

Solvent Controlled Synthesis of 2,3-Diarylepoxy Indenone, α -Hydroxy Diarylindanones and their Evaluation as Inhibitors of DNA Alkylation Repair

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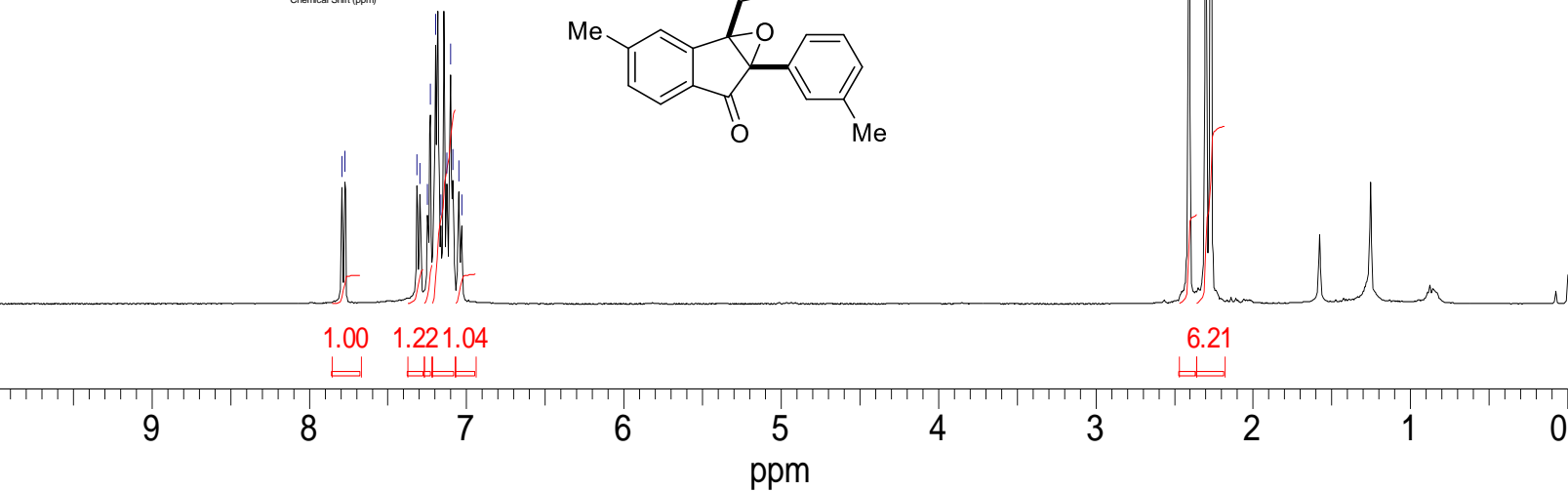
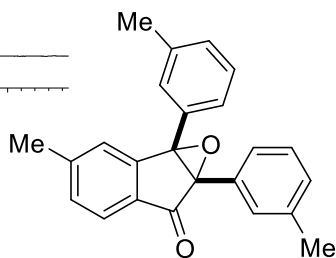
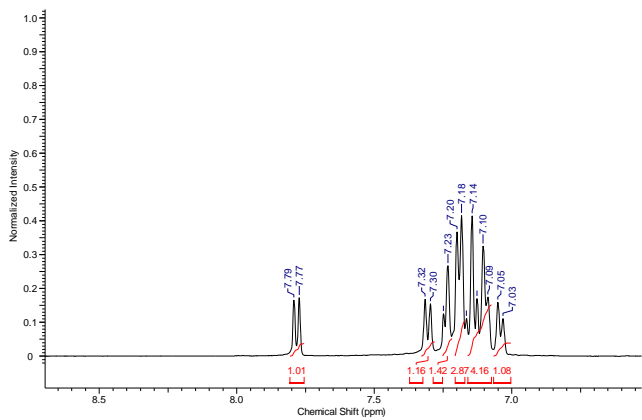
1. ¹H, ¹³C NMR spectrum of compounds **2**, **3**, **1**, **4**.....S2-S79
2. X-ray crystallography Data of compound **2a**, **3ab**, **3ac**.....S80-S84

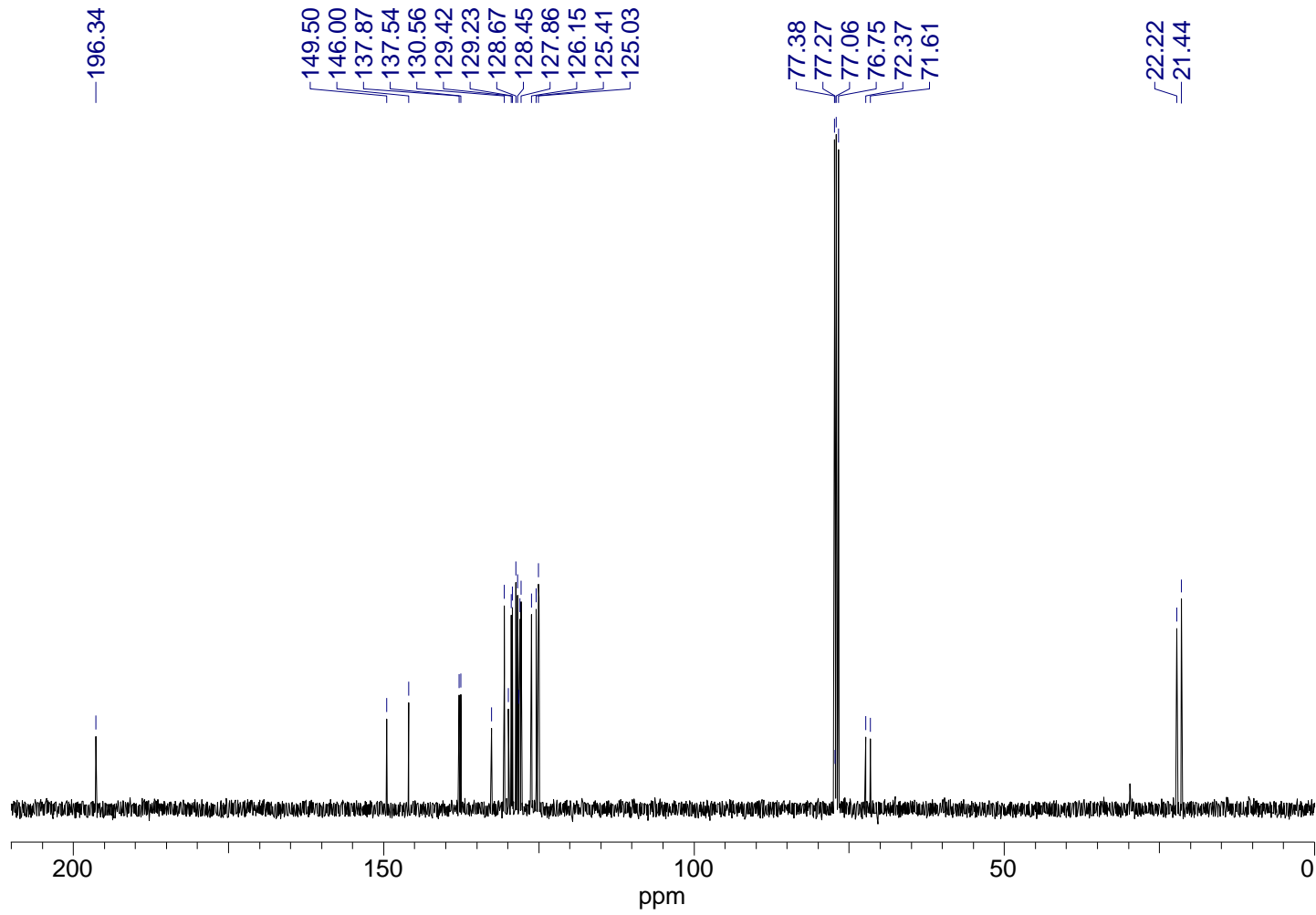
1. Copies of NMR Spectrum:

^1H (400 MHz) and ^{13}C (100 MHz) NMR in CDCl_3 of Compound 2a

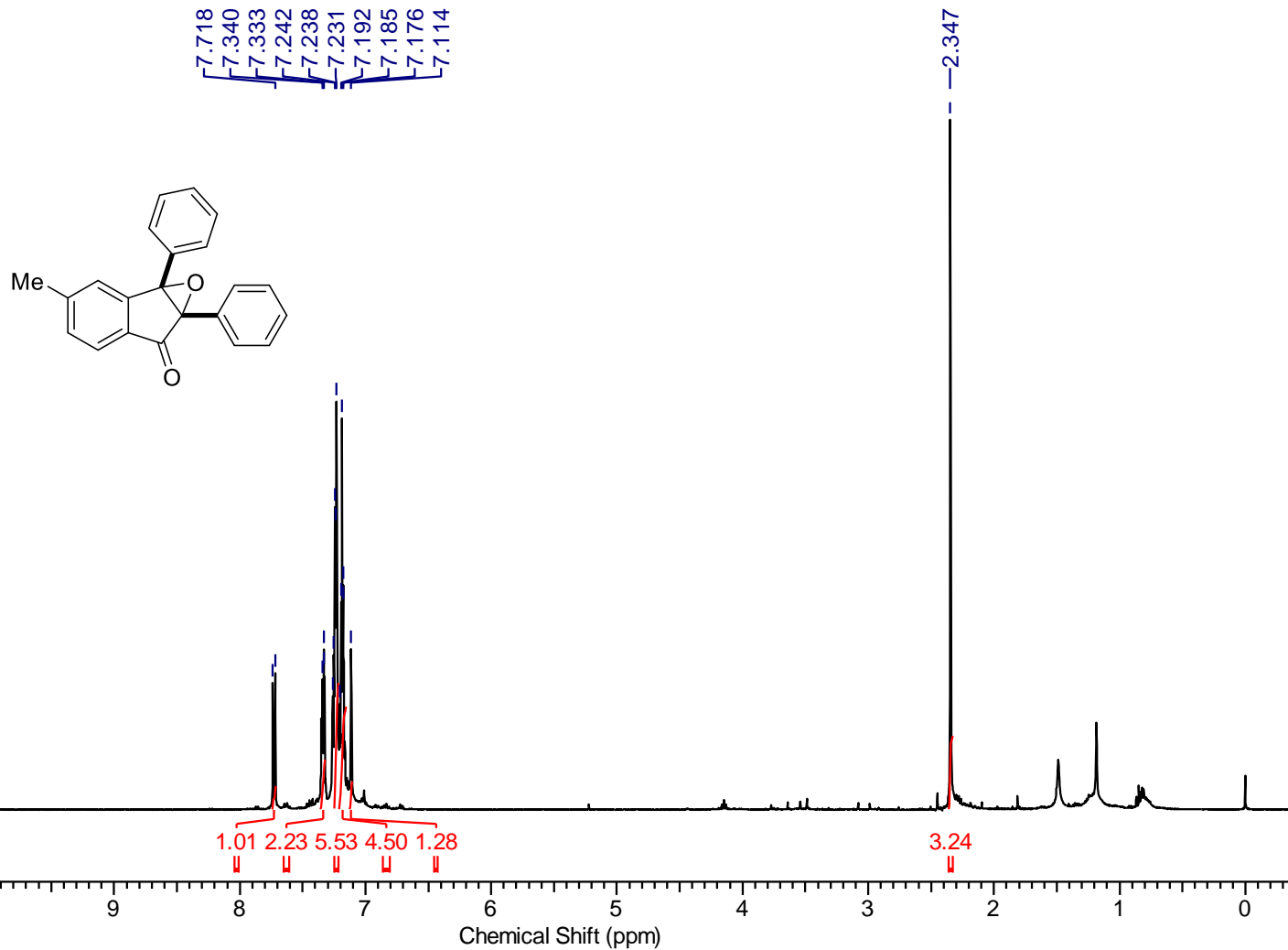
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7.32
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7.23
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7.14
7.13
7.10
7.09

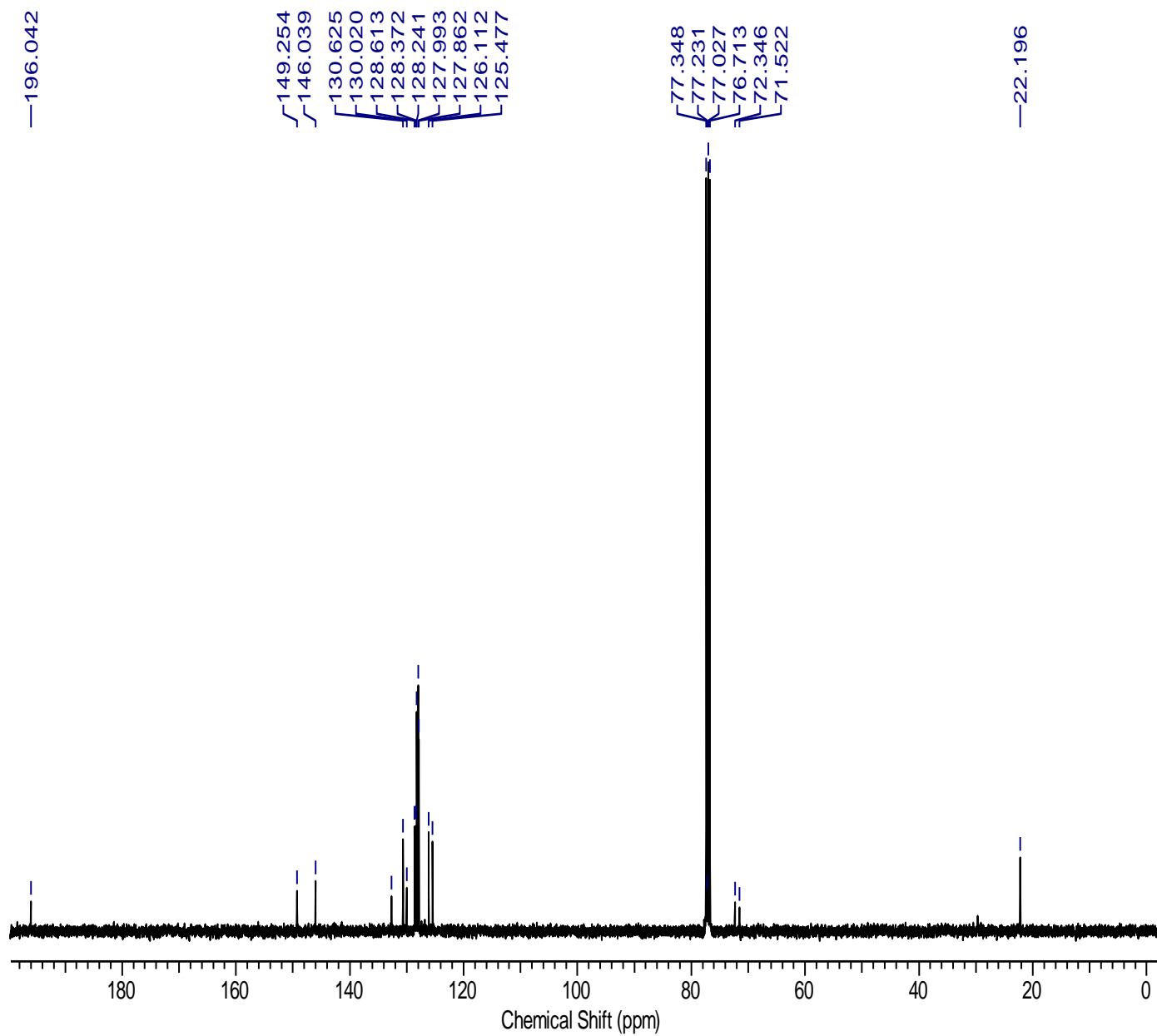
2.41
2.30
2.27



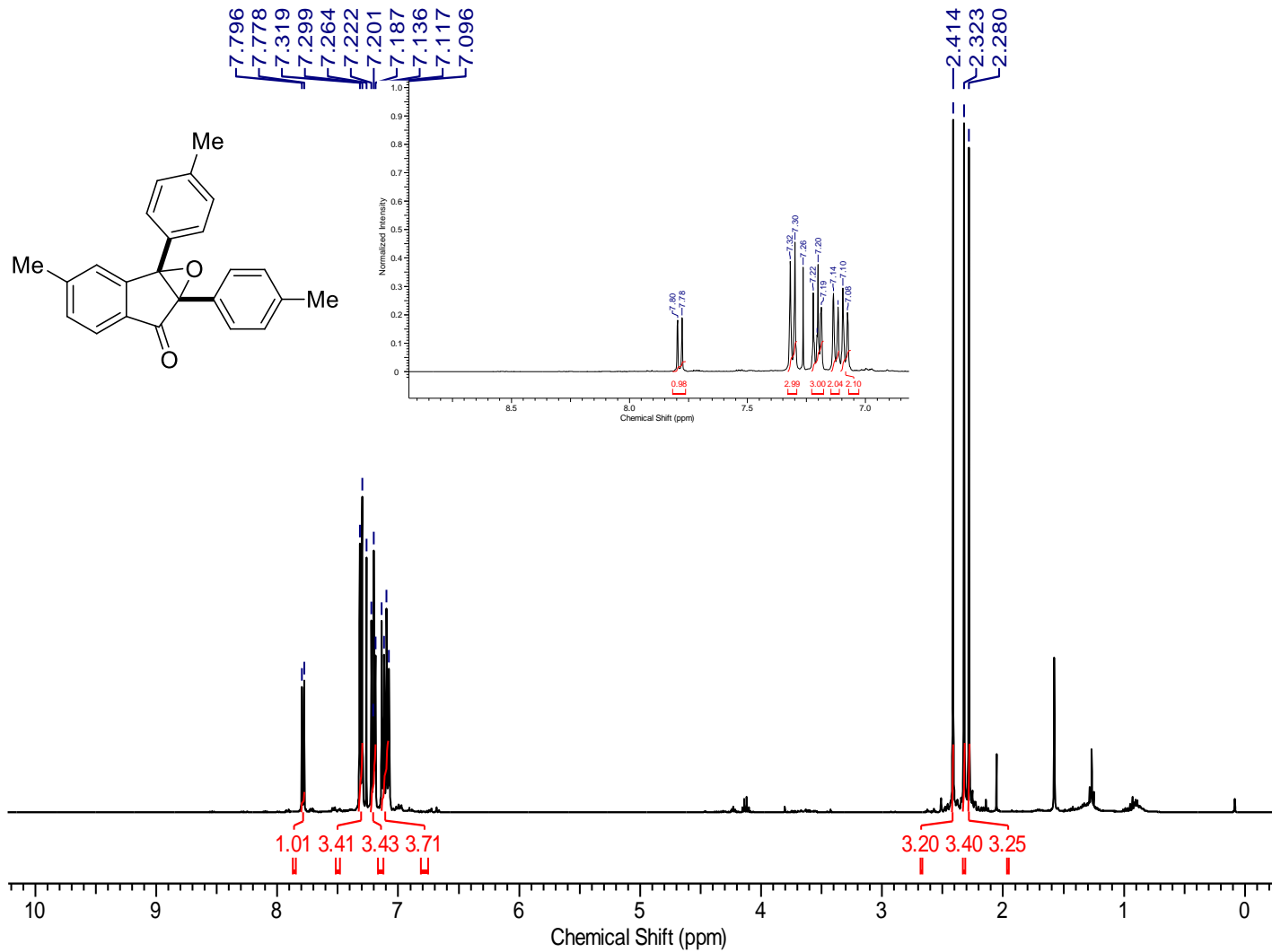


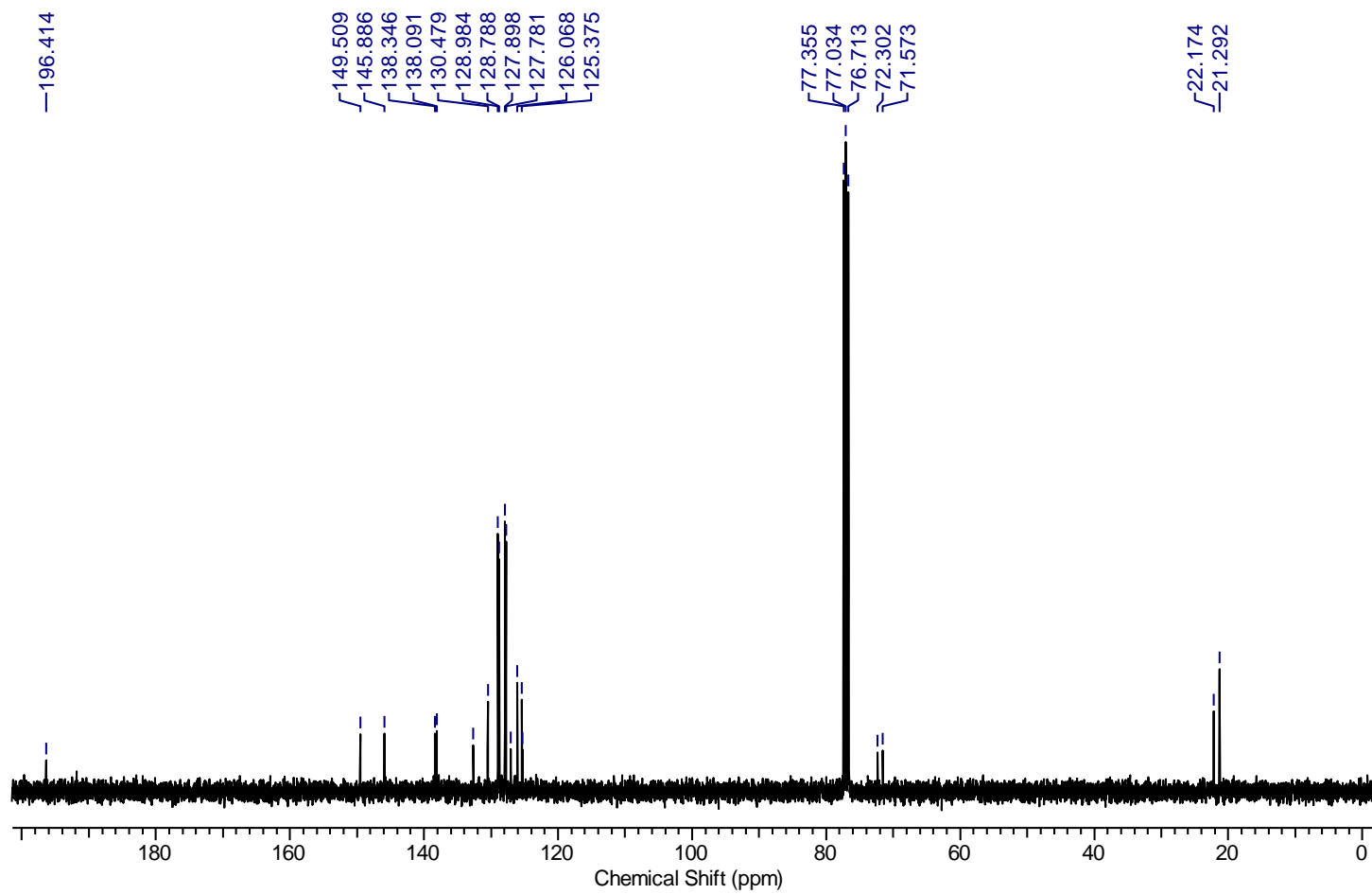
¹H (400 MHz) and ¹³C (100 MHz) NMR in CDCl₃ of Compound 2b



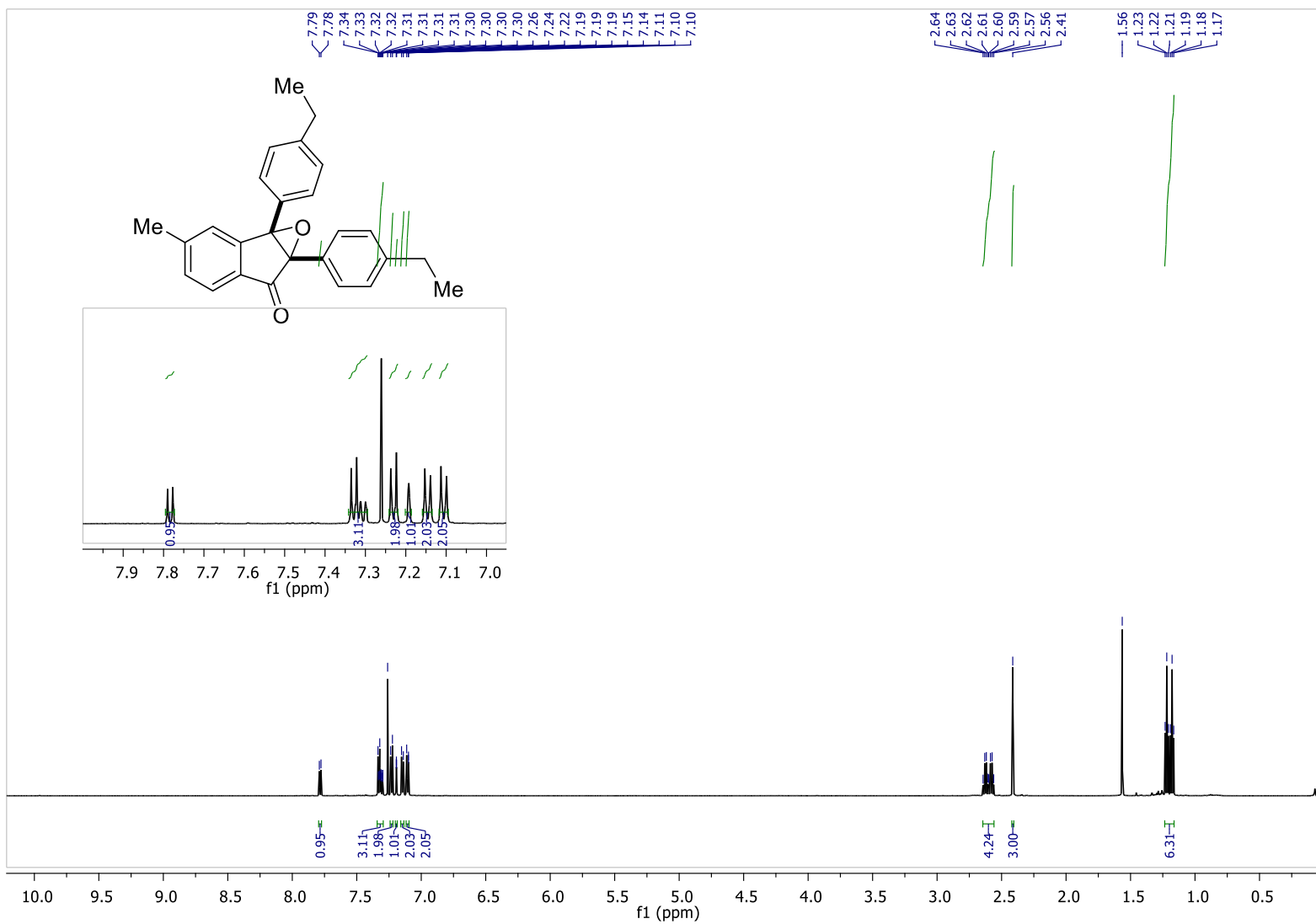


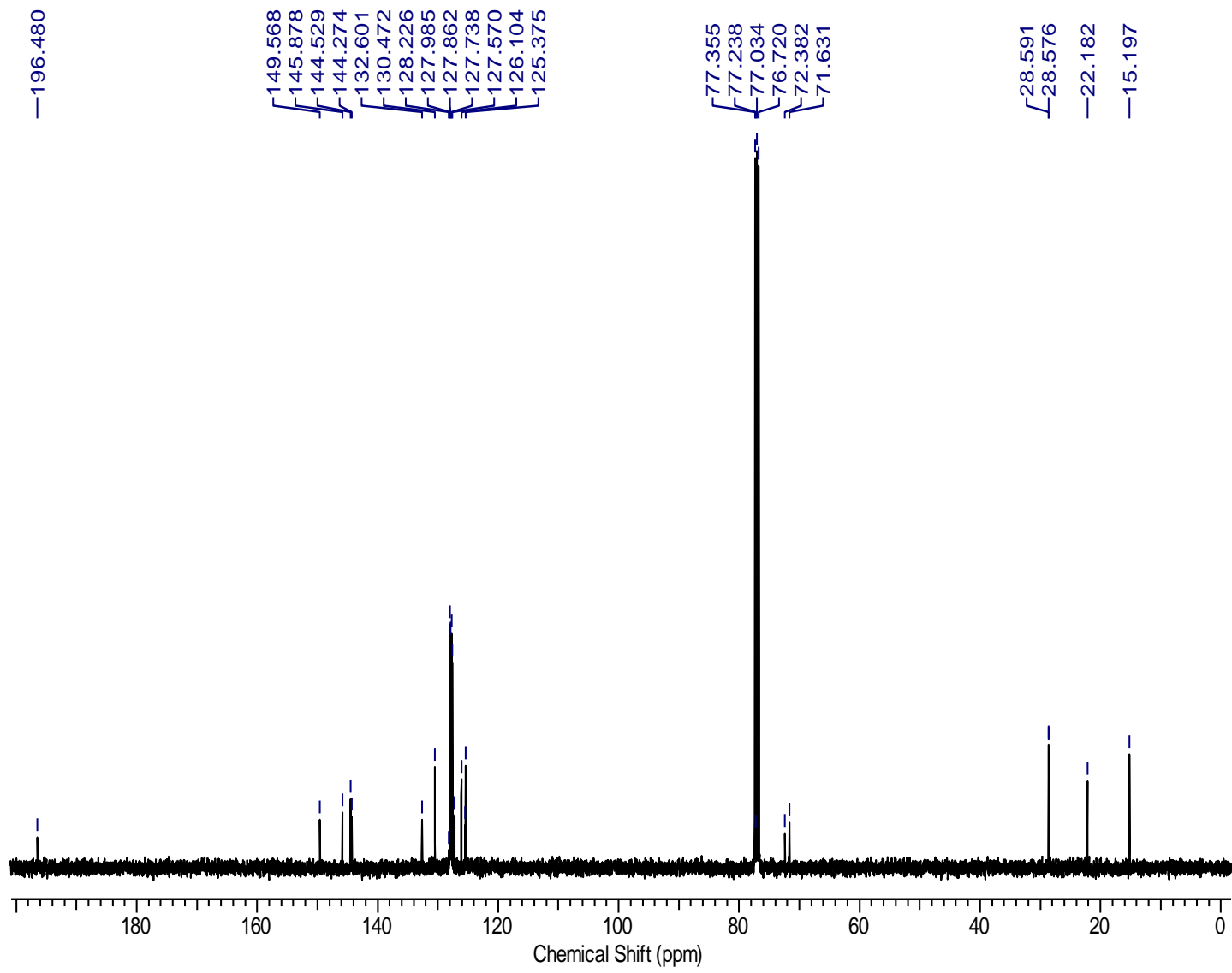
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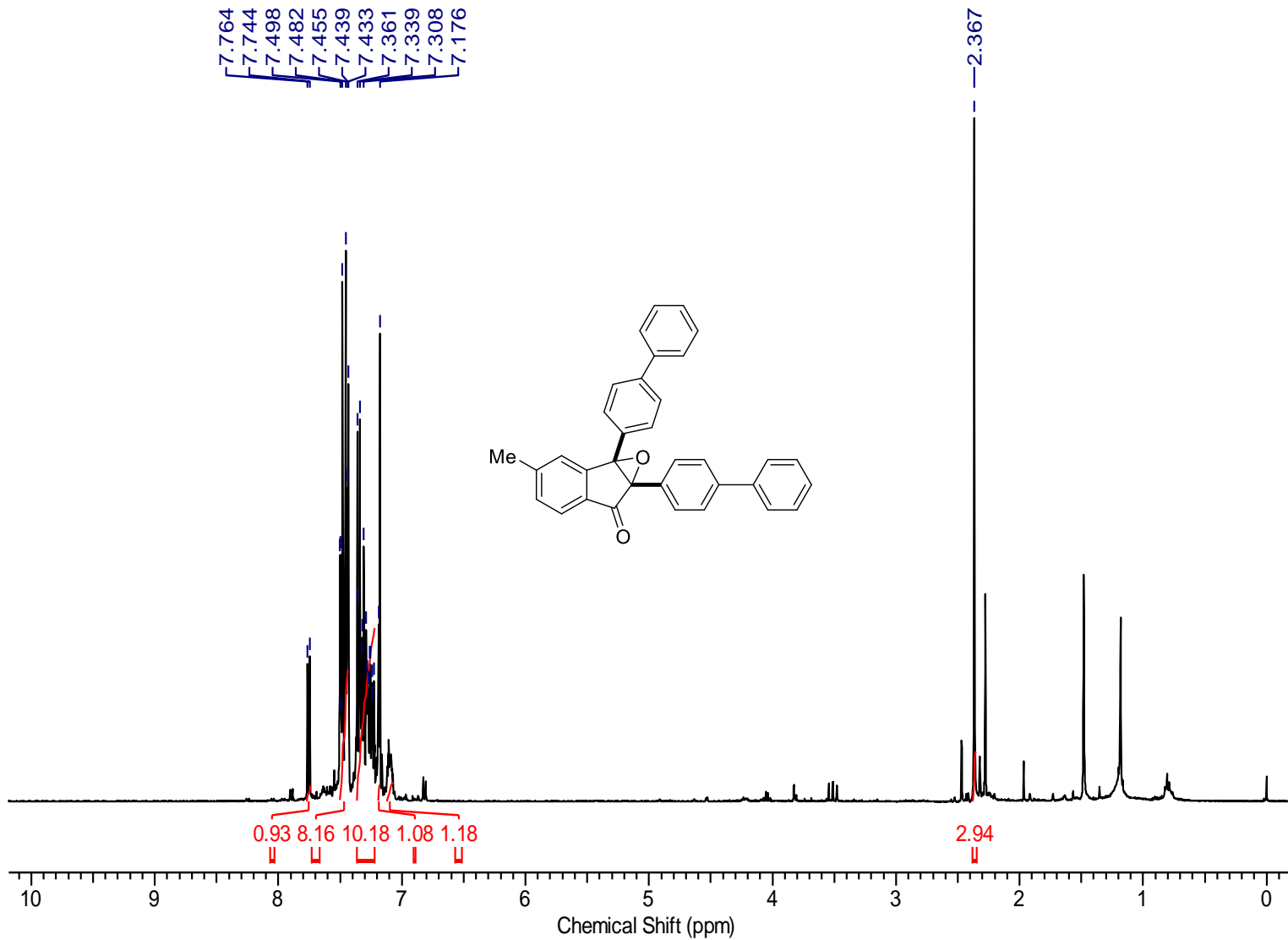


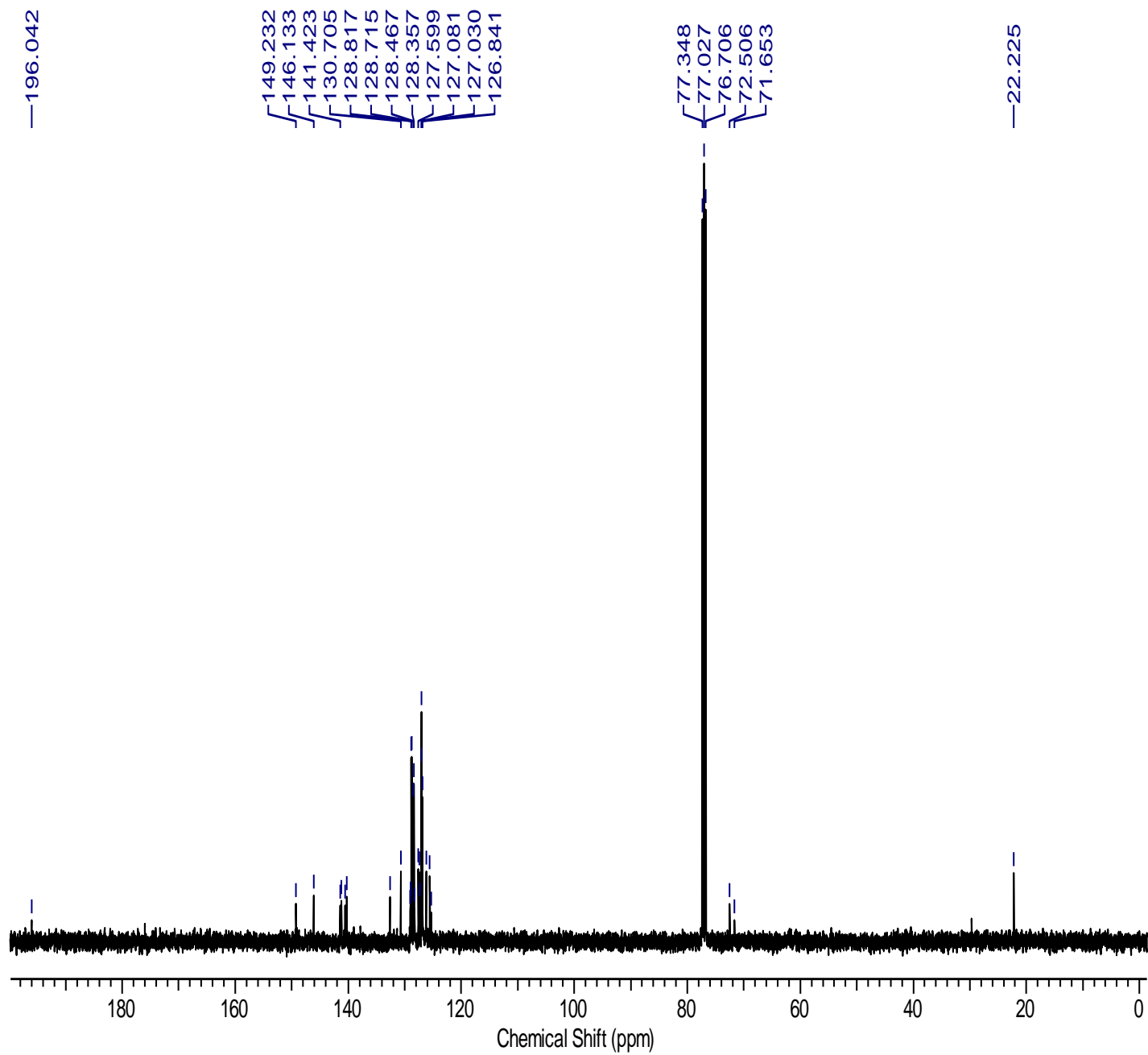
^1H (400 MHz) and ^{13}C (100 MHz) NMR in CDCl_3 of Compound 2d



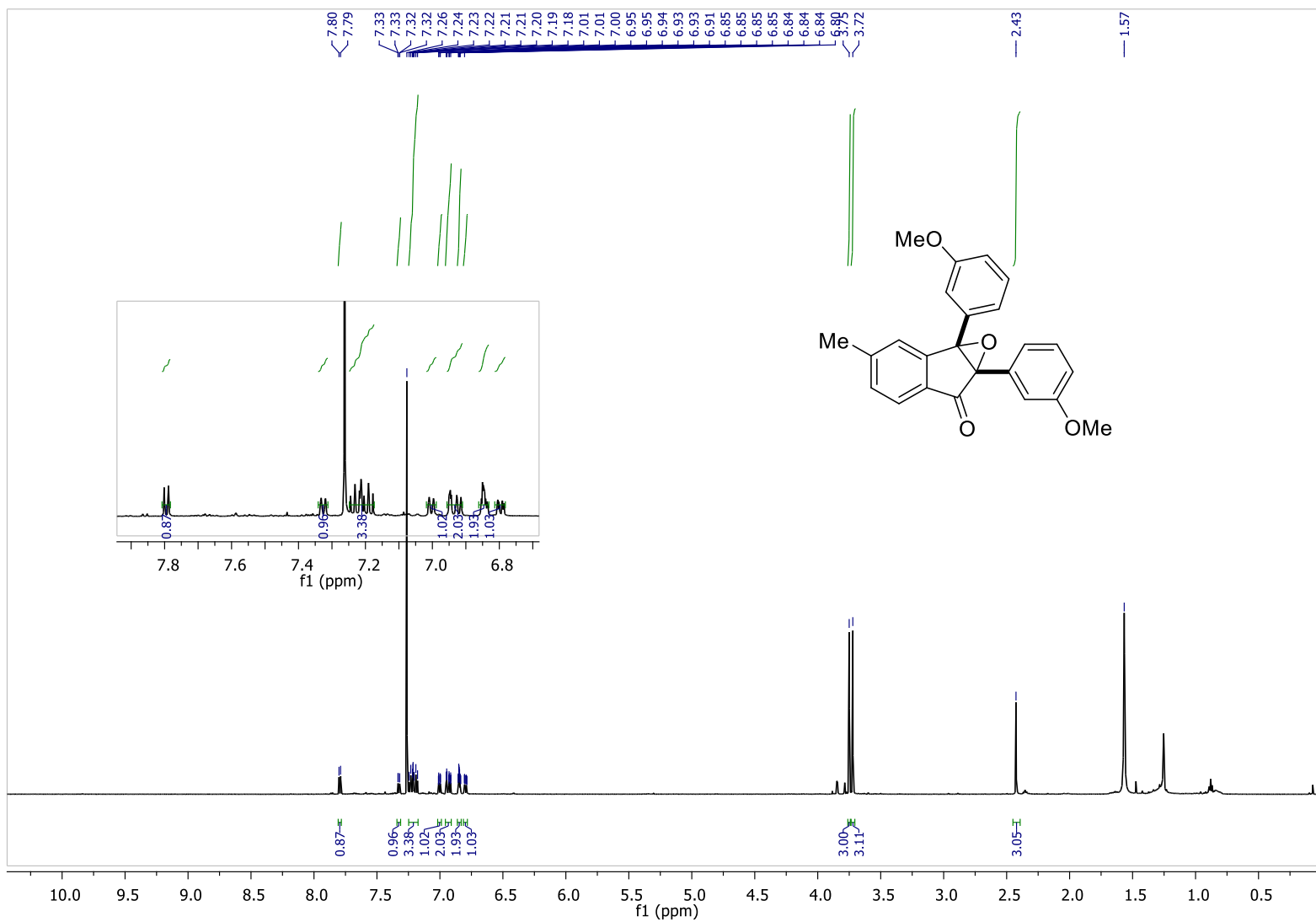


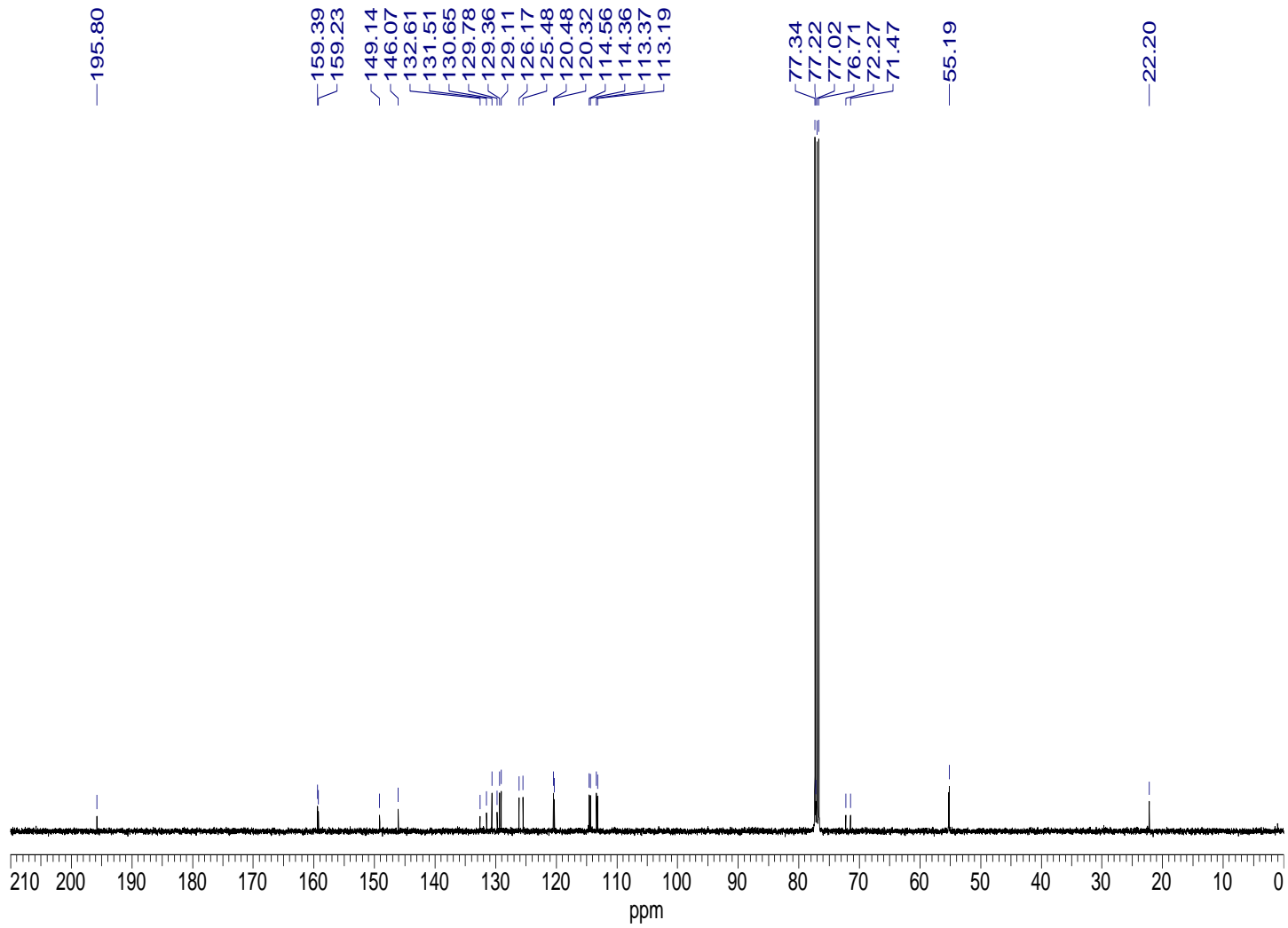
¹H (400 MHz) and ¹³C (100 MHz) NMR in CDCl₃ of Compound 2e



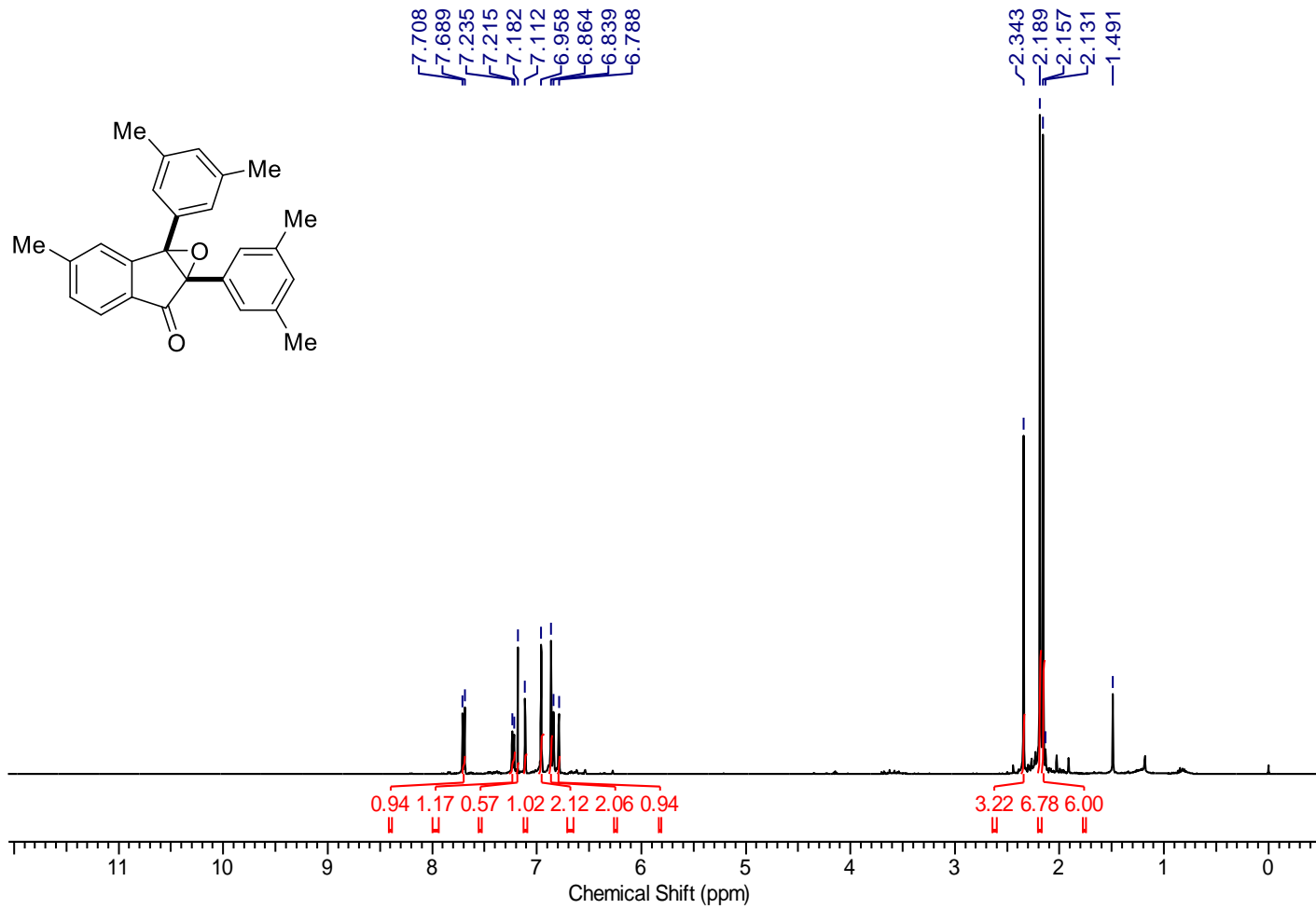


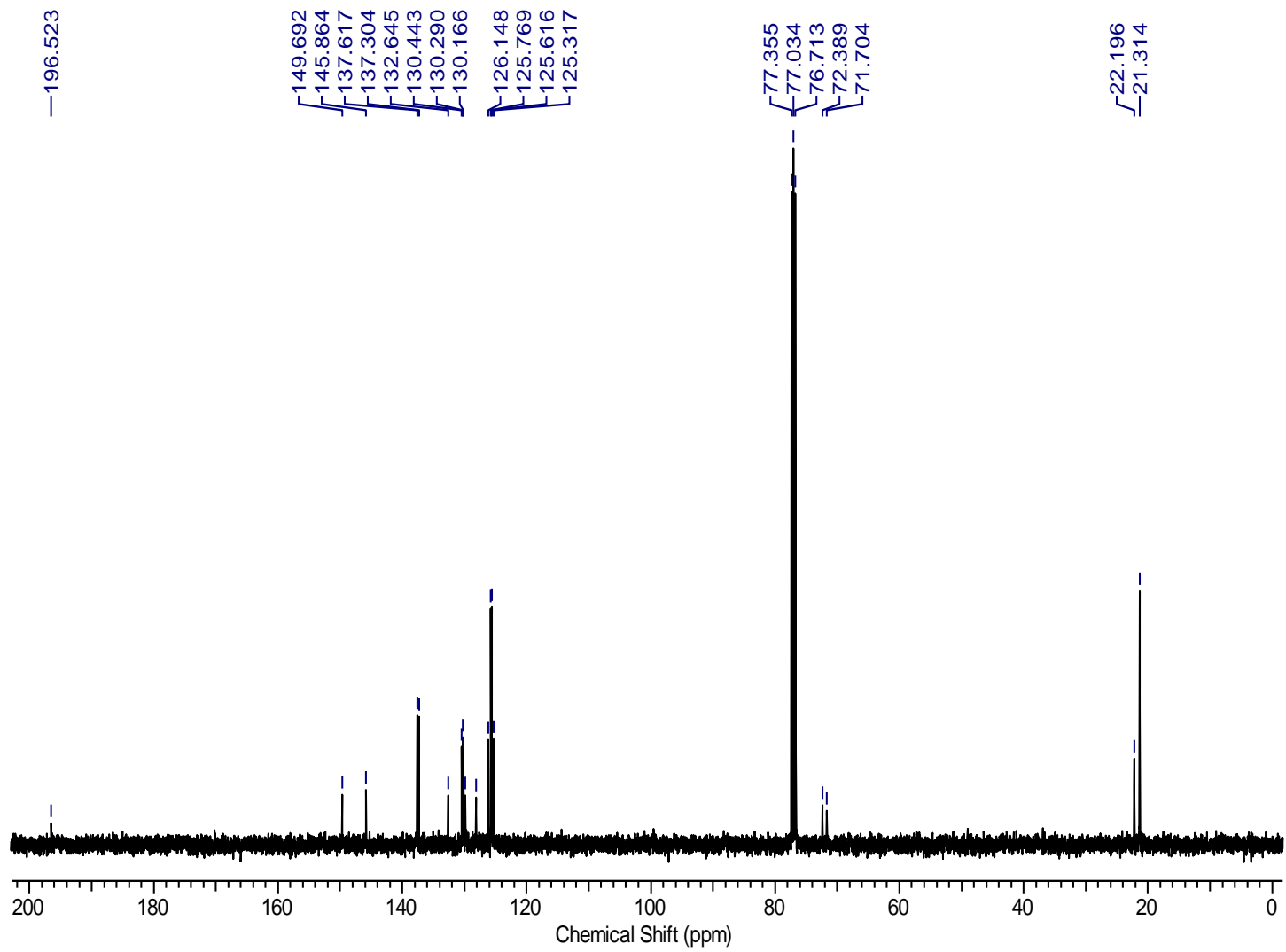
¹H (400 MHz) and ¹³C (100 MHz) NMR in CDCl₃ of Compound 2f



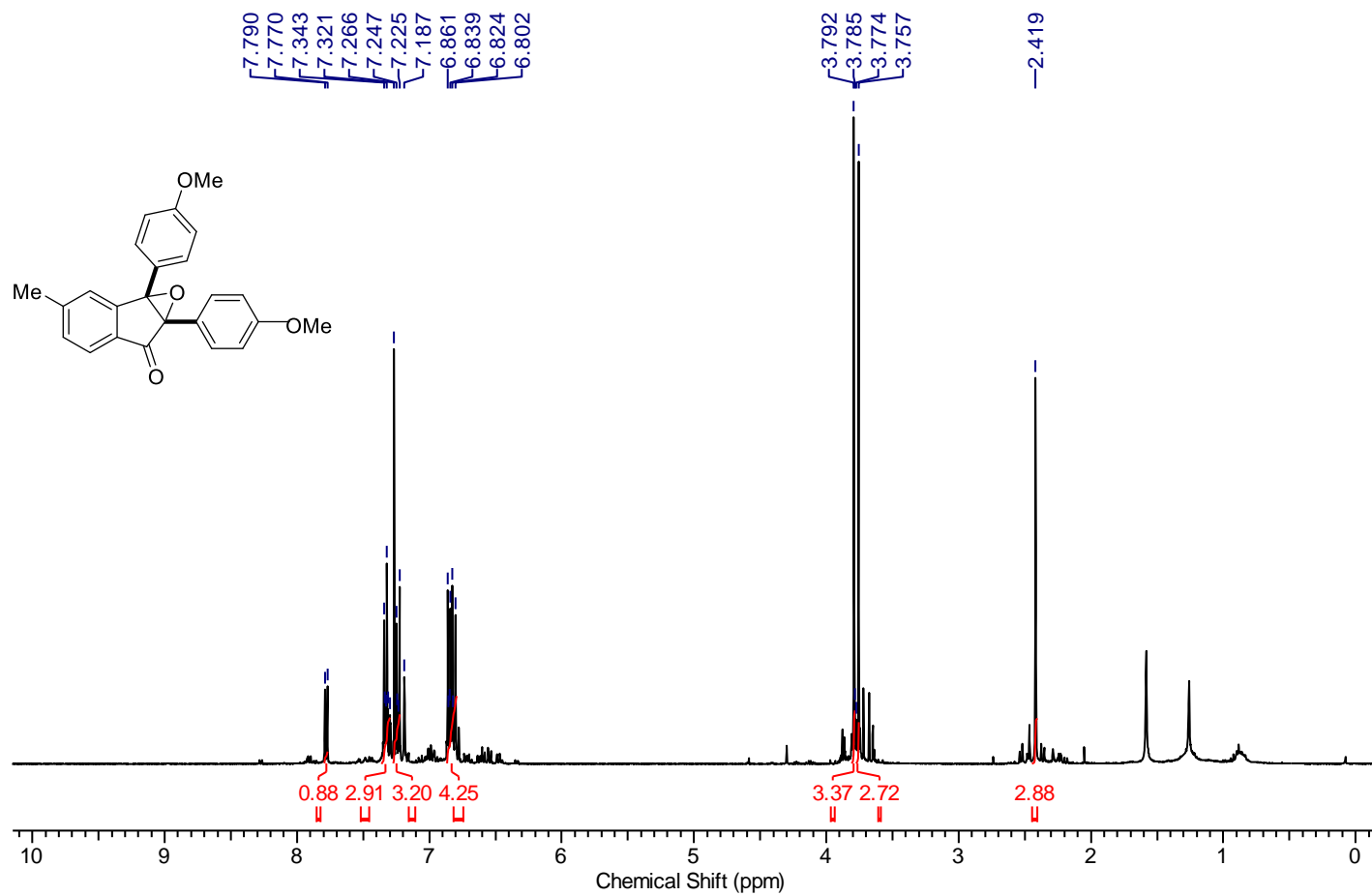


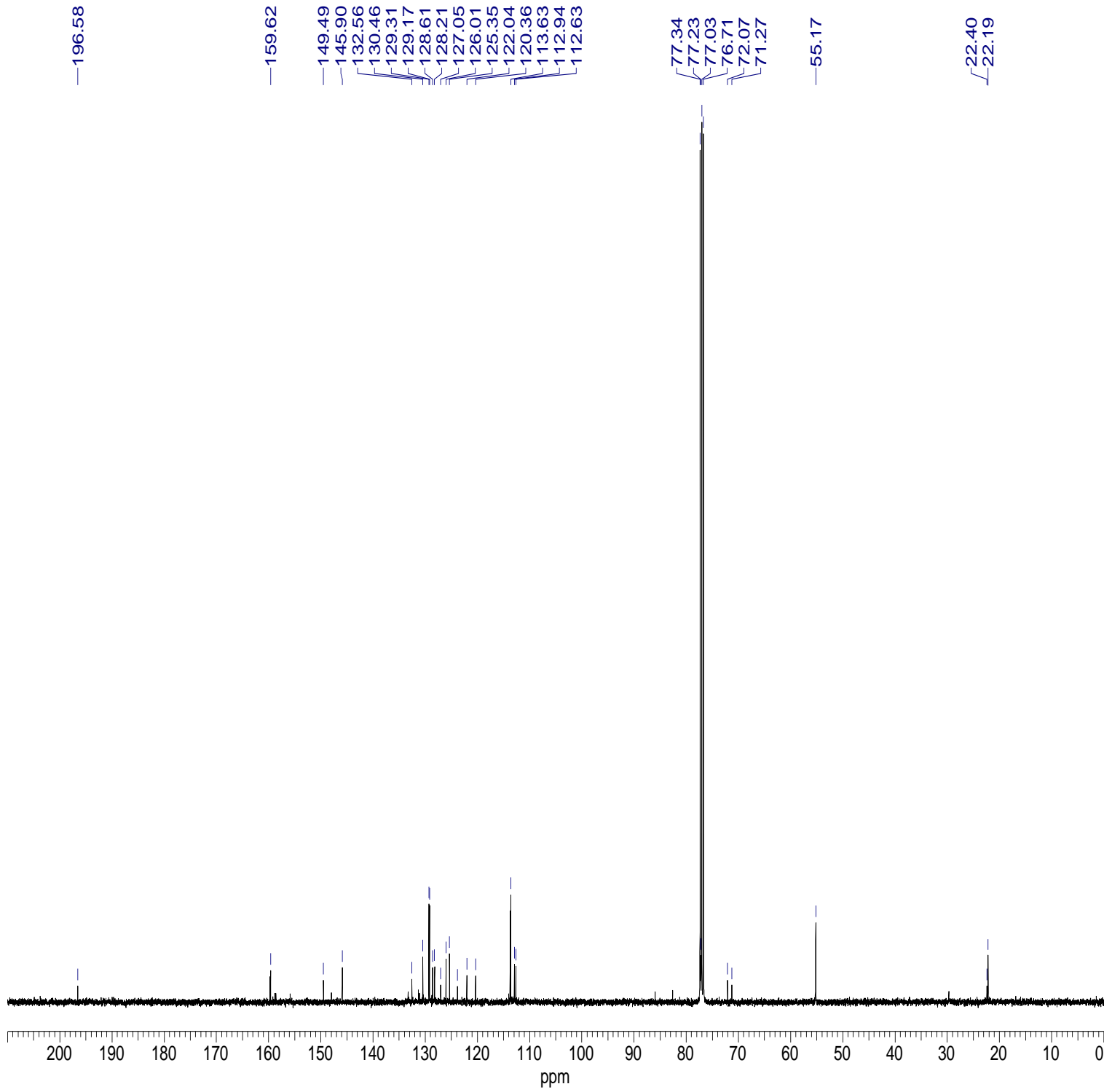
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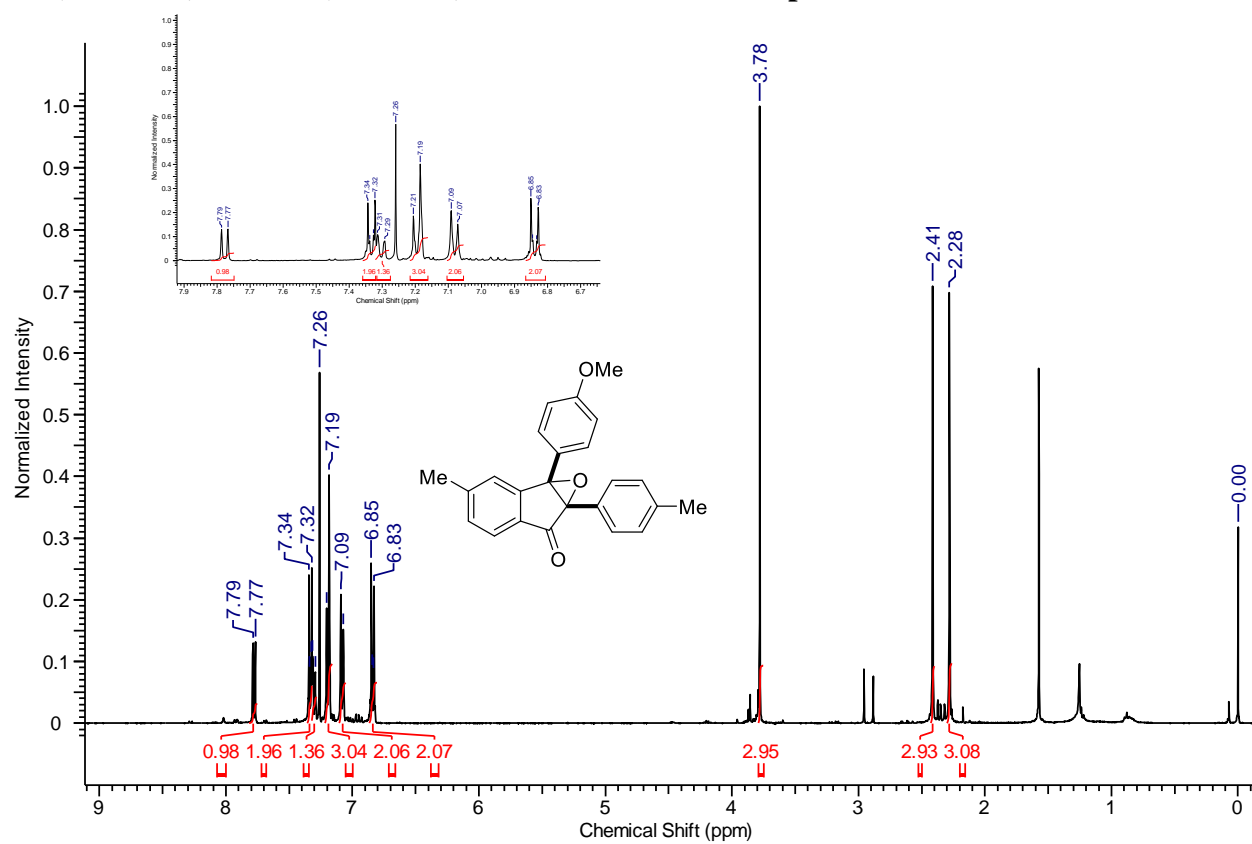


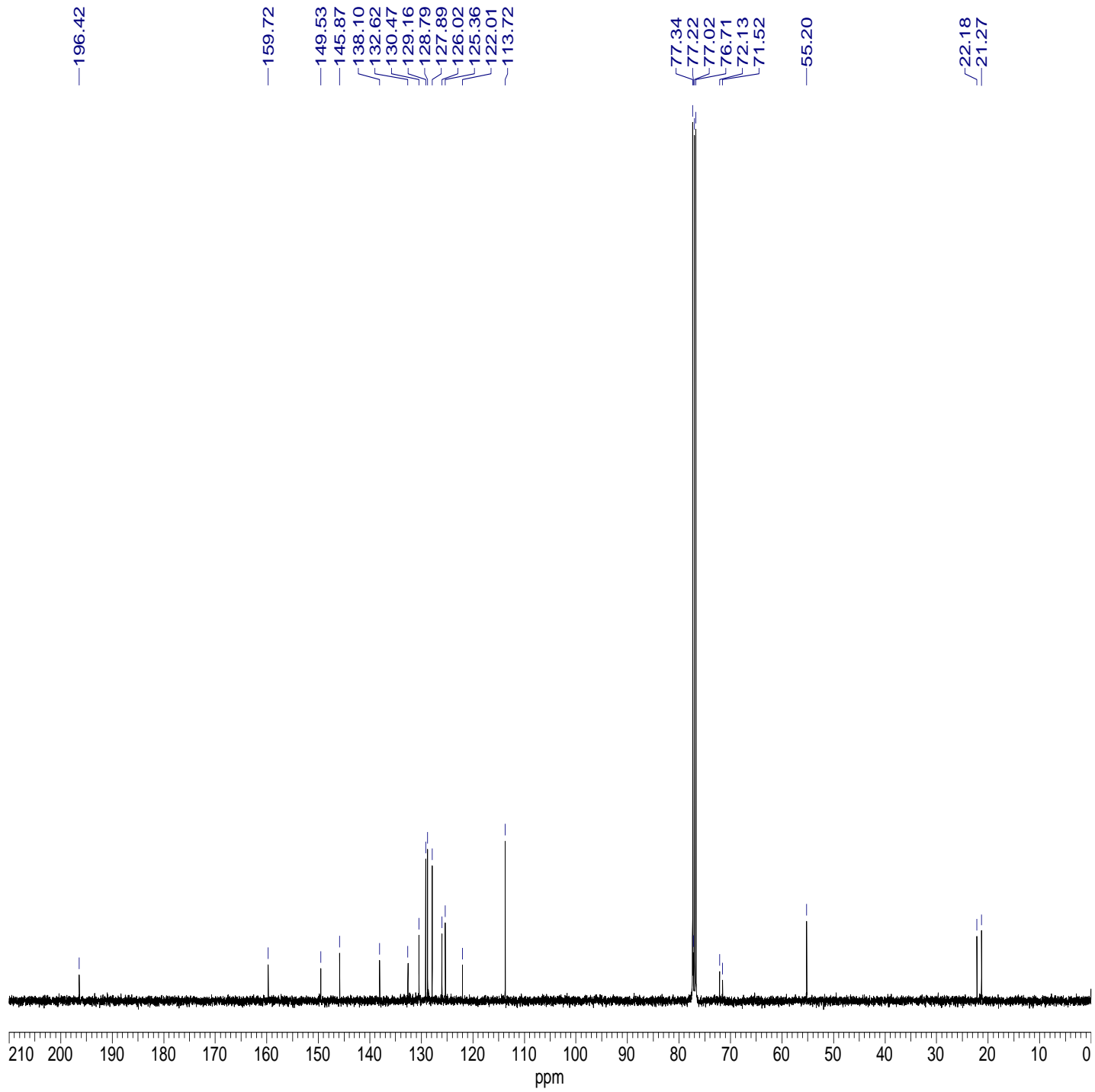
^1H (400 MHz) and ^{13}C (100 MHz) NMR in CDCl_3 of Compound 2h



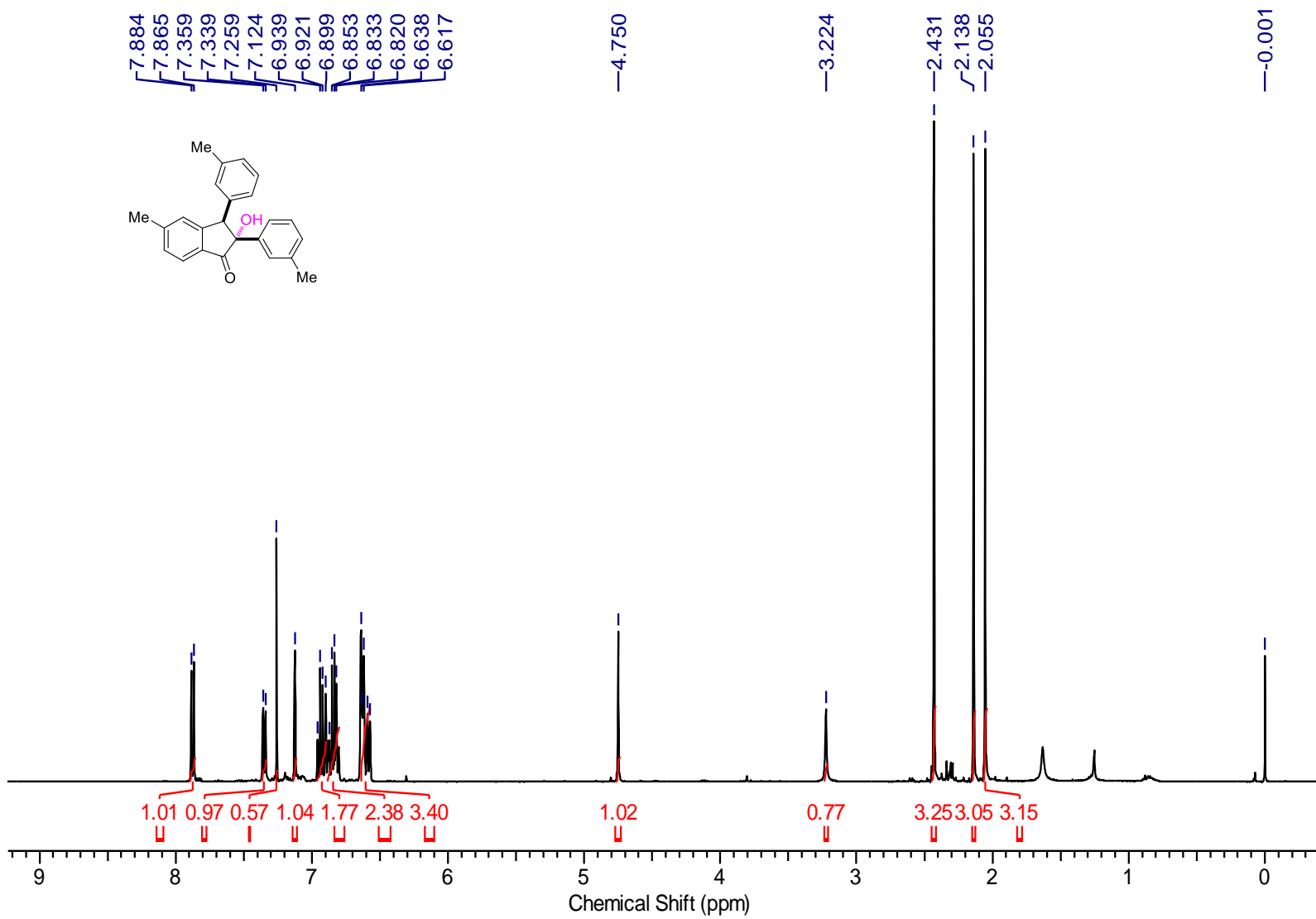


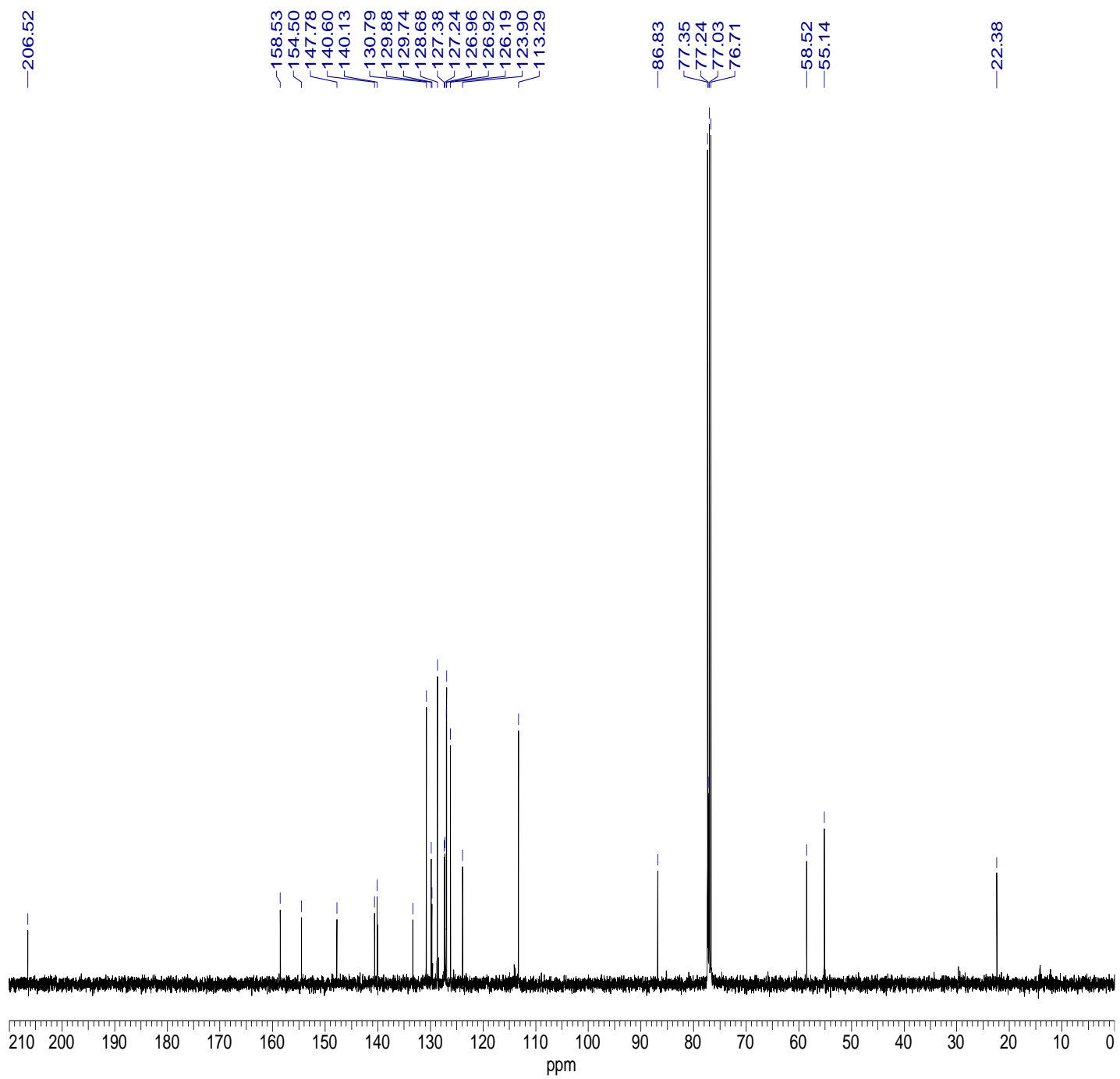
^1H (400 MHz) and ^{13}C (100 MHz) NMR in CDCl_3 of Compound 2i



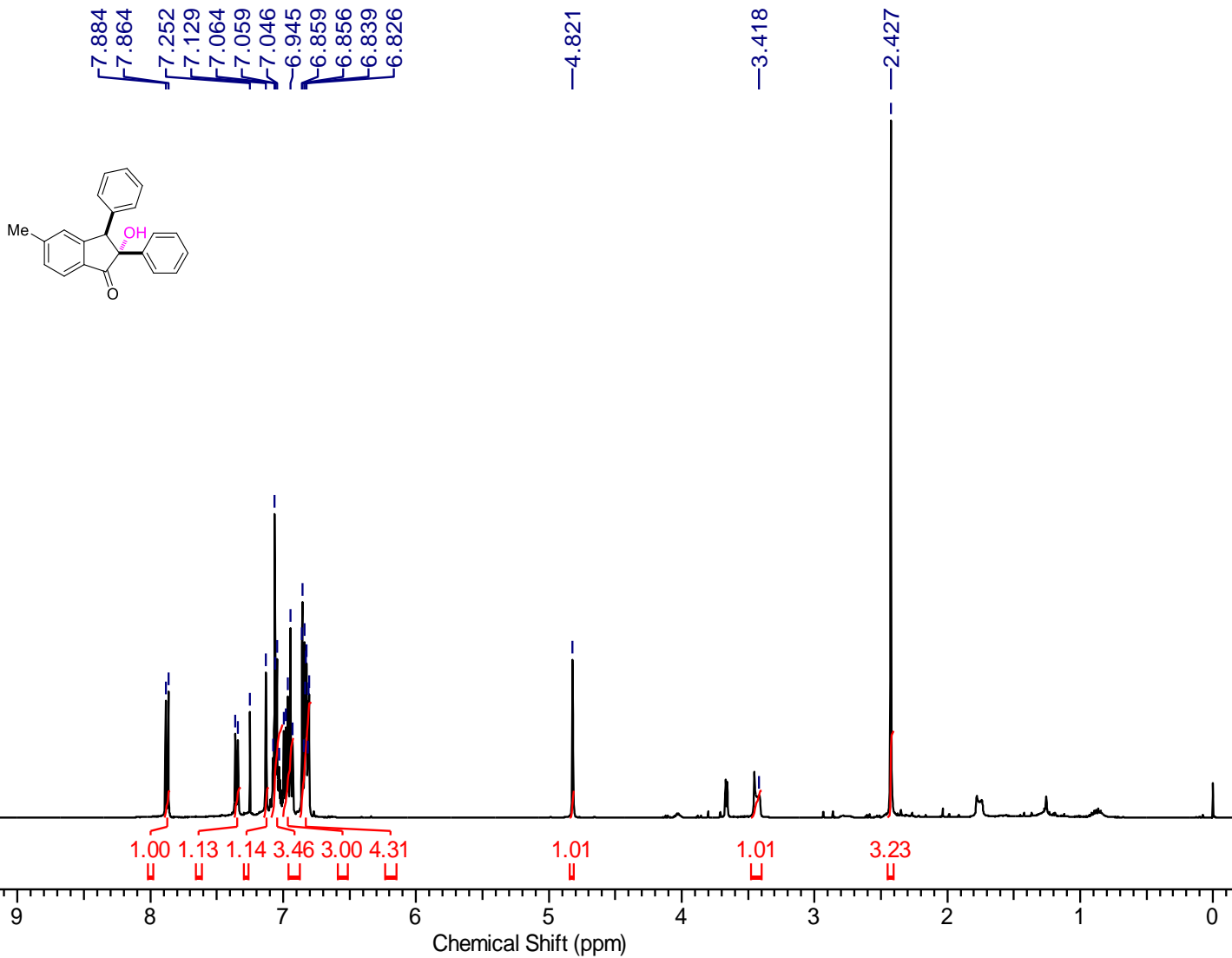


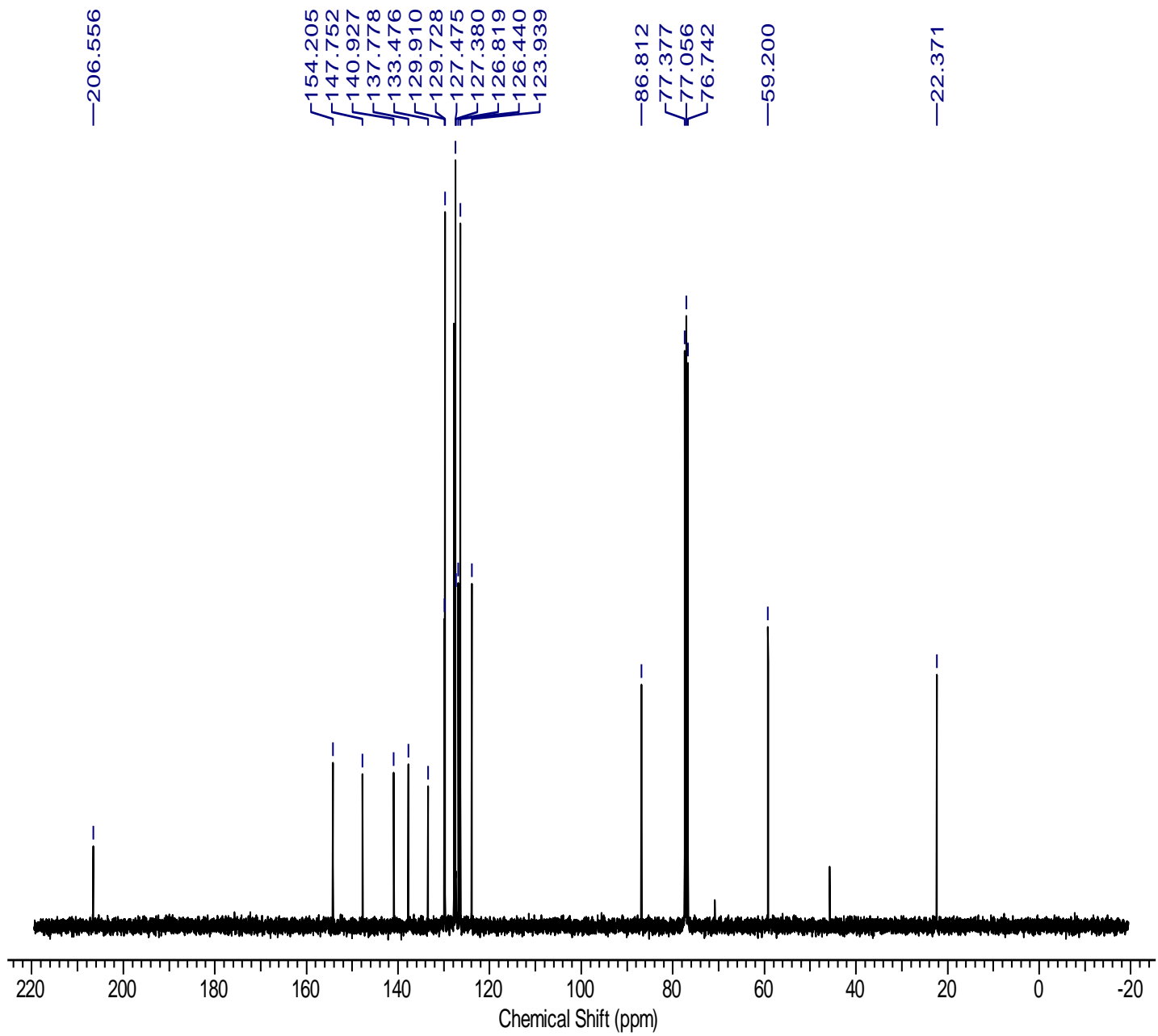
^1H (400 MHz) and ^{13}C (100 MHz) NMR in CDCl_3 of Compound 3aa



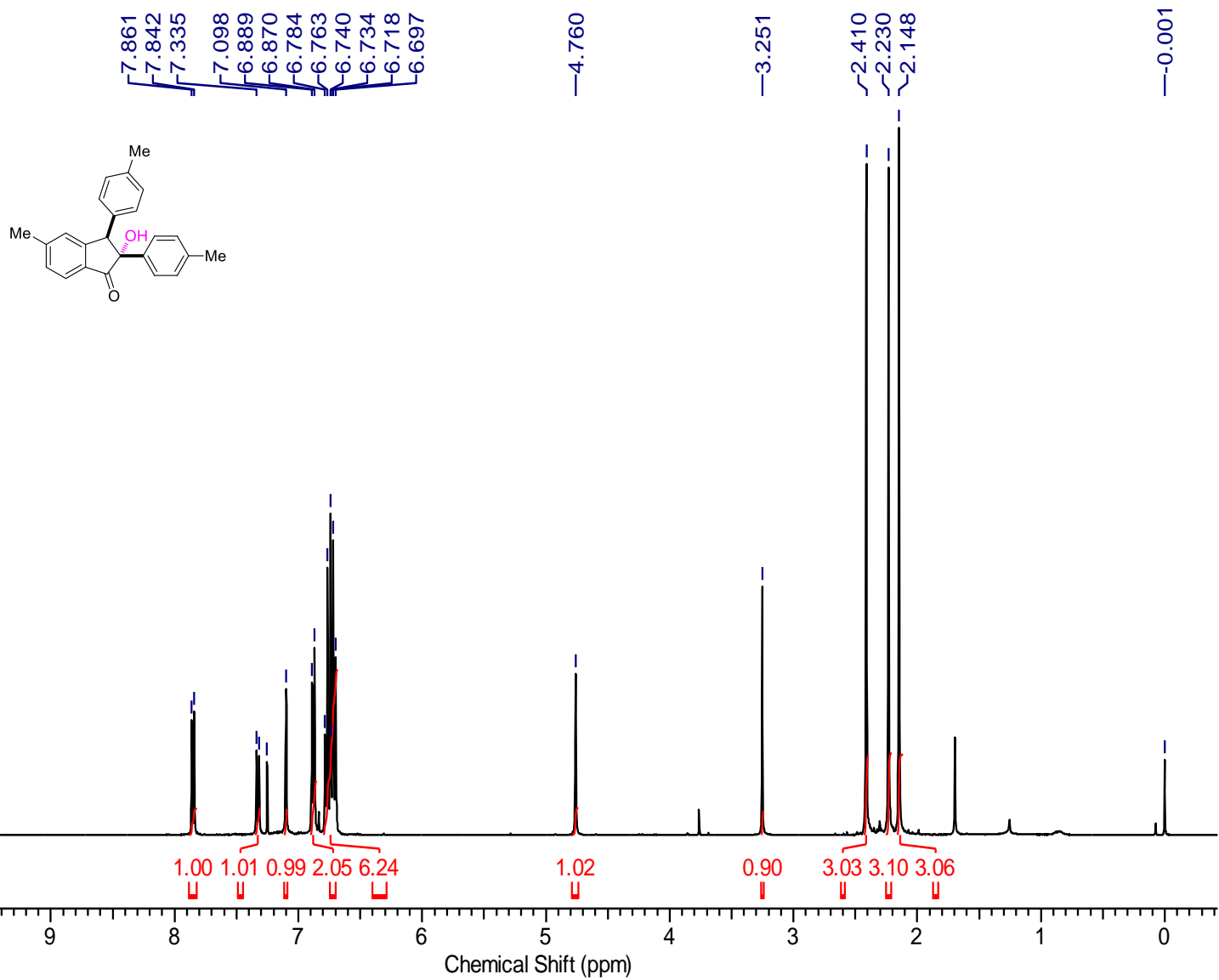


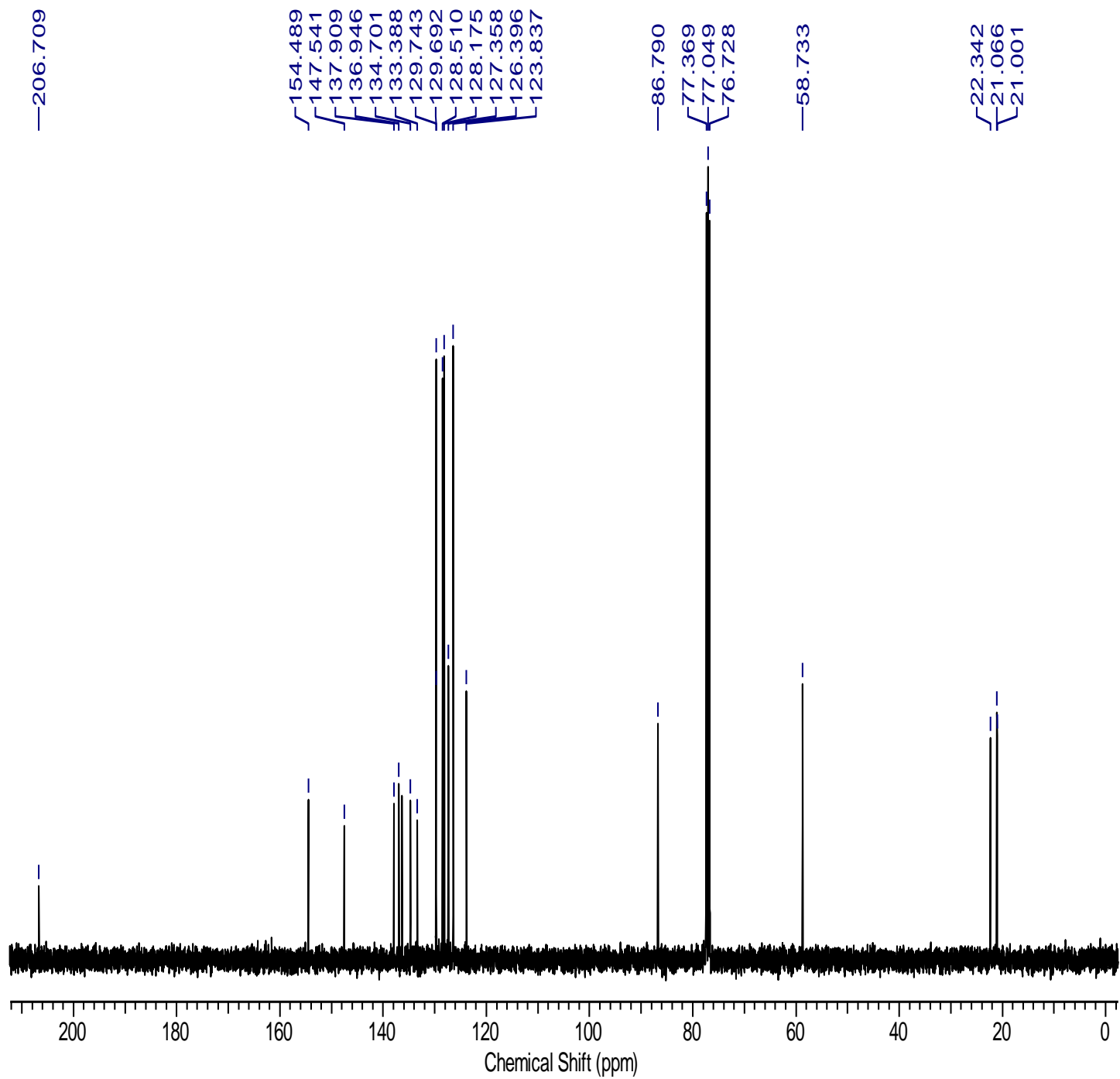
^1H (400 MHz) and ^{13}C (100 MHz) NMR in CDCl_3 of Compound 3ab



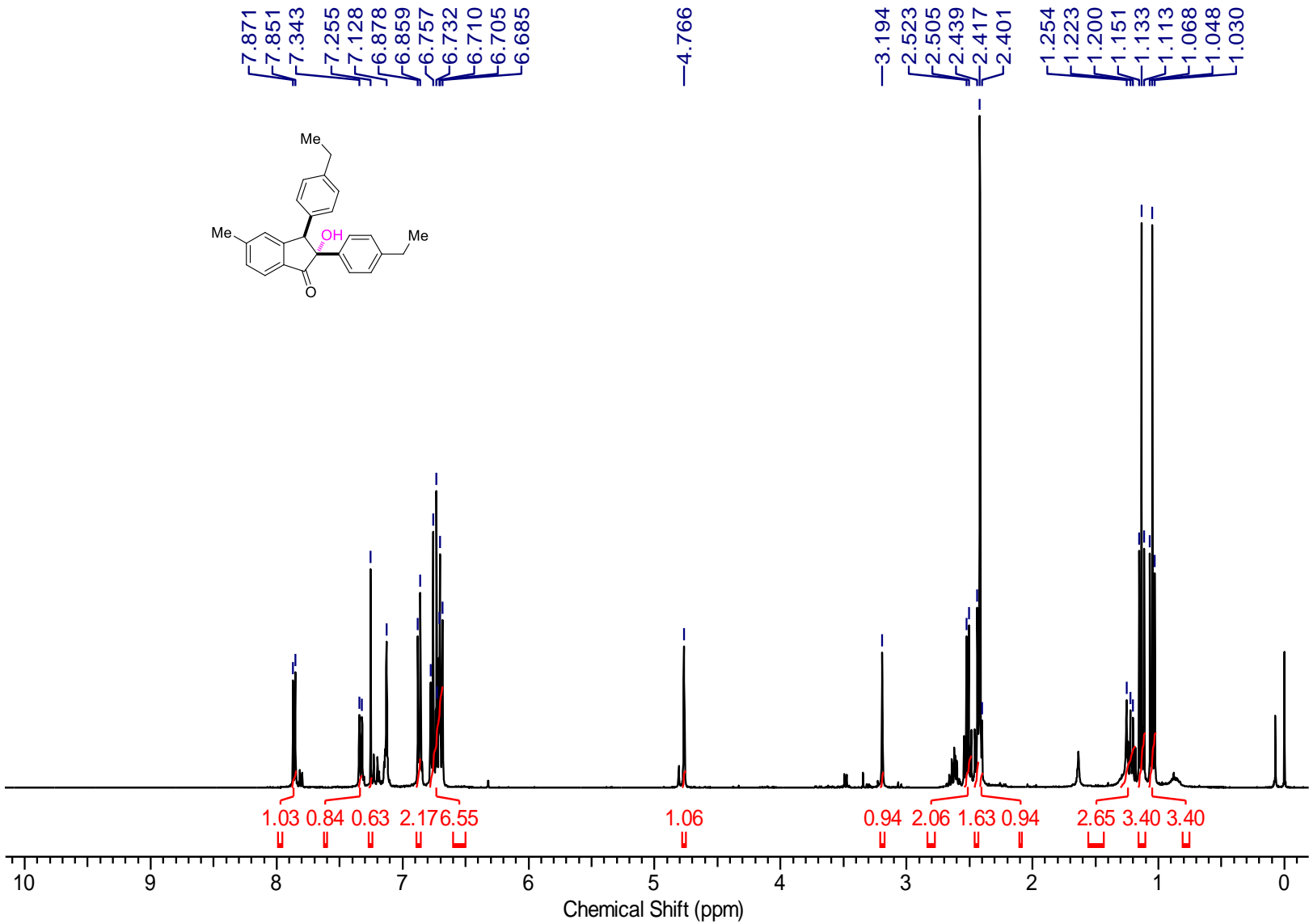


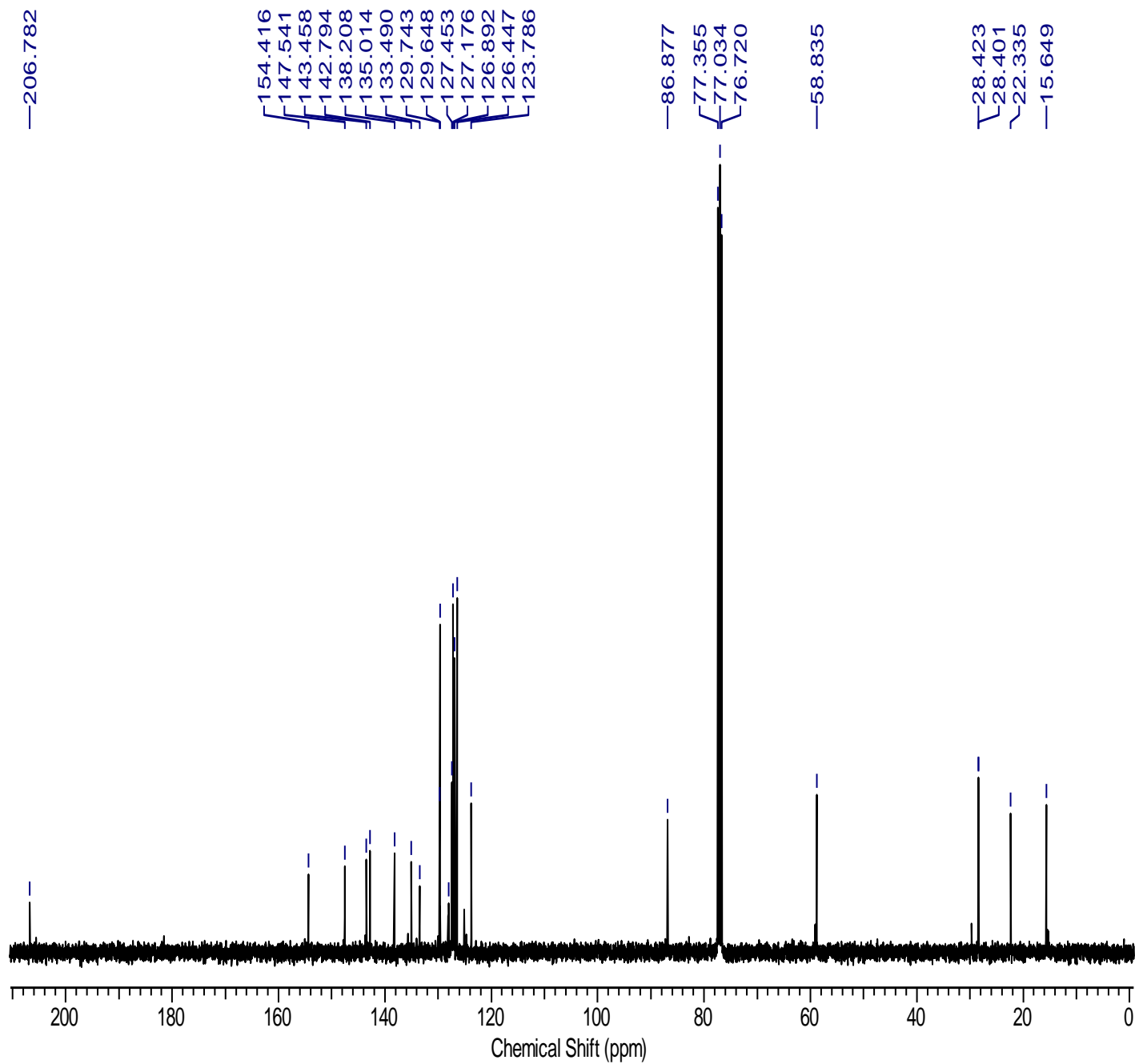
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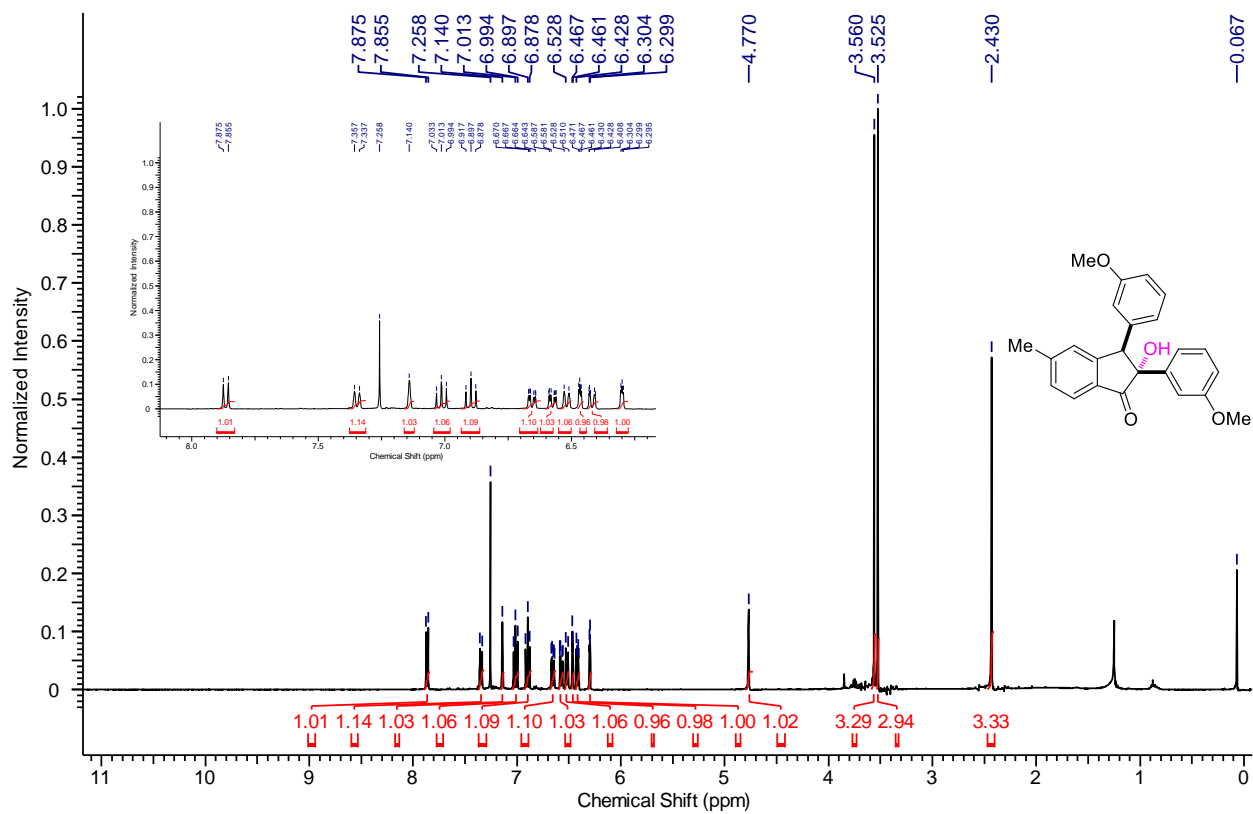


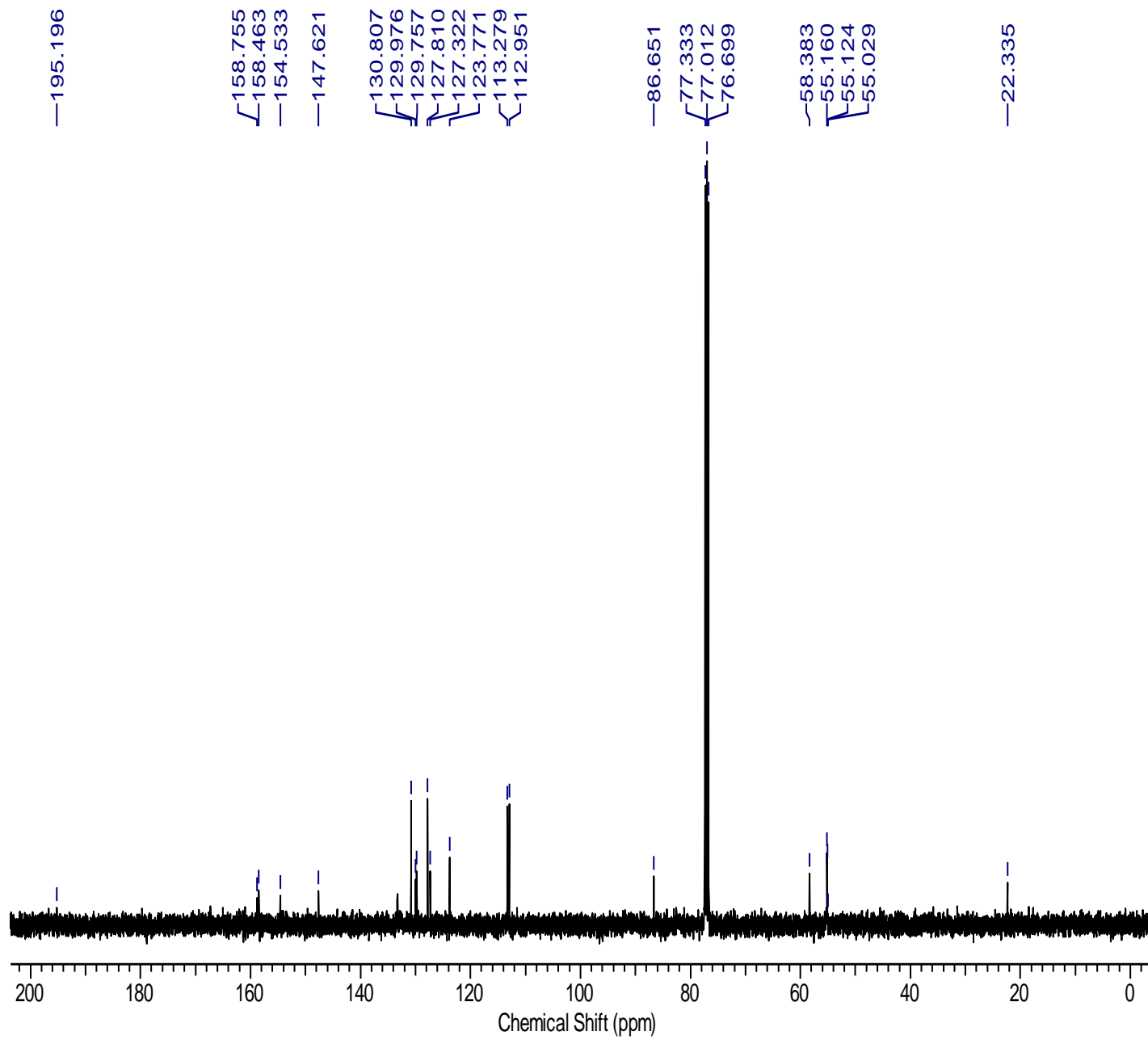
¹H (400 MHz) and ¹³C (100 MHz) NMR in CDCl₃ of Compound 3ad





¹H (400 MHz) and ¹³C (100 MHz) NMR in CDCl₃ of Compound 3ae





¹H (400 MHz) and ¹³C (100 MHz) NMR in CDCl₃ of Compound 3af

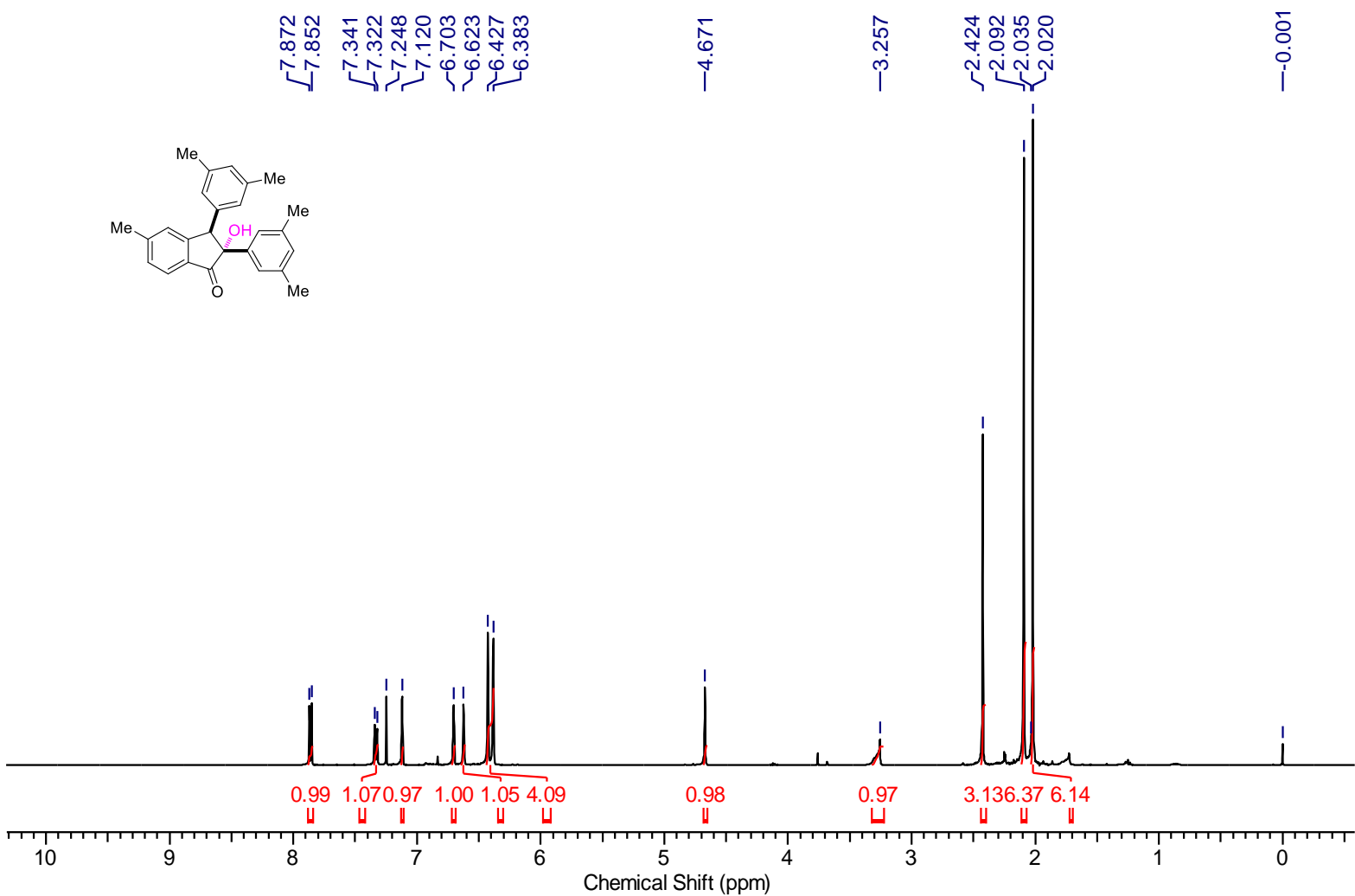
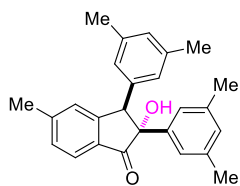
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7.120
6.703
6.623
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6.383

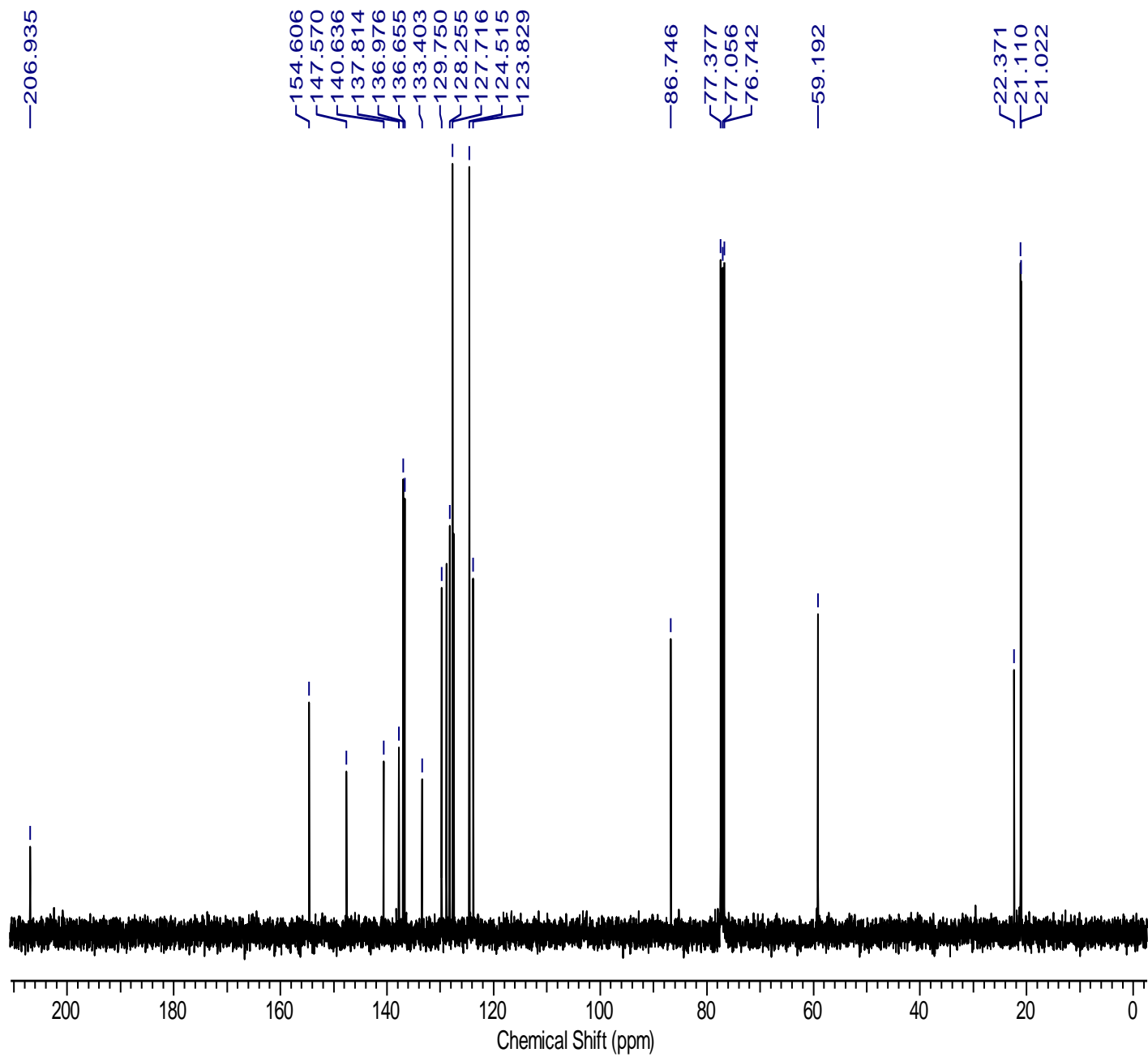
4.671

3.257

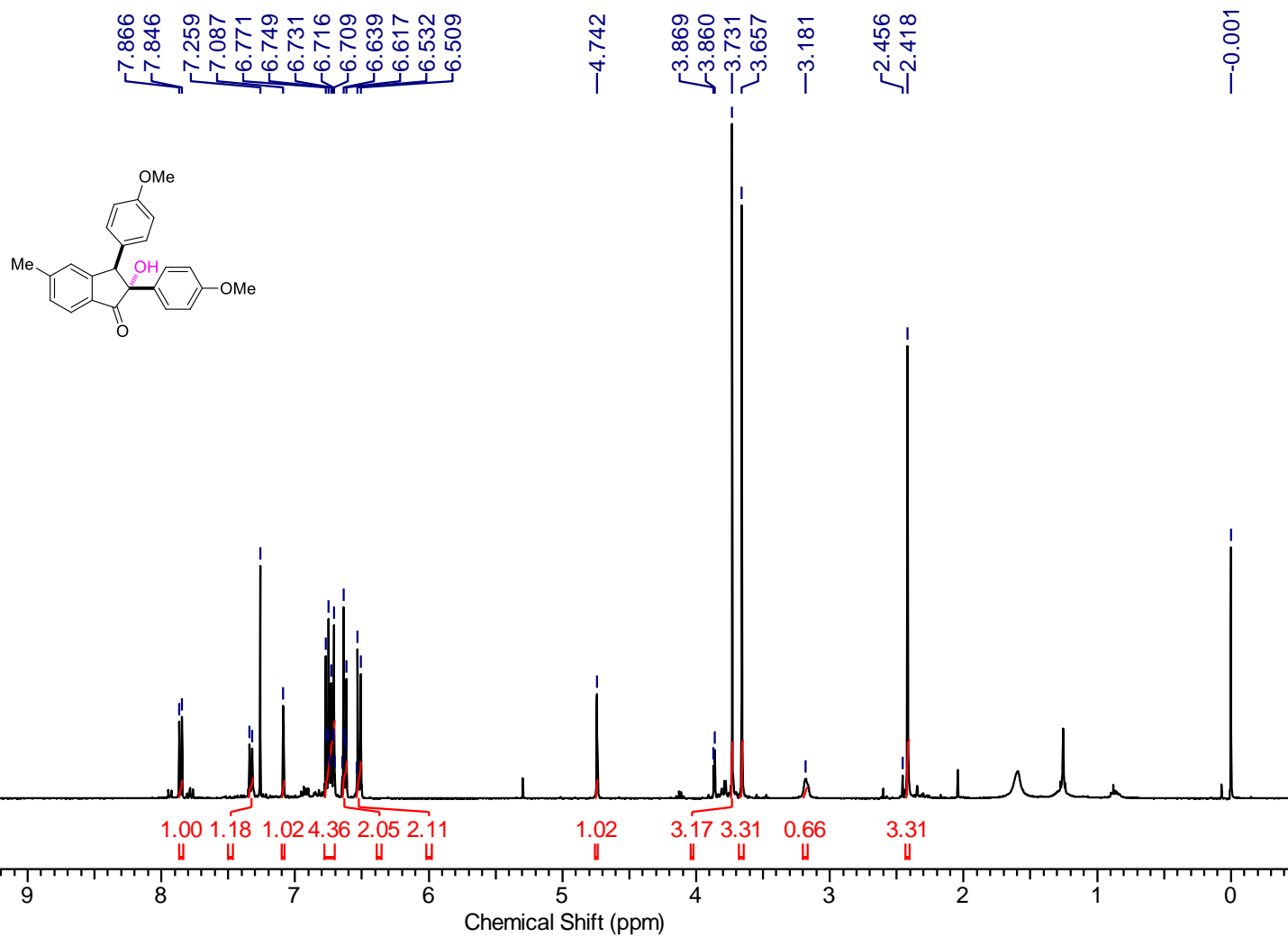
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2.092
2.035
2.020

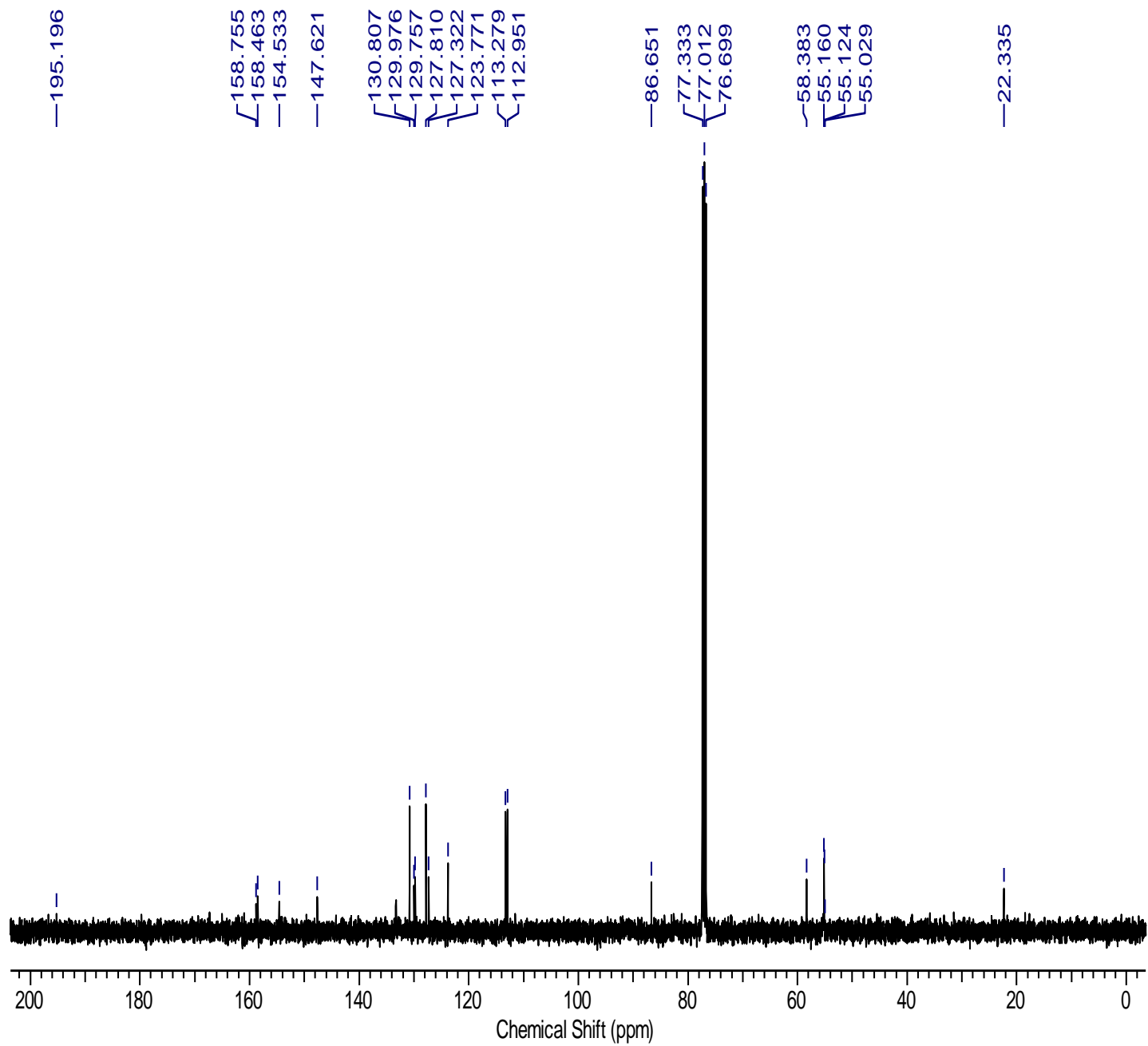
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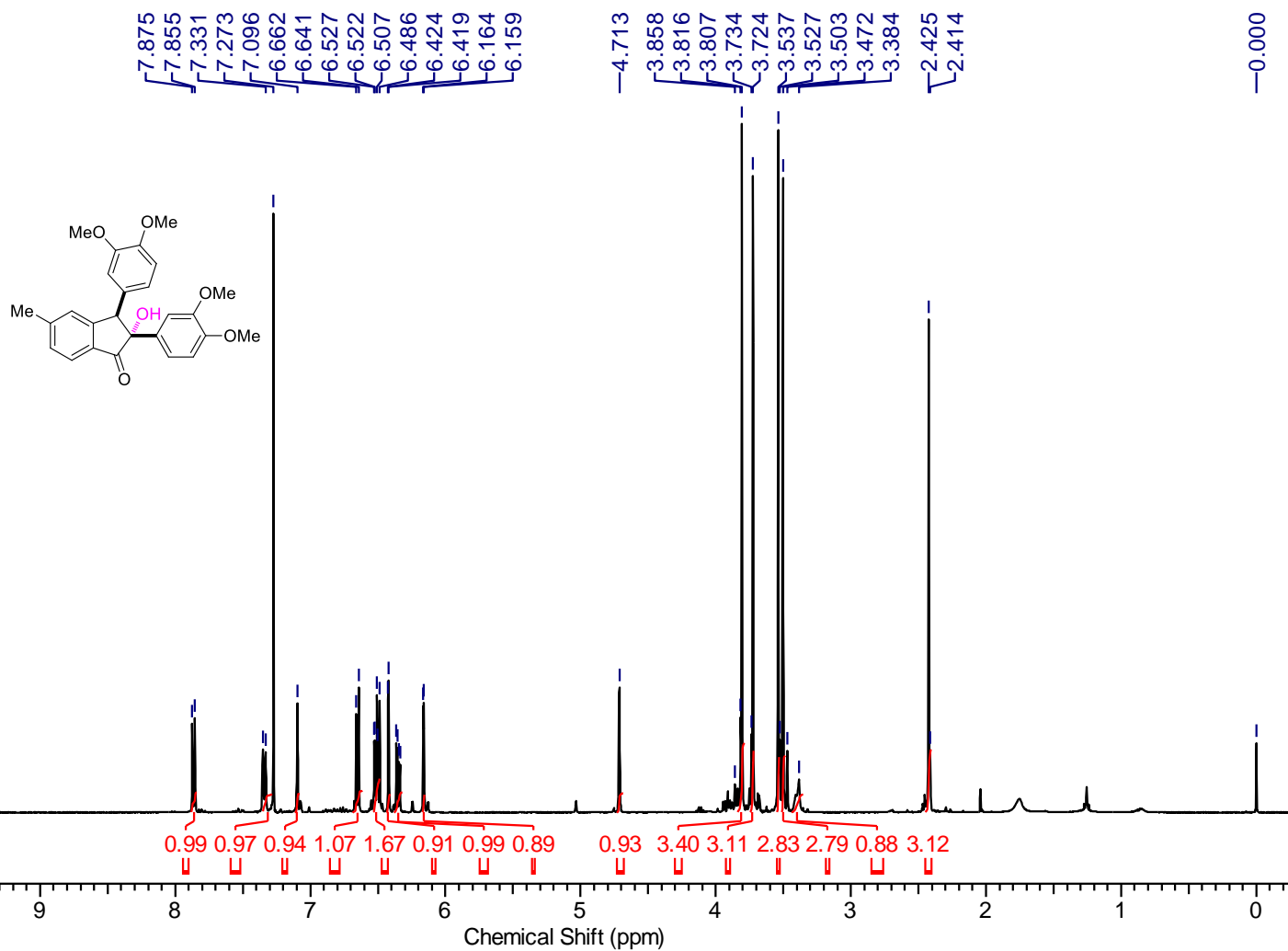


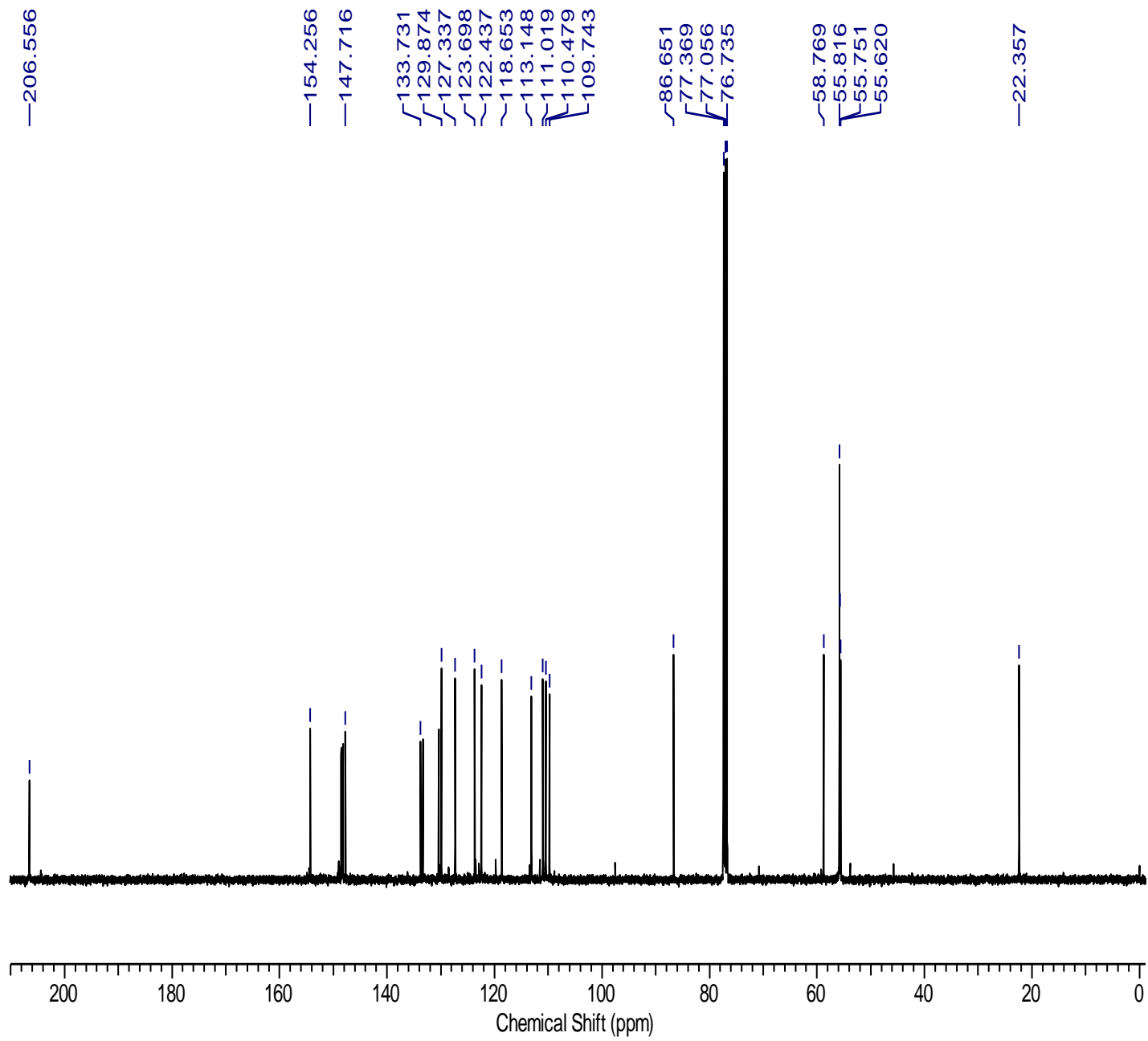
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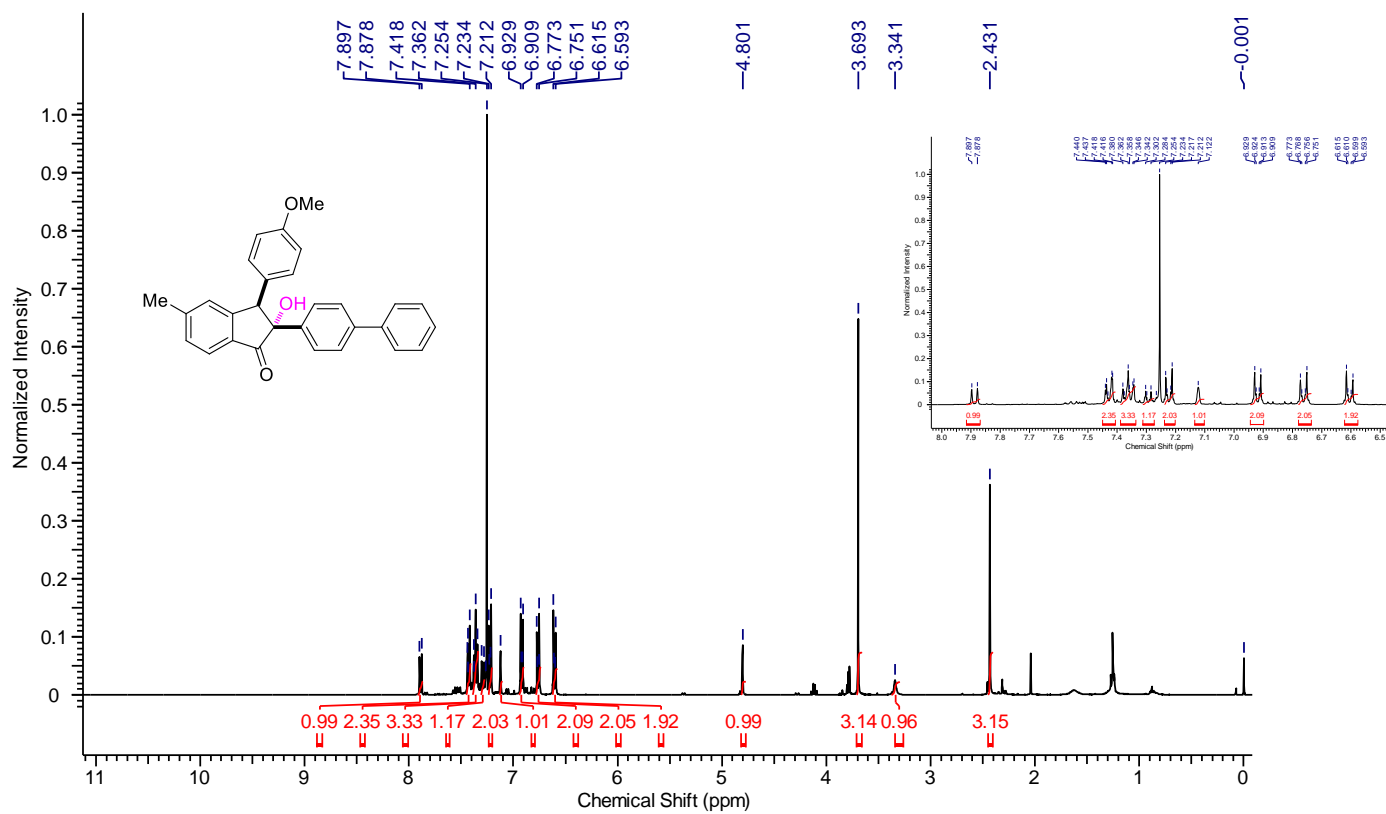


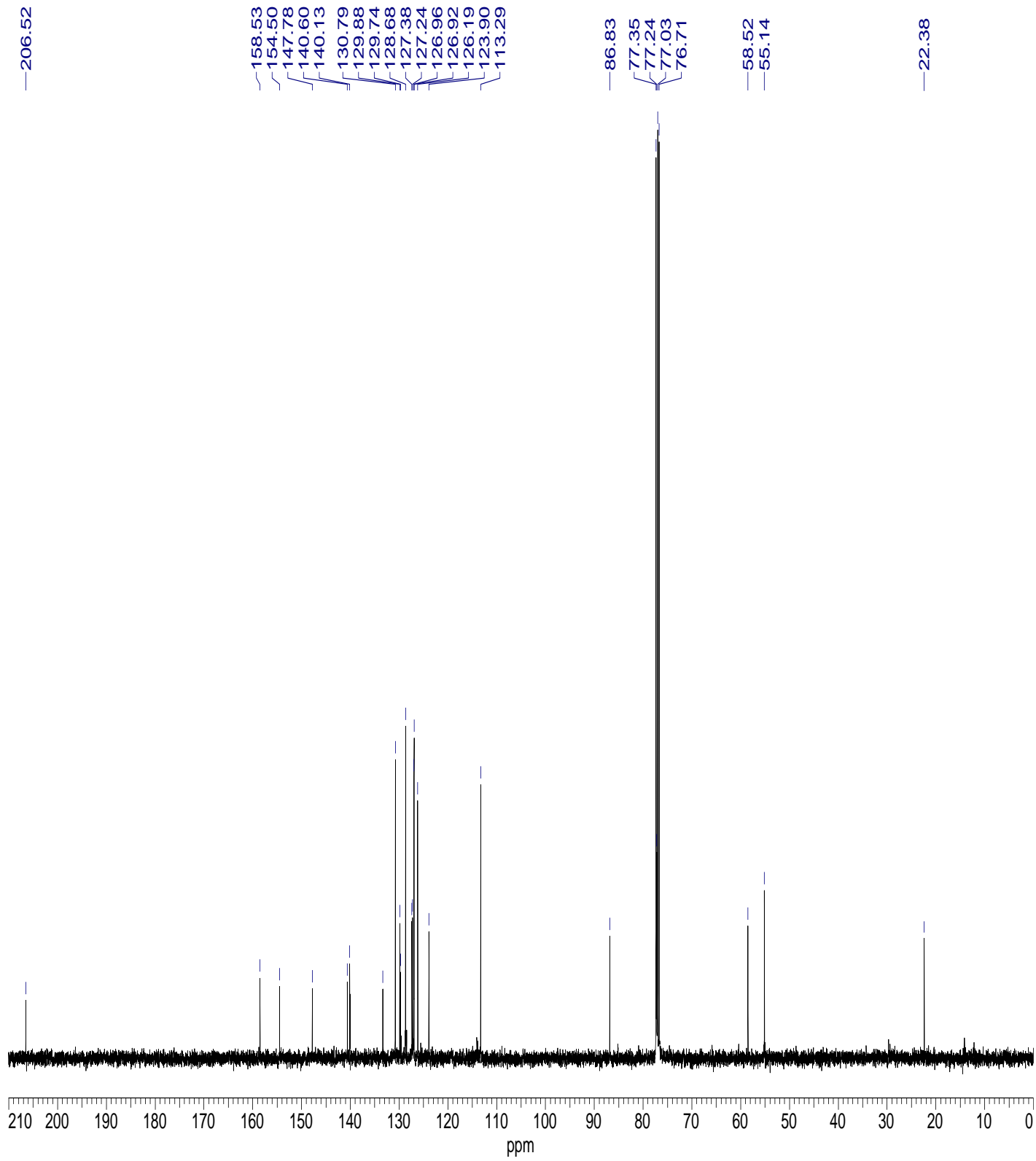
^1H (400 MHz) and ^{13}C (100 MHz) NMR in CDCl_3 of Compound 3ah





^1H (400 MHz) and ^{13}C (100 MHz) NMR in CDCl_3 of Compound 3ai





^1H (400 MHz) and ^{13}C (100 MHz) NMR in CDCl_3 of Compound 3aj

7.80
7.78
7.27
7.25
7.19
7.03
6.66
6.63
6.57
6.55
6.53
6.35

4.66

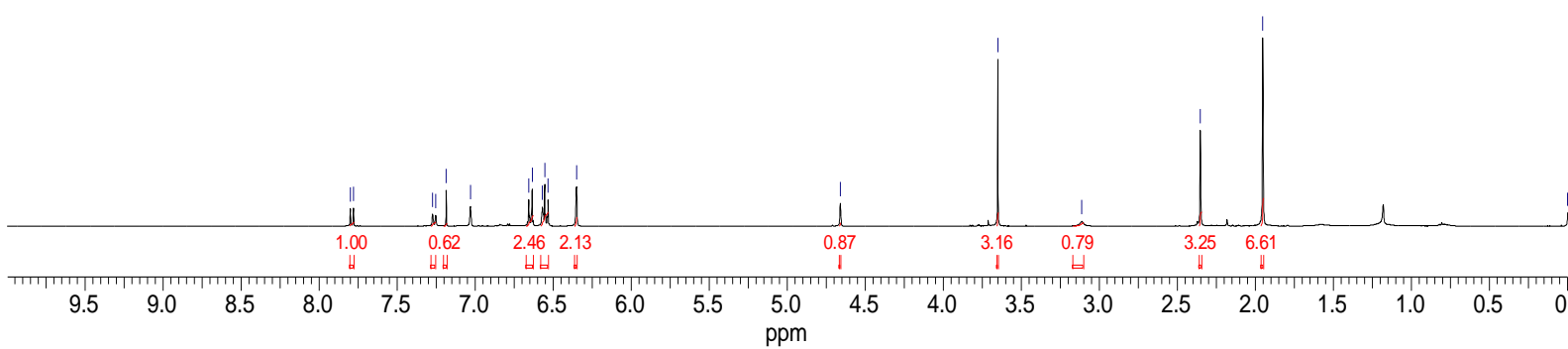
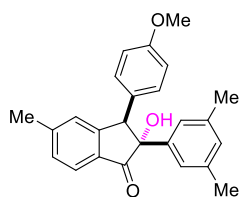
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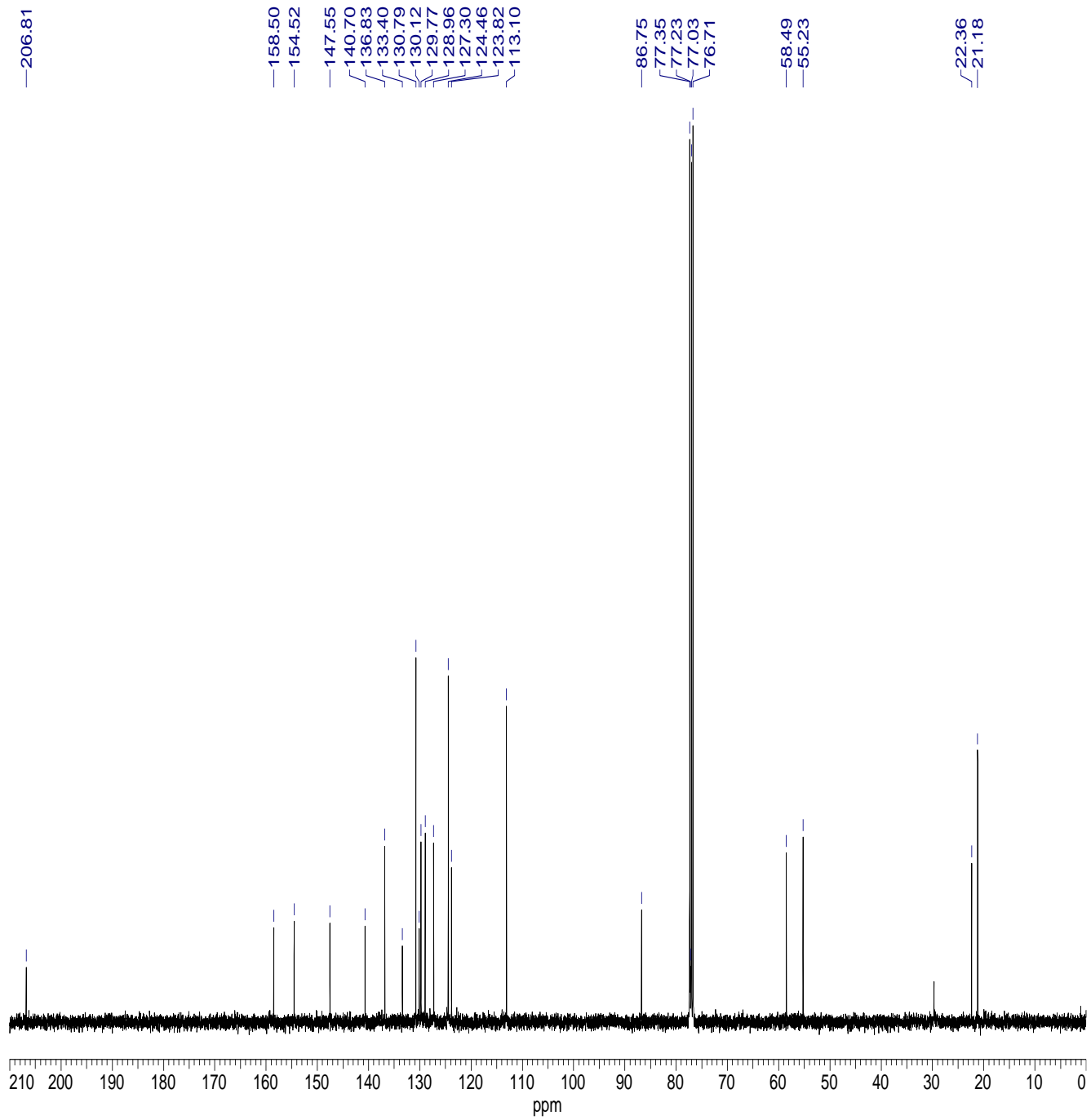
3.11

2.35

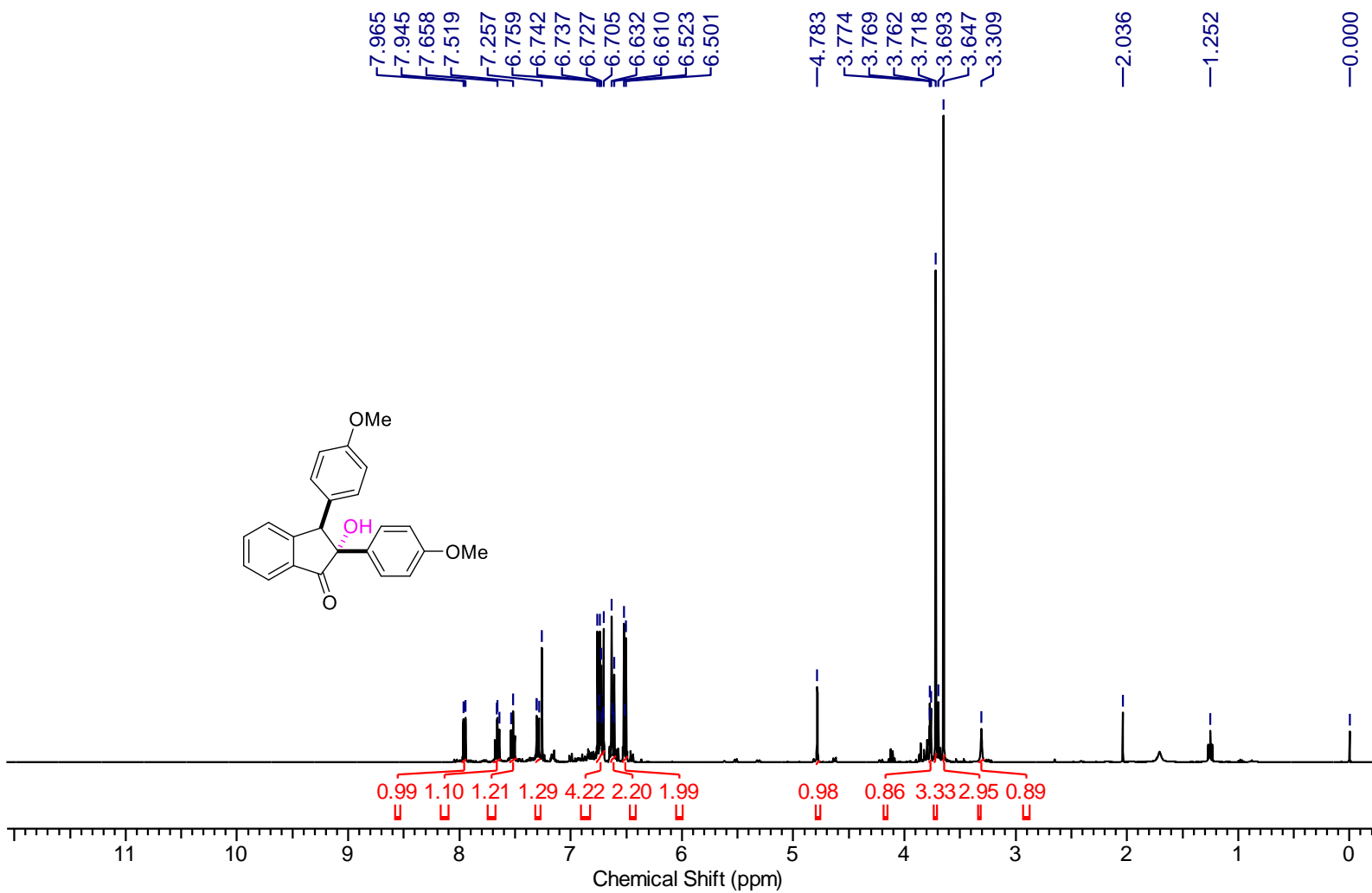
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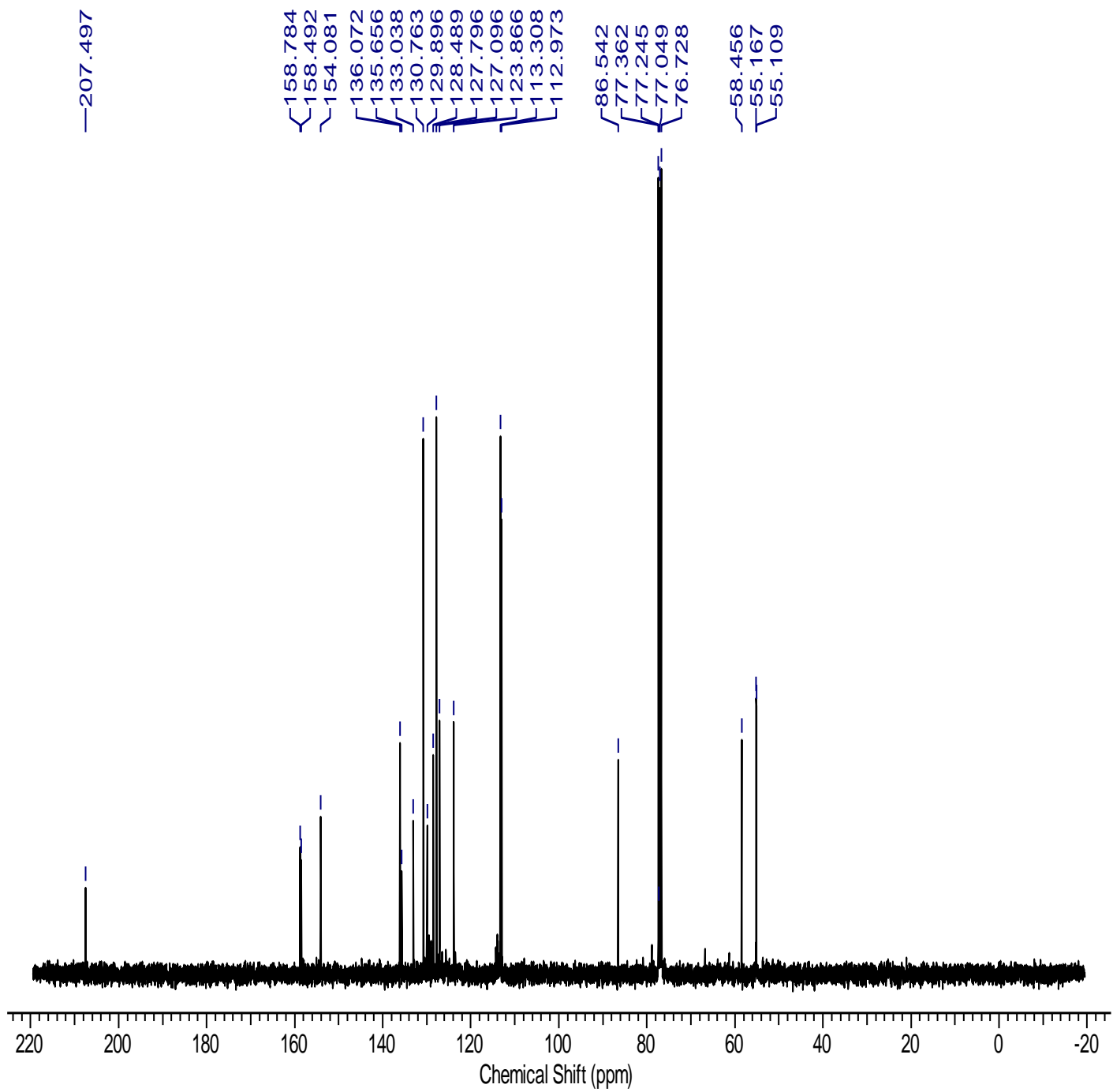
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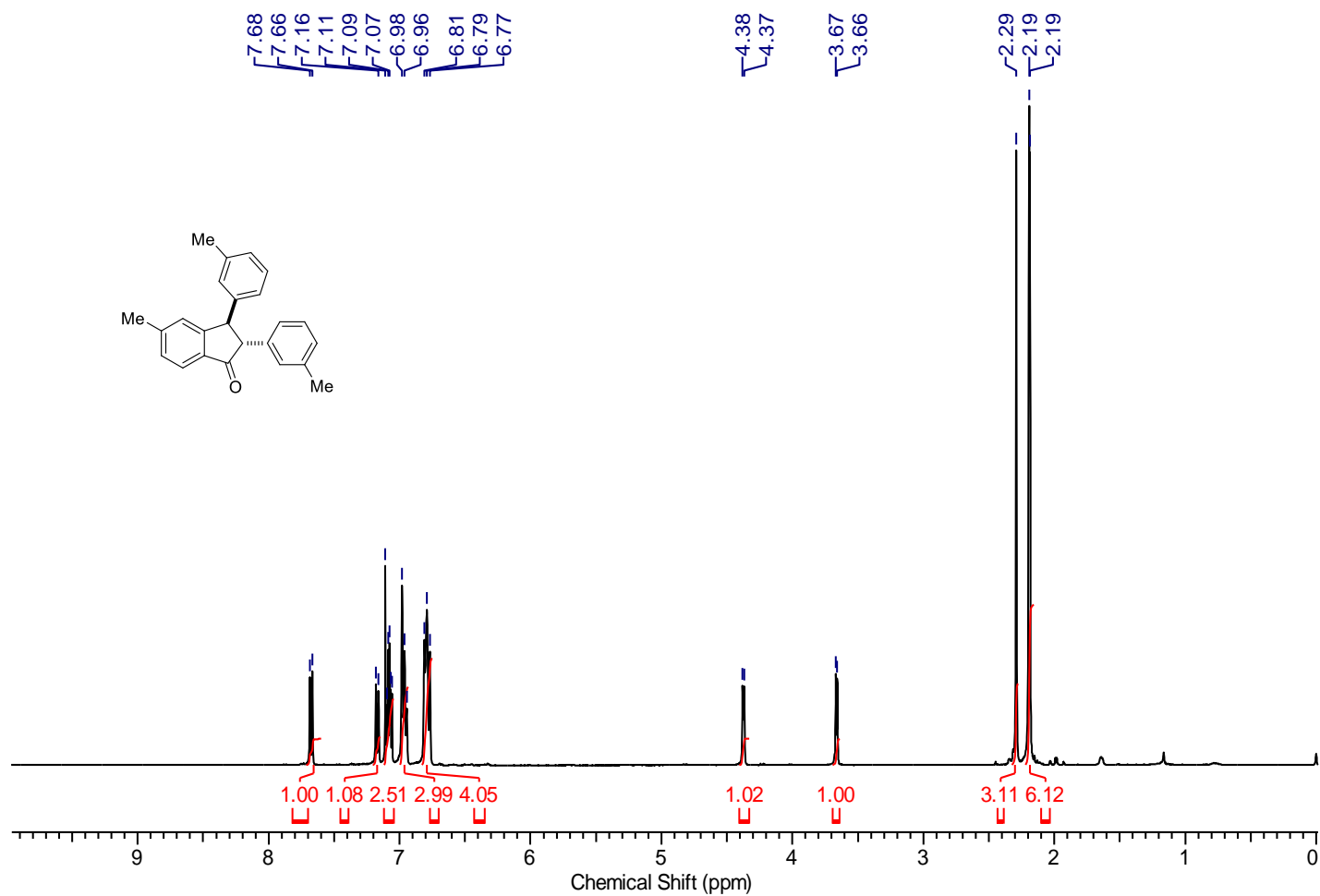


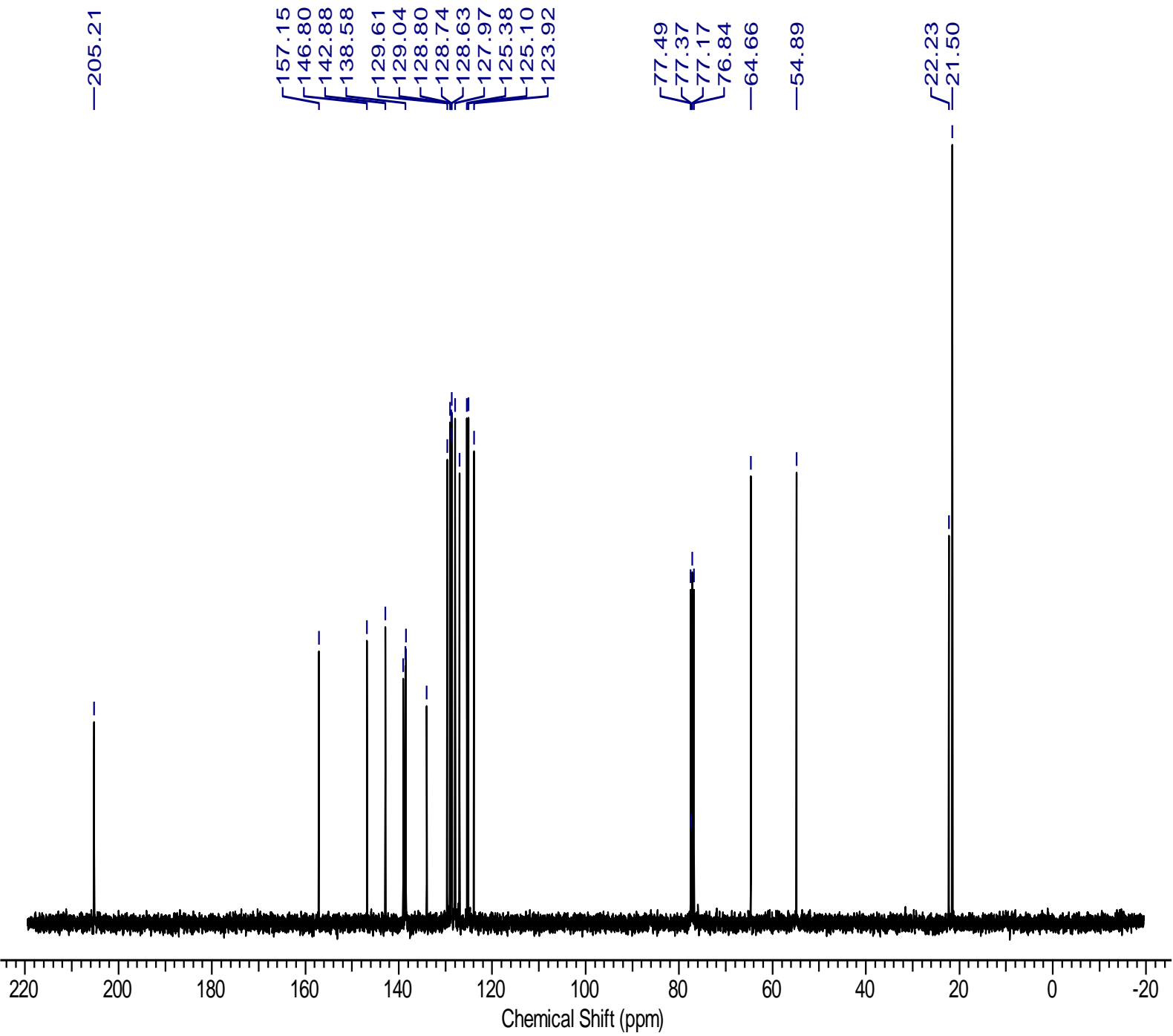
¹H (400 MHz) and ¹³C (100 MHz) NMR in CDCl₃ of Compound 3ak



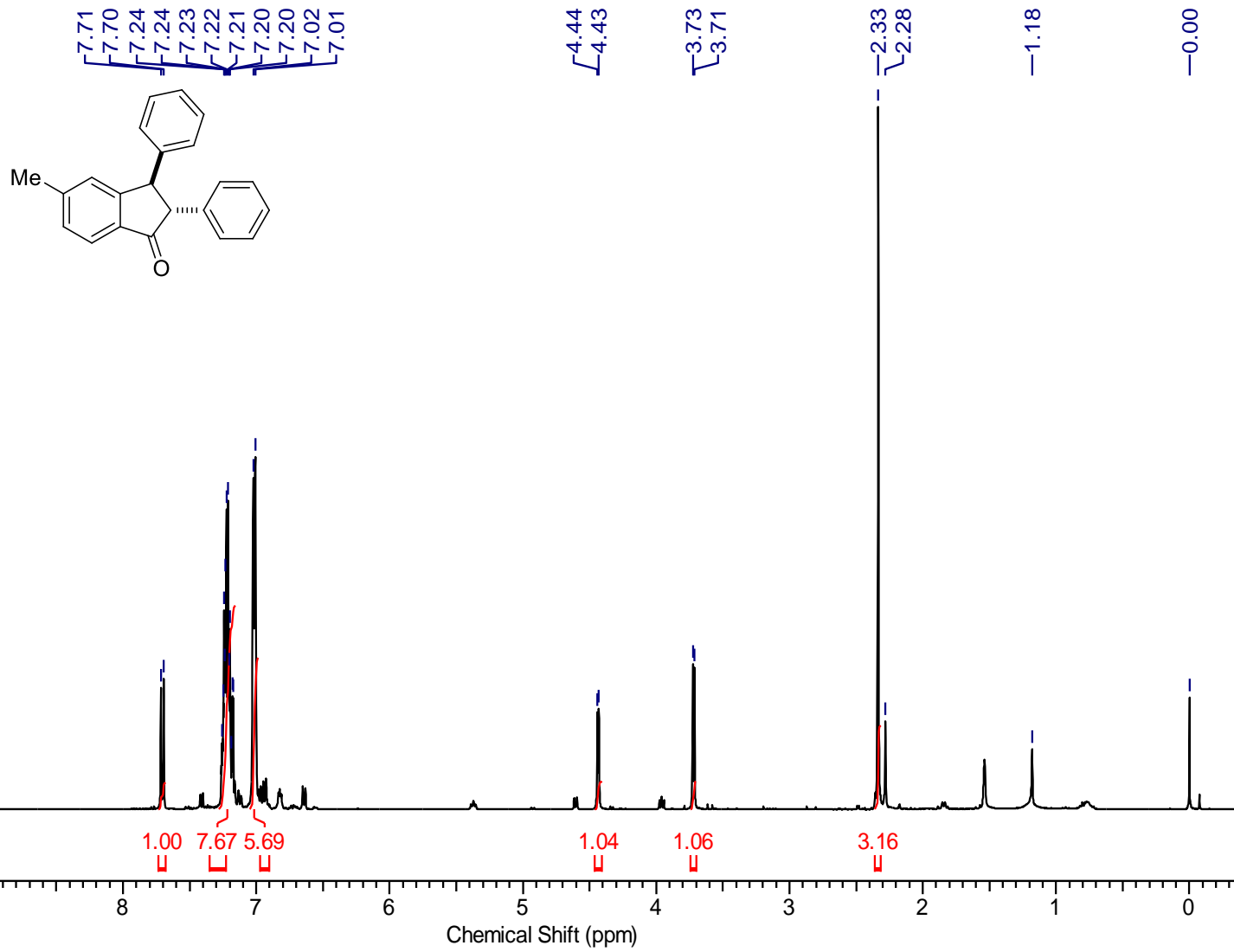


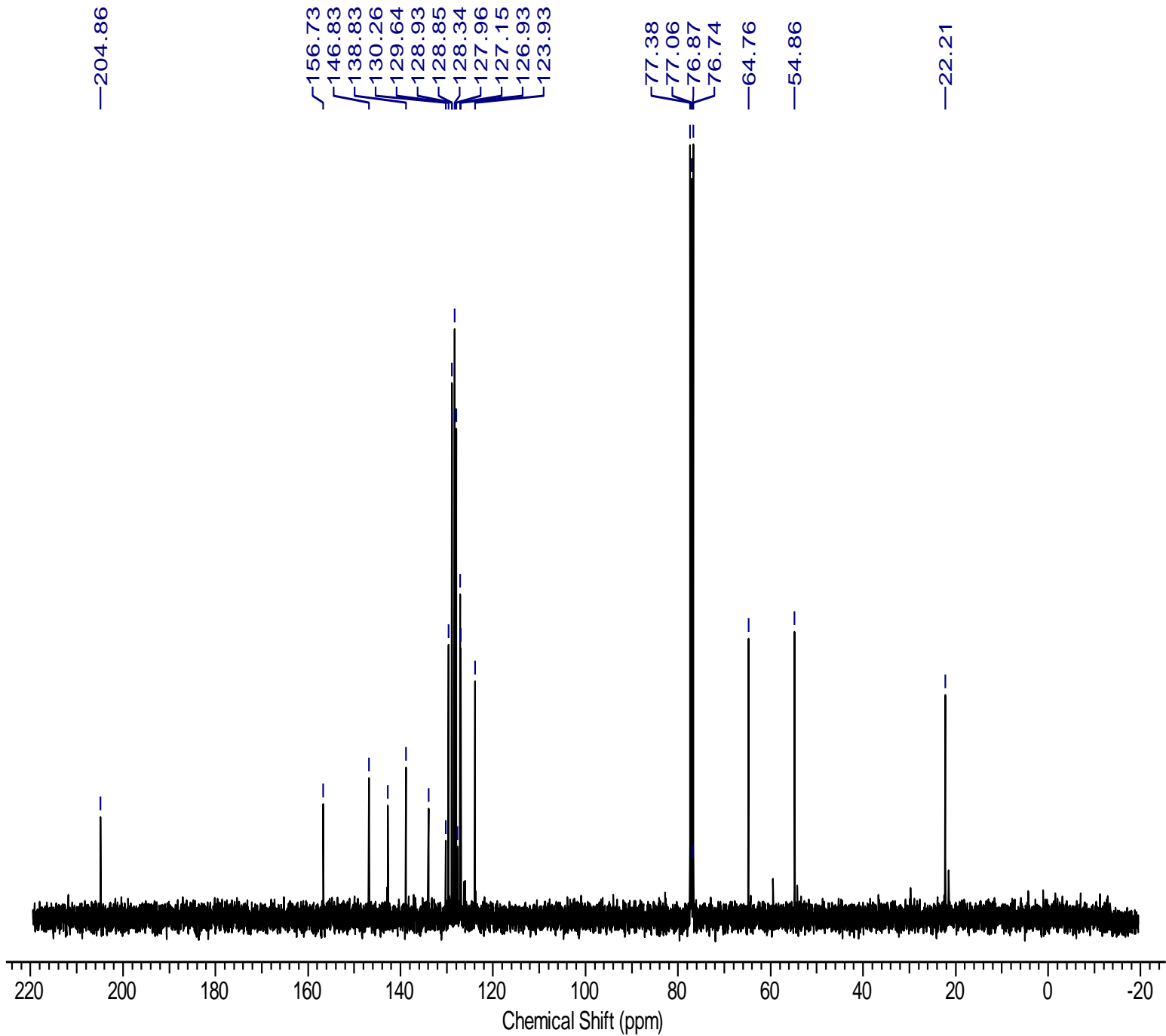
^1H (400 MHz) and ^{13}C (100 MHz) NMR in CDCl_3 of Compound 1a



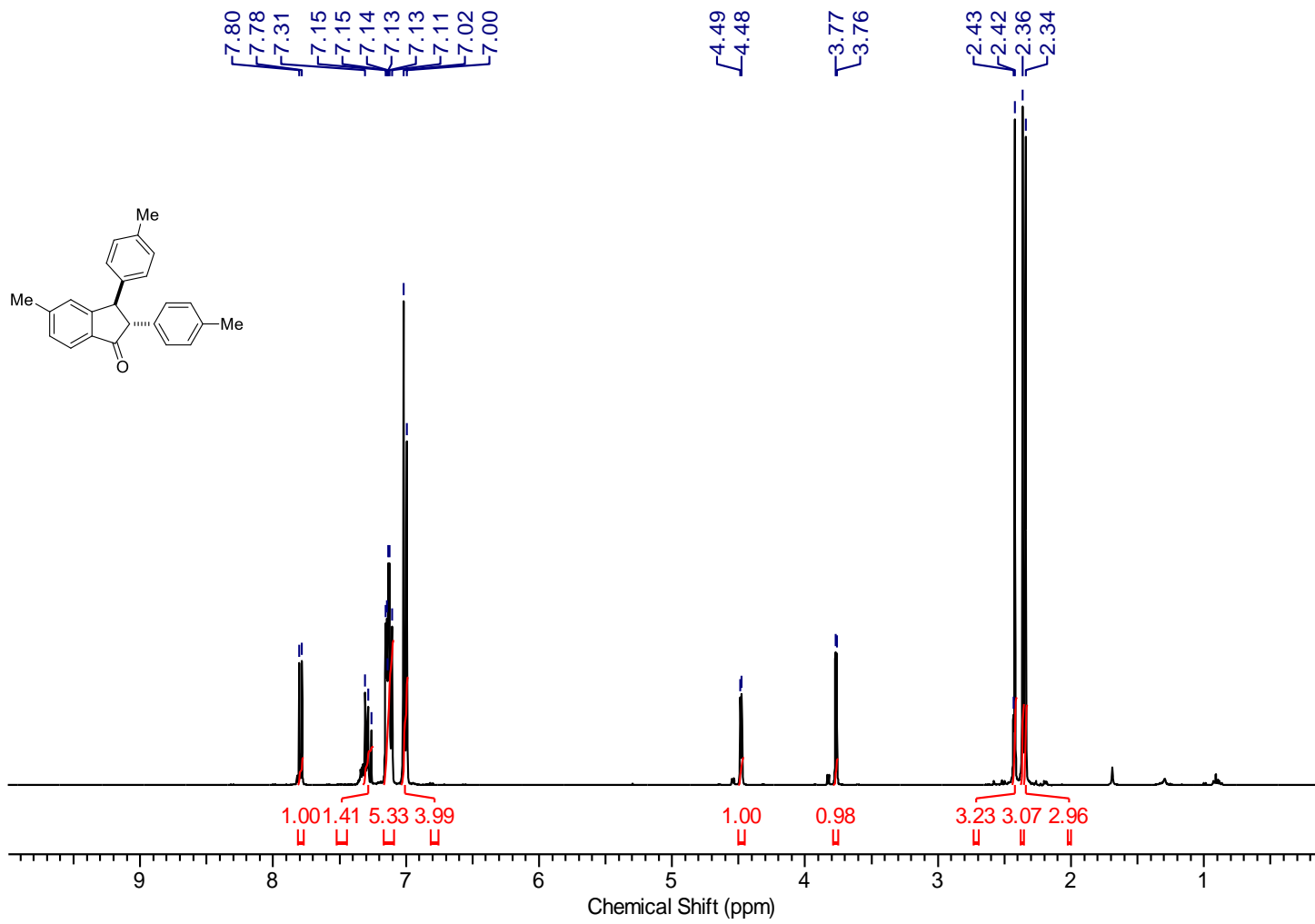


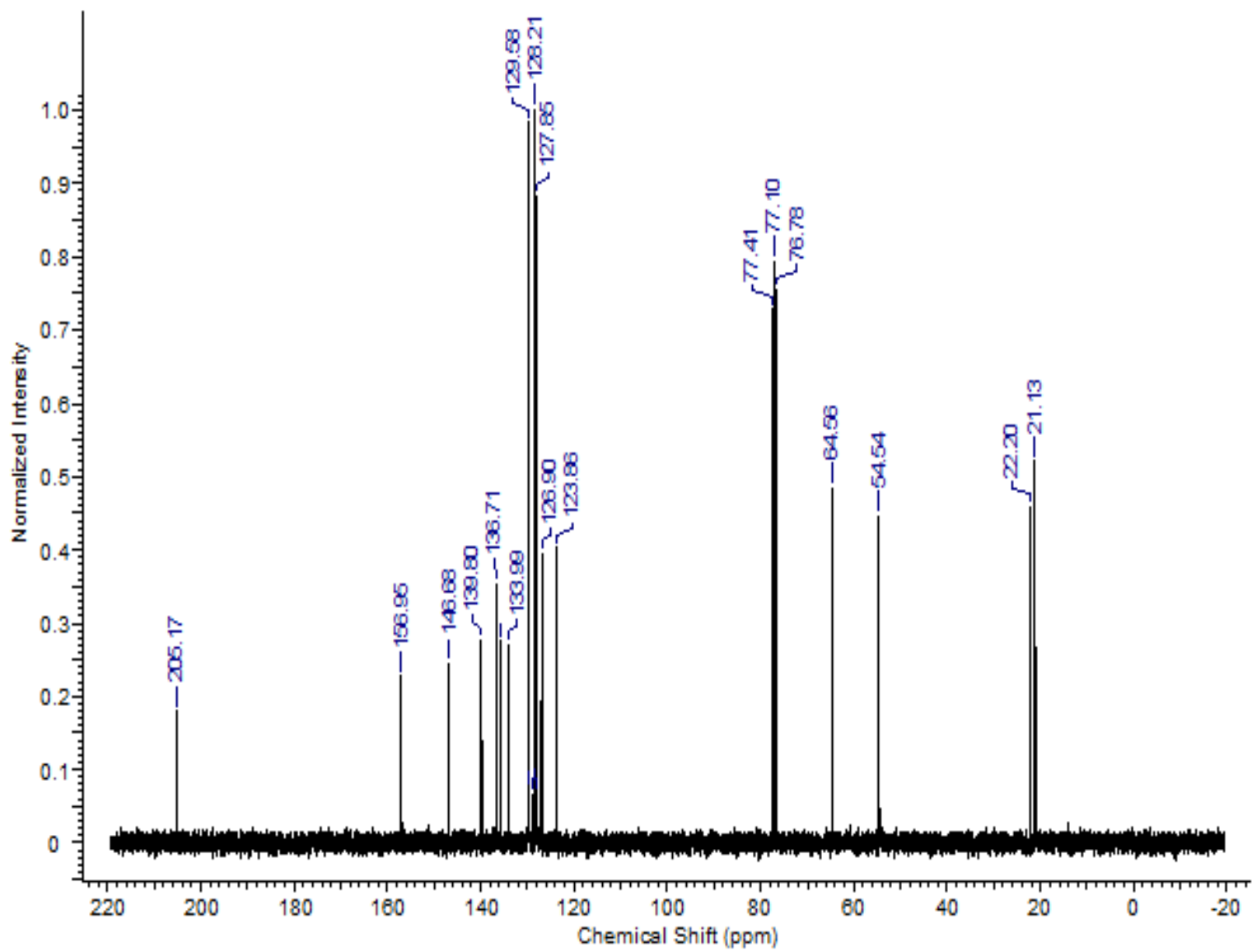
^1H (400 MHz) and ^{13}C (100 MHz) NMR in CDCl_3 of Compound 1b



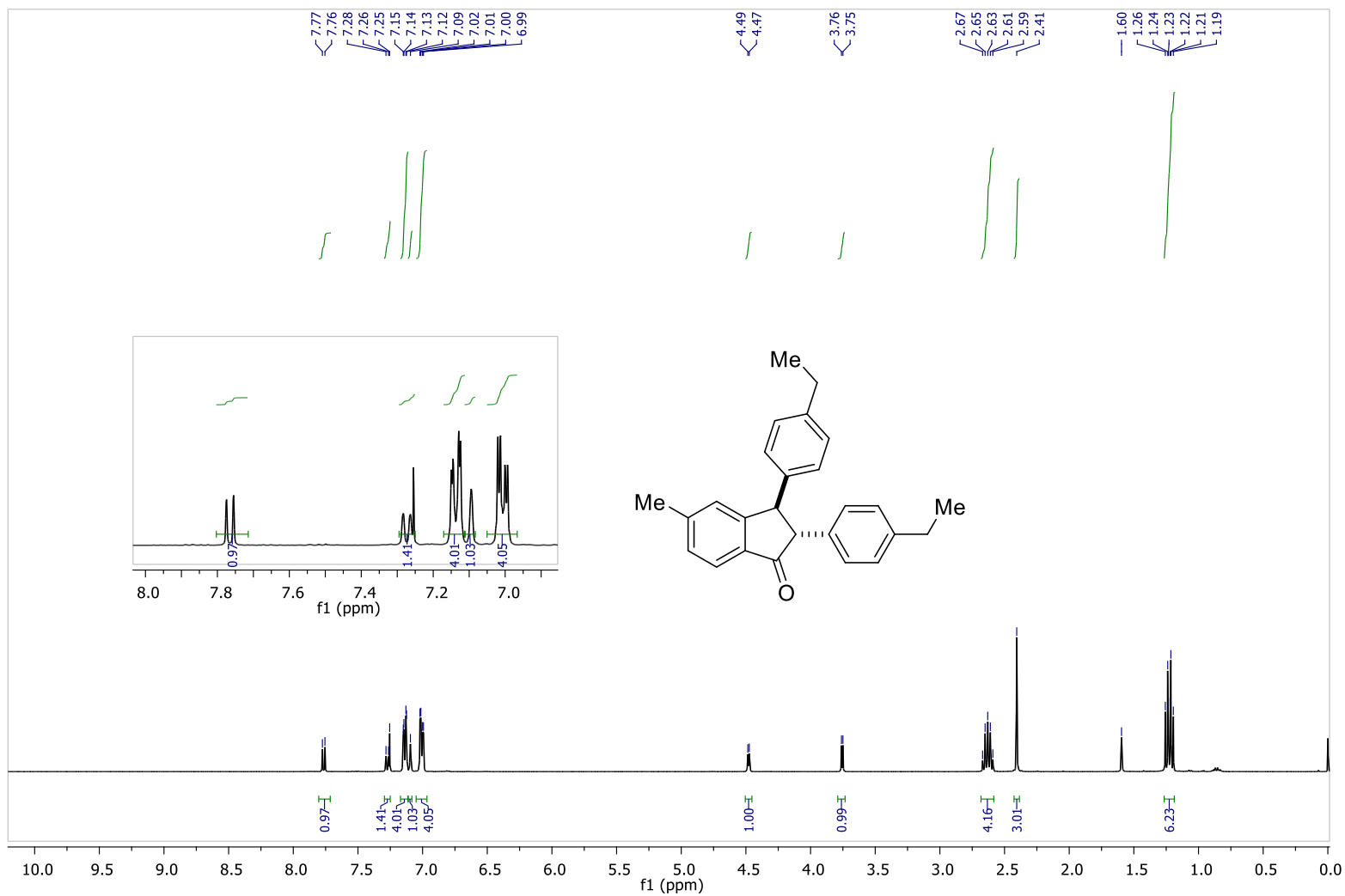


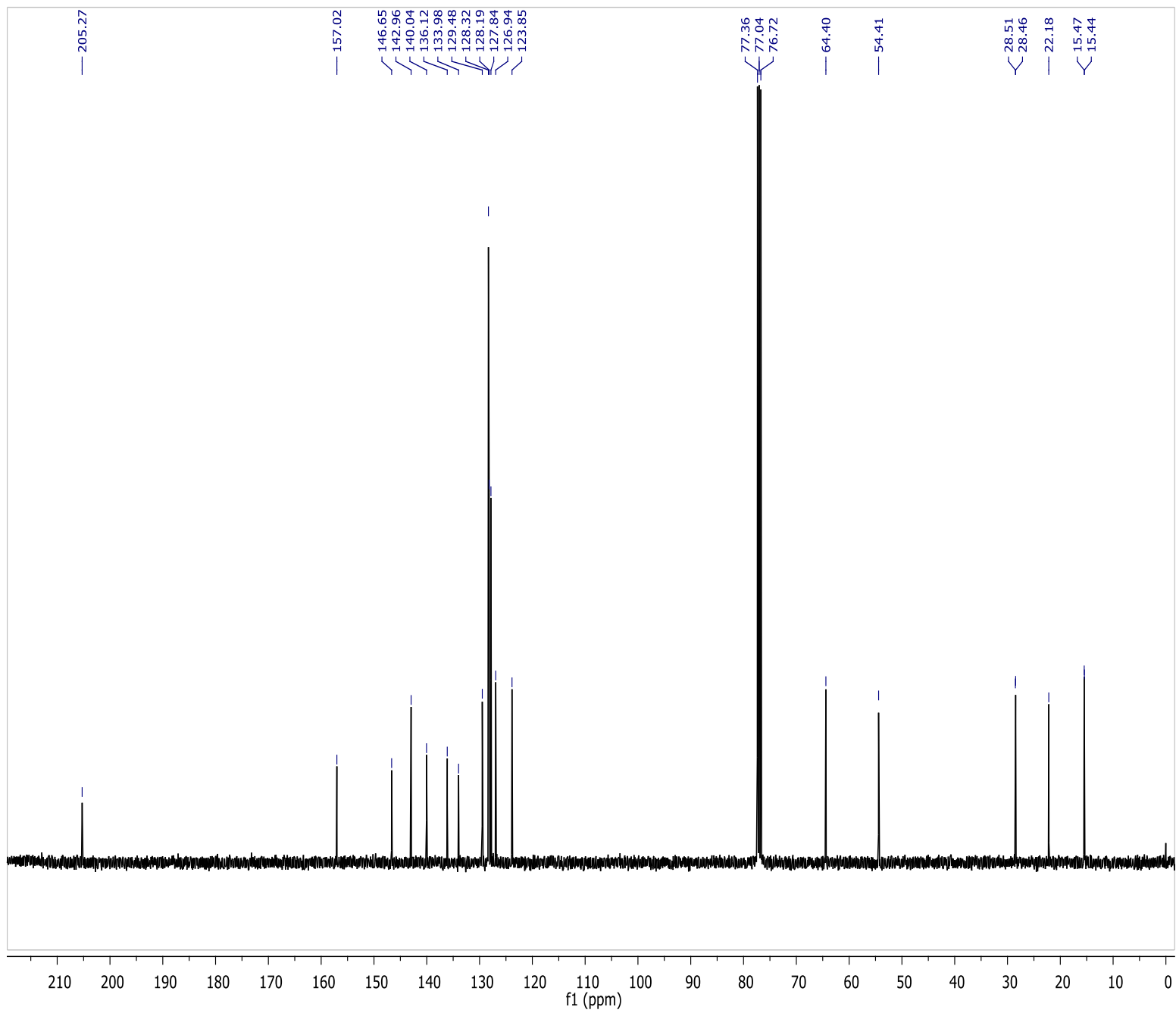
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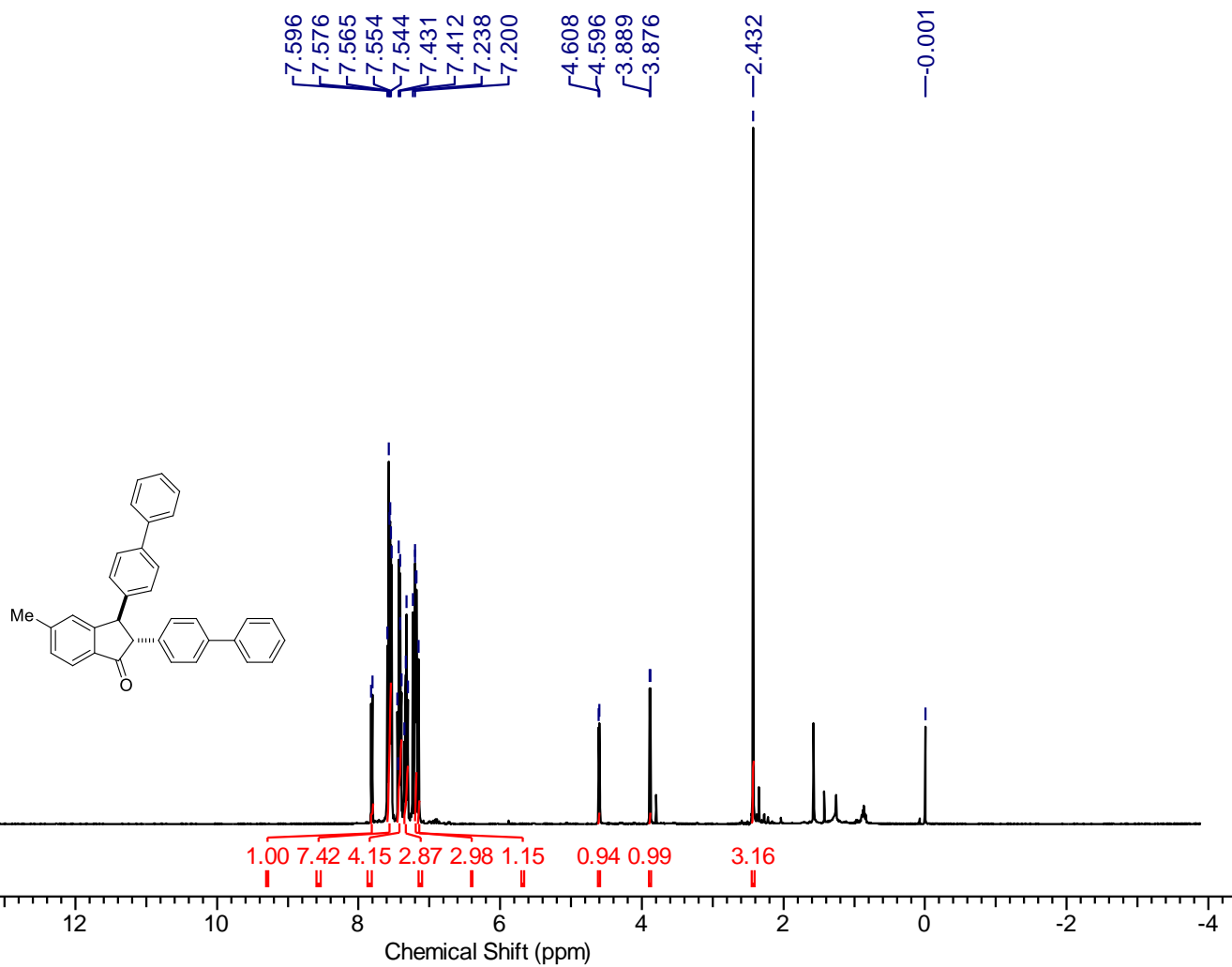


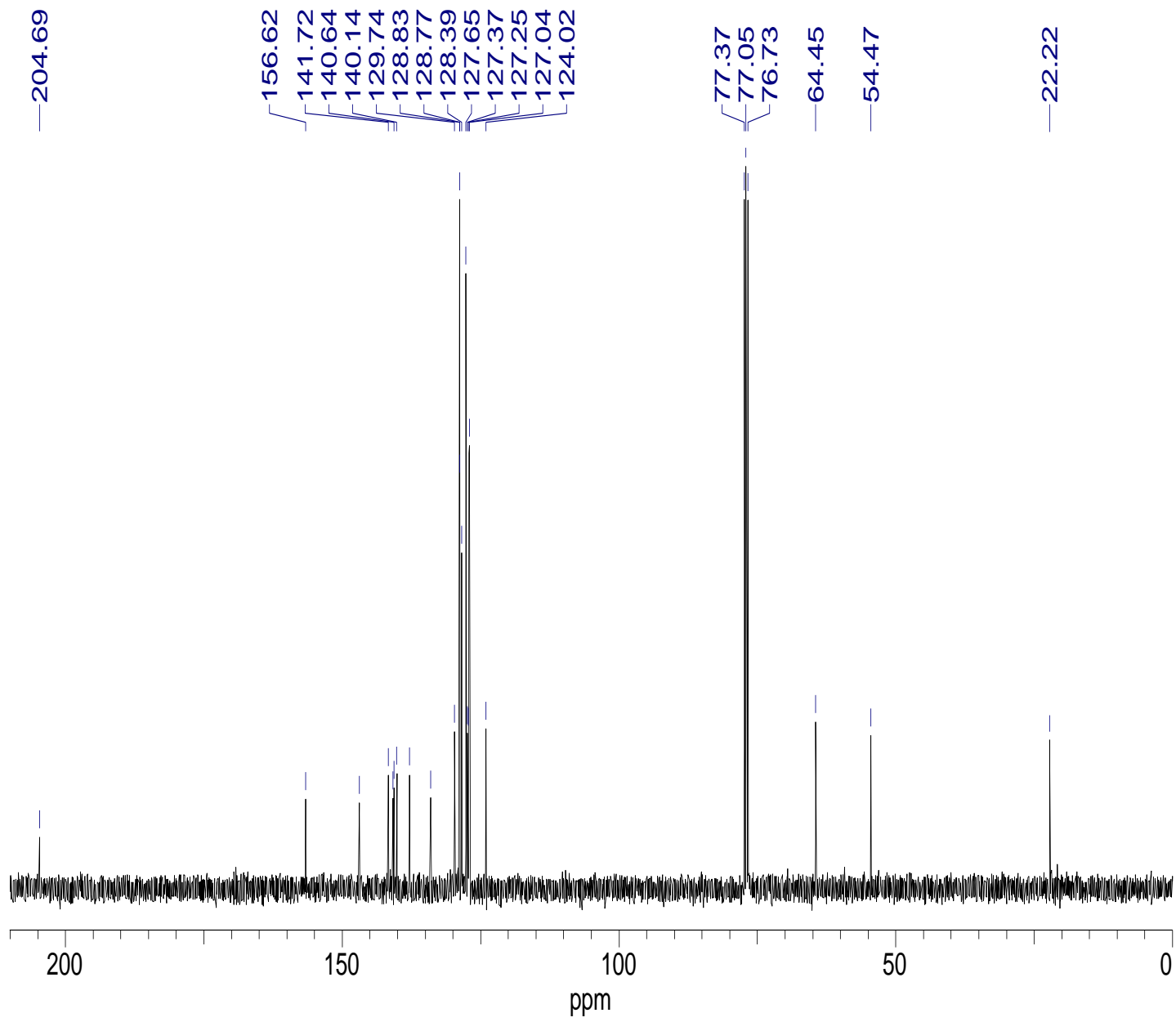
^1H (400 MHz) and ^{13}C (100 MHz) NMR in CDCl_3 of Compound 1d



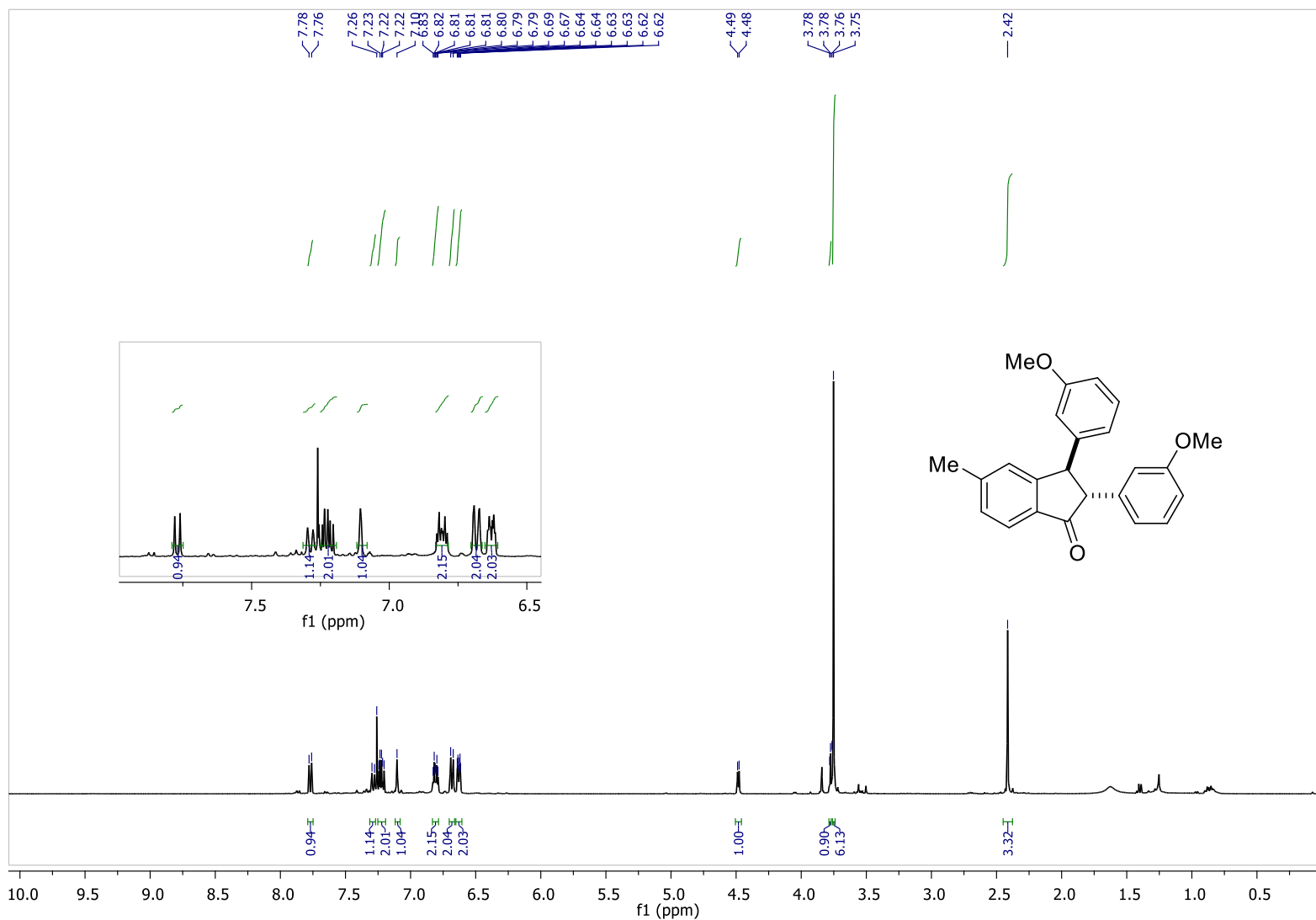


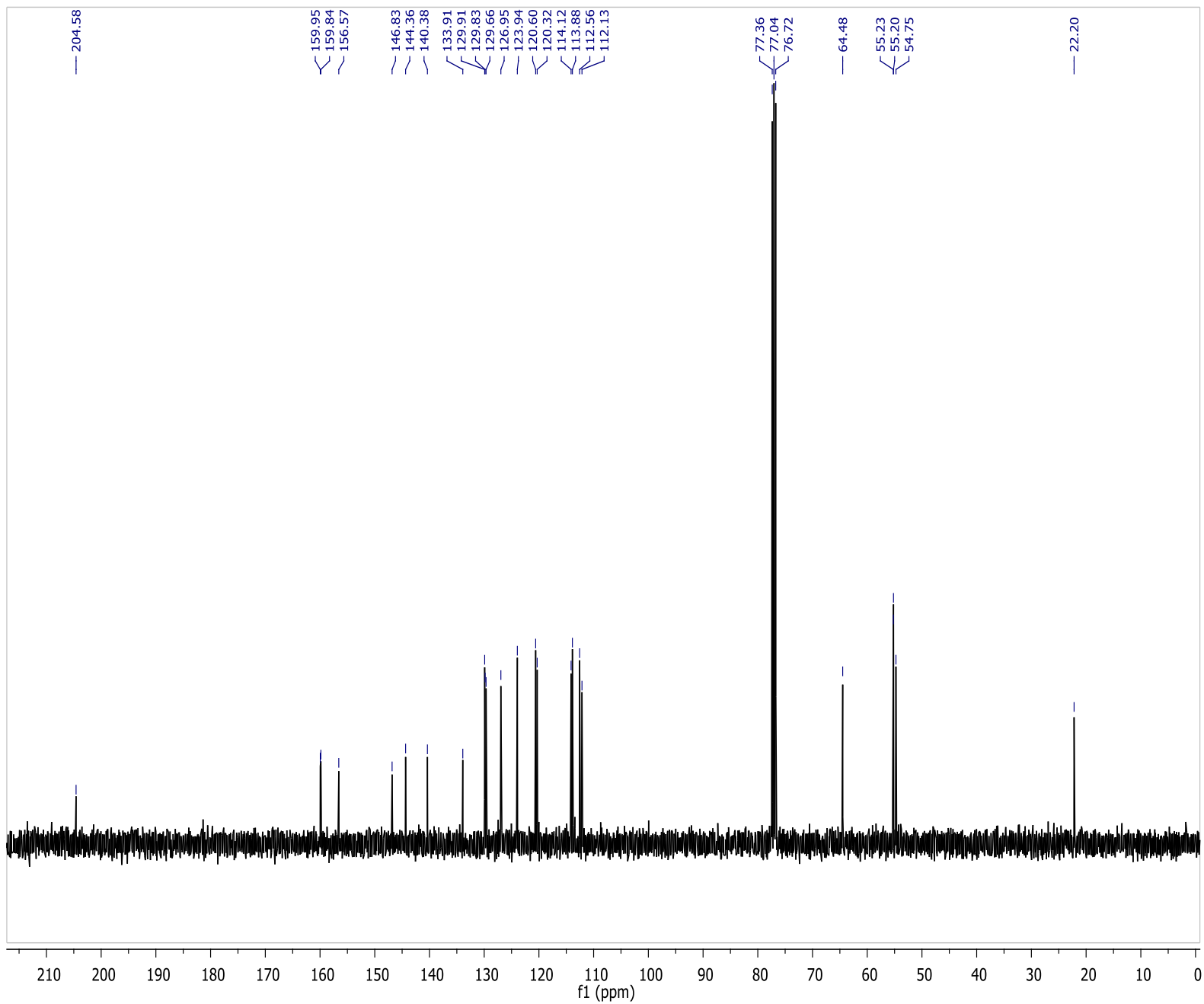
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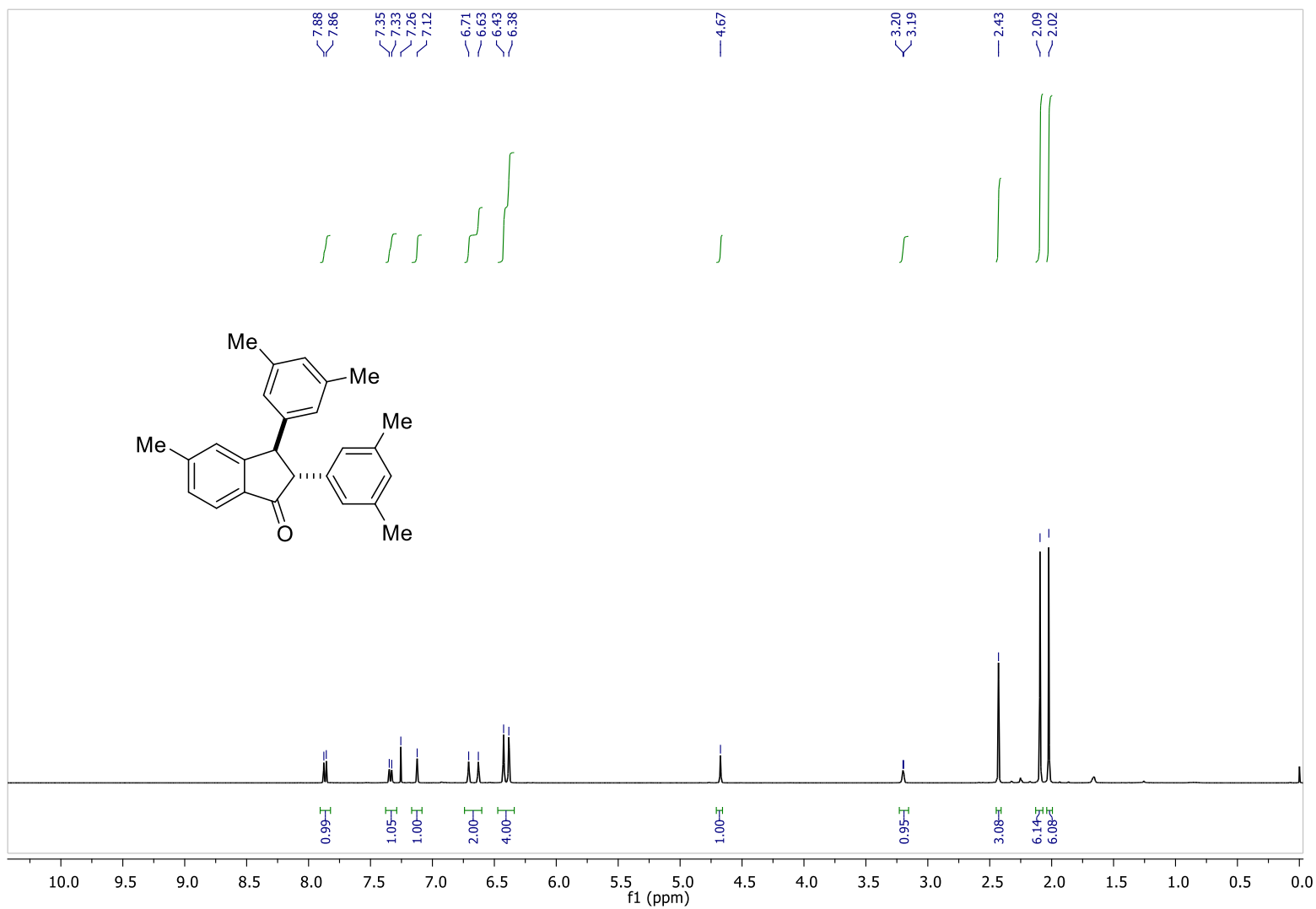


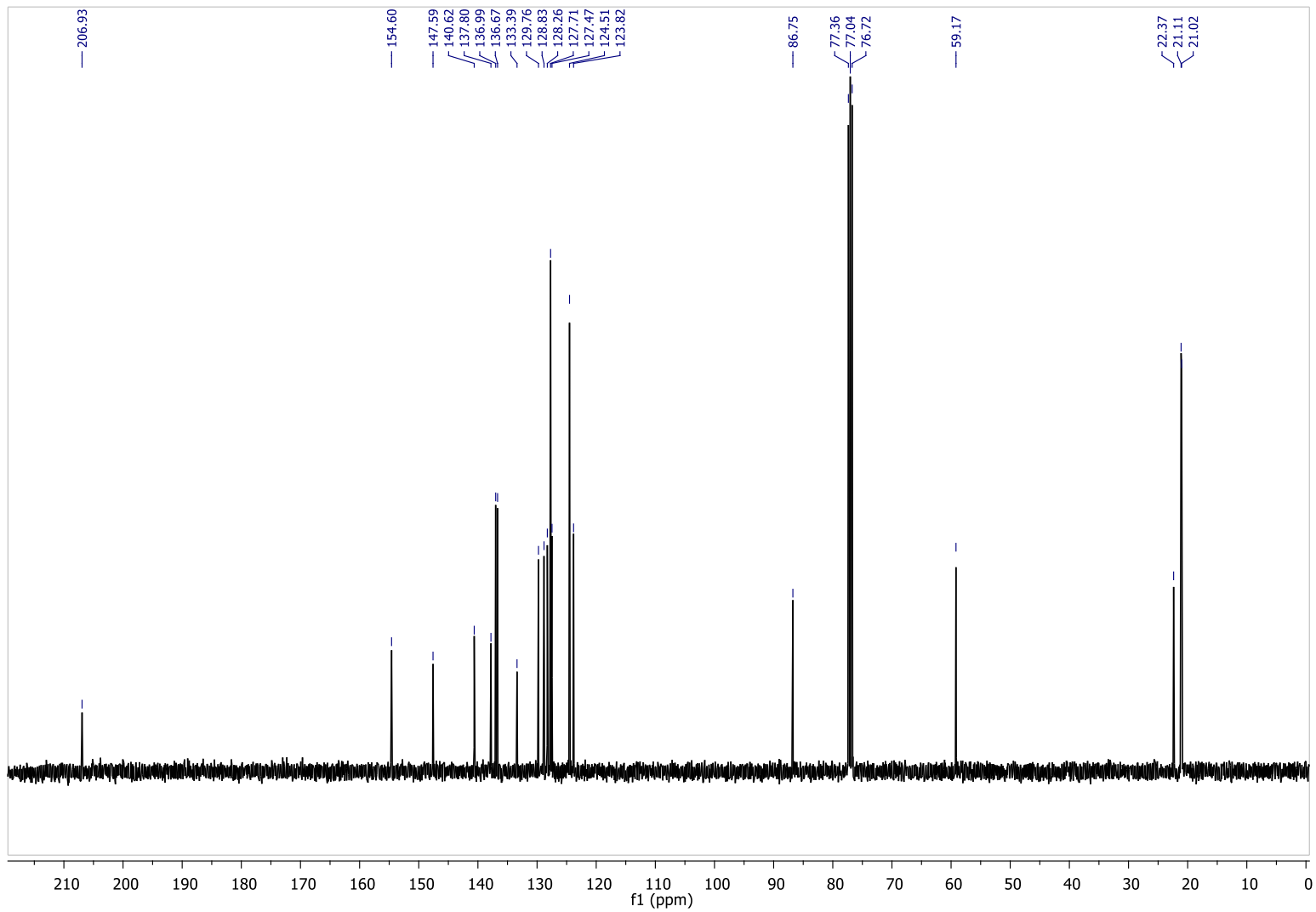
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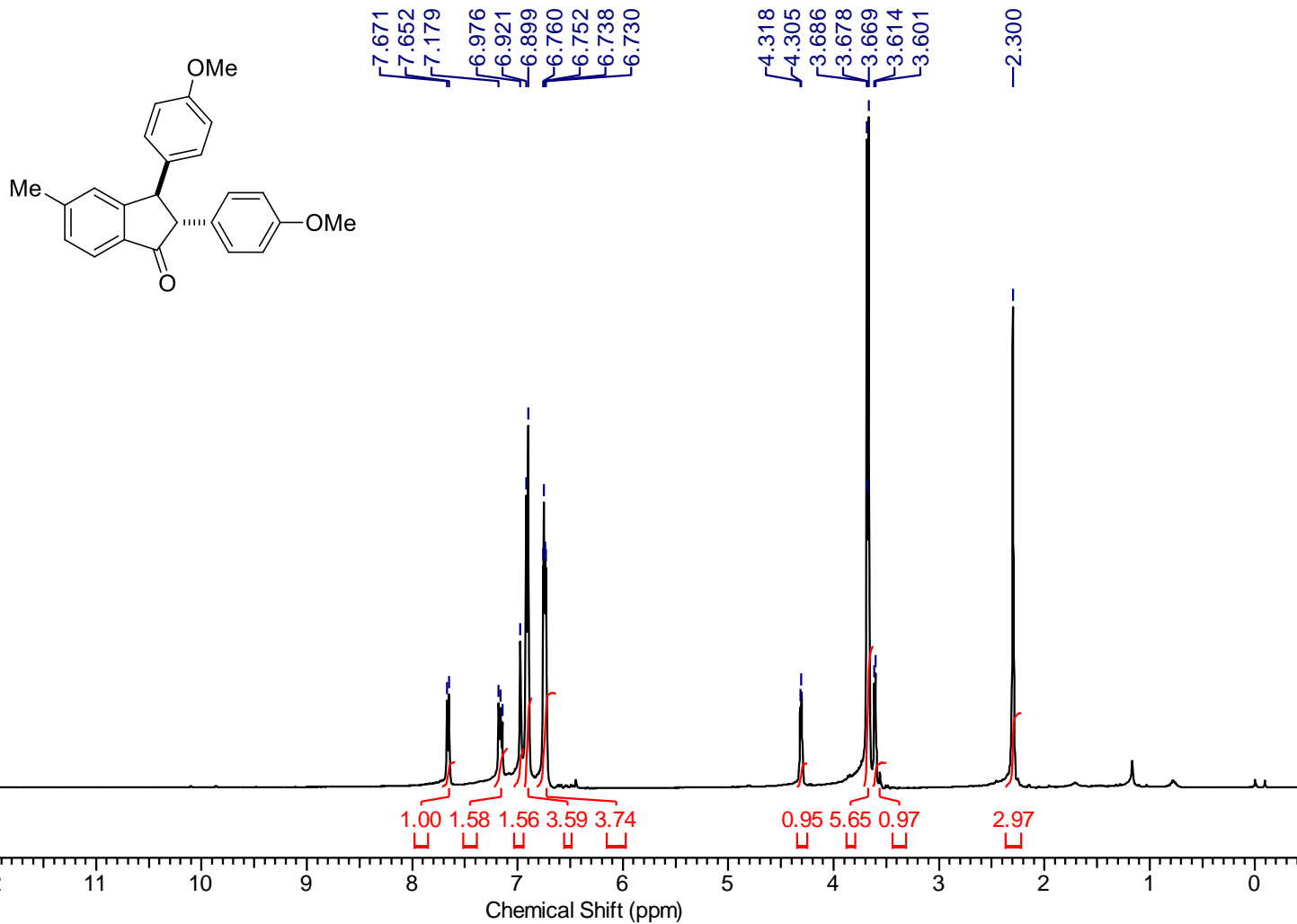


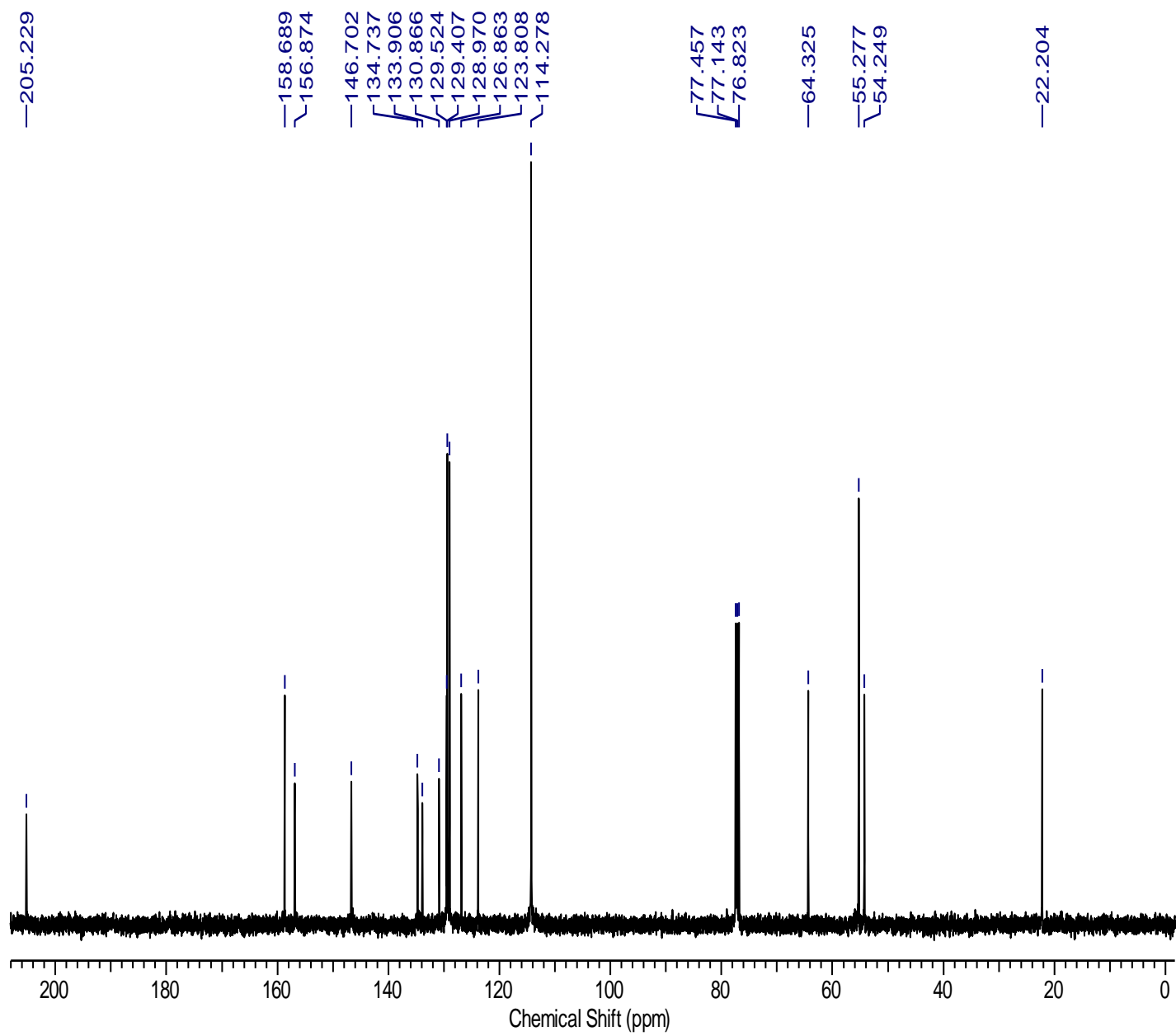
^1H (400 MHz) and ^{13}C (100 MHz) NMR in CDCl_3 of Compound **1g**



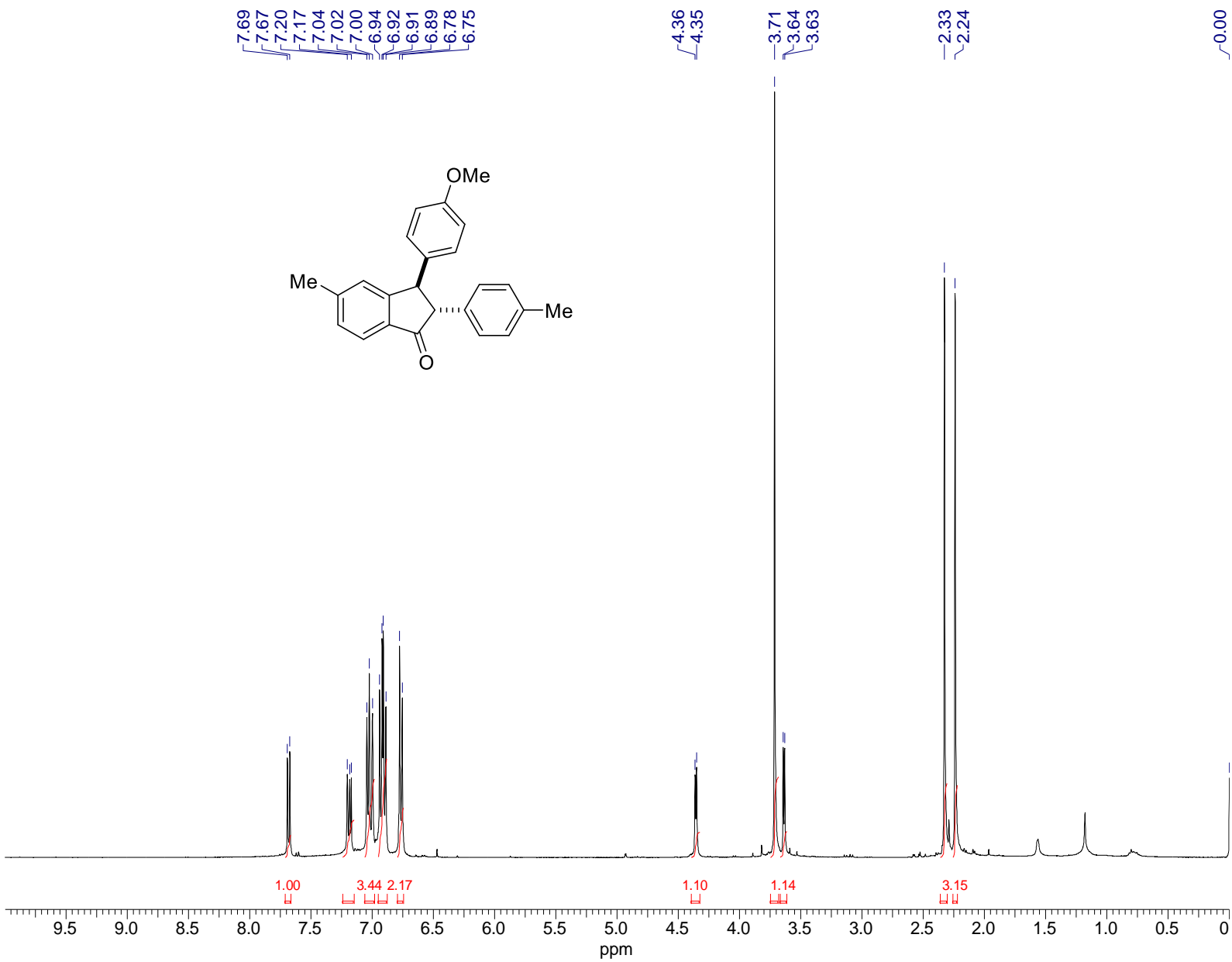


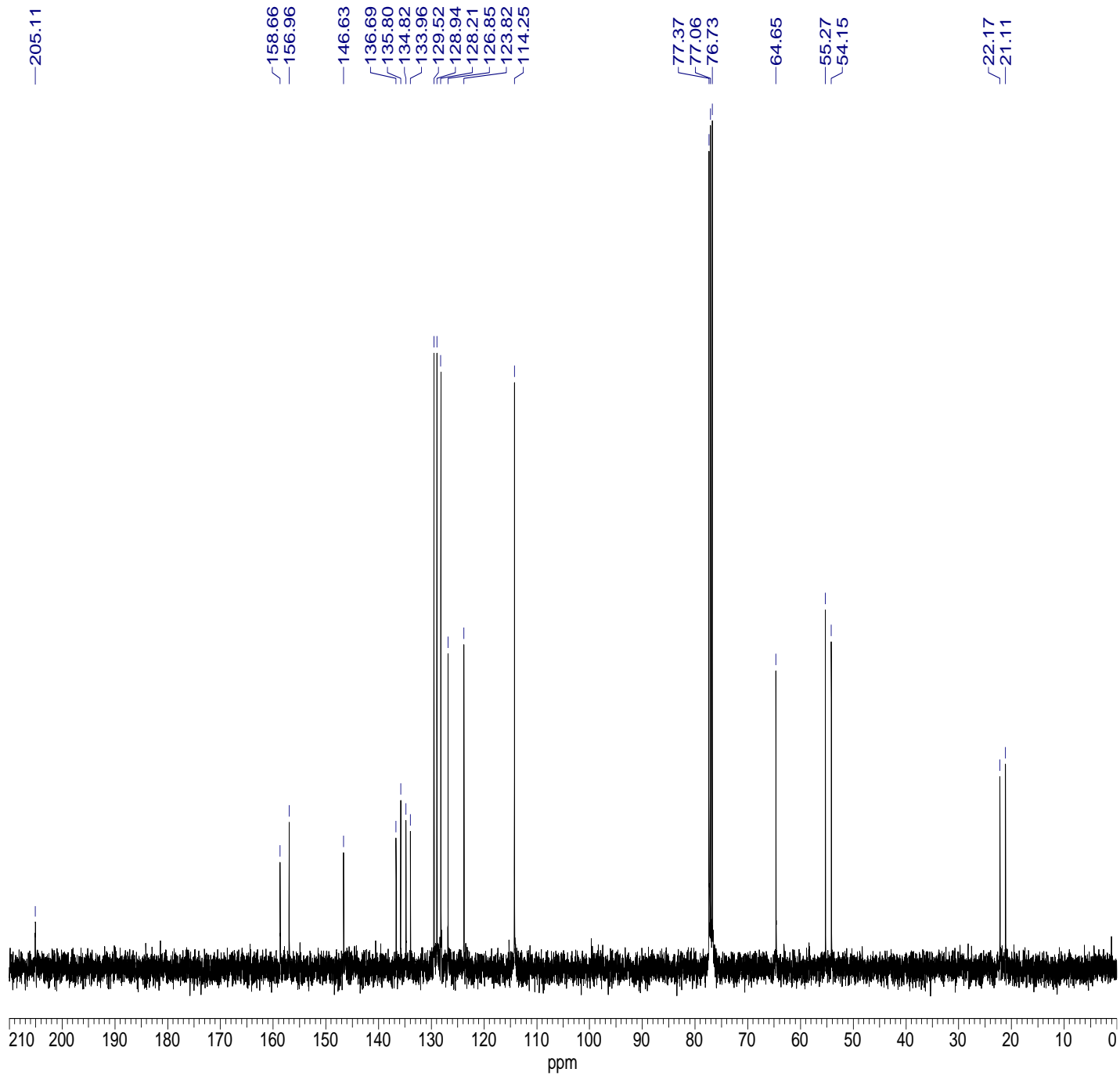
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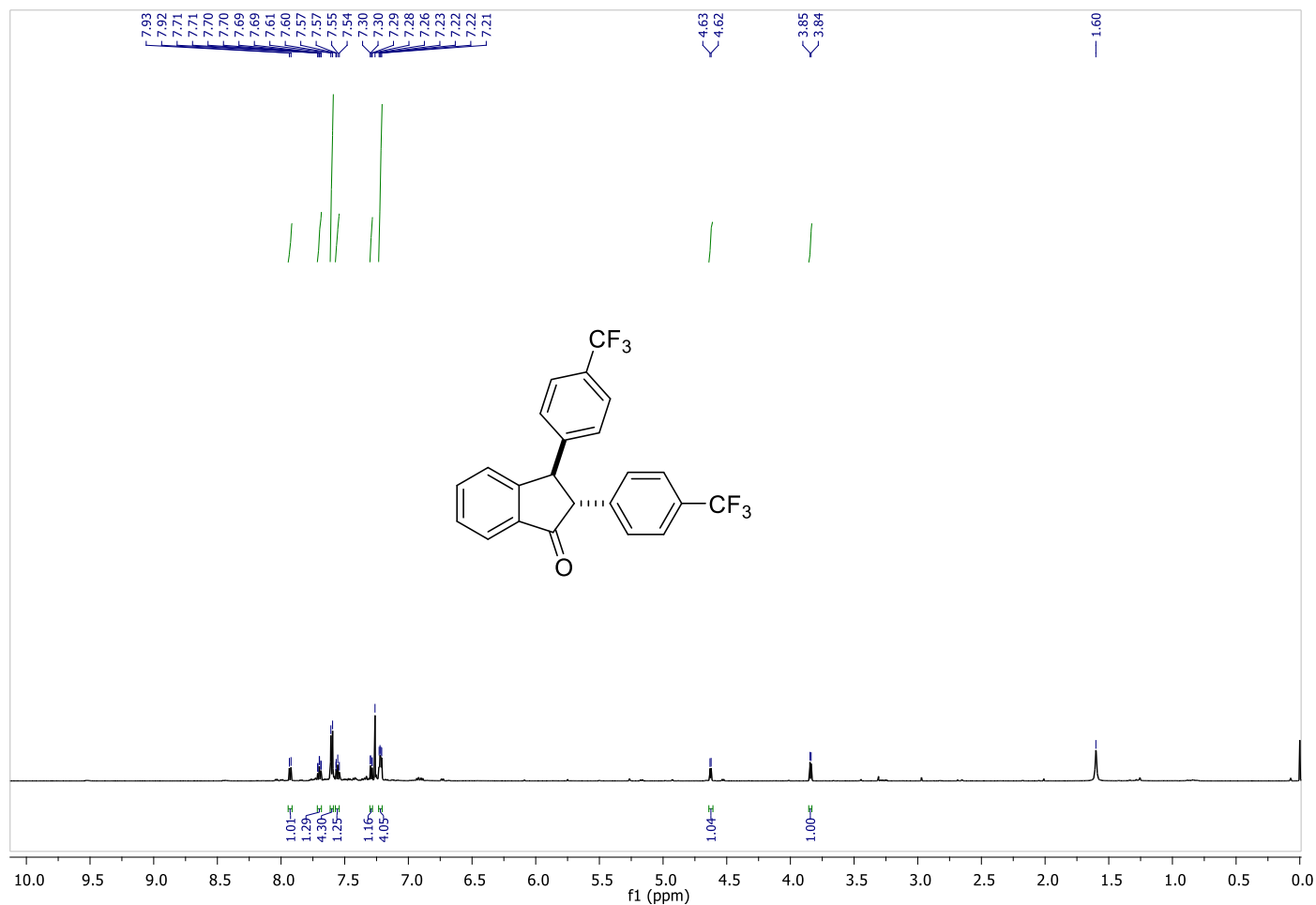


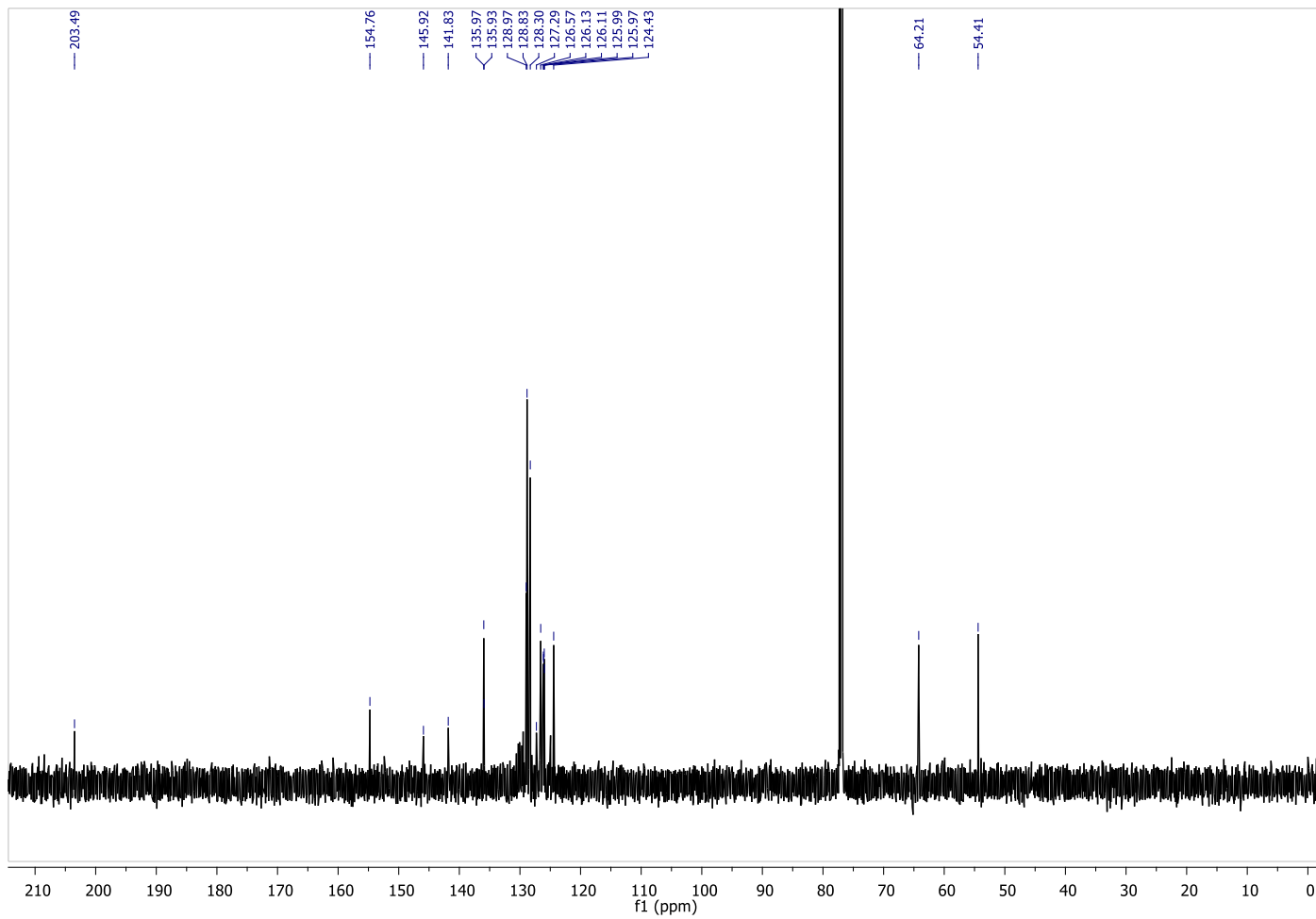
¹H (400 MHz) and ¹³C (100 MHz) NMR in CDCl₃ of Compound 1i



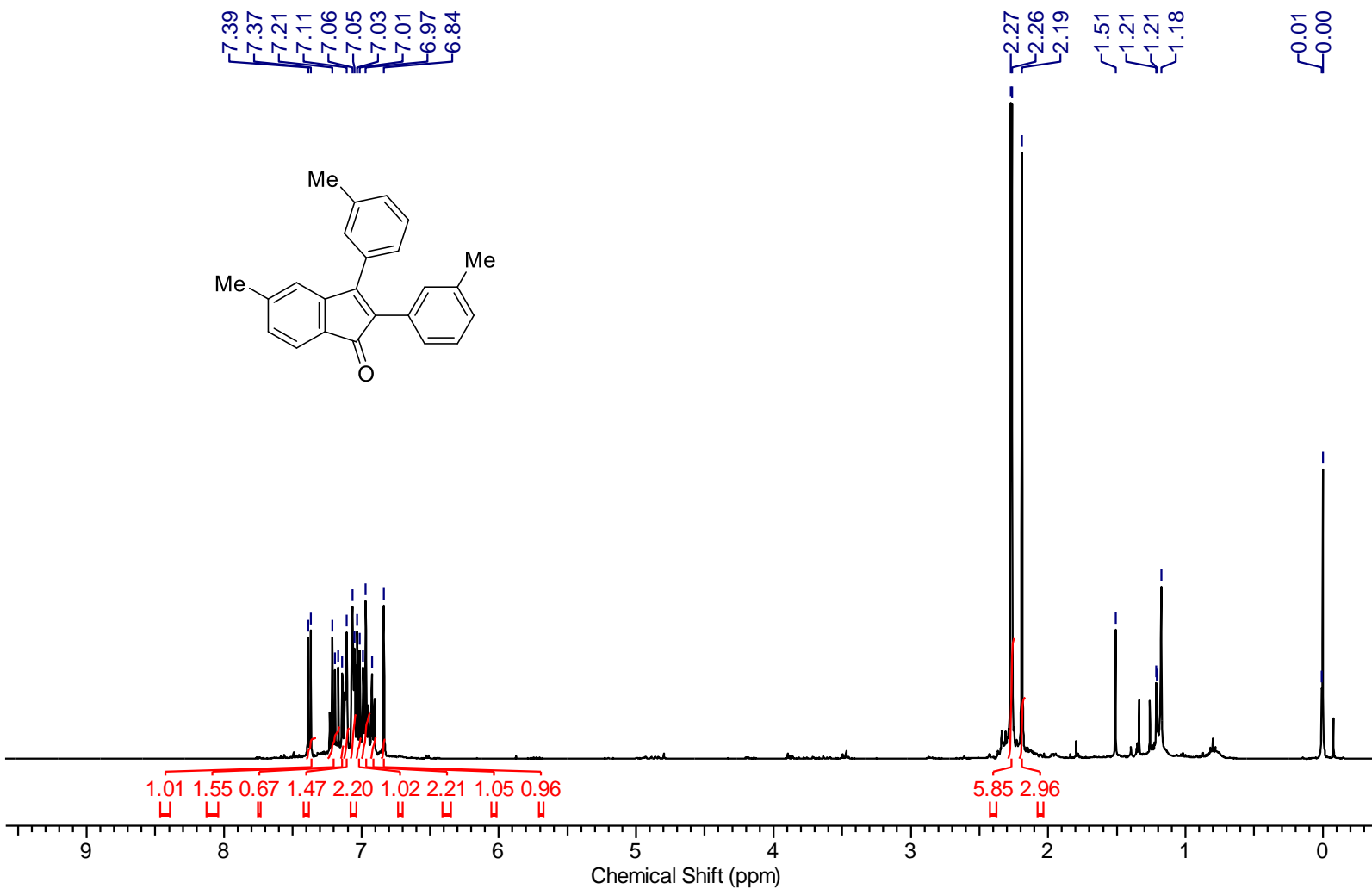
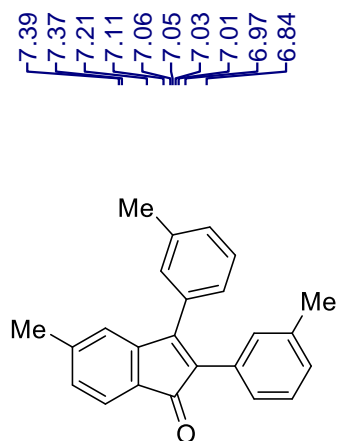


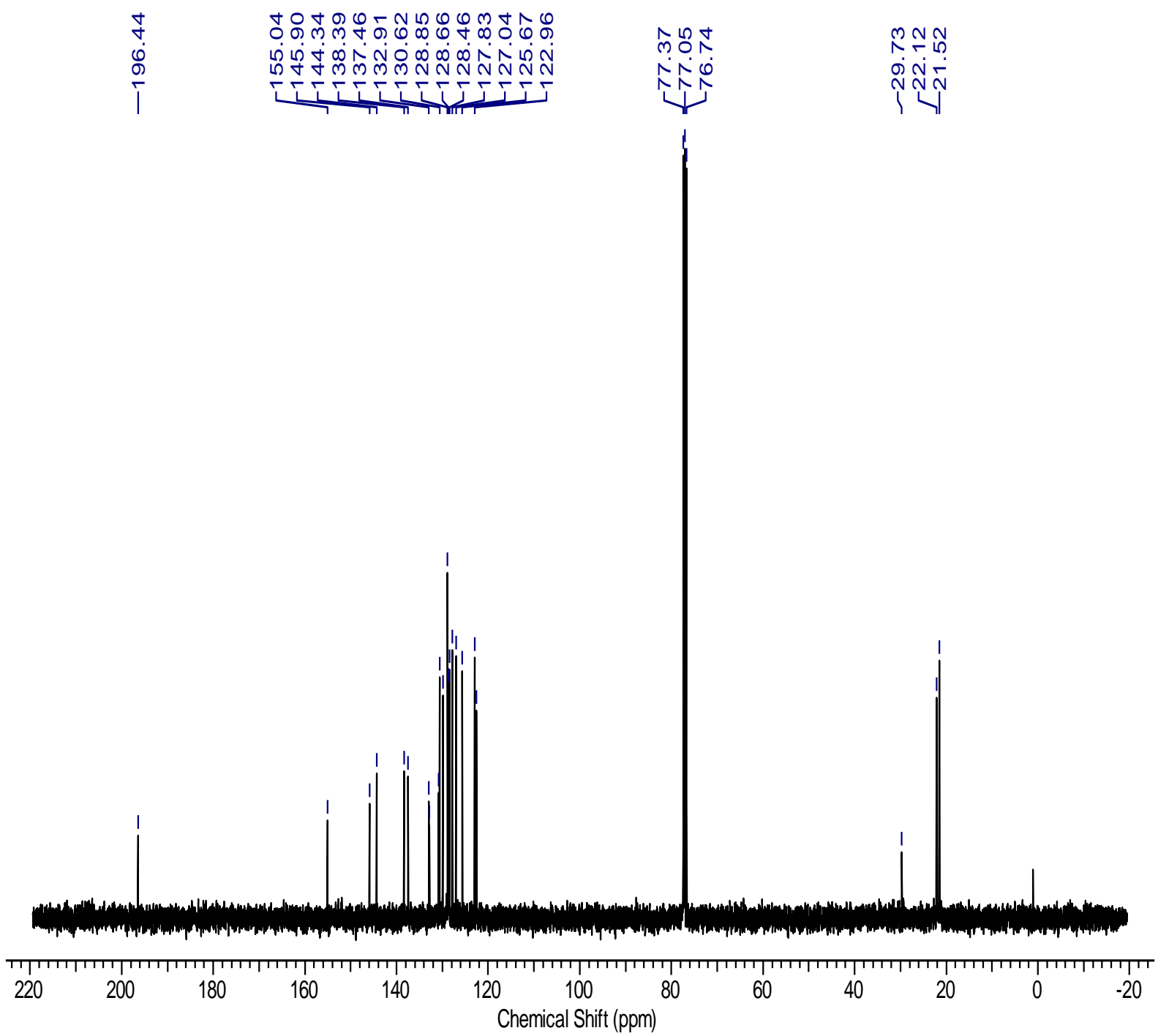
^1H (600 MHz) and ^{13}C (151 MHz) NMR in CDCl_3 of Compound 1o



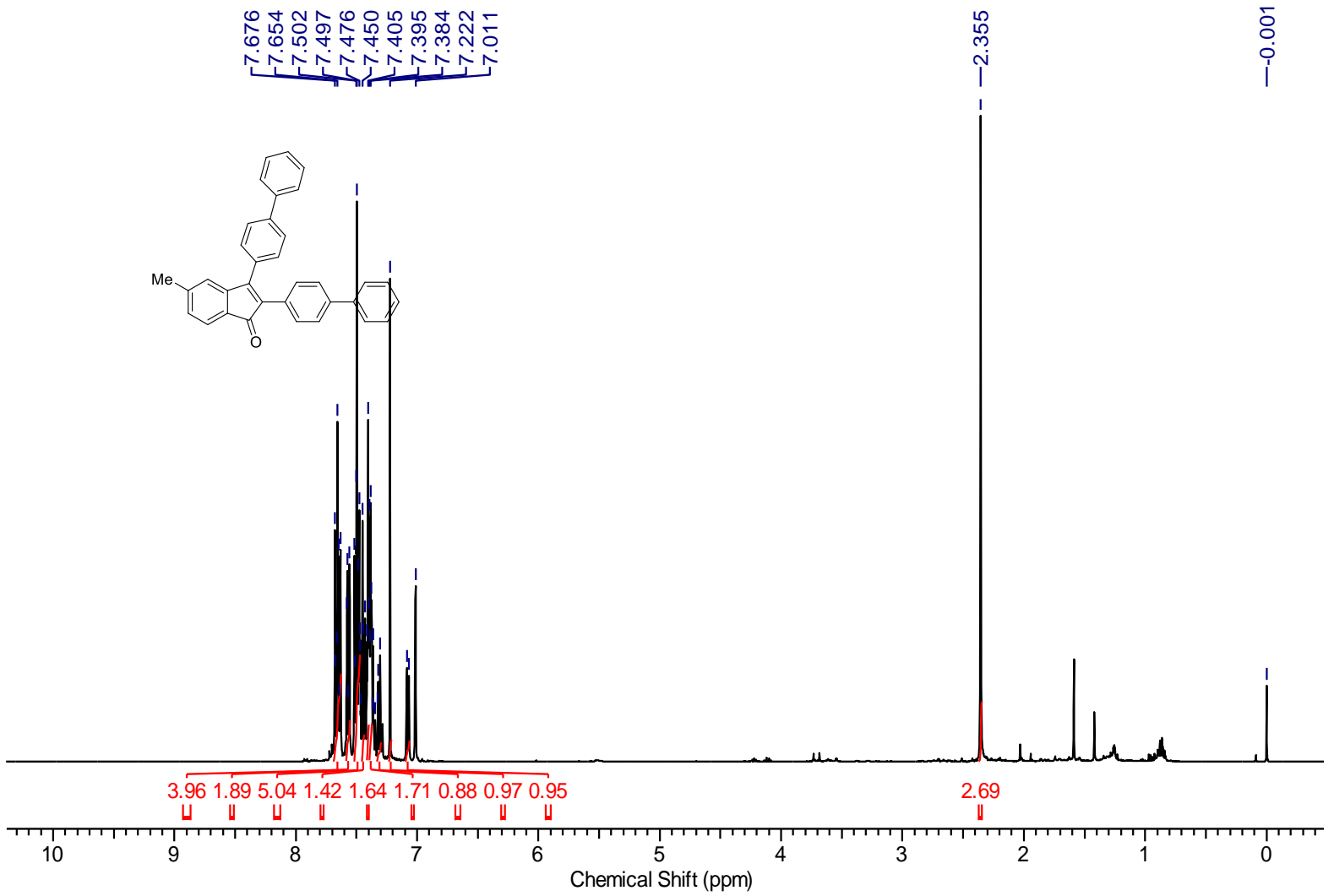


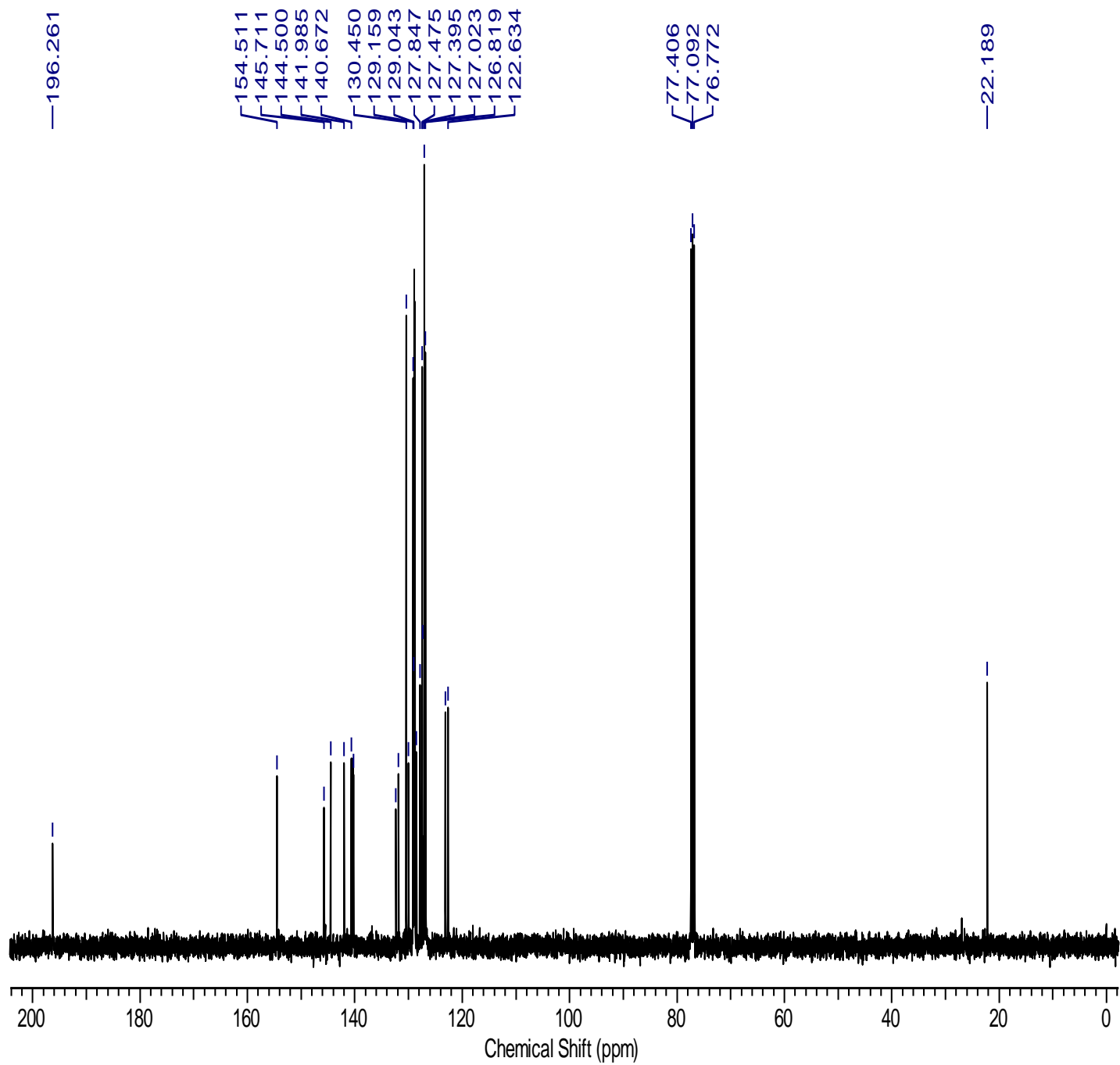
¹H (400 MHz) and ¹³C (100 MHz) NMR in CDCl₃ of Compound 4a



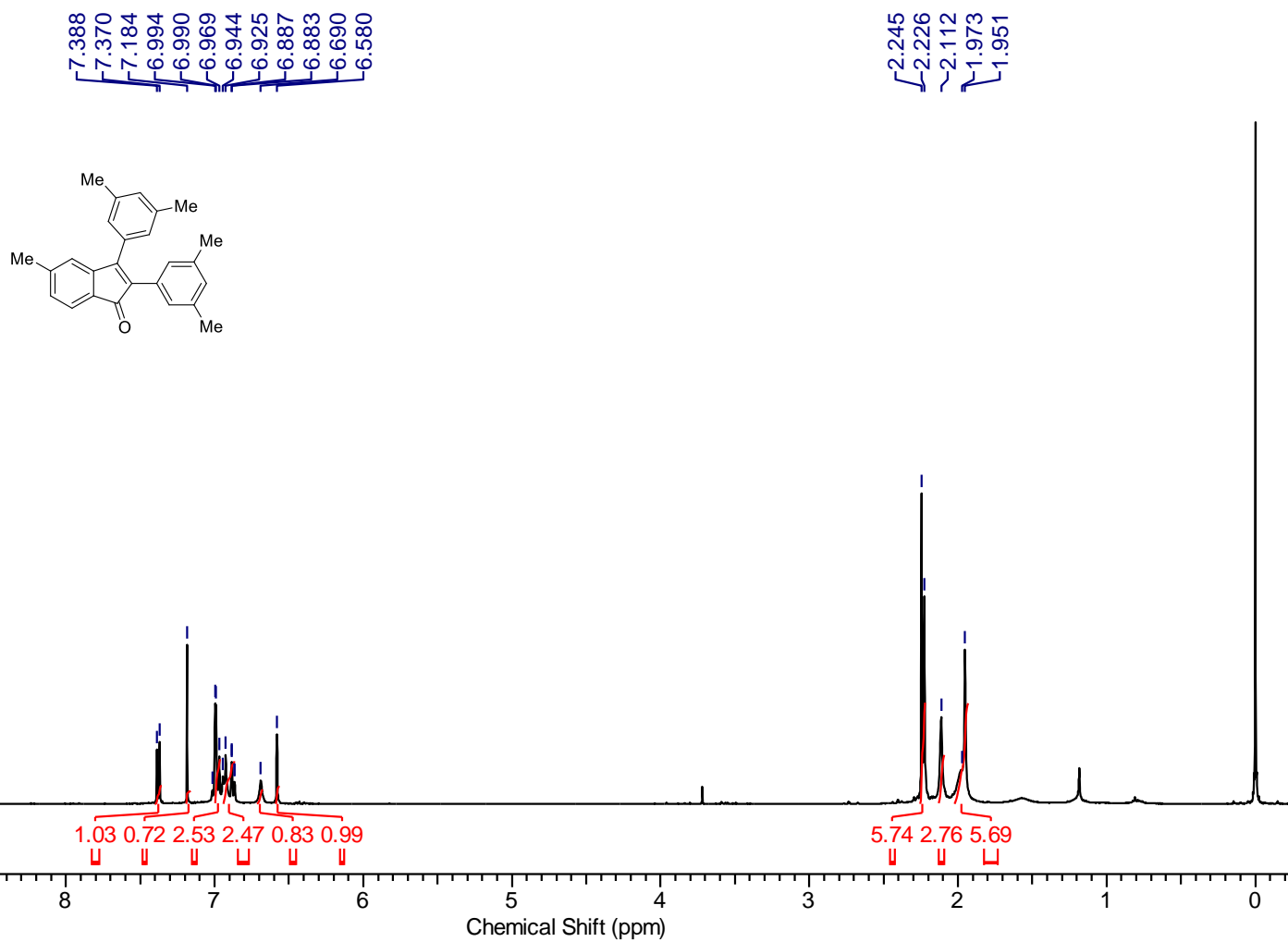


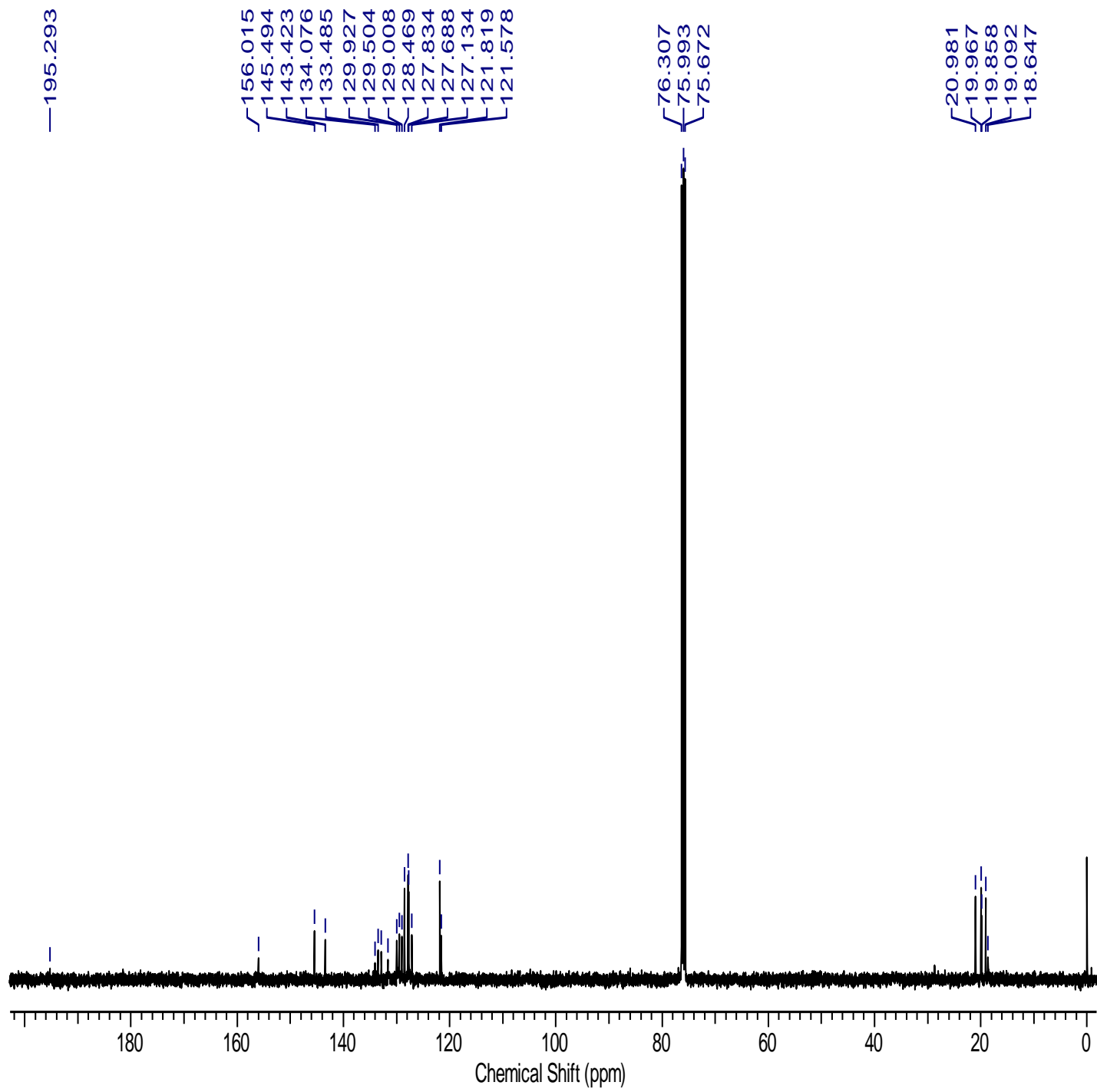
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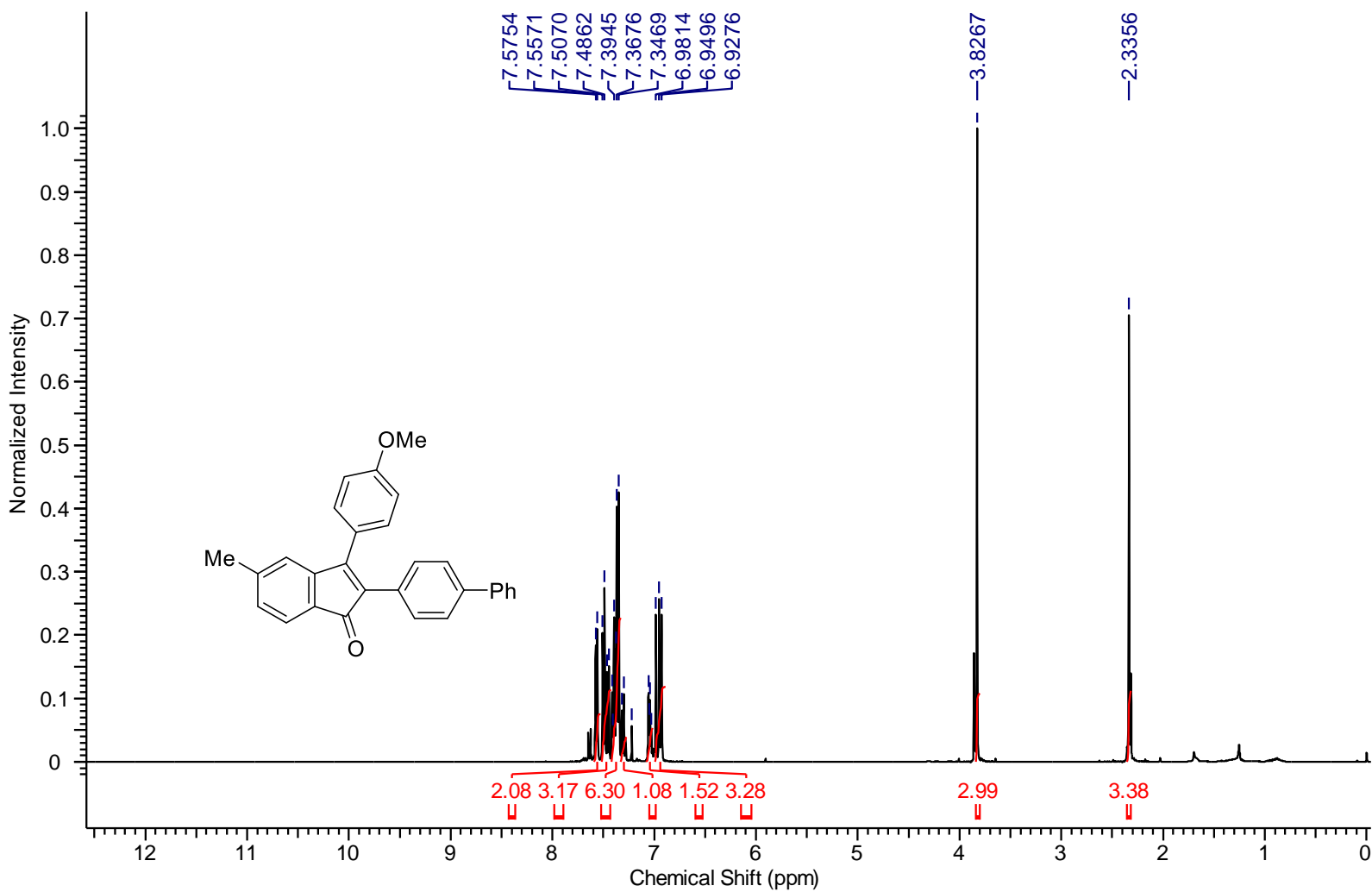


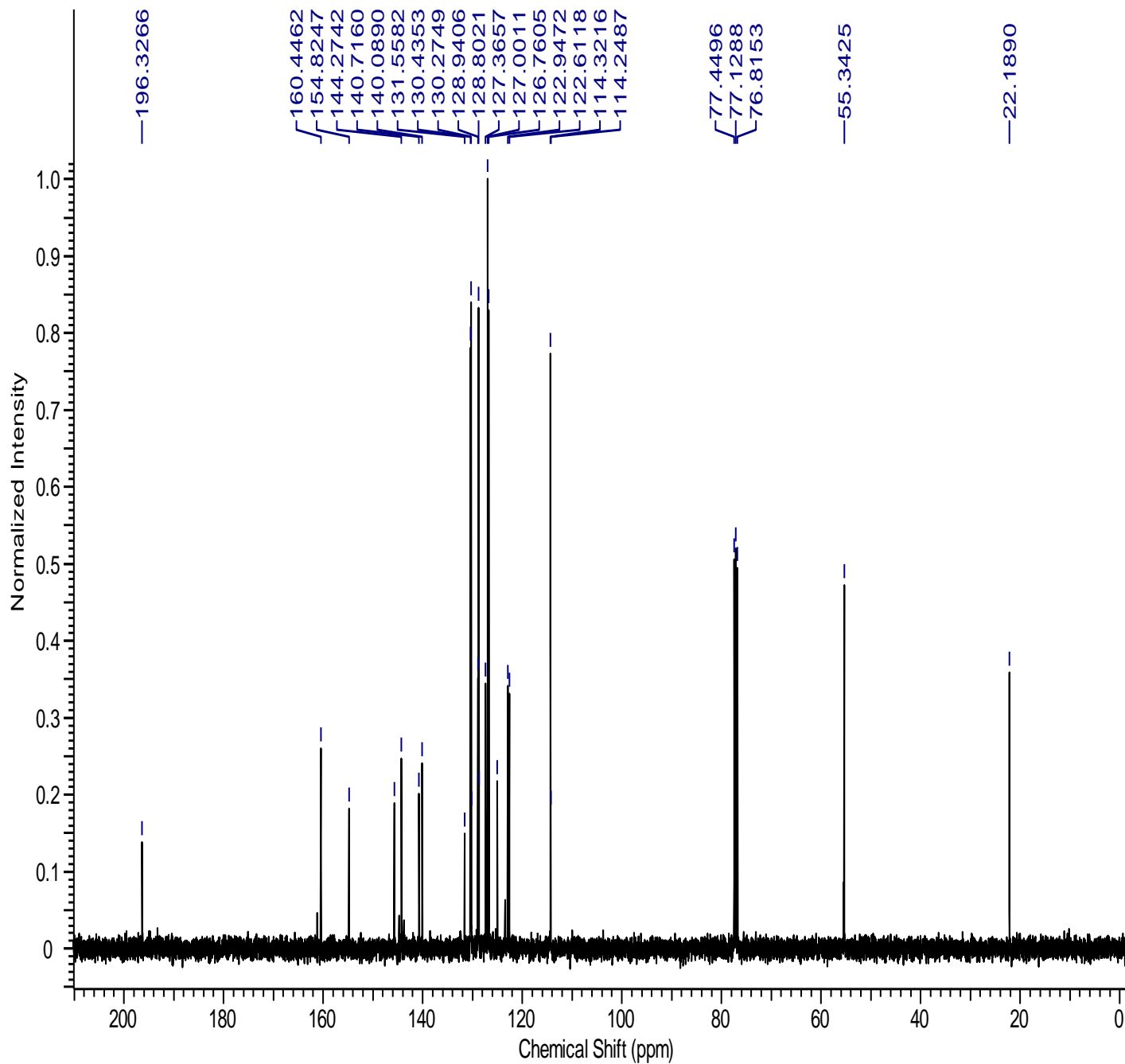
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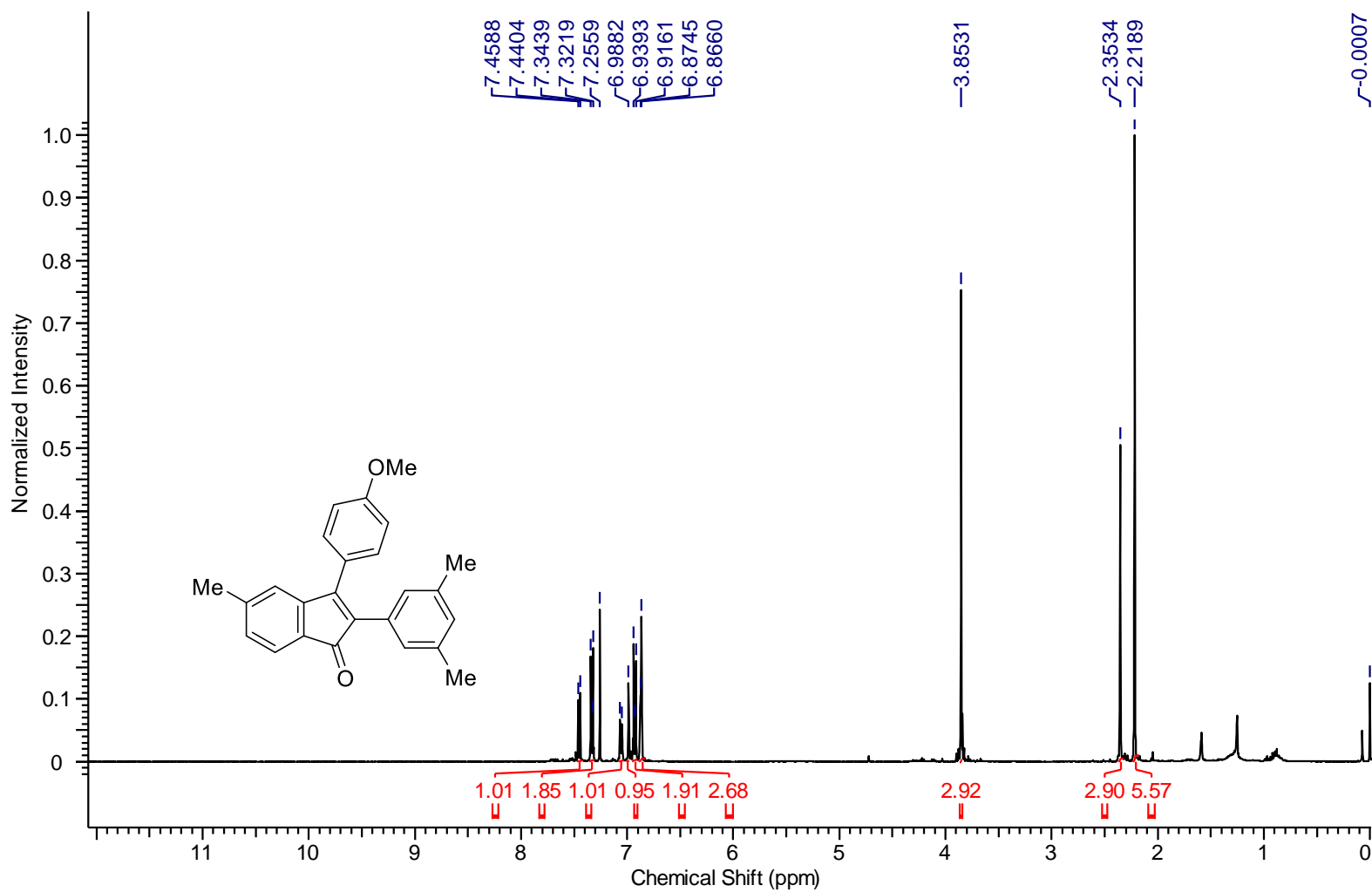


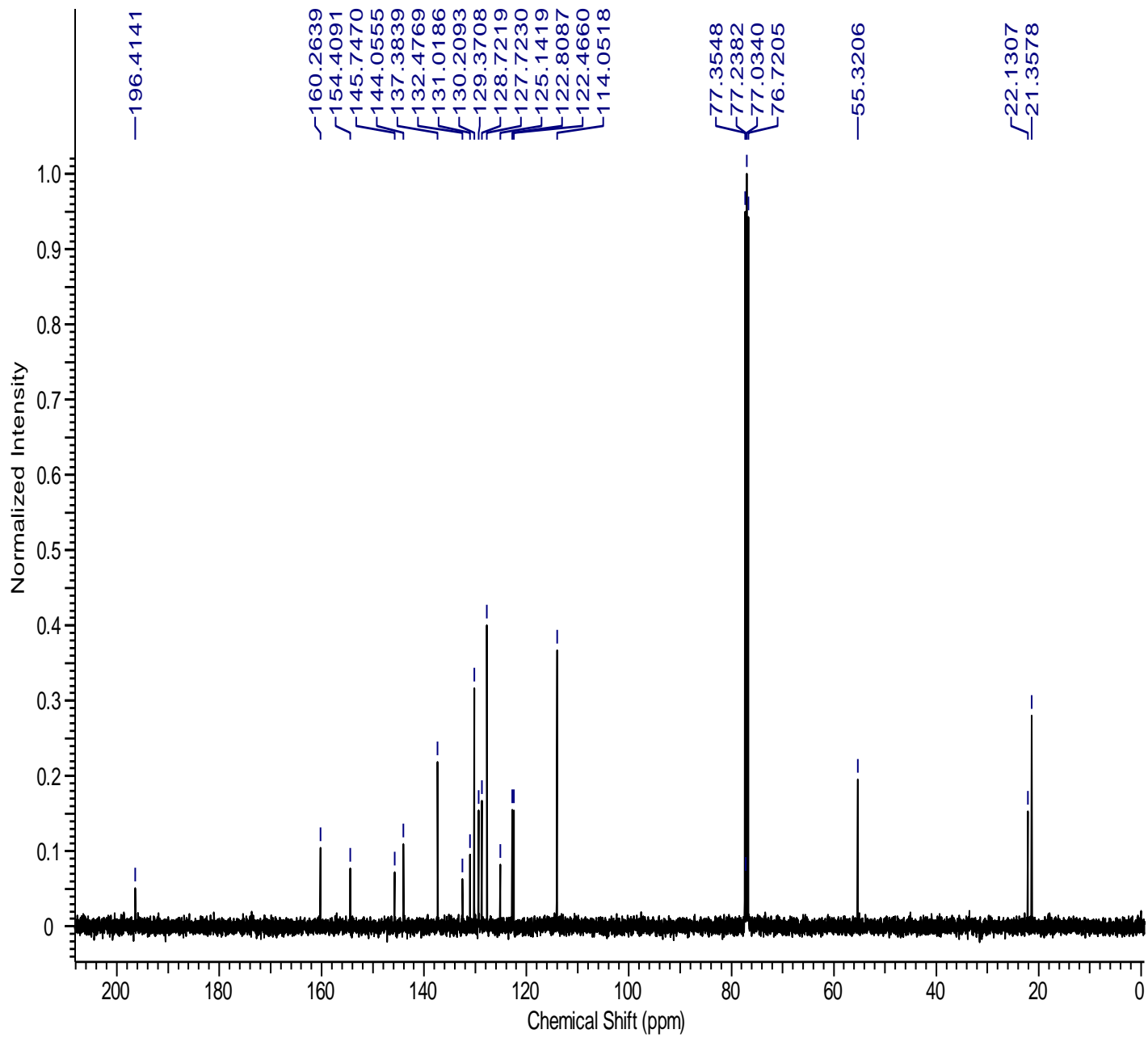
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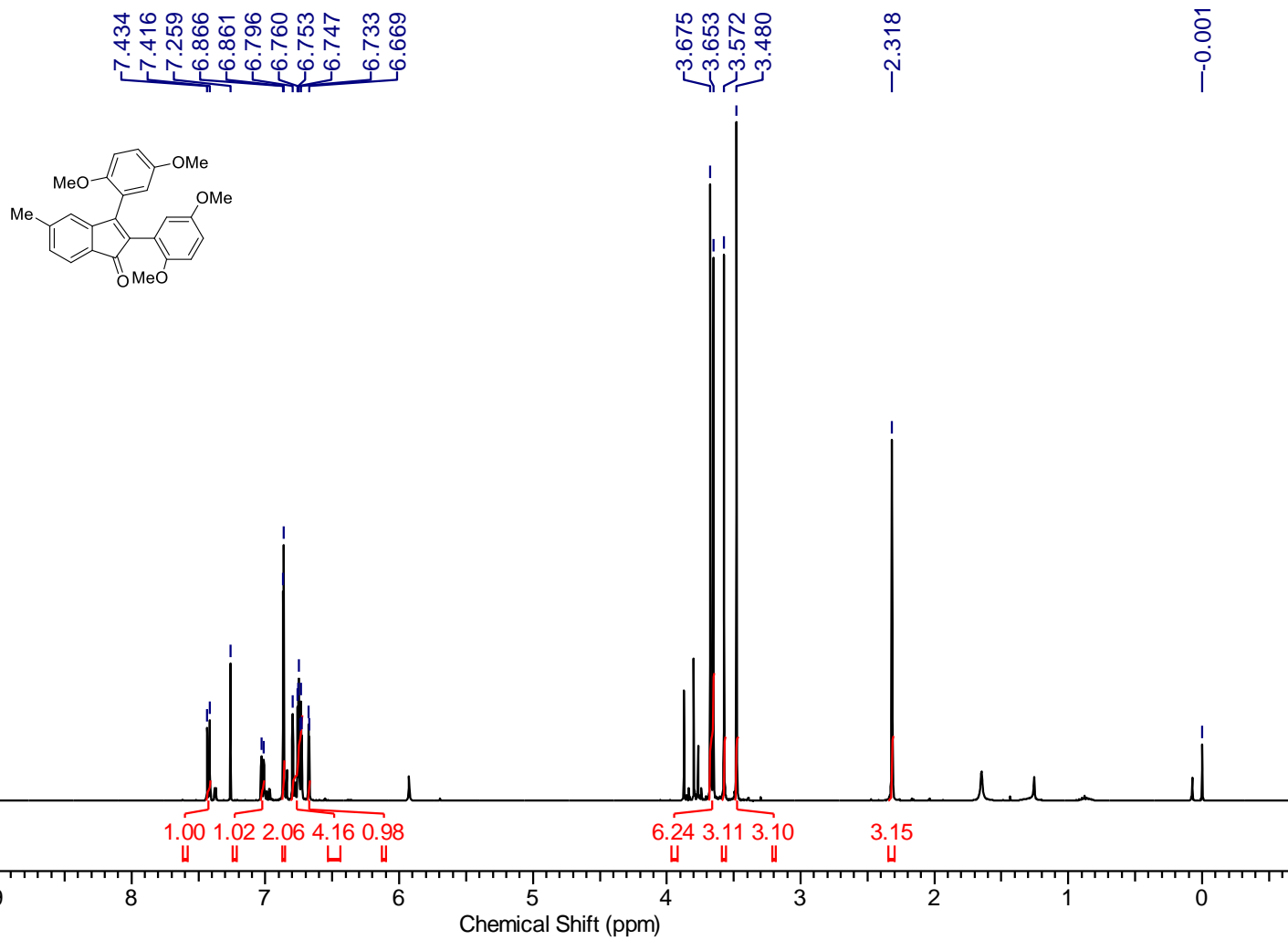


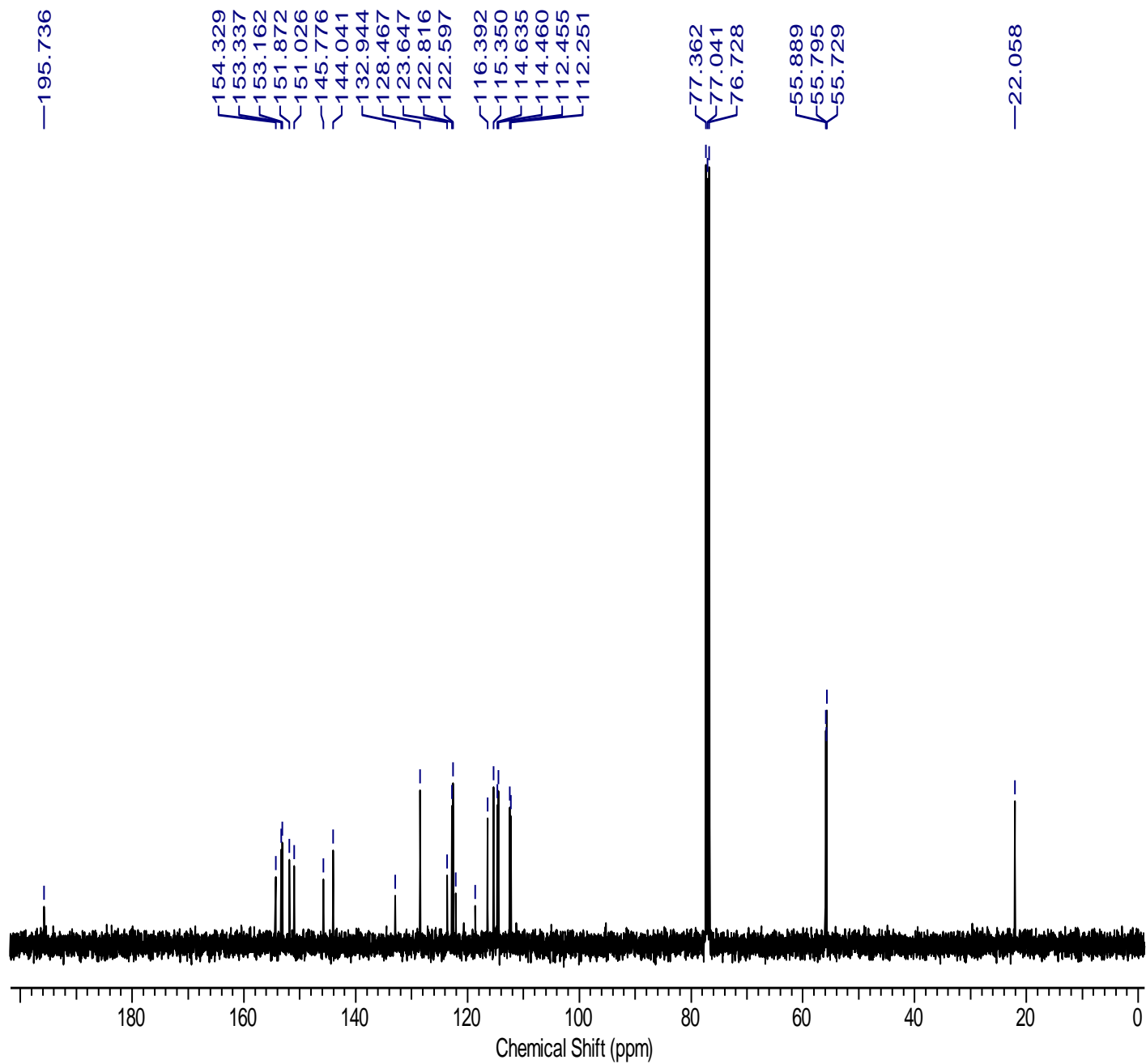
¹H (400 MHz) and ¹³C (100 MHz) NMR in CDCl₃ of Compound 4j



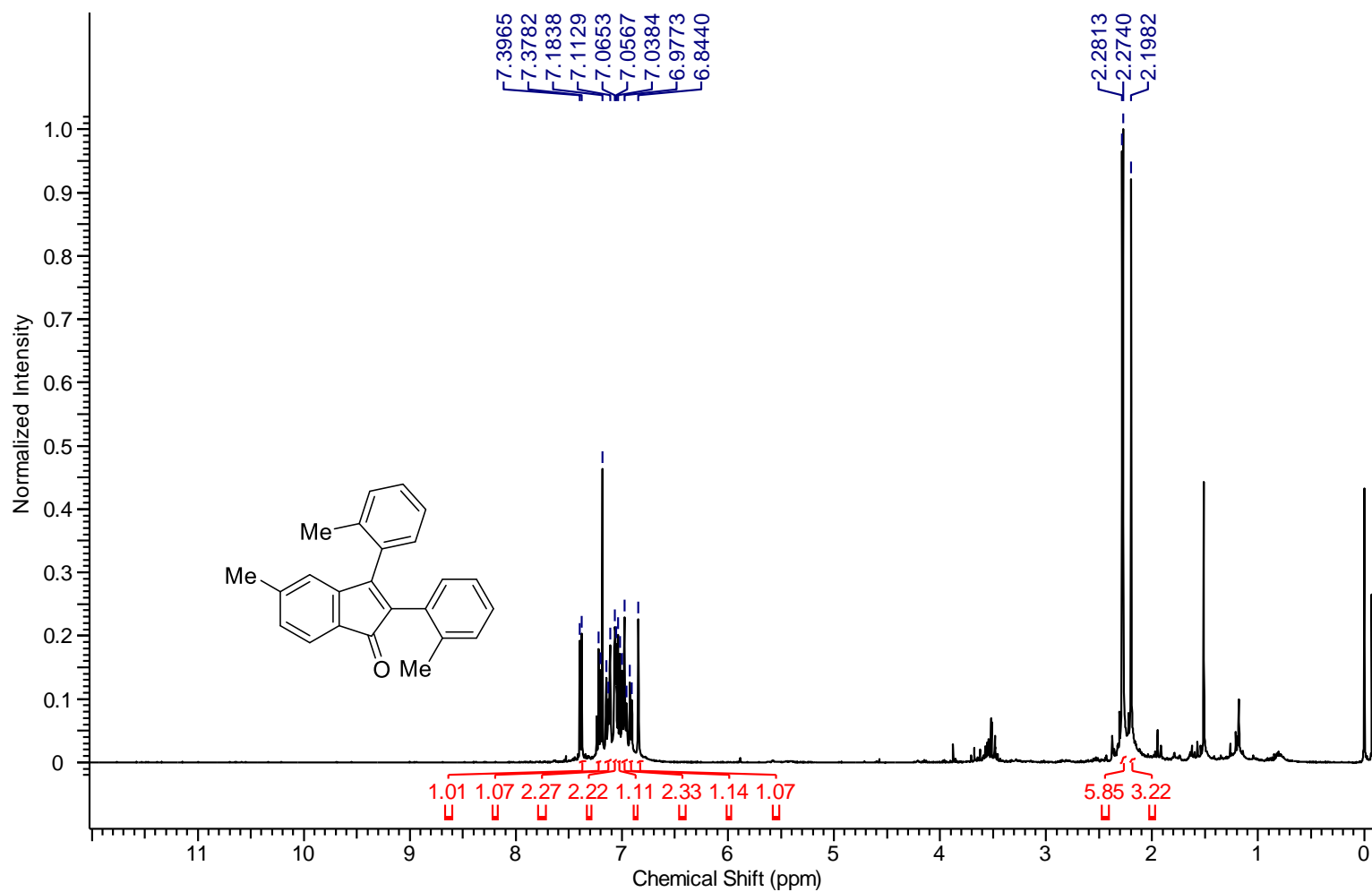


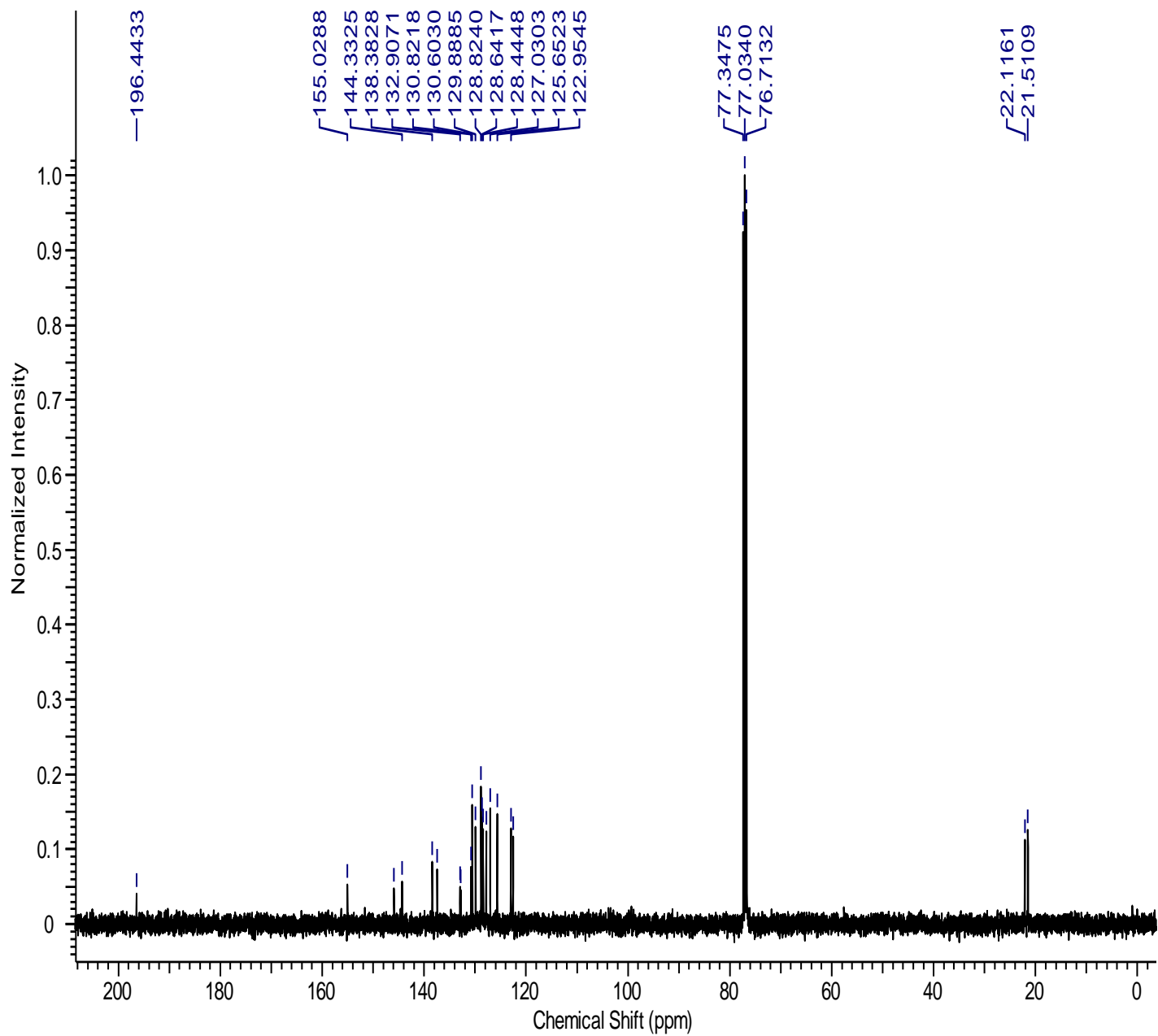
^1H (400 MHz) and ^{13}C (100 MHz) NMR in CDCl_3 of Compound 4l



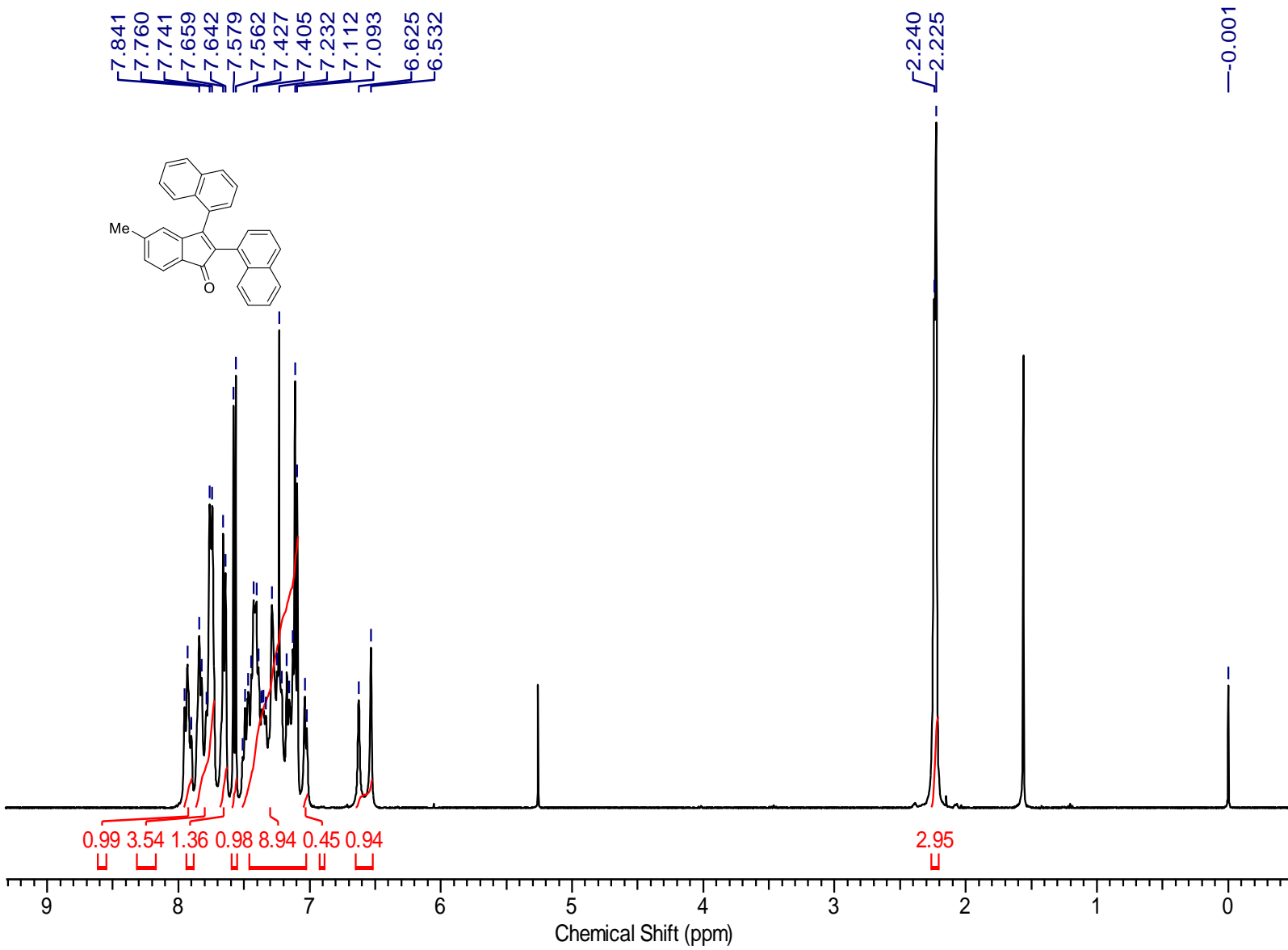


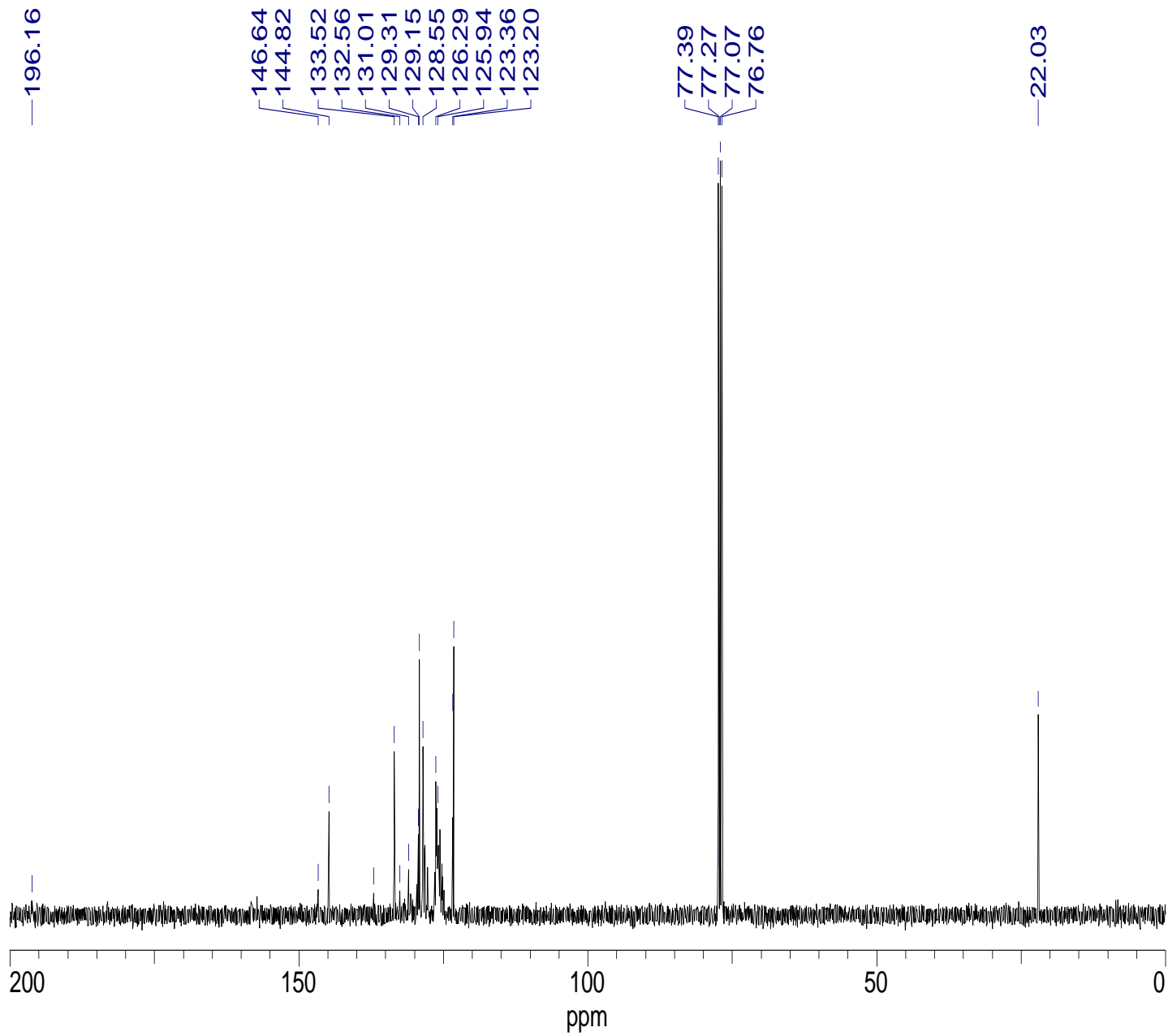
¹H (400 MHz) and ¹³C (100 MHz) NMR in CDCl₃ of Compound 4m



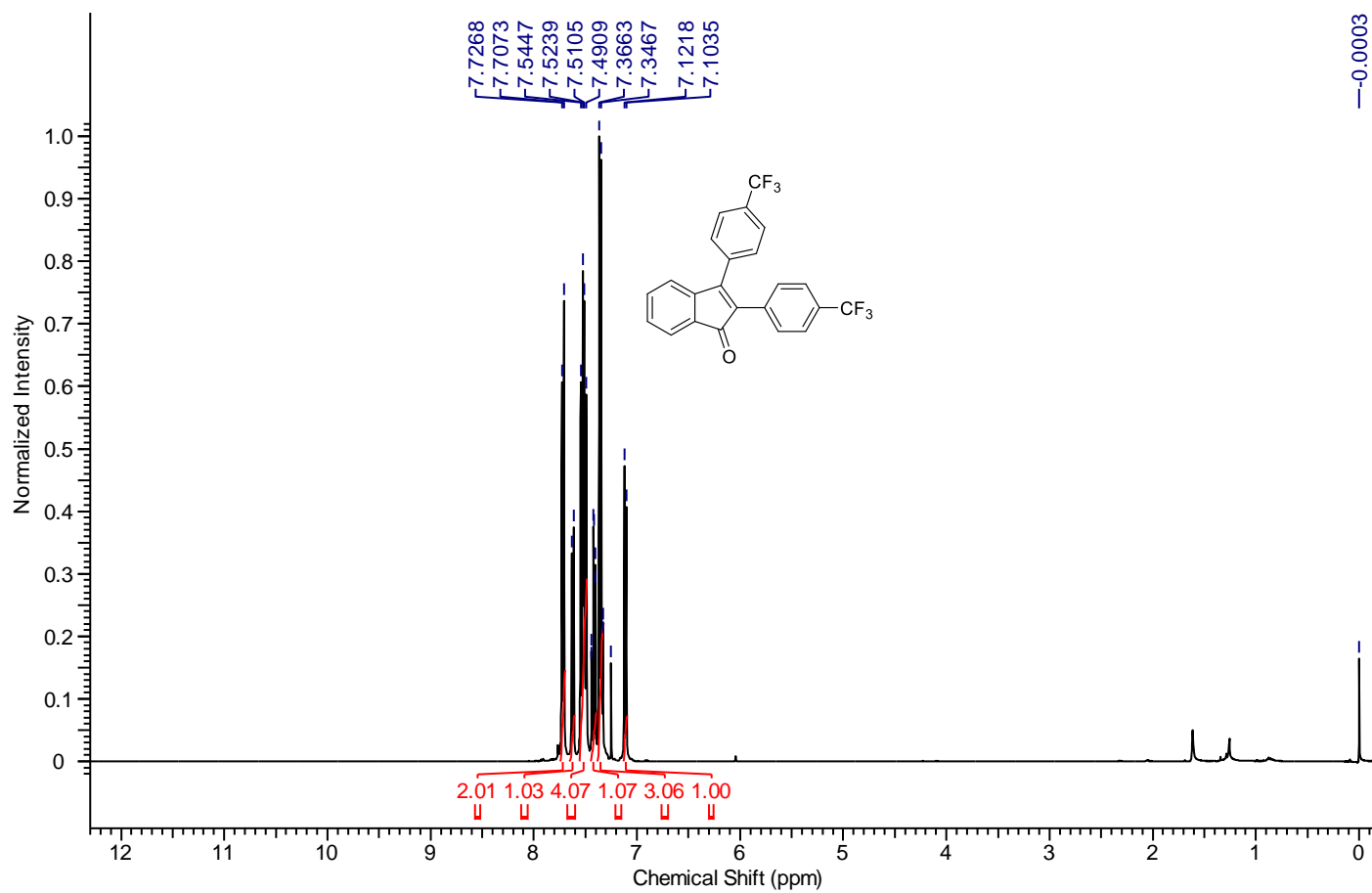


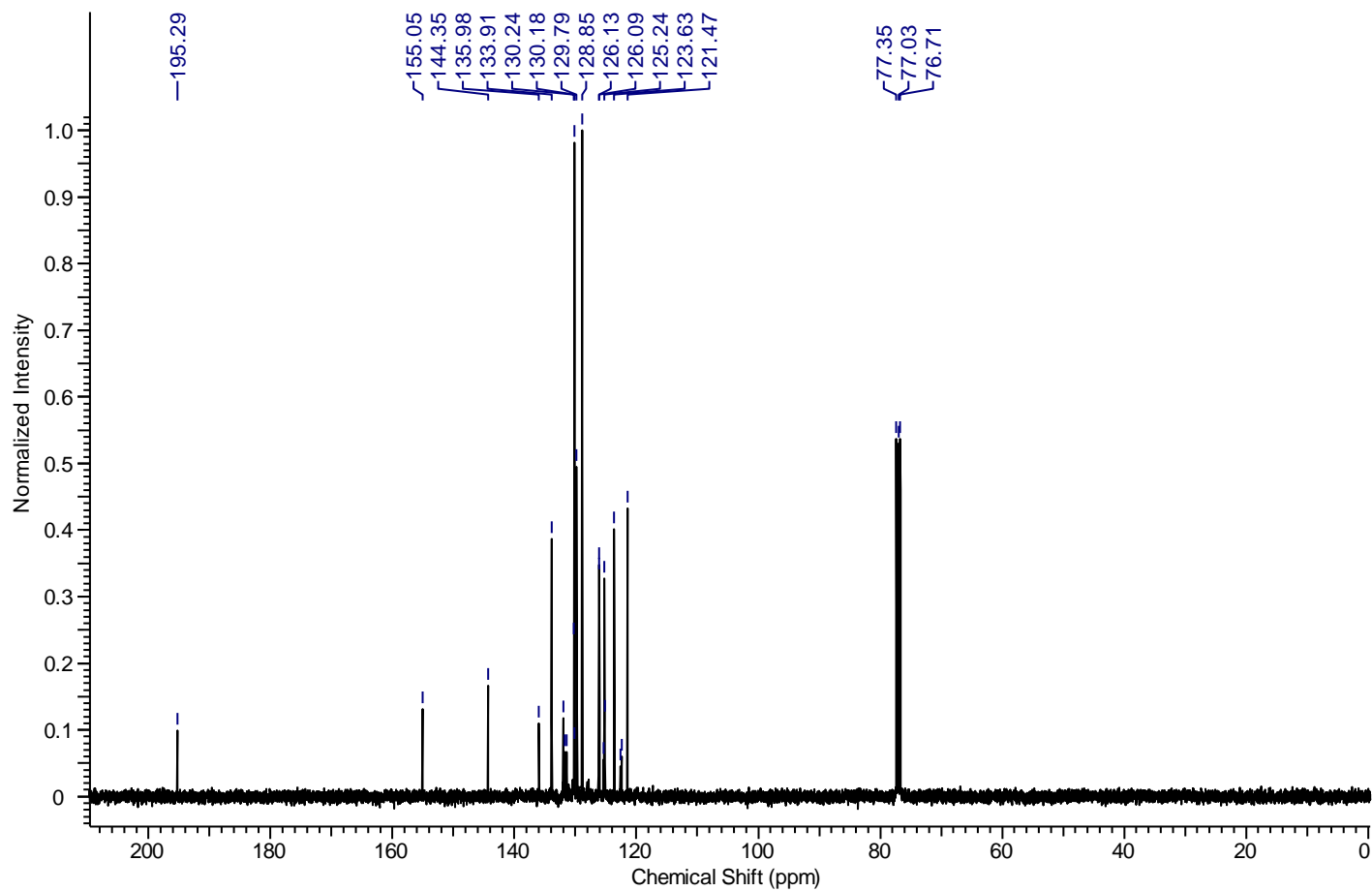
¹H (400 MHz) and ¹³C (100 MHz) NMR in CDCl₃ of Compound 4n





¹H (400 MHz) and ¹³C (100 MHz) NMR in CDCl₃ of Compound 4o





2. Single crystal X-ray data:

- X-ray crystal structure data for 1a,6a-di-m-tolyl-1a,6a-dihydro-6H-indeno[1,2-b]oxiren-6-one (2a): CCDC Number-2110069

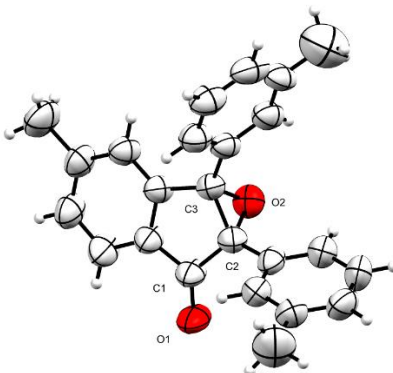


Figure S1. Perspective Drawing of **2a** with 40% Ellipsoid Probability.

Single crystals of **2a** was obtained by slow evaporation of chloroform-methanol solution at room temperature.

Table S1. Single Crystallographic Data of 2a

| | |
|------------------------------------|--|
| Empirical formula | C ₂₄ H ₂₀ O ₂ |
| Formula weight | 340.40 |
| Temperature/K | 293.0 |
| Crystal system | monoclinic |
| Space group | P2 ₁ /n |
| a/Å | 18.013(2) |
| b/Å | 10.9258(16) |
| c/Å | 19.1838(15) |
| α/° | 90 |
| β/° | 93.691(7) |
| γ/° | 90 |
| Volume/Å ³ | 3767.7(8) |
| Z | 8 |
| ρ _{calc} /cm ³ | 1.200 |
| μ/mm ⁻¹ | 0.075 |

| | |
|---|---|
| F(000) | 1440.0 |
| Crystal size/mm ³ | 0.08 × 0.06 × 0.04 |
| Radiation | MoK α (λ = 0.71073) |
| 2 Θ range for data collection/° | 5.87 to 58.21 |
| Index ranges | -24 ≤ h ≤ 14, -14 ≤ k ≤ 13, -23 ≤ l ≤ 24 |
| Reflections collected | 19874 |
| Independent reflections | 8706 [R _{int} = 0.0400, R _{sigma} = 0.0702] |
| Data/restraints/parameters | 8706/0/475 |
| Goodness-of-fit on F ² | 1.036 |
| Final R indexes [I ≥ 2 σ (I)] | R ₁ = 0.1144, wR ₂ = 0.3373 |
| Final R indexes [all data] | R ₁ = 0.2117, wR ₂ = 0.4170 |
| Largest diff. peak/hole / e Å ⁻³ | 1.11/-0.41 |

- **X-ray crystal structure data for 2-hydroxy-5-methyl-2,3-diphenyl-2,3-dihydro-1H-inden-1-one (3ab): CCDC Number-2110074**

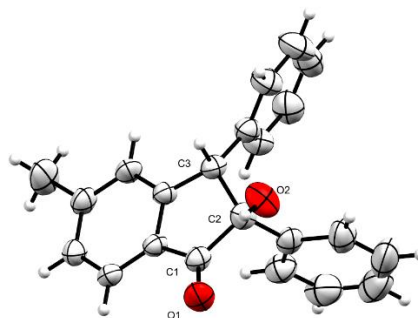


Figure S2. Perspective Drawing of **3ab** with 40% Ellipsoid Probability.

Single crystals of **3ab** was obtained by slow evaporation of DCM-Hexane solution at room temperature.

Table S2. Single Crystallographic Data of **3ab**.

| | |
|-------------------|--|
| Empirical formula | C ₂₂ H ₁₈ O ₂ |
| Formula weight | 314.36 |
| Temperature/K | 301.41(10) |
| Crystal system | monoclinic |

| | |
|---|---|
| Space group | P2 ₁ /n |
| a/Å | 8.6355(11) |
| b/Å | 6.4732(8) |
| c/Å | 30.237(3) |
| α/° | 90 |
| β/° | 91.300(11) |
| γ/° | 90 |
| Volume/Å ³ | 1689.8(4) |
| Z | 4 |
| ρ _{calc} /cm ³ | 1.236 |
| μ/mm ⁻¹ | 0.078 |
| F(000) | 664.0 |
| Crystal size/mm ³ | 0.18 × 0.14 × 0.12 |
| Radiation | Mo Kα (λ = 0.71073) |
| 2Θ range for data collection/° | 6.144 to 58.06 |
| Index ranges | -11 ≤ h ≤ 9, -8 ≤ k ≤ 8, -40 ≤ l ≤ 38 |
| Reflections collected | 15595 |
| Independent reflections | 3961 [R _{int} = 0.1080, R _{sigma} = 0.0752] |
| Data/restraints/parameters | 3961/0/219 |
| Goodness-of-fit on F ² | 1.097 |
| Final R indexes [I >= 2σ (I)] | R ₁ = 0.1086, wR ₂ = 0.2983 |
| Final R indexes [all data] | R ₁ = 0.1361, wR ₂ = 0.3138 |
| Largest diff. peak/hole / e Å ⁻³ | 0.35/-0.29 |

- **X-ray crystal structure data for 2-hydroxy-5-methyl-2,3-di-*p*-tolyl-2,3-dihydro-1*H*-inden-1-one (3ac): CCDC Number-2109773**

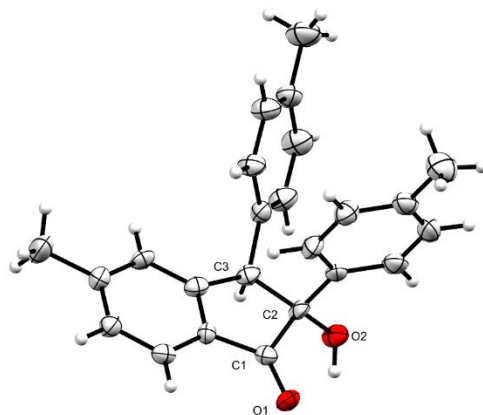


Figure S3. Perspective Drawing of **3ac** with 40% Ellipsoid Probability.

Single crystals of **3ac** was obtained by slow evaporation of DCM-Hexane solution at room temperature.

Table 3. Single Crystallographic Data of 3ac.

| | |
|------------------------------------|--|
| Empirical formula | C ₂₄ H ₂₂ O ₂ |
| Formula weight | 342.16 |
| Temperature/K | 296.15 |
| Crystal system | monoclinic |
| Space group | P2 ₁ /n |
| a/Å | 8.739(5) |
| b/Å | 6.410(4) |
| c/Å | 33.594(18) |
| α/° | 90 |
| β/° | 94.590(17) |
| γ/° | 90 |
| Volume/Å ³ | 1875.7(18) |
| Z | 8 |
| ρ _{calc} /cm ³ | 1.213 |
| μ/mm ⁻¹ | 0.076 |

| | |
|--|---|
| F(000) | 728.0 |
| Crystal size/mm ³ | 0.027 × 0.023 × 0.012 |
| Radiation | MoK α (λ = 0.71073) |
| 2 Θ range for data collection/ $^{\circ}$ | 2.432 to 56.67 |
| Index ranges | -6 ≤ h ≤ 11, -8 ≤ k ≤ 8, -32 ≤ l ≤ 44 |
| Reflections collected | 7261 |
| Independent reflections | 4476 [R _{int} = 0.1120, R _{sigma} = 0.2695] |
| Data/restraints/parameters | 4476/0/240 |
| Goodness-of-fit on F ² | 0.901 |
| Final R indexes [I ≥ 2 σ (I)] | R ₁ = 0.0828, wR ₂ = 0.1533 |
| Final R indexes [all data] | R ₁ = 0.2604, wR ₂ = 0.2431 |
| Largest diff. peak/hole / e \AA^{-3} | 0.42/-0.48 |