

Supplementary Material

Combination of $\text{NH}_2\text{OH}\cdot\text{HCl}$ and NaIO_4 : a new and mild reagent for the synthesis of vicinal diiodo carbonyl compounds

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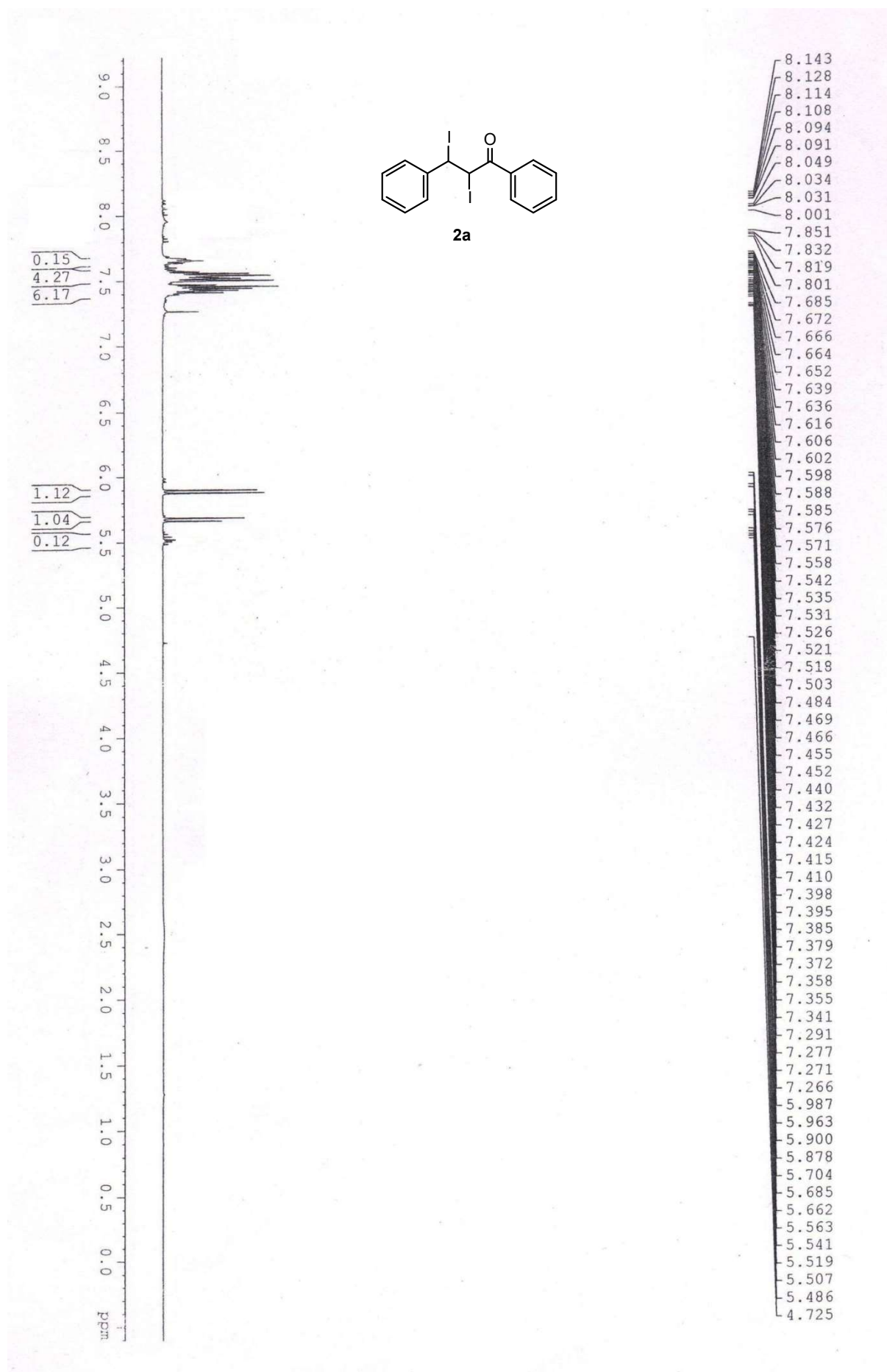
^b*Jhargram Raj College, Jhargram, West Midnapore 721 507, West Bengal, India*

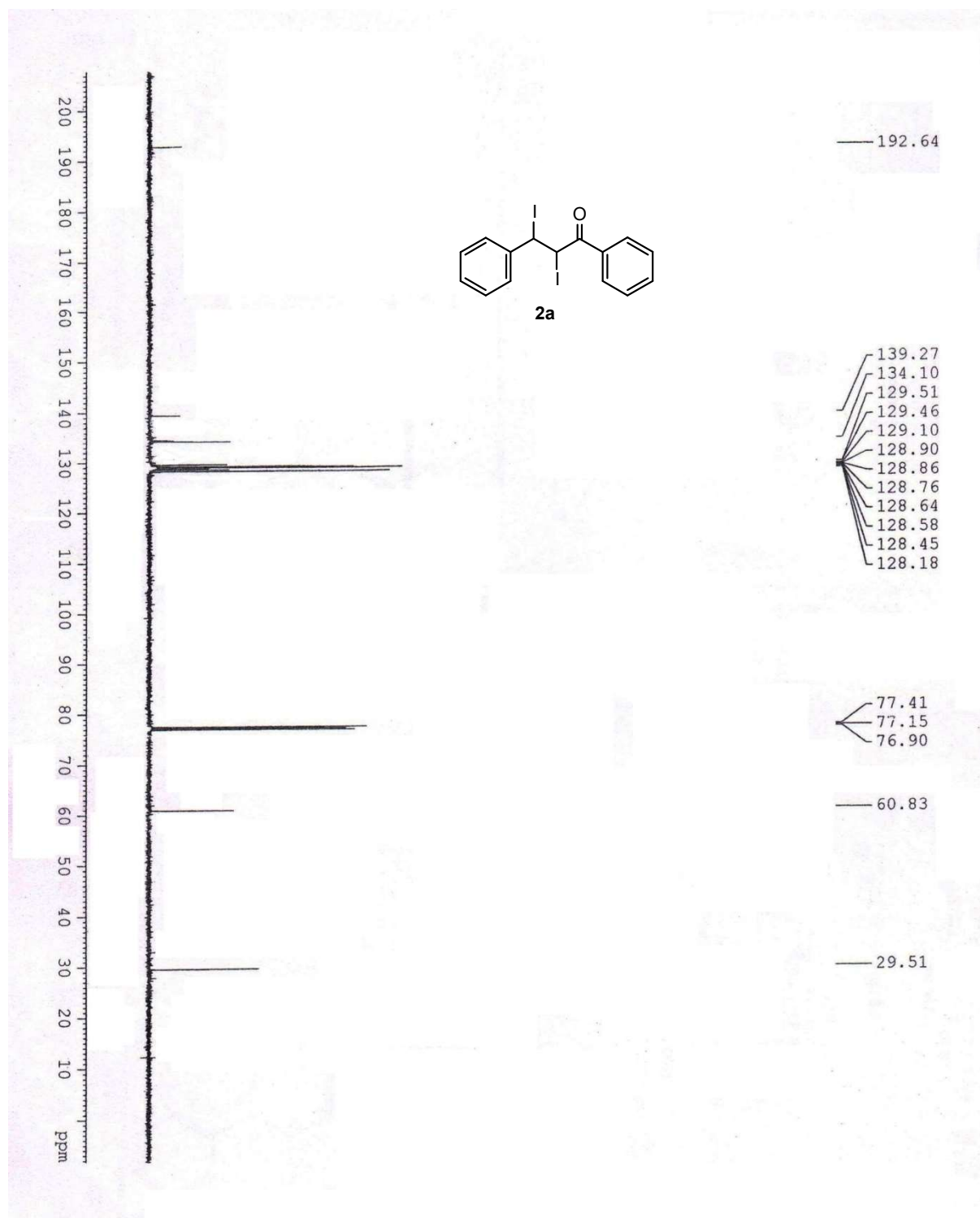
^c*Department of Chemistry, Visva-Bharati, Santiniketan 731 235, West Bengal, India*

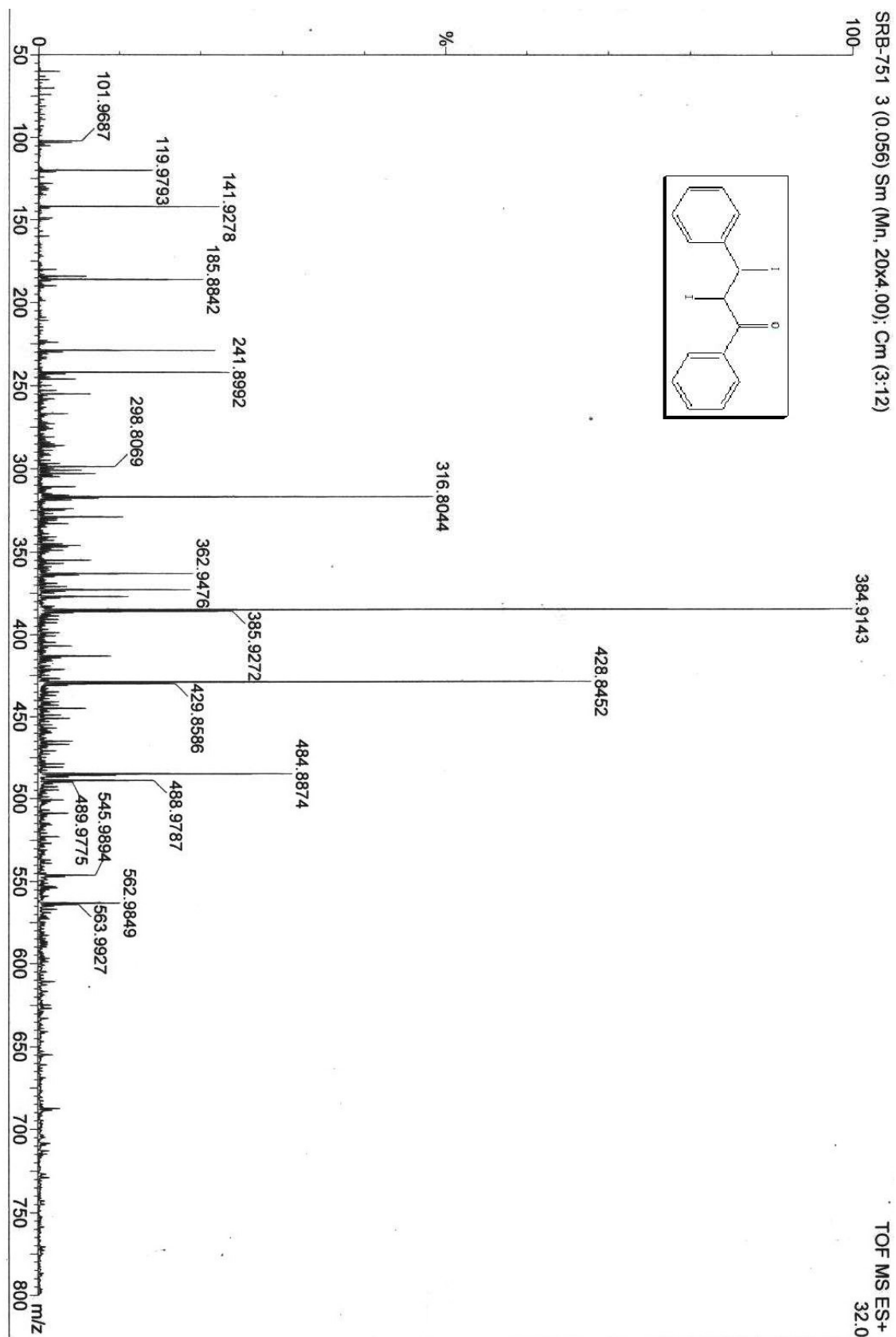
E-mail: adinath.majee@visva-bharati.ac.in

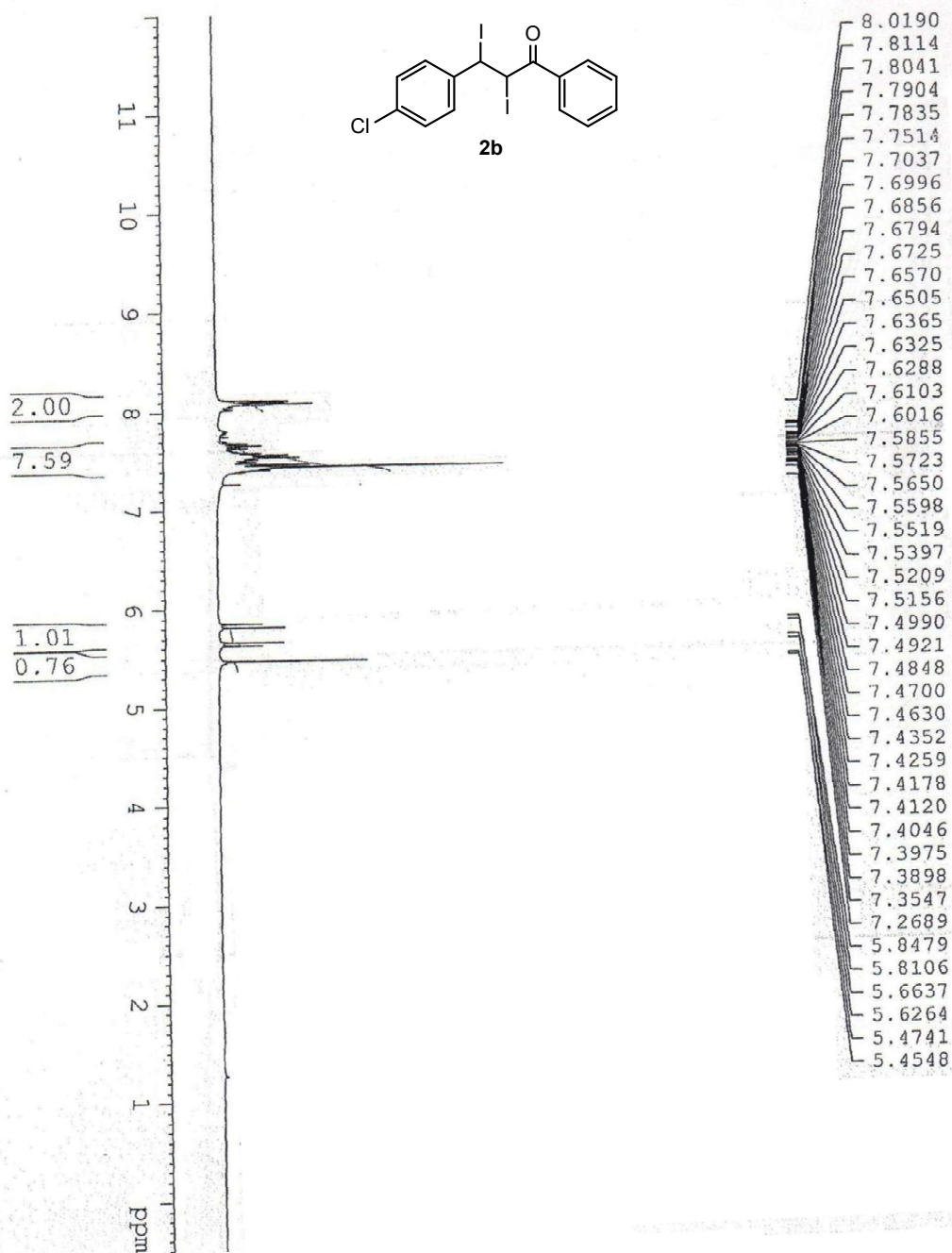
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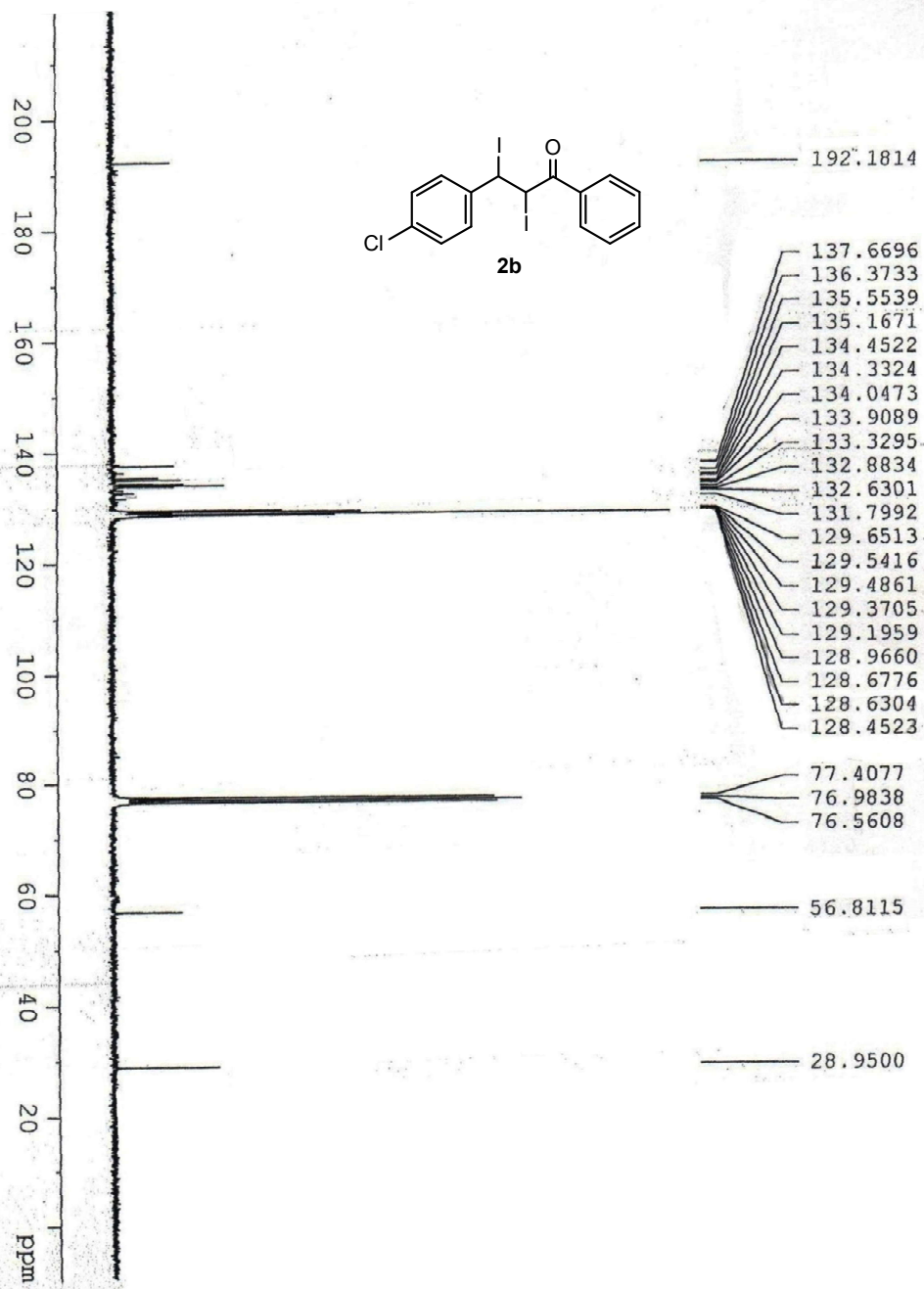
Copies of NMR spectra	2-3 & 5-21
Mass spectra of the compound 2a			4
Structure Determination	22-23

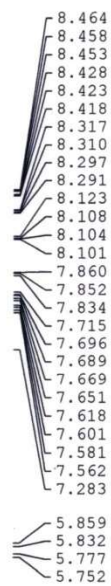










¹H of VBNC-80DINO2

Current Data Parameters
 NAME Dr. A MAJEE 2015
 EXENO 19
 PROCNO 1

F2 - Acquisition Parameters
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 Time 14.14

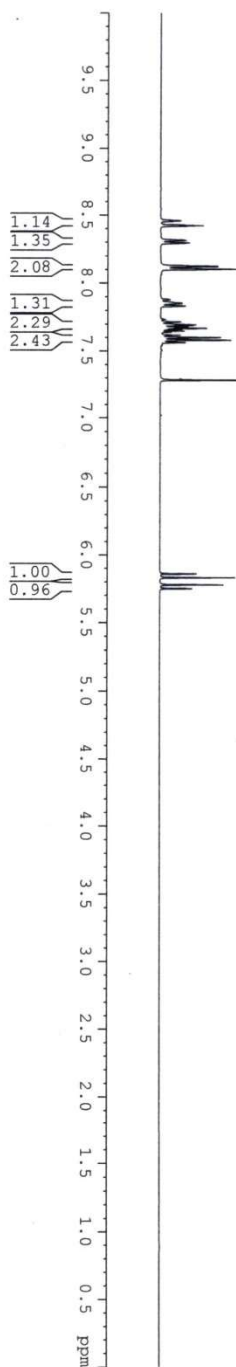
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 PROBHD 5 mm PABBO BB/
 PULPROG zg30
 TD 32768
 SOLVENT CDCl3
 NS 64
 DS 2

SWH 8223.685 Hz
 FIDRES 0.1250967 Hz
 AQ 1.9923444 sec
 RG 186.42

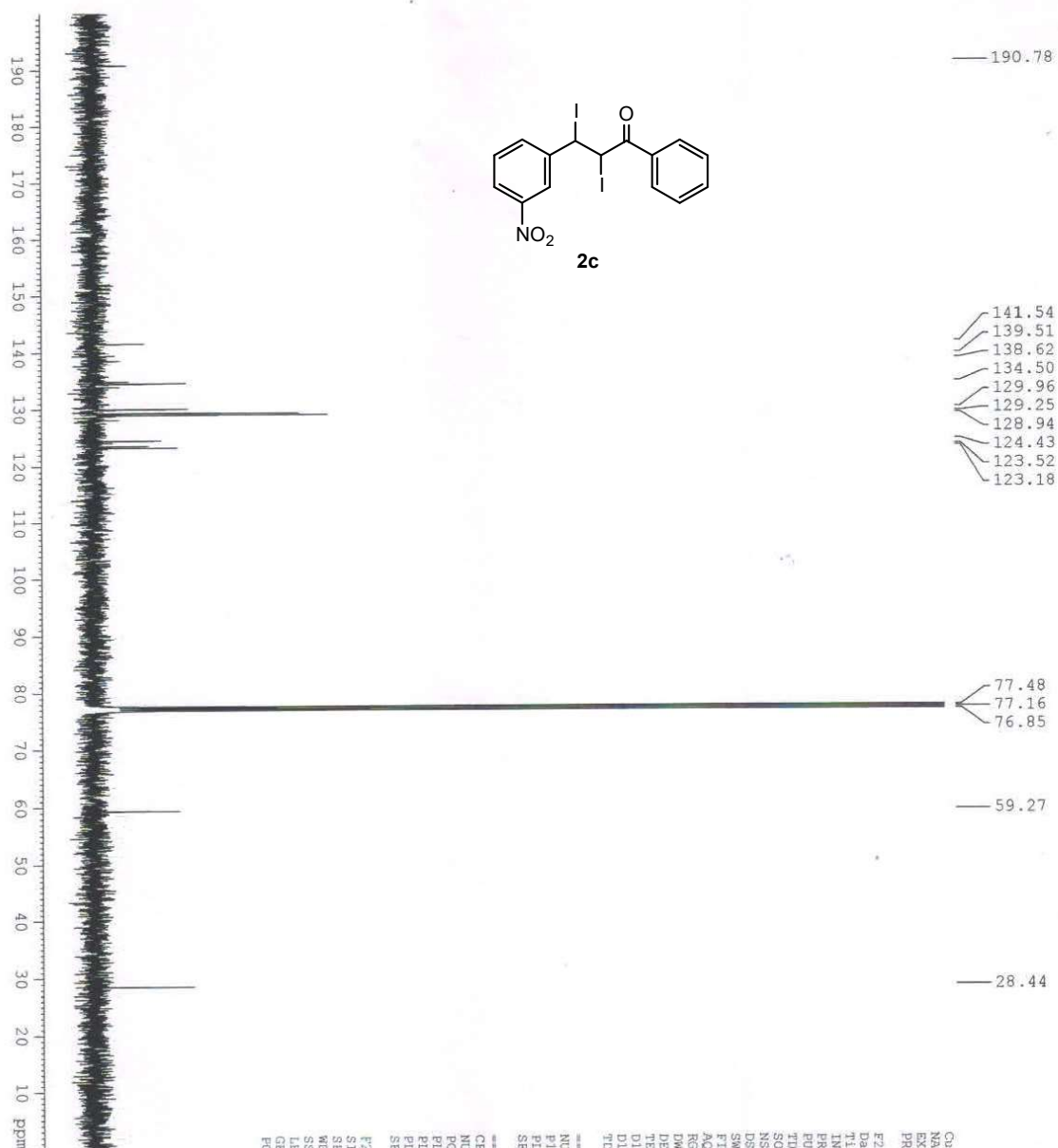
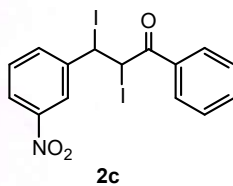
DW 60.800 usec
 DE 6.50 usec
 TE 296.7 K
 D1 1.00000000 sec
 TD0 1

===== CHANNEL f1 =====
 NUC1 ¹H
 P1 14.75 usec
 PLM1 11.99499989 W
 SFO1 400.1524711 MHz

F2 - Processing parameters
 SI 16384
 SF 400.1500000 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00



13C OF VBNC-DINO2



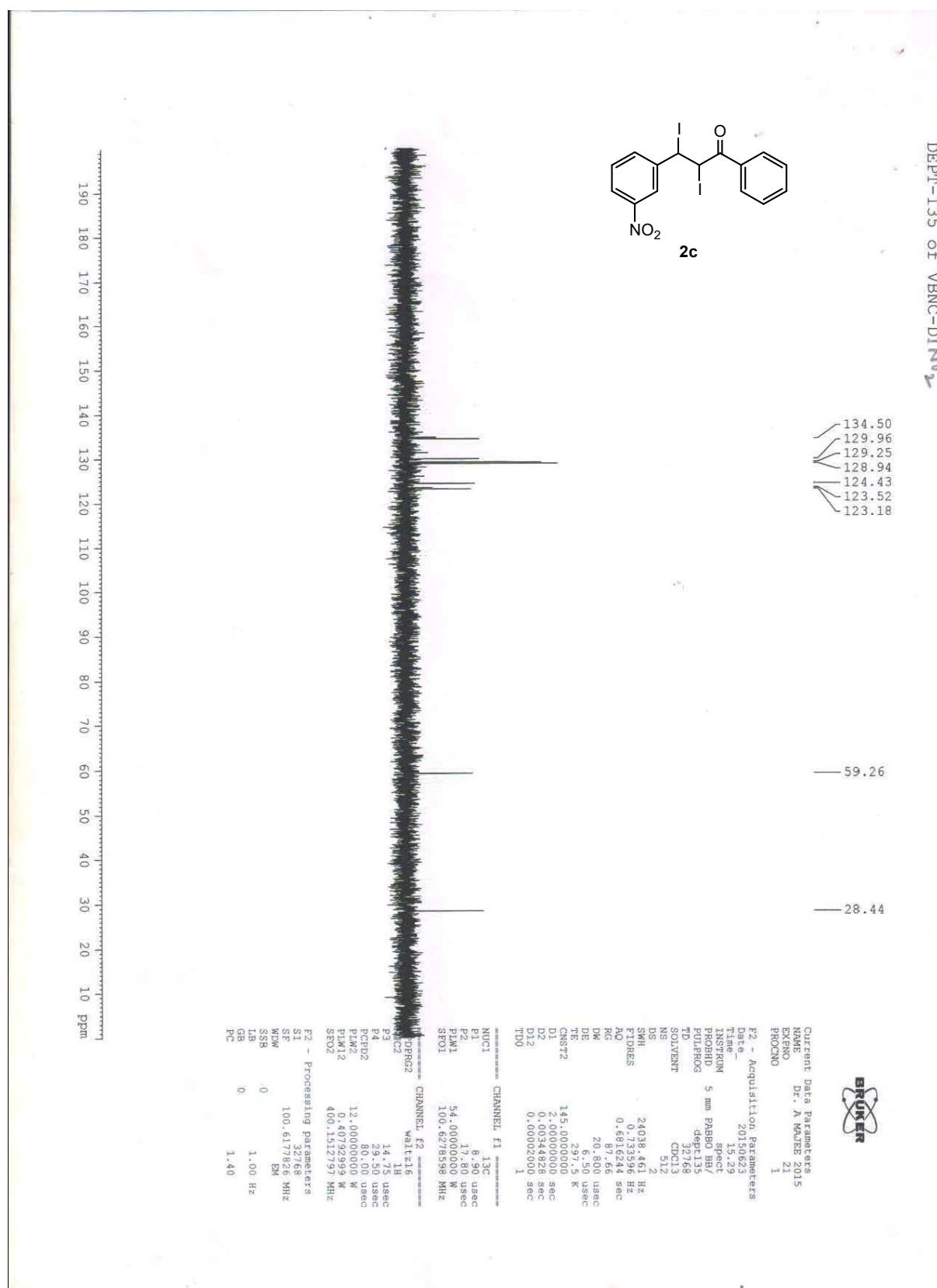
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NAME Dr. A. MATE 2015
EXPNO 20
PROCNO 1

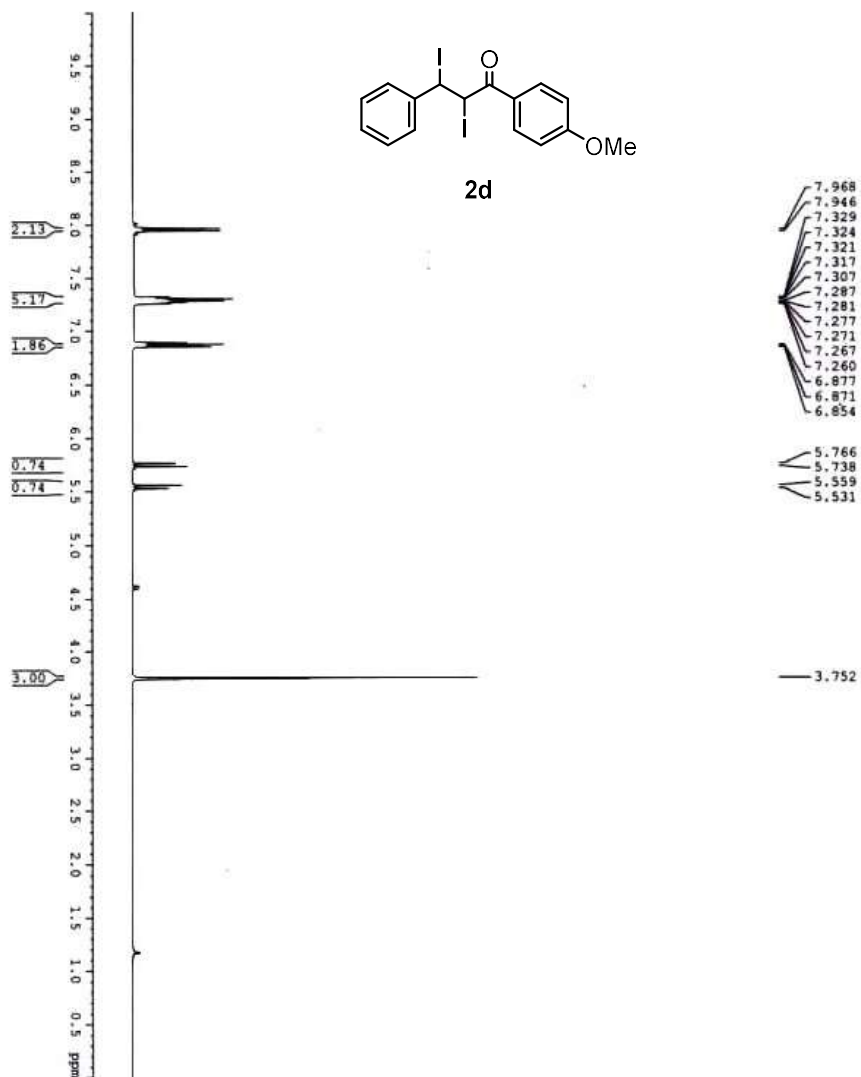
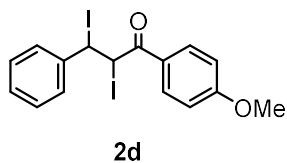
F2 - Acquisition Parameters
Date_ 20150623
Time 15.06
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 1024
DS 2
SWH 24038.461 Hz
FIDRES 0.733596 Hz
AQ 0.6816244 sec
RG 87.66
DE 20.800 usec
TE 298.1 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

CHANNEL f1
NUC1 13C
P1 8.30 usec
PL1 54.0000000 W
SFO1 100.6278386 MHz

CHANNEL f2
CPDPRG2 waltz16
MTC2 1H
PCPD2 80.00 usec
PLW2 12.0000000 W
PLW12 0.40792999 W
PLW13 0.26107001 W
SFO2 400.1516006 MHz

F2 - Processing parameters
SI 16384
SE 100.6177821 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
FC 1.40



¹H of VBSS-Chal (L)

7.968
7.946
7.329
7.324
7.321
7.317
7.307
7.287
7.281
7.277
7.271
7.267
7.260
6.877
6.871
6.854

5.766
5.738
5.559
5.531

3.752

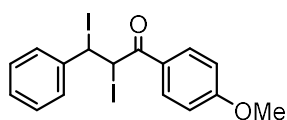
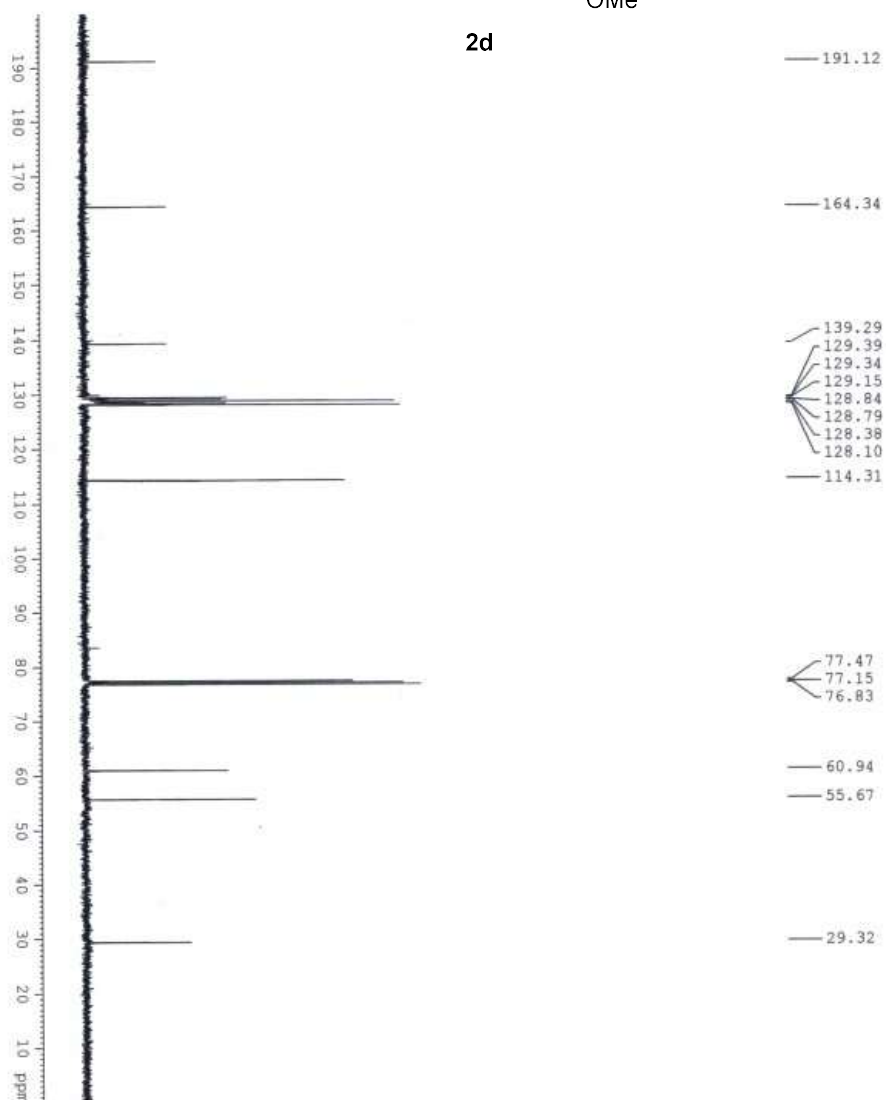


Current Data Parameters
NAME Dr. A. MATE 2016
EXPNO 280
PROCNO 1

F2 - Acquisition Parameters
Date_ 20160911
Time 18.01
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 1
SWH 8223.685 Hz
FIDRES 0.230967 Hz
AQ 1.932984 sec
RG 327.743
DM 60.800 usec
DE 6.50 usec
TE 298.0 K
D1 1.00000000 sec
TD0 1

===== CHANNEL f1 =====
SFO1 400.1524711 MHz
NUC1 13C
P1 12.00
PL1 0.00000000 W

F2 - Processing parameters
SI 16384
SF 400.1500609 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

**2d**¹³C of VBSS-Chal (L)

Current Data Parameters
NAME: DR. A. POLHE
EXPNO: 251
PROCNO: 1

F2 - Acquisition Parameters
Date_: 20160911
Time: 15.10

INSTRUM: spect
PROBHD: 5 mm PABBO 1H/

PULPROG: zgpg30
TD: 32768

SOLVENT: CDCl3
NS: 120

DS: 4
SWH: 34036.441 Hz

FIDRES: 0.131566 Hz
AQ: 0.6615744 sec

RG: 37.83
DM: 20.800 usec

DE: 6.50 usec
TE: 300.2 K

D1: 3.0000000 sec
D11: 0.03000000 sec

TD0: 1

===== CHANNEL f1 =====
SFO1: 100.627808 MHz
NUC1: 13C
P1: 1.50 usec
PL1: 54.00000000 W

===== CHANNEL f2 =====
SFO2: 400.151606 MHz
NUC2: 1H
P2: 1.50 usec
PL2: 54.00000000 W

===== CHANNEL f3 =====
SFO3: 100.627808 MHz
NUC3: 13C
P3: 1.50 usec
PL3: 54.00000000 W

===== CHANNEL f4 =====
SFO4: 100.627808 MHz
NUC4: 13C
P4: 1.50 usec
PL4: 54.00000000 W

===== CHANNEL f5 =====
SFO5: 100.627808 MHz
NUC5: 13C
P5: 1.50 usec
PL5: 54.00000000 W

===== CHANNEL f6 =====
SFO6: 100.627808 MHz
NUC6: 13C
P6: 1.50 usec
PL6: 54.00000000 W

===== CHANNEL f7 =====
SFO7: 100.627808 MHz
NUC7: 13C
P7: 1.50 usec
PL7: 54.00000000 W

===== CHANNEL f8 =====
SFO8: 100.627808 MHz
NUC8: 13C
P8: 1.50 usec
PL8: 54.00000000 W

===== CHANNEL f9 =====
SFO9: 100.627808 MHz
NUC9: 13C
P9: 1.50 usec
PL9: 54.00000000 W

===== CHANNEL f10 =====
SFO10: 100.627808 MHz
NUC10: 13C
P10: 1.50 usec
PL10: 54.00000000 W

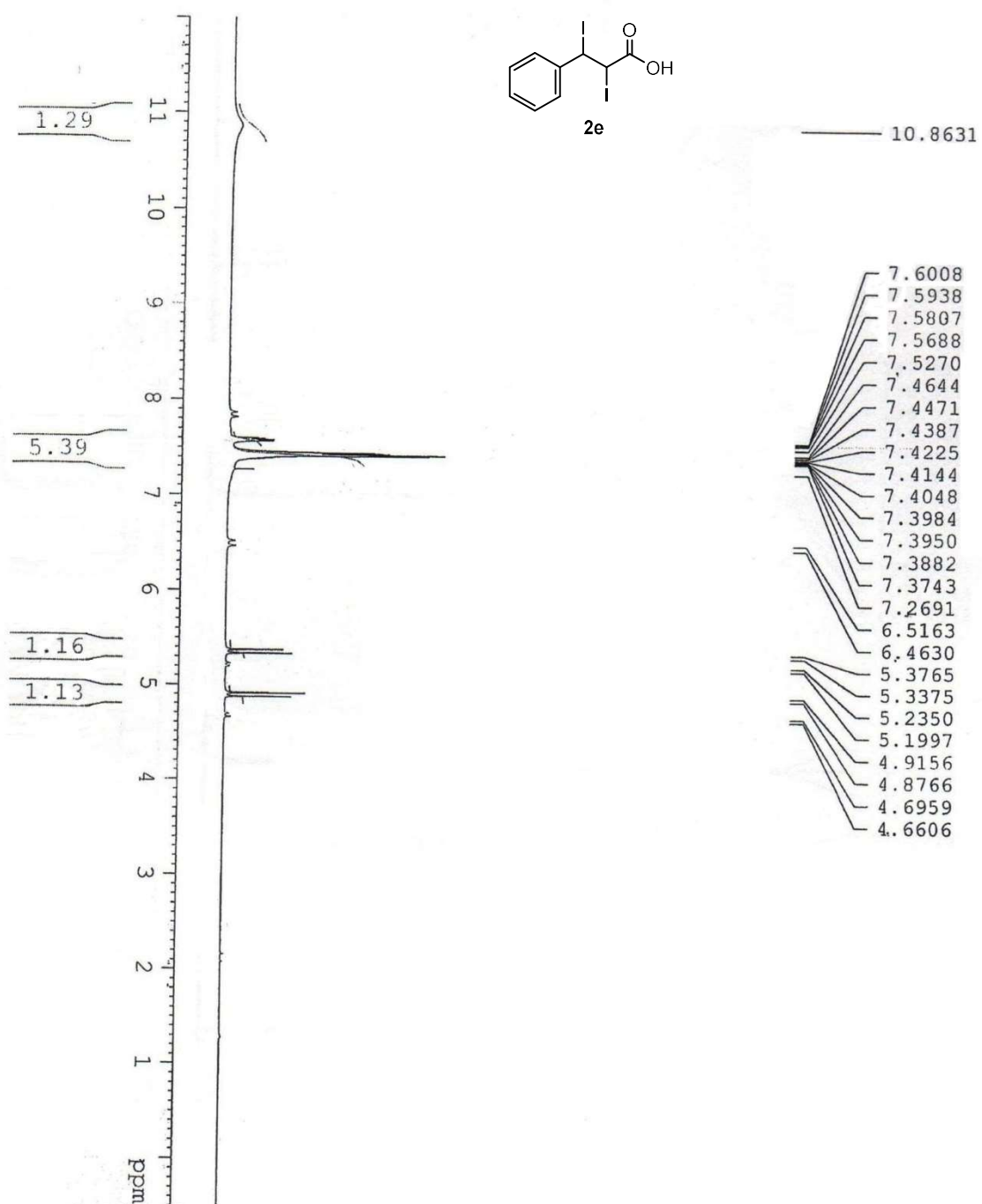
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SFO11: 100.627808 MHz
NUC11: 13C
P11: 1.50 usec
PL11: 54.00000000 W

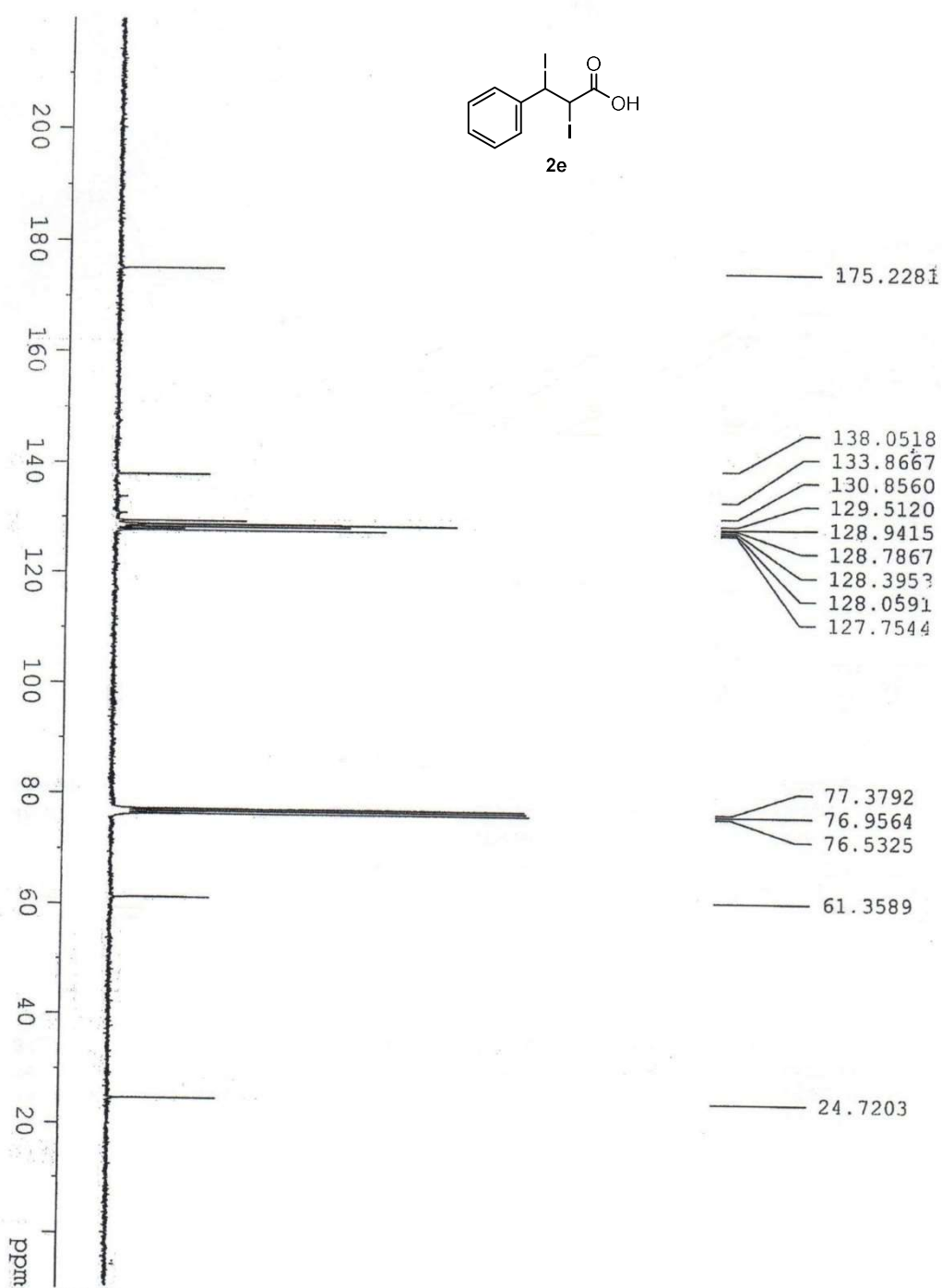
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SFO12: 100.627808 MHz
NUC12: 13C
P12: 1.50 usec
PL12: 54.00000000 W

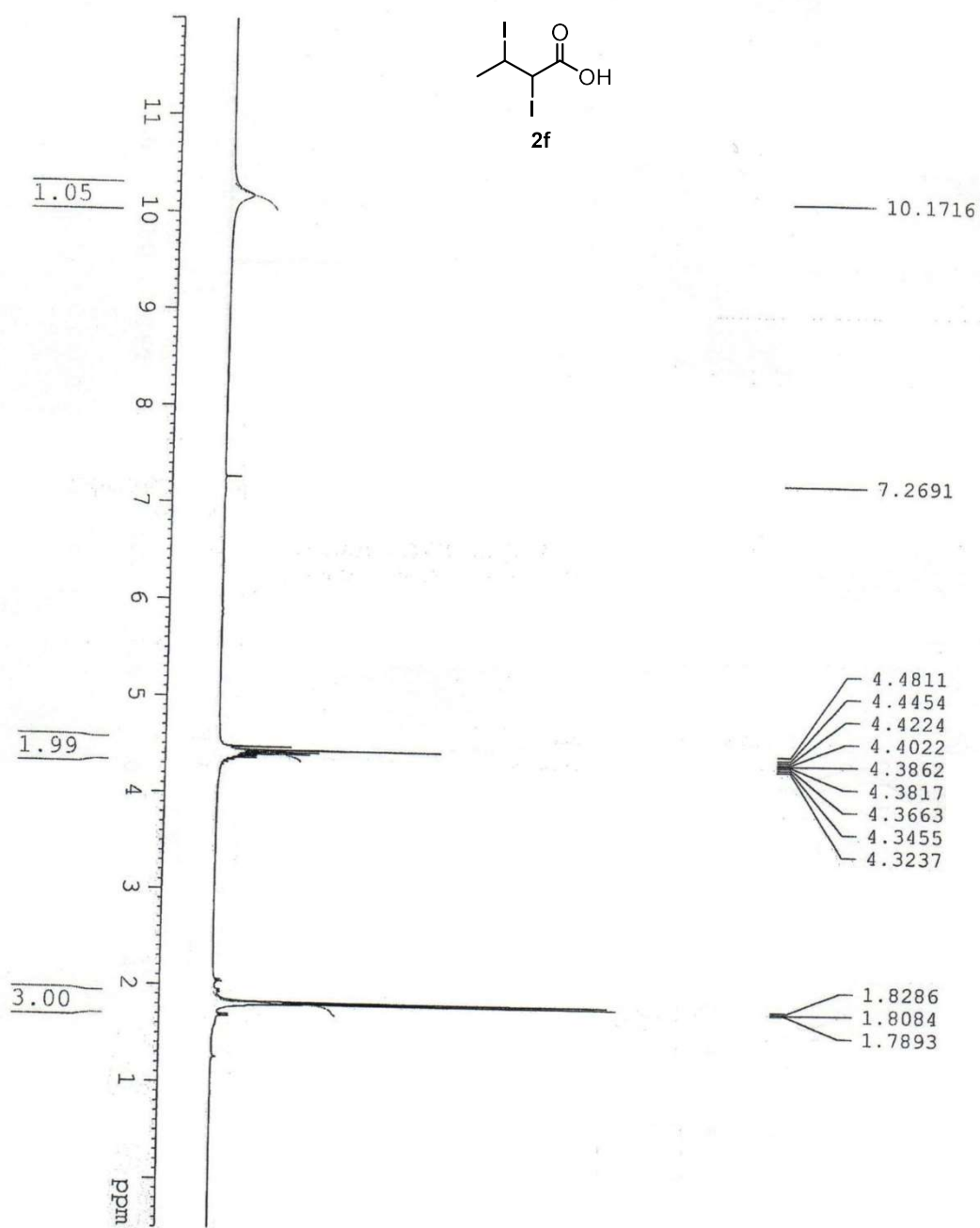
===== CHANNEL f13 =====
SFO13: 100.627808 MHz
NUC13: 13C
P13: 1.50 usec
PL13: 54.00000000 W

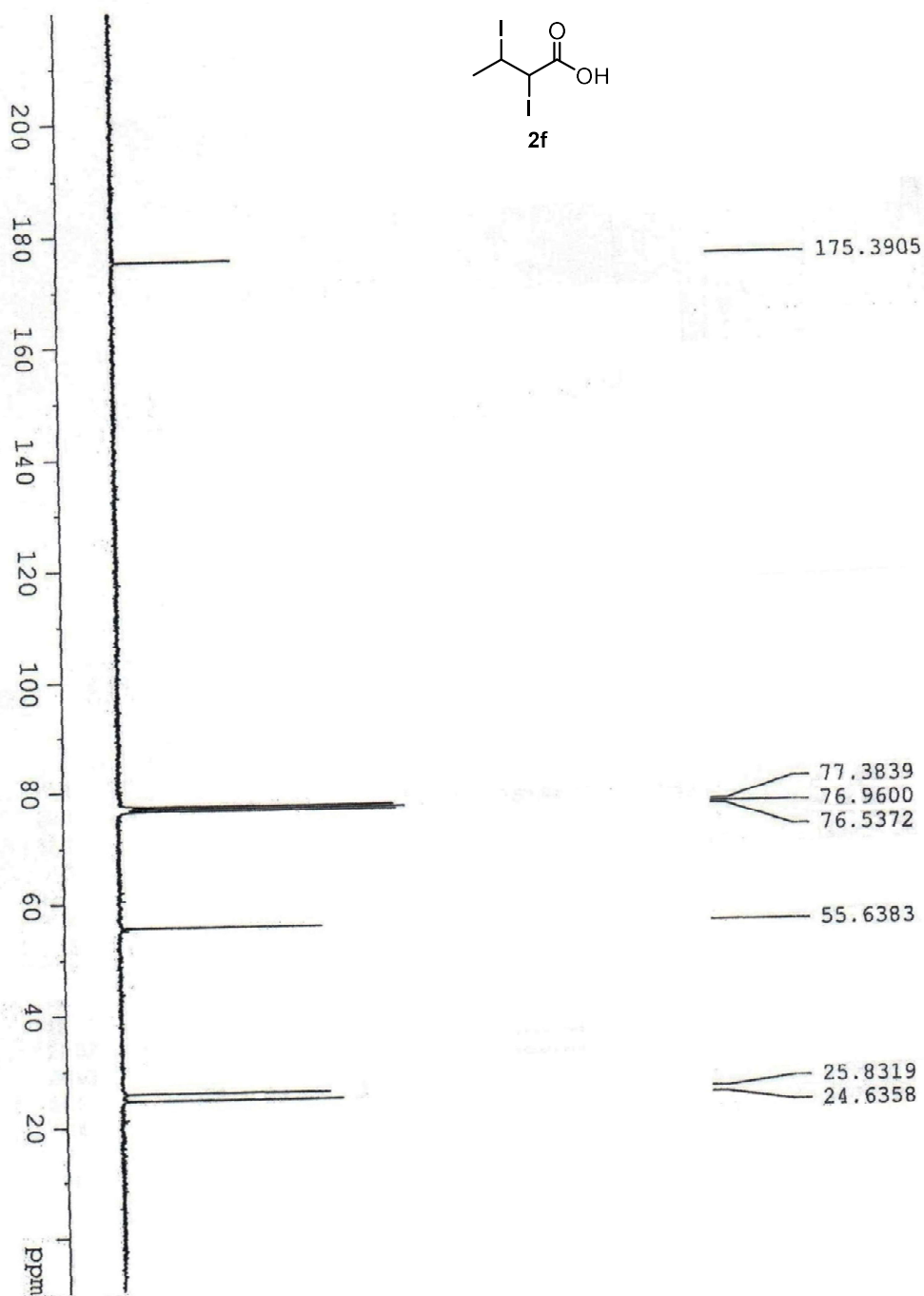
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SFO14: 100.627808 MHz
NUC14: 13C
P14: 1.50 usec
PL14: 54.00000000 W

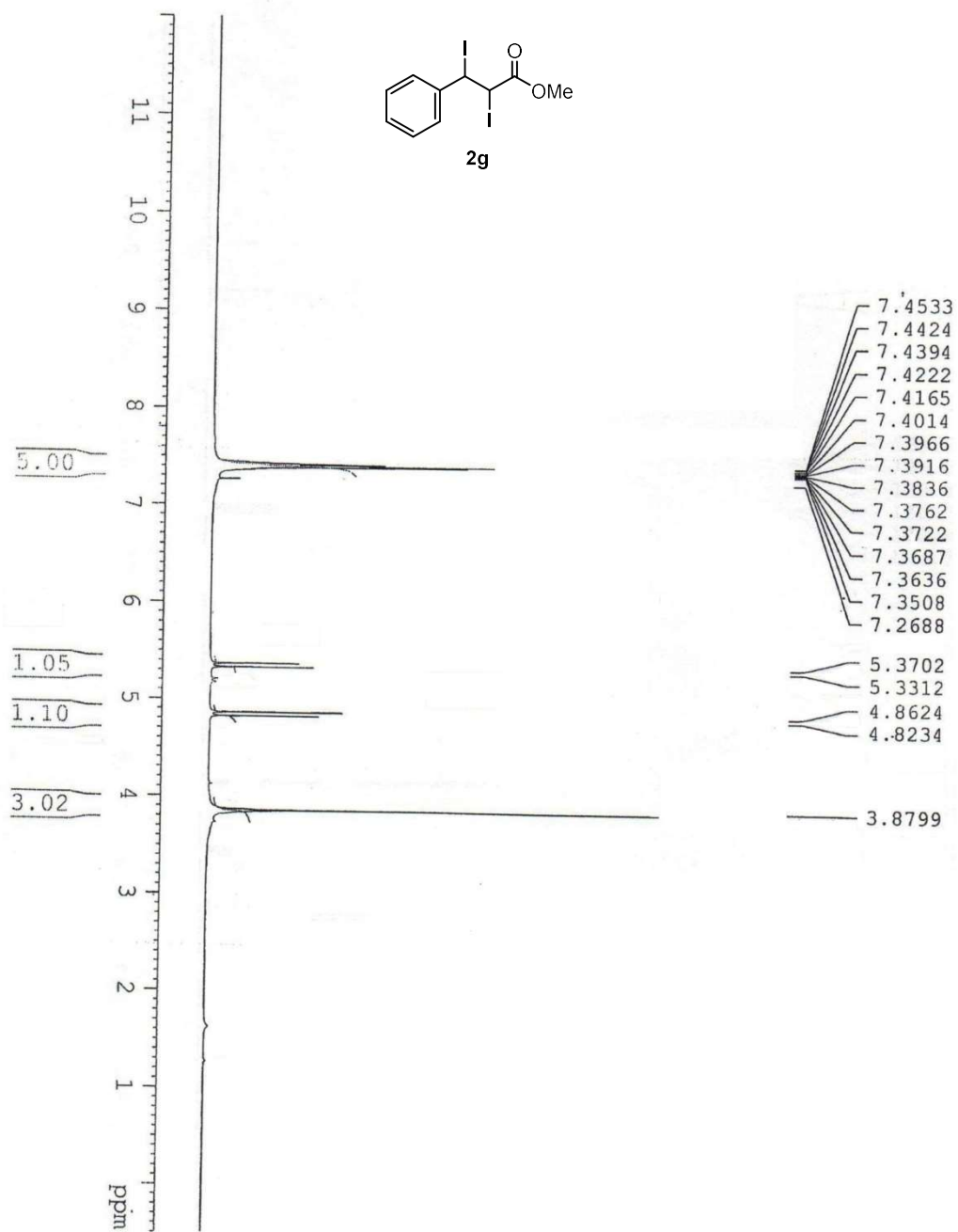
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NUC15: 13C
P15: 1.50 usec
PL15: 54.00000000 W

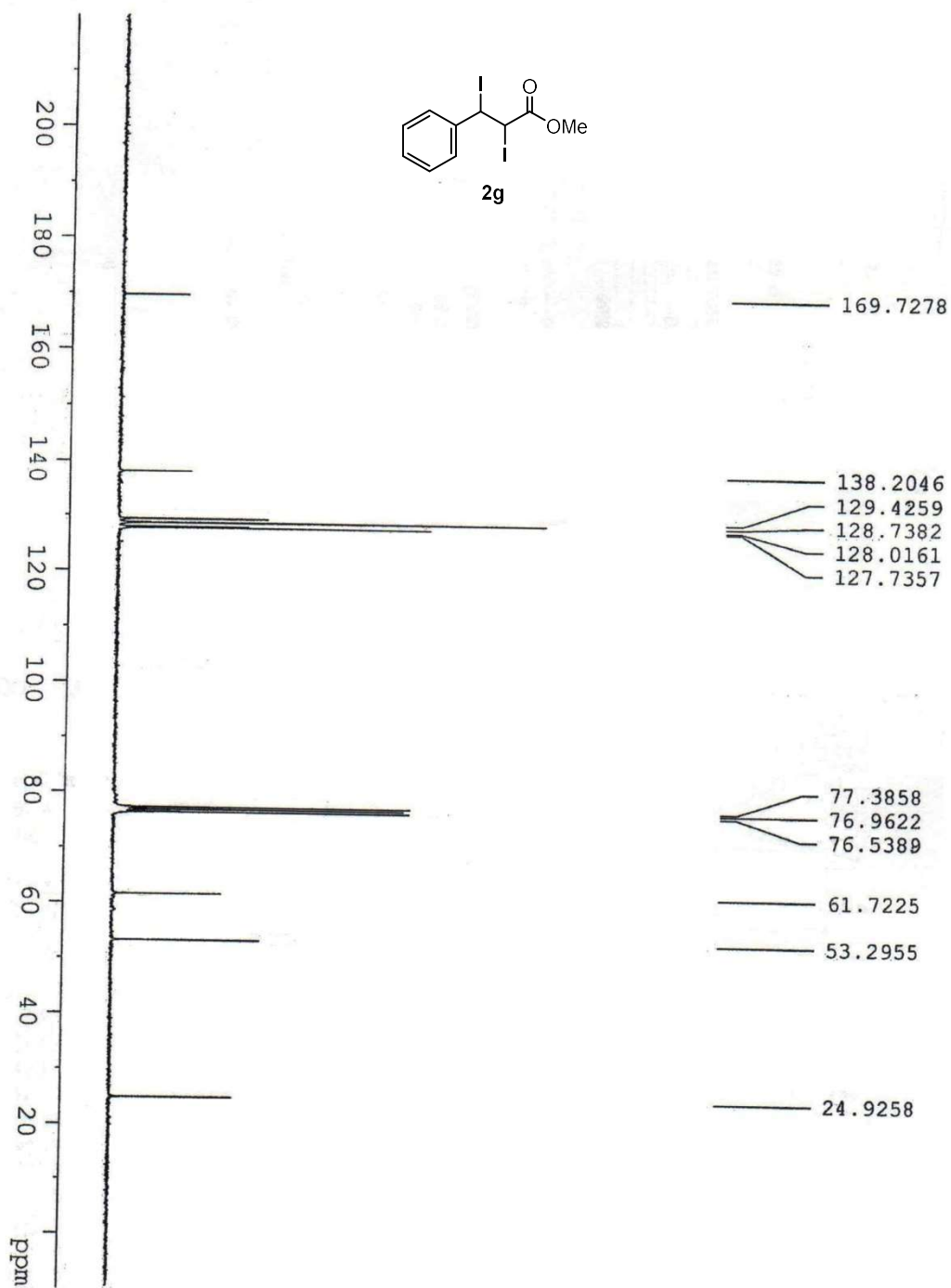


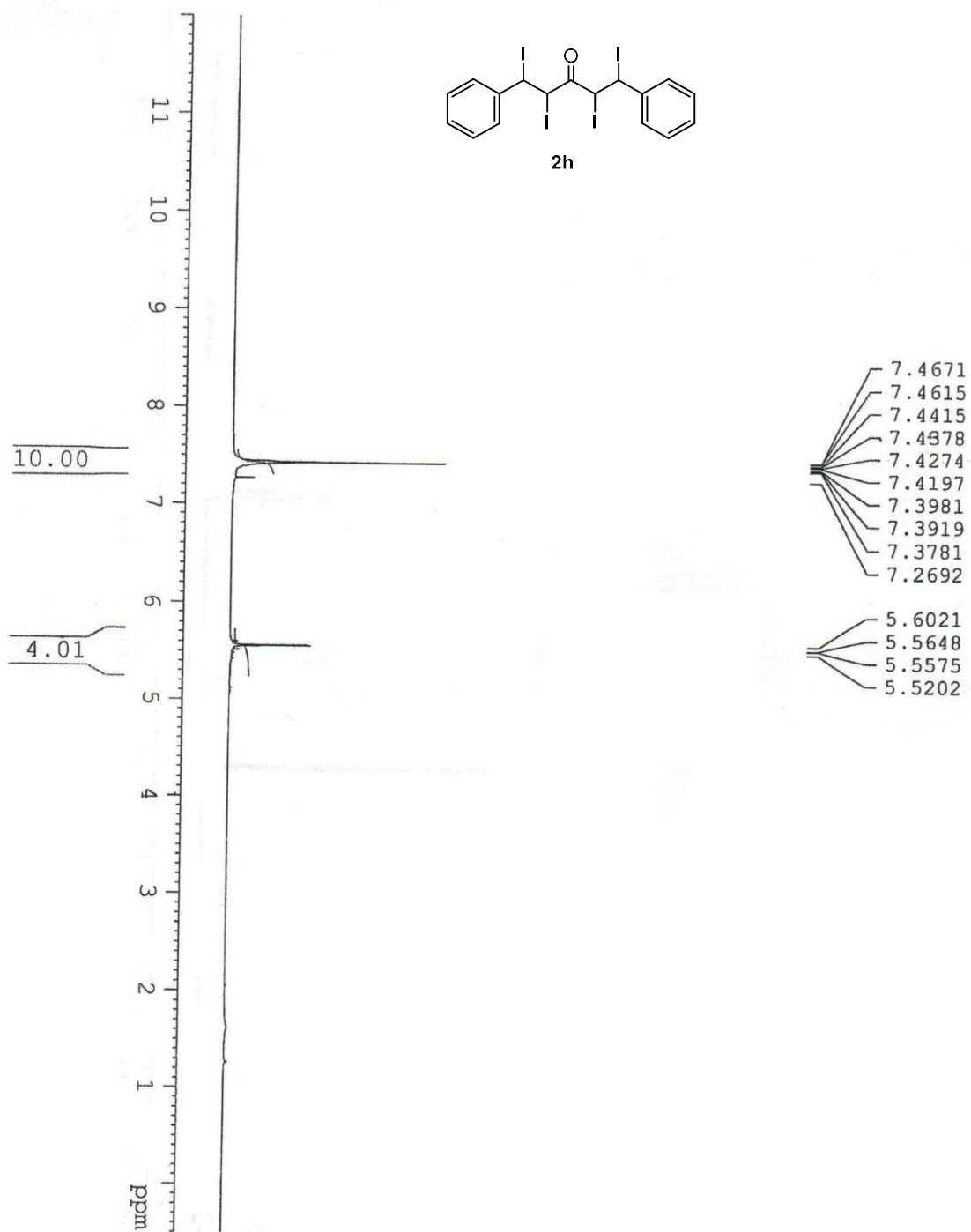


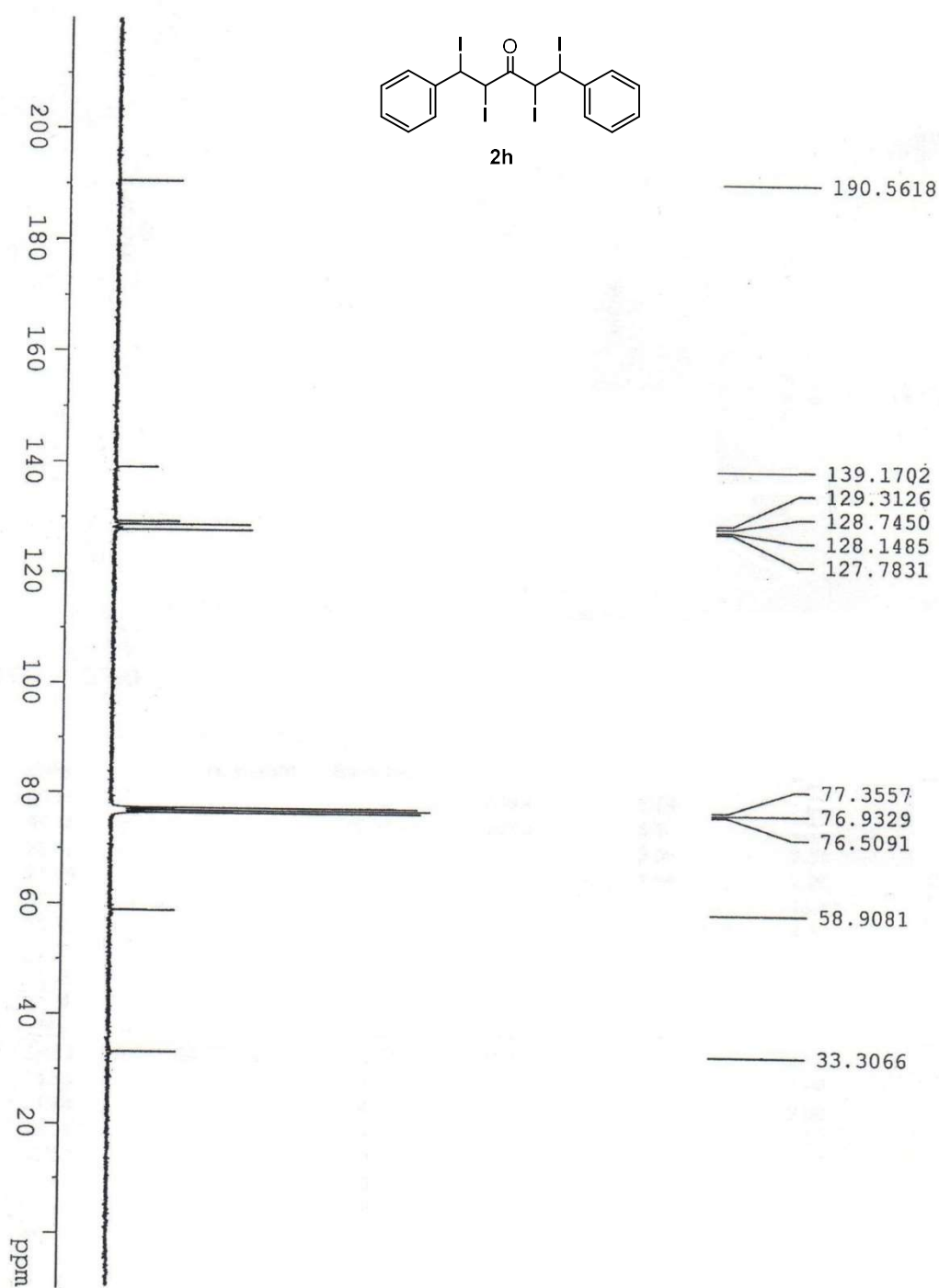


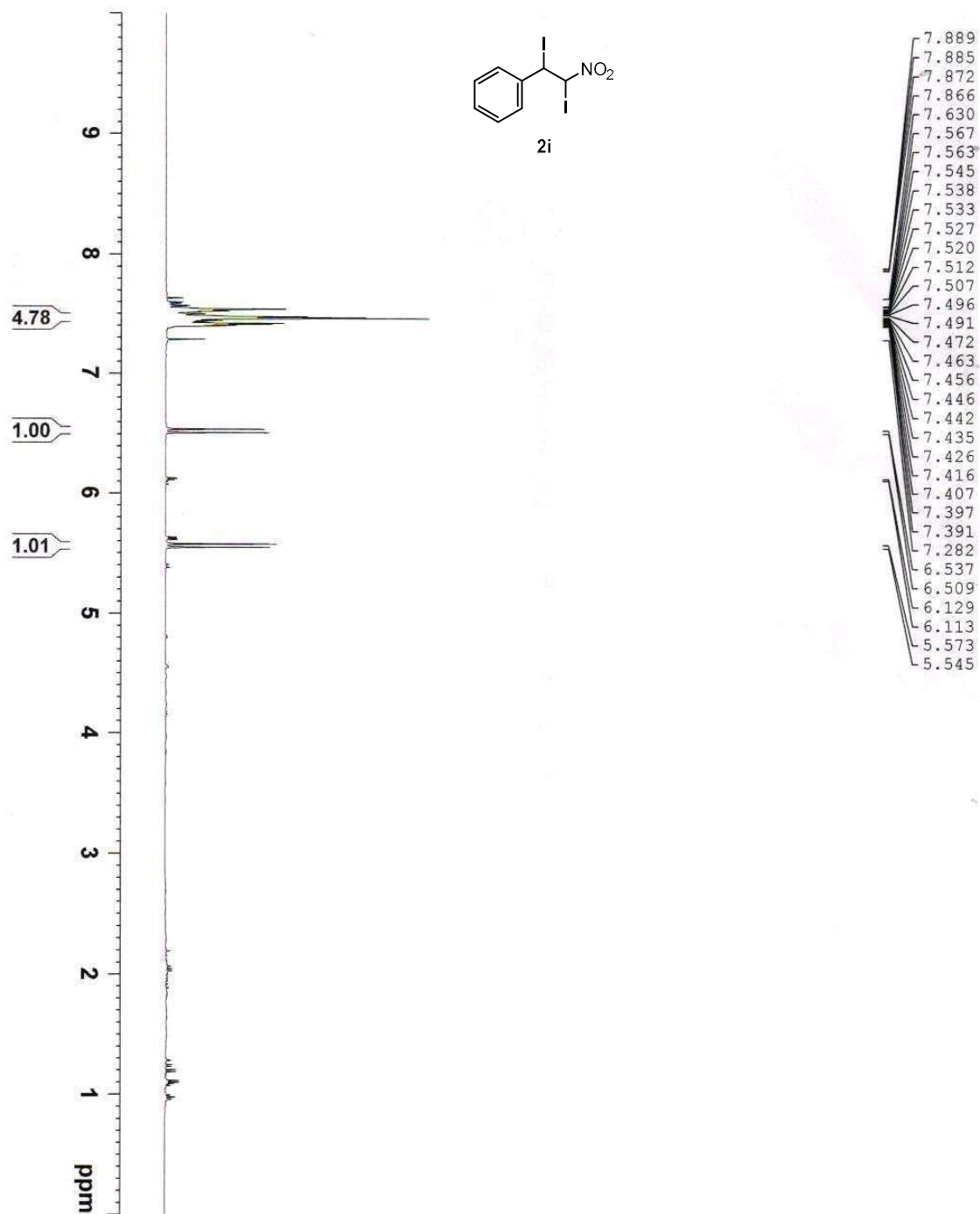


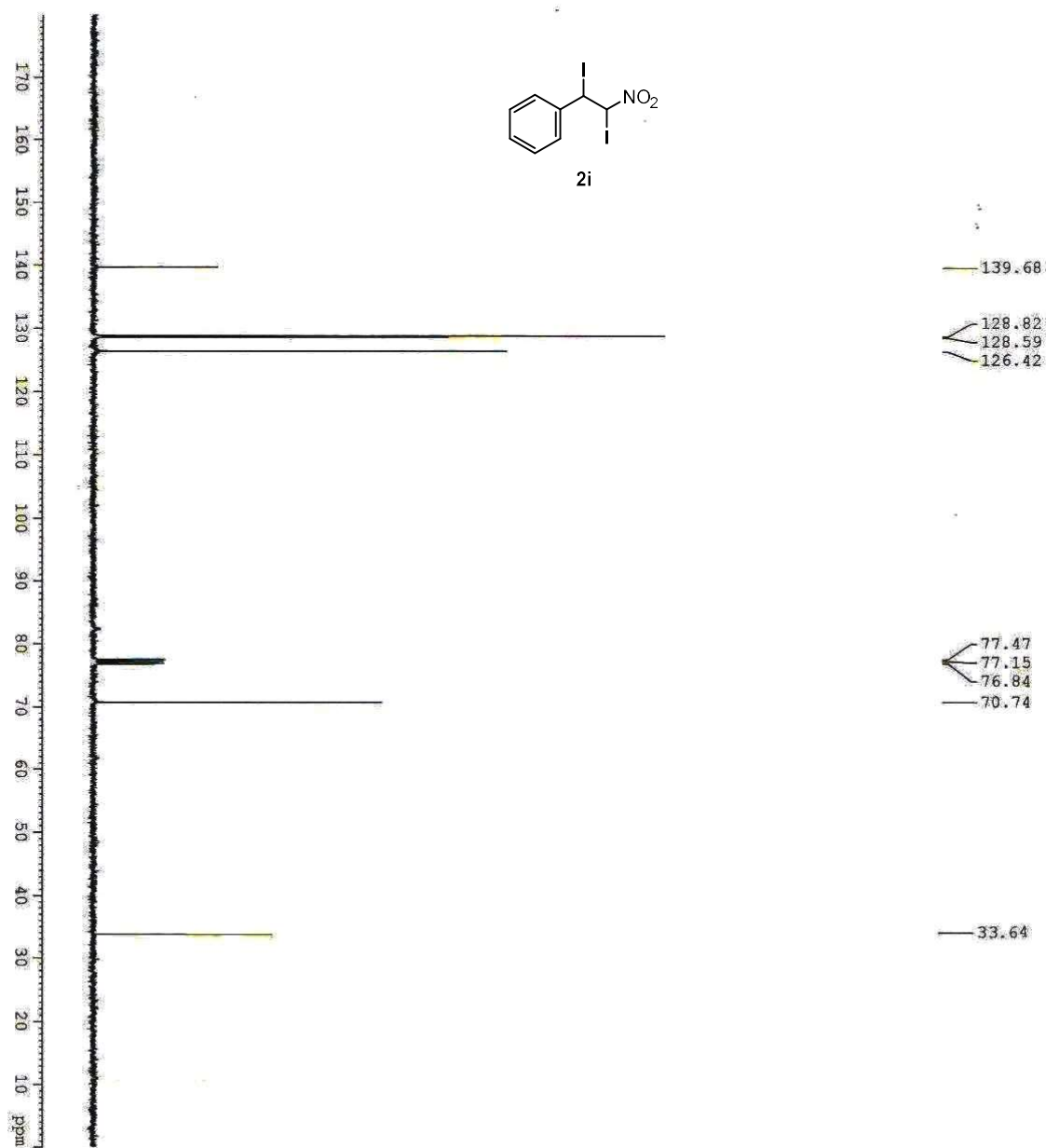


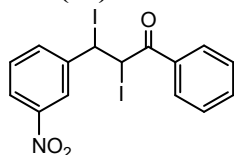










Structure Determination of compound 2,3-diiodo-3-(3-nitro-phenyl)-1-phenyl-propan-1-one (2c):

Single crystal suitable for X-ray diffraction of 2,3-diiodo-3-(3-nitro-phenyl)-1-phenyl-propan-1-one was grown from pet ether/EtOAc.

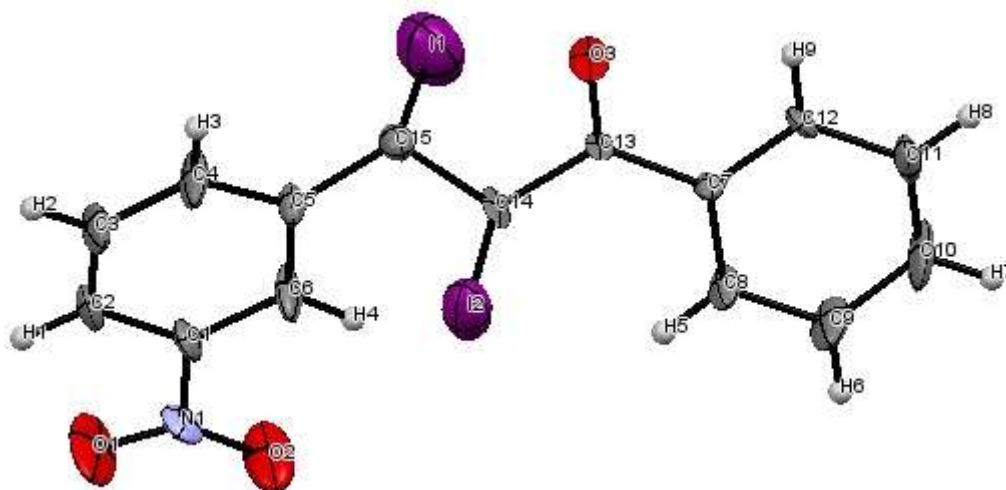
Molecular Formula: $C_{15}H_{11}I_2NO_3$; Mol. Wt.: 507.06

A specimen of $C_{15}H_{11}I_2NO_3$ was used for the X-ray crystallographic analysis. The X-ray intensity data were measured.

The total exposure time was 5.55 hours. The frames were integrated with the Bruker SAINT Software package using a narrow-frame algorithm. The integration of the data using a triclinic unit cell yielded a total of 25807 reflections to a maximum θ angle of 30.04° (0.71 \AA resolution), of which 4207 were independent (average redundancy 6.134, completeness = 94.5%, $R_{\text{int}} = 20.19\%$, $R_{\text{sig}} = 8.23\%$) and 3405 (80.94%) were greater than $2\sigma(F^2)$. The final cell constants of $a = 9.1267(9) \text{ \AA}$, $b = 9.2452(10) \text{ \AA}$, $c = 10.5181(10) \text{ \AA}$, $\alpha = 89.390(5)^\circ$, $\beta = 68.455(4)^\circ$, $\gamma = 68.534(4)^\circ$, volume = $760.34(14) \text{ \AA}^3$, are based upon the refinement of the XYZ-centroids of 9860 reflections above $20 \sigma(I)$ with $4.783^\circ < 2\theta < 59.09^\circ$. Data were corrected for absorption effects using the multi-scan method (SADABS).

The final anisotropic full-matrix least-squares refinement on F^2 with 190 variables converged at $R1 = 16.53\%$, for the observed data and $wR2 = 48.10\%$ for all data. The goodness-of-fit was 3.053. The largest peak in the final difference electron density synthesis was $6.298 \text{ e}^-/\text{\AA}^3$ and the largest hole was $-4.469 \text{ e}^-/\text{\AA}^3$ with an RMS deviation of $0.538 \text{ e}^-/\text{\AA}^3$. On the basis of the final model, the calculated density was 2.969 g/cm^3 and $F(000)$, 600 e^- .

Chemical formula	C ₁₅ H ₁₁ I ₂ NO ₃
Formula weight	169.93 g/mol
Temperature	296(2) K
Wavelength	0.71073 Å
Crystal system	triclinic
Space group	P -1
Unit cell dimensions	a = 9.1267(9) Å α = 89.390(5)° b = 9.2452(10) Å β = 68.455(4)° c = 10.5181(10) Å γ = 68.534(4)°
Volume	760.34(14) Å ³
Z	8
Density (calculated)	2.969 g/cm ³
Absorption coefficient	8.201 mm ⁻¹
F(000)	600



ORTEP diagram for the structure of 2,3-diiodo-3-(3-nitro-phenyl)-1-phenyl-propan-1-one (**2c**)