719147 sec
RG                113.31
DW                69.333 usec
DE                10.06 usec
TE                297.7 K
D1             2.00000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1         400.1324008 MHz
NUC1                   1H
P1                 15.00 usec
PLW1         13.50000000 W

F2 - Processing parameters
SI                 16384
SF           400.1300090 MHz
WDW                   EM
SSB                    0
LB                  0 Hz
GB                     0
PC                  1.00
```



BRUKER

```
Current Data Parameters
NAME          Suresh-750
EXPNO                  3
PROCNO                 1

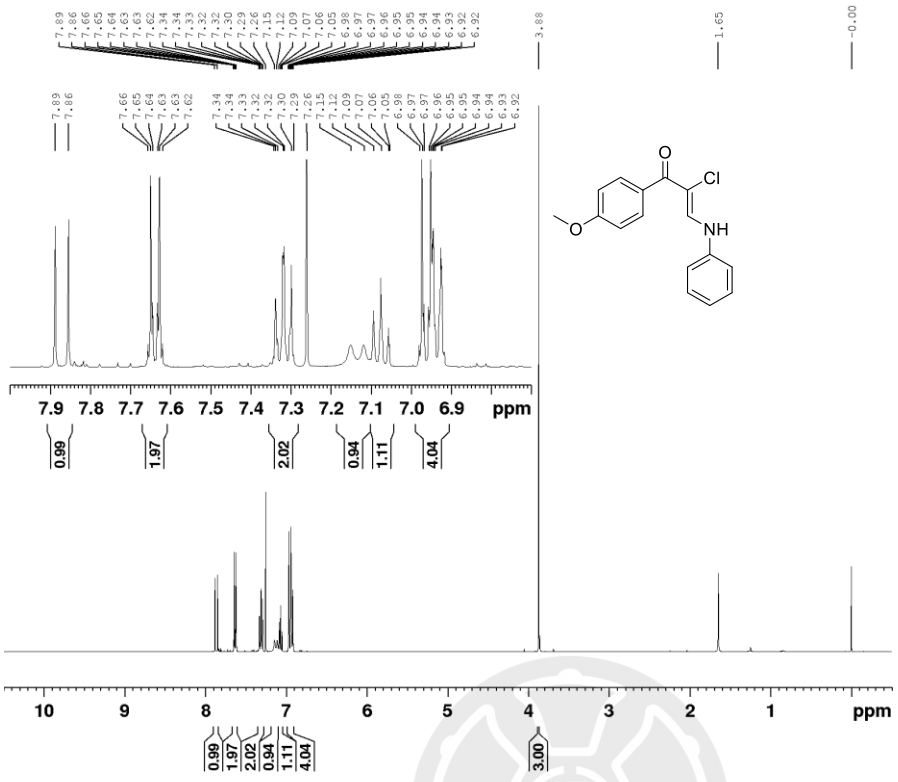
F2 - Acquisition Parameters
Date_           20231223
Time               18.05
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG          zgpg30
TD                32768
SOLVENT           CDC13
NS                  682
DS                    0
SWH           24038.461 Hz
FIDRES         0.733596 Hz
AQ             0.6815744 sec
RG                198.09
DW                20.800 usec
DE                 6.50 usec
TE                298.9 K
D1             2.00000000 sec
D11            0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1         100.6228298 MHz
NUC1                  13C
P1                 10.00 usec
PLW1         49.50000000 W

======== CHANNEL f2 ========
SFO2         400.1316005 MHz
NUC2                   1H
CPDPRG[2          waltz16
PCPD2              90.00 usec
PLW2         12.50000000 W
PLW12         0.34722000 W
PLW13         0.28125000 W

F2 - Processing parameters
SI                 32768
SF           100.6127685 MHz
WDW                   EM
SSB                    0
LB                  2.00 Hz
GB                     0
PC                  1.00
```

(*Z*)-2-chloro-1-(4-methoxyphenyl)-3-(phenylamino)prop-2-en-1-one (3ca)

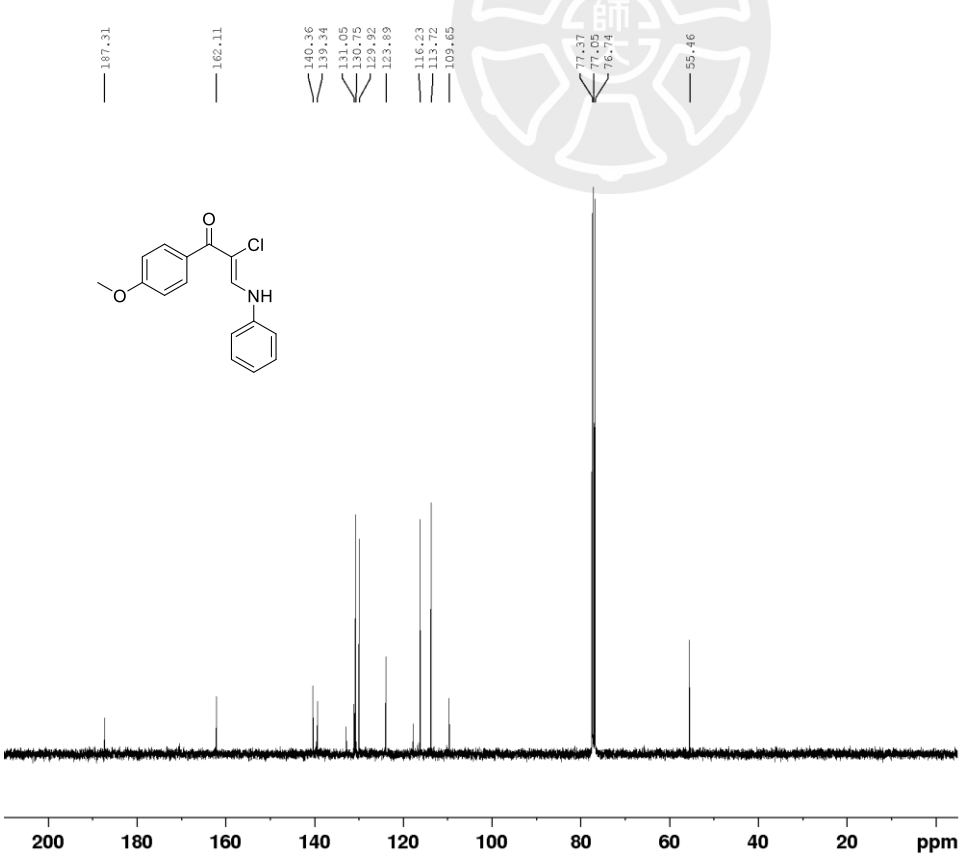



```
Current Data Parameters
NAME                  Enaminone
EXPNO                        87
PROCNO                        1

F2 - Acquisition Parameters
Date_               20240710
Time                    14.15
INSTRUM                 spect
PROBHD    5 mm PABBO BB/
PULPROG                 zg30
TD                     32768
SOLVENT                CDC13
NS                        16
DS                         0
SWH                 7211.539 Hz
FIDRES                0.220079 Hz
AQ                  2.2719147 sec
RG                      128.9
DW                     69.333 usec
DE                      10.06 usec
TE                      299.6 K
D1                  2.00000000 sec
TD0                           1

======== CHANNEL f1 ========
SFO1                400.1324008 MHz
NUC1                        1H
P1                       15.00 usec
PLW1                13.50000000 W

F2 - Processing parameters
SI                        16384
SF                  400.1300098 MHz
WDW                          EM
SSB                           0
LB                            0 Hz
GB                            0
PC                         1.00
```




```
Current Data Parameters
NAME                   exp 738
EXPNO                        2
PROCNO                       1

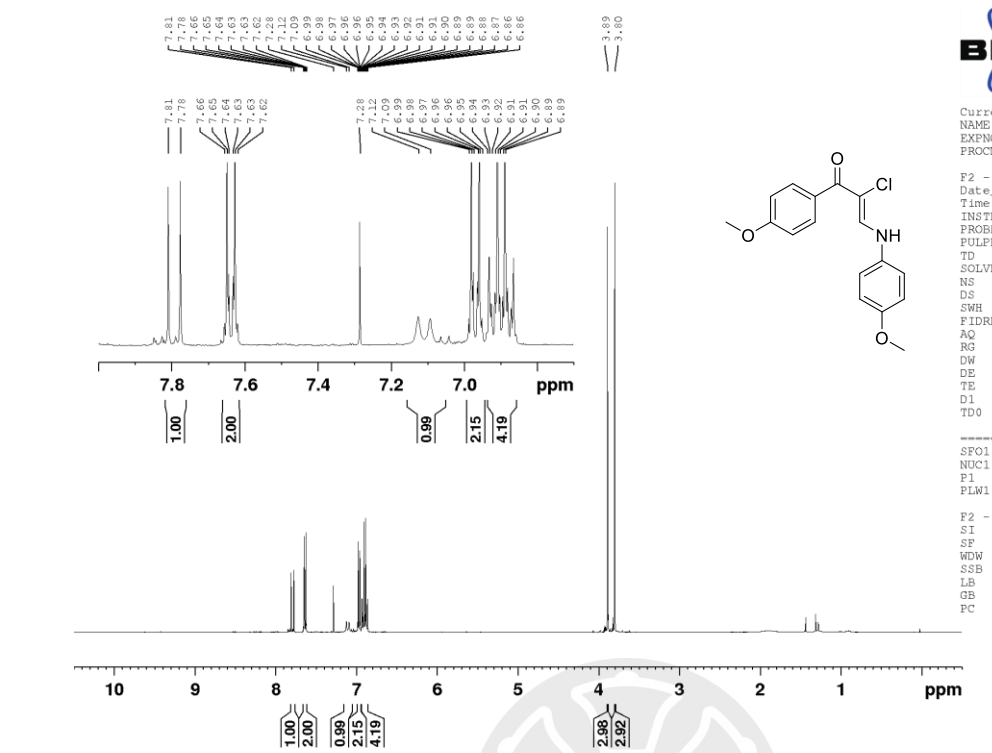
F2 - Acquisition Parameters
Date_               20231213
Time                    21.54
INSTRUM                 spect
PROBHD    5 mm BBO BB-1H
PULPROG                zgpg30
TD                     32768
SOLVENT                CDC13
NS                       232
DS                         0
SWH                24038.461 Hz
FIDRES                0.733596 Hz
AQ                  0.6815744 sec
RG                      32768
DW                     20.800 usec
DE                      6.50 usec
TE                      296.5 K
D1                  2.00000000 sec
D11                 0.03000000 sec
TD0                           1

======== CHANNEL f1 ========
NUC1                        13C
P1                       10.00 usec
PL1                       4.20 dB
SFO1               100.6233325 MHz

======== CHANNEL f2 ========
CPDPRG[2                waltz16
NUC2                        1H
PCPD2                    90.00 usec
PL2                      10.20 dB
PL12                     26.00 dB
PL13                     29.00 dB
SFO2               400.1316005 MHz

F2 - Processing parameters
SI                        32768
SF                 100.6127690 MHz
WDW                          EM
SSB                           0
LB                         1.00 Hz
GB                            0
PC                         1.00
```

(*Z*)-2-chloro-1-(4-methoxyphenyl)-3-((4-methoxyphenyl)amino)prop-2-en-1-one (3cj)

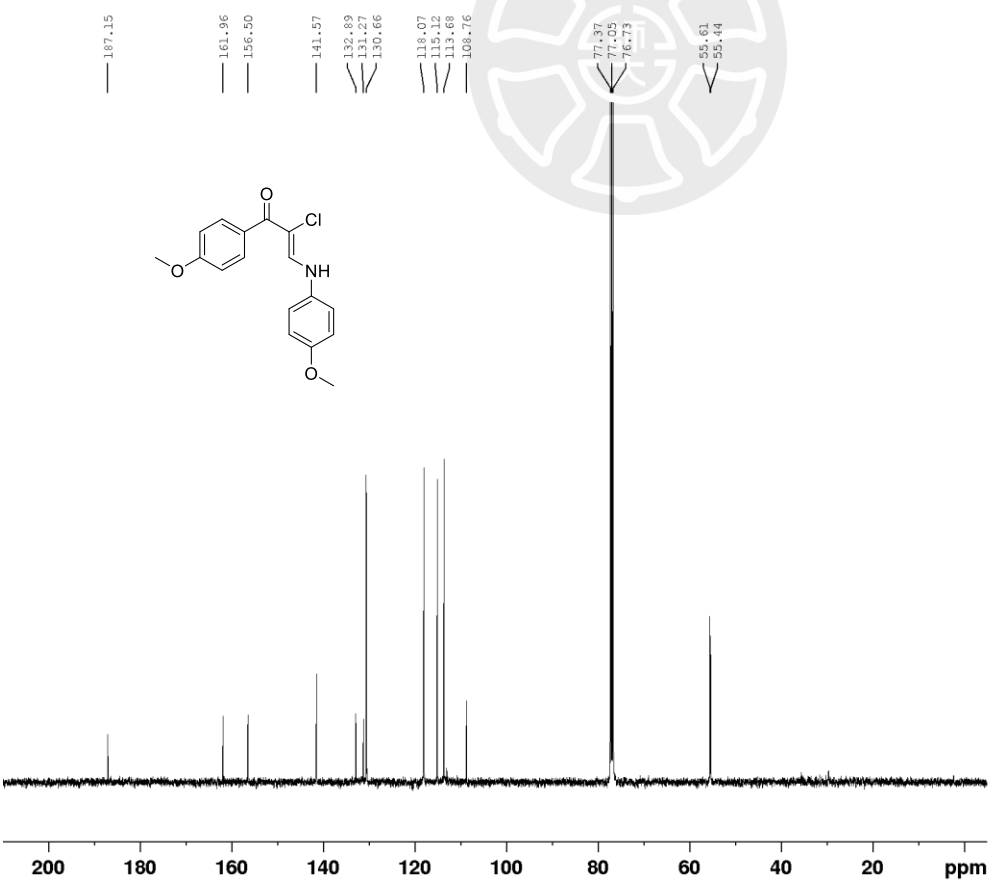


Current Data Parameters
NAME Suresh-736
EXPNO 4
PROCNO 1

F2 - Acquisition Parameters
Date_ 20231211
Time 17.14
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 32768
SOLVENT CDC13
NS 16
DS 0
SWH 7211.539 Hz
FIDRES 0.220079 Hz
AQ 2.2719147 sec
RG 99.72
DW 69.333 usec
DE 10.06 usec
TE 298.5 K
D1 2.00000000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 400.1324008 MHz
NUC1 1H
P1 15.00 usec
PLW1 13.50000000 W

F2 - Processing parameters
SI 16384
SF 400.1300000 MHz
WDW EM
SSB 0
LB 0 Hz
GB 0
PC 1.00



Current Data Parameters
NAME Suresh-736
EXPNO 3
PROCNO 1

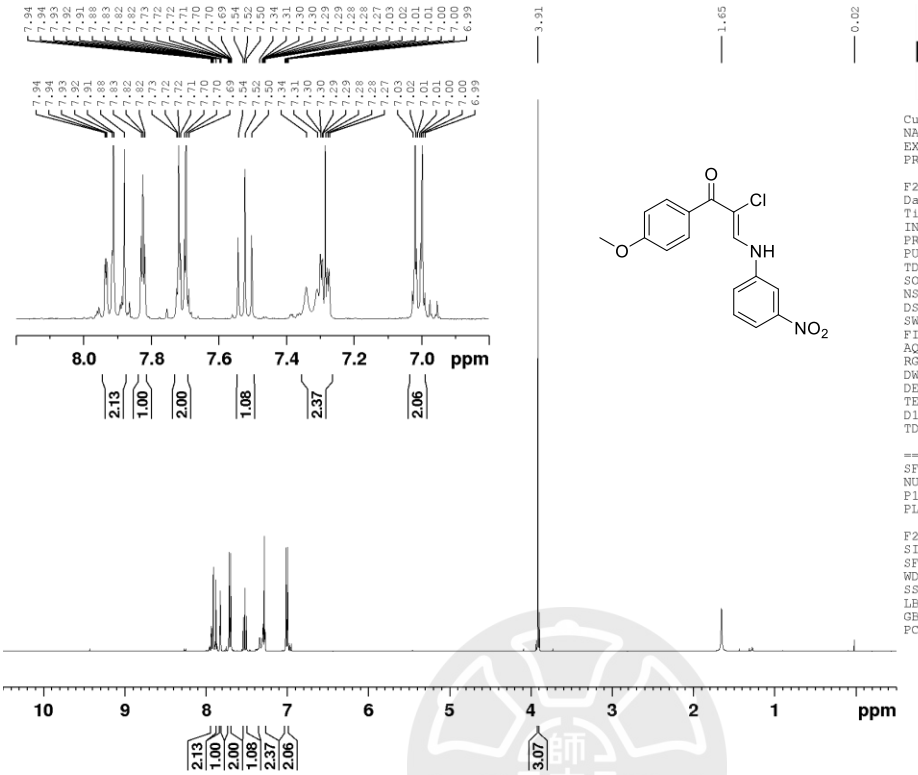
F2 - Acquisition Parameters
Date_ 20231210
Time 15.01
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 32768
SOLVENT CDC13
NS 512
DS 0
SWH 24038.461 Hz
FIDRES 0.733596 Hz
AQ 0.6815744 sec
RG 198.09
DW 20.800 usec
DE 6.50 usec
TE 298.6 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 100.6228298 MHz
NUC1 13C
P1 10.00 usec
PLW1 49.50000000 W

======== CHANNEL f2 ========
SFO2 400.1316005 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 90.00 usec
PLW2 12.50000000 W
PLW12 0.34722000 W
PLW13 0.28125000 W

F2 - Processing parameters
SI 32768
SF 100.6127685 MHz
WDW EM
SSB 0
LB 2.00 Hz
GB 0
PC 1.00

(*Z*)-2-chloro-1-(4-methoxyphenyl)-3-((3-nitrophenyl)amino)prop-2-en-1-one (3cl)

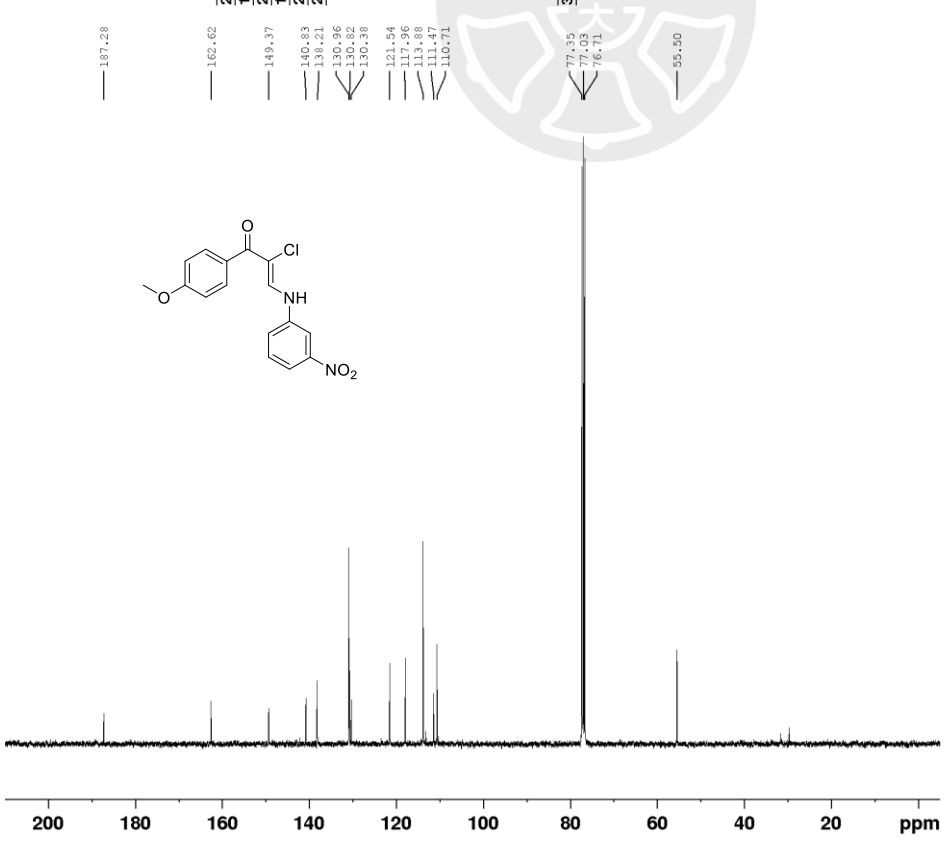



```
Current Data Parameters
NAME          Suresh-737
EXPNO                  4
PROCNO                 1

F2 - Acquisition Parameters
Date_           20231213
Time              21.44
INSTRUM           spect
PROBHD   5 mm PABBO BB/
PULPROG            zg30
TD                32768
SOLVENT           CDC13
NS                   16
DS                    0
SWH           7211.539 Hz
FIDRES        0.220079 Hz
AQ           2.2719147 sec
RG              158.74
DW              69.333 usec
DE               10.06 usec
TE               298.4 K
D1           2.00000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1        400.1324008 MHz
NUC1                   1H
P1                 15.00 usec
PLW1        13.50000000 W

F2 - Processing parameters
SI                 16384
SF          400.1300000 MHz
WDW                   EM
SSB                    0
LB                  0 Hz
GB                     0
PC                  1.00
```




```
Current Data Parameters
NAME          Suresh-737
EXPNO                  2
PROCNO                 1

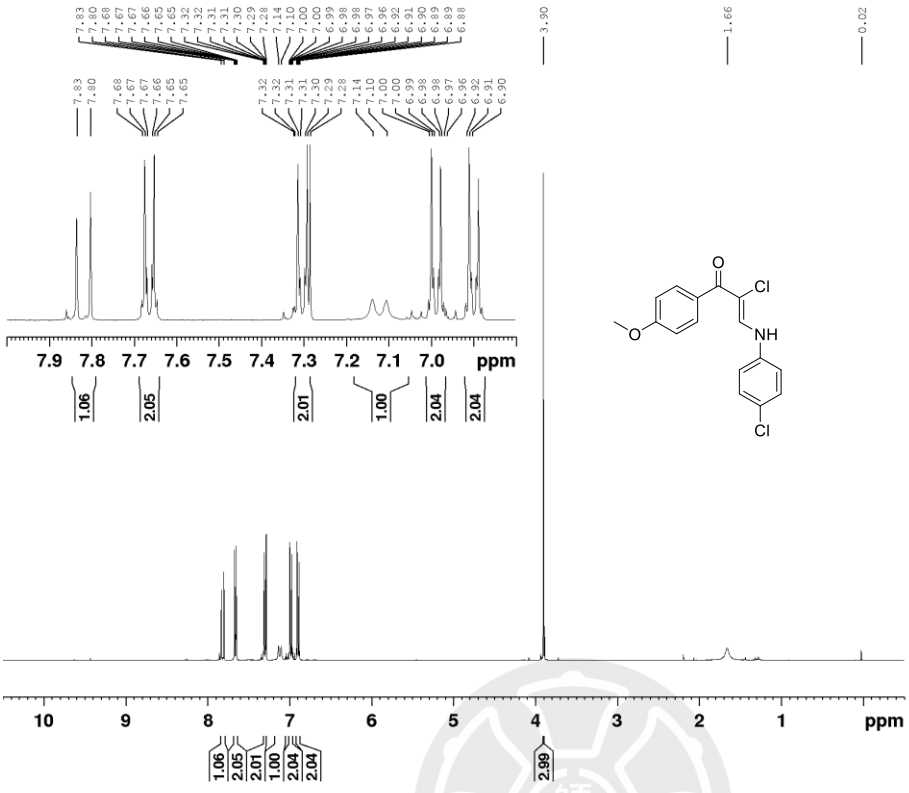
F2 - Acquisition Parameters
Date_           20231210
Time              16.00
INSTRUM           spect
PROBHD   5 mm PABBO BB/
PULPROG          zgpg30
TD                32768
SOLVENT           CDC13
NS                  512
DS                    0
SWH          24038.461 Hz
FIDRES        0.733596 Hz
AQ           0.6815744 sec
RG              198.09
DW              20.800 usec
DE               6.50 usec
TE               298.9 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1        100.6228298 MHz
NUC1                   13C
P1                 10.00 usec
PLW1        49.50000000 W

======== CHANNEL f2 ========
SFO2        400.1316005 MHz
NUC2                   1H
CPDPRG[2          waltz16
PCPD2              90.00 usec
PLW2        12.50000000 W
PLW12        0.34722000 W
PLW13        0.28125000 W

F2 - Processing parameters
SI                 32768
SF          100.6127685 MHz
WDW                   EM
SSB                    0
LB                 2.00 Hz
GB                     0
PC                  1.00
```

(*Z*)-2-chloro-3-((4-chlorophenyl)amino)-1-(4-methoxyphenyl)prop-2-en-1-one (3cp)

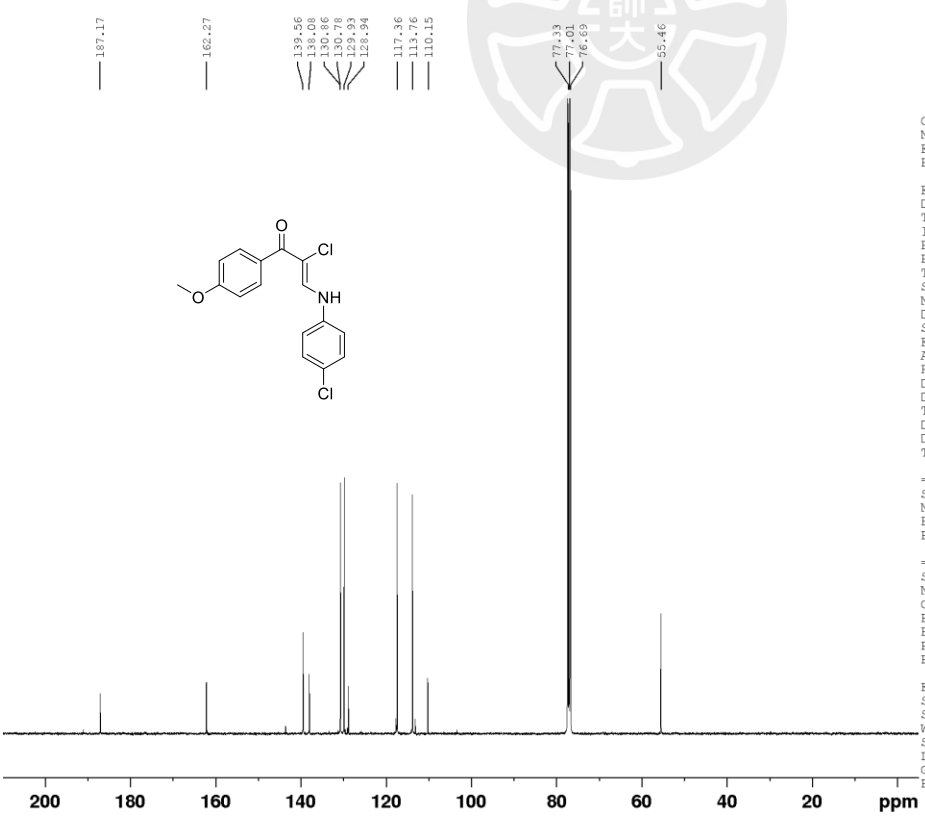



```
Current Data Parameters
NAME          Suresh-739
EXPNO                  6
PROCNO                 1

F2 - Acquisition Parameters
Date_           20231216
Time               21.05
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG             zg30
TD                 32768
SOLVENT            CDCl3
NS                    16
DS                     0
SWH             7211.539 Hz
FIDRES          0.220079 Hz
AQ              2.2719147 sec
RG                144.49
DW                69.333 usec
DE                 10.06 usec
TE                298.2 K
D1             2.00000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1          400.1324008 MHz
NUC1                  1H
P1                 15.00 usec
PLW1          13.50000000 W

F2 - Processing parameters
SI                 16384
SF          400.1300000 MHz
WDW                   EM
SSB                    0
LB                  0 Hz
GB                     0
PC                  1.00
```




```
Current Data Parameters
NAME          Suresh-739
EXPNO                  7
PROCNO                 1

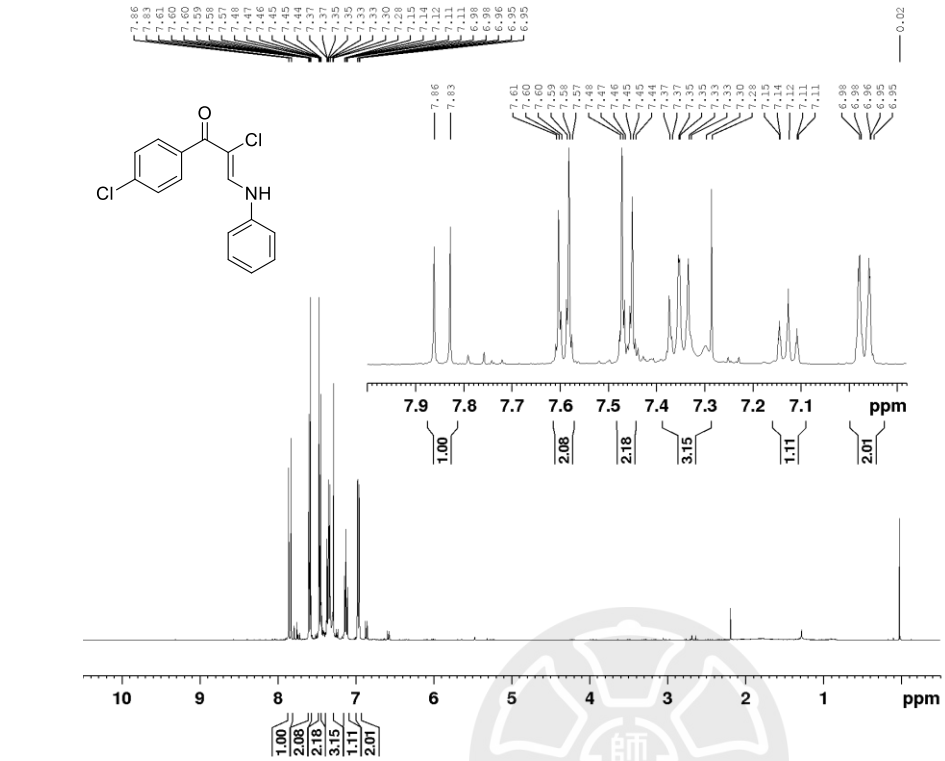
F2 - Acquisition Parameters
Date_           20231216
Time               21.07
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG           zgpg30
TD                 32768
SOLVENT            CDCl3
NS                 15000
DS                     0
SWH            24038.461 Hz
FIDRES          0.733596 Hz
AQ              0.6815744 sec
RG                198.09
DW                20.800 usec
DE                 6.50 usec
TE                298.2 K
D1             2.00000000 sec
D11            0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1          100.6228298 MHz
NUC1                  13C
P1                 10.00 usec
PLW1          49.50000000 W

======== CHANNEL f2 ========
SFO2          400.1316005 MHz
NUC2                  1H
CPDPRG[2          waltz16
PCPD2              90.00 usec
PLW2          12.50000000 W
PLW12          0.34722000 W
PLW13          0.28125000 W

F2 - Processing parameters
SI                 32768
SF          100.6127685 MHz
WDW                   EM
SSB                    0
LB                  2.00 Hz
GB                     0
PC                  1.00
```

(*Z*)-2-chloro-1-(4-chlorophenyl)-3-(phenylamino)prop-2-en-1-one (3da)



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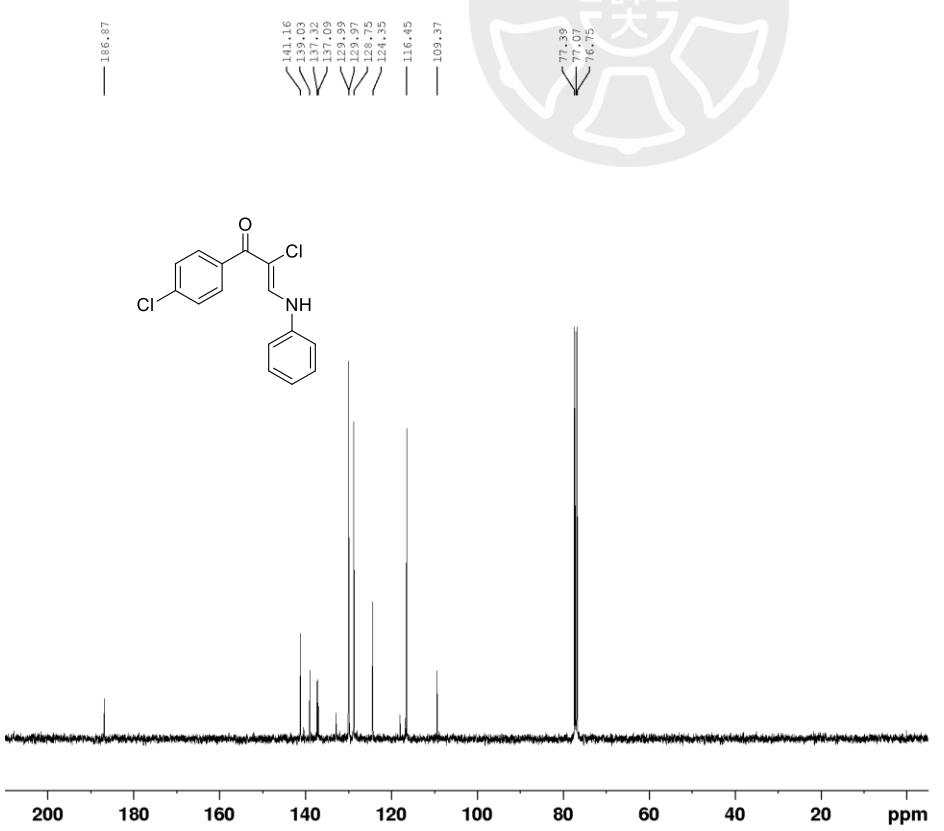
```
Current Data Parameters
NAME                  20230412
EXPNO                        2
PROCNO                       1

F2 - Acquisition Parameters
Date_                 20230413
Time                     15.43
INSTRUM                  spect
PROBHD     5 mm PABBO BB/
PULPROG                  zg30
TD                      32768
SOLVENT                 CDCl3
NS                           8
DS                           0
SWH                  7211.539 Hz
FIDRES               0.220079 Hz
AQ                   2.2719147 sec
RG                       89.08
DW                     69.333 usec
DE                      10.06 usec
TE                      297.7 K
D1                   2.00000000 sec
TD0                          1

======== CHANNEL f1 ========
SFO1           400.1324008 MHz
NUC1                     1H
P1                    15.00 usec
PLW1           13.50000000 W

F2 - Processing parameters
SI                       16384
SF             400.1300000 MHz
WDW                         EM
SSB                          0
LB                        0 Hz
GB                           0
PC                        1.00
```



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```
Current Data Parameters
NAME                  20230412
EXPNO                        3
PROCNO                       1

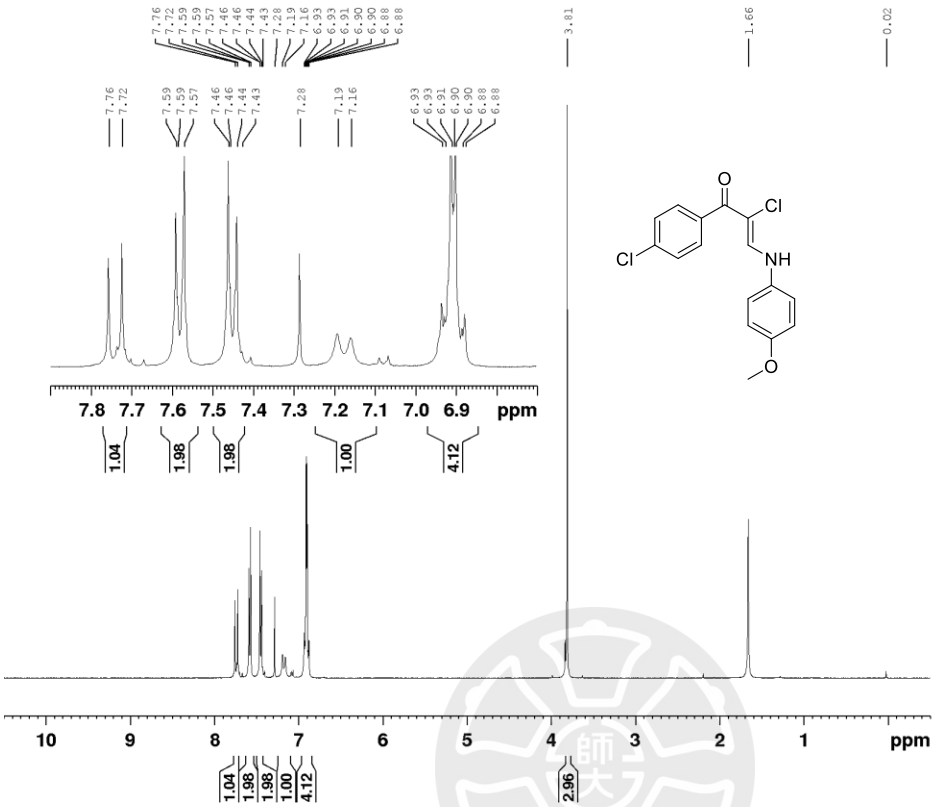
F2 - Acquisition Parameters
Date_                 20230413
Time                     15.44
INSTRUM                  spect
PROBHD     5 mm PABBO BB/
PULPROG                zgpg30
TD                      32768
SOLVENT                 CDCl3
NS                         123
DS                           0
SWH                24038.461 Hz
FIDRES              0.733596 Hz
AQ                  0.6815744 sec
RG                      198.09
DW                      20.800 usec
DE                       6.50 usec
TE                      298.1 K
D1                   2.00000000 sec
D11                  0.03000000 sec
TD0                          1

======== CHANNEL f1 ========
SFO1           100.6228298 MHz
NUC1                     13C
P1                    10.00 usec
PLW1           49.50000000 W

======== CHANNEL f2 ========
SFO2           400.1316005 MHz
NUC2                     1H
CPDPRG[2             waltz16
PCPD2                 90.00 usec
PLW2           12.50000000 W
PLW12           0.34722000 W
PLW13           0.28125000 W

F2 - Processing parameters
SI                       32768
SF             100.6127685 MHz
WDW                         EM
SSB                          0
LB                        2.00 Hz
GB                           0
PC                        1.00
```

(*Z*)-2-chloro-1-(4-chlorophenyl)-3-((4-methoxyphenyl)amino)prop-2-en-1-one (3dj)

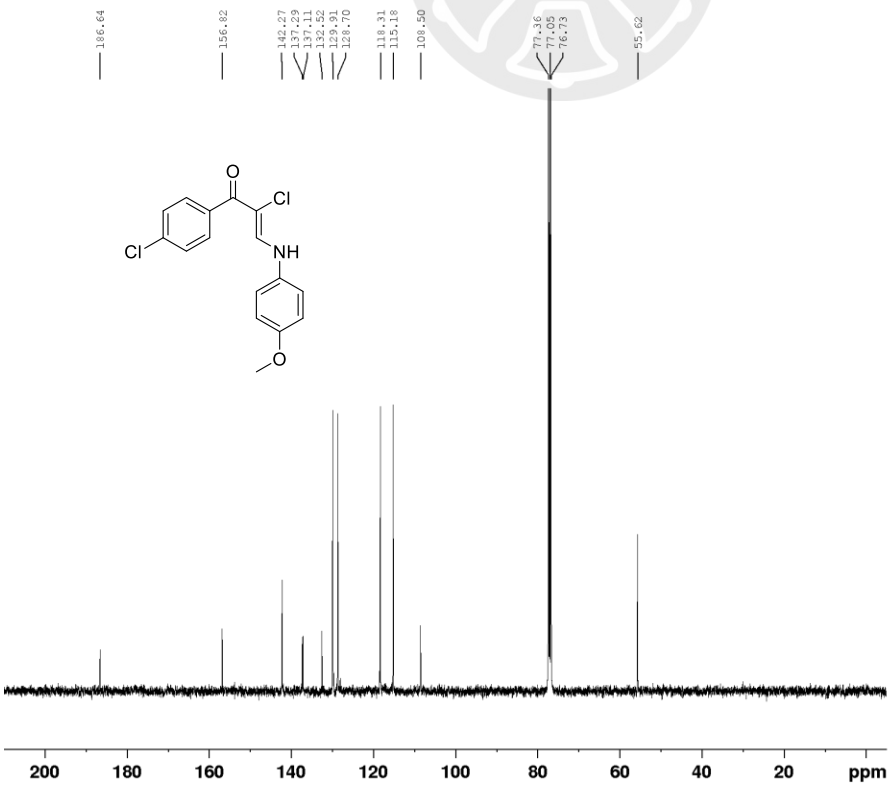


```
Current Data Parameters
NAME          Suresh-754
EXPNO                  4
PROCNO                 1

F2 - Acquisition Parameters
Date_           20231228
Time               17.26
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG             zg30
TD                 32768
SOLVENT            CDCl3
NS                    16
DS                     0
SWH             7211.539 Hz
FIDRES          0.220079 Hz
AQ              2.2719147 sec
RG                144.49
DW                69.333 usec
DE                10.06 usec
TE                298.1 K
D1             2.00000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1         400.1324008 MHz
NUC1                   1H
P1                 15.00 usec
PLW1         13.50000000 W

F2 - Processing parameters
SI                 16384
SF            400.1300000 MHz
WDW                   EM
SSB                    0
LB                  0 Hz
GB                     0
PC                  1.00
```



```
Current Data Parameters
NAME          Suresh-754
EXPNO                  3
PROCNO                 1

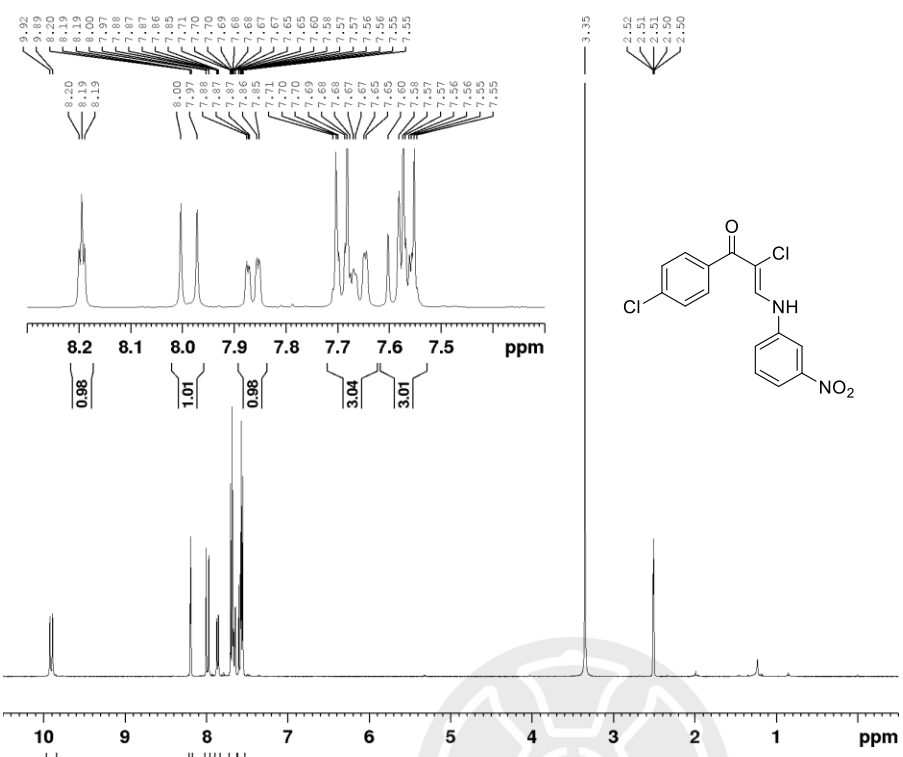
F2 - Acquisition Parameters
Date_           20231228
Time               15.57
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG          zgpg30
TD                 32768
SOLVENT            CDCl3
NS                   429
DS                     0
SWH            24038.461 Hz
FIDRES          0.733596 Hz
AQ              0.6815744 sec
RG                198.09
DW                20.800 usec
DE                 6.50 usec
TE                298.5 K
D1             2.00000000 sec
D11            0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1         100.6228298 MHz
NUC1                   13C
P1                 10.00 usec
PLW1         49.50000000 W

======== CHANNEL f2 ========
SFO2         400.1316005 MHz
NUC2                   1H
CPDPRG[2          waltz16
PCPD2              90.00 usec
PLW2         12.50000000 W
PLW12         0.34722000 W
PLW13         0.28125000 W

F2 - Processing parameters
SI                 32768
SF            100.6127685 MHz
WDW                   EM
SSB                    0
LB                  2.00 Hz
GB                     0
PC                  1.00
```

(*Z*)-2-chloro-1-(4-chlorophenyl)-3-((3-nitrophenyl)amino)prop-2-en-1-one (3dl)

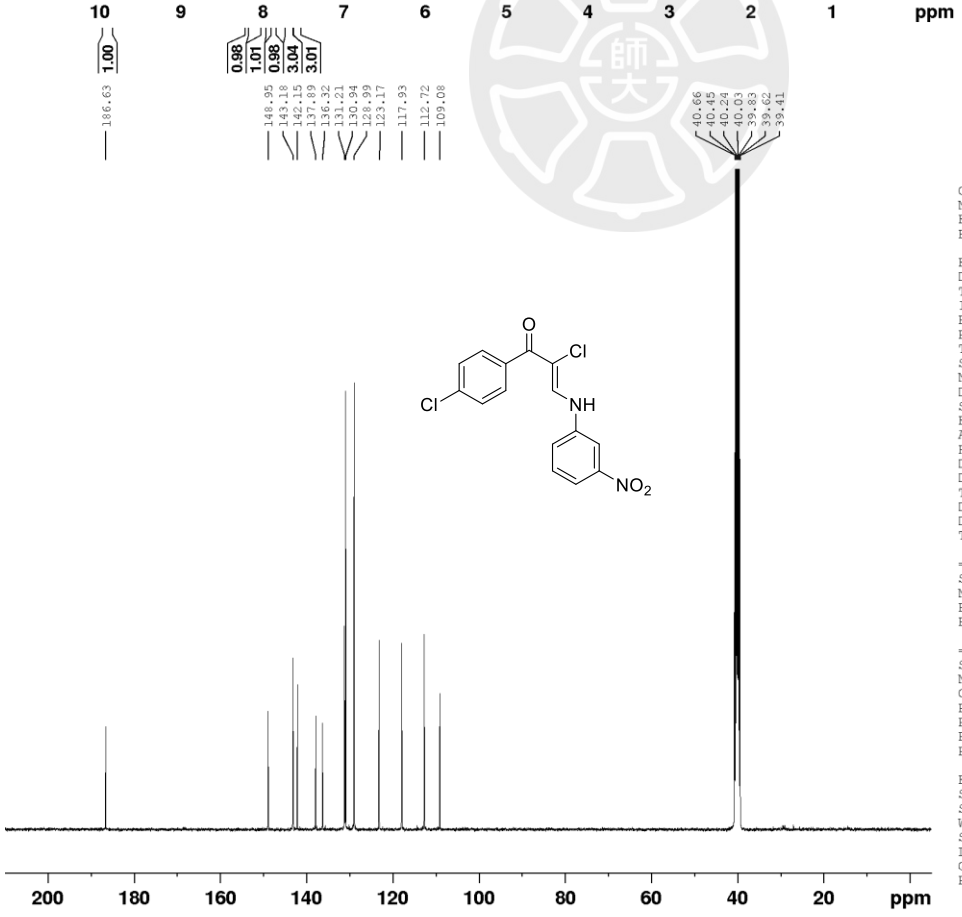


Current Data Parameters
NAME Suresh-755
EXPNO 4
PROCNO 1

F2 - Acquisition Parameters
Date_ 20240116
Time 22.19
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 32768
SOLVENT DMSO
NS 16
DS 0
SWH 7211.539 Hz
FIDRES 0.220079 Hz
AQ 2.2719147 sec
RG 113.31
DW 69.333 usec
DE 10.06 usec
TE 298.1 K
D1 2.00000000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 400.1324008 MHz
NUC1 1H
P1 15.00 usec
PLW1 13.50000000 W

F2 - Processing parameters
SI 16384
SF 400.1300000 MHz
WDW EM
SSB 0
LB 0 Hz
GB 0
PC 1.00



Current Data Parameters
NAME Suresh-755
EXPNO 5
PROCNO 1

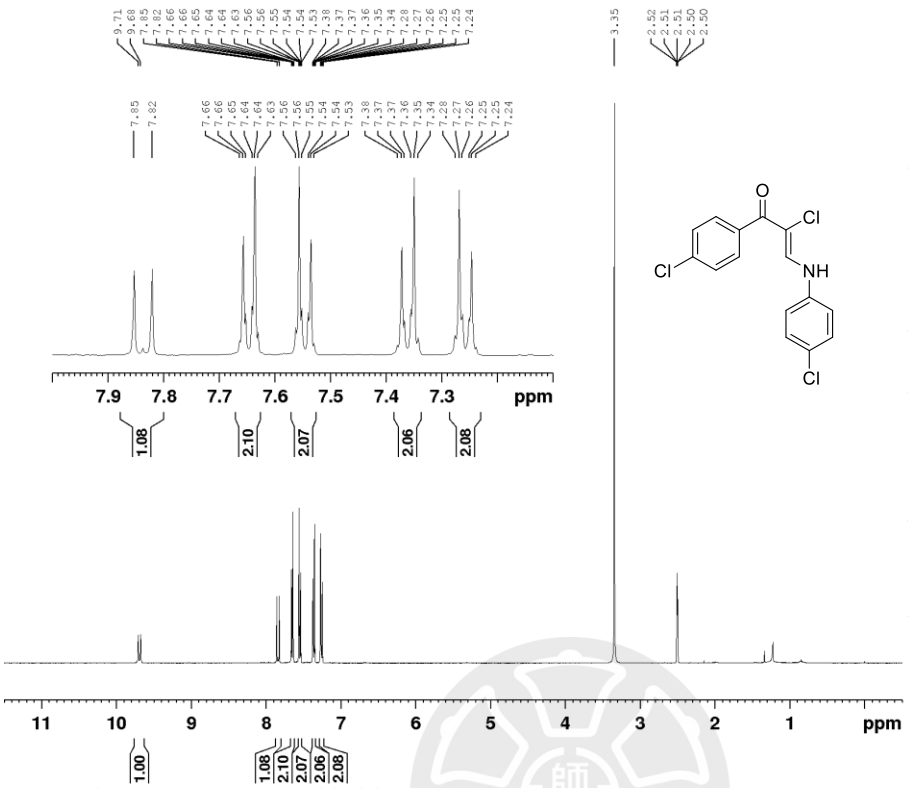
F2 - Acquisition Parameters
Date_ 20240116
Time 22.21
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 32768
SOLVENT DMSO
NS 14950
DS 0
SWH 24038.461 Hz
FIDRES 0.733596 Hz
AQ 0.6815744 sec
RG 198.09
DW 20.800 usec
DE 6.50 usec
TE 298.2 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

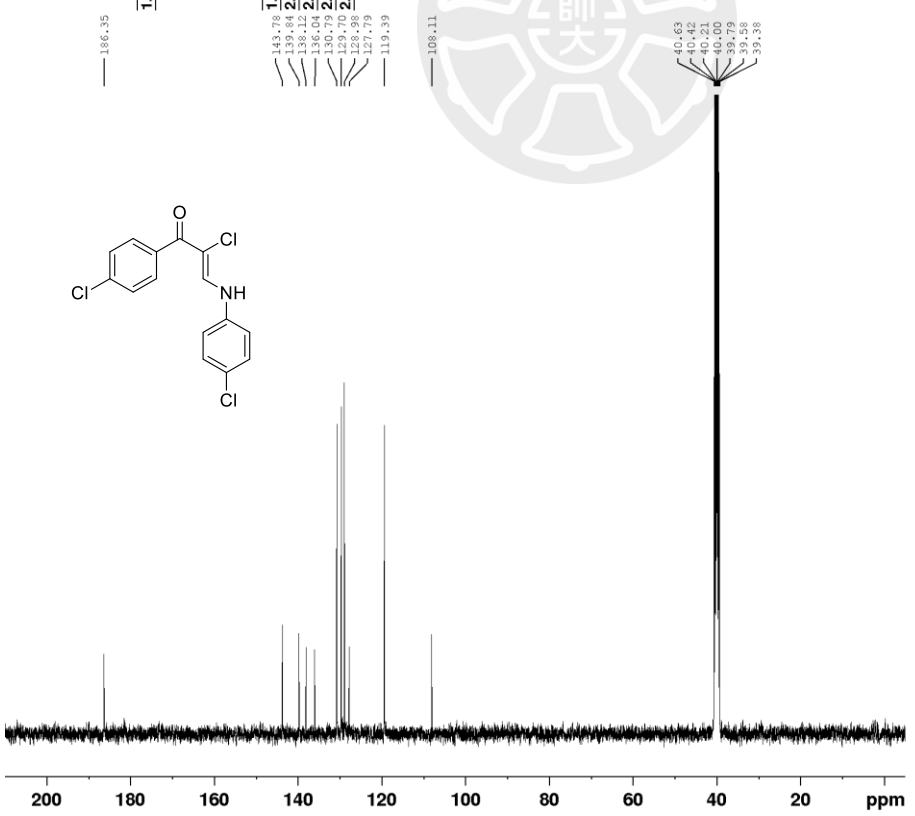
======== CHANNEL f1 ========
SFO1 100.6228298 MHz
NUC1 13C
P1 10.00 usec
PLW1 49.50000000 W

======== CHANNEL f2 ========
SFO2 400.1316005 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 90.00 usec
PLW2 12.50000000 W
PLW12 0.34722000 W
PLW13 0.28125000 W

F2 - Processing parameters
SI 32768
SF 100.6127685 MHz
WDW EM
SSB 0
LB 2.00 Hz
GB 0
PC 1.00

(*Z*)-2-chloro-1-(4-chlorophenyl)-3-((4-chlorophenyl)amino)prop-2-en-1-one (3dp)





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```
Current Data Parameters
NAME          Suresh-756
EXPNO                  3
PROCNO                 1

F2 - Acquisition Parameters
Date_           20240108
Time               20.23
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG             zg30
TD                 32768
SOLVENT             DMSO
NS                    16
DS                     0
SWH             7211.539 Hz
FIDRES          0.220079 Hz
AQ              2.2719147 sec
RG                 128.9
DW                69.333 usec
DE                10.06 usec
TE                298.3 K
D1             2.00000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1         400.1324008 MHz
NUC1                   1H
P1                  15.00 usec
PLW1        13.50000000 W

F2 - Processing parameters
SI                 16384
SF          400.1300000 MHz
WDW                   EM
SSB                    0
LB                     0 Hz
GB                     0
PC                  1.00
```

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```
Current Data Parameters
NAME          Suresh-756
EXPNO                  4
PROCNO                 1

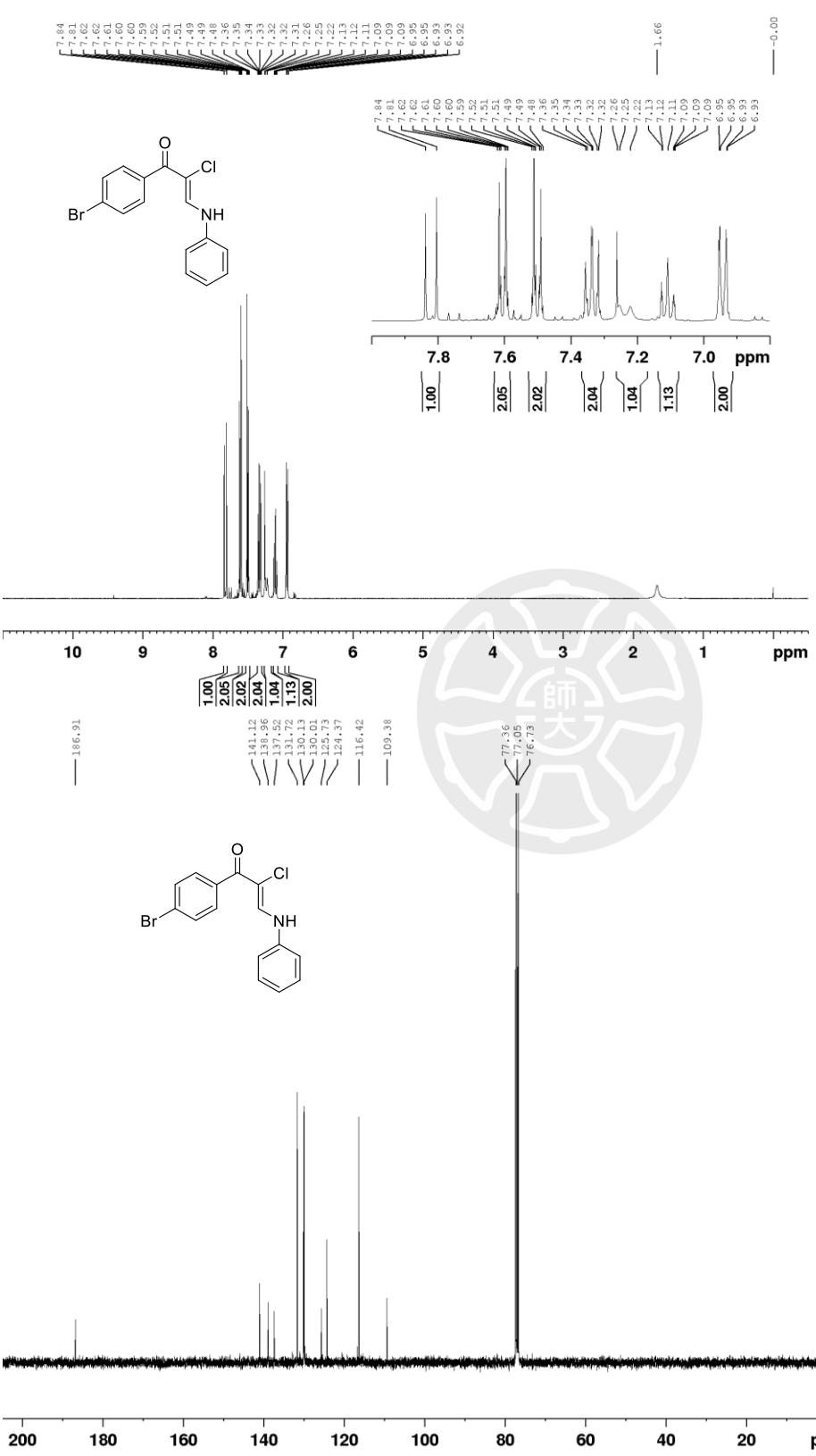
F2 - Acquisition Parameters
Date_           20240108
Time               20.25
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG          zgpg30
TD                 32768
SOLVENT             DMSO
NS                   400
DS                     0
SWH            24038.461 Hz
FIDRES          0.733596 Hz
AQ              0.6815744 sec
RG                 198.09
DW                20.800 usec
DE                 6.50 usec
TE                298.3 K
D1             2.00000000 sec
D11            0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1         100.6228298 MHz
NUC1                   13C
P1                  10.00 usec
PLW1        49.50000000 W

======== CHANNEL f2 ========
SFO2         400.1316005 MHz
NUC2                   1H
CPDPRG[2         waltz16
PCPD2               90.00 usec
PLW2        12.50000000 W
PLW12        0.34722000 W
PLW13        0.28125000 W

F2 - Processing parameters
SI                 32768
SF          100.6127685 MHz
WDW                   EM
SSB                    0
LB                  2.00 Hz
GB                     0
PC                  1.00
```

(*Z*)-1-(4-bromophenyl)-2-chloro-3-(phenylamino)prop-2-en-1-one (3ea)



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```
Current Data Parameters
NAME          Suresh-741
EXPNO                  2
PROCNO                 1

F2 - Acquisition Parameters
Date_           20231212
Time               11.08
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG             zg30
TD                 32768
SOLVENT            CDC13
NS                    16
DS                     0
SWH             7211.539 Hz
FIDRES          0.220079 Hz
AQ              2.2719147 sec
RG                 128.9
DW                69.333 usec
DE                10.06 usec
TE                298.4 K
D1             2.00000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1         400.1324008 MHz
NUC1                   1H
P1                  15.00 usec
PLW1        13.50000000 W

F2 - Processing parameters
SI                 16384
SF           400.1300094 MHz
WDW                   EM
SSB                    0
LB                     0 Hz
GB                     0
PC                  1.00
```

BRUKER

```
Current Data Parameters
NAME              exp 741
EXPNO                   2
PROCNO                  1

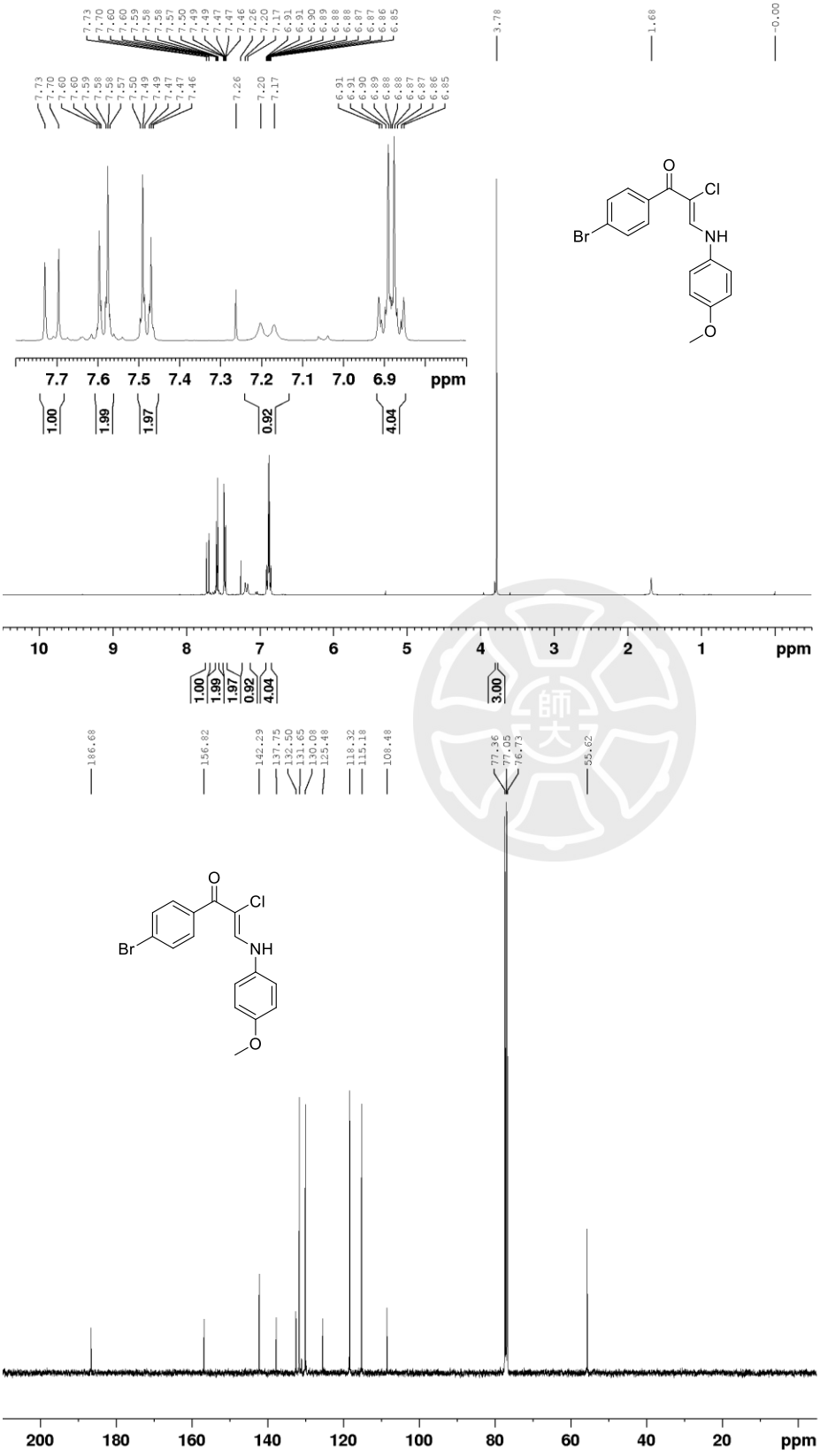
F2 - Acquisition Parameters
Date_           20231211
Time               22.14
INSTRUM            spect
PROBHD   5 mm BBO BB-1H
PULPROG          zgpg30
TD                 32768
SOLVENT            CDC13
NS                   512
DS                     0
SWH            24038.461 Hz
FIDRES          0.733596 Hz
AQ              0.6815744 sec
RG                 32768
DW                20.800 usec
DE                 6.50 usec
TE                295.2 K
D1             2.00000000 sec
D11            0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  13C
P1                 10.00 usec
PL1                 4.20 dB
SFO1         100.6233325 MHz

======== CHANNEL f2 ========
CPDPRG[2          waltz16
NUC2                   1H
PCPD2              90.00 usec
PL2                10.20 dB
PL12               26.00 dB
PL13               29.00 dB
SFO2         400.1316005 MHz

F2 - Processing parameters
SI                 32768
SF           100.6127690 MHz
WDW                   EM
SSB                    0
LB                  1.00 Hz
GB                     0
PC                  1.00
```

(*Z*)-1-(4-bromophenyl)-2-chloro-3-((4-methoxyphenyl)amino)prop-2-en-1-one (3ej)




```
Current Data Parameters
NAME          Suresh-742
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20231212
Time               18.13
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG             zg30
TD                 32768
SOLVENT            CDCl3
NS                    16
DS                     0
SWH             7211.539 Hz
FIDRES          0.220079 Hz
AQ             2.2719147 sec
RG                113.31
DW                69.333 usec
DE                 10.06 usec
TE                299.3 K
D1             2.00000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1          400.1324008 MHz
NUC1                  1H
P1                 15.00 usec
PLW1          13.50000000 W

F2 - Processing parameters
SI                 16384
SF            400.1300090 MHz
WDW                   EM
SSB                    0
LB                     0 Hz
GB                     0
PC                  1.00
```

```
Current Data Parameters
NAME          Suresh-742
EXPNO                  2
PROCNO                 1

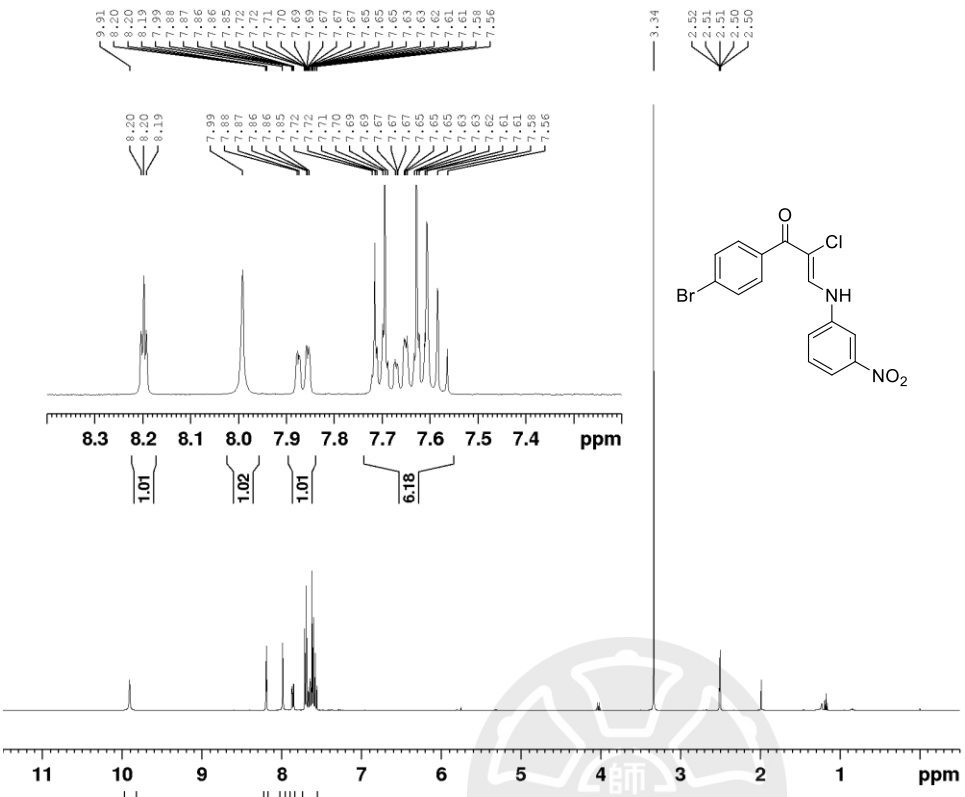
F2 - Acquisition Parameters
Date_           20231212
Time               18.16
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG          zgpg30
TD                 32768
SOLVENT            CDCl3
NS                   412
DS                     0
SWH            24038.461 Hz
FIDRES          0.733596 Hz
AQ             0.6815744 sec
RG                198.09
DW                20.800 usec
DE                 6.50 usec
TE                299.4 K
D1             2.00000000 sec
D11            0.03000000 sec
TD0                    1

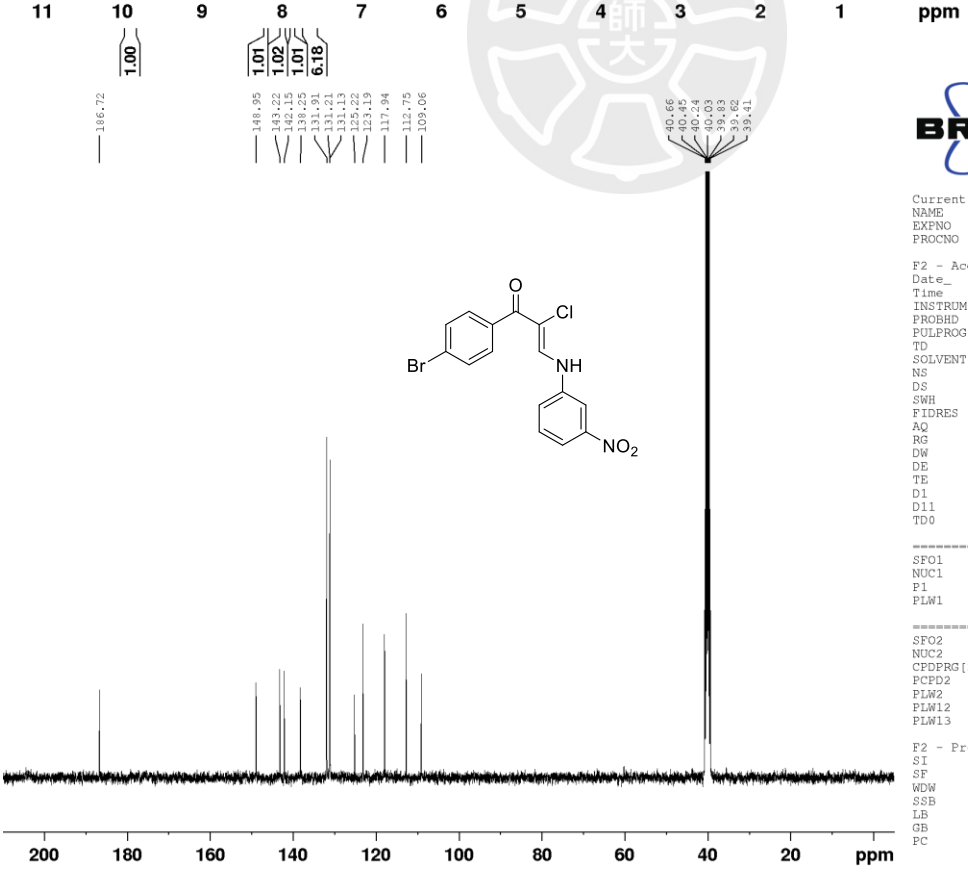
======== CHANNEL f1 ========
SFO1          100.6228298 MHz
NUC1                  13C
P1                 10.00 usec
PLW1          49.50000000 W

======== CHANNEL f2 ========
SFO2          400.1316005 MHz
NUC2                  1H
CPDPRG[2          waltz16
PCPD2              90.00 usec
PLW2          12.50000000 W
PLW12          0.34722000 W
PLW13          0.28125000 W

F2 - Processing parameters
SI                 32768
SF            100.6127685 MHz
WDW                   EM
SSB                    0
LB                  2.00 Hz
GB                     0
PC                  1.00
```

(*Z*)-1-(4-bromophenyl)-2-chloro-3-((3-nitrophenyl)amino)prop-2-en-1-one (3el)





BRUKER

```
Current Data Parameters
NAME          Suresh-744
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20231215
Time               10.39
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG             zg30
TD                 32768
SOLVENT             DMSO
NS                    16
DS                     0
SWH             7211.539 Hz
FIDRES          0.220079 Hz
AQ              2.2719147 sec
RG                 113.31
DW                 69.333 usec
DE                 10.06 usec
TE                 298.3 K
D1              2.00000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1          400.1324008 MHz
NUC1                  1H
P1                 15.00 usec
PLW1          13.50000000 W

F2 - Processing parameters
SI                 16384
SF            400.1300000 MHz
WDW                   EM
SSB                    0
LB                     0 Hz
GB                     0
PC                  1.00
```

BRUKER

```
Current Data Parameters
NAME          Suresh-744
EXPNO                  2
PROCNO                 1

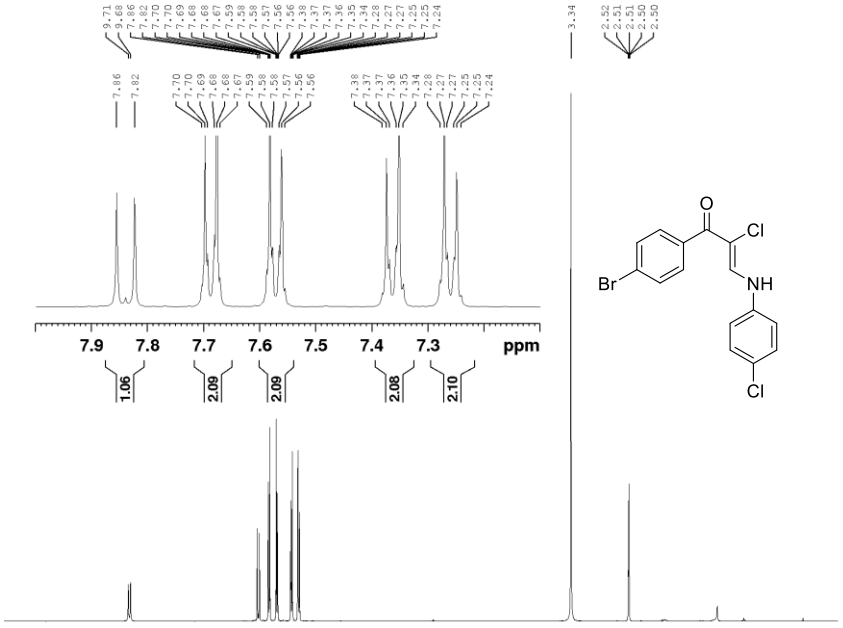
F2 - Acquisition Parameters
Date_           20231215
Time               10.42
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG           zgpg30
TD                 32768
SOLVENT             DMSO
NS                   512
DS                     0
SWH            24038.461 Hz
FIDRES          0.733596 Hz
AQ              0.6815744 sec
RG                 198.09
DW                 20.800 usec
DE                  6.50 usec
TE                 298.9 K
D1              2.00000000 sec
D11             0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1          100.6228298 MHz
NUC1                  13C
P1                 10.00 usec
PLW1          49.50000000 W

======== CHANNEL f2 ========
SFO2          400.1316005 MHz
NUC2                  1H
CPDPRG[2          waltz16
PCPD2              90.00 usec
PLW2          12.50000000 W
PLW12          0.34722000 W
PLW13          0.28125000 W

F2 - Processing parameters
SI                 32768
SF            100.6127685 MHz
WDW                   EM
SSB                    0
LB                  2.00 Hz
GB                     0
PC                  1.00
```

(*Z*)-1-(4-bromophenyl)-2-chloro-3-((4-chlorophenyl)amino)prop-2-en-1-one (3ep)

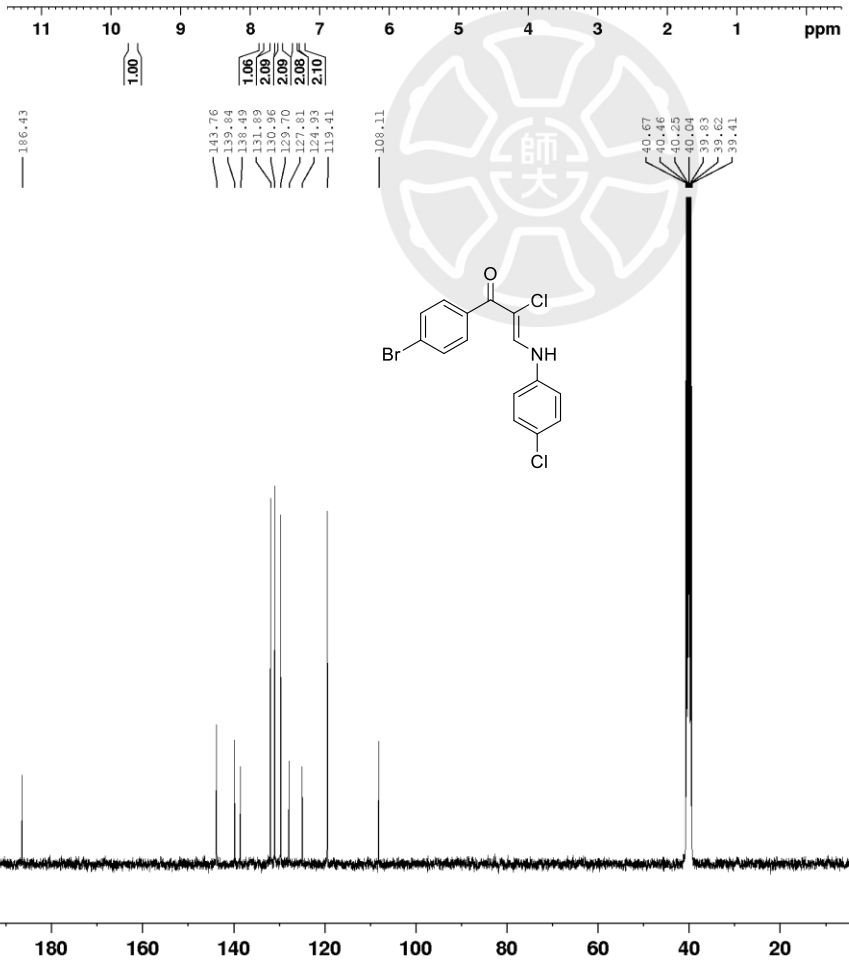


```
Current Data Parameters
NAME          Suresh-743
EXPNO                  2
PROCNO                 1

F2 - Acquisition Parameters
Date_           20231215
Time               11.17
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG             zg30
TD                 32768
SOLVENT             DMSO
NS                    16
DS                     0
SWH             7211.539 Hz
FIDRES          0.220079 Hz
AQ             2.2719147 sec
RG                 128.9
DW                69.333 usec
DE                 10.06 usec
TE                298.9 K
D1             2.00000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1         400.1324008 MHz
NUC1                   1H
P1                 15.00 usec
PLW1         13.50000000 W

F2 - Processing parameters
SI                 16384
SF           400.1300000 MHz
WDW                   EM
SSB                    0
LB                  0 Hz
GB                     0
PC                  1.00
```



```
Current Data Parameters
NAME          Suresh-743
EXPNO                  5
PROCNO                 1

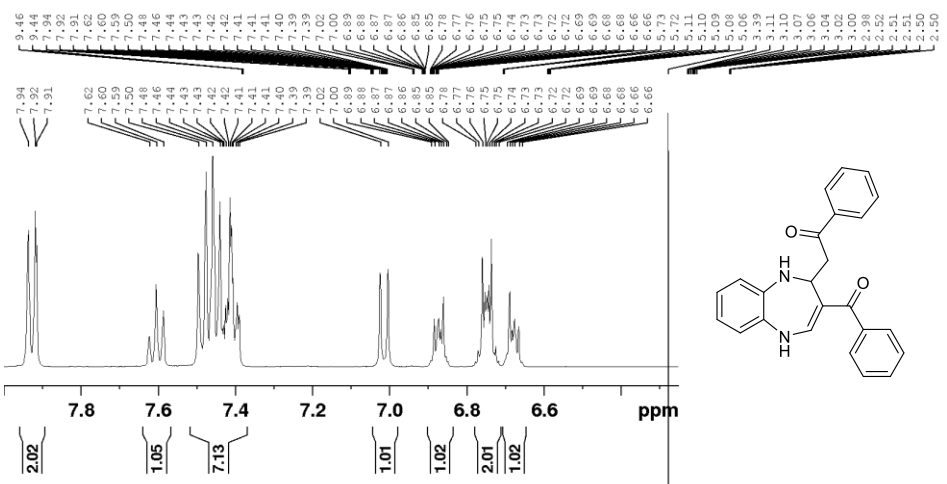
F2 - Acquisition Parameters
Date_           20231215
Time               13.05
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG          zgpg30
TD                 32768
SOLVENT             DMSO
NS                  1324
DS                     0
SWH            24038.461 Hz
FIDRES          0.733596 Hz
AQ             0.6815744 sec
RG                 198.09
DW                20.800 usec
DE                 6.50 usec
TE                298.7 K
D1             2.00000000 sec
D11            0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1         100.6228298 MHz
NUC1                  13C
P1                 10.00 usec
PLW1         49.50000000 W

======== CHANNEL f2 ========
SFO2         400.1316005 MHz
NUC2                   1H
CPDPRG[2         waltz16
PCPD2              90.00 usec
PLW2         12.50000000 W
PLW12         0.34722000 W
PLW13         0.28125000 W

F2 - Processing parameters
SI                 32768
SF           100.6127685 MHz
WDW                   EM
SSB                    0
LB                  2.00 Hz
GB                     0
PC                  1.00
```

2-(3-benzoyl-2,5-dihydro-1*H*-benzo[*b*][1,4]diazepin-2-yl)-1-phenylethan-1-one 6aa

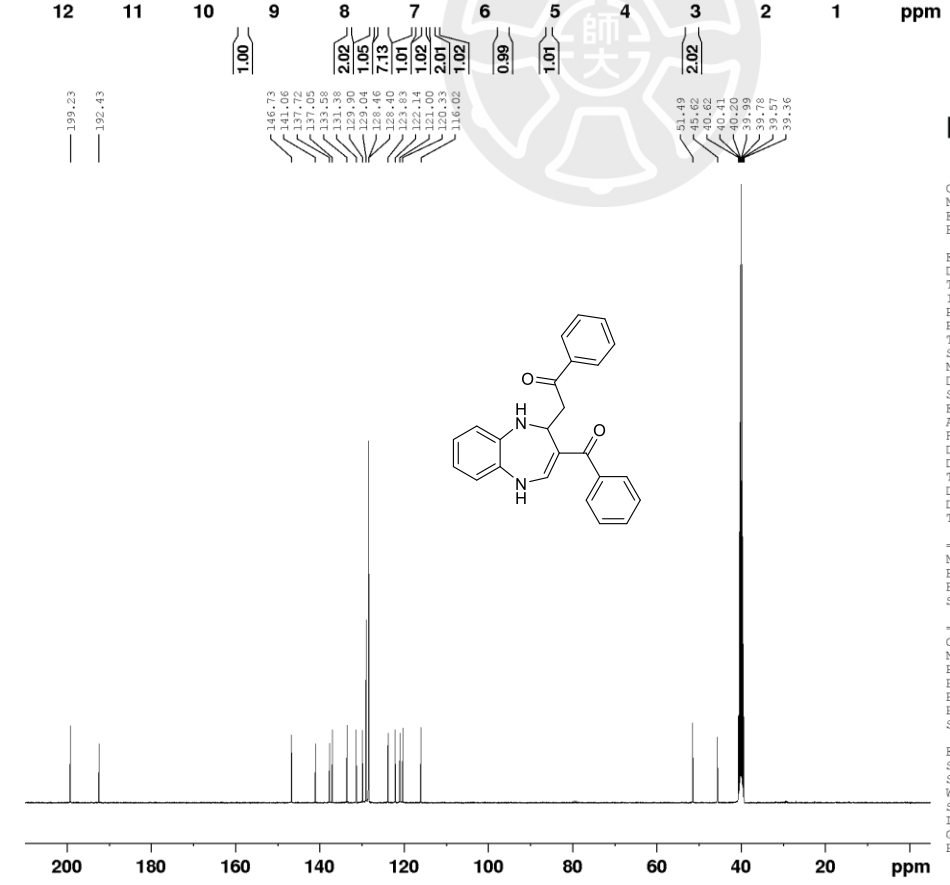


```
Current Data Parameters
NAME          Suresh-777
EXPNO                  5
PROCNO                 1

F2 - Acquisition Parameters
Date_           20240321
Time               21.26
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG             zg30
TD                 32768
SOLVENT             DMSO
NS                    16
DS                     0
SWH             7211.539 Hz
FIDRES             0.220079 Hz
AQ              2.2719147 sec
RG                  63.58
DW                 69.333 usec
DE                 10.06 usec
TE                 298.2 K
D1              2.00000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1          400.1324008 MHz
NUC1                  1H
P1                 15.00 usec
PLW1          13.50000000 W

F2 - Processing parameters
SI                 16384
SF            400.1300000 MHz
WDW                   EM
SSB                    0
LB                     0 Hz
GB                     0
PC                  1.00
```



```
Current Data Parameters
NAME             exp 777
EXPNO                  4
PROCNO                 1

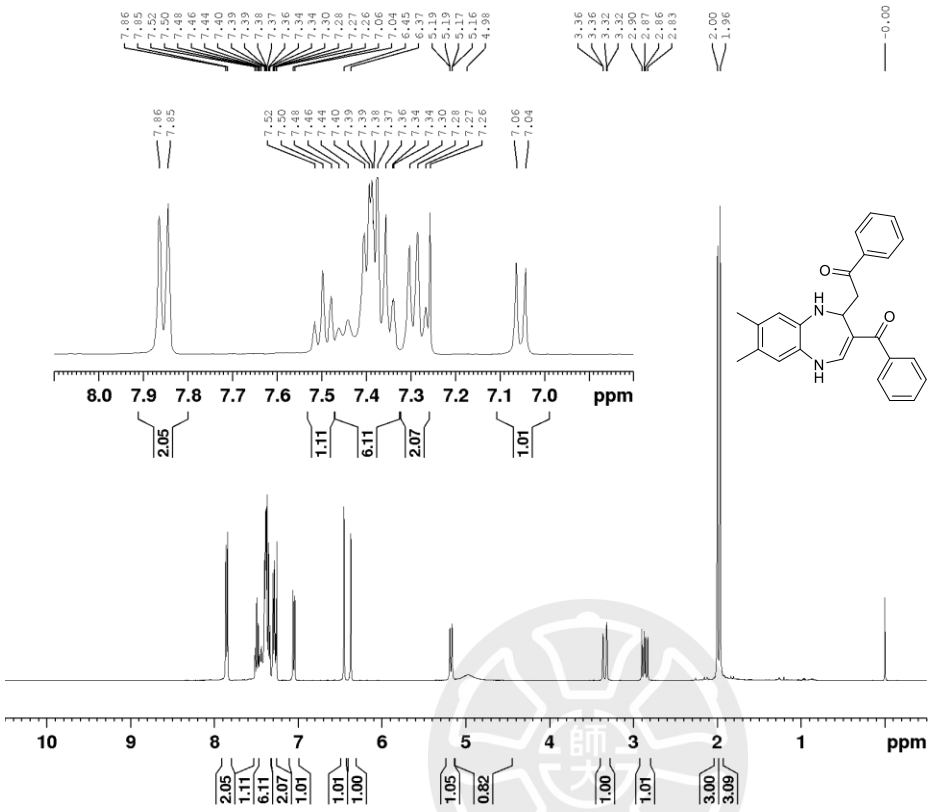
F2 - Acquisition Parameters
Date_           20240321
Time               21.51
INSTRUM            spect
PROBHD   5 mm BBO BB-1H
PULPROG          zgpg30
TD                 32768
SOLVENT             DMSO
NS                 15000
DS                     0
SWH            24038.461 Hz
FIDRES             0.733596 Hz
AQ              0.6815744 sec
RG                 32768
DW                 20.800 usec
DE                  6.50 usec
TE                 296.2 K
D1              2.00000000 sec
D11             0.03000000 sec
TD0                    1

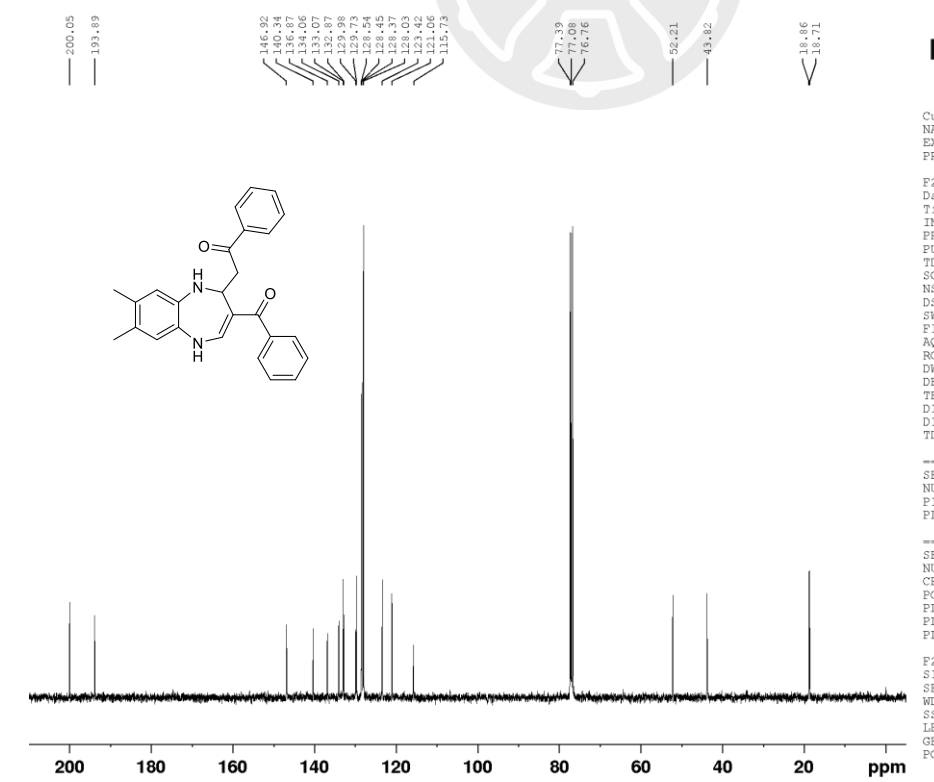
======== CHANNEL f1 ========
NUC1                  13C
P1                 10.00 usec
PL1                 4.20 dB
SFO1          100.6233325 MHz

======== CHANNEL f2 ========
CPDPRG[2          waltz16
NUC2                  1H
PCPD2              90.00 usec
PL2                10.20 dB
PL12               26.00 dB
PL13               29.00 dB
SFO2          400.1316005 MHz

F2 - Processing parameters
SI                 32768
SF            100.6127690 MHz
WDW                   EM
SSB                    0
LB                  1.00 Hz
GB                     0
PC                  1.00
```

2-(3-benzoyl-7,8-dimethyl-2,5-dihydro-1*H*-benzo[*b*][1,4]diazepin-2-yl)-1-phenylethan-1-one
6ab






```
Current Data Parameters
NAME          Suresh-785
EXPNO                  2
PROCNO                 1

F2 - Acquisition Parameters
Date_           20240402
Time               20.42
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG            zg30
TD                32768
SOLVENT           CDC13
NS                   16
DS                    0
SWH           7211.539 Hz
FIDRES        0.220079 Hz
AQ            2.2719147 sec
RG                57.42
DW               69.333 usec
DE                10.06 usec
TE                298.3 K
D1           2.00000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1        400.1324008 MHz
NUC1                   1H
P1                 15.00 usec
PLW1        13.50000000 W

F2 - Processing parameters
SI                 16384
SF          400.1300112 MHz
WDW                   EM
SSB                    0
LB                  0 Hz
GB                     0
PC                  1.00
```

```
Current Data Parameters
NAME          Suresh-785
EXPNO                  3
PROCNO                 1

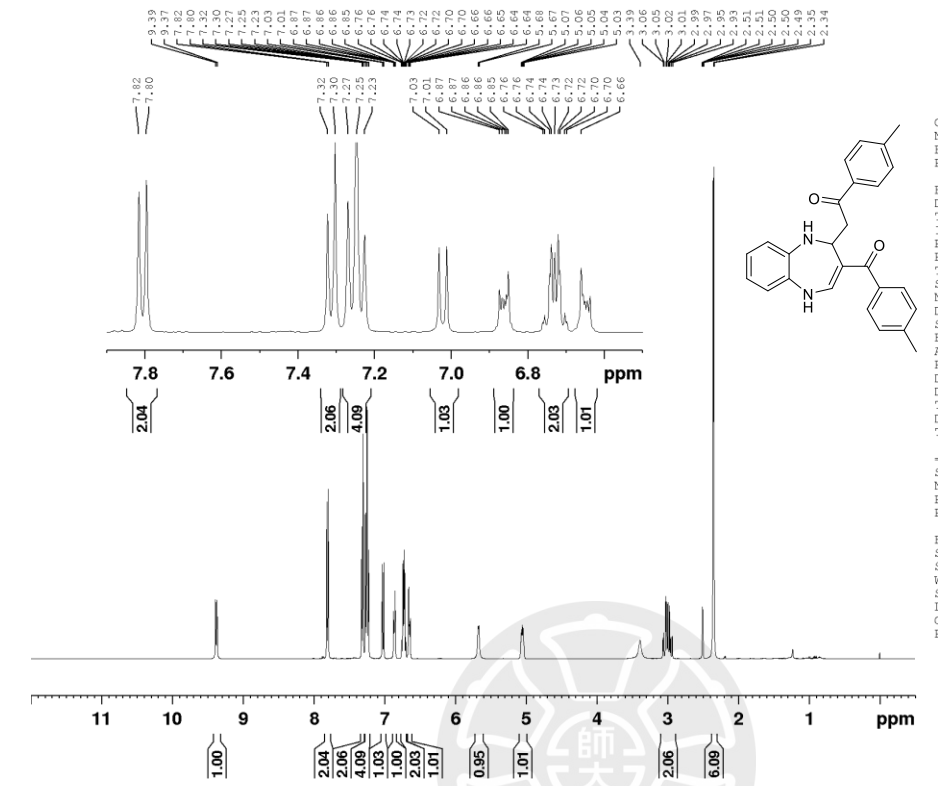
F2 - Acquisition Parameters
Date_           20240402
Time               20.44
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG          zgpg30
TD                32768
SOLVENT           CDC13
NS                  200
DS                    0
SWH          24038.461 Hz
FIDRES        0.733596 Hz
AQ            0.6815744 sec
RG                198.09
DW               20.800 usec
DE                 6.50 usec
TE                298.4 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1        100.6228298 MHz
NUC1                   13C
P1                 10.00 usec
PLW1        49.50000000 W

======== CHANNEL f2 ========
SFO2        400.1316005 MHz
NUC2                   1H
CPDPRG[2         waltz16
PCPD2              90.00 usec
PLW2        12.50000000 W
PLW12        0.34722000 W
PLW13        0.28125000 W

F2 - Processing parameters
SI                 32768
SF          100.6127685 MHz
WDW                   EM
SSB                    0
LB                  2.00 Hz
GB                     0
PC                  1.00
```

2-(3-(4-methylbenzoyl)-2,5-dihydro-1*H*-benzo[*b*][1,4]diazepin-2-yl)-1-(*p*-tolyl)ethan-1-one 6ba

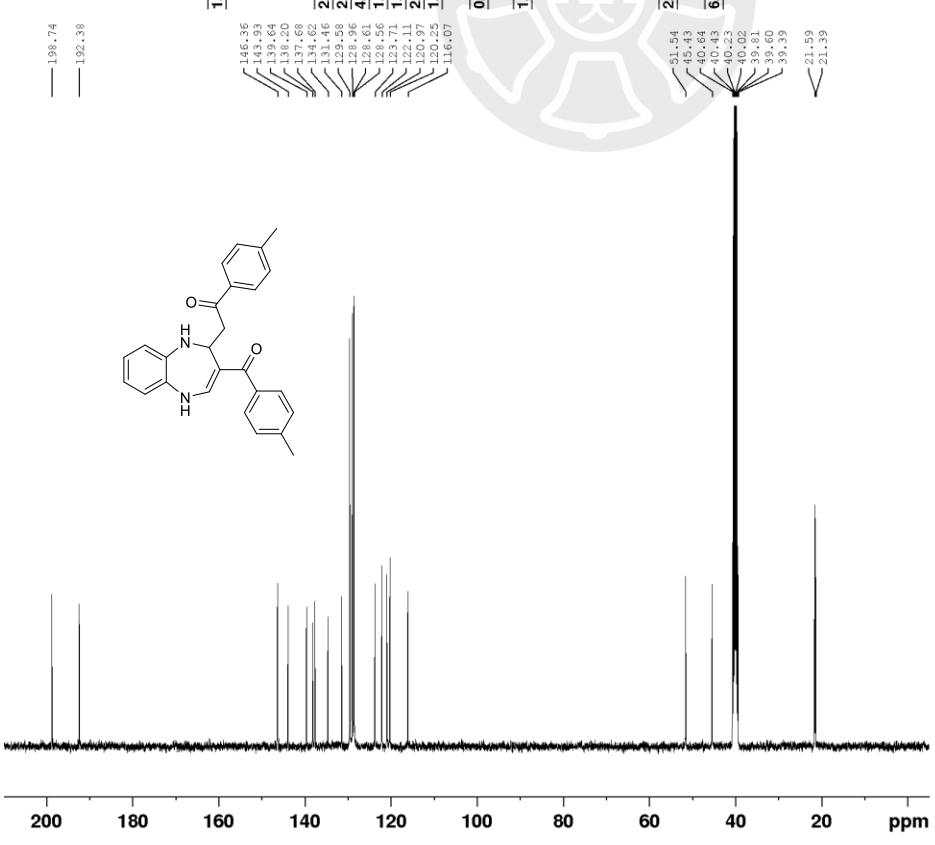


```
Current Data Parameters
NAME            Suresh-833
EXPNO                    2
PROCNO                   1

F2 - Acquisition Parameters
Date_             20240531
Time                 11.14
INSTRUM              spect
PROBHD   5 mm PABBO BB/
PULPROG              zg30
TD                  32768
SOLVENT              DMSO
NS                      16
DS                       0
SWH             7211.539 Hz
FIDRES          0.220079 Hz
AQ              2.2719147 sec
RG                   57.42
DW                  69.333 usec
DE                  10.06 usec
TE                  298.0 K
D1              2.00000000 sec
TD0                      1

======== CHANNEL f1 ========
SFO1          400.1324008 MHz
NUC1                    1H
P1                   15.00 usec
PLW1          13.50000000 W

F2 - Processing parameters
SI                   16384
SF            400.1300028 MHz
WDW                     EM
SSB                      0
LB                       0 Hz
GB                       0
PC                    1.00
```



```
Current Data Parameters
NAME            Suresh-833
EXPNO                    3
PROCNO                   1

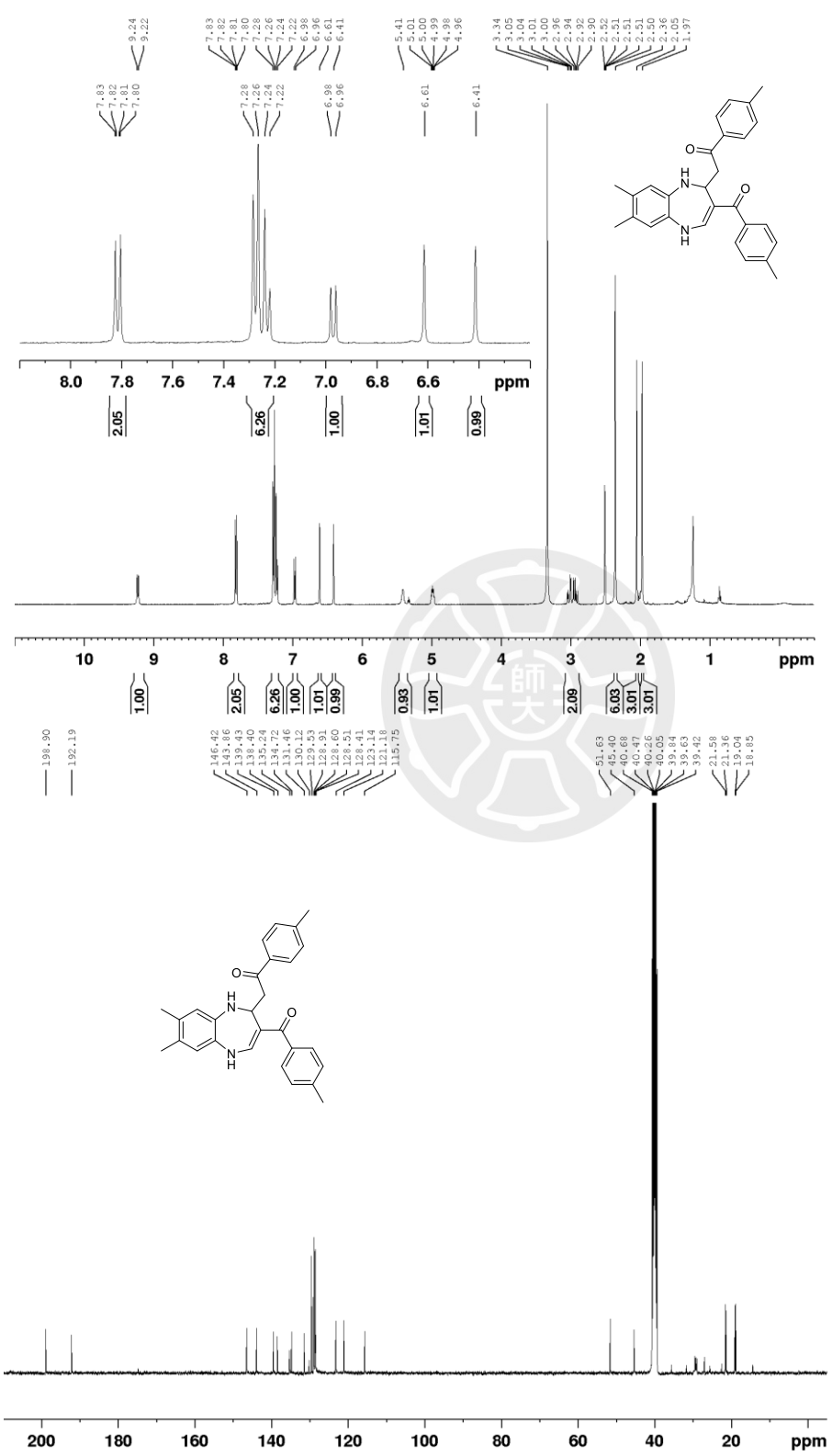
F2 - Acquisition Parameters
Date_             20240531
Time                 11.19
INSTRUM              spect
PROBHD   5 mm PABBO BB/
PULPROG            zgpg30
TD                  32768
SOLVENT              DMSO
NS                     512
DS                       0
SWH            24038.461 Hz
FIDRES          0.733596 Hz
AQ              0.6815744 sec
RG                  198.09
DW                  20.800 usec
DE                   6.50 usec
TE                  298.8 K
D1              2.00000000 sec
D11             0.03000000 sec
TD0                      1

======== CHANNEL f1 ========
SFO1          100.6228298 MHz
NUC1                    13C
P1                   10.00 usec
PLW1          49.50000000 W

======== CHANNEL f2 ========
SFO2          400.1316005 MHz
NUC2                    1H
CPDPRG[2             waltz16
PCPD2                90.00 usec
PLW2          12.50000000 W
PLW12          0.34722000 W
PLW13          0.28125000 W

F2 - Processing parameters
SI                   32768
SF            100.6127685 MHz
WDW                     EM
SSB                      0
LB                    2.00 Hz
GB                       0
PC                    1.00
```

2-(7,8-dimethyl-3-(4-methylbenzoyl)-2,5-dihydro-1*H*-benzo[*b*][1,4]diazepin-2-yl)-1-(*p*-tolyl)ethan-1-one 6bb



```
Current Data Parameters
NAME      Exp-1-revision work
EXPNO                   26
PROCNO                   1

F2 - Acquisition Parameters
Date_           20240430
Time              22.04
INSTRUM           spect
PROBHD    5 mm PABBO BB/
PULPROG           zg30
TD               32768
SOLVENT           DMSO
NS                  16
DS                   0
SWH           7211.539 Hz
FIDRES        0.220079 Hz
AQ            2.2719147 sec
RG                128.9
DW               69.333 usec
DE               10.06 usec
TE               298.3 K
D1            2.00000000 sec
TD0                  1

======== CHANNEL f1 ========
SFO1        400.1324008 MHz
NUC1               1H
P1              15.00 usec
PLW1        13.50000000 W

F2 - Processing parameters
SI                16384
SF          400.1300000 MHz
WDW                EM
SSB                 0
LB                  0 Hz
GB                  0
PC               1.00
```



```
Current Data Parameters
NAME      Exp-1-revision work
EXPNO                   27
PROCNO                   2

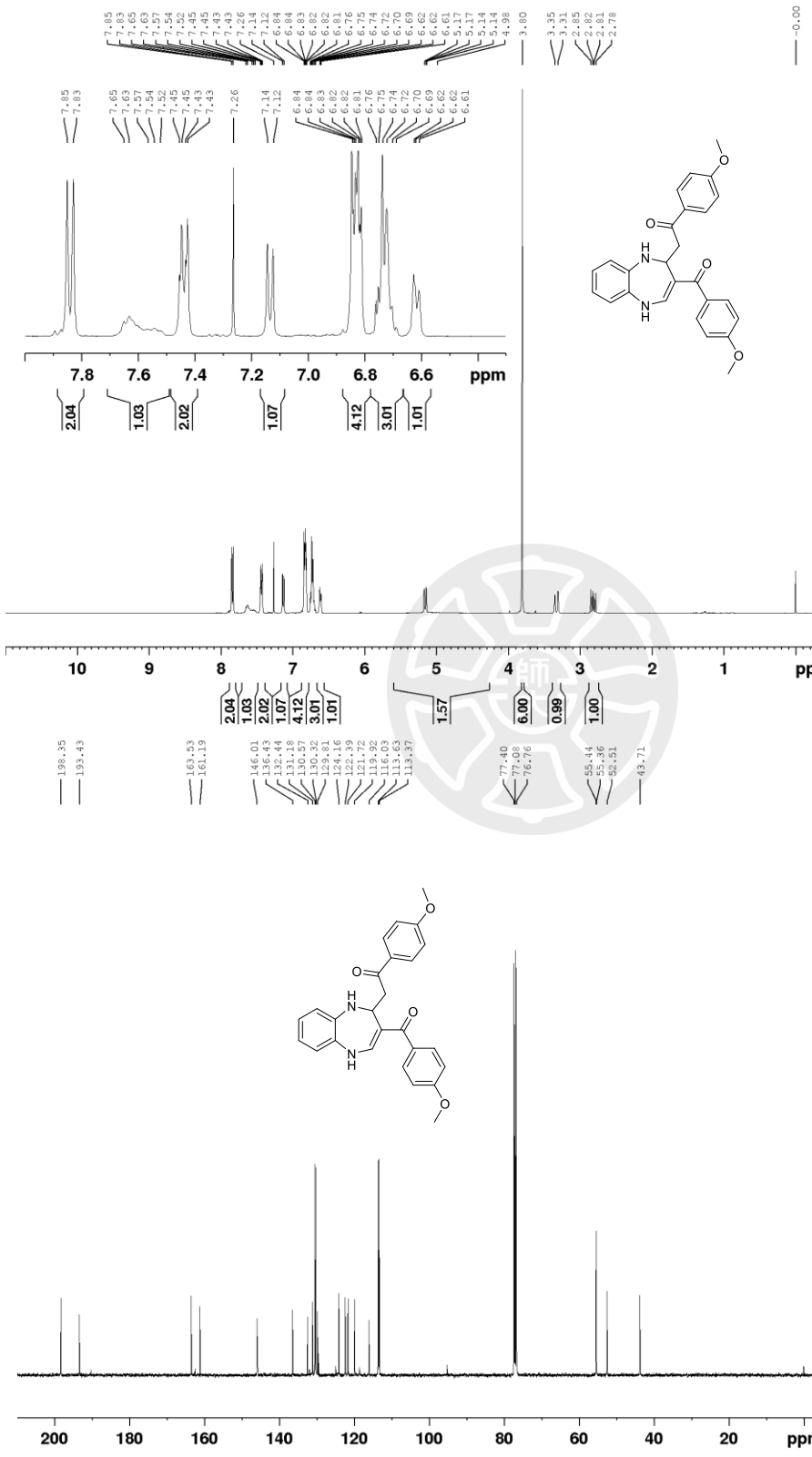
F2 - Acquisition Parameters
Date_           20240501
Time              9.32
INSTRUM           spect
PROBHD    5 mm PABBO BB/
PULPROG          zgpg30
TD               32768
SOLVENT           DMSO
NS                15000
DS                    0
SWH          24038.461 Hz
FIDRES        0.733596 Hz
AQ            0.6815744 sec
RG               198.09
DW               20.800 usec
DE                6.50 usec
TE               300.4 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0                  1

======== CHANNEL f1 ========
SFO1        100.6228298 MHz
NUC1               13C
P1              10.00 usec
PLW1        49.50000000 W

======== CHANNEL f2 ========
SFO2        400.1316005 MHz
NUC2               1H
CPDPRG[2         waltz16
PCPD2           90.00 usec
PLW2        12.50000000 W
PLW12        0.34722000 W
PLW13        0.28125000 W

F2 - Processing parameters
SI                32768
SF          100.6127685 MHz
WDW                EM
SSB                 0
LB               2.00 Hz
GB                  0
PC               1.00
```

2-(3-(4-methoxybenzoyl)-2,5-dihydro-1*H*-benzo[*b*][1,4]diazepin-2-yl)-1-(4-methoxyphenyl)ethan-1-one 6ca



```
Current Data Parameters
NAME          Suresh-829
EXPNO                  2
PROCNO                 1

F2 - Acquisition Parameters
Date_           20240522
Time               17.20
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG             zg30
TD                 32768
SOLVENT            CDC13
NS                    16
DS                     0
SWH             7211.539 Hz
FIDRES          0.220079 Hz
AQ              2.2719147 sec
RG                 63.58
DW                69.333 usec
DE                10.06 usec
TE                297.8 K
D1             2.00000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1         400.1324008 MHz
NUC1                   1H
P1                  15.00 usec
PLW1        13.50000000 W

F2 - Processing parameters
SI                 16384
SF           400.1300085 MHz
WDW                   EM
SSB                    0
LB                     0 Hz
GB                     0
PC                  1.00
```



```
Current Data Parameters
NAME          Suresh-829
EXPNO                  3
PROCNO                 1

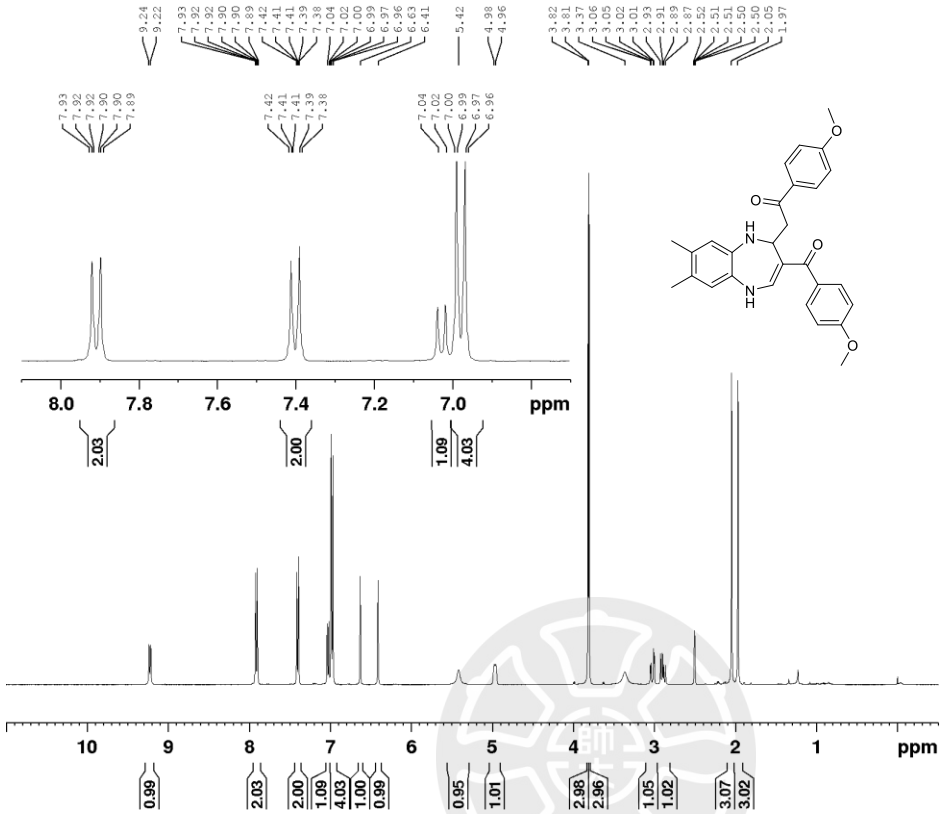
F2 - Acquisition Parameters
Date_           20240522
Time               17.25
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG          zgpg30
TD                 32768
SOLVENT            CDC13
NS                   978
DS                     0
SWH            24038.461 Hz
FIDRES          0.733596 Hz
AQ              0.6815744 sec
RG                198.09
DW                20.800 usec
DE                 6.50 usec
TE                298.5 K
D1             2.00000000 sec
D11            0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1         100.6228298 MHz
NUC1                   13C
P1                  10.00 usec
PLW1        49.50000000 W

======== CHANNEL f2 ========
SFO2         400.1316005 MHz
NUC2                   1H
CPDPRG[2          waltz16
PCPD2              90.00 usec
PLW2        12.50000000 W
PLW12        0.34722000 W
PLW13        0.28125000 W

F2 - Processing parameters
SI                 32768
SF           100.6127685 MHz
WDW                   EM
SSB                    0
LB                  2.00 Hz
GB                     0
PC                  1.00
```

2-(3-(4-methoxybenzoyl)-7,8-dimethyl-2,5-dihydro-1H-benzo[b][1,4]diazepin-2-yl)-1-(4-methoxyphenyl)ethan-1-one 6cb

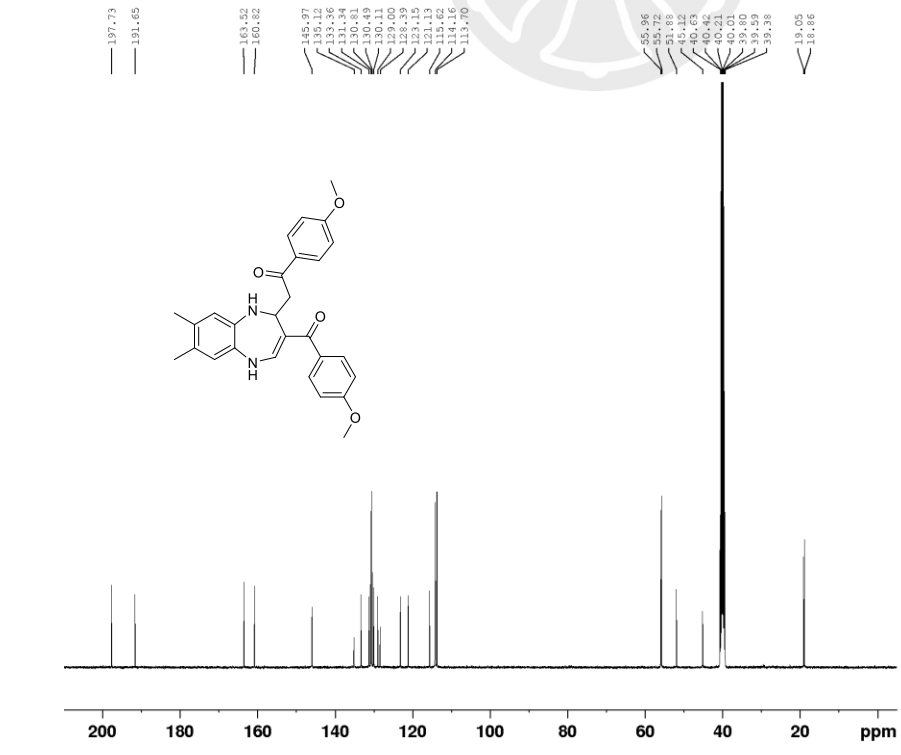


```
Current Data Parameters
NAME          Suresh-830
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20240523
Time               20.00
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG             zg30
TD                 32768
SOLVENT             DMSO
NS                    16
DS                     0
SWH             7211.539 Hz
FIDRES          0.220079 Hz
AQ              2.2719147 sec
RG                 71.42
DW                69.333 usec
DE                 10.06 usec
TE                297.7 K
D1             2.00000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1         400.1324008 MHz
NUC1                   1H
P1                 15.00 usec
PLW1         13.50000000 W

F2 - Processing parameters
SI                 16384
SF           400.1300000 MHz
WDW                   EM
SSB                    0
LB                     0 Hz
GB                     0
PC                  1.00
```



```
Current Data Parameters
NAME             exp 830
EXPNO                  3
PROCNO                 1

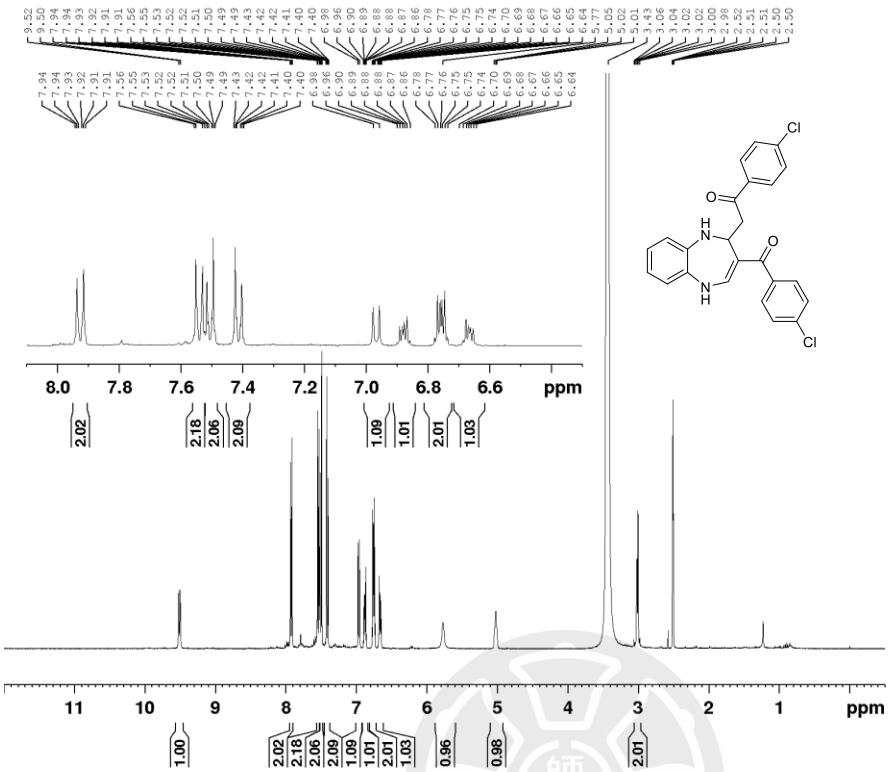
F2 - Acquisition Parameters
Date_           20240523
Time               22.11
INSTRUM            spect
PROBHD   5 mm BBO BB-1H
PULPROG           zgpg30
TD                 32768
SOLVENT             DMSO
NS                 12000
DS                     0
SWH            24038.461 Hz
FIDRES          0.733596 Hz
AQ              0.6815744 sec
RG                 32768
DW                20.800 usec
DE                 6.50 usec
TE                295.9 K
D1             2.00000000 sec
D11            0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  13C
P1                 10.00 usec
PL1                 4.20 dB
SFO1         100.6233325 MHz

======== CHANNEL f2 ========
CPDPRG[2          waltz16
NUC2                   1H
PCPD2              90.00 usec
PL2                10.20 dB
PL12               26.00 dB
PL13               29.00 dB
SFO2         400.1316005 MHz

F2 - Processing parameters
SI                 32768
SF           100.6127690 MHz
WDW                   EM
SSB                    0
LB                  1.00 Hz
GB                     0
PC                  1.00
```

2-(3-(4-chlorobenzoyl)-2,5-dihydro-1*H*-benzo[*b*][1,4]diazepin-2-yl)-1-(4-chlorophenyl)ethan-1-one 6da

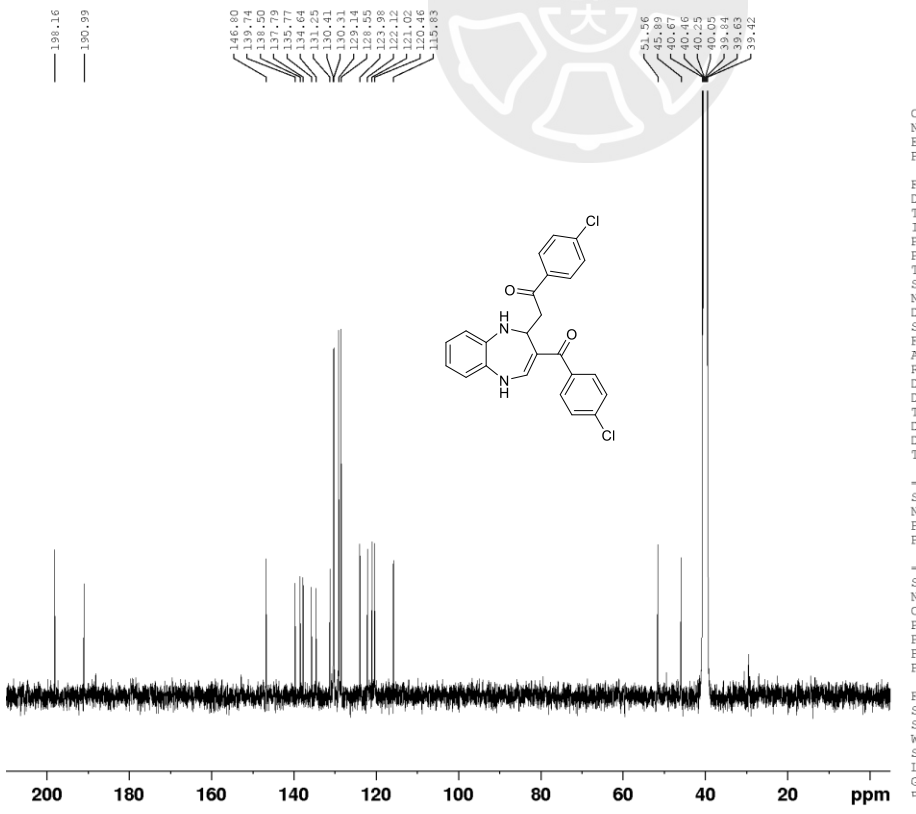


```
Current Data Parameters
NAME      Exp-1-revision work
EXPNO                   22
PROCNO                   1

F2 - Acquisition Parameters
Date_           20240425
Time              20.55
INSTRUM           spect
PROBHD   5 mm PABBO BB/
PULPROG           zg30
TD               32768
SOLVENT           DMSO
NS                   16
DS                    0
SWH            7211.539 Hz
FIDRES        0.220079 Hz
AQ            2.2719147 sec
RG                71.42
DW               69.333 usec
DE               10.06 usec
TE               298.4 K
D1            2.00000000 sec
TD0                   1

======== CHANNEL f1 ========
SFO1         400.1324008 MHz
NUC1                1H
P1                15.00 usec
PLW1        13.50000000 W

F2 - Processing parameters
SI                16384
SF           400.1300000 MHz
WDW                EM
SSB                 0
LB                  0 Hz
GB                  0
PC               1.00
```



```
Current Data Parameters
NAME      Exp-1-revision work -NJC
EXPNO                   24
PROCNO                   1

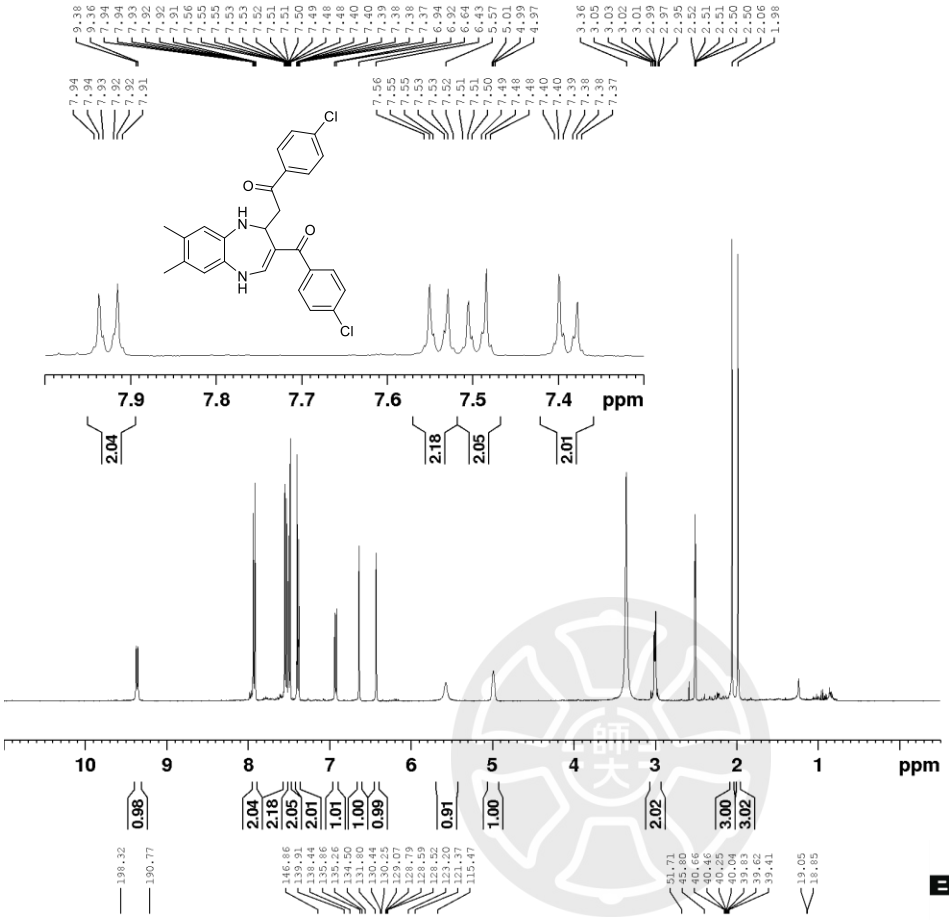
F2 - Acquisition Parameters
Date_           20240426
Time              7.19
INSTRUM           spect
PROBHD   5 mm PABBO BB/
PULPROG          zgpg30
TD               32768
SOLVENT           DMSO
NS                12017
DS                    0
SWH           24038.461 Hz
FIDRES        0.733596 Hz
AQ            0.6815744 sec
RG               198.09
DW               20.800 usec
DE                6.50 usec
TE               299.8 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
SFO1         100.6228298 MHz
NUC1                13C
P1                10.00 usec
PLW1         49.50000000 W

======== CHANNEL f2 ========
SFO2         400.1316005 MHz
NUC2                1H
CPDPRG[2         waltz16
PCPD2             90.00 usec
PLW2         12.50000000 W
PLW12         0.34722000 W
PLW13         0.28125000 W

F2 - Processing parameters
SI                32768
SF           100.6127685 MHz
WDW                EM
SSB                 0
LB                2.00 Hz
GB                  0
PC               1.00
```

2-(3-(4-chlorobenzoyl)-7,8-dimethyl-2,5-dihydro-1*H*-benzo[*b*][1,4]diazepin-2-yl)-1-(4-chlorophenyl)ethan-1-one 6db

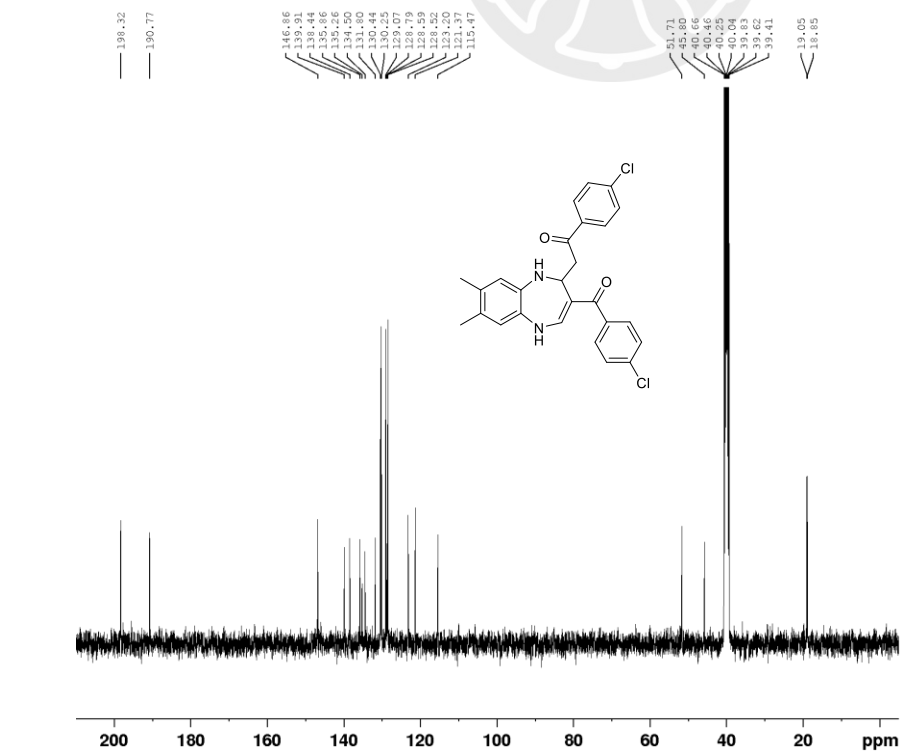


```
Current Data Parameters
NAME      Exp-1-revision work -N
EXPNO                 20
PROCNO                 1

F2 - Acquisition Parameters
Date_           20240425
Time              20.47
INSTRUM           spect
PROBHD   5 mm PABBO BB/
PULPROG           zg30
TD               32768
SOLVENT           DMSO
NS                  20
DS                   0
SWH           7211.539 Hz
FIDRES        0.220079 Hz
AQ            2.2719147 sec
RG              113.31
DW             69.333 usec
DE              10.06 usec
TE              298.4 K
D1          2.00000000 sec
TD0                  1

======== CHANNEL f1 ========
SFO1        400.1324008 MHz
NUC1                1H
P1                15.00 usec
PLW1        13.50000000 W

F2 - Processing parameters
SI                16384
SF          400.1300000 MHz
WDW                 EM
SSB                  0
LB                   0 Hz
GB                   0
PC                1.00
```



```
Current Data Parameters
NAME      Exp-1-revision work -I
EXPNO                 22
PROCNO                 1

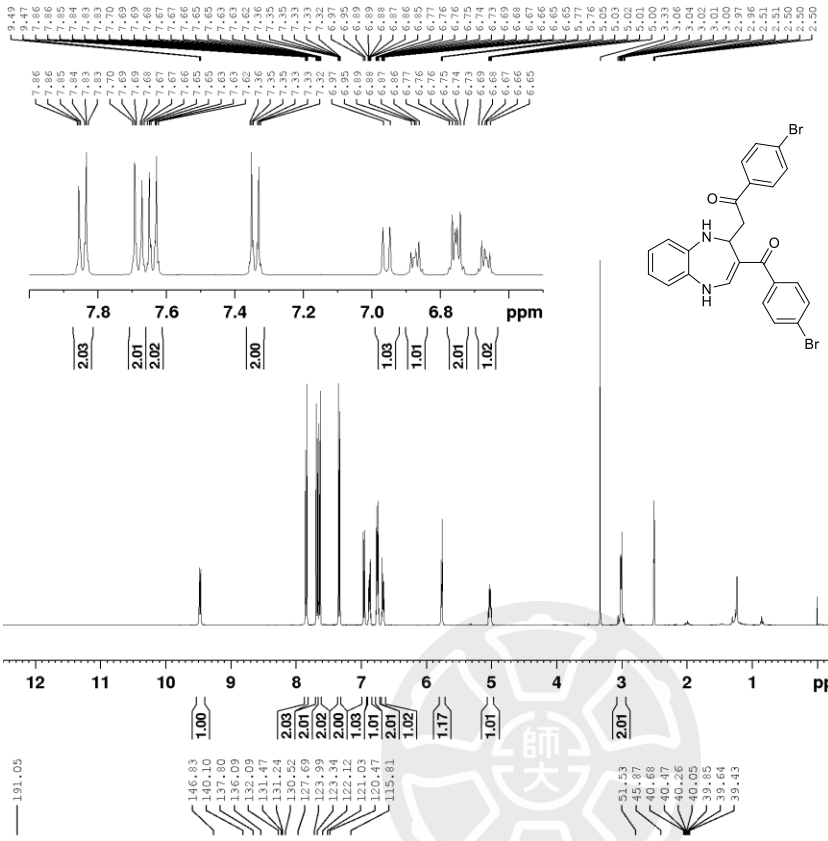
F2 - Acquisition Parameters
Date_           20240425
Time              21.44
INSTRUM           spect
PROBHD   5 mm PABBO BB/
PULPROG         zgpg30
TD               32768
SOLVENT           DMSO
NS                 505
DS                   0
SWH          24038.461 Hz
FIDRES        0.733596 Hz
AQ            0.6815744 sec
RG              198.09
DW             20.800 usec
DE              6.50 usec
TE              300.0 K
D1          2.00000000 sec
D11         0.03000000 sec
TD0                  1

======== CHANNEL f1 ========
SFO1        100.6228298 MHz
NUC1                13C
P1                10.00 usec
PLW1        49.50000000 W

======== CHANNEL f2 ========
SFO2        400.1316005 MHz
NUC2                1H
CPDPRG[2        waltz16
PCPD2             90.00 usec
PLW2        12.50000000 W
PLW12        0.34722000 W
PLW13        0.28125000 W

F2 - Processing parameters
SI                32768
SF          100.6127685 MHz
WDW                 EM
SSB                  0
LB                2.00 Hz
GB                   0
PC                1.00
```

2-(3-(4-bromobenzoyl)-2,5-dihydro-1*H*-benzo[*b*][1,4]diazepin-2-yl)-1-(4-bromophenyl)ethan-1-one 6ea

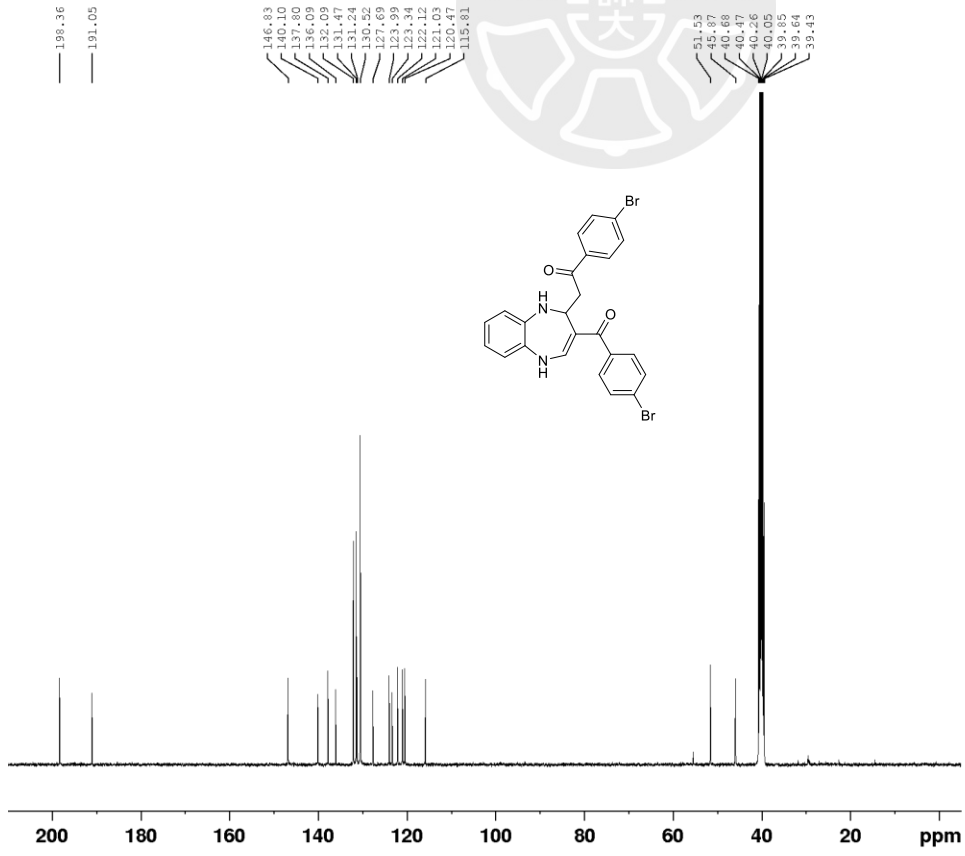


```
Current Data Parameters
NAME          Suresh-787
EXPNO                  3
PROCNO                 1

F2 - Acquisition Parameters
Date_           20240419
Time               15.55
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG             zg30
TD                 32768
SOLVENT             DMSO
NS                    16
DS                     0
SWH             7211.539 Hz
FIDRES          0.220079 Hz
AQ              2.2719147 sec
RG                113.31
DW                69.333 usec
DE                10.06 usec
TE                298.8 K
D1             2.00000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1          400.1324008 MHz
NUC1                  1H
P1                 15.00 usec
PLW1          13.50000000 W

F2 - Processing parameters
SI                 16384
SF            400.1300019 MHz
WDW                   EM
SSB                    0
LB                     0 Hz
GB                     0
PC                  1.00
```



```
Current Data Parameters
NAME          Suresh-787
EXPNO                  5
PROCNO                 1

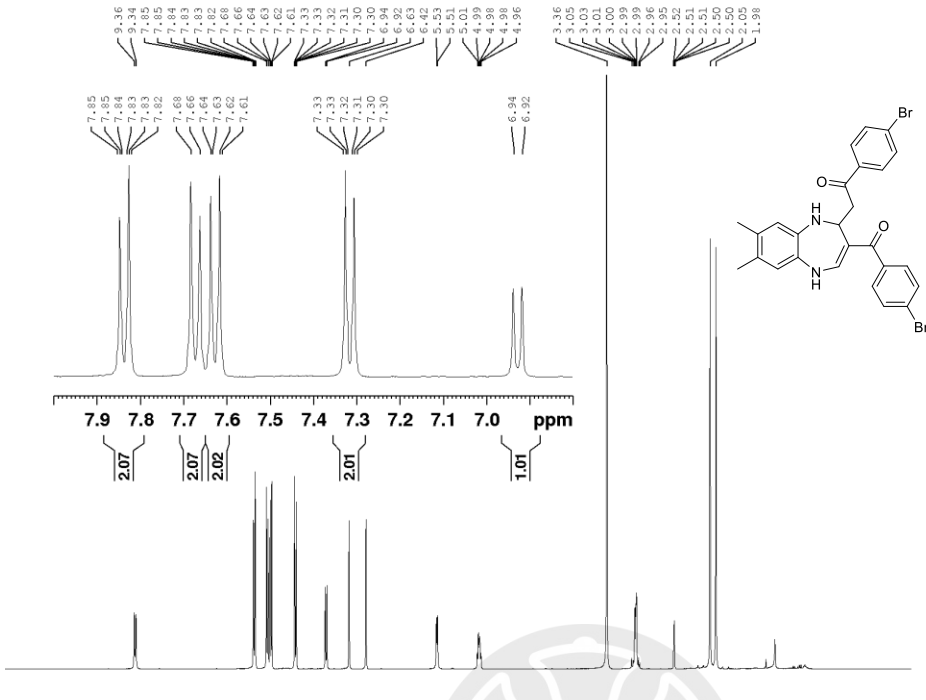
F2 - Acquisition Parameters
Date_           20240419
Time               21.59
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG           zgpg30
TD                 32768
SOLVENT             DMSO
NS                  6000
DS                     0
SWH            24038.461 Hz
FIDRES          0.733596 Hz
AQ              0.6815744 sec
RG                198.09
DW                20.800 usec
DE                 6.50 usec
TE                298.7 K
D1             2.00000000 sec
D11            0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1          100.6228298 MHz
NUC1                  13C
P1                 10.00 usec
PLW1          49.50000000 W

======== CHANNEL f2 ========
SFO2          400.1316005 MHz
NUC2                  1H
CPDPRG[2          waltz16
PCPD2              90.00 usec
PLW2          12.50000000 W
PLW12          0.34722000 W
PLW13          0.28125000 W

F2 - Processing parameters
SI                 32768
SF            100.6127685 MHz
WDW                   EM
SSB                    0
LB                  2.00 Hz
GB                     0
PC                  1.00
```

2-(3-(4-bromobenzoyl)-7,8-dimethyl-2,5-dihydro-1*H*-benzo[*b*][1,4]diazepin-2-yl)-1-(4-bromophenyl)ethan-1-one 6eb

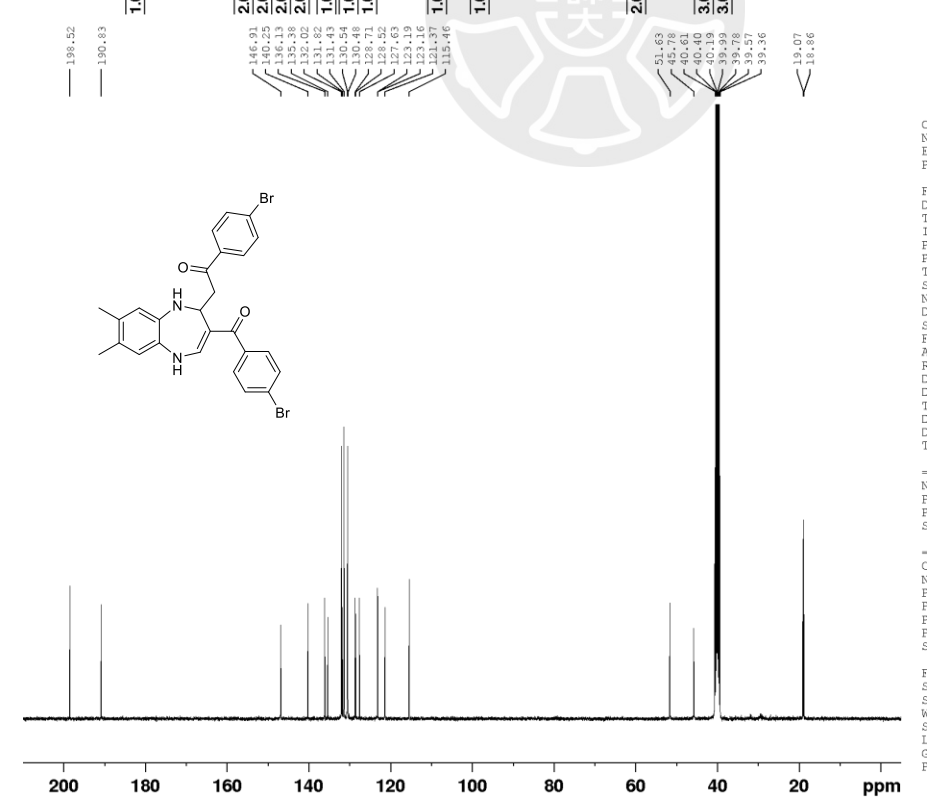


```
Current Data Parameters
NAME          Suresh-791
EXPNO                  5
PROCNO                 1

F2 - Acquisition Parameters
Date_           20240409
Time               20.50
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG             zg30
TD                 32768
SOLVENT             DMSO
NS                    16
DS                     0
SWH           7211.539 Hz
FIDRES        0.220079 Hz
AQ            2.2719147 sec
RG                 78.51
DW                69.333 usec
DE                10.06 usec
TE                298.3 K
D1            2.00000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1        400.1324008 MHz
NUC1                   1H
P1                 15.00 usec
PLW1        13.50000000 W

F2 - Processing parameters
SI                 16384
SF          400.1300000 MHz
WDW                   EM
SSB                    0
LB                     0 Hz
GB                     0
PC                  1.00
```



```
Current Data Parameters
NAME             exp 791
EXPNO                  2
PROCNO                 1

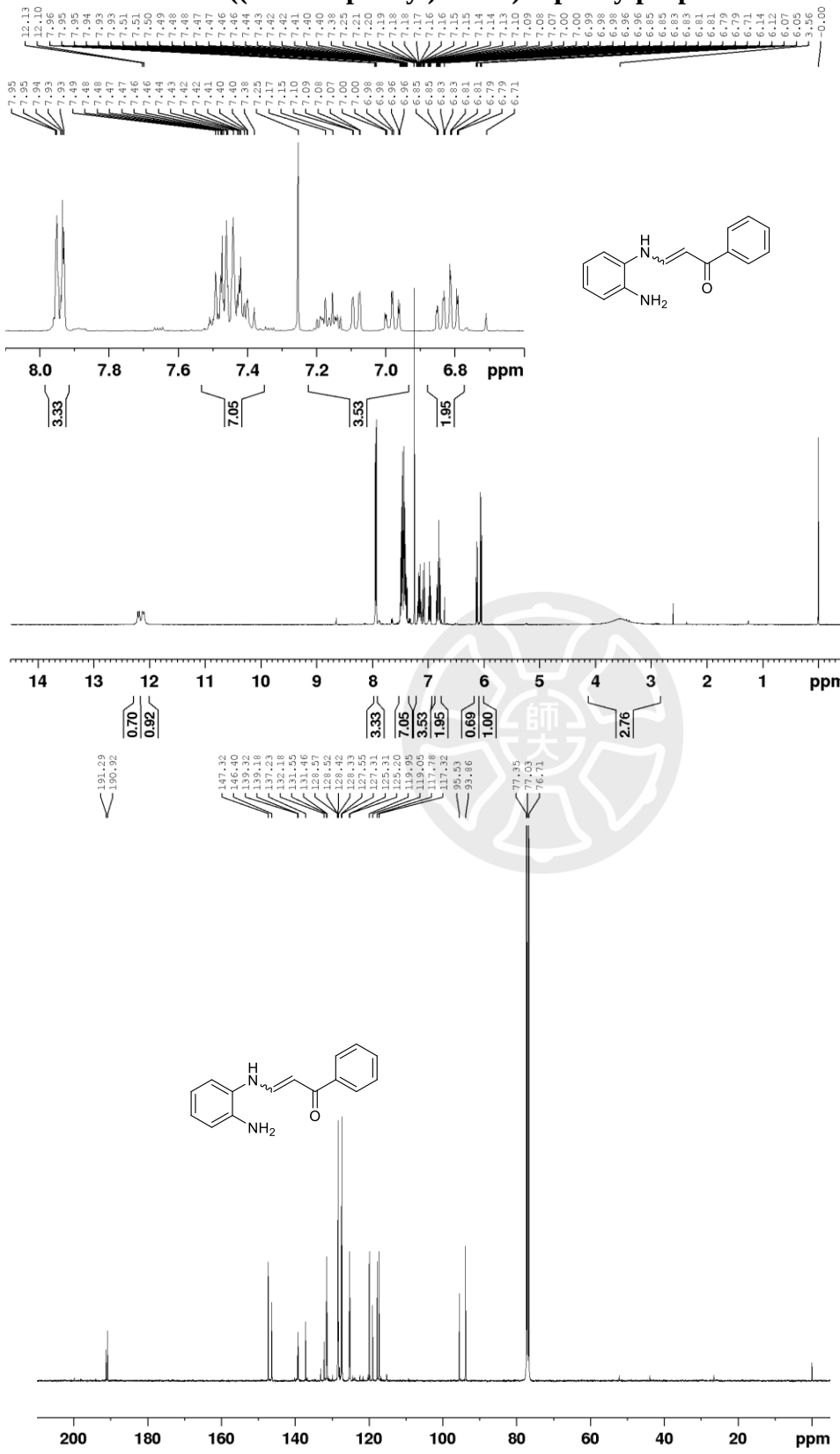
F2 - Acquisition Parameters
Date_           20240409
Time               22.11
INSTRUM            spect
PROBHD   5 mm BBO BB-1H
PULPROG          zgpg30
TD                 32768
SOLVENT              DMSO
NS                 12000
DS                     0
SWH           24038.461 Hz
FIDRES         0.733596 Hz
AQ            0.6815744 sec
RG                 32768
DW                20.800 usec
DE                 6.50 usec
TE                294.8 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  13C
P1                10.00 usec
PL1                4.20 dB
SFO1         100.6233325 MHz

======== CHANNEL f2 ========
CPDPRG[2         waltz16
NUC2                   1H
PCPD2             90.00 usec
PL2               10.20 dB
PL12              26.00 dB
PL13              29.00 dB
SFO2         400.1316005 MHz

F2 - Processing parameters
SI                 32768
SF          100.6127690 MHz
WDW                   EM
SSB                    0
LB                  1.00 Hz
GB                     0
PC                  1.00
```

3-((2-aminophenyl)amino)-1-phenylprop-2-en-1-one 7aa



```
Current Data Parameters
NAME          Suresh-821
EXPNO                  3
PROCNO                 1

F2 - Acquisition Parameters
Date_           20240515
Time               22.04
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG             zg30
TD                 32768
SOLVENT            CDC13
NS                    16
DS                     0
SWH             7211.539 Hz
FIDRES          0.220079 Hz
AQ              2.2719147 sec
RG                 99.72
DW                69.333 usec
DE                10.06 usec
TE                299.4 K
D1             2.00000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1         400.1324008 MHz
NUC1                   1H
P1                 15.00 usec
PLW1         13.50000000 W

F2 - Processing parameters
SI                 16384
SF           400.1300125 MHz
WDW                   EM
SSB                    0
LB                     0 Hz
GB                     0
PC                  1.00
```

```
Current Data Parameters
NAME          Suresh-821
EXPNO                  4
PROCNO                 1

F2 - Acquisition Parameters
Date_           20240515
Time               22.08
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG          zgpg30
TD                 32768
SOLVENT            CDC13
NS                 16297
DS                     0
SWH            24038.461 Hz
FIDRES          0.733596 Hz
AQ              0.6815744 sec
RG                198.09
DW                20.800 usec
DE                 6.50 usec
TE                300.1 K
D1             2.00000000 sec
D11            0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1         100.6228298 MHz
NUC1                   13C
P1                 10.00 usec
PLW1         49.50000000 W

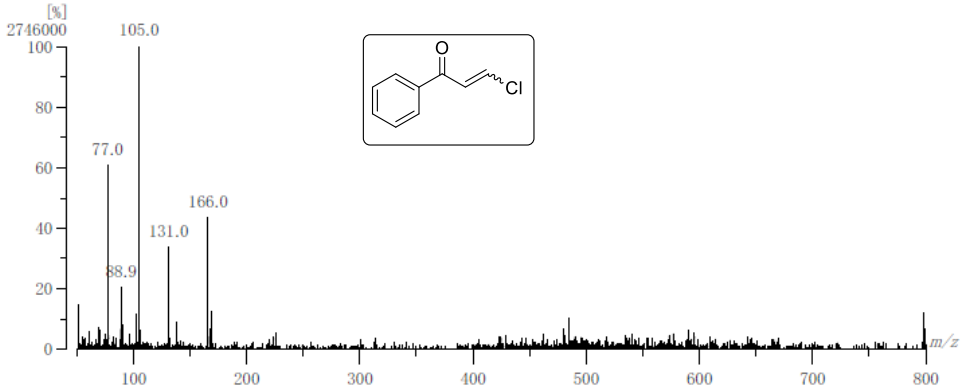
======== CHANNEL f2 ========
SFO2         400.1316005 MHz
NUC2                   1H
CPDPRG[2         waltz16
PCPD2              90.00 usec
PLW2         12.50000000 W
PLW12         0.34722000 W
PLW13         0.28125000 W

F2 - Processing parameters
SI                 32768
SF           100.6127685 MHz
WDW                   EM
SSB                    0
LB                  2.00 Hz
GB                     0
PC                  1.00
```

4. Mass Spectrometry Data

HRMS of **1a**

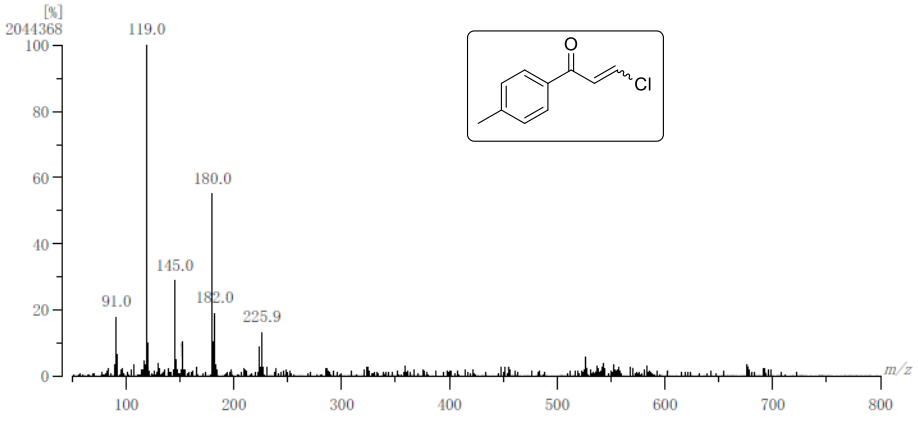
[Mass Spectrum]
Data : 20231128_HREI_2a002 Date : 28-Nov-2023 15:11
RT : 0.00 min Scan# : 1
Elements : C 10/0, H 10/0, 35Cl 1/0, 37Cl 1/0, O 1/0
Mass Tolerance : 100ppm, 5mmu if m/z < 50, 50mmu if m/z > 500
Unsaturation (U.S.) : -0.5 - 100.0



	Observed m/z	Int%							
	166.0181	43.36							
	Estimated m/z	Err[ppm / mmu]	U.S.	C	H	35Cl	37Cl	O	
1	166.0130	+30.7 / +5.1	2.0	7	10	1	1	-	
2	166.0185	-2.7 / -0.4	6.0	9	7	1	-	1	

HRMS of **1b**

[Mass Spectrum]
Data : 20231220_HREI_Expt-745001 Date : 20-Dec-2023 16:15
RT : 0.00 min Scan# : 1
Elements : C 10/0, H 10/0, 35Cl 1/0, 37Cl 1/0, O 1/0
Mass Tolerance : 100ppm, 5mmu if m/z < 50, 50mmu if m/z > 500
Unsaturation (U.S.) : -0.5 - 100.0

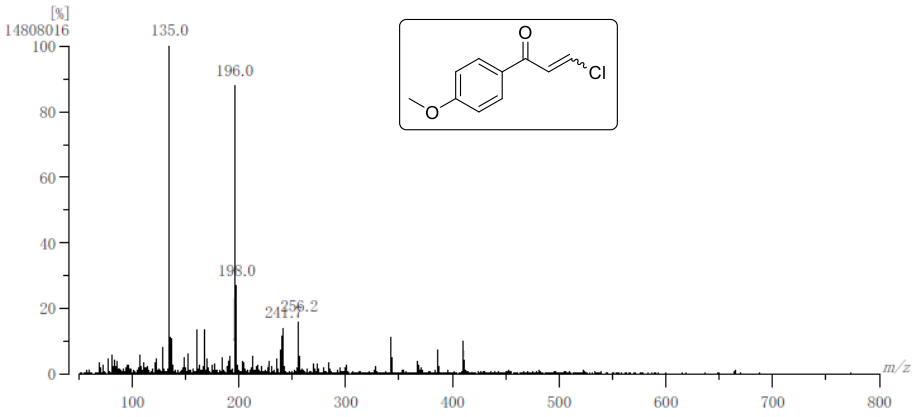


	Observed m/z	Int%							
	180.0338	55.02							
	Estimated m/z	Err[ppm / mmu]	U.S.	C	H	35Cl	37Cl	O	
1	180.0342	-2.2 / -0.4	6.0	10	9	1	-	1	

	Observed m/z	Int%							
	182.0325	18.99							
	Estimated m/z	Err[ppm / mmu]	U.S.	C	H	35Cl	37Cl	O	
2	182.0312	+6.9 / +1.3	6.0	10	9	-	1	1	

HRMS of **1c**

[Mass Spectrum]
Data : 20231220_HREI_Expt-735002 Date : 20-Dec-2023 11:52
RT : 0.15 min Scan# : 2
Elements : C 10/0, H 10/0, 35Cl 1/0, 37Cl 1/0, O 2/0
Mass Tolerance : 100ppm, 5mmu if m/z < 50, 50mmu if m/z > 500
Unsaturation (U.S.) : -0.5 - 100.0



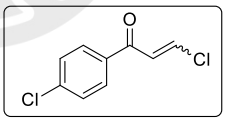
	Observed m/z	Int%							
	196.0284	87.93							
	Estimated m/z	Err[ppm / mmu]	U.S.	C	H	35Cl	37Cl		O
1	196.0291	-3.6 / -0.7	6.0	10	9	1	-		2
2	196.0105	+91.3 / +17.9	7.0	10	7	-	1		2

	Observed m/z	Int%							
	198.0277	27.00							
	Estimated m/z	Err[ppm / mmu]	U.S.	C	H	35Cl	37Cl		O
3	198.0262	+7.8 / +1.5	6.0	10	9	-	1		2

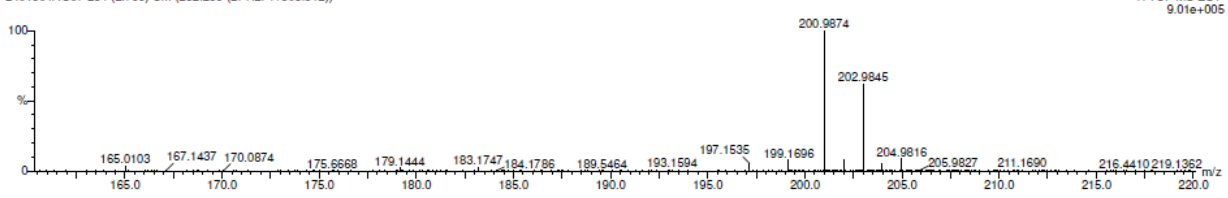
HRMS of **1d**

Elemental Composition Report Page 1

Single Mass Analysis
Tolerance = 10.0 mDa / DBE: min = -1.5, max = 50.0
Element prediction: Off
Number of isotope peaks used for i-FIT = 3



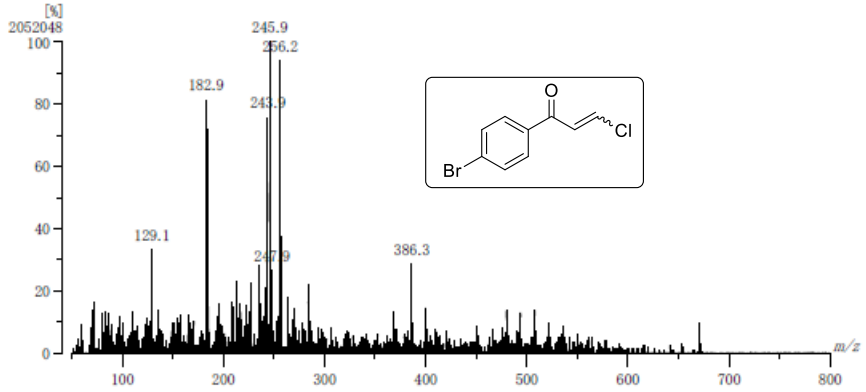
Monoisotopic Mass, Even Electron Ions
35 formula(e) evaluated with 3 results within limits (up to 25 closest results for each mass)
Elements Used:
C: 1-100 H: 1-100 O: 1-10 Cl: 1-3
Expt-752
240130YA007 284 (2.769) Cm (282:286-(271:274+308:312))



Minimum:	80.00				-1.5
Maximum:	100.00		10.0	500.0	50.0

Mass	RA	Calc. Mass	mDa	PPM	DBE	Formula
200.9874	100.00	200.9874	0.0	0.0	5.5	C9 H7 O C12
		200.9802	7.2	35.8	1.5	C4 H6 O7 C1
		200.9955	-8.1	-40.3	5.5	C8 H6 O4 C1

HRMS of **1e**



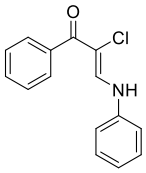
	Observed m/z	Int%									
	243.9287	75.76									
	Estimated m/z	Err[ppm / mmu]	U.S.	C	H	79Br	81Br	35Cl	37Cl	O	
1	243.9477	-78.1 / -19.0	6.0	10	8	-	1	1	-	-	
2	243.9468	-74.4 / -18.1	6.0	10	8	1	-	-	1	-	
3	243.9291	-1.8 / -0.4	7.0	10	6	-	1	-	1	-	
4	243.9235	+21.2 / +5.2	2.0	7	9	1	-	1	1	-	
5	243.9058	+93.8 / +22.9	3.0	7	7	-	1	1	1	-	
6	243.9291	-1.4 / -0.4	6.0	9	6	1	-	1	-	1	
7	243.9114	+71.1 / +17.3	7.0	9	4	-	1	1	-	1	
8	243.9105	+74.8 / +18.2	7.0	9	4	1	-	-	1	1	

	Observed m/z	Int%									
	245.9276	100.00									
	Estimated m/z	Err[ppm / mmu]	U.S.	C	H	79Br	81Br	35Cl	37Cl	O	
9	245.9448	-69.9 / -17.2	6.0	10	8	-	1	-	1	-	
10	245.9215	+24.9 / +6.1	2.0	7	9	-	1	1	1	-	
11	245.9078	+80.5 / +19.8	0.0	5	10	1	1	-	-	1	
12	245.9447	-69.5 / -17.1	5.0	9	8	1	-	1	-	1	
13	245.9270	+2.4 / +0.6	6.0	9	6	-	1	1	-	1	
14	245.9261	+6.1 / +1.5	6.0	9	6	1	-	-	1	1	
15	245.9084	+78.0 / +19.2	7.0	9	4	-	1	-	1	1	

	Observed m/z	Int%									
	247.9265	26.44									
	Estimated m/z	Err[ppm / mmu]	U.S.	C	H	79Br	81Br	35Cl	37Cl	O	
16	247.9427	-65.2 / -16.2	5.0	9	8	-	1	1	-	1	
17	247.9418	-61.5 / -15.3	5.0	9	8	1	-	-	1	1	
18	247.9241	+9.9 / +2.4	6.0	9	6	-	1	-	1	1	
19	247.9184	+32.5 / +8.1	1.0	6	9	1	-	1	1	1	

HRMS of **3aa**

Elemental Composition Report Page 1

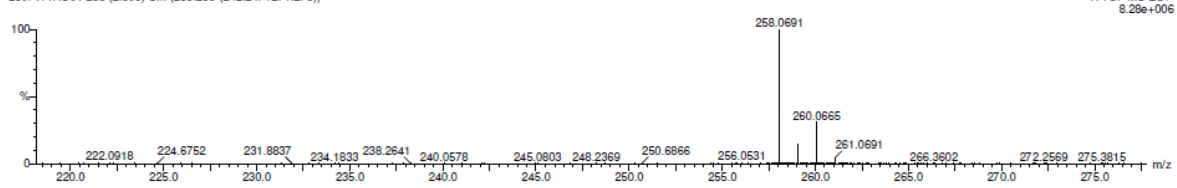


Single Mass Analysis
Tolerance = 5.0 mDa / DBE: min = -1.5, max = 50.0
Element prediction: Off
Number of isotope peaks used for i-FIT = 3

Monoisotopic Mass, Even Electron Ions
326 formula(e) evaluated with 4 results within limits (up to 20 closest results for each mass)
Elements Used:
C: 1-100 H: 1-100 N: 1-10 O: 1-10 Cl: 1-3

1
230711YAO01 256 (2.509) Cm (255:258-(242:247+271:275))

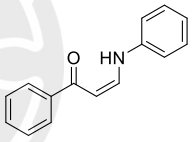
1: TOF MS ES+
8.28e+006



Minimum:			-1.5		
Maximum:		5.0	500.0	50.0	
Mass	Calc. Mass	mDa	PPM	DBE	Formula
258.0691	258.0686	0.5	1.9	9.5	C15 H13 N O Cl
	258.0664	2.7	10.5	0.5	C9 H18 N O3 Cl2
	258.0718	-2.7	-10.5	1.5	C4 H13 N7 O4 Cl
	258.0645	4.6	17.8	5.5	C10 H13 N3 O3 Cl

HRMS of **4aa**

Elemental Composition Report Page 1

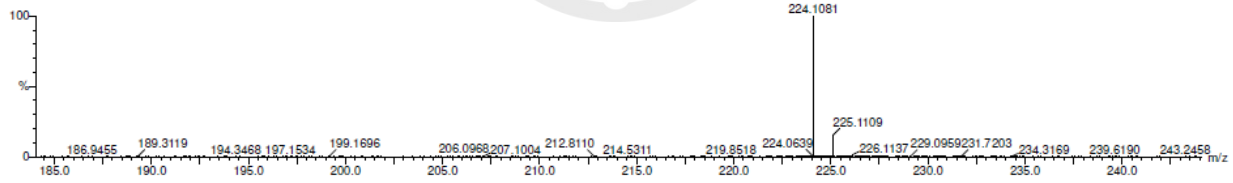


Single Mass Analysis
Tolerance = 5.0 mDa / DBE: min = -1.5, max = 50.0
Element prediction: Off
Number of isotope peaks used for i-FIT = 3

Monoisotopic Mass, Even Electron Ions
165 formula(e) evaluated with 3 results within limits (up to 20 closest results for each mass)
Elements Used:
C: 1-100 H: 1-100 N: 1-10 O: 1-10

5
230711YAO05 281 (2.743) Cm (277:285-(254:262+301:306))

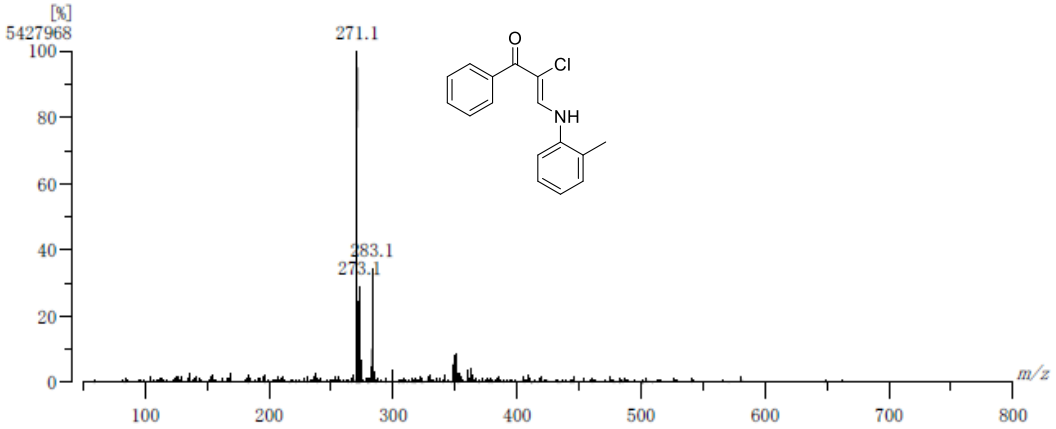
1: TOF MS ES+
4.27e+007



Minimum:			-1.5		
Maximum:		5.0	500.0	50.0	
Mass	Calc. Mass	mDa	PPM	DBE	Formula
224.1081	224.1075	0.6	2.7	9.5	C15 H14 N O
	224.1107	-2.6	-11.6	1.5	C4 H14 N7 O4
	224.1035	4.6	20.5	5.5	C10 H14 N3 O3

HRMS of **3ab**

[Mass Spectrum]
Data : 20231128_HREI_3ab005 Date : 28-Nov-2023 14:44
RT : 0.15 min Scan# : 2
Elements : C 20/0, H 20/0, 35Cl 1/0, 37Cl 1/0, N 1/0, O 1/0
Mass Tolerance : 100ppm, 5mmu if m/z < 50, 50mmu if m/z > 500
Unsaturation (U.S.) : -0.5 - 100.0

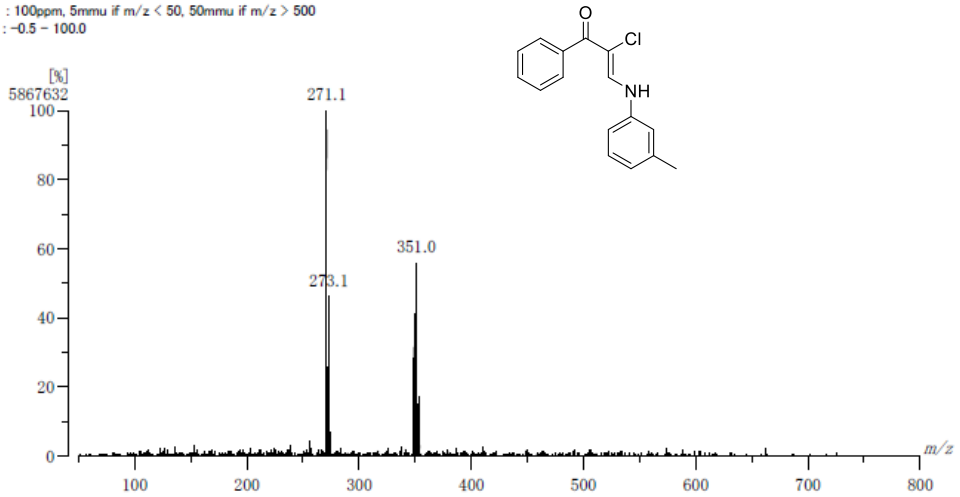


	Observed m/z	Int%								
	271.0758	100.00								
	Estimated m/z	Err[ppm / mmu]	U.S.	C	H	35Cl	37Cl	N	O	
1	271.0834	-28.2 / -7.6	5.5	15	19	1	1	-	-	
2	271.0942	-67.8 / -18.4	10.0	17	16	-	1	1	-	
3	271.0709	+18.2 / +4.9	6.0	14	17	1	1	1	-	
4	271.0890	-48.6 / -13.2	9.5	17	16	1	-	-	1	
5	271.0704	+20.0 / +5.4	10.5	17	14	-	1	-	1	
6	271.0997	-88.2 / -23.9	14.0	19	13	-	-	1	1	
7	271.0764	-2.2 / -0.6	10.0	16	14	1	-	1	1	
8	271.0578	+66.4 / +18.0	11.0	16	12	-	1	1	1	

	Observed m/z	Int%								
	273.0747	29.06								
	Estimated m/z	Err[ppm / mmu]	U.S.	C	H	35Cl	37Cl	N	O	
9	273.0865	-43.2 / -11.8	5.0	14	19	1	1	1	-	
10	273.0860	-41.4 / -11.3	9.5	17	16	-	1	-	1	
11	273.0627	+44.0 / +12.0	5.5	14	17	1	1	-	1	
12	273.0920	-63.5 / -17.3	9.0	16	16	1	-	1	1	
13	273.0734	+4.6 / +1.3	10.0	16	14	-	1	1	1	
14	273.0501	+90.0 / +24.6	6.0	13	15	1	1	1	1	

HRMS of **3ac**

[Mass Spectrum]
Data : 20231128_HREI_3ac002 Date : 28-Nov-2023 14:24
RT : 0.30 min Scan# : 3
Elements : C 20/0, H 20/0, 35Cl 1/0, 37Cl 1/0, N 1/0, O 1/0
Mass Tolerance : 100ppm, 5mmu if m/z < 50, 50mmu if m/z > 500
Unsaturation (U.S.) : -0.5 - 100.0

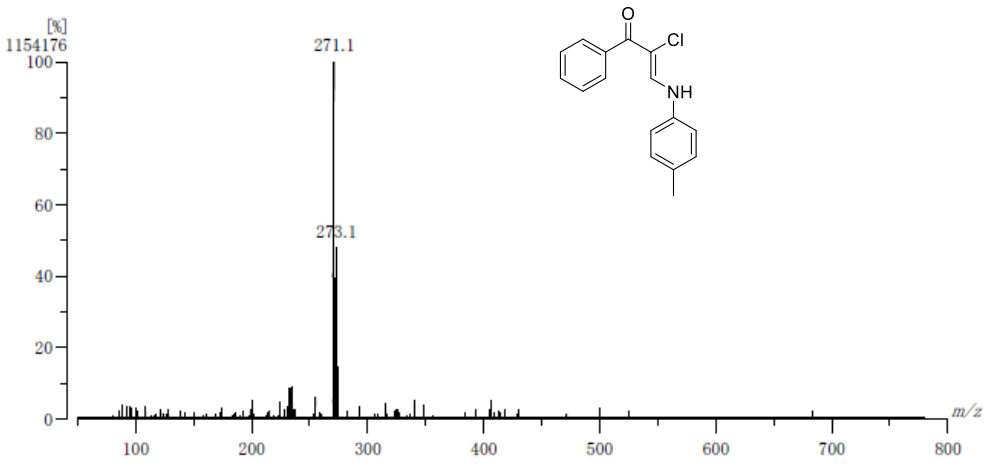


	Observed m/z	Int%							
	271.0780	100.00							
	Estimated m/z	Err[ppm / mmu]	U.S.	C	H	35Cl	37Cl	N	O
1	271.0834	-20.0 / -5.4	5.5	15	19	1	1	-	-
2	271.0942	-59.7 / -16.2	10.0	17	16	-	1	1	-
3	271.0709	+26.4 / +7.1	6.0	14	17	1	1	1	-
4	271.0890	-40.5 / -11.0	9.5	17	16	1	-	-	1
5	271.0704	+28.2 / +7.6	10.5	17	14	-	1	-	1
6	271.0997	-80.1 / -21.7	14.0	19	13	-	-	1	1
7	271.0764	+5.9 / +1.6	10.0	16	14	1	-	1	1
8	271.0578	+74.5 / +20.2	11.0	16	12	-	1	1	1

	Observed m/z	Int%							
	273.0772	46.12							
	Estimated m/z	Err[ppm / mmu]	U.S.	C	H	35Cl	37Cl	N	O
9	273.0865	-34.1 / -9.3	5.0	14	19	1	1	1	-
10	273.0860	-32.3 / -8.8	9.5	17	16	-	1	-	1
11	273.0627	+53.1 / +14.5	5.5	14	17	1	1	-	1
12	273.0920	-54.4 / -14.8	9.0	16	16	1	-	1	1
13	273.0734	+13.8 / +3.8	10.0	16	14	-	1	1	1
14	273.0501	+99.2 / +27.1	6.0	13	15	1	1	1	1

HRMS of **3ad**

[Mass Spectrum]
Data : 20231128_HREI_3ad001 Date : 28-Nov-2023 14:54
RT : 0.45 min Scan# : 4
Elements : C 20/0, H 20/0, 35Cl 1/0, 37Cl 1/0, N 1/0, O 1/0
Mass Tolerance : 100ppm, 5mmu if m/z < 50, 50mmu if m/z > 500
Unsaturation (U.S.) : -0.5 - 100.0

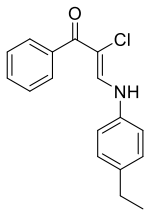


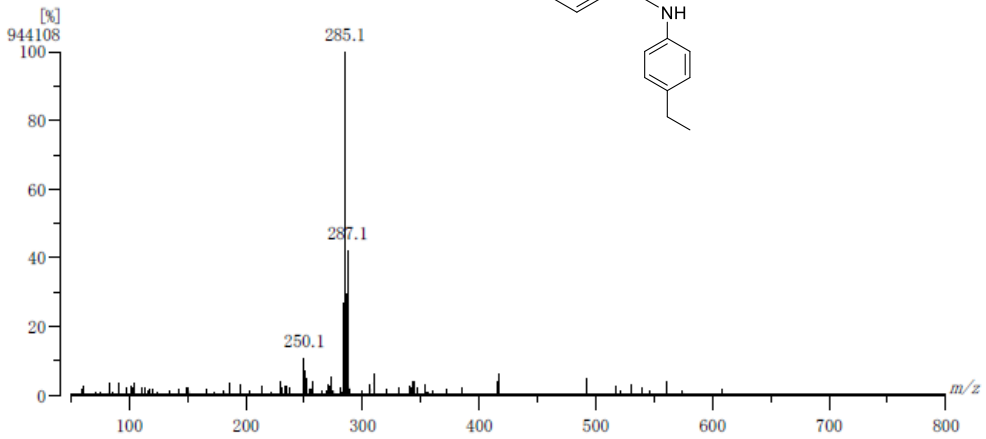
	Observed m/z	Int%								
	271.0755	100.00								
	Estimated m/z	Err[ppm / mmu]	U.S.	C	H	35Cl	37Cl	N	O
1	271.0834	-29.3 / -7.9	5.5	15	19	1	1	-	-
2	271.0942	-68.9 / -18.7	10.0	17	16	-	1	1	-
3	271.0709	+17.1 / +4.6	6.0	14	17	1	1	1	-
4	271.0890	-49.7 / -13.5	9.5	17	16	1	-	-	1
5	271.0704	+18.9 / +5.1	10.5	17	14	-	1	-	1
6	271.0997	-89.3 / -24.2	14.0	19	13	-	-	1	1
7	271.0764	-3.3 / -0.9	10.0	16	14	1	-	1	1
8	271.0578	+65.3 / +17.7	11.0	16	12	-	1	1	1

	Observed m/z	Int%								
	273.0739	47.75								
	Estimated m/z	Err[ppm / mmu]	U.S.	C	H	35Cl	37Cl	N	O
9	273.0471	+98.1 / +26.8	14.5	19	10	1	-	-	-
10	273.0865	-46.2 / -12.6	5.0	14	19	1	1	1	-
11	273.0860	-44.4 / -12.1	9.5	17	16	-	1	-	1
12	273.0627	+41.0 / +11.2	5.5	14	17	1	1	-	1
13	273.0920	-66.4 / -18.1	9.0	16	16	1	-	1	1
14	273.0734	+1.7 / +0.5	10.0	16	14	-	1	1	1
15	273.0501	+87.1 / +23.8	6.0	13	15	1	1	1	1

HRMS of **3ae**

[Mass Spectrum]
Data : 20231128_HREI_3ae001 Date : 28-Nov-2023 15:24
RT : 0.60 min Scan# : 5
Elements : C 20/0, H 20/0, 35Cl 1/0, 37Cl 1/0, N 1/0, O 1/0
Mass Tolerance : 100ppm, 5mmu if m/z < 50, 50mmu if m/z > 500
Unsaturation (U.S.) : -0.5 - 100.0



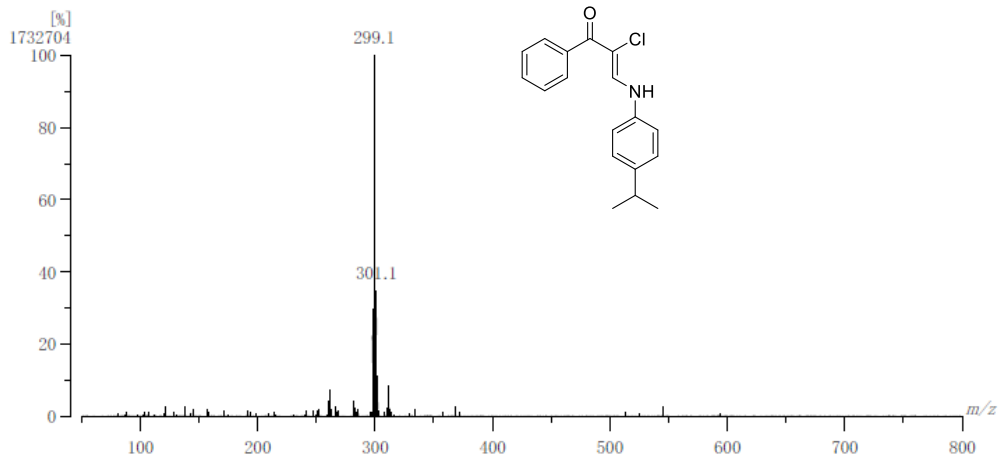


	Observed m/z	Int%								
	285.0913	100.00								
	Estimated m/z	Err[ppm / mmu]	U.S.	C	H	35Cl	37Cl	N	O
1	285.1098	-65.0 / -18.5	10.0	18	18	-	1	1	-
2	285.0865	+16.8 / +4.8	6.0	15	19	1	1	1	-
3	285.1046	-46.7 / -13.3	9.5	18	18	1	-	-	1
4	285.0860	+18.5 / +5.3	10.5	18	16	-	1	-	1
5	285.1154	-84.4 / -24.1	14.0	20	15	-	-	1	1
6	285.0920	-2.6 / -0.7	10.0	17	16	1	-	1	1
7	285.0734	+62.6 / +17.9	11.0	17	14	-	1	1	1

	Observed m/z	Int%							
	287.0902	42.19							
	Estimated m/z	Err[ppm / mmu]	U.S.	C	H	35Cl	37Cl	N	O
8	287.0628	+95.6 / +27.4	14.5	20	12	1	-	-	-
9	287.1017	-39.9 / -11.5	9.5	18	18	-	1	-	1
10	287.0783	+41.3 / +11.9	5.5	15	19	1	1	-	1
11	287.1077	-60.9 / -17.5	9.0	17	18	1	-	1	1
12	287.0891	+3.9 / +1.1	10.0	17	16	-	1	1	1
13	287.0658	+85.1 / +24.4	6.0	14	17	1	1	1	1

HRMS of **3af**

[Mass Spectrum]
Data : 20231128_HREI_3af001 Date : 28-Nov-2023 15:34
RT : 0.45 min Scan# : 4
Elements : C 20/0, H 20/0, 35Cl 1/0, 37Cl 1/0, N 1/0, O 1/0
Mass Tolerance : 100ppm, 5mmu if m/z < 50, 50mmu if m/z > 500
Unsaturation (U.S.) : -0.5 - 100.0

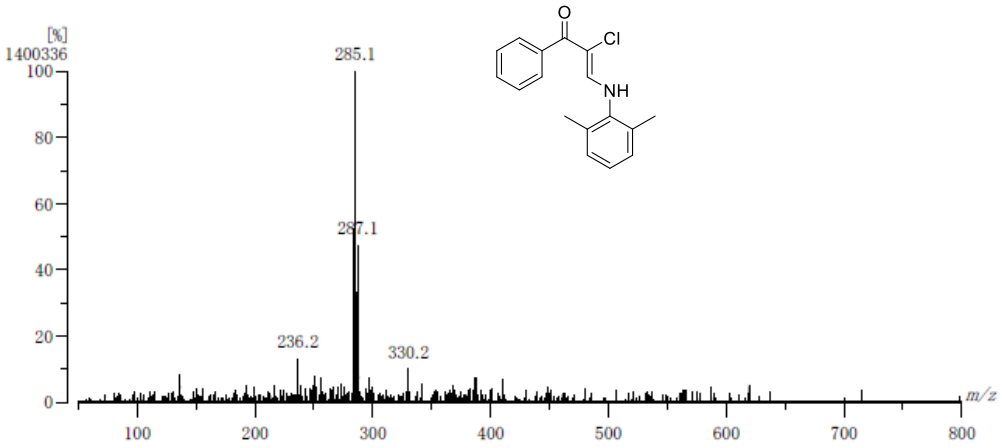


	Observed m/z	Int%							
	299.1074	100.00							
	Estimated m/z	Err[ppm / mmu]	U.S.	C	H	35Cl	37Cl	N	O
1	299.1255	-60.4 / -18.1	10.0	19	20	-	1	1	-
2	299.1203	-43.0 / -12.9	9.5	19	20	1	-	-	1
3	299.1017	+19.2 / +5.7	10.5	19	18	-	1	-	1
4	299.0783	+97.1 / +29.1	6.5	16	19	1	1	-	1
5	299.1077	-1.0 / -0.3	10.0	18	18	1	-	1	1
6	299.0891	+61.2 / +18.3	11.0	18	16	-	1	1	1

	Observed m/z	Int%							
	301.1058	34.80							
	Estimated m/z	Err[ppm / mmu]	U.S.	C	H	35Cl	37Cl	N	O
7	301.1173	-38.3 / -11.5	9.5	19	20	-	1	-	1
8	301.1233	-58.3 / -17.5	9.0	18	20	1	-	1	1
9	301.1047	+3.5 / +1.1	10.0	18	18	-	1	1	1
10	301.0814	+81.0 / +24.4	6.0	15	19	1	1	1	1

HRMS of **3ag**

[Mass Spectrum]
Data : 20231128_HREI_3ag001 Date : 28-Nov-2023 15:42
RT : 0.75 min Scan# : 6
Elements : C 20/0, H 20/0, 35Cl 1/0, 37Cl 1/0, N 1/0, O 1/0
Mass Tolerance : 100ppm, 5mmu if m/z < 50, 50mmu if m/z > 500
Unsaturation (U.S.) : -0.5 - 100.0



Observed m/z Int%
285.0910 100.00

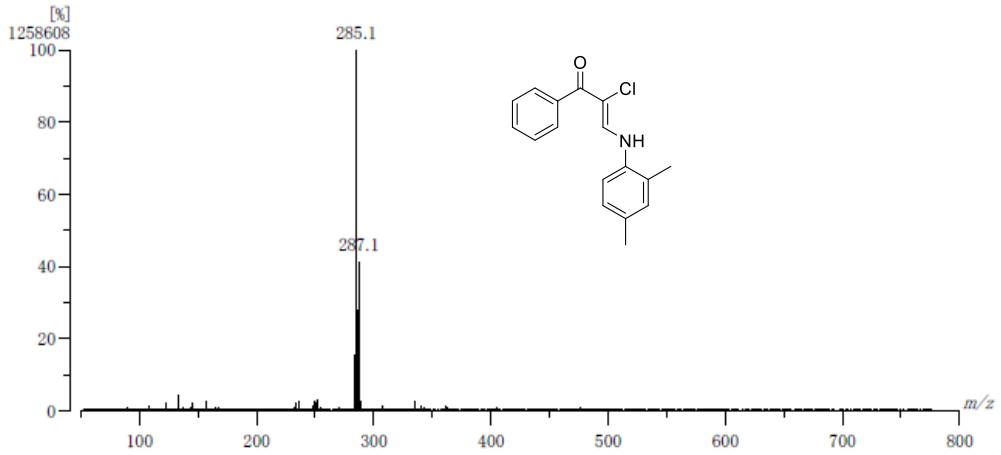
	Estimated m/z	Err[ppm / mmu]	U.S.	C	H	35Cl	37Cl	N	O
1	285.1098	-66.0 / -18.8	10.0	18	18	-	1	1	-
2	285.0865	+15.8 / +4.5	6.0	15	19	1	1	1	-
3	285.1046	-47.8 / -13.6	9.5	18	18	1	-	-	1
4	285.0860	+17.5 / +5.0	10.5	18	16	-	1	-	1
5	285.0627	+99.3 / +28.3	6.5	15	17	1	1	-	1
6	285.1154	-85.5 / -24.4	14.0	20	15	-	-	1	1
7	285.0920	-3.7 / -1.0	10.0	17	16	1	-	1	1
8	285.0734	+61.6 / +17.6	11.0	17	14	-	1	1	1

Observed m/z Int%
287.0939 47.22

	Estimated m/z	Err[ppm / mmu]	U.S.	C	H	35Cl	37Cl	N	O
9	287.1203	-91.8 / -26.4	8.5	18	20	1	-	-	1
10	287.1017	-27.1 / -7.8	9.5	18	18	-	1	-	1
11	287.0783	+54.2 / +15.6	5.5	15	19	1	1	-	1
12	287.1077	-48.0 / -13.8	9.0	17	18	1	-	1	1
13	287.0891	+16.7 / +4.8	10.0	17	16	-	1	1	1
14	287.0658	+98.0 / +28.1	6.0	14	17	1	1	1	1

HRMS of **3ah**

[Mass Spectrum]
Data : 20231128_HREI_3ah001 Date : 28-Nov-2023 15:50
RT : 0.30 min Scan# : 3
Elements : C 20/0, H 20/0, 35Cl 1/0, 37Cl 1/0, N 1/0, O 1/0
Mass Tolerance : 100ppm, 5mmu if m/z < 50, 50mmu if m/z > 500
Unsaturation (U.S.) : -0.5 - 100.0



Observed m/z	Int%
285.0910	100.00

	Estimated m/z	Err[ppm / mmu]	U.S.	C	H	35Cl	37Cl	N	O
1	285.1098	-66.0 / -18.8	10.0	18	18	-	1	1	-
2	285.0865	+15.8 / +4.5	6.0	15	19	1	1	1	-
3	285.1046	-47.8 / -13.6	9.5	18	18	1	-	-	1
4	285.0860	+17.5 / +5.0	10.5	18	16	-	1	-	1
5	285.0627	+99.3 / +28.3	6.5	15	17	1	1	-	1
6	285.1154	-85.5 / -24.4	14.0	20	15	-	-	1	1
7	285.0920	-3.7 / -1.0	10.0	17	16	1	-	1	1
8	285.0734	+61.6 / +17.6	11.0	17	14	-	1	1	1

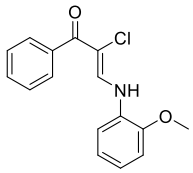
Observed m/z	Int%
287.0902	41.13

	Estimated m/z	Err[ppm / mmu]	U.S.	C	H	35Cl	37Cl	N	O
9	287.0628	+95.6 / +27.4	14.5	20	12	1	-	-	-
10	287.1017	-39.9 / -11.5	9.5	18	18	-	1	-	1
11	287.0783	+41.3 / +11.9	5.5	15	19	1	1	-	1
12	287.1077	-60.9 / -17.5	9.0	17	18	1	-	1	1
13	287.0891	+3.9 / +1.1	10.0	17	16	-	1	1	1
14	287.0658	+85.1 / +24.4	6.0	14	17	1	1	1	1

HRMS of **3ai**

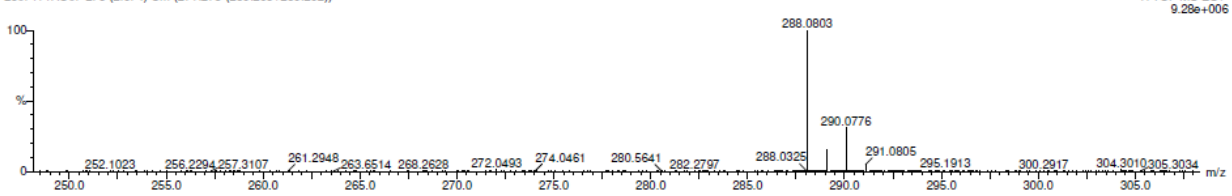
Elemental Composition Report Page 1

Single Mass Analysis
Tolerance = 5.0 mDa / DBE: min = -1.5, max = 50.0
Element prediction: Off
Number of isotope peaks used for i-FIT = 3



Monoisotopic Mass, Even Electron Ions
451 formula(e) evaluated with 4 results within limits (up to 20 closest results for each mass)
Elements Used:
C: 1-100 H: 1-100 N: 1-10 O: 1-10 Cl: 1-3
7
230711YAO07 273 (2.674) Cm (271:275-(259:263+289:292))

1: TOF MS ES+
9.28e+006

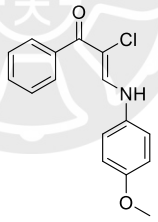


Minimum:			-1.5		
Maximum:		5.0	500.0	50.0	
Mass	Calc. Mass	mDa	PPM	DBE	Formula
288.0803	288.0791	1.2	4.2	9.5	C16 H15 N O2 Cl
	288.0823	-2.0	-6.9	1.5	C5 H15 N7 O5 Cl
	288.0769	3.4	11.8	0.5	C10 H20 N O4 Cl2
	288.0850	-4.7	-16.3	0.5	C9 H19 N O7 Cl

HRMS of **3aj**

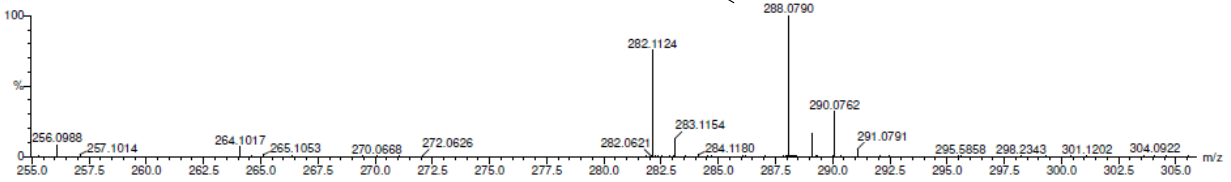
Elemental Composition Report Page 1

Single Mass Analysis
Tolerance = 5.0 mDa / DBE: min = -1.5, max = 50.0
Element prediction: Off
Number of isotope peaks used for i-FIT = 3



Monoisotopic Mass, Even Electron Ions
451 formula(e) evaluated with 5 results within limits (up to 20 closest results for each mass)
Elements Used:
C: 1-100 H: 1-100 N: 1-10 O: 1-10 Cl: 1-3
6
230711YAO06 252 (2.455) Cm (251:253-(237:243+277:284))

1: TOF MS ES+
1.31e+007

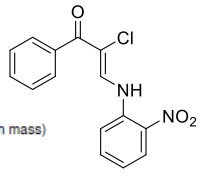


Minimum:			-1.5		
Maximum:		5.0	500.0	50.0	
Mass	Calc. Mass	mDa	PPM	DBE	Formula
288.0790	288.0791	-0.1	-0.3	9.5	C16 H15 N O2 Cl
	288.0769	2.1	7.3	0.5	C10 H20 N O4 Cl2
	288.0823	-3.3	-11.5	1.5	C5 H15 N7 O5 Cl
	288.0751	3.9	13.5	5.5	C11 H15 N3 O4 Cl
	288.0743	4.7	16.3	1.5	C6 H16 N7 O2 Cl2

HRMS of **3ak**

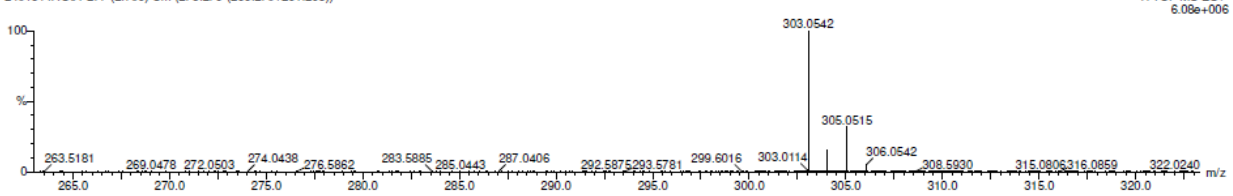
Elemental Composition Report Page 1

Single Mass Analysis
Tolerance = 10.0 mDa / DBE: min = -1.5, max = 50.0
Element prediction: Off
Number of isotope peaks used for i-FIT = 3



Monoisotopic Mass, Even Electron Ions
512 formula(e) evaluated with 14 results within limits (up to 25 closest results for each mass)
Elements Used:
C: 1-100 H: 1-100 N: 1-10 O: 1-10 Cl: 1-3
3ak
240131YAO01 277 (2.709) Cm (275:279-(266:270+291:295))

1: TOF MS ES+
6.08e+006

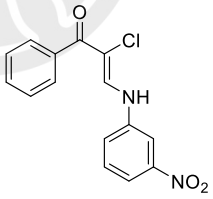


Minimum:	80.00				-1.5	
Maximum:	100.00		10.0	500.0	50.0	
Mass	RA	Calc. Mass	mDa	PPM	DBE	Formula
303.0542	100.00	303.0536	0.6	2.0	10.5	C15 H12 N2 O3 Cl
		303.0515	2.7	8.9	1.5	C9 H17 N2 O5 Cl2
		303.0595	-5.3	-17.5	1.5	C8 H16 N2 O8 Cl
		303.0546	-0.4	-1.3	1.5	C9 H18 N4 O Cl3
		303.0627	-8.5	-28.0	1.5	C8 H17 N4 O4 Cl2
		303.0496	4.6	15.2	6.5	C10 H12 N4 O5 Cl
		303.0609	-6.7	-22.1	6.5	C9 H12 N6 O4 Cl
		303.0456	8.6	28.4	2.5	C5 H12 N6 O7 Cl
		303.0528	1.4	4.6	6.5	C10 H13 N6 O Cl2
		303.0488	5.4	17.8	2.5	C5 H13 N8 O3 Cl2
		303.0568	-2.6	-8.6	2.5	C4 H12 N8 O6 Cl
		303.0510	3.2	10.6	11.5	C11 H8 N8 O Cl
		303.0469	7.3	24.1	7.5	C6 H8 N10 O3 Cl
		303.0600	-5.8	-19.1	2.5	C4 H13 N10 O2 Cl2

HRMS of **3al**

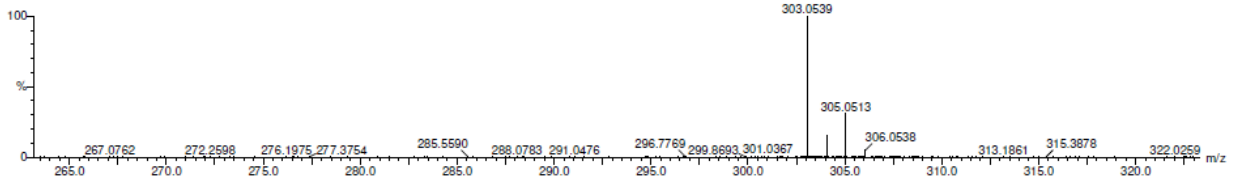
Elemental Composition Report Page 1

Single Mass Analysis
Tolerance = 5.0 mDa / DBE: min = -1.5, max = 50.0
Element prediction: Off
Number of isotope peaks used for i-FIT = 3



Monoisotopic Mass, Even Electron Ions
512 formula(e) evaluated with 7 results within limits (up to 20 closest results for each mass)
Elements Used:
C: 1-100 H: 1-100 N: 1-10 O: 1-10 Cl: 1-3
3
230711YAO03 256 (2.509) Cm (255:259-(245:250+271:275))

1: TOF MS ES+
6.10e+006



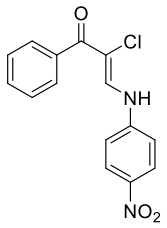
Minimum:				-1.5	
Maximum:		5.0	500.0	50.0	
Mass	Calc. Mass	mDa	PPM	DBE	Formula
303.0539	303.0536	0.3	1.0	10.5	C15 H12 N2 O3 Cl
	303.0546	-0.7	-2.3	1.5	C9 H18 N4 O Cl3
	303.0528	1.1	3.6	6.5	C10 H13 N6 O Cl2
	303.0515	2.4	7.9	1.5	C9 H17 N2 O5 Cl2
	303.0568	-2.9	-9.6	2.5	C4 H12 N8 O6 Cl
	303.0510	2.9	9.6	11.5	C11 H8 N8 O Cl
	303.0496	4.3	14.2	6.5	C10 H12 N4 O5 Cl

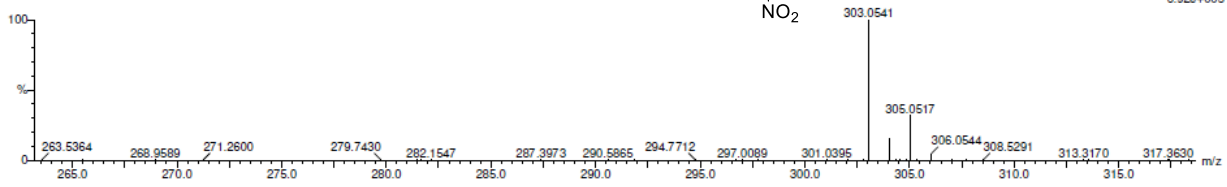
HRMS of **3am**

Elemental Composition Report Page 1

Single Mass Analysis
Tolerance = 10.0 mDa / DBE: min = -1.5, max = 50.0
Element prediction: Off
Number of isotope peaks used for i-FIT = 3

Monoisotopic Mass, Even Electron Ions
512 formula(e) evaluated with 14 results within limits (up to 25 closest results for each mass)
Elements Used:
C: 1-100 H: 1-100 N: 1-10 O: 1-10 Cl: 1-3
3am
240205YAO003 255 (2.500) Cm (254:257-(240:247+272:279))





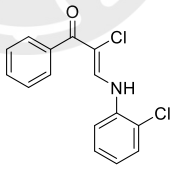
Minimum:	80.00				-1.5	
Maximum:	100.00		10.0	500.0	50.0	
Mass	RA	Calc. Mass	mDa	PPM	DBE	Formula
303.0541	100.00	303.0536	0.5	1.6	10.5	C15 H12 N2 O3 Cl
		303.0546	-0.5	-1.6	1.5	C9 H18 N4 O C13
		303.0528	1.3	4.3	6.5	C10 H13 N6 O C12
		303.0515	2.6	8.6	1.5	C9 H17 N2 O5 C12
		303.0568	-2.7	-8.9	2.5	C4 H12 N8 O6 Cl
		303.0510	3.1	10.2	11.5	C11 H8 N8 O Cl
		303.0496	4.5	14.8	6.5	C10 H12 N4 O5 Cl
		303.0488	5.3	17.5	2.5	C5 H13 N8 O3 C12
		303.0595	-5.4	-17.8	1.5	C8 H16 N2 O8 Cl
		303.0600	-5.9	-19.5	2.5	C4 H13 N10 O2 C12
		303.0609	-6.8	-22.4	6.5	C9 H12 N6 O4 Cl
		303.0469	7.2	23.8	7.5	C6 H8 N10 O3 Cl
		303.0456	8.5	28.0	2.5	C5 H12 N6 O7 Cl
		303.0627	-8.6	-28.4	1.5	C8 H17 N4 O4 C12

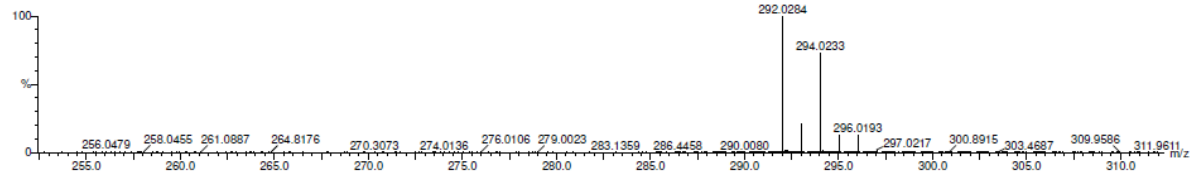
HRMS of **3an**

Elemental Composition Report Page 1

Single Mass Analysis
Tolerance = 5.0 mDa / DBE: min = -1.5, max = 50.0
Element prediction: Off
Number of isotope peaks used for i-FIT = 3

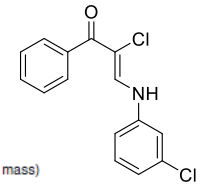
Monoisotopic Mass, Even Electron Ions
469 formula(e) evaluated with 9 results within limits (up to 20 closest results for each mass)
Elements Used:
C: 1-100 H: 1-100 N: 1-10 O: 1-10 Cl: 1-3
10
230711YAO10 289 (2.831) Cm (289:293-(272:279+310:318))





Minimum:					-1.5	
Maximum:			5.0	500.0	50.0	
Mass	Calc. Mass		mDa	PPM	DBE	Formula
292.0284	292.0296		-1.2	-4.1	9.5	C15 H12 N O C12
	292.0278		0.6	2.1	14.5	C16 H7 N3 O Cl
	292.0247		3.7	12.7	1.5	C5 H13 N7 O C13
	292.0237		4.7	16.1	10.5	C11 H7 N5 O3 Cl
	292.0256		2.8	9.6	5.5	C10 H12 N3 O3 C12
	292.0274		1.0	3.4	0.5	C9 H17 N O3 C13
	292.0310		-2.6	-8.9	6.5	C5 H7 N9 O4 Cl
	292.0328		-4.4	-15.1	1.5	C4 H12 N7 O4 C12
	292.0296		-1.2	-4.1	1.5	C4 H11 N5 O8 Cl

HRMS of **3ao**

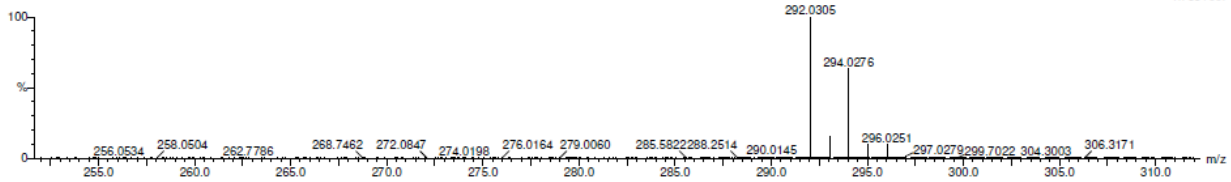


Elemental Composition Report Page 1

Single Mass Analysis
Tolerance = 10.0 mDa / DBE: min = -1.5, max = 50.0
Element prediction: Off
Number of isotope peaks used for i-FIT = 3

Monoisotopic Mass, Even Electron Ions
469 formula(e) evaluated with 18 results within limits (up to 25 closest results for each mass)
Elements Used:
C: 1-100 H: 1-100 N: 1-10 O: 1-10 Cl: 1-3
Expt-723
240130YAO01 278 (2.717) Cm (276:280-(265:270+292:297))

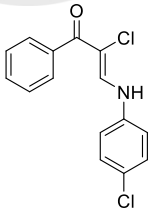
1: TOF MS ES+
1.75e+007



Minimum:	80.00			-1.5	
Maximum:	100.00		10.0	500.0	50.0

Mass	RA	Calc. Mass	mDa	PPM	DBE	Formula
292.0305	100.00	292.0296	0.9	3.1	9.5	C15 H12 N O C12
		292.0274	3.1	10.6	0.5	C9 H17 N O3 C13
		292.0224	8.1	27.7	5.5	C10 H11 N O7 C1
		292.0355	-5.0	-17.1	0.5	C8 H16 N O6 C12
		292.0377	-7.2	-24.7	9.5	C14 H11 N O4 C1
		292.0256	4.9	16.8	5.5	C10 H12 N3 O3 C12
		292.0386	-8.1	-27.7	0.5	C8 H17 N3 O2 C13
		292.0278	2.7	9.2	14.5	C16 H7 N3 O C1
		292.0336	-3.1	-10.6	5.5	C9 H11 N3 O6 C1
		292.0296	0.9	3.1	1.5	C4 H11 N5 O8 C1
		292.0368	-6.3	-21.6	5.5	C9 H12 N5 O2 C12
		292.0237	6.8	23.3	10.5	C11 H7 N5 O3 C1
		292.0215	9.0	30.8	1.5	C5 H12 N5 O5 C12
		292.0247	5.8	19.9	1.5	C5 H13 N7 O C13
		292.0350	-4.5	-15.4	10.5	C10 H7 N7 O2 C1
		292.0328	-2.3	-7.9	1.5	C4 H12 N7 O4 C12
		292.0229	7.6	26.0	6.5	C6 H8 N9 O C12
		292.0310	-0.5	-1.7	6.5	C5 H7 N9 O4 C1

HRMS of **3ap**

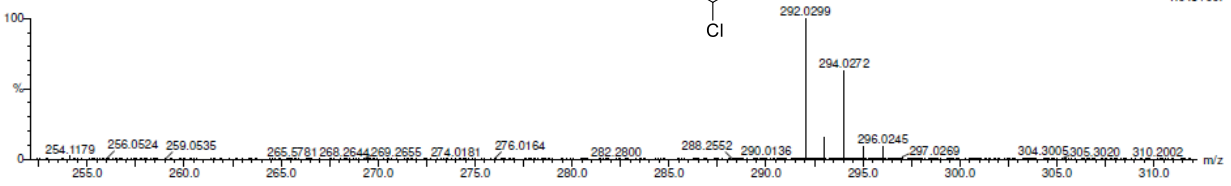


Elemental Composition Report Page 1

Single Mass Analysis
Tolerance = 5.0 mDa / DBE: min = -1.5, max = 50.0
Element prediction: Off
Number of isotope peaks used for i-FIT = 3

Monoisotopic Mass, Even Electron Ions
469 formula(e) evaluated with 8 results within limits (up to 20 closest results for each mass)
Elements Used:
C: 1-100 H: 1-100 N: 1-10 O: 1-10 Cl: 1-3
4
230711YAO04 277 (2.709) Cm (275:279-(266:269+289:292))

1: TOF MS ES+
1.04e+007



Minimum:			-1.5	
Maximum:		5.0	500.0	50.0

Mass	Calc. Mass	mDa	PPM	DBE	Formula
292.0299	292.0296	0.3	1.0	9.5	C15 H12 N O C12
	292.0296	0.3	1.0	1.5	C4 H11 N5 O8 C1
	292.0310	-1.1	-3.8	6.5	C5 H7 N9 O4 C1
	292.0278	2.1	7.2	14.5	C16 H7 N3 O C1
	292.0274	2.5	8.6	0.5	C9 H17 N O3 C13
	292.0328	-2.9	-9.9	1.5	C4 H12 N7 O4 C12
	292.0336	-3.7	-12.7	5.5	C9 H11 N3 O6 C1
	292.0256	4.3	14.7	5.5	C10 H12 N3 O3 C12

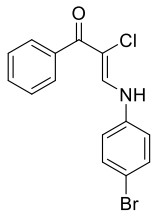
HRMS of **3aq**

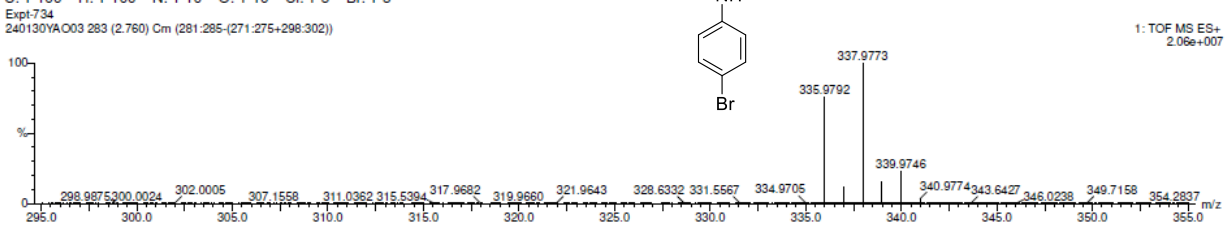
Elemental Composition Report Page 1

Single Mass Analysis
Tolerance = 10.0 mDa / DBE: min = -1.5, max = 50.0
Element prediction: Off
Number of isotope peaks used for i-FIT = 3

Monoisotopic Mass, Even Electron Ions
386 formula(e) evaluated with 10 results within limits (up to 25 closest results for each mass)
Elements Used:
C: 1-100 H: 1-100 N: 1-10 O: 1-10 Cl: 1-3 Br: 1-3

Expt-734
240130YAO03 283 (2.760) Cm (281:285-(271:275+298:302))





Minimum:				-1.5	
Maximum:		10.0	500.0	50.0	

Mass	Calc. Mass	mDa	PPM	DBE	Formula
335.9792	335.9791	0.1	0.3	9.5	C15 H12 N O Cl Br
	335.9769	2.3	6.8	0.5	C9 H17 N O3 Cl2 Br
	335.9823	-3.1	-9.2	1.5	C4 H12 N7 O4 Cl Br
	335.9751	4.1	12.2	5.5	C10 H12 N3 O3 Cl Br
	335.9742	5.0	14.9	1.5	C5 H13 N7 O Cl2 Br
	335.9850	-5.8	-17.3	0.5	C8 H16 N O6 Cl Br
	335.9724	6.8	20.2	6.5	C6 H8 N9 O Cl Br
	335.9863	-7.1	-21.1	5.5	C9 H12 N5 O2 Cl Br
	335.9710	8.2	24.4	1.5	C5 H12 N5 O5 Cl Br
	335.9881	-8.9	-26.5	0.5	C8 H17 N3 O2 Cl2 Br

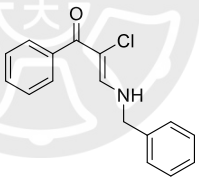
HRMS of **3ar**

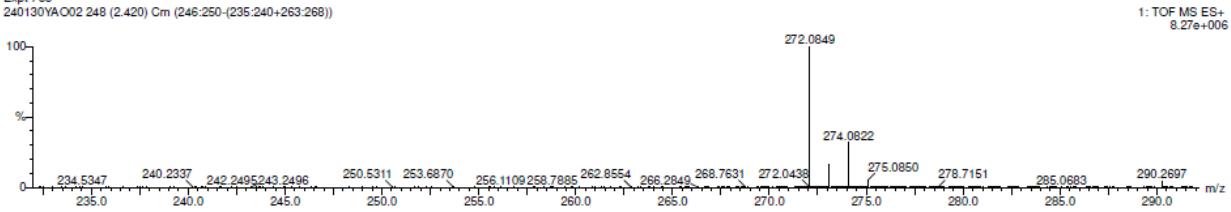
Elemental Composition Report Page 1

Single Mass Analysis
Tolerance = 10.0 mDa / DBE: min = -1.5, max = 50.0
Element prediction: Off
Number of isotope peaks used for i-FIT = 3

Monoisotopic Mass, Even Electron Ions
381 formula(e) evaluated with 10 results within limits (up to 25 closest results for each mass)
Elements Used:
C: 1-100 H: 1-100 N: 1-10 O: 1-10 Cl: 1-3

Expt-733
240130YAO02 248 (2.420) Cm (246:250-(235:240+263:268))



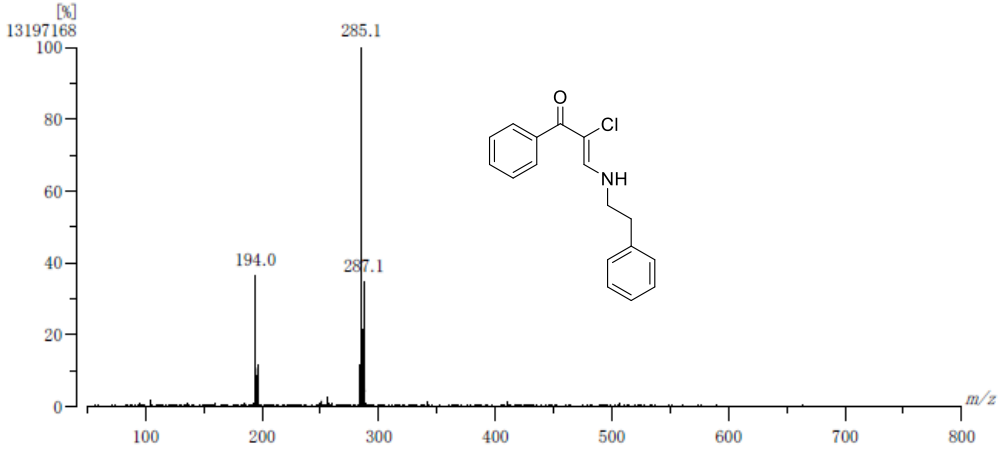


Minimum:	80.00			-1.5	
Maximum:	100.00	10.0	500.0	50.0	

Mass	RA	Calc. Mass	mDa	PPM	DBE	Formula
272.0849	100.00	272.0842	0.7	2.6	9.5	C16 H15 N O Cl
		272.0874	-2.5	-9.2	1.5	C5 H15 N7 O4 Cl
		272.0820	2.9	10.7	0.5	C10 H20 N O3 Cl2
		272.0802	4.7	17.3	5.5	C11 H15 N3 O3 Cl
		272.0901	-5.2	-19.1	0.5	C9 H19 N O6 Cl
		272.0793	5.6	20.6	1.5	C6 H16 N7 O Cl2
		272.0914	-6.5	-23.9	5.5	C10 H15 N5 O2 Cl
		272.0775	7.4	27.2	6.5	C7 H11 N9 O Cl
		272.0933	-8.4	-30.9	0.5	C9 H20 N3 O2 Cl2
		272.0762	8.7	32.0	1.5	C6 H15 N5 O5 Cl

HRMS of **3as**

[Mass Spectrum]
Data : 20231128_HREI_3as001 Date : 28-Nov-2023 15:57
RT : 0.15 min Scan# : 2
Elements : C 20/0, H 20/0, 35Cl 1/0, 37Cl 1/0, N 1/0, O 1/0
Mass Tolerance : 100ppm, 5mmu if m/z < 50, 50mmu if m/z > 500
Unsaturation (U.S.) : -0.5 - 100.0



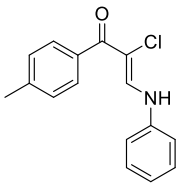
	Observed m/z	Int%								
	285.0912	100.00								
	Estimated m/z	Err[ppm / mmu]	U.S.	C	H	35Cl	37Cl	N	O	
1	285.1098	-65.3 / -18.6	10.0	18	18	-	1	1	-	
2	285.0865	+16.5 / +4.7	6.0	15	19	1	1	1	-	
3	285.1046	-47.1 / -13.4	9.5	18	18	1	-	-	1	
4	285.0860	+18.2 / +5.2	10.5	18	16	-	1	-	1	
5	285.0627	+100.0 / +28.5	6.5	15	17	1	1	-	1	
6	285.1154	-84.8 / -24.2	14.0	20	15	-	-	1	1	
7	285.0920	-3.0 / -0.8	10.0	17	16	1	-	1	1	
8	285.0734	+62.3 / +17.8	11.0	17	14	-	1	1	1	

	Observed m/z	Int%								
	287.0902	34.43								
	Estimated m/z	Err[ppm / mmu]	U.S.	C	H	35Cl	37Cl	N	O	
9	287.0628	+95.6 / +27.4	14.5	20	12	1	-	-	-	
10	287.1017	-39.9 / -11.5	9.5	18	18	-	1	-	1	
11	287.0783	+41.3 / +11.9	5.5	15	19	1	1	-	1	
12	287.1077	-60.9 / -17.5	9.0	17	18	1	-	1	1	
13	287.0891	+3.9 / +1.1	10.0	17	16	-	1	1	1	
14	287.0658	+85.1 / +24.4	6.0	14	17	1	1	1	1	

HRMS of **3ba**

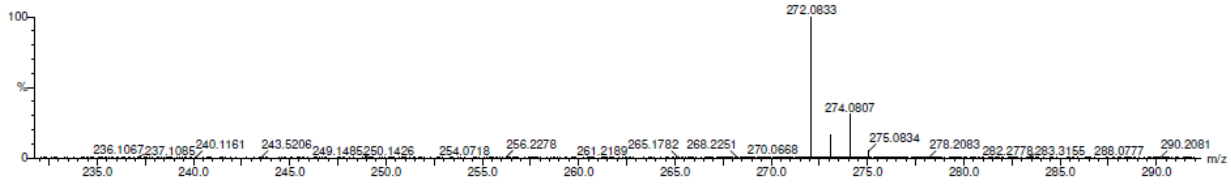
Elemental Composition Report Page 1

Single Mass Analysis
Tolerance = 5.0 mDa / DBE: min = -1.5, max = 50.0
Element prediction: Off
Number of isotope peaks used for i-FIT = 3



Monoisotopic Mass, Even Electron Ions
381 formula(e) evaluated with 5 results within limits (up to 20 closest results for each mass)
Elements Used:
C: 1-100 H: 1-100 N: 1-10 O: 1-10 Cl: 1-3
13
230711YAO13 277 (2.709) Cm (275:279-(254:257+297:299))

1: TOF MS ES+
1.40e+007



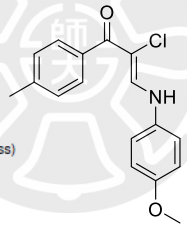
Minimum:				-1.5	
Maximum:		5.0	500.0	50.0	

Mass	Calc. Mass	mDa	PPM	DBE	Formula
272.0833	272.0842	-0.9	-3.3	9.5	C16 H15 N O Cl
	272.0820	1.3	4.8	0.5	C10 H20 N O3 Cl2
	272.0802	3.1	11.4	5.5	C11 H15 N3 O3 Cl
	272.0793	4.0	14.7	1.5	C6 H16 N7 O Cl2
	272.0874	-4.1	-15.1	1.5	C5 H15 N7 O4 Cl

HRMS of **3bj**

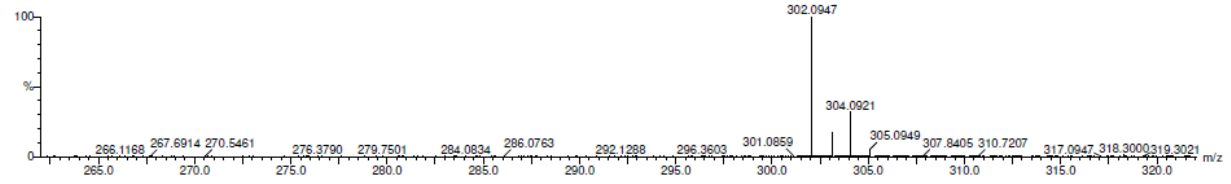
Elemental Composition Report Page 1

Single Mass Analysis
Tolerance = 10.0 mDa / DBE: min = -1.5, max = 50.0
Element prediction: Off
Number of isotope peaks used for i-FIT = 3



Monoisotopic Mass, Even Electron Ions
509 formula(e) evaluated with 11 results within limits (up to 25 closest results for each mass)
Elements Used:
C: 1-100 H: 1-100 N: 1-10 O: 1-10 Cl: 1-3
Expt-748
240130YAO05 268 (2.612) Cm (266:270-(254:259+283:288))

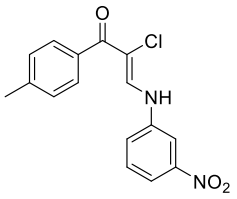
1: TOF MS ES+
5.67e+006



Minimum:	80.00			-1.5	
Maximum:	100.00		10.0	500.0	50.0

Mass	RA	Calc. Mass	mDa	PPM	DBE	Formula
302.0947	100.00	302.0948	-0.1	-0.3	9.5	C17 H17 N O2 Cl
		302.0926	2.1	7.0	0.5	C11 H22 N O4 Cl2
		302.0980	-3.3	-10.9	1.5	C6 H17 N7 O5 Cl
		302.0908	3.9	12.9	5.5	C12 H17 N3 O4 Cl
		302.0899	4.8	15.9	1.5	C7 H18 N7 O2 Cl2
		302.1007	-6.0	-19.9	0.5	C10 H21 N O7 Cl
		302.1011	-6.4	-21.2	1.5	C6 H18 N9 O Cl2
		302.0881	6.6	21.8	6.5	C8 H13 N9 O2 Cl
		302.1020	-7.3	-24.2	5.5	C11 H17 N5 O3 Cl
		302.0867	8.0	26.5	1.5	C7 H17 N5 O6 Cl
		302.1038	-9.1	-30.1	0.5	C10 H22 N3 O3 Cl2

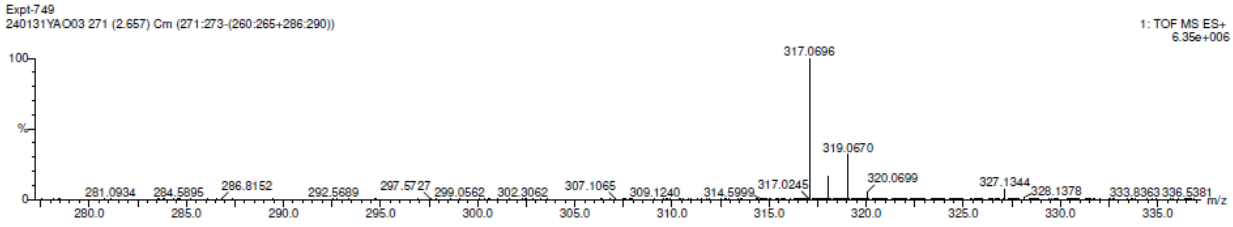
HRMS of **3bl**



Elemental Composition Report Page 1

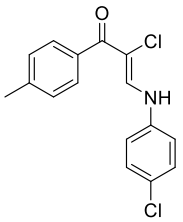
Single Mass Analysis
Tolerance = 10.0 mDa / DBE: min = -1.5, max = 50.0
Element prediction: Off
Number of isotope peaks used for i-FIT = 3

Monoisotopic Mass, Even Electron Ions
571 formula(e) evaluated with 15 results within limits (up to 25 closest results for each mass)
Elements Used:
C: 1-100 H: 1-100 N: 1-10 O: 1-10 Cl: 1-3
Expt-749
240131YAO03 271 (2.657) Cm (271:273-(260:265+286:290))



Minimum:	80.00				-1.5	
Maximum:	100.00		10.0	500.0	50.0	
Mass	RA	Calc. Mass	mDa	PPM	DBE	Formula
317.0696	100.00	317.0693	0.3	0.9	10.5	C16 H14 N2 O3 Cl
		317.0703	-0.7	-2.2	1.5	C10 H20 N4 O C13
		317.0684	1.2	3.8	6.5	C11 H15 N6 O C12
		317.0671	2.5	7.9	1.5	C10 H19 N2 O5 C12
		317.0725	-2.9	-9.1	2.5	C5 H14 N8 O6 Cl
		317.0666	3.0	9.5	11.5	C12 H10 N8 O Cl
		317.0653	4.3	13.6	6.5	C11 H14 N4 O5 Cl
		317.0644	5.2	16.4	2.5	C6 H15 N8 O3 C12
		317.0752	-5.6	-17.7	1.5	C9 H18 N2 O8 Cl
		317.0757	-6.1	-19.2	2.5	C5 H15 N10 O2 C12
		317.0765	-6.9	-21.8	6.5	C10 H14 N6 O4 Cl
		317.0626	7.0	22.1	7.5	C7 H10 N10 O3 Cl
		317.0612	8.4	26.5	2.5	C6 H14 N6 O7 Cl
		317.0783	-8.7	-27.4	1.5	C9 H19 N4 O4 C12
		317.0604	9.2	29.0	-1.5	C H15 N10 O5 C12

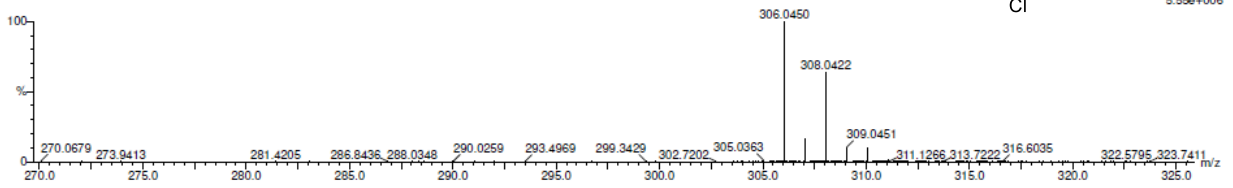
HRMS of **3bp**



Elemental Composition Report Page 1

Single Mass Analysis
Tolerance = 10.0 mDa / DBE: min = -1.5, max = 50.0
Element prediction: Off
Number of isotope peaks used for i-FIT = 3

Monoisotopic Mass, Even Electron Ions
533 formula(e) evaluated with 19 results within limits (up to 25 closest results for each mass)
Elements Used:
C: 1-100 H: 1-100 N: 1-10 O: 1-10 Cl: 1-3
3bp
240205YAO002 289 (2.831) Cm (289:291-(273:281+307:313))



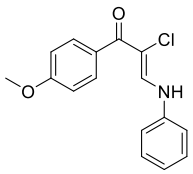
Minimum:	80.00				-1.5	
Maximum:	100.00		10.0	500.0	50.0	
Mass	RA	Calc. Mass	mDa	PPM	DBE	Formula
306.0450	100.00	306.0452	-0.2	-0.7	9.5	C16 H14 N O C12
		306.0453	-0.3	-1.0	1.5	C5 H13 N5 O8 Cl
		306.0434	1.6	5.2	14.5	C17 H9 N3 O Cl
		306.0466	-1.6	-5.2	6.5	C6 H9 N9 O4 Cl
		306.0431	1.9	6.2	0.5	C10 H19 N O3 C13
		306.0484	-3.4	-11.1	1.5	C5 H14 N7 O4 C12
		306.0412	3.8	12.4	5.5	C11 H14 N3 O3 C12
		306.0493	-4.3	-14.1	5.5	C10 H13 N3 O6 Cl
		306.0404	4.6	15.0	1.5	C6 H15 N7 O C13
		306.0506	-5.6	-18.3	10.5	C11 H9 N7 O2 Cl
		306.0394	5.6	18.3	10.5	C12 H9 N5 O3 Cl
		306.0511	-6.1	-19.9	0.5	C9 H18 N O6 C12
		306.0385	6.5	21.2	6.5	C7 H10 N9 O C12
		306.0381	6.9	22.5	5.5	C11 H13 N O7 Cl
		306.0525	-7.5	-24.5	5.5	C10 H14 N5 O2 C12
		306.0372	7.8	25.5	1.5	C6 H14 N5 O5 C12
		306.0533	-8.3	-27.1	9.5	C15 H13 N O4 Cl
		306.0543	-9.3	-30.4	0.5	C9 H19 N3 O2 C13
		306.0354	9.6	31.4	6.5	C7 H9 N7 O5 Cl

HRMS of **3ca**

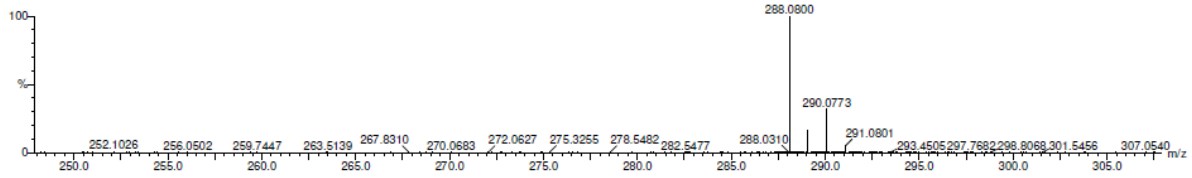
Elemental Composition Report Page 1

Single Mass Analysis
Tolerance = 5.0 mDa / DBE: min = -1.5, max = 50.0
Element prediction: Off
Number of isotope peaks used for i-FIT = 3

Monoisotopic Mass, Even Electron Ions
451 formula(e) evaluated with 4 results within limits (up to 20 closest results for each mass)
Elements Used:
C: 1-100 H: 1-100 N: 1-10 O: 1-10 Cl: 1-3
9
230711YAO09 257 (2.517) Cm (255:259-(239:246+275:280))



1: TOF MS ES+
1.86e+007

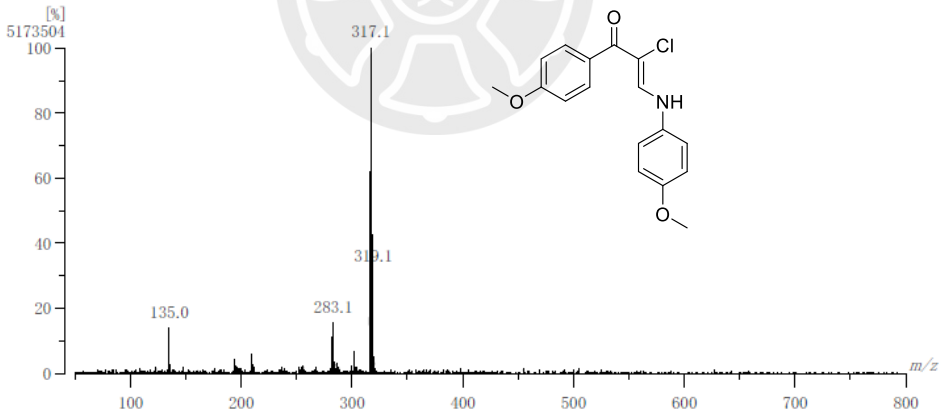


		mDa	PPM	DBE
Minimum:				-1.5
Maximum:		5.0	500.0	50.0

Mass	Calc. Mass	mDa	PPM	DBE	Formula
288.0800	288.0791	0.9	3.1	9.5	C16 H15 N O2 Cl
	288.0823	-2.3	-8.0	1.5	C5 H15 N7 O5 Cl
	288.0769	3.1	10.8	0.5	C10 H20 N O4 Cl2
	288.0751	4.9	17.0	5.5	C11 H15 N3 O4 Cl

HRMS of **3cj**

[Mass Spectrum]
Data : 20231220_HREI_Expt-736001 Date : 20-Dec-2023 11:59
RT : 0.45 min Scan# : 4
Elements : C 20/0, H 20/0, 35Cl 1/0, 37Cl 1/0, N 1/0, O 3/0
Mass Tolerance : 100ppm, 5mmu if m/z < 50, 50mmu if m/z > 500
Unsaturation (U.S.) : -0.5 - 100.0



Observed m/z Int%
317.0812 100.00

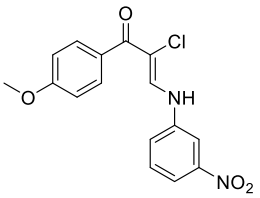
	Estimated m/z	Err[ppm / mmu]	U.S.	C	H	35Cl	37Cl	N	O
1	317.0678	+42.3 / +13.4	10.5	19	17	1	1	-	-
2	317.0552	+82.0 / +26.0	11.0	18	15	1	1	1	-
3	317.0607	+64.5 / +20.5	15.0	20	12	1	-	1	1
4	317.1122	-97.9 / -31.0	9.5	19	20	-	1	-	2
5	317.0997	-58.2 / -18.5	10.0	18	18	-	1	1	2
6	317.0763	+15.3 / +4.9	6.0	15	19	1	1	1	2
7	317.0944	-41.8 / -13.2	9.5	18	18	1	-	-	3
8	317.0758	+16.9 / +5.4	10.5	18	16	-	1	-	3
9	317.0525	+90.4 / +28.7	6.5	15	17	1	1	-	3
10	317.1052	-75.7 / -24.0	14.0	20	15	-	-	1	3
11	317.0819	-2.1 / -0.7	10.0	17	16	1	-	1	3
12	317.0633	+56.5 / +17.9	11.0	17	14	-	1	1	3

HRMS of **3cl**

Elemental Composition Report Page 1

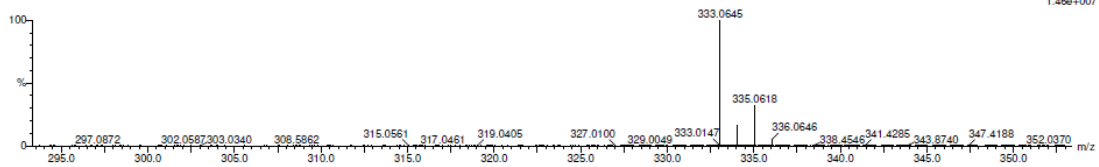
Single Mass Analysis
Tolerance = 10.0 mDa / DBE: min = -1.5, max = 50.0
Element prediction: Off
Number of isotope peaks used for i-FIT = 3

Monoisotopic Mass, Even Electron Ions
643 formula(e) evaluated with 17 results within limits (up to 25 closest results for each mass)
Elements Used:
C: 1-100 H: 1-100 N: 1-10 O: 1-10 Cl: 1-3



Expt-737
240131YA002 259 (2.534) Cm (258:261-(247:252+276:281))

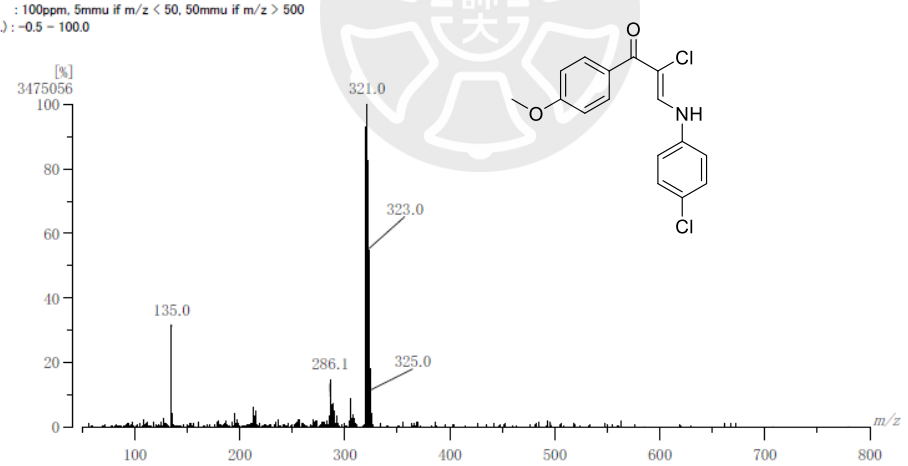
1: TOF MS ES+
1.46e+007



Minimum:	80.00				-1.5	
Maximum:	100.00		10.0	500.0	50.0	
Mass	RA	Calc. Mass	mDa	PPM	DBE	Formula
333.0645	100.00	333.0642	0.3	0.9	10.5	C16 H14 N2 O4 Cl
		333.0652	-0.7	-2.1	1.5	C10 H20 N4 O2 C13
		333.0634	1.1	3.3	6.5	C11 H15 N6 O2 C12
		333.0620	2.5	7.5	1.5	C10 H19 N2 O6 C12
		333.0674	-2.9	-8.7	2.5	C5 H14 N8 O7 Cl
		333.0615	3.0	9.0	11.5	C12 H10 N8 O2 Cl
		333.0602	4.3	12.9	6.5	C11 H14 N4 O6 Cl
		333.0593	5.2	15.6	2.5	C6 H15 N8 O4 C12
		333.0701	-5.6	-16.8	1.5	C9 H18 N2 O9 Cl
		333.0706	-6.1	-18.3	2.5	C5 H15 N10 O3 C12
		333.0714	-6.9	-20.7	6.5	C10 H14 N6 O5 Cl
		333.0575	7.0	21.0	7.5	C7 H10 N10 O4 Cl
		333.0562	8.3	24.9	2.5	C6 H14 N6 O8 Cl
		333.0728	-8.3	-24.9	11.5	C11 H10 N10 O Cl
		333.0561	8.4	25.2	10.5	C17 H15 N2 O C12
		333.0733	-8.8	-26.4	1.5	C9 H19 N4 O5 C12
		333.0553	9.2	27.6	-1.5	C H15 N10 O6 C12

HRMS of **3cp**

[Mass Spectrum]
Data : 20231220_HREI_Expt-739002 Date : 20-Dec-2023 15:01
RT : 0.60 min Scan# : 5
Elements : C 16/0, H 15/0, 35Cl 2/0, 37Cl 2/0, N 1/0, O 2/0
Mass Tolerance : 100ppm, 5mmu if m/z < 50, 50mmu if m/z > 500
Unsaturation (U.S.) : -0.5 - 100.0

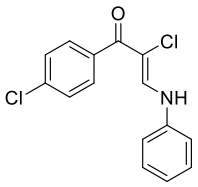


	Observed m/z	Int%							
	321.0318	100.00							
	Estimated m/z	Err[ppm / mmu]	U.S.	C	H	35Cl	37Cl	N	O
1	321.0082	+73.5 / +23.6	7.0	14	14	1	2	1	1
2	321.0030	+89.7 / +28.8	6.5	14	14	2	1	-	2
3	321.0323	-1.7 / -0.5	10.0	16	13	2	-	1	2
4	321.0137	+56.3 / +18.1	11.0	16	11	1	1	1	2

	Observed m/z	Int%							
	322.0260	82.43							
	Estimated m/z	Err[ppm / mmu]	U.S.	C	H	35Cl	37Cl	N	O
5	322.0160	+31.0 / +10.0	6.5	14	15	1	2	1	1
6	322.0108	+47.2 / +15.2	6.0	14	15	2	1	-	2
7	322.0402	-44.0 / -14.2	9.5	16	14	2	-	1	2
8	322.0216	+13.8 / +4.4	10.5	16	12	1	1	1	2
9	321.9982	+86.2 / +27.8	6.5	13	13	2	1	1	2
10	322.0030	+71.6 / +23.0	11.5	16	10	-	2	1	2

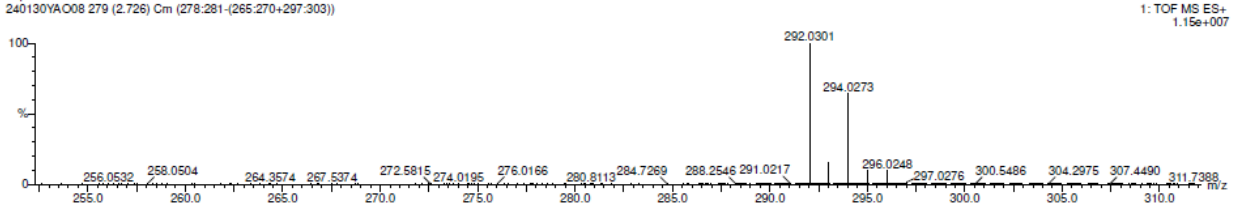
HRMS of **3da**

Elemental Composition Report Page 1



Single Mass Analysis
Tolerance = 10.0 mDa / DBE: min = -1.5, max = 50.0
Element prediction: Off
Number of isotope peaks used for i-FIT = 3

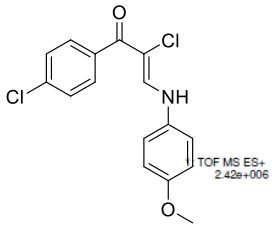
Monoisotopic Mass, Even Electron Ions
469 formula(e) evaluated with 18 results within limits (up to 25 closest results for each mass)
Elements Used:
C: 1-100 H: 1-100 N: 1-10 O: 1-10 Cl: 1-3
Expt-753
240130YAO08 279 (2.726) Cm (278:281-(265:270+297:303))



Minimum:	80.00				-1.5	
Maximum:	100.00		10.0	500.0	50.0	
Mass	RA	Calc. Mass	mDa	PPM	DBE	Formula
292.0301	100.00	292.0296	0.5	1.7	9.5	C15 H12 N O C12
		292.0296	0.5	1.7	1.5	C4 H11 N5 O8 C1
		292.0310	-0.9	-3.1	6.5	C5 H7 N9 O4 C1
		292.0278	2.3	7.9	14.5	C16 H7 N3 O C1
		292.0328	-2.7	-9.2	1.5	C4 H12 N7 O4 C12
		292.0274	2.7	9.2	0.5	C9 H17 N O3 C13
		292.0336	-3.5	-12.0	5.5	C9 H11 N3 O6 C1
		292.0256	4.5	15.4	5.5	C10 H12 N3 O3 C12
		292.0350	-4.9	-16.8	10.5	C10 H7 N7 O2 C1
		292.0355	-5.4	-18.5	0.5	C8 H16 N O6 C12
		292.0247	5.4	18.5	1.5	C5 H13 N7 O C13
		292.0237	6.4	21.9	10.5	C11 H7 N5 O3 C1
		292.0368	-6.7	-22.9	5.5	C9 H12 N5 O2 C12
		292.0229	7.2	24.7	6.5	C6 H8 N9 O C12
		292.0377	-7.6	-26.0	9.5	C14 H11 N O4 C1
		292.0224	7.7	26.4	5.5	C10 H11 N O7 C1
		292.0386	-8.5	-29.1	0.5	C8 H17 N3 O2 C13
		292.0215	8.6	29.4	1.5	C5 H12 N5 O5 C12

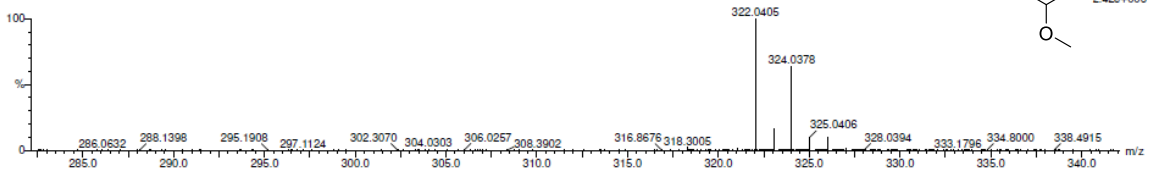
HRMS of **3dj**

Elemental Composition Report Page 1



Single Mass Analysis
Tolerance = 10.0 mDa / DBE: min = -1.5, max = 50.0
Element prediction: Off
Number of isotope peaks used for i-FIT = 3

Monoisotopic Mass, Even Electron Ions
604 formula(e) evaluated with 20 results within limits (up to 25 closest results for each mass)
Elements Used:
C: 1-100 H: 1-100 N: 1-10 O: 1-10 Cl: 1-3
Expt-754
240130YAO09 273 (2.674) Cm (271:275-(259:265+291:296))



Minimum:	80.00				-1.5	
Maximum:	100.00		10.0	500.0	50.0	
Mass	RA	Calc. Mass	mDa	PPM	DBE	Formula
322.0405	100.00	322.0402	0.3	0.9	9.5	C16 H14 N O2 C12
		322.0402	0.3	0.9	1.5	C5 H13 N5 O9 C1
		322.0415	-1.0	-3.1	6.5	C6 H9 N9 O5 C1
		322.0383	2.2	6.8	14.5	C17 H9 N3 O2 C1
		322.0380	2.5	7.8	0.5	C10 H19 N O4 C13
		322.0433	-2.8	-8.7	1.5	C5 H14 N7 O5 C12
		322.0442	-3.7	-11.5	5.5	C10 H13 N3 O7 C1
		322.0361	4.4	13.7	5.5	C11 H14 N3 O4 C12
		322.0455	-5.0	-15.5	10.5	C11 H9 N7 O3 C1
		322.0353	5.2	16.1	1.5	C6 H15 N7 O2 C13
		322.0460	-5.5	-17.1	0.5	C9 H18 N O7 C12
		322.0465	-6.0	-18.6	1.5	C5 H15 N9 O C13
		322.0343	6.2	19.3	10.5	C12 H9 N5 O4 C1
		322.0474	-6.9	-21.4	5.5	C10 H14 N5 O3 C12
		322.0335	7.0	21.7	6.5	C7 H10 N9 O2 C12
		322.0330	7.5	23.3	5.5	C11 H13 N O8 C1
		322.0482	-7.7	-23.9	9.5	C15 H13 N O5 C1
		322.0321	8.4	26.1	1.5	C6 H14 N5 O6 C12
		322.0492	-8.7	-27.0	0.5	C9 H19 N3 O3 C13
		322.0496	-9.1	-28.3	14.5	C16 H9 N5 O C1

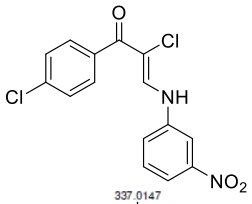
HRMS of **3dl**

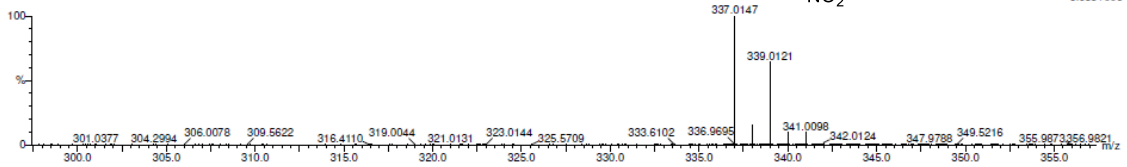
Elemental Composition Report Page 1

Single Mass Analysis
Tolerance = 10.0 mDa / DBE: min = -1.5, max = 50.0
Element prediction: Off
Number of isotope peaks used for i-FIT = 3

Monoisotopic Mass, Even Electron Ions
661 formula(e) evaluated with 25 results within limits (up to 25 closest results for each mass)
Elements Used:
C: 1-100 H: 1-100 N: 1-10 O: 1-10 Cl: 1-3

Expt-755
240131YAO04 277 (2.709) Cm (276:280-(265:270+293:299))





Minimum:	80.00				-1.5	
Maximum:	100.00		10.0	500.0	50.0	

Mass	RA	Calc. Mass	mDa	PPM	DBE	Formula
337.0147	100.00	337.0147	0.0	0.0	10.5	C15 H11 N2 O3 C12
		337.0075	7.2	21.4	6.5	C10 H10 N2 O9 Cl
		337.0125	2.2	6.5	1.5	C9 H16 N2 O5 C13
		337.0169	-2.2	-6.5	19.5	C21 H6 N2 O Cl
		337.0205	-5.8	-17.2	1.5	C8 H15 N2 O8 C12
		337.0227	-8.0	-23.7	10.5	C14 H10 N2 O6 Cl
		337.0128	1.9	5.6	15.5	C16 H6 N4 O3 Cl
		337.0187	-4.0	-11.9	6.5	C9 H10 N4 O8 Cl
		337.0106	4.1	12.2	6.5	C10 H11 N4 O5 C12
		337.0237	-9.0	-26.7	1.5	C8 H16 N4 O4 C13
		337.0219	-7.2	-21.4	6.5	C9 H11 N6 O4 C12
		337.0147	0.0	0.0	2.5	C4 H10 N6 O10 Cl
		337.0138	0.9	2.7	6.5	C10 H12 N6 O C13
		337.0066	8.1	24.0	2.5	C5 H11 N6 O7 C12
		337.0241	-9.4	-27.9	15.5	C15 H6 N6 O2 Cl
		337.0088	5.9	17.5	11.5	C11 H6 N6 O5 Cl
		337.0179	-3.2	-9.5	2.5	C4 H11 N8 O6 C12
		337.0098	4.9	14.5	2.5	C5 H12 N8 O3 C13
		337.0201	-5.4	-16.0	11.5	C10 H6 N8 O4 Cl
		337.0048	9.9	29.4	7.5	C6 H6 N8 O7 Cl
		337.0120	2.7	8.0	11.5	C11 H7 N8 O C12
		337.0210	-6.3	-18.7	2.5	C4 H12 N10 O2 C13
		337.0102	4.5	13.4	16.5	C12 H2 N10 O Cl
		337.0160	-1.3	-3.9	7.5	C5 H6 N10 O6 Cl
		337.0080	6.7	19.9	7.5	C6 H7 N10 O3 C12

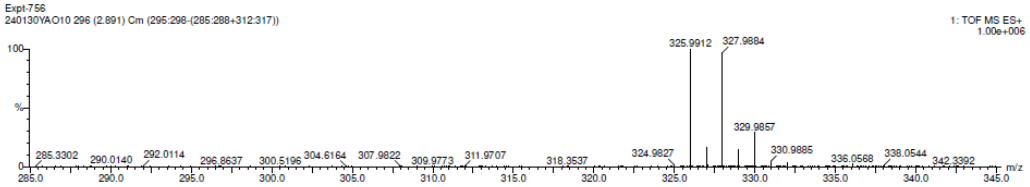
HRMS of **3dp**

Elemental Composition Report Page 1

Single Mass Analysis
Tolerance = 10.0 mDa / DBE: min = -1.5, max = 50.0
Element prediction: Off
Number of isotope peaks used for i-FIT = 3

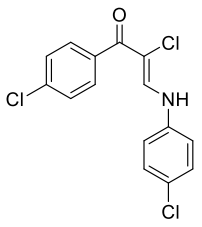
Monoisotopic Mass, Even Electron Ions
615 formula(e) evaluated with 26 results within limits (up to 25 closest results for each mass)
Elements Used:
C: 1-100 H: 1-100 N: 1-10 O: 1-10 Cl: 1-3

Expt-756
240130YAO10 296 (2.891) Cm (295:298-(285:288+312:317))



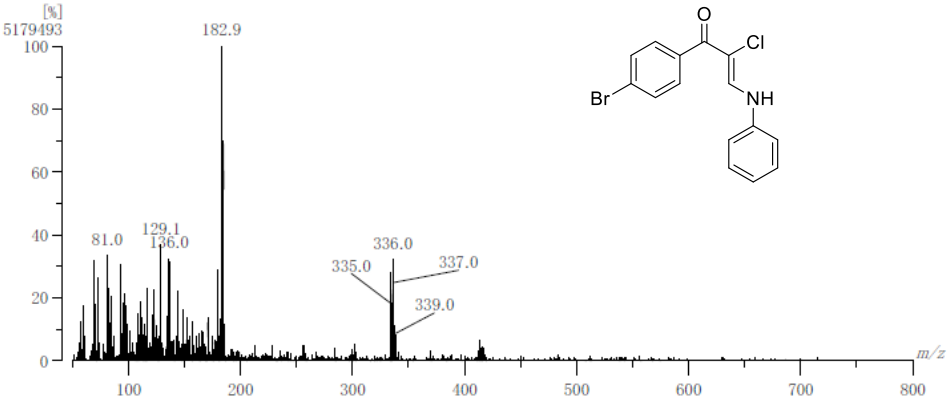
Minimum:					-1.5	
Maximum:			10.0	500.0	50.0	

Mass	Calc. Mass	mDa	PPM	DBE	Formula
325.9912	325.9906	0.6	1.8	9.5	C15 H11 N O C13
	325.9834	7.8	23.9	5.5	C10 H10 N O7 C12
	325.9987	-7.5	-23.0	9.5	C14 H10 N O4 C12
	325.9965	-5.3	-16.3	0.5	C8 H15 N O6 C13
	325.9856	5.6	17.2	14.5	C16 H5 N O5 Cl
	325.9915	-0.3	-0.9	5.5	C9 H9 N O10 Cl
	325.9888	2.4	7.4	14.5	C16 H6 N3 O C12
	325.9969	-5.7	-17.5	14.5	C15 H5 N3 O4 Cl
	325.9816	9.6	29.4	10.5	C11 H5 N3 O7 Cl
	325.9947	-3.5	-10.7	5.5	C9 H10 N3 O6 C12
	325.9866	4.6	14.1	5.5	C10 H11 N3 O3 C13
	325.9870	4.2	12.9	19.5	C17 H N5 O Cl
	325.9826	8.6	26.4	1.5	C5 H11 N5 O5 C13
	325.9978	-6.6	-20.2	5.5	C9 H11 N5 O2 C13
	325.9848	6.4	19.6	10.5	C11 H6 N5 O3 C12
	325.9928	-1.6	-4.9	10.5	C10 H5 N5 O6 Cl
	325.9906	0.6	1.8	1.5	C4 H10 N5 O8 C12
	325.9960	-4.8	-14.7	10.5	C10 H6 N7 O2 C12
	325.9829	8.3	25.5	15.5	C12 H N7 O3 Cl
	325.9938	-2.6	-8.0	1.5	C4 H11 N7 O4 C13
	325.9888	2.4	7.4	6.5	C5 H5 N7 O8 Cl
	325.9942	-3.0	-9.2	15.5	C11 H N9 O2 Cl
	326.0000	-8.8	-27.0	6.5	C4 H5 N9 O7 Cl
	325.9839	7.3	22.4	6.5	C6 H7 N9 O C13
	325.9920	-0.8	-2.5	6.5	C5 H6 N9 O4 C12



HRMS of **3ea**

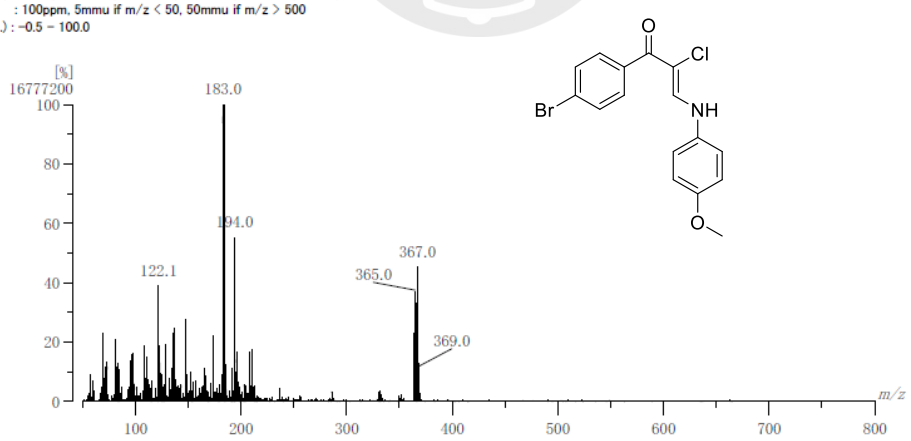
[Mass Spectrum]
Data : 20231220_HREI_Expt-741001 Date : 20-Dec-2023 15:33
RT : 0.30 min Scan# : (3,4)
Elements : C 15/0, H 15/0, 79Br 1/0, 81Br 1/0, 35Cl 1/0, 37Cl 1/0, N 1/0, O 1/0
Mass Tolerance : 100ppm, 5mmu if m/z < 50, 50mmu if m/z > 500
Unsaturation (U.S.) : -0.5 - 100.0



	Observed m/z	Int%										
	334.9703	17.49										
	Estimated m/z	Err[ppm / mmu]	U.S.	C	H	79Br	81Br	35Cl	37Cl	N	O	
1	334.9606	+29.0 / +9.7	6.5	14	14	-	1	1	1	-	-	
2	334.9657	+13.7 / +4.6	6.0	13	14	1	-	1	1	1	-	
3	334.9480	+66.5 / +22.3	7.0	13	12	-	1	1	1	1	-	
4	334.9469	+69.8 / +23.4	4.5	12	15	1	1	-	-	-	1	
5	334.9419	+84.8 / +28.4	6.5	13	12	1	-	1	1	-	1	
6	334.9713	-2.8 / -1.0	10.0	15	11	1	-	1	-	1	1	
7	334.9536	+50.0 / +16.7	11.0	15	9	-	1	1	-	1	1	
8	334.9527	+52.7 / +17.6	11.0	15	9	1	-	-	1	1	1	

HRMS of **3ej**

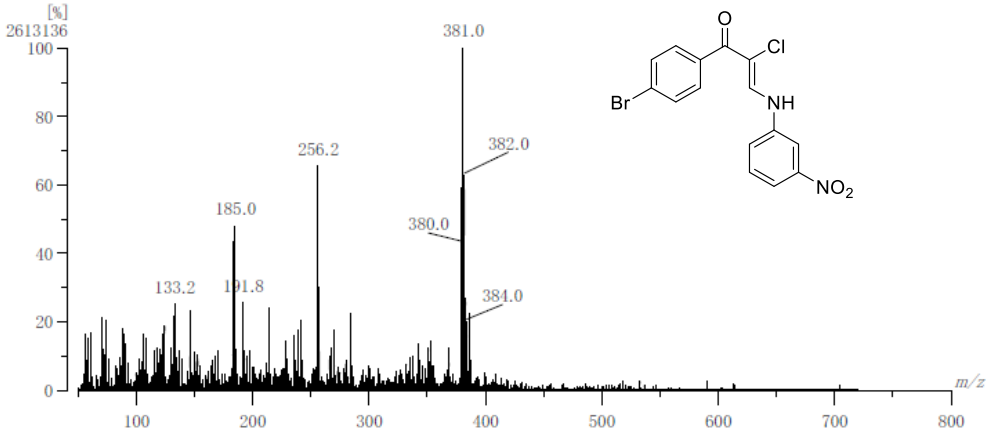
[Mass Spectrum]
Data : 20231220_HREI_Expt-742001 Date : 20-Dec-2023 15:48
RT : 0.30 min Scan# : 3
Elements : C 16/0, H 15/0, 79Br 1/0, 81Br 1/0, 35Cl 1/0, 37Cl 1/0, N 1/0, O 2/0
Mass Tolerance : 100ppm, 5mmu if m/z < 50, 50mmu if m/z > 500
Unsaturation (U.S.) : -0.5 - 100.0



	Observed m/z	Int%									
	364.9812	36.93									
	Estimated m/z	Err[ppm / mmu]	U.S.	C	H	79Br	81Br	35Cl	37Cl	N	O
1	364.9586	+62.0 / +22.6	7.0	14	14	-	1	1	1	1	1
2	364.9525	+78.7 / +28.7	6.5	14	14	1	-	1	1	-	2
3	364.9449	+99.4 / +36.3	5.0	12	15	1	1	-	-	1	2
4	364.9818	-1.7 / -0.6	10.0	16	13	1	-	1	-	1	2
5	364.9641	+46.8 / +17.1	11.0	16	11	-	1	1	-	1	2
6	364.9632	+49.3 / +18.0	11.0	16	11	1	-	-	1	1	2
7	364.9455	+97.8 / +35.7	12.0	16	9	-	1	-	1	1	2

HRMS of **3el**

[Mass Spectrum]
Data : 20231220_HREI_Expt-744001 Date : 20-Dec-2023 15:55
RT : 0.15 min Scan# : 2
Elements : C 15/0, H 10/0, 79Br 1/0, 81Br 1/0, 35Cl 1/0, 37Cl 1/0, N 2/0, O 3/0
Mass Tolerance : 100ppm, 5mmu if m/z < 50, 50mmu if m/z > 500
Unsaturation (U.S.) : -0.5 - 100.0



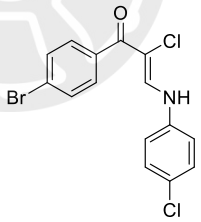
	Observed m/z	Int%									
	379.9554	42.91									
	Estimated m/z	Err[ppm / mmu]	U.S.	C	H	79Br	81Br	35Cl	37Cl	N	O
1	379.9563	-2.5 / -0.9	11.0	15	10	1	-	1	-	2	3
2	379.9386	+44.1 / +16.8	12.0	15	8	-	1	1	-	2	3
3	379.9377	+46.5 / +17.7	12.0	15	8	1	-	-	1	2	3
4	379.9200	+93.1 / +35.4	13.0	15	6	-	1	-	1	2	3

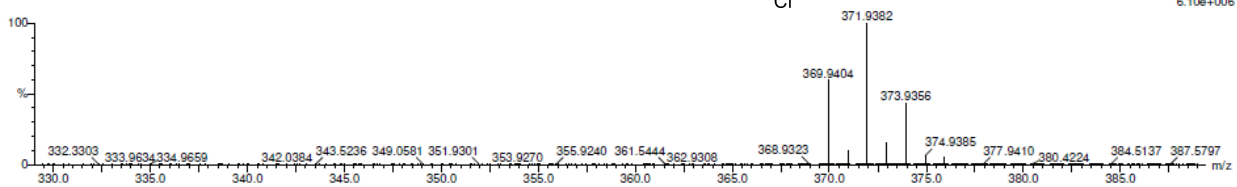
HRMS of **3ep**

Elemental Composition Report Page 1

Single Mass Analysis
Tolerance = 10.0 mDa / DBE: min = -1.5, max = 50.0
Element prediction: Off
Number of isotope peaks used for i-FIT = 3

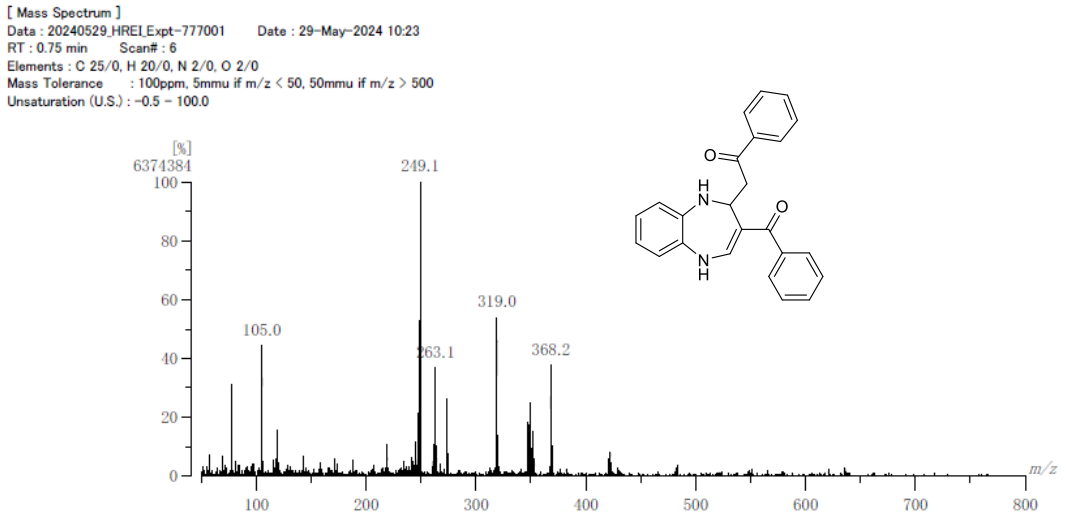
Monoisotopic Mass, Even Electron Ions
607 formula(e) evaluated with 20 results within limits (up to 25 closest results for each mass)
Elements Used:
C: 1-100 H: 1-100 N: 1-10 O: 1-10 Cl: 1-3 Br: 1-3
Expt-743
240130YAO04 300 (2.926) Cm (299:303-(287:292+313:317))





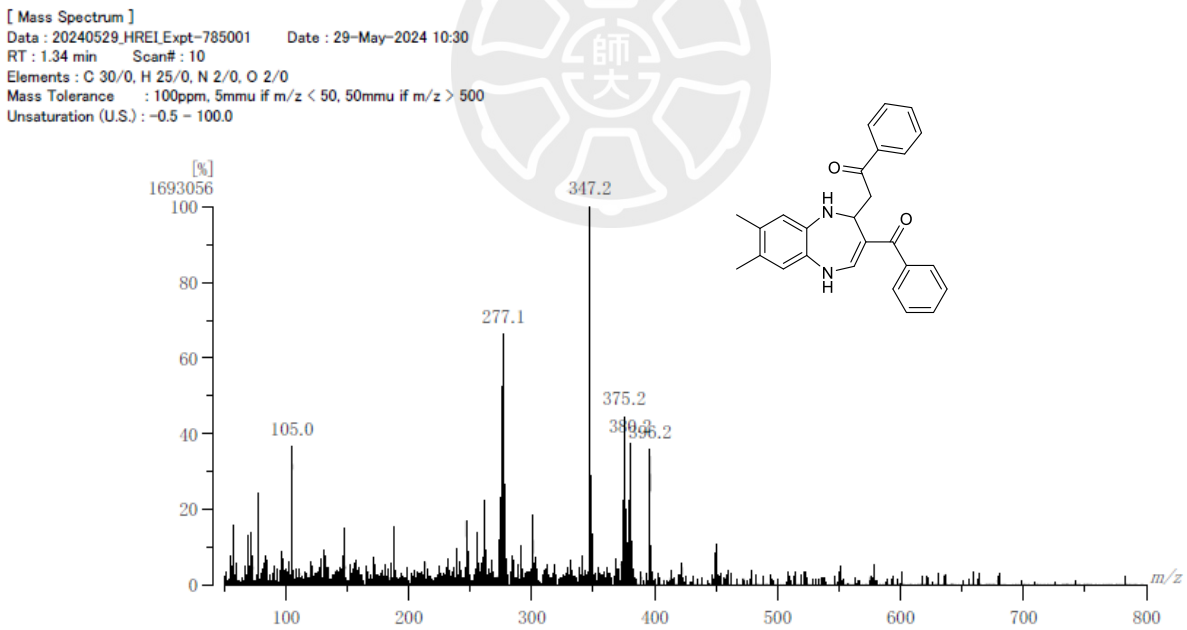
	Minimum:			-1.5	
	Maximum:	10.0	500.0	50.0	
Mass	Calc. Mass	mDa	PPM	DBE	Formula
369.9404	369.9401	0.3	0.8	9.5	C15 H11 N O Cl2 Br
	369.9401	0.3	0.8	1.5	C4 H10 N5 O8 Cl Br
	369.9415	-1.1	-3.0	6.5	C5 H6 N9 O4 Cl Br
	369.9393	1.1	3.0	-0.5	C4 H15 N7 O Cl Br2
	369.9420	-1.6	-4.3	-1.5	C8 H19 N O3 Cl Br2
	369.9383	2.1	5.7	14.5	C16 H6 N3 O Cl Br
	369.9379	2.5	6.8	0.5	C9 H16 N O3 Cl3 Br
	369.9433	-2.9	-7.8	1.5	C4 H11 N7 O4 Cl2 Br
	369.9442	-3.8	-10.3	5.5	C9 H10 N3 O6 Cl Br
	369.9361	4.3	11.6	5.5	C10 H11 N3 O3 Cl2 Br
	369.9455	-5.1	-13.8	10.5	C10 H6 N7 O2 Cl Br
	369.9352	5.2	14.1	1.5	C5 H12 N7 O Cl3 Br
	369.9460	-5.6	-15.1	0.5	C8 H15 N O6 Cl2 Br
	369.9343	6.1	16.5	10.5	C11 H6 N5 O3 Cl Br
	369.9473	-6.9	-18.7	5.5	C9 H11 N5 O2 Cl2 Br
	369.9334	7.0	18.9	6.5	C6 H7 N9 O Cl2 Br
	369.9329	7.5	20.3	5.5	C10 H10 N O7 Cl Br
	369.9482	-7.8	-21.1	9.5	C14 H10 N O4 Cl Br
	369.9321	8.3	22.4	1.5	C5 H11 N5 O5 Cl2 Br
	369.9491	-8.7	-23.5	0.5	C8 H16 N3 O2 Cl3 Br

HRMS of **6aa**



	Observed m/z	Int%	Estimated m/z	Err[ppm / mmu]	U.S.	C	H	N	O
	368.1537	37.77							
1			368.1525	+3.3 / +1.2	16.0	24	20	2	2

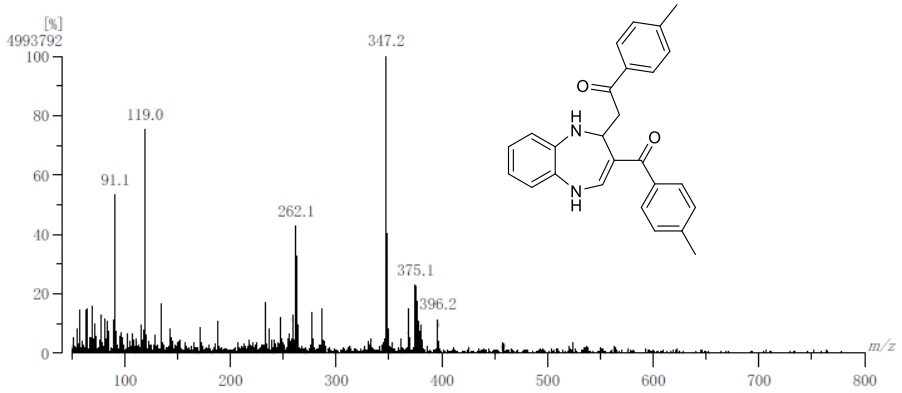
HRMS of **6ab**



	Observed m/z	Int%	Estimated m/z	Err[ppm / mmu]	U.S.	C	H	N	O
	396.1834	35.87							
1			396.1752	+20.6 / +8.2	20.5	30	22	1	-
2			396.1626	+52.4 / +20.8	21.0	29	20	2	-
3			396.1514	+80.7 / +32.0	21.0	30	20	-	1
4			396.1838	-1.0 / -0.4	16.0	26	24	2	2

HRMS of **6ba**

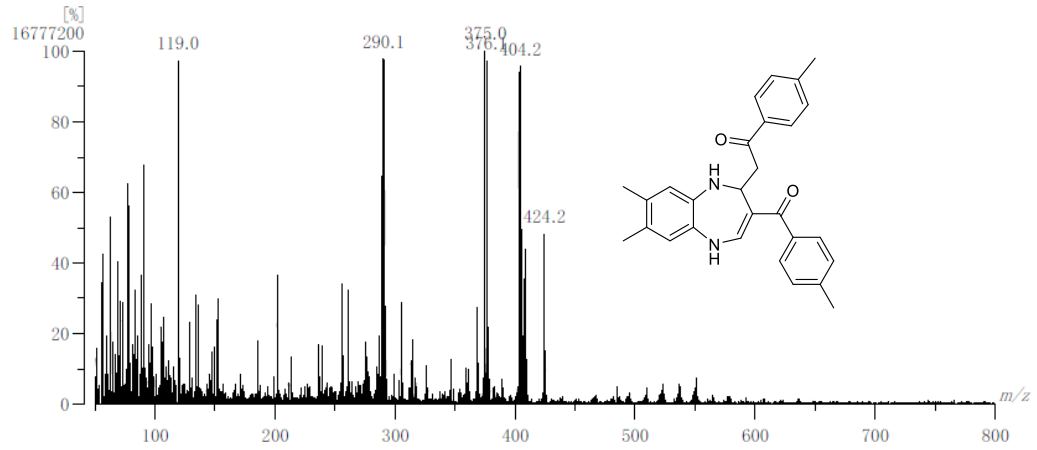
[Mass Spectrum]
Data : 20240603_HREI_6ba001 Date : 03-Jun-2024 10:39
RT : 1.49 min Scan# : 11
Elements : C 30/0, H 30/0, N 2/0, O 2/0
Mass Tolerance : 100ppm, 5mmu if m/z < 50, 50mmu if m/z > 500
Unsaturation (U.S.) : -0.5 - 100.0



	Observed m/z	Int%					
	396.1850	11.30					
	Estimated m/z	Err[ppm / mmu]	U.S.	C	H	N	O
1	396.1752	+24.7 / +9.8	20.5	30	22	1	-
2	396.1626	+56.4 / +22.4	21.0	29	20	2	-
3	396.1514	+84.8 / +33.6	21.0	30	20	-	1
4	396.2202	-88.8 / -35.2	15.0	27	28	2	1
5	396.2089	-60.4 / -23.9	15.0	28	28	-	2
6	396.1964	-28.7 / -11.4	15.5	27	26	1	2
7	396.1838	+3.1 / +1.2	16.0	26	24	2	2

HRMS of **6bb**

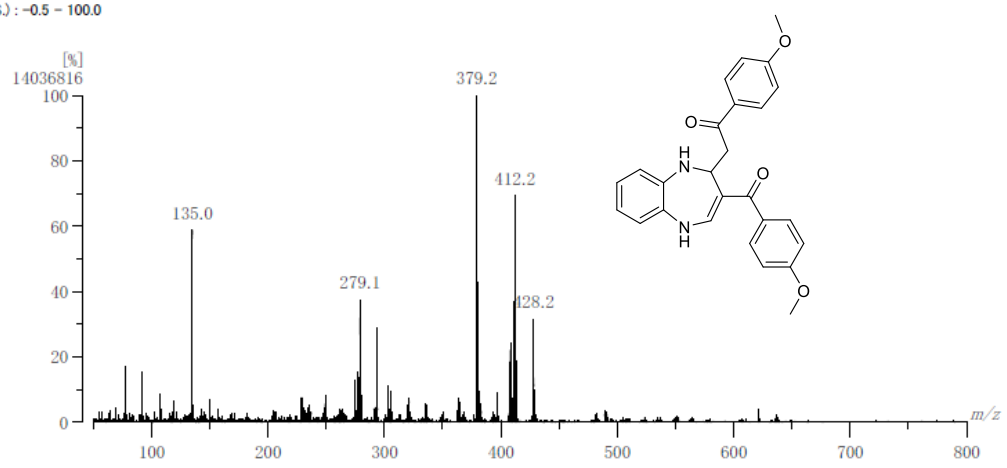
[Mass Spectrum]
Data : 20240603_HREI_6bb001 Date : 03-Jun-2024 10:48
RT : 1.64 min Scan# : 12
Elements : C 30/0, H 30/0, N 2/0, O 2/0
Mass Tolerance : 100ppm, 5mmu if m/z < 50, 50mmu if m/z > 500
Unsaturation (U.S.) : -0.5 - 100.0



	Observed m/z	Int%					
	424.2136	48.24					
	Estimated m/z	Err[ppm / mmu]	U.S.	C	H	N	O
1	424.2277	-33.1 / -14.1	15.5	29	30	1	2
2	424.2151	-3.5 / -1.5	16.0	28	28	2	2

HRMS of **6ca**

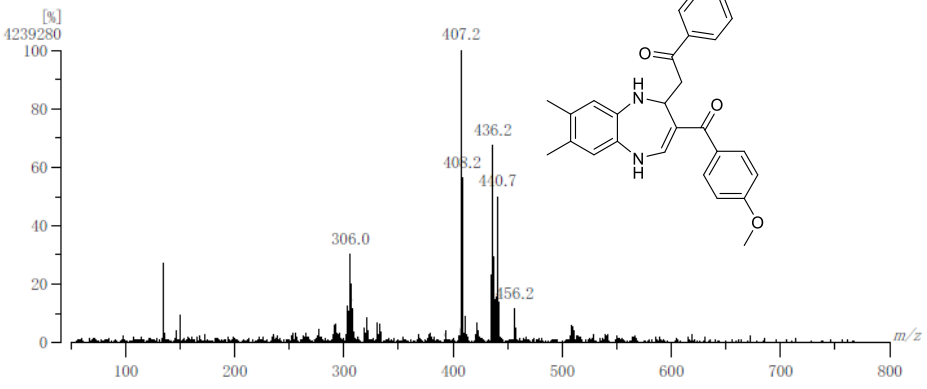
[Mass Spectrum]
Data : 20240529_HREI_Expt-829001 Date : 29-May-2024 10:55
RT : 1.19 min Scan# : 9
Elements : C 30/0, H 30/0, N 2/0, O 4/0
Mass Tolerance : 100ppm, 5mmu if m/z < 50, 50mmu if m/z > 500
Unsaturation (U.S.) : -0.5 - 100.0



	Observed m/z	Int%					
	428.1731	31.62					
	Estimated m/z	Err[ppm / mmu]	U.S.	C	H	N	O
1	428.1889	-36.8 / -15.8	20.0	30	24	2	1
2	428.1651	+18.8 / +8.0	20.5	30	22	1	2
3	428.1525	+48.2 / +20.6	21.0	29	20	2	2
4	428.1412	+74.4 / +31.9	21.0	30	20	-	3
5	428.2100	-86.2 / -36.9	15.0	27	28	2	3
6	428.1988	-59.9 / -25.7	15.0	28	28	-	4
7	428.1862	-30.6 / -13.1	15.5	27	26	1	4
8	428.1736	-1.2 / -0.5	16.0	26	24	2	4

HRMS of **6cb**

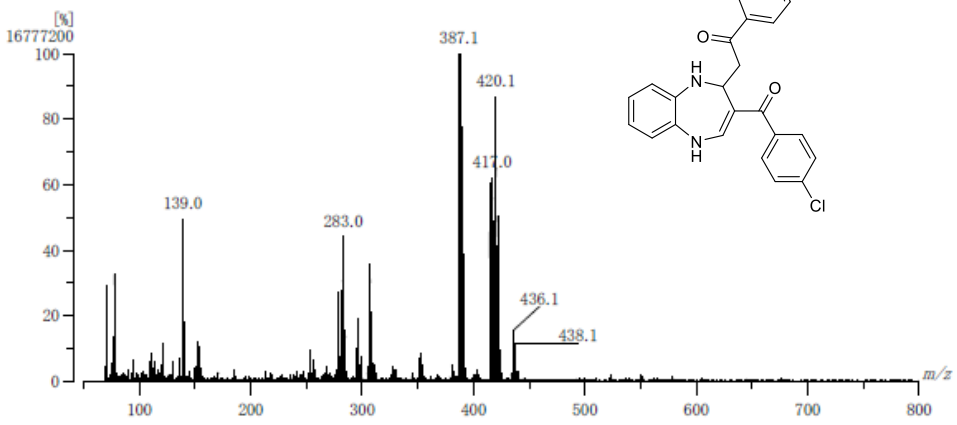
[Mass Spectrum]
Data : 20240529_HREI_Expt-830002 Date : 29-May-2024 11:13
RT : 1.34 min Scan# : 10
Elements : C 30/0, H 30/0, N 2/0, O 4/0
Mass Tolerance : 100ppm, 5mmu if m/z < 50, 50mmu if m/z > 500
Unsaturation (U.S.) : -0.5 - 100.0



	Observed m/z	Int%					
	456.2033	11.45					
	Estimated m/z	Err[ppm / mmu]	U.S.	C	H	N	O
1	456.2175	-31.1 / -14.2	15.5	29	30	1	4
2	456.2049	-3.5 / -1.6	16.0	28	28	2	4

HRMS of **6da**

[Mass Spectrum]
Data : 20240603_HREI_6da004 Date : 03-Jun-2024 11:34
RT : 0.15 min Scan# : 2
Elements : C 25/0, H 20/0, 35Cl 2/0, 37Cl 2/0, N 2/0, O 2/0
Mass Tolerance : 100ppm, 5mmu if m/z < 50, 50mmu if m/z > 500
Unsaturation (U.S.) : -0.5 - 100.0

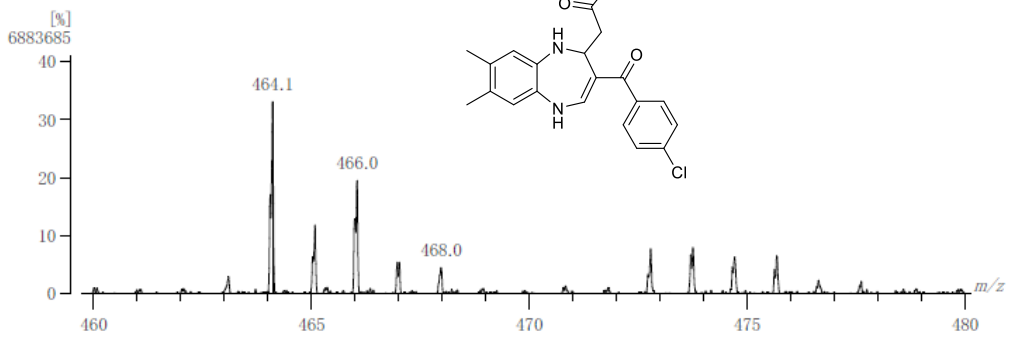


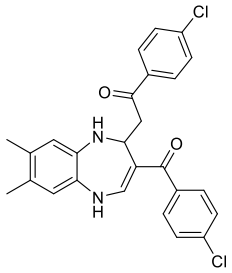
	Observed m/z	Int%								
	436.0736	14.90								
	Estimated m/z	Err[ppm / mmu]	U.S.	C	H	35Cl	37Cl	N	O	
1	436.0923	-42.9 / -18.7	16.0	25	20	1	1	2	1	
2	436.0737	-0.3 / -0.1	17.0	25	18	-	2	2	1	
3	436.0504	+53.2 / +23.2	13.0	22	19	1	2	2	1	
4	436.0392	+79.0 / +34.4	13.0	23	19	1	2	-	2	
5	436.0871	-31.0 / -13.5	15.5	25	20	2	-	1	2	
6	436.0685	+11.7 / +5.1	16.5	25	18	1	1	1	2	
7	436.0452	+65.2 / +28.4	12.5	22	19	2	1	1	2	
8	436.0499	+54.3 / +23.7	17.5	25	16	-	2	1	2	
9	436.0745	-2.1 / -0.9	16.0	24	18	2	-	2	2	
10	436.0559	+40.5 / +17.7	17.0	24	16	1	1	2	2	
11	436.0326	+94.0 / +41.0	13.0	21	17	2	1	2	2	
12	436.0373	+83.2 / +36.3	18.0	24	14	-	2	2	2	

	Observed m/z	Int%								
	438.0722	10.98								
	Estimated m/z	Err[ppm / mmu]	U.S.	C	H	35Cl	37Cl	N	O	
13	438.0397	+74.2 / +32.5	16.5	25	17	2	1	1	-	
14	438.0894	-39.2 / -17.2	16.0	25	20	-	2	2	1	
15	438.0842	-27.3 / -12.0	15.5	25	20	1	1	1	2	
16	438.0656	+15.2 / +6.6	16.5	25	18	-	2	1	2	
17	438.0422	+68.4 / +30.0	12.5	22	19	1	2	1	2	
18	438.0902	-41.1 / -18.0	15.0	24	20	2	-	2	2	
19	438.0716	+1.4 / +0.6	16.0	24	18	1	1	2	2	
20	438.0483	+54.6 / +23.9	12.0	21	19	2	1	2	2	
21	438.0530	+43.9 / +19.2	17.0	24	16	-	2	2	2	
22	438.0297	+97.1 / +42.5	13.0	21	17	1	2	2	2	

HRMS of **6db**

[Mass Spectrum]
Data : 20240429_HREI_1001 Date : 29-Apr-2024 15:32
RT : 1.04 min Scan# : 8
Elements : C 26/0, H 25/0, 35Cl 2/0, 37Cl 2/0, N 2/0, O 2/0
Mass Tolerance : 100ppm, 5mmu if m/z < 50, 50mmu if m/z > 500
Unsaturation (U.S.) : -0.5 - 100.0

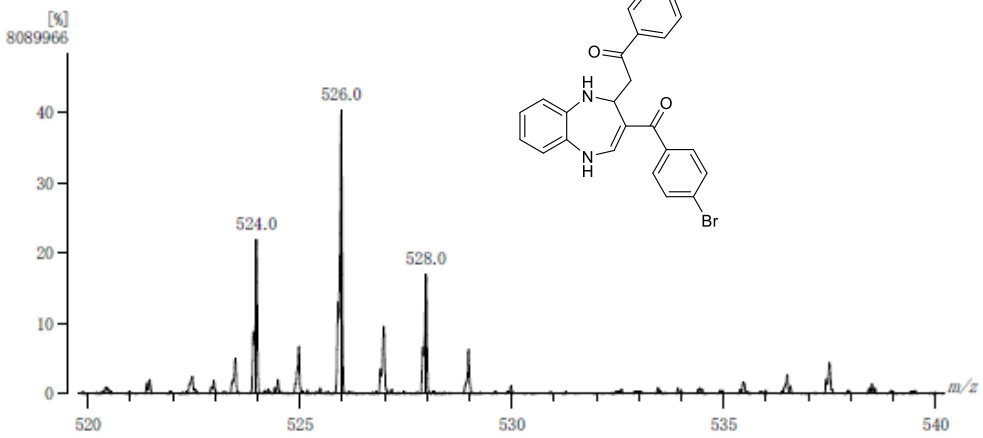




	Observed m/z	Int%							
	464.1051	33.06							
	Estimated m/z	Err[ppm / mmu]	U.S.	C	H	35Cl	37Cl	N	O
1	464.0943	+23.3 / +10.8	12.5	25	25	1	2	1	1
2	464.1003	+10.3 / +4.8	12.0	24	25	2	1	2	1
3	464.0817	+50.4 / +23.4	13.0	24	23	1	2	2	1
4	464.0891	+34.6 / +16.0	12.0	25	25	2	1	-	2
5	464.0705	+74.6 / +34.6	13.0	25	23	1	2	-	2
6	464.0765	+61.7 / +28.6	12.5	24	23	2	1	1	2
7	464.1058	-1.6 / -0.7	16.0	26	22	2	-	2	2
8	464.0872	+38.5 / +17.9	17.0	26	20	1	1	2	2
9	464.0639	+88.7 / +41.2	13.0	23	21	2	1	2	2
10	464.0686	+78.6 / +36.5	18.0	26	18	-	2	2	2

HRMS of **6ea**

[Mass Spectrum]
Data : 20240429_HREI_787001 Date : 29-Apr-2024 17:15
RT : 1.19 min Scan# : 9
Elements : C 25/0, H 20/0, 79Br 2/0, 81Br 2/0, N 2/0, O 2/0
Mass Tolerance : 100ppm, 5mmu if m/z < 50, 50mmu if m/z > 500
Unsaturation (U.S.) : -0.5 - 100.0

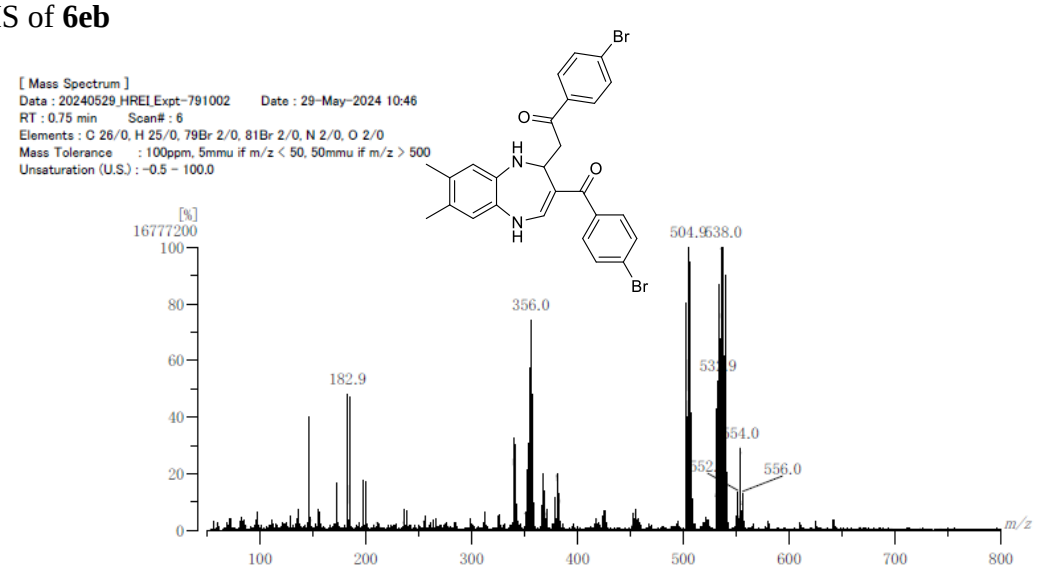


	Observed m/z	Int%							
	523.9738	21.90							
	Estimated m/z	Err[ppm / mmu]	U.S.	C	H	79Br	81Br	N	O
1	523.9922	-35.1 / -18.4	16.0	25	20	1	1	2	1
2	523.9745	-1.3 / -0.7	17.0	25	18	-	2	2	1
3	523.9861	-23.4 / -12.3	15.5	25	20	2	-	1	2
4	523.9684	+10.3 / +5.4	16.5	25	18	1	1	1	2
5	523.9507	+44.1 / +23.1	17.5	25	16	-	2	1	2
6	523.9735	+0.6 / +0.3	16.0	24	18	2	-	2	2
7	523.9558	+34.3 / +18.0	17.0	24	16	1	1	2	2
8	523.9381	+68.1 / +35.7	18.0	24	14	-	2	2	2

	Observed m/z	Int%							
	525.9759	40.19							
	Estimated m/z	Err[ppm / mmu]	U.S.	C	H	79Br	81Br	N	O
9	525.9901	-27.1 / -14.2	16.0	25	20	-	2	2	1
10	525.9840	-15.5 / -8.1	15.5	25	20	1	1	1	2
11	525.9663	+18.2 / +9.6	16.5	25	18	-	2	1	2
12	525.9892	-25.2 / -13.3	15.0	24	20	2	-	2	2
13	525.9715	+8.5 / +4.4	16.0	24	18	1	1	2	2
14	525.9538	+42.1 / +22.1	17.0	24	16	-	2	2	2

	Observed m/z	Int%							
	527.9737	16.98							
	Estimated m/z	Err[ppm / mmu]	U.S.	C	H	79Br	81Br	N	O
15	527.9820	-15.7 / -8.3	15.5	25	20	-	2	1	2
16	527.9871	-25.4 / -13.4	15.0	24	20	1	1	2	2
17	527.9694	+8.1 / +4.3	16.0	24	18	-	2	2	2

HRMS of **6eb**



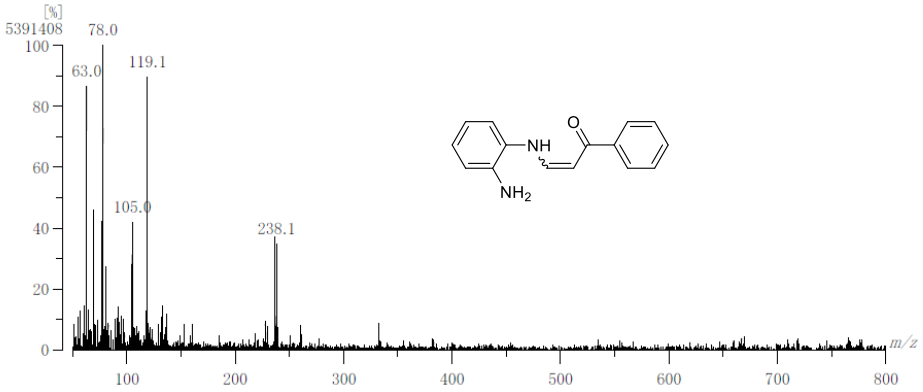
	Observed m/z	Int%							
	552.0033	13.46							
	Estimated m/z	Err[ppm / mmu]	U.S.	C	H	79Br	81Br	N	O
1	552.0048	-2.7 / -1.5	16.0	26	22	2	-	2	2
2	551.9871	+29.3 / +16.2	17.0	26	20	1	1	2	2
3	551.9694	+61.4 / +33.9	18.0	26	18	-	2	2	2

	Observed m/z	Int%							
	554.0021	29.01							
	Estimated m/z	Err[ppm / mmu]	U.S.	C	H	79Br	81Br	N	O
4	554.0205	-33.1 / -18.4	15.0	26	24	2	-	2	2
5	554.0028	-1.2 / -0.7	16.0	26	22	1	1	2	2
6	553.9851	+30.8 / +17.0	17.0	26	20	-	2	2	2

	Observed m/z	Int%							
	556.0003	13.14							
	Estimated m/z	Err[ppm / mmu]	U.S.	C	H	79Br	81Br	N	O
7	555.9547	+81.9 / +45.6	10.0	22	25	2	1	2	-
8	556.0184	-32.6 / -18.1	15.0	26	24	1	1	2	2
9	556.0007	-0.7 / -0.4	16.0	26	22	-	2	2	2

HRMS of **7aa**

[Mass Spectrum]
Data : 20240603_HREI_7aa001 Date : 03-Jun-2024 12:02
RT : 0.45 min Scan# : 4
Elements : C 15/0, H 15/0, N 2/0, O 1/0
Mass Tolerance : 100ppm, 5mmu if m/z < 50, 50mmu if m/z > 500
Unsaturation (U.S.) : -0.5 - 100.0



	Observed m/z	Int%	Err[ppm / mmu]	U.S.	Composition
1	238.1102	34.82	-1.7 / -0.4	10.0	C15 H14 N2 O

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Part III

Iodine-Catalyzed Cascade Reactions of 2-Styrylbenzaldehydes with Indoles for the Synthesis of 1*H*-Indenes via 4π-Electrocyclization

摘要

一種合成 1H-茚類化合物的級聯反應已被開發，通過 2-苯乙烯基苯甲醛與吲哚反應實現。該過程包括碘催化的吲哚對 2-苯乙烯基苯甲醛醛基的親核加成，形成碳陽離子中間體。這些中間體隨後經歷 4π-電子環化反應，生成具有三取代雙鍵的茚類化合物。在反應條件下，這些茚類化合物進一步轉化為熱力學更穩定的全取代雙鍵產物。此外，反應還生成少量吲哚基苯并[b]咔唑類副產物。

Abstract

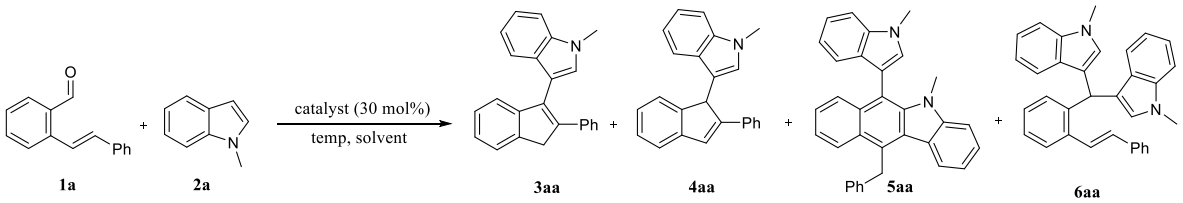
A cascade reaction has been developed for the synthesis of 1H-indenes by reacting 2-styrylbenzaldehydes with indoles. The process involves an iodine-catalyzed nucleophilic addition of indoles to the aldehyde group of 2-styrylbenzaldehydes, resulting in the formation of carbocation intermediates. These intermediates undergo 4π-electrocyclization to yield indenes with a trisubstituted double bond. Under the reaction conditions, these indenes are further transformed into more thermodynamically stable products with a fully substituted double bond. Minor amounts of indolylbenzo[b]carbazoles are also generated as byproducts.

1. Introduction

In 2018, molecular iodine-catalyzed cycloisomerization of conjugated dienals was reported, leading to the synthesis of cyclopentenones. However, transition-metal-free methods for the synthesis of heterocyclic compounds using iodine as a catalyst remain underexplored. Some iodine-catalyzed reactions involving indoles as nucleophiles have been documented, including [4+2] annulation strategies to form carbazole derivatives and iodine-promoted electrocyclization of 4-arylhex-2-en-5-ynals with indoles to yield polysubstituted cyclopentene derivatives or cyclopenta[b]carbazoles. Additionally, iodine-promoted cascade reactions between 2-(phenylethynyl)benzaldehydes and indoles have been developed for the generation of indole-fused azulene derivatives. However, to the best of our knowledge, the iodine-catalyzed reaction of 2-styrylbenzaldehydes with indoles to access 1H-indene derivatives has not been previously reported in the literature.

Given the possible applications of substituted 1H-indenes in medicinal chemistry, a general and practical synthetic route is highly desirable. Motivated by our previous work on iodine-catalyzed reactions, we present here a cascade reaction of 2-styrylbenzaldehydes with indoles, which is catalyzed by molecular iodine, for the synthesis of a range of substituted 1H-indene derivatives. In our study, the reaction between 2-styrylbenzaldehyde 1a and indole 2a was carried out at 70°C using 30 mol% iodine as a catalyst. The product 4aa was obtained in 23% yield (Table 1, entry 1). The reaction was then performed under microwave heating conditions, yielding 4aa in 36% at 50°C (entry 2). When conducted at 80°C, the yield of 4aa increased to 53%, accompanied by a 10% yield of 3aa (entry 3). Similar results were obtained when toluene was used as the solvent (entry 4).

Table 1. Optimization of the reaction conditions.[a]



1a **2a** **3aa** **4aa** **5aa** **6aa**

Entry	Catalyst	Solvent	Temp (°C)	Time (h)	Yield (%) **3aa**[b]	Yield (%) **4aa**[b]	Yield (%) **5aa**[b]	Yield (%) **6aa**[b]
1[c]	I_2	EA	70	2	-	23	-	-
2	I_2	EA	50	2	-	36	-	-
3	I_2	EA	80	2	10	53	-	-
4	I_2	toluene	80	2	9	54	-	-
5	I_2	CH_3CN	80	2	3	23	-	-
6	I_2	$(CH_2)_2Cl_2$	80	2	34	-	5	-
7	I_2	DMSO	80	2	-	-	-	13
8	I_2	DMF	80	2	-	36	-	19
9	I_2	toluene	90	2	59	21	5	-
10	I_2	toluene	100	1.5	65	10	10	-
11	I_2	toluene	100	1	55	8	6	-
12	I_2	toluene	110	1	72	trace	5	-
13	I_2	toluene	120	1	82 (77)[d]	trace	9	-
14[e]	I_2	toluene	120	1	75	trace	8	-
15[f]	I_2	toluene	120	1	trace	18	trace	-
16	$TsOH•H_2O$	toluene	120	1	-	83	-	-
17	$TsOH•H_2O$	toluene	90	0.5	-	81	-	-
18	$TsOH•H_2O$	toluene	70	0.5	-	80 (75)[d]	-	-

[a]All of the reactions were carried out with **1a** (0.25 mmol, 52 mg), **2a** (0.3 mmol, 39 mg), and catalyst (0.075 mmol) in solvent (5 mL) under microwave heating conditions. [b]Yields were determined by ^1H NMR analyses of the crude reaction mixtures using CH_2Br_2 as the internal standard. [c]The reaction was carried out using oil-bath heating conditions. [d]Isolated yield. [e]20 mol% I_2 was used. [f]10 mol% I_2 was used. EA = ethyl acetate. DMSO = dimethyl sulfoxide. DMF = dimethylformamide.

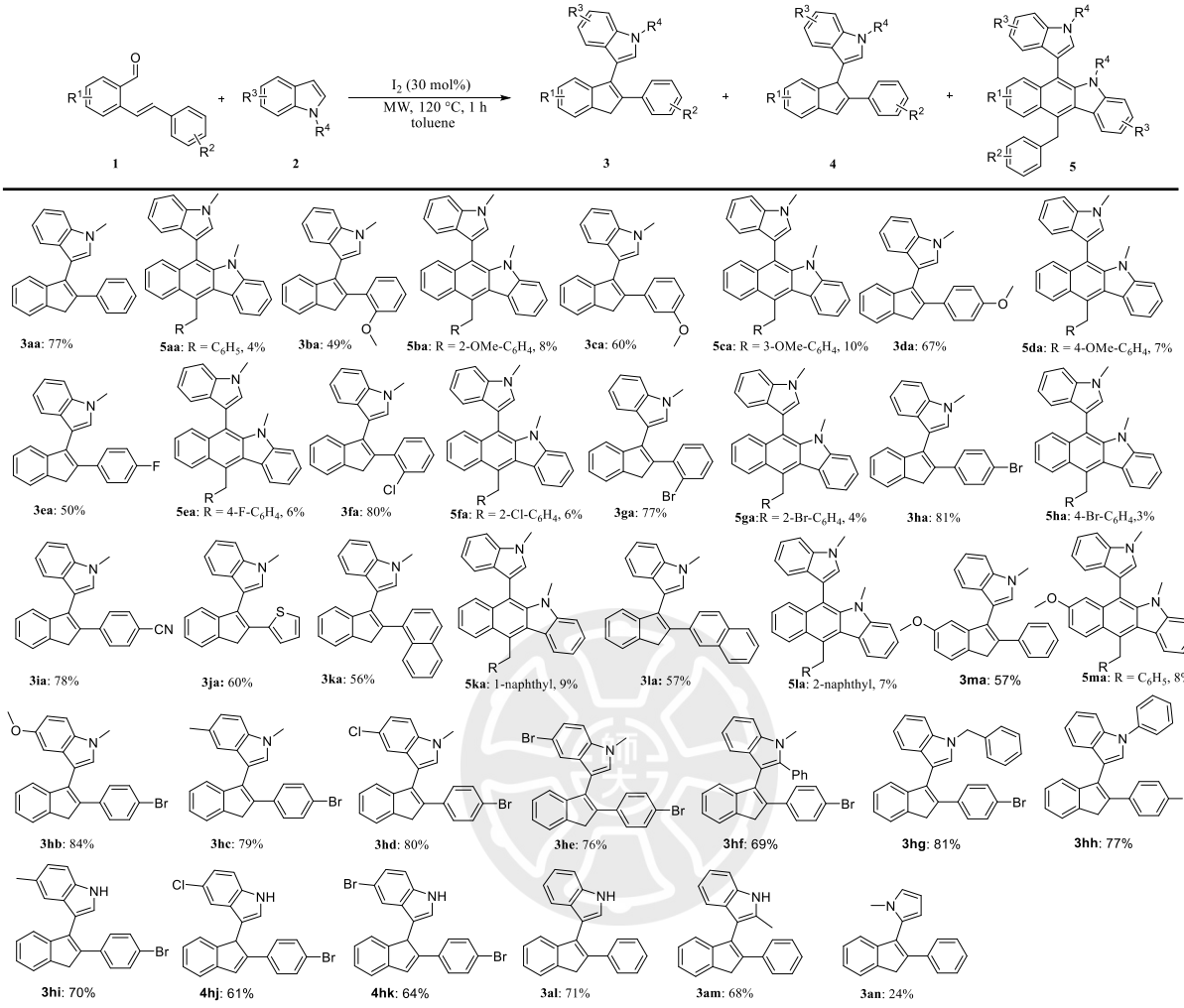
The efficiency of the reaction was reduced when CH_3CN or $(CH_2Cl)_2$ was used as the solvent system (entries 5 and 6). Additionally, reactions in DMSO and DMF resulted in the formation of 6aa, but the yields were relatively low, ranging from 13–19% (entries 7 and 8). Reactions performed at higher temperatures using toluene as the solvent improved the yields, with 1H-indene 3aa formed in 59% yield and 4aa in 21% yield when conducted at 90°C in a microwave reactor (entry 9). Notably, carbazole derivative 5aa was also formed as a minor product in 5% yield. Increasing the reaction temperature to 100°C resulted in a higher yield of 3aa (65%), along with 4aa and 5aa in 10% yields (entry 10). When the reaction time was reduced to 1 hour, 3aa and 4aa

were produced in 55% and 8% yields, respectively (entry 11). A further increase in temperature to 110°C showed slightly improved selectivity (entry 12), and when the temperature reached 120°C, 1H-indene 3aa was obtained in 82% yield (entry 13). These results suggest that the reaction outcome can be fine-tuned by adjusting the reaction conditions. Using 20 mol% iodine as a catalyst decreased the efficiency to some extent (entry 14), and using 10 mol% iodine resulted in the formation of 4aa in only 18% yield (entry 15). The selective formation of 4aa was observed when TsOH · H_2O was used as the catalyst (entries 16–18); however, 3aa did not form even at 120 °C under these conditions. It appears that iodine plays a critical role in the isomerization step. Overall, the optimal reaction conditions for synthesizing 1H-indene derivatives were found to be those in entry 13.

With the optimized reaction conditions established for the synthesis of 1H-indene 3aa, we sought to test the generality of the cascade reaction using different 2-styrylbenzaldehydes and indoles (Table 2). When 1b, containing an ortho-methoxy group at the styryl position, was reacted with 2a, the desired product 3ba was obtained in 49% yield, along with 4ba in 8% yield. Similarly, substrates 1c and 1d with electron-donating methoxy groups reacted with 2a to produce the corresponding 1H-indenes in yields up to 67%, with minor quantities of benzo[b]carbazoles (5ca and 5da). Substrate 1e, when coupled with 2a, yielded 3ea in 50% yield, with a minor product in 6% yield. A substrate with an ortho-chloro group (1f) demonstrated enhanced reactivity, producing 3fa in 80% yield, along with 5fa in 6% yield. Substrate 1g, bearing an ortho-bromo styryl group, gave the desired product 3ga in 77% yield. When 1h was used, 1H-indene 3ha was obtained in 81% yield, while substrate 1i with an electron-withdrawing cyano group gave 3ia in 78% yield. These results suggest that substrates with electron-withdrawing substituents exhibit good reactivity, leading to high yields and selectivity for 1H-indene products. Substrate 1j, with a

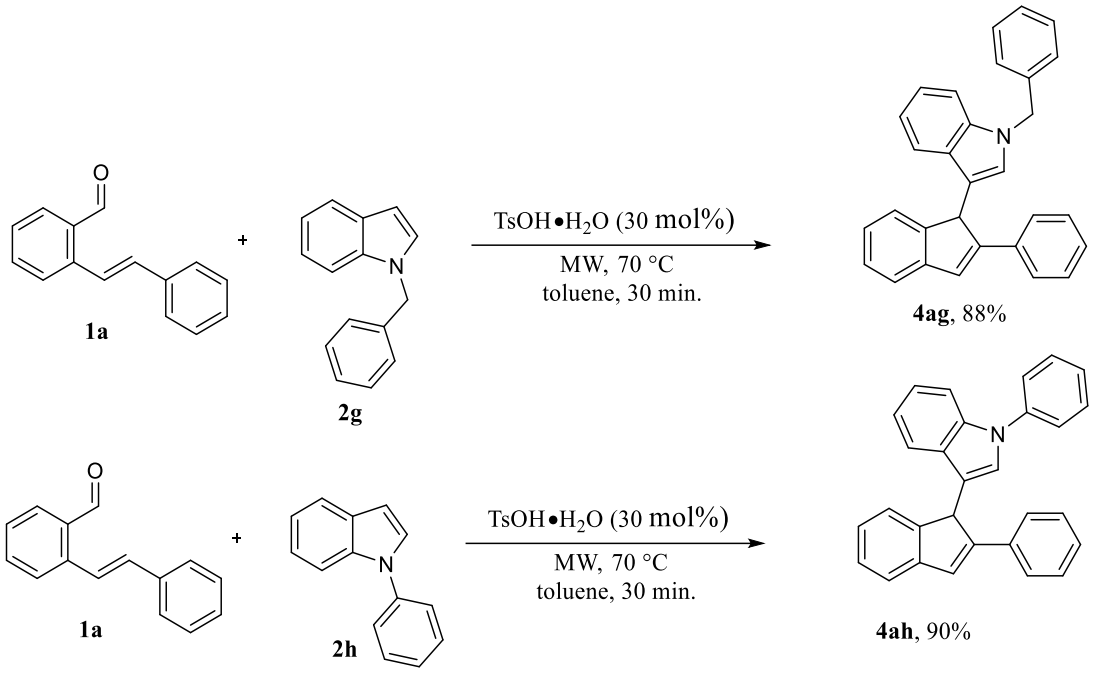
thiophene moiety, gave 3ja in a moderate yield of 60%. Finally, reactions with substrates containing a naphthyl group (1k and 1l) resulted in the formation of 3ka and 3la in 56% and 57% yields, respectively, along with carbazole derivatives (5ka and 5la) in 9% yields. The reaction with 2-styrylbenzaldehyde containing a methoxy group at the meta-position (1m) yielded the corresponding product 3ma in 57% yield. Reactions of 1h with various indoles were performed to assess the versatility of this cascade reaction for synthesizing 1H-indenes. N-Methylindoles with electron-donating groups at the 5-position (2b and 2c) reacted efficiently, yielding the desired products 3hb and 3hc in 79% and 84% yields, respectively. Similarly, when N-methylindoles with electron-withdrawing groups (2d and 2e) were used, 1H-indenes 3hd and 3he were obtained in good yields. Additionally, 2-phenyl-substituted indole gave the expected product in a 69% yield. Next, N-benzyl and N-phenyl indoles were employed as reaction partners with 1h, resulting in the formation of 1H-indenes 3hg and 3hh in 81% and 77% yields, respectively. To further investigate the reaction mechanism, (NH)-indoles were tested. When 5-methylindole was reacted with 1h under the optimized conditions, the expected product 3hi was obtained in 70% yield. However, indoles 2j and 2k produced products 4hj and 4hk with a trisubstituted double bond, and no isomerization occurred to form the more stable 3hj and 3hk with a fully substituted double bond. This suggests that an electron-rich indole favors isomerization, leading to a more conjugated system. Finally, reactions of indoles 2l and 2m with 1h resulted in the formation of 1H-indenes 3al and 3am, each with a fully substituted double bond. A reaction with N-methylpyrrole as the nucleophile also led to the formation of product 3an, but with a much lower yield of 24%. $TsOH \cdot H_2O$-catalyzed reactions were conducted to selectively synthesize 1H-indenes with a trisubstituted double bond. Indoles 2g and 2h reacted efficiently with 1a, yielding the corresponding indenes 4ag and 4ah in 88% and 90% yields, respectively (Scheme 2).

Table 2. Substrate scope for the synthesis of 1H-indene derivatives.[a,b,c,d]



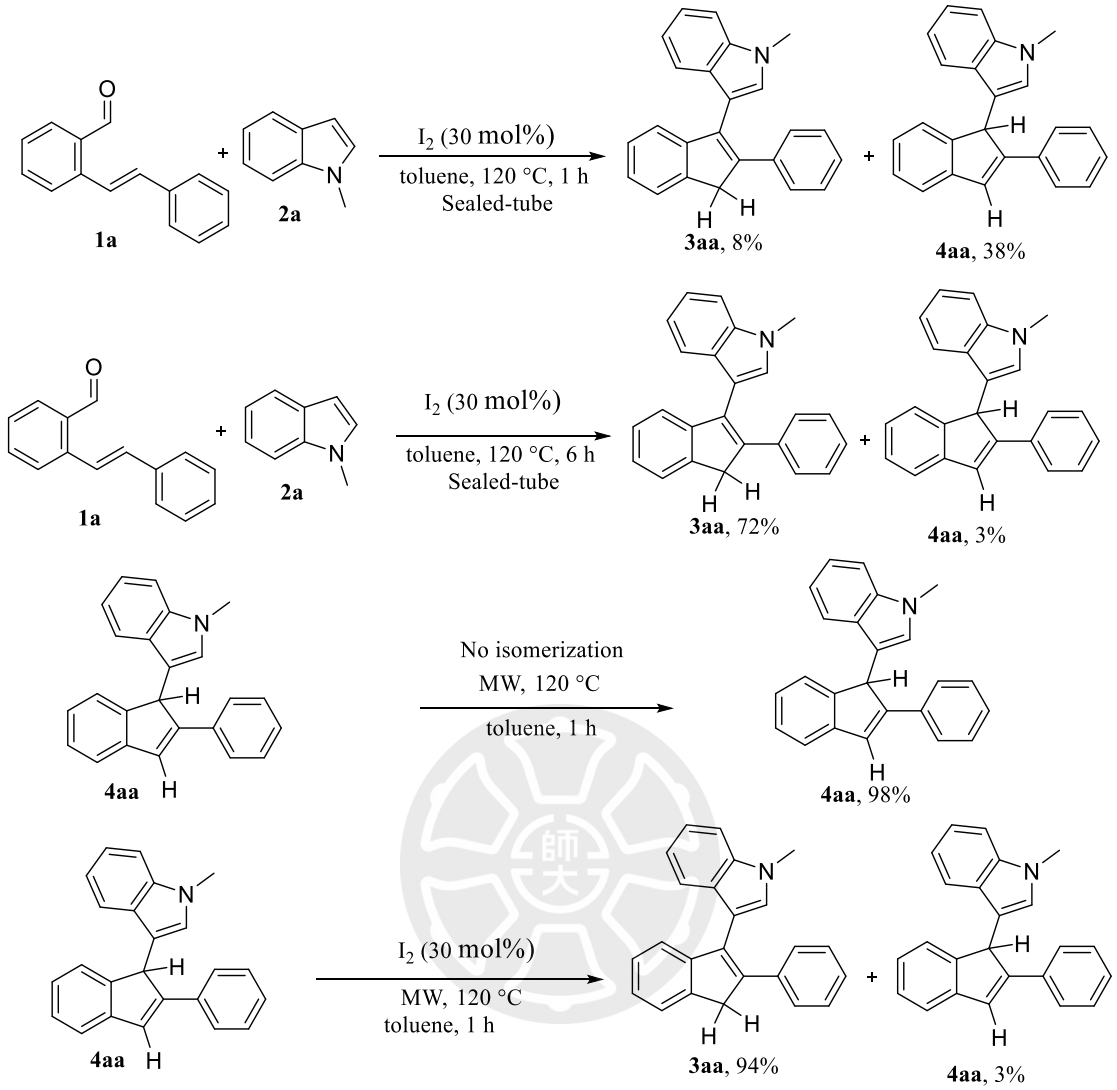
[a]All of the reactions were carried out with **1** (0.25 mmol), **2** (0.3 mmol), and I_2 (0.075 mmol, 19 mg) in toluene (5 mL) under microwave heating conditions at 120 ° C. [b]Isolated yield. [c]The product **4** was formed in trace quantities. [d]The formation of **5** was observed in traces.

To investigate the reaction mechanism, several control experiments were conducted. Analysis of the crude reaction mixture via ^{1}H NMR showed a significant decrease in reaction efficiency when performed for 1 hour under sealed-tube heating conditions (Scheme 3). However, product 3aa was obtained in a 72% yield with a longer reaction time. Indene 4aa did not undergo isomerization when heated at 120 °C in toluene. In contrast, when 4aa was treated with iodine (30 mol%) at 120 °C, it was converted to the more stable 3aa. This observation suggests that iodine plays a crucial role in facilitating the isomerization process.



Scheme 2. Synthesis of 1H-indenes with a trisubstituted olefinic moiety.

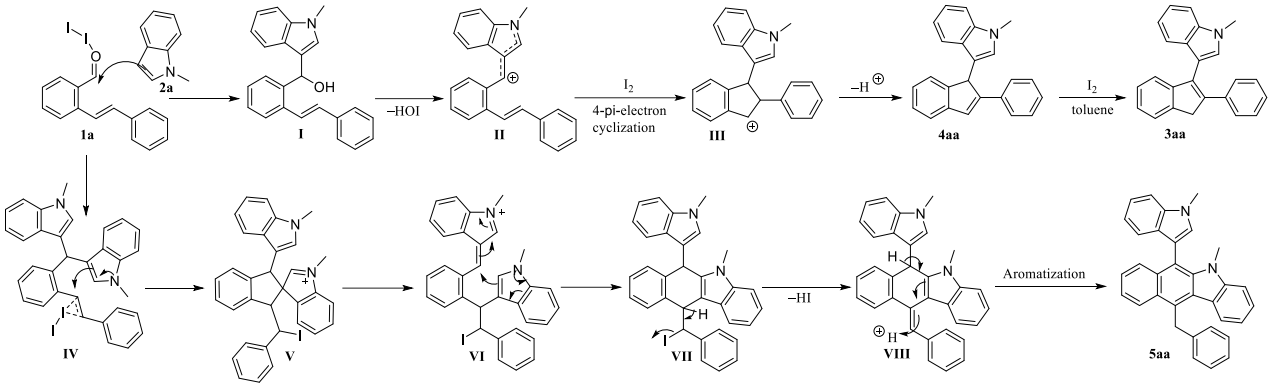
Based on our observations and previous literature reports [13,15], we propose a plausible mechanism for the formation of 1H-indene 3aa (Scheme 4). Initially, indole 2a undergoes nucleophilic addition to the aldehyde group of 1a, forming intermediate I. Under the reaction conditions, this intermediate can undergo carbocation formation, resulting in intermediate II. Following 4π-electrocyclization, intermediate II is converted to intermediate III. The elimination of a proton leads to the formation of 1H-indene 4aa, which contains a trisubstituted double bond. Subsequently, the thermodynamically more stable 1H-indene 3aa, with a tetrasubstituted double bond, is formed. Additionally, the byproduct, indolylbenzo[b]carbazole 5aa, is produced from bis(indolyl)methane intermediate IV, as described in Scheme 3.[16].



Scheme 3. Control experiments.

In summary, we have developed a one-pot, metal-free method for synthesizing 1H-indenes using iodine as a catalyst. The reaction proceeds through a sequence of nucleophilic addition, 4π-electrocyclization, and isomerization steps to form the desired products. This method is compatible with a wide range of substrates and allows for the efficient synthesis of 1H-indenes in a short time. Along with the 1H-indenes, benzo[b]carbazole derivatives are also produced as minor byproducts. Additionally, we demonstrated that TsOH · H2O can selectively synthesize indenes with a trisubstituted double bond. A plausible reaction mechanism has been proposed to explain the

formation of the desired products. Given the significant biological activities of indene derivatives and their presence in many natural products, the approach outlined here offers a valuable synthetic route.



Scheme 4. Proposed reaction mechanism.

Experimental Section

General information

Unless otherwise specified, all microwave reactions were performed in a closed vessel using a commercial CEM microwave reactor equipped with an external IR sensor. The 35 mL microwave vials were cleaned, dried in an oven, and allowed to cool to room temperature before use. All reagents and solvents, unless stated otherwise, were obtained from commercial suppliers and used as received without further purification. Round-bottom flasks were also cleaned, dried, and cooled to room temperature prior to use. Reactions were carried out under a nitrogen atmosphere at room temperature. Flash column chromatography was performed on 63-200 mesh silica gel, with a hexane/ethyl acetate mixture used as the eluent. 1H and 13C NMR spectra were recorded on a Bruker Ascend spectrometer at 400 MHz and 100 MHz, respectively. Chemical shifts are reported in parts per million (ppm) on the δ scale, using $CDCl_3$ as the internal standard. Multiplicities are denoted as follows: s = singlet, d = doublet, t = triplet, q = quartet, dd = doublet of doublets, m = multiplet, br = broad signal. Coupling constants are expressed in Hertz (Hz). High-resolution mass

spectrometry (HRMS) was conducted in ESI-TOF mode. Melting points (mp) were determined using an Electro-Thermal FARGO MP-2D capillary melting point apparatus. The diethyl benzylphosphonates were synthesized according to a previously reported procedure. The preparation of 2-styrylbenzaldehyde 1m followed the method described in the literature. Indoles 2g and 2h were synthesized using established methods.

General Procedure for the Synthesis of 1H-indenes

To a 35 mL microwave vial, dried in an oven and cooled to room temperature, were added 2-styrylbenzaldehyde (0.25 mmol), toluene (3 mL), and I2 (0.075 mmol, 19 mg). Indole (0.3 mmol) and toluene (2 mL) were then introduced into the vial, which was sealed with a silicone cap. The vial was placed in the microwave reactor and heated at 120 °C for 1 hour. After cooling the reaction mixture to room temperature, it was transferred to a separatory funnel containing ethyl acetate (30 mL). A 1 M solution of $Na_2S_2O_3$ (5 mL) was added, and the organic layer was separated. The aqueous layer was then extracted with ethyl acetate (20 mL). The combined organic layers were washed with brine and dried over $MgSO_4$. The solvent was evaporated under reduced pressure, and the crude product was purified by silica gel column chromatography using a mixture of ethyl acetate and hexane as the eluent.

2. General procedure for the synthesis of 2-styrylbenzaldehydes

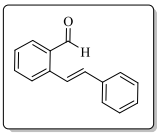
Step 1: An oven-dried 100 mL reaction flask was cooled to room temperature under a nitrogen atmosphere. To this flask, 2-(2-bromophenyl)-1,3-dioxolane (2 mmol, 0.458 g, 0.3 mL) and dry THF (20 mL) were added, and the mixture was cooled to -15 °C using an ice-salt bath. The 2.5 M solution of n-BuLi (2.5 mmol, 1.0 mL) was then added dropwise over 5 minutes, and the reaction was stirred for 10 minutes. DMF (4 mmol, 0.293 g, 0.31 mL) was subsequently added, and the reaction continued for 1 hour. The reaction was quenched by the addition of 1 M HCl (2 mL). The mixture was extracted with ethyl acetate and water. The organic layer was separated, and the

aqueous layer was re-extracted with ethyl acetate (20 mL). The combined organic layers were washed with brine and dried over $MgSO_4$. The solvent was evaporated, and the crude product was used directly in the next step.

Step 2: The 65% NaH solution (4 mmol, 0.148 g) was added to an oven-dried reaction flask, which was cooled to room temperature under a nitrogen atmosphere. The reaction mixture was then cooled to 0 °C using an ice bath. The crude diethyl benzylphosphonate (3 mmol) in THF (10 mL) was added via an addition funnel. The crude reaction mixture from Step 1 was dissolved in dry THF (10 mL) and added to the reaction flask via an addition funnel. The reaction mixture was allowed to warm to room temperature and continued for 16 hours. The reaction was cooled to 0 °C and quenched with ice. The crude mixture was extracted with ethyl acetate (35 mL), and the aqueous layer was re-extracted with ethyl acetate (20 mL). The solvent was removed under reduced pressure, and the crude product was dissolved in acetone (20 mL). HCl (5 mL, 1 M) was added, and the mixture was stirred for 6 hours at room temperature. The solvent was evaporated under reduced pressure, and a standard work-up procedure was followed using ethyl acetate and water. The organic layer was dried over $MgSO_4$. After solvent removal, the crude product was purified by silica gel column chromatography.

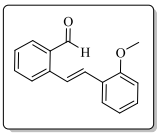
3. NMR Data of 2-Styrylbenzaldehydes

(*E*)-2-styrylbenzaldehyde (1a)[2]



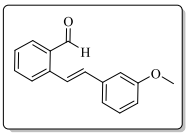
The crude product was purified by column chromatography on silica gel using hexane/ethyl acetate = 96:4 (v/v) as the eluent. Yield 75% (312 mg); Pale yellow liquid. ^{1}H NMR (400 MHz, $CDCl_3$) δ 10.33 (s, 1H), 8.04 (d, *J* = 16.0 Hz, 1H), 7.84 (dd, *J* = 8.0, 4.0 Hz, 1H), 7.73 (d, *J* = 8.0 Hz, 1H), 7.61-7.56 (m, 3H), 7.46-7.36 (m, 3H), 7.32-7.29 (m, 1H), 7.05 (d, *J* = 16.0 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 192.5, 139.8, 136.7, 133.8, 133.6, 132.7, 132.2, 128.6, 128.2, 127.5, 127.0, 126.8, 124.6.

(*E*)-2-(2-methoxystyryl)benzaldehyde (1b)



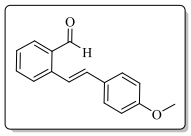
The crude product was purified by column chromatography on silica gel using hexane/ethyl acetate = 95:5 (v/v) as the eluent. Yield 65% (309 mg); Yellow solid. m.p.: 75.0-76.5 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 10.35 (s, 1H), 8.04 (d, *J* = 16.0 Hz, 1H), 7.84 (d, *J* = 8.0 Hz, 1H), 7.75 (d, *J* = 8.0 Hz, 1H), 7.66 (d, *J* = 8.0 Hz, 1H), 7.59 (t, *J* = 8.0 Hz, 1H), 7.44-7.38 (m, 2H), 7.28 (t, *J* = 8.0 Hz, 1H), 7.01 (t, *J* = 8.0 Hz, 1H), 6.92 (d, *J* = 8.0 Hz, 1H), 3.89 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 192.5, 157.1, 140.7, 133.6, 132.9, 131.5, 129.3, 129.1, 127.3, 127.3, 127.1, 125.9, 124.8, 120.8, 110.9, 55.5; HRMS (EI) m/z calcd. For $C_{16}H_{14}O_2$ $[M]^+$ 238.0994, found 238.0988.

(*E*)-2-(3-methoxystyryl)benzaldehyde (1c)[4]



The crude product was purified by column chromatography on silica gel using hexane/ethyl acetate = 95:5 (v/v) as the eluent. Yield 71% (338 mg); Yellow solid. m.p.: 83.5-84.5 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 10.31 (s, 1H), 8.02 (d, *J* = 16.0 Hz, 1H), 7.82 (dd, *J* = 8.0, 1.2 Hz, 1H), 7.69 (d, *J* = 8.0 Hz, 1H), 7.57 (td, *J* = 8.0, 1.2 Hz, 1H), 7.42 (t, *J* = 8.0 Hz, 1H), 7.29 (t, *J* = 8.0 Hz, 1H), 7.15 (d, *J* = 8.0 Hz, 1H), 7.08 (t, *J* = 1.6 Hz, 1H), 7.01 (d, *J* = 16.0 Hz, 1H), 6.87-6.84 (m, 1H), 3.84 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 192.6, 159.9, 139.9, 138.3, 133.9, 133.7, 133.0, 132.2, 129.7, 127.6, 127.2, 125.1, 119.6, 114.0, 112.2, 55.3

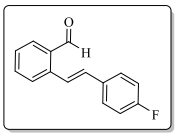
(*E*)-2-(4-methoxystyryl)benzaldehyde (1d)[2]



The crude product was purified by column chromatography on silica gel using hexane/ethyl acetate = 95:5 (v/v) as the eluent. Yield 78% (371 mg); Yellow solid. m.p.: 90.0-91.5 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 10.34 (s, 1H), 7.95 (d, *J* = 16.0 Hz, 1H), 7.86 (d, *J* = 8.0 Hz, 1H), 7.73 (d, *J* = 8.0 Hz, 1H), 7.60 (t, *J* = 8.0 Hz, 1H), 7.54-7.51 (m, 2H), 7.44 (t, *J* = 8.0 Hz, 1H), 7.05 (d, *J* = 16.0 Hz, 1H), 6.95-6.92 (m, 2H), 3.86 (s, 3H); ^{13}C NMR (100 MHz,

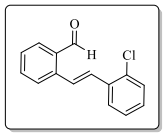
$CDCl_3$) δ 192.6, 159.8, 140.3, 133.6, 133.6, 132.1, 129.7, 128.2, 127.1, 126.9, 122.4, 114.1, 55.3

(*E*)-2-(4-fluorostyryl)benzaldehyde (1e)[2]



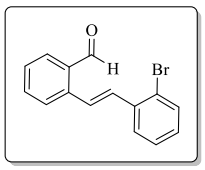
The crude product was purified by column chromatography on silica gel using hexane/ethyl acetate = 97:3 (v/v) as the eluent. Yield 76% (344 mg); Yellow liquid. ^{1}H NMR (400 MHz, $CDCl_3$) δ 10.3 (s, 1H), 7.98 (d, *J* = 16.0 Hz, 1H), 7.83 (dd, *J* = 8.0, 1.2 Hz, 1H), 7.70 (d, *J* = 8.0 Hz, 1H), 7.58(td, *J* = 8.0, 1.2 Hz, 1H), 7.55-7.51 (m, 2H), 7.45 (td, *J* = 8.0, 1.2 Hz, 1H), 7.10-7.03 (m, 2H), 7.01 (d, *J* = 16.0 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 192.7, 163.9, 161.4, 139.6, 133.7, 133.1, 132.9, 132.7, 132.5, 128.5, 128.4, 127.6, 127.0, 124.7, 124.7, 115.8, 115.6

(*E*)-2-(2-chlorostyryl)benzaldehyde (1f)



The crude product was purified by column chromatography on silica gel using hexane/ethyl acetate = 97:3 (v/v) as the eluent. Yield 82% (397 mg); White solid. m.p.: 91.5-92.5 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 10.29 (s, 1H), 8.07 (d, *J* = 16.0 Hz, 1H), 7.84 (d, *J* = 8.0 Hz, 1H), 7.78 (d, *J* = 8.0 Hz, 2H), 7.62 (t, *J* = 8.0 Hz, 1H), 7.49-7.44 (m, 2H), 7.40 (d, *J* = 8.0 Hz, 1H), 7.31 (t, *J* = 8.0 Hz, 1H), 7.25 (t, *J* = 8.0 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 192.7, 139.4, 135.0, 133.7, 133.6, 133.0, 132.8, 129.8, 129.6, 129.1, 128.0, 127.6, 127.4, 127.0; HRMS (EI) m/z calcd. For $C_{15}H_{11}ClO$ $[M]^+$ 244.0469, found 244.0467.

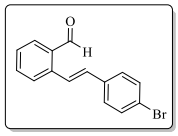
(*E*)-2-(2-Bromostyryl)benzaldehyde (1g)



The crude product was purified by column chromatography on silica gel using hexane/ethyl acetate = 97:3 (v/v) as the eluent. Yield 72% (412 mg); White solid. m.p.: 93.5-94.5 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 10.30 (s, 1H), 8.00 (d, *J* = 16.0 Hz, 1H), 7.84 (dd, *J* = 8.0, 1.3 Hz, 1H), 7.78-7.75 (m, 2H), 7.63-7.58 (m, 2H), 7.47 (td, *J* = 8.0, 1.0 Hz, 1H), 7.41 (d, *J* = 16.0 Hz, 1H), 7.36-7.32 (m, 1H), 7.15 (td, *J* = 8.0, 1.6 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 192.8, 139.4, 136.8, 133.8, 132.9, 132.3, 129.4, 128.0, 127.8,

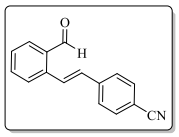
127.7, 127.5, 127.3, 124.3; HRMS (EI) m/z calcd. For $C_{15}H_{11}BrO$ $[M]^+$ 285.9993, found 285.9982.

(*E*)-2-(4-bromostyryl)benzaldehyde (1h)



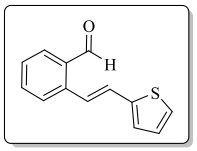
The crude product was purified by column chromatography on silica gel using hexane/ethyl acetate = 97:3 (v/v) as the eluent. Yield 80% (458 mg); White solid. m.p.: 64.5-65.5 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 10.31 (s, 1H), 8.09 (d, *J* = 20 Hz, 1H), 7.85 (dd, J = 8.0, 1.2 Hz, 1H), 7.73 (d, *J* = 8.0 Hz, 1H), 7.61 (td, *J* = 8.0, 1.2 Hz, 1H), 7.54-7.43 (m, 5H), 7.00 (d, *J* = 16.0 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 192.7, 139.3, 135.8, 133.7, 132.9, 132.9,132.4, 131.8, 128.4, 127.8, 127.0, 125.7, 122.0; HRMS (EI) m/z calcd. For $C_{15}H_{11}BrO$ $[M]^+$ 285.9993, found 286.0000.

(E)-4-(2-Formylstyryl)benzonitrile (1i)



The crude product was purified by column chromatography on silica gel using hexane/ethyl acetate = 95:5 (v/v) as the eluent. Yield 60% (280 mg); Pale yellow solid. m.p.: 65.5-66.5 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 10.25 (s, 1H), 8.23 (d, *J* = 16.0 Hz, 1H), 7.85-7.83 (m, 1H), 7.74 (d, *J* = 8.0 Hz, 1H), 7.65-7.60 (m, 5H), 7.54-7.49 (m, 1H), 7.04 (d, *J* = 16.0 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 193.0, 141.4, 138.4, 133.8, 133.1, 132.5, 131.4, 129.2, 128.5, 127.4, 127.2, 118.9, 111.2; HRMS (EI) m/z calcd. For $C_{16}H_{11}NO$ $[M]^+$ 233.0841, found 233.0834.

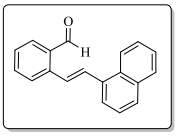
(E)-2-(2-(thiophen-2-yl)vinyl)benzaldehyde (1j)



The crude product was purified by column chromatography on silica gel using hexane/ethyl acetate = 97:3 (v/v) as the eluent. Yield 68% (291 mg); Pale yellow liquid. ^{1}H NMR (400 MHz, $CDCl_3$) δ 10.31 (s, 1H), 7.88-7.81 (m, 2H), 7.67 (d, *J* = 8.0 Hz, 1H), 7.55 (t, *J* = 8.0 Hz, 1H), 7.41 (t, *J* = 8.0 Hz, 1H), 7.26-7.25 (m, 1H), 7.18 (d, *J* = 16.0 Hz, 1H), 7.14-7.13 (m, 1H), 7.03-7.01 (m, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 192.4, 142.3, 139.4, 133.6, 132.7, 132.2, 127.7, 127.5, 127.1, 126.8, 126.7, 125.5, 124.0; HRMS (EI) m/z calcd.

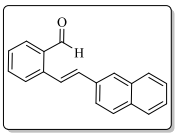
For $C_{13}H_{10}OS$ $[M]^+$ 214.0452, found 214.0443.

(*E*)-2-(2-(naphthalen-1-yl)vinyl)benzaldehyde (1k)[5]



The crude product was purified by column chromatography on silica gel using hexane/ethyl acetate = 96:4 (v/v) as the eluent. Yield 68% (351 mg); Yellow solid. m.p.: 80.0-81.5 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 10.34 (s, 1H), 8.20 (d, *J* = 8.0 Hz, 1H), 8.06 (d, *J* = 16.0 Hz, 1H), 7.87-7.78 (m, 6H), 7.63 (t, *J* = 8.0 Hz, 1H), 7.55-7.43 (m, 4H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 192.5, 140.1, 134.4, 133.7, 133.6, 133.0, 132.1, 131.3, 131.0, 128.6, 128.6, 127.7, 127.7, 127.5, 126.2, 125.8, 125.6, 124.2, 123.5

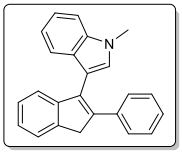
(*E*)-2-(2-(naphthalen-2-yl)vinyl)benzaldehyde (1l)[2]



The crude product was purified by column chromatography on silica gel using hexane/ethyl acetate = 96:4 (v/v) as the eluent. Yield 72% (372 mg); White solid. m.p.: 84.5-85.5 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 10.35 (s, 1H), 8.21 (d, *J* = 16.0 Hz, 1H), 7.90 (s, 1H), 7.86-7.77 (m, 6H), 7.62 (t, *J* = 8.0 Hz, 1H), 7.49-7.43 (m, 3H), 7.21 (s, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 192.7, 139.8, 134.4, 134.0, 133.6, 133.5, 133.3, 132.9, 132.5, 128.4, 128.1, 127.7, 127.6, 127.6, 127.3, 127.0, 126.4, 126.2, 125.0, 123.6

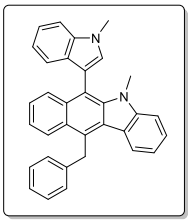
5. NMR Data of 1*H*-indenes

1-Methyl-3-(2-phenyl-1*H*-inden-3-yl)-1*H*-indole (3aa)



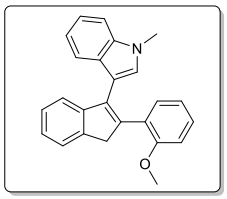
Yield 77% (62 mg); White solid. m.p.: 108.5-109.5 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.55-7.53 (m, 1H), 7.41-7.36 (m, 3H), 7.30-7.21 (m, 5H), 7.17-7.10 (m, 4H), 6.97 (t, *J* = 8.0 Hz, 1H), 3.97 (s, 2H), 3.84 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 147.6, 142.4, 141.1, 137.4, 132.8, 128.0, 127.97, 127.92, 127.0, 126.6, 126.3, 124.8, 123.5, 121.7, 121.2, 120.8, 119.2, 109.7, 109.2, 41.2, 32.9; HRMS (ESI) m/z calcd. For $C_{24}H_{20}N$ $[M+H]^+$ 322.1596, found 322.1597.

11-benzyl-5-methyl-6-(1-methyl-1*H*-indol-3-yl)-5*H*-benzo[*b*]carbazole (5aa)



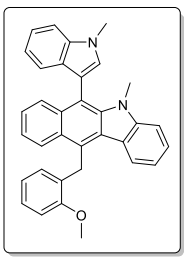
Yield 4% (5 mg); White solid. m.p.: 155.5-156.5 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.23 (d, *J* = 8.0 Hz, 1H), 8.18 (d, *J* = 8.0 Hz, 1H), 7.81 (d, *J* = 8.0 Hz, 1H), 7.52-7.46 (m, 2H), 7.37-7.31 (m, 4H), 7.28-7.25 (m, 5H), 7.21-7.13 (m, 3H), 7.08 (t, *J* = 8.0 Hz, 1H), 5.23 (s, 2H), 4.00 (s, 3H), 3.29 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 144.9, 139.9, 139.6, 136.7, 134.1, 130.7, 129.8, 129.7, 128.6, 128.4, 127.4, 126.7, 126.0, 125.9, 124.7, 124.6, 124.1, 123.6, 122.7, 122.1, 120.7, 119.8, 119.0, 111.5, 109.6, 109.3, 108.1, 35.1, 33.1, 31.7; HRMS (ESI) m/z calcd. For $C_{33}H_{27}N_2$ $[M+H]^+$ 451.2174, found 451.2176.

3-(2-(2-methoxyphenyl)-1*H*-inden-3-yl)-1-methyl-1*H*-indole (3ba)



Yield 49% (43 mg); Yellow solid. m.p.: 166.5-167.5 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.52 (d, *J* = 8.0 Hz, 1H), 7.40 (d, *J* = 8.0 Hz, 1H), 7.31-7.25 (m, 3H), 7.21-7.10 (m, 3H), 7.05 (d, *J* = 8.0 Hz, 1H), 6.99 (s, 1H), 6.94 (t, *J* = 8.0 Hz, 1H), 6.86 (t, *J* = 8.0 Hz, 1H), 6.67 (t, *J* = 8.0 Hz, 1H), 3.99 (s, 2H), 3.75 (s, 3H), 3.69 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 157.2, 146.5, 143.7, 140.2, 136.9, 133.8, 131.1, 128.0, 127.9, 127.3, 127.2, 125.9, 124.4, 123.3, 121.3, 121.0, 120.7, 120.2, 118.8, 110.9, 109.9, 108.9, 55.2, 42.2, 32.8; HRMS (ESI) m/z calcd. For $C_{25}H_{22}NO$ $[M+H]^+$ 352.1701, found 352.1701.

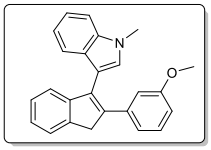
1-(2-methoxybenzyl)-5-methyl-6-(1-methyl-1*H*-indol-3-yl)-5*H*-benzo[b]carbazole (5ba)



Yield 8% (10 mg); Orange solid. m.p.: 216.5-217.5 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.18 (d, *J* = 8.0 Hz, 1H), 8.04 (d, *J* = 8.0 Hz, 1H), 7.81 (d, *J* = 8.0 Hz, 1H), 7.51 (d, *J* = 8.0 Hz,1H), 7.45 (t, *J* = 8.0 Hz, 1H), 7.34-7.26 (m, 4H), 7.24-7.15 (m, 3H), 7.11 (t, *J* = 8.0 Hz, 2H), 7.05 (t, *J* = 8.0 Hz, 1H), 6.69 (d, *J* = 6.3 Hz, 1H), 6.61 (t, *J* = 8.0 Hz, 1H), 5.11 (s, 2H), 4.12 (s, 3H), 3.99 (s, 3H), 3.27 (s, 3H); ^{13}C NMR

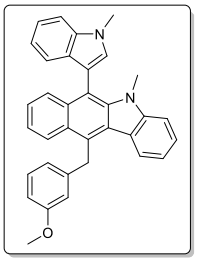
(100 MHz, $CDCl_3$) δ 157.5, 144.9, 139.9, 136.7, 134.0, 130.7, 130.0, 129.6, 128.8, 127.7, 127.7, 127.0, 126.5, 125.8, 124.7, 124.5, 124.2, 123.5, 122.8, 122.6, 122.0, 120.7, 120.7, 119.7, 118.9, 111.5, 109.7, 109.3, 109.2, 107.8; HRMS (EI) m/z calcd. For $C_{34}H_{28}N_2O$ $[M]^+$ 480.2202, found 480.2210.

3-(2-(3-methoxyphenyl)-1*H*-inden-3-yl)-1-methyl-1*H*-indole (3ca)



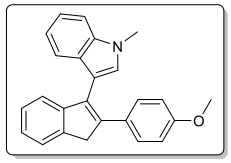
Yield 60% (53 mg); White solid. m.p.: 170.0-171.5 °C. ^1H NMR (400 MHz, $CDCl_3$) δ 7.56 (d, *J* = 8.0 Hz, 1H), 7.38 (d, *J* = 8.0 Hz, 1H), 7.33-7.22 (m, 5H), 7.15 (s, 1H), 7.10 (t, *J* = 8.0 Hz, 1H), 7.05-6.96 (m, 3H), 6.69 (d, *J* = 8.0 Hz, 1H), 3.99 (s, 2H), 3.87 (s, 3H), 3.41 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 159.1, 147.6, 142.3, 140.9, 138.4, 137.1, 133.0, 128.9, 127.8, 126.9, 126.3, 124.9, 123.4, 121.7, 121.1, 120.7, 120.4, 119.3, 113.0, 112.8, 109.7, 109.1, 54.6, 41.1, 32.9; HRMS (ESI) m/z calcd. For $C_{25}H_{22}NO$ $[M+H]^+$ 352.1701, found 352.1697.

11-(3-methoxybenzyl)-5-methyl-6-(1-methyl-1*H*-indol-3-yl)-5*H*-benzo[b]carbazole (5ca)



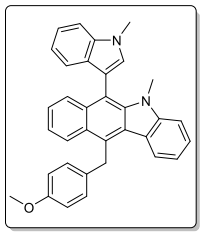
Yield 10% (12 mg); White solid. m.p.: 191.5-192.5 °C ^1H NMR (400 MHz, $CDCl_3$) δ 8.21 (d, *J* = 8.0 Hz, 2H), 8.17 (d, *J* = 8.0 Hz, 1H), 7.80-7.78 (m, 1H), 7.51-7.44 (m, *2*H), 7.36-7.27 (m, 5H), 7.23-7.19 (m, 2H), 7.17-7.11 (m, 2H), 7.08-7.05 (m, 1H), 6.91-6.88 (m, 2H), 6.72 (dd, *J* = 2.4, 8.0 Hz, 1H), 5.19 (s, 2H), 3.99 (s, 3H), 3.71 (s, 3H), 3.28 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 159.8, 44.9, 141.3, 139.9,136.6, 134.0, 130.7, 129.6, 129.6, 129.5, 127.4, 126.7, 125.9, 124.6, 124.5, 124.0, 123.5, 122.7, 122.0, 120.8, 120.6, 119.7, 118.9, 114.6, 111.4, 110.8, 109.5, 109.2, 108.0, 55.0, 35.0, 33.1, 31.6; HRMS (ESI) m/z calcd. For $C_{34}H_{29}N_2O$ $[M+H]^+$ 481.2280, found 481.2264.

3-(2-(4-Methoxyphenyl)-1*H*-inden-3-yl)-1-methyl-1*H*-indole (3da)



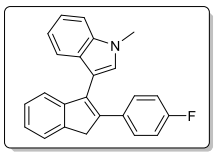
Yield 67% (59 mg); Brown solid. m.p.: 176.5-177.5 °C ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.53 (d, *J* = 8.0 Hz, 1H), 7.40-7.34 (m, 3H), 7.30 (d, *J* = 8.0 Hz, 1H), 7.27-7.19 (m, 4H), 7.11 (s, 1H), 7.01 (t, *J* = 8.0 Hz, 1H), 6.72-6.69 (m, 2H), 3.96 (s, 2H), 3.86 (s, 3H), 3.75 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 158.3, 147.9, 142.1, 140.9, 137.2, 131.2, 130.0, 129.1, 127.8, 127.1, 126.3, 124.5, 123.3, 121.7, 121.2, 120.5, 119.2, 113.5, 109.9, 109.1, 55.1, 41.2, 32.9; HRMS (ESI) m/z calcd. For $C_{25}H_{22}NO$ $[M+H]^+$ 352.1701, found 352.1705.

11-(4-methoxybenzyl)-5-methyl-6-(1-methyl-1H-indol-3-yl)-5H-benzo[b]carbazole (5da)



Yield 7% (9 mg); White solid. m.p.: 191.0-192.0 °C ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.22 (d, *J* = 8.0 Hz, 1H), 8.18 (d, *J* = 8.0 Hz, 1H), 7.79 (dd, *J* = 8.0, 1.0 Hz, 1H), 7.51-7.44 (m, 2H), 7.36-7.32 (m, 2H), 7.30-7.25 (m, 2H), 7.24-7.20 (m, 4H), 7.16-7.12 (m, 1H), 7.08-7.05 (m, 1H), 6.81-6.78 (m, 2H), 5.15 (s, 2H), 3.99 (s, 3H), 3.74 (s, 3H), 3.28 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 157.9, 144.9, 139.9, 136.7, 134.1, 131.6, 130.7, 130.3, 129.6, 129.3, 127.4, 126.7, 125.9, 124.5, 124.1, 123.6, 122.8, 122.7, 122.1, 120.7, 119.8, 119.0, 114.5, 111.5, 109.5, 109.3, 108.0, 55.2, 34.2, 33.1, 31.6; HRMS (ESI) m/z calcd. For $C_{34}H_{28}N_2O$ $[M]^+$ 480.2202, found 480.2198.

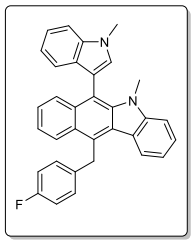
3-(2-(4-fluorophenyl)-1*H*-inden-3-yl)-1-methyl-1*H*-indole (3ea)



Yield 50% (42 mg); White solid. m.p.: 176.5-177.5 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.54-7.52 (m, 1H), 7.39-7.33 (m, 3H), 7.30-7.26 (m, 2H), 7.24-7.20 (m, 3H), 7.11 (s, 1H), 7.00-6.96 (m, 1H), 6.85-6.81 (m, 2H), 3.94 (s, 2H), 3.86 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 162.8, 160.3, 147.4, 142.2, 139.9, 137.2, 133.5, 133.4,

132.6, 129.5, 129.4, 127.9, 126.8, 126.4, 124.8, 123.4, 121.8, 121.0, 119.3, 115.0, 114.8, 109.5, 109.2; HRMS (EI) m/z calcd. For $C_{24}H_{18}NF$ $[M]^+$ 339.1423, found 339.1413.

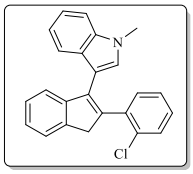
11-(4-fluorobenzyl)-5-methyl-6-(1-methyl-1*H*-indol-3-yl)-5*H*-benzo[b]carbazole (5ea)



Yield 6% (7 mg); White solid. m.p.: 227.5-229.0 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.17 (d, *J* = 8.0 Hz, 1H), 8.12 (d, *J* = 8.0 Hz, 1H), 7.81 (d, *J* = 8.0 Hz, 1H), 7.49-7.44 (m, 2H), 7.35-7.20 (m, 7H), 7.16 (s, 1H), 7.13 (t, *J* = 8.0 Hz, 1H), 7.05 (t, *J* = 8.0 Hz, 1H), 6.95-6.89 (m, 2H), 5.16 (s, 2H), 3.95 (s, 3H), 3.26 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 162.5, 160.1, 144.9, 139.9, 136.6, 135.1, 135.1, 134.0, 130.6, 129.7, 129.6, 129.6, 129.5, 127.2, 126.8,126.0, 124.6, 124.5, 1238, 123.3,122.8, 122.5, 122.1, 120.5, 119.8, 119.0, 115.4, 115.2, 111.3, 109.7, 109.3, 108.1, 34.2, 33.0, 31.6; HRMS (ESI) m/z calcd. For $C_{33}H_{25}N_2F$ $[M]^+$ 468.2002, found 468.1992.

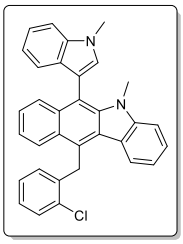
3-(2-(2-chlorophenyl)-1*H*-inden-3-yl)-1-methyl-1*H*-indole (3fa)

Yield 80% (71 mg); White solid. m.p.: 192.5-193.5 °C ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.55 (d, *J* =



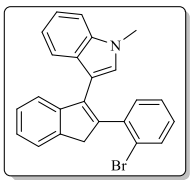
8.0 Hz, 1H), 7.45 (d, *J* = 8.0 Hz, 1H), 7.39 (d, *J* = 8.0 Hz, 1H), 7.36 (d, *J* = 8.0 Hz, 1H), 7.29-7.26 (m, 3H), 7.18 (t, *J* = 8.0 Hz, 1H), 7.12 (t, *J* = 8.0 Hz, 2H), 7.01-6.95 (m, 3H), 3.98 (s, 2H), 3.73 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 145.9, 123.4, 140.1, 137.6, 136.9, 135.6, 133.5, 131.7, 129.5, 128.2, 128.1, 127.2, 126.3, 126.2, 124.9, 123.5, 121.5, 121.3, 120.9, 119.1, 109.1, 109.0, 42.2, 32.8; HRMS (ESI) m/z calcd. For $C_{24}H_{19}NCl$ $[M+H]^+$ 356.1206, found 356.1203.

11-(2-chlorobenzyl)-5-methyl-6-(1-methyl-1*H*-indol-3-yl)-5*H*-benzo[b]carbazole (5fa)



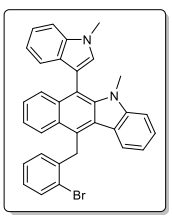
Yield 6% (7 mg); Yellow solid. m.p.: 182.5-183.5 °C ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.14 (d, *J* = 8.0 Hz, 1H), 7.98 (d, *J* = 8.0 Hz, 1H), 7.85 (dd, *J* = 1.0, 8.0 Hz, 1H), 7.58 (dd, *J* = 1.0, 8.0 Hz, 1H), 7.54 (d, *J* = 8.0, 1H), 7.51-7.47 (m, 1H), 7.41-7.31 (m, 4H), 7.29-7.28 (m, 1H), 7.26 (s, 1H), 7.19-7.11 (m, 2H), 7.12-7.09 (m, 1H), 6.99-6.92 (m, 1H), 6.76 (dd, *J* = 1.0, 8.0 Hz, 1H), 5.25 (s, 2H), 4.03 (s, 3H), 3.32 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 144.9, 139.9, 137.0, 136.6, 134.1, 134.0, 129.6, 129.5, 129.2, 128.4, 127.5, 127.4, 127.0, 126.8, 126.0, 124.8, 124.6, 123.8, 123.2, 122.9, 122.4, 122.1, 120.6, 119.8, 119.1, 111.3, 109.9, 109.3, 108.0, 33.1, 32.8, 31.6; HRMS (ESI) m/z calcd. For $C_{33}H_{26}N_2Cl$ $[M+H]^+$ 485.1785, found 485.1774.

3-(2-(2-bromophenyl)-1*H*-inden-3-yl)-1-methyl-1*H*-indole (3ga)



Yield 77% (77 mg); White solid. m.p.: 185.5-186.5 °C ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.55 (dd, *J* = 1.2, 8.0 Hz, 2H), 7.46-7.43 (m, 2H), 7.30-7.26 (m, 3H), 7.20 (dt, *J* = 1.0, 8.0 Hz, 1H), 7.12-7.00 (m, 4H), 6.94 (s, 1H), 3.98 (s, 2H), 3.72 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 145.9, 143.3, 141.7, 139.8, 136.8, 135.4, 132.6, 131.6, 128.3, 128.2, 127.2, 126.9, 126.2, 124.9, 123.7, 123.5, 121.4, 121.4, 121.0, 119.0, 109.0, 42.3, 32.8; HRMS (ESI) m/z calcd. For $C_{24}H_{19}NBr$ $[M+2+H]^+$ 402.0680, found 402.0683.

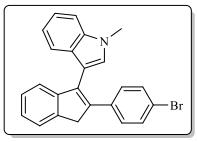
11-(2-bromobenzyl)-5-methyl-6-(1-methyl-1*H*-indol-3-yl)-5*H*-benzo[b]carbazole (5ga)



Yield 4% (5 mg); Brown solid. m.p.: 162.5-163.5 °C ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.11 (d, *J* = 8.0 Hz, 1H), 7.95 (d, *J* = 8.0 Hz, 1H), 7.84 (d, *J* = 8.0 Hz, 1H), 7.75 (d, *J* = 8.0, 1H), 7.52 (d, *J* = 8.0, 1H), 7.47 (t, *J* = 8.0 Hz, 1H), 7.39-7.24 (m, 5H), 7.23 (s, 1H), 7.15-7.05 (m, 3H), 6.96 (t, *J* = 8.0 Hz, 1H), 6.73 (d, *J* = 8.0 Hz, 1H), 5.20 (s, 2H),

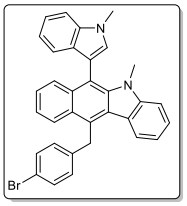
4.01 (s, 3H), 3.29 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 144.9, 139.8, 138.6, 136.6, 134.0, 132.5, 130.6, 129.6, 128.5, 127.8, 127.6, 127.4, 126.8, 126.0, 124.9, 124.8, 124.6, 123.8, 123.3, 122.9, 122.4, 122.1, 120.6, 119.8, 119.1, 111.3, 109.9, 109.3, 108.0, 35.8, 33.1, 31.6; HRMS (ESI) m/z calcd. For $C_{33}H_{26}N_2Br$ $[M+H]^+$ 529.1279, found 529.1286.

3-(2-(4-bromophenyl)-1*H*-inden-3-yl)-1-methyl-1*H*-indole (3ha)



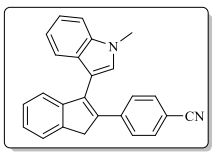
Yield 81% (81 mg); White solid. m.p.: 171.5-172.5 °C ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.59-7.57 (m, 1H), 7.43 (d, *J* = 8.0 Hz, 1H), 7.36-7.27 (m, 9H), 7.15 (s, 1H), 7.07-7.03 (m, 1H), 3.98 (s, 2H), 3.90 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 147.3, 142.3, 139.6, 137.3, 136.3, 133.6, 131.2, 129.5, 127.9, 126.7, 126.5, 125.2, 123.5, 121.9, 121.1, 120.9, 120.4, 119.4, 109.4, 109.3, 41.0, 33.0; HRMS (ESI) m/z calcd. For $C_{24}H_{19}NBr$ $[M+H]^+$ 400.0701, found 400.0699.

11-(4-bromobenzyl)-5-methyl-6-(1-methyl-1*H*-indol-3-yl)-5*H*-benzo[b]carbazole (5ha)



Yield 3% (4 mg); Yellow solid. m.p.: 135.5-136.5 °C ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.15 (d, *J* = 8.0 Hz, 1H), 8.10 (d, *J* = 8.0 Hz, 1H), 7.81 (d, *J* = 8.0 Hz, 1H), 7.51-7.46 (m, 2H), 7.37-7.24 (m, 7H), 7.19-7.12 (m, 4H), 7.06 (t, *J* = 8.0 Hz, 1H), 5.15 (s, 2H), 3.98 (s, 3H), 3.28 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 144.9, 139.8, 138.6, 136.6, 134.0, 131.6, 130.6, 130.1, 129.6, 128.9, 127.2, 126.8, 126.0, 124.6, 124.6, 123.7, 123.3, 122.8, 122.4, 122.1, 120.5, 119.8, 119.8, 119.0, 111.2, 109.8, 109.3, 108.1, 34.4, 33.0, 31.6; HRMS (EI) m/z calcd. For $C_{33}H_{25}N_2Br$ $[M]^+$ 528.1201, found 528.1194.

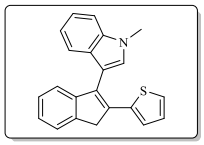
4-(3-(1-methyl-1*H*-indol-3-yl)-1*H*-inden-2-yl)benzonitrile (3ia)



Yield 78% (68 mg); Brown solid. m.p.: 209.0-210.5 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.57-7.55 (m, 1H), 7.47-7.45 (m, 2H), 7.41-7.38 (m, 3H), 7.36-7.33 (m, 1H), 7.30-7.26 (m, 3H),7.16-7.14 (m, 2H), 7.00-6.96 (m, 1H), 3.97 (s, 2H),

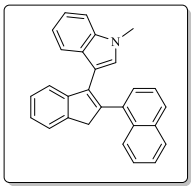
3.88 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 146.8, 142.5, 141.9, 138.3, 137.3, 136.2, 131.8, 128.1, 127.9, 126.6, 126.3, 125.8, 123.6, 122.1, 121.3, 120.8, 119.6, 119.2, 109.4, 109.3, 108.9, 40.7, 33.0; HRMS (ESI) m/z calcd. For $C_{25}H_{19}N_2$ $[M+H]^+$ 347.1548, found 347.1557.

1-methyl-3-(2-(thiophen-2-yl)-1*H*-inden-3-yl)-1*H*-indole (3ja)



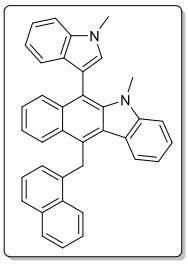
Yield 60% (49 mg); White solid. m.p.: 155.5-156.5 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.49-7.47 (m, 1H), 7.42 (d, *J* = 8.0 Hz, 1H), 7.33 (dd, *J* = 8.0, 0.7 Hz, 1H), 7.28-7.24 (m, 1H), 7.21-7.18 (m, 3H), 7.14-7.11 (m, 1H), 7.09-7.08 (m, 1H), 7.06-7.02 (m, 2H), 6.89-6.86 (m, 1H), 4.01 (s, 2H), 3.90 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 147.7, 141.4, 140.0, 137.4, 136.2, 132.0, 128.4, 127.1, 126.5, 126.5, 125.1, 124.9, 124.9, 123.3, 121.8, 120.8, 120.6, 119.3, 109.2, 108.6, 41.0, 33.0; HRMS (EI) m/z calcd. For $C_{22}H_{17}NS$ $[M]^+$ 327.1082, found 327.1071.

1-methyl-3-(2-(naphthalen-1-yl)-1*H*-inden-3-yl)-1*H*-indole (3ka)



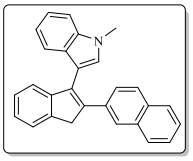
Yield 56% (52 mg); Orange solid. m.p.: 236.5-237.5 °C ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.90 (d, *J* = 8.0 Hz, 1H), 7.79 (d, *J* = 8.0 Hz, 1H), 7.72 (t, *J* = 8.0 Hz, 1H), 7.58 (d, *J* = 8.0 Hz, 1H), 7.50 (d, *J* = 8.0 Hz, 1H), 7.46 (d, *J* = 8.0 Hz, 1H), 7.39-7.24 (m, 6H), 7.19 (d, *J* = 8.0 Hz, 1H), 7.14 (t, *J* = 8.0 Hz, 1H), 6.96 (t, *J* = 8.0 Hz, 1H), 6.73 (s, 1H), 3.99 (s, 2H), 3.56 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 146.4, 143.3, 141.2, 136.7, 136.7, 135.5, 133.5, 132.1, 128.1, 128.0, 127.2, 127.2, 127.0, 126.3, 126.2, 125.6, 125.5, 125.2, 124.7, 123.5, 121.4, 121.3, 121.0, 118.9, 109.3, 109.0, 44.4, 32.6; HRMS (ESI) m/z calcd. For $C_{28}H_{22}N$ $[M+H]^+$ 372.1752, found 372.1762.

5-methyl-6-(1-methyl-1*H*-indol-3-yl)-11-(naphthalen-1-ylmethyl)-5*H*-benzo[*b*]carbazole (5ka)



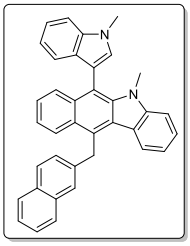
Yield 9% (11 mg); White solid. m.p.: 201.0-202.5 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.57 (d, *J* = 8.0 Hz, 1H), 8.10-8.08 (m, 1H), 7.98 (d, *J* = 8.0 Hz, 1H), 7.86-7.83 (m, 1H), 7.80-7.70 (m, 3H), 7.64 (t, *J* = 8.0 Hz, 1H), 7.51 (d, *J* = 8.0, 1H), 7.41-7.27 (m, 6H), 7.23-7.21 (m, 1H), 7.14-7.07 (m, 2H), 6.93 (t, *J* = 8.0 Hz, 1H), 6.86 (d, *J* = 8.0 Hz, 1H), 5.58 (s, 2H), 4.00 (s, 3H), 3.30 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 144.9, 139.9, 136.7, 134.6, 134.0, 133.8, 132.1, 130.7, 129.6, 129.2, 129.0, 127.6, 126.8, 126.7, 126.3, 26.0, 125.6, 125.3, 125.0, 124.6, 124.1, 123.4, 123.2, 122.7, 122.5, 122.1, 120.6, 119.8, 119.0, 111.4, 109.6, 109.3, 107.9, 33.1, 32.2, 31.6; HRMS (EI) m/z calcd. For $C_{37}H_{28}N_2$ $[M]^+$ 500.2252, found 500.2241.

1-methyl-3-(2-(naphthalen-2-yl)-1*H*-inden-3-yl)-1*H*-indole (3la)



Yield 57% (53 mg); White solid. m.p.: 176.5-177.5 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.92 (s, 1H), 7.71-7.67 (m, 2H), 7.57-7.55 (m, 1H), 7.48-7.31 (m, 7H), 7.26-7.24 (m, 3H), 7.10 (s, 1H), 6.98-6.94 (m, 1H), 4.09 (s, 2H), 3.83 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 147.5, 142.4, 140.9, 137.2, 135.1, 133.4, 133.3, 132.2, 128.1, 127.9, 127.4, 127.1, 126.5, 126.4, 126.4, 125.8, 125.6, 124.9, 123.4, 121.7, 121.2, 120.9, 119.3, 109.7, 109.2, 41.3, 32.9; HRMS (EI) m/z calcd. For $C_{28}H_{21}N$ $[M]^+$ 371.1674, found 371.1667.

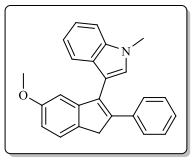
5-methyl-6-(1-methyl-1*H*-indol-3-yl)-11-(naphthalen-2-ylmethyl)-5*H*-benzo[b]carbazole (5la)



Yield 7 % (9 mg); White solid. m.p.: 166.5-167.5 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.25 (d, *J* = 8.0 Hz, 1H), 8.16 (d, *J* = 8.0 Hz, 1H), 7.84-7.79 (m, 3H), 7.60-7.56 (m, 3H), 7.50 (d, *J* = 8.0, 1H), 7.44 (t, *J* = 8.0 Hz, 1H), 7.38-7.26 (m, 7H), 7.23

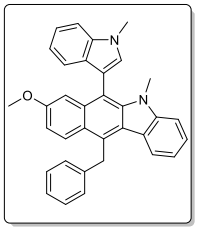
(s, 1H), 7.10-7.07 (m, 2H), 5.37 (s, 2H), 3.99 (s, 3H), 3.30 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 144.9, 139.9, 137.2, 136.6, 134.1, 133.7, 132.1, 130.7, 129.6, 129.5, 128.2, 127.7, 127.5, 127.5,127.1, 126.7, 126.4, 125.9, 125.7, 125.1, 124.7, 124.6, 124.1, 123.5, 122.7, 122.6, 122.1, 120.6, 119.8, 119.0, 111.4, 109.6, 109.3, 108.0, 35.3, 33.1, 31.6; HRMS (EI) m/z calcd. For $C_{37}H_{28}N_2$ $[M]^+$ 500.2252, found 500.2243.

3-(5-methoxy-2-phenyl-1*H*-inden-3-yl)-1-methyl-1*H*-indole (3ma)



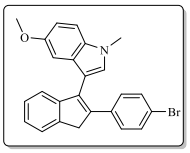
Yield 57% (50 mg); White solid. m.p.: 174.5-175.8 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.42 (d, *J* = 8.0 Hz, 1H), 7.40-7.37 (m, 3H), 7.27 (d, *J* = 8.0 Hz, 1H), 7.23-7.21 (m, 1H), 7.17-7.11 (m, 4H), 7.00 (t, *J* = 8.0 Hz, 1H), 6.87 (d, *J* = 2.5 Hz, 1H), 6.81 (dd, *J* = 8.0, 2.5 Hz, 1H), 3.92 (s, 2H), 3.85 (s, 3H), 3.74 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 159.0, 149.0, 142.5, 137.3, 137.2, 128.0, 127.9, 127.8, 126.9, 126.5, 123.8, 121.6, 121.1, 119.2, 110.8, 109.6, 109.1, 106.5, 55.5, 40.4, 32.9; HRMS (EI) m/z calcd. For $C_{25}H_{21}NO$ $[M]^+$351.1623, found 351.1619.

11-benzyl-9-methoxy-5-methyl-6-(1-methyl-1*H*-indol-3-yl)-5*H*-benzo[*b*]carbazole (5ma)



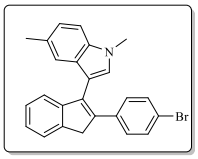
Yield 8% (10 mg); Yellow solid. m.p.: 174.5-175.5 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.22 (d, *J* = 8.0 Hz, 1H), 7.71 (d, *J* = 8.0 Hz, 1H), 7.49-7.43 (m, 3H), 7.36-7.25 (m, 6H), 7.23 (s, 1H), 7.19 (t, *J* = 4.0 Hz, 2H), 7.14 (t, *J* = 7.5 Hz, 1H), 7.08 (dt, *J* = 7.5, 1.0 Hz, 1H), 6.99 (dd, *J* = 8.0, 2.5 Hz, 1H), 5.16 (s, 2H), 3.98 (s, 3H), 3.82 (s, 3H), 3.26 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 155.4, 144.8, 139.6, 138.6, 136.6, 130.6, 129.7, 129.5, 128.6, 128.4, 128.4, 127.6, 126.6, 126.0, 124.9, 123.4, 122.6, 122.0, 120.6, 119.7, 118.7, 117.3, 111.4, 109.9, 109.2, 108.0, 102.3, 55.2, 35.4, 33.0, 31.6; HRMS (EI) m/z calcd. For $C_{34}H_{28}N_2O$ $[M]^+$ 480.2202 found 480.2206

3-(2-(4-bromophenyl)-1*H*-inden-3-yl)-5-methoxy-1-methyl-1*H*-indole (3hb)



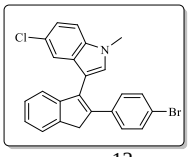
Yield 84% (90 mg); White solid. m.p.: 145.5-146.5 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.54 (dd, *J* = 7.0, 1.5 Hz, 1H), 7.36 (dd, *J* = 7.0, 1.5 Hz, 1H), 7.29-7.21 (m, 7H), 7.13 (s, 1H), 6.86 (dd, *J* = 8.0, 2.4 Hz, 1H), 6.55 (d, *J* = 2.5 Hz, 1H), 3.93 (s, 2H), 3.83 (s, 3H), 3.53 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 153.9, 147.1, 142.3, 139.0, 136.5, 133.5, 132.5, 131.1, 129.5, 128.4, 126.7, 126.4, 125.1, 123.5, 120.9, 120.3, 112.5, 110.0, 108.8, 102.5, 55.6, 41.0, 33.1; HRMS (ESI) m/z calcd. For $C_{25}H_{21}NOBr$ $[M+H]^+$430.0807, found 430.0800.

3-(2-(4-bromophenyl)-1*H*-inden-3-yl)-1,5-dimethyl-1*H*-indole (3hc)



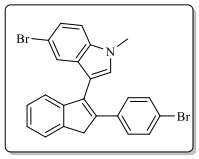
Yield 79% (82 mg); Red solid. m.p.: 96.5-97.5 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.53-7.51 (m, 1H), 7.29-7.22 (m, 8H), 7.08-7.01 (m, 3H), 3.92 (s, 2H), 3.80 (s, 3H), 2.31 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 147.3, 142.2, 139.5, 136.2, 135.6, 133.8, 131.0, 129.4, 128.6, 127.8, 127.0, 126.4, 125.0, 123.4, 123.4, 121.0, 120.5, 120.3, 108.9, 108.7, 40.9, 32.9, 21.3; HRMS (ESI) m/z calcd. For $[M+H]^+$, found. HRMS (ESI) m/z calcd. For $C_{25}H_{21}NBr$ $[M+H]^+$ 414.0857, found 414.0876.

3-(2-(4-bromophenyl)-1*H*-inden-3-yl)-5-chloro-1-methyl-1*H*-indole (3hd)



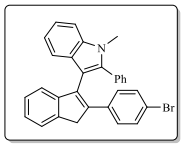
Yield 80% (87 mg); White solid. m.p.: 152.5-153.5 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.55-7.53 (m, 1H), 7.30-7.17 (m, 10H), 7.09 (s, 1H), 3.93 (s, 2H), 3.84 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 147.0, 142.1, 140.3, 135.9, 135.6, 132.8, 131.2, 129.4, 129.0, 127.8, 126.5, 125.4, 125.2, 123.5, 122.3, 120.7, 120.7, 120.2, 110.3, 109.1, 41.0, 33.1; HRMS (ESI) m/z calcd. For $C_{24}H_{18}NClBr$ $[M+H]^+$ 434.0311, found 434.0296.

5-bromo-3-(2-(4-bromophenyl)-1*H*-inden-3-yl)-1-methyl-1*H*-indole (3he)



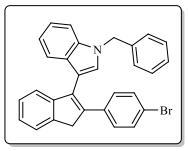
Yield 76% (91 mg); Yellow solid. m.p.: 158.5-159.5 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.53-7.51 (m, 1H), 7.36 (d, *J* = 1.7 Hz, 1H), 7.31-7.18 (m, 9H), 7.04 (s, 1H), 3.90 (s, 2H), 3.80 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 146.9, 142.1, 140.4, 135.9, 135.9, 132.8, 131.2, 129.4, 128.8, 128.4, 126.5, 125.2, 124.8, 123.5, 123.2, 120.7, 112.9, 110.8, 109.0, 41.0, 33.1; HRMS (ESI) m/z calcd. For $C_{24}H_{17}Br_2N$ $[M]^+$ 476.9728, found 476.9717.

3-(2-(4-bromophenyl)-1*H*-inden-3-yl)-1-methyl-2-phenyl-1*H*-indole (3hf)



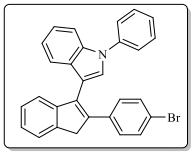
Yield 69% (82 mg); White solid. m.p.: 130.5-131.5 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.49-7.43 (m, 3H), 7.31 (t, *J* = 8.0 Hz, 1H), 7.21-7.08 (m, 9H), 6.94-6.92 (m, 2H), 6.86 (d, *J* = 8.0 Hz, 2H), 3.81-3.66 (m, 5H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 147.3, 142.2, 141.4, 138.8, 138.0, 136.3, 133.9, 131.4, 130.9, 129.9, 129.1, 127.9, 127.45, 127.41, 126.3, 124.8, 123.3, 122.0, 121.1, 120.6, 120.1, 119.8, 109.6, 108.2, 40.7, 31.3; HRMS (ESI) m/z calcd. For $C_{30}H_{23}NBr$ $[M+2+H]^+$ 478.0993, found 478.0985.

1-benzyl-3-(2-(4-bromophenyl)-1*H*-inden-3-yl)-1*H*-indole (3hg)



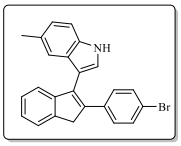
Yield 81% (97 mg); White solid. m.p.: 162.0-163.5 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.55-7.53 (m, 1H), 7.37-7.19 (m, 13H), 7.14-7.13 (m, 3H), 7.03 (t, *J* = 8.0 Hz, 1H), 5.37 (s, 2H), 3.94 (s, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 147.1, 142.2, 140.0, 137.3, 136.8, 136.1, 133.6, 131.1, 129.5, 128.8, 127.7, 127.4, 127.1, 126.7, 126.5, 125.1, 123.5, 122.1, 121.1, 121.0, 120.4, 119.6, 110.0, 109.8, 50.1, 40.9; HRMS (EI) m/z calcd. For $C_{30}H_{22}BrN$ $[M+2]^+$ 477.0915, found 477.0921.

3-(2-(4-bromophenyl)-1*H*-inden-3-yl)-1-phenyl-1*H*-indole (3hh)



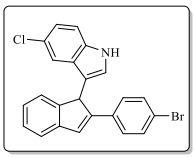
Yield 77% (89 mg); White solid. m.p.: 186.0-187.5 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.64 (d, *J* = 8.0 Hz, 1H), 7.56-7.54 (m, 5H), 7.41 (s, 1H), 7.39-7.35 (m, 2H), 7.33-7.23 (m, 8H), 7.08-7.04 (m, 1H), 3.97 (s, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 147.0, 142.7, 140.3, 139.5, 136.4, 136.0, 133.0, 131.2, 129.6, 129.4, 127.6, 126.9, 126.6, 126.5, 125.2, 124.3, 123.5, 122.7, 121.3, 120.9, 120.4, 111.9, 110.6, 41.1; HRMS (EI) m/z calcd. For $C_{29}H_{20}NBr$ $[M+2]^+$ 463.0759, found 463.0752.

3-(2-(4-bromophenyl)-1*H*-inden-3-yl)-5-methyl-1*H*-indole (3hi)



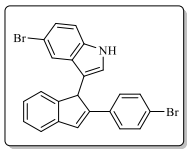
Yield 70% (70 mg); White solid. m.p.: 166.5-167.5 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.20 (brs, 1H), 7.55-7.53 (m, 1H), 7.34 (d, *J* = 8.0 Hz, 1H), 7.28-7.23 (m, 7H), 7.15 (d, *J* = 2.5 Hz, 1H), 7.08-7.04 (m, 2H), 3.94 (s, 2H), 2.32 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ147.3, 142.1, 140.0, 136.1, 134.6, 133.8, 131.1, 129.4, 129.1, 126.6, 126.5, 125.1, 124.0, 123.4, 123.3, 121.0, 120.4, 110.7, 110.5, 40.9, 21.4; HRMS (ESI) m/z calcd. For $C_{24}H_{19}NBr$ $[M+2+H]^+$ 402.0680, found 402.0668.

3-(2-(4-bromophenyl)-1*H*-inden-3-yl)-5-chloro-1*H*-indole (4hj)



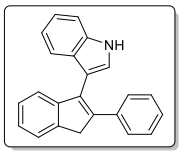
Yield 61% (64 mg); White solid. m.p.: 215.5-216.5 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.96 (brs, 1H), 7.46-7.44 (m, 1H), 7.39-7.32 (m, 6H), 7.28 (d, *J* = 8.0 Hz, 2H), 7.19 (d, *J* = 8.0 Hz, 1H), 7.12-7.06 (m, 2H), 7.00 (d, *J* = 2.0 Hz, 2H), 5.19 (s, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 148.2, 148.0, 142.6, 134.7, 134.1, 131.5, 128.2, 128.1, 127.6, 127.1, 125.7, 125.3, 123.6, 123.5, 122.5, 121.3, 121.1, 118.6, 113.4, 112.1, 47.4; HRMS (ESI) m/z calcd. For $C_{23}H_{16}NClBr$ $[M+H]^+$ 420.0155, found 420.0157.

5-bromo-3-(2-(4-bromophenyl)-1*H*-inden-3-yl)-1*H*-indole (4hk)



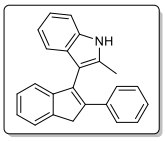
Yield 64% (74 mg); White solid. m.p.: 224.5-225.5 °C. ^{1}H NMR (400 MHz, DMSO) δ 11.11 (brs, 1H), 7.61 (d, *J* = 8.0 Hz, 3H), 7.53 (d, *J* = 8.0 Hz, 1H), 7.48-7.44 (m, 3H), 7.29-7.23 (m, 2H), 7.17 (d, *J* = 8.0 Hz, 1H), 7.12 (dd, *J* = 8.0, 0.6 Hz, 1H), 7.08-7.04 (m, 2H), 5.46 (s, 1H); ^{13}C NMR (100 MHz, DMSO) δ 149.0, 148.9, 143.1, 135.6, 134.7, 11.7, 128.9, 128.3, 128.0, 127.3, 126.4, 125.8, 123.9, 123.8, 121.7, 121.0, 120.8, 113.9, 111.6, 111.3, 55.3, 47.6; HRMS (EI) m/z calcd. For $C_{23}H_{15}Br_2N$ $[M]^+$ 462.9571, found 462.9568.

3-(2-Phenyl-1*H*-inden-3-yl)-1*H*-indole (3al)



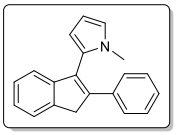
Yield 71% (55 mg); White solid. m.p.: 203-204.5 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.23 (brs, 1H), 7.55-7.53 (m, 1H), 7.43 (d, *J* = 8.0 Hz, 1H), 7.39-7.37 (m, 2H), 7.30-7.18 (m, 6H), 7.16-7.08 (m, 3H), 6.99 (t, *J* = 8.0 Hz, 1H), 3.98 (s, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 147.5, 142.4, 141.6, 137.2, 136.3, 132.8, 128.1, 127.9, 126.7, 126.6, 126.4, 124.9, 123.5, 123.3, 122.2, 121.0, 120.8, 119.7, 111.4, 111.0, 41.2; HRMS (ESI) m/z calcd. For $C_{23}H_{18}N$ $[M+H]^+$ 308.1439, found 308.1436.

2-Methyl-3-(2-phenyl-1H-inden-3-yl)-1H-indole (3am)



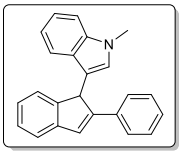
Yield 68% (55 mg); White solid. m.p.: 75.5-76.5 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.95 (brs, 1H), 7.56-7.54 (m, 1H), 7.37-7.34 (m, 4H), 7.24-7.20 (m, 2H), 7.19-7.10 (m, 5H), 7.05-7.00 (m, 1H), 4.11 (d, *J* = 24 Hz, 1H), 3.89 (d, *J* = 24 Hz, 1H), 2.00 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 147.5, 142.3, 142.1, 137.5, 135.8, 133.1, 132.5, 128.3, 128.2, 127.5, 126.6, 126.4, 124.8, 123.4, 121.4, 121.3, 120.1, 119.6, 110.3, 108.1, 40.9, 12.5; HRMS (ESI) m/z calcd. For $C_{24}H_{20}N$ $[M+H]^+$ 322.1596, found 322.1602.

1-methyl-2-(2-phenyl-1*H*-inden-3-yl)-1*H*-pyrrole (3an)



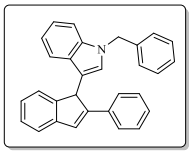
Yield 24% (17 mg); Brown gummy. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.52 (d, *J* = 8.0 Hz, 1H), 7.35-7.20 (m, 8H), 6.73 (t, *J* = 4.0 Hz, 1H), 6.32-6.30 (m, 2H), 4.09 (d, *J* = 24 Hz, 1H), 3.80 (d, *J* = 24 Hz, 1H), 3.15 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 147.1, 142.8, 142.0, 136.8, 131.3, 128.5, 127.3, 127.2, 127.1, 126.6, 125.2, 123.5, 122.6, 120.8, 109.0, 108.1, 40.6, 34.0 ; HRMS (ESI) m/z calcd. For $C_{20}H_{18}N$ $[M+H]^+$272.1439, found 272.1436.

1-Methyl-3-(2-phenyl-1*H*-inden-1-yl)-1*H*-indole (4aa)



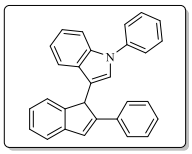
Yield 75% (60 mg); White solid. m.p.: 160.0-161.5 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.58-7.54 (m, 3H), 7.44 (d, *J* = 8.0 Hz, 1H), 7.39 (s, 1H), 7.33 (d, *J* = 8.0 Hz, 1H), 7.24-7.22 (m, 4H), 7.20-7.14 (m, 2H), 7.08-7.04 (m, 2H), 6.76 (s, 1H), 5.32 (s, 1H), 3.63 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 149.8, 149.3, 142.9, 137.2, 135.5, 128.4, 127.5, 127.4, 127.2, 126.83, 126.81, 126.7, 125.3, 123.6, 121.5, 121.0, 119.4, 118.9, 112.2, 109.2, 47.4, 32.6; HRMS (ESI) m/z calcd. For $C_{24}H_{19}N$ $[M]^+$321.1517, found 321.1512.

1-benzyl-3-(2-phenyl-1*H*-inden-1-yl)-1*H*-indole (4ag)



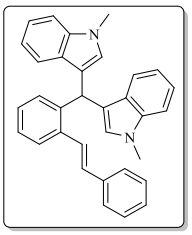
Yield 88% (87 mg); White solid. m.p.: 182.0-183.5 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.55 (d, *J* = 8.0 Hz, 2H), 7.45 (d, *J* = 8.0 Hz, 2H), 7.38 (d, *J* = 1.0 Hz, 1H), 7.35 (d, *J* = 8.0 Hz, 1H), 7.27-7.13 (m, 8H), 7.08 (t, *J* = 8.0 Hz, 2H), 6.99 (t, *J* = 8.0 Hz, 1H), 6.91 (s, 1H), 6.89-6.86 (m, 2H), 5.31 (s, 1H), 5.24-5.15 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 149.7, 148.8, 143.0, 137.6, 136.8, 135.4, 128.6, 128.2, 127.6, 127.3, 127.2, 127.1, 126.9, 126.8, 126.7, 126.3, 125.3, 123.6, 121.7, 121.0, 119.5, 119.1, 112.9, 109.6, 49.7, 47.7; HRMS (ESI) m/z calcd. For $C_{30}H_{24}N$ $[M+H]^+$ 398.1909, found 398.1915.

1-phenyl-3-(2-phenyl-1*H*-inden-1-yl)-1*H*-indole (4ah)



Yield 90% (86 mg); Orange solid. m.p.: 199.5-200.5 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.60 (d, *J* = 8.0 Hz, 2H), 7.51 (t, *J* = 8.0 Hz, 2H), 7.46-7.37 (m, 7H), 7.29-7.22 (m, 4H), 7.18-7.05 (m, 5H), 5.36 (s, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 149.5, 148.6, 143.1, 139.6, 136.3, 135.3, 129.4, 128.3, 127.6, 127.2, 126.9, 126.7, 126.1, 125.9, 125.3, 124.1, 123.7, 122.3, 121.0, 120.0, 119.7, 114.9, 110.5, 47.5; HRMS (ESI) m/z calcd. For $C_{29}H_{21}N$ $[M]^+$ 383.1674, found 383.1668.

1-benzyl-3-(2-phenyl-1*H*-inden-1-yl)-1*H*-indole (6aa)

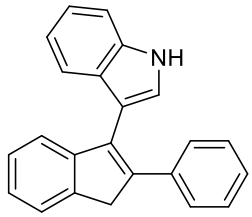


Yield 10% (12 mg); White solid. m.p.: 213.5-214.5 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.66 (d, *J* = 8.0 Hz, 1H), 7.53 (d, *J* = 16.0 Hz, 1H), 7.38 (d, *J* = 8.0 Hz, 2H), 7.30-7.11 (m, 12H), 7.03-6.96 (m, 3H), 6.54 (s, 2H), 6.24 (s, 1H), 3.67 (s, 6H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 142.0, 137.6, 137.5, 136.2, 130.4, 128.9, 128.7, 128.5, 127.5, 127.4, 127.38, 126.8, 126.5, 126.3, 126.0, 121.4, 119.9, 118.7, 117.7, 109.0, 36.3, 32.7; HRMS (ESI) m/z calcd. For $C_{33}H_{29}N_2$ $[M+H]^+$ 453.2330, found 453.2234.

5. Single crystal X-ray analytical data

Crystals of **3al, 4ag,** and **6aa** were obtained by a slow evaporation method using ethyl acetate as a solvent system at ambient temperature. Toluene was used to obtain the crystals of **5aa** by a slow evaporation method. X-ray reflections were collected using Mo Kα X-radiation (λ = 0.71073 Å) on the single crystals at 200 K using a Bruker Kappa APEX-II diffractometer. All the crystal structures were solved and refined using SHELX-97. The details of crystal data collection and data refinement parameters are given in Tables S1, S2, S3, and S4.

Ortep (ellipsoid counter 30% probability**) diagram and crystal data of compound 3al: CCDC Number 2298811**



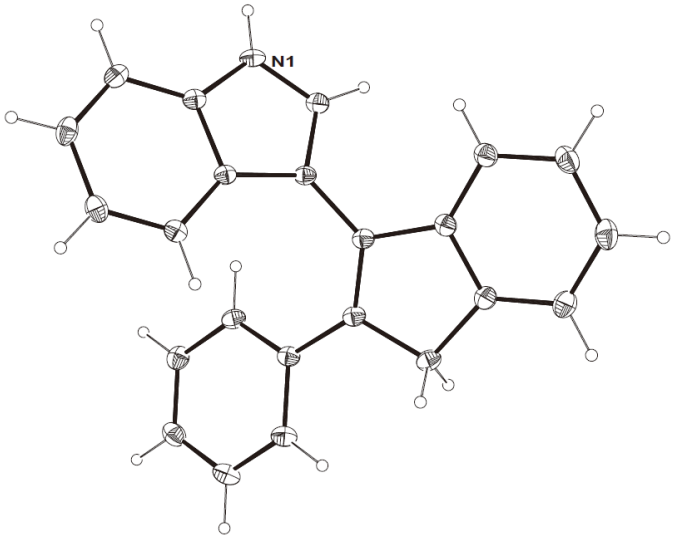


Table S1. Crystal data and structure refinement for d22733.

Identification code	d22733	
Empirical formula	C_{23} H_{17} N	
Formula weight	307.38	
Temperature	200(2) K	
Wavelength	0.71073 Å	
Crystal system	Monoclinic	
Space group	P 21	
Unit cell dimensions	a = 11.7440(9) Å	α= 90°.
	b = 5.7445(3) Å	β= 110.748(2)°.
	c = 12.5787(9) Å	γ = 90°.
Volume	793.57(9) $Å^3$	
Z	2	
Density (calculated)	1.286 Mg/m^3	
Absorption coefficient	0.074 mm^{-1}	
F(000)	324	
Crystal size	0.79 x 0.07 x 0.03 mm^3	
Theta range for data collection	2.95 to 25.12°.	
Index ranges	-14<=h<=13, -6<=k<=6, -15<=l<=13	
Reflections collected	10099	
Independent reflections	2786 [R(int) = 0.0370]	

Completeness to theta = 25.12°	98.8 %
Absorption correction	multi-scan
Max. and min. transmission	0.9978 and 0.9437
Refinement method	Full-matrix least-squares on F^2
Data/restraints/parameters	2786 / 1 / 217
Goodness-of-fit on F^2	1.041
Final R indices [I>2sigma(I)]	R1 = 0.0324, wR2 = 0.0791
R indices (all data)	R1 = 0.0387, wR2 = 0.0832
Absolute structure parameter	-1(3)
Largest diff. peak and hole	0.105 and -0.117 e.$Å^{-3}$

Ortep (ellipsoid counter 30% probability**) diagram and crystal data of compound 5aa: CCDC Number 2298812**

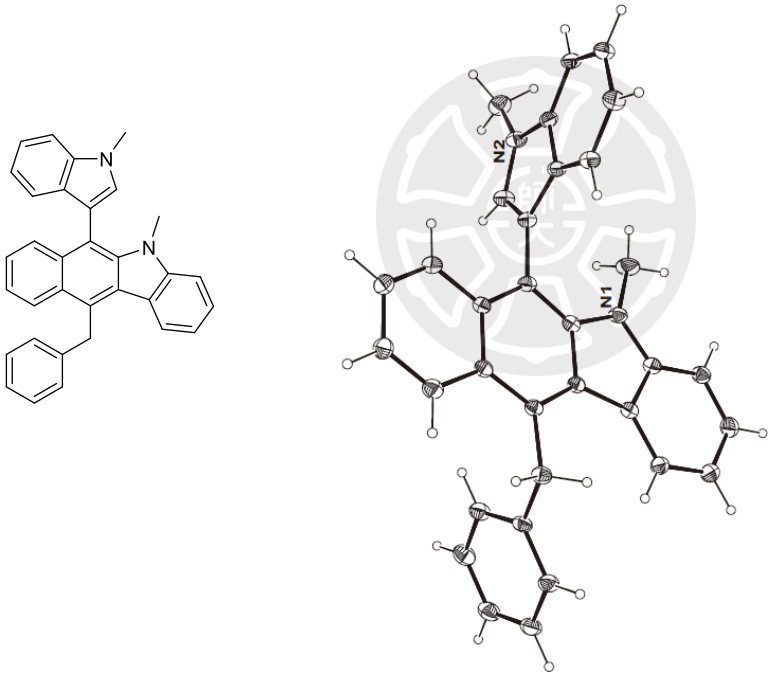


Table S2. Crystal data and structure refinement for d23600.

Identification code	d23600
Empirical formula	C_{33} H_{26} N_2
Formula weight	450.56
Temperature	200(2) K
Wavelength	0.71073 Å
Crystal system	Triclinic

Space group	P -1	
Unit cell dimensions	a = 9.4922(17) Å	α= 86.648(4)°.
	b = 9.8766(16) Å	β= 83.418(5)°.
	c = 12.783(3) Å	γ = 80.488(4)°.
Volume	1173.2(4) $Å^3$	
Z	2	
Density (calculated)	1.275 Mg/m^3	
Absorption coefficient	0.074 mm^{-1}	
F(000)	476	
Crystal size	0.09 x 0.08 x 0.01 mm^3	
Theta range for data collection	2.19 to 25.07°.	
Index ranges	-11<=h<=11, -11<=k<=11, -15<=l<=15	
Reflections collected	20716	
Independent reflections	4153 [R(int) = 0.0961]	
Completeness to theta = 25.07°	99.7 %	
Absorption correction	multi-scan	
Max. and min. transmission	0.9993 and 0.9934	
Refinement method	Full-matrix least-squares on F^2	
Data/restraints/parameters	4153 / 0 / 318	
Goodness-of-fit on F^2	1.026	
Final R indices [I>2sigma(I)]	R1 = 0.0551, wR2 = 0.1284	
R indices (all data)	R1 = 0.1095, wR2 = 0.1621	
Largest diff. peak and hole	0.224 and -0.198 $e.Å^{-3}$	

Ortep (ellipsoid counter 30% probability**) diagram and crystal data of compound 4ag: CCDC Number 2298813**

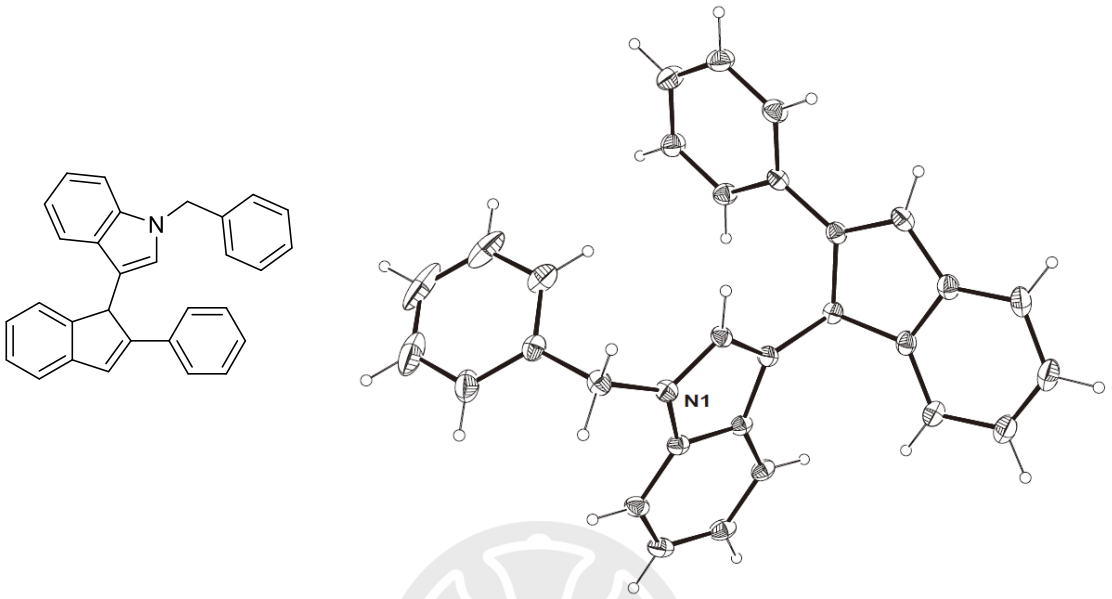
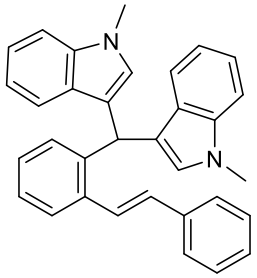


Table S3. Crystal data and structure refinement for d24632.

Identification code	d24632	
Empirical formula	C_{30} H_{23} N	
Formula weight	397.49	
Temperature	200(2) K	
Wavelength	0.71073 Å	
Crystal system	Orthorhombic	
Space group	P n a 21	
Unit cell dimensions	a = 9.4304(6) Å	α= 90°.
	b = 16.0120(11) Å	β= 90°.
	c = 14.3171(10) Å	γ = 90°.
Volume	2161.9(3) $Å^3$	
Z	4	
Density (calculated)	1.221 Mg/m^3	
Absorption coefficient	0.070 mm^{-1}	
F(000)	840	
Crystal size	0.62 x 0.07 x 0.03 mm^3	
Theta range for data collection	2.51 to 25.01°.	

Index ranges	-11<=h<=9, -19<=k<=19, -17<=l<=17
Reflections collected	26115
Independent reflections	3804 [R(int) = 0.1080]
Completeness to theta = 25.01°	99.9 %
Absorption correction	None
Max. and min. transmission	0.9979 and 0.9578
Refinement method	Full-matrix least-squares on F^2
Data/restraints/parameters	3804 / 1 / 280
Goodness-of-fit on F^2	1.067
Final R indices [I>2sigma(I)]	R1 = 0.0557, wR2 = 0.1192
R indices (all data)	R1 = 0.1052, wR2 = 0.1496
Absolute structure parameter	6(6)
Largest diff. peak and hole	0.165 and -0.244 e.Å^{-3}

Ortep (ellipsoid counter 30% probability**) diagram and crystal data of compound 6aa: CCDC Number 2298814**



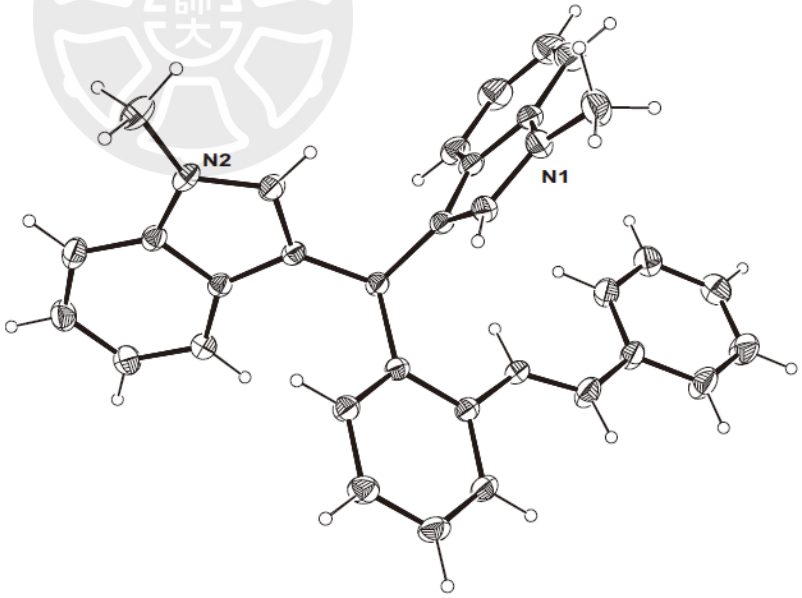
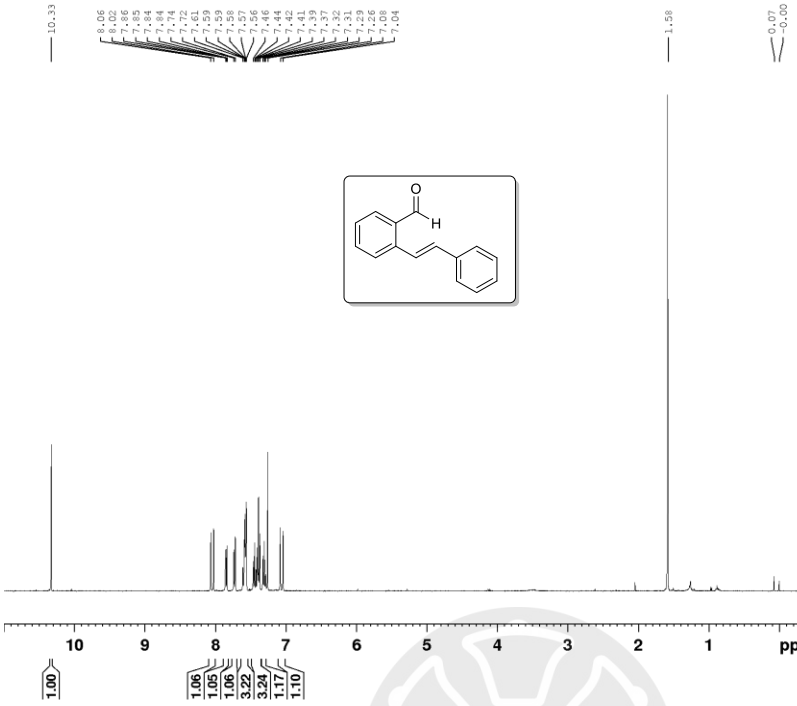


Table S4. Crystal data and structure refinement for d23609.

Identification code	d23609	
Empirical formula	C_{33} H_{28} N_2	
Formula weight	452.57	
Temperature	200(2) K	
Wavelength	0.71073 Å	
Crystal system	Triclinic	
Space group	P -1	
Unit cell dimensions	a = 8.817(3) Å	α= 101.527(12)°.
	b = 11.894(5) Å	β= 93.825(10)°.
	c = 12.370(4) Å	γ = 97.448(11)°.
Volume	1254.7(8) $Å^3$	
Z	2	
Density (calculated)	1.198 Mg/m^3	
Absorption coefficient	0.070 mm^{-1}	
F(000)	480	
Crystal size	0.38 x 0.30 x 0.06 mm^3	
Theta range for data collection	2.17 to 25.17°.	
Index ranges	-10<=h<=10, -14<=k<=14, -14<=l<=14	
Reflections collected	27917	
Independent reflections	4486 [R(int) = 0.0833]	
Completeness to theta = 25.17°	99.5 %	
Absorption correction	multi-scan	
Max. and min. transmission	0.9958 and 0.9741	
Refinement method	Full-matrix least-squares on F^2	
Data/restraints/parameters	4486 / 0 / 318	
Goodness-of-fit on F^2	1.031	
Final R indices [I>2sigma(I)]	R1 = 0.0486, wR2 = 0.1182	
R indices (all data)	R1 = 0.0872, wR2 = 0.1441	
Largest diff. peak and hole	0.170 and -0.169 $e.Å^{-3}$	

6. NMR Spectra Copies

(*E*)-2-styrylbenzaldehyde (1a)

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Current Data Parameters
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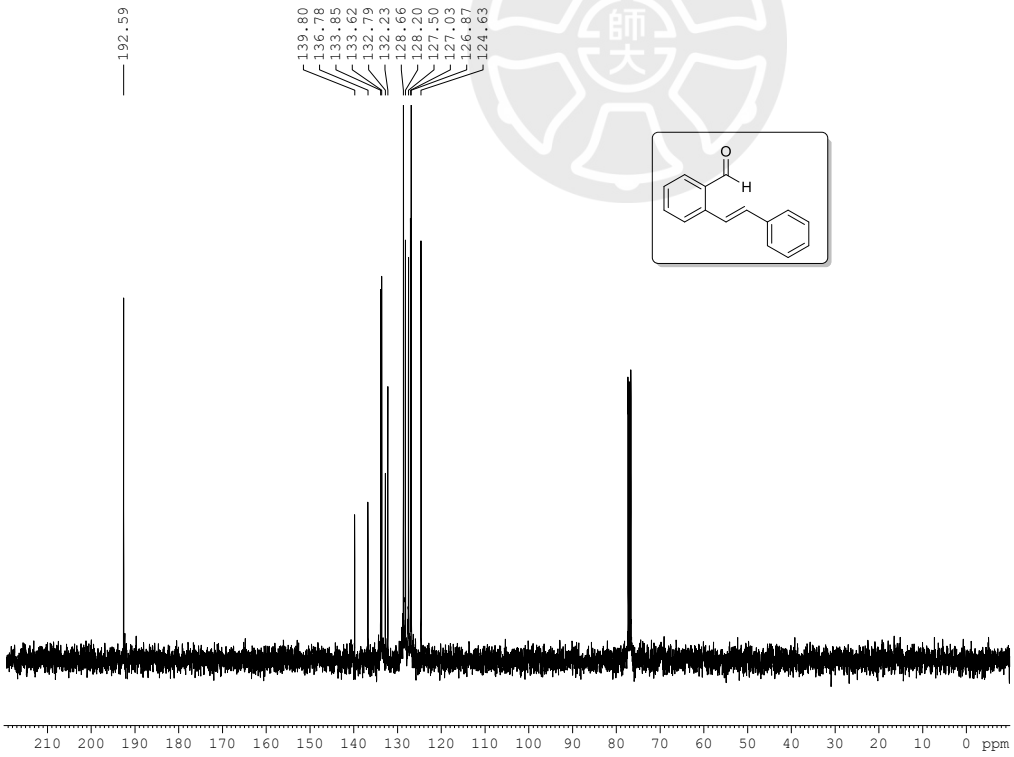
EXPNO                   3
PROCNO                  1

F2 - Acquisition Parameters
Date_            20200905
Time                10.27
INSTRUM             spect
PROBHD    5 mm BBO BB-1H
PULPROG              zg30
TD                  32768
SOLVENT             CDCl3
NS                     16
DS                      0
SWH             7246.377 Hz
FIDRES          0.221142 Hz
AQ              2.2609921 sec
RG                  322.5
DW                 69.000 usec
DE                  6.50 usec
TE                 297.6 K
D1              2.00000000 sec
TD0                     1

======== CHANNEL f1 ========
NUC1                   1H
P1                  15.00 usec
PL1                 11.10 dB
SFO1         400.1324008 MHz

F2 - Processing parameters
SI                  16384
SF            400.1300118 MHz
WDW                    EM
SSB                     0
LB                   0 Hz
GB                      0
PC                   1.00
```

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EXPNO                   4
PROCNO                  1
Date_            20230712
Time                7.32
INSTRUM             spect
PROBHD    5 mm PABBO BB/
PULPROG            zgpg30
TD                  32768
SOLVENT             CDCl3
NS                      9
DS                      0
SWH             24038.461 Hz
FIDRES           0.733596 Hz
AQ               0.6816244 sec
RG                  198.09
DW                 20.800 usec
DE                  6.50 usec
TE                 298.9 K
D1              2.00000000 sec
D11             0.03000000 sec
TD0                     1

======== CHANNEL f1 ========
SFO1         100.6228298 MHz
NUC1                  13C
P1                  10.00 usec
SI                  32768
SF           100.6127860 MHz
WDW                    EM
SSB                     0
LB                   2.00 Hz
GB                      0
PC                   1.00
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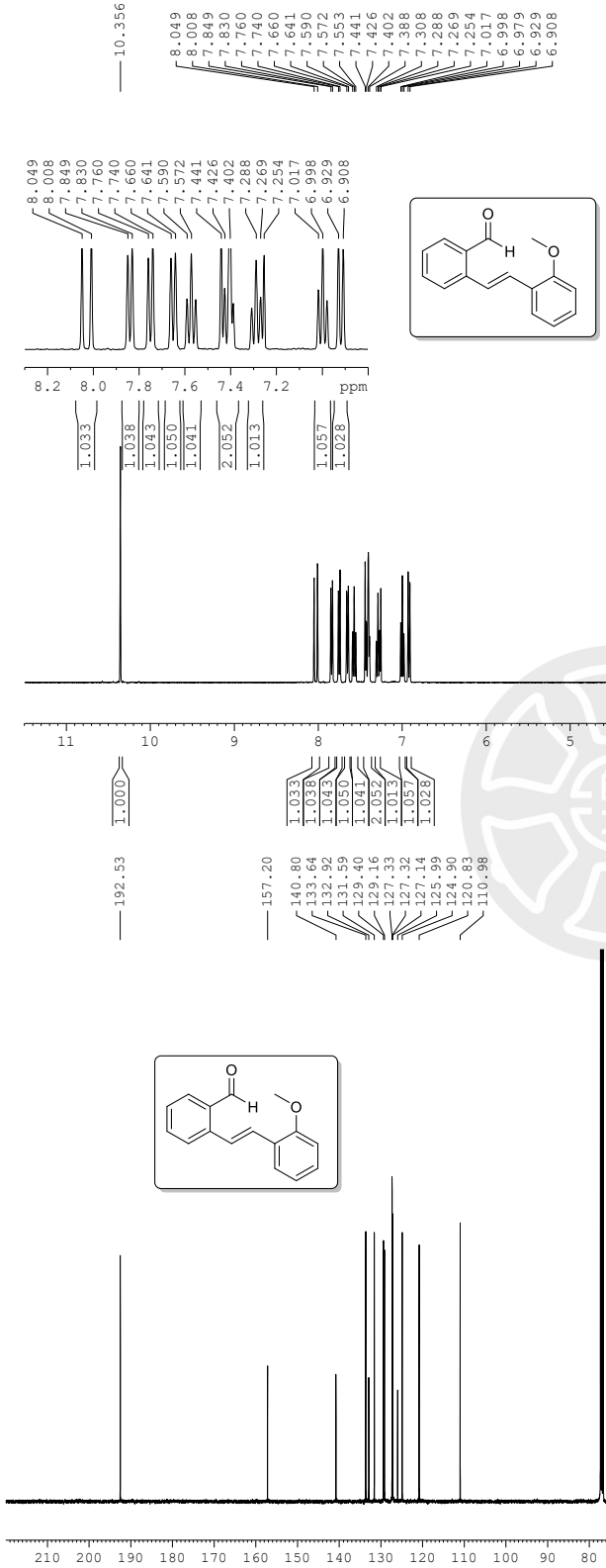
(*E*)-2-(2-methoxystyryl)benzaldehyde (1b)




```
NAME      Aldehyde+indole
EXPNO                  81
PROCNO                  1
Date_            20211118
Time                22.41
INSTRUM             spect
PROBHD   5 mm BBO BB-1H
PULPROG              zg30
TD                  32768
SOLVENT             CDCl3
NS                     16
DS                      0
SWH             7246.377 Hz
FIDRES          0.221142 Hz
AQ              2.2611110 sec
RG                    114
DW                 69.000 usec
DE                  6.50 usec
TE                 300.6 K
D1              2.00000000 sec
TD0                     1

======== CHANNEL f1 ========
NUC1                   1H
P1                  15.00 usec
PL1                 11.10 dB
SFO1          400.1324008 MHz
SI                  16384
SF            400.1300118 MHz
WDW                    EM
SSB                     0
LB                   0.00 Hz
GB                      0
PC                   1.00
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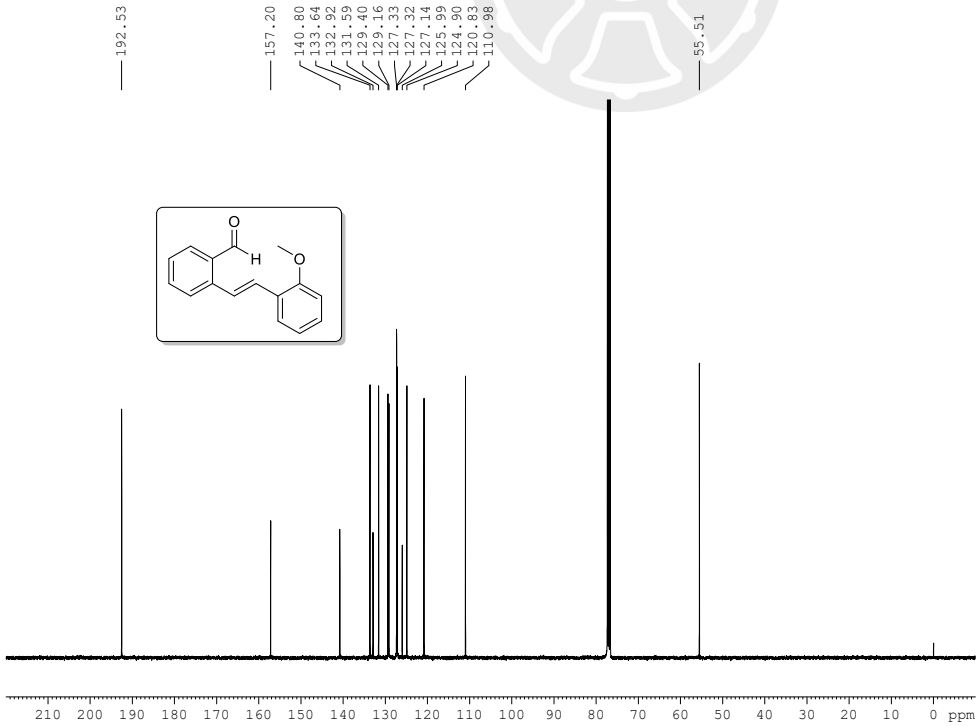


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NAME      Aldehyde+indole
EXPNO                  82
PROCNO                  1
Date_            20211118
Time                22.47
INSTRUM             spect
PROBHD   5 mm BBO BB-1H
PULPROG            zgpg30
TD                  32768
SOLVENT             CDCl3
NS                   5000
DS                      0
SWH            24038.461 Hz
FIDRES          0.733596 Hz
AQ              0.6816452 sec
RG                    32768
DW                 20.800 usec
DE                  6.50 usec
TE                 301.2 K
D1              2.00000000 sec
D11             0.03000000 sec
TD0                     1

======== CHANNEL f1 ========
NUC1                  13C
P1                  10.00 usec
PL1                  3.80 dB
SFO1          100.6233325 MHz

======== CHANNEL f2 ========
CPDPRG2           waltz16
NUC2                   1H
PCPD2               90.00 usec
PL2                 10.20 dB
PL12                26.00 dB
PL13                29.00 dB
SFO2          400.1316005 MHz
SI                  32768
SF            100.6127723 MHz
WDW                    EM
SSB                     0
LB                   1.00 Hz
GB                      0
PC                   1.00
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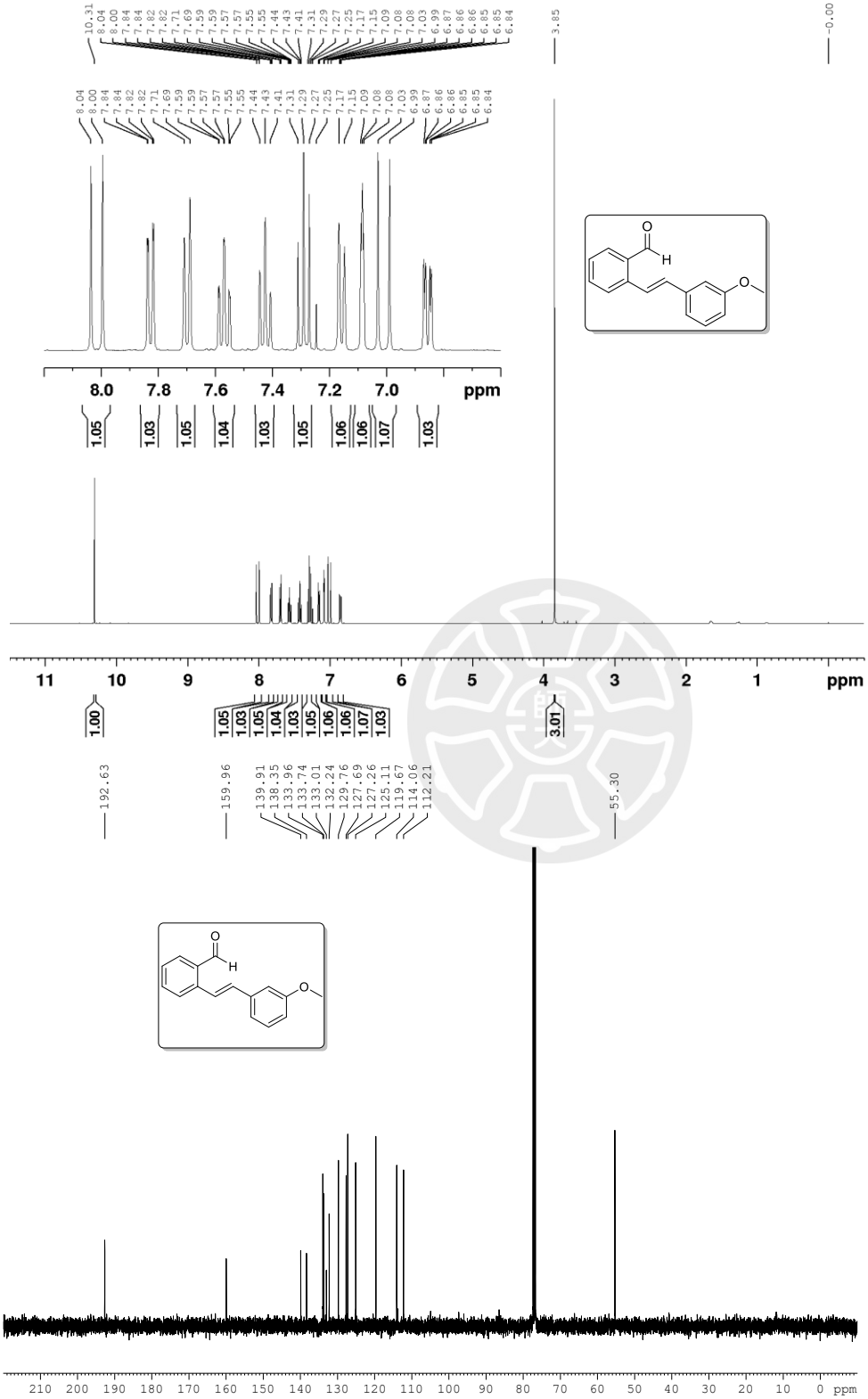
(*E*)-2-(3-methoxystyryl)benzaldehyde (1c)

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PROCNO                   1

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PULPROG              zg30
TD                  32768
SOLVENT             CDCl3
NS                     16
DS                      0
SWH            7211.539 Hz
FIDRES         0.220079 Hz
AQ            2.2719147 sec
RG                  63.58
DW                69.333 usec
DE                 10.06 usec
TE                 298.2 K
D1            2.00000000 sec
TD0                     1

======== CHANNEL f1 ========
SFO1        400.1324008 MHz
NUC1                   1H
P1                  15.00 usec
PLW1        13.50000000 W

F2 - Processing parameters
SI                  16384
SF          400.1300151 MHz
WDW                    EM
SSB                     0
LB                      0 Hz
GB                      0
PC                   1.00
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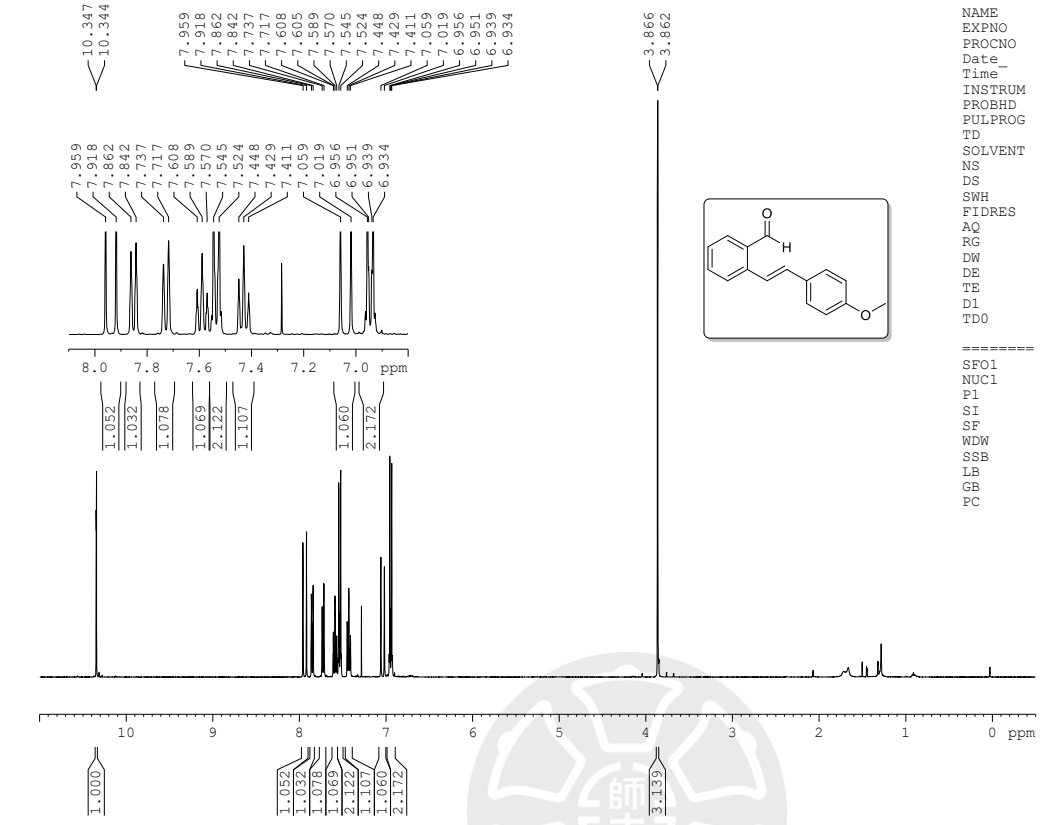
NAME      Aldehyde+indole
EXPNO                  84
PROCNO                  1
Date_           20211001
Time                20.34
INSTRUM             spect
PROBHD   5 mm BBO BB-1H
PULPROG           zgpg30
TD                  32768
SOLVENT             CDCl3
NS                    129
DS                      0
SWH           24038.461 Hz
FIDRES         0.733596 Hz
AQ            0.6816452 sec
RG                  32768
DW                20.800 usec
DE                 6.50 usec
TE                 298.4 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0                     1

======== CHANNEL f1 ========
NUC1                  13C
P1                  10.00 usec
PL1                  3.80 dB
SFO1        100.6233325 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                   1H
PCPD2               90.00 usec
PL2                 10.20 dB
PL12                26.00 dB
PL13                29.00 dB
SFO2        400.1316005 MHz
SI                  32768
SF          100.6127690 MHz
WDW                    EM
SSB                     0
LB                   1.00 Hz
GB                      0
PC                   1.00
```

(*E*)-2-(4-methoxystyryl)benzaldehyde (1d)

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NAME      Aldehyde+indole
EXPNO                  85
PROCNO                  1
Date_            20210909
Time                23.07
INSTRUM             spect
PROBHD    5 mm PABBO BB/
PULPROG              zg30
TD                  32768
SOLVENT             CDCl3
NS                     16
DS                      0
SWH              7211.539 Hz
FIDRES           0.220079 Hz
AQ             2.2719646 sec
RG                  78.51
DW                69.333 usec
DE                 10.06 usec
TE                 295.9 K
D1            2.00000000 sec
TD0                     1

======== CHANNEL f1 ========
SFO1         400.1324008 MHz
NUC1                   1H
P1                  15.00 usec
SI                  16384
SF           400.1300000 MHz
WDW                    EM
SSB                     0
LB                   0.00 Hz
GB                      0
PC                   1.00
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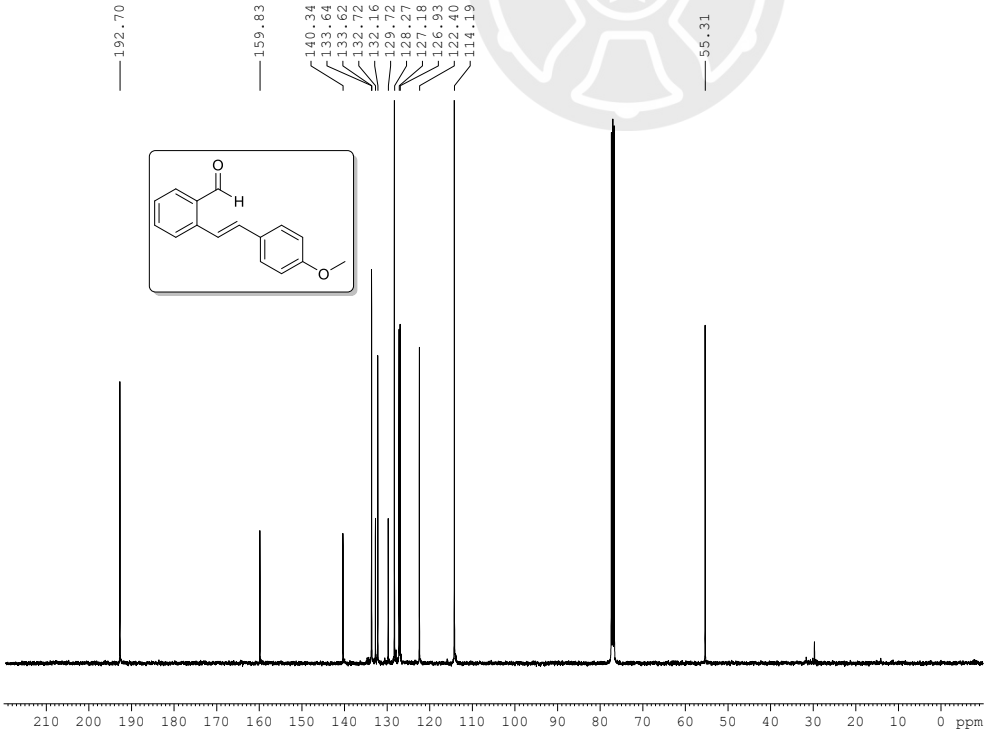


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NAME      Aldehyde+indole
EXPNO                  86
PROCNO                  1
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Time                23.11
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PROBHD    5 mm PABBO BB/
PULPROG           zgpg30
TD                  32768
SOLVENT             CDCl3
NS                   3000
DS                      0
SWH             24038.461 Hz
FIDRES           0.733596 Hz
AQ               0.6816244 sec
RG                  198.09
DW                20.800 usec
DE                 6.50 usec
TE                 296.2 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0                     1

======== CHANNEL f1 ========
SFO1         100.6228298 MHz
NUC1                   13C
P1                  10.00 usec
SI                  32768
SF           100.6127738 MHz
WDW                    EM
SSB                     0
LB                   2.00 Hz
GB                      0
PC                   1.00
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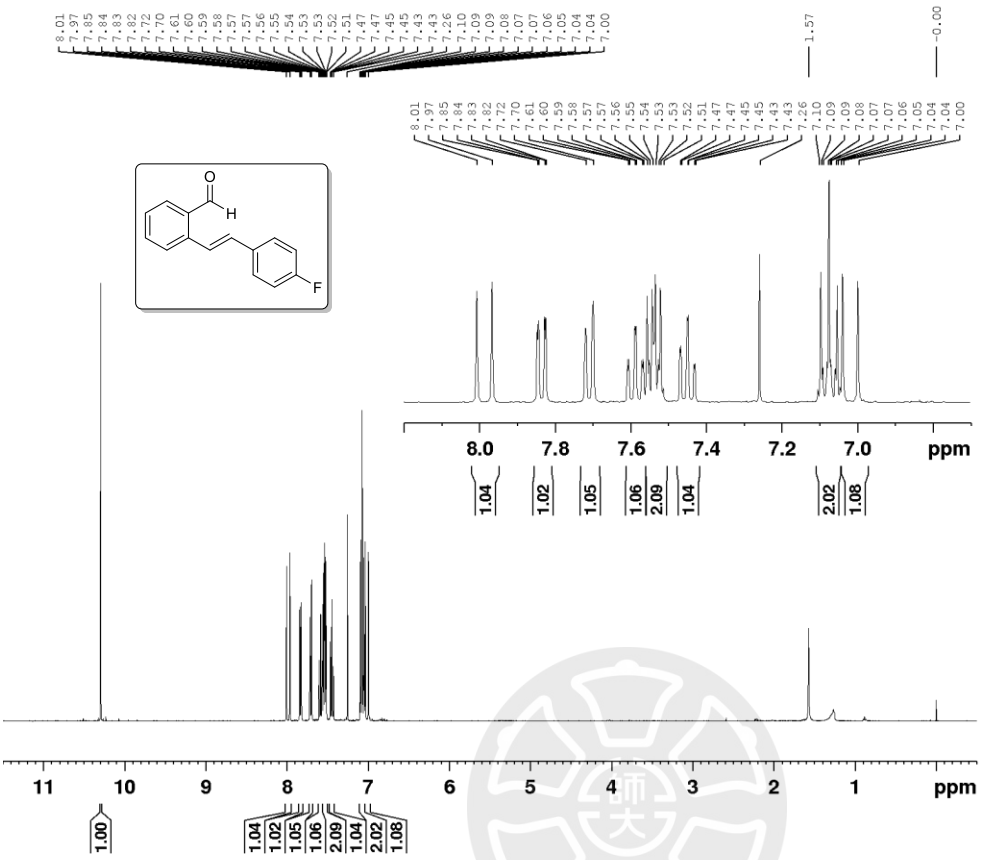
(*E*)-2-(4-fluorostyryl)benzaldehyde (1e)



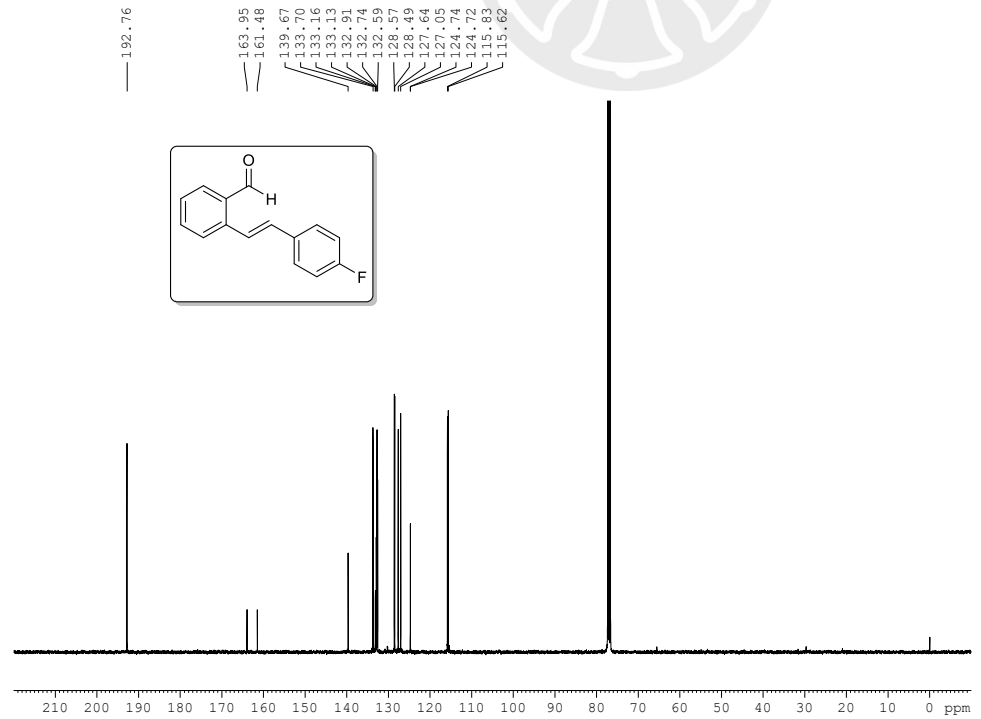
BRUKER

```
Current Data Parameters
NAME                  ADSC
EXPNO                   10
PROCNO                   1

F2 - Acquisition Parameters
Date_           20240118
Time                20.37
INSTRUM             spect
PROBHD   5 mm PABBO BB/
PULPROG             zg30
TD                 32768
SOLVENT            CDCl3
NS                    16
DS                     0
SWH             7211.539 Hz
FIDRES          0.220079 Hz
AQ              2.2719147 sec
RG                144.49
DW                69.333 usec
DE                10.06 usec
TE                298.2 K
D1              2.00000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1          400.1324008 MHz
NUC1                  1H
P1                 15.00 usec
PLW1          13.50000000 W

F2 - Processing parameters
SI                 16384
SF            400.1300107 MHz
WDW                   EM
SSB                    0
LB                     0 Hz
GB                     0
PC                  1.00
```

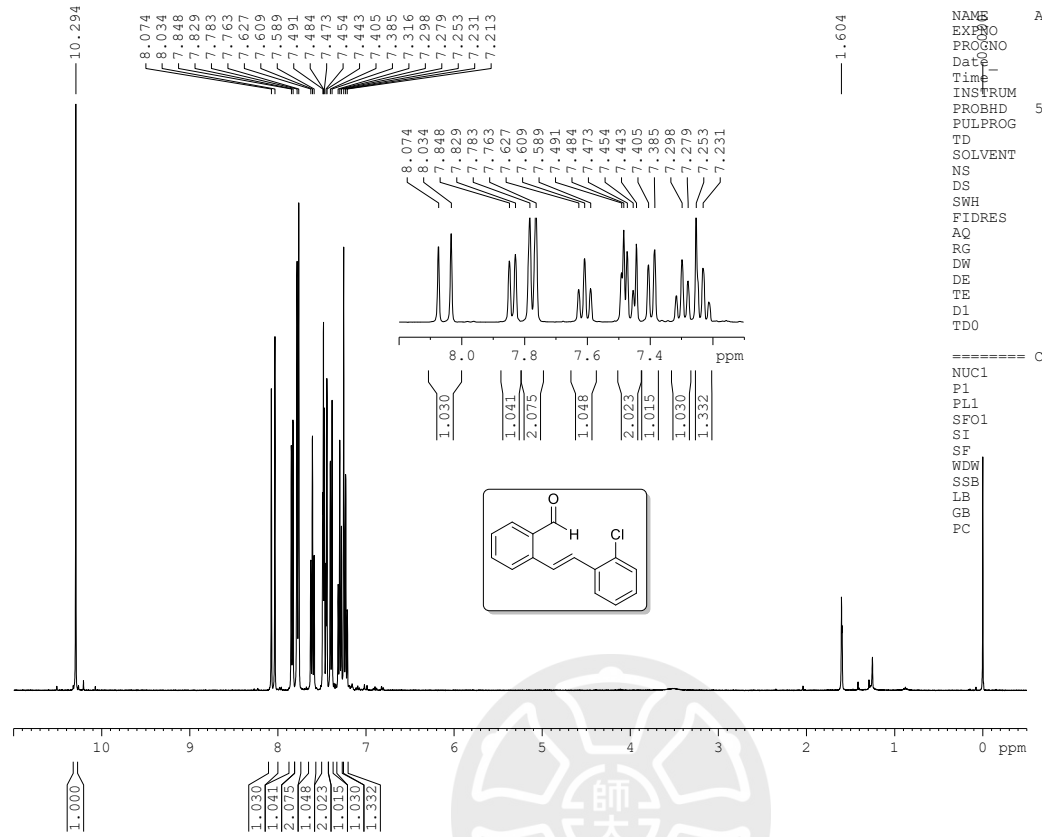



```
NAME      Aldehyde+indole
EXPNO                  96
PROCNO                  1
Date_           20211123
Time                23.05
INSTRUM             spect
PROBHD   5 mm BBO BB-1H
PULPROG           zgpg30
TD                 32768
SOLVENT            CDCl3
NS                  5000
DS                     0
SWH            24038.461 Hz
FIDRES          0.733596 Hz
AQ              0.6816452 sec
RG                 32768
DW                20.800 usec
DE                 6.50 usec
TE                297.9 K
D1              2.00000000 sec
D11             0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  13C
P1                10.00 usec
PL1                3.80 dB
SFO1          100.6233325 MHz

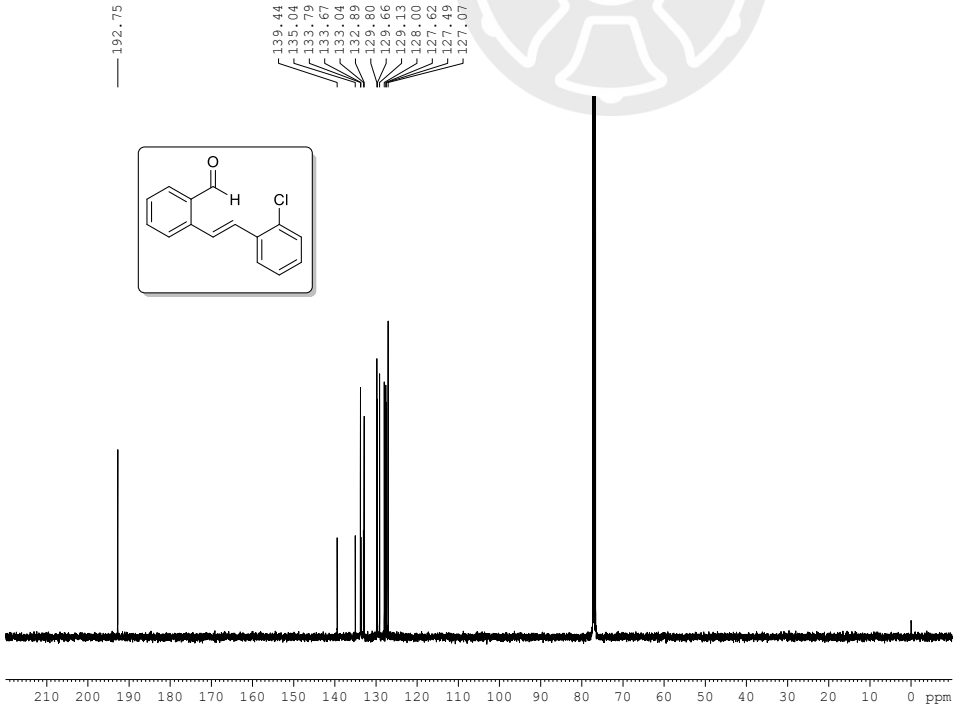
======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                  1H
PCPD2             90.00 usec
PL2               10.20 dB
PL12              26.00 dB
PL13              29.00 dB
SFO2          400.1316005 MHz
SI                 32768
SF            100.6127720 MHz
WDW                   EM
SSB                    0
LB                  1.00 Hz
GB                     0
PC                  1.00
```

(*E*)-2-(2-chlorostyryl)benzaldehyde (1f)




```
NAME      Aldehyde+indole
EXPNO               91
PROCNO               1
Date_         20211115
Time_            20.49
INSTRUM          spect
PROBHD   5 mm BBO BB-1H
PULPROG            zg30
TD                32768
SOLVENT           CDCl3
NS                   16
DS                    0
SWH           7246.377 Hz
FIDRES        0.221142 Hz
AQ            2.2611110 sec
RG                161.3
DW             69.000 usec
DE               6.50 usec
TE               297.3 K
D1           2.00000000 sec
TD0                  1

======== CHANNEL f1 ========
NUC1                1H
P1              15.00 usec
PL1             11.10 dB
SFO1        400.1324008 MHz
SI                16384
SF          400.1300118 MHz
WDW                EM
SSB                 0
LB               0.00 Hz
GB                  0
PC               1.00
```

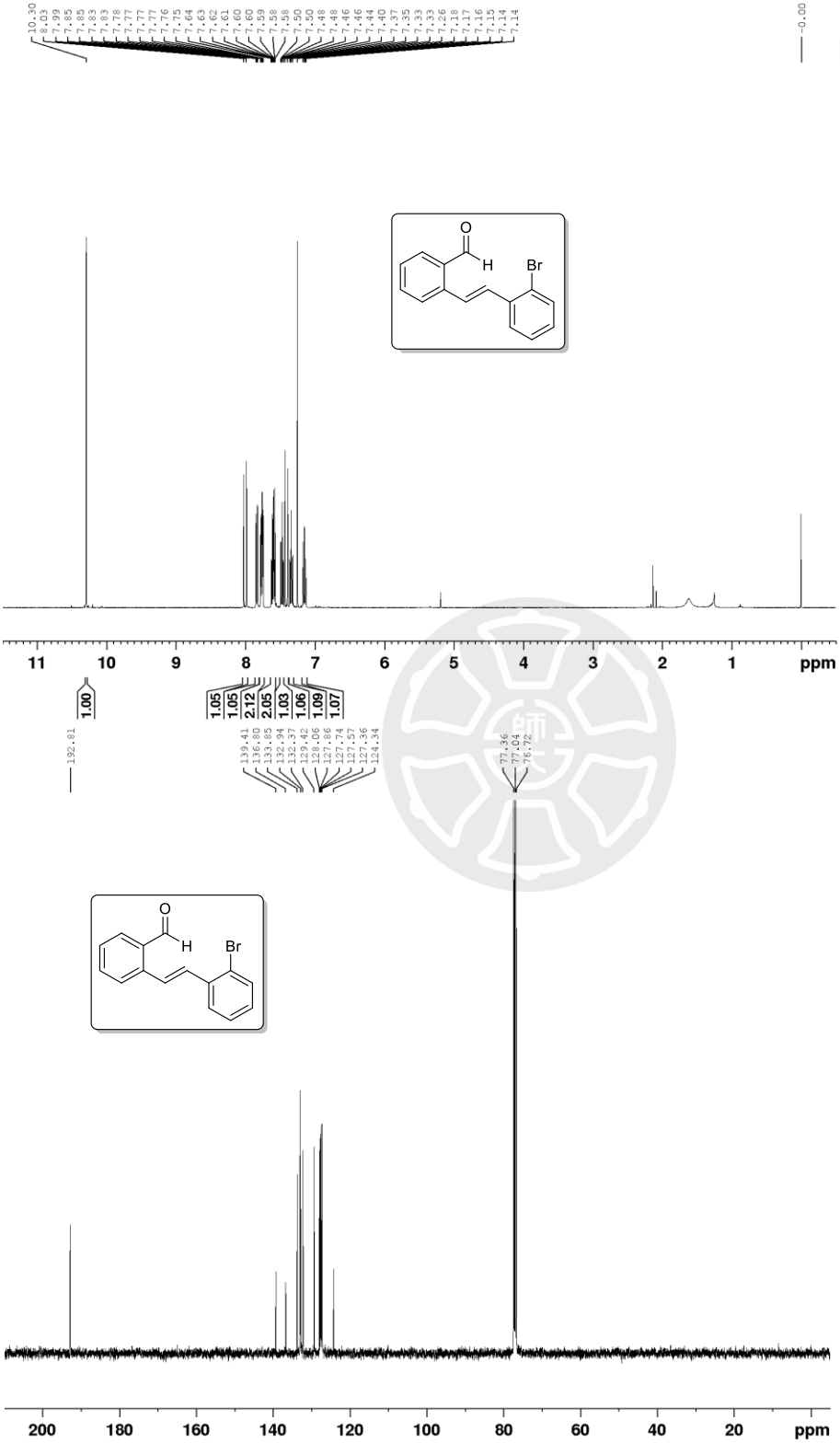



```
NAME      Aldehyde+indole
EXPNO               92
PROCNO               1
Date_         20211115
Time_            22.11
INSTRUM          spect
PROBHD   5 mm BBO BB-1H
PULPROG          zgpg30
TD                32768
SOLVENT           CDCl3
NS                  802
DS                    0
SWH          24038.461 Hz
FIDRES        0.733596 Hz
AQ            0.6816452 sec
RG                32768
DW             20.800 usec
DE               6.50 usec
TE               297.3 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                  1

======== CHANNEL f1 ========
NUC1                13C
P1              10.00 usec
PL1              3.80 dB
SFO1        100.6233325 MHz

======== CHANNEL f2 ========
CPDPRG2         waltz16
NUC2                1H
PCPD2           90.00 usec
PL2             10.20 dB
PL12            26.00 dB
PL13            29.00 dB
SFO2        400.1316005 MHz
SI                32768
SF          100.6127723 MHz
WDW                EM
SSB                 0
LB               1.00 Hz
GB                  0
PC               1.00
```

(*E*)-2-(2-Bromostyryl)benzaldehyde (1g)



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```
Current Data Parameters
NAME          Suresh-383
EXPNO                  4
PROCNO                 1

F2 - Acquisition Parameters
Date_           20230725
Time              10.17
INSTRUM           spect
PROBHD   5 mm PABBO BB/
PULPROG            zg30
TD                32768
SOLVENT           CDC13
NS                   16
DS                    0
SWH            7211.539 Hz
FIDRES         0.220079 Hz
AQ             2.2719147 sec
RG                113.31
DW                69.333 usec
DE                10.06 usec
TE                298.1 K
D1             2.00000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1          400.1324008 MHz
NUC1                  1H
P1                 15.00 usec
PLW1          13.50000000 W

F2 - Processing parameters
SI                 16384
SF            400.1300112 MHz
WDW                   EM
SSB                    0
LB                     0 Hz
GB                     0
PC                  1.00
```

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```
Current Data Parameters
NAME          Suresh-383
EXPNO                  3
PROCNO                 1

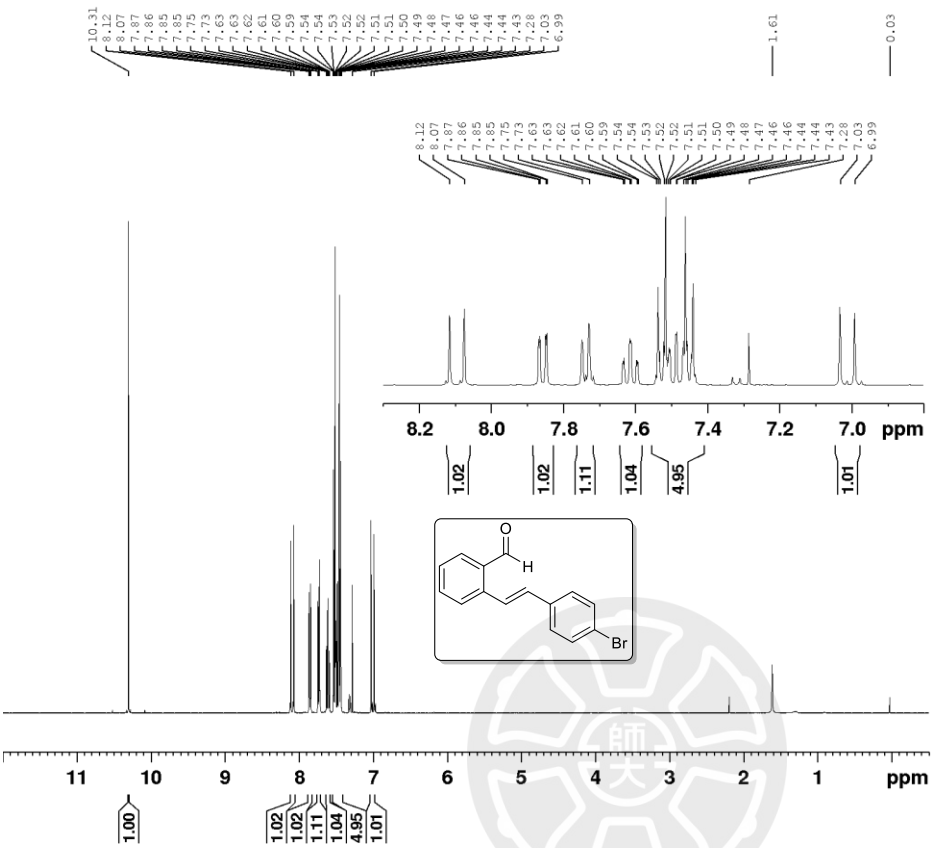
F2 - Acquisition Parameters
Date_           20230725
Time              10.18
INSTRUM           spect
PROBHD   5 mm PABBO BB/
PULPROG          zgpg30
TD                32768
SOLVENT           CDC13
NS                  356
DS                    0
SWH           24038.461 Hz
FIDRES         0.733596 Hz
AQ             0.6815744 sec
RG                198.09
DW                20.800 usec
DE                 6.50 usec
TE                298.2 K
D1             2.00000000 sec
D11            0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1          100.6228298 MHz
NUC1                  13C
P1                 10.00 usec
PLW1          49.50000000 W

======== CHANNEL f2 ========
SFO2          400.1316005 MHz
NUC2                  1H
CPDPRG[2         waltz16
PCPD2              90.00 usec
PLW2          12.50000000 W
PLW12          0.34722000 W
PLW13          0.28125000 W

F2 - Processing parameters
SI                 32768
SF            100.6127685 MHz
WDW                   EM
SSB                    0
LB                  2.00 Hz
GB                     0
PC                  1.00
```

(*E*)-2-(4-bromostyryl)benzaldehyde (1h)

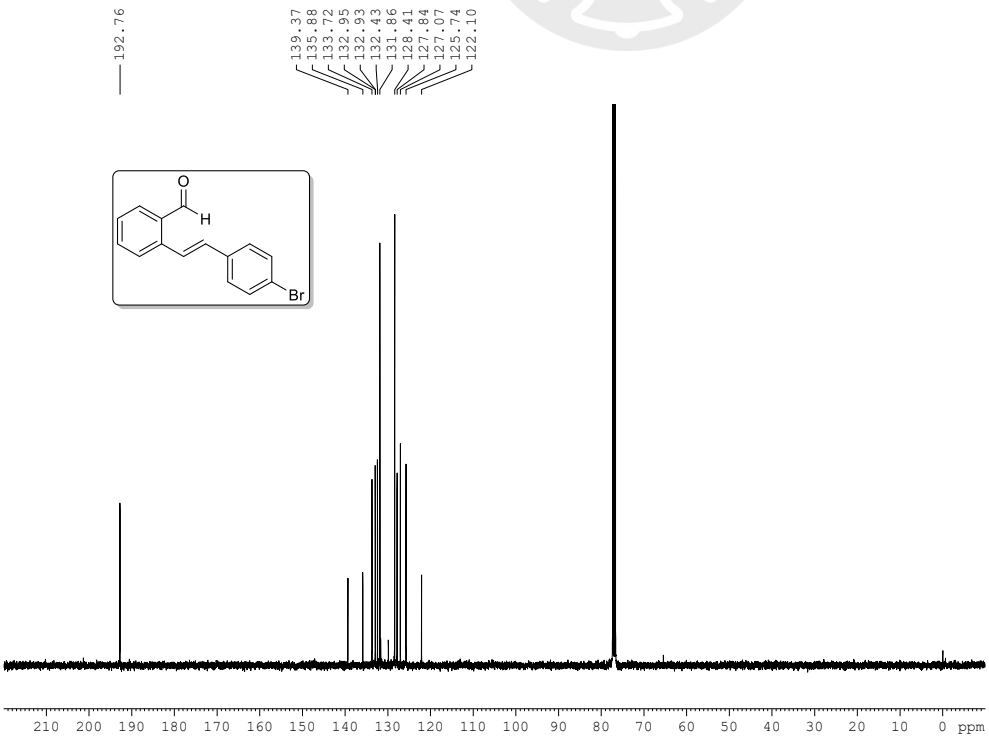



```
Current Data Parameters
NAME      Exp-1-revision work -1h
EXPNO                 23
PROCNO                 1

F2 - Acquisition Parameters
Date_           20240115
Time              12.09
INSTRUM           spect
PROBHD   5 mm PABBO BB/
PULPROG           zg30
TD               32768
SOLVENT          CDCl3
NS                  16
DS                   0
SWH           7211.539 Hz
FIDRES        0.220079 Hz
AQ            2.2719147 sec
RG               113.31
DW               69.333 usec
DE               10.06 usec
TE               298.0 K
D1           2.00000000 sec
TD0                  1

======== CHANNEL f1 ========
SFO1        400.1324008 MHz
NUC1               1H
P1              15.00 usec
PLW1        13.50000000 W

F2 - Processing parameters
SI               16384
SF          400.1300000 MHz
WDW                EM
SSB                 0
LB                  0 Hz
GB                  0
PC               1.00
```

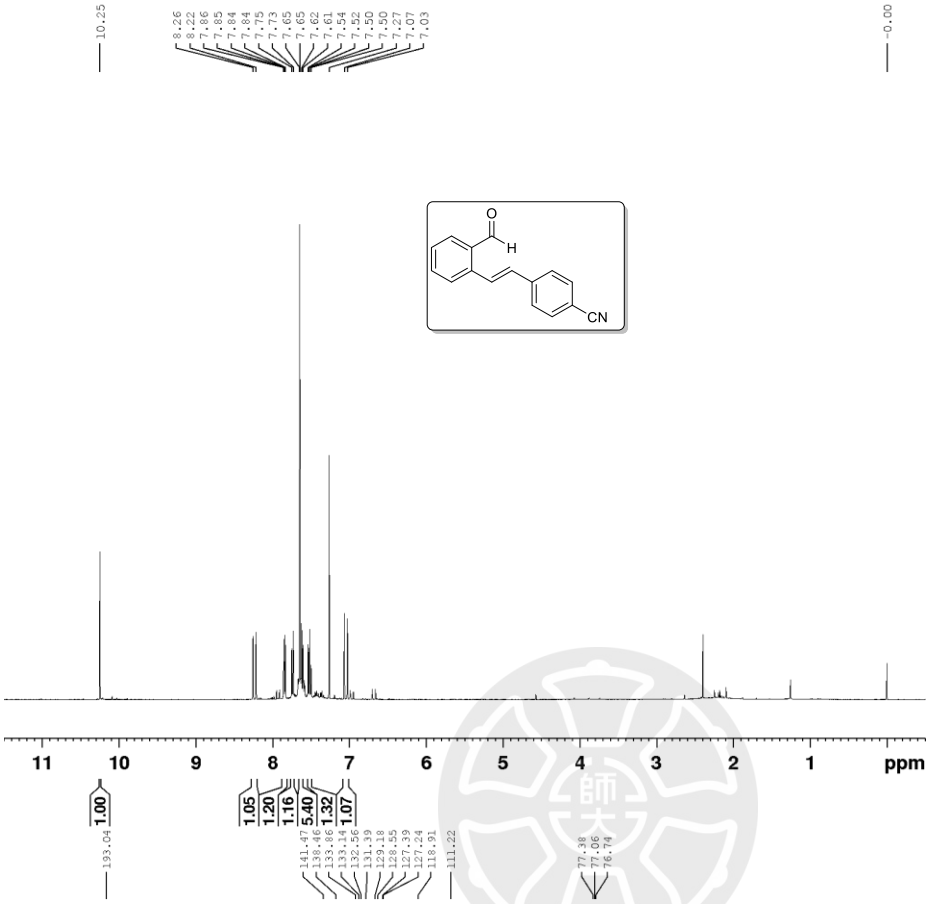



```
NAME      Aldehyde+indole
EXPNO                94
PROCNO                1
Date_          20211115
Time              22.57
INSTRUM           spect
PROBHD   5 mm BBO BB-1H
PULPROG          zgpg30
TD                32768
SOLVENT           CDCl3
NS                  770
DS                    0
SWH           24038.461 Hz
FIDRES         0.733596 Hz
AQ             0.6816452 sec
RG                32768
DW               20.800 usec
DE                6.50 usec
TE                297.8 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                13C
P1               10.00 usec
PL1               3.80 dB
SFO1        100.6233325 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                1H
PCPD2            90.00 usec
PL2              10.20 dB
PL12             26.00 dB
PL13             29.00 dB
SFO2        400.1316005 MHz
SI                32768
SF          100.6127730 MHz
WDW                EM
SSB                 0
LB               1.00 Hz
GB                  0
PC               1.00
```

(*E*)-4-(2-Formylstyryl)benzonitrile (1i)



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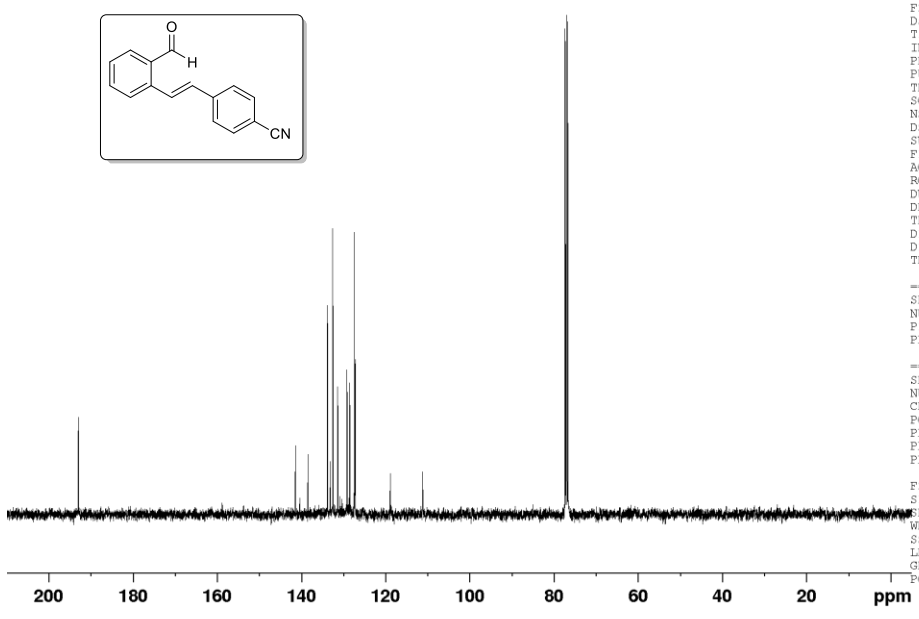
```
Current Data Parameters
NAME          Suresh-383
EXPNO                  5
PROCNO                 1

F2 - Acquisition Parameters
Date_           20230725
Time               10.42
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG             zg30
TD                 32768
SOLVENT            CDC13
NS                    16
DS                     0
SWH             7211.539 Hz
FIDRES          0.220079 Hz
AQ             2.2719147 sec
RG                 99.72
DW                69.333 usec
DE                 10.06 usec
TE                 298.4 K
D1            2.00000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1         400.1324008 MHz
NUC1                   1H
P1                  15.00 usec
PLW1         13.50000000 W

F2 - Processing parameters
SI                 16384
SF          400.1300072 MHz
WDW                   EM
SSB                    0
LB                  0 Hz
GB                     0
PC                  1.00
```



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```
Current Data Parameters
NAME          Suresh-383
EXPNO                  6
PROCNO                 1

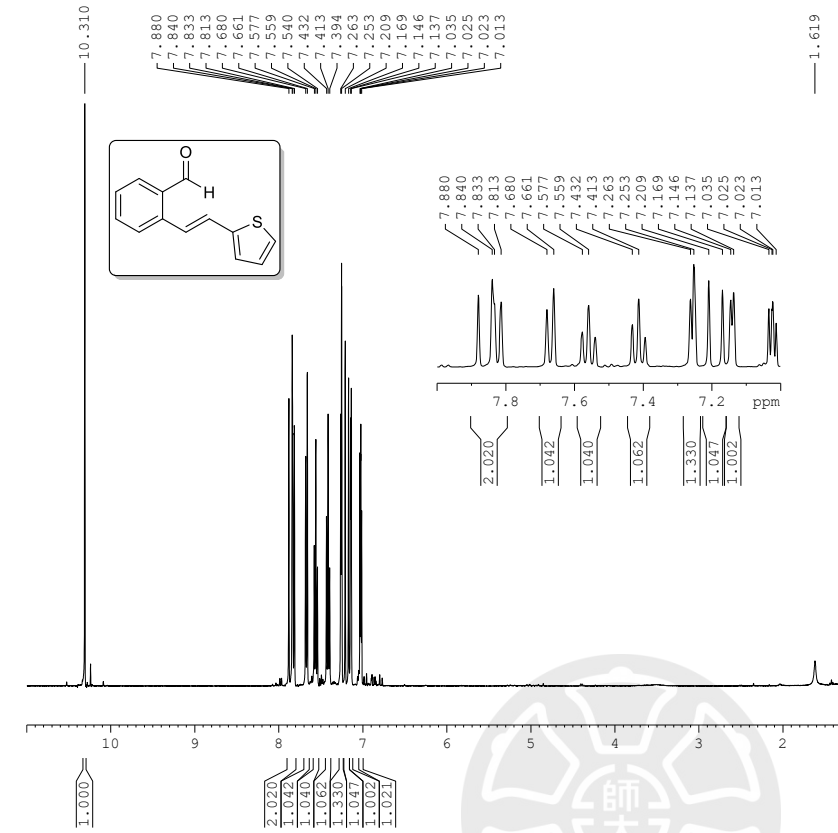
F2 - Acquisition Parameters
Date_           20230725
Time               10.44
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG          zgpg30
TD                 32768
SOLVENT            CDC13
NS                   106
DS                     0
SWH            24038.461 Hz
FIDRES          0.733596 Hz
AQ             0.6815744 sec
RG                198.09
DW                20.800 usec
DE                 6.50 usec
TE                 298.4 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1         100.6228298 MHz
NUC1                  13C
P1                  10.00 usec
PLW1         49.50000000 W

======== CHANNEL f2 ========
SFO2         400.1316005 MHz
NUC2                   1H
CPDPRG[2          waltz16
PCPD2               90.00 usec
PLW2         12.50000000 W
PLW12         0.34722000 W
PLW13         0.28125000 W

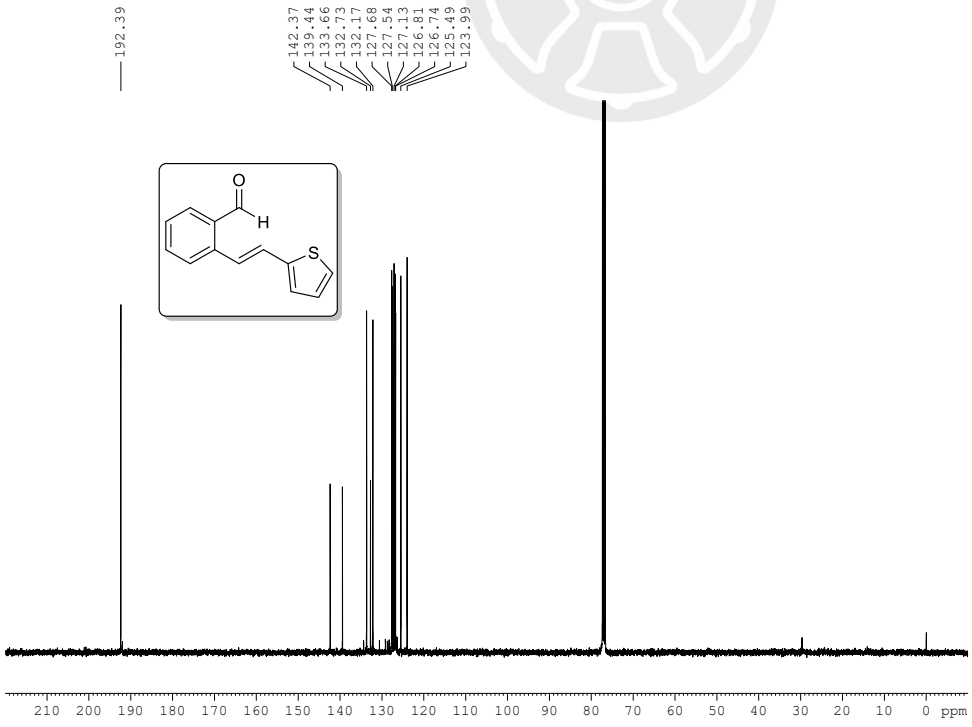
F2 - Processing parameters
SI                 32768
SF          100.6127685 MHz
WDW                   EM
SSB                    0
LB                  2.00 Hz
GB                     0
PC                  1.00
```

(*E*)-2-(2-(thiophen-2-yl)vinyl)benzaldehyde (1j)




```
NAME      Aldehyde+indole
EXPNO                  97
PROCNO                  1
Date_           20211115
Time                23.34
INSTRUM             spect
PROBHD    5 mm BBO BB-1H
PULPROG              zg30
TD                  32768
SOLVENT             CDCl3
NS                     16
DS                      0
SWH             7246.377 Hz
FIDRES          0.221142 Hz
AQ             2.2611110 sec
RG                    114
DW                 69.000 usec
DE                  6.50 usec
TE                 297.6 K
D1            2.00000000 sec
TD0                     1

======== CHANNEL f1 ========
NUC1                   1H
P1                  15.00 usec
PL1                 11.10 dB
SFO1         400.1324008 MHz
SI                  16384
SF           400.1300118 MHz
WDW                    EM
SSB                     0
LB                   0.00 Hz
GB                      0
PC                   1.00
```

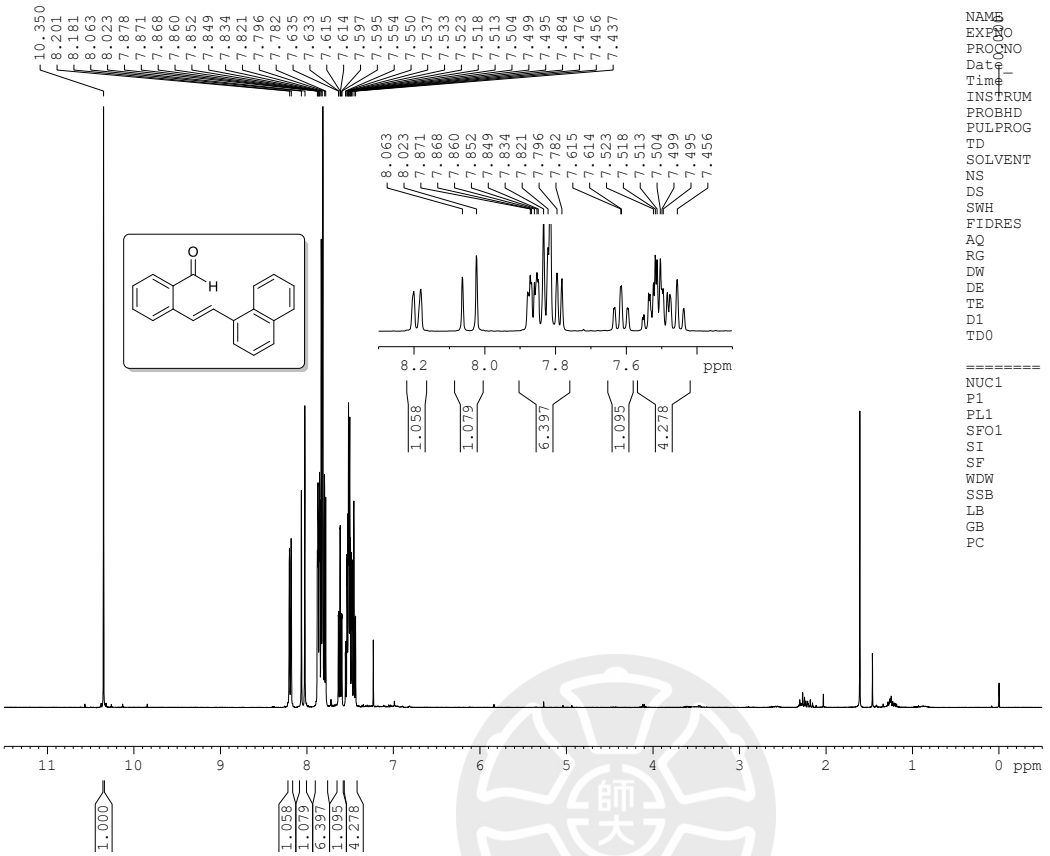



```
NAME      Aldehyde+indole
EXPNO                  98
PROCNO                  1
Date_           20211115
Time                23.37
INSTRUM             spect
PROBHD    5 mm BBO BB-1H
PULPROG           zgpg30
TD                  32768
SOLVENT             CDCl3
NS                   2054
DS                      0
SWH            24038.461 Hz
FIDRES          0.733596 Hz
AQ             0.6816452 sec
RG                  32768
DW                 20.800 usec
DE                  6.50 usec
TE                 297.7 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0                     1

======== CHANNEL f1 ========
NUC1                  13C
P1                  10.00 usec
PL1                  3.80 dB
SFO1        100.6233325 MHz

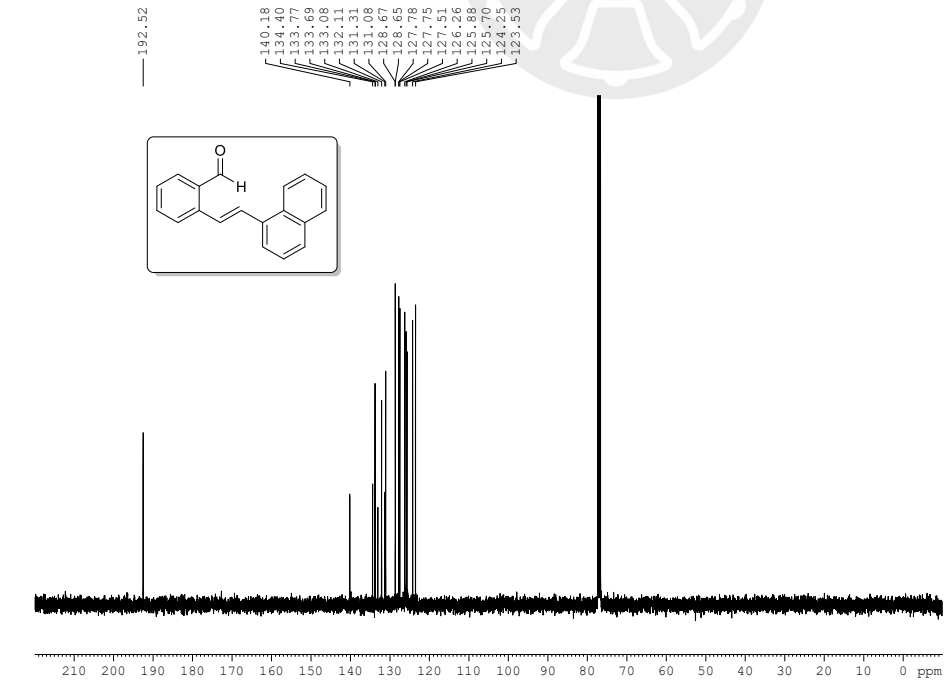
======== CHANNEL f2 ========
CPDPRG2           waltz16
NUC2                   1H
PCPD2               90.00 usec
PL2                 10.20 dB
PL12                26.00 dB
PL13                29.00 dB
SFO2         400.1316005 MHz
SI                  32768
SF           100.6127738 MHz
WDW                    EM
SSB                     0
LB                   1.00 Hz
GB                      0
PC                   1.00
```

(*E*)-2-(2-(naphthalen-1-yl)vinyl)benzaldehyde (1k)




```
NAME      Aldehyde+indole
EXPNO                  87
PROCNO                  1
Date_           20210929
Time                13.13
INSTRUM             spect
PROBHD   5 mm BBO BB-1H
PULPROG              zg30
TD                  32768
SOLVENT             CDCl3
NS                     16
DS                      0
SWH              7246.377 Hz
FIDRES           0.221142 Hz
AQ               2.2611110 sec
RG                    114
DW                 69.000 usec
DE                  6.50 usec
TE                 297.9 K
D1              2.00000000 sec
TD0                     1

======== CHANNEL f1 ========
NUC1                   1H
P1                  15.00 usec
PL1                 11.10 dB
SFO1         400.1324008 MHz
SI                  16384
SF           400.1300207 MHz
WDW                    EM
SSB                     0
LB                   0.00 Hz
GB                      0
PC                   1.00
```

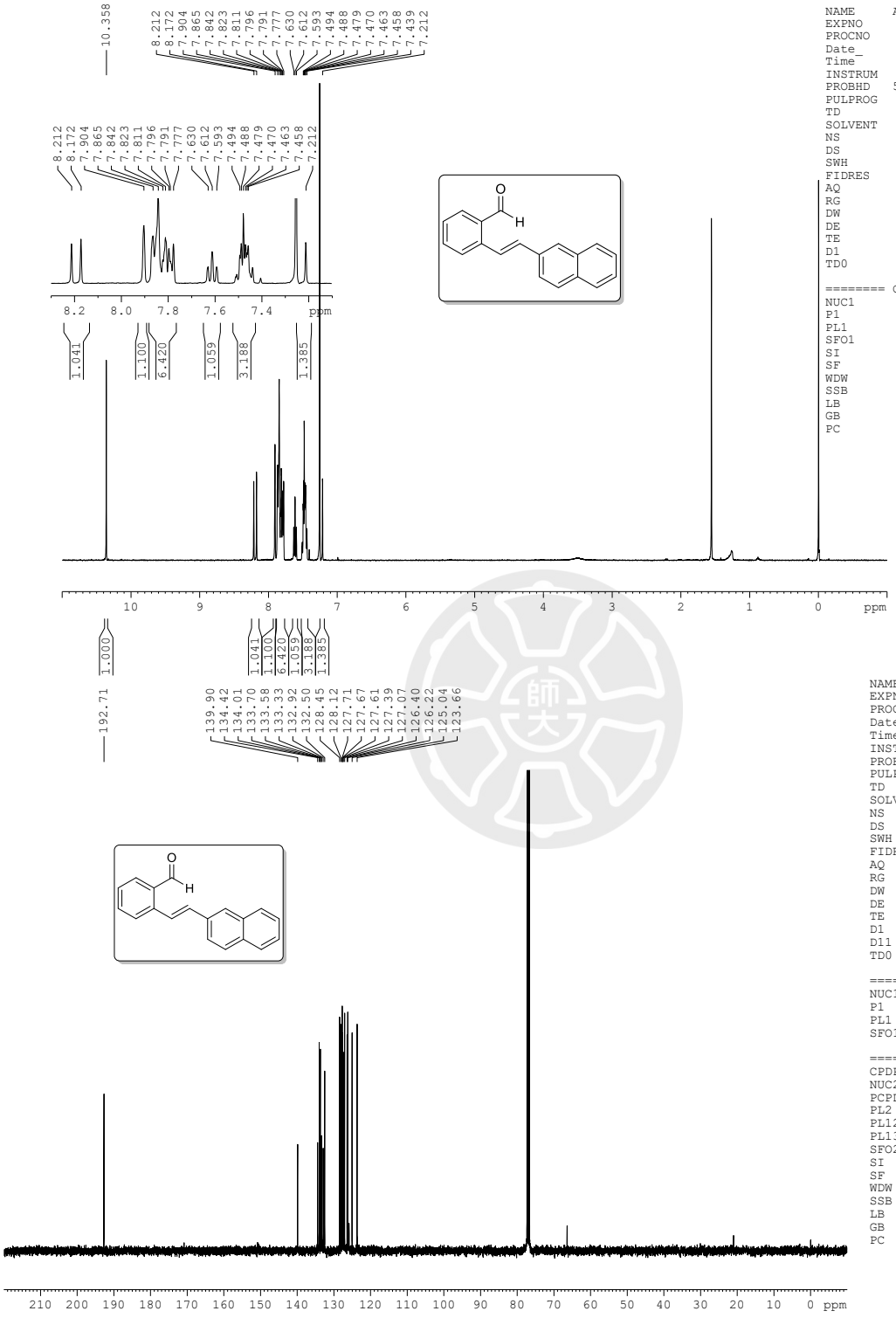



```
NAME      Aldehyde+indole
EXPNO                  88
PROCNO                  1
Date_           20210929
Time                13.18
INSTRUM             spect
PROBHD   5 mm BBO BB-1H
PULPROG           zgpg30
TD                  32768
SOLVENT             CDCl3
NS                    151
DS                      0
SWH             24038.461 Hz
FIDRES           0.733596 Hz
AQ               0.6816452 sec
RG                  32768
DW                 20.800 usec
DE                  6.50 usec
TE                 298.4 K
D1              2.00000000 sec
D11             0.03000000 sec
TD0                     1

======== CHANNEL f1 ========
NUC1                  13C
P1                  10.00 usec
PL1                  3.80 dB
SFO1         100.6233325 MHz

======== CHANNEL f2 ========
CPDPRG2           waltz16
NUC2                   1H
PCPD2               90.00 usec
PL2                 10.20 dB
PL12                26.00 dB
PL13                29.00 dB
SFO2         400.1316005 MHz
SI                  32768
SF           100.6127760 MHz
WDW                    EM
SSB                     0
LB                   1.00 Hz
GB                      0
PC                   1.00
```

(*E*)-2-(2-(naphthalen-2-yl)vinyl)benzaldehyde (1l)




```
NAME      Aldehyde+indole
EXPNO                  89
PROCNO                  1
Date_            20211104
Time                21.52
INSTRUM             spect
PROBHD   5 mm BBO BB-1H
PULPROG              zg30
TD                  32768
SOLVENT             CDCl3
NS                     16
DS                      0
SWH              7246.377 Hz
FIDRES           0.221142 Hz
AQ             2.2611110 sec
RG                    362
DW                69.000 usec
DE                 6.50 usec
TE                296.4 K
D1           2.00000000 sec
TD0                     1

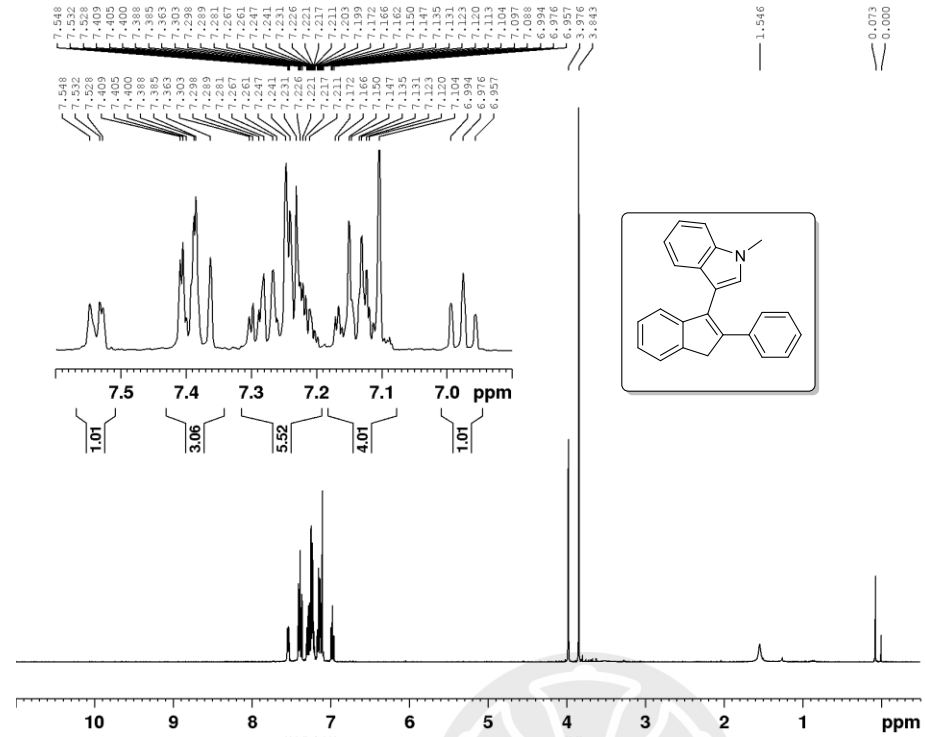
======== CHANNEL f1 ========
NUC1                   1H
P1                 15.00 usec
PL1                11.10 dB
SFO1         400.1324008 MHz
SI                  16384
SF           400.1300118 MHz
WDW                    EM
SSB                     0
LB                 0.00 Hz
GB                      0
PC                 1.00
```

```
NAME      Aldehyde+indole
EXPNO                  90
PROCNO                  1
Date_            20211005
Time                23.10
INSTRUM             spect
PROBHD   5 mm BBO BB-1H
PULPROG            zgpg30
TD                  32768
SOLVENT             CDCl3
NS                    512
DS                      0
SWH             24038.461 Hz
FIDRES           0.733596 Hz
AQ               0.6816452 sec
RG                  32768
DW                20.800 usec
DE                 6.50 usec
TE                298.4 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                     1

======== CHANNEL f1 ========
NUC1                  13C
P1                 10.00 usec
PL1                 3.80 dB
SFO1         100.6233325 MHz

======== CHANNEL f2 ========
CPDPRG2           waltz16
NUC2                   1H
PCPD2              90.00 usec
PL2                10.20 dB
PL12               26.00 dB
PL13               29.00 dB
SFO2         400.1316005 MHz
SI                  32768
SF           100.6127745 MHz
WDW                    EM
SSB                     0
LB                 1.00 Hz
GB                      0
PC                 1.00
```

1-Methyl-3-(2-phenyl-1*H*-inden-3-yl)-1*H*-indole (3aa)

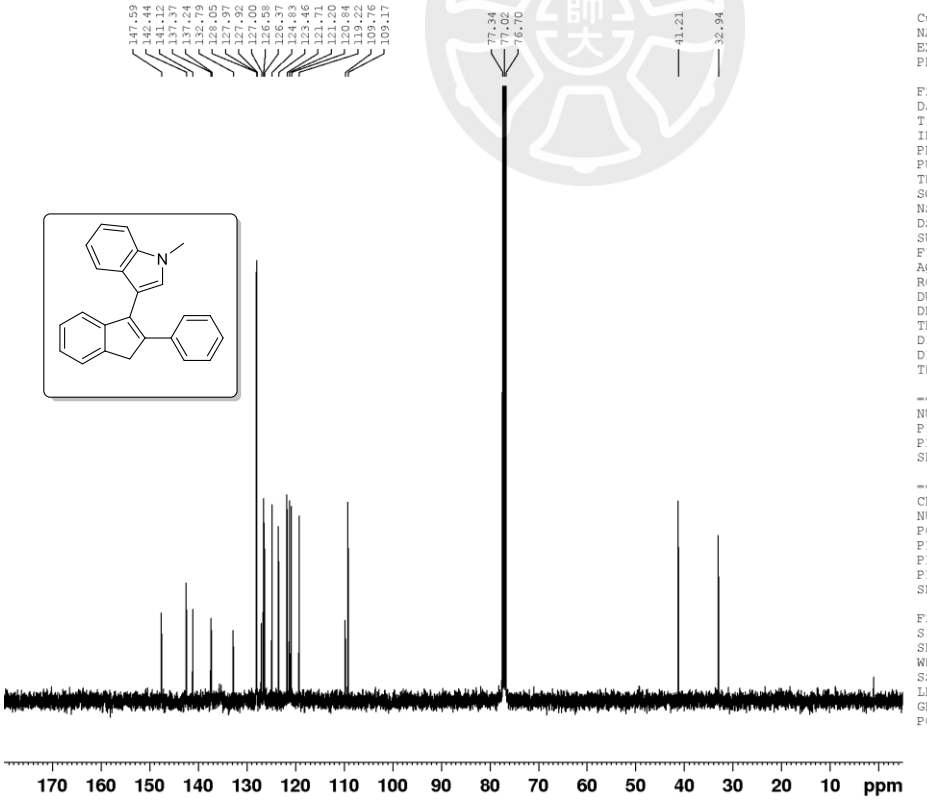



```
Current Data Parameters
NAME                  exp 252
EXPNO                       3
PROCNO                      1

F2 - Acquisition Parameters
Date_               20210825
Time                   21.05
INSTRUM                spect
PROBHD    5 mm BBO BB-1H
PULPROG                zg30
TD                     32768
SOLVENT                CDCl3
NS                        16
DS                         0
SWH                7246.377 Hz
FIDRES            0.221142 Hz
AQ                2.2609921 sec
RG                     228.1
DW                  69.000 usec
DE                    6.50 usec
TE                   299.0 K
D1                2.00000000 sec
TD0                        1

======== CHANNEL f1 ========
NUC1                      1H
P1                     15.00 usec
PL1                    11.10 dB
SFO1            400.1324008 MHz

F2 - Processing parameters
SI                        16384
SF                400.1300171 MHz
WDW                        EM
SSB                         0
LB                          0 Hz
GB                          0
PC                       1.00
```




```
Current Data Parameters
NAME                  exp 252
EXPNO                       5
PROCNO                      1

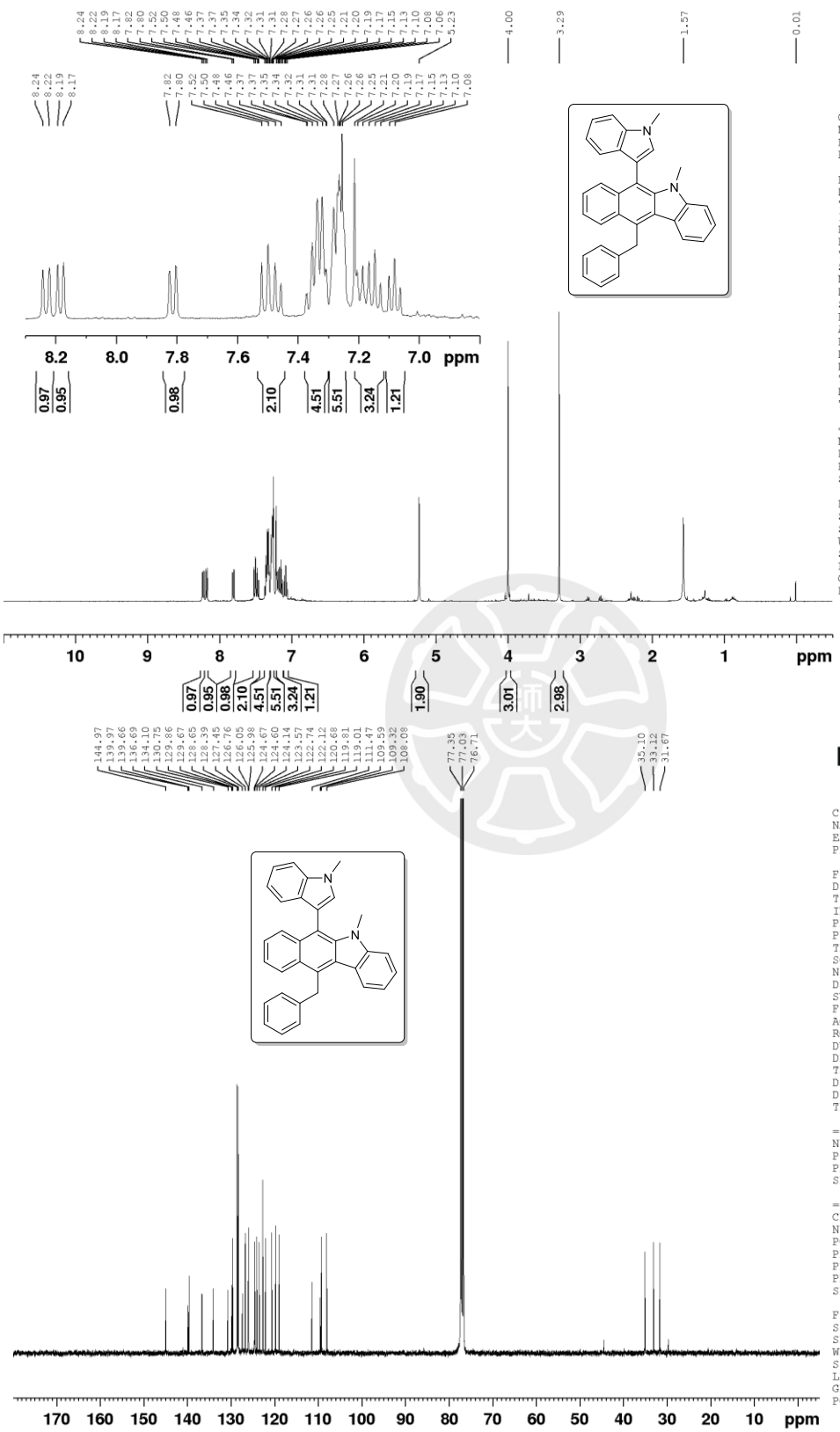
F2 - Acquisition Parameters
Date_               20210825
Time                   21.11
INSTRUM                spect
PROBHD    5 mm BBO BB-1H
PULPROG             zgpg30
TD                     32768
SOLVENT                CDCl3
NS                       593
DS                         0
SWH               24038.461 Hz
FIDRES            0.733596 Hz
AQ                0.6815744 sec
RG                     32768
DW                  20.800 usec
DE                    6.50 usec
TE                   299.7 K
D1                2.00000000 sec
D11               0.03000000 sec
TD0                        1

======== CHANNEL f1 ========
NUC1                      13C
P1                     10.00 usec
PL1                     3.80 dB
SFO1            100.6233325 MHz

======== CHANNEL f2 ========
CPDPRG[2          waltz16
NUC2                      1H
PCPD2                  90.00 usec
PL2                    10.20 dB
PL12                   26.00 dB
PL13                   29.00 dB
SFO2            400.1316005 MHz

F2 - Processing parameters
SI                        32768
SF                100.6127690 MHz
WDW                        EM
SSB                         0
LB                       1.00 Hz
GB                          0
PC                       1.00
```

11-benzyl-5-methyl-6-(1-methyl-1*H*-indol-3-yl)-5*H*-benzo[*b*]carbazole (5aa)



```
Current Data Parameters
NAME                  exp 252
EXPNO                      15
PROCNO                      1

F2 - Acquisition Parameters
Date_              20210926
Time                  15.01
INSTRUM               spect
PROBHD    5 mm BBO BB-1H
PULPROG                zg30
TD                    32768
SOLVENT               CDC13
NS                       16
DS                        0
SWH                7246.377 Hz
FIDRES             0.221142 Hz
AQ                 2.2609921 sec
RG                      256
DW                   69.000 usec
DE                    6.50 usec
TE                   297.6 K
D1                 2.00000000 sec
TD0                         1

======== CHANNEL f1 ========
NUC1                       1H
P1                      15.00 usec
PL1                     11.10 dB
SFO1            400.1324008 MHz

F2 - Processing parameters
SI                      16384
SF                400.1300118 MHz
WDW                        EM
SSB                         0
LB                       0 Hz
GB                          0
PC                       1.00
```

```
Current Data Parameters
NAME                  exp 252
EXPNO                      14
PROCNO                      1

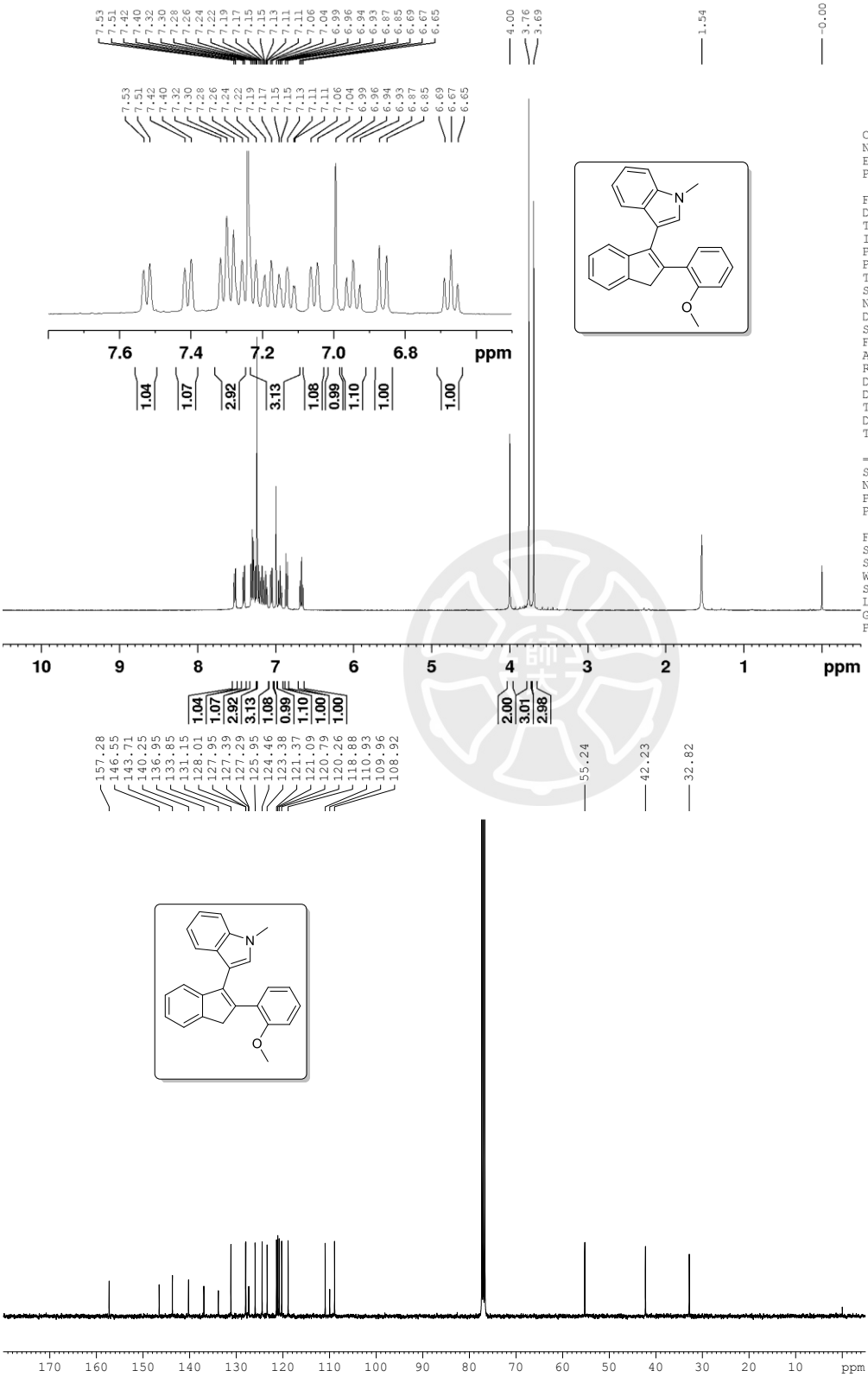
F2 - Acquisition Parameters
Date_              20210925
Time                  22.31
INSTRUM               spect
PROBHD    5 mm BBO BB-1H
PULPROG             zgpg30
TD                    32768
SOLVENT               CDC13
NS                    15000
DS                        0
SWH               24038.461 Hz
FIDRES             0.733596 Hz
AQ                 0.6815744 sec
RG                      32768
DW                   20.800 usec
DE                    6.50 usec
TE                   297.9 K
D1                 2.00000000 sec
D11                0.03000000 sec
TD0                         1

======== CHANNEL f1 ========
NUC1                       13C
P1                      10.00 usec
PL1                      3.80 dB
SFO1            100.6233325 MHz

======== CHANNEL f2 ========
CPDPRG[2              waltz16
NUC2                       1H
PCPD2                   90.00 usec
PL2                     10.20 dB
PL12                    26.00 dB
PL13                    29.00 dB
SFO2            400.1316005 MHz

F2 - Processing parameters
SI                      32768
SF                100.6127690 MHz
WDW                        EM
SSB                         0
LB                       1.00 Hz
GB                          0
PC                       1.00
```

3-(2-(2-methoxyphenyl)-1*H*-inden-3-yl)-1-methyl-1*H*-indole (3ba)




```
Current Data Parameters
NAME         20221018(3)
EXPNO                 1
PROCNO                1

F2 - Acquisition Parameters
Date_           20221018
Time              18.40
INSTRUM           spect
PROBHD    5 mm PABBO BB/
PULPROG           zg30
TD               32768
SOLVENT          CDCl3
NS                  16
DS                   0
SWH           7211.539 Hz
FIDRES        0.220079 Hz
AQ            2.2719147 sec
RG               113.31
DW               69.333 usec
DE                10.06 usec
TE                297.4 K
D1           2.00000000 sec
TD0                  1

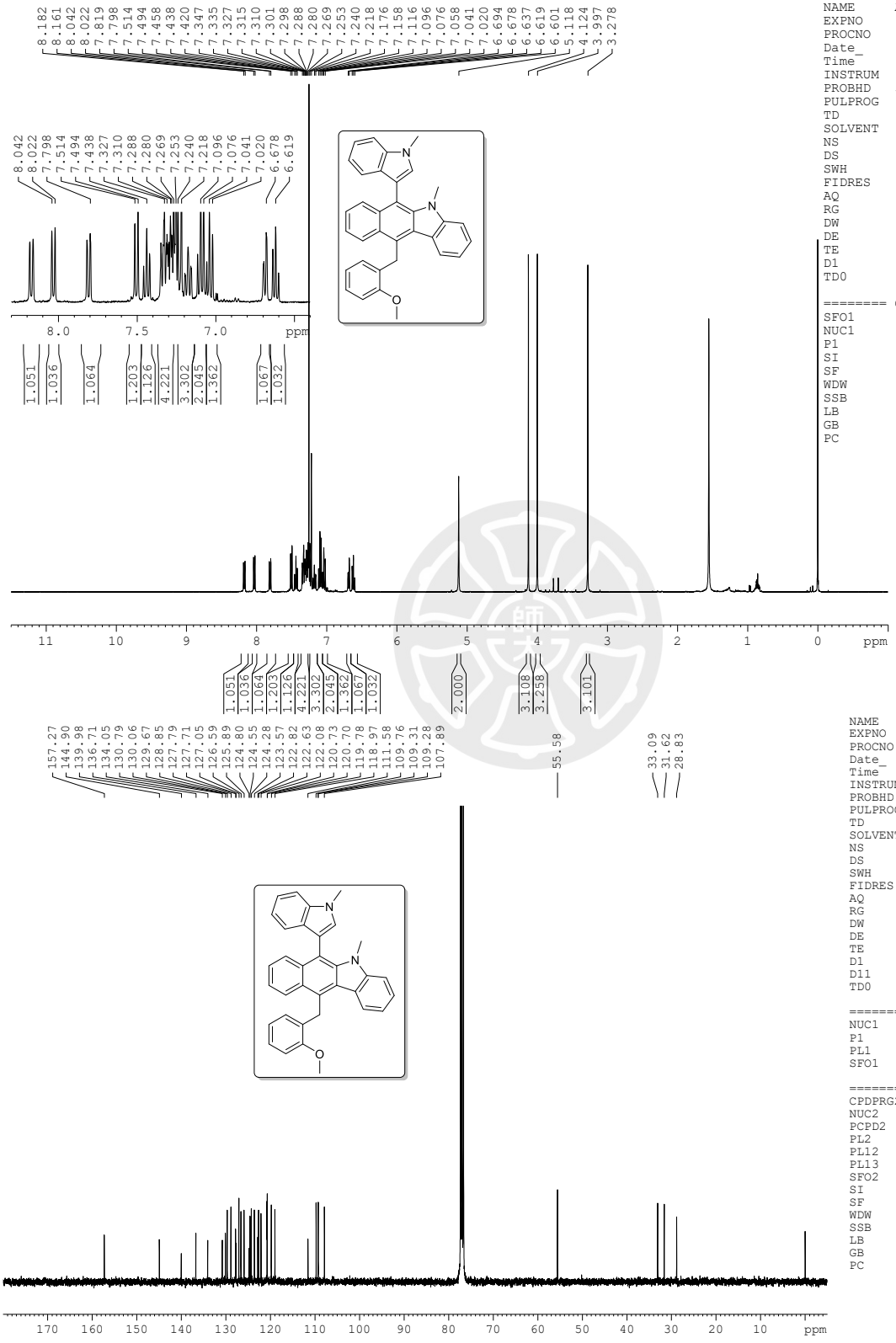
======== CHANNEL f1 ========
SFO1        400.1324008 MHz
NUC1                1H
P1                15.00 usec
PLW1        11.39999962 W

F2 - Processing parameters
SI                16384
SF          400.1300179 MHz
WDW                EM
SSB                 0
LB                  0 Hz
GB                  0
PC               1.00
```

```
NAME      Aldehyde+indole
EXPNO                  16
PROCNO                  1
Date_            20221020
Time               18.26
INSTRUM            spect
PROBHD    5 mm PABBO BB/
PULPROG          zgpg30
TD                32768
SOLVENT           CDCl3
NS                 1325
DS                    0
SWH           24038.461 Hz
FIDRES         0.733596 Hz
AQ            0.6816244 sec
RG               198.09
DW               20.800 usec
DE                6.50 usec
TE                298.7 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                  1

======== CHANNEL f1 ========
SFO1        100.6228298 MHz
NUC1                13C
P1                10.00 usec
SI                32768
SF          100.6127720 MHz
WDW                EM
SSB                 0
LB                2.00 Hz
GB                  0
PC               1.00
```

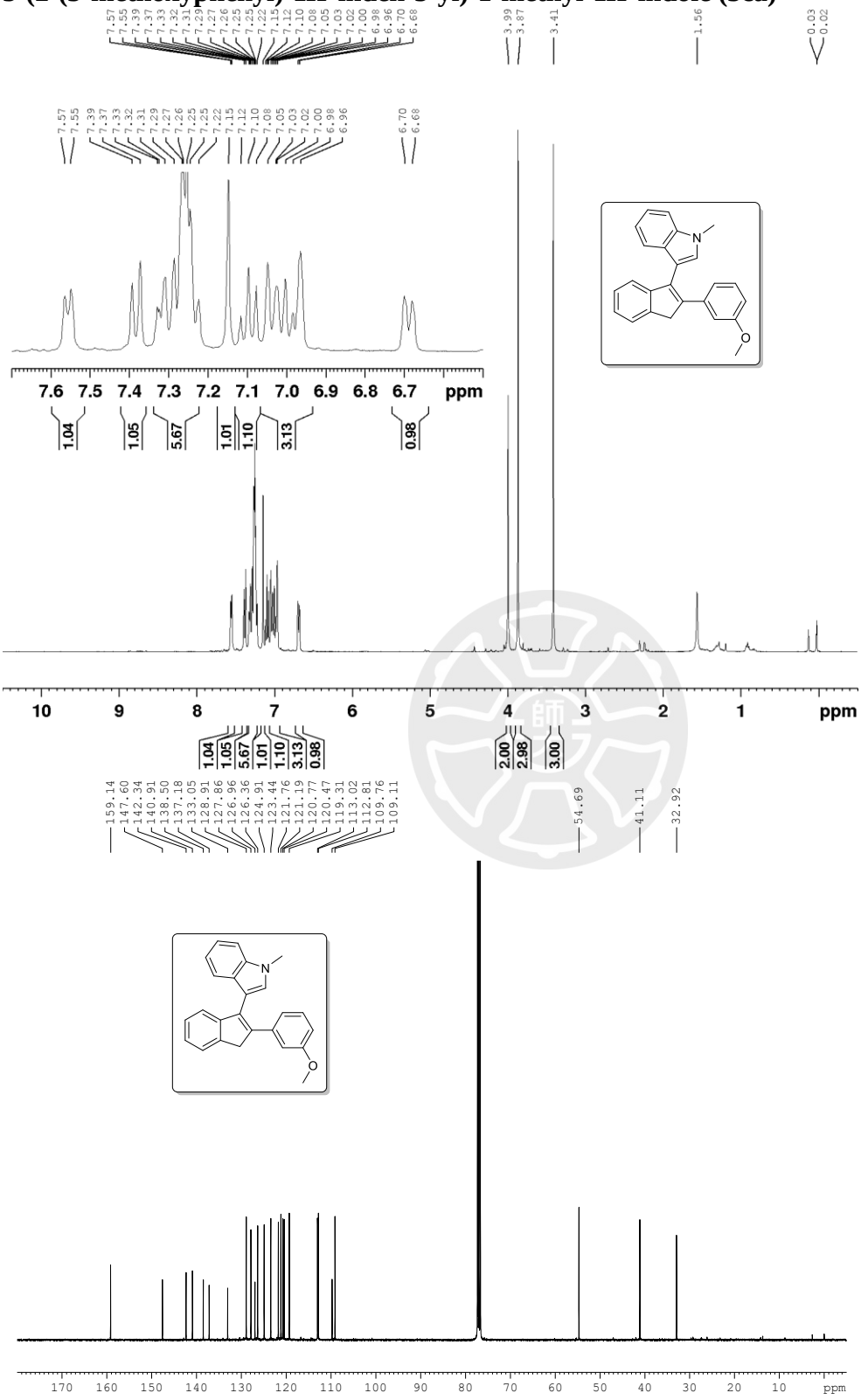
1-(2-methoxybenzyl)-5-methyl-6-(1-methyl-1*H*-indol-3-yl)-5*H*-benzo[*b*]carbazole (5ba)



NAME	Aldehyde+indole
EXPNO	17
PROCNO	1
Date_	20221018
Time	19.43
INSTRUM	spect
PROBHD	5 mm PABBO BB/
PULPROG	zg30
TD	32768
SOLVENT	CDCl3
NS	16
DS	0
SWH	7211.539 Hz
FIDRES	0.220079 Hz
AQ	2.2719646 sec
RG	158.74
DW	69.333 usec
DE	10.06 usec
TE	297.9 K
D1	2.00000000 sec
TD0	1
======== CHANNEL f1 ========	
SFO1	400.1324008 MHz
NUC1	1H
P1	15.00 usec
SI	16384
SF	400.1300125 MHz
WDW	EM
SSB	0
LB	0.00 Hz
GB	0
PC	1.00

NAME	Aldehyde+indole
EXPNO	18
PROCNO	1
Date_	20221018
Time	22.10
INSTRUM	spect
PROBHD	5 mm BBO BB-1H
PULPROG	zgpg30
TD	32768
SOLVENT	CDCl3
NS	13419
DS	0
SWH	24038.461 Hz
FIDRES	0.733596 Hz
AQ	0.6816452 sec
RG	14596.5
DW	20.800 usec
DE	6.50 usec
TE	300.3 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	13C
P1	10.00 usec
PL1	3.80 dB
SFO1	100.6233325 MHz
======== CHANNEL f2 ========	
CPDPRG2	waltz16
NUC2	1H
PCPD2	90.00 usec
PL2	10.20 dB
PL12	26.00 dB
PL13	29.00 dB
SFO2	400.1316005 MHz
SI	32768
SF	100.6127688 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.00

3-(2-(3-methoxyphenyl)-1*H*-inden-3-yl)-1-methyl-1*H*-indole (3ca)




```
Current Data Parameters
NAME                  20221105
EXPNO                        6
PROCNO                       1

F2 - Acquisition Parameters
Date_                 20221105
Time                     22.12
INSTRUM                  spect
PROBHD    5 mm BBO BB-1H
PULPROG                  zg30
TD                      32768
SOLVENT                  CDCl3
NS                          16
DS                           0
SWH                  7246.377 Hz
FIDRES               0.221142 Hz
AQ                   2.2609921 sec
RG                         114
DW                      69.000 usec
DE                       6.50 usec
TE                      296.5 K
D1                   2.00000000 sec
TD0                          1

======== CHANNEL f1 ========
NUC1                        1H
P1                       15.00 usec
PL1                      11.10 dB
SFO1                400.1324008 MHz

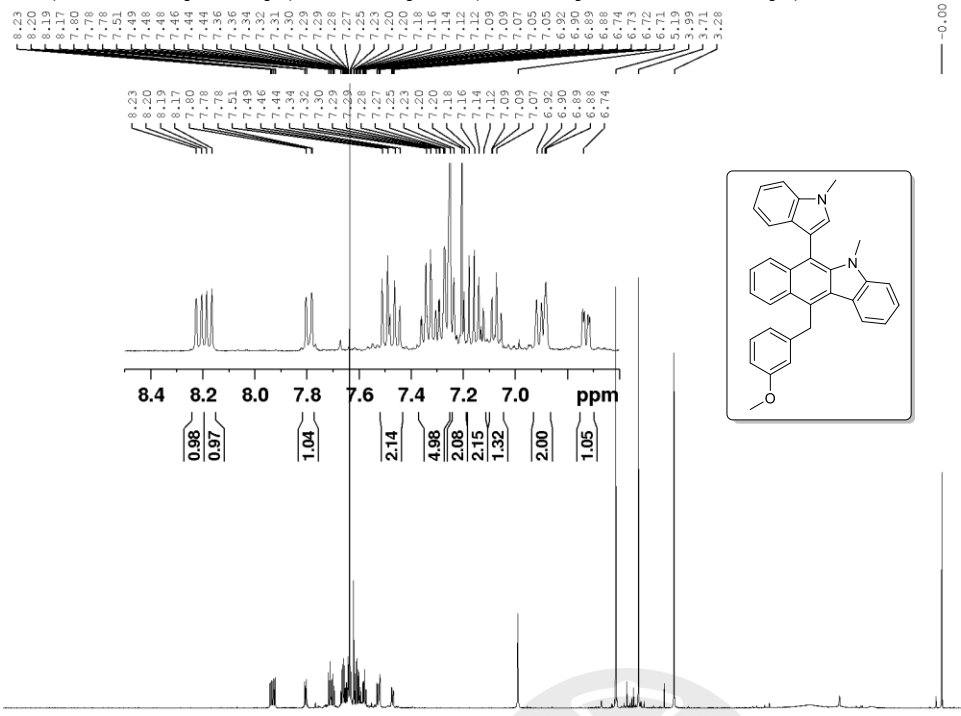
F2 - Processing parameters
SI                          16384
SF                  400.1300118 MHz
WDW                            EM
SSB                          0
LB                           0 Hz
GB                           0
PC                        1.00
```

```
NAME      Aldehyde+indole
EXPNO                    32
PROCNO                    1
Date_              20221106
Time                   9.40
INSTRUM               spect
PROBHD    5 mm BBO BB-1H
PULPROG             zgpg30
TD                     32768
SOLVENT                CDCl3
NS                     15000
DS                         0
SWH                24038.461 Hz
FIDRES             0.733596 Hz
AQ                 0.6816452 sec
RG                    9195.2
DW                    20.800 usec
DE                     6.50 usec
TE                    297.6 K
D1                 2.00000000 sec
D11                0.03000000 sec
TD0                        1

======== CHANNEL f1 ========
NUC1                      13C
P1                     10.00 usec
PL1                     3.80 dB
SFO1              100.6233325 MHz

======== CHANNEL f2 ========
CPDPRG2              waltz16
NUC2                      1H
PCPD2                 90.00 usec
PL2                   10.20 dB
PL12                  26.00 dB
PL13                  29.00 dB
SFO2              400.1316005 MHz
SI                     32768
SF                100.6127736 MHz
WDW                       EM
SSB                        0
LB                      1.00 Hz
GB                         0
PC                      1.00
```

11-(3-methoxybenzyl)-5-methyl-6-(1-methyl-1*H*-indol-3-yl)-5*H*-benzo[*b*]carbazole (5ca)

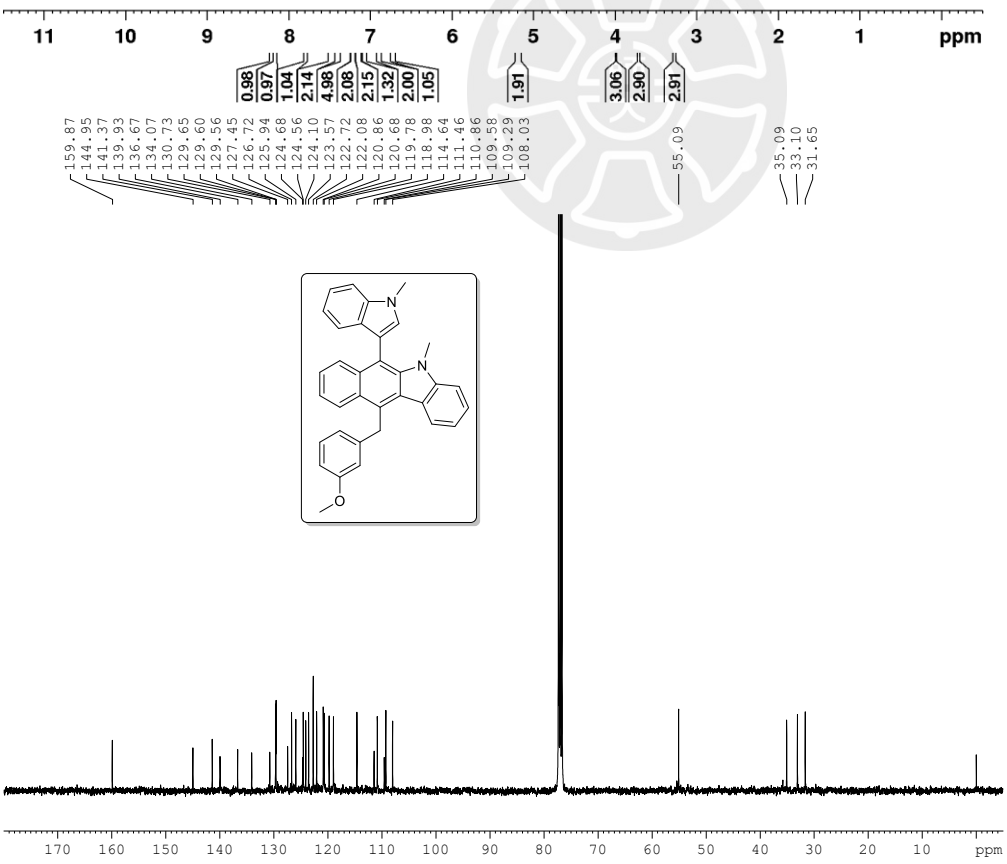


```
Current Data Parameters
NAME                  20230201(3)
EXPNO                           3
PROCNO                          1

F2 - Acquisition Parameters
Date_                    20230201
Time                        22.41
INSTRUM                     spect
PROBHD    5 mm PABBO BB/
PULPROG                     zg30
TD                         32768
SOLVENT                    CDC13
NS                            16
DS                             0
SWH                     7211.539 Hz
FIDRES                  0.220079 Hz
AQ                      2.2719147 sec
RG                          128.9
DW                         69.333 usec
DE                         10.06 usec
TE                         299.0 K
D1                    2.00000000 sec
TD0                            1

======== CHANNEL f1 ========
SFO1                 400.1324008 MHz
NUC1                          1H
P1                         15.00 usec
PLW1                13.50000000 W

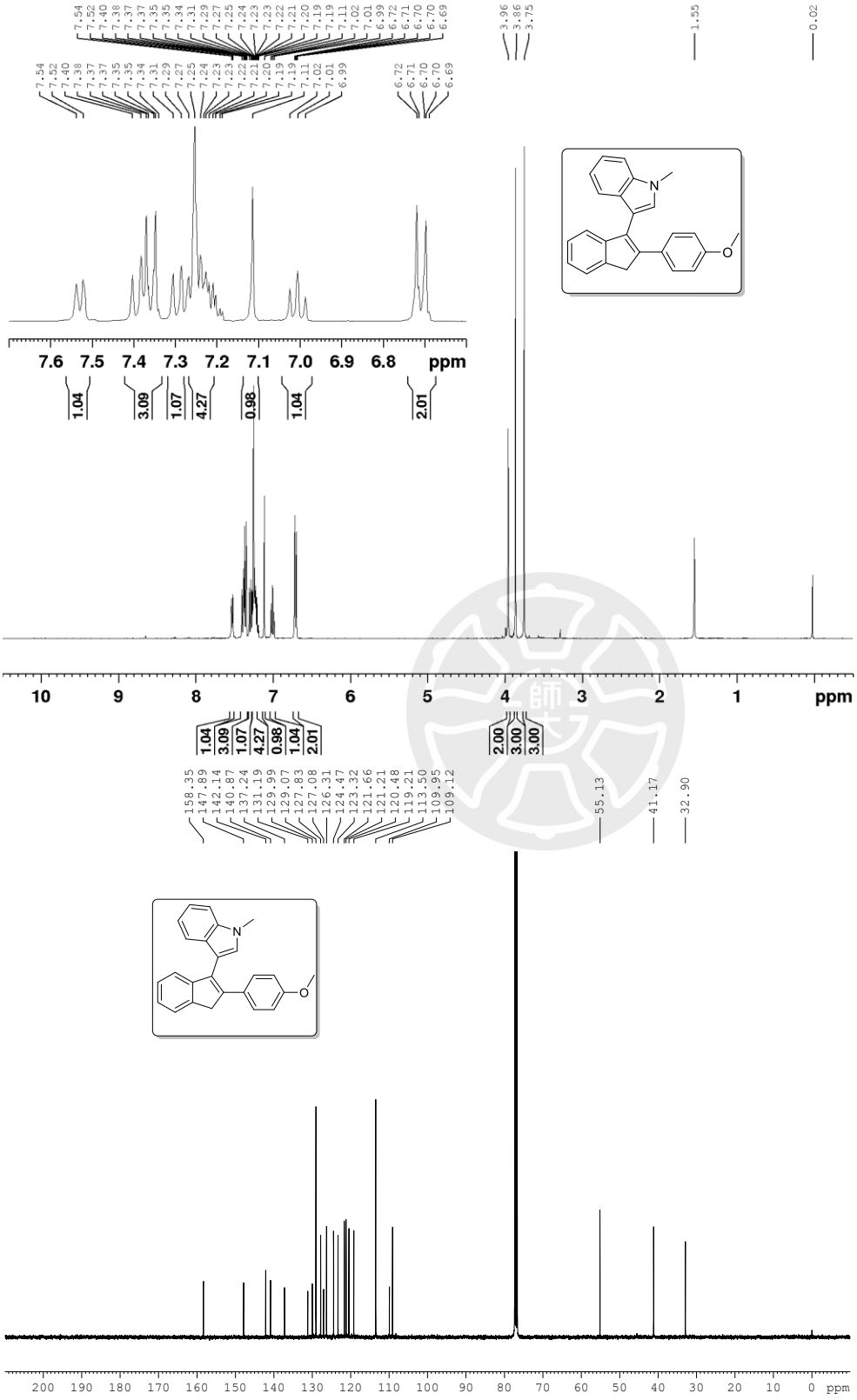
F2 - Processing parameters
SI                         16384
SF                   400.1300142 MHz
WDW                           EM
SSB                            0
LB                             0 Hz
GB                             0
PC                          1.00
```



```
NAME          Aldehyde+indole
EXPNO                      34
PROCNO                      1
Date_                20230202
Time                     1.00
INSTRUM                 spect
PROBHD    5 mm PABBO BB/
PULPROG                 zgpg30
TD                         32768
SOLVENT                    CDC13
NS                          3000
DS                             0
SWH                    24038.461 Hz
FIDRES                  0.733596 Hz
AQ                      0.6816244 sec
RG                          198.09
DW                         20.800 usec
DE                          6.50 usec
TE                         300.5 K
D1                    2.00000000 sec
D11                   0.03000000 sec
TD0                            1

======== CHANNEL f1 ========
SFO1                 100.6228298 MHz
NUC1                          13C
P1                         10.00 usec
SI                         32768
SF                   100.6127710 MHz
WDW                           EM
SSB                            0
LB                          2.00 Hz
GB                             0
PC                          1.00
```

3-(2-(4-Methoxyphenyl)-1*H*-inden-3-yl)-1-methyl-1*H*-indole (3da)



```
Current Data Parameters
NAME                 20221020
EXPNO                       1
PROCNO                      1

F2 - Acquisition Parameters
Date_               20221020
Time                   22.30
INSTRUM                spect
PROBHD    5 mm BBO BB-1H
PULPROG                zg30
TD                    32768
SOLVENT               CDCl3
NS                       16
DS                        0
SWH                7246.377 Hz
FIDRES            0.221142 Hz
AQ                2.2609921 sec
RG                      181
DW                   69.000 usec
DE                    6.50 usec
TE                   300.3 K
D1                2.00000000 sec
TD0                         1

======== CHANNEL f1 ========
NUC1                       1H
P1                      15.00 usec
PL1                     11.10 dB
SFO1            400.1324008 MHz

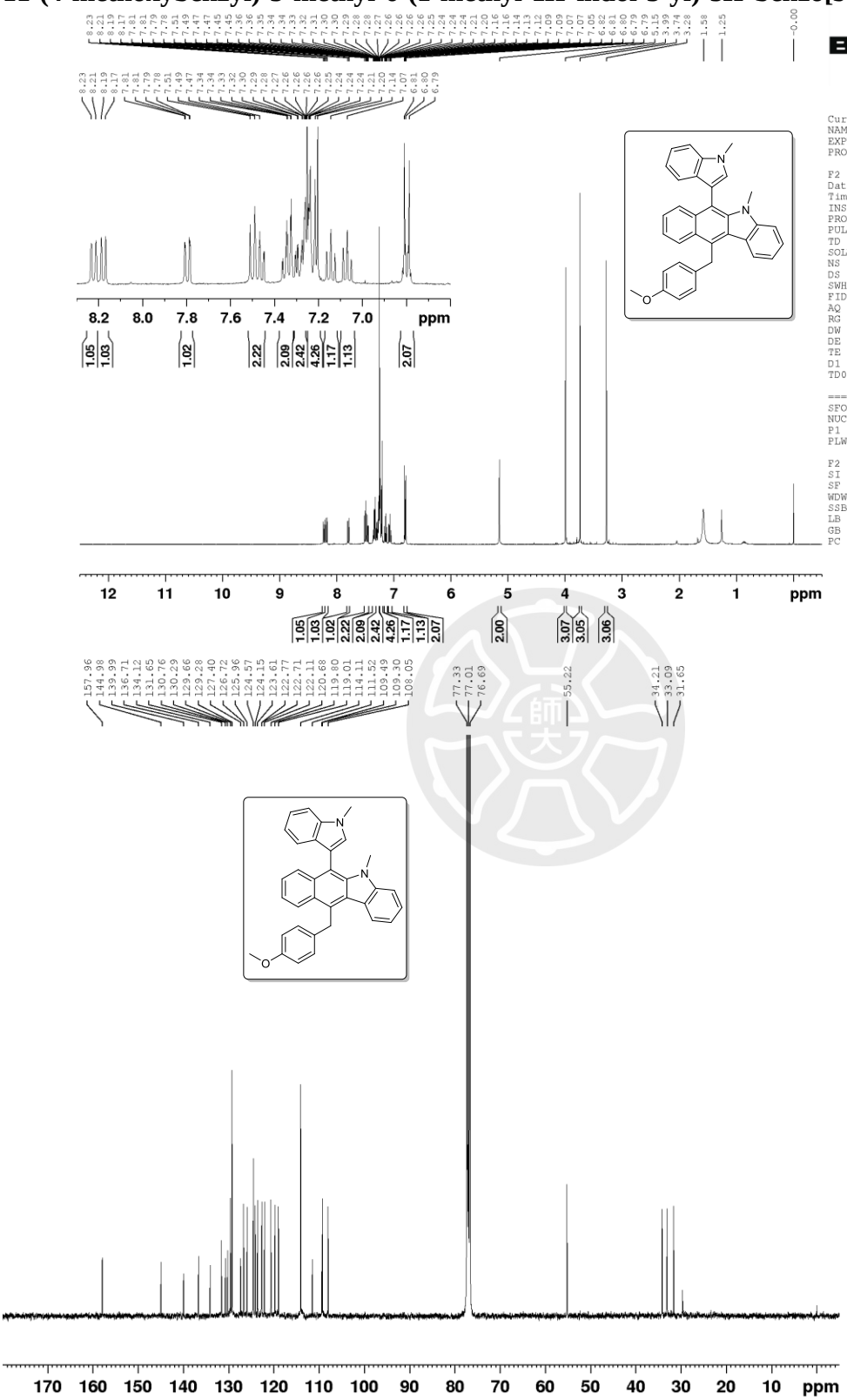
F2 - Processing parameters
SI                        16384
SF                400.1300118 MHz
WDW                          EM
SSB                           0
LB                            0 Hz
GB                            0
PC                         1.00
```

```
NAME          Aldehyde+indole
EXPNO                      20
PROCNO                      1
Date_               20221021
Time                    1.13
INSTRUM                spect
PROBHD    5 mm BBO BB-1H
PULPROG             zgpg30
TD                    32768
SOLVENT               CDCl3
NS                     3557
DS                        0
SWH               24038.461 Hz
FIDRES            0.733596 Hz
AQ                0.6816452 sec
RG                     4096
DW                   20.800 usec
DE                    6.50 usec
TE                   301.6 K
D1                2.00000000 sec
D11               0.03000000 sec
TD0                         1

======== CHANNEL f1 ========
NUC1                      13C
P1                      10.00 usec
PL1                      3.80 dB
SFO1            100.6233325 MHz

======== CHANNEL f2 ========
CPDPRG2             waltz16
NUC2                       1H
PCPD2                  90.00 usec
PL2                    10.20 dB
PL12                   26.00 dB
PL13                   29.00 dB
SFO2            400.1316005 MHz
SI                    32768
SF                100.6127719 MHz
WDW                        EM
SSB                         0
LB                       1.00 Hz
GB                          0
PC                       1.00
```

11-(4-methoxybenzyl)-5-methyl-6-(1-methyl-1*H*-indol-3-yl)-5*H*-benzo[*b*]carbazole (5da)



```
Current Data Parameters
NAME          Suresh-660
EXPNO                  2
PROCNO                 1

F2 - Acquisition Parameters
Date_           20230821
Time              22.09
INSTRUM           spect
PROBHD   5 mm PABBO BB/
PULPROG            zg30
TD                32768
SOLVENT           CDCl3
NS                   16
DS                    0
SWH            7211.539 Hz
FIDRES        0.220079 Hz
AQ            2.2719147 sec
RG               144.49
DW              69.333 usec
DE              10.06 usec
TE              298.6 K
D1            2.00000000 sec
TD0                   1

======== CHANNEL f1 ========
SFO1         400.1324008 MHz
NUC1                 1H
P1                15.00 usec
PLW1         13.50000000 W

F2 - Processing parameters
SI                16384
SF           400.1300134 MHz
WDW                  EM
SSB                   0
LB                 0 Hz
GB                    0
PC                 1.00
```

```
Current Data Parameters
NAME          Suresh-660
EXPNO                  3
PROCNO                 1

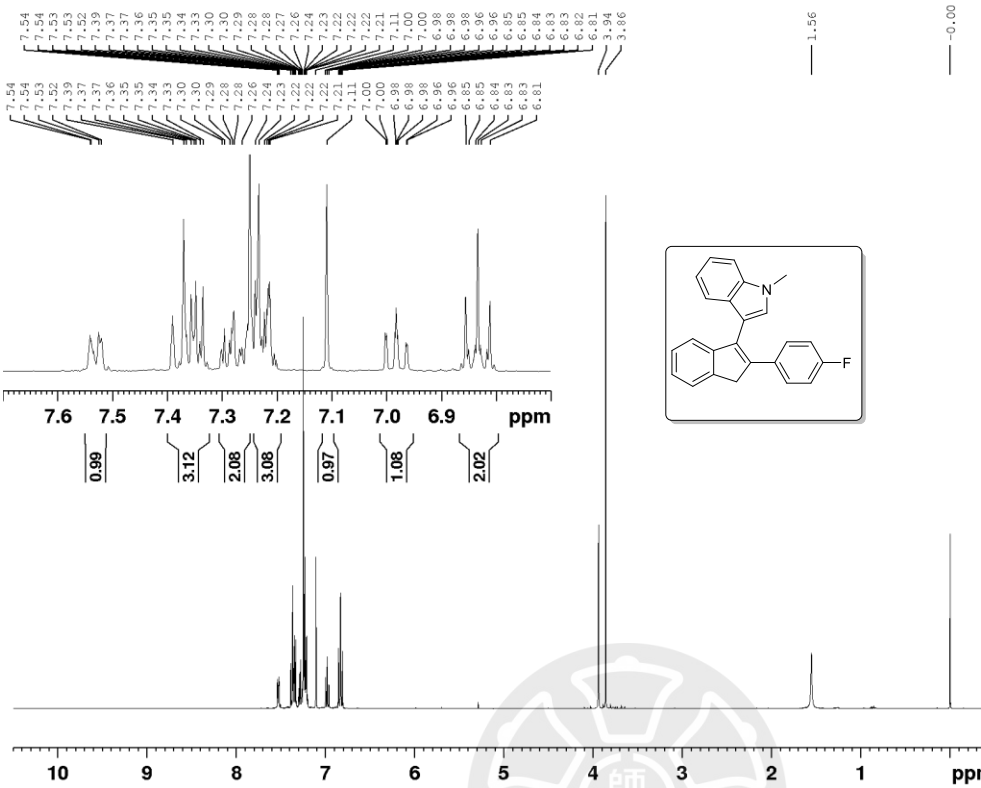
F2 - Acquisition Parameters
Date_           20230821
Time              22.11
INSTRUM           spect
PROBHD   5 mm PABBO BB/
PULPROG          zgpg30
TD                32768
SOLVENT           CDCl3
NS                14139
DS                    0
SWH           24038.461 Hz
FIDRES         0.733596 Hz
AQ            0.6815744 sec
RG               198.09
DW              20.800 usec
DE               6.50 usec
TE              298.6 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
SFO1         100.6228298 MHz
NUC1                 13C
P1                10.00 usec
PLW1         49.50000000 W

======== CHANNEL f2 ========
SFO2         400.1316005 MHz
NUC2                 1H
CPDPRG[2          waltz16
PCPD2             90.00 usec
PLW2         12.50000000 W
PLW12         0.34722000 W
PLW13         0.28125000 W

F2 - Processing parameters
SI                32768
SF           100.6127685 MHz
WDW                  EM
SSB                   0
LB                 2.00 Hz
GB                    0
PC                 1.00
```

3-(2-(4-fluorophenyl)-1*H*-inden-3-yl)-1-methyl-1*H*-indole (3ea)

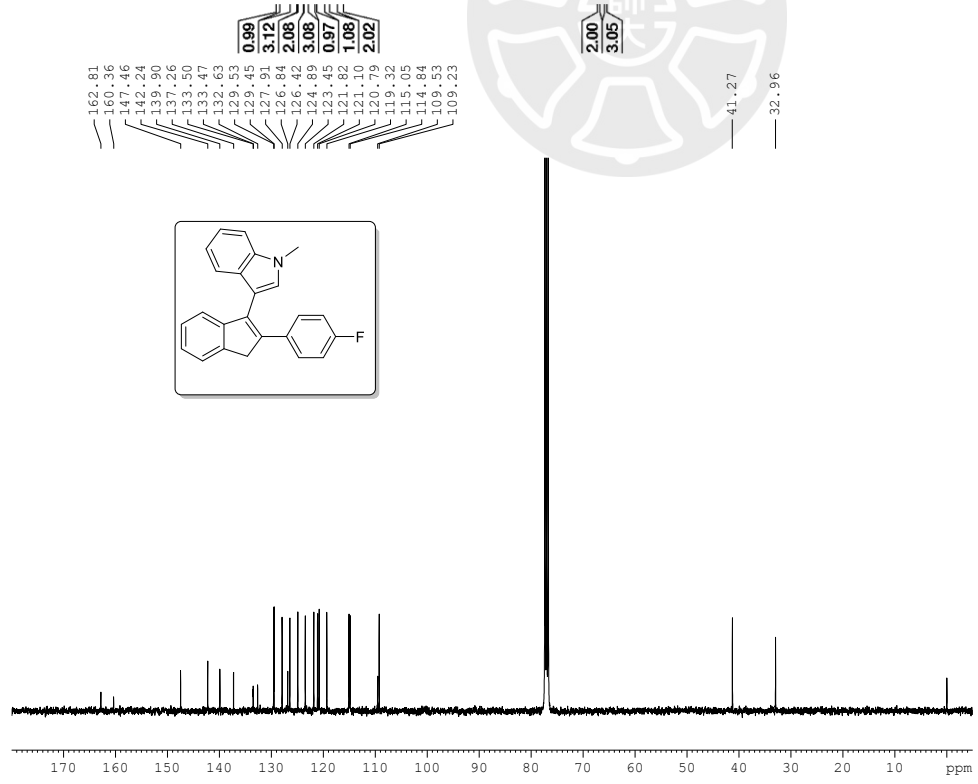


Current Data Parameters
NAME Aldehyde+indole
EXPNO 43
PROCNO 1

F2 - Acquisition Parameters
Date_ 20230624
Time 2.13
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 32768
SOLVENT CDC13
NS 12
DS 0
SWH 7211.539 Hz
FIDRES 0.220079 Hz
AQ 2.2719147 sec
RG 144.49
DW 69.333 usec
DE 10.06 usec
TE 299.1 K
D1 2.00000000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 400.1324008 MHz
NUC1 1H
P1 15.00 usec
PLW1 13.50000000 W

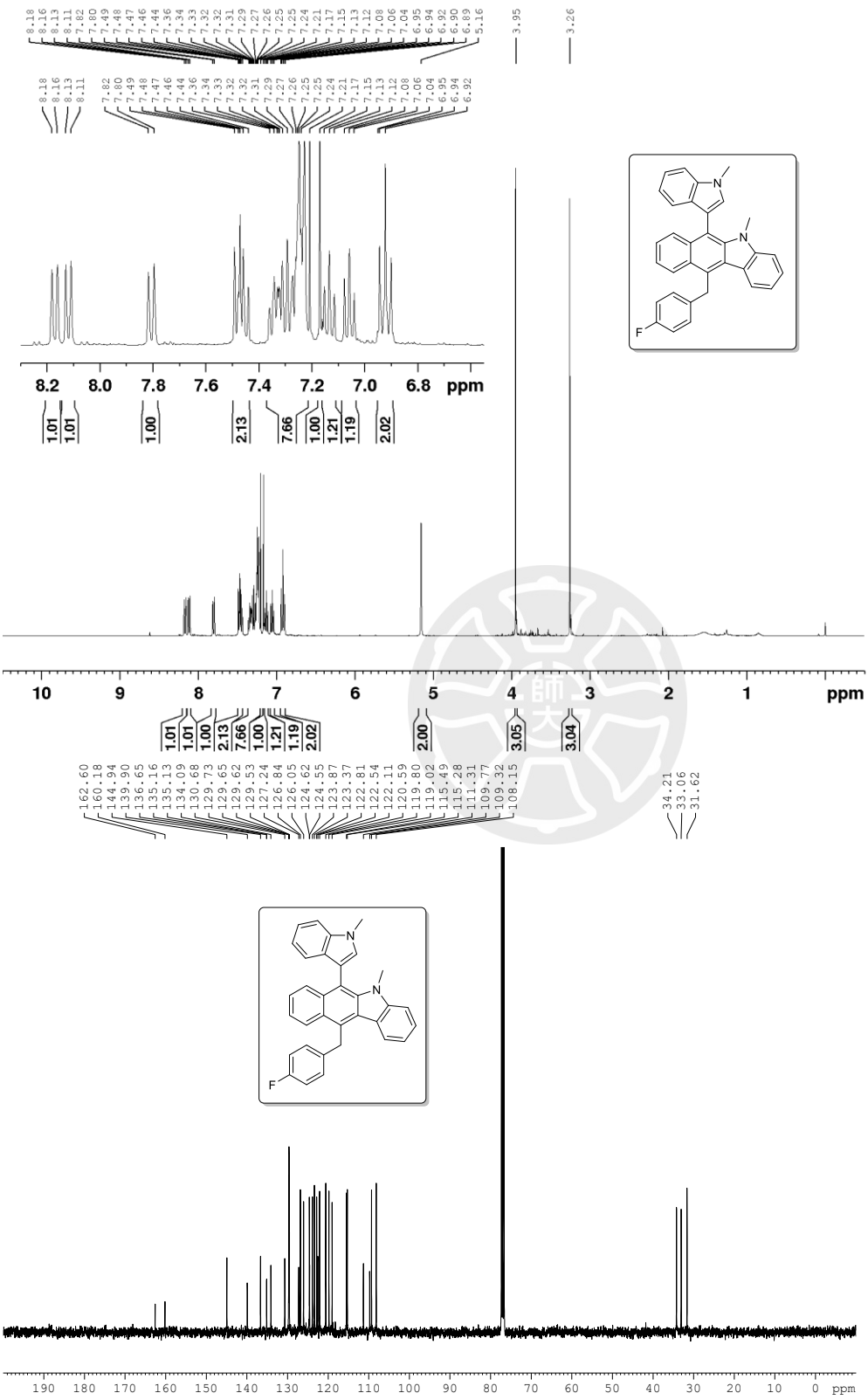
F2 - Processing parameters
SI 16384
SF 400.1300140 MHz
WDW EM
SSB 0
LB 0 Hz
GB 0
PC 1.00



NAME Aldehyde+indole
EXPNO 64
PROCNO 1
Date_ 20230614
Time 5.11
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 32768
SOLVENT CDC13
NS 2274
DS 0
SWH 24038.461 Hz
FIDRES 0.733596 Hz
AQ 0.6816244 sec
RG 198.09
DW 20.800 usec
DE 6.50 usec
TE 298.5 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 100.6228298 MHz
NUC1 13C
P1 10.00 usec
SI 32768
SF 100.6127685 MHz
WDW EM
SSB 0
LB 2.00 Hz
GB 0
PC 1.00

11-(4-fluorobenzyl)-5-methyl-6-(1-methyl-1*H*-indol-3-yl)-5*H*-benzo[*b*]carbazole (5ea)



```
Current Data Parameters
NAME         20230729(3)
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_          20230729
Time             16.10
INSTRUM           spect
PROBHD   5 mm PABBO BB/
PULPROG          zg30
TD              32768
SOLVENT          CDCl3
NS                  12
DS                   0
SWH           7211.539 Hz
FIDRES        0.220079 Hz
AQ            2.2719147 sec
RG                71.42
DW               69.333 usec
DE               10.06 usec
TE               298.2 K
D1          2.00000000 sec
TD0                  1

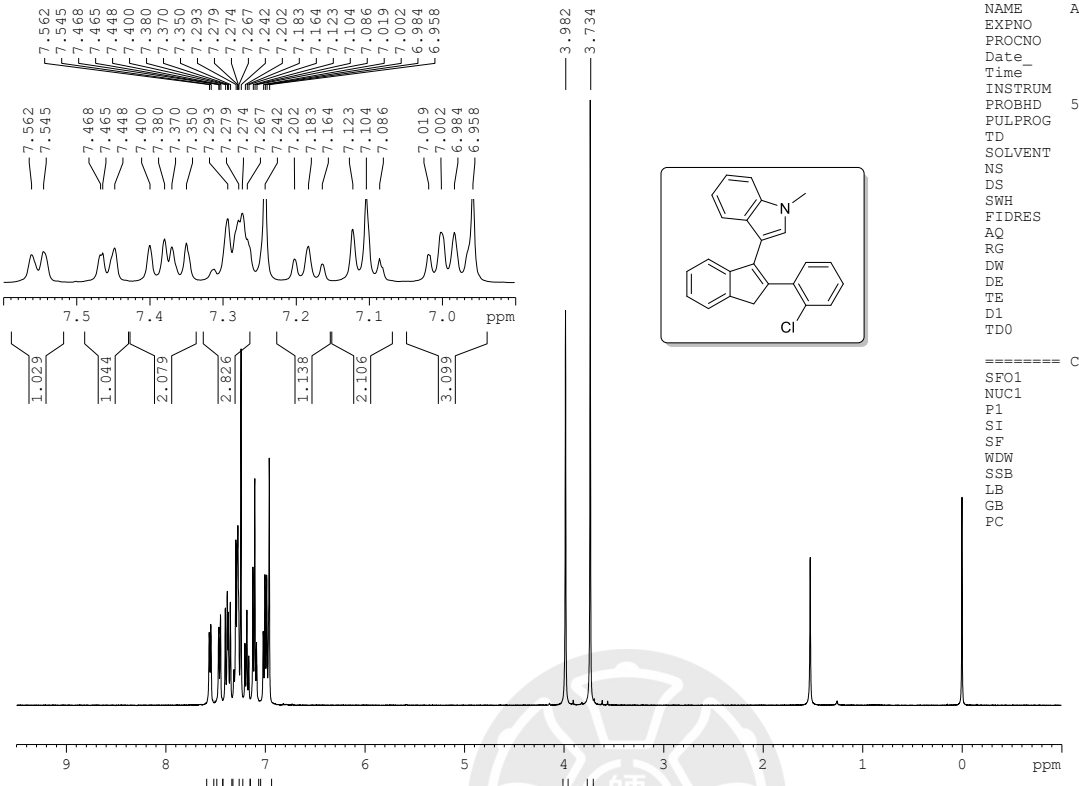
======== CHANNEL f1 ========
SFO1       400.1324008 MHz
NUC1               1H
P1               15.00 usec
PLW1       13.50000000 W

F2 - Processing parameters
SI               16384
SF         400.1300302 MHz
WDW                EM
SSB                 0
LB                  0 Hz
GB                  0
PC               1.00
```

```
NAME         20230729(3)
EXPNO                  2
PROCNO                 1
Date_          20230729
Time             16.16
INSTRUM           spect
PROBHD   5 mm PABBO BB/
PULPROG        zgpg30
TD              32768
SOLVENT          CDCl3
NS                 217
DS                   0
SWH          24038.461 Hz
FIDRES        0.733596 Hz
AQ            0.6816244 sec
RG               198.09
DW               20.800 usec
DE                6.50 usec
TE               299.2 K
D1          2.00000000 sec
D11         0.03000000 sec
TD0                  1

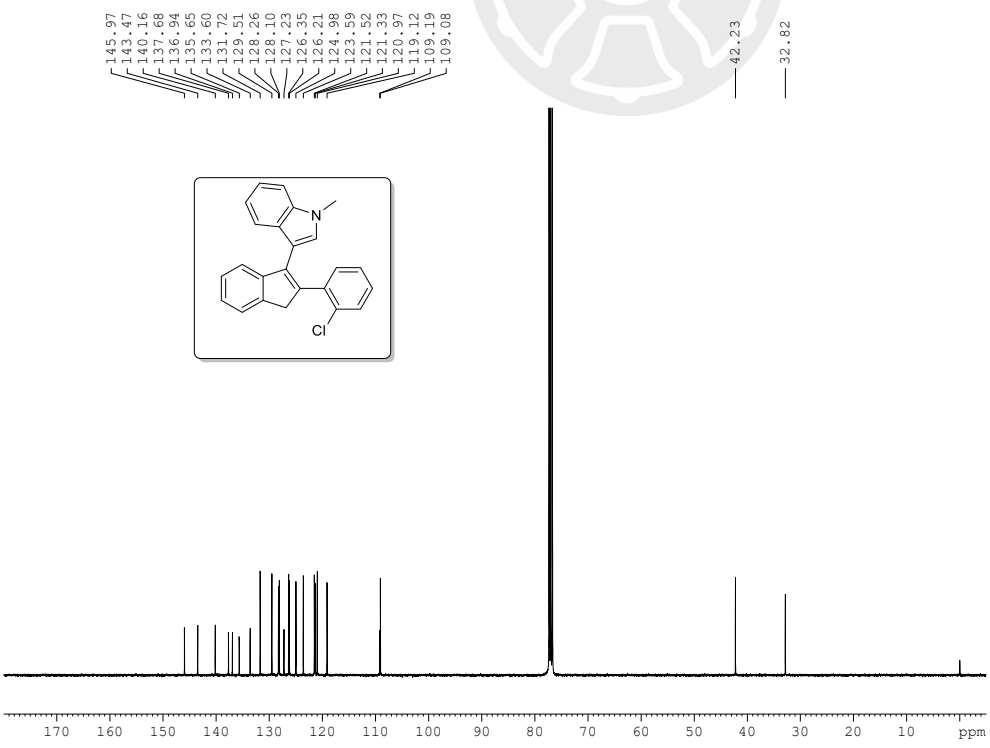
======== CHANNEL f1 ========
SFO1       100.6228298 MHz
NUC1               13C
P1               10.00 usec
SI               32768
SF         100.6127758 MHz
WDW                EM
SSB                 0
LB               2.00 Hz
GB                  0
PC               1.00
```

3-(2-(2-chlorophenyl)-1*H*-inden-3-yl)-1-methyl-1*H*-indole (3fa)




```
NAME      Aldehyde+indole
EXPNO                  11
PROCNO                  1
Date_            20221010
Time                22.32
INSTRUM             spect
PROBHD   5 mm PABBO BB/
PULPROG              zg30
TD                  32768
SOLVENT             CDCl3
NS                     16
DS                      0
SWH             7211.539 Hz
FIDRES          0.220079 Hz
AQ             2.2719646 sec
RG                  128.9
DW                 69.333 usec
DE                 10.06 usec
TE                 297.6 K
D1            2.00000000 sec
TD0                     1

======== CHANNEL f1 ========
SFO1         400.1324008 MHz
NUC1                   1H
P1                  15.00 usec
SI                  16384
SF           400.1300168 MHz
WDW                    EM
SSB                     0
LB                   0.00 Hz
GB                      0
PC                   1.00
```

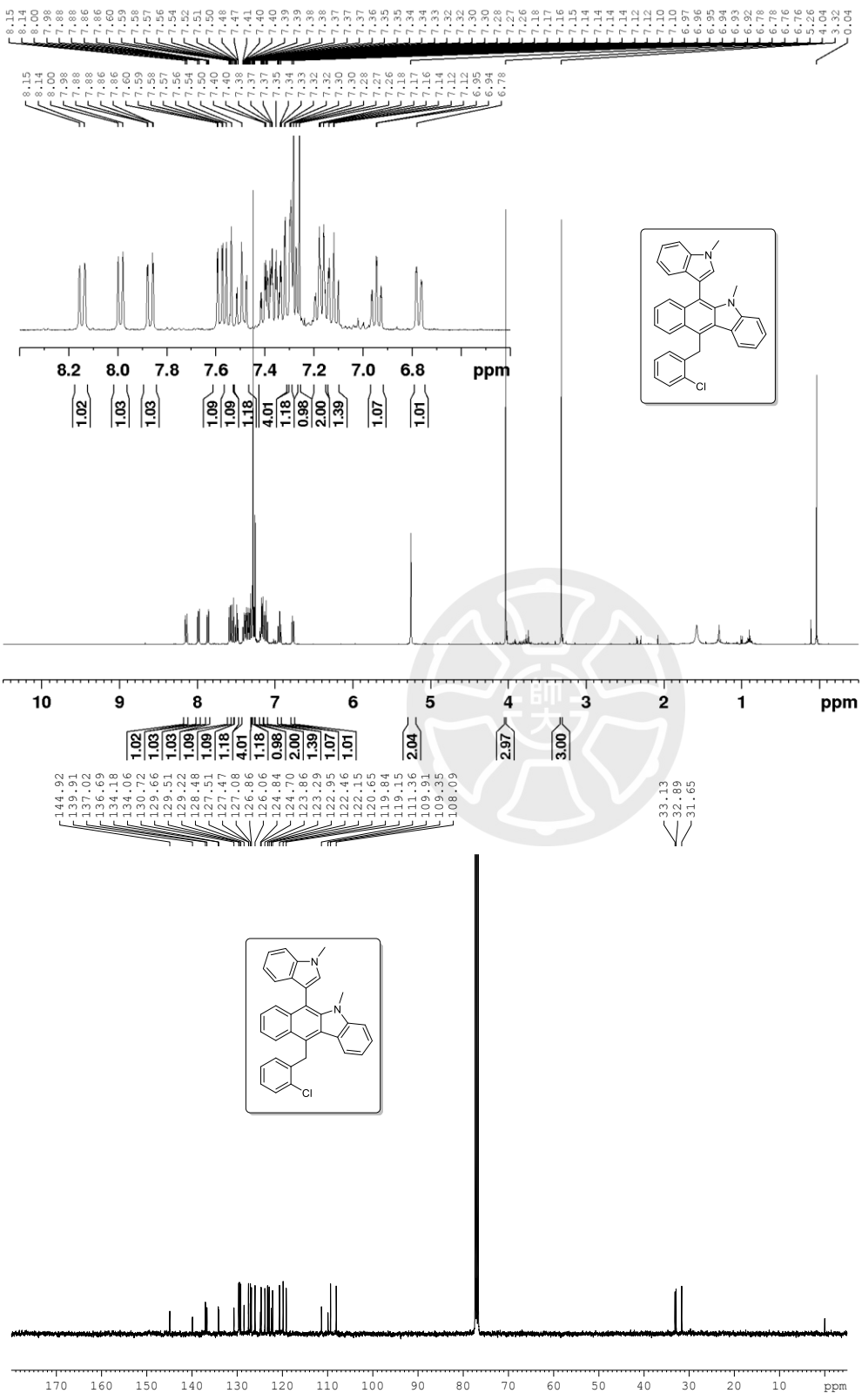



```
NAME      Aldehyde+indole
EXPNO                  12
PROCNO                  1
Date_            20221014
Time                22.04
INSTRUM             spect
PROBHD   5 mm BBO BB-1H
PULPROG           zgpg30
TD                  32768
SOLVENT             CDCl3
NS                  14532
DS                      0
SWH            24038.461 Hz
FIDRES          0.733596 Hz
AQ             0.6816452 sec
RG                 9195.2
DW                 20.800 usec
DE                  6.50 usec
TE                 300.4 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0                     1

======== CHANNEL f1 ========
NUC1                   13C
P1                  10.00 usec
PL1                  3.80 dB
SFO1         100.6233325 MHz

======== CHANNEL f2 ========
CPDPRG2             waltz16
NUC2                   1H
PCPD2               90.00 usec
PL2                 10.20 dB
PL12                26.00 dB
PL13                29.00 dB
SFO2         400.1316005 MHz
SI                  32768
SF           100.6127699 MHz
WDW                    EM
SSB                     0
LB                   1.00 Hz
GB                      0
PC                   1.00
```

11-(2-chlorobenzyl)-5-methyl-6-(1-methyl-1*H*-indol-3-yl)-5*H*-benzo[*b*]carbazole (5fa)



```
Current Data Parameters
NAME         20230131(3)
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20230131
Time               17.33
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG             zg30
TD                 32768
SOLVENT            CDCl3
NS                    16
DS                     0
SWH             7211.539 Hz
FIDRES          0.220079 Hz
AQ              2.2719147 sec
RG                 128.9
DW                69.333 usec
DE                10.06 usec
TE                299.2 K
D1             2.00000000 sec
TD0                    1

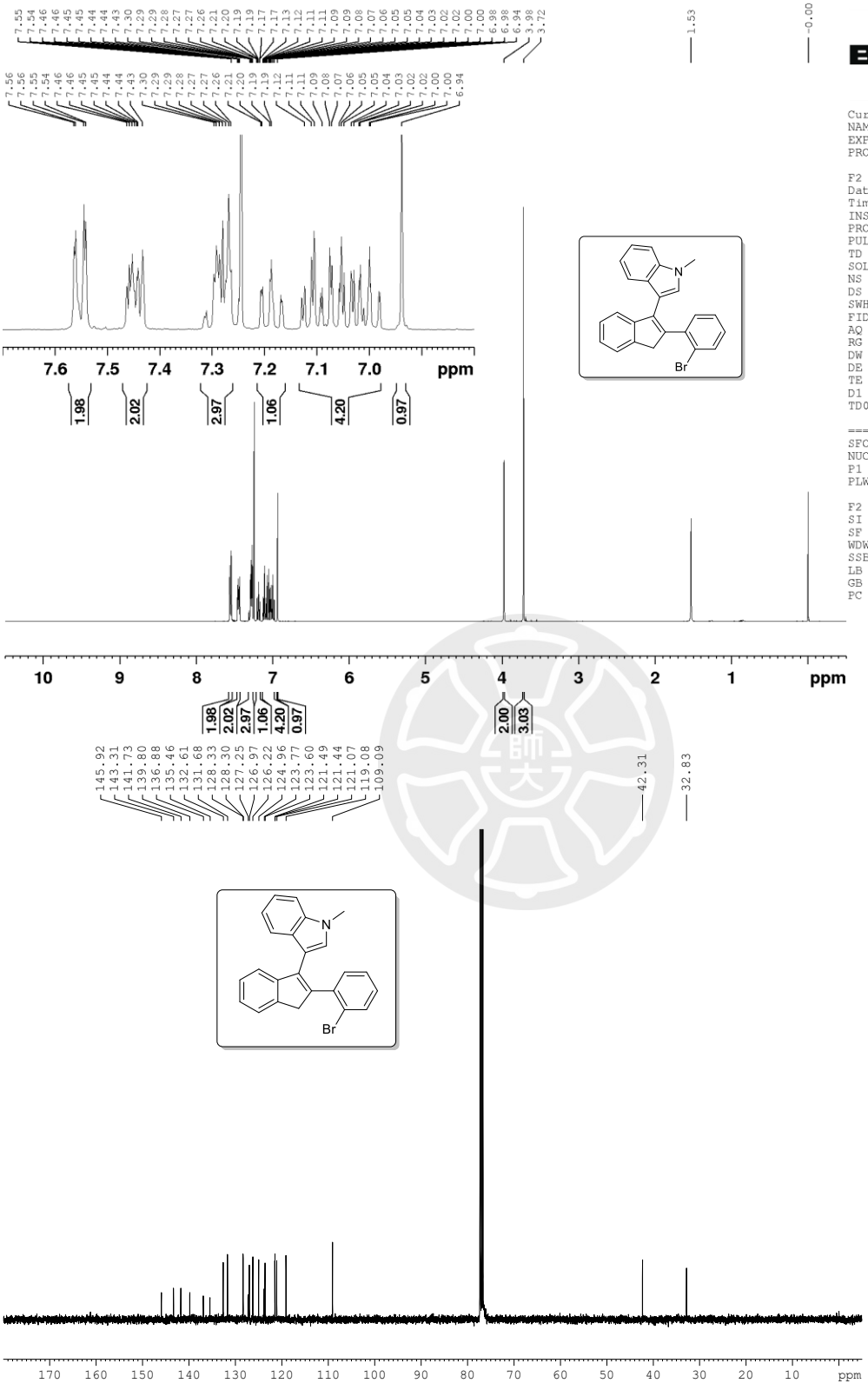
======== CHANNEL f1 ========
SFO1          400.1324008 MHz
NUC1                  1H
P1                 15.00 usec
PLW1          13.50000000 W

F2 - Processing parameters
SI                 16384
SF            400.1300000 MHz
WDW                   EM
SSB                    0
LB                  0 Hz
GB                     0
PC                  1.00
```

```
NAME      Aldehyde+indole
EXPNO                  14
PROCNO                  1
Date_           20230131
Time               17.36
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG          zgpg30
TD                 32768
SOLVENT            CDCl3
NS                  1015
DS                     0
SWH            24038.461 Hz
FIDRES          0.733596 Hz
AQ              0.6816244 sec
RG                 198.09
DW                20.800 usec
DE                 6.50 usec
TE                299.9 K
D1             2.00000000 sec
D11            0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1          100.6228298 MHz
NUC1                  13C
P1                 10.00 usec
SI                 32768
SF            100.6127697 MHz
WDW                   EM
SSB                    0
LB                  2.00 Hz
GB                     0
PC                  1.00
```

3-(2-(2-bromophenyl)-1*H*-inden-3-yl)-1-methyl-1*H*-indole (3ga)



```
Current Data Parameters
NAME      Aldehyde+indole
EXPNO                  27
PROCNO                  1

F2 - Acquisition Parameters
Date_           20230624
Time                1.36
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG             zg30
TD                 32768
SOLVENT            CDCl3
NS                    12
DS                     0
SWH             7211.539 Hz
FIDRES          0.220079 Hz
AQ             2.2719147 sec
RG                 128.9
DW                69.333 usec
DE                 10.06 usec
TE                 299.2 K
D1            2.00000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1         400.1324008 MHz
NUC1                   1H
P1                 15.00 usec
PLW1         13.50000000 W

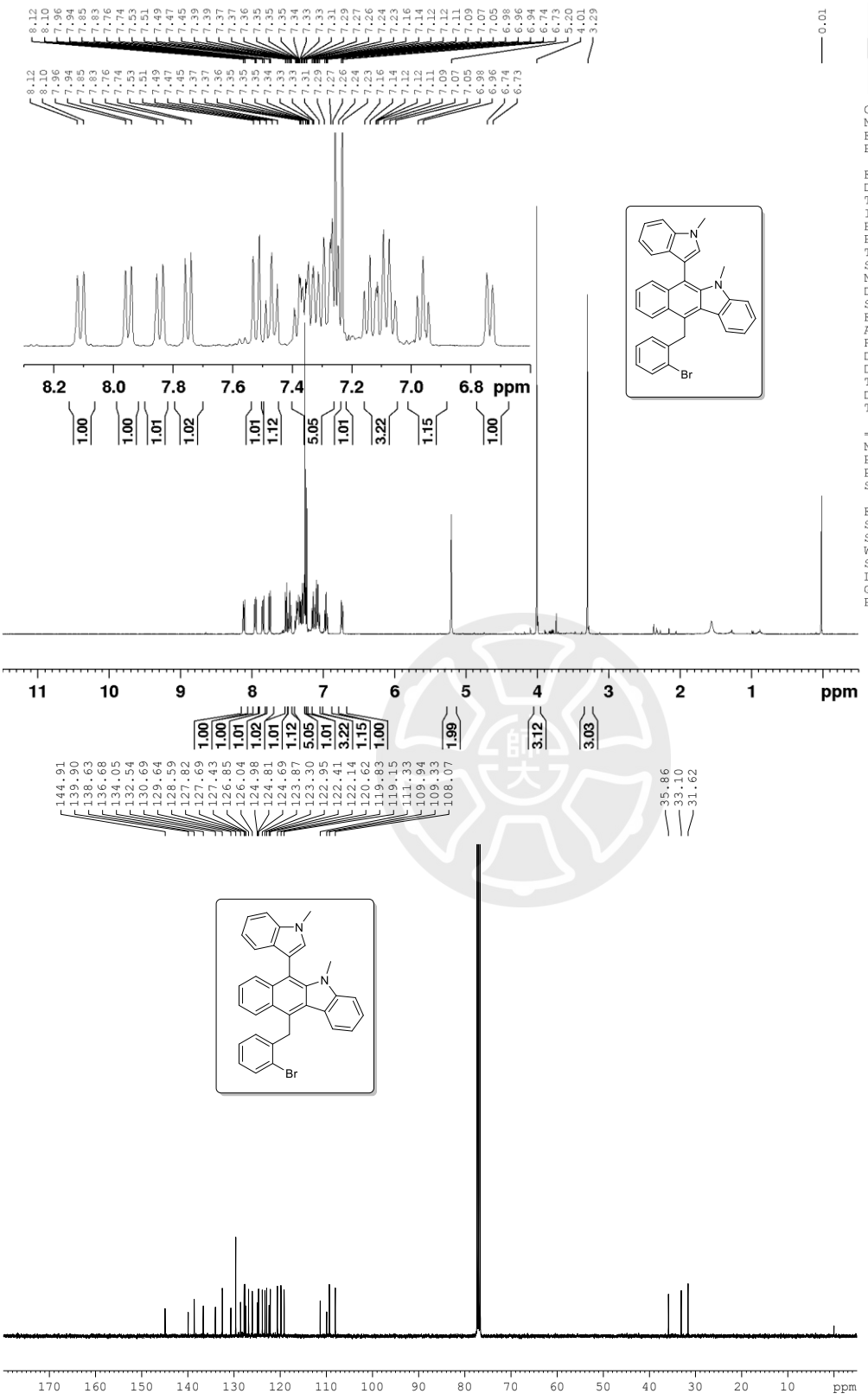
F2 - Processing parameters
SI                 16384
SF           400.1300156 MHz
WDW                   EM
SSB                    0
LB                     0 Hz
GB                     0
PC                  1.00
```

```
NAME      Aldehyde+indole
EXPNO                  28
PROCNO                  1
Date_           20221105
Time               21.48
INSTRUM            spect
PROBHD   5 mm BBO BB-1H
PULPROG           zgpg30
TD                 32768
SOLVENT            CDCl3
NS                  1013
DS                     0
SWH            24038.461 Hz
FIDRES          0.733596 Hz
AQ             0.6816452 sec
RG                9195.2
DW                20.800 usec
DE                 6.50 usec
TE                297.5 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  13C
P1                 10.00 usec
PL1                 3.80 dB
SFO1         100.6233325 MHz

======== CHANNEL f2 ========
CPDPRG2           waltz16
NUC2                   1H
PCPD2              90.00 usec
PL2                10.20 dB
PL12               26.00 dB
PL13               29.00 dB
SFO2         400.1316005 MHz
SI                 32768
SF           100.6127710 MHz
WDW                   EM
SSB                    0
LB                  1.00 Hz
GB                     0
PC                  1.00
```

11-(2-bromobenzyl)-5-methyl-6-(1-methyl-1*H*-indol-3-yl)-5*H*-benzo[*b*]carbazole (5ga)



```
Current Data Parameters
NAME      Aldehyde+indole
EXPNO                  29
PROCNO                  1

F2 - Acquisition Parameters
Date_           20230119
Time               6.10
INSTRUM           spect
PROBHD   5 mm BBO BB-1H
PULPROG            zg30
TD                32768
SOLVENT           CDCl3
NS                   16
DS                    0
SWH            7246.377 Hz
FIDRES        0.221142 Hz
AQ            2.2609921 sec
RG                  181
DW               69.000 usec
DE                6.50 usec
TE               297.6 K
D1            2.00000000 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                 1H
P1                15.00 usec
PL1               11.10 dB
SFO1        400.1324008 MHz

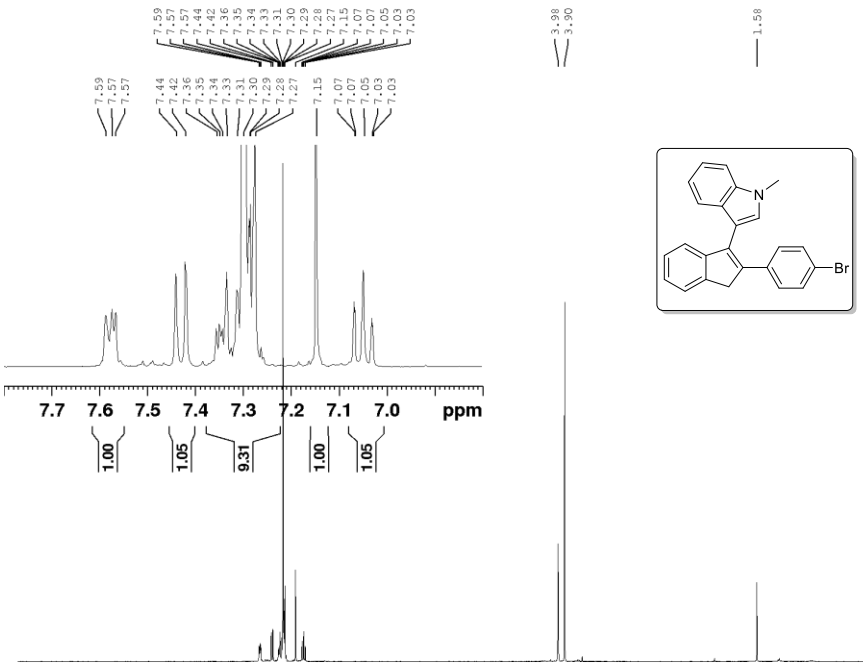
F2 - Processing parameters
SI                16384
SF          400.1300118 MHz
WDW                  EM
SSB                   0
LB                 0 Hz
GB                    0
PC                 1.00
```

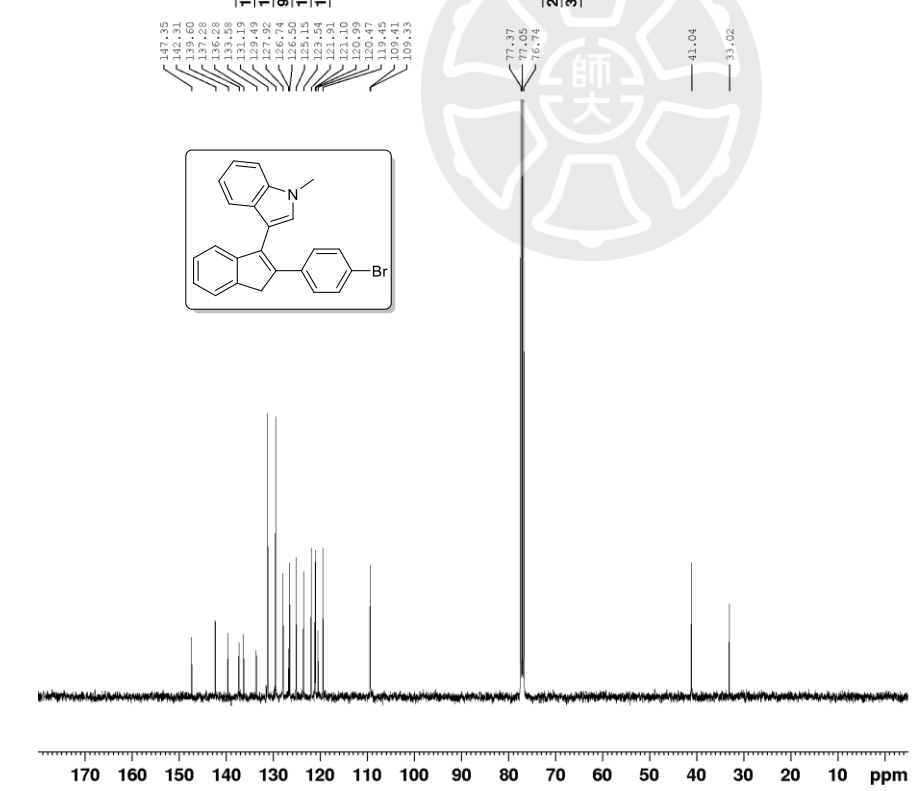
```
NAME      Aldehyde+indole
EXPNO                30
PROCNO                1
Date_          20230119
Time              10.58
INSTRUM           spect
PROBHD   5 mm BBO BB-1H
PULPROG         zgpg30
TD                32768
SOLVENT           CDCl3
NS                 6253
DS                    0
SWH           24038.461 Hz
FIDRES         0.733596 Hz
AQ            0.6816452 sec
RG               9195.2
DW               20.800 usec
DE                6.50 usec
TE               298.5 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                 13C
P1                52.00 usec
PL1               -6.00 dB
SFO1        100.6233325 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                 1H
PCPD2             90.00 usec
PL2               10.20 dB
PL12              26.00 dB
PL13              29.00 dB
SFO2        400.1316005 MHz
SI                32768
SF          100.6127727 MHz
WDW                  EM
SSB                   0
LB                 1.00 Hz
GB                    0
PC                 1.00
```

3-(2-(4-bromophenyl)-1*H*-inden-3-yl)-1-methyl-1*H*-indole (3ha)






```
Current Data Parameters
NAME          Suresh-692
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20230922
Time               10.29
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG             zg30
TD                 32768
SOLVENT            CDC13
NS                    16
DS                     0
SWH             7211.539 Hz
FIDRES          0.220079 Hz
AQ              2.2719147 sec
RG                 99.72
DW                69.333 usec
DE                10.06 usec
TE                298.1 K
D1            2.00000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1         400.1324008 MHz
NUC1                   1H
P1                 15.00 usec
PLW1         13.50000000 W

F2 - Processing parameters
SI                 16384
SF           400.1300000 MHz
WDW                   EM
SSB                    0
LB                     0 Hz
GB                     0
PC                  1.00
```

```
Current Data Parameters
NAME          Suresh-692
EXPNO                  2
PROCNO                 1

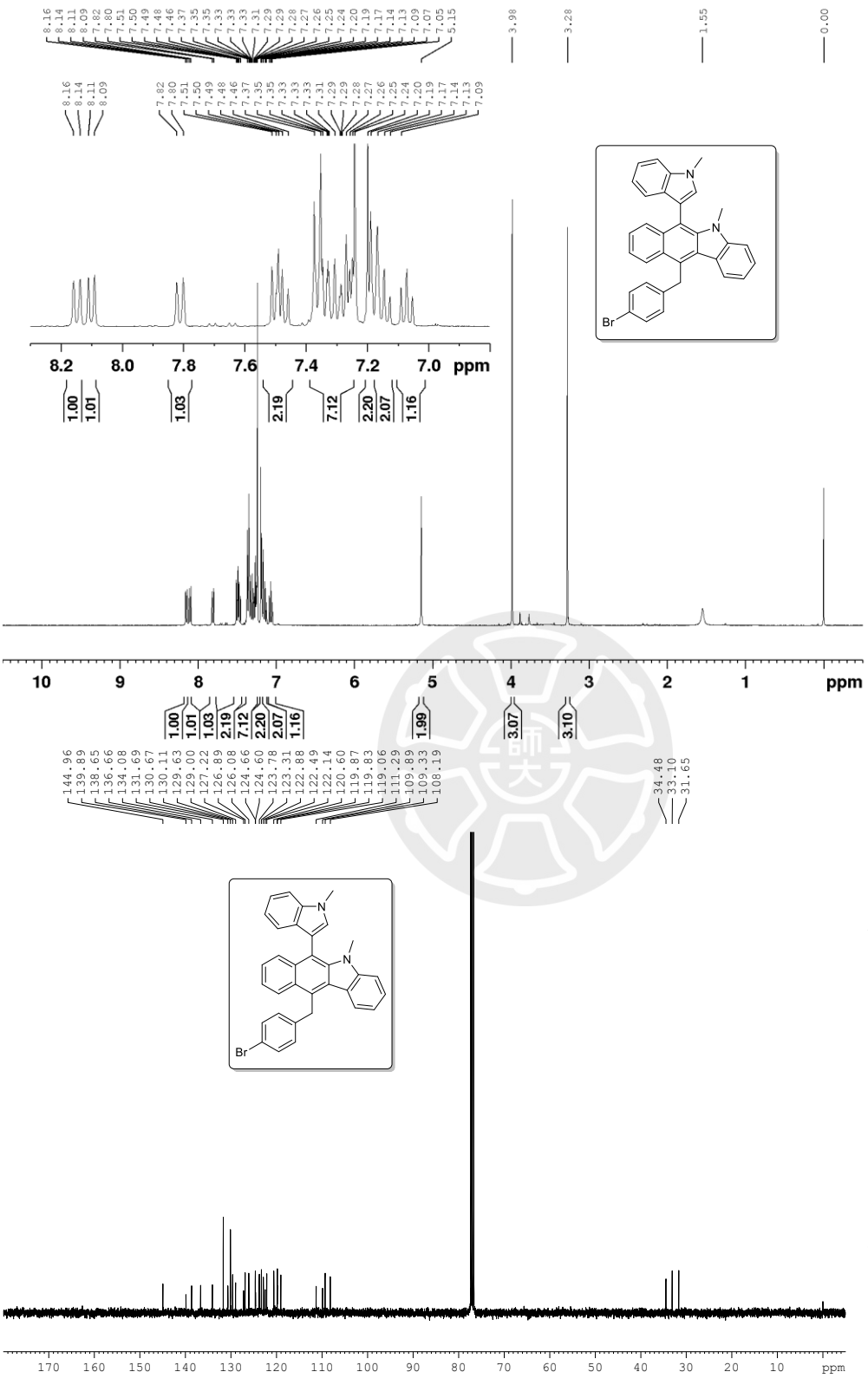
F2 - Acquisition Parameters
Date_           20230922
Time               10.30
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG           zgpg30
TD                 32768
SOLVENT            CDC13
NS                   252
DS                     0
SWH            24038.461 Hz
FIDRES          0.733596 Hz
AQ              0.6815744 sec
RG                198.09
DW                20.800 usec
DE                 6.50 usec
TE                298.1 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1         100.6228298 MHz
NUC1                  13C
P1                 10.00 usec
PLW1         49.50000000 W

======== CHANNEL f2 ========
SFO2         400.1316005 MHz
NUC2                   1H
CPDPRG[2         waltz16
PCPD2              90.00 usec
PLW2         12.50000000 W
PLW12         0.34722000 W
PLW13         0.28125000 W

F2 - Processing parameters
SI                 32768
SF           100.6127685 MHz
WDW                   EM
SSB                    0
LB                  2.00 Hz
GB                     0
PC                  1.00
```

11-(4-bromobenzyl)-5-methyl-6-(1-methyl-1*H*-indol-3-yl)-5*H*-benzo[*b*]carbazole (5ha)



```
Current Data Parameters
NAME      Aldehyde+indole
EXPNO                  25
PROCNO                  1

F2 - Acquisition Parameters
Date_           20230203
Time              18.03
INSTRUM           spect
PROBHD   5 mm BBO BB-1H
PULPROG             zg30
TD                 32768
SOLVENT            CDCl3
NS                    16
DS                     0
SWH             7246.377 Hz
FIDRES          0.221142 Hz
AQ              2.2609921 sec
RG                   181
DW                69.000 usec
DE                 6.50 usec
TE                296.4 K
D1            2.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                15.00 usec
PL1               11.10 dB
SFO1         400.1324008 MHz

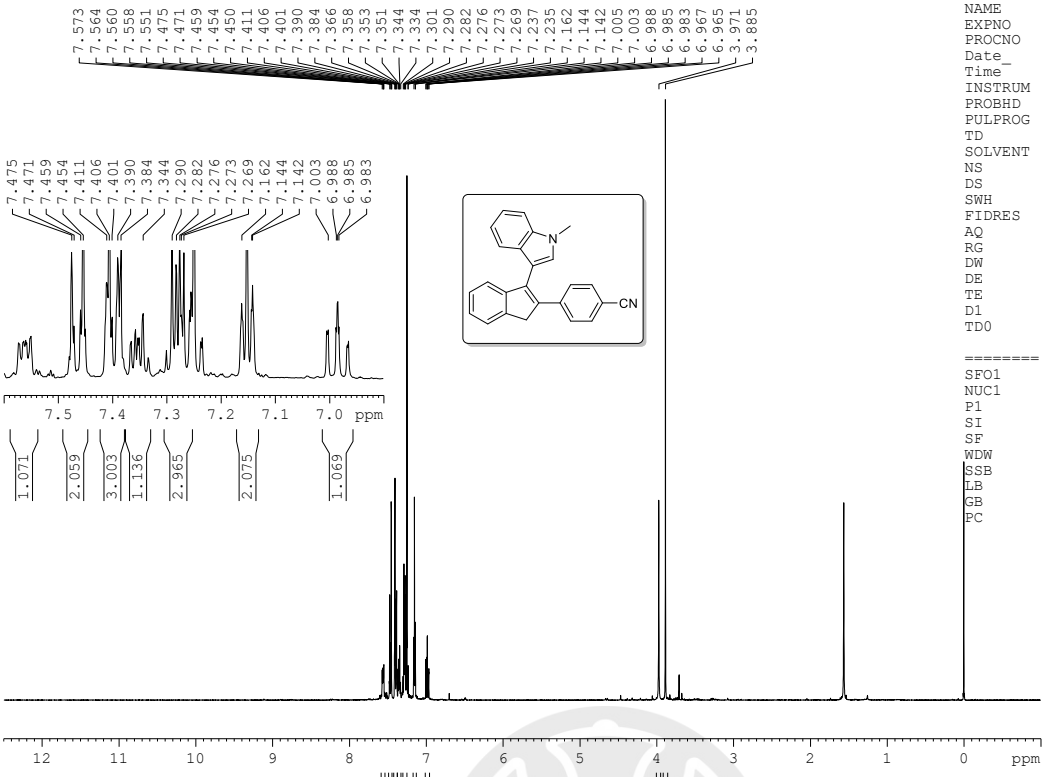
F2 - Processing parameters
SI                 16384
SF          400.1300171 MHz
WDW                   EM
SSB                    0
LB                     0 Hz
GB                     0
PC                  1.00
```

```
NAME      Aldehyde+indole
EXPNO                  26
PROCNO                  1
Date_           20230203
Time              19.29
INSTRUM           spect
PROBHD   5 mm BBO BB-1H
PULPROG           zgpg30
TD                 32768
SOLVENT            CDCl3
NS                  1963
DS                     0
SWH            24038.461 Hz
FIDRES          0.733596 Hz
AQ              0.6816452 sec
RG                9195.2
DW                20.800 usec
DE                 6.50 usec
TE                297.7 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                 13C
P1                52.00 usec
PL1               -6.00 dB
SFO1         100.6233325 MHz

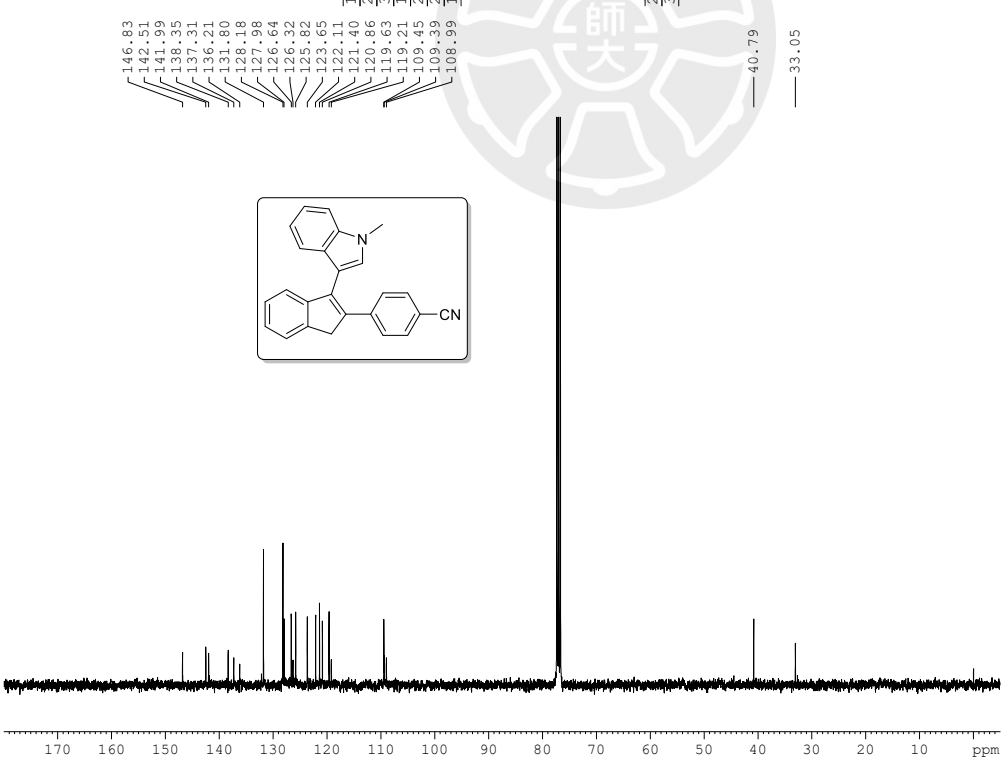
======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                  1H
PCPD2             90.00 usec
PL2               10.20 dB
PL12              26.00 dB
PL13              29.00 dB
SFO2         400.1316005 MHz
SI                 32768
SF          100.6127722 MHz
WDW                   EM
SSB                    0
LB                  1.00 Hz
GB                     0
PC                  1.00
```

4-(3-(1-methyl-1*H*-indol-3-yl)-1*H*-inden-2-yl)benzonitrile (3ia)



```
NAME      Aldehyde+indole
EXPNO                  67
PROCNO                  1
Date_            20230613
Time_                5.02
INSTRUM             spect
PROBHD    5 mm PABBO BB/
PULPROG              zg30
TD                  32768
SOLVENT             CDCl3
NS                     16
DS                      0
SWH             7211.539 Hz
FIDRES          0.220079 Hz
AQ              2.2719646 sec
RG                  128.9
DW                 69.333 usec
DE                 10.06 usec
TE                 298.3 K
D1             2.00000000 sec
TD0                     1

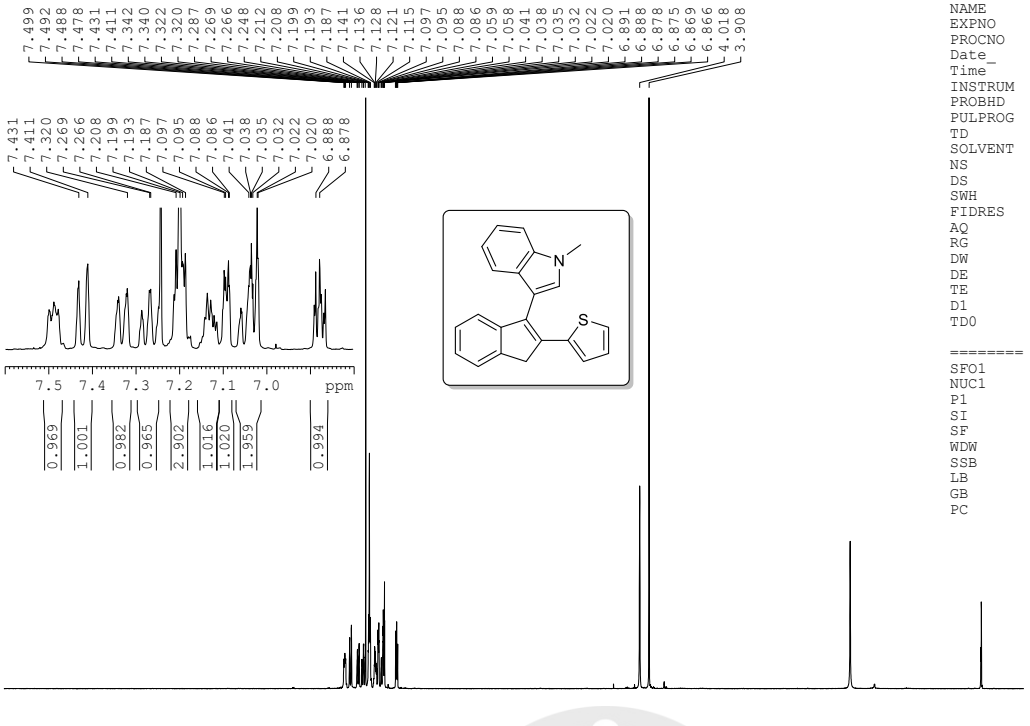
======== CHANNEL f1 ========
SFO1          400.1324008 MHz
NUC1                   1H
P1                  15.00 usec
SI                  16384
SF            400.1300138 MHz
WDW                    EM
SSB                     0
LB                   0.00 Hz
GB                      0
PC                   1.00
```



```
NAME      Aldehyde+indole
EXPNO                  68
PROCNO                  1
Date_            20230613
Time_                5.09
INSTRUM             spect
PROBHD    5 mm PABBO BB/
PULPROG            zgpg30
TD                  32768
SOLVENT             CDCl3
NS                    238
DS                      0
SWH            24038.461 Hz
FIDRES          0.733596 Hz
AQ              0.6816244 sec
RG                  198.09
DW                 20.800 usec
DE                  6.50 usec
TE                 299.4 K
D1             2.00000000 sec
D11            0.03000000 sec
TD0                     1

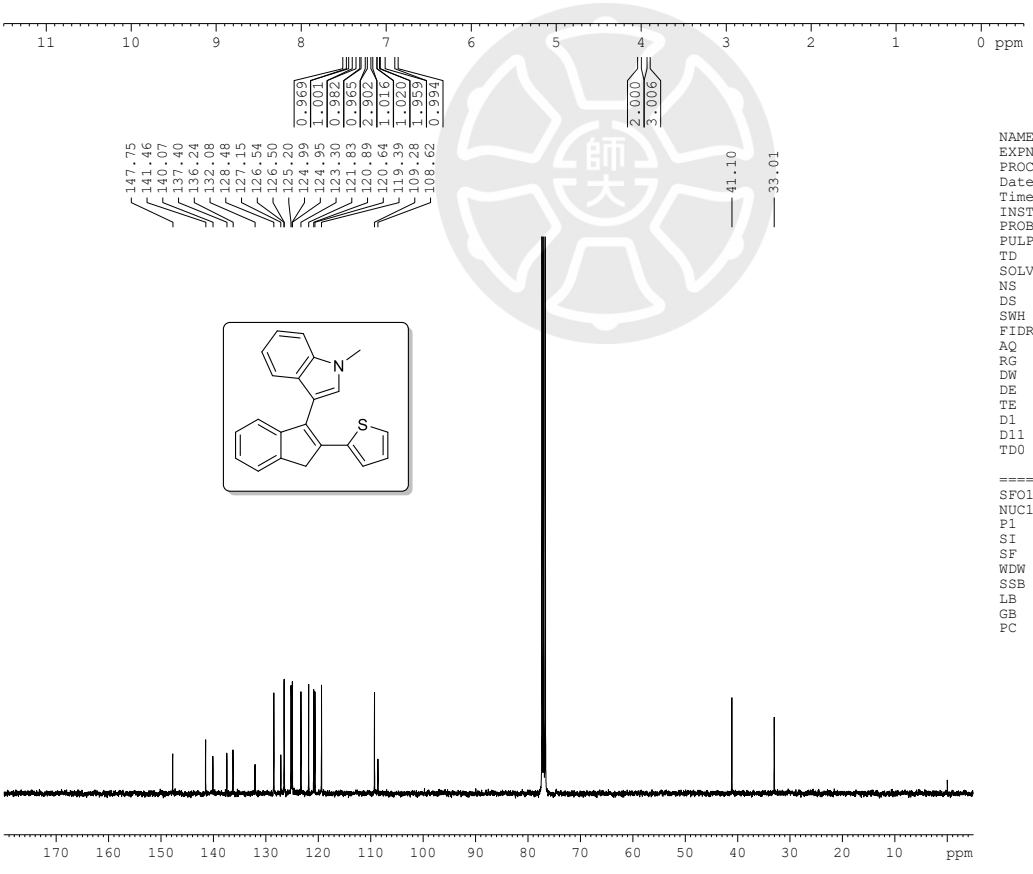
======== CHANNEL f1 ========
SFO1          100.6228298 MHz
NUC1                   13C
P1                  10.00 usec
SI                  32768
SF            100.6127713 MHz
WDW                    EM
SSB                     0
LB                   2.00 Hz
GB                      0
PC                   1.00
```

1-methyl-3-(2-(thiophen-2-yl)-1*H*-inden-3-yl)-1*H*-indole (3ja)



```
NAME      Aldehyde+indole
EXPNO                  65
PROCNO                  1
Date_            20230613
Time                5.24
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG             zg30
TD                 32768
SOLVENT            CDCl3
NS                    16
DS                     0
SWH             7211.539 Hz
FIDRES          0.220079 Hz
AQ             2.2719646 sec
RG                 128.9
DW                69.333 usec
DE                10.06 usec
TE                298.7 K
D1             2.00000000 sec
TD0                    1

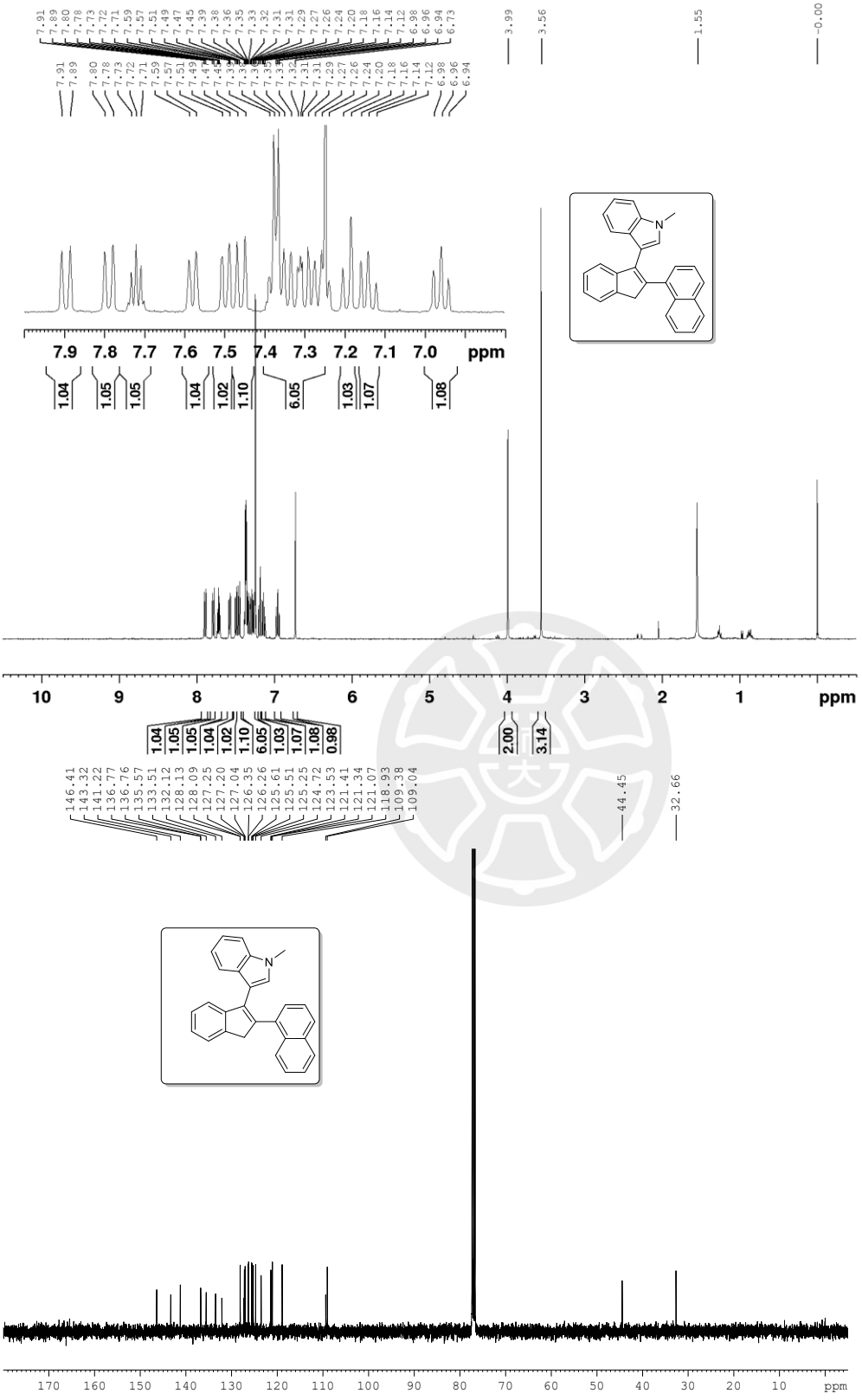
======== CHANNEL f1 ========
SFO1         400.1324008 MHz
NUC1                   1H
P1                 15.00 usec
SI                 16384
SF           400.1300165 MHz
WDW                   EM
SSB                    0
LB                  0.00 Hz
GB                     0
PC                  1.00
```



```
NAME      Aldehyde+indole
EXPNO                  66
PROCNO                  1
Date_            20230613
Time                6.10
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG           zgpg30
TD                 32768
SOLVENT            CDCl3
NS                  1001
DS                     0
SWH            24038.461 Hz
FIDRES          0.733596 Hz
AQ             0.6816244 sec
RG                 198.09
DW                20.800 usec
DE                 6.50 usec
TE                300.4 K
D1             2.00000000 sec
D11            0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1         100.6228298 MHz
NUC1                   13C
P1                10.00 usec
SI                 32768
SF           100.6127710 MHz
WDW                   EM
SSB                    0
LB                  2.00 Hz
GB                     0
PC                  1.00
```

1-methyl-3-(2-(naphthalen-1-yl)-1*H*-inden-3-yl)-1*H*-indole (3ka)



```
Current Data Parameters
NAME      Aldehyde+indole
EXPNO                  35
PROCNO                  1

F2 - Acquisition Parameters
Date_           20230207
Time              12.09
INSTRUM           spect
PROBHD   5 mm BBO BB-1H
PULPROG             zg30
TD                 32768
SOLVENT            CDCl3
NS                     8
DS                     0
SWH             7246.377 Hz
FIDRES          0.221142 Hz
AQ              2.2609921 sec
RG                   256
DW                69.000 usec
DE                 6.50 usec
TE                296.4 K
D1             2.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                 15.00 usec
PL1                11.10 dB
SFO1         400.1324008 MHz

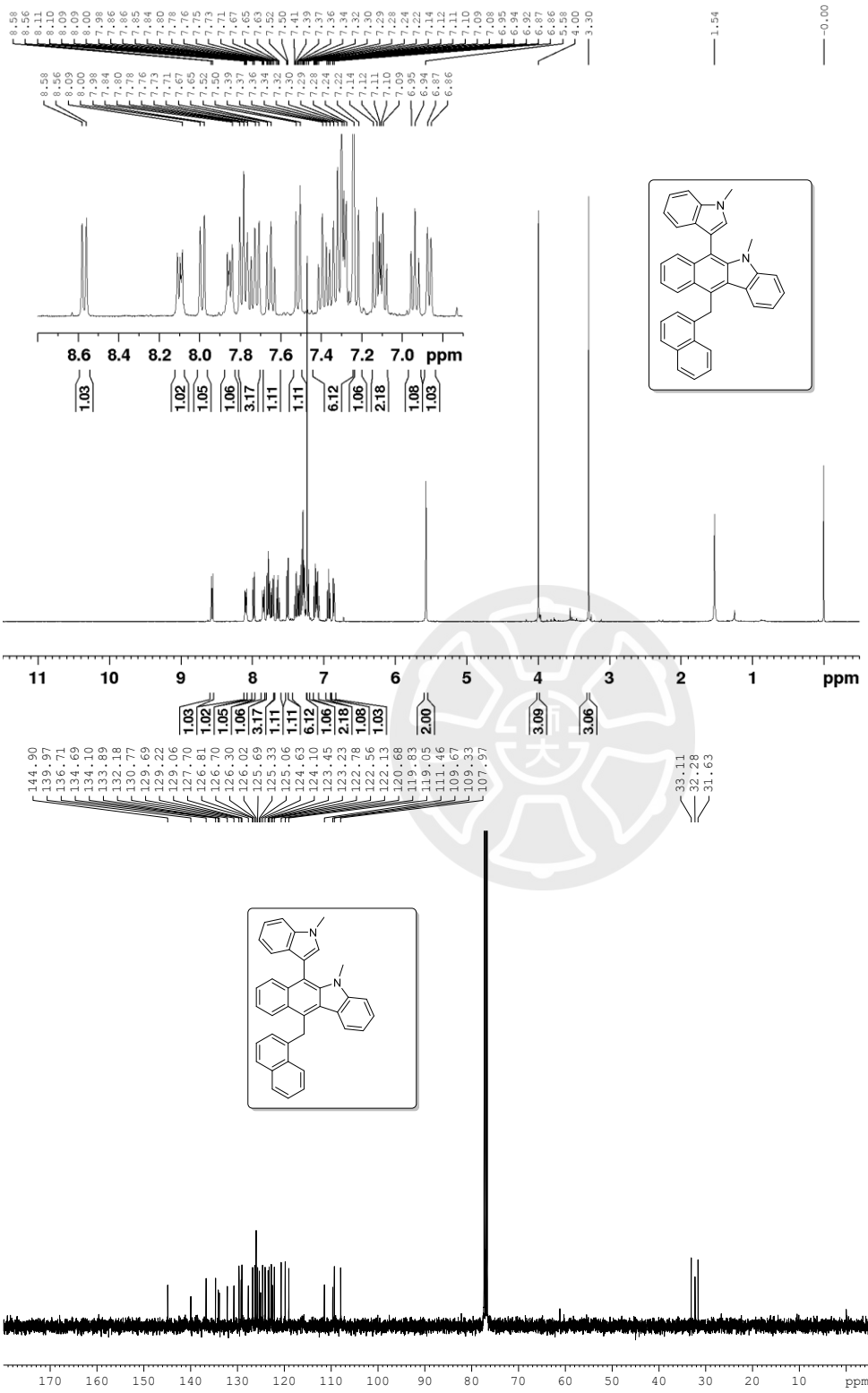
F2 - Processing parameters
SI                 16384
SF           400.1300145 MHz
WDW                   EM
SSB                    0
LB                  0 Hz
GB                     0
PC                  1.00
```

```
NAME      Aldehyde+indole
EXPNO                  36
PROCNO                  1
Date_           20230207
Time              12.10
INSTRUM           spect
PROBHD   5 mm BBO BB-1H
PULPROG          zgpg30
TD                 32768
SOLVENT            CDCl3
NS                  2516
DS                     0
SWH            24038.461 Hz
FIDRES          0.733596 Hz
AQ              0.6816452 sec
RG                  8192
DW                20.800 usec
DE                 6.50 usec
TE                296.5 K
D1             2.00000000 sec
D11            0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  13C
P1                 52.00 usec
PL1                -6.00 dB
SFO1         100.6233325 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                  1H
PCPD2              90.00 usec
PL2                10.20 dB
PL12               26.00 dB
PL13               29.00 dB
SFO2         400.1316005 MHz
SI                 32768
SF           100.6127708 MHz
WDW                   EM
SSB                    0
LB                  1.00 Hz
GB                     0
PC                  1.00
```

5-methyl-6-(1-methyl-1*H*-indol-3-yl)-11-(naphthalen-1-ylmethyl)-5*H*-benzo[*b*]carbazole (5ka)



```
Current Data Parameters
NAME      Aldehyde+indole
EXPNO                  37
PROCNO                  1

F2 - Acquisition Parameters
Date_           20230208
Time              18.03
INSTRUM           spect
PROBHD    5 mm BBO BB-1H
PULPROG            zg30
TD                32768
SOLVENT           CDCl3
NS                   16
DS                    0
SWH            7246.377 Hz
FIDRES        0.221142 Hz
AQ            2.2609921 sec
RG                228.1
DW               69.000 usec
DE                6.50 usec
TE               298.6 K
D1            2.00000000 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                  1H
P1                15.00 usec
PL1               11.10 dB
SFO1         400.1324008 MHz

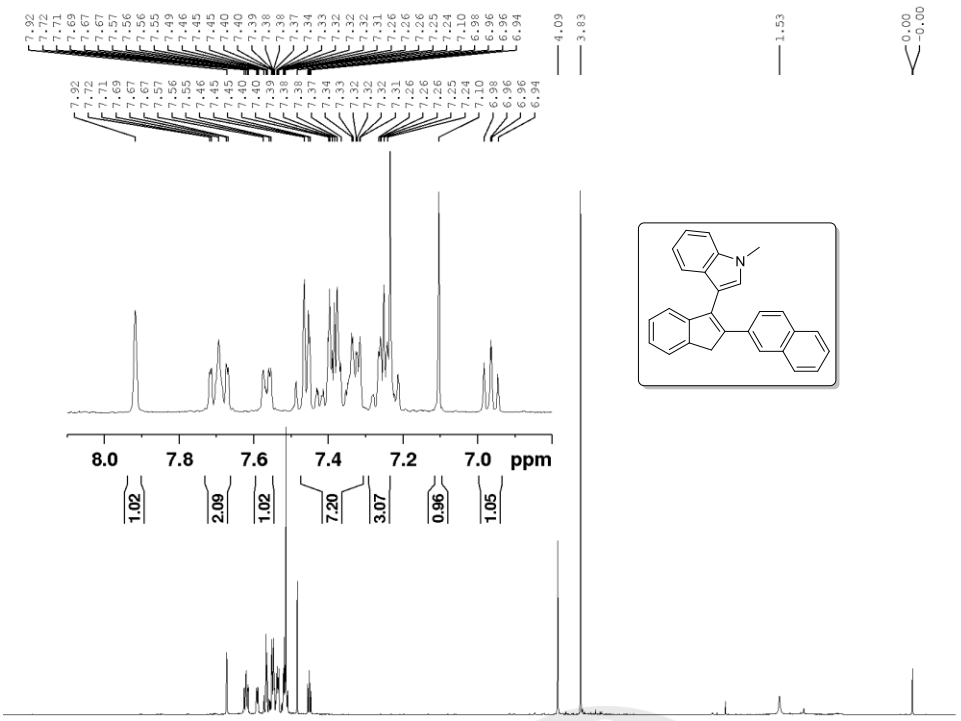
F2 - Processing parameters
SI                16384
SF           400.1300178 MHz
WDW                  EM
SSB                   0
LB                    0 Hz
GB                    0
PC                 1.00
```

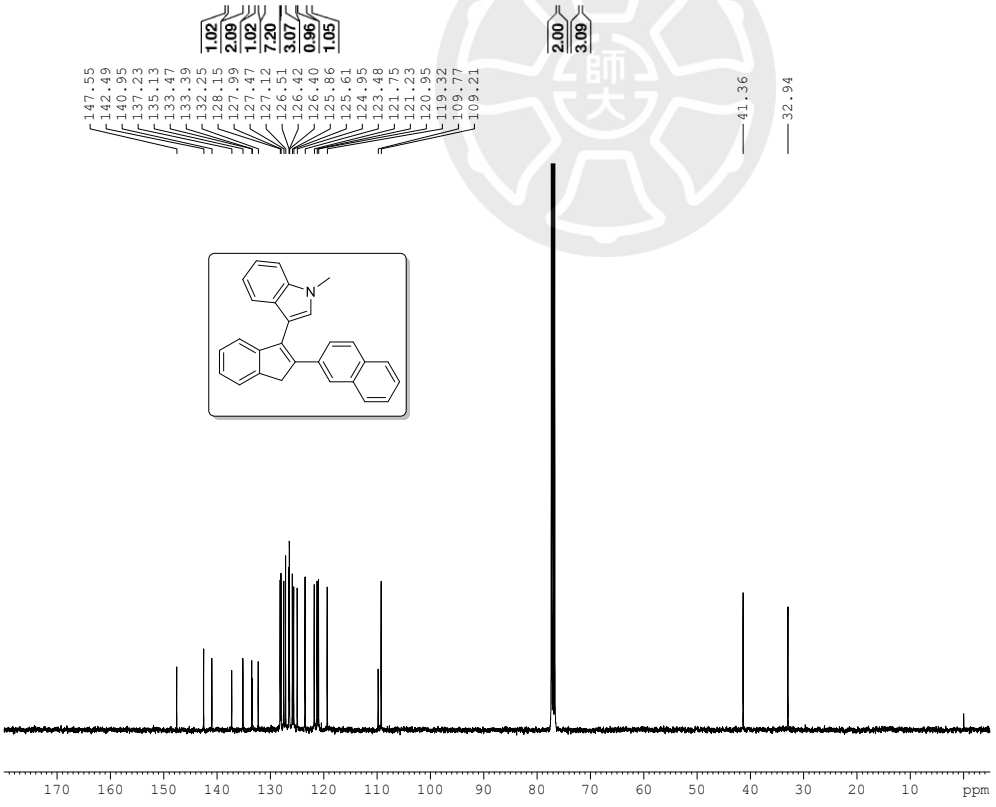
```
NAME      Aldehyde+indole
EXPNO                  38
PROCNO                  1
Date_           20230208
Time              19.47
INSTRUM           spect
PROBHD    5 mm BBO BB-1H
PULPROG         zgpg30
TD                32768
SOLVENT           CDCl3
NS                 2221
DS                    0
SWH           24038.461 Hz
FIDRES        0.733596 Hz
AQ            0.6816452 sec
RG                9195.2
DW               20.800 usec
DE                6.50 usec
TE               300.0 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                 13C
P1                52.00 usec
PL1               -6.00 dB
SFO1        100.6233325 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                  1H
PCPD2             90.00 usec
PL2               10.20 dB
PL12              26.00 dB
PL13              29.00 dB
SFO2        400.1316005 MHz
SI                32768
SF          100.6127716 MHz
WDW                  EM
SSB                   0
LB                 1.00 Hz
GB                    0
PC                 1.00
```

1-methyl-3-(2-(naphthalen-2-yl)-1*H*-inden-3-yl)-1*H*-indole (3la)





```
Current Data Parameters
NAME      Aldehyde+indole
EXPNO                  39
PROCNO                  1

F2 - Acquisition Parameters
Date_           20230215
Time               22.35
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG             zg30
TD                 32768
SOLVENT            CDCl3
NS                    16
DS                     0
SWH             7211.539 Hz
FIDRES          0.220079 Hz
AQ              2.2719147 sec
RG                113.31
DW                69.333 usec
DE                10.06 usec
TE                299.6 K
D1             2.00000000 sec
TD0                    1

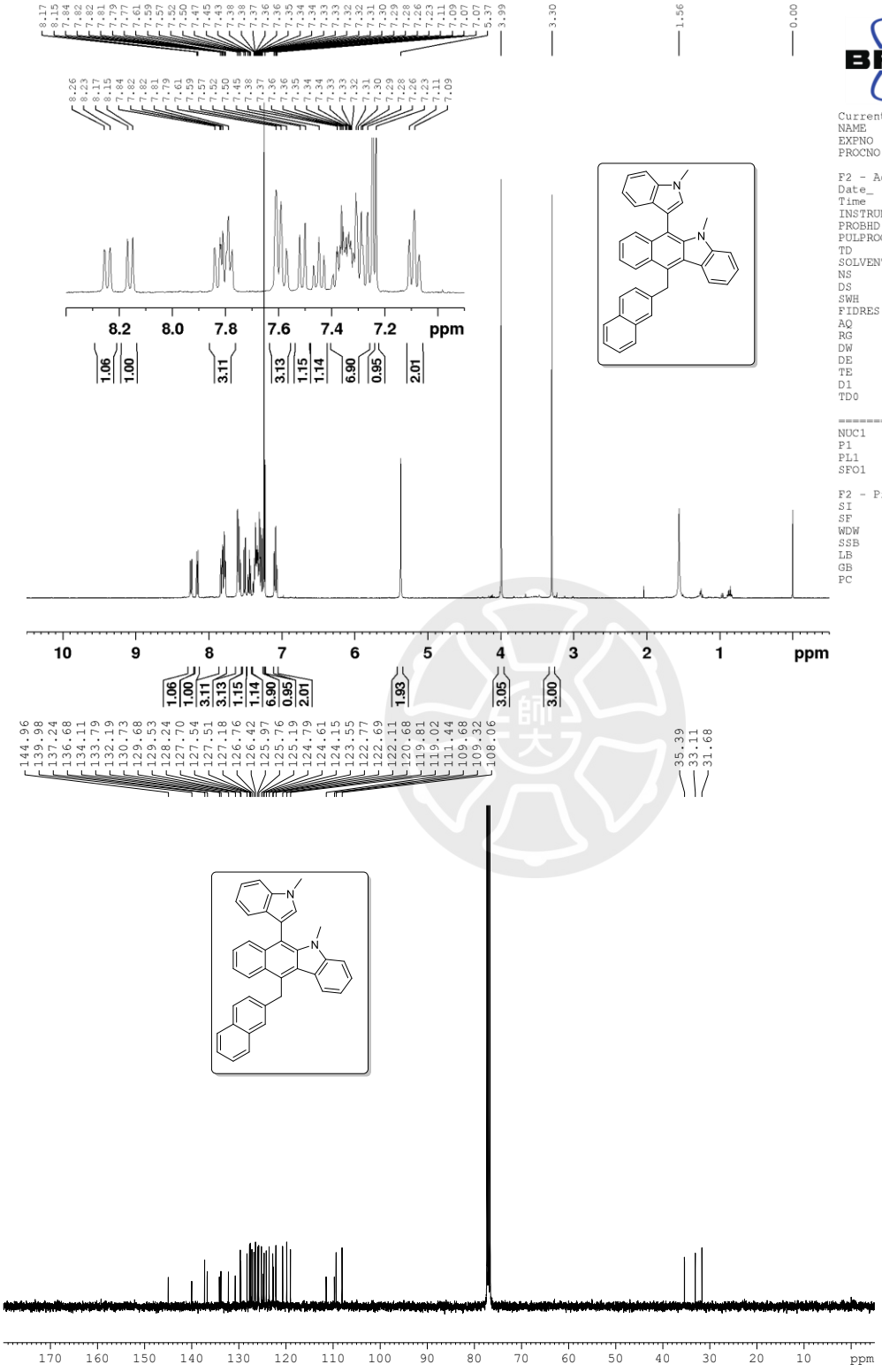
======== CHANNEL f1 ========
SFO1         400.1324008 MHz
NUC1                   1H
P1                 15.00 usec
PLW1         13.50000000 W

F2 - Processing parameters
SI                 16384
SF            400.1300201 MHz
WDW                   EM
SSB                    0
LB                     0 Hz
GB                     0
PC                  1.00
```

```
NAME      Aldehyde+indole
EXPNO                  40
PROCNO                  1
Date_           20230216
Time                0.01
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG          zgpg30
TD                 32768
SOLVENT            CDCl3
NS                  1858
DS                     0
SWH            24038.461 Hz
FIDRES          0.733596 Hz
AQ              0.6816244 sec
RG                198.09
DW                20.800 usec
DE                 6.50 usec
TE                300.9 K
D1             2.00000000 sec
D11            0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1         100.6228298 MHz
NUC1                   13C
P1                 10.00 usec
SI                 32768
SF           100.6127722 MHz
WDW                   EM
SSB                    0
LB                  2.00 Hz
GB                     0
PC                  1.00
```

5-methyl-6-(1-methyl-1*H*-indol-3-yl)-11-(naphthalen-2-ylmethyl)-5*H*-benzo[*b*]carbazole (5la)



```
Current Data Parameters
NAME      Aldehyde+indole
EXPNO                  41
PROCNO                  1

F2 - Acquisition Parameters
Date_            20230624
Time                 8.12
INSTRUM             spect
PROBHD   5 mm BBO BB-1H
PULPROG              zg30
TD                  32768
SOLVENT             CDCl3
NS                     12
DS                      0
SWH             7246.377 Hz
FIDRES          0.221142 Hz
AQ              2.2609921 sec
RG                    256
DW                 69.000 usec
DE                  6.50 usec
TE                 296.7 K
D1              2.00000000 sec
TD0                     1

======== CHANNEL f1 ========
NUC1                   1H
P1                 15.00 usec
PL1                11.10 dB
SFO1          400.1324008 MHz

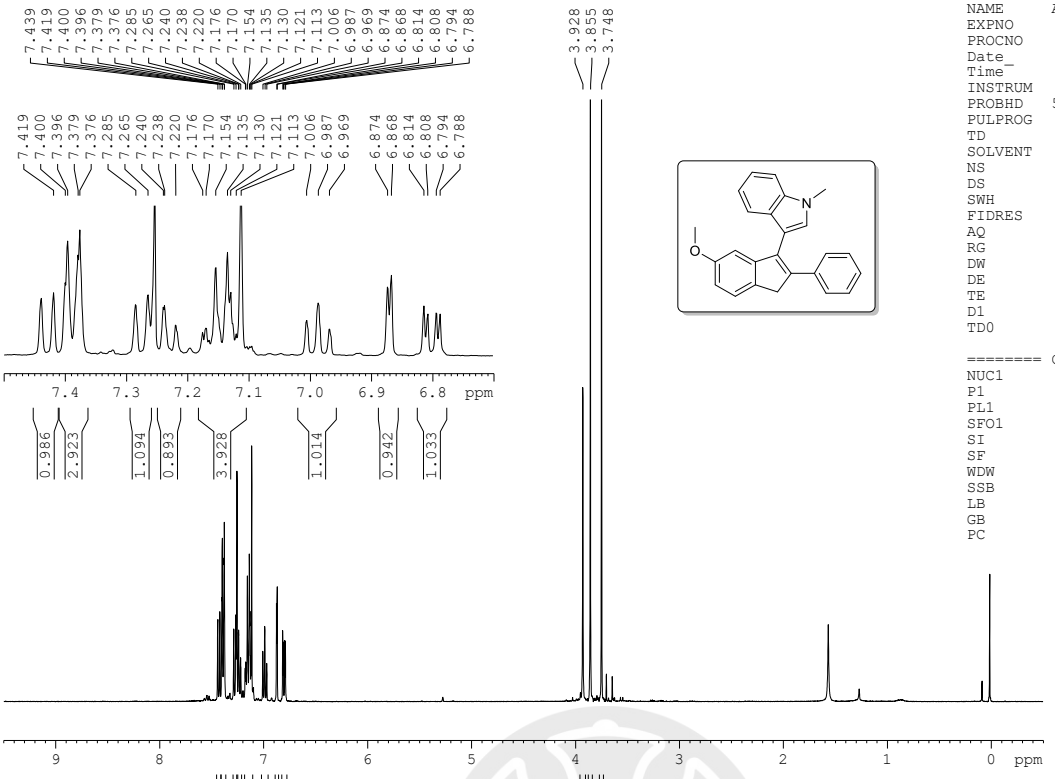
F2 - Processing parameters
SI                  16384
SF            400.1300154 MHz
WDW                    EM
SSB                     0
LB                      0 Hz
GB                      0
PC                   1.00
```

```
NAME      Aldehyde+indole
EXPNO                  42
PROCNO                  1
Date_            20230624
Time                 8.14
INSTRUM             spect
PROBHD   5 mm BBO BB-1H
PULPROG          zgpg30
TD                  32768
SOLVENT             CDCl3
NS                   1275
DS                      0
SWH            24038.461 Hz
FIDRES          0.733596 Hz
AQ              0.6816452 sec
RG                   8192
DW                20.800 usec
DE                  6.50 usec
TE                 296.7 K
D1              2.00000000 sec
D11             0.03000000 sec
TD0                     1

======== CHANNEL f1 ========
NUC1                  13C
P1                 10.00 usec
PL1                 4.20 dB
SFO1          100.6233325 MHz

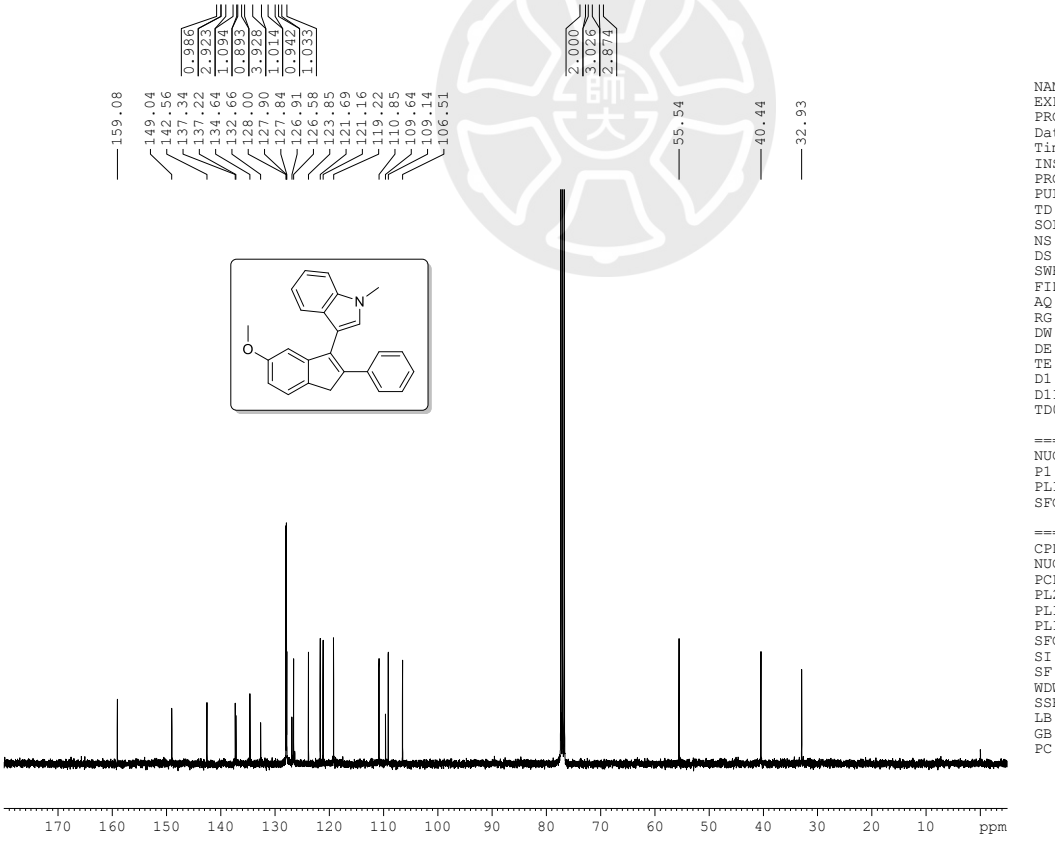
======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                   1H
PCPD2              90.00 usec
PL2                10.20 dB
PL12               26.00 dB
PL13               29.00 dB
SFO2          400.1316005 MHz
SI                  32768
SF            100.6127716 MHz
WDW                    EM
SSB                     0
LB                   1.00 Hz
GB                      0
PC                   1.00
```

3-(5-methoxy-2-phenyl-1*H*-inden-3-yl)-1-methyl-1*H*-indole (3ma)




```
NAME      Aldehyde+indole
EXPNO                  79
PROCNO                  1
Date_            20230624
Time                 7.20
INSTRUM             spect
PROBHD   5 mm BBO BB-1H
PULPROG              zg30
TD                  32768
SOLVENT             CDCl3
NS                     12
DS                      0
SWH             7246.377 Hz
FIDRES          0.221142 Hz
AQ             2.2611110 sec
RG                    181
DW                 69.000 usec
DE                  6.50 usec
TE                 296.5 K
D1            2.00000000 sec
TD0                     1

======== CHANNEL f1 ========
NUC1                   1H
P1                  15.00 usec
PL1                 11.10 dB
SFO1         400.1324008 MHz
SI                  16384
SF           400.1300118 MHz
WDW                    EM
SSB                     0
LB                   0.00 Hz
GB                      0
PC                   1.00
```

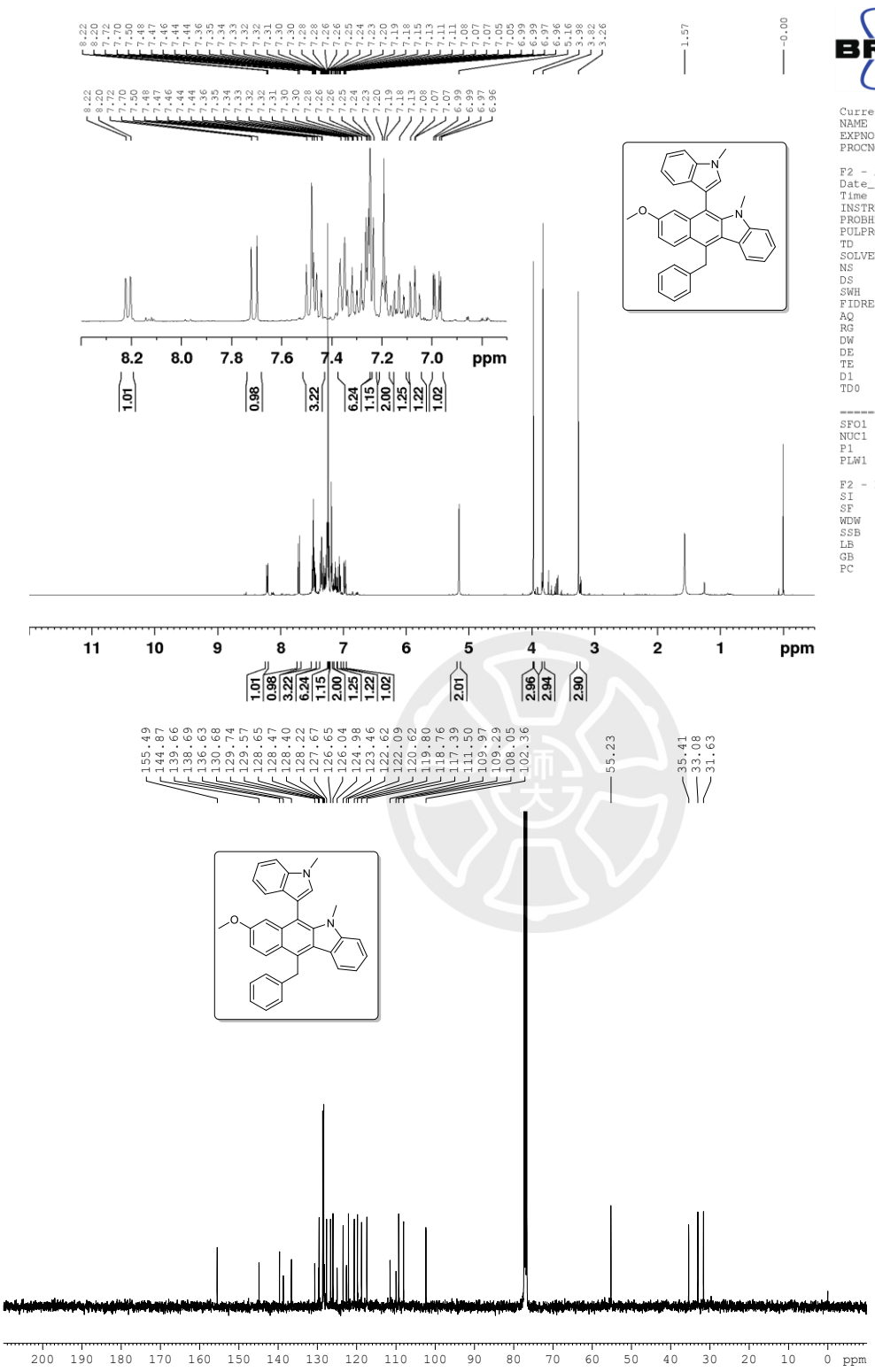



```
NAME      Aldehyde+indole
EXPNO                  80
PROCNO                  1
Date_            20230624
Time                 7.21
INSTRUM             spect
PROBHD   5 mm BBO BB-1H
PULPROG           zgpg30
TD                  32768
SOLVENT             CDCl3
NS                    909
DS                      0
SWH            24038.461 Hz
FIDRES          0.733596 Hz
AQ             0.6816452 sec
RG                 1625.5
DW                 20.800 usec
DE                  6.50 usec
TE                 296.5 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0                     1

======== CHANNEL f1 ========
NUC1                  13C
P1                  10.00 usec
PL1                  4.20 dB
SFO1        100.6233325 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                   1H
PCPD2               90.00 usec
PL2                 10.20 dB
PL12                26.00 dB
PL13                29.00 dB
SFO2         400.1316005 MHz
SI                  32768
SF           100.6127723 MHz
WDW                    EM
SSB                     0
LB                   1.00 Hz
GB                      0
PC                   1.00
```

11-benzyl-9-methoxy-5-methyl-6-(1-methyl-1*H*-indol-3-yl)-5*H*-benzo[*b*]carbazole (5ma)



```
Current Data Parameters
NAME          20230722(3)
EXPNO                   3
PROCNO                  1

F2 - Acquisition Parameters
Date_           20230723
Time               18.55
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG            zg30
TD                32768
SOLVENT           CDC13
NS                   16
DS                    0
SWH           7211.539 Hz
FIDRES         0.220079 Hz
AQ            2.2719147 sec
RG               113.31
DW               69.333 usec
DE                10.06 usec
TE                298.1 K
D1            2.00000000 sec
TD0                   1

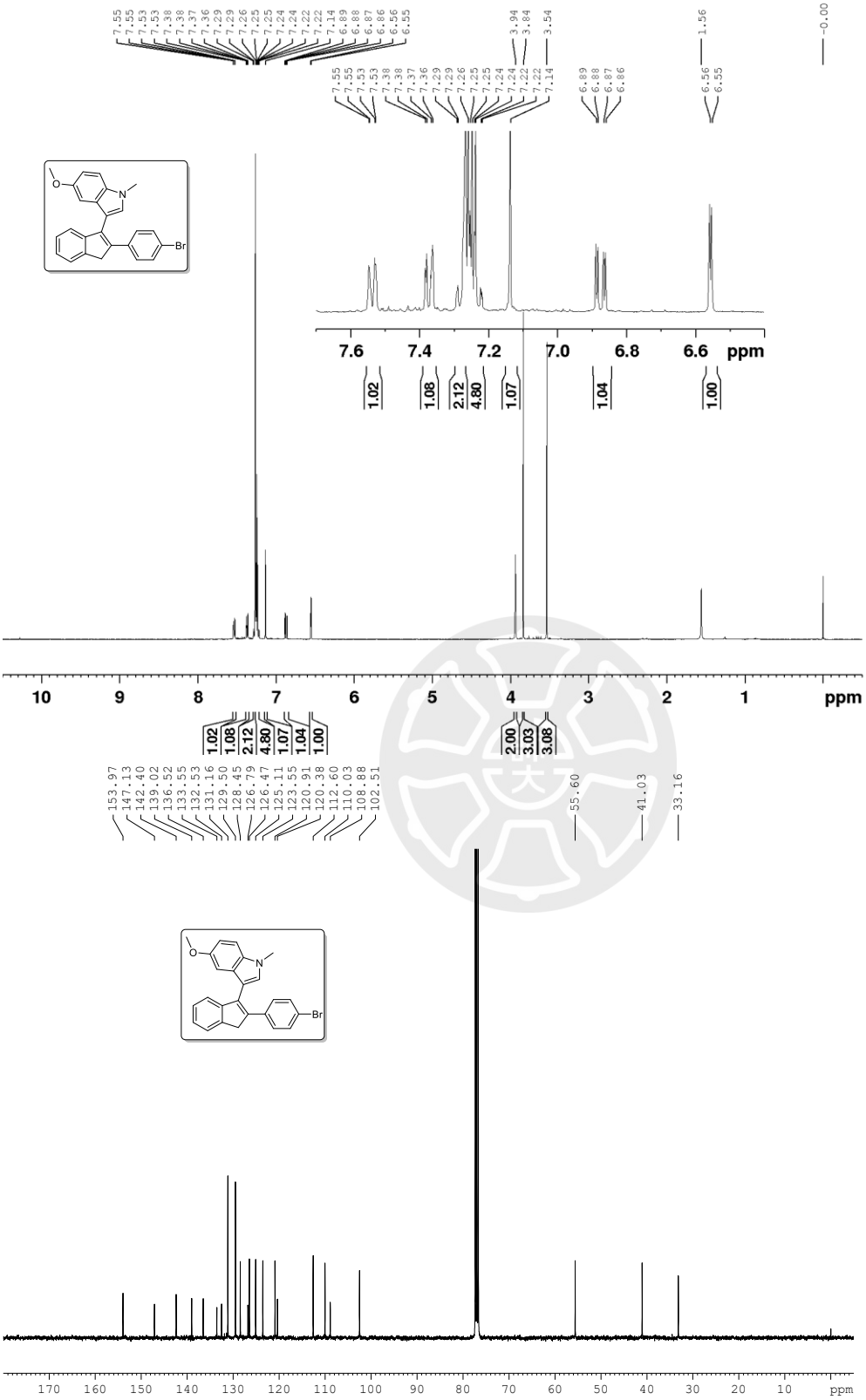
======== CHANNEL f1 ========
SFO1        400.1324008 MHz
NUC1                   1H
P1                 15.00 usec
PLW1        13.50000000 W

F2 - Processing parameters
SI                 16384
SF          400.1300159 MHz
WDW                   EM
SSB                    0
LB                     0 Hz
GB                     0
PC                  1.00
```

```
NAME          20230722(3)
EXPNO                   2
PROCNO                  1
Date_           20230723
Time               17.15
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG          zgpg30
TD                32768
SOLVENT           CDC13
NS                 1000
DS                    0
SWH          24038.461 Hz
FIDRES         0.733596 Hz
AQ            0.6816244 sec
RG               198.09
DW                20.800 usec
DE                 6.50 usec
TE                299.2 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
SFO1        100.6228298 MHz
NUC1                  13C
P1                10.00 usec
SI                32768
SF          100.6127714 MHz
WDW                   EM
SSB                    0
LB                  2.00 Hz
GB                     0
PC                  1.00
```

3-(2-(4-bromophenyl)-1*H*-inden-3-yl)-5-methoxy-1-methyl-1*H*-indole (3hb)



```
Current Data Parameters
NAME      Aldehyde+indole
EXPNO                  75
PROCNO                  1

F2 - Acquisition Parameters
Date_           20230618
Time               4.08
INSTRUM           spect
PROBHD   5 mm PABBO BB/
PULPROG            zg30
TD                32768
SOLVENT           CDCl3
NS                   16
DS                    0
SWH           7211.539 Hz
FIDRES        0.220079 Hz
AQ            2.2719147 sec
RG               113.31
DW               69.333 usec
DE                10.06 usec
TE                298.5 K
D1           2.00000000 sec
TD0                   1

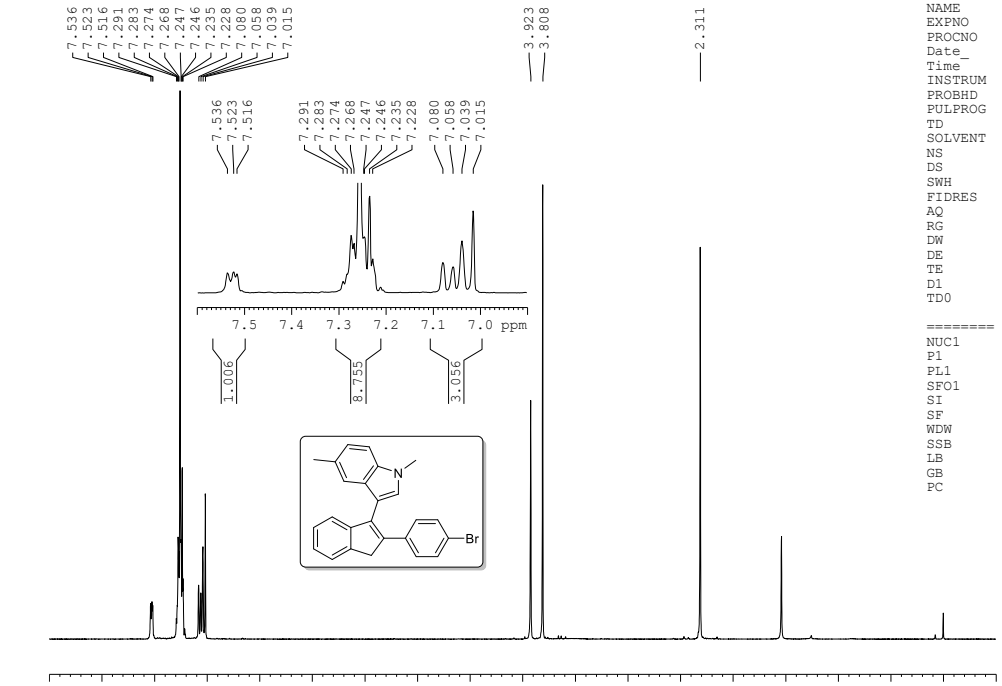
======== CHANNEL f1 ========
SFO1        400.1324008 MHz
NUC1               1H
P1                15.00 usec
PLW1       13.50000000 W

F2 - Processing parameters
SI                16384
SF          400.1300151 MHz
WDW                EM
SSB                 0
LB                  0 Hz
GB                  0
PC               1.00
```

```
NAME      Aldehyde+indole
EXPNO                  76
PROCNO                  1
Date_           20230618
Time               5.10
INSTRUM           spect
PROBHD   5 mm PABBO BB/
PULPROG          zgpg30
TD                32768
SOLVENT           CDCl3
NS                 1342
DS                    0
SWH           24038.461 Hz
FIDRES        0.733596 Hz
AQ            0.6816244 sec
RG               198.09
DW               20.800 usec
DE                6.50 usec
TE                300.1 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                   1

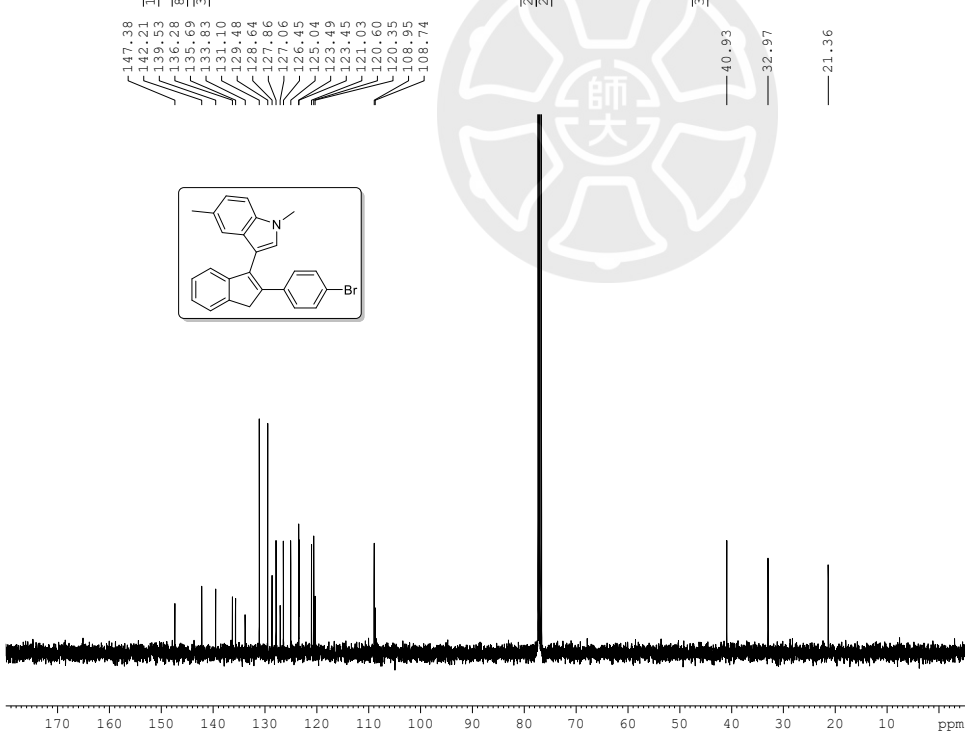
======== CHANNEL f1 ========
SFO1        100.6228298 MHz
NUC1               13C
P1                10.00 usec
SI                32768
SF          100.6127707 MHz
WDW                EM
SSB                 0
LB                2.00 Hz
GB                  0
PC               1.00
```

3-(2-(4-bromophenyl)-1*H*-inden-3-yl)-1,5-dimethyl-1*H*-indole (3hc)




```
NAME      Aldehyde+indole
EXPNO                  77
PROCNO                  1
Date_            20230619
Time                14.39
INSTRUM             spect
PROBHD   5 mm BBO BB-1H
PULPROG               zg30
TD                   32768
SOLVENT              CDCl3
NS                      16
DS                       0
SWH               7246.377 Hz
FIDRES           0.221142 Hz
AQ              2.2611110 sec
RG                     114
DW                69.000 usec
DE                 6.50 usec
TE                 297.1 K
D1             2.00000000 sec
TD0                     1

======== CHANNEL f1 ========
NUC1                   1H
P1                 15.00 usec
PL1                11.10 dB
SFO1         400.1324008 MHz
SI                   16384
SF           400.1300193 MHz
WDW                    EM
SSB                     0
LB                 0.00 Hz
GB                      0
PC                 1.00
```

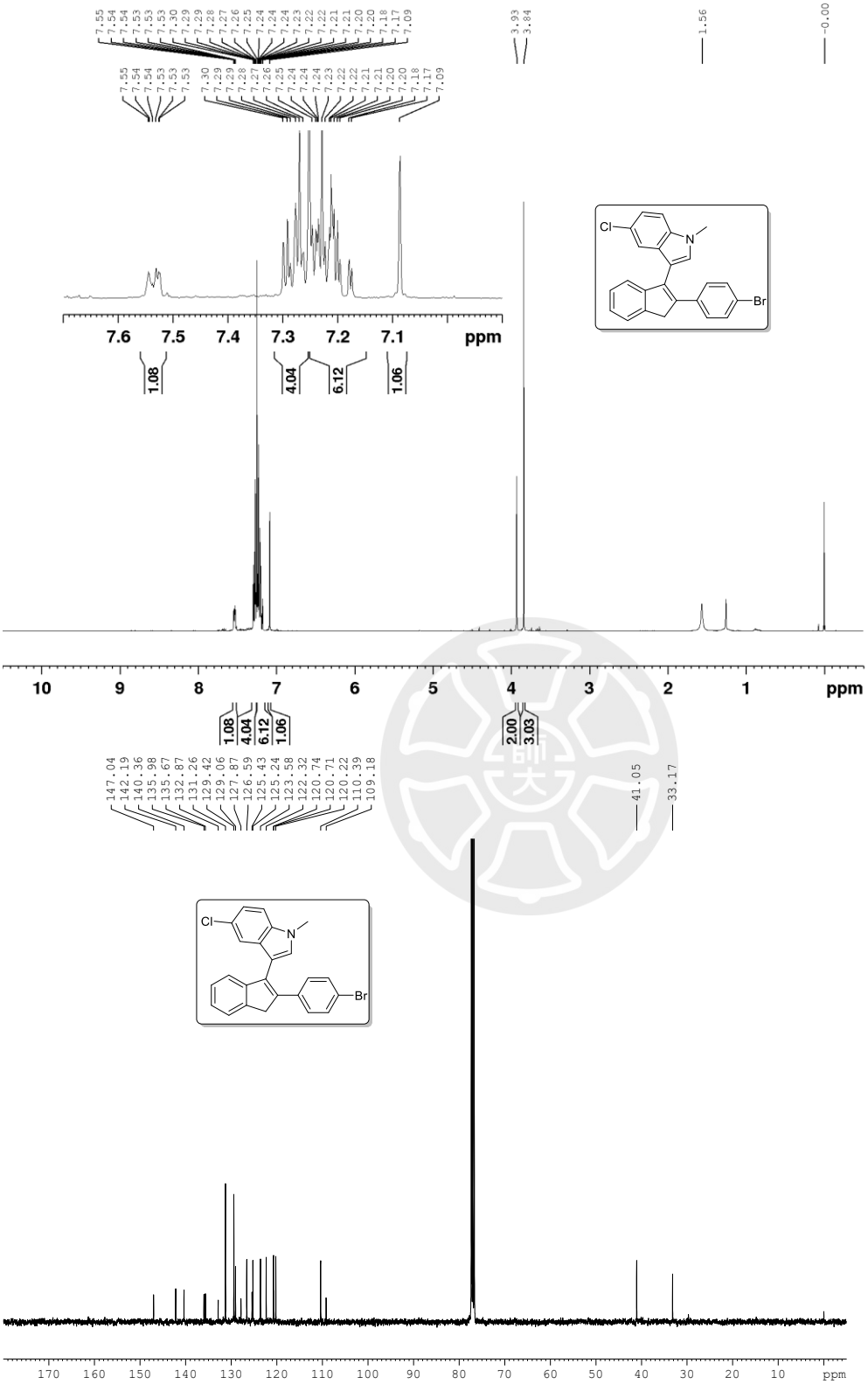



```
NAME      Aldehyde+indole
EXPNO                  78
PROCNO                  1
Date_            20230619
Time                14.41
INSTRUM             spect
PROBHD   5 mm BBO BB-1H
PULPROG            zgpg30
TD                   32768
SOLVENT              CDCl3
NS                     100
DS                       0
SWH              24038.461 Hz
FIDRES           0.733596 Hz
AQ               0.6816452 sec
RG                    3251
DW                20.800 usec
DE                 6.50 usec
TE                 297.3 K
D1             2.00000000 sec
D11            0.03000000 sec
TD0                     1

======== CHANNEL f1 ========
NUC1                  13C
P1                10.00 usec
PL1                4.20 dB
SFO1         100.6233325 MHz

======== CHANNEL f2 ========
CPDPRG2           waltz16
NUC2                   1H
PCPD2              90.00 usec
PL2                10.20 dB
PL12               26.00 dB
PL13               29.00 dB
SFO2         400.1316005 MHz
SI                   32768
SF           100.6127738 MHz
WDW                    EM
SSB                     0
LB                 1.00 Hz
GB                      0
PC                 1.00
```

3-(2-(4-bromophenyl)-1*H*-inden-3-yl)-5-chloro-1-methyl-1*H*-indole (3hd)



```
Current Data Parameters
NAME      Aldehyde+indole
EXPNO                  73
PROCNO                  1

F2 - Acquisition Parameters
Date_            20230615
Time                9.45
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG             zg30
TD                 32768
SOLVENT            CDCl3
NS                    16
DS                     0
SWH             7211.539 Hz
FIDRES          0.220079 Hz
AQ              2.2719147 sec
RG                 128.9
DW                69.333 usec
DE                10.06 usec
TE                298.2 K
D1             2.00000000 sec
TD0                    1

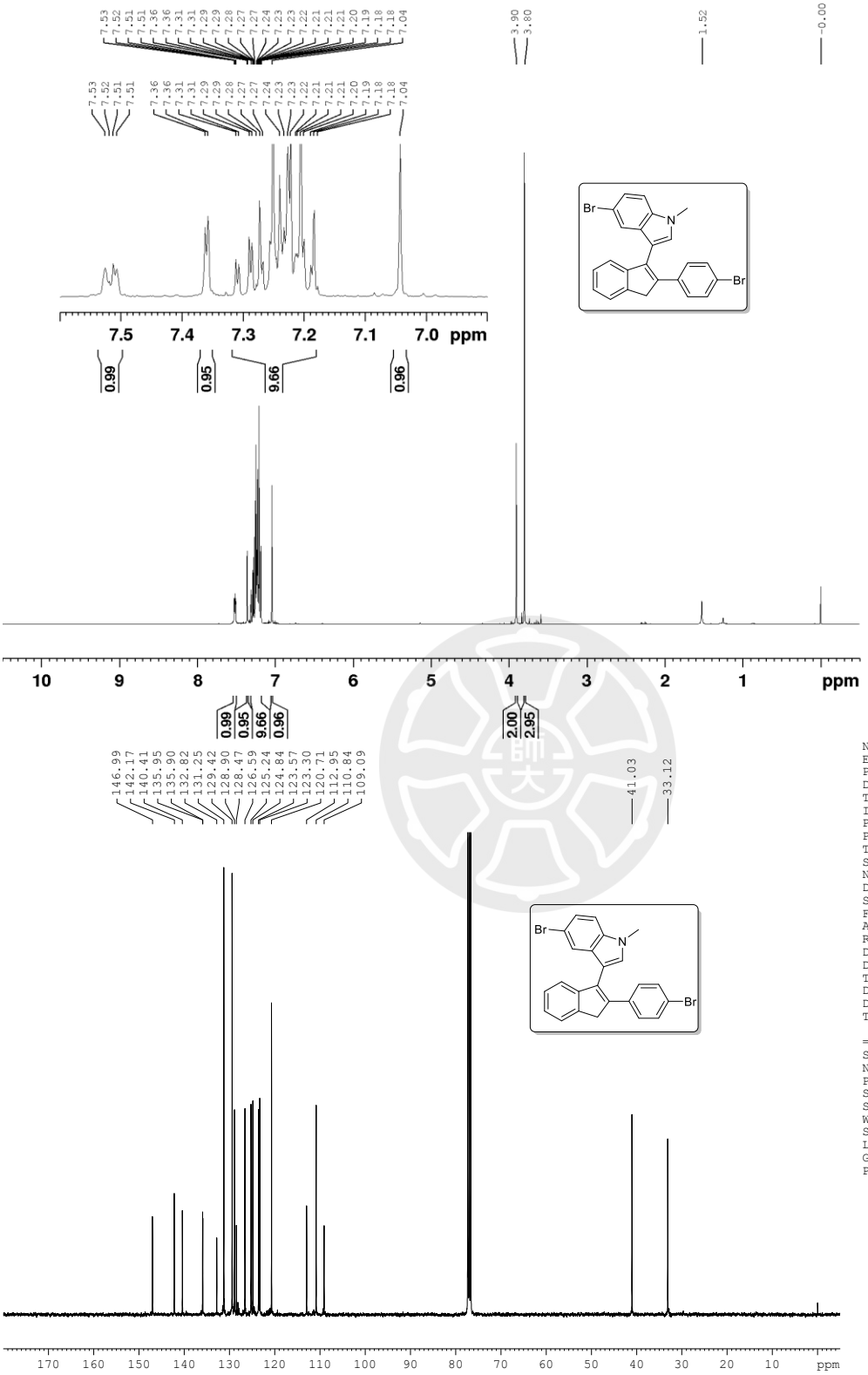
======== CHANNEL f1 ========
SFO1          400.1324008 MHz
NUC1                  1H
P1                 15.00 usec
PLW1          13.50000000 W

F2 - Processing parameters
SI                 16384
SF            400.1300130 MHz
WDW                   EM
SSB                    0
LB                     0 Hz
GB                     0
PC                  1.00
```

```
NAME      Aldehyde+indole
EXPNO                  74
PROCNO                  1
Date_            20230615
Time                9.51
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG          zgpg30
TD                 32768
SOLVENT            CDCl3
NS                   638
DS                     0
SWH            24038.461 Hz
FIDRES          0.733596 Hz
AQ              0.6816244 sec
RG                 198.09
DW                20.800 usec
DE                 6.50 usec
TE                299.3 K
D1             2.00000000 sec
D11            0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1          100.6228298 MHz
NUC1                  13C
P1                 10.00 usec
SI                 32768
SF            100.6127698 MHz
WDW                   EM
SSB                    0
LB                  2.00 Hz
GB                     0
PC                  1.00
```

5-bromo-3-(2-(4-bromophenyl)-1*H*-inden-3-yl)-1-methyl-1*H*-indole (3he)




```
Current Data Parameters
NAME      Aldehyde+indole
EXPNO                  71
PROCNO                  1

F2 - Acquisition Parameters
Date_           20230615
Time               10.22
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG             zg30
TD                 32768
SOLVENT            CDCl3
NS                    16
DS                     0
SWH             7211.539 Hz
FIDRES          0.220079 Hz
AQ              2.2719147 sec
RG                 71.42
DW                69.333 usec
DE                10.06 usec
TE                298.7 K
D1             2.00000000 sec
TD0                    1

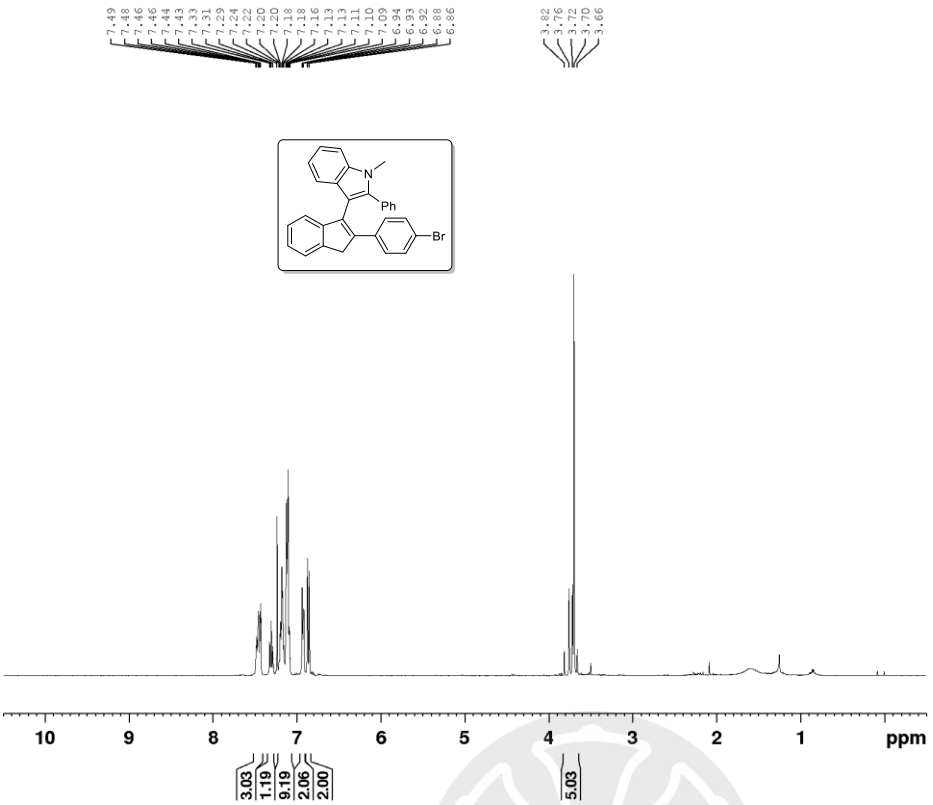
======== CHANNEL f1 ========
SFO1         400.1324008 MHz
NUC1                   1H
P1                 15.00 usec
PLW1         13.50000000 W

F2 - Processing parameters
SI                 16384
SF          400.1300247 MHz
WDW                   EM
SSB                    0
LB                     0 Hz
GB                     0
PC                  1.00
```

```
NAME      Aldehyde+indole
EXPNO                  72
PROCNO                  1
Date_           20230615
Time               11.54
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG           zgpg30
TD                 32768
SOLVENT            CDCl3
NS                  2000
DS                     0
SWH            24038.461 Hz
FIDRES          0.733596 Hz
AQ              0.6816244 sec
RG                198.09
DW                20.800 usec
DE                 6.50 usec
TE                300.5 K
D1             2.00000000 sec
D11            0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1         100.6228298 MHz
NUC1                   13C
P1                 10.00 usec
SI                 32768
SF          100.6127740 MHz
WDW                   EM
SSB                    0
LB                  2.00 Hz
GB                     0
PC                  1.00
```

3-(2-(4-bromophenyl)-1*H*-inden-3-yl)-1-methyl-2-phenyl-1*H*-indole (3hf)

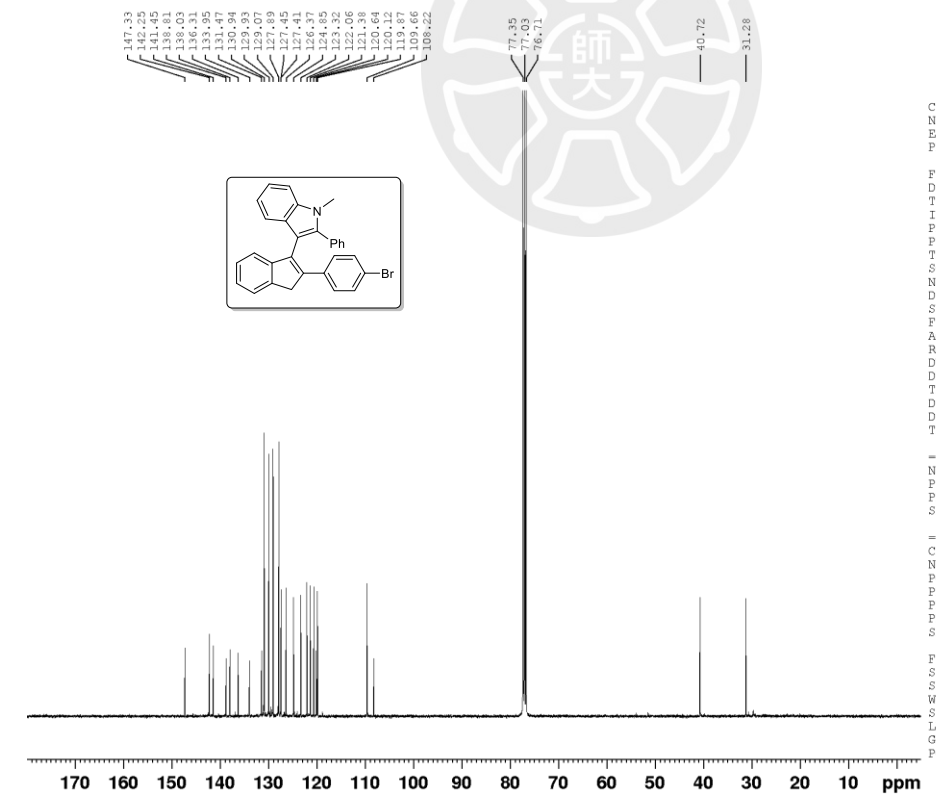



```
Current Data Parameters
NAME                 exp 659
EXPNO                      3
PROCNO                     1

F2 - Acquisition Parameters
Date_               20230816
Time                   21.58
INSTRUM                spect
PROBHD    5 mm BBO BB-1H
PULPROG                zg30
TD                    32768
SOLVENT               CDCl3
NS                       16
DS                        0
SWH                7246.377 Hz
FIDRES            0.221142 Hz
AQ                2.2609921 sec
RG                     161.3
DW                  69.000 usec
DE                    6.50 usec
TE                   297.5 K
D1                2.00000000 sec
TD0                        1

======== CHANNEL f1 ========
NUC1                      1H
P1                     15.00 usec
PL1                    11.10 dB
SFO1           400.1324008 MHz

F2 - Processing parameters
SI                        16384
SF                400.1300175 MHz
WDW                          EM
SSB                           0
LB                         0 Hz
GB                            0
PC                         1.00
```




```
Current Data Parameters
NAME                 exp 659
EXPNO                      4
PROCNO                     1

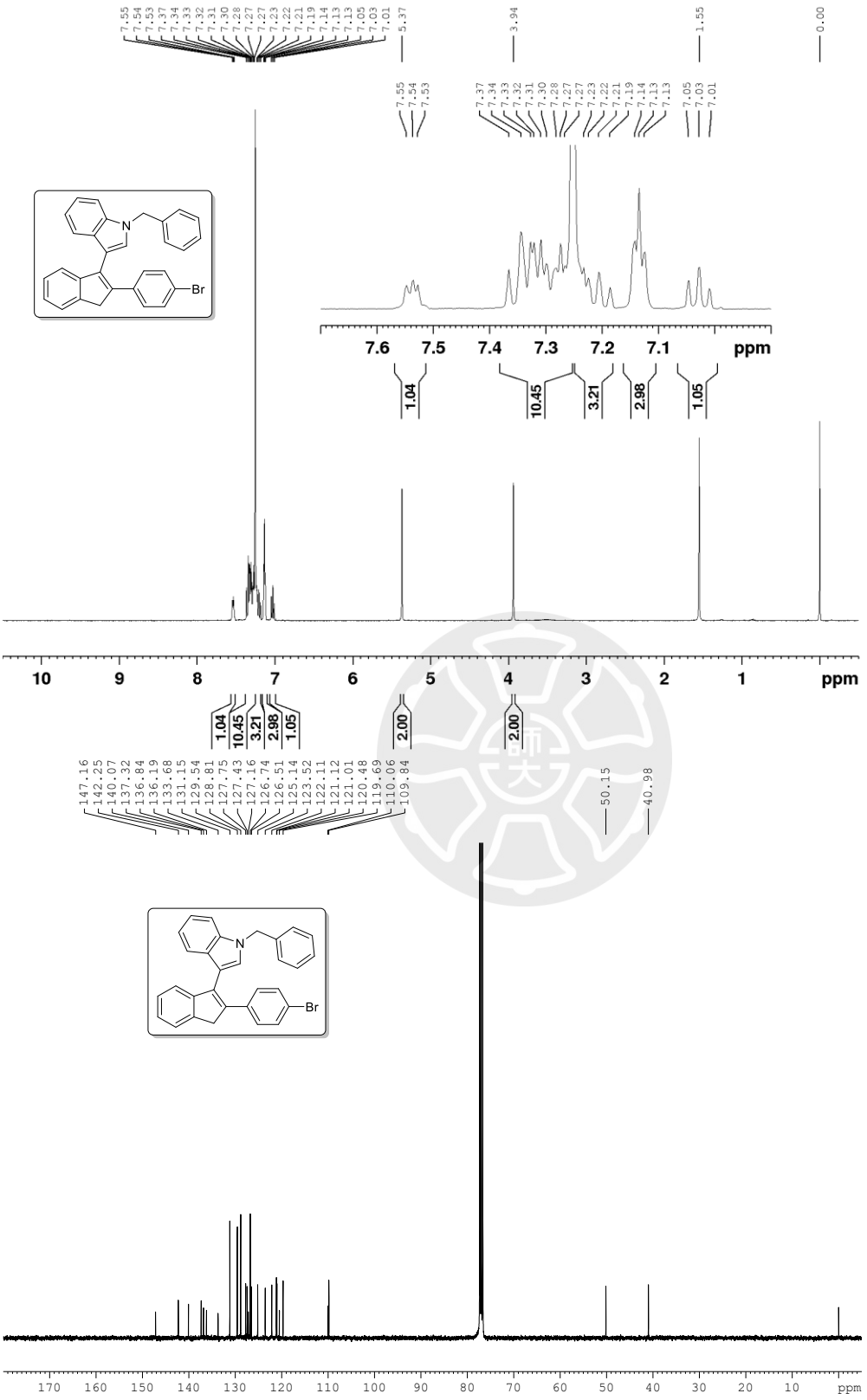
F2 - Acquisition Parameters
Date_               20230816
Time                   22.00
INSTRUM                spect
PROBHD    5 mm BBO BB-1H
PULPROG             zgpg30
TD                    32768
SOLVENT               CDCl3
NS                    12000
DS                        0
SWH               24038.461 Hz
FIDRES            0.733596 Hz
AQ                0.6815744 sec
RG                    32768
DW                  20.800 usec
DE                    6.50 usec
TE                   297.5 K
D1                2.00000000 sec
D11               0.03000000 sec
TD0                        1

======== CHANNEL f1 ========
NUC1                     13C
P1                     10.00 usec
PL1                     4.20 dB
SFO1           100.6233325 MHz

======== CHANNEL f2 ========
CPDPRG[2          waltz16
NUC2                      1H
PCPD2                  90.00 usec
PL2                    10.20 dB
PL12                   26.00 dB
PL13                   29.00 dB
SFO2           400.1316005 MHz

F2 - Processing parameters
SI                        32768
SF                100.6127690 MHz
WDW                          EM
SSB                           0
LB                      1.00 Hz
GB                            0
PC                         1.00
```

1-benzyl-3-(2-(4-bromophenyl)-1*H*-inden-3-yl)-1*H*-indole (3hg)




```
Current Data Parameters
NAME      Aldehyde+indole
EXPNO                  55
PROCNO                  1

F2 - Acquisition Parameters
Date_           20230606
Time                4.41
INSTRUM            spect
PROBHD   5 mm BBO BB-1H
PULPROG             zg30
TD                 32768
SOLVENT            CDCl3
NS                    16
DS                     0
SWH             7246.377 Hz
FIDRES          0.221142 Hz
AQ              2.2609921 sec
RG                 322.5
DW                69.000 usec
DE                 6.50 usec
TE                297.2 K
D1             2.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                15.00 usec
PL1               11.10 dB
SFO1         400.1324008 MHz

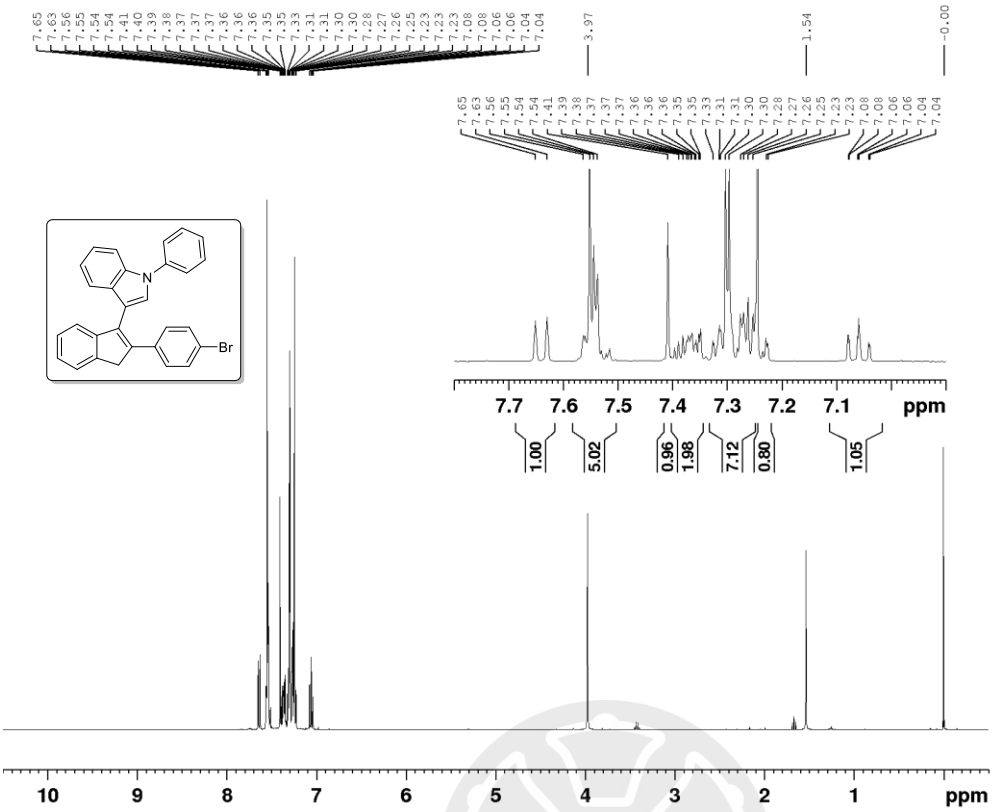
F2 - Processing parameters
SI                 16384
SF           400.1300119 MHz
WDW                   EM
SSB                    0
LB                  0 Hz
GB                     0
PC                  1.00
```

```
NAME      Aldehyde+indole
EXPNO                  56
PROCNO                  1
Date_           20230606
Time                10.09
INSTRUM            spect
PROBHD   5 mm BBO BB-1H
PULPROG           zgpg30
TD                 32768
SOLVENT            CDCl3
NS                  7844
DS                     0
SWH            24038.461 Hz
FIDRES          0.733596 Hz
AQ              0.6816452 sec
RG                3649.1
DW                20.800 usec
DE                 6.50 usec
TE                298.5 K
D1             2.00000000 sec
D11            0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                 13C
P1                10.00 usec
PL1                4.20 dB
SFO1         100.6233325 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                  1H
PCPD2             90.00 usec
PL2               10.20 dB
PL12              26.00 dB
PL13              29.00 dB
SFO2         400.1316005 MHz
SI                 32768
SF           100.6127698 MHz
WDW                   EM
SSB                    0
LB                1.00 Hz
GB                     0
PC                  1.00
```

3-(2-(4-bromophenyl)-1*H*-inden-3-yl)-1-phenyl-1*H*-indole (3hh)

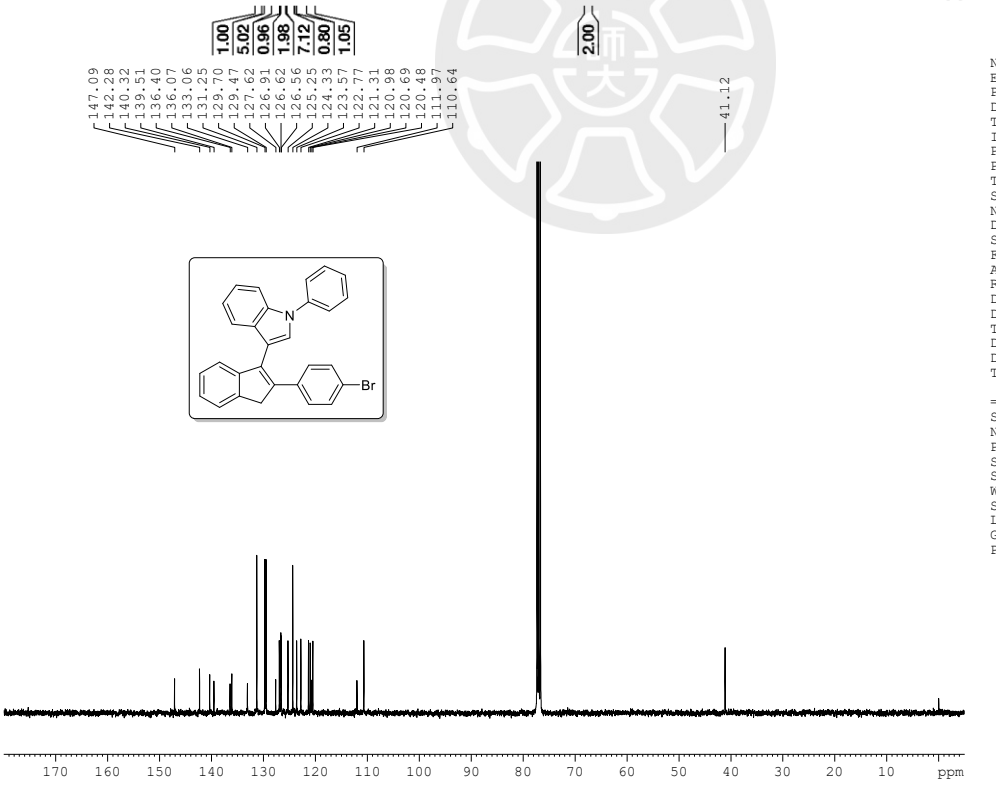



```
Current Data Parameters
NAME      Aldehyde+indole
EXPNO                  53
PROCNO                  1

F2 - Acquisition Parameters
Date_           20230607
Time                0.05
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG             zg30
TD                 32768
SOLVENT            CDCl3
NS                    16
DS                     0
SWH             7211.539 Hz
FIDRES          0.220079 Hz
AQ              2.2719147 sec
RG                177.16
DW                69.333 usec
DE                10.06 usec
TE                298.6 K
D1           2.00000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1         400.1324008 MHz
NUC1                   1H
P1                 15.00 usec
PLW1         13.50000000 W

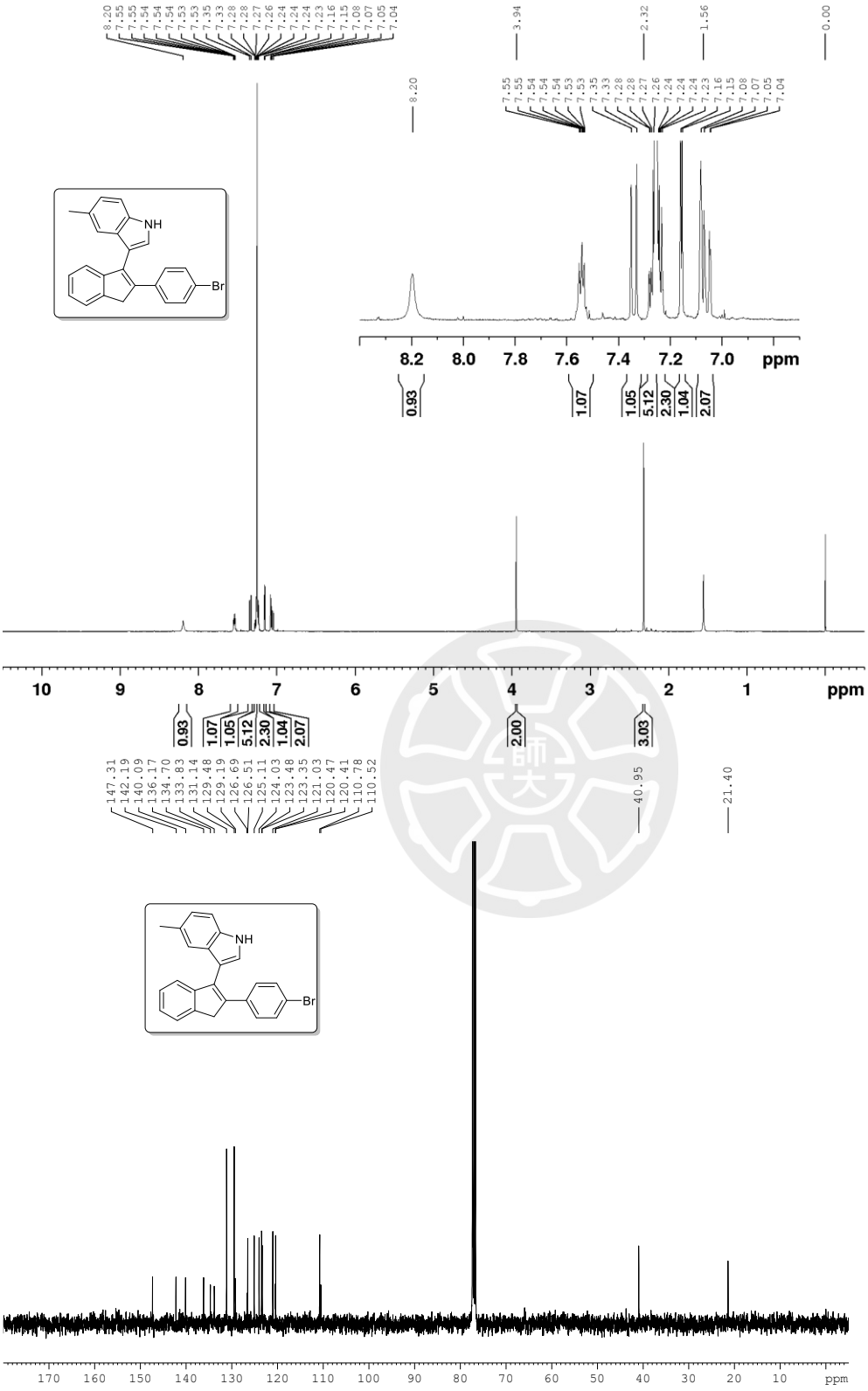
F2 - Processing parameters
SI                 16384
SF          400.1300156 MHz
WDW                   EM
SSB                    0
LB                     0 Hz
GB                     0
PC                  1.00
```




```
NAME      Aldehyde+indole
EXPNO                  54
PROCNO                  1
Date_           20230607
Time                0.12
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG          zgpg30
TD                 32768
SOLVENT            CDCl3
NS                   725
DS                     0
SWH            24038.461 Hz
FIDRES          0.733596 Hz
AQ              0.6816244 sec
RG                198.09
DW                20.800 usec
DE                 6.50 usec
TE                299.8 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1         100.6228298 MHz
NUC1                   13C
P1                10.00 usec
SI                 32768
SF          100.6127700 MHz
WDW                   EM
SSB                    0
LB                  2.00 Hz
GB                     0
PC                  1.00
```

3-(2-(4-bromophenyl)-1*H*-inden-3-yl)-5-methyl-1*H*-indole (3hi)




```
Current Data Parameters
NAME      Aldehyde+indole
EXPNO                  51
PROCNO                  1

F2 - Acquisition Parameters
Date_           20230325
Time               21.37
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG             zg30
TD                 32768
SOLVENT            CDC13
NS                    16
DS                     0
SWH             7211.539 Hz
FIDRES          0.220079 Hz
AQ              2.2719147 sec
RG                 158.74
DW                 69.333 usec
DE                 10.06 usec
TE                 299.0 K
D1              2.00000000 sec
TD0                    1

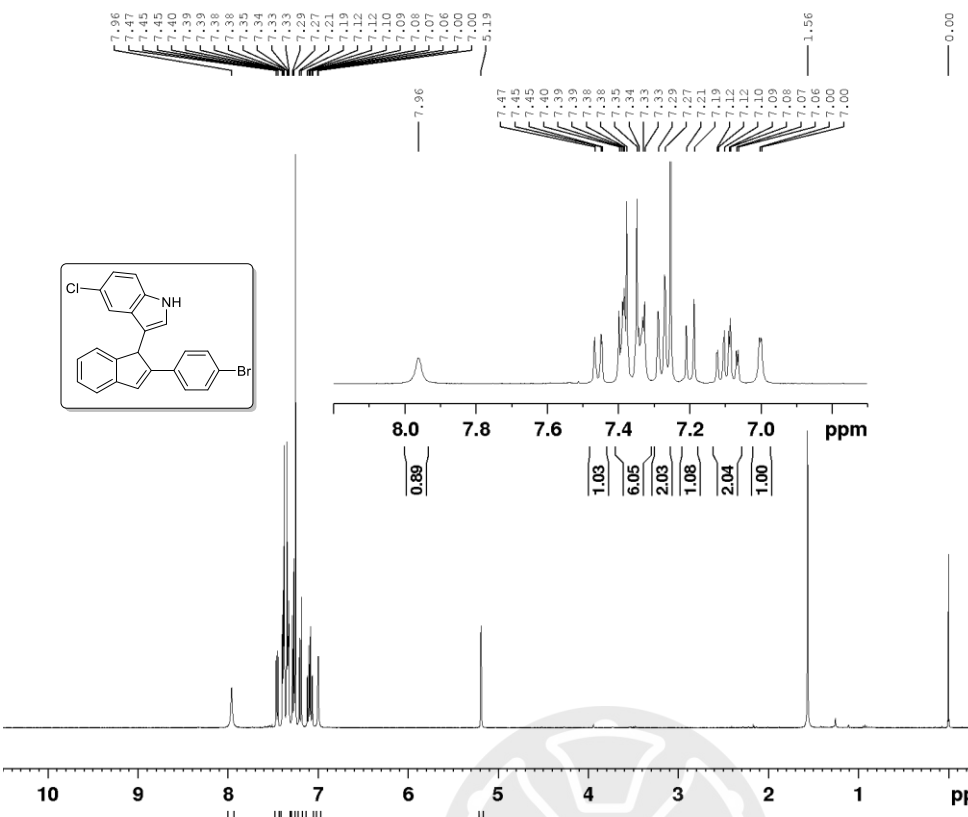
======== CHANNEL f1 ========
SFO1          400.1324008 MHz
NUC1                  1H
P1                 15.00 usec
PLW1          13.50000000 W

F2 - Processing parameters
SI                 16384
SF            400.1300129 MHz
WDW                   EM
SSB                    0
LB                     0 Hz
GB                     0
PC                  1.00
```

```
NAME      Aldehyde+indole
EXPNO                  52
PROCNO                  1
Date_           20230325
Time               21.43
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG           zgpg30
TD                 32768
SOLVENT            CDC13
NS                   214
DS                     0
SWH            24038.461 Hz
FIDRES          0.733596 Hz
AQ              0.6816244 sec
RG                 198.09
DW                 20.800 usec
DE                  6.50 usec
TE                 299.9 K
D1              2.00000000 sec
D11             0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1          100.6228298 MHz
NUC1                  13C
P1                 10.00 usec
SI                 32768
SF            100.6127710 MHz
WDW                   EM
SSB                    0
LB                  2.00 Hz
GB                     0
PC                  1.00
```

3-(2-(4-bromophenyl)-1*H*-inden-3-yl)-5-chloro-1*H*-indole (4hj)

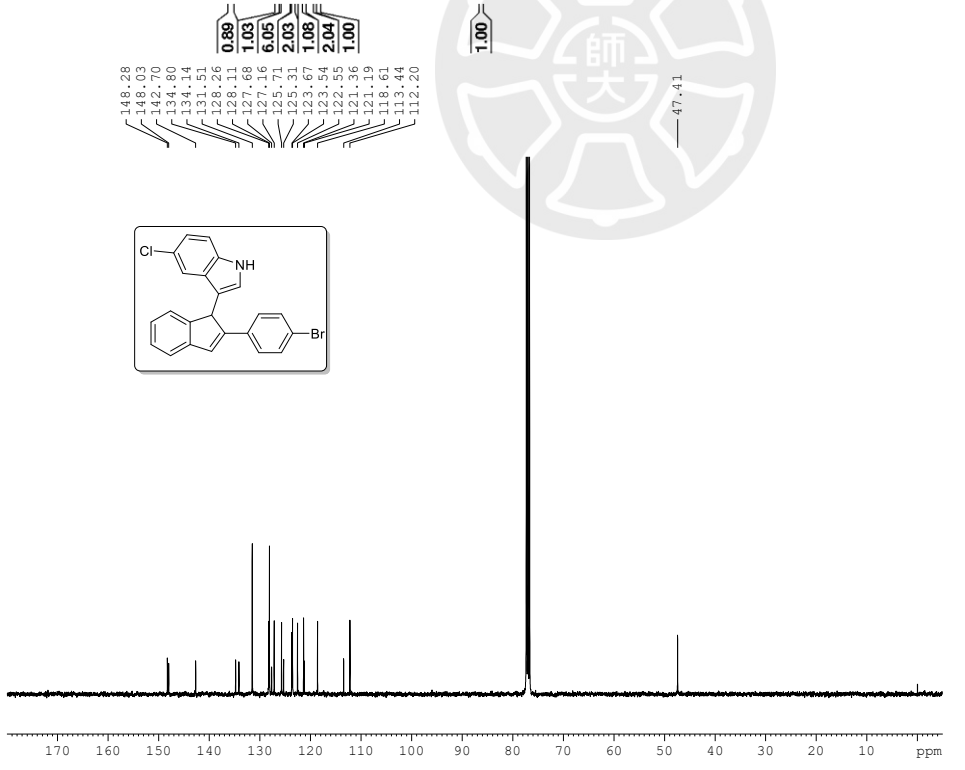



```
Current Data Parameters
NAME      Aldehyde+indole
EXPNO                  49
PROCNO                  1

F2 - Acquisition Parameters
Date_           20230401
Time              19.18
INSTRUM           spect
PROBHD   5 mm PABBO BB/
PULPROG            zg30
TD                32768
SOLVENT           CDCl3
NS                   16
DS                    0
SWH            7211.539 Hz
FIDRES         0.220079 Hz
AQ            2.2719147 sec
RG               198.09
DW               69.333 usec
DE               10.06 usec
TE               299.4 K
D1            2.00000000 sec
TD0                   1

======== CHANNEL f1 ========
SFO1         400.1324008 MHz
NUC1                 1H
P1                15.00 usec
PLW1         13.50000000 W

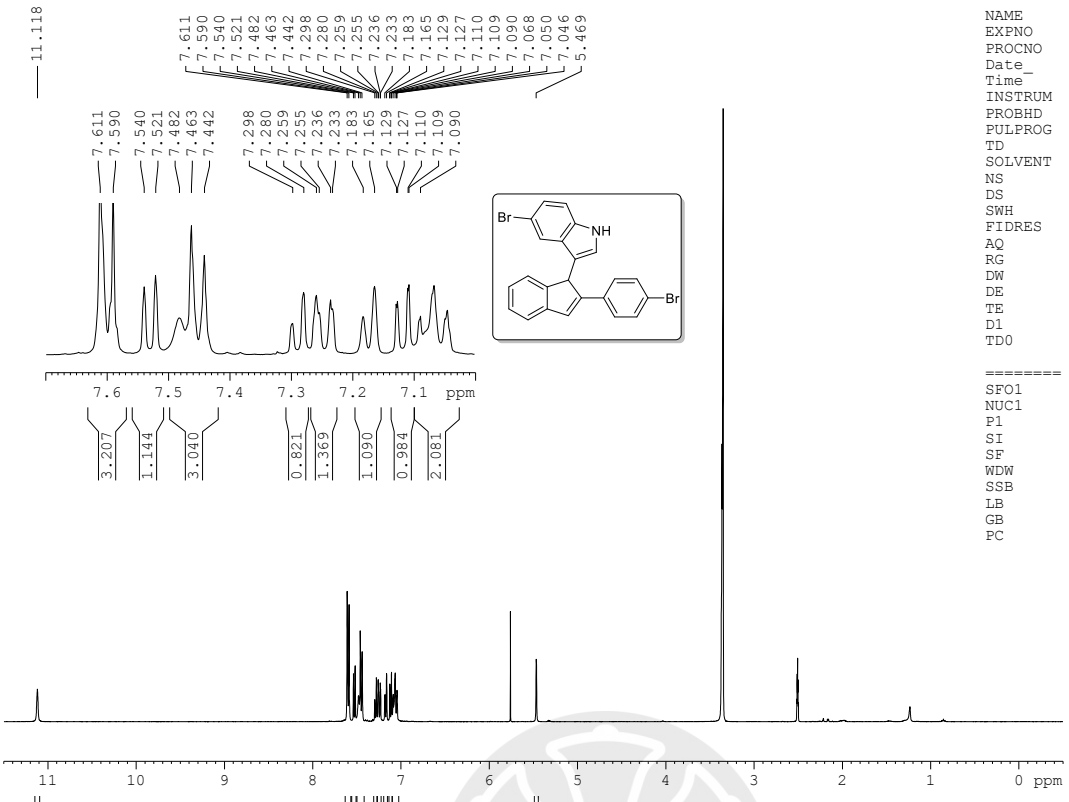
F2 - Processing parameters
SI                16384
SF           400.1300126 MHz
WDW                  EM
SSB                   0
LB                    0 Hz
GB                    0
PC                 1.00
```




```
NAME      Aldehyde+indole
EXPNO                  50
PROCNO                  1
Date_           20230401
Time              20.21
INSTRUM           spect
PROBHD   5 mm PABBO BB/
PULPROG          zgpg30
TD                32768
SOLVENT           CDCl3
NS                 1364
DS                    0
SWH           24038.461 Hz
FIDRES         0.733596 Hz
AQ            0.6816244 sec
RG               198.09
DW               20.800 usec
DE                6.50 usec
TE               300.4 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0                   1

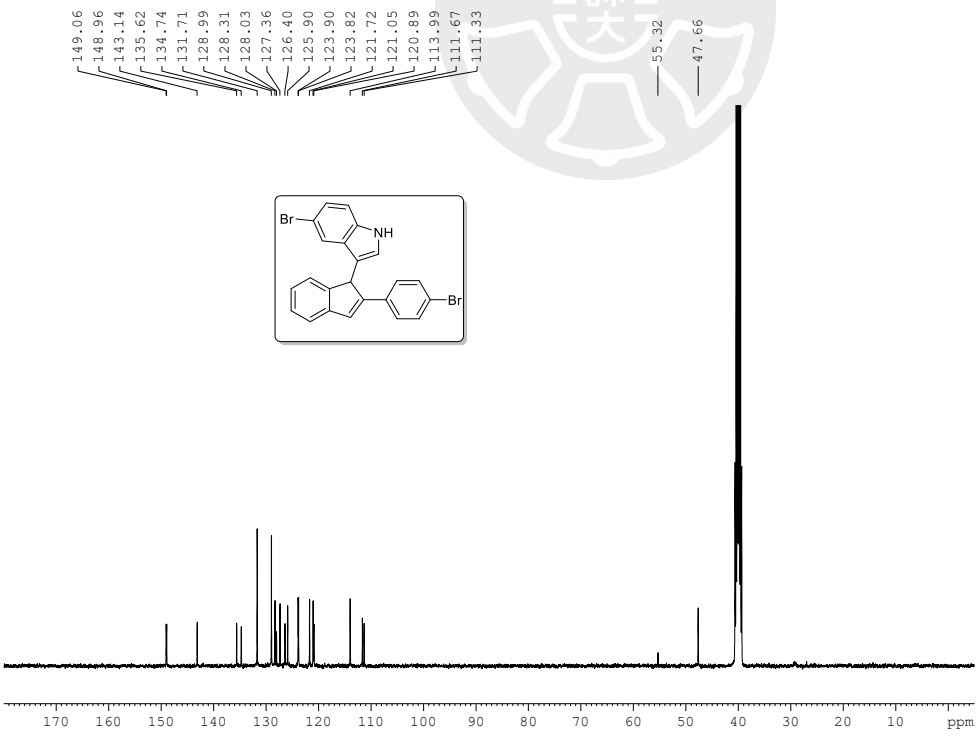
======== CHANNEL f1 ========
SFO1         100.6228298 MHz
NUC1                 13C
P1                10.00 usec
SI                32768
SF           100.6127713 MHz
WDW                  EM
SSB                   0
LB                 2.00 Hz
GB                    0
PC                 1.00
```

5-bromo-3-(2-(4-bromophenyl)-1*H*-inden-3-yl)-1*H*-indole (4hk)




```
NAME      Aldehyde+indole
EXPNO                  47
PROCNO                  1
Date_            20230619
Time                3.36
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG             zg30
TD                 32768
SOLVENT              DMSO
NS                     16
DS                      0
SWH            7211.539 Hz
FIDRES         0.220079 Hz
AQ            2.2719646 sec
RG                113.31
DW                69.333 usec
DE                 10.06 usec
TE                 298.1 K
D1           2.00000000 sec
TD0                     1

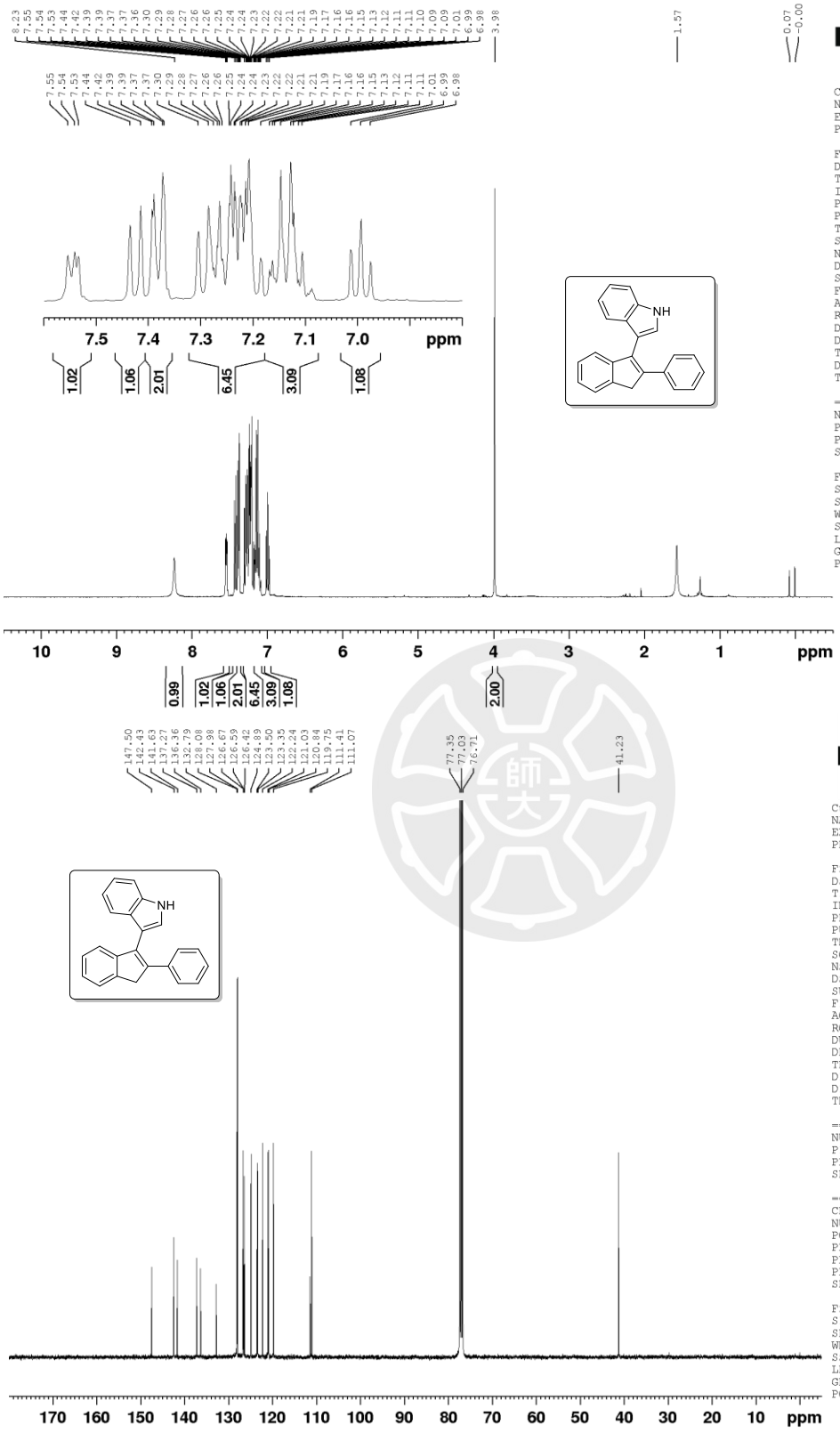
======== CHANNEL f1 ========
SFO1         400.1324008 MHz
NUC1                   1H
P1                  15.00 usec
SI                  16384
SF           400.1300000 MHz
WDW                    EM
SSB                     0
LB                   0.00 Hz
GB                      0
PC                   1.00
```




```
NAME      Aldehyde+indole
EXPNO                  48
PROCNO                  1
Date_            20230619
Time                5.09
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG           zgpg30
TD                 32768
SOLVENT              DMSO
NS                   4737
DS                      0
SWH           24038.461 Hz
FIDRES         0.733596 Hz
AQ            0.6816244 sec
RG                198.09
DW                20.800 usec
DE                 6.50 usec
TE                 300.2 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                     1

======== CHANNEL f1 ========
SFO1         100.6228298 MHz
NUC1                   13C
P1                  10.00 usec
SI                  32768
SF           100.6127726 MHz
WDW                    EM
SSB                     0
LB                   2.00 Hz
GB                      0
PC                   1.00
```

3-(2-phenyl-1*H*-inden-3-yl)-1*H*-indole (3al)



```
Current Data Parameters
NAME                  exp 270
EXPNO                       2
PROCNO                      1

F2 - Acquisition Parameters
Date_              20210913
Time                  20.47
INSTRUM               spect
PROBHD    5 mm BBO BB-1H
PULPROG                zg30
TD                    32768
SOLVENT               CDCl3
NS                       16
DS                        0
SWH                7246.377 Hz
FIDRES            0.221142 Hz
AQ                2.2609921 sec
RG                      181
DW                 69.000 usec
DE                   6.50 usec
TE                  297.6 K
D1              2.00000000 sec
TD0                       1

======== CHANNEL f1 ========
NUC1                      1H
P1                 15.00 usec
PL1                 11.10 dB
SFO1          400.1324008 MHz

F2 - Processing parameters
SI                    16384
SF            400.1300188 MHz
WDW                      EM
SSB                       0
LB                     0 Hz
GB                        0
PC                     1.00
```



```
Current Data Parameters
NAME                  exp 270
EXPNO                       6
PROCNO                      1

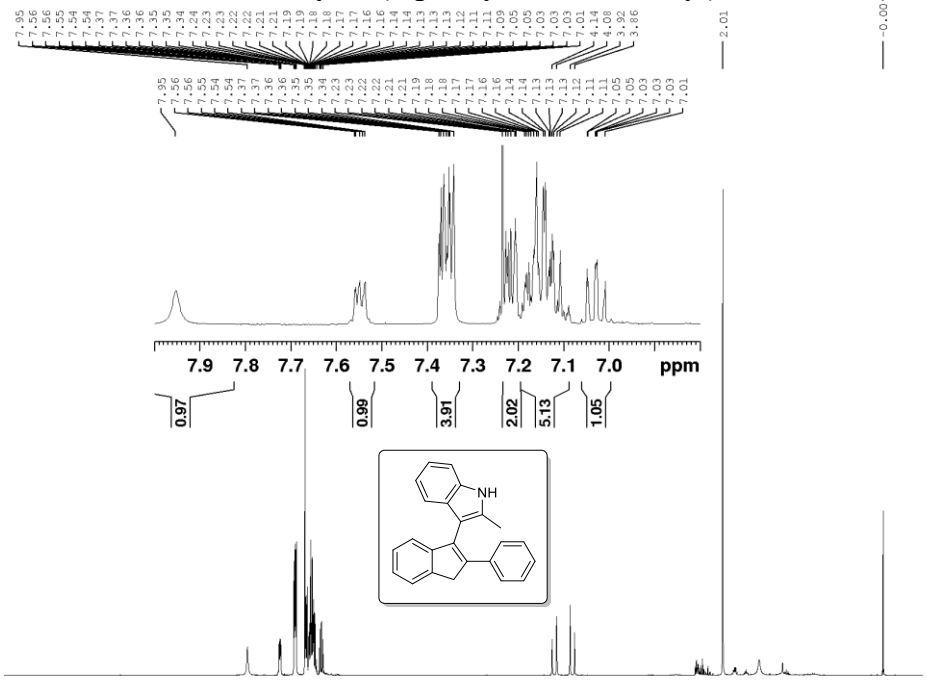
F2 - Acquisition Parameters
Date_              20210913
Time                  22.11
INSTRUM               spect
PROBHD    5 mm BBO BB-1H
PULPROG             zgpg30
TD                    32768
SOLVENT               CDCl3
NS                    12000
DS                        0
SWH            24038.461 Hz
FIDRES          0.733596 Hz
AQ              0.6815744 sec
RG                    32768
DW                 20.800 usec
DE                   6.50 usec
TE                  298.1 K
D1              2.00000000 sec
D11             0.03000000 sec
TD0                       1

======== CHANNEL f1 ========
NUC1                     13C
P1                 10.00 usec
PL1                  3.80 dB
SFO1          100.6233325 MHz

======== CHANNEL f2 ========
CPDPRG[2           waltz16
NUC2                      1H
PCPD2              90.00 usec
PL2                 10.20 dB
PL12                26.00 dB
PL13                29.00 dB
SFO2          400.1316005 MHz

F2 - Processing parameters
SI                    32768
SF            100.6127690 MHz
WDW                      EM
SSB                       0
LB                  1.00 Hz
GB                        0
PC                     1.00
```

2-methyl-3-(2-phenyl-1*H*-inden-3-yl)-1*H*-indole (3am)

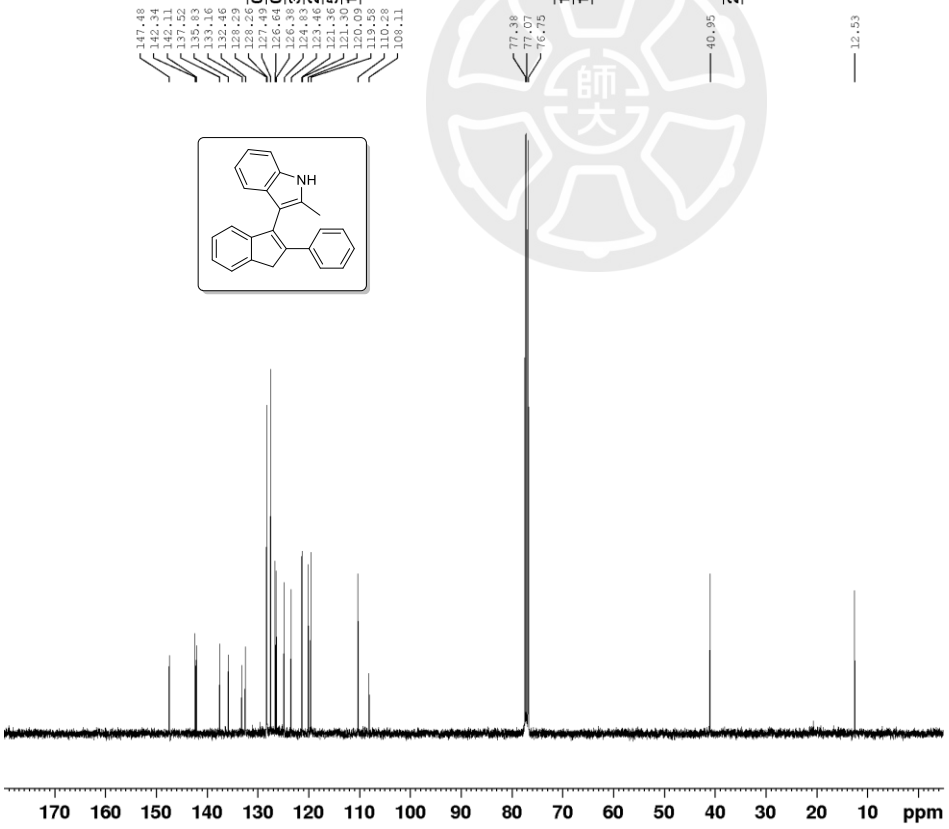



```
Current Data Parameters
NAME          Suresh-273
EXPNO                  4
PROCNO                 1

F2 - Acquisition Parameters
Date_           20230607
Time               23.34
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG             zg30
TD                 32768
SOLVENT            CDC13
NS                    16
DS                     0
SWH             7211.539 Hz
FIDRES          0.220079 Hz
AQ              2.2719147 sec
RG                 89.08
DW                69.333 usec
DE                10.06 usec
TE                298.6 K
D1              2.00000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1          400.1324008 MHz
NUC1                  1H
P1                 15.00 usec
PLW1          13.50000000 W

F2 - Processing parameters
SI                 16384
SF            400.1300200 MHz
WDW                   EM
SSB                    0
LB                  0 Hz
GB                     0
PC                  1.00
```




```
Current Data Parameters
NAME             exp 273
EXPNO                  4
PROCNO                 1

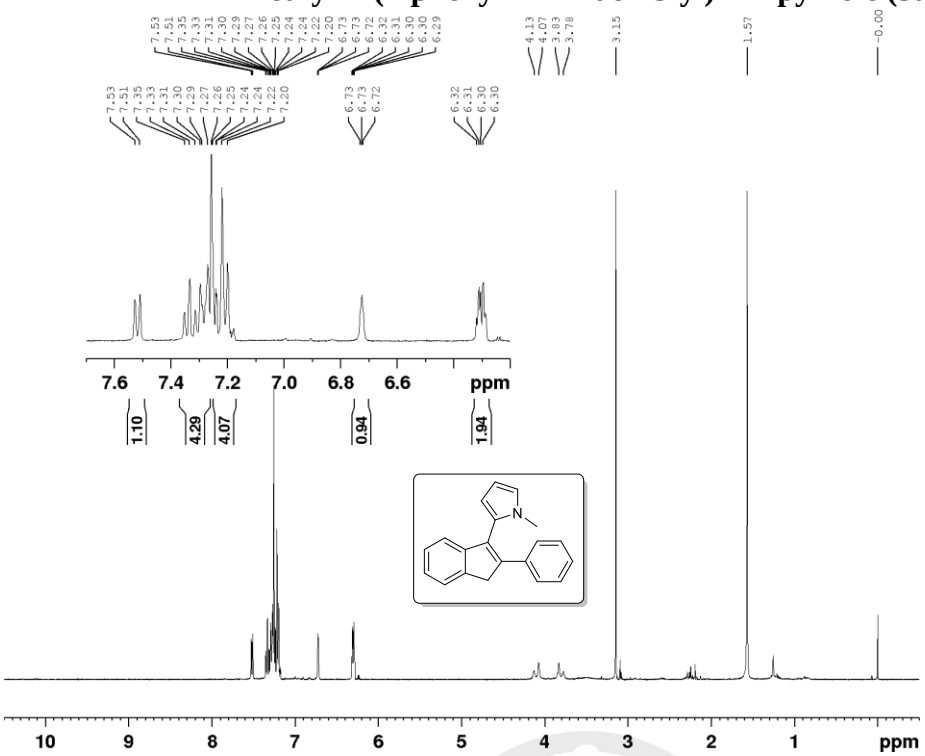
F2 - Acquisition Parameters
Date_           20210930
Time               21.15
INSTRUM            spect
PROBHD   5 mm BBO BB-1H
PULPROG          zgpg30
TD                 32768
SOLVENT            CDC13
NS                   412
DS                     0
SWH            24038.461 Hz
FIDRES          0.733596 Hz
AQ              0.6815744 sec
RG                 32768
DW                20.800 usec
DE                 6.50 usec
TE                298.3 K
D1              2.00000000 sec
D11             0.03000000 sec
TD0                    1

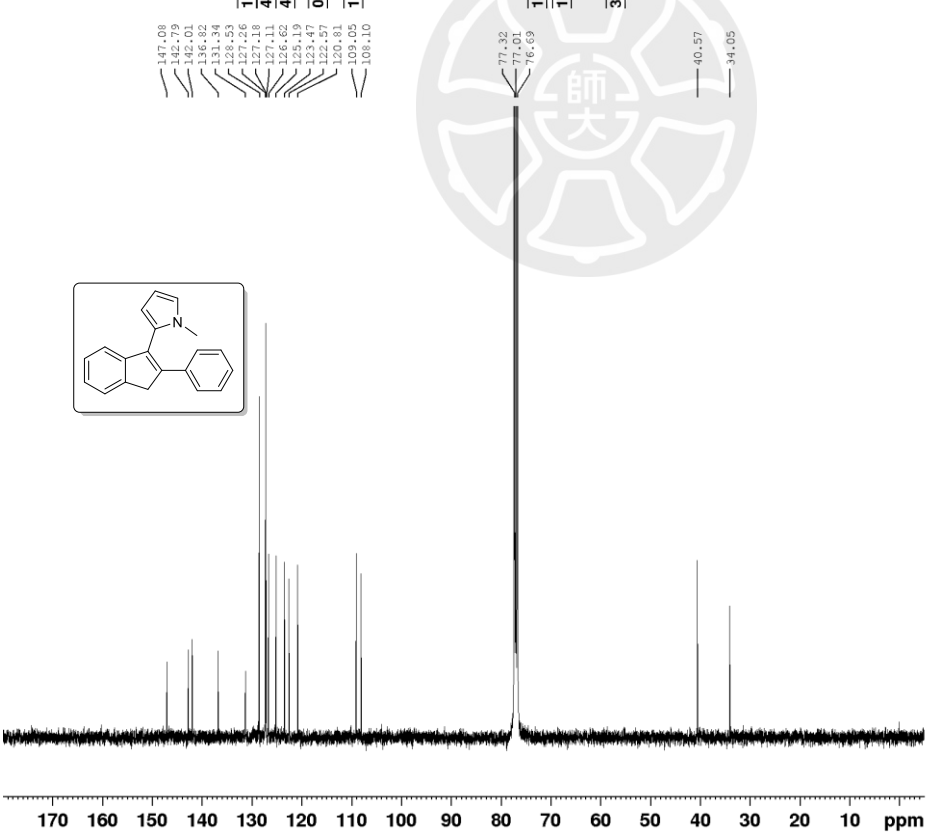
======== CHANNEL f1 ========
NUC1                  13C
P1                10.00 usec
PL1                3.80 dB
SFO1          100.6233325 MHz

======== CHANNEL f2 ========
CPDPRG[2          waltz16
NUC2                  1H
PCPD2             90.00 usec
PL2               10.20 dB
PL12              26.00 dB
PL13              29.00 dB
SFO2          400.1316005 MHz

F2 - Processing parameters
SI                 32768
SF            100.6127690 MHz
WDW                   EM
SSB                    0
LB                  1.00 Hz
GB                     0
PC                  1.00
```

1-methyl-2-(2-phenyl-1*H*-inden-3-yl)-1*H*-pyrrole (3an)





BRUKER

```
Current Data Parameters
NAME                 exp 266
EXPNO                      2
PROCNO                     1

F2 - Acquisition Parameters
Date_               20210906
Time                   17.31
INSTRUM                spect
PROBHD    5 mm BBO BB-1H
PULPROG                zg30
TD                    32768
SOLVENT               CDC13
NS                       16
DS                        0
SWH                7246.377 Hz
FIDRES             0.221142 Hz
AQ                 2.2609921 sec
RG                      362
DW                   69.000 usec
DE                    6.50 usec
TE                   296.8 K
D1                2.00000000 sec
TD0                        1

======== CHANNEL f1 ========
NUC1                      1H
P1                     15.00 usec
PL1                    11.10 dB
SFO1            400.1324008 MHz

F2 - Processing parameters
SI                        16384
SF              400.1300102 MHz
WDW                        EM
SSB                         0
LB                      0 Hz
GB                          0
PC                       1.00
```

BRUKER

```
Current Data Parameters
NAME                 exp 266
EXPNO                      5
PROCNO                     1

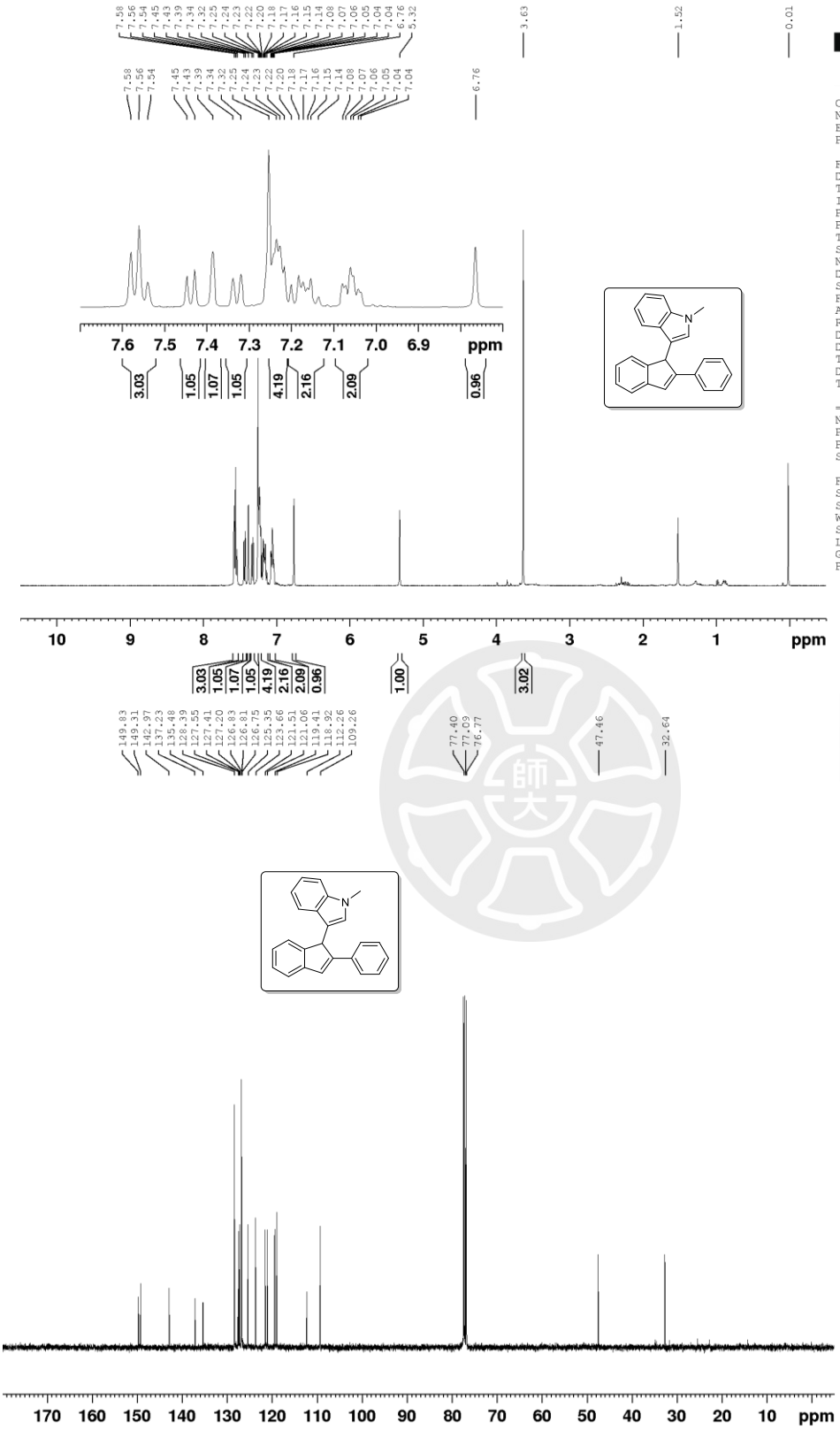
F2 - Acquisition Parameters
Date_               20210906
Time                   23.25
INSTRUM                spect
PROBHD    5 mm BBO BB-1H
PULPROG             zgpg30
TD                    32768
SOLVENT               CDC13
NS                    12277
DS                        0
SWH               24038.461 Hz
FIDRES             0.733596 Hz
AQ                 0.6815744 sec
RG                      32768
DW                   20.800 usec
DE                    6.50 usec
TE                   297.5 K
D1                2.00000000 sec
D11               0.03000000 sec
TD0                        1

======== CHANNEL f1 ========
NUC1                     13C
P1                     10.00 usec
PL1                     3.80 dB
SFO1            100.6233325 MHz

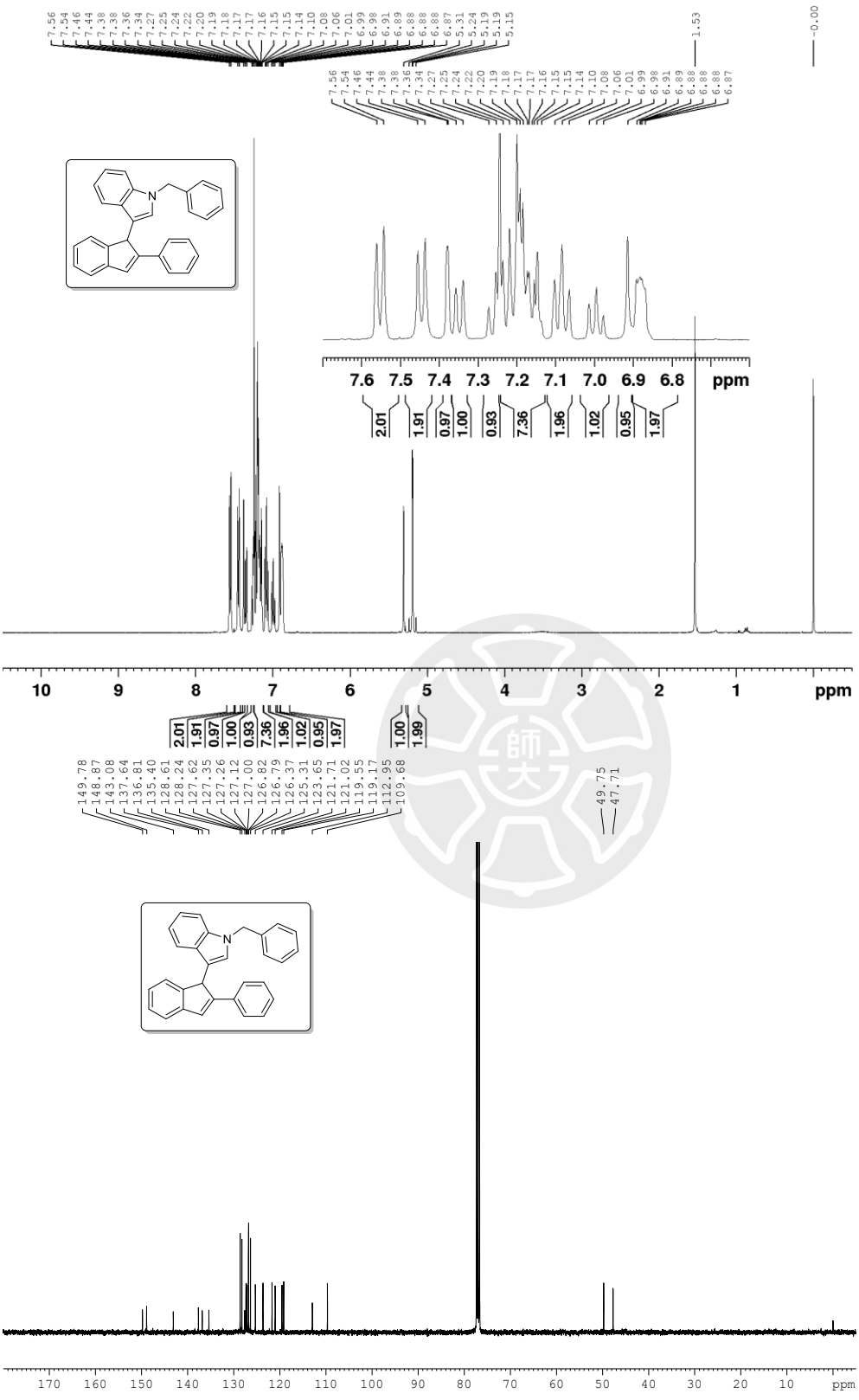
======== CHANNEL f2 ========
CPDPRG[2            waltz16
NUC2                      1H
PCPD2                  90.00 usec
PL2                    10.20 dB
PL12                   26.00 dB
PL13                   29.00 dB
SFO2            400.1316005 MHz

F2 - Processing parameters
SI                        32768
SF              100.6127690 MHz
WDW                        EM
SSB                         0
LB                      1.00 Hz
GB                          0
PC                       1.00
```

1-methyl-3-(2-phenyl-1*H*-inden-1-yl)-1*H*-indole (4aa)



1-benzyl-3-(2-phenyl-1*H*-inden-1-yl)-1*H*-indole (4ag)



```
Current Data Parameters
NAME      Aldehyde+indole
EXPNO                  59
PROCNO                  1

F2 - Acquisition Parameters
Date_           20230610
Time              17.08
INSTRUM           spect
PROBHD   5 mm BBO BB-1H
PULPROG             zg30
TD                 32768
SOLVENT            CDCl3
NS                    16
DS                     0
SWH             7246.377 Hz
FIDRES          0.221142 Hz
AQ              2.2609921 sec
RG                 228.1
DW                69.000 usec
DE                 6.50 usec
TE                296.7 K
D1             2.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                15.00 usec
PL1               11.10 dB
SFO1         400.1324008 MHz

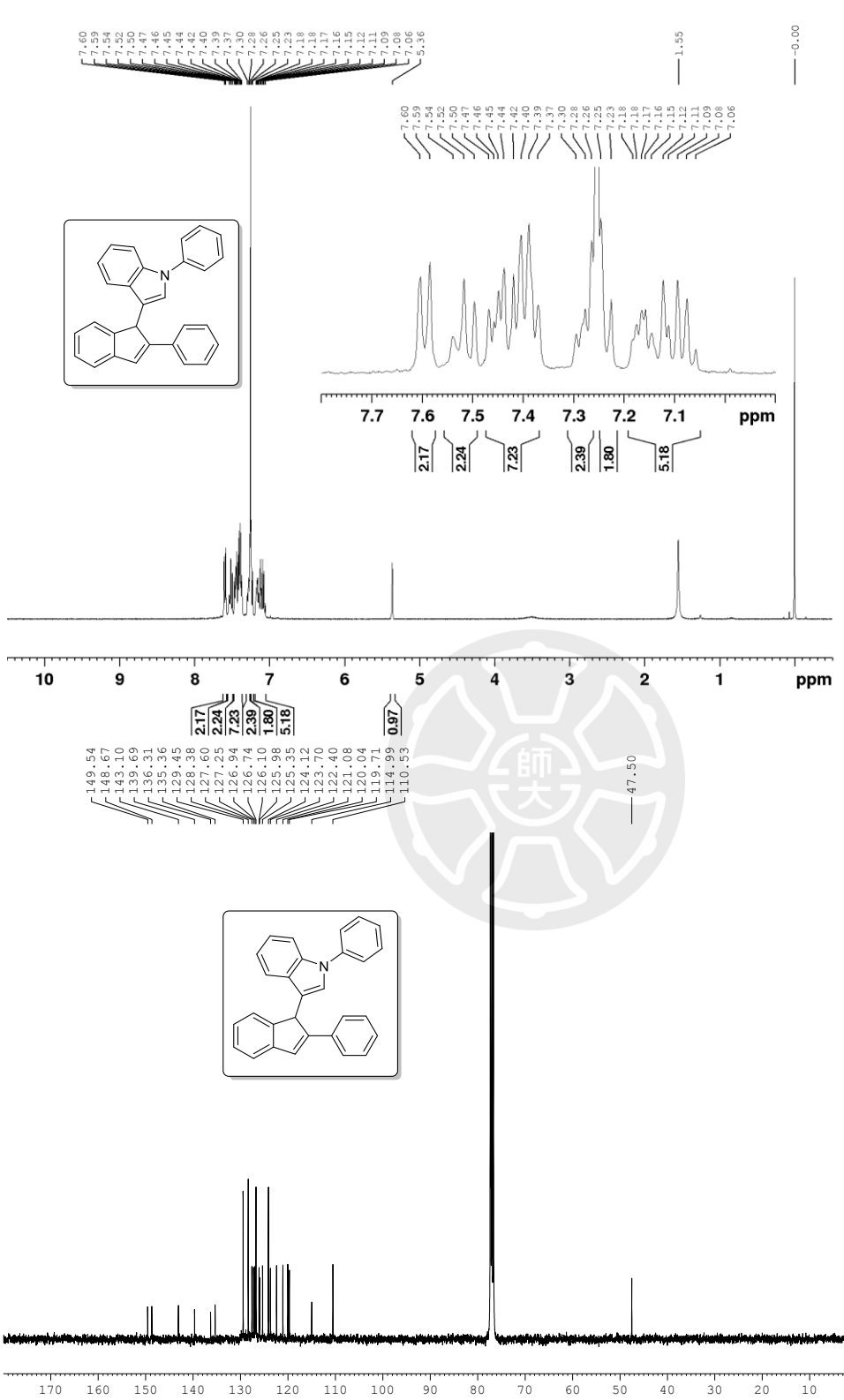
F2 - Processing parameters
SI                 16384
SF          400.1300158 MHz
WDW                   EM
SSB                    0
LB                     0 Hz
GB                     0
PC                  1.00
```

```
NAME      Aldehyde+indole
EXPNO                  60
PROCNO                  1
Date_           20230610
Time              19.12
INSTRUM           spect
PROBHD   5 mm BBO BB-1H
PULPROG           zgpg30
TD                 32768
SOLVENT            CDCl3
NS                  2660
DS                     0
SWH            24038.461 Hz
FIDRES          0.733596 Hz
AQ              0.6816452 sec
RG                5792.6
DW                20.800 usec
DE                 6.50 usec
TE                297.9 K
D1             2.00000000 sec
D11            0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                 13C
P1                10.00 usec
PL1                4.20 dB
SFO1         100.6233325 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                  1H
PCPD2             90.00 usec
PL2               10.20 dB
PL12              26.00 dB
PL13              29.00 dB
SFO2         400.1316005 MHz
SI                 32768
SF          100.6127712 MHz
WDW                   EM
SSB                    0
LB                  1.00 Hz
GB                     0
PC                  1.00
```

1-phenyl-3-(2-phenyl-1*H*-inden-1-yl)-1*H*-indole (4ah)



```
Current Data Parameters
NAME      Aldehyde+indole
EXPNO                  57
PROCNO                  1

F2 - Acquisition Parameters
Date_           20230603
Time              21.20
INSTRUM           spect
PROBHD   5 mm BBO BB-1H
PULPROG            zg30
TD                32768
SOLVENT           CDCl3
NS                   16
DS                    0
SWH            7246.377 Hz
FIDRES         0.221142 Hz
AQ             2.2609921 sec
RG                  362
DW               69.000 usec
DE                6.50 usec
TE               297.9 K
D1            2.00000000 sec
TD0                   1

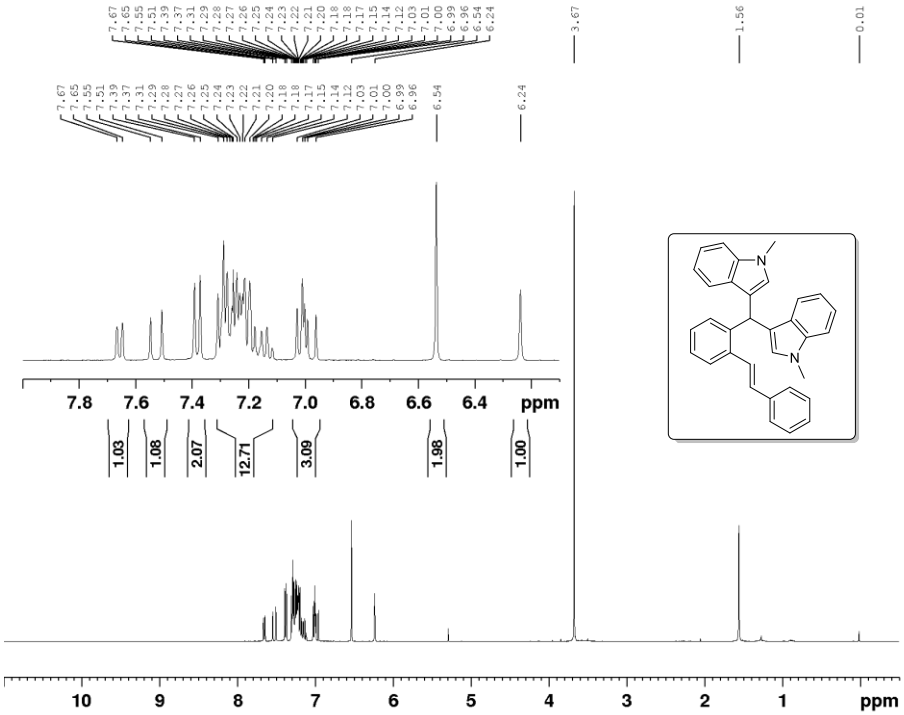
======== CHANNEL f1 ========
NUC1                  1H
P1                15.00 usec
PL1               11.10 dB
SFO1         400.1324008 MHz

F2 - Processing parameters
SI                16384
SF          400.1300116 MHz
WDW                  EM
SSB                   0
LB                 0 Hz
GB                    0
PC                 1.00
```

```
NAME      Aldehyde+indole
EXPNO                  58
PROCNO                  1
Date_           20230615
Time              14.45
INSTRUM           spect
PROBHD   5 mm PABBO BB/
PULPROG          zgpg30
TD                32768
SOLVENT           CDCl3
NS                 4265
DS                    0
SWH           24038.461 Hz
FIDRES         0.733596 Hz
AQ             0.6816244 sec
RG                198.09
DW               20.800 usec
DE                6.50 usec
TE               300.2 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
SFO1         100.6228298 MHz
NUC1               13C
P1               10.00 usec
SI                32768
SF          100.6127689 MHz
WDW                  EM
SSB                   0
LB                2.00 Hz
GB                    0
PC                 1.00
```

(*E*)-3,3'-((2-styrylphenyl)methylene)bis(1-methyl-1*H*-indole) (6aa)

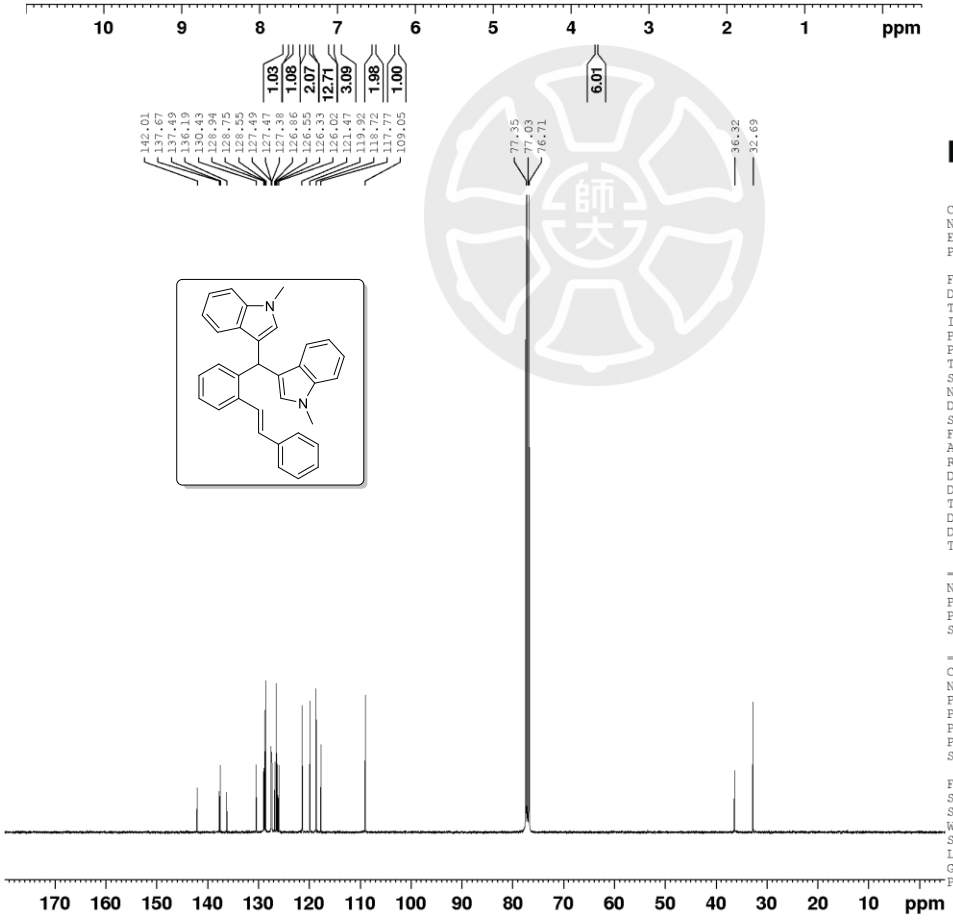



```
Current Data Parameters
NAME                  exp 276
EXPNO                       5
PROCNO                      1

F2 - Acquisition Parameters
Date_               20210923
Time                   22.00
INSTRUM                spect
PROBHD    5 mm BBO BB-1H
PULPROG                 zg30
TD                     32768
SOLVENT                CDC13
NS                        16
DS                         0
SWH                7246.377 Hz
FIDRES            0.221142 Hz
AQ                2.2609921 sec
RG                     228.1
DW                    69.000 usec
DE                     6.50 usec
TE                    297.9 K
D1                2.00000000 sec
TD0                        1

======== CHANNEL f1 ========
NUC1                      1H
P1                     15.00 usec
PL1                    11.10 dB
SFO1           400.1324008 MHz

F2 - Processing parameters
SI                     16384
SF               400.1300118 MHz
WDW                       EM
SSB                        0
LB                      0 Hz
GB                         0
PC                      1.00
```




```
Current Data Parameters
NAME                  exp 276
EXPNO                       7
PROCNO                      1

F2 - Acquisition Parameters
Date_               20210923
Time                   22.28
INSTRUM                spect
PROBHD    5 mm BBO BB-1H
PULPROG             zgpg30
TD                     32768
SOLVENT                CDC13
NS                     12000
DS                         0
SWH               24038.461 Hz
FIDRES            0.733596 Hz
AQ                0.6815744 sec
RG                     32768
DW                   20.800 usec
DE                     6.50 usec
TE                    298.3 K
D1                2.00000000 sec
D11               0.03000000 sec
TD0                        1

======== CHANNEL f1 ========
NUC1                      13C
P1                     10.00 usec
PL1                     3.80 dB
SFO1          100.6233325 MHz

======== CHANNEL f2 ========
CPDPRG[2              waltz16
NUC2                      1H
PCPD2                  90.00 usec
PL2                    10.20 dB
PL12                   26.00 dB
PL13                   29.00 dB
SFO2          400.1316005 MHz

F2 - Processing parameters
SI                     32768
SF               100.6127690 MHz
WDW                       EM
SSB                        0
LB                     1.00 Hz
GB                         0
PC                      1.00
```

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