Supporting Information for

Regioselective Cope Rearrangement and Prenyltransfers on Indole Scaffold Mimicking Fungal and Bacterial Dimethyl Allyl Tryptophan Synthases

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and Rajesh Viswanathan[‡,*]

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Abbreviations

DMATS	=	Dimethyl Allyl Tryptophan Synthase
EtOAc	=	Ethylacetate
Et_3N	=	Triethylamine
NCS	=	*N*-chlorosuccinimide
TLC	=	Thin layer chromatography
IR	=	Infrared
NMR	=	Nuclear Magnetic Resonance
MHz	=	Mega Hertz
$CDCl_3$	=	Deuterated chloroform
DMSO-d_6	=	Deuterated dimethyl sulfoxide
CD_3OD	=	Deuterated methanol
HRMS	=	High Resolution Mass Spectrometry
LC-MS	=	Liquid Chromatography-Mass Spectrometry
ESI	=	Electrospray Ionization
δ	=	Chemical shift
rt	=	Room temperature
h	=	Hour
d	=	Day(s)
satd.	=	Saturated
anhyd.	=	Anhydrous
DMAc	=	*N*,*N*-Dimethyl acetamide
KOH	=	Potassium hydroxide
DMAc	=	*N*,*N*-dimethylacetamide
AIBN	=	2,2’-azo*bis*isobutyronitrile
DMF	=	*N*,*N*-dimethylformamide
CD_3OD	=	Deuterated methanol
DKP	=	Diketopiperazine
BaO	=	Barium oxide
THF	=	Tetrahydrofuran
L-Trp	=	L-tryptophan
Phth	=	Phthalyl

Reagents, Solvents and Glassware:

All commercially available reagents were purchased as analytically pure substances and were used as received. Their storage followed recommended manufacturer's protocols. All small-scale dry reactions were carried out under a blanket of nitrogen, using standard syringe-septum, and cannulation techniques.[1] Reactions were monitored by thin-layer chromatography (TLC) on silica gel plates (60 F_{254}) with a fluorescent indicator, and independently visualized with UV light (254 nm). Prenyl alcohol was freshly distilled from potassium carbonate before use. Similarly, geraniol, nerol, farnesol and allyl alcohol were freshly distilled prior to use. NCS was recrystallized from benzene. 1,4-Dimethyl piperazine was distilled from KOH. CCl_3COOH was dried by azeotropic distillation from benzene and stored under nitrogen. *N*,*N*-Dimethyl acetamide (DMAc) was distilled from BaO and used within 3 days from the time of drying. Anhydrous ether, hexanes, dichloromethane and THF were obtained from an m-Braun solvent purification system (charged with A2 alumina as a desiccant).[2] All other solvents were purified according to specific literature procedures.[3] Thermal reactions involving *N*,*N*-dimethyl acetamide and decalin were carried out in Schlenk flasks[4] (analogous to Chemglass [AF-0523] or [AF-0523]) Schlenk flask. Indole C3-reverse prenylation to prepare substrates for Cope rearrangement were carried out according to procedure published by Booker-Milburn et al.[5]. Isatin-derived C3-reverse

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[1] Pirrung, M. C.; Chapter 8: Conducting the Reaction Itself, *The Synthetic Organic Chemist's Companion*, John Wiley & Sons Inc., Hoboken, NJ, 2007, 69-91.

[2] Pangborn, A. B.; Giardello, M. A.; Grubbs, R. H.; Rosen, R. K. Timmers, F. J. Safe and Convenient Procedure for Solvent Purification. *Organometallics*, **1996**, *15*, 1518 -1520.

[3] Armarego, W. L. F.; Chai, C. L. L.; *Purification of Laboratory Chemicals*, 5th Ed. Elsevier Butterworth-Heinemann, 2003.

[4] Custom-made by NavCour Glassware, Cleveland.

prenylated **21a**, **21b**, and **21c** were prepared using published literature methods.[6] C3-substituted indoles used in this study as precursors for reverse prenylation were prepared through published literature methods.[7] C3-reverse prenyl oxindole **16b** was synthesized as reported in reference 7i.

Chromatography:

Silica Gel 60 Å (230-400 mesh) was obtained from Sorbent Technologies. For the purification of C3-reverse prenylated compounds (**16a-r**), the silica gel was deactivated with Et_3N (10% in hexane) prior to use. For the purification of C4-prenyl rearrangement products (**17a-r**), silica gel was pretreated with 1:4 v/v of Et_3N and hexane and stirred overnight. Silica gel was filtered and dried under vacuo (ca. 100 – 200 mbar) and stored in an air-tight container until further use. Resulting silica was treated with a v:v:v mixture of Et_3N:EtOAc:hexanes (1:1.5:8.5) prior to use in flash column chromatography. Separations of compounds were carried out using flash technique[8] (Silica gel grade: 200-400 mesh, 40-63 μm) at medium pressure (20 psi). Preparatory thin-layer chromatography (TLC) was performed on pre-coated glass plates (7.5 x 2.5 and 7.5 x 5.0 cm) coated with 60 Å silica gel. The TLC plate was pretreated by eluting with a mixture of

[5] Booker-Milburn, K. I; Fedouloff, M; Paknoham, S. J; Strachan, J. B; Melville, J. L; Voyle, M. *Tetrahedron Letters*, **2000**, *41*, 4657-4661.
[6] Nair, V; Ros, S; Jayan C. N.; Viji, S. *Synthesis* **2003**, *16*, 2542-2546.

[7] a) Yadav, J. S.; Abraham, S.; Reddy, B. V. S; Sabitha, G. *Synthesis* **2001**, *14*, 2165-2169. b) Ferreira, E. M; Stoltz, B. M. *J. Am. Chem. Soc*. **2003**, *125*, 9578-9579. c) Adla, S. K.; Golz, G.; Jones, P. G.; Lindel, T. *Synthesis* **2010**, 2161-2170. d) Giraud, F.; Marchand, P.; Carbonnelle, D.; Sartor, M.; Lang, F.; Duflos, M. *Bioorg. Med. Chem. Lett.* **2010**, *20*, 5203-5206. e) Kumar, V.; Kuar, S.; Kumar, S. *Tetrahedron Letters*, **2006**, *47*, 7001-7005. f) Wu, J.; Zhao, M.; Qian, K.; Lee, K. H.; Morris-Natschke, S; Peng, S. *Eur. J. Med. Chem.* **2009**, *44*, 4153-4161. g) Brooks, S; Sainsbury, M; Weerasinge, D. K. *Tetrahedron*, **1982**, *38*, 3019-3022. h) Dolusic, E.; Larrieu, P.; Moineaux, L.; Stroobant, V.; Pilotte, L.; Colau, D.; Pochet, L.; Van den Eynde, B.; Masereel, B.; Wouters, J.; Frederick, R. *J. Med. Chem.***2011**, *54*, 5320-5334. i) Ignatenko, V. A.; Zhang, P.; Viswanathan, R. *Tetrahedron Letters*, **2011**, *52*, 1269-1272.
[8] Still, W. C.; Kahn, M.; Mitra, A. Rapid chromatographic technique for preparative separations with moderate resolution. *J. Org. Chem.* **1978**, *43*, 2923-2925.

Et_3N:EtOAc:hexanes (1:4:5). The loaded preparative plates were eluted with 40% EtOAc in hexanes.

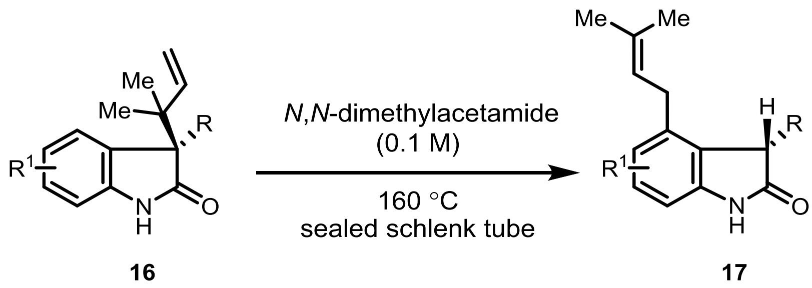
Structural Characterization of Intermediates and Products:

NMR spectroscopic analyses (^{1}H NMR, ^{13}C NMR) were conducted for all new compounds. ^{1}H (400 MHz) and ^{13}C (100 MHz) spectra were recorded on a 400 MHz spectrophotometer, with the exception of a few compounds recorded on a 600 M Hz spectrophotometer (^{1}H: 600 MHz and ^{13}C: 150 MHz). Pertinent frequency is specifically reported for each compound. Chemical shift values (δ) for NMR spectra are reported in parts per million (ppm) relative to the residual (indicated) solvent peak ($CDCl_3$ or CD_3OD).[9] Coupling constants are reported in Hz. Data for ^{1}H NMR are reported as follows: chemical shift (δ, ppm), multiplicity (s = singlet, brs = broad singlet, d = doublet, t = triplet, q = quartet, ddd = double double doublet, m = multiplet), integration corresponding to the number of protons followed by coupling constants in Hz. For ^{13}C NMR spectra, the nature of the carbons (C, CH, CH_2 or CH_3) was determined by recording the DEPT experiment, and notations are provided in parentheses. ^{13}C NMR data is reported in parts per million (δ) relative to the residual (indicated) solvent peak. All melting points for solids are reported uncorrected. IR spectra were recorded on a FT-IR spectrometer. In order to avoid temperature fluctuations that cause large aberrations in the result of [3,3] and [1,3] prenyl transfer reactions, a stirrer with digitally controlled hot plate was used for monitoring all thermal reactions. Positive ion-mode ElectroSpray Ionization High Resolution-Mass Spectrometry

[9] (a). Gottileb, H. E.; Kotlyar, V.; Nudelman, A. NMR Chemical Shifts of Common Laboratory Solvents as Tace Impurities *J. Org. Chem.* **1997**, *62*, 7512-7515 and (b). Fulmer, G. R.; Miller, A. J. M.; Sherden, N. H.; Gottileb, H. E.; Nudelman, A.; Stoltz, B. M.; Bercaw, J. E.; Goldberg, K. I. NMR Chemical Shifts of Trace Impurities: Common Laboratory Solvents, Organics, and Gases in Deuterated Solvents Relevant to the Organometallic Chemist *Organometallics*, **2010**, *29*, 2176-2179.

(HRMS-ESI$^+$) data for compounds reported herein were obtained by direct infusion of methanolic solutions on a QTOF mass spectrometer. Direct Electron-Impact (EI) mode was applied for a few compounds. HPLC analyses for **16s** and **17s** were performed using a Shimadzu LC-20-AT Series separations module. equipped with a photo diode array and multiple wavelength detectors (180 nm-800 nm). For these oxindoles, UV detector was set at 310 nm with a 5 nm slit-width. The overall system was controlled using LC Solutions software. Raw traces were plotted using Origin® software after exporting absorbance data as an ASCII-formatted file. Analytical separations of diastereomers of **17s** were carried out on Daicel® (normal phase) AS chiral column. A 10% isopropanol/ 90% hexanes mixture was used as elution medium with a flow rate of 1 mL/min in an isocratic mode. Individual retention times for are reported along with analytical data for each isomer. A CEM Discover microwave reactor was used for reactions. An internal sensor mode was employed for careful tuning of the temperature for Cope rearrangement reactions reported herein.

General notes for Cope rearrangement



General Notes:

i) The reverse prenylated oxindole should be free of residual chlorinated solvents.

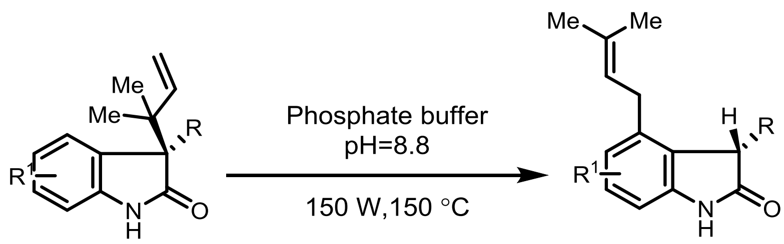
ii) *N*, *N*–Dimethylacetamide should be freshly distilled over BaO.

iii) Volume of reaction solvent in the dry Schlenk tube should be controlled to minimize empty air space (after addition of reactants).

iv) Precise control of heating temperature of the reaction using a digital thermostat helps prevent thermal decomposition of the reactants.

The C3-reverse prenylated oxindole **16s** was taken in a vial and capped with a septum. Under nitrogen, the oxindole (**16b**) was dissolved in DMAc to form a 0.1 M solution. The solution was transferred to a Schlenk tube using a syringe and capped under nitrogen and heated in an oil bath at 155 °C for 40 h. The reaction mixture was allowed to cool to rt and DMAc was removed *in vacuo*. The reaction mixture was diluted with ethylacetate and washed with brine. The organic layer was dried with anhyd. Na_2SO_4 and evaporated to get the crude that was subjected to silica gel chromatography (refer to general experimental details) eluted with ca.1:4 v/v ethylacetate and hexane and the product oxindole **17s** (as a representative example) was isolated.

General notes for Cope rearrangement under microwave irradiation

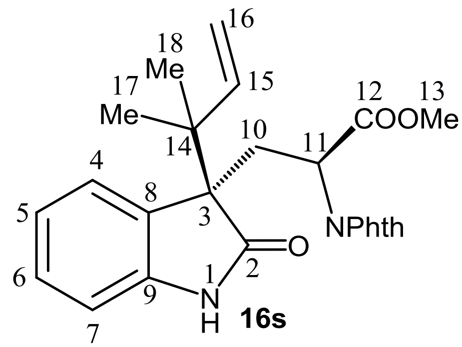


16 17

A 10 mM phosphate buffer (pH 8.8) was prepared by dissolving 3 mg of monosodium phosphate monohydrate and 262 mg of disodium hydrogen phosphate in 100 mL of doubly deionized water. C3-reverse prenylated oxindole (**16b**, 30 mg) was dissolved in 0.75mL of buffer, all in a microwave-compatible sealed tube, and subjected to irradiation of 150 W (watts) and 150 °C over 40 minutes. The reaction mass was extracted with ethylacetate (10 mL) and washed with brine (10 mL). Organic layer was dried with anhyd. Na_2SO_4 and evaporated to get the crude reaction mixture which upon purification by preparative TLC (refer general experimental details) gave the product **17b** in 75% yield.

Table S1. ^{1}H NMR assignment for mixture of two diastereomers of 16s.

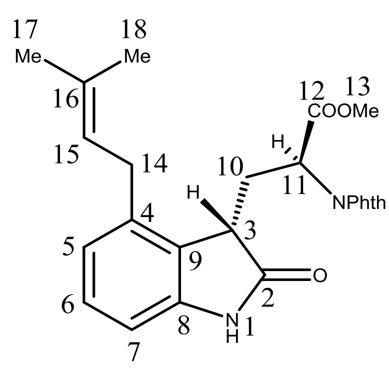
S. No	Chemical shift	Description
1	1.15 (s, 3H), 1.03 (s, 3H)	Gem di methyl (one diastereomer)
2	1.12 (s, 3H), 0.99 (s, 3H)	Gem di methyl (one diastereomer)
3	3.21 (ABX, *J* = 14.8, 11.1 Hz, 1H), 3.05-2.95 (m, 2H), 2.90 (ABX, *J* = 14.8, 2.2 Hz, 1H)	C10 protons
4	3.68 (s, 3H), 3.63 (s, 3H)	-$COOCH_3$
5	4.79 (dd, *J* = 10.3, 4.6 Hz, 1H), 4.25 (dd, *J* = 11.1, 2.1 Hz, 1H)	C11 protons
6	6.07 (t, *J* = 11.2 Hz, 1H), 6.02 (t, *J* = 11.2 Hz, 1H)	C15 proton
7	5.13 (dd, *J* = 7.4, 1.1 Hz, 1H), 5.10 (dd, *J* = 7.4, 1.1 Hz, 1H), 5.03 (dd, *J* = 15.1, 1.1 Hz, 1H), 4.98 (dd, *J* = 15.1, 1.1 Hz, 1H)	C16 proton
8	7.33-7.24 (m, 2H), 7.09 (td, *J* = 7.6, 1.2 Hz, 1H), 6.87 (dd, *J* = 16.5, 7.6 Hz, 2H), 6.48 (d, *J* = 4.0 Hz, 2H), 6.26 (dt, *J* = 7.4, 4.4 Hz, 1H)	Oxindole aromatic protons
9	7.85-7.79 (m, 2H), 7.72 (brs, 1H), 7.67-7.61 (m, 2H), 7.60-7.51 (m, 4H)	Phthalyl protons
10	8.34 (brs, 1H), 7.72 (brs, 1H)	NH protons



16s

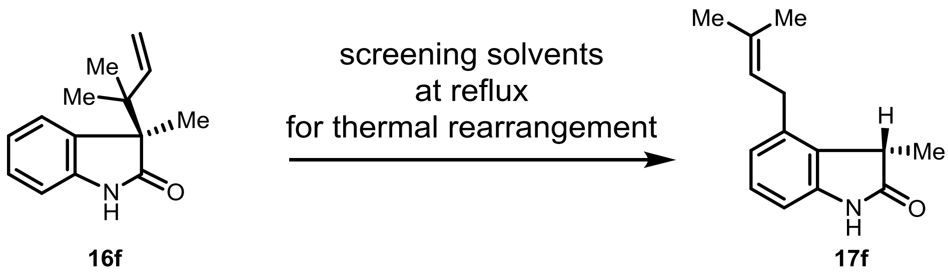
Table S2. [1]H NMR assignment for mixture of two diastereomers of 17s.

S. No	Chemical shift	Description
1	1.74 (s, 6H), 1.73 (s, 6H)	C17,18 methyl protons
2	3.39–3.27 (m, 4H)	C10,C3 & C14 protons
3	3.75–3.70 (m, 1H), 3.69–3.65 (m, 1H)	
4	3.22 (ABX, *J* = 16.0, 6.8 Hz, 2H), 3.43 (ABX, *J* = 15.7, 6.8 Hz, 1H)	
5	3.71 (s, 3H), 3.68 (s, 3H)	$COOCH_3$
6	5.05 (dd, *J* = 9.4, 5.6 Hz, 1H)	C11 protons
7	4.79 (dd, *J* = 11.8, 2.5 Hz, 1H)	
8	5.24 (t, *J* = 6.9 Hz, 1H)	C15 protons
9	5.12 (t, *J* = 6.9 Hz, 1H)	
10	7.20 (t, *J* = 7.7 Hz, 1H), 6.89 (d, *J* = 7.8 Hz, 1H), 6.67 (d, *J* = 7.7 Hz, 1H), 6.61 (t, *J* = 7.8 Hz, 1H), 6.41 (d, *J* = 7.7 Hz, 1H), 6.38 (d, *J* = 7.9 Hz, 1H)	Oxindole aromatic protons
11	7.72 (dd, *J* = 5.5, 3.0 Hz, 2H), 7.47 (dd, *J* = 5.5, 3.0 Hz, 2H)	Phthalyl aromatic protons
12	7.67–7.58 (m, 4H)	
13	8.68 (s, 1H)	NH protons
14	8.89 (s, 1H)	



17s

Table S3. List of Solvents[10] and Corresponding Efficiency of [3,3] and [1,3] prenyltransfer reactions.



Sl.No	Solvent*	% yield[b]	
		[3,3]	[1,3]
1	**Decalin**	**0**	**98** [b]
2	Toluene	< 5[a]	
3	Xylene	< 5[a]	
4	Benzonitrile	< 5[a]	
5	Benzylcyanide	< 5[a]	
6	Butyl ether	< 5[a]	
7	DMSO	< 5[a]	
8	DMF	< 5[a]	
9	Acetic acid	< 5[a]	
10	Nitrobenzene	< 5[a]	
11	*N*-Methyl pyrrolidine	10[a]	
12	*N*-methyl acetamide	< 5[a]	
13	***N,N*-dimethyl acetamide**	**60[b] (75[c])**	**23** [b]
14	H_2O	< 5[a]	
15	^{t}BuOH	< 5[a]	
16	HMPA	10	

* - each reaction was attempted at or slightly above the boiling temperature of respective solvents. [a] – no noticeable conversion. [b] – isolated yield after chromatography. [c] - % conversion estimated from NMR analysis.

[10] Pirrung, M. C.; from "Synthetic Solvent Selection Chart" *The Synthetic Organic Chemist's Companion*, John Wiley & Sons Inc., Hoboken, NJ, 2007, 169-171.

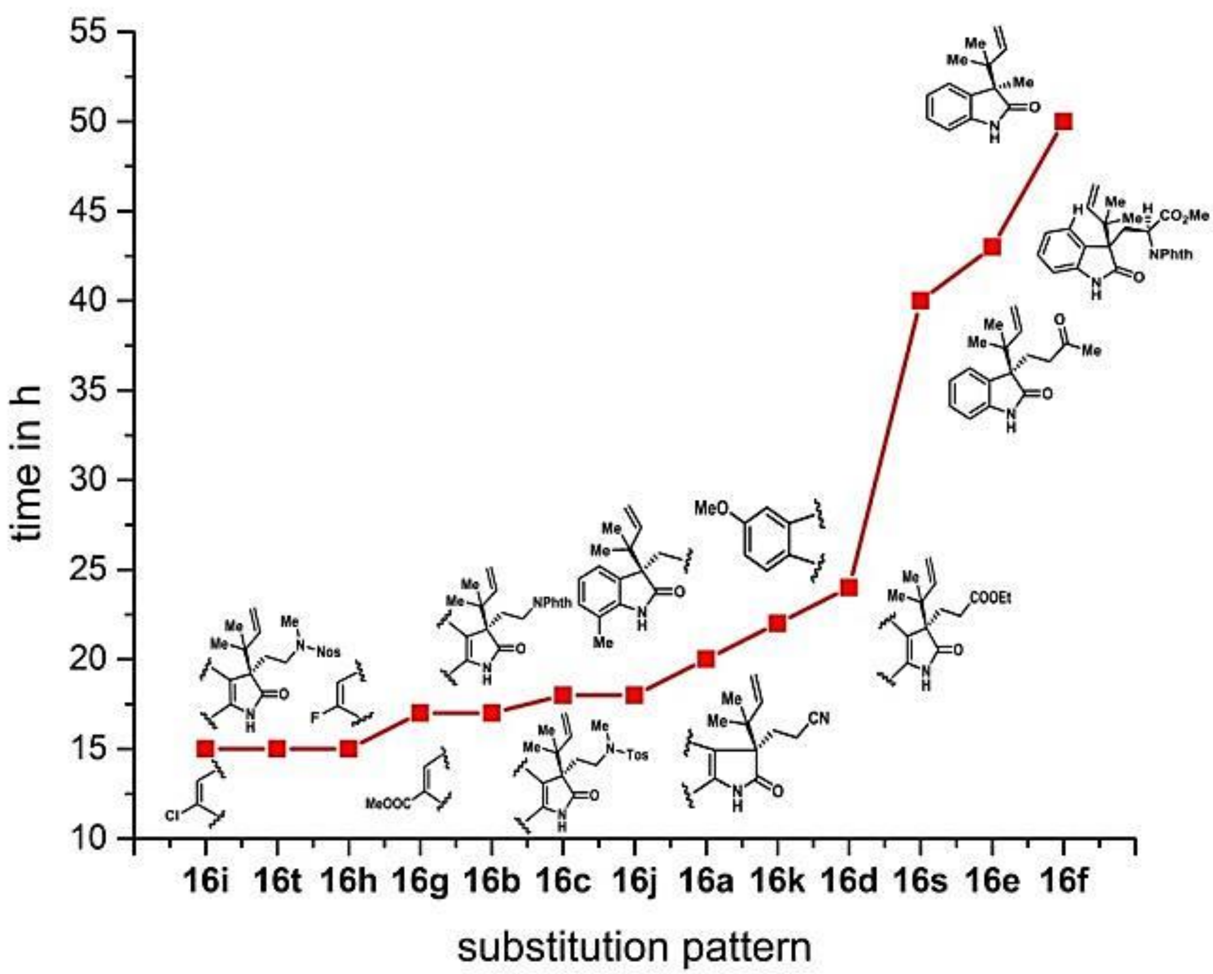


Figure S1. Plot of % conversion as a function of substitution on oxindole 16 upon conversion to 17 under thermal conditions in DMAc.

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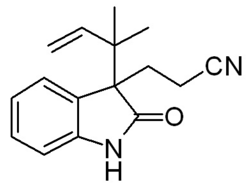

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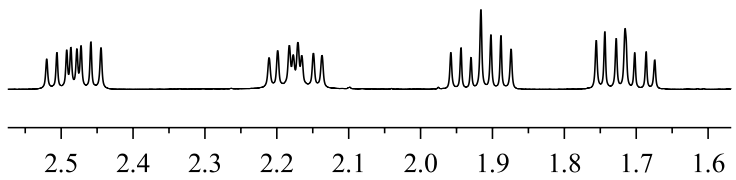

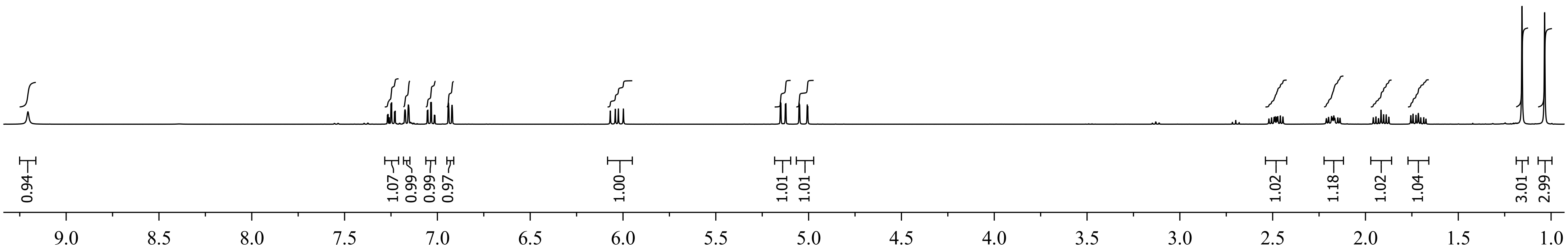

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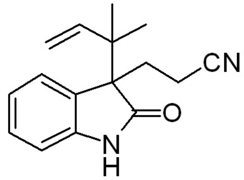

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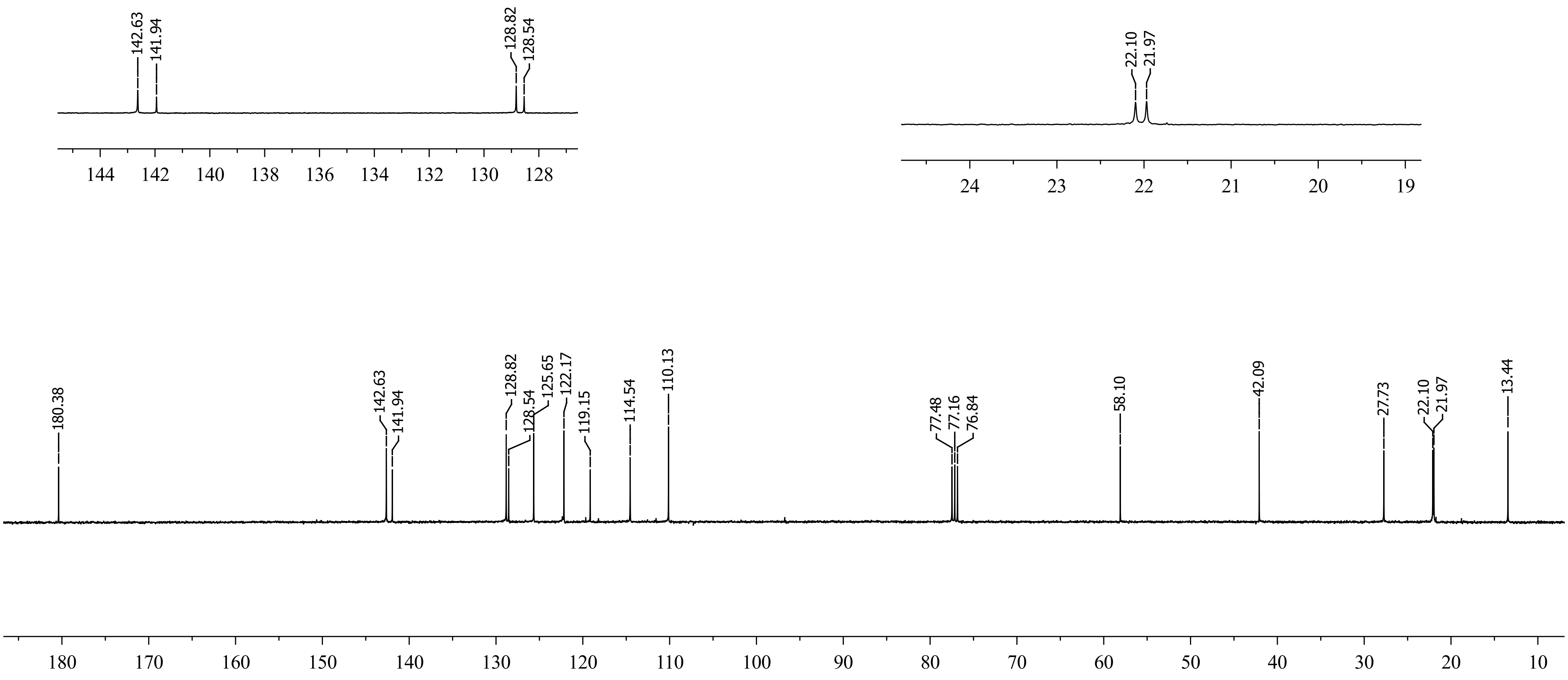

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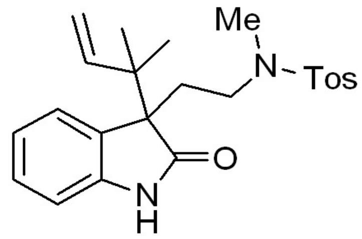

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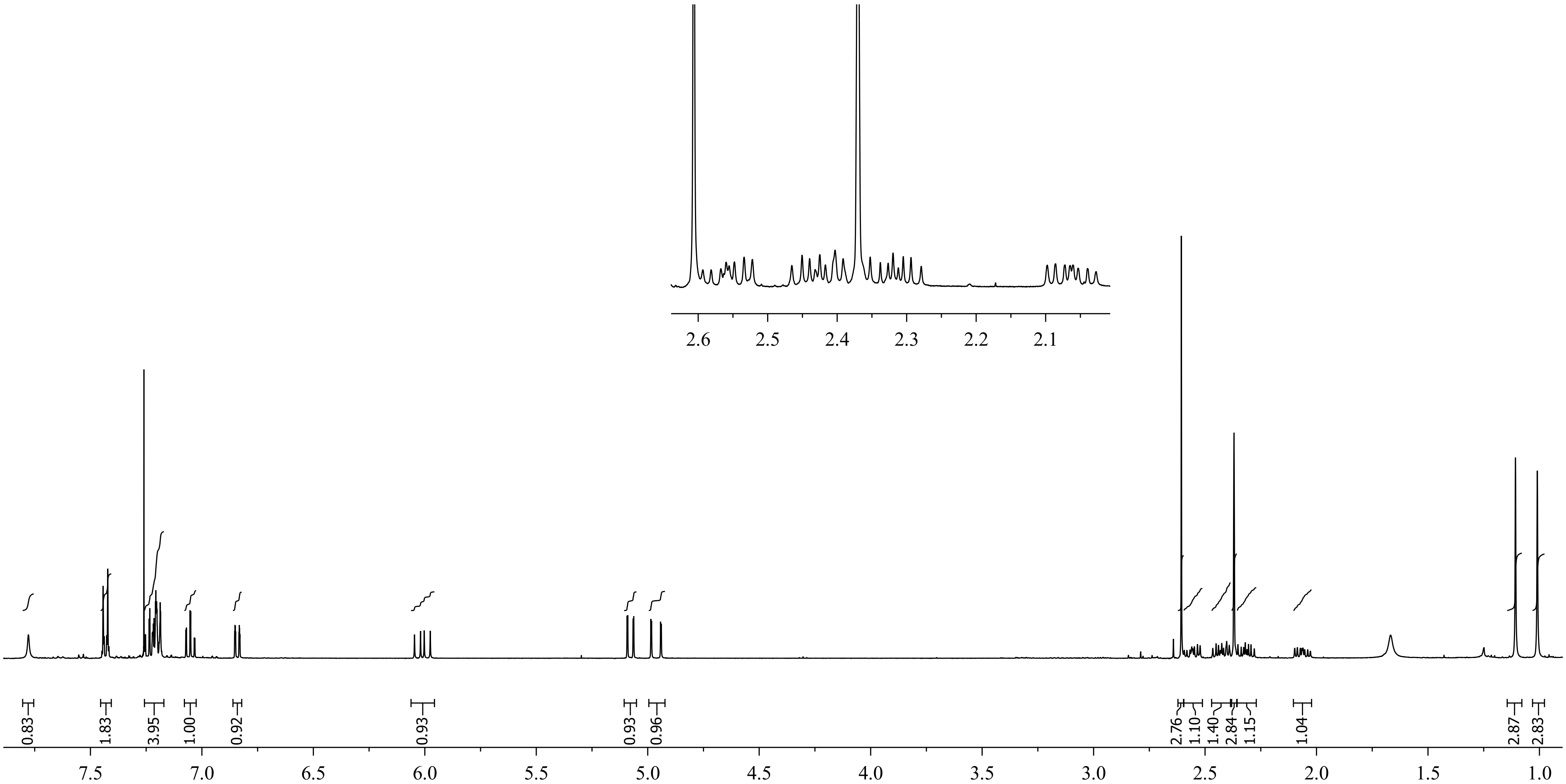

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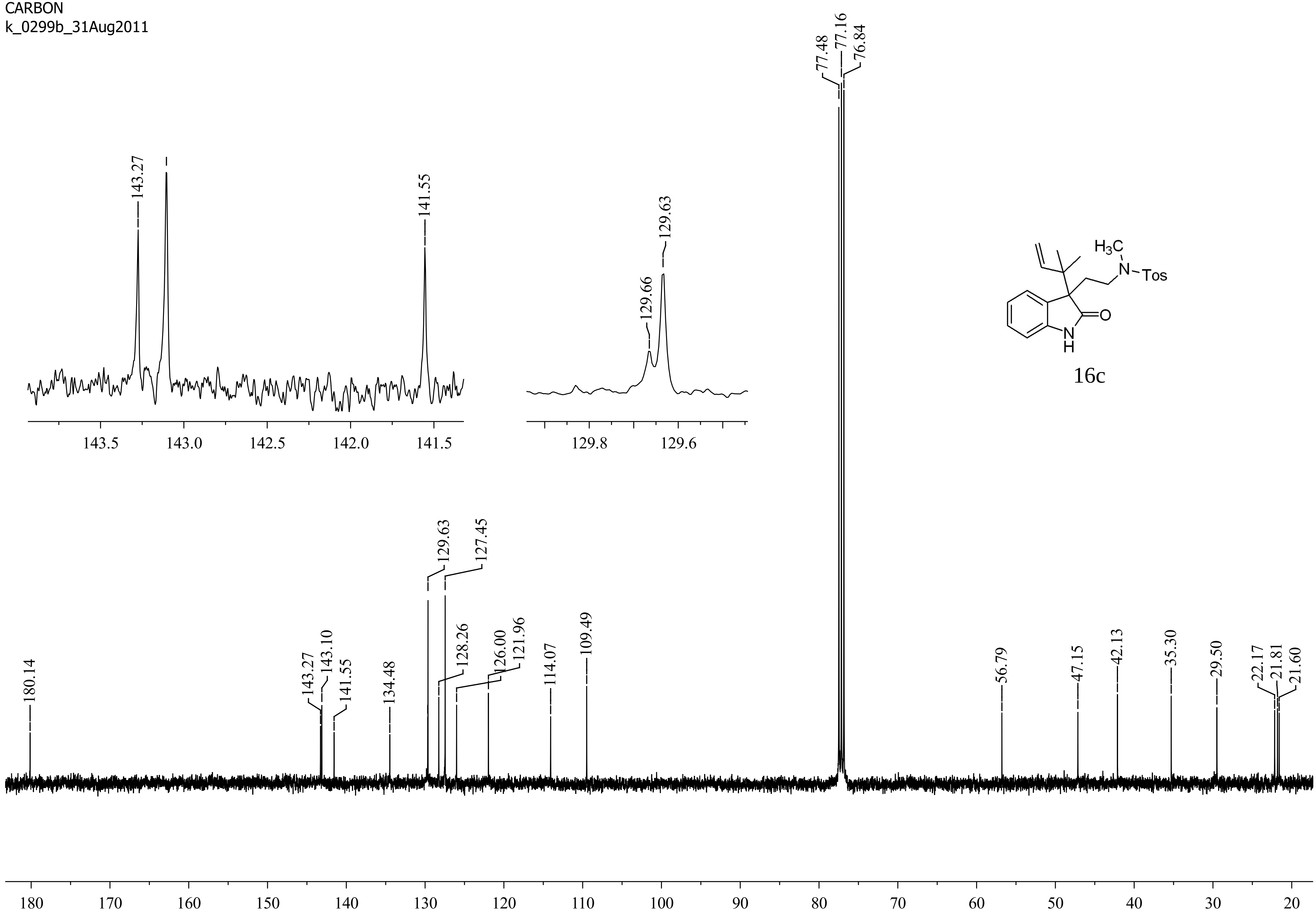

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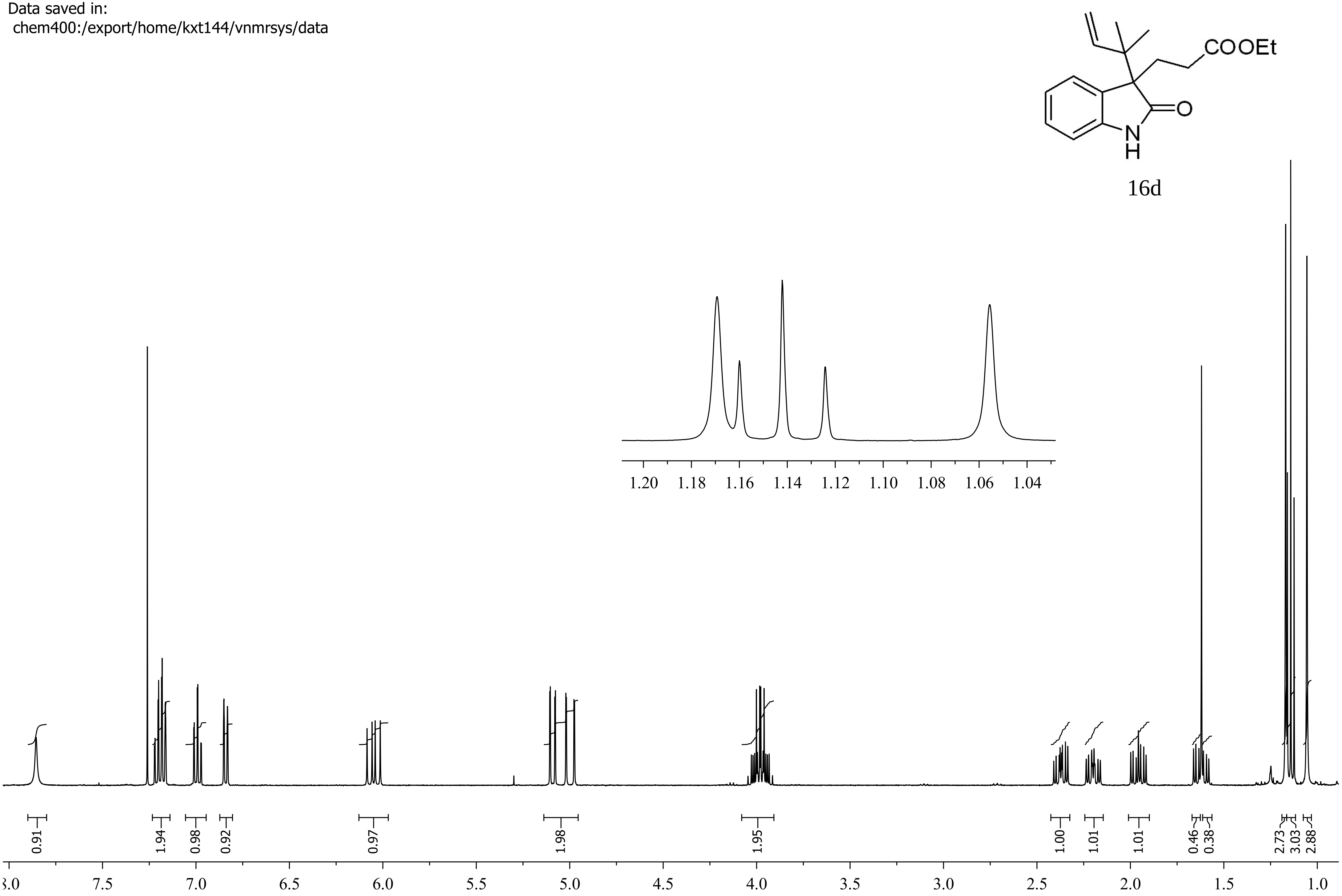

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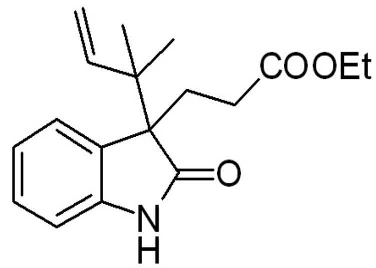

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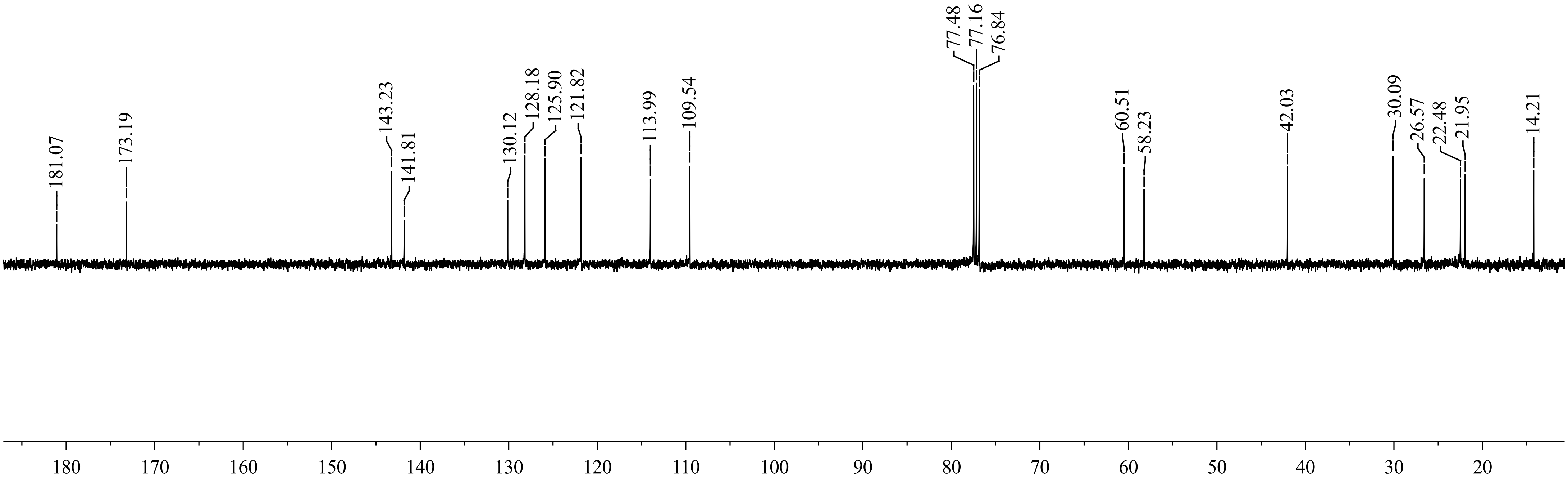

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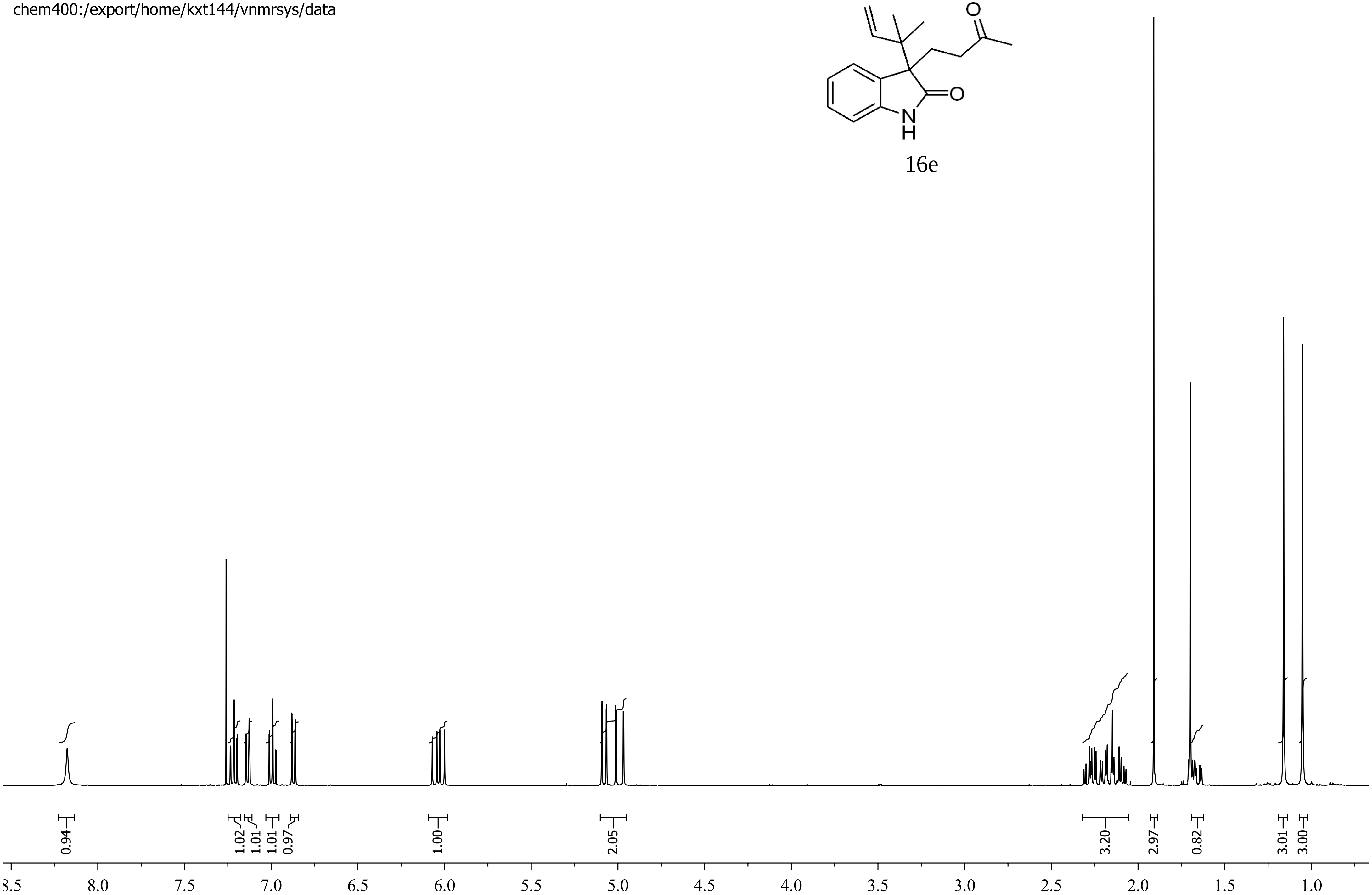

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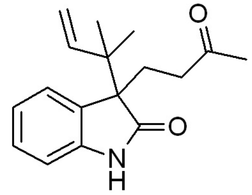

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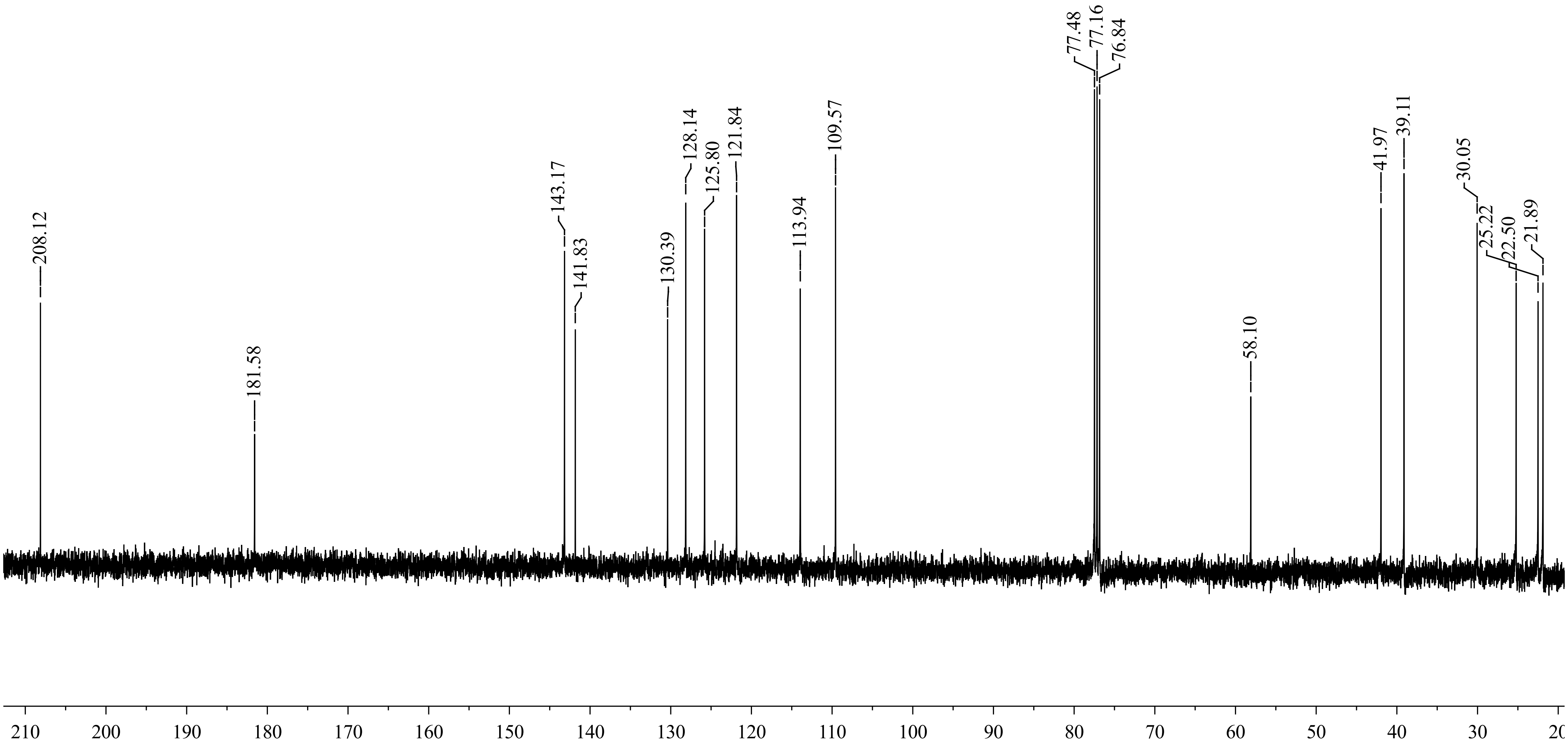

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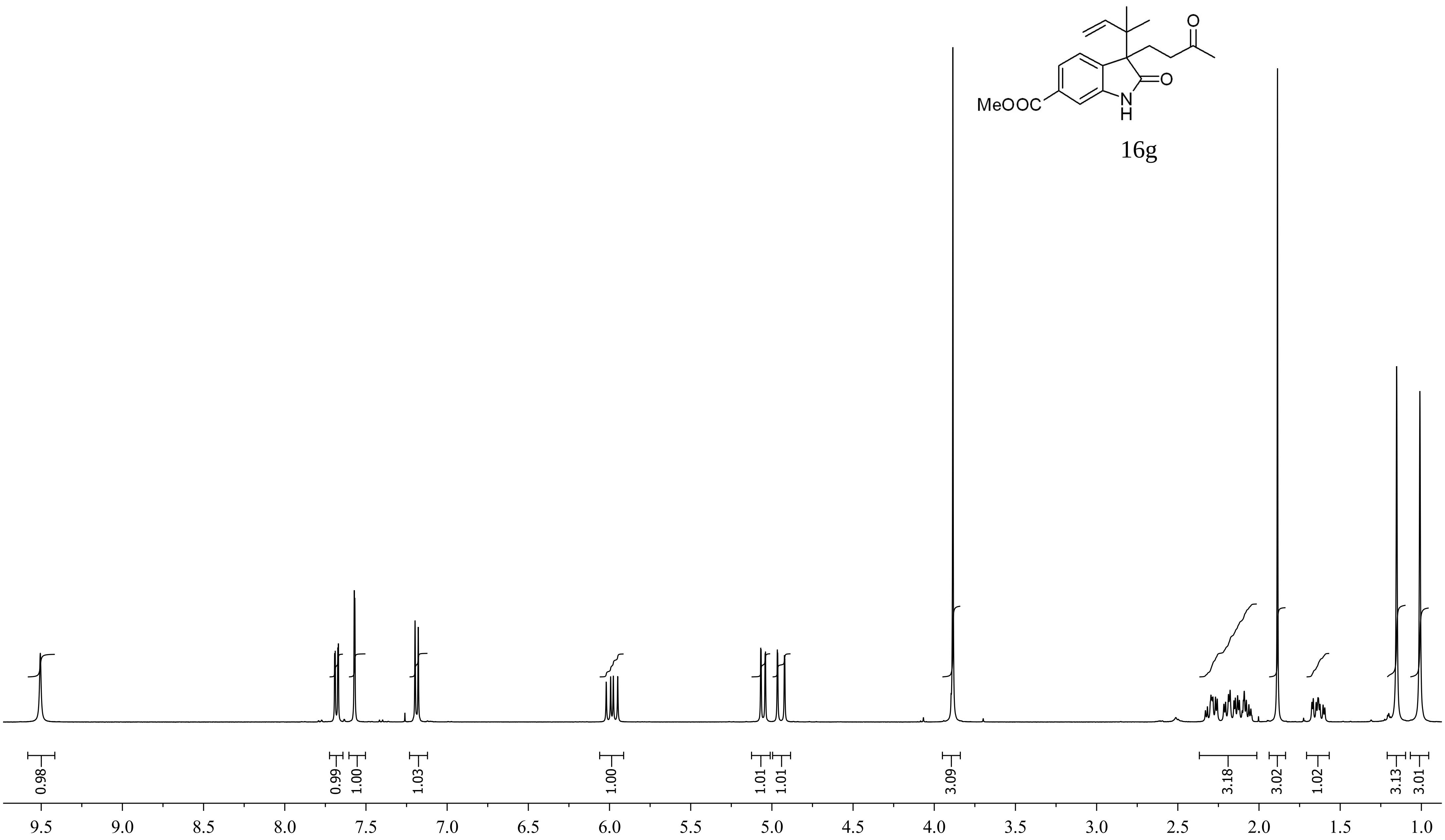

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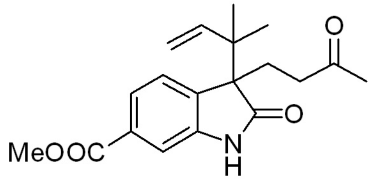

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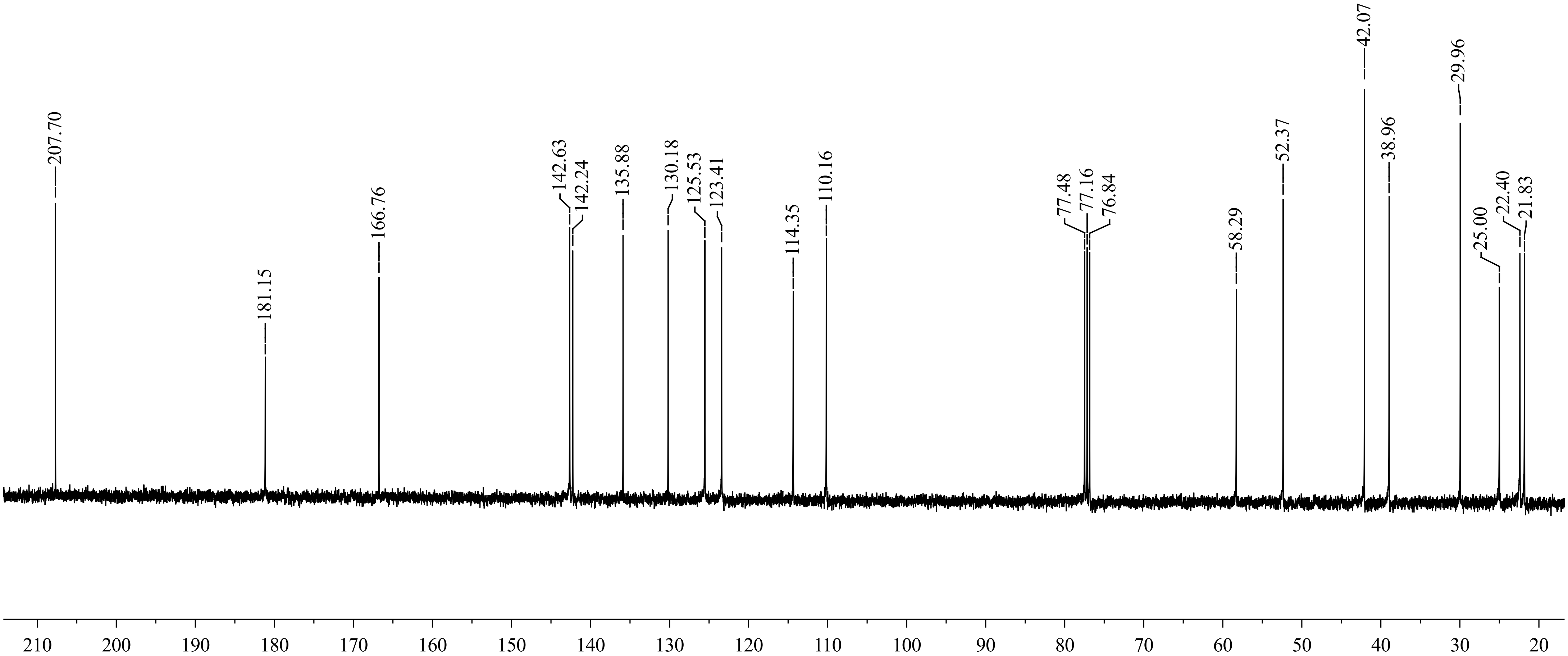

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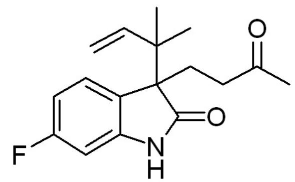

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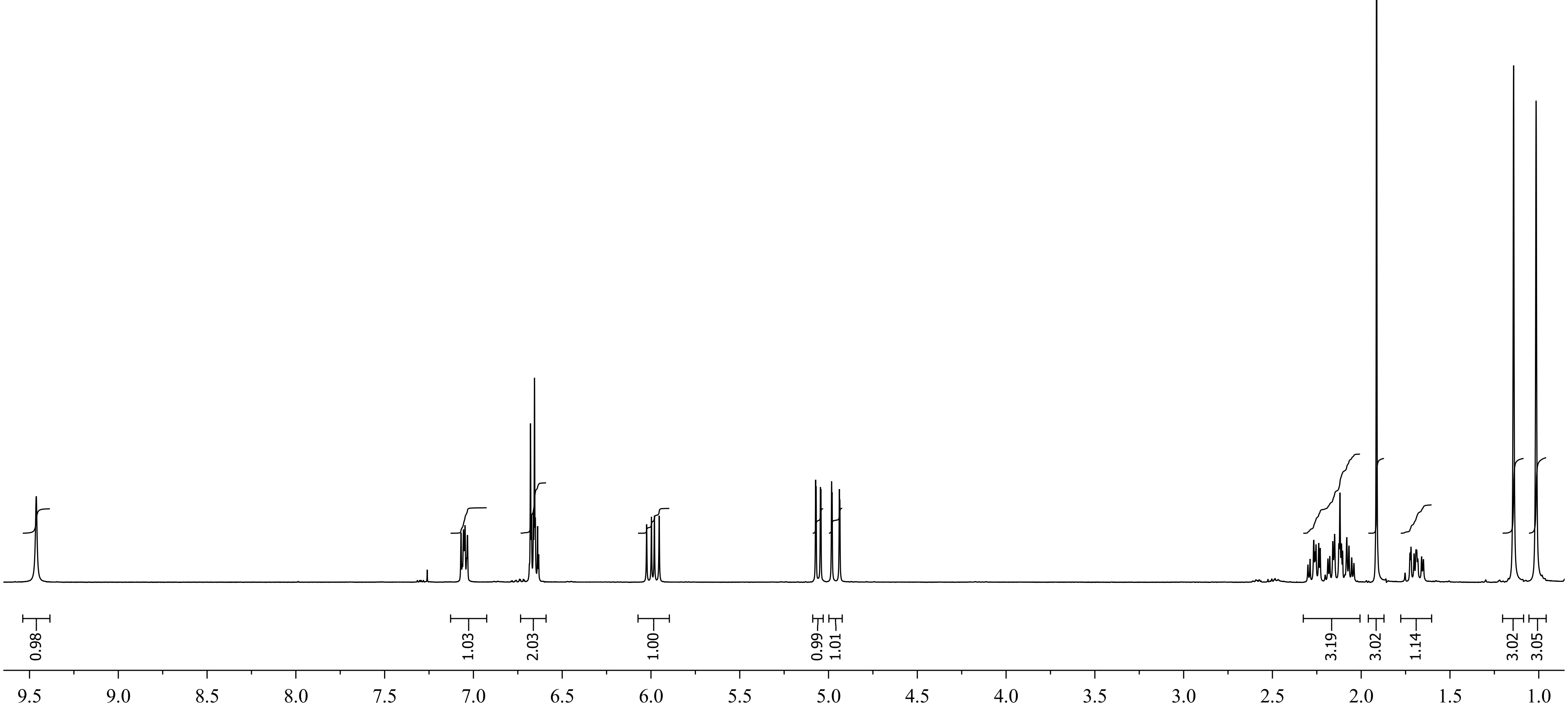

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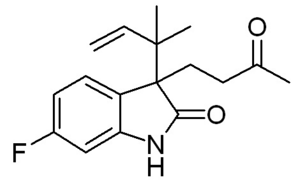

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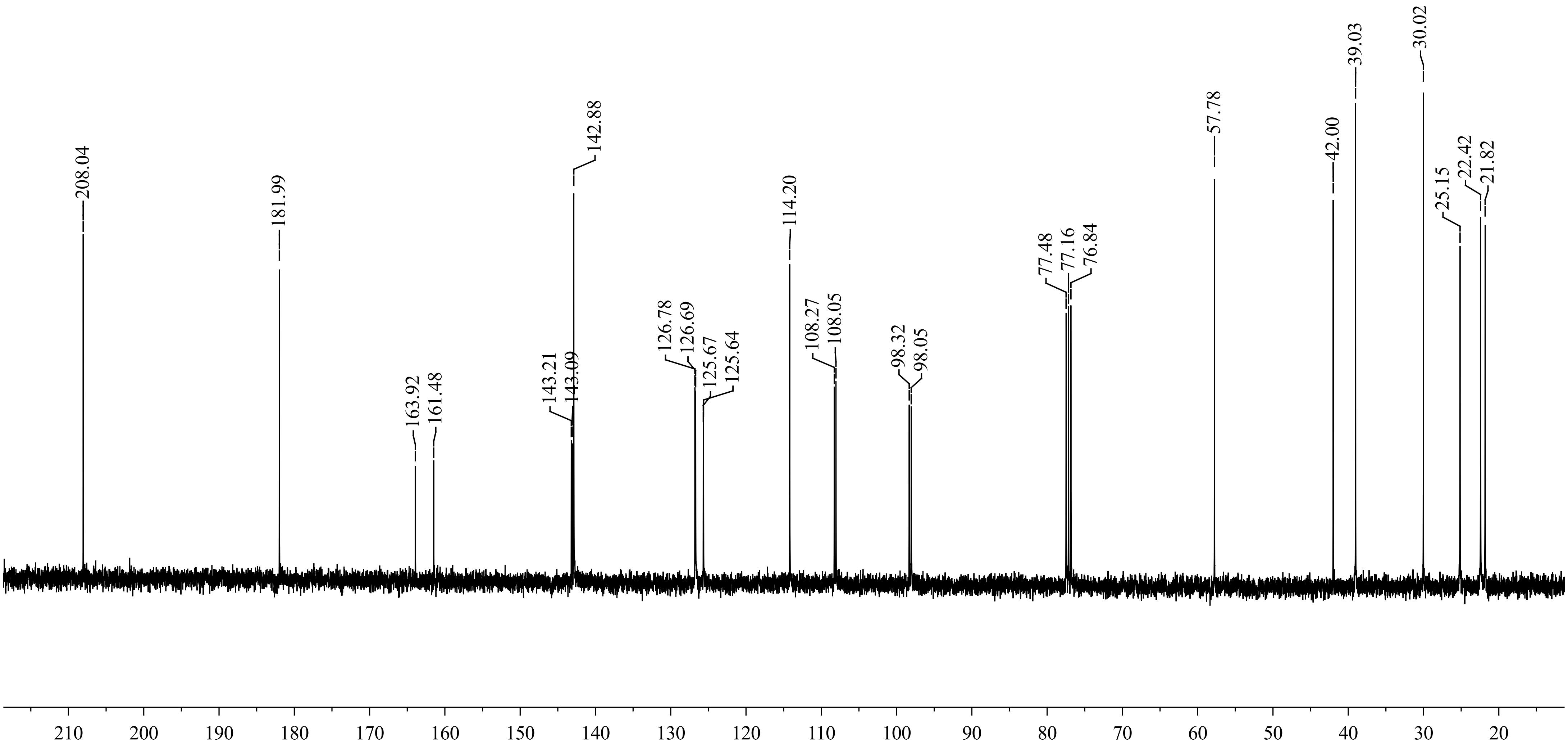

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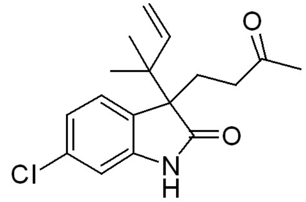

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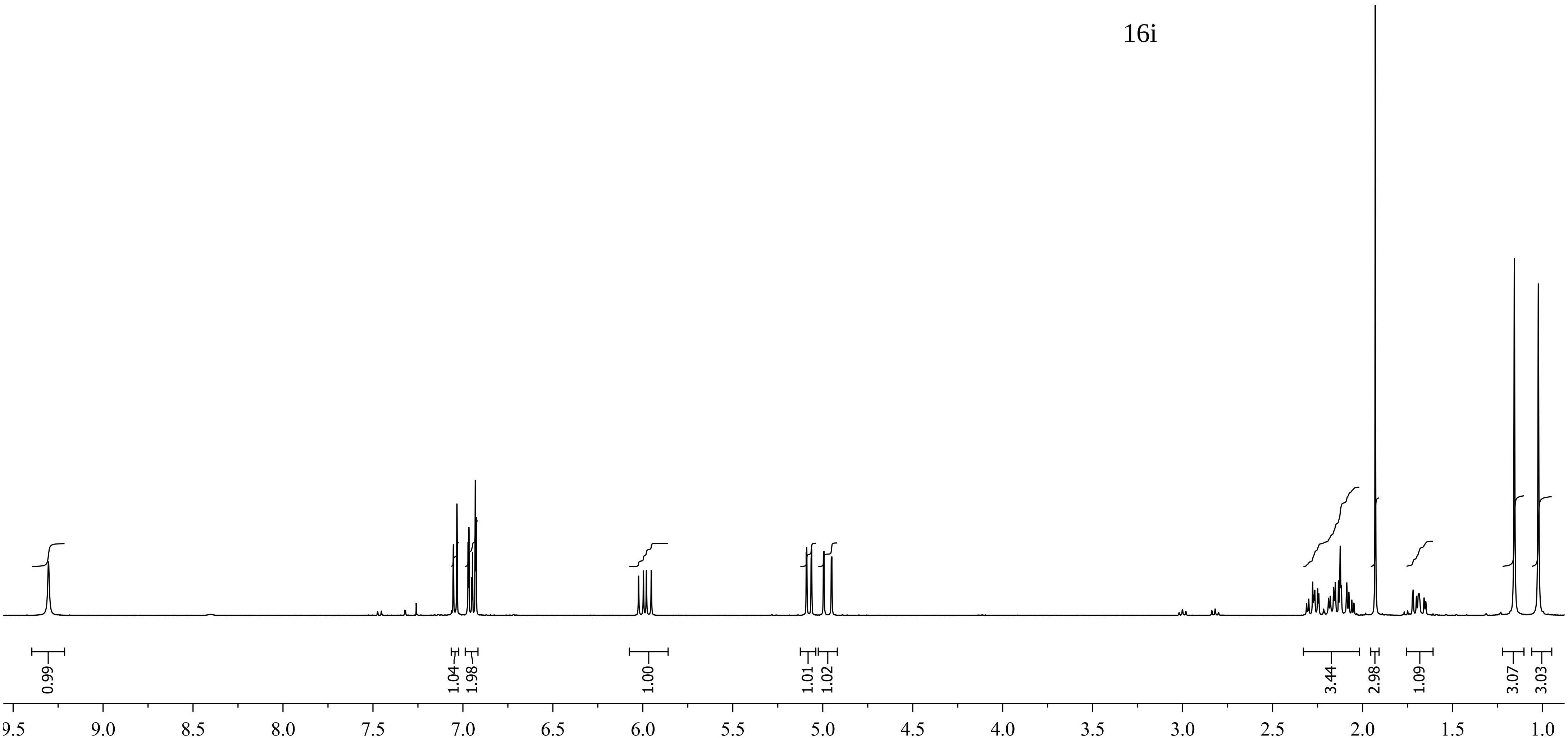

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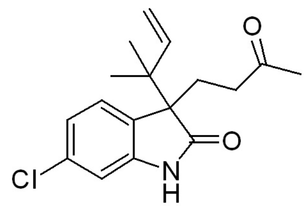

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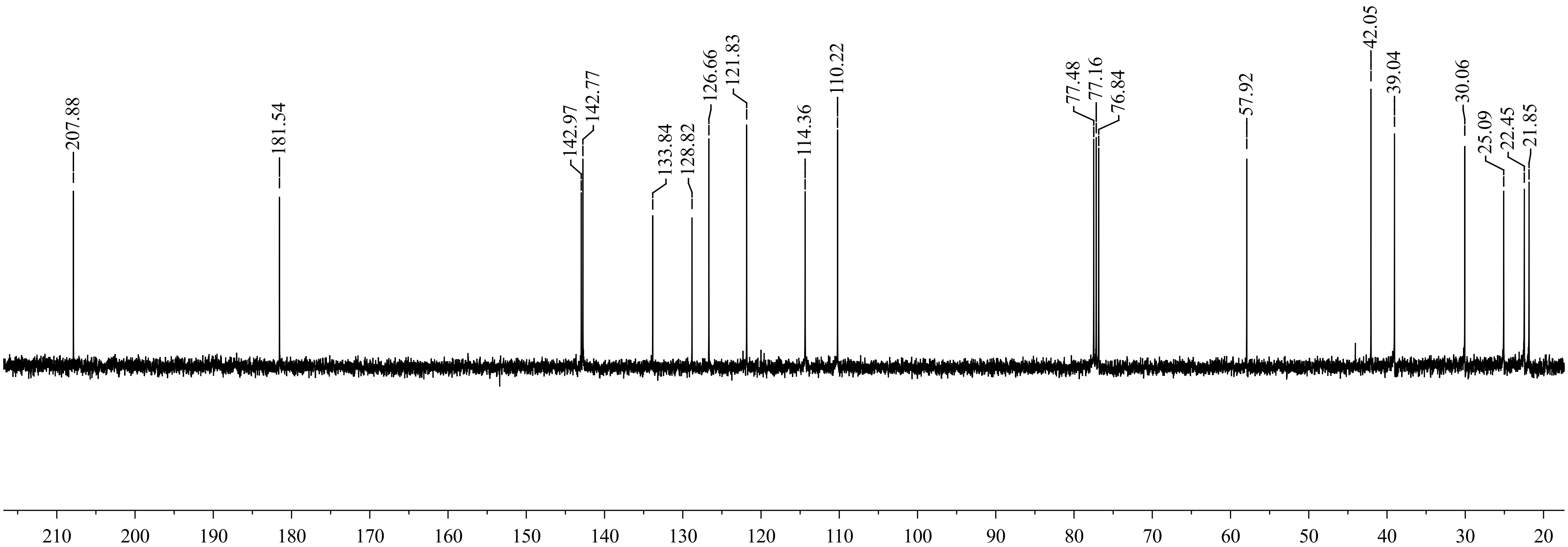

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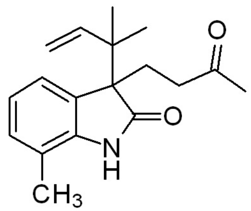

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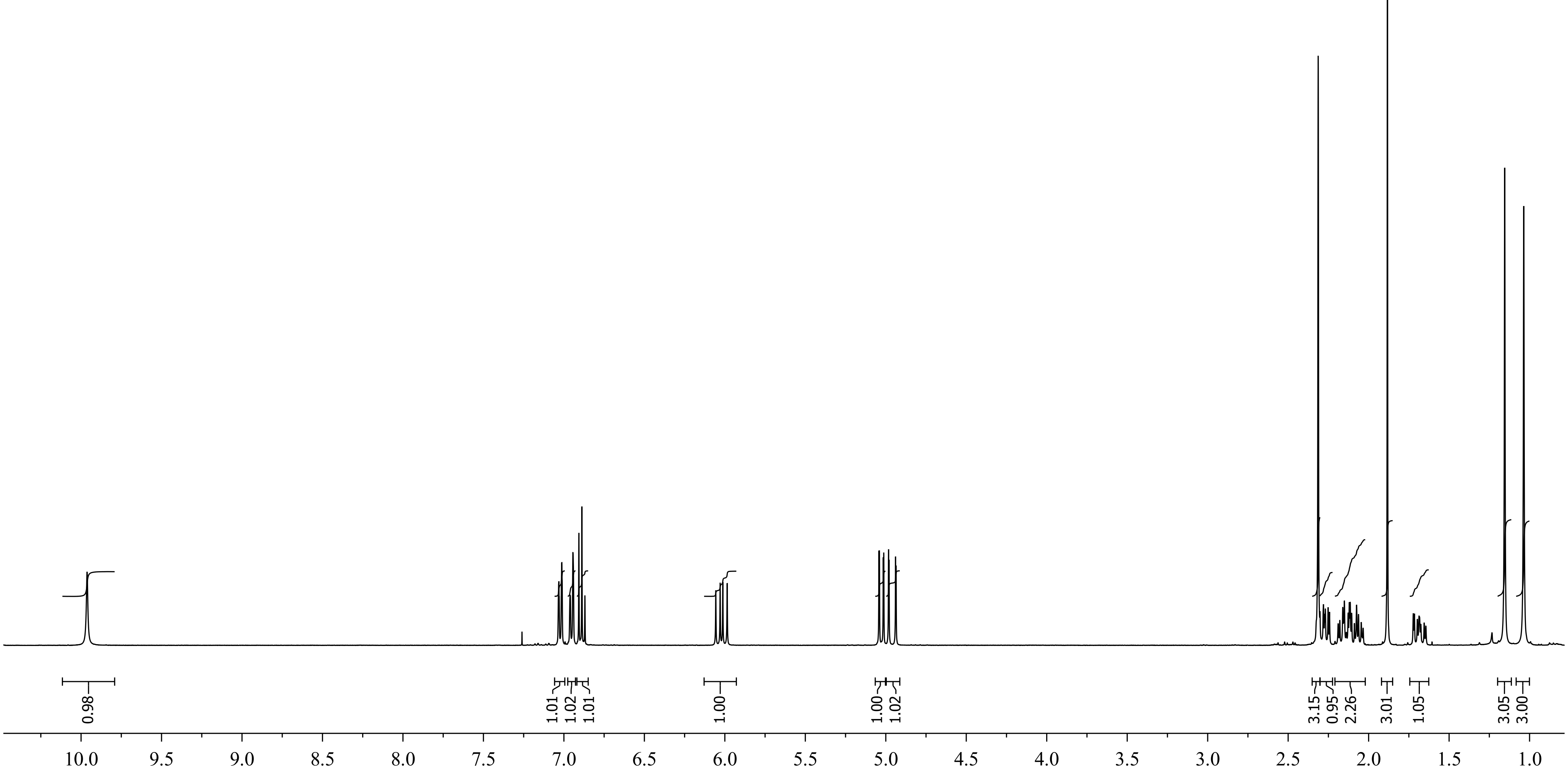

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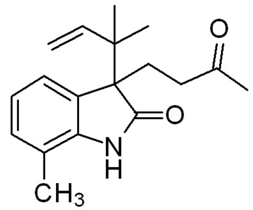

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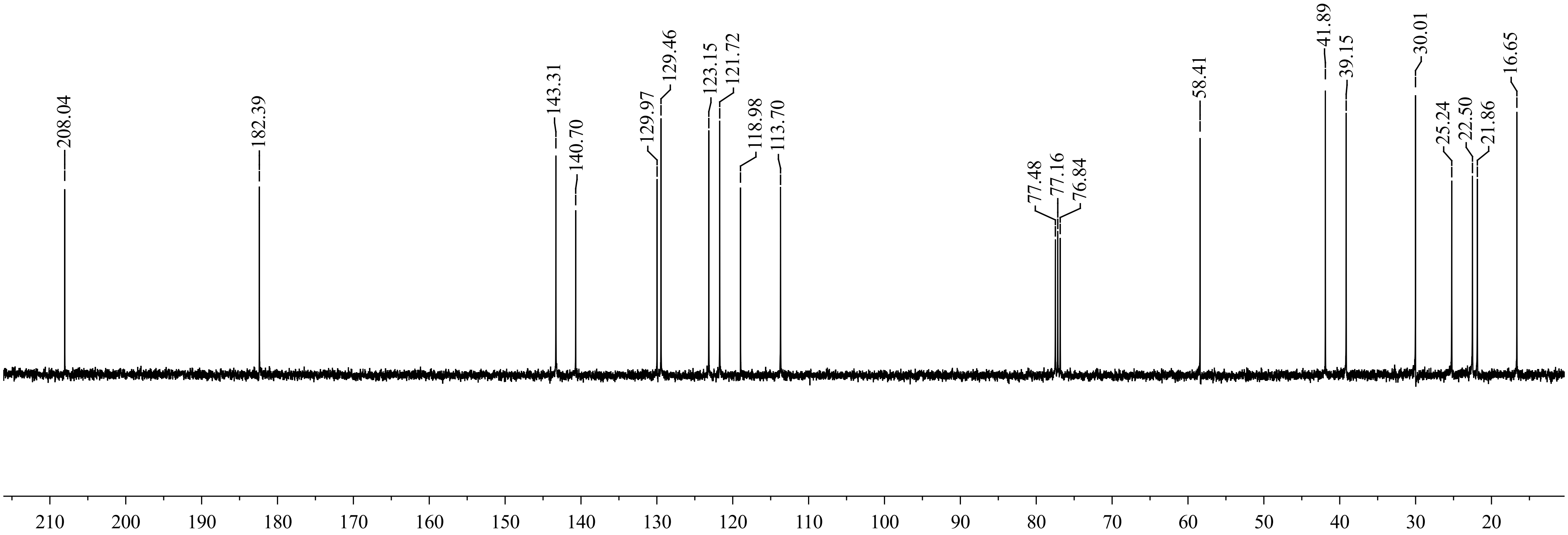

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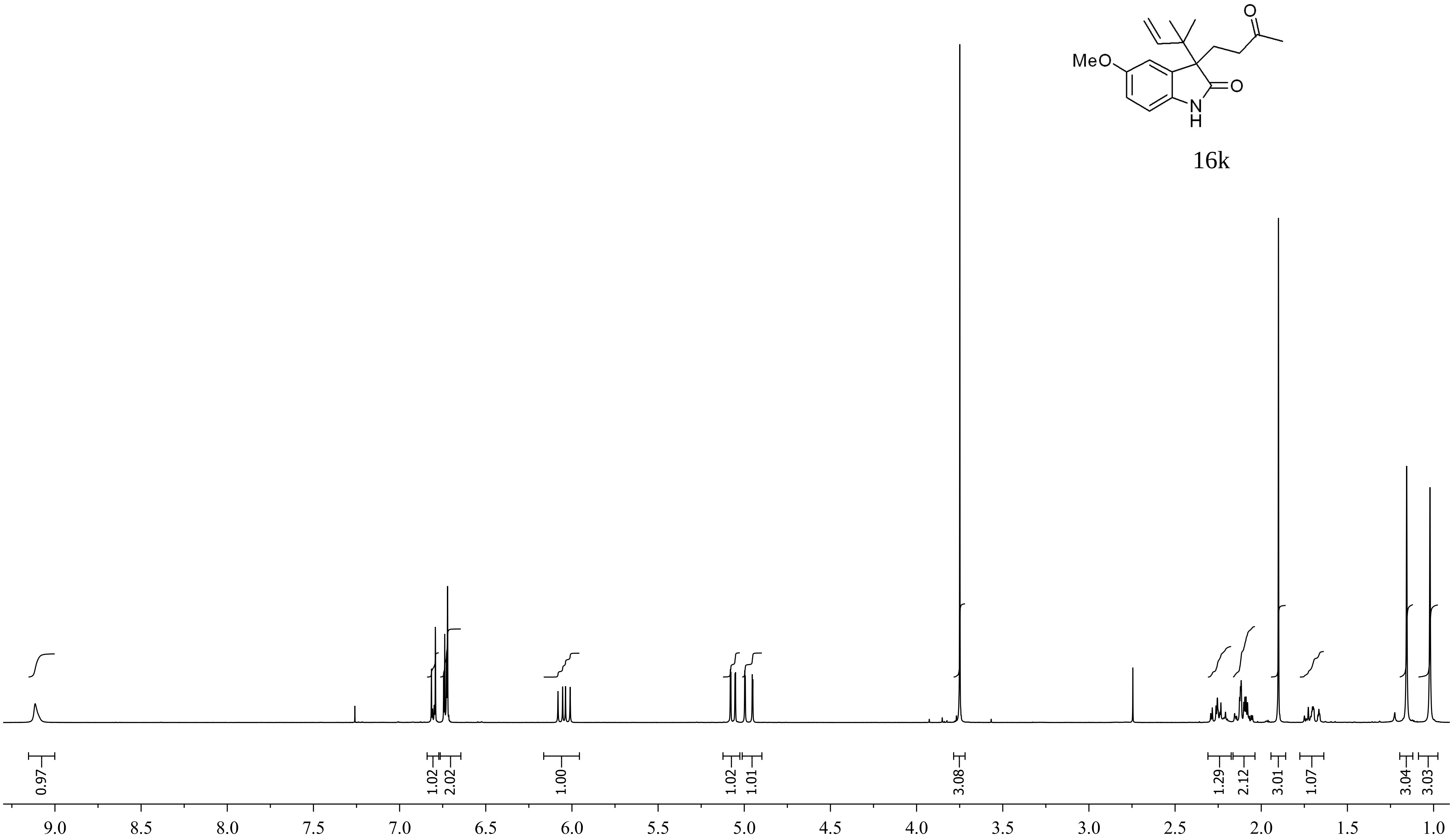

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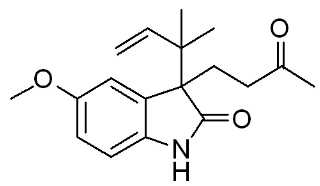

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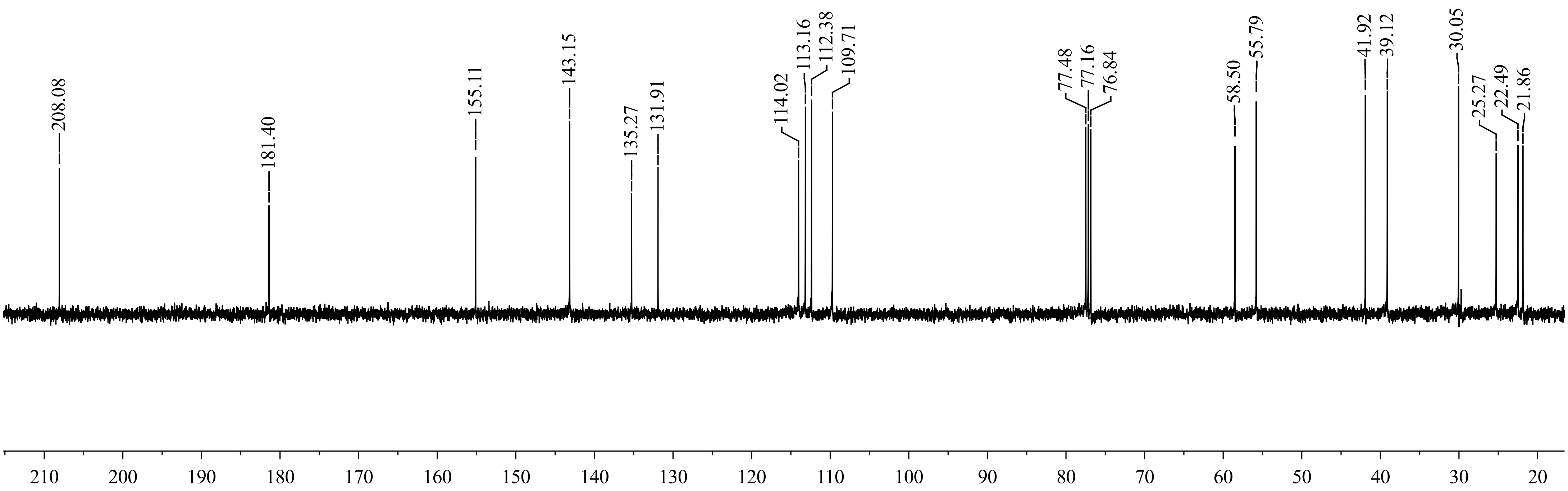

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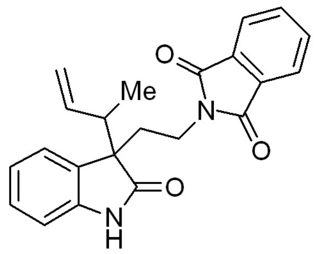

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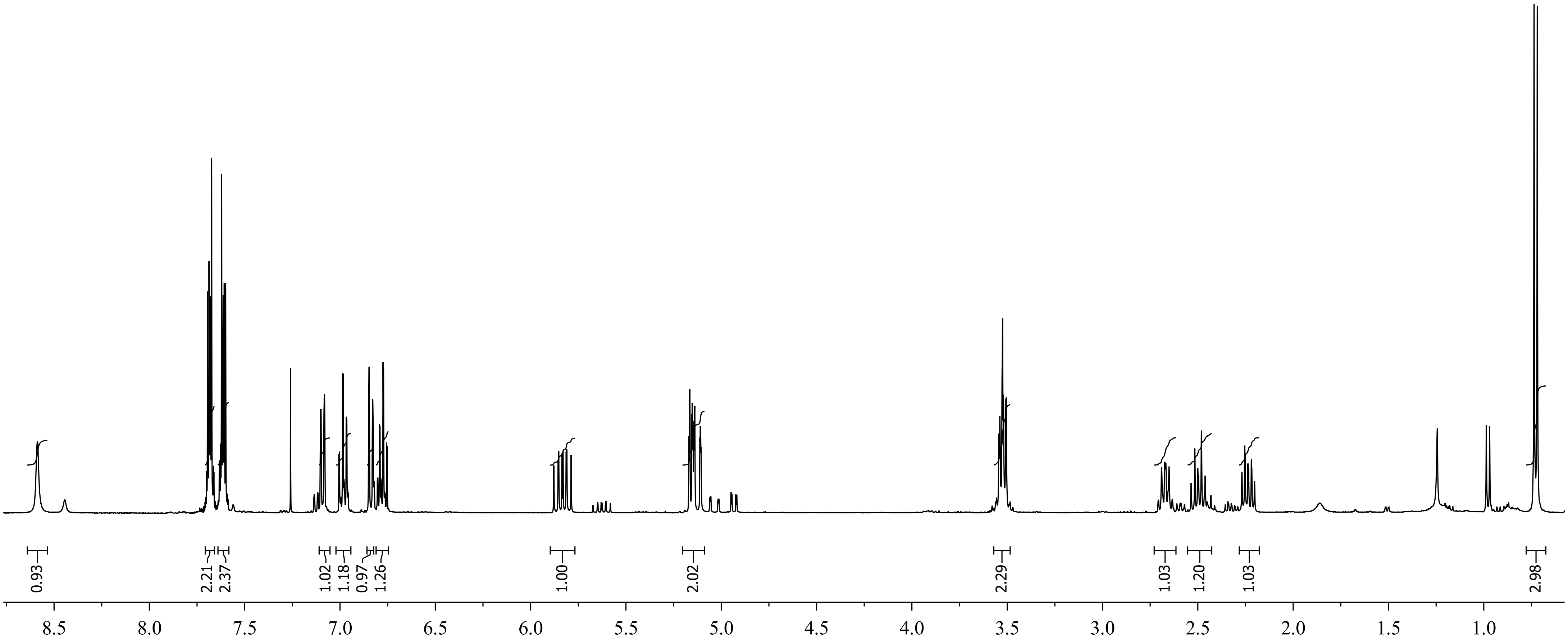

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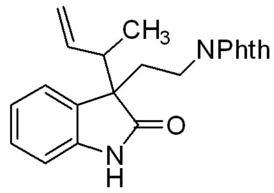

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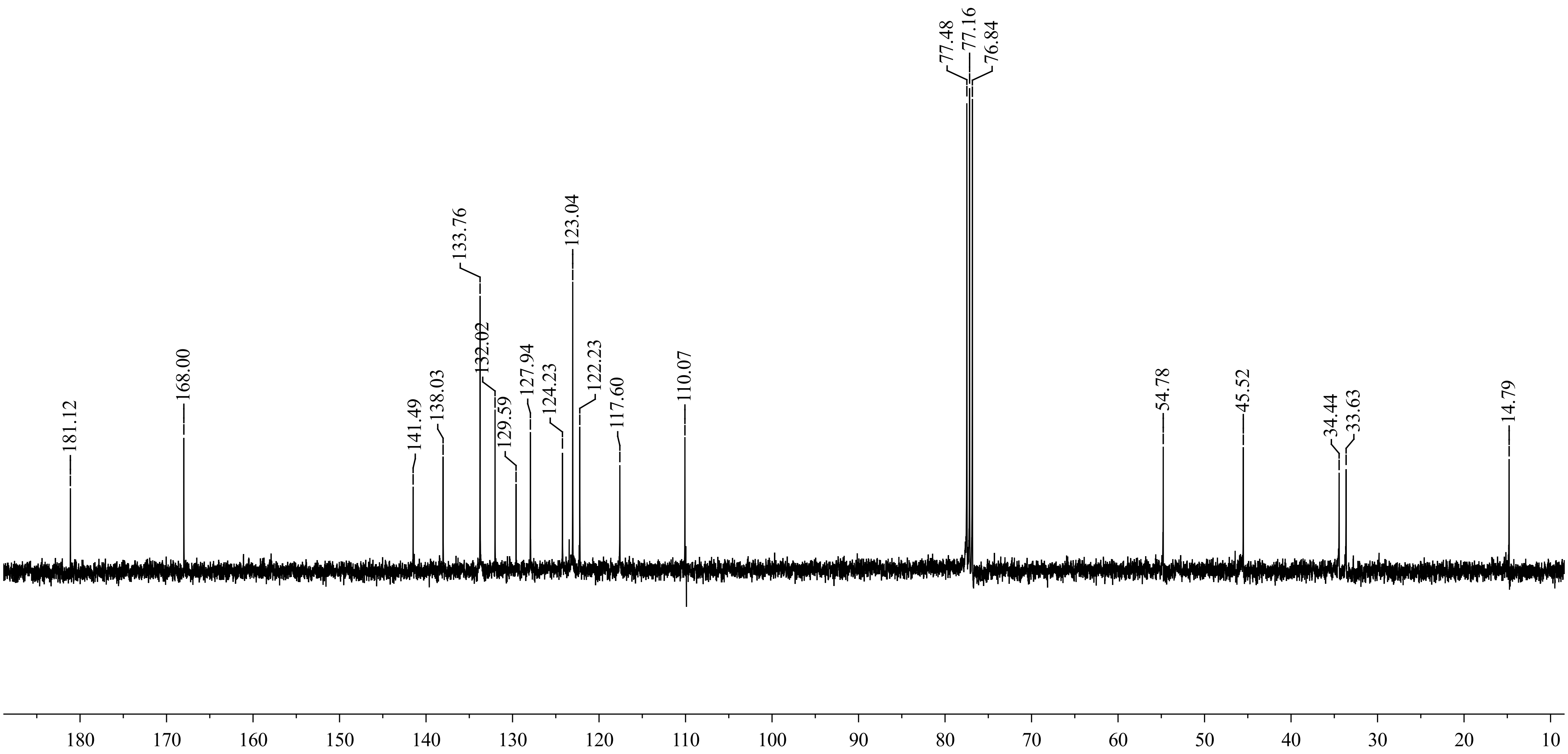

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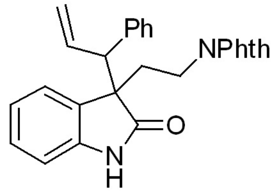

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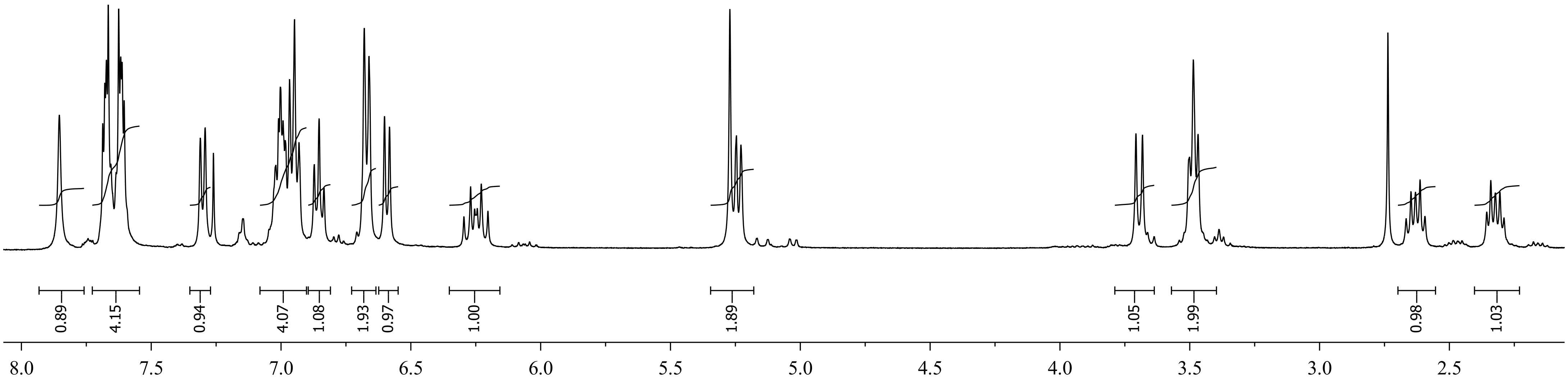

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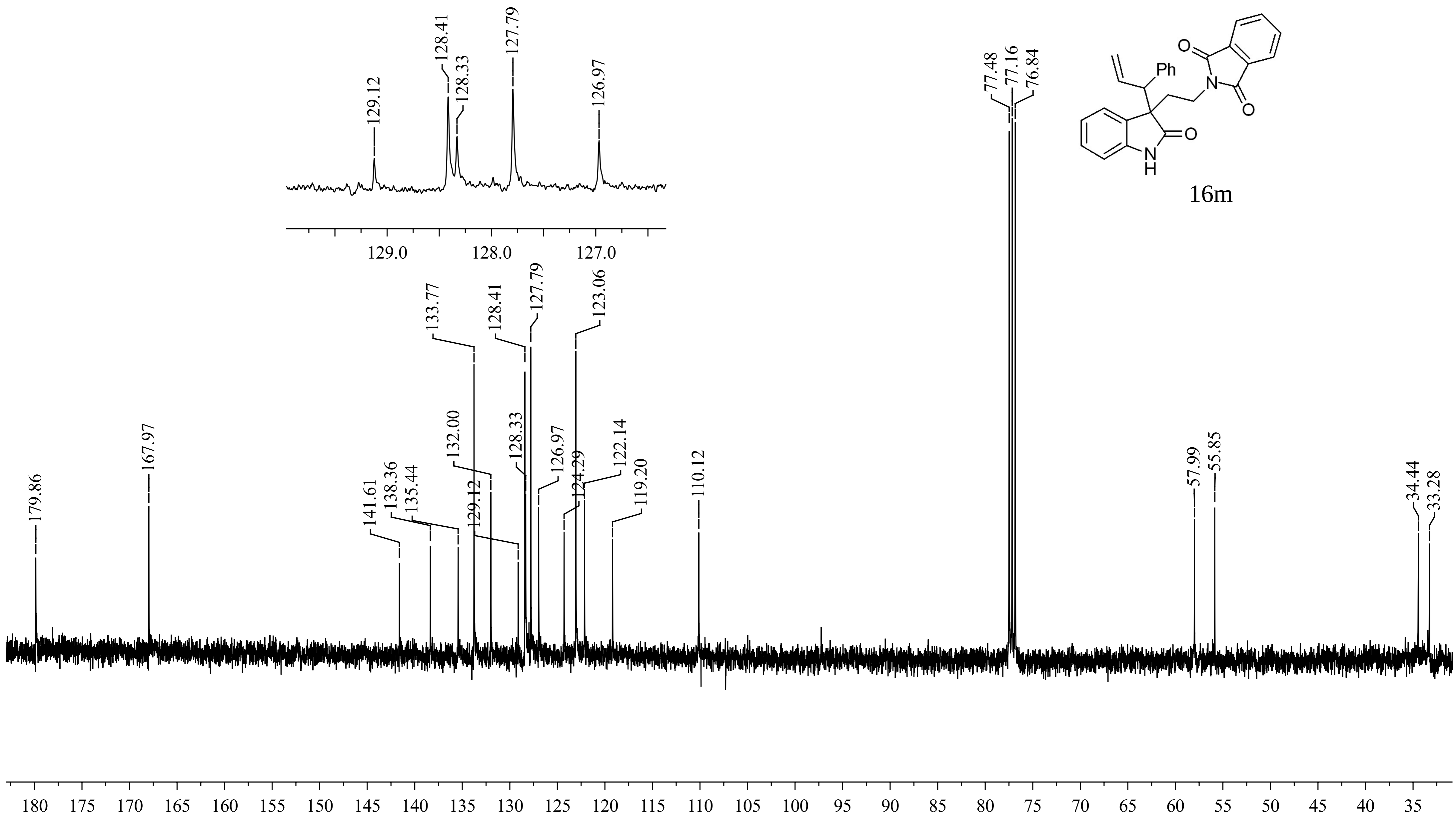

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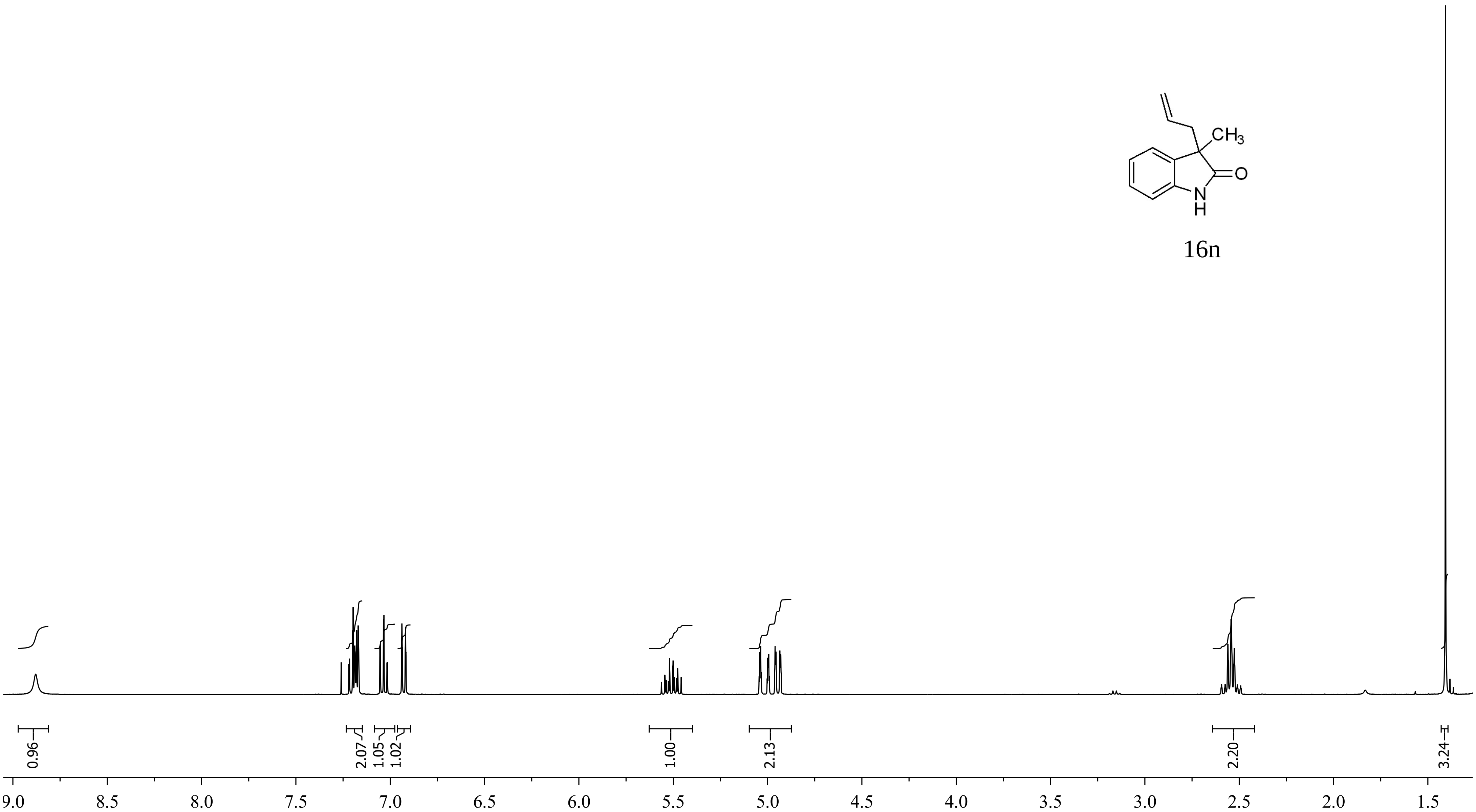

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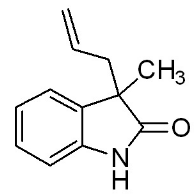

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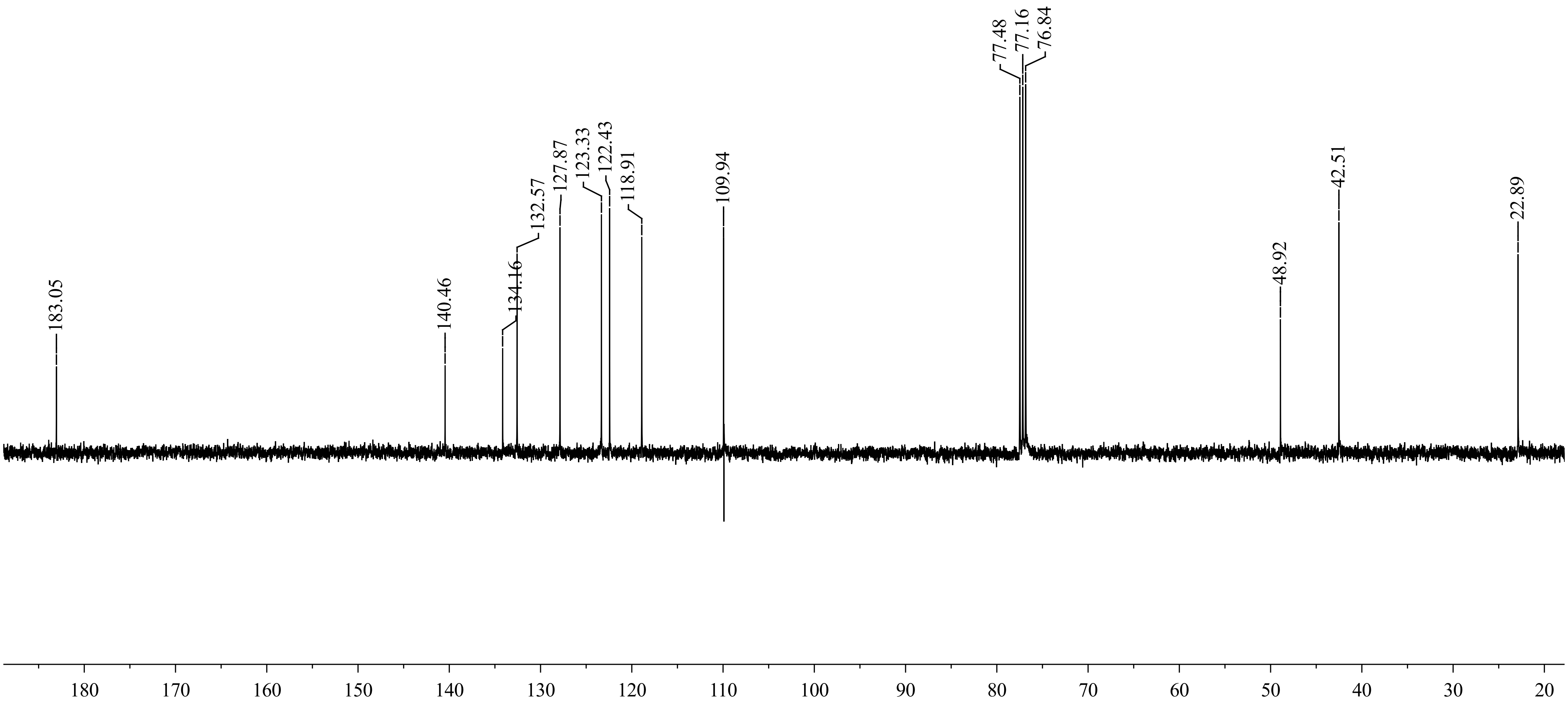

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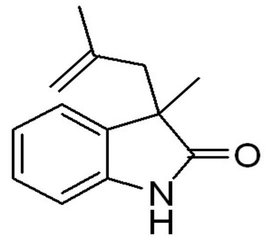

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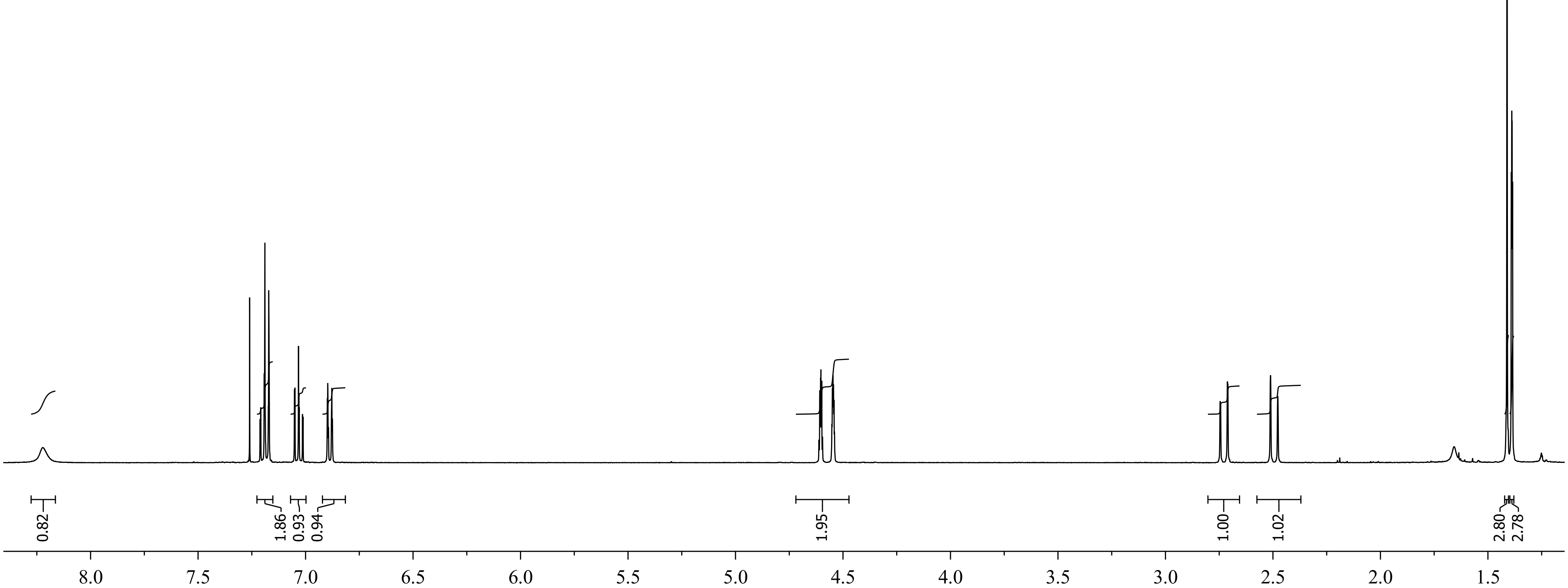

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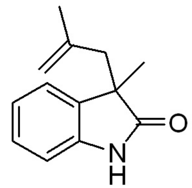

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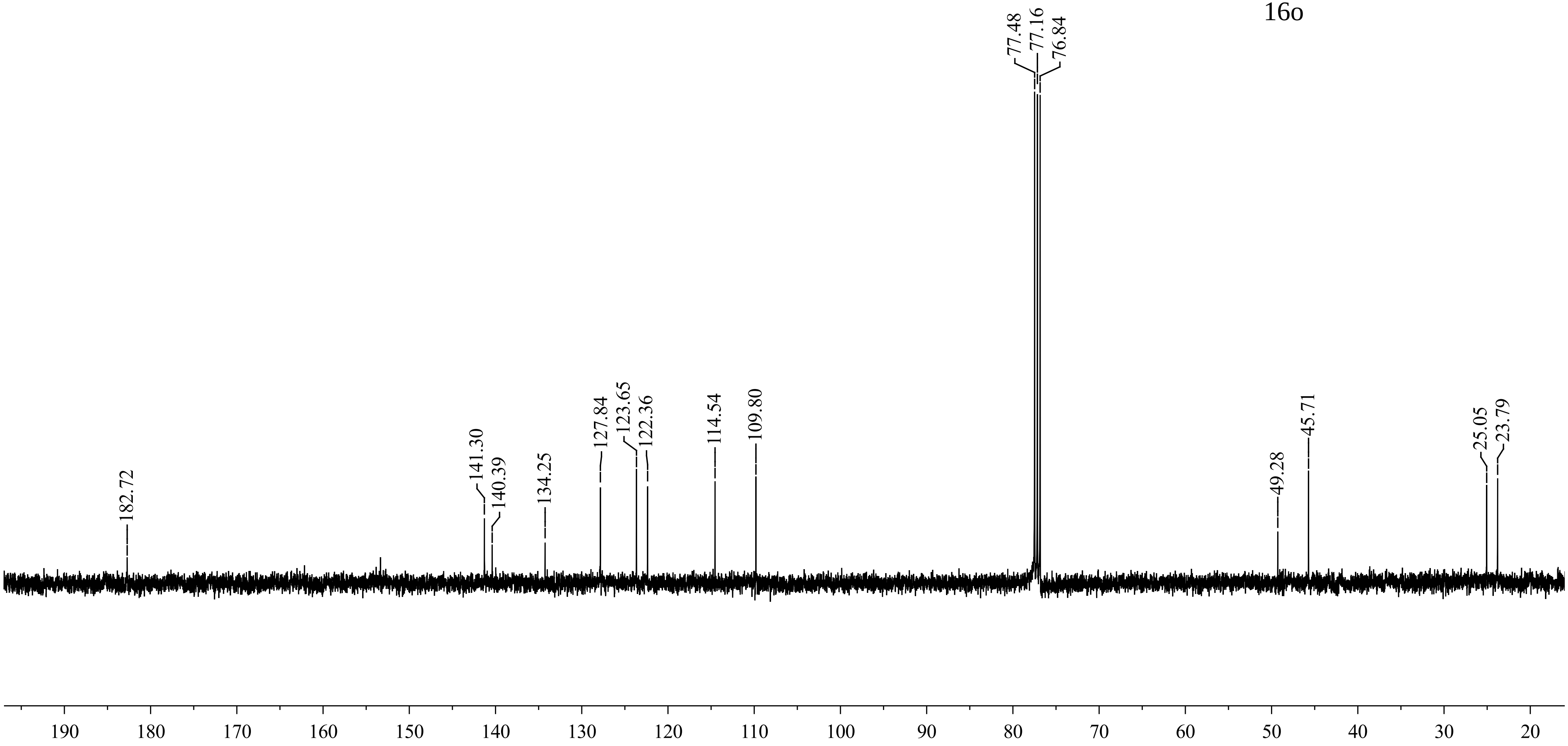

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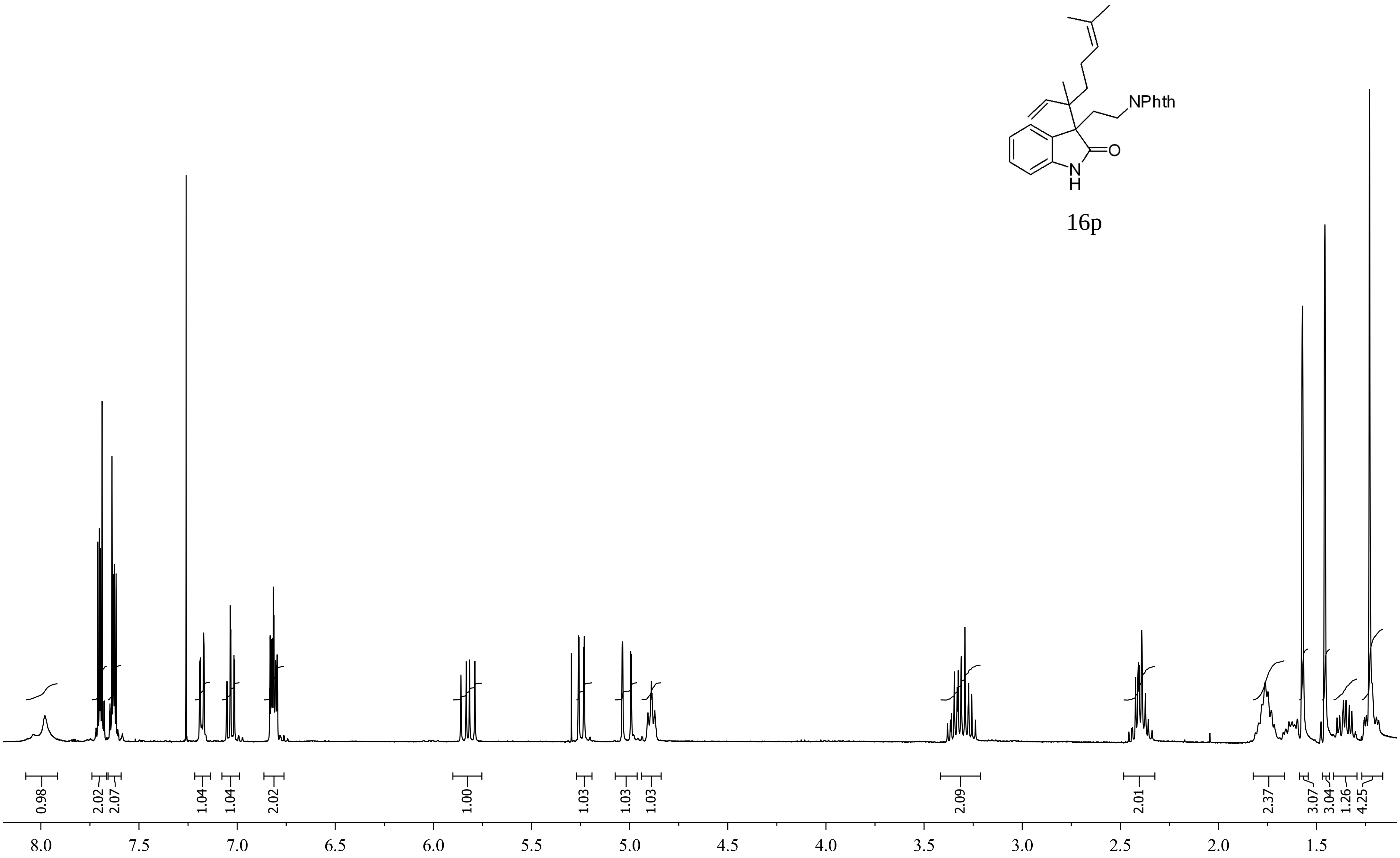

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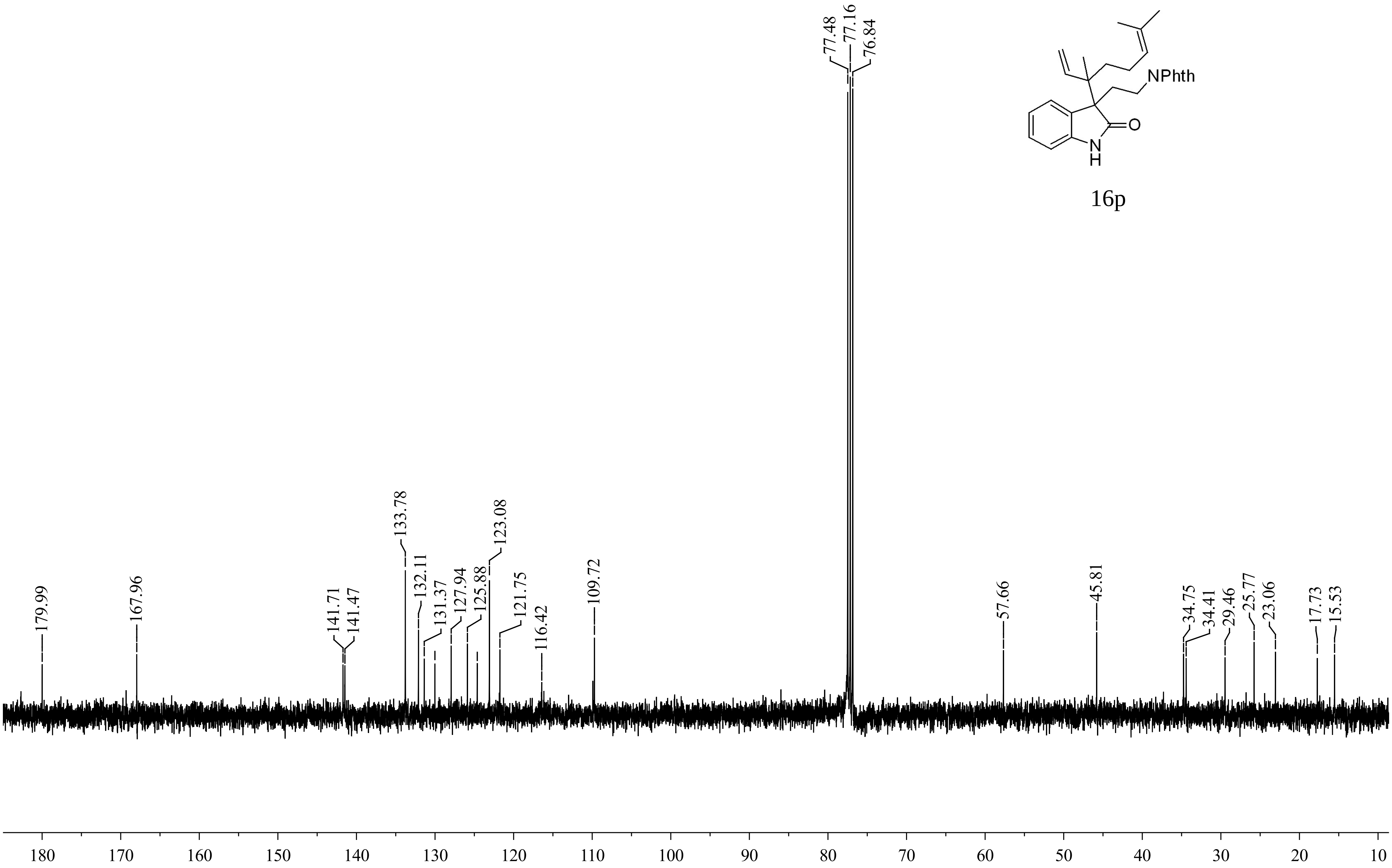

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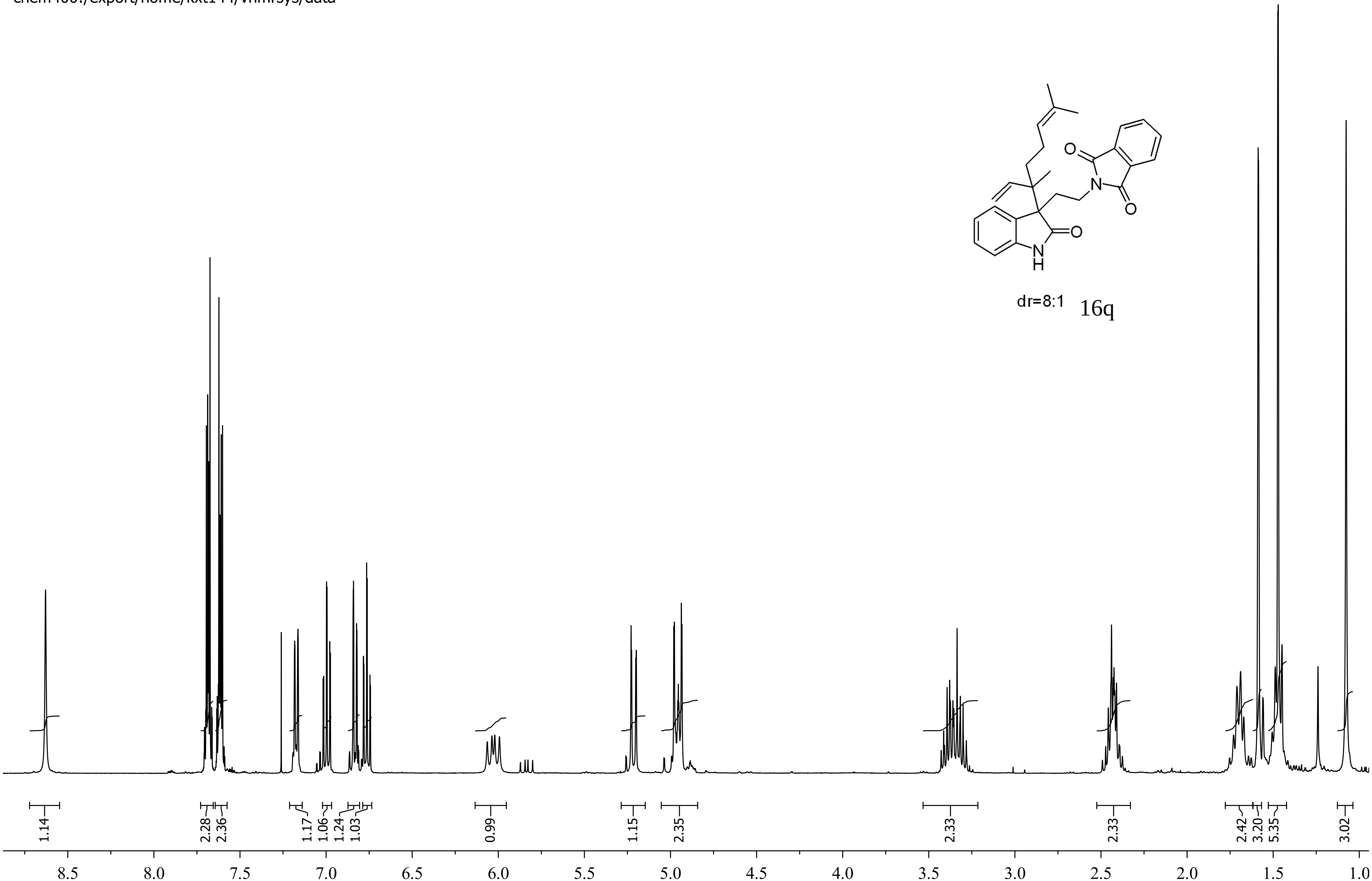

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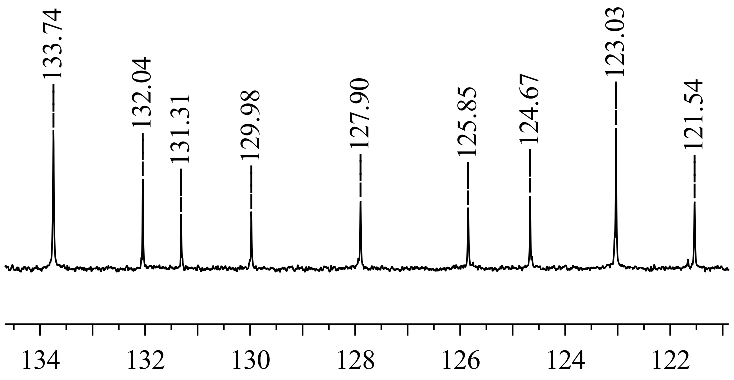

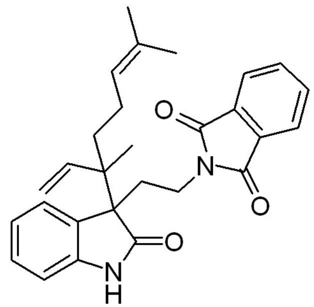

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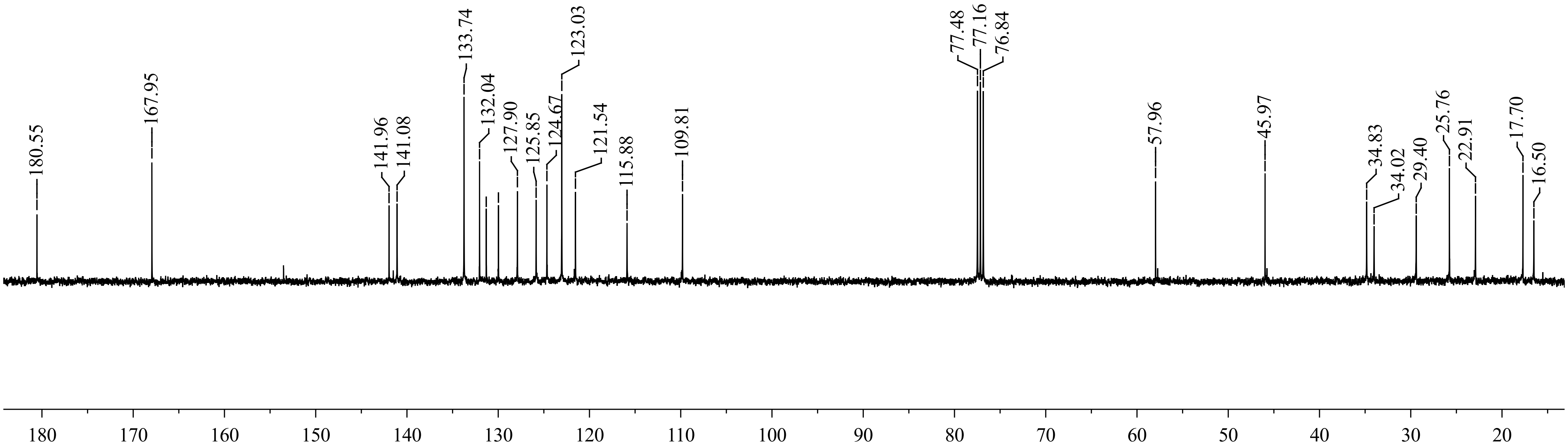

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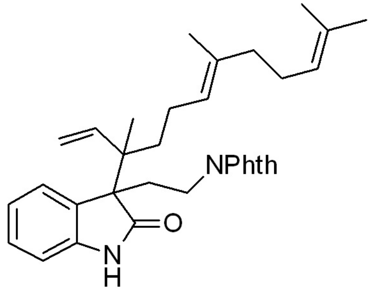

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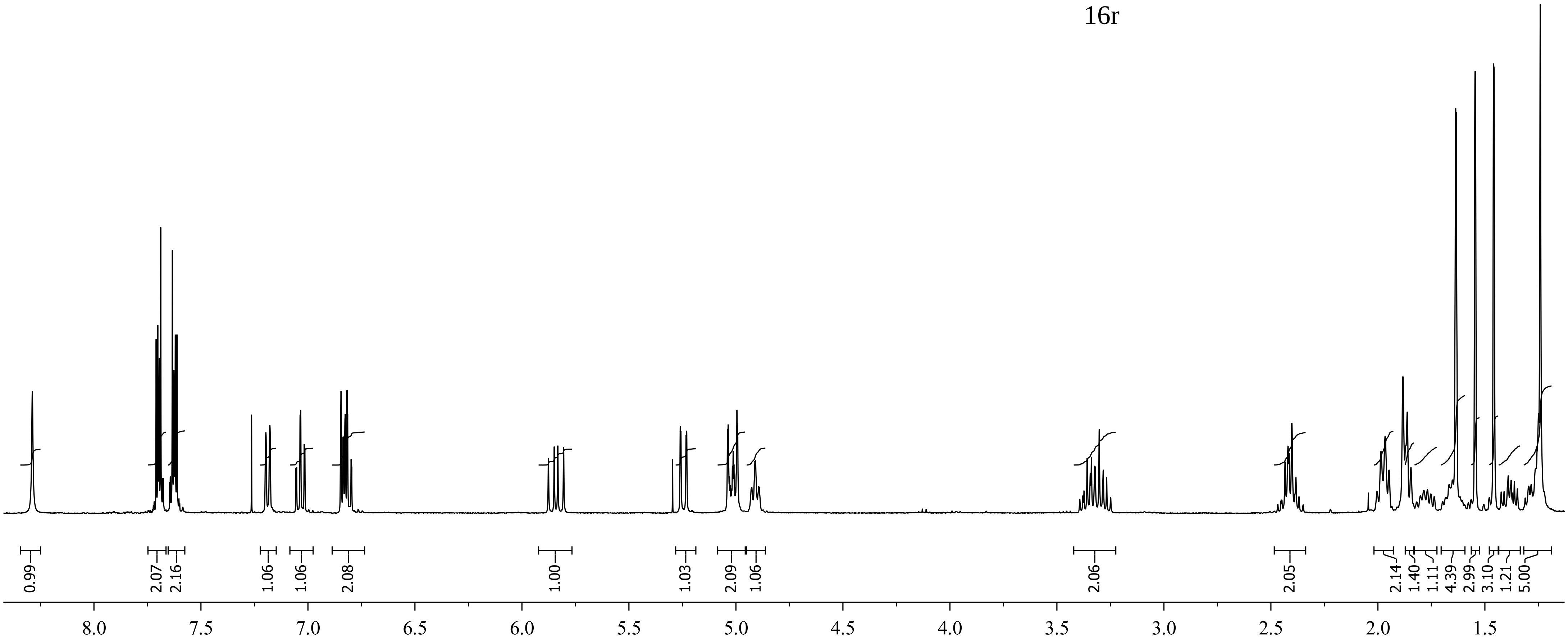

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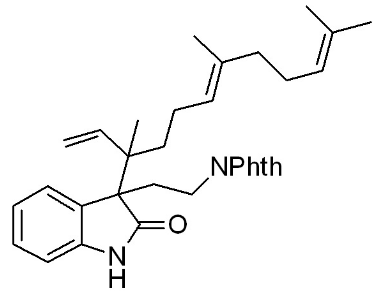

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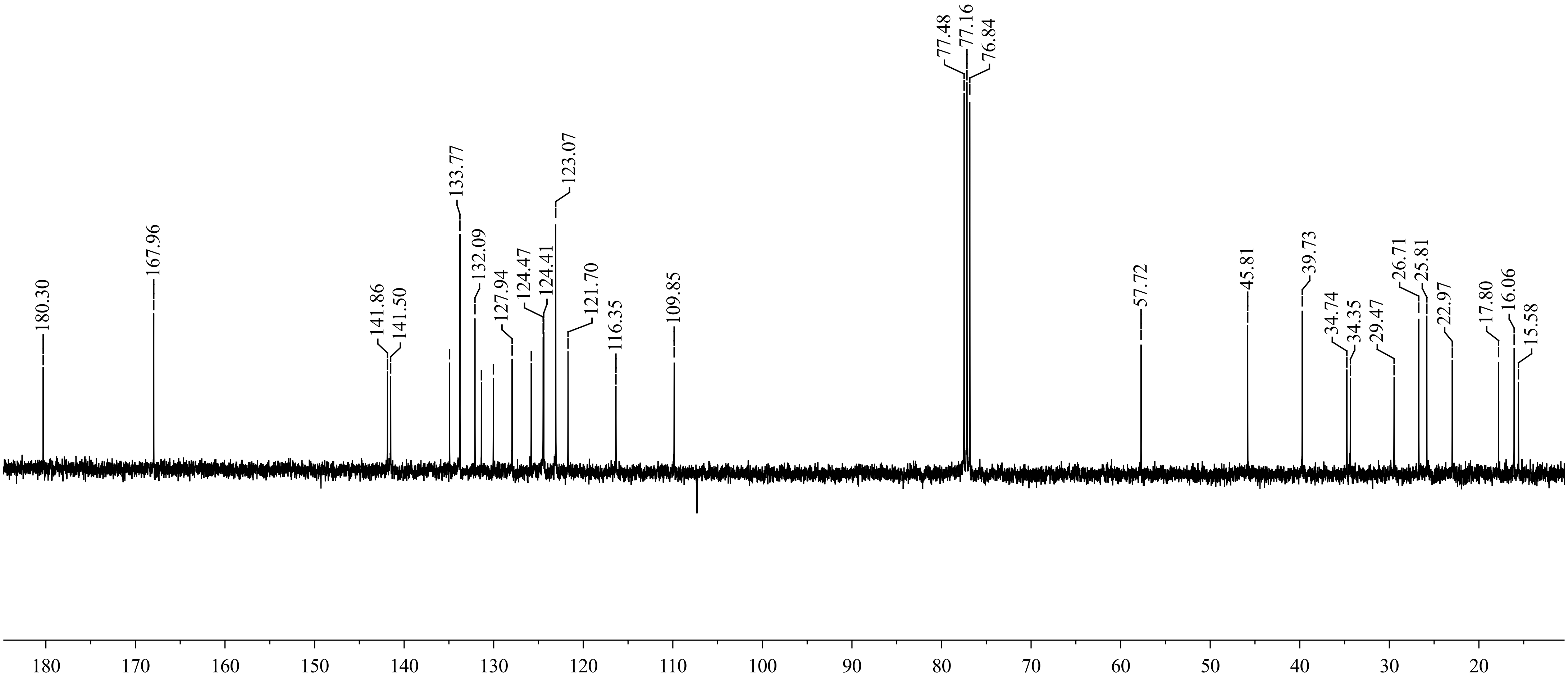

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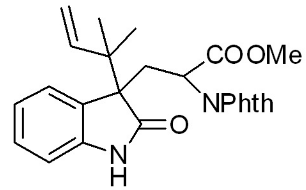

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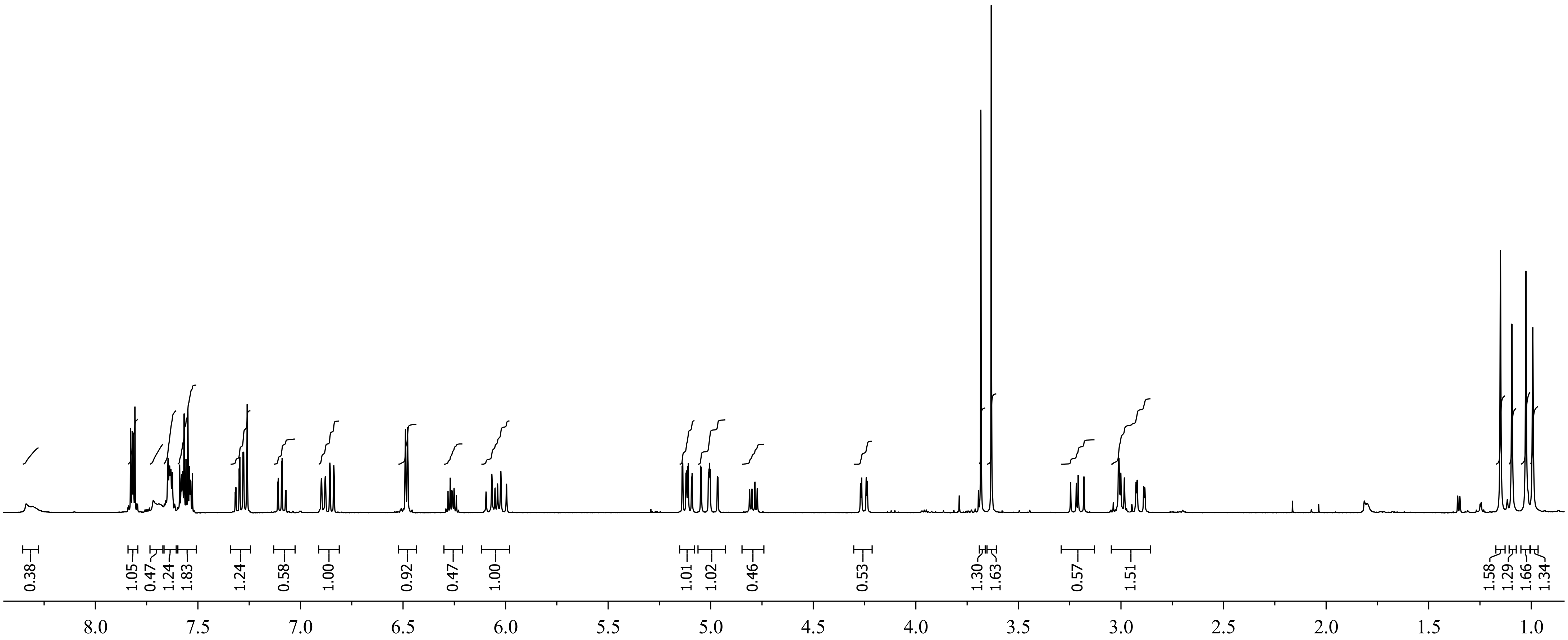

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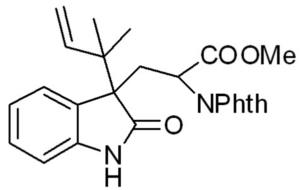

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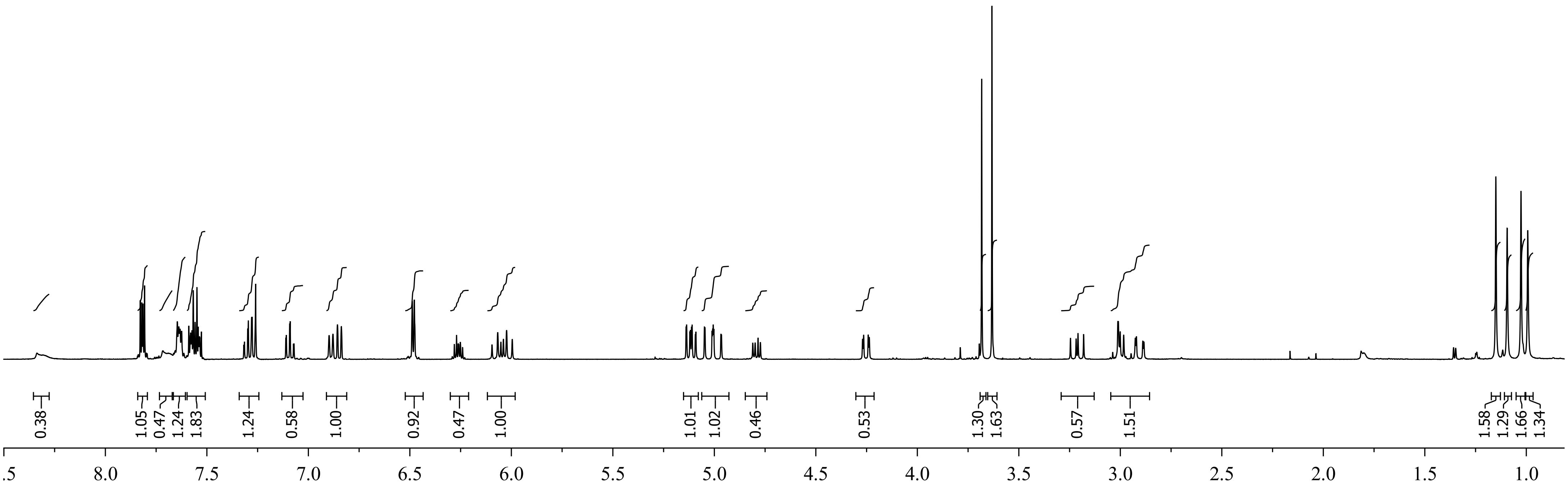

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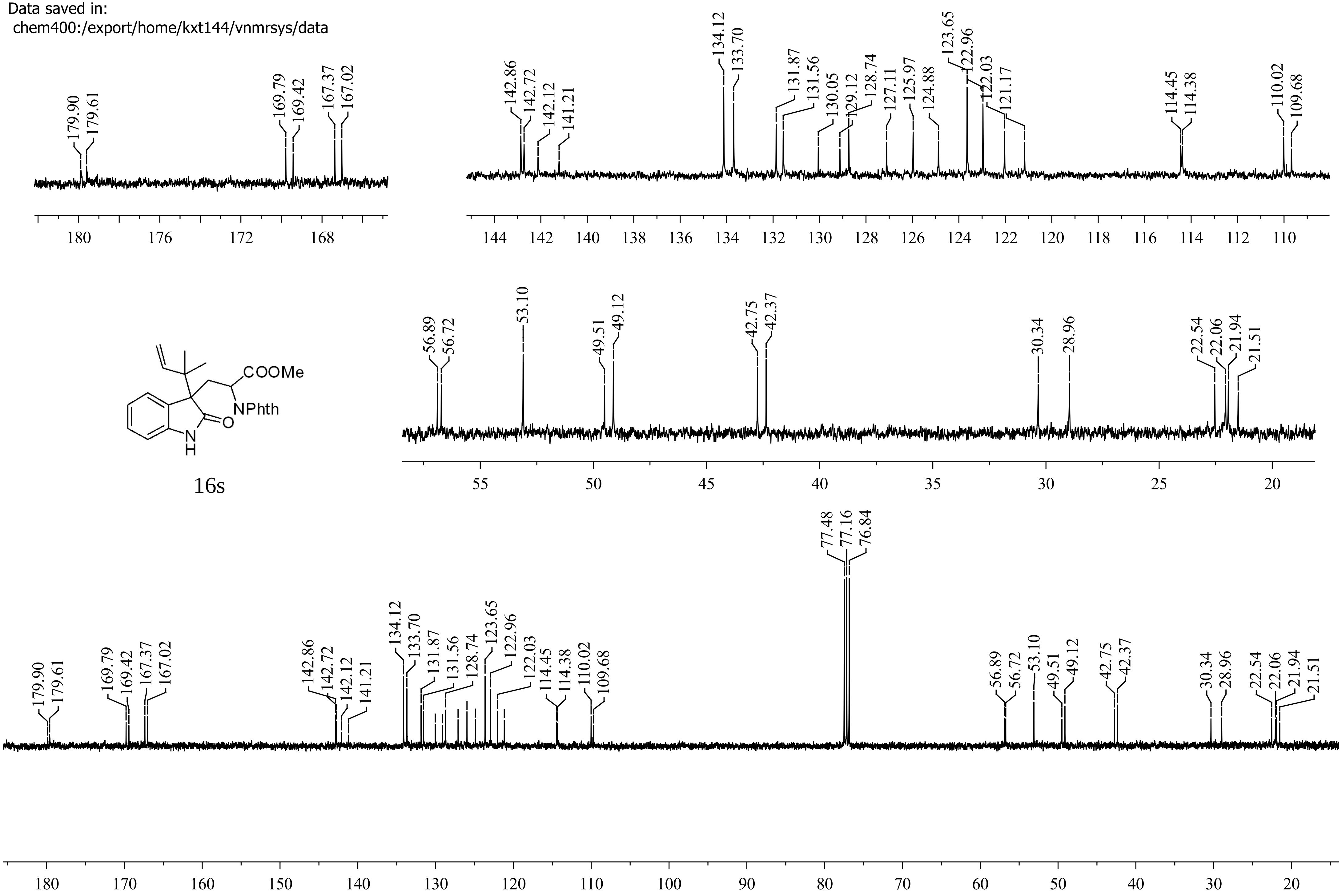

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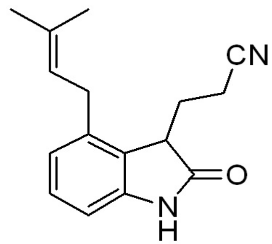

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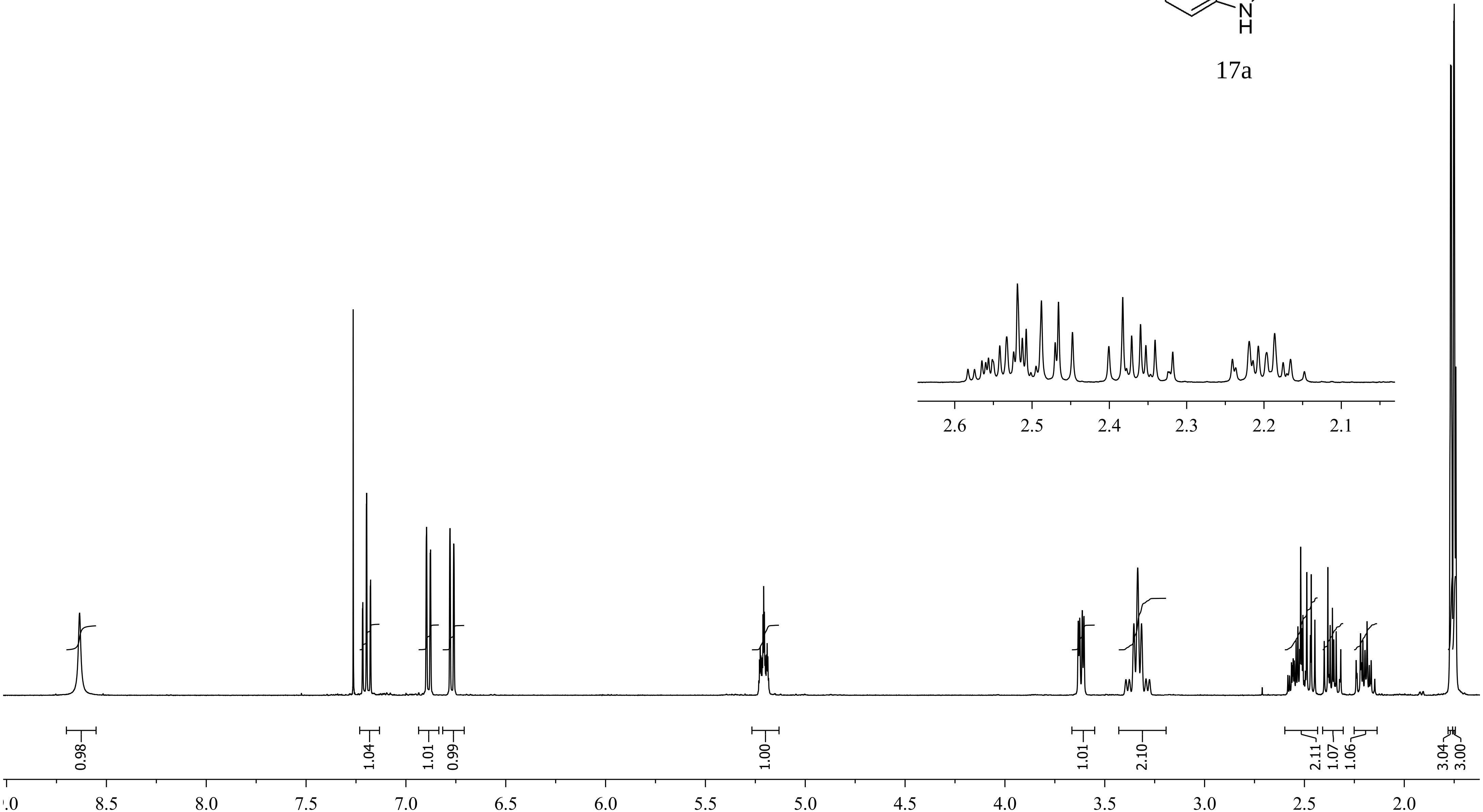

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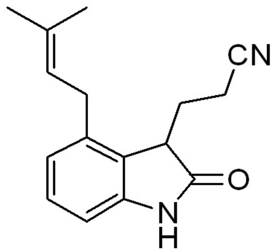

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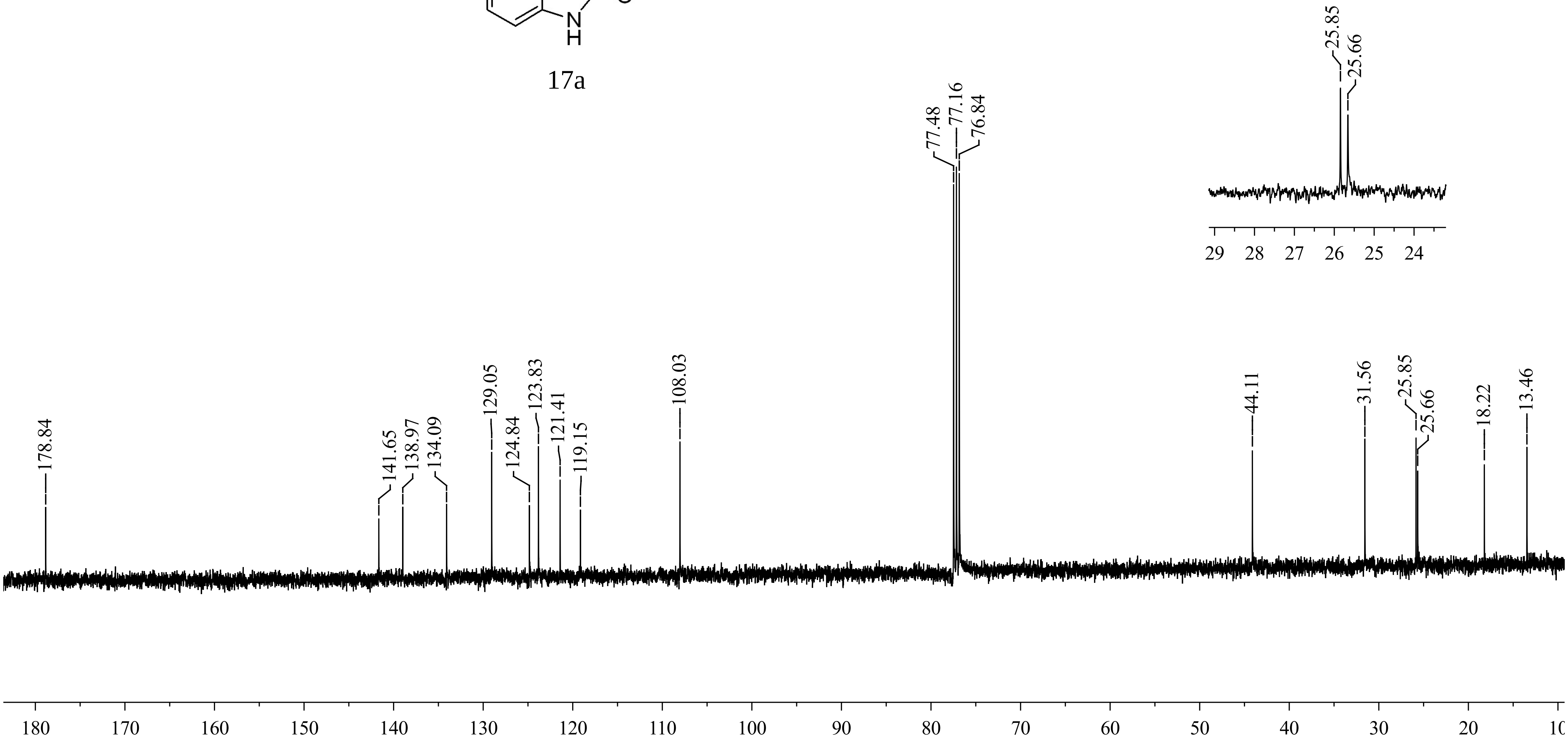

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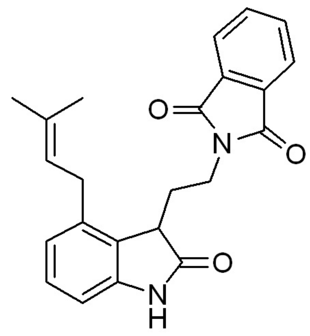

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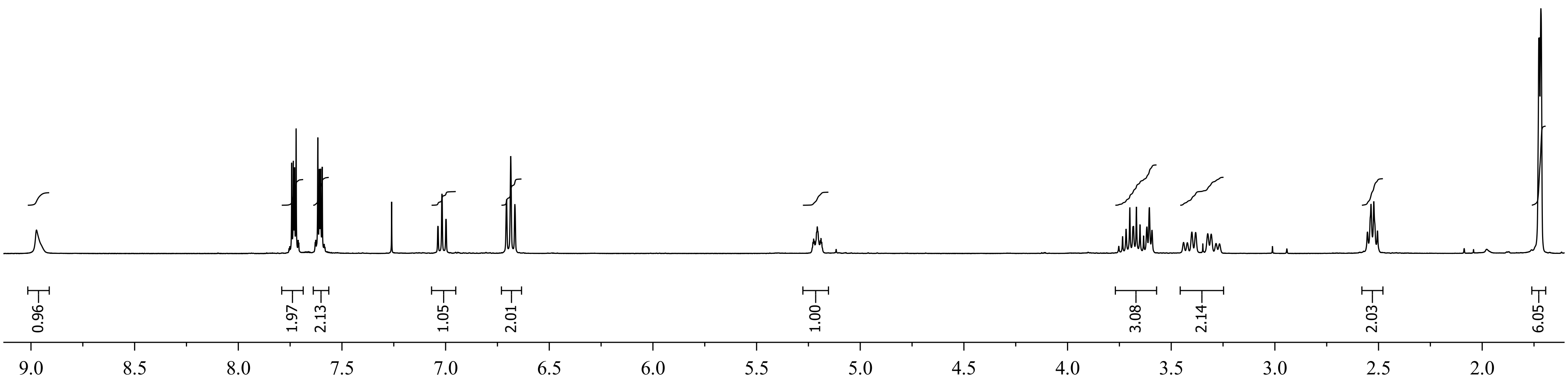

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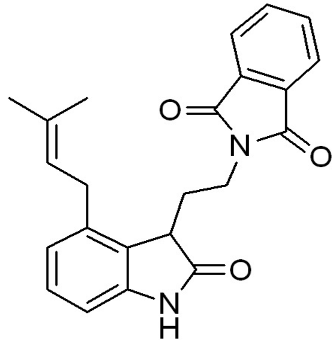

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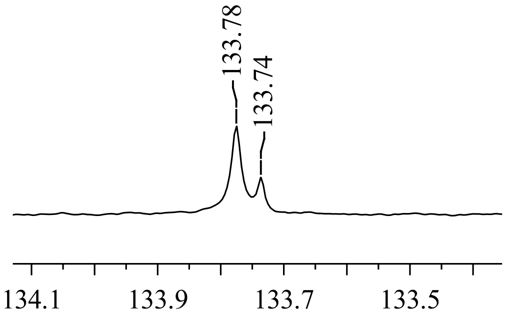

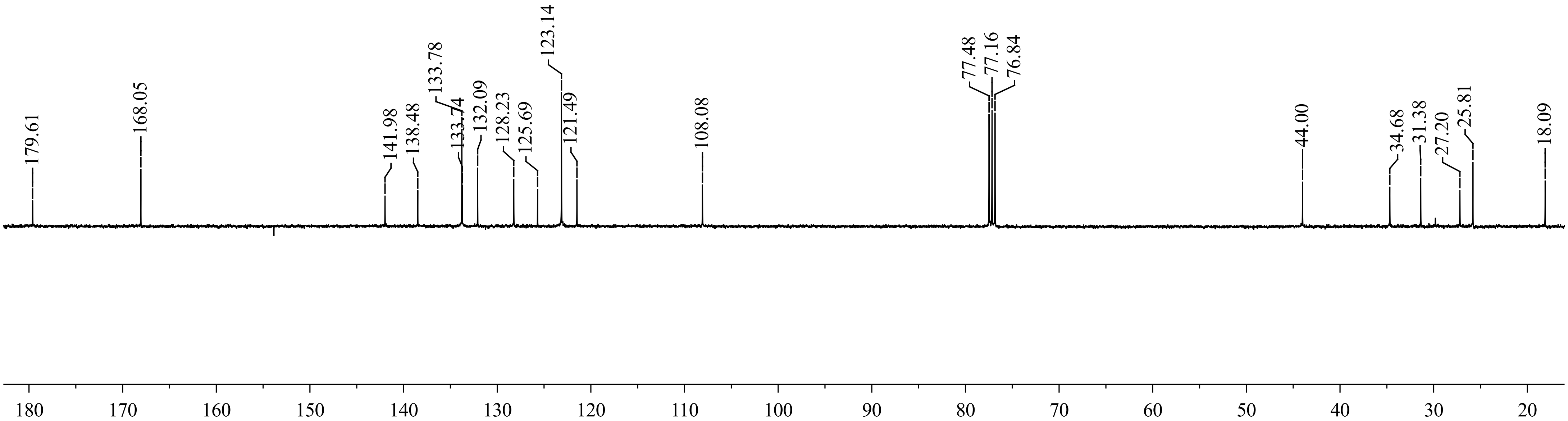

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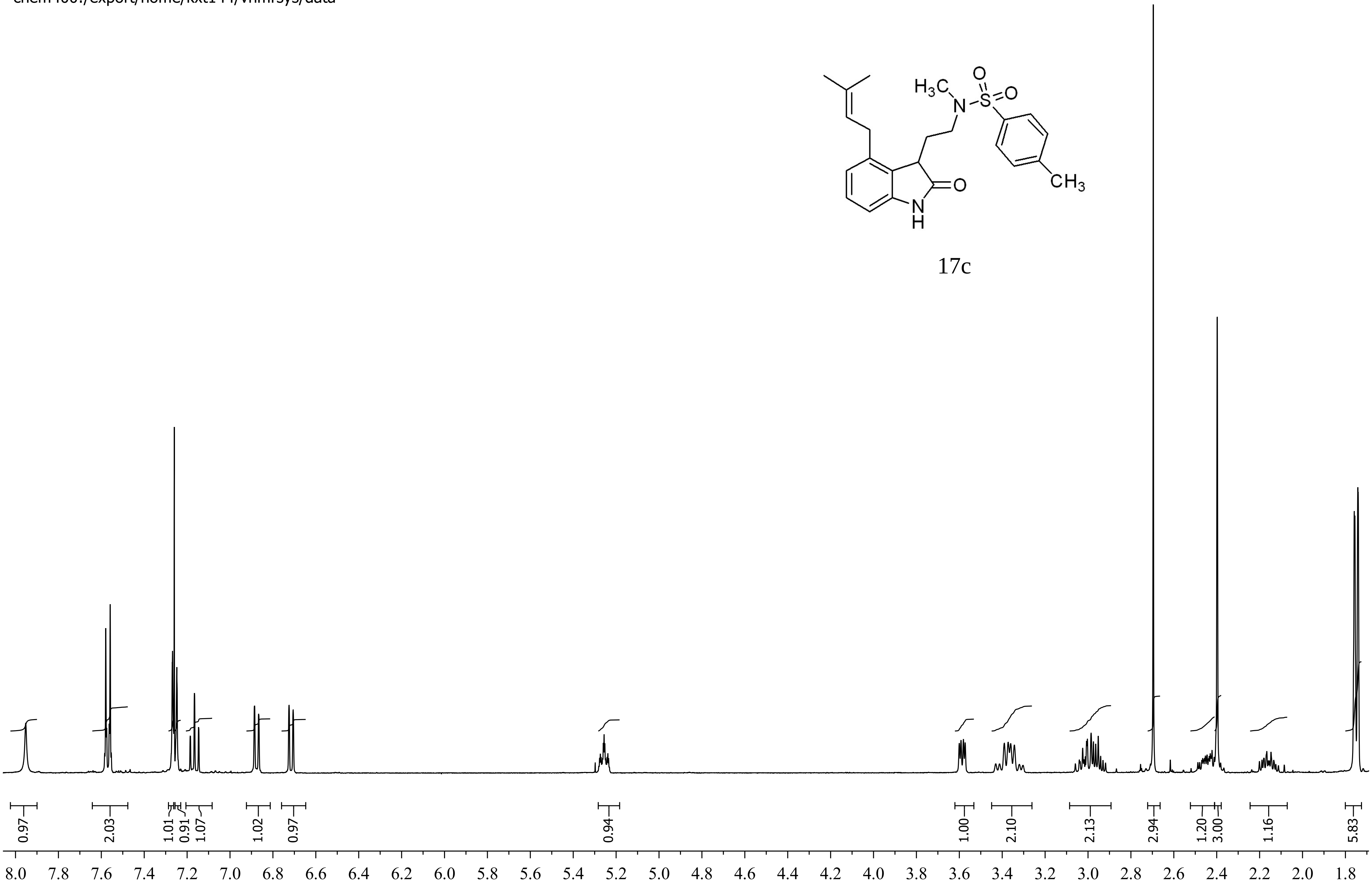

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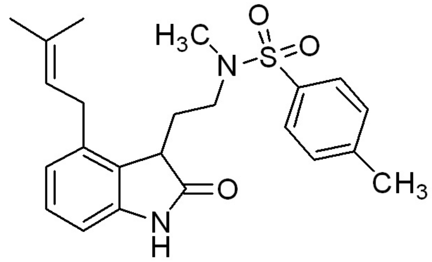

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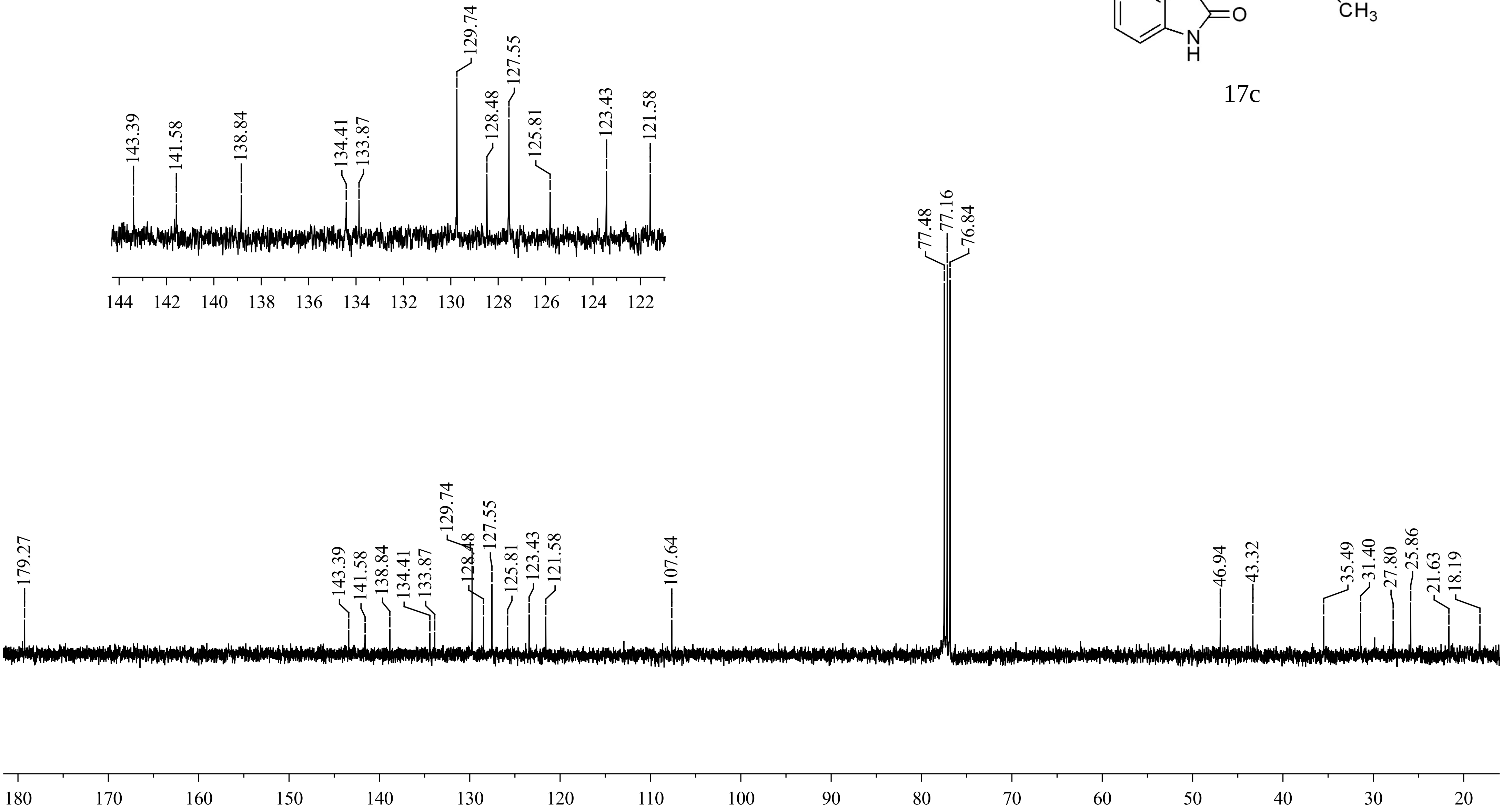

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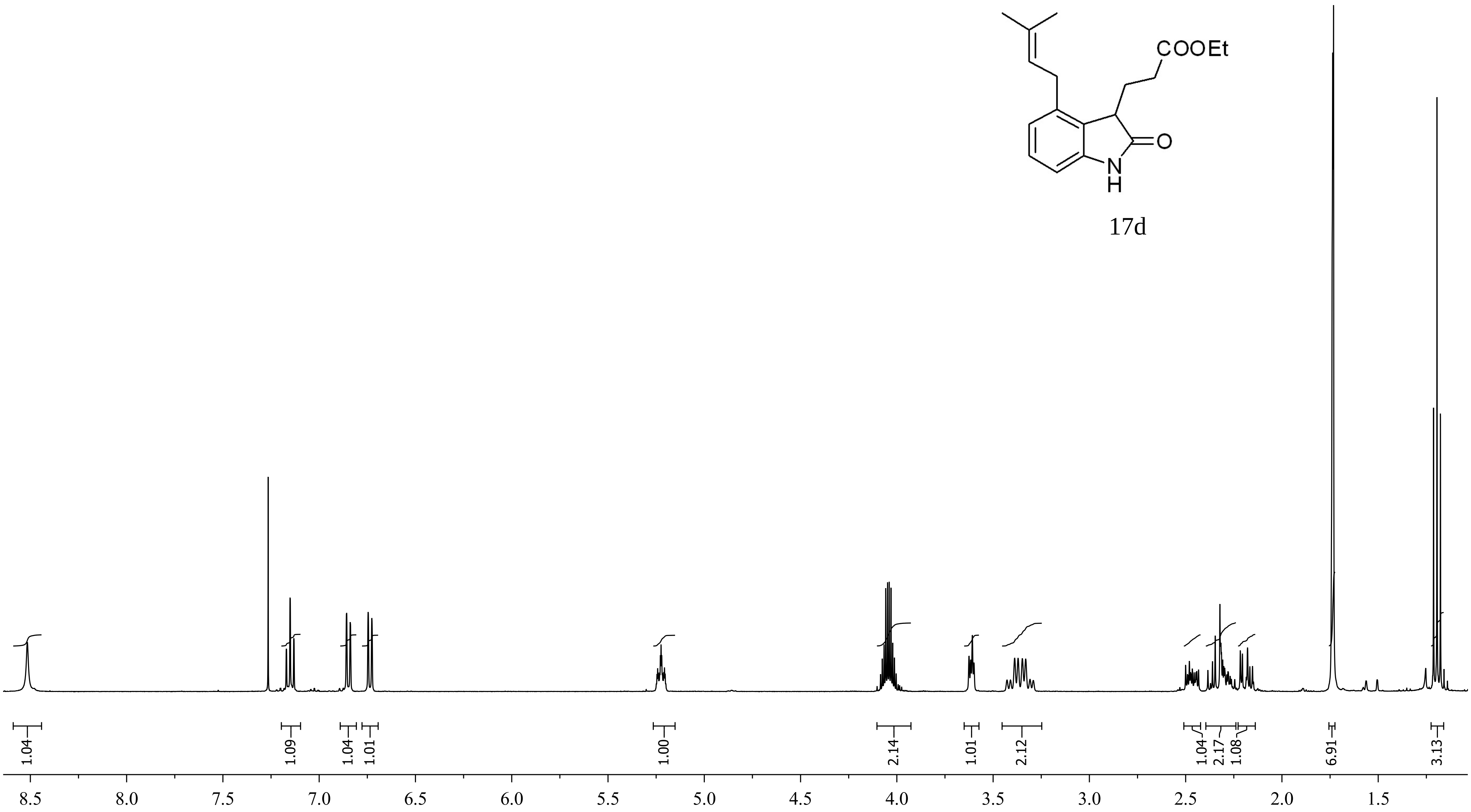

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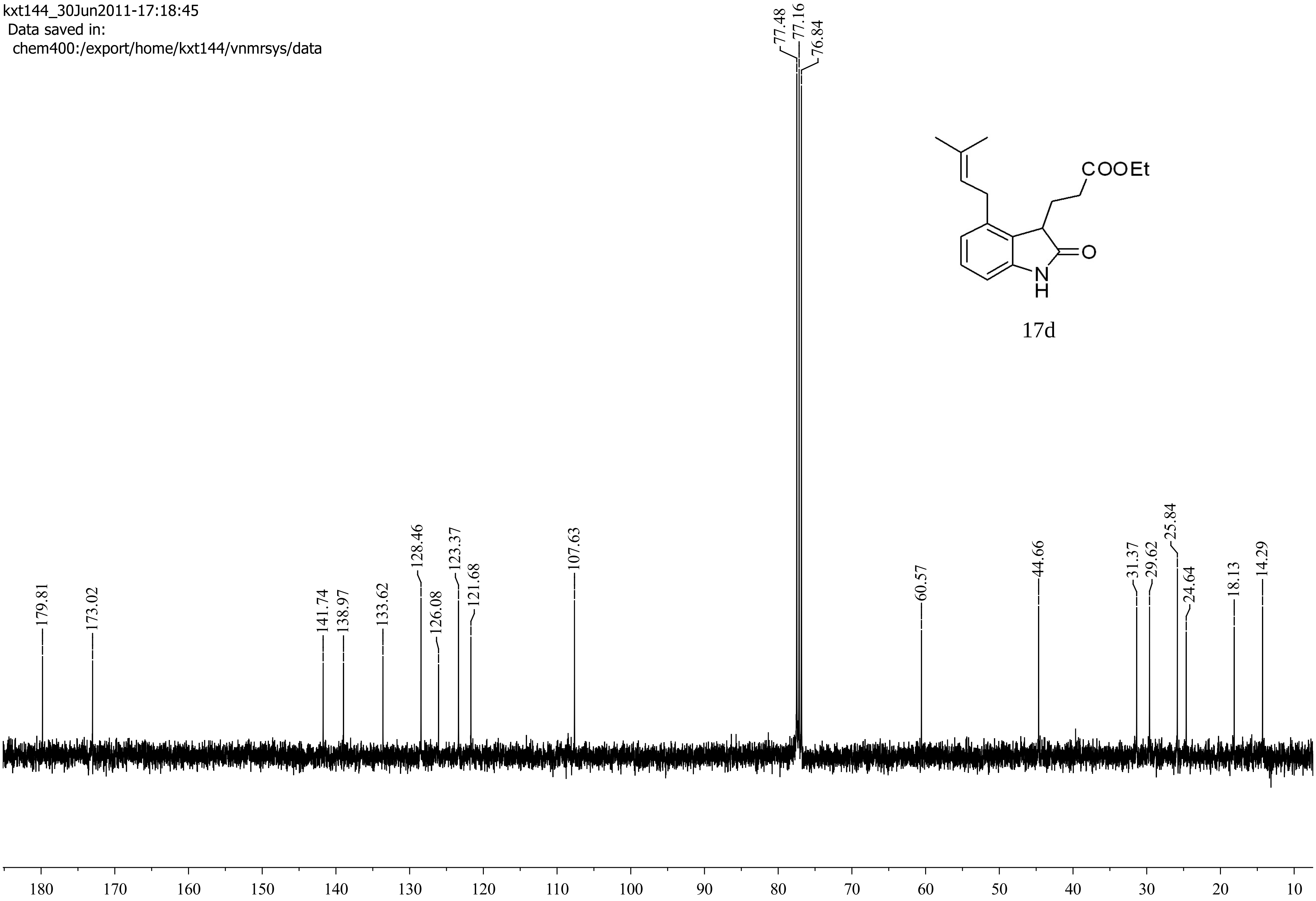

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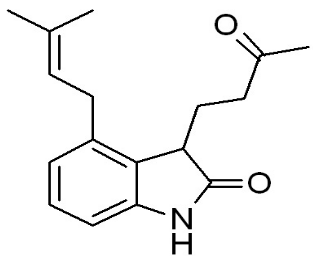

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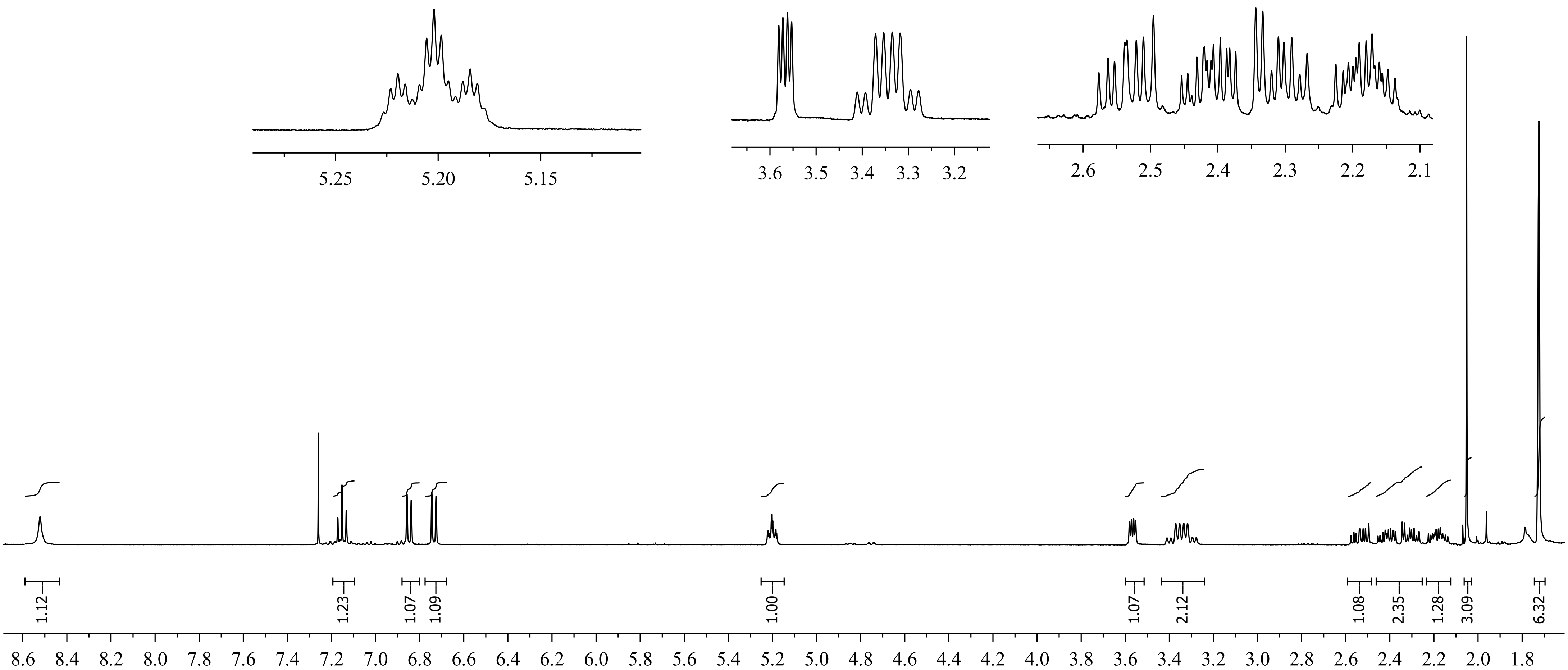

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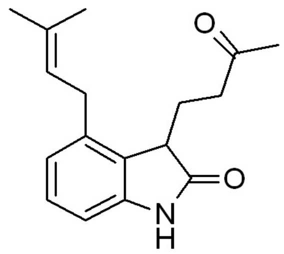

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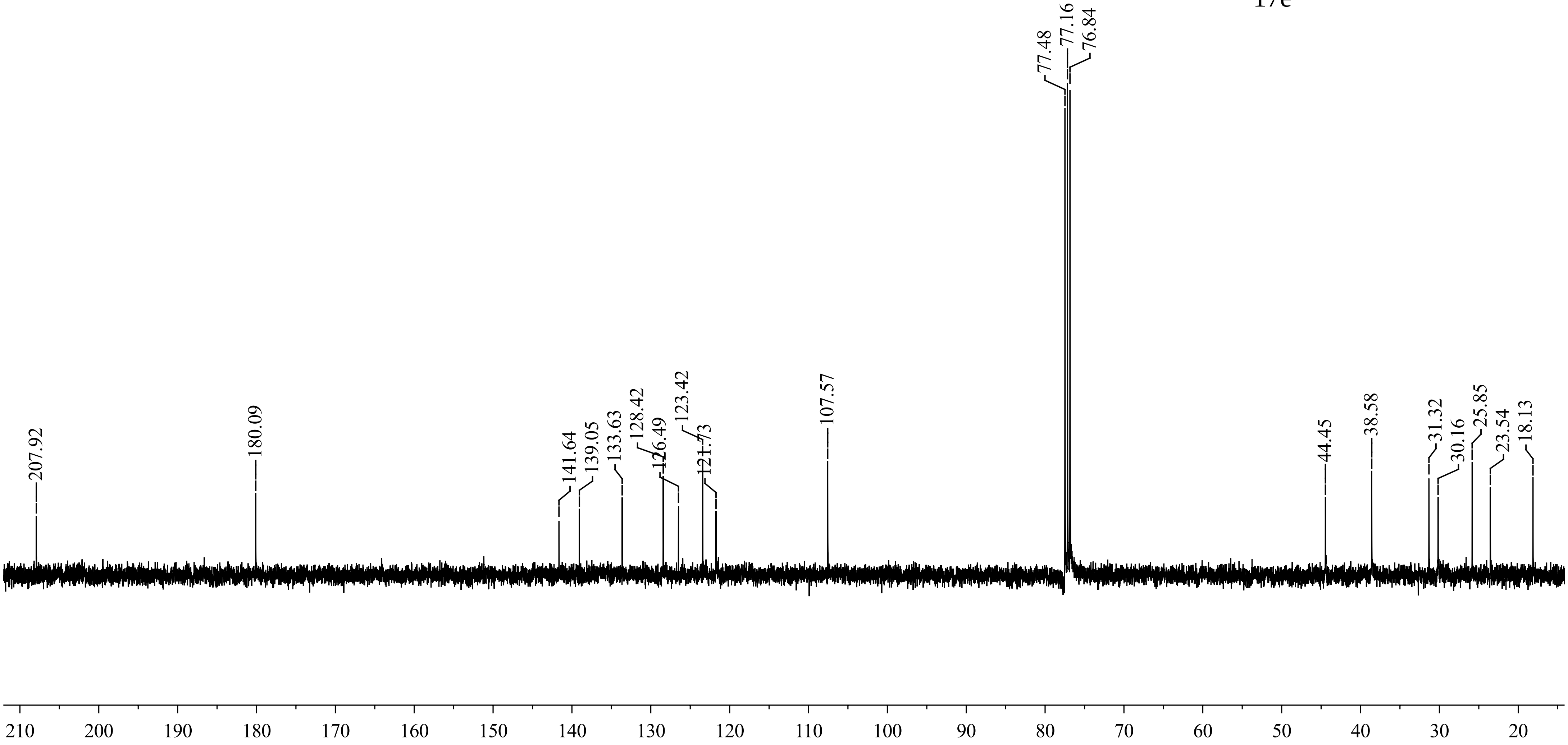

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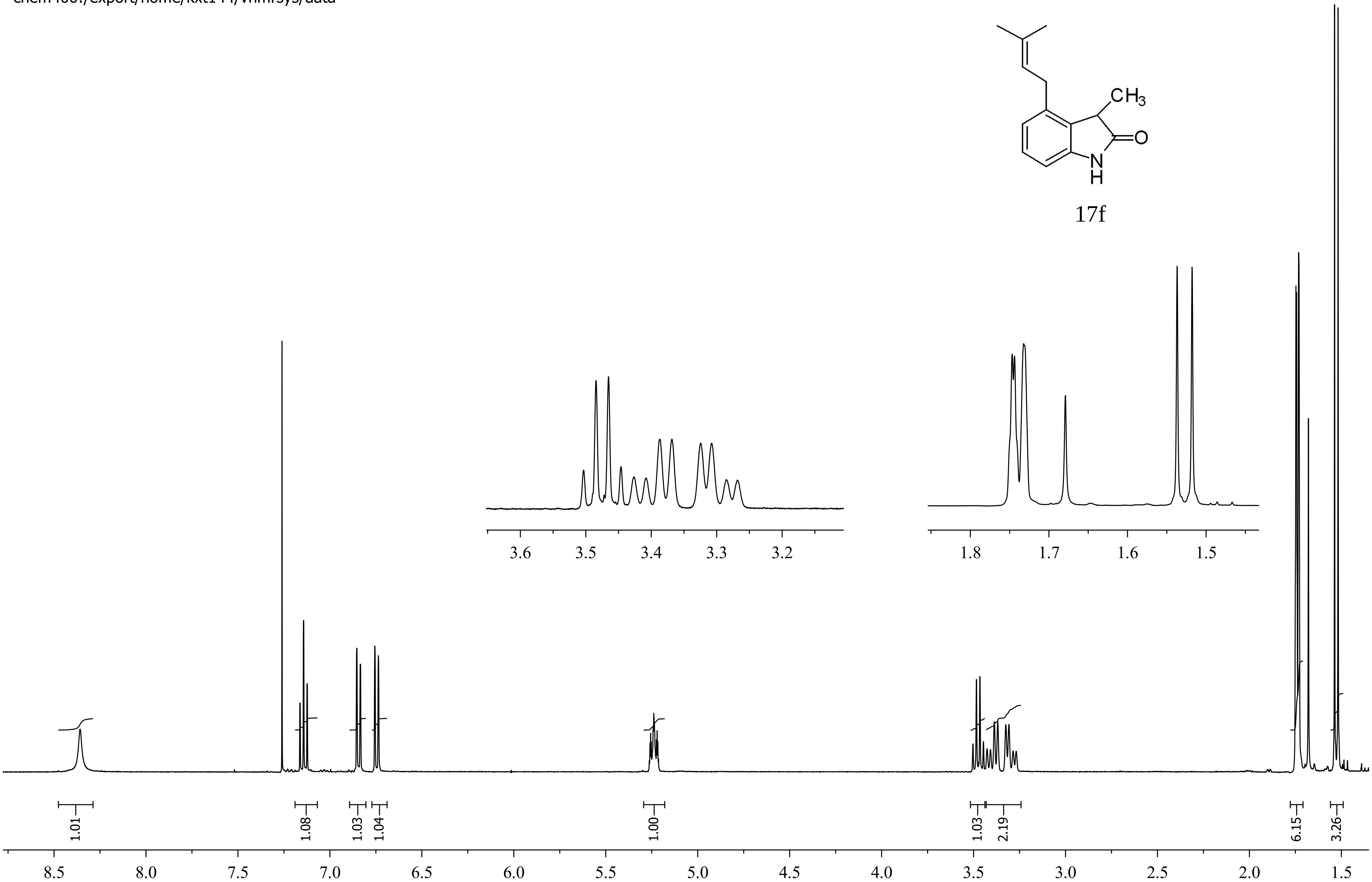

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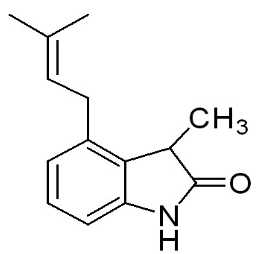

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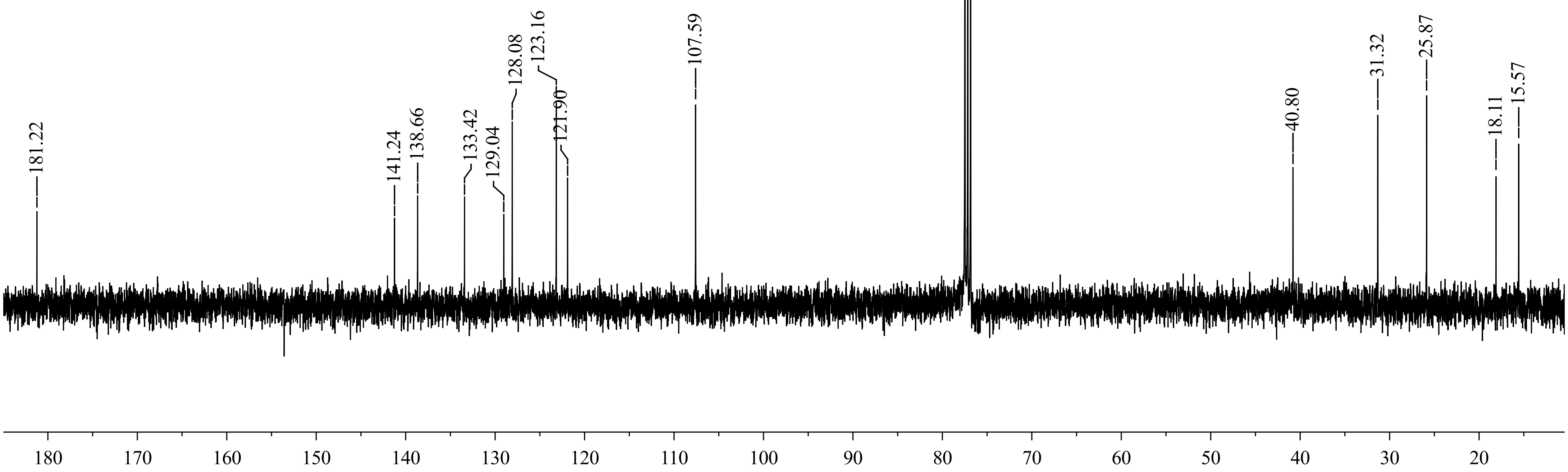

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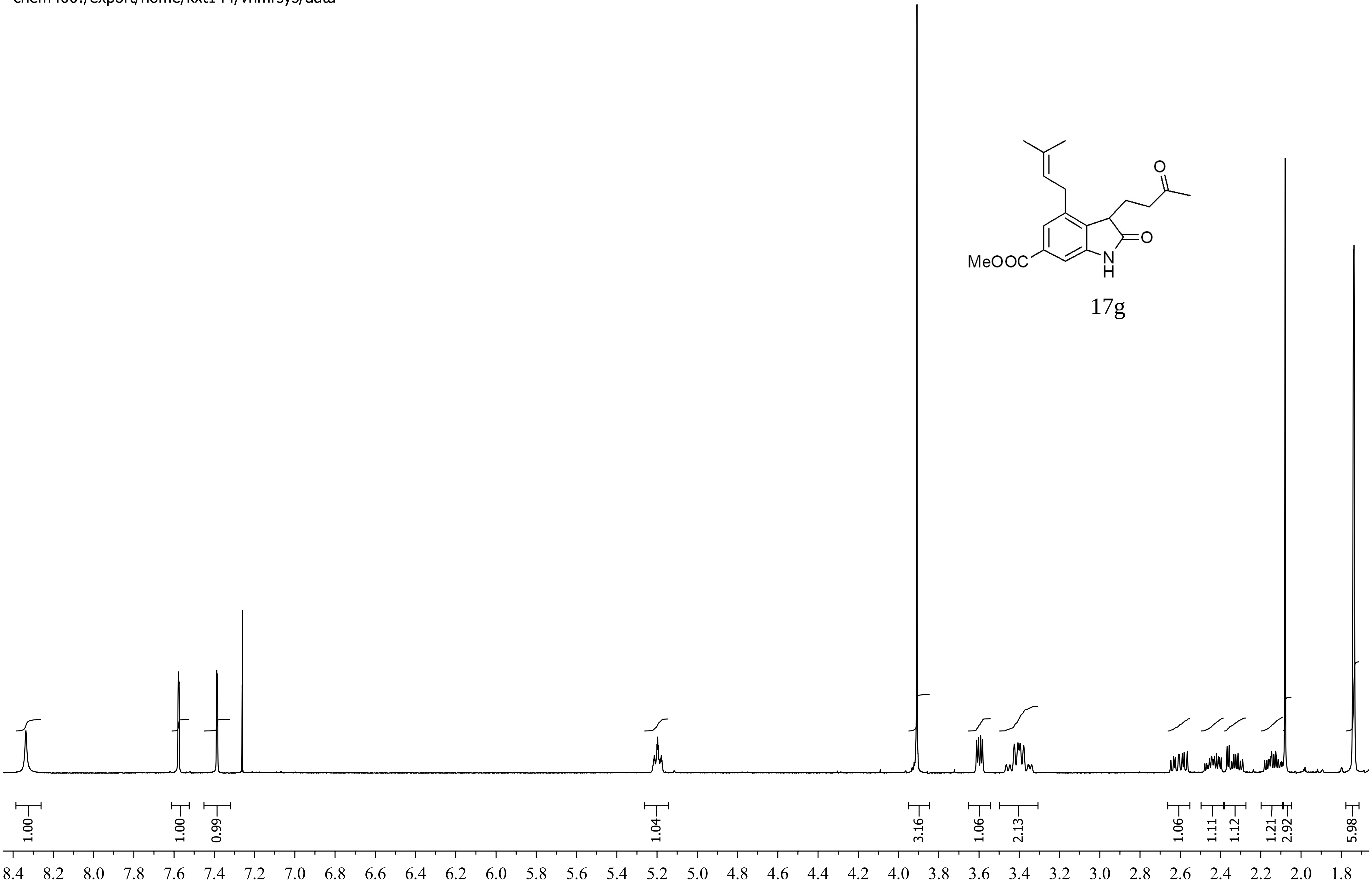

d83-2
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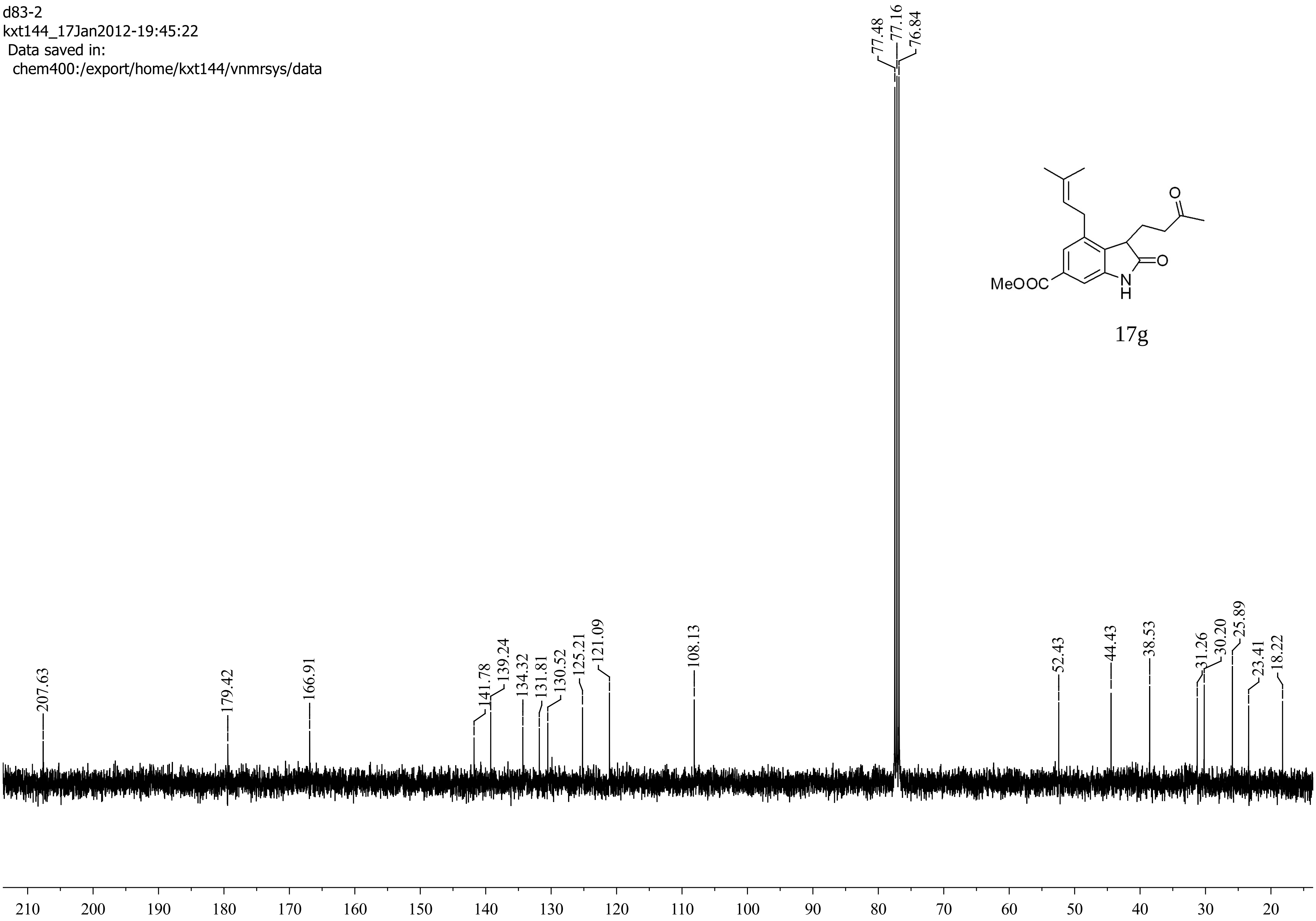

17g

PROTON
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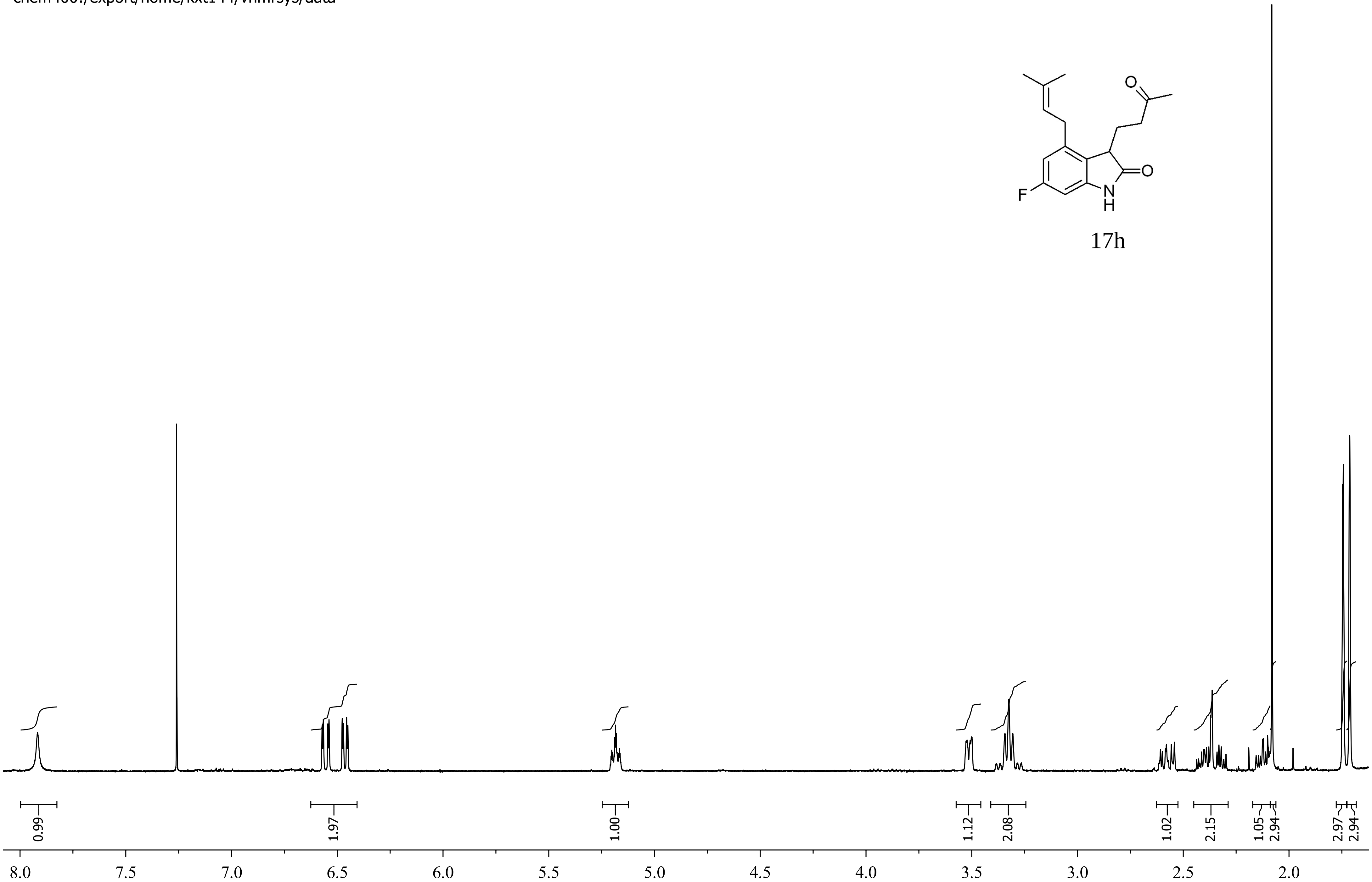

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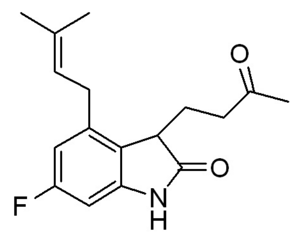
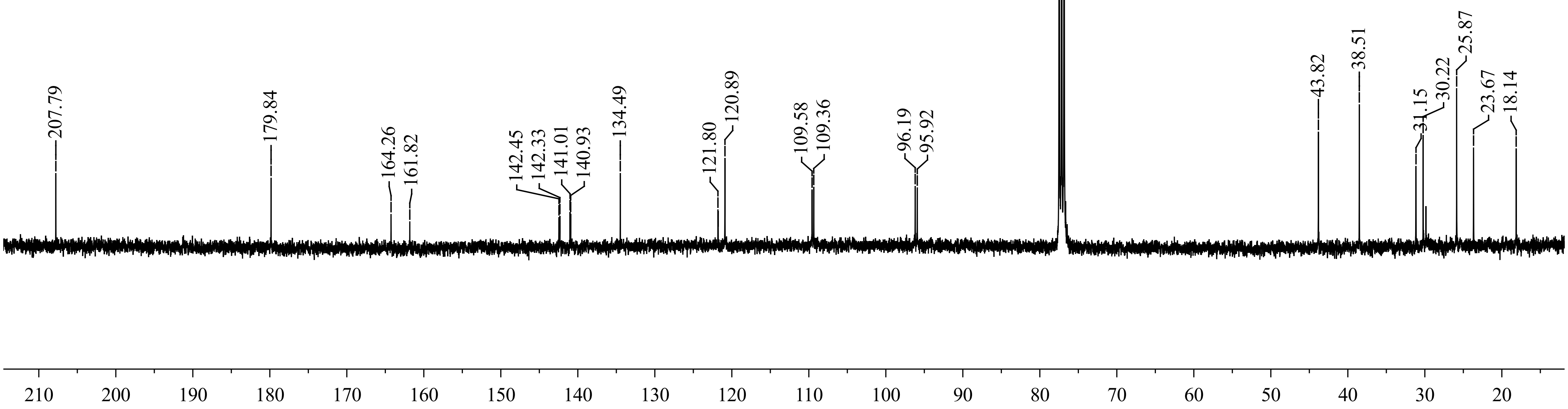


17h



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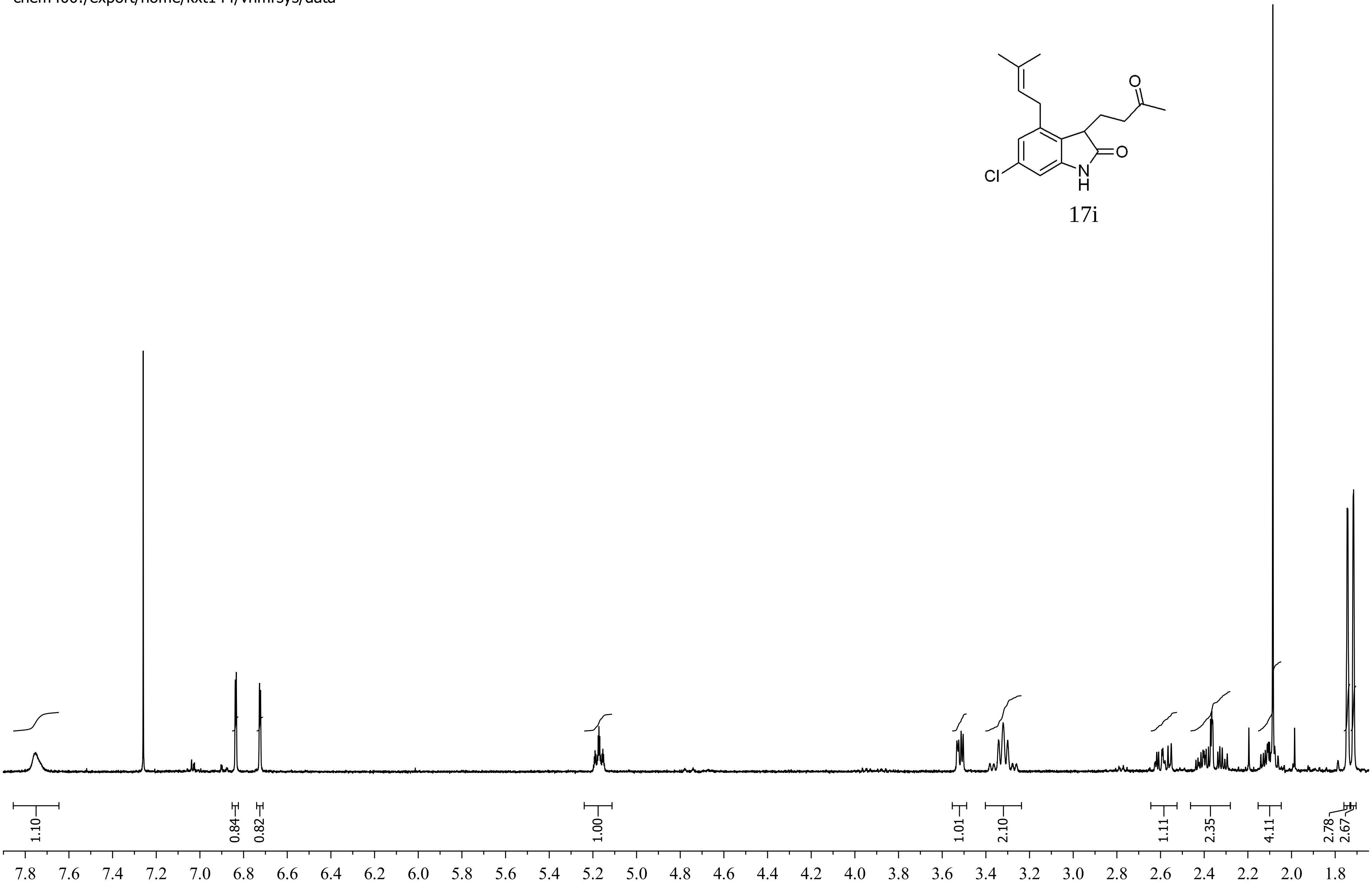

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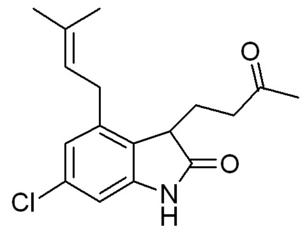
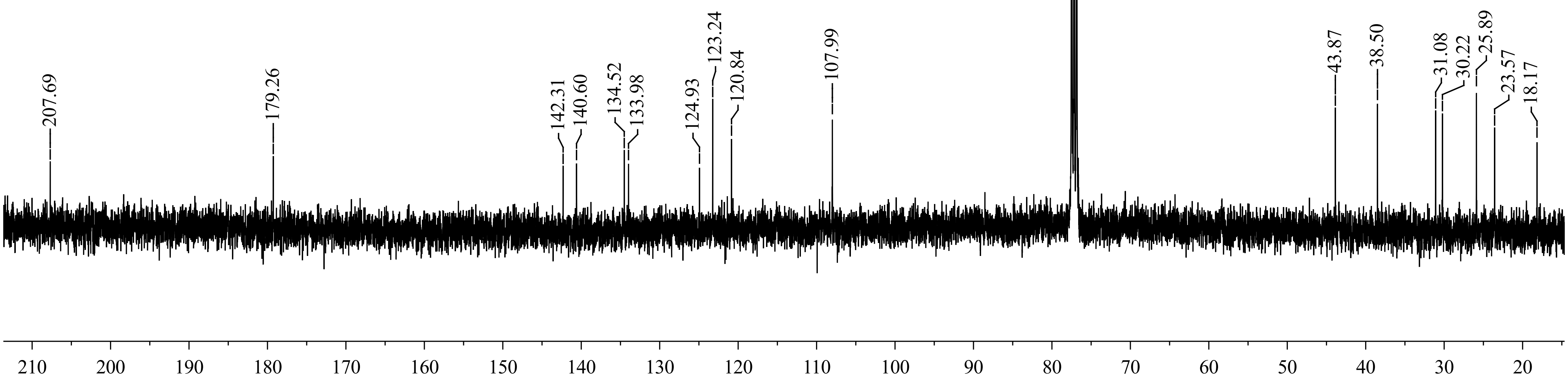


17i



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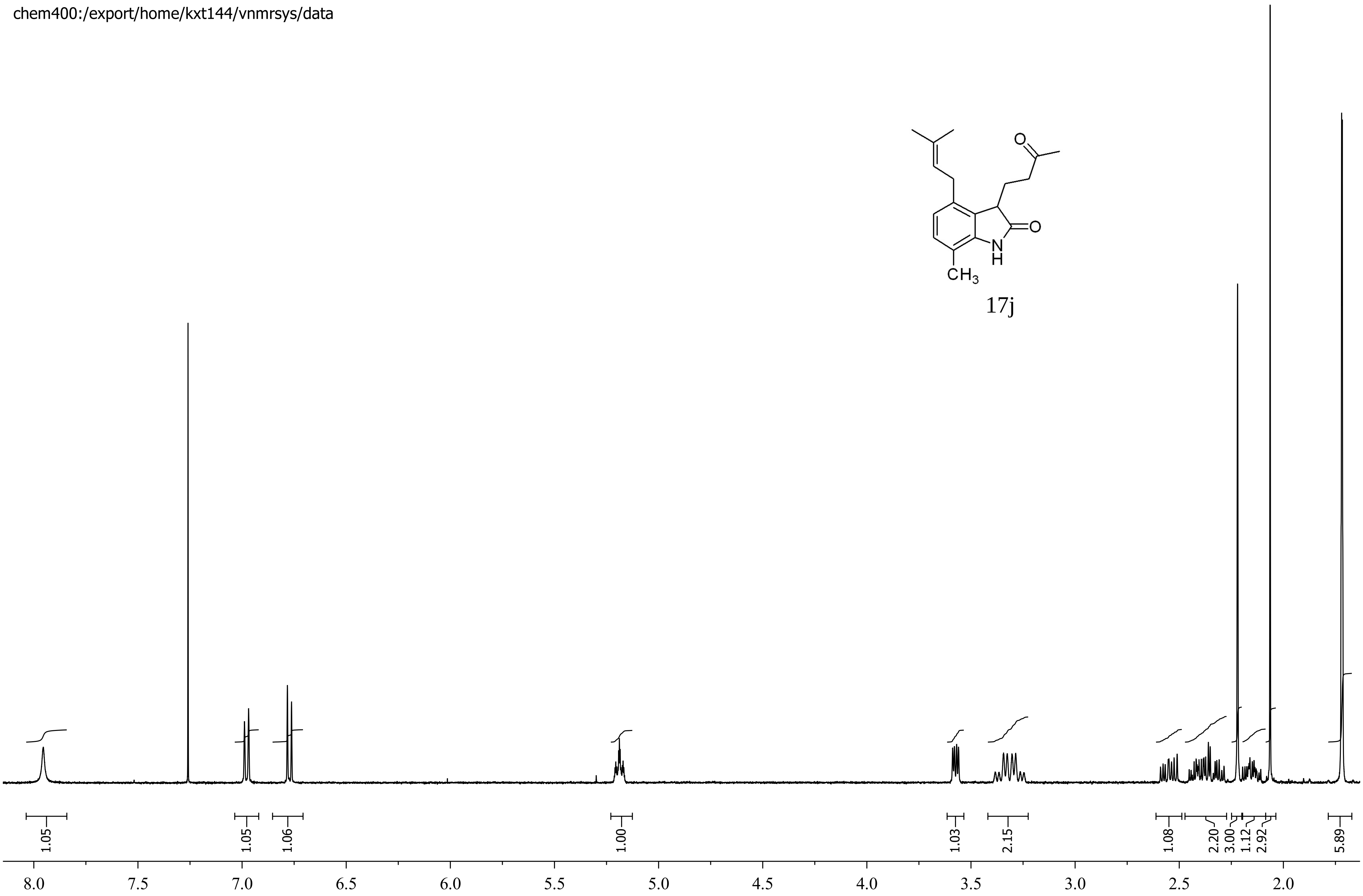

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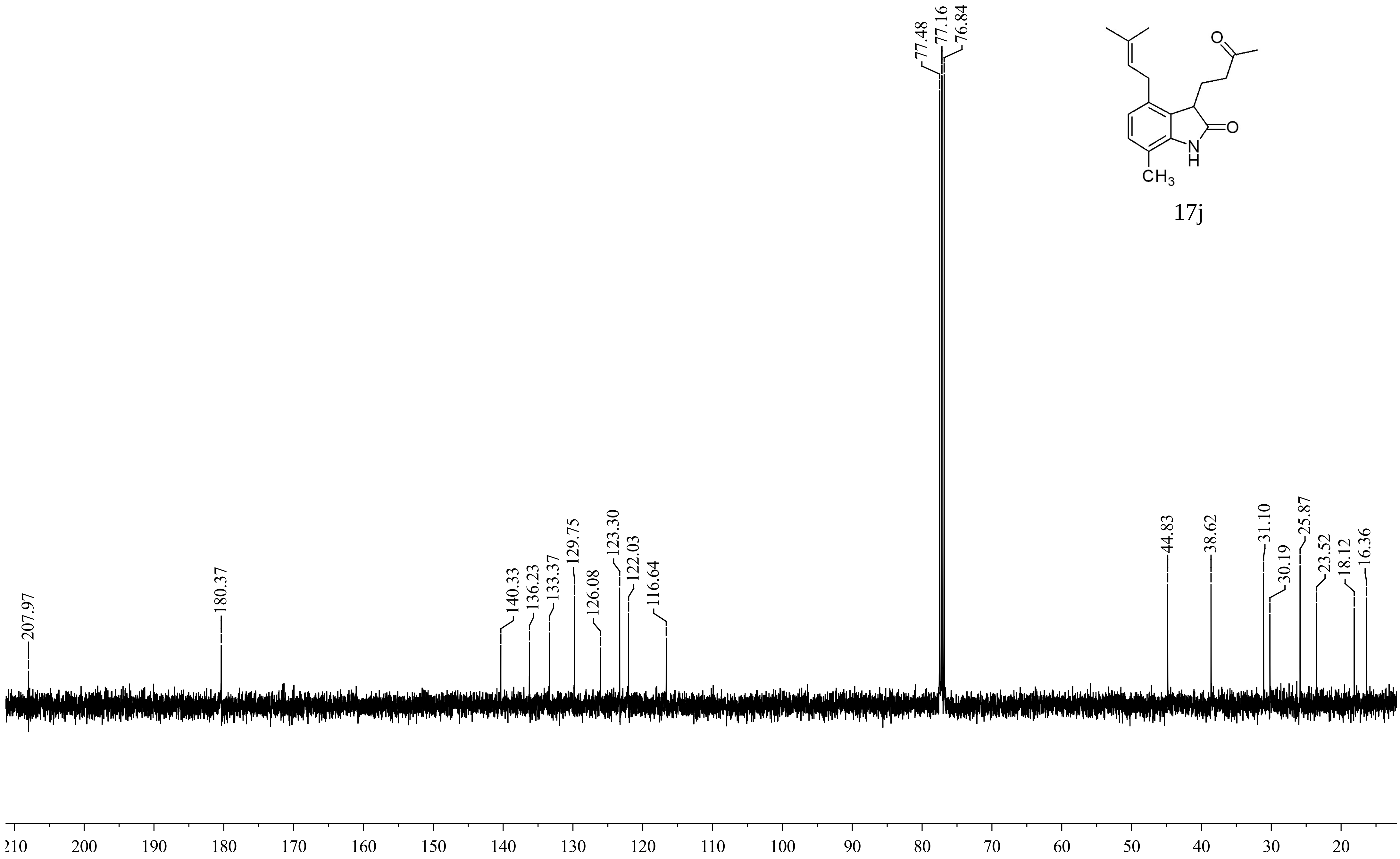

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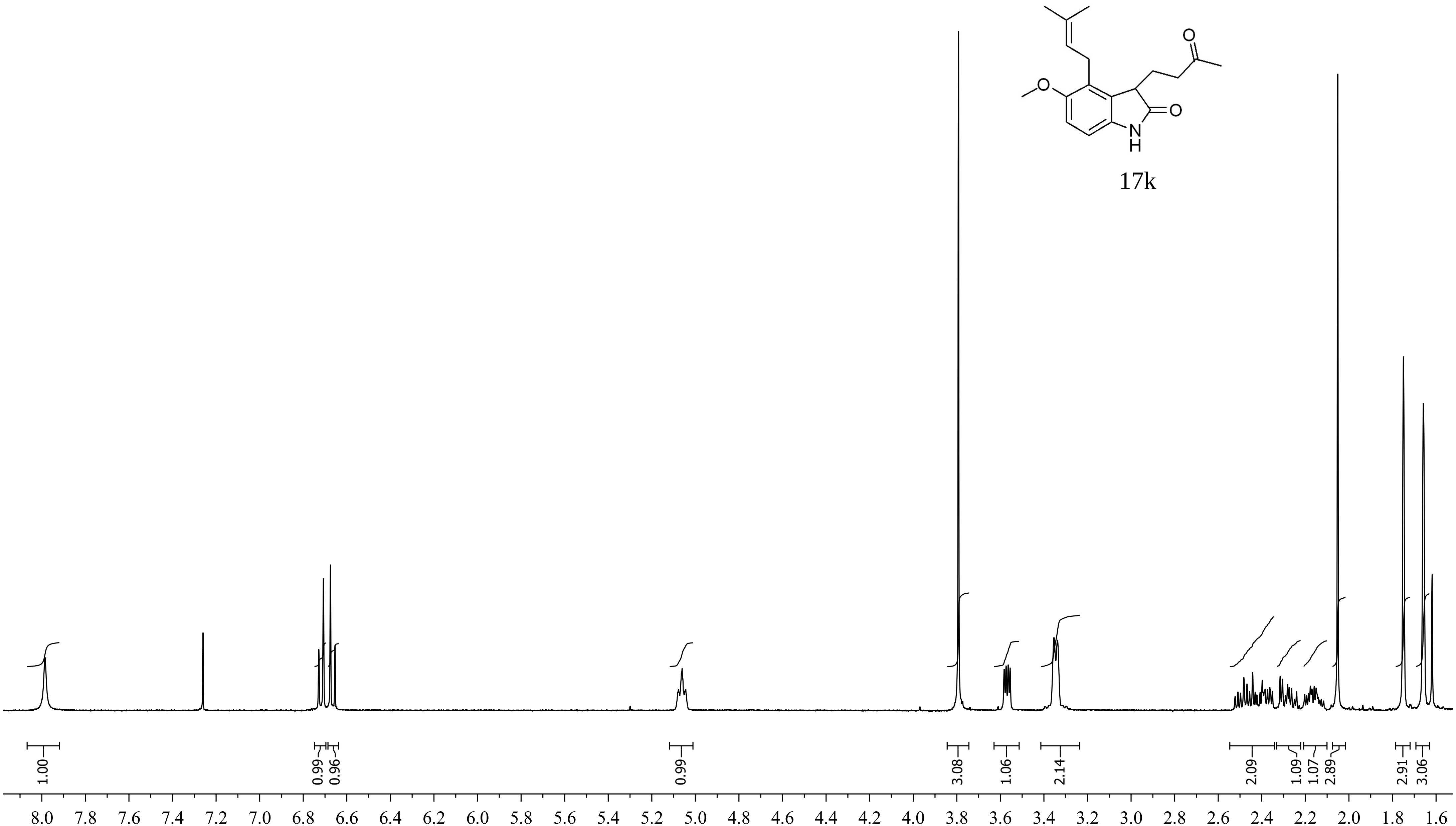

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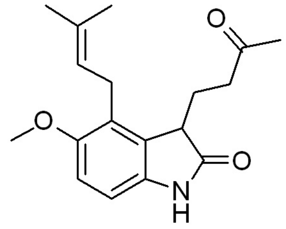
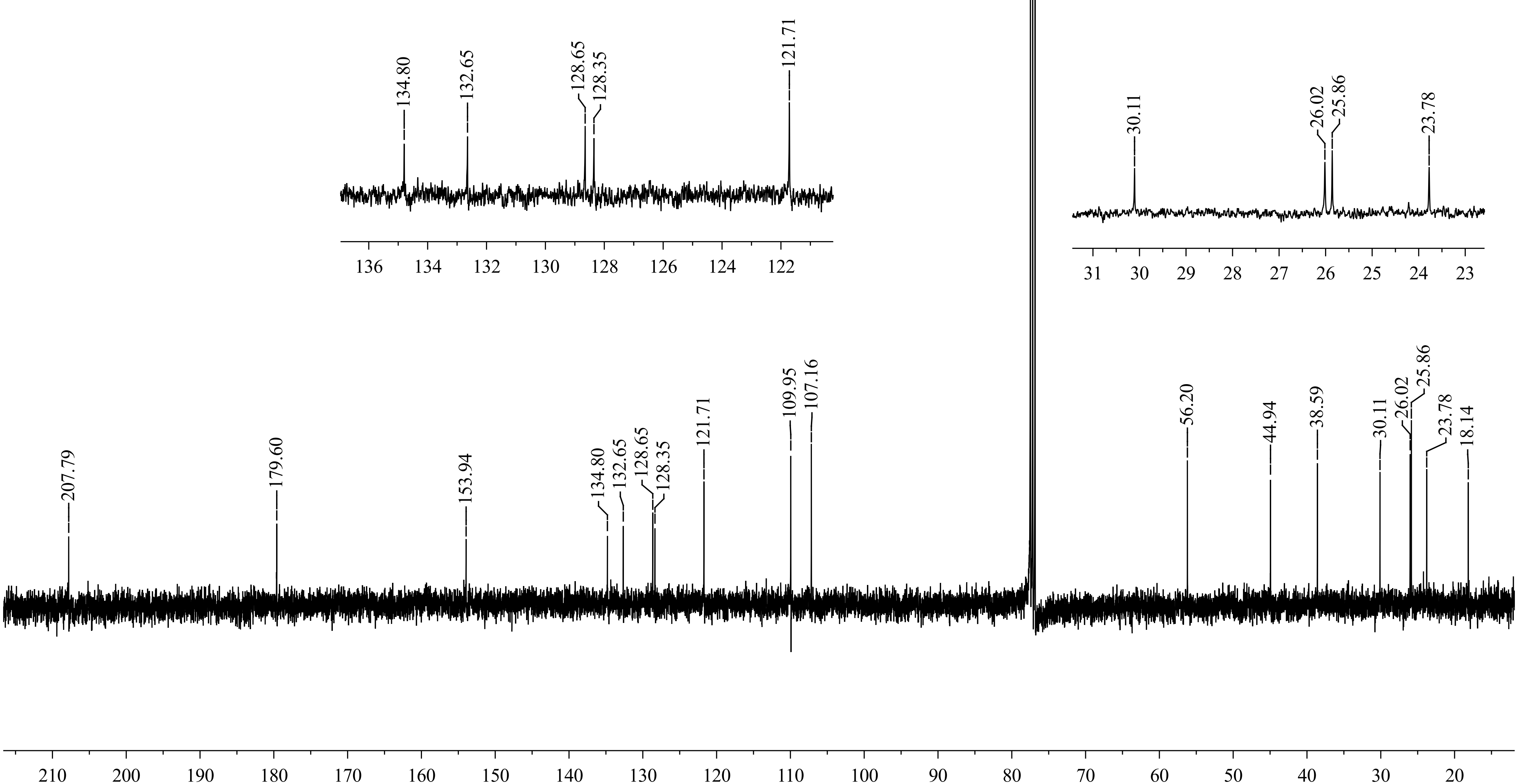


17k



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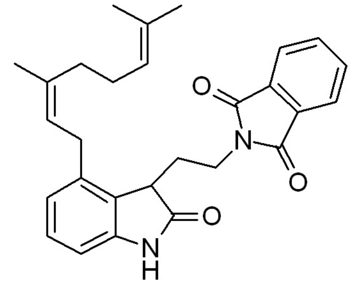
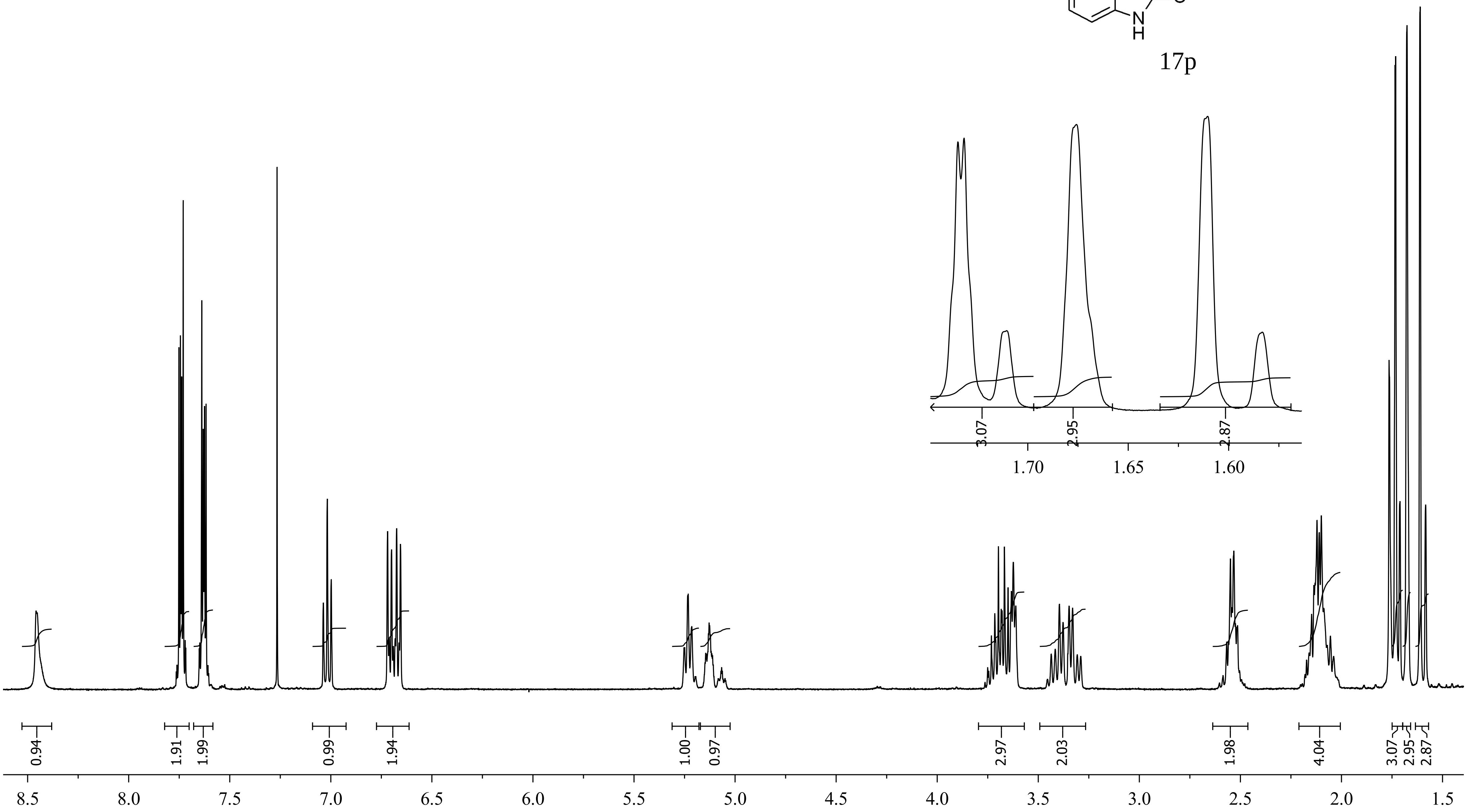


17p



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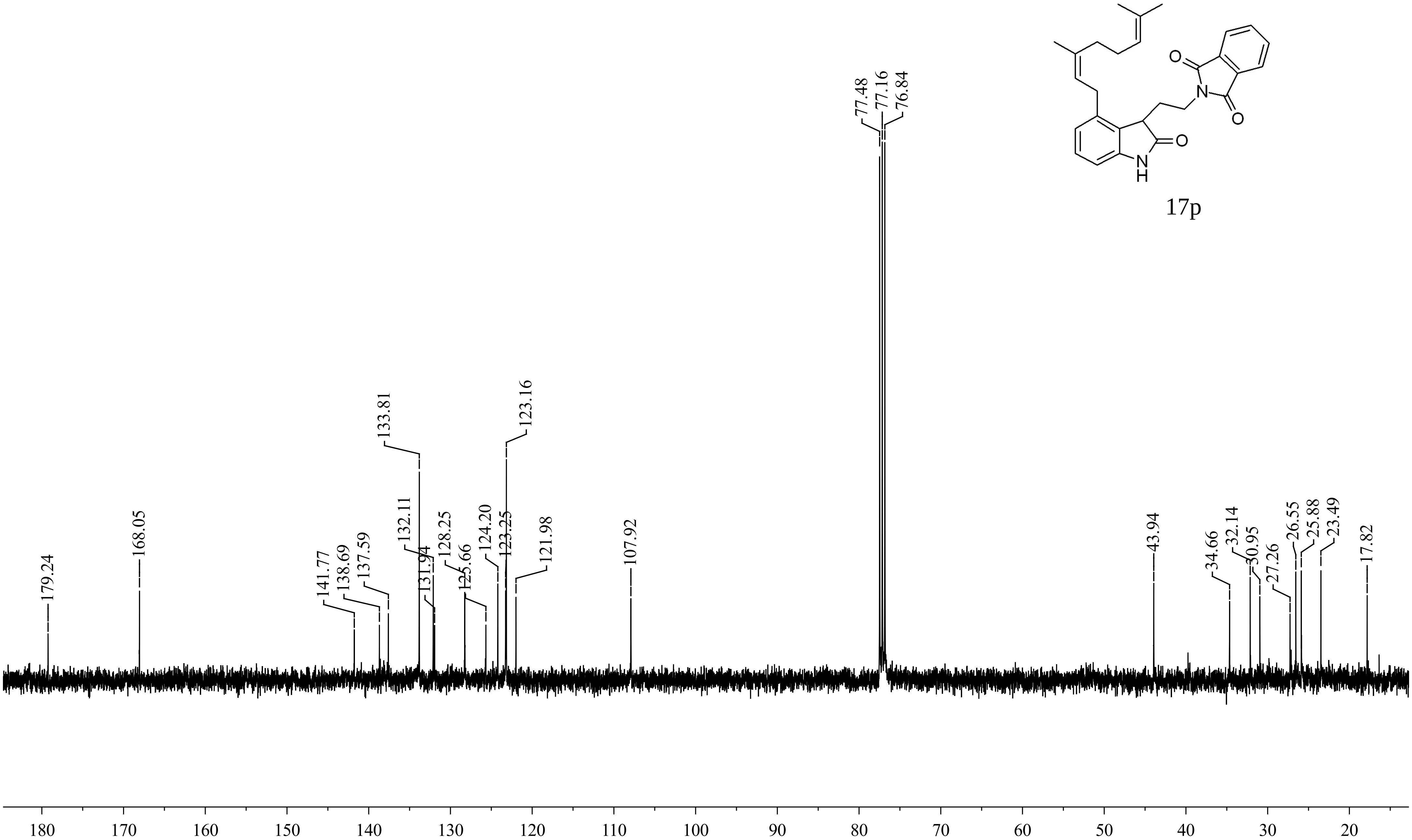

17p

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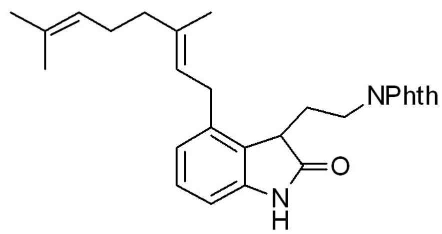
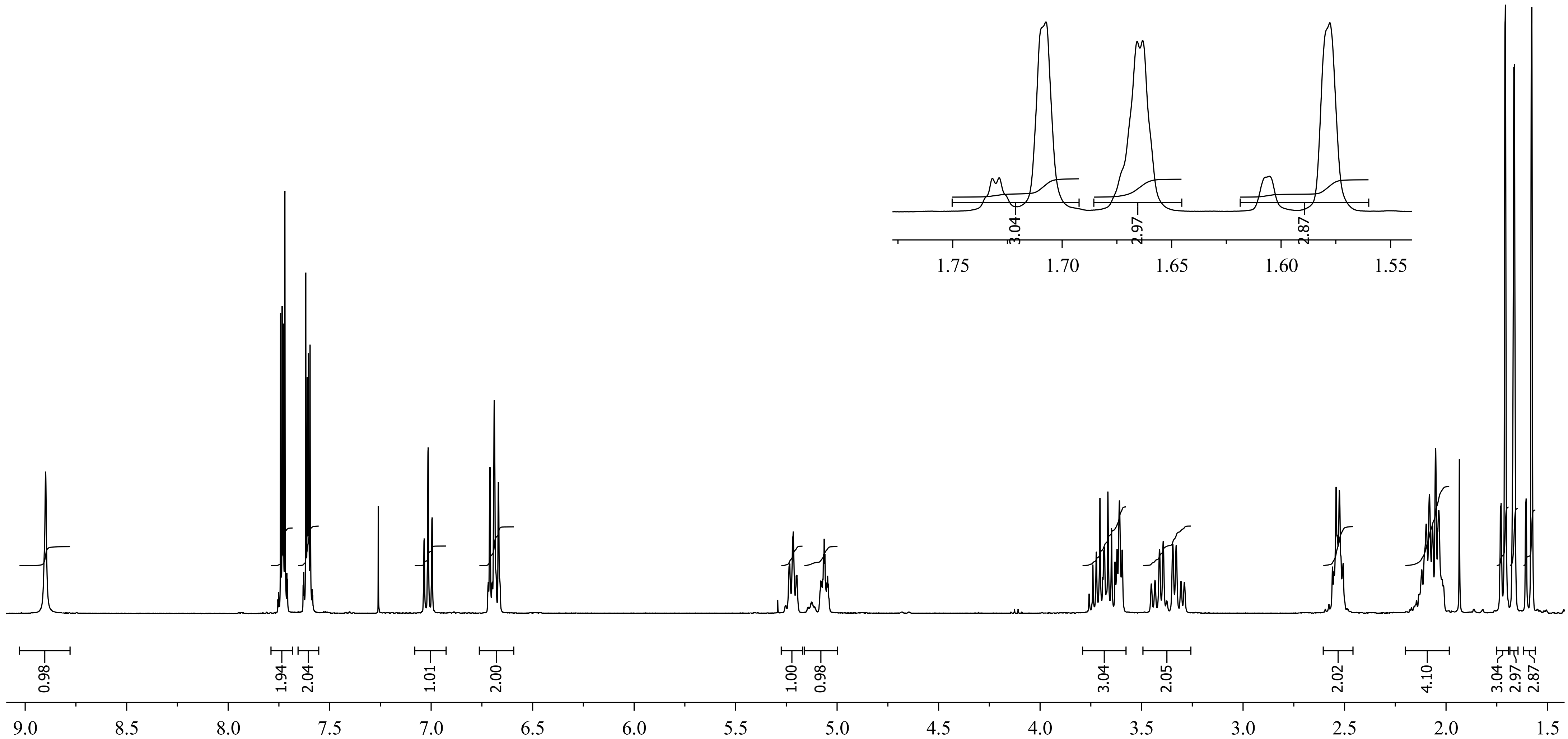


17q



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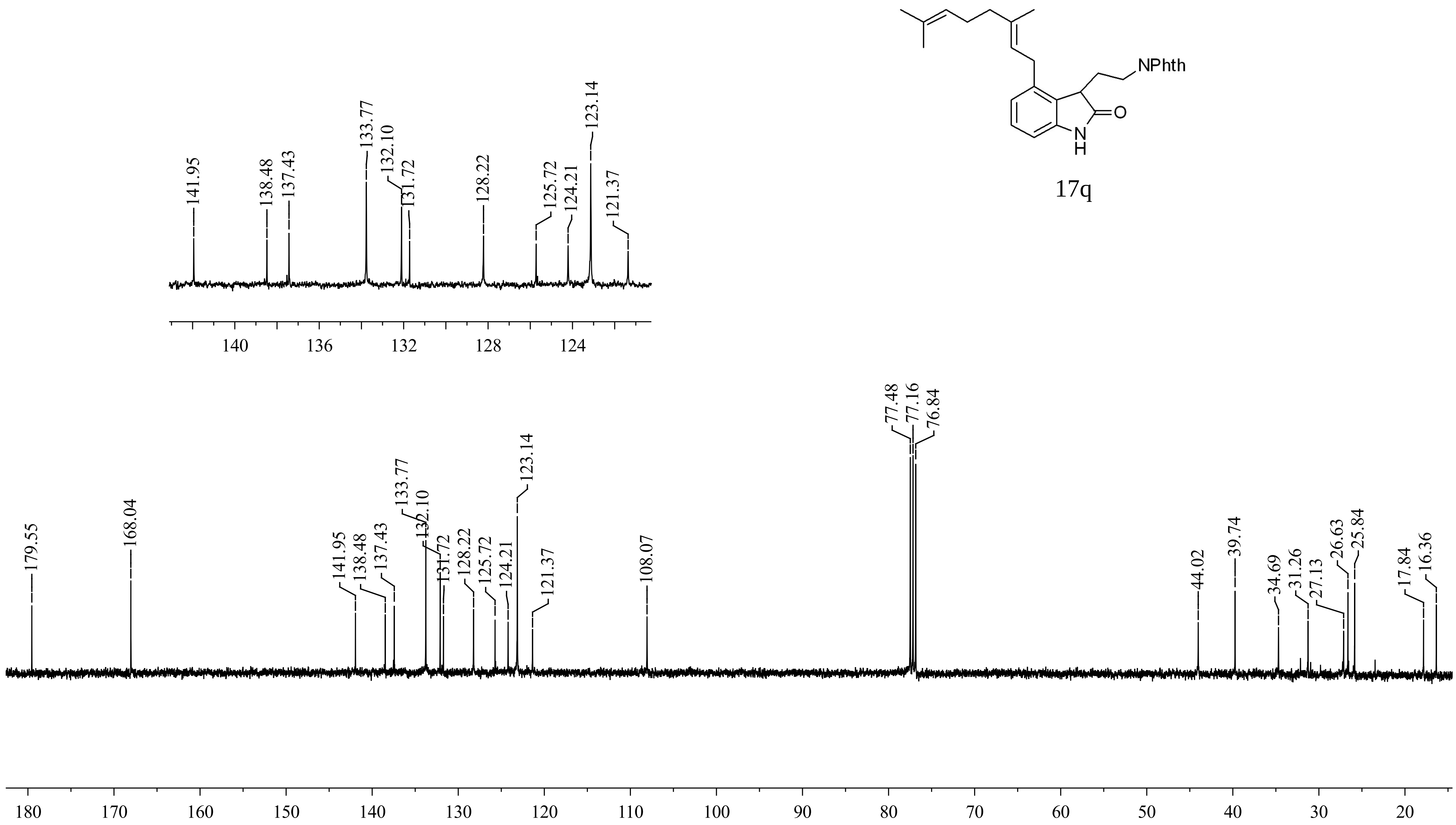

17q

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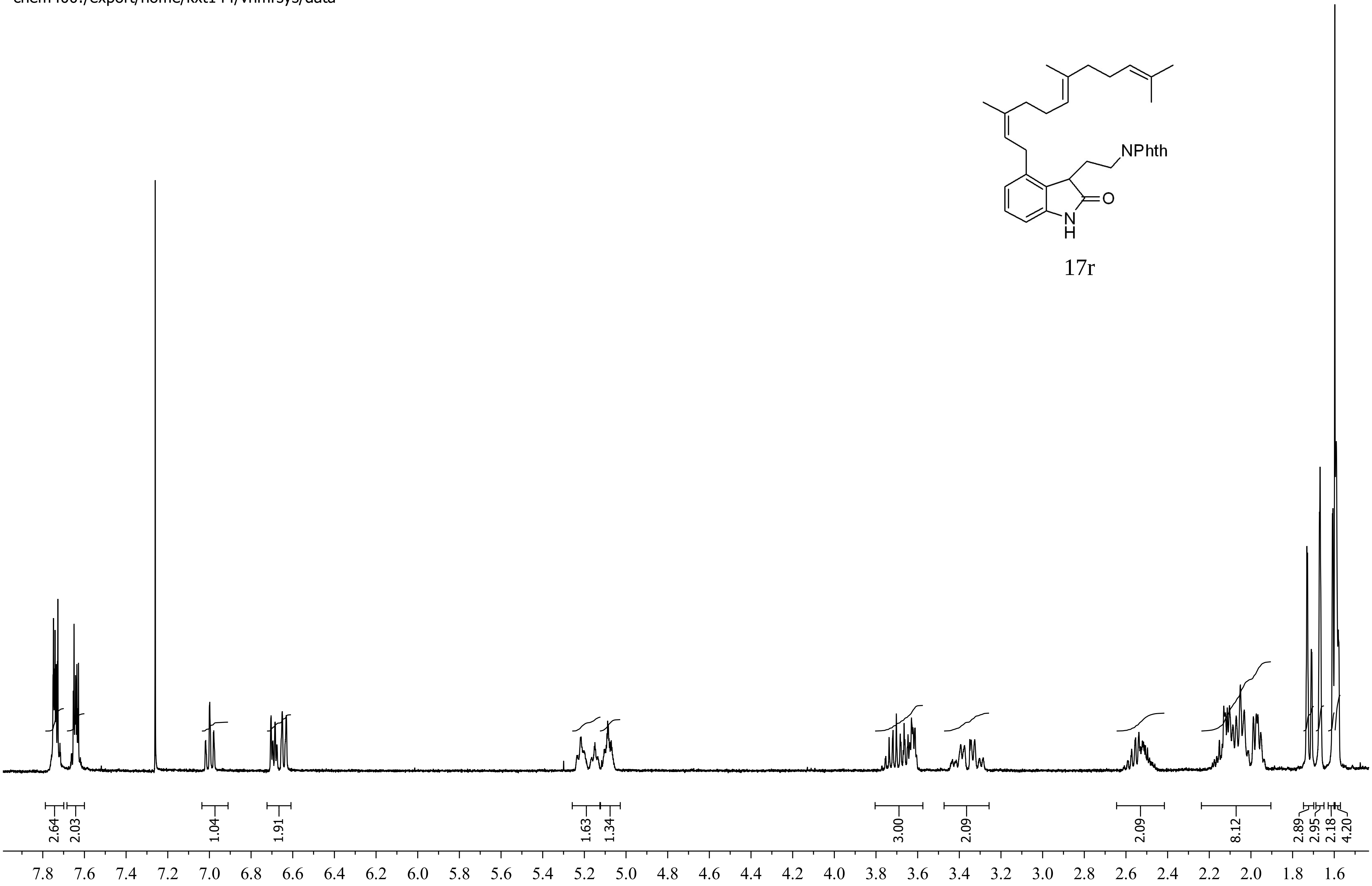

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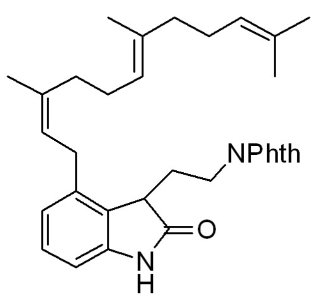
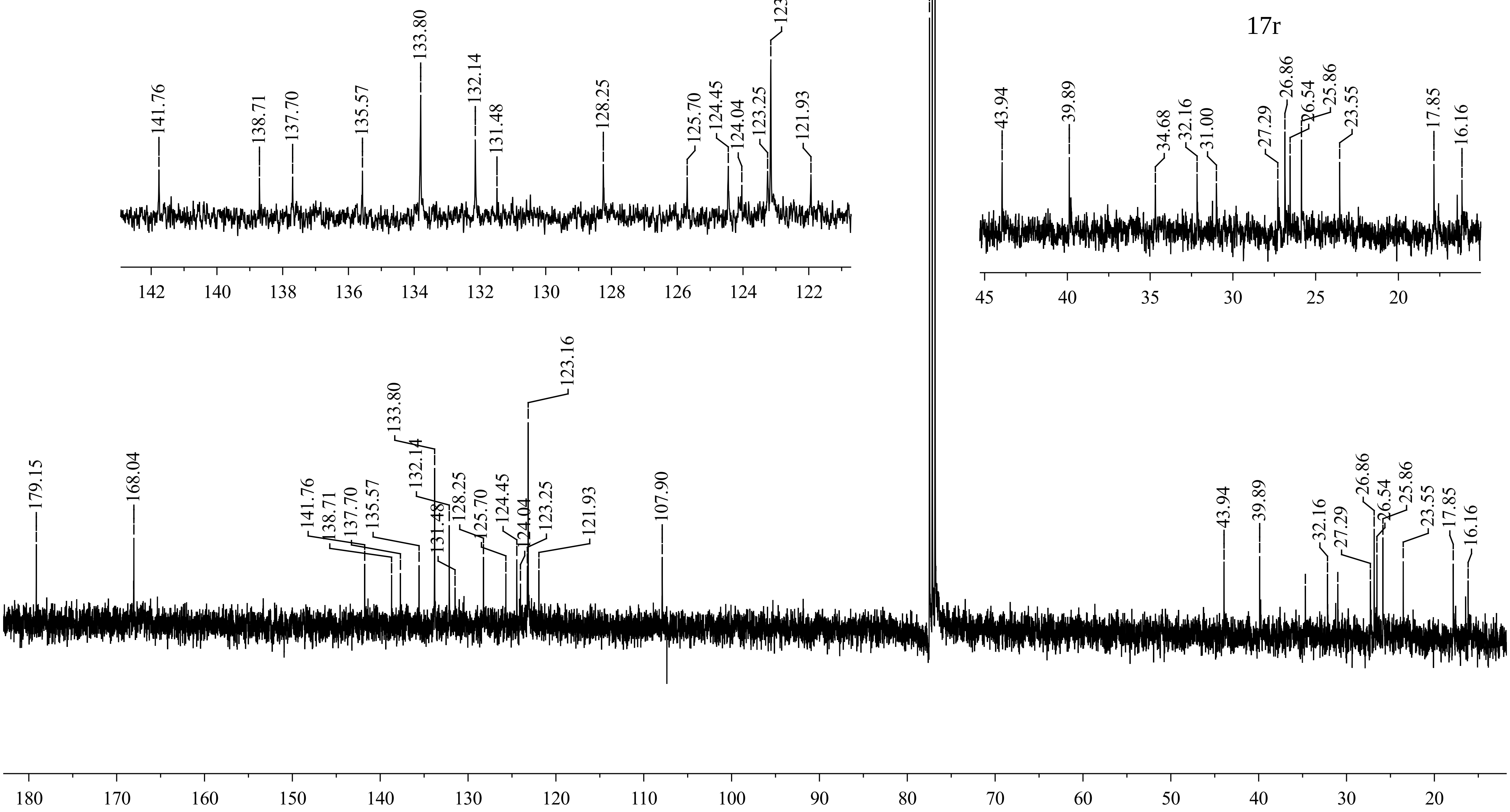


17r



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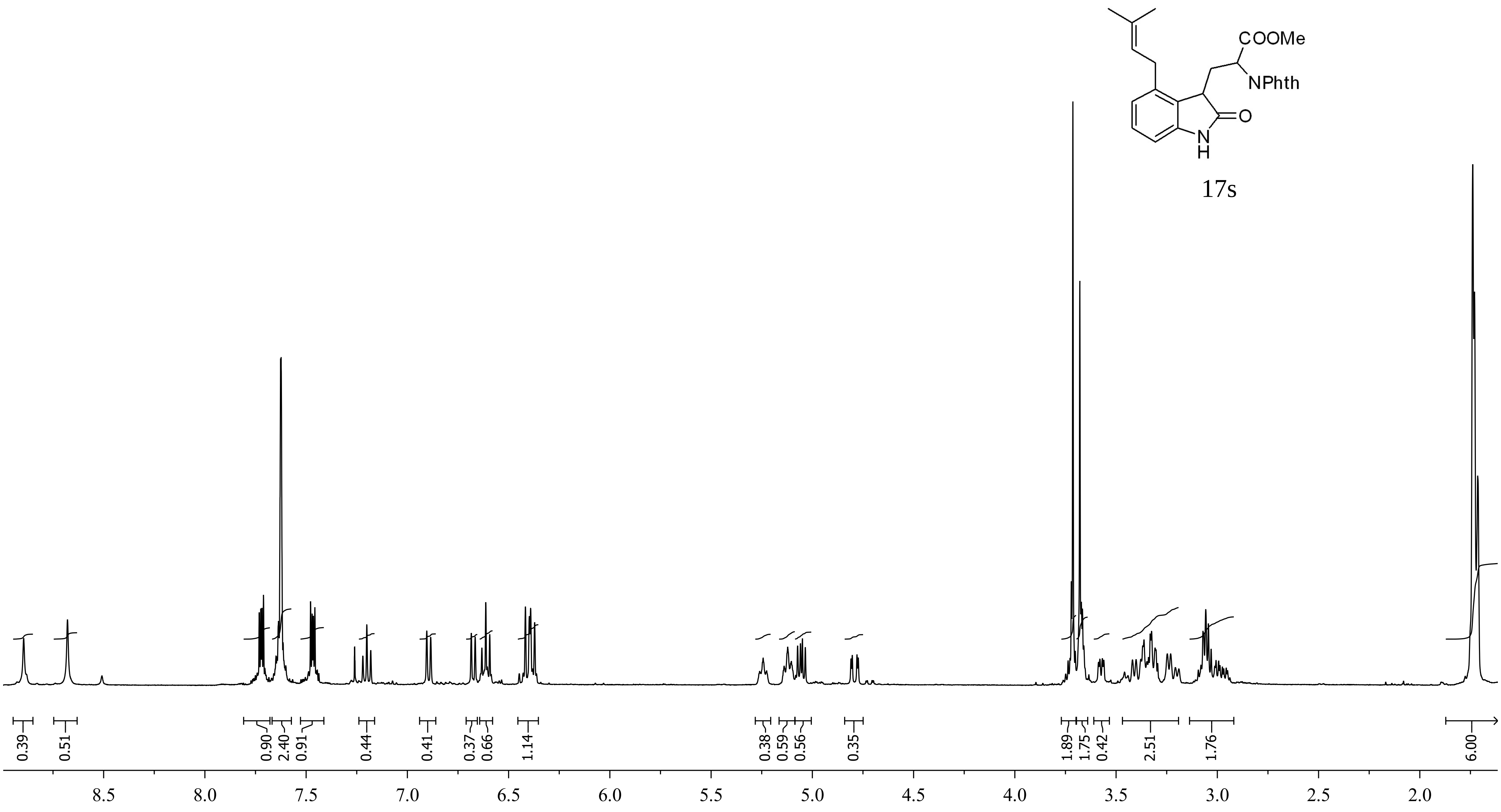

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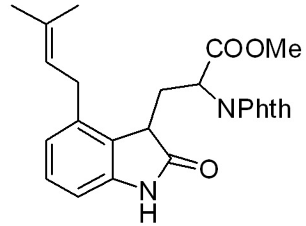
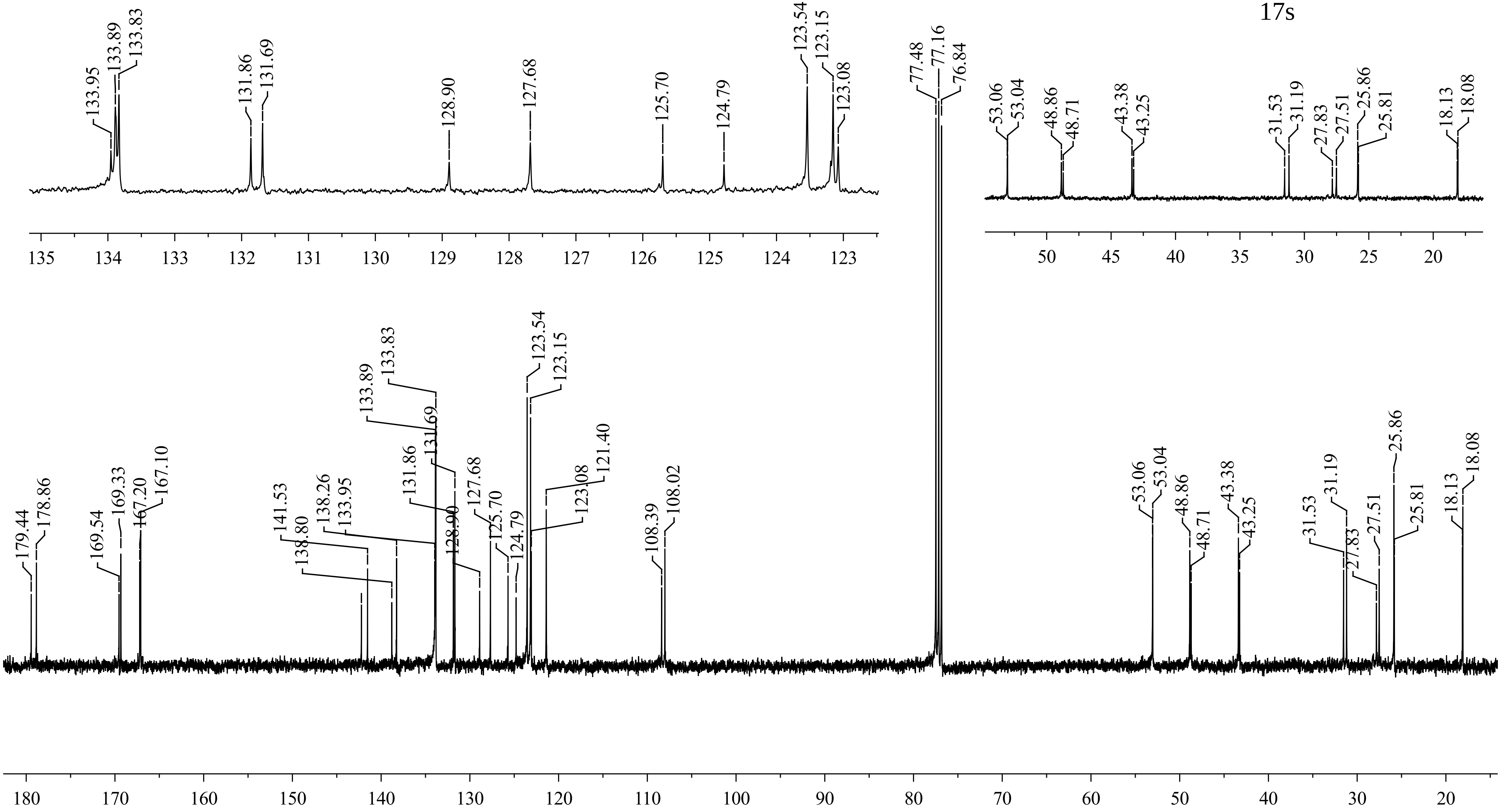


17s



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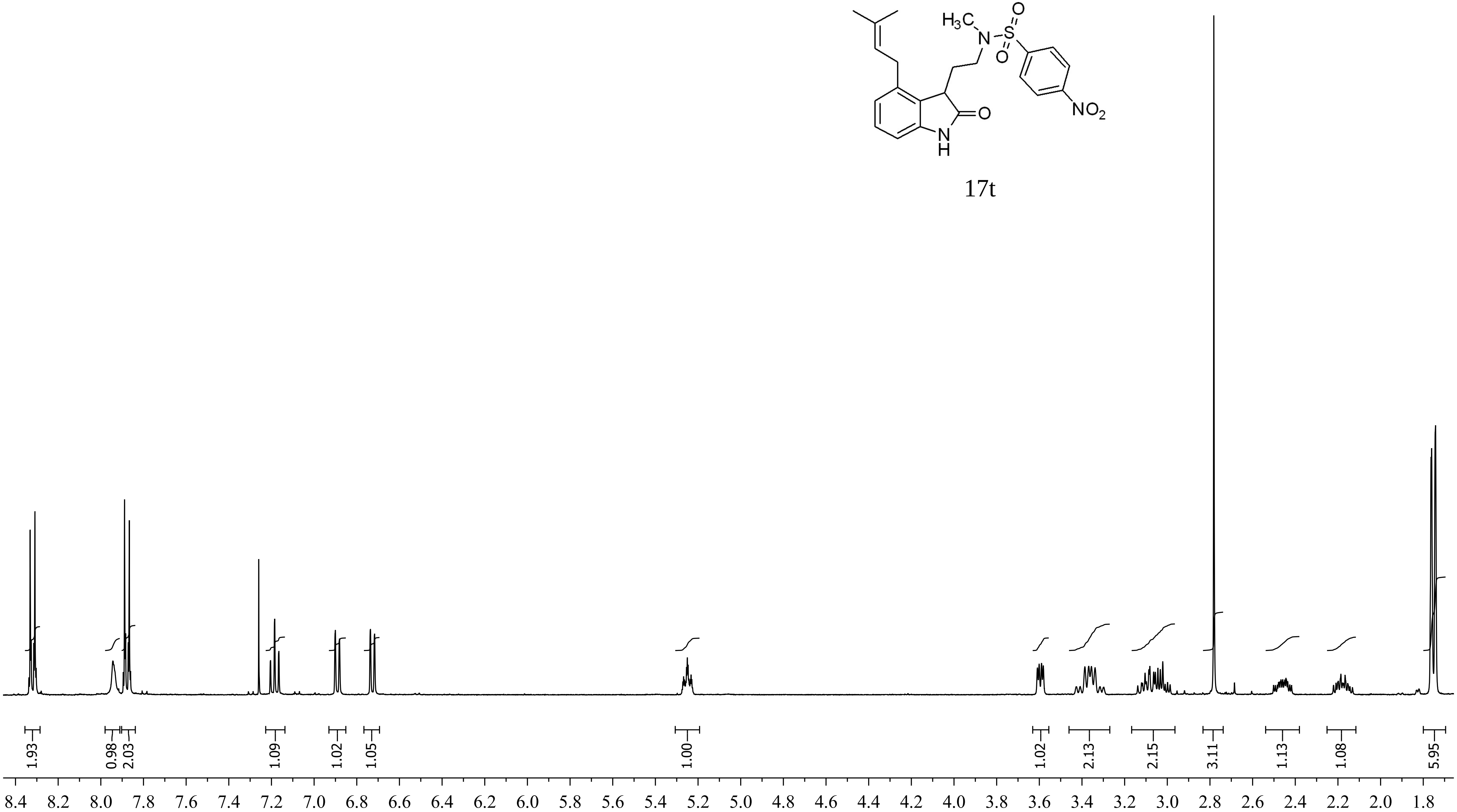

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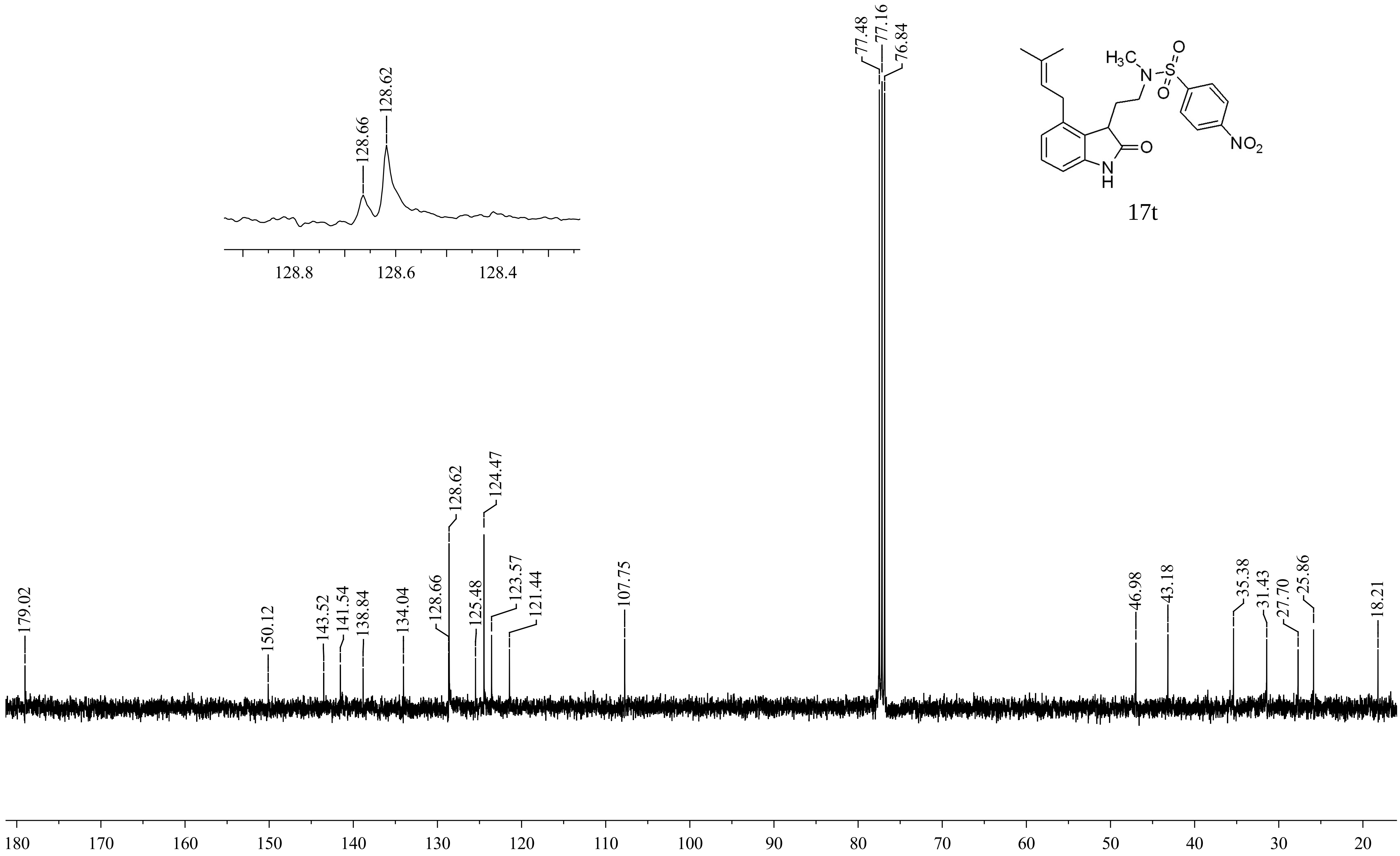

17t

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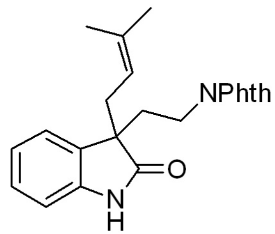
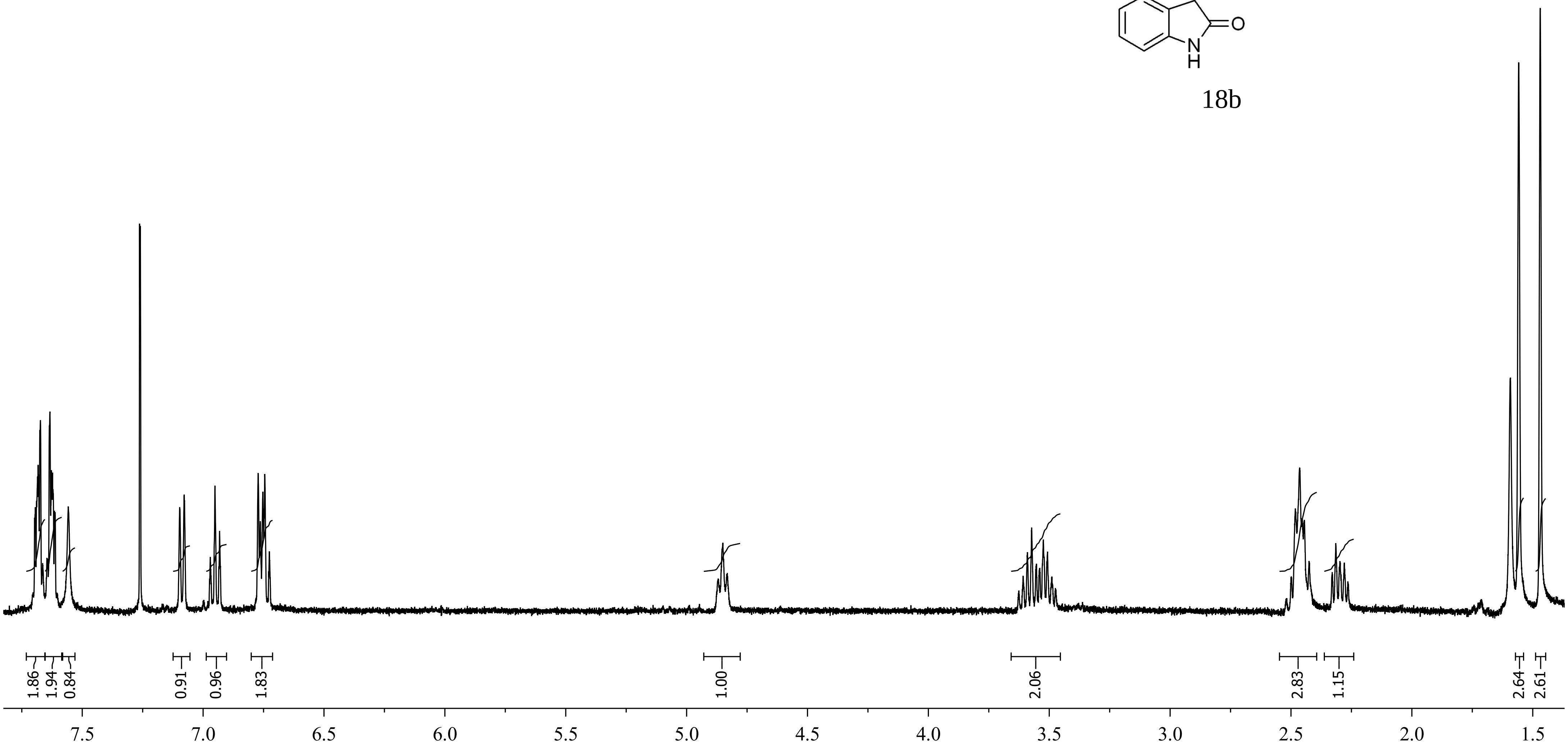


18b



0296c
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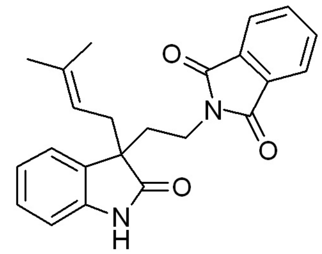

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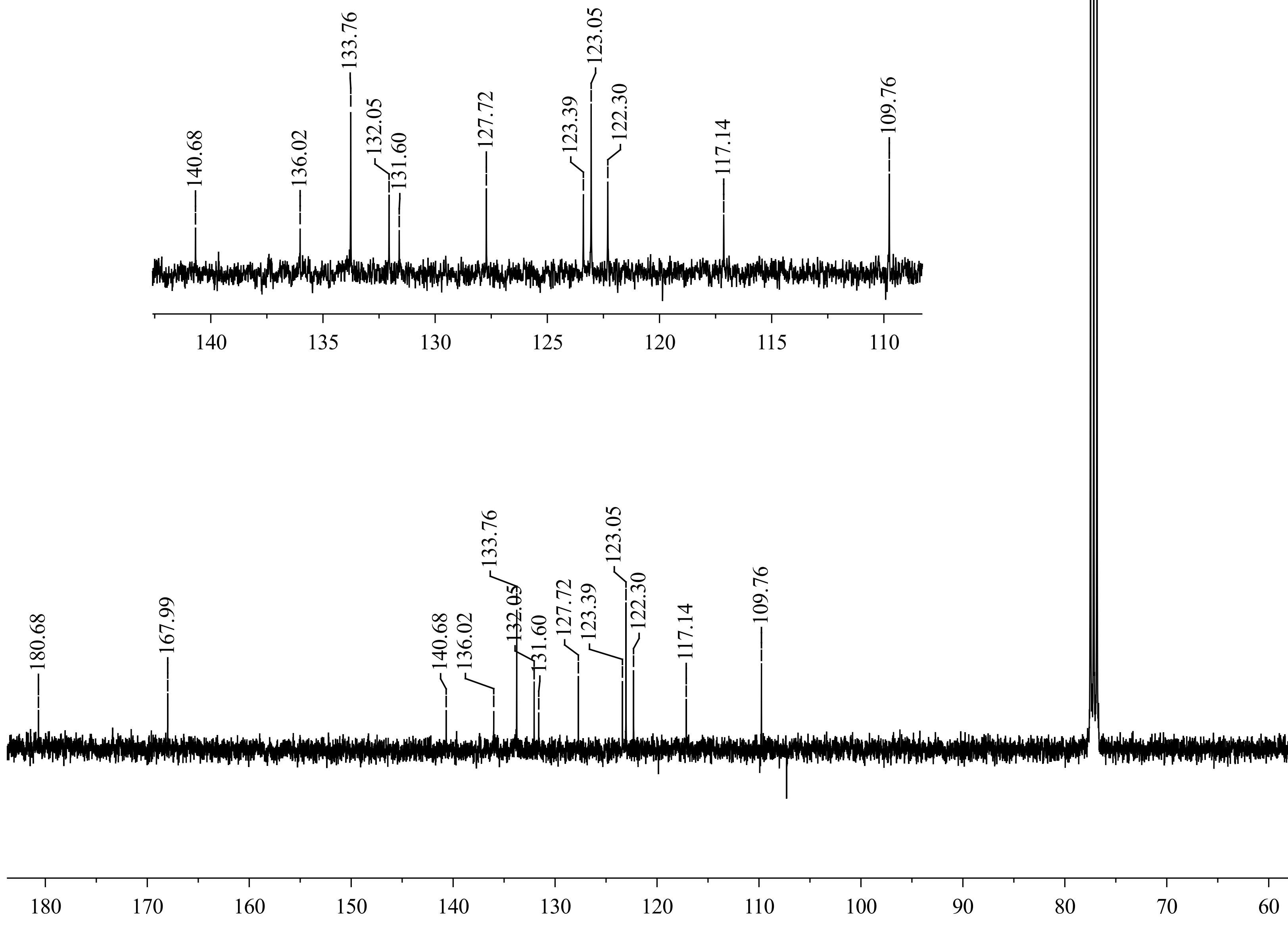

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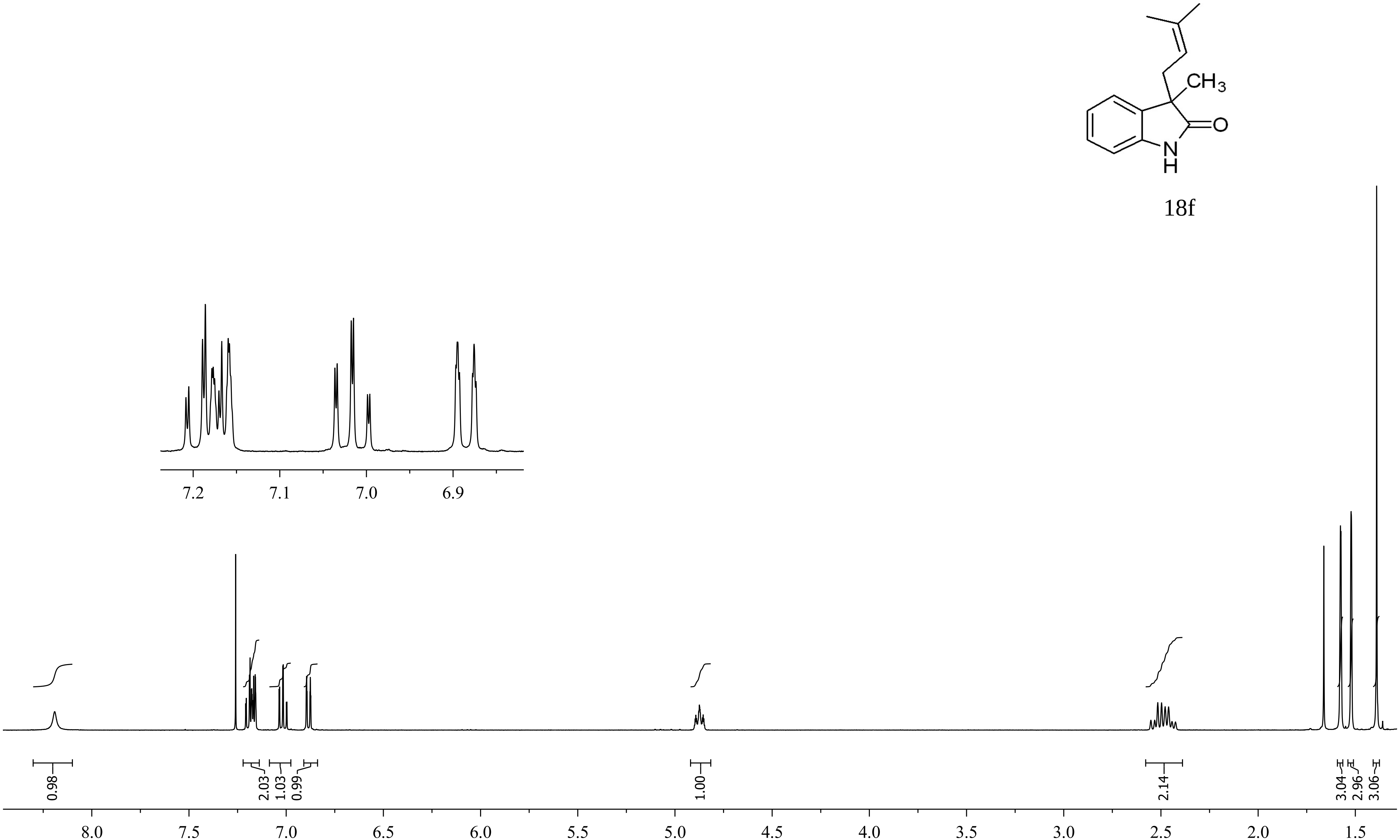

18f

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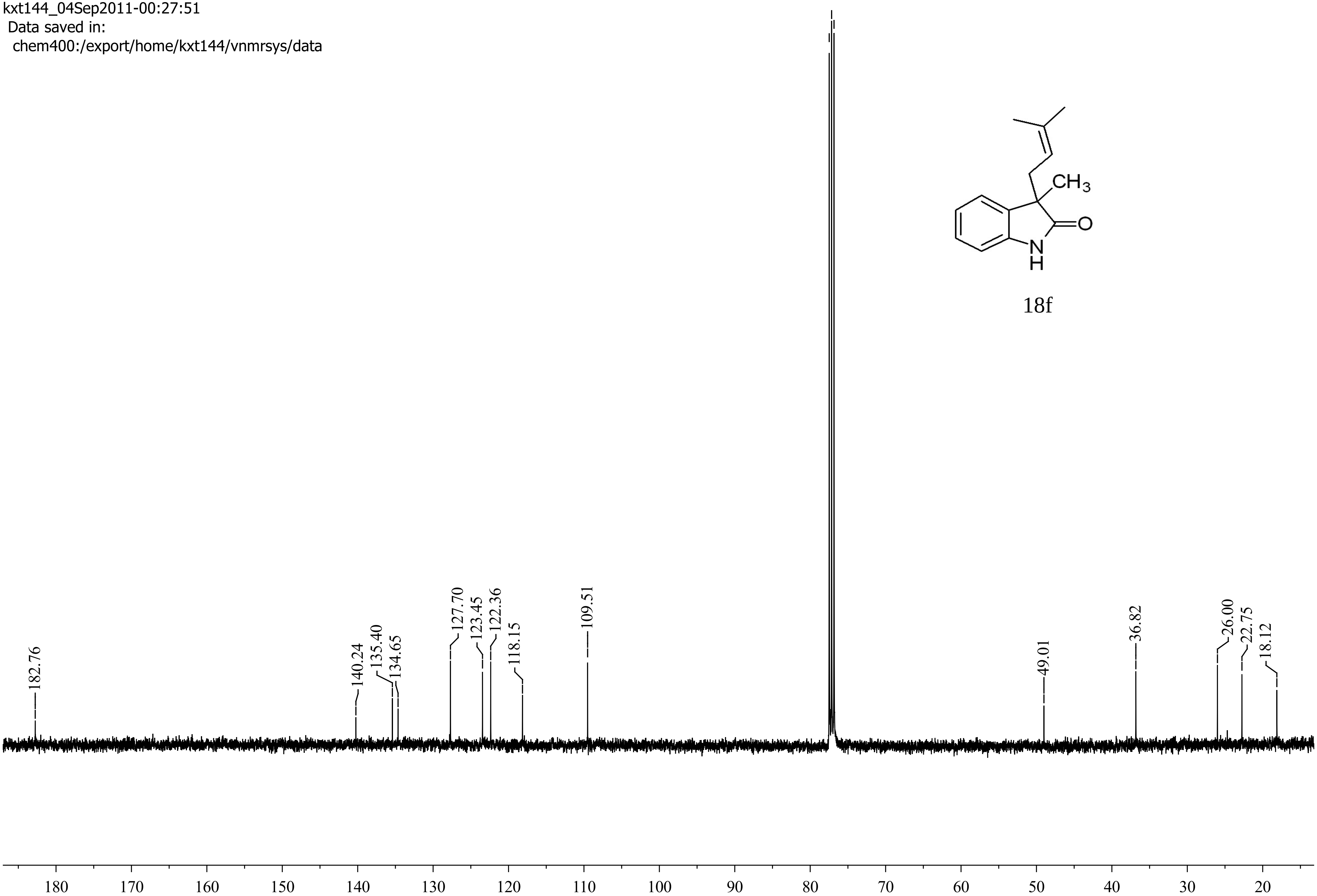

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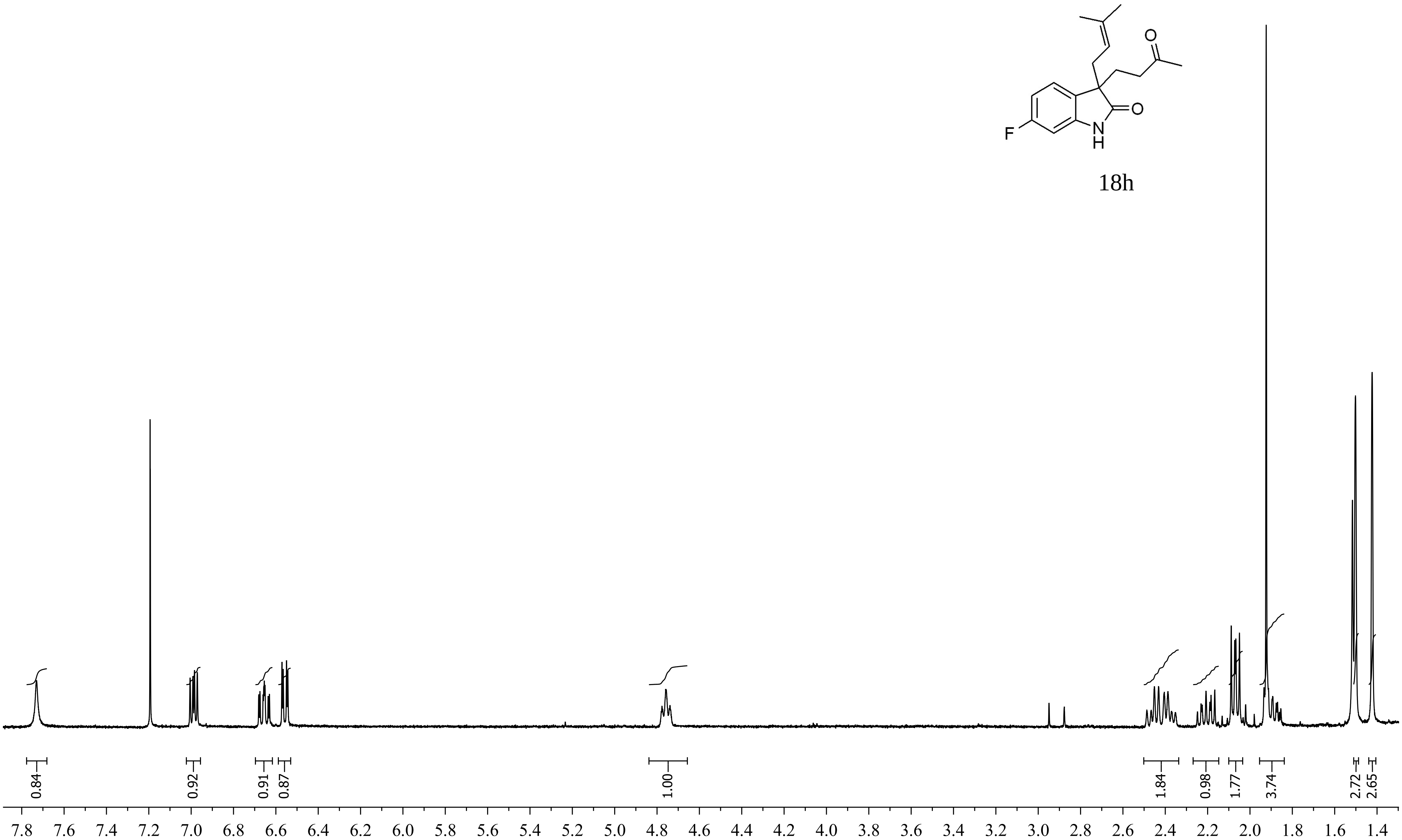

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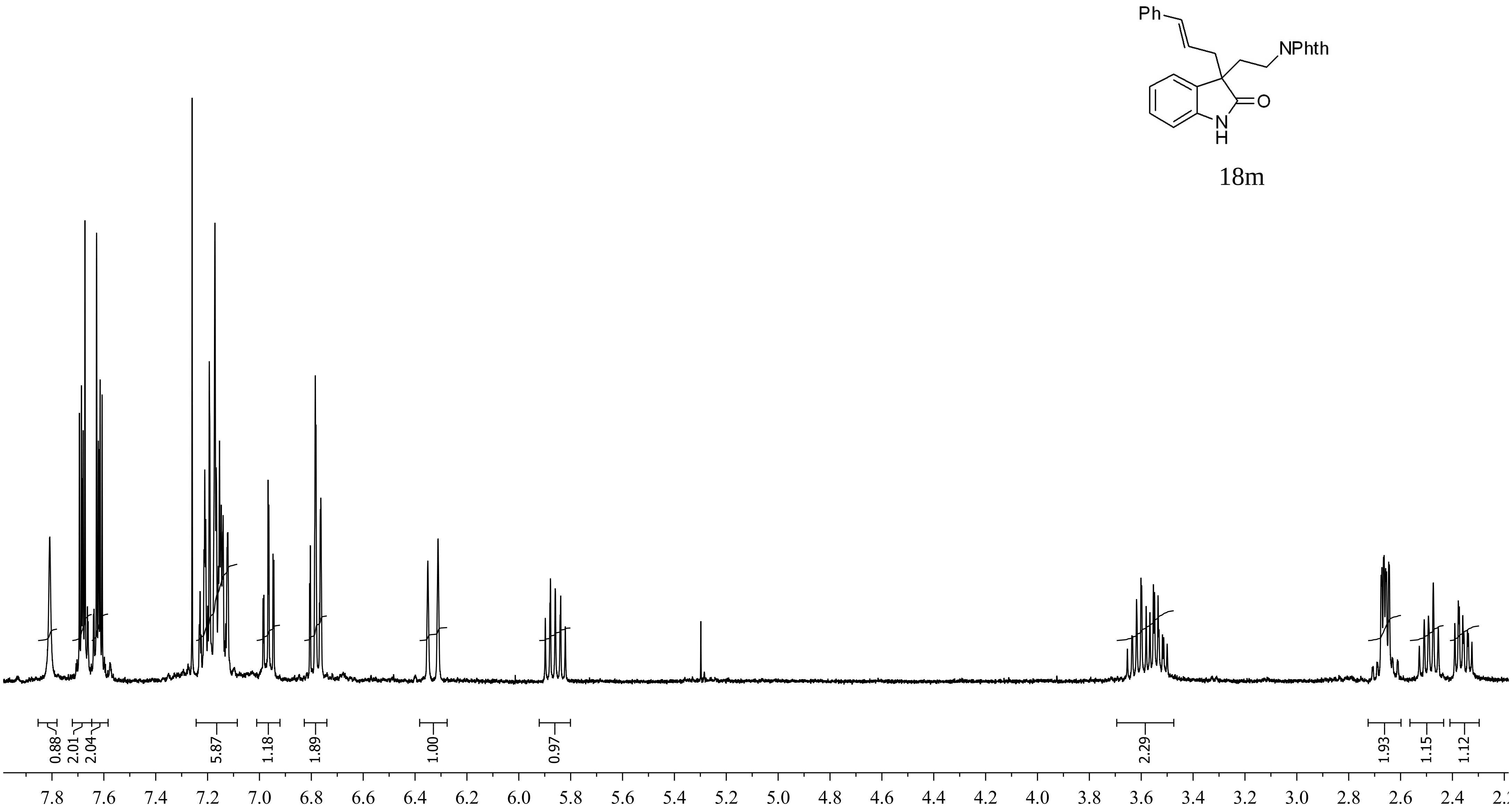

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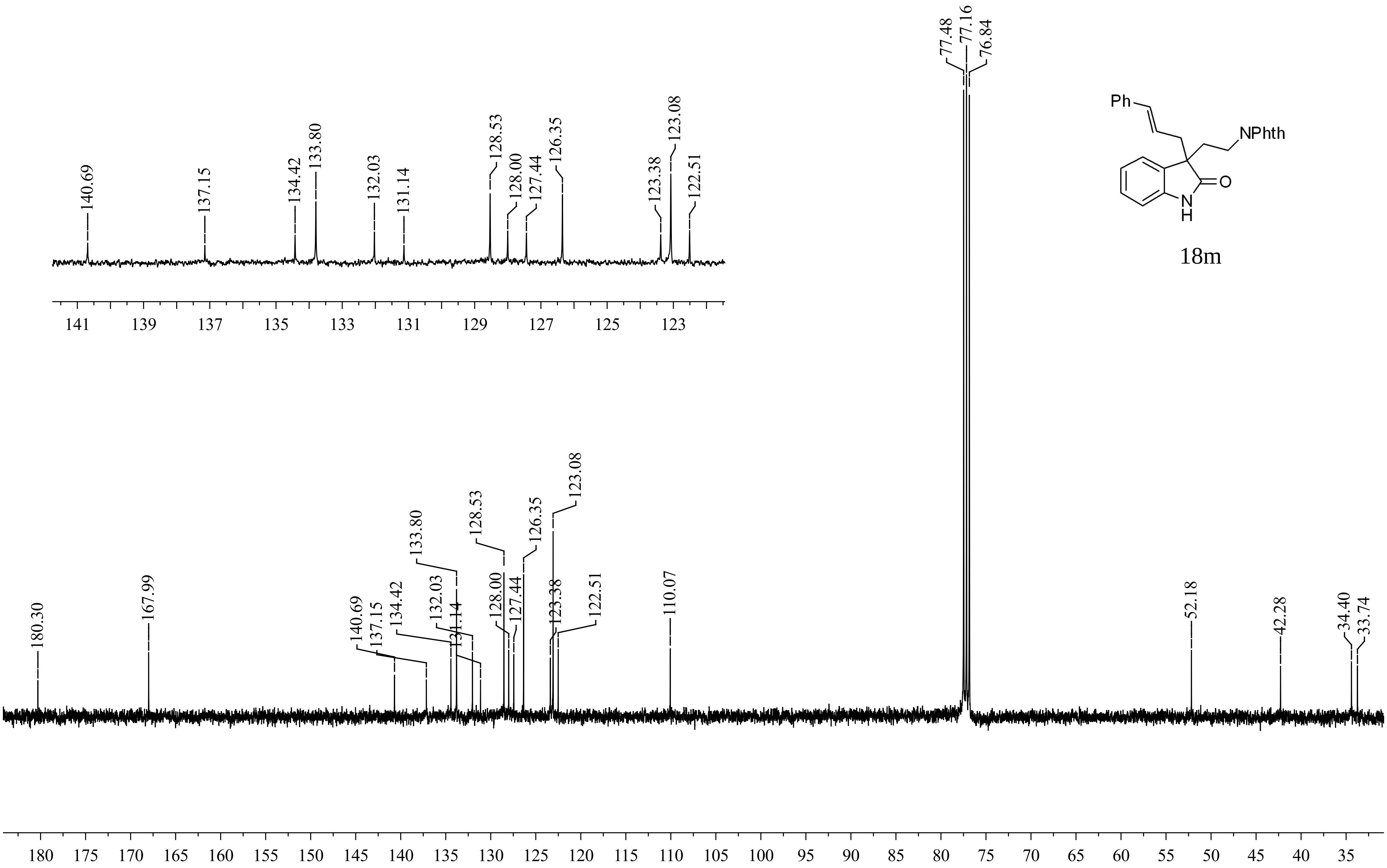

18m

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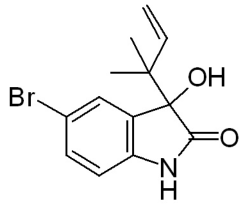
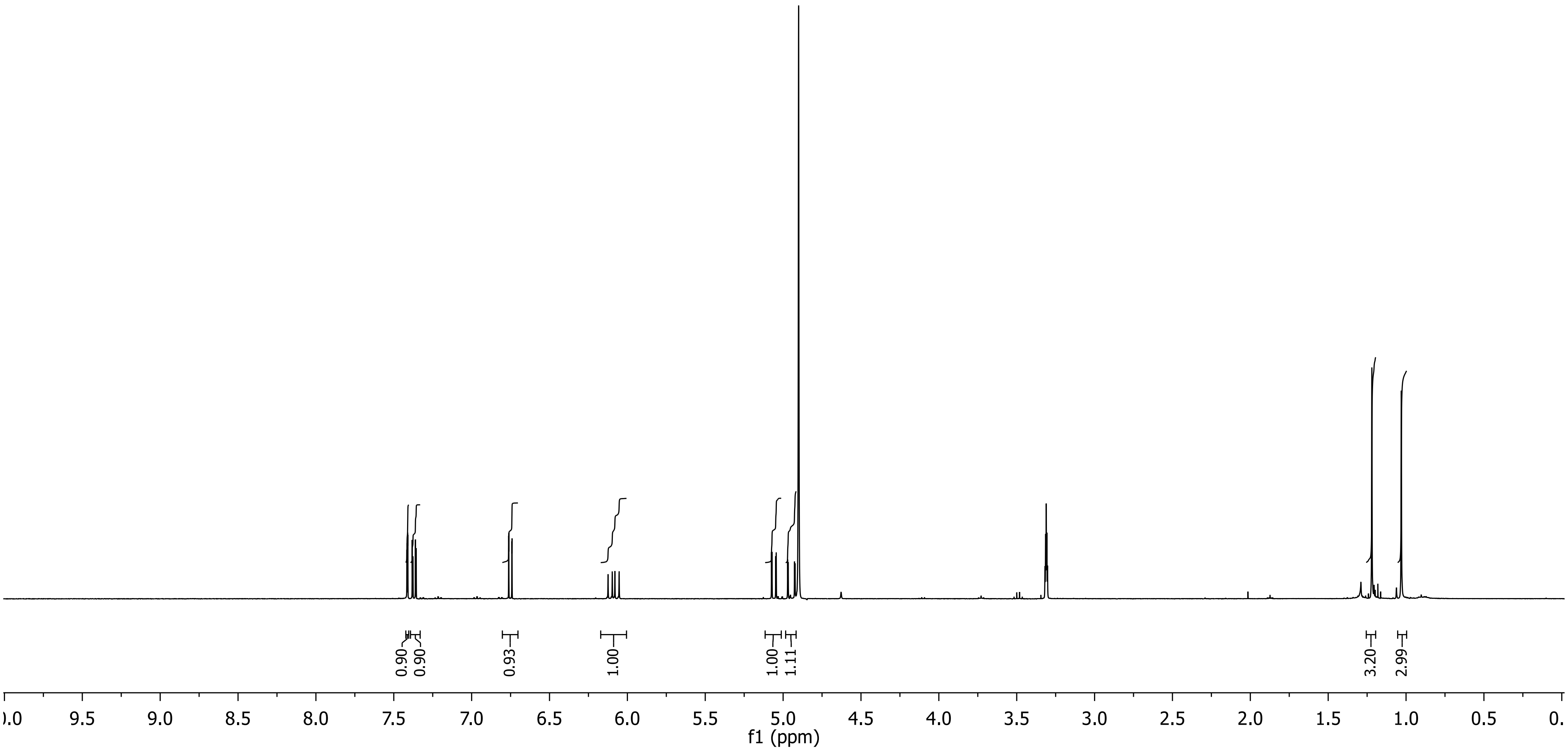
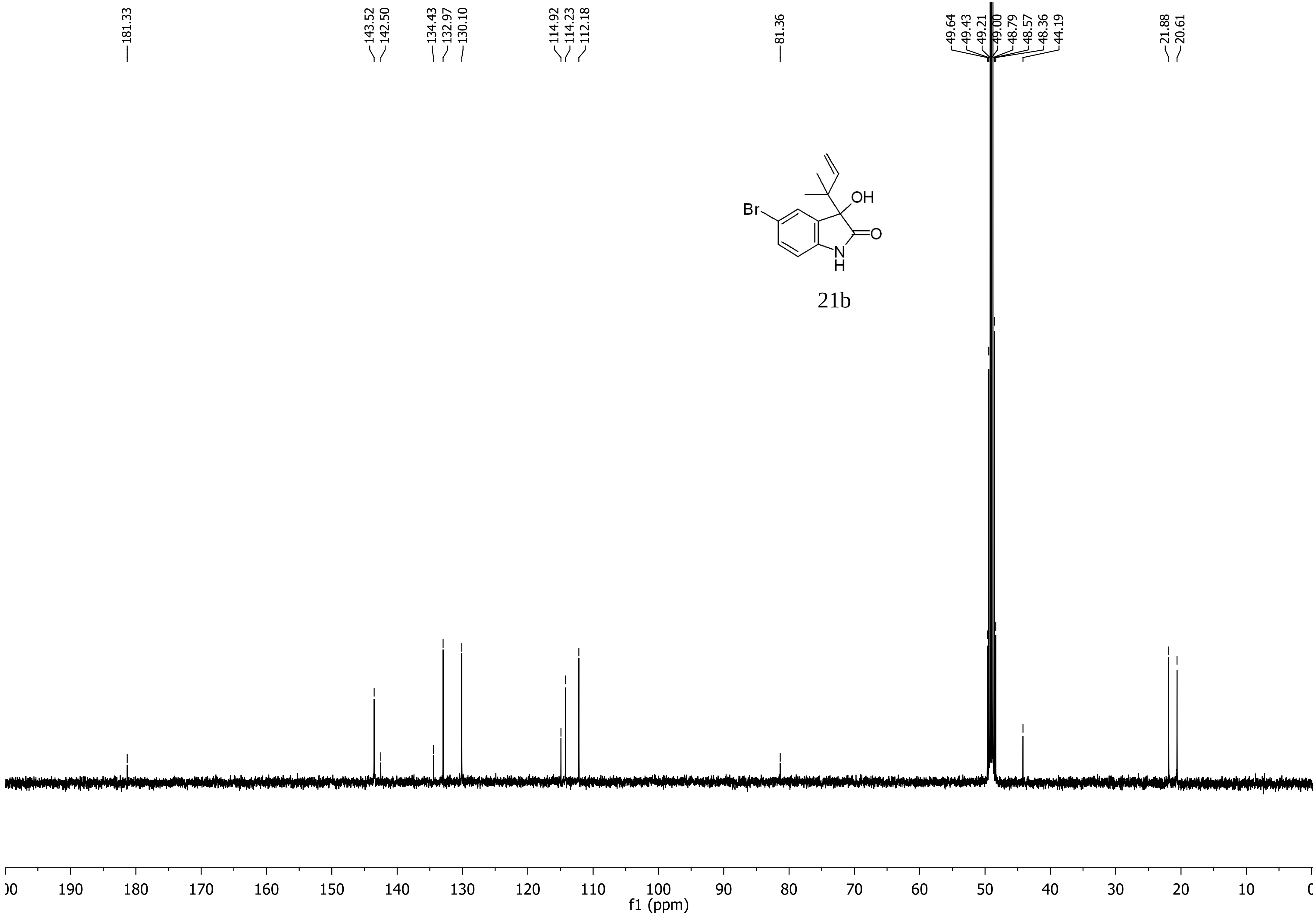


21b

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143.52
142.50
134.43
132.97
130.10
114.92
114.23
112.18
81.36
49.64
49.43
49.21
49.00
48.79
48.57
48.36
44.19
21.88
20.61
Br
OH
O
N
H
21b
f1 (ppm)

PROTON
kxt144_18Aug2011
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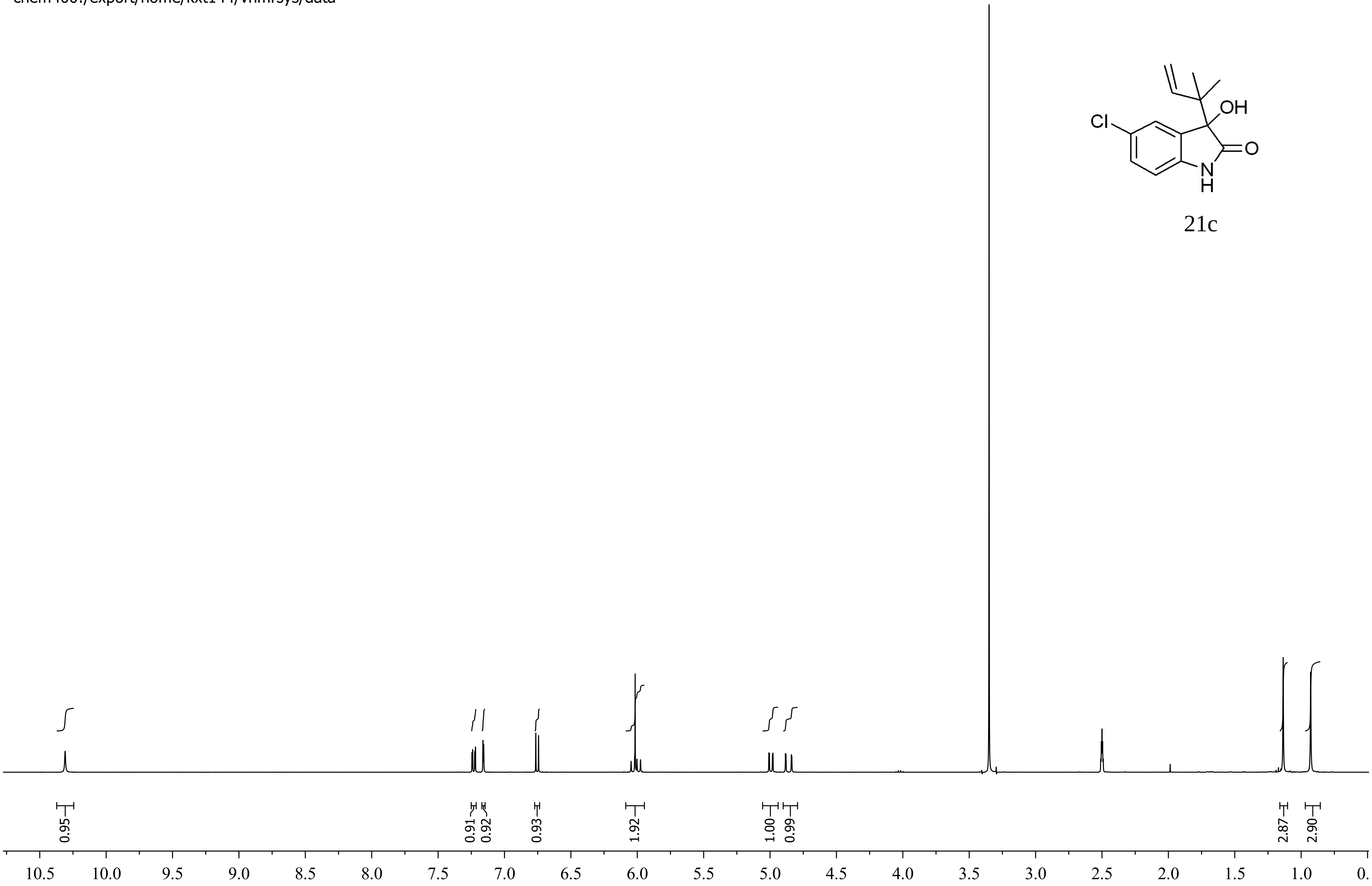

21c

CARBON
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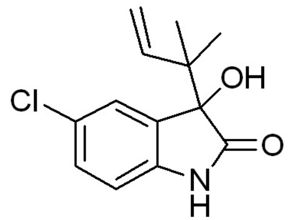
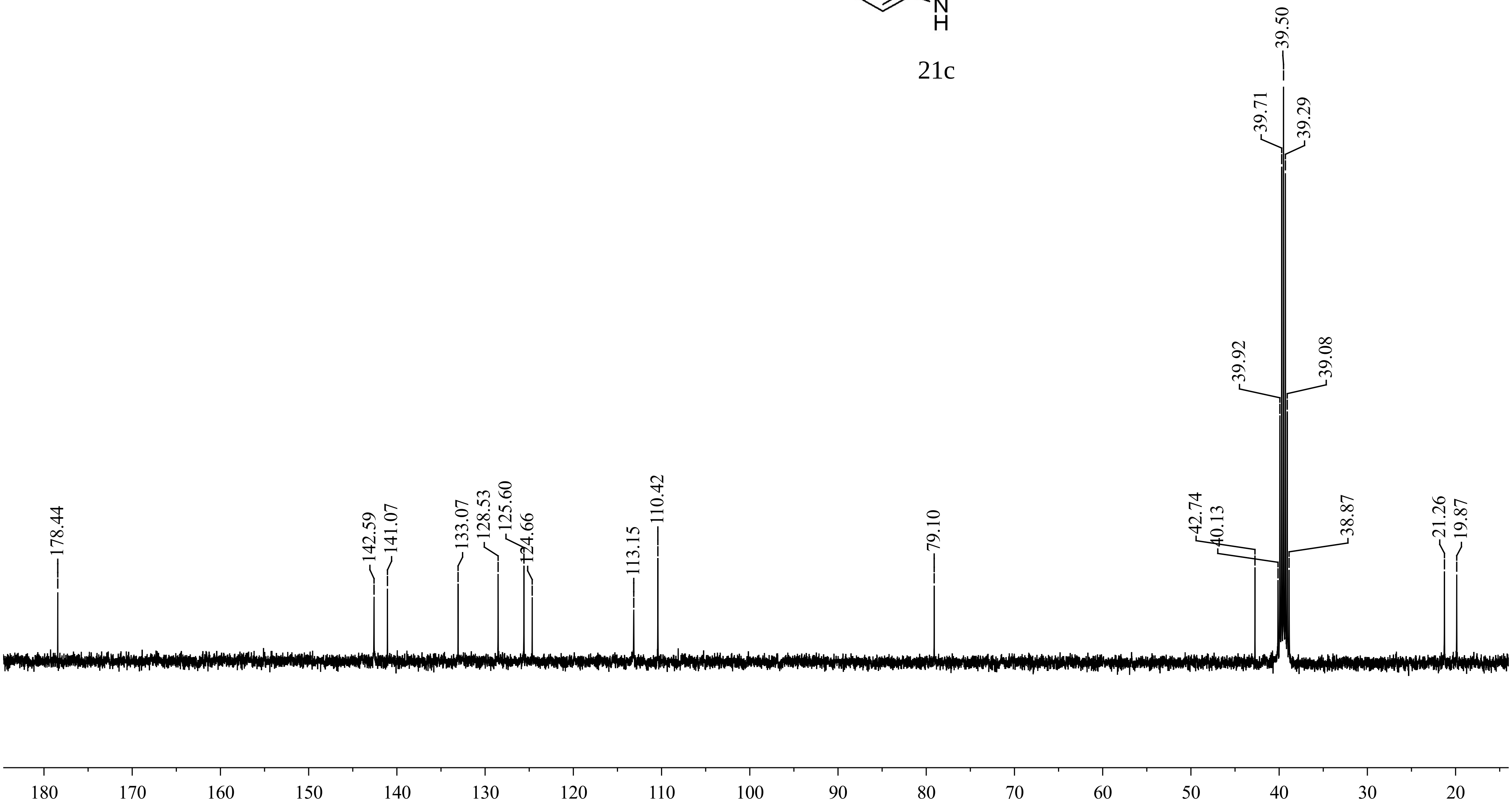


21c



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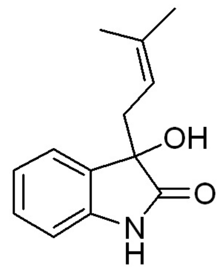

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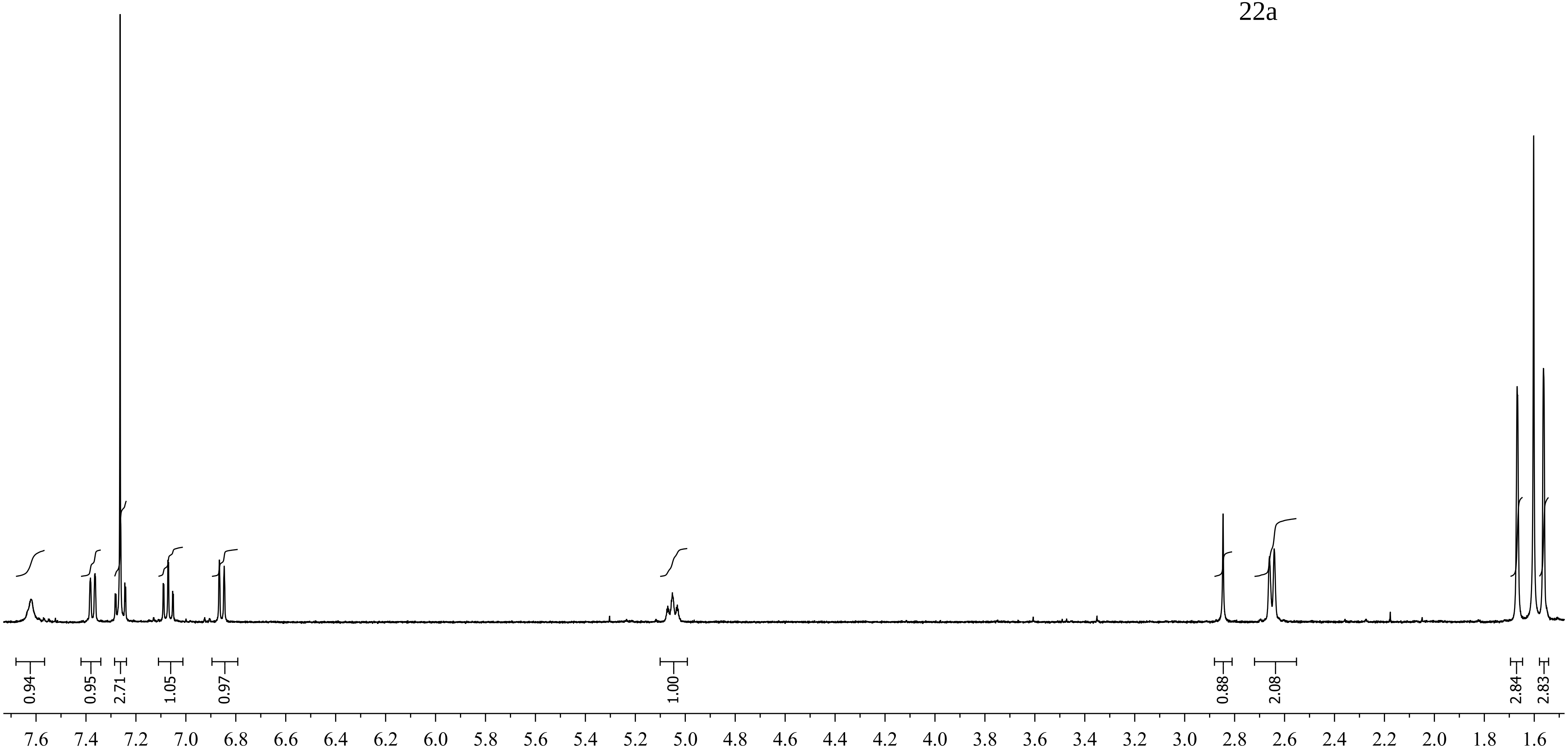
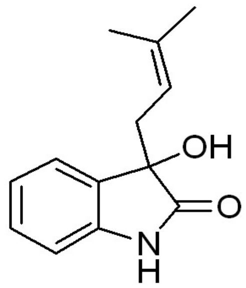
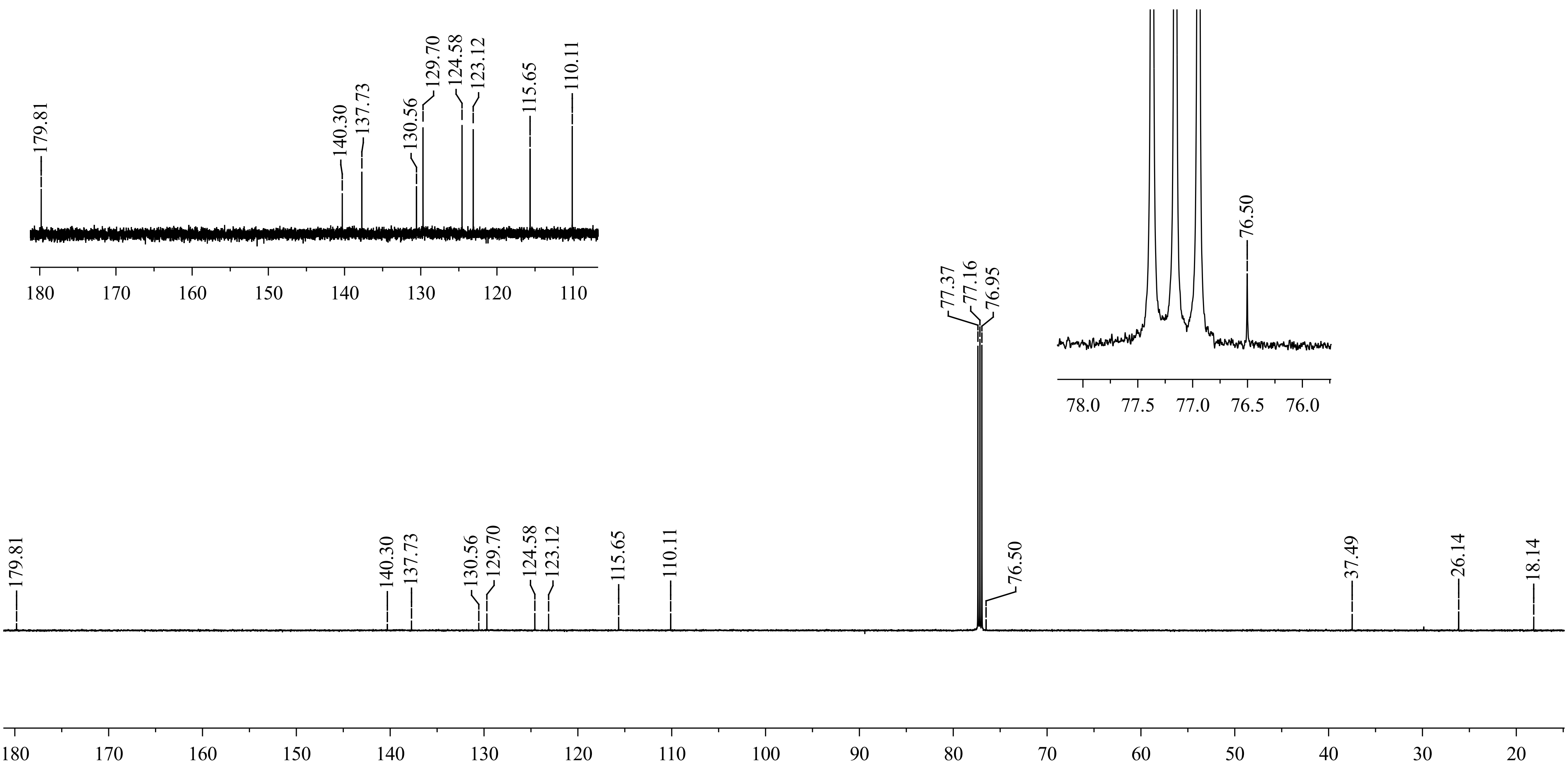


Kar_02-37
Karthik
02-37



22a



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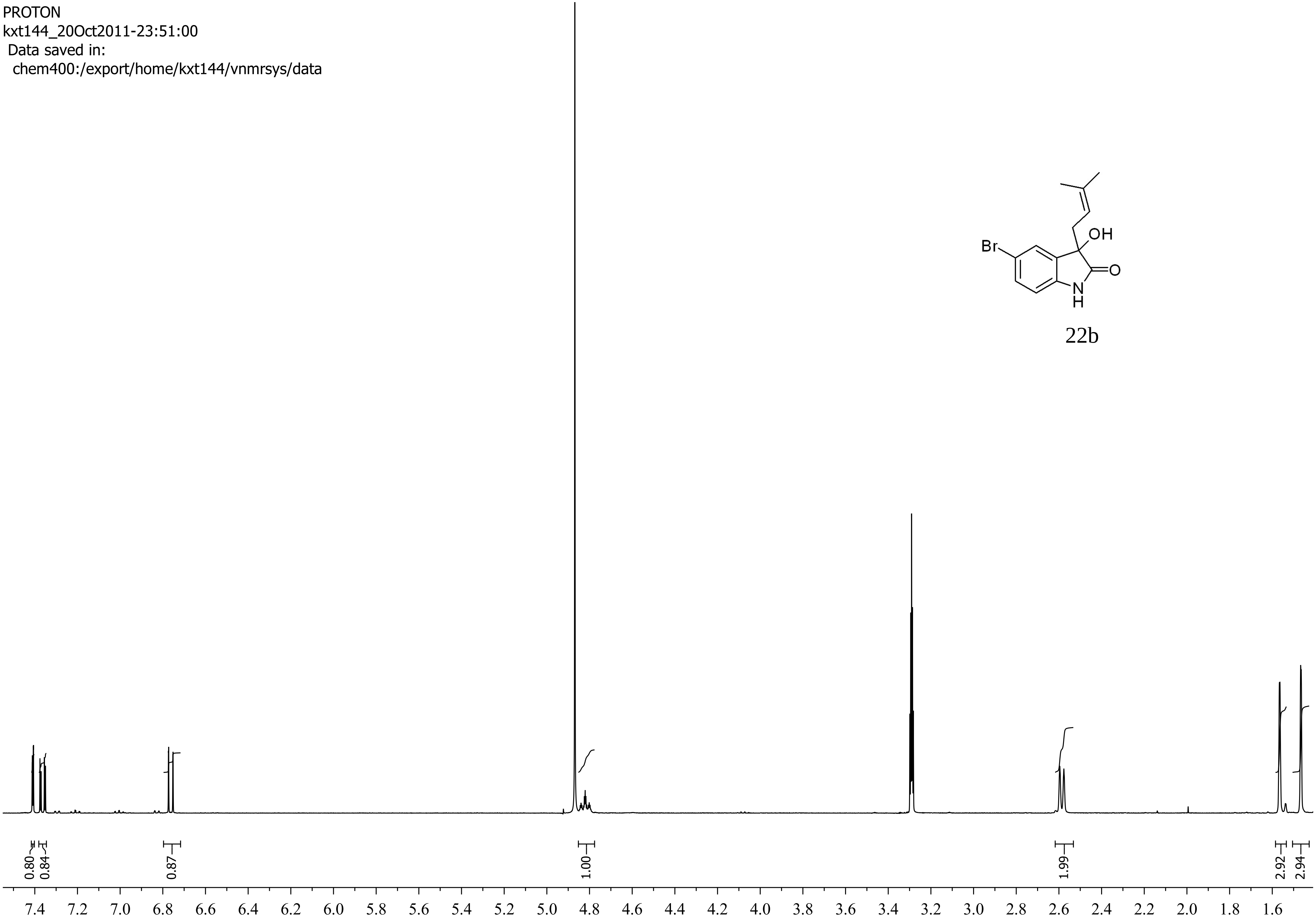

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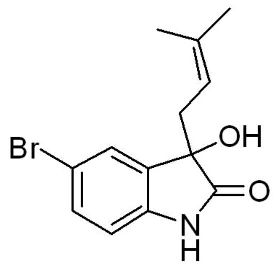
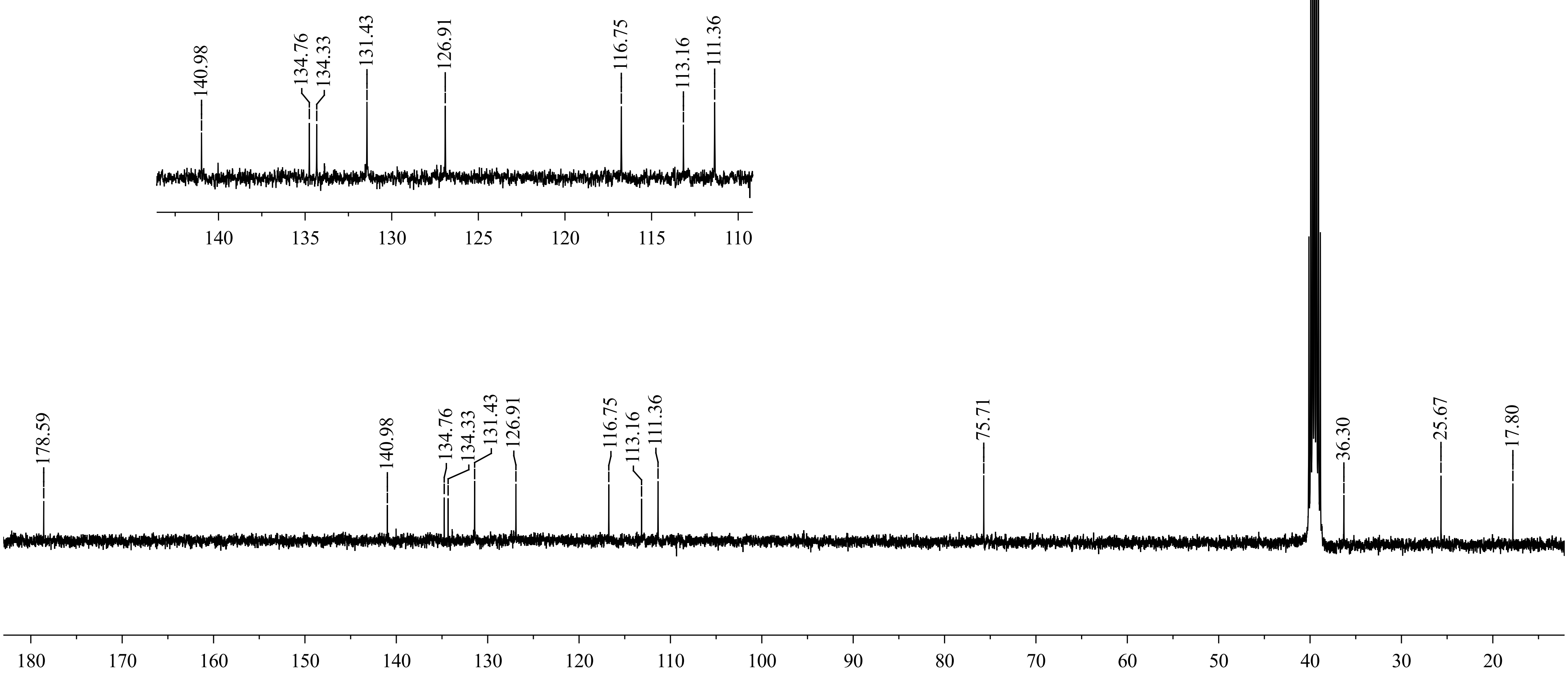


22b



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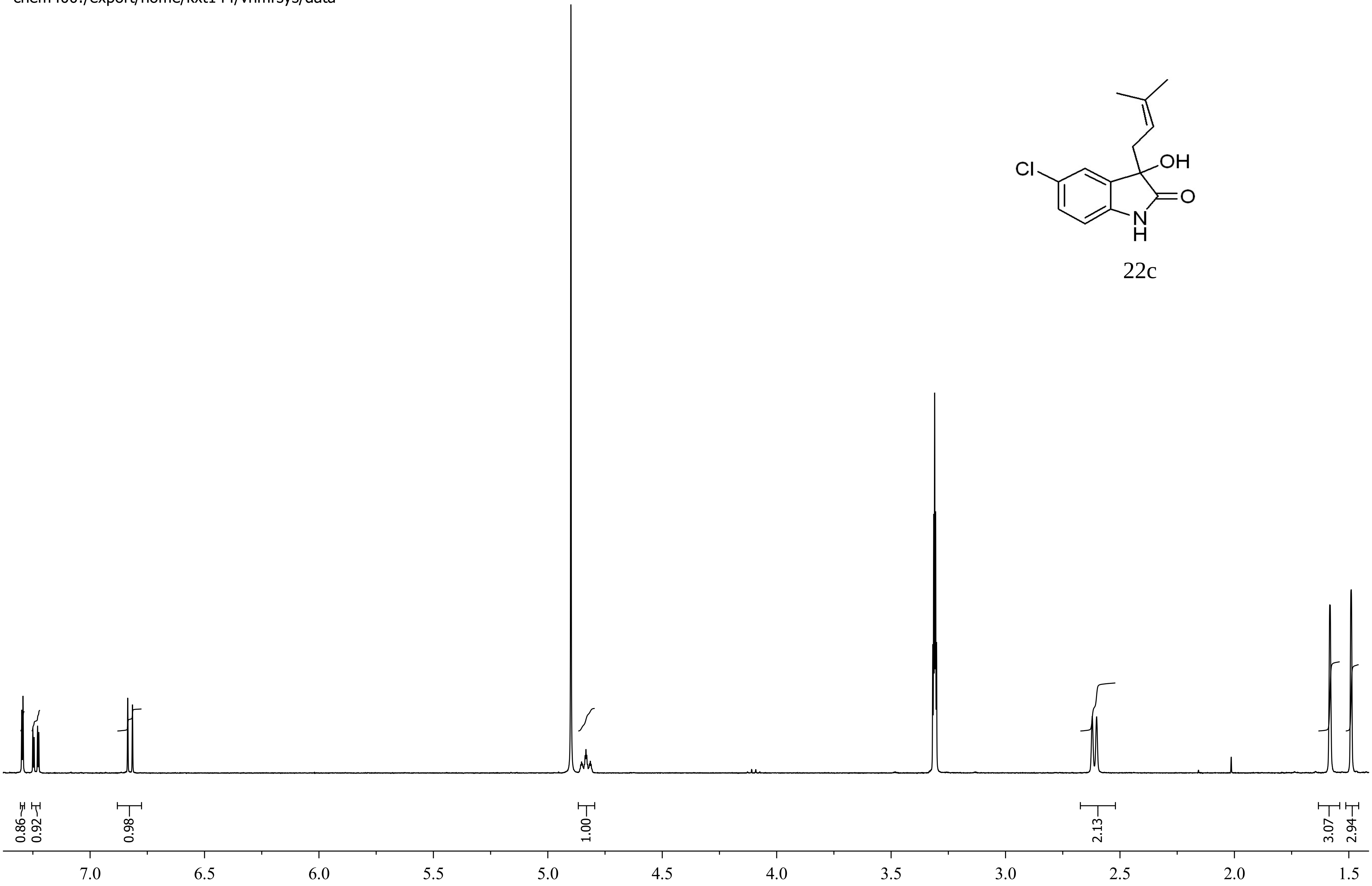

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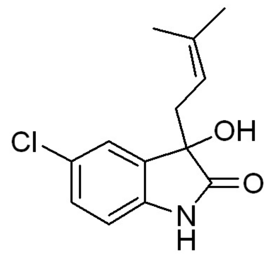
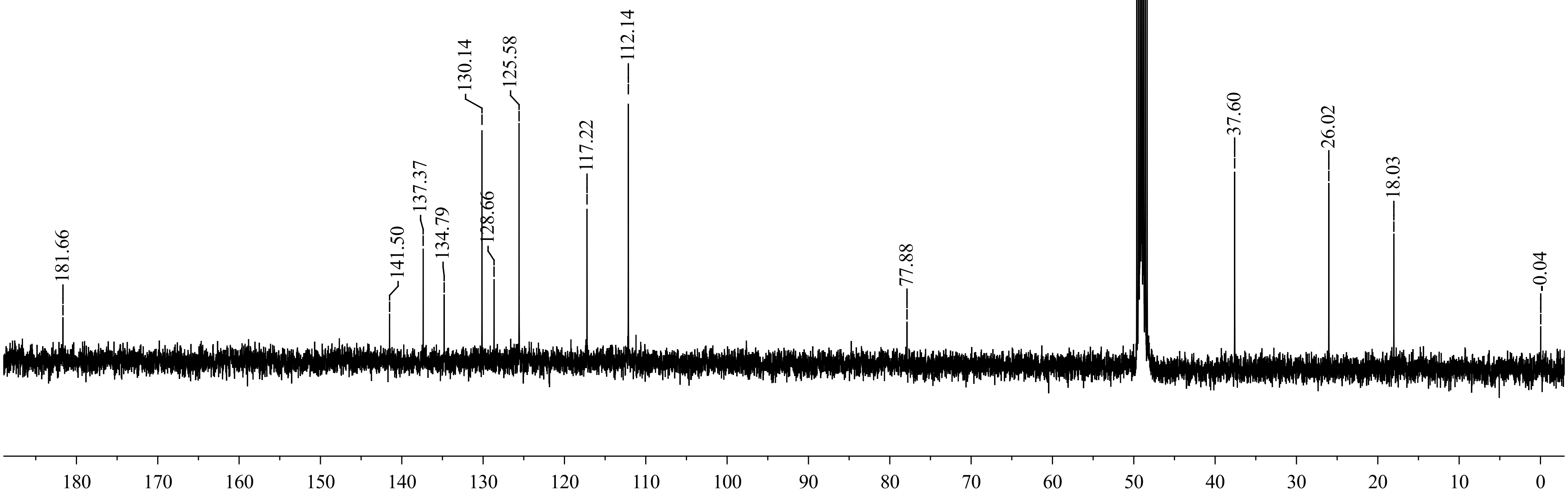
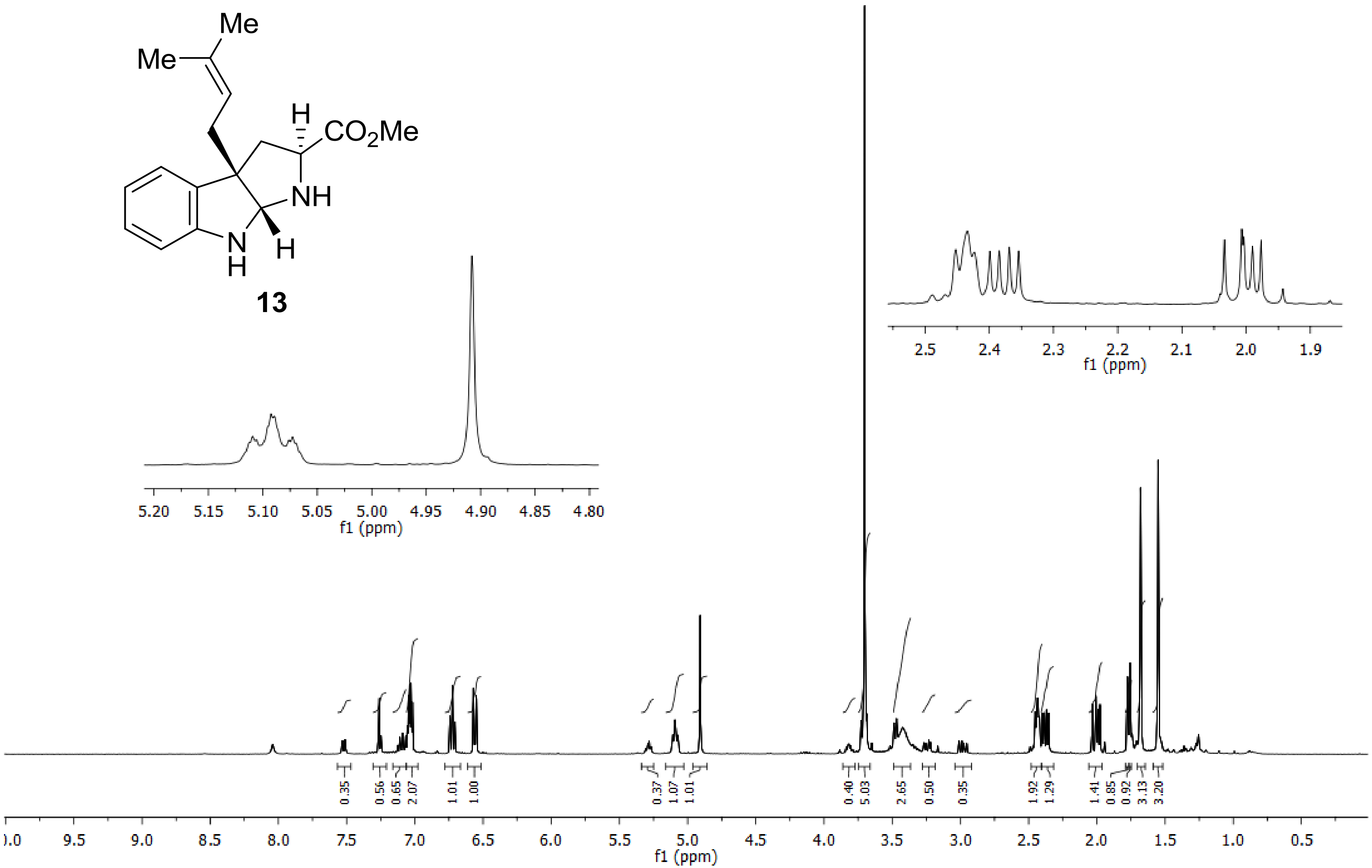
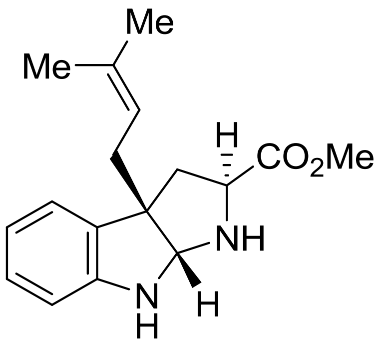


22c



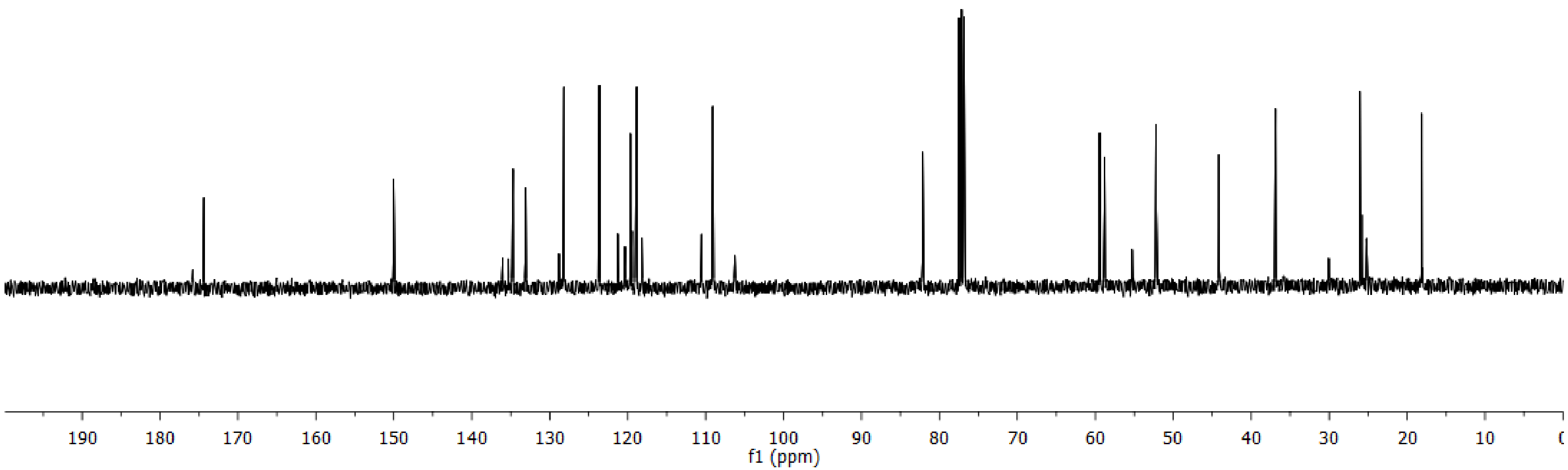


Me
Me
H
CO_2Me
NH
N
H
H
13
5.20
5.15
5.10
5.05
5.00
4.95
4.90
4.85
4.80
f1 (ppm)
2.5
2.4
2.3
2.2
2.1
2.0
1.9
f1 (ppm)
0.35
0.56
0.65
2.07
1.01
1.00
0.37
1.07
1.01
0.40
5.03
2.65
0.50
0.35
1.92
1.29
1.41
0.85
0.92
3.13
3.20
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
f1 (ppm)

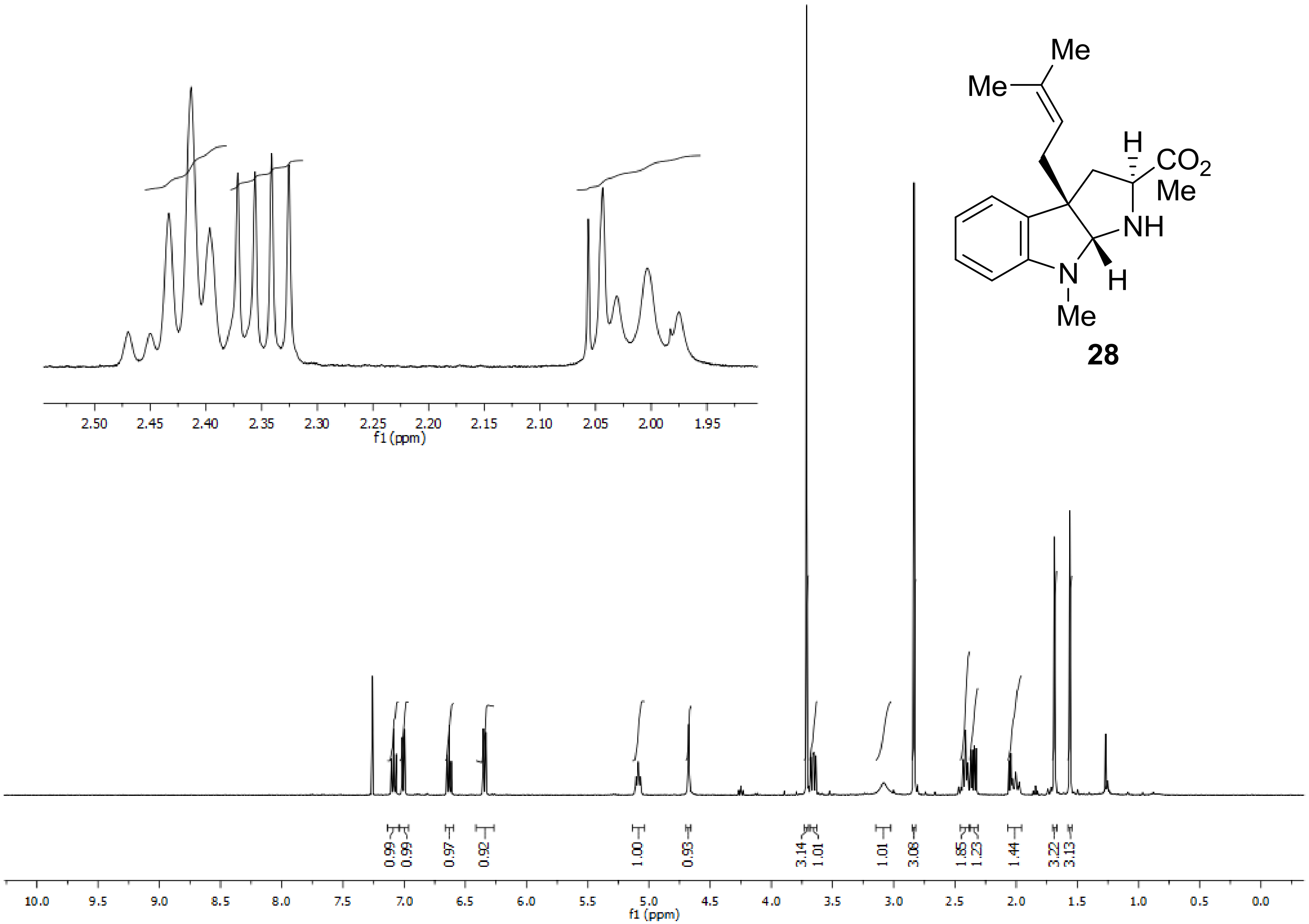


Me
Me
H
CO2Me
NH
N
H
H

13

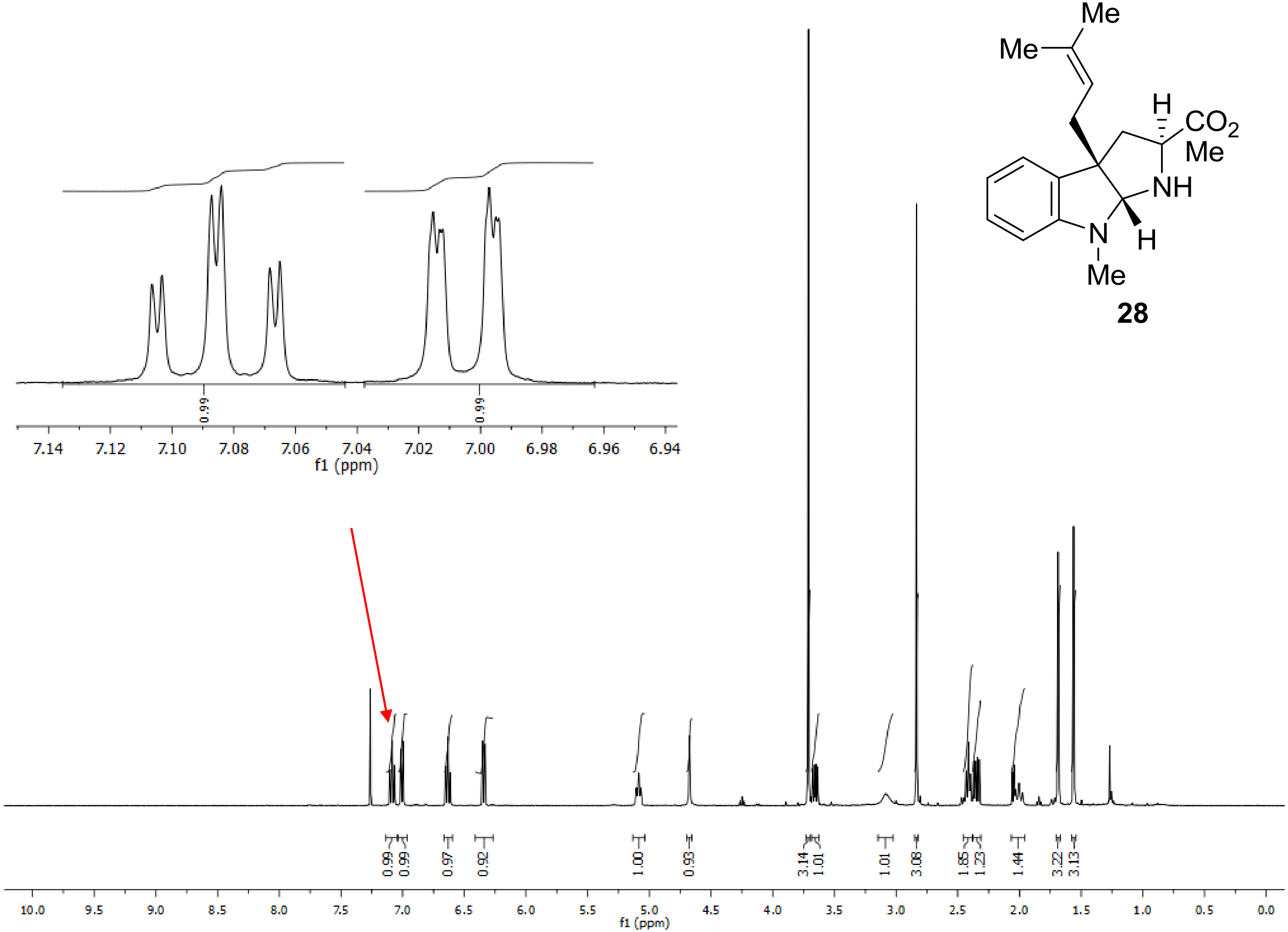


190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
f1 (ppm)



2.50
2.45
2.40
2.35
2.30
2.25
2.20
2.15
2.10
2.05
2.00
1.95
f1 (ppm)
0.99
0.99
0.97
0.92
1.00
0.93
3.14
1.01
1.01
3.08
1.85
1.23
1.44
3.22
3.13
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)
Me
Me
H
CO_2
Me
NH
N
H
Me

28



7.14
7.12
7.10
7.08
7.06
7.04
7.02
7.00
6.98
6.96
6.94
f1 (ppm)
0.99
0.99
Me
Me
H
CO2
Me
NH
N
H
Me
0.99
0.99
0.97
0.92
1.00
0.93
3.14
1.01
1.01
3.08
1.85
1.23
1.44
3.22
3.13
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
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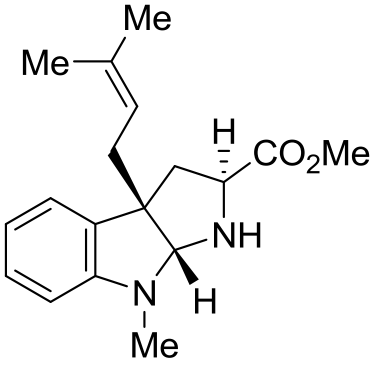
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

28



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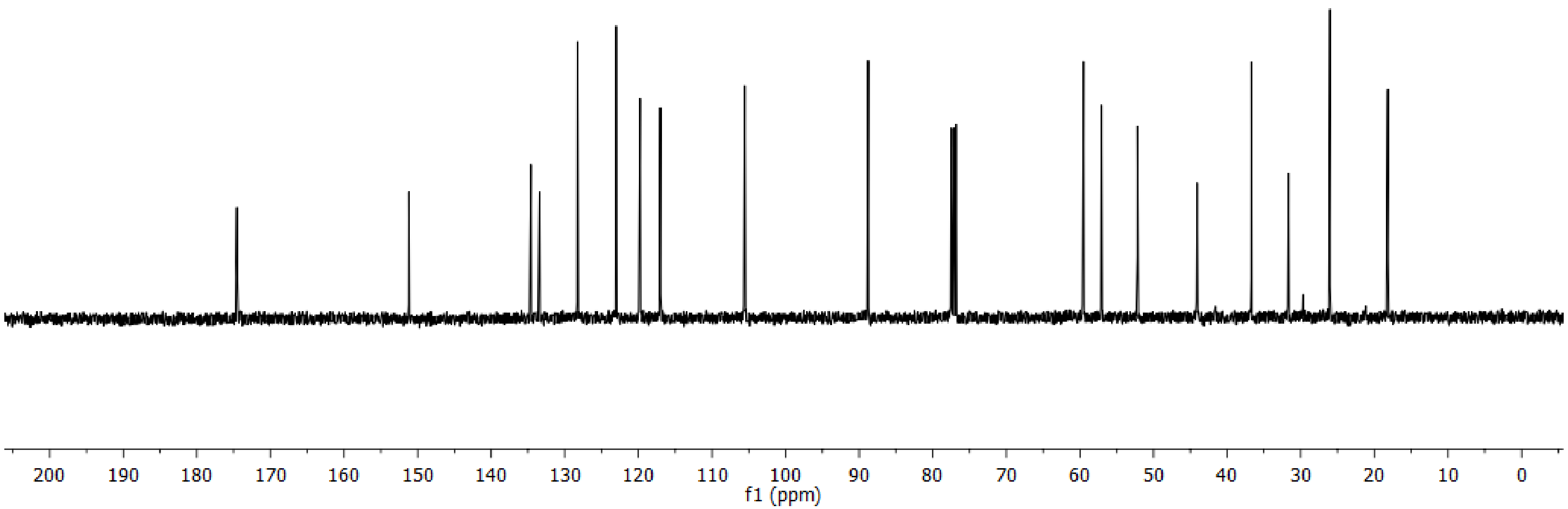
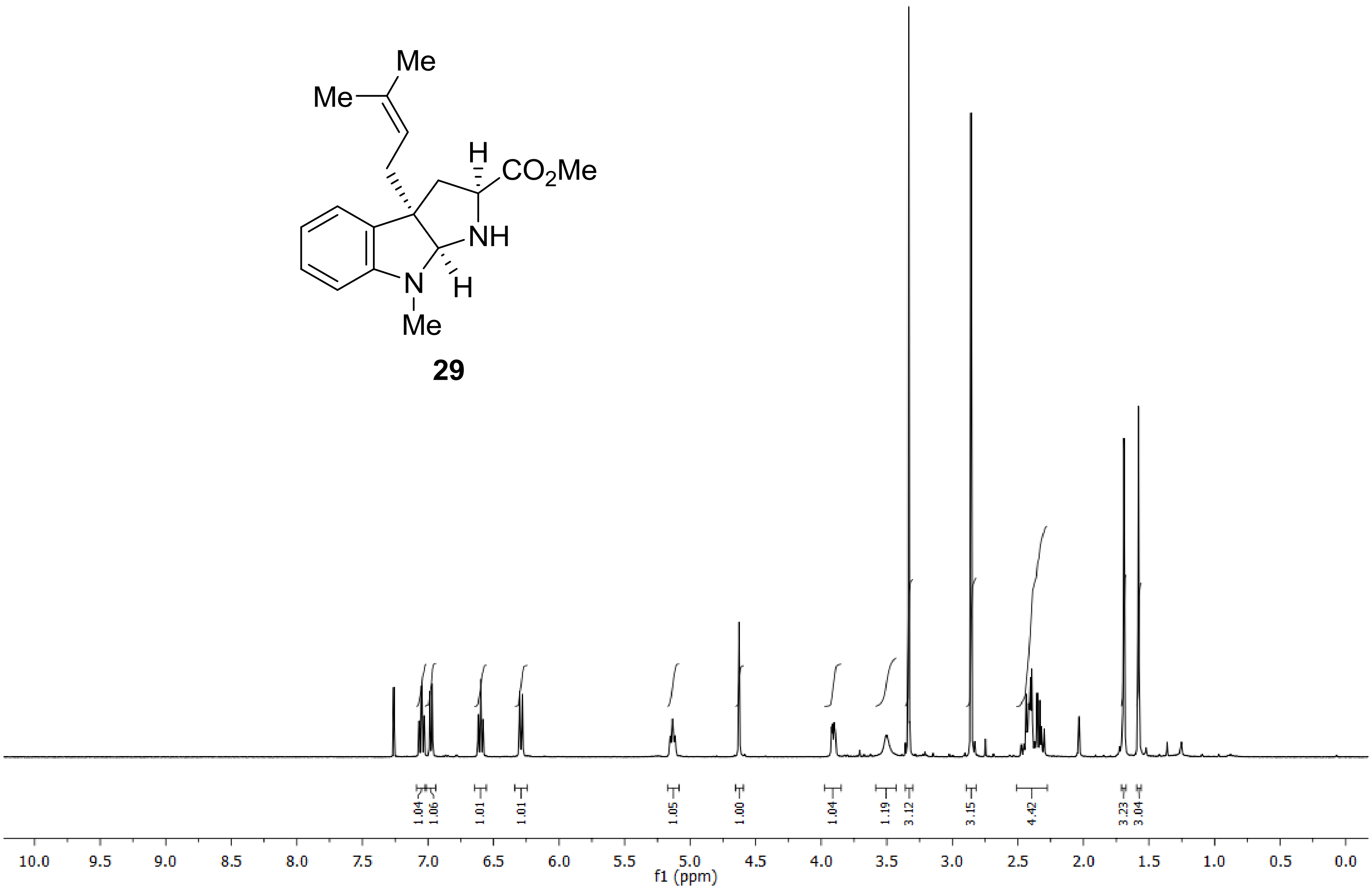
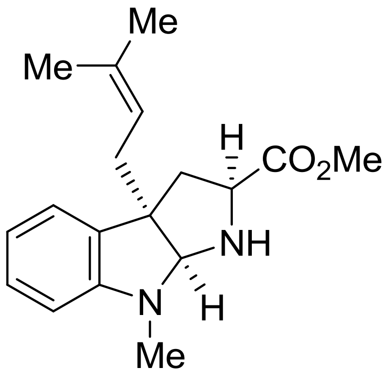


200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

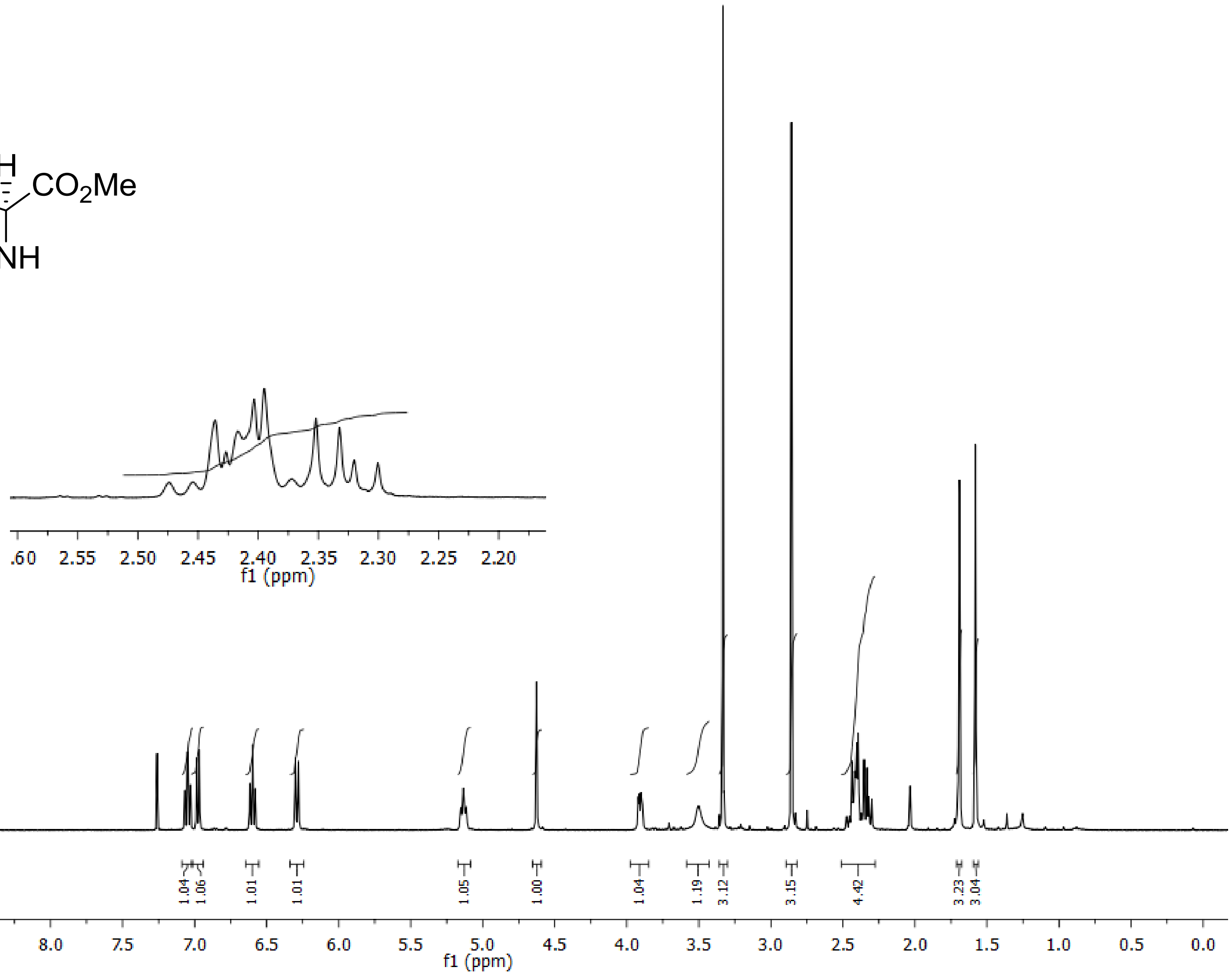

Me
Me
H
CO2Me
NH
N
H
Me

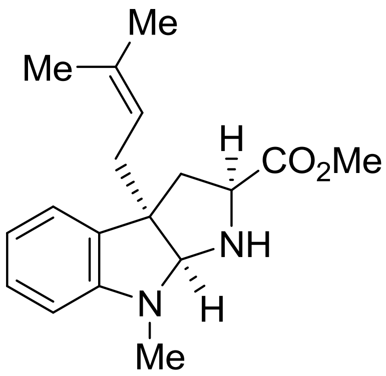
29

1.04
1.06
1.01
1.01
1.05
1.00
1.04
1.19
3.12
3.15
4.42
3.23
3.04
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)


Me
Me
H
CO2Me
NH
N
H
Me

29


.60
2.55
2.50
2.45
2.40
2.35
2.30
2.25
2.20
f1 (ppm)
1.04
1.06
1.01
1.01
1.05
1.00
1.04
1.19
3.12
3.15
4.42
3.23
3.04
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)



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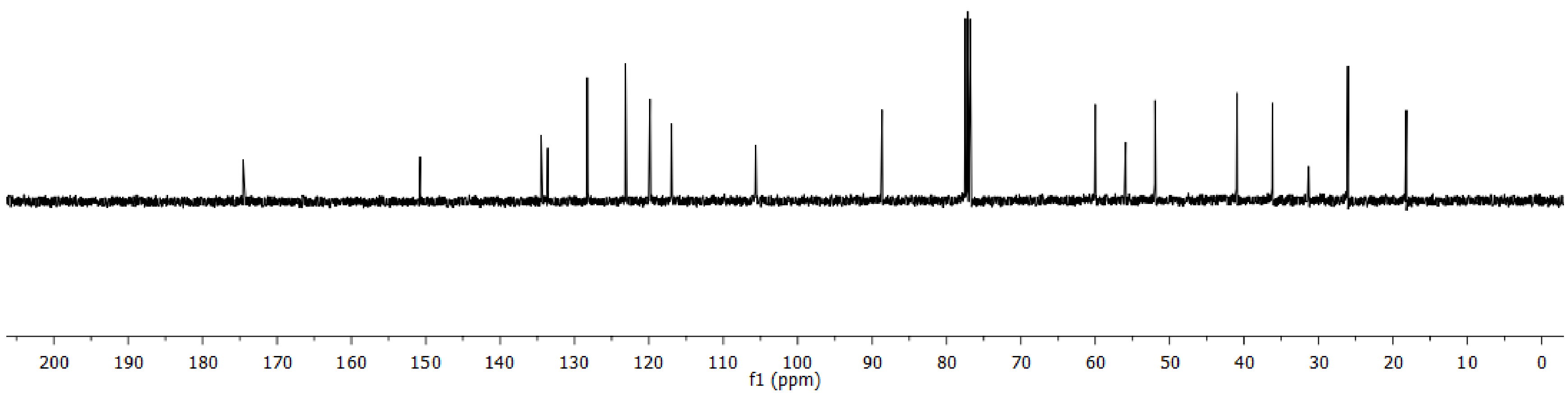
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)