

## Supplementary Material

### Combination of $\text{NH}_2\text{OH}\cdot\text{HCl}$ and $\text{NaIO}_4$ : a new and mild reagent for the synthesis of vicinal diiodo carbonyl compounds

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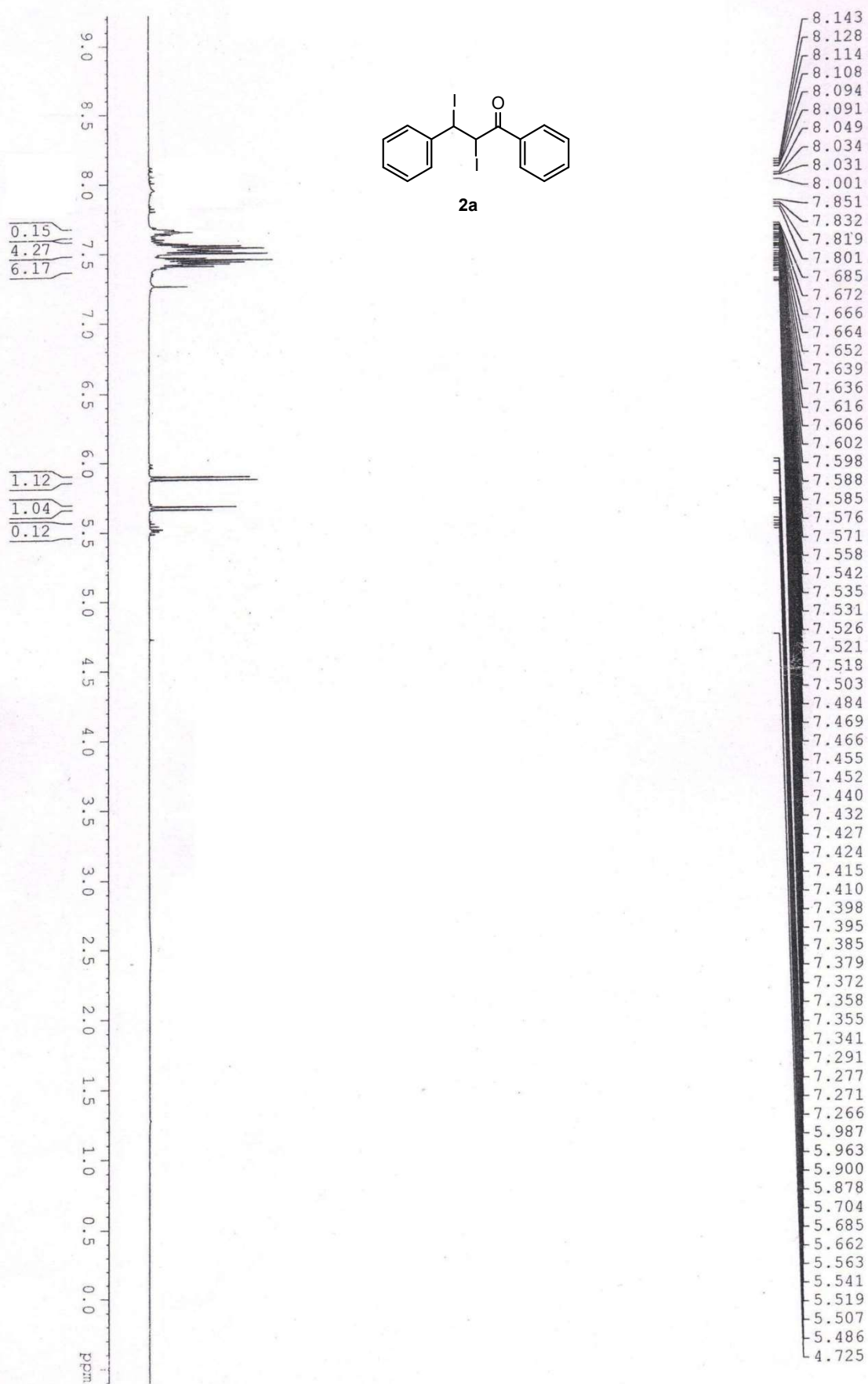
<sup>b</sup>Jhargram Raj College, Jhargram, West Midnapore 721 507, West Bengal, India

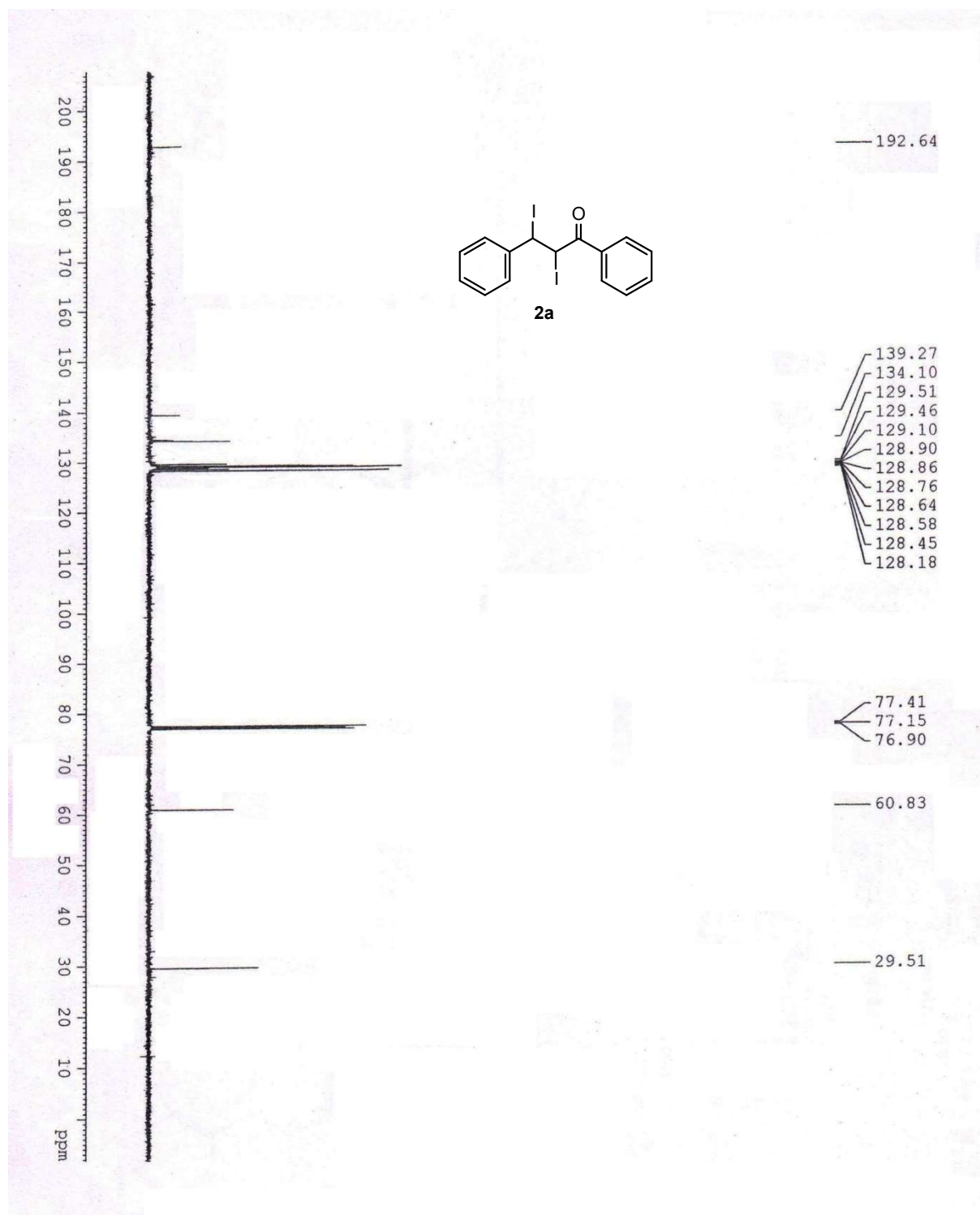
<sup>c</sup>Department of Chemistry, Visva-Bharati, Santiniketan 731 235, West Bengal, India

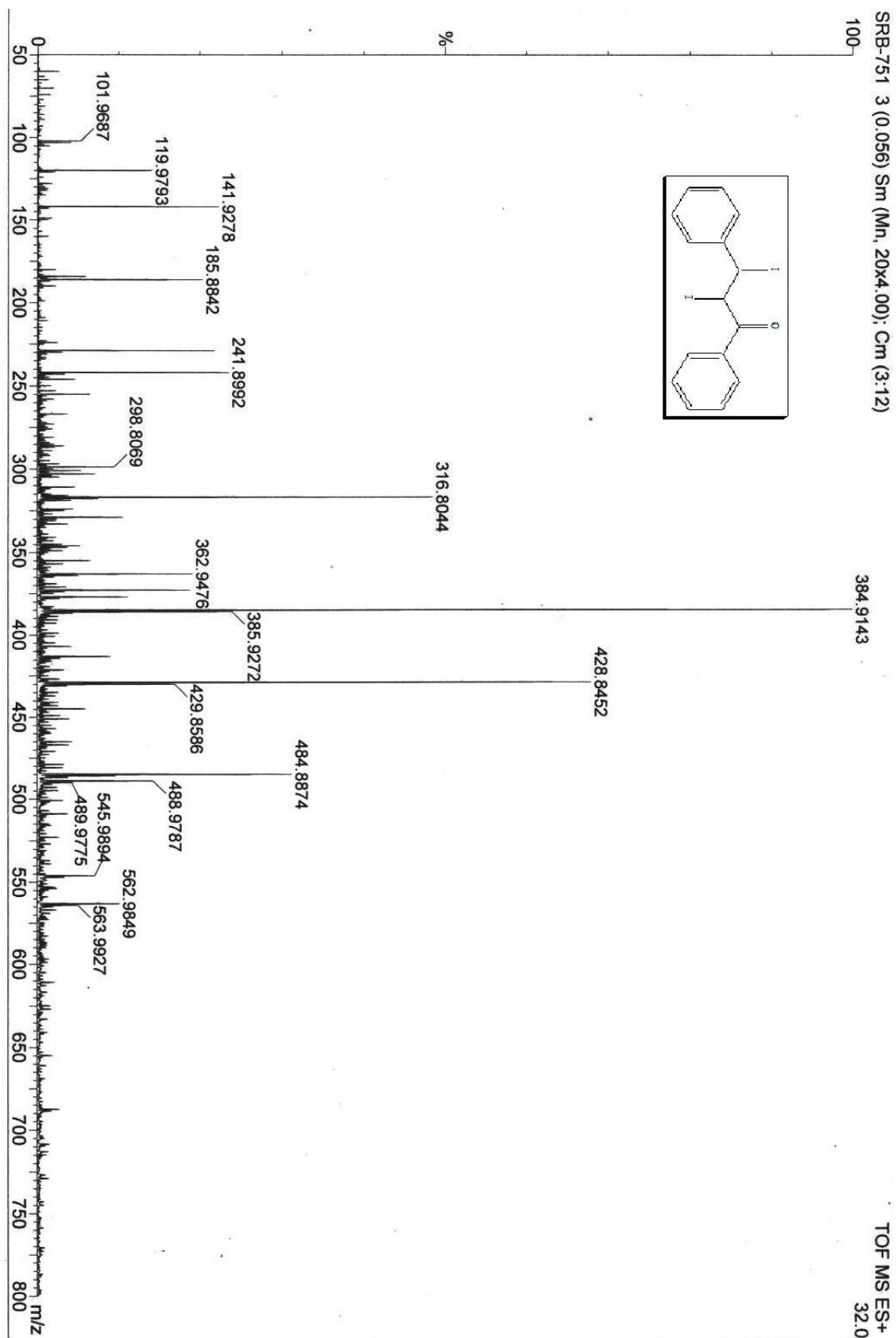
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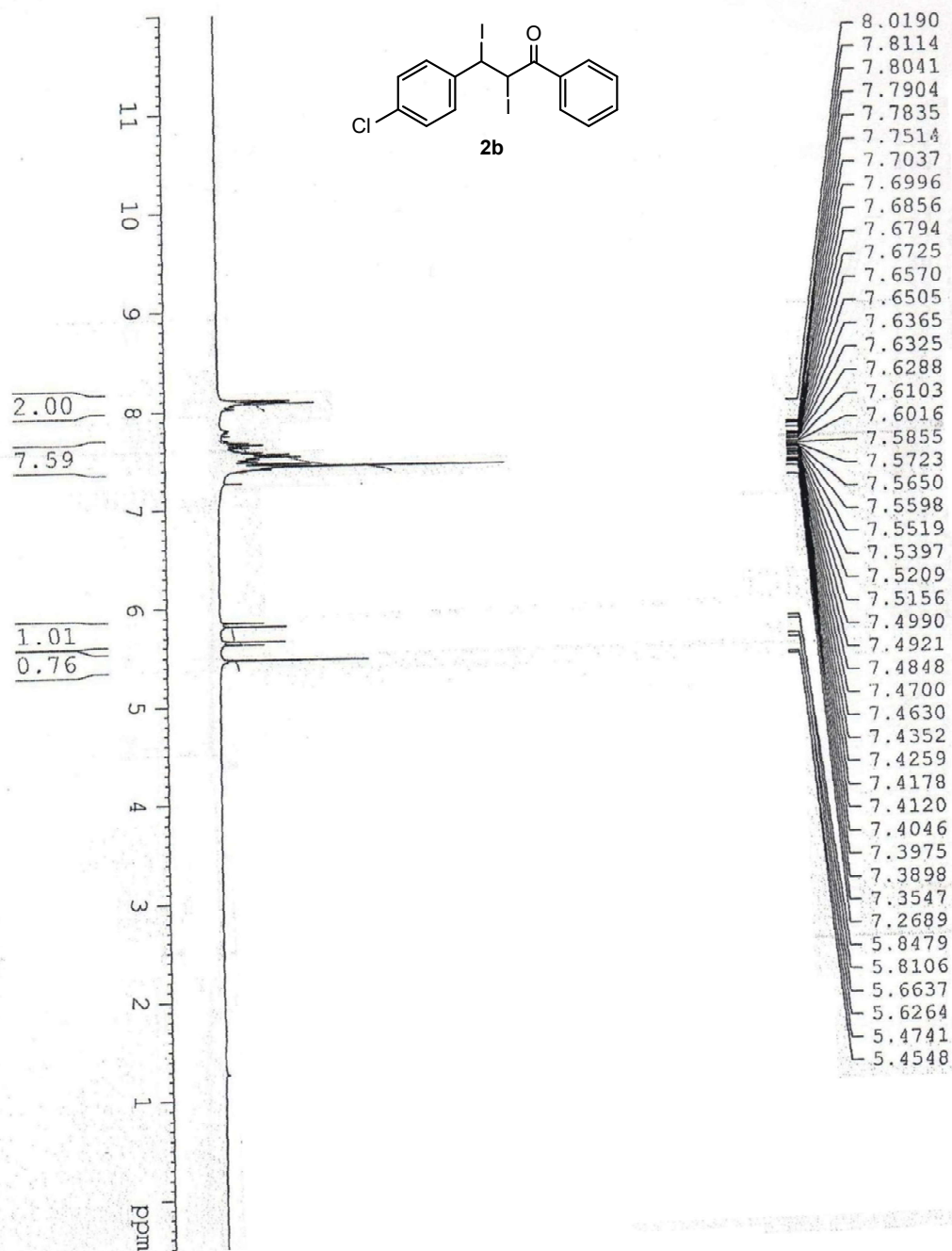
#### Contents

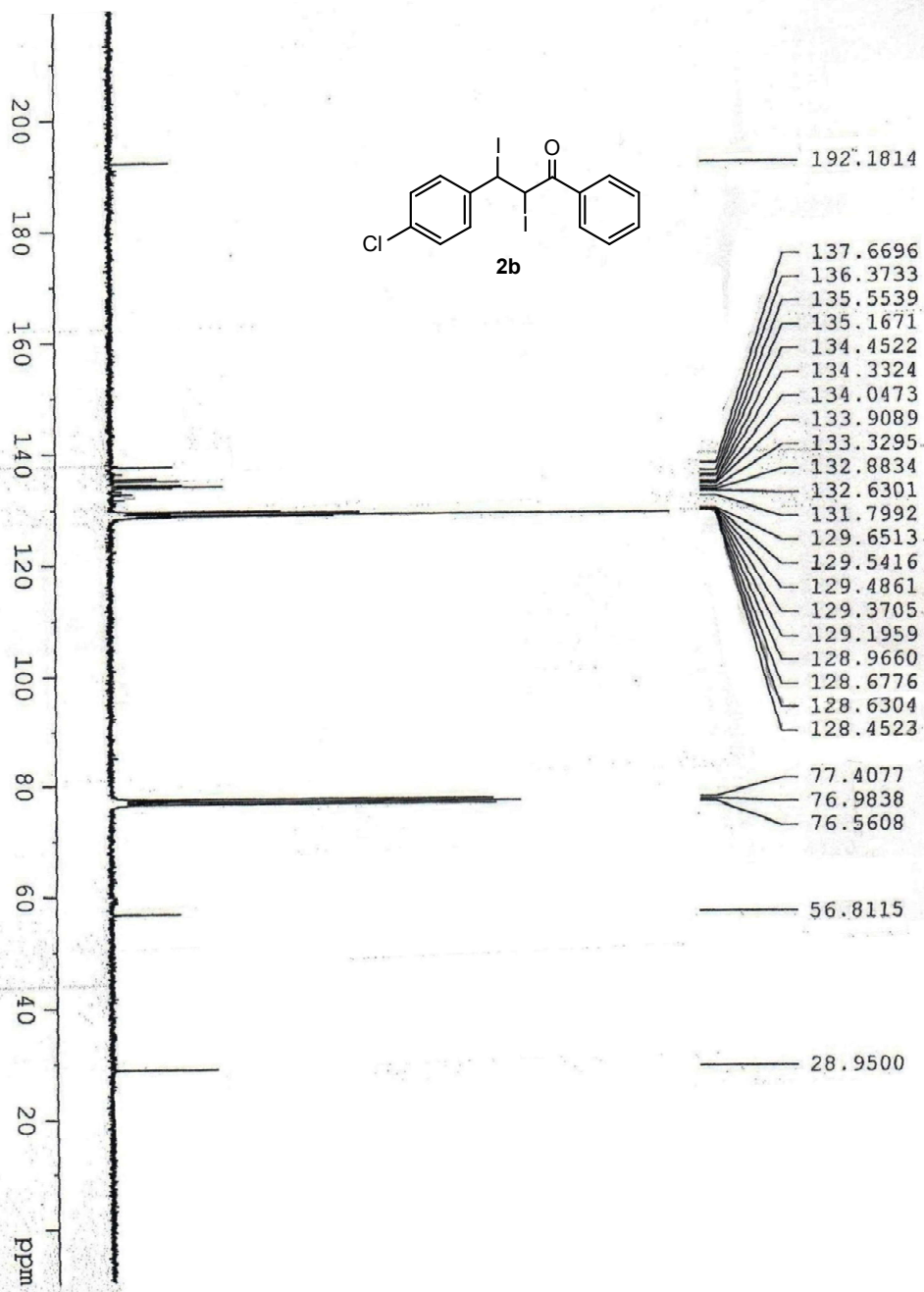
Copies of NMR spectra	.....	.....	.....	2-3 & 5-21
Mass spectra of the compound 2a			.....	4
Structure Determination	.....	.....	.....	22-23





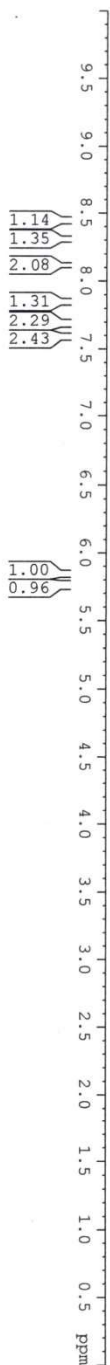
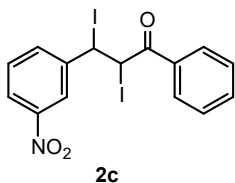






1H of VBNC-80DINO2

- 8.464
- 8.458
- 8.453
- 8.428
- 8.423
- 8.418
- 8.317
- 8.310
- 8.297
- 8.291
- 8.123
- 8.108
- 8.104
- 8.101
- 7.860
- 7.852
- 7.834
- 7.715
- 7.696
- 7.689
- 7.669
- 7.651
- 7.618
- 7.601
- 7.581
- 7.562
- 7.283
- 5.859
- 5.832
- 5.777
- 5.752



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 NAME Dr. A MAJEE 2015  
 EXPNO 19  
 PROCNO 1

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 SOLVENT CDCl3  
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 DS 2  
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 FIDRES 0.1250967 Hz  
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 TDO 1

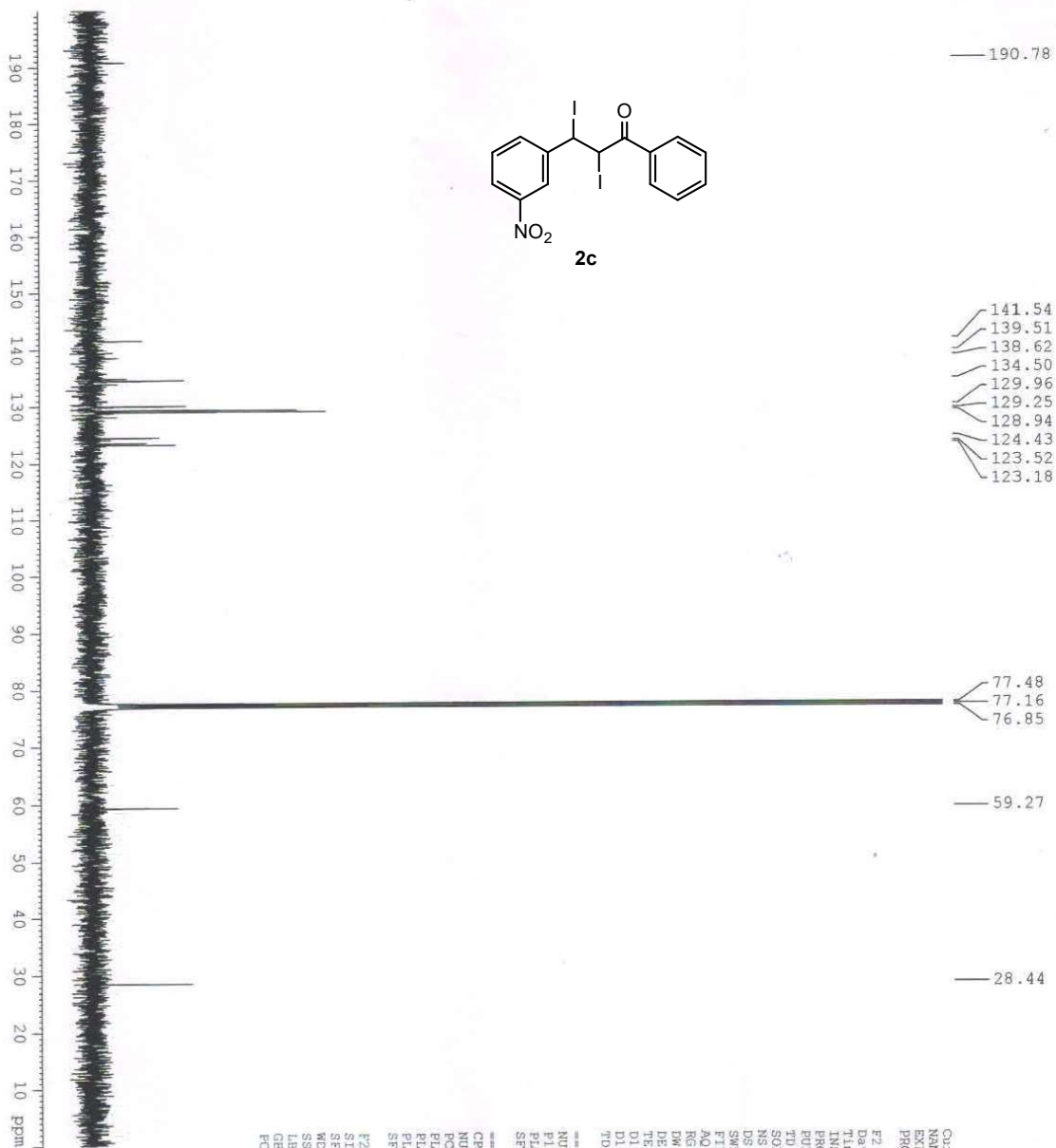
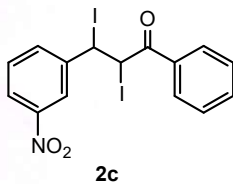
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13C OF VBNC-DINOZ



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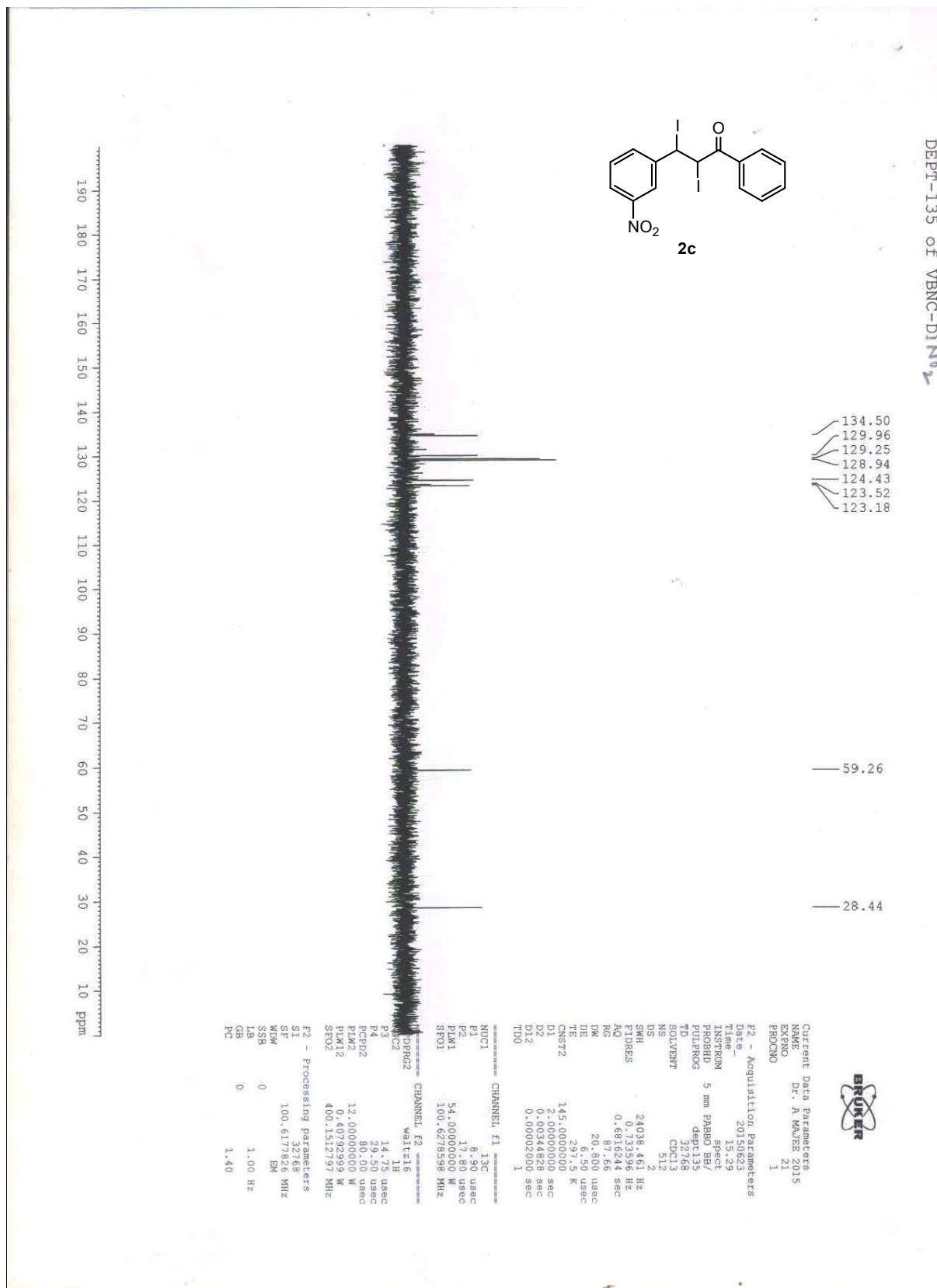
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 FIDRES 0.26107001  
 SOLVENT CDCl3  
 NS 1024  
 DS 2  
 SFO 24038.461 Hz  
 FIDRES 0.733596 Hz  
 AQ 0.6815244 sec  
 RG 87.66  
 DW 20.800 usec  
 DE 6.50 usec  
 TE 298.1 K  
 D1 2.00000000 sec  
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 TD0 1

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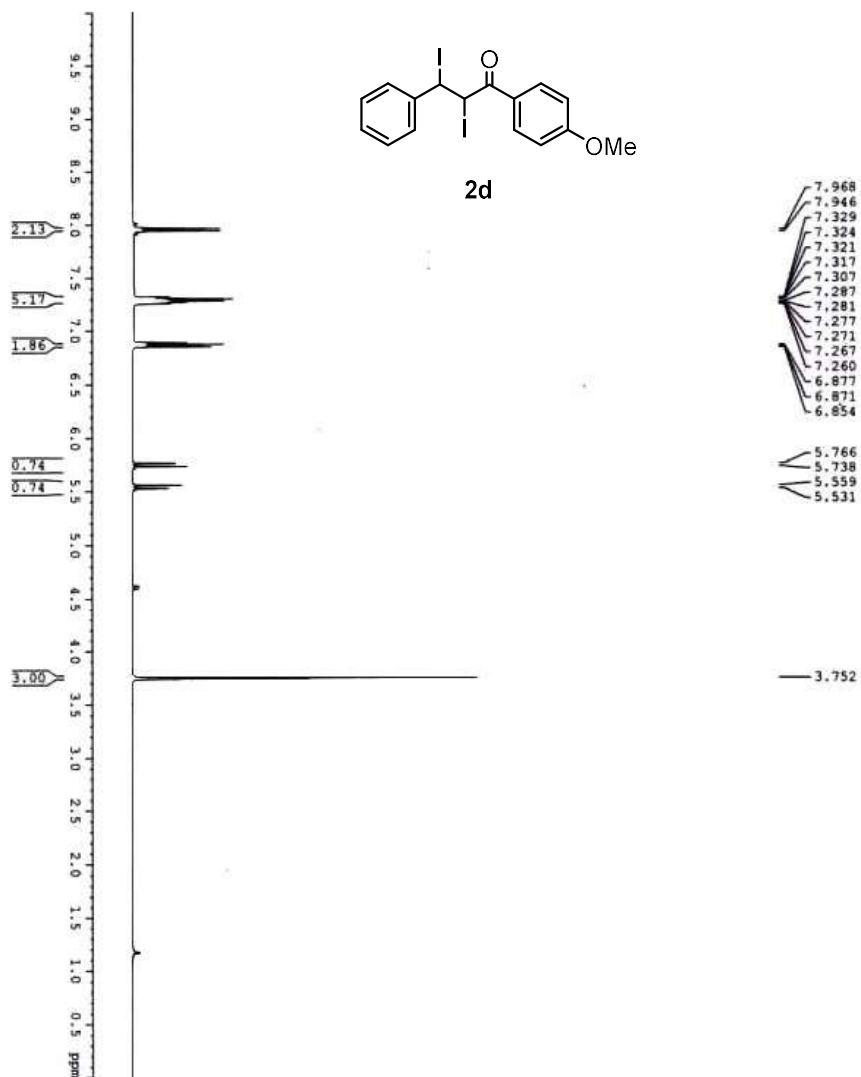
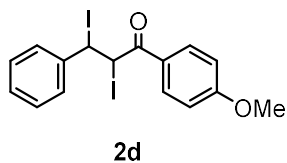
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 PLW13 0.26107001 W  
 SFO2 400.1516006 MHz

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





<sup>1</sup>H 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

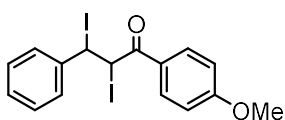


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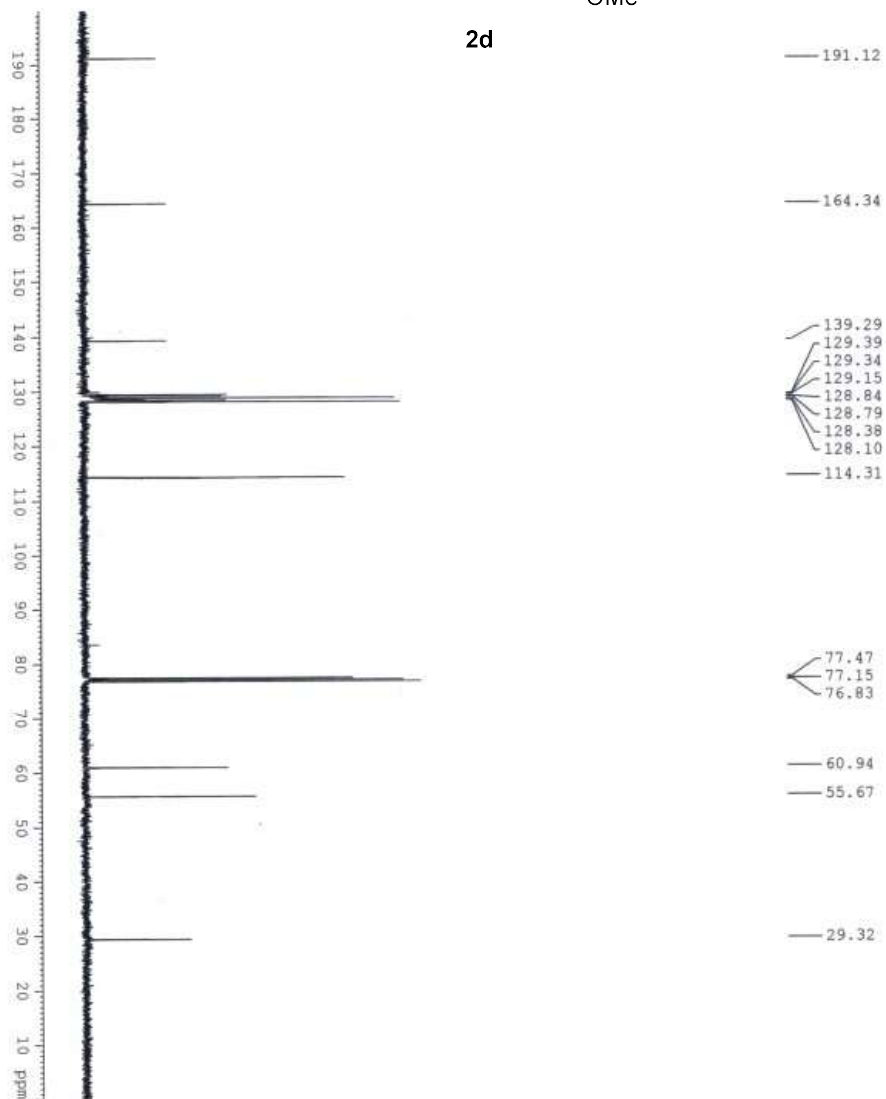
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 SFO 400.1500609  
 SOLVENT CDCl3  
 NS 16  
 DS 1  
 SMH 8223.685 Hz  
 FIDRES 0.250987 Hz  
 AQ 0.250987 sec  
 RQ 1.993743  
 DE 60.800 usec  
 TE 5.50 usec  
 TD0 298.0 K  
 D1 1.00000000 sec  
 TD0 1

CHANNEL f1  
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 P P0C1 14.75 usec  
 PLW1 12.00000000 W

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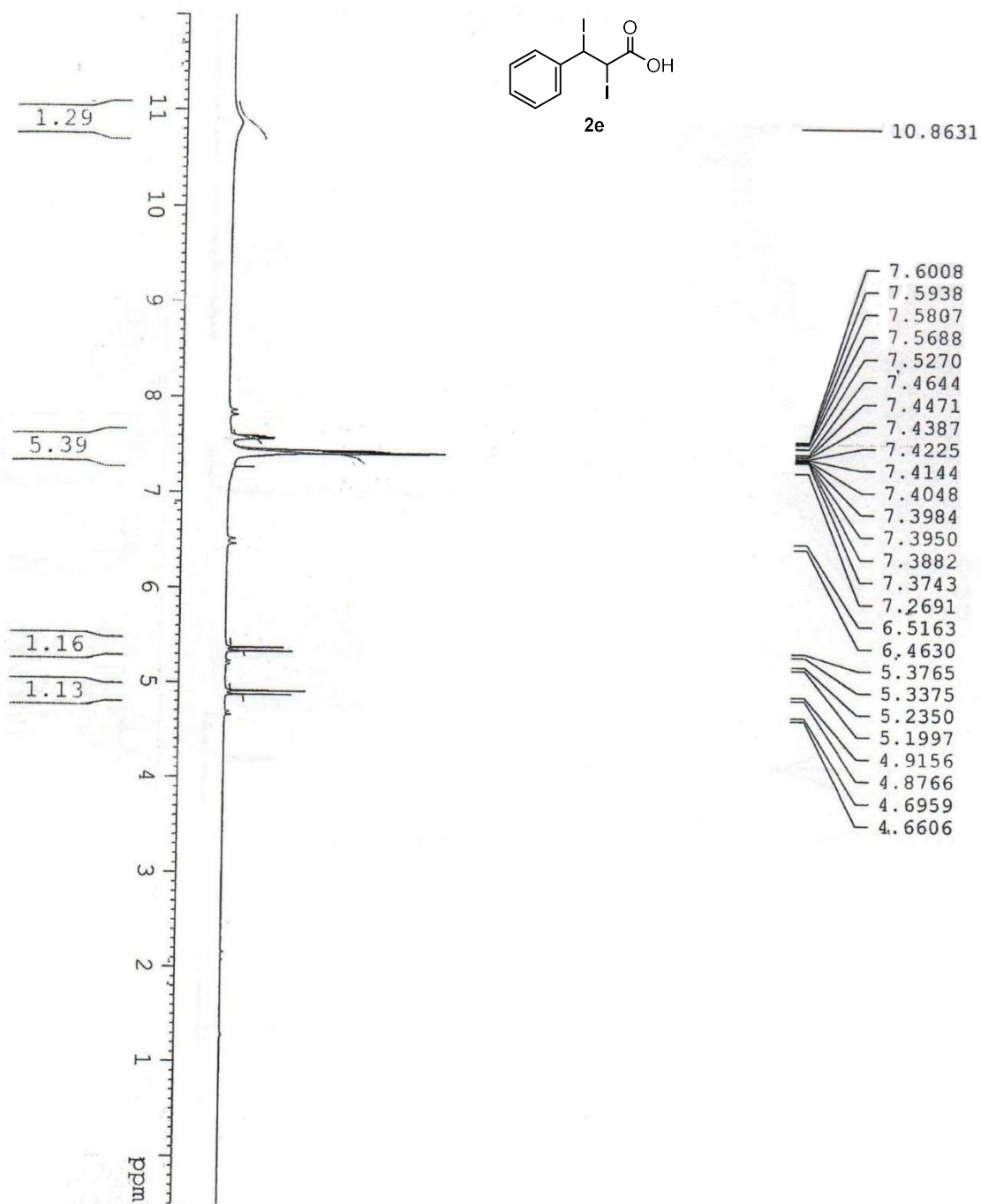
<sup>13</sup>C of VBSS-Chal (L)

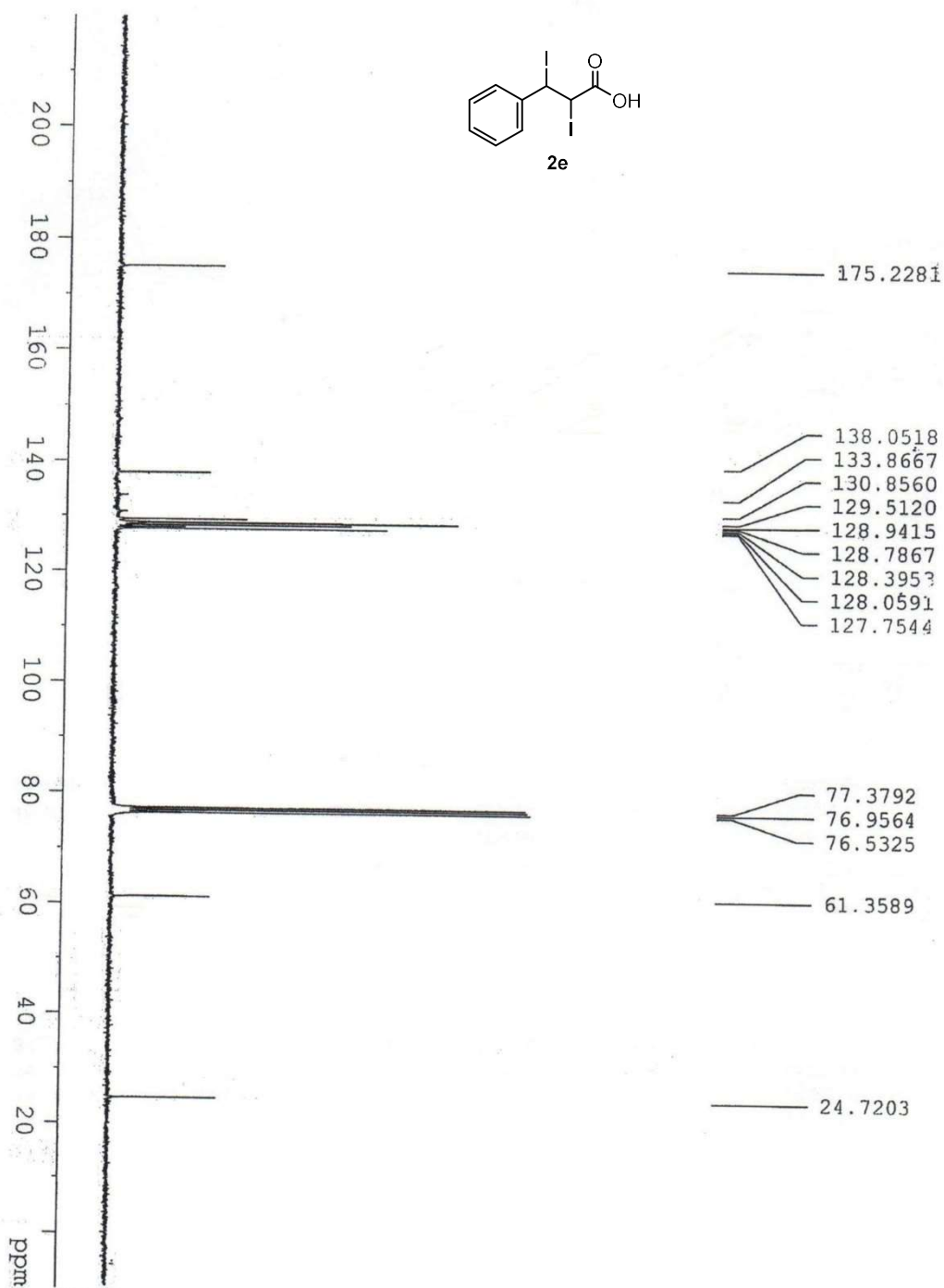


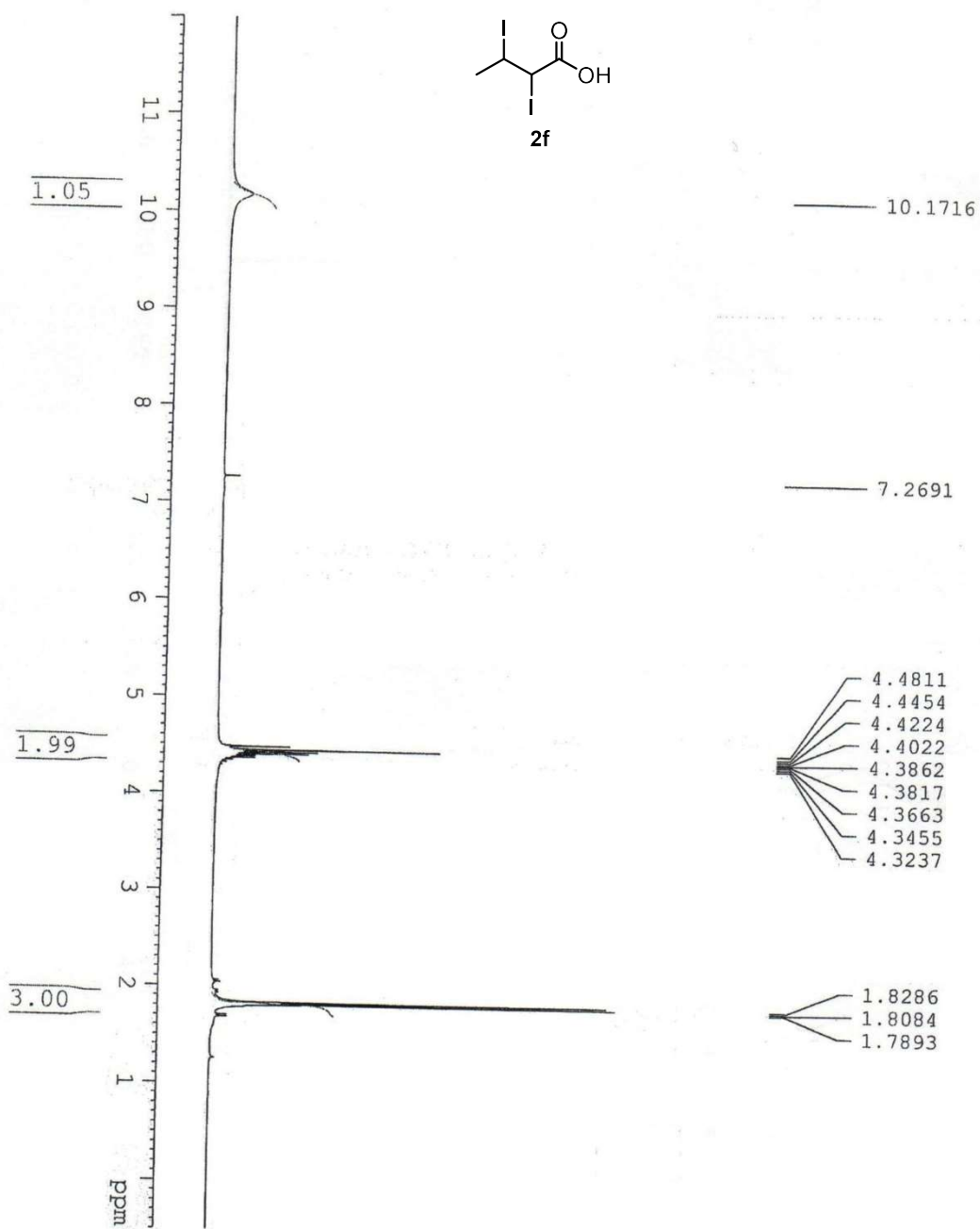
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 SOLVENT: CDCl3  
 NS: 128  
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 SWH: 24038.447 Hz  
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 SOLVENT: CDCl3  
 NS: 128  
 DS: 4  
 SWH: 24038.447 Hz  
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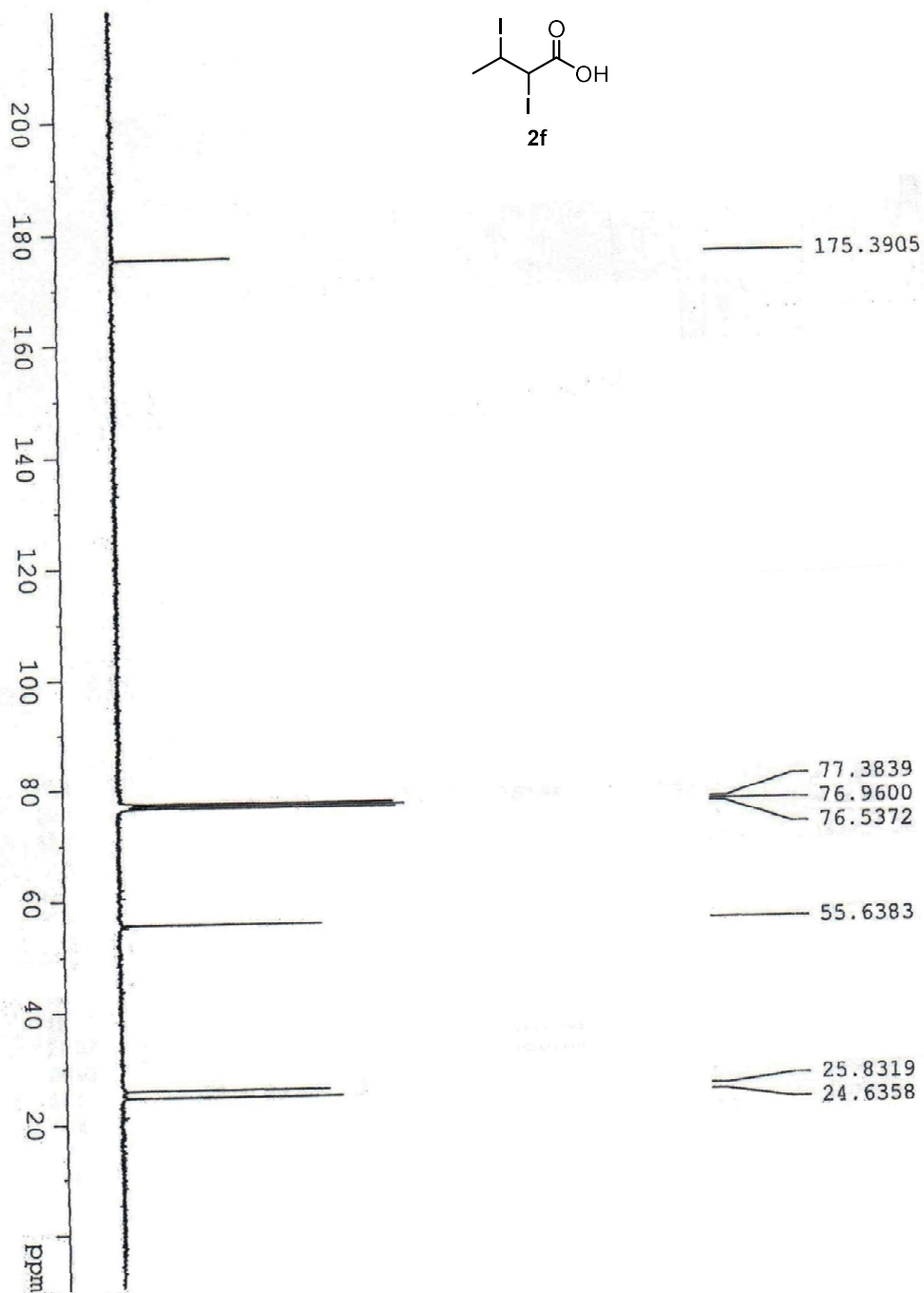
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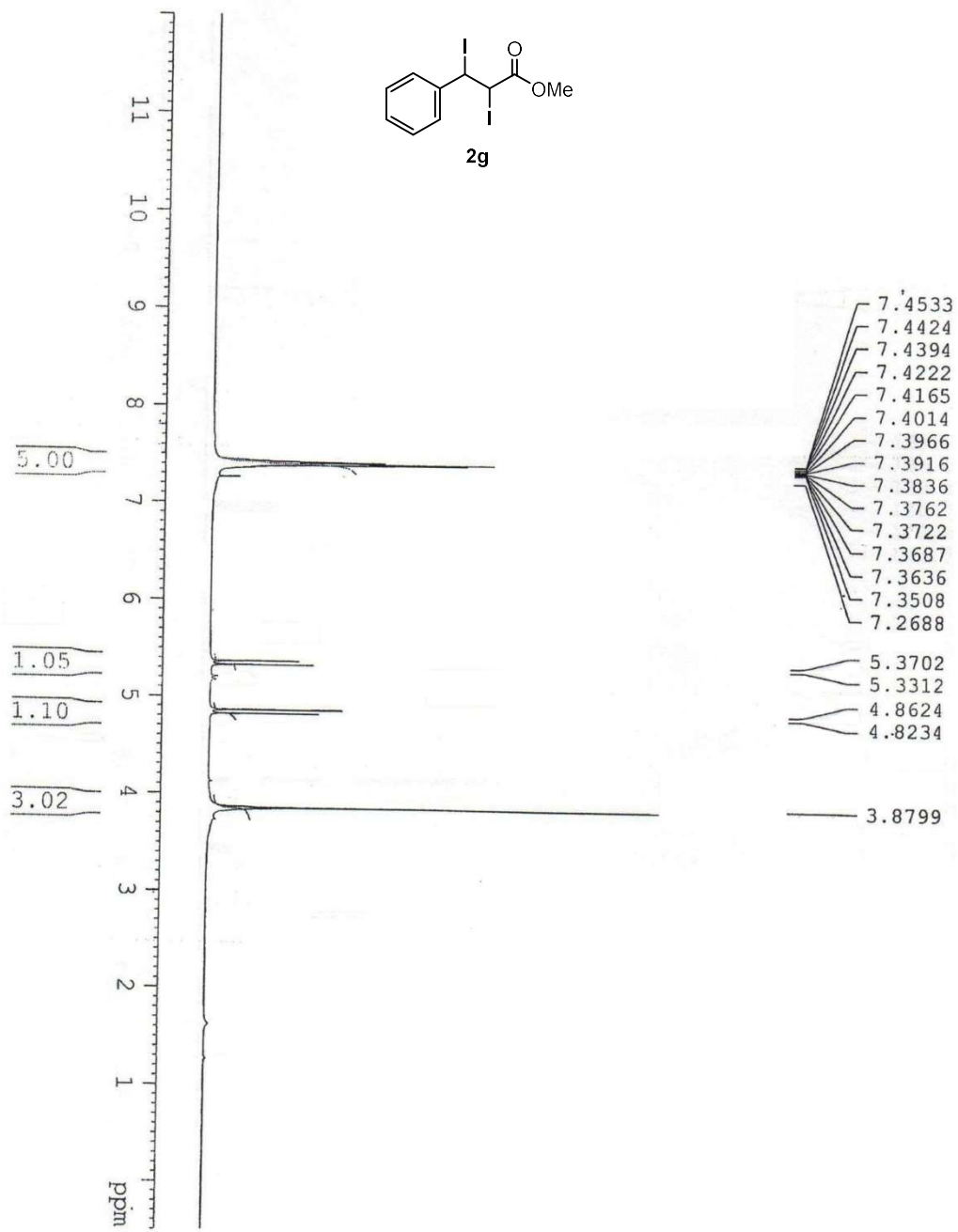
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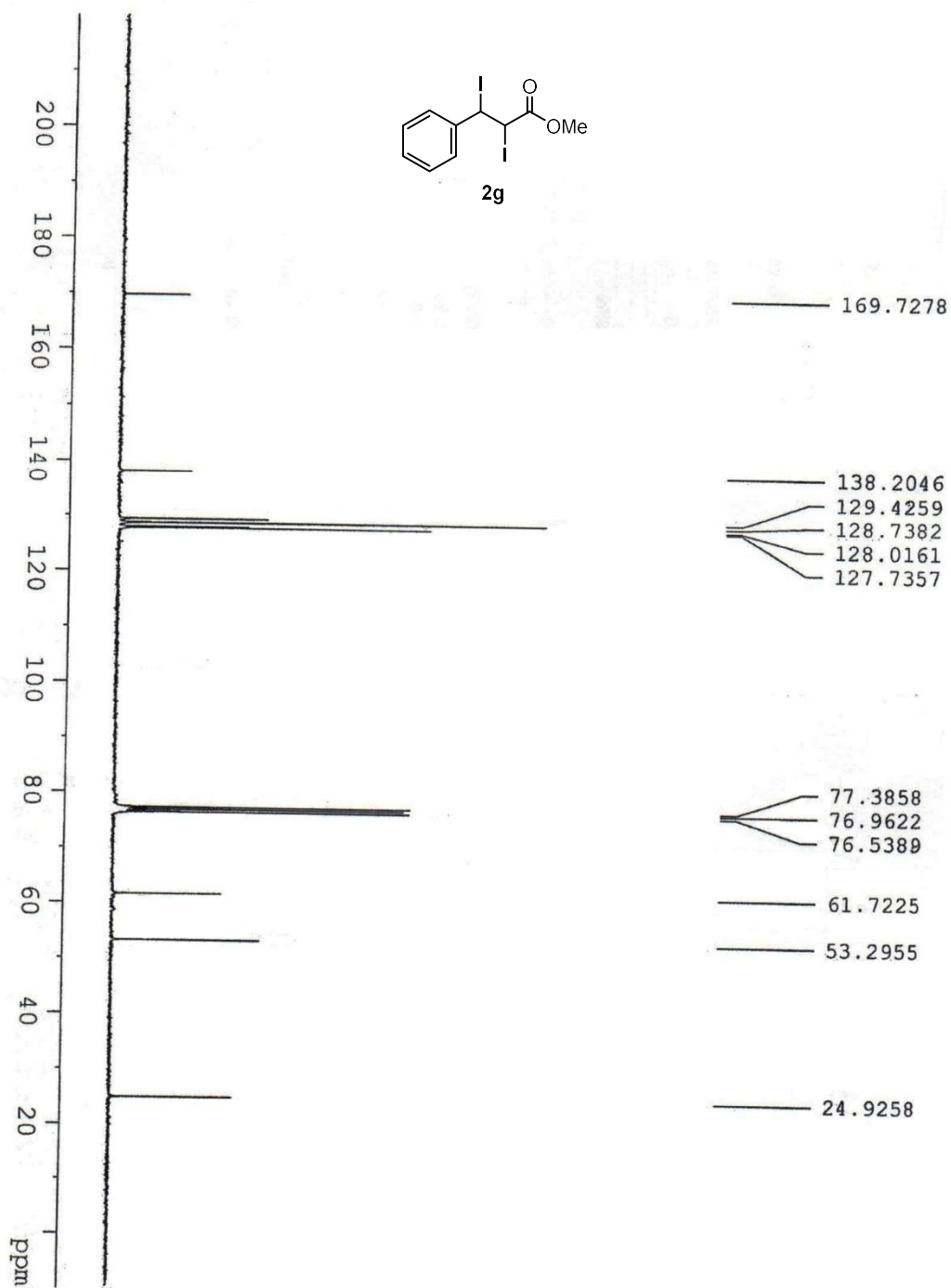


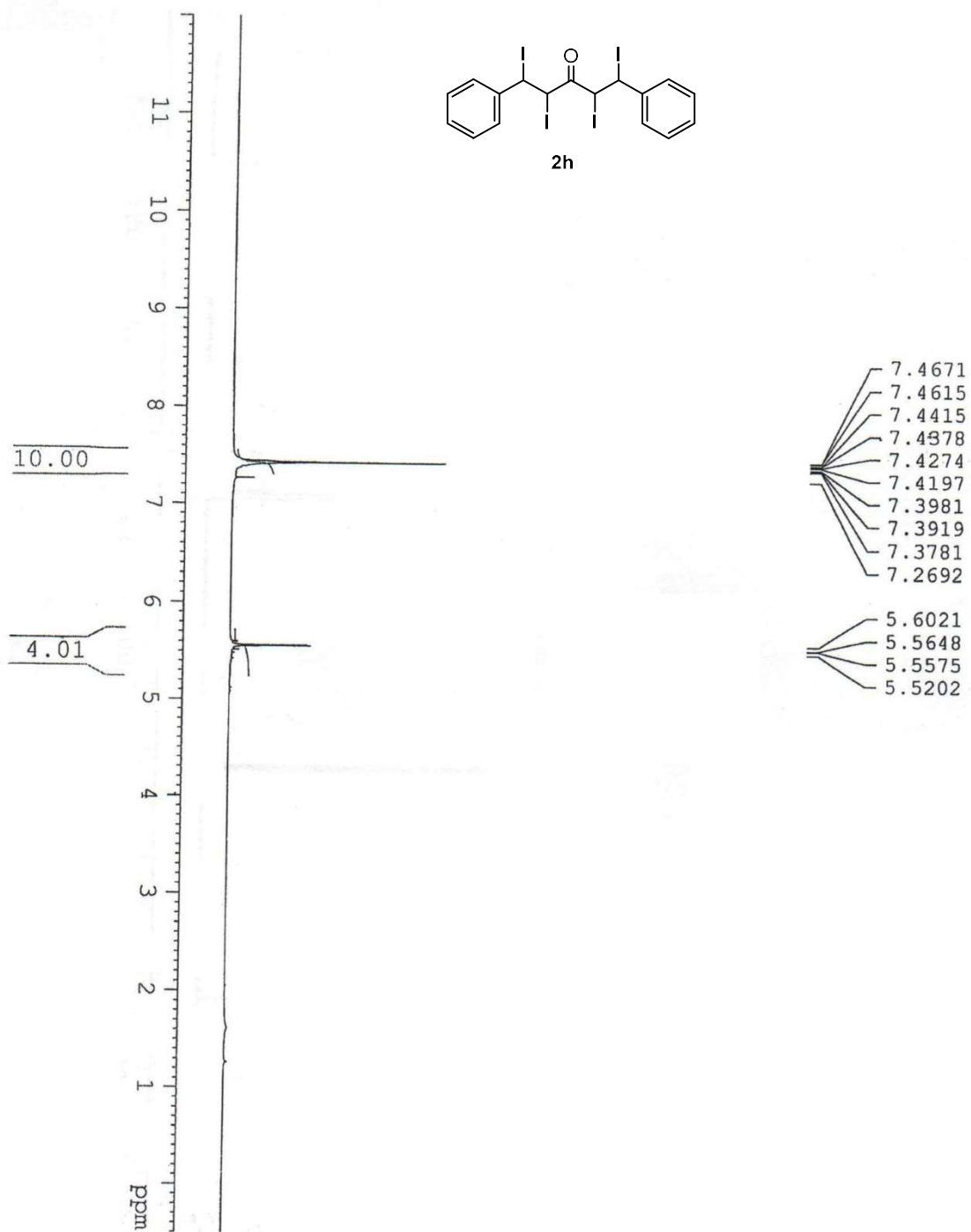


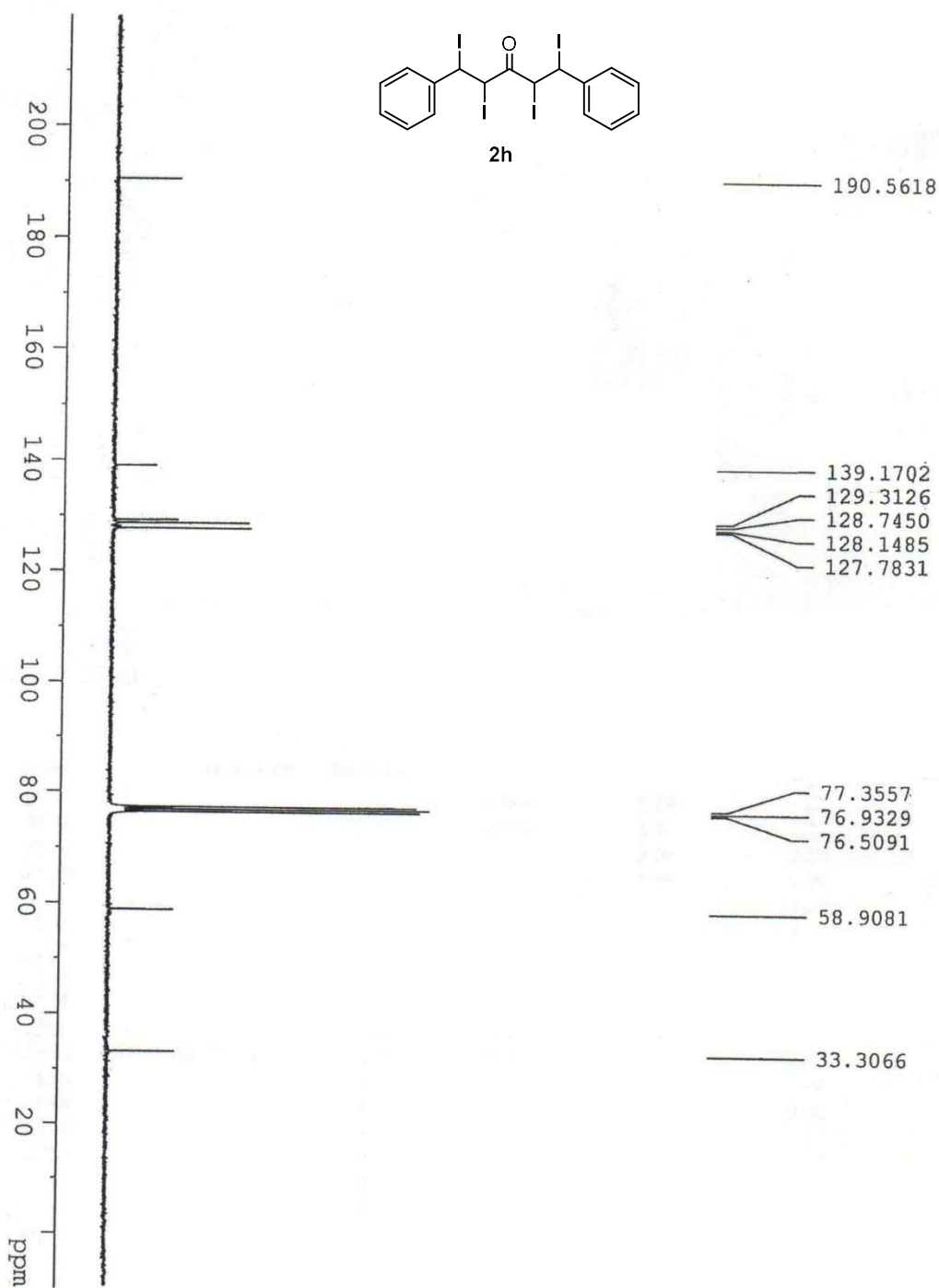


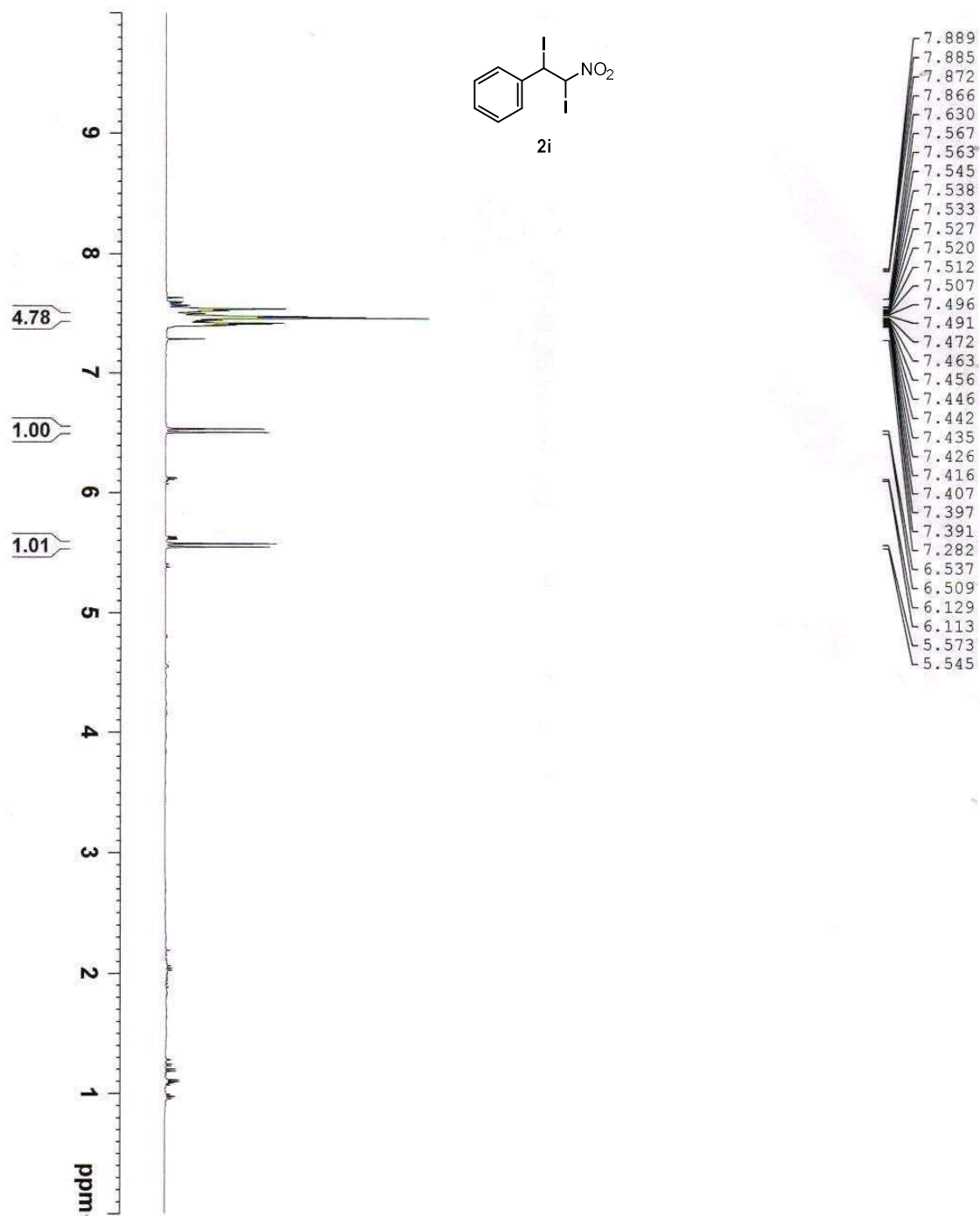


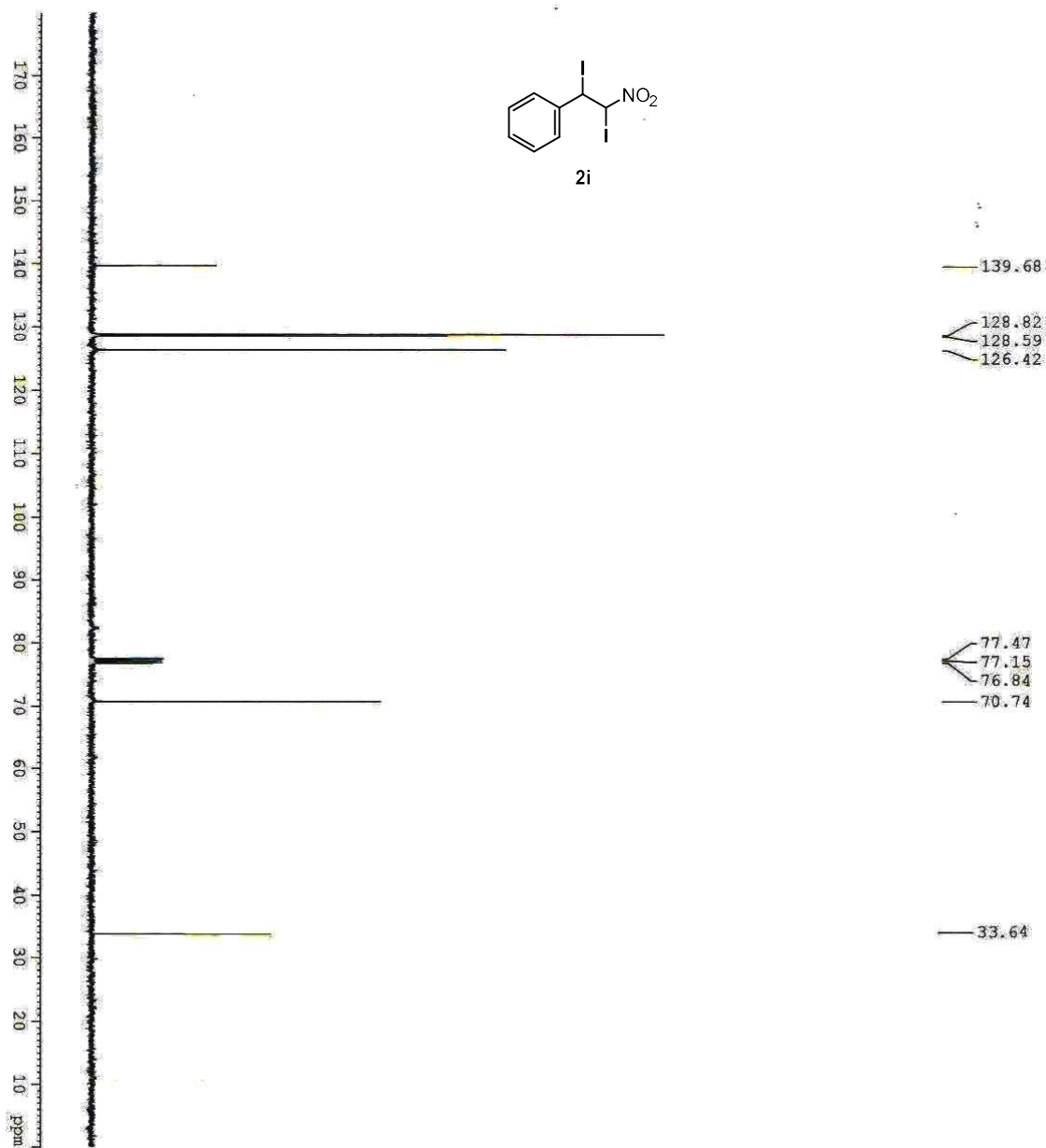


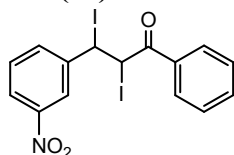










**Structure Determination of compound 2,3-diiodo-3-(3-nitro-phenyl)-1-phenyl-propan-1-one (2c):**

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<sub>15</sub>H<sub>11</sub>I<sub>2</sub>NO<sub>3</sub>; Mol. Wt.: 507.06

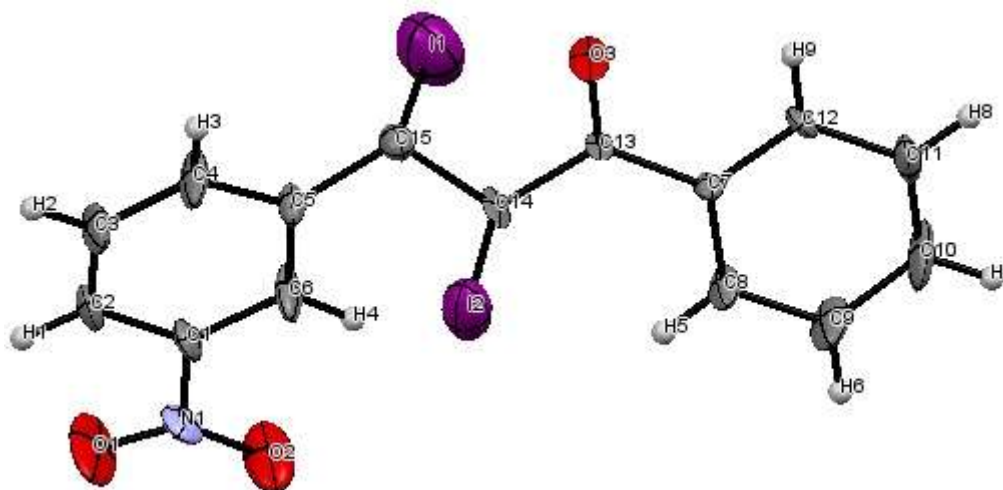
A specimen of C<sub>15</sub>H<sub>11</sub>I<sub>2</sub>NO<sub>3</sub> was used for the X-ray crystallographic analysis. The X-ray intensity data were measured.

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  $\theta$  angle of 30.04° (0.71 Å 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  $a = 9.1267(9)$  Å,  $b = 9.2452(10)$  Å,  $c = 10.5181(10)$  Å,  $\alpha = 89.390(5)^\circ$ ,  $\beta = 68.455(4)^\circ$ ,  $\gamma = 68.534(4)^\circ$ , volume = 760.34(14) Å<sup>3</sup>, 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  $6.298 e^-/\text{Å}^3$  and the largest hole was  $-4.469 e^-/\text{Å}^3$  with an RMS deviation of  $0.538 e^-/\text{Å}^3$ . On the basis of the final model, the calculated density was  $2.969 \text{ g/cm}^3$  and  $F(000)$ , 600  $e^-$ .

Identification code	vbchem005
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<b>Chemical formula</b>	C <sub>15</sub> H <sub>11</sub> I <sub>2</sub> NO <sub>3</sub>
<b>Formula weight</b>	169.93 g/mol
<b>Temperature</b>	296(2) K
<b>Wavelength</b>	0.71073 Å
<b>Crystal system</b>	triclinic
<b>Space group</b>	P -1
<b>Unit cell dimensions</b>	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)°
<b>Volume</b>	760.34(14) Å <sup>3</sup>
<b>Z</b>	8
<b>Density (calculated)</b>	2.969 g/cm <sup>3</sup>
<b>Absorption coefficient</b>	8.201 mm <sup>-1</sup>
<b>F(000)</b>	600



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