Electronic Supplementary Material (ESI) for Chemical Science.

Supporting Information

Enantioselective Radical Process for Synthesis of Chiral Indolines by Co(II)-Based Metalloradical Alkylation of Diverse C(sp^3)–H Bonds

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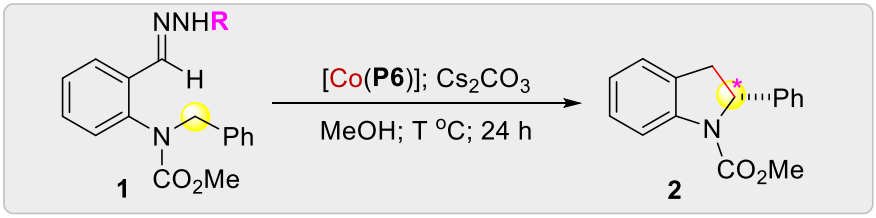
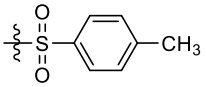
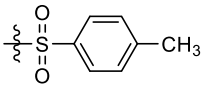
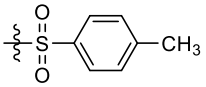
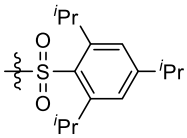
I. General Considerations

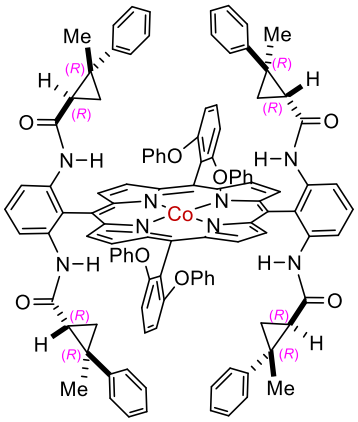
^{1}H NMR spectra were recorded on a Varian INOVA 400 (400 MHz), 500 (500 MHz) or a 600 (600 MHz) spectrometer. Chemical shifts are internally referenced to residual $CHCl_3$ signal (δ 7.26 ppm). Data are reported as follows: chemical shift, integration, multiplicity (s = singlet, d = doublet, t = triplet, q = quartet, p = pentet, hept = heptet, br = broad, m = multiplet), and coupling constants (Hz). ^{13}C NMR spectra were recorded on a Varian INOVA 500 (125 MHz), or 600 (150 MHz) spectrometers with complete proton decoupling. Chemical shifts are reported in ppm with residual $CHCl_3$ as the internal standard (δ 77.0 ppm). High-resolution mass spectrometry was performed on a Micromass LCT ESI-MS and JEOL Accu TOF Dart at the Mass Spectrometry Facility, Boston College. The UV-Vis absorption spectra in the range 200-700 nm were measured with an Evolution 300 UV-VIS spectrophotometer using quartz cuvettes with 1.0 cm optical path length. HPLC measurements were carried out on a Shimadzu HPLC system with ChiralPak Immobilized columns: IA, IB and IC. Infrared (IR) spectra were recorded on a Termo Scientific Nicolet Is5 System. Frequencies are reported in wavenumbers (cm^{-1}). HRMS data was obtained on an Agilent 6210 Time-of-Flight LC/MS with ESI as the ion source. Optical rotations were measured on a Rudolph Research Analytical AUTOPOL® IV digital polarimeter. The X-ray diffraction data were collected using Bruker Kappa APEX DUO diffractometer and a Rigaku HighFlux Homelab diffractometer. X-band EPR spectra were recorded on a Bruker EMX-Plus spectrometer (Bruker BioSpin). Spartan modelling was performed using Spartan'14 software from Wavefunction, Inc.

Unless otherwise noted, all C–H alkylation reactions were performed in oven-dried glassware under dry N_2 atmosphere with standard vacuum line techniques. Gastight syringes were used to transfer liquid reagents and solvents in catalytic reactions. Anhydrous solvents as well as other commercial reagents were purchased from Sigma-Aldrich, Acros, Alfa Aesar, Strem, Oakwood Products Inc., TCI, or Matrix Scientific and used as received unless otherwise stated. Thin layer chromatography was performed on Merck TLC plates (silica gel 60 F254). Flash column chromatography was performed with ICN silica gel (60 Å, 230-400 mesh, 32-63 μm).

II. Table S1. The Effect of Sulfonyl Group on Co(II)-Catalyzed Enantioselective Radical C–H Alkylation of Aryldiazomethanes[a]



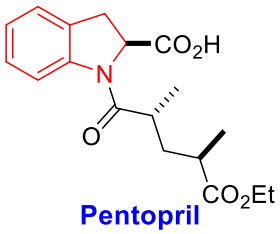
entry	R	T (°C)	yield	*ee*
1	 (Ts)	60	95%	89%
2	 (Ts)	40	92%	91%
3	 (Ts)	RT	47%	93%
4	 (TPS)	**RT**	**92%**	**94%**



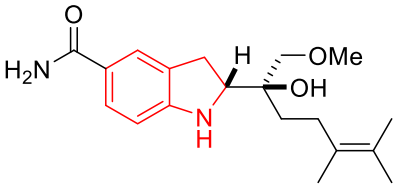
[a] Carried out with **1** (0.1 mmol) in the presence of Cs_2CO_3 (2.0 equiv.) by [Co(**P6**)] (2 mol %) in methanol (1.0 mL). Yield refers to isolated yield. *ee* was determined by chiral HPLC.

III. Figures S1-S4

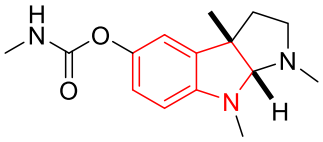
Figure S1. Examples of natural products and biologically active compounds containing indoline moiety



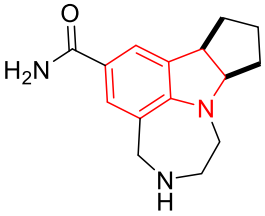
Pentopril



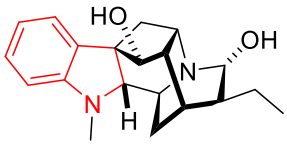
Benzastatin E



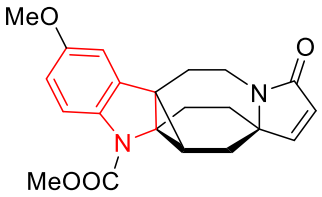
Physostigmine



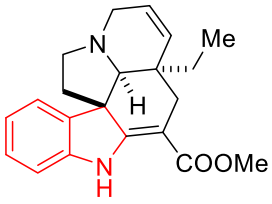
WAY-163909



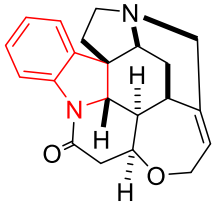
Ajmaline



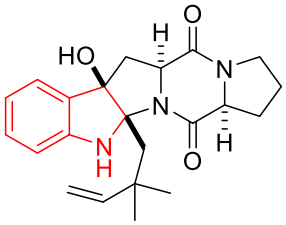
Lundurine A



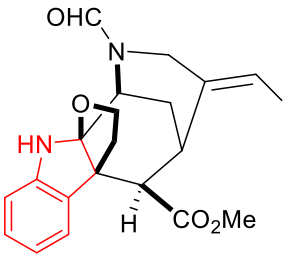
tabersonine



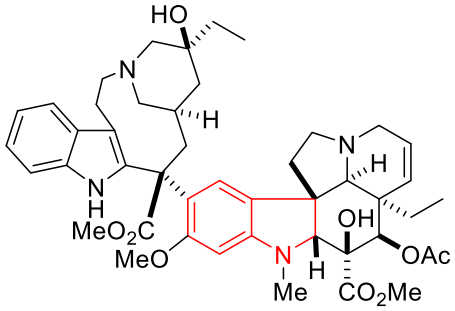
Strychnine



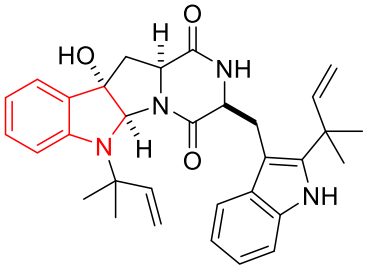
Brebianamide E



Aspidophylline A

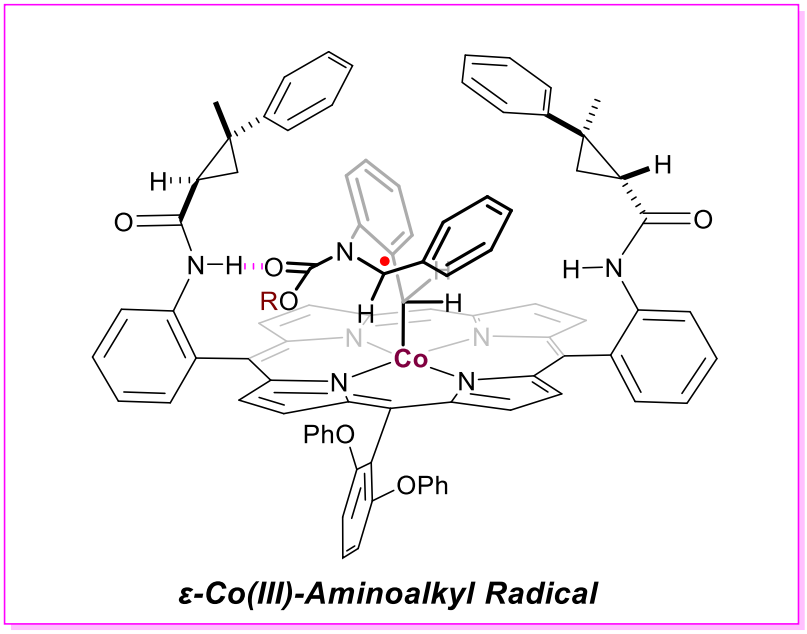


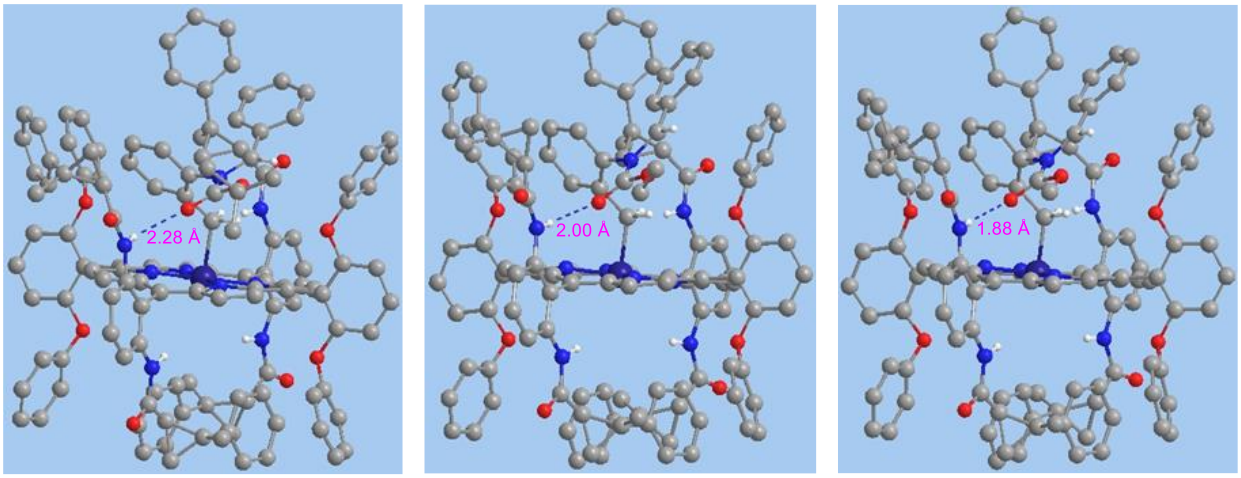
Vinblastine



Okaramine C

Figure S2. Molecular modeling of proposed ε-Co(III)-aminoalkyl radical intermediates showing potential hydrogen-bonding interactions.[a]





[a] The molecular modelings were carried out using Spartan' 14 Software from Wavefunction, Inc.

[b] Corresponding ε-Co(III)-aminoalkyl radical intermediate from reaction of substrate **1a**.

[c] Corresponding ε-Co(III)-aminoalkyl radical intermediate from reaction of substrate **1b**.

[d] Corresponding ε-Co(III)-aminoalkyl radical intermediate from reaction of substrate **1c**.

Figure S3. High resolution mass spectroscopy (HRMS) spectrum for Co(III)-supported alkyl radical intermediate**.**

sCLIPS Report - F:\Agilent6220_1257.d\AcqData\MSProfile.bin

Self-Calibration Mass Range (Da)
Start: -0.43
End: 0.43

RT Windows
Scan 29 at 0.466

sCLIPS Parameters

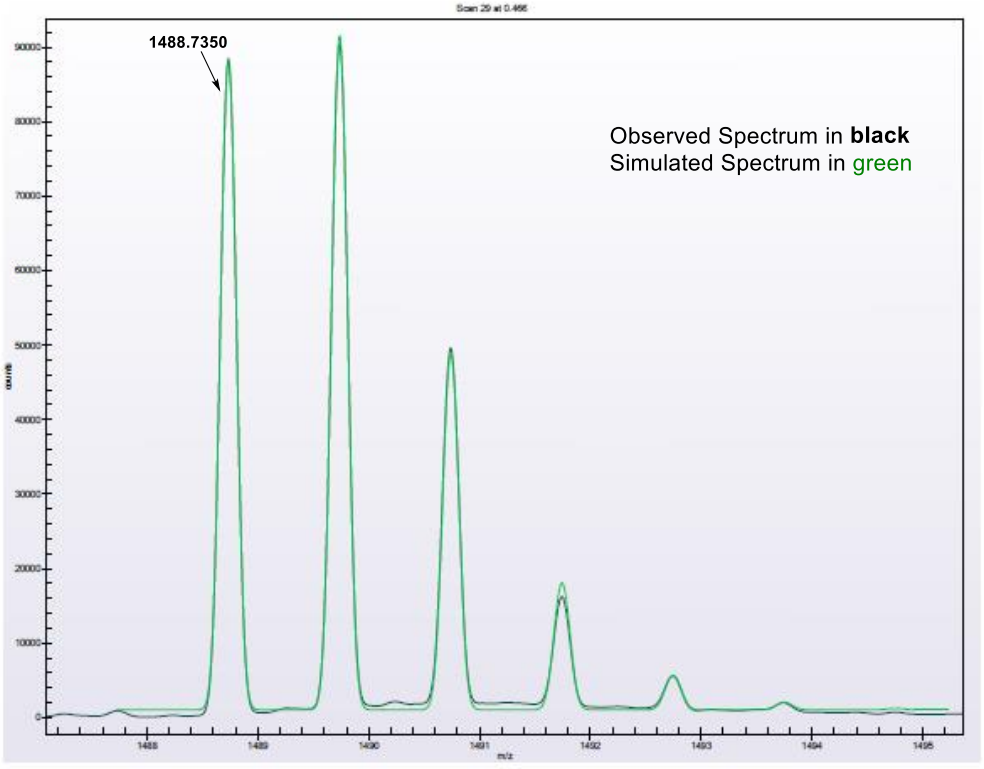
Accurate Mass:	1488.7350
Charge:	1
Mass Tolerance (mDa):	250.69
Electron State:	Both
Double Bond Equivalent Range	
Minimum:	-200.00
Maximum:	200.00
Profile Mass Range (Da)	
Start:	-1.00
End:	6.50
Empirical Rules:	Enabled
Empirical Elemental Limits:	Wiley
H/C Ratio:	Extended
Heteroatom Ratios:	Extended

Element	Minimum	Maximum
C	1	100
H	0	110
Co	1	1
N	0	9
O	0	6

Chemical Formula: $C_{92}H_{103}CoN_9O_6^+$
Calculated Mass: ***1488.7358***
Found Mass: ***1488.7350***

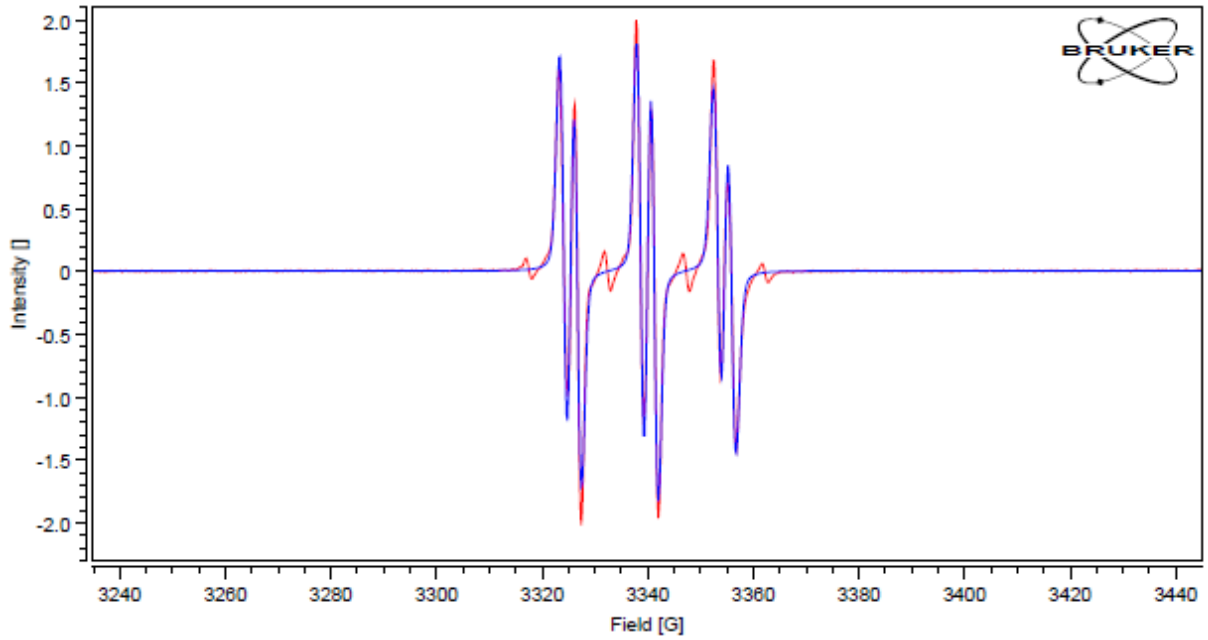
sCLIPS Search Results

	Formula	Mono Isotope Mass	Error (mDa)	Mass Error (PPM)	Spectral Accuracy	RMSE	DBE
1	C92H103CoN9O6	1,488.7358	-0.7837	-0.5264	96.6621	600	46.0
2	C93H105CoN8O6	1,488.7484	-13.3598	-8.9739	96.5953	612	45.5
3	C93H107CoN9O5	1,488.7722	-37.1692	-24.9670	96.5795	615	45.0



Procedure for HRMS Experiment: To an over-dried Schlenk tube, sulfonylhydrazone **1c** (0.05 mmol) and Cs_2CO_3 (2.0 equiv.) were added. The Schlenk tube was then evacuated and backfilled with nitrogen for 3 times. The Teflon screw cap was replaced with a rubber septum, and CH_3CN (0.5 mL) was added via a gastight syringe. The mixture was then stirred at 60 ^oC for 0.5 h. The resulting light yellow solution was then passed through a short pad of Celite (to get rid of base and salt) under the flow of nitrogen and the filtrate was collected in a HPLC vial (vial A, degassed and backfilled with argon). During the time, [Co(**P1**)] (2 mol %) was charged into another HPLC vial (vial B, degassed and backfilled with argon) and dissolved in CH_3CN (0.5 mL). After mixing equal amount of solutions from vial A (0.1 mL) and vial B (0.1 mL), the sample was further diluted with CH_3CN and immediately injected into HRMS instrument. The HRMS experiment was carried out in the absence of any additives such as formic acid, which commonly act as electron carriers for ionization, allowing for the detection of the molecular ion signals corresponding to Co(III)-alkyl radical ($C_{92}H_{103}CoN_9O_6^{\cdot}$) by the loss of one electron.

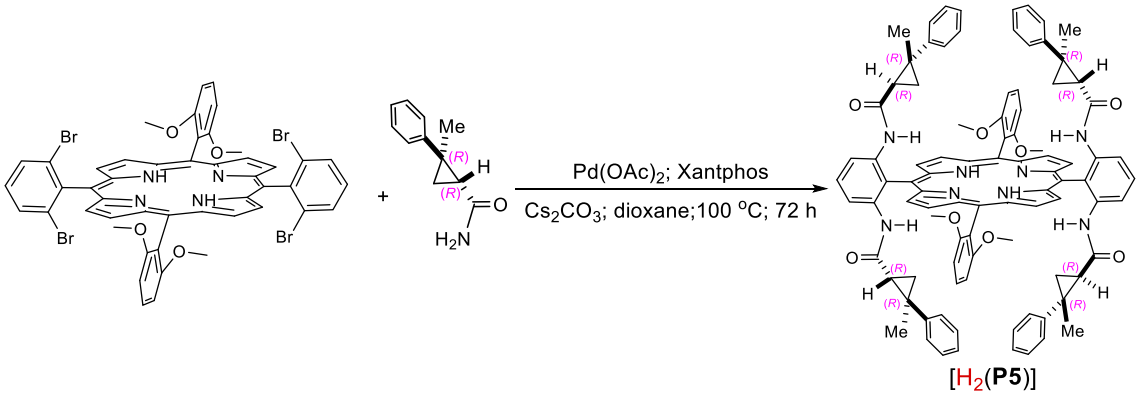
Figure S4. Isotropic X-band EPR spectrum of phenyl *N*-*tert*-butylnitrone (PBN)-trapped Co(III)-supported alkyl radical intermediate.



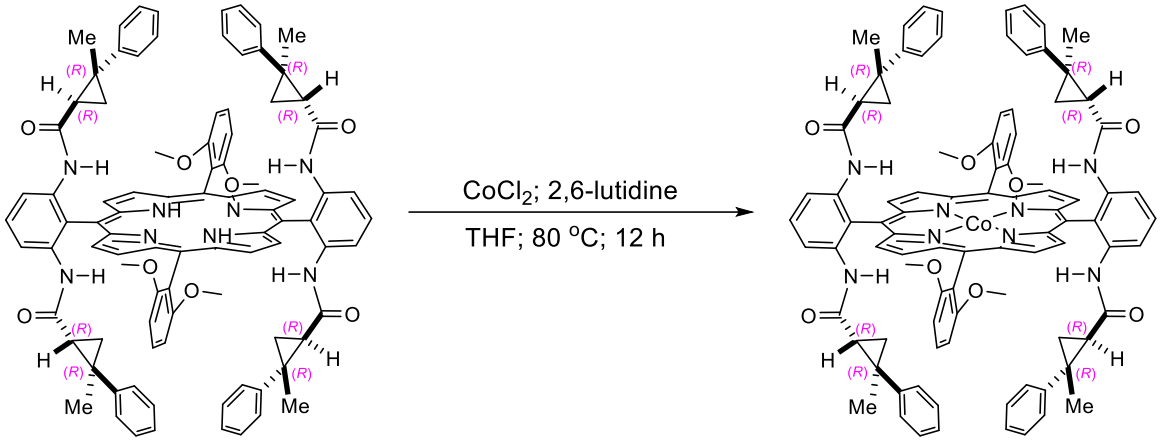
The resulting strong EPR signal (in red) has been simulated (in blue) with g = 2.006, A_N = 14.6 G, A_H = 2.6 G, which is assigned to PBN-trapped Co(III)-supported alkyl radical intermediate. The values are consistent with that of the reported similar species.[1] *[The simulation of the EPR spectrum was performed by iteration of the isotropic g-values and line widths using the EPR simulation program SpinFit Xenon]*

Procedure for EPR Experiment: To an oven-dried Schlenk tube A, sulfonylhydrazone **1c** (0.05 mmol) and Cs_2CO_3 (2.0 equiv.) were added. The Schlenk tube was then evacuated and backfilled with nitrogen for 3 times. The Teflon screw cap was replaced with a rubber septum, and benzene (0.5 mL) was added via a gastight syringe. The mixture was then stirred at 60 °C for 0.5 h. During the time, [Co(**P1**)] (4 mol %) was charged into another oven-dried Schlenk tube B. The Schlenk tube B was then evacuated and backfilled with nitrogen for 3 times. After 0.5 h, the resulting light yellow solution from tube A was passed through a short pad of Celite (to get rid of base and salt) under the flow of nitrogen and transferred to Schlenk tube B. The mixture was stirred for 1 min, followed by the addition of phenyl *N*-*tert*-butylnitrone (PBN, 0.05 mmol). The reaction mixture was stirred for 3 min and transferred into a degassed EPR tube (filled with argon) through a gastight syringe. The sample was then carried out for EPR experiment at room temperature (EPR settings: T = 298 K; microwave frequency: 9.37762 GHz; power: 6.325 mW; modulation amplitude: 1.0 G).

IV. Synthesis of Catalyst [Co(P5)]

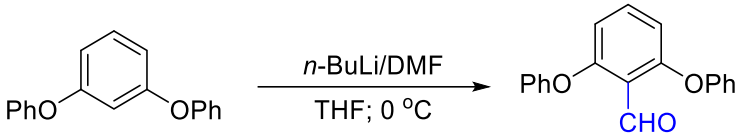


[H$_2$(P5)] was synthesized according to our previous reported procedure[1-2] with 58% yield. The 5,15-bis(2,6-dibromophenyl)-10,20-bis(2,6-dimethoxyphenyl)porphyrin (0.2 mmol), (1*R*, 2*R*)-2-methyl-2-phenylcyclopropane-1-carboxamide[3] (3.2 mmol), $Pd(OAc)_2$ (0.08 mmol), Xantphos (0.16 mmol), and Cs_2CO_3 (3.2 mmol) were placed in an oven-dried, resealable Schlenk tube. The tube was capped with a Teflon screwcap, evacuated, and backfilled with nitrogen. The screwcap was replaced with a rubber septum, and dioxane (10 mL) was added via a gastight syringe. The tube was purged with nitrogen for 2 minutes, and then the septum was replaced with the Teflon screwcap. The tube was sealed and stirred at 100 °C for 72 h. The resulting mixture was cooled down to room temperature, diluted in ethyl acetate, filtrated through a silica pad and concentrated under vacuum. The pure compound was obtained as a purple solid after purification by flash column chromatography (hexanes/DCM/ethyl acetate: 10/10/2 to 10/10/3). ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.88 (s, 8H), 8.48 (d, *J* = 5.9 Hz, 4H), 7.89 (t, *J* = 8.3 Hz, 2H), 7.69 (t, *J* = 8.5 Hz, 2H), 6.90 (d, *J* = 8.6 Hz, 4H), 6.68 (s, 4H), 5.98 (s, 4H), 5.31 (br, 16H), 2.96 (s, 12H), 0.99 – 0.96 (m, 16H), 0.56 (br, 4H), 0.18 (br, 4H), -2.12 (s, 2H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 168.60, 160.36, 144.50, 139.05, 132.77, 130.79, 130.28, 130.01, 126.81, 125.94, 124.97, 121.46, 117.81, 117.15, 114.78, 106.81, 103.80, 55.07, 30.01, 29.92, 19.42, 17.71. IR (neat, cm^{-1}): 3409.14, 3313.83, 2928.10, 2836.12, 1690.30, 1586.35, 1464.60, 1249.90, 1108.38, 731.66. HRMS (ESI) ($M+H^+$) Calcd. for $C_{92}H_{83}N_8O_8^+$: 1427.6328, found 1427.6301. UV–vis (CH_2Cl_2), λ_{max} nm (log ε): 421(5.43), 514(4.23), 588(3.74), 643(3.34).

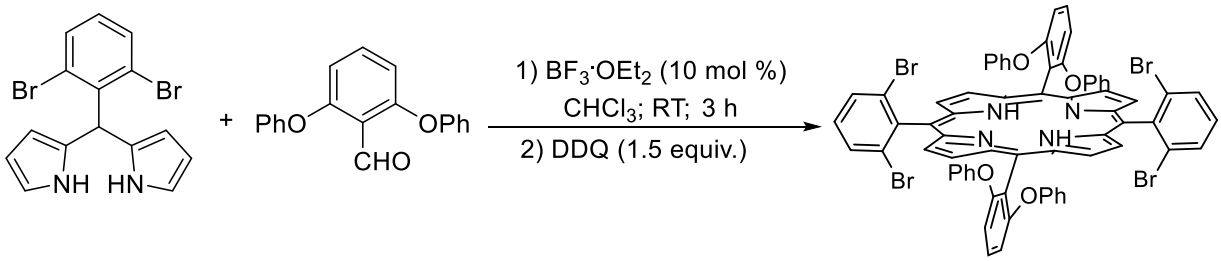


[Co(P5)] was synthesized according to our previous reported procedure[2] with 92% yield. Free base porphyrin [H_2(**P5**)] and anhydrous $CoCl_2$ (8 equiv.) were placed in an oven-dried, resealable Schlenk tube. The tube was capped with a Teflon screwcap, evacuated, and backfilled with nitrogen. The screwcap was then replaced with a rubber septum, 2,6-lutidine (4 equiv.) and anhydrous THF were added via a gastight syringe. The tube was purged with nitrogen for 2 minutes, and then the septum was replaced with the Teflon screwcap. The reaction was conducted at 80 °C for 12 h. The resulting mixture was cooled down to room temperature, diluted with ethyl acetate, and transferred to a separatory funnel. The reaction mixture was washed with water 3 times and concentrated under vacuum. The target compound [Co(**P5**)] was isolated as a purple solid after purification by flash column chromatography with hexanes/ethyl acetate (2/1) as eluent. IR (neat, cm^{-1}): 3405.77, 2930.13, 1691.39, 1584.23, 1464.43, 1249.49, 1107.50, 997.65, 762.32. HRMS (ESI) (M^{*+}) Calcd. for C92H80CoN8O8: 1483.5426, found 1483.5488. UV–vis (CH_2Cl_2), λ_{max} nm (log ε): 413(4.90), 532(3.77).

V. Synthesis of Catalyst [Co(P6)]

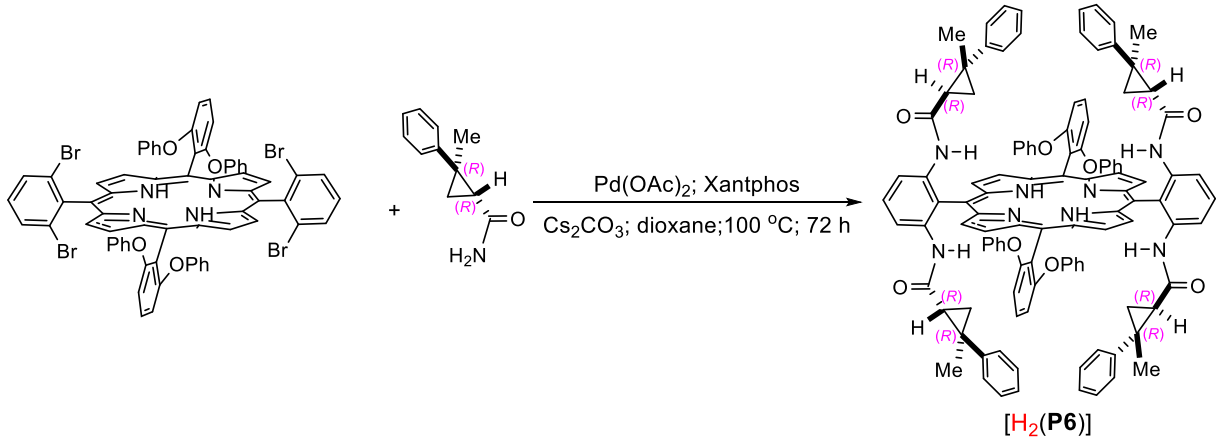


2, 6-diphenoxybenzaldehyde was synthesized according to previous reported procedure.[4] To a stirred solution of 1,3-diphenoxybenzene (10 mmol) in dry THF (60 mL) at 0 ℃, *n*-BuLi (8 mL, 1.5 M in hexanes) was added dropwise for 1 h. Then the mixture was stirred at room temperature for 2 h and followed by the slow addition of DMF (1.83 g, 25 mmol). After 2 h, the mixture was poured into ice water. The organic phase was separated and the aqueous phase was extracted with ether (3×30 mL). The combined organic layer was dried over anhydrous Na_2SO_4. After the removal of solvent under vacuum, the product was purified by column chromatography with hexanes/ethyl acetate (9:1 to 6:1) as eluent to afford 2, 6-diphenoxybenzaldehyde as a white solid in 70% yield. ^{1}H NMR (600 MHz, $CDCl_3$) δ 10.61 (s, 1H), 7.39 (t, J = 7.9 Hz, 4H), 7.32 (t, J = 8.4 Hz, 1H), 7.18 (t, J = 7.4 Hz, 2H), 7.10 – 7.08 (m, 4H), 6.58 (d, J = 8.4 Hz, 2H).^{13}C NMR (150 MHz, $CDCl_3$) δ 188.15, 159.85, 156.13, 135.09, 129.95, 124.31, 119.61, 118.47, 112.72. IR (neat, cm^{-1}): 2774.28, 1688.49, 1598.71, 1570.31, 1487.76, 1454.31, 1409.35, 1204.09, 1030.85, 772.93, 717.85, 685.53. HRMS (ESI) ($M+H^+$) Calcd. for $C19H15O3^+$: 291.1021, found 291.1034.



5,15-bis(2,6-dibromophenyl)-10,20-bis(2,6-diphenoxyphenyl)porphyrin was synthesized according to our previous reported procedure[2] with 60% yield. A mixture of *meso*-(2,6-dibromophenyl)dipyrromethane (5 mmol), 2, 6-diphenoxybenzaldehyde (5 mmol) in chloroform (500 mL) was purged with nitrogen for 10 min. The flask was wrapped with aluminum foil to shield it from light. Then boron trifluoride diethyl etherate was added dropwise via a syringe. After the solution was stirred under the nitrogen atmosphere at room temperature for 3 h, 2,3-dichloro-5,6-dicyano-1,4-benzoquinone (DDQ) (7.5 mmol) was added at one time. After 1 h, triethylamine

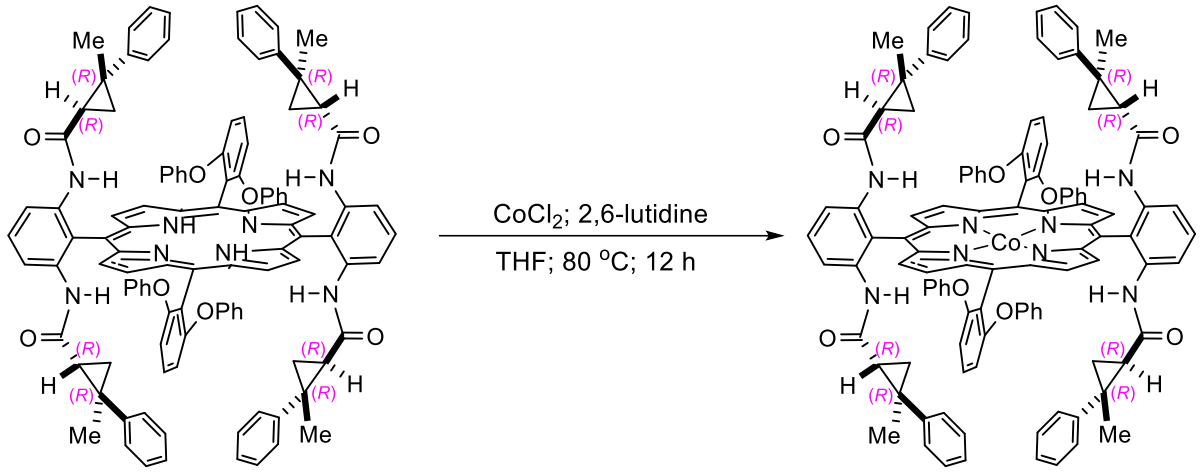
(10 mL) was added. The reaction solution was then directly poured into a silica gel column that was rinsed with dichloromethane. The column was eluted with dichloromethane. The fractions containing the product were collected and concentrated under vacuum. The residue was washed several times with methanol to afford the pure compound. ^{1}H NMR (600 MHz, CD_2Cl_2) δ 9.00 (s, 4H), 8.62 (s, 4H), 8.06 (d, J = 7.7 Hz, 4H), 7.68 (t, J = 7.7 Hz, 2H), 7.57 (t, J = 7.5 Hz, 2H), 7.11 (d, J = 8.0 Hz, 4H), 6.89 (d, J = 5.8 Hz, 8H), 6.70 (d, J = 6.5 Hz, 12H), -2.73 (s, 2H). ^{13}C NMR (150 MHz, CD_2Cl_2) δ 159.40, 156.70, 143.47, 131.93, 131.61, 130.68, 129.58, 128.78, 124.59, 123.70, 119.58, 118.31, 113.27, 111.14. IR (neat, cm^{-1}): 3311.65, 1570.06, 1487.58, 1449.43, 1230.13, 1209.38, 1023.02, 1012.10, 979.66, 796.47, 721.44. HRMS (ESI) ($M+H^+$) Calcd. for $C68H43Br4N4O4^+$: 1295.0012, found 1295.0050. UV–vis (CH_2Cl_2), λ_{max} nm (log ε): 422(5.63), 516(4.37), 592(3.87), 646(3.11).



[H_2(**P6**)]

[H_2(P6)] was synthesized according to our previous reported procedure[2] with 61% yield. The 5,15-bis(2,6-dibromophenyl)-10,20-bis(2,6-diphenoxyphenyl)porphyrin (0.2 mmol), (1*R*, 2*R*)-2-methyl-2-phenylcyclopropane-1-carboxamide[3] (3.2 mmol), $Pd(OAc)_2$ (0.08 mmol), Xantphos (0.16 mmol), and Cs_2CO_3 (3.2 mmol) were placed in an oven-dried, resealable Schlenk tube. The tube was capped with a Teflon screwcap, evacuated, and backfilled with nitrogen. The screwcap was replaced with a rubber septum, and dioxane (10 mL) was added via a gastight syringe. The tube was purged with nitrogen for 2 minutes, and then the septum was replaced with the Teflon screwcap. The tube was sealed and stirred at 100 °C for 72 h. The resulting mixture was cooled down to room temperature, diluted in ethyl acetate, filtrated through a silica pad and concentrated under vacuum. The pure compound was obtained as a purple solid after purification by flash

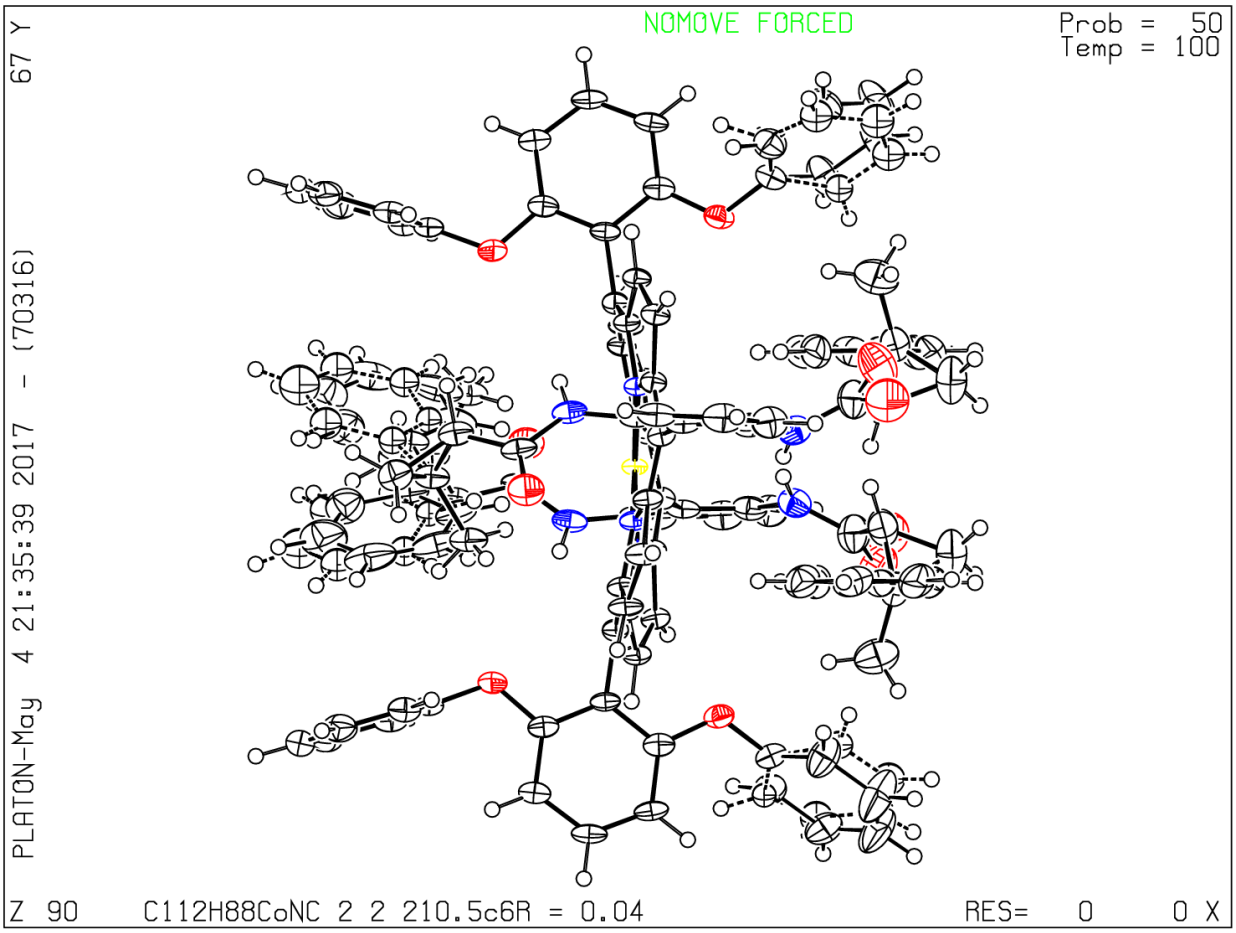
column chromatography (hexanes/ethyl acetate: 3/1 to 2/1). ^{1}H NMR (600 MHz, $CDCl_3$) δ 9.18 (d, *J* = 4.4 Hz, 4H), 8.92 (d, *J* = 4.4 Hz, 4H), 8.49 (br, 4H), 7.89 (t, *J* = 8.3 Hz, 2H), 7.52 (t, *J* = 8.6 Hz, 2H), 6.87 (t, *J* = 7.9 Hz, 8H), 6.82 (d, *J* = 8.5 Hz, 4H), 6.74 (t, *J* = 7.3 Hz, 4H), 6.58 (br, 4H), 6.52 (d, *J* = 7.9 Hz, 8H), 6.04 (br, 4H), 5.60 (br, 8H), 5.44 (br, 8H), 0.77 (s, 12H), 0.60 (br, 4H), 0.22 (br, 4H), 0.09 (br, 4H), -2.27(s, 2H). ^{13}C NMR (150 MHz, $CDCl_3$) δ 168.77, 159.50, 155.55, 144.27, 139.06, 132.58, 130.48, 130.35, 129.21, 126.92, 125.71, 125.07, 123.63, 121.64, 121.44, 119.90, 117.38, 113.00, 110.70, 107.55, 30.13, 29.36, 19.10, 18.31. IR (neat, cm^{-1}): 3409.85, 3009.75, 1686.60, 1686.60, 1488.40, 1450.74, 1208.39, 1160.10, 978.20, 749.56, 692.35. HRMS (ESI) ($M+H^+$) Calcd. for $C_{112}H_{91}N_8O_8^+$: 1675.6954, found 1675.6960. UV–vis (CH_2Cl_2), λ_{max} nm (log ε): 422(5.46), 515(4.25), 591(3.75), 645(3.12).



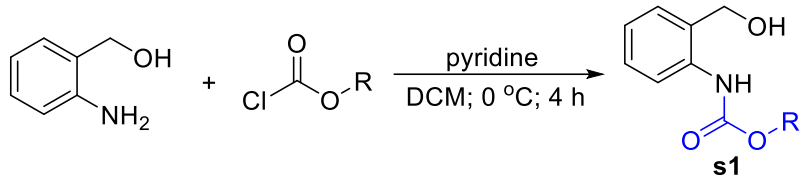
[H_2(**P6**)] [Co(**P6**)]

[Co(P6)] was synthesized according to our previous reported procedure[2] with 92% yield. Free base porphyrin [H_2(**P6**)] and anhydrous $CoCl_2$ (8 equiv.) were placed in an oven-dried, resealable Schlenk tube. The tube was capped with a Teflon screwcap, evacuated, and backfilled with nitrogen. The screwcap was then replaced with a rubber septum, 2,6-lutidine (4 equiv.) and anhydrous THF were added via a gastight syringe. The tube was purged with nitrogen for 2 minutes, and then the septum was replaced with the Teflon screwcap. The reaction was conducted at 80 °C for 12 h. The resulting mixture was cooled down to room temperature, diluted with ethyl acetate, and transferred to a separatory funnel. The reaction mixture was washed with water for 3 times and concentrated under vacuum. The target compound [Co(**P6**)] was isolated as a purple solid after purification by flash column chromatography with hexanes/ethyl acetate (2/1) as eluent. IR (neat,

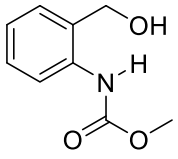
cm^{-1}): 3407.80, 1692.64, 1488.16, 1449.06, 1206.20, 1159.67, 998.09, 759.70, 690.98. HRMS (ESI) (M^{*+}) Calcd. for $C_{112}H_{88}CoN_8O_8$: 1731.6057, found 1731.6057. UV–vis (CH_2Cl_2), λ_{max} nm (log ε): 414(4.84), 534(3.67).



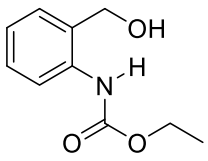
VI. The Synthetic Procedure for (2-(Hydroxymethyl)phenyl)carbamate s1



The compound **s1** was synthesized according to the previous reported procedure.[5] To a solution of 2-aminobenzyl alcohol (20 mmol) and pyridine (26 mmol) in DCM (80.0 mL) at 0 °C, methyl chloroformate (or ethyl chloroformate) (22 mmol) was added dropwise. The reaction was then stirred at 0 °C for 4 h. After that, the reaction was quenched by the addition of 0.1 M HCl and extracted with DCM (80 mL) for 3 times. The combined organic layers were then dried over anhydrous Na_2SO_4 and concentrated under vacuum. The mixture was then purified by flash column chromatography.

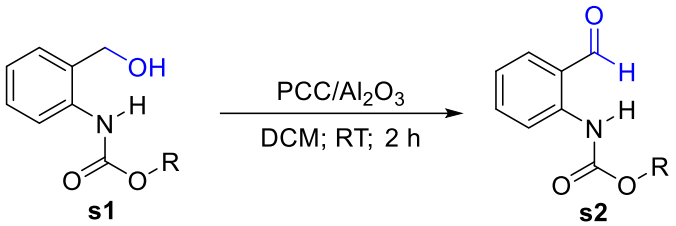


methyl (2-(hydroxymethyl)phenyl)carbamate s1-a White solid. Yield: 81%. Hexanes/ethyl acetate = 3/1. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.94 (d, *J* = 7.6 Hz, 1H), 7.90 (s, 1H), 7.34 (td, *J* = 8.0, 1.6 Hz, 1H), 7.17 (dd, *J* = 7.5, 1.6 Hz, 1H), 7.04 (td, *J* = 7.5, 1.1 Hz, 1H), 4.72 (d, *J* = 5.8 Hz, 2H), 3.78 (s, 3H), 1.96 (t, *J* = 5.8 Hz, 1H).^{13}C NMR (125 MHz, $CDCl_3$) δ 154.57, 137.61, 129.17, 128.89, 128.78, 123.42, 120.98, 64.22, 52.29. IR (neat, cm^{-1}): 3288.39, 1697.47, 1528.92, 1455.34, 1294.08, 1247.81, 1024.00, 664.01. HRMS (ESI) ($M+H^+$) Calcd. for $C9H12NO3^+$: 182.0812, found 182.0810.

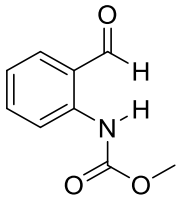


ethyl (2-(hydroxymethyl)phenyl)carbamate s1-b, known compound.[6] White solid. Yield: 77%. Hexanes/ethyl acetate = 3/1. ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.93 – 7.86 (m, 2H), 7.32 (t, *J* = 7.8 Hz, 1H), 7.16 (d, *J* = 7.1 Hz, 1H), 7.03 (t, *J* = 7.5 Hz, 1H), 4.69 (d, *J* = 5.7 Hz, 2H), 4.21 (q, *J* = 7.1 Hz, 2H), 2.25 (t, *J* = 5.7 Hz, 1H), 1.31 (t, *J* = 7.1 Hz, 3H). ^{13}C NMR (150 MHz, $CDCl_3$) δ 154.16, 137.74, 129.21, 128.83, 123.31, 120.98, 64.29, 61.21, 14.52.

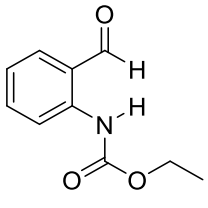
VII. The Synthetic Procedure for (2-Formylphenyl)carbamate s2



The compound **s2** was synthesized according to the previous reported procedure.[7] To a solution of carbamate **s1** (15 mmol) in 150 mL of DCM was added pyridinium chlorochromate (PCC, 30 mmol) and Al_2O_3 (use same amount as PCC in order to ease the separation of the desired product from the PCC residue). The reaction mixture was stirred at room temperature for 2 h and then filtered through a pad of silica. The filtrate was concentrated under reduced pressure and purified by flash column chromatography with hexanes/ethyl acetate (4/1) as eluent.

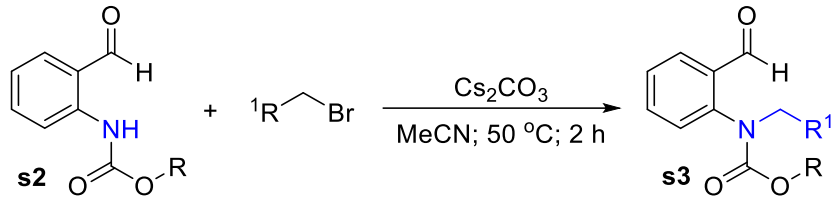


methyl (2-formylphenyl)carbamate s2-a White solid. Yield: 90%. ^{1}H NMR (500 MHz, $CDCl_3$) δ 10.61 (s, 1H), 9.90 (s, 1H), 8.45 (d, *J* = 8.5 Hz, 1H), 7.64 (d, *J* = 7.7 Hz, 1H), 7.59 (t, *J* = 7.9 Hz, 1H), 7.16 (t, *J* = 7.5 Hz, 1H), 3.80 (s, 3H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 195.03, 154.09, 141.22, 135.99, 135.97, 121.92, 121.31, 118.24, 52.39. IR (neat, cm^{-1}): 3277.15, 3023.56, 2957.46, 2843.22, 2764.22, 1731.62, 1522.48, 1455.43, 1214.36, 1058.89, 1044.52, 769.03, 695.17. HRMS (ESI) ($M+H^+$) Calcd. for $C9H10NO3^+$: 180.0661, found 180.0666.

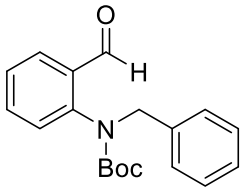


ethyl (2-formylphenyl)carbamate s2-b White solid. Yield: 85%. ^{1}H NMR (600 MHz, $CDCl_3$) δ 10.56 (s, 1H), 9.91 (s, 1H), 8.46 (d, *J* = 8.5 Hz, 1H), 7.64 (dd, *J* = 7.6, 1.4 Hz, 1H), 7.61 – 7.58 (m, 1H), 7.16 (td, *J* = 7.6, 0.9 Hz, 1H), 4.24 (q, *J* = 7.1 Hz, 2H), 1.34 (t, *J* = 7.1 Hz, 3H). ^{13}C NMR (150 MHz, $CDCl_3$) δ 195.03, 153.70, 141.37, 135.99, 121.80, 121.28, 118.27, 61.39, 14.46. IR (neat, cm^{-1}): 3278.69, 2985.48, 1729.45, 1654.96, 1585.28, 1522.65, 1451.42, 1242.86, 1191.63, 1058.03, 1042.13, 871.07, 764.43. HRMS (ESI) ($M+H^+$) Calcd. for $C10H12NO3^+$: 194.0812, found 194.0809.

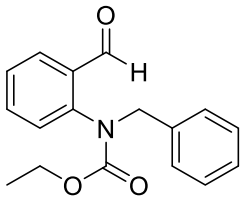
VIII. The Synthetic Procedure for Benzyl(2-formylphenyl)carbamate Derivatives s3



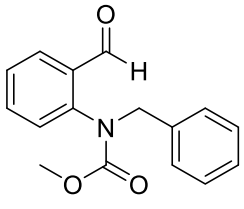
To a solution of **s2** (2 mmol) and Cs_2CO_3 (2.4 mmol) in MeCN (20 mL) was added alkyl bromide (2.4 mmol) at room temperature. The reaction was heated at 50 °C for 2 h. The resulting mixture was cooled down to room temperature and filtered through a short pad of silica. The combined organic mixture was concentrated under vacuum and purified by flash column chromatography.



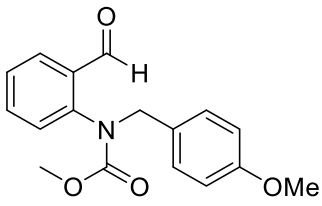
***tert*-butyl benzyl(2-formylphenyl)carbamate s3-a** Yield: 95%. Hexanes/ethyl acetate = 10/1. ^{1}H NMR (500 MHz, $CDCl_3$) δ 9.75 (s, 1H), 7.82 (d, J = 6.5 Hz, 1H), 7.57 – 7.54 (m, 1H), 7.36 (t, J = 7.4 Hz, 1H), 7.25 – 7.14 (m, 6H), 4.99 and 4.78 (br, 2H), 1.33 (br, 9H). ^{13}C NMR (150 MHz, $CDCl_3$) δ 189.80, 154.58, 144.27, 136.99, 134.59, 132.88, 128.73, 128.55, 128.37, 128.15, 127.76, 127.36, 81.36, 54.29, 28.12. IR (neat, cm^{-1}): 2978.49, 2873.61, 1682.42, 1595.02, 1484.35, 1445.89, 1367.81, 1152.81, 1016.51, 861.22, 740.86. HRMS (ESI) ($M+H^+$) Calcd. for $C19H22NO3^+$: 312.1594, found 312.1596.



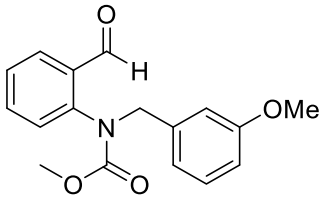
ethyl benzyl(2-formylphenyl)carbamate s3-b Yield: 94%. Hexanes/ethyl acetate = 6/1. ^{1}H NMR (600 MHz, $CDCl_3$) δ 9.72 (br, 1H), 7.84 (d, J = 7.2 Hz, 1H), 7.56 (td, J = 7.7, 1.6 Hz, 1H), 7.40 (t, J = 7.5 Hz, 1H), 7.26 – 7.25 (m, 3H), 7.18 – 7.13 (m, 3H), 4.96 and 4.82 (br, 2H), 4.18 and 4.07 (br, 2H), 1.09 (br, 3H). ^{13}C NMR (150 MHz, $CDCl_3$) δ 189.58, 155.64, 143.50, 136.48, 134.71, 132.87, 128.91, 128.59, 127.93, 127.77, 62.28, 54.92, 14.45. IR (neat, cm^{-1}): 2917.25, 2848.98, 1709.73, 1598.20, 1455.24, 1378.29, 1216.68, 1019.37, 701.86. HRMS (ESI) ($M+H^+$) Calcd. for $C17H18NO3^+$: 284.1289, found 284.1287.



methyl benzyl(2-formylphenyl)carbamate s3-c Yield: 99%. Hexanes/ethyl acetate = 6/1. ^{1}H NMR (600 MHz, $CDCl_3$) δ 9.72 (s, 1H), 7.84 (d, *J* = 7.6 Hz, 1H), 7.58 – 7.55 (m, 1H), 7.43 – 7.39 (m, 1H), 7.28 – 7.23 (m, 3H), 7.17 (d, *J* = 3.3 Hz, 2H), 7.12 (d, *J* = 7.8 Hz, 1H), 4.92 and 4.84 (br, 2H), 3.65 (br, 3H). ^{13}C NMR (150 MHz, $CDCl_3$) δ 189.55, 156.13, 143.26, 134.78, 132.85, 129.16, 129.00, 128.80, 128.62, 128.00, 127.95, 127.76, 55.12, 53.38. IR (neat, cm^{-1}): 3030.97, 2954.16, 2855.87, 1713.47, 1598.69, 1454.69, 1382.03, 1270.69, 734.74. HRMS (ESI) ($M+H^+$) Calcd. for $C16H16NO3^+$: 270.1125, found 270.1126.

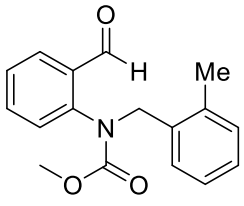


methyl (2-formylphenyl)(4-methoxybenzyl)carbamate s3-d Yield: 98%. Hexanes/ethyl acetate = 4/1. ^{1}H NMR (600 MHz, $CDCl_3$) δ 9.71 (s, 1H), 7.84 (d, *J* = 7.6 Hz, 1H), 7.57 (td, *J* = 7.7, 1.5 Hz, 1H), 7.41 (t, *J* = 7.5 Hz, 1H), 7.08 (d, *J* = 8.0 Hz, 3H), 6.77 (d, *J* = 8.5 Hz, 2H), 4.89 and 4.75 (br, 2H), 3.76 (s, 3H), 3.63 (s, 3H). ^{13}C NMR (150 MHz, $CDCl_3$) δ 189.57, 159.31, 156.07, 143.30, 134.75, 132.91, 130.34, 128.89, 128.60, 128.44, 127.89, 113.92, 55.18, 54.51, 53.29. IR (neat, cm^{-1}): 2955.10, 2837.50, 2758.23, 1711.53, 1611.89, 1598.52, 1514.07, 1459.69, 1251.04, 1034.18. HRMS (ESI) ($M+H^+$) Calcd. for $C17H18NO4^+$: 300.1230, found 300.1232.

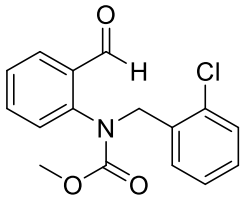


methyl (2-formylphenyl)(3-methoxybenzyl)carbamate s3-e Yield: 99%. Hexanes/ethyl acetate = 4/1. ^{1}H NMR (600 MHz, $CDCl_3$) δ 9.76 (s, 1H), 7.85 (d, *J* = 7.5 Hz, 1H), 7.59 – 7.54 (m, 1H), 7.41 (t, *J* = 7.5 Hz, 1H), 7.17 (t, *J* = 7.8 Hz, 1H), 7.14 (d, *J* = 7.5 Hz, 1H), 6.80 (dd, *J* = 8.3, 2.5 Hz, 1H), 6.75 (d, *J* = 7.6 Hz, 1H), 6.73 (s, 1H), 4.88 and 4.82 (br, 2H), 3.73 (s, 3H), 3.83 and 3.64 (br, 3H).^{13}C NMR (150 MHz, $CDCl_3$) δ 189.59, 159.70, 156.11, 143.25, 137.85, 134.75, 132.85, 129.63, 129.20, 128.72, 127.92, 121.20, 114.43, 113.50, 55.17, 55.02,

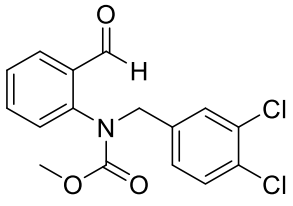
53.37. IR (neat, cm^{-1}): 3002.67, 2953.35, 2836.31, 1692.94, 1597.51, 1488.11, 1445.75, 1377.15, 1299.06, 1264.77, 1192.76, 1051.67, 769.19, 740.54. HRMS (ESI) ($M+H^+$) Calcd. for $C17H18NO4^+$: 300.1230, found 300.1236.



methyl (2-formylphenyl)(2-methylbenzyl)carbamate s3-f Yield: 99%. Hexanes/ethyl acetate = 5/1. ^1H NMR (600 MHz, $CDCl_3$) δ 9.74 (s, 1H), 7.82 (d, *J* = 7.6 Hz, 1H), 7.55 – 7.53 (m, 1H), 7.40 (t, *J* = 7.5 Hz, 1H), 7.16 – 7.15 (m, 1H), 7.10 (t, *J* = 8.0 Hz, 2H), 7.05 (br, 2H), 4.98 and 4.90 (br, 2H), 3.66 (s, 3H), 2.13 (s, 3H). ^{13}C NMR (150 MHz, $CDCl_3$) δ 189.42, 156.06, 143.11, 136.59, 134.70, 134.24, 133.06, 130.54, 130.12, 129.23, 128.89, 128.06, 127.98, 126.07, 53.39, 52.16, 19.02. IR (neat, cm^{-1}): 3022.23, 2953.91, 2860.00, 1711.08, 1598.43, 1486.9, 1457.43, 1377.82, 1304.30, 1272.43, 1194.06, 743.36. HRMS (ESI) ($M+H^+$) Calcd. for $C17H18NO3^+$: 284.1281, found 284.1284.

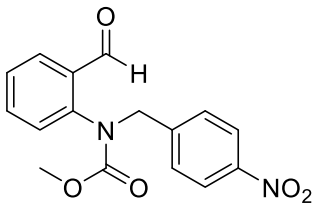


methyl (2-chlorobenzyl)(2-formylphenyl)carbamate s3-g Yield: 99%. Hexanes/ethyl acetate = 5/1. ^1H NMR (600 MHz, $CDCl_3$) δ 9.88 (s, 1H), 7.83 (d, *J* = 7.6 Hz, 1H), 7.55 (td, *J* = 7.8, 1.4 Hz, 1H), 7.41 (t, *J* = 7.5 Hz, 2H), 7.29 – 7.27 (m, 1H), 7.21 – 7.18 (m, 2H), 7.16 (d, *J* = 7.8 Hz, 1H), 5.05 (br, 2H), 3.79 and 3.67 (s, 3H). ^{13}C NMR (150 MHz, $CDCl_3$) δ 189.61, 156.12, 143.00, 134.68, 134.04, 132.89, 131.34, 129.63, 129.31, 128.55, 127.96, 127.56, 127.02, 126.85, 53.47, 51.90. IR (neat, cm^{-1}): 3004.63, 2953.62, 2360.00, 1712.93, 1598.56, 1444.12, 1379.73, 1302.25, 1277.42, 765.24, 742.13. HRMS (ESI) ($M+H^+$) Calcd. for $C16H15ClNO3^+$: 304.0735, found 304.0739.

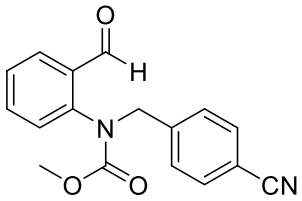


methyl (3,4-dichlorobenzyl)(2-formylphenyl)carbamate s3-h Yield: 98%. Hexanes/ethyl acetate = 5/1. ^1H NMR (600 MHz, $CDCl_3$) δ 9.87 (s, 1H), 7.88 (dd, *J* = 7.7,

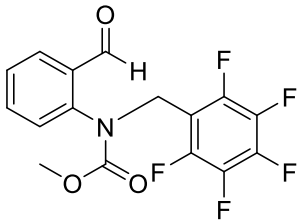
1.5 Hz, 1H), 7.59 (td, *J* = 7.7, 1.7 Hz, 1H), 7.46 (t, *J* = 7.5 Hz, 1H), 7.35 (d, *J* = 8.2 Hz, 1H), 7.32 (s, 1H), 7.06 (t, *J* = 8.3 Hz, 2H), 4.91 and 4.70 (br, 2H), 3.66 (s, 3H). ^{13}C NMR (150 MHz, $CDCl_3$) δ 189.46, 156.08, 142.69, 142.52, 136.88, 134.87, 134.69, 132.63, 132.46, 132.08, 130.75, 130.57, 128.82, 128.24, 53.94, 53.51. IR (neat, cm^{-1}): 3002.67, 2953.42, 2860.35, 2746.50, 1712.18, 1598.16, 1447.29, 1374.56, 1297.34, 1216.67, 1032.47, 738.51. HRMS (ESI) ($M+H^+$) Calcd. for $C16H14Cl2NO3^+$: 338.0345, found 338.0347.



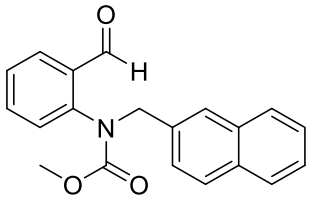
methyl (2-formylphenyl)(4-nitrobenzyl)carbamate s3-i Yield: 80%. Hexanes/ethyl acetate = 3/1. ^{1}H NMR (600 MHz, $CDCl_3$) δ 9.87 (s, 1H), 8.14 (d, *J* = 8.2 Hz, 2H), 7.87 (d, *J* = 7.5 Hz, 1H), 7.58 (t, *J* = 7.6 Hz, 1H), 7.47 (t, *J* = 7.5 Hz, 1H), 7.41 (d, *J* = 8.1 Hz, 2H), 7.09 (d, *J* = 5.9 Hz, 1H), 5.05 and 4.83 (br, 2H), 3.66 (s, 3H).^{13}C NMR (150 MHz, $CDCl_3$) δ 189.44, 156.12, 147.57, 143.97, 142.31, 134.91, 132.37, 131.10, 129.60, 128.72, 128.33, 123.83, 54.33, 53.58. IR (neat, cm^{-1}): 3077.25, 2955.54, 2857.16, 2756.27, 1691.77, 1597.46, 1518.47, 1445.74, 1344.17, 1307.21, 1267.61, 1192.18, 732.85. HRMS (ESI) ($M+H^+$) Calcd. for $C16H15N2O5^+$: 315.0975, found 315.0977.



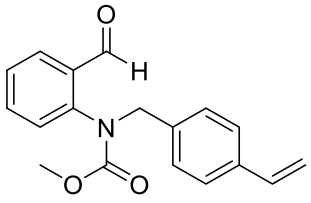
methyl (4-cyanobenzyl)(2-formylphenyl)carbamate s3-j Yield: 98%. Hexanes/ethyl acetate = 3/1. ^{1}H NMR (600 MHz, $CDCl_3$) δ 9.85 (s, 1H), 7.86 (d, *J* = 7.6 Hz, 1H), 7.59 – 7.56 (m, 3H), 7.46 (t, *J* = 7.5 Hz, 1H), 7.34 (d, *J* = 7.9 Hz, 2H), 7.07 (d, *J* = 7.4 Hz, 1H), 4.99 and 4.80 (br, 2H), 3.65 (br, 3H).^{13}C NMR (125 MHz, $CDCl_3$) δ 189.40, 156.10, 142.41, 141.95, 134.88, 134.72, 132.39, 130.85, 129.44, 128.68, 128.27, 118.45, 111.86, 54.62, 53.53. IR (neat, cm^{-1}): 2954.98, 2851.81, 2751.36, 2228.37, 1708.46, 1598.31, 1456.80, 1379.91, 1316.36, 1269.35, 1193.78, 778.53, 735.44, HRMS (ESI) ($M+H^+$) Calcd. for $C17H15N2O3^+$: 295.1077, found 295.1079.



methyl (2-formylphenyl)((perfluorophenyl)methyl)carbamate s3-k Yield: 97%. Hexanes/ethyl acetate = 4/1. ^{1}H NMR (500 MHz, $CDCl_3$) δ 9.94 (s, 1H), 7.87 (dd, *J* = 7.7, 1.4 Hz, 1H), 7.61 (td, *J* = 7.7, 1.6 Hz, 1H), 7.49 (t, *J* = 7.6 Hz, 1H), 7.18 (d, *J* = 7.8 Hz, 1H), 5.02 (br, 2H), 3.64 (br, 3H). ^{13}C NMR (150 MHz, $CDCl_3$) δ 189.48, 155.50, 145.45 (md, *J* = 250.0 Hz), 141.50, 141.12 (md, *J* = 255.3 Hz), 137.31 (md, *J* = 252.6 Hz), 134.82, 132.69, 131.10, 129.04, 128.53, 109.95, 53.58, 41.88. IR (neat, cm^{-1}): 2957.90, 2838.25, 2751.54, 1706.21, 1526.90, 1503.64, 1456.77, 1439.58, 1383.57, 1278.56, 966.81, 945.34, 760.50. HRMS (ESI) ($M+H^+$) Calcd. for $C16H11F5NO3^+$: 360.0654, found 360.0657.

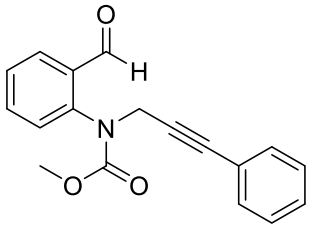


methyl (2-formylphenyl)(naphthalen-2-ylmethyl)carbamate s3-l Yield: 99%. Hexanes/ethyl acetate = 5/1. ^{1}H NMR (600 MHz, $CDCl_3$) δ 9.83 (s, 1H), 7.84 – 7.70 (m, 3H), 7.73 – 7.72 (m, 1H), 7.57 (s, 1H), 7.53 (td, *J* = 7.7, 1.5 Hz, 1H), 7.47 – 7.44 (m, 2H), 7.41-7.38 (m, 2H), 7.11 (br, 1H), 5.08 and 5.02 (br, 2H), 3.67 (br, 3H). ^{13}C NMR (150 MHz, $CDCl_3$) δ 189.58, 156.26, 143.16, 134.73, 133.93, 133.15, 132.87, 132.72, 129.46, 129.42, 128.81, 128.52, 127.94, 127.81, 127.67, 126.63, 126.22, 126.11, 55.20, 53.42. IR (neat, cm^{-1}): 3015.95, 2952.55, 2856.83, 2752.36, 1708.72, 1597.93, 1446.53, 1365.74, 755.23. HRMS (ESI) ($M+H^+$) Calcd. for $C20H18NO3^+$: 320.1281, found 320.1282.

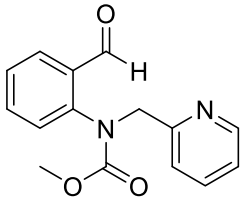


methyl (2-formylphenyl)(4-vinylbenzyl)carbamate s3-m Yield: 80%. Hexanes/ethyl acetate = 5/1. ^{1}H NMR (600 MHz, $CDCl_3$) δ 9.78 (s, 1H), 7.85 (d, *J* = 7.5 Hz, 1H), 7.56 (td, *J* = 7.6, 1.6 Hz, 1H), 7.40 – 7.43 (m, 1H), 7.30 (d, *J* = 8.0 Hz, 2H), 7.17 – 7.07 (m, 3H), 6.66 (dd, *J* = 17.6, 10.9 Hz, 1H), 5.71 (d, *J* = 17.6 Hz, 1H), 5.23 (d, *J* = 10.9 Hz, 1H), 4.86 (s, 2H), 3.65 (br, 3H).^{13}C NMR (150 MHz, $CDCl_3$) δ 189.56, 156.13, 137.26, 136.26, 135.90, 134.75,

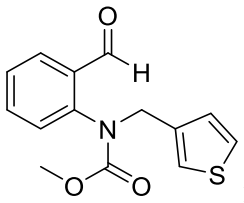
132.78, 129.33, 129.14, 128.79, 127.93, 126.42, 114.17, 54.80, 53.36. IR (neat, cm^{-1}): 2953.28, 2855.09, 2754.32, 1707.44, 1597.93, 1446.39, 1379.05, 1269.78, 1193.14, 990.30, 910.56, 779.90. HRMS (ESI) ($M+H^+$) Calcd. for $C18H18NO3^+$: 296.1281, found 296.1282.



methyl (2-formylphenyl)(3-phenylprop-2-yn-1-yl)carbamate s3-n Yield: 95%. Hexanes/ethyl acetate = 5/1. ^1H NMR (400 MHz, $CDCl_3$) δ 10.21 (s, 1H), 7.97 (dd, *J* = 7.7, 1.7 Hz, 1H), 7.70 – 7.66 (m, 1H), 7.50 (t, *J* = 7.5 Hz, 1H), 7.43 (d, *J* = 7.6 Hz, 1H), 7.31 – 7.27 (m, 5H), 4.85 and 4.67 (br, 2H), 3.67 (br, 3H). ^{13}C NMR (150 MHz, $CDCl_3$) δ 189.77, 155.48, 142.56, 134.87, 133.24, 131.45, 129.18, 128.84, 128.44, 128.38, 128.19, 122.12, 85.46, 83.36, 53.51, 41.33. IR (neat, cm^{-1}): 2955.69, 2861.87, 1698.24, 1597.94, 1489.13, 1375.69, 1443.87, 1271.73, 758.84. HRMS (ESI) ($M+H^+$) Calcd. for $C18H16NO3^+$: 294.1125, found 294.1126.

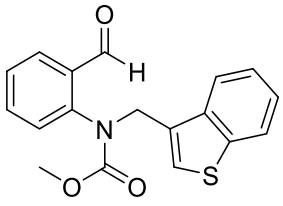


methyl (2-formylphenyl)(pyridin-2-ylmethyl)carbamate s3-o Yield: 50%. Hexanes/ethyl acetate = 1/1. ^1H NMR (600 MHz, $CDCl_3$) δ 10.03 (s, 1H), 8.49 (s, 1H), 7.85 (d, *J* = 7.5 Hz, 1H), 7.63 (td, *J* = 7.7, 1.7 Hz, 1H), 7.57 (t, *J* = 7.8 Hz, 1H), 7.41 – 7.37 (m, 2H), 7.31 (d, *J* = 7.7 Hz, 1H), 7.17 – 7.15 (m, 1H), 5.03 and 5.00 (br, 2H), 3.65 (br, 3H). ^{13}C NMR (150 MHz, $CDCl_3$) δ 190.08, 156.50, 156.12, 149.36, 143.70, 136.64, 134.73, 132.67, 129.34, 128.23, 127.77, 122.95, 122.57, 56.45, 53.40. IR (neat, cm^{-1}): 2955.07, 2854.05, 2359.77, 2343.66, 1711.48, 1598.24, 1486.57, 1459.02, 1310.65, 1271.14, 1193.08, 779.37. HRMS (ESI) ($M+H^+$) Calcd. for $C15H15N2O3^+$: 271.1077, found 271.1081.

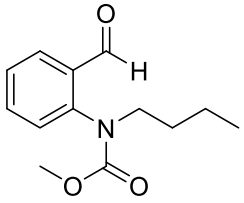


methyl (2-formylphenyl)(thiophen-3-ylmethyl)carbamate s3-p Yield: 99%. Hexanes/ethyl acetate = 5/1. ^1H NMR (600 MHz, $CDCl_3$) δ 9.69 (s, 1H), 7.86 (d, *J* = 7.6 Hz, 1H),

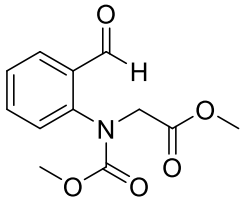
7.59 (td, *J* = 7.7, 1.4 Hz, 1H), 7.43 (t, *J* = 7.5 Hz, 1H), 7.25 – 7.24 (m, 1H), 7.13 (d, *J* = 7.7 Hz, 1H), 7.00 (d, *J* = 1.5 Hz, 1H), 6.97 (d, *J* = 2.2 Hz, 1H), 4.94 and 4.79 (br, 2H), 3.63 (br, 3H).^{13}C NMR (150 MHz, $CDCl_3$) δ 189.41, 155.90, 143.20, 136.85, 134.81, 132.96, 129.05, 128.82, 128.02, 126.42, 124.39, 53.32, 49.64. IR (neat, cm^{-1}): 2953.44, 2852.10, 2360.47, 2339.74, 1713.03, 1598.52, 1458.39, 1374.07, 1270.54, 1193.41, 738.61. HRMS (ESI) ($M+H^+$) Calcd. for $C14H14NO3S^+$: 276.0689, found 276.0691.



methyl (benzo[*b*]thiophen-3-ylmethyl)(2-formylphenyl)carbamate s3-q Yield: 80%. Hexanes/ethyl acetate = 5/1. ^{1}H NMR (600 MHz, $CDCl_3$) δ 9.63 (s, 1H), 7.83 – 7.80 (m, 3H), 7.53 (td, *J* = 7.5, 1.3 Hz, 1H), 7.41 (t, *J* = 7.4 Hz, 1H), 7.37 – 7.33 (m, 2H), 7.05 (s, 2H), 5.19 and 5.11 (br, 2H), 3.67 (s, 3H). ^{13}C NMR (150 MHz, $CDCl_3$) δ 189.30, 156.02, 142.75, 140.34, 137.86, 134.70, 133.09, 131.19, 129.39, 129.01, 128.12, 126.74, 124.62, 124.45, 122.87, 122.04, 53.48, 48.07. IR (neat, cm^{-1}): 2952.77, 2852.10, 1692.59, 1597.84, 1446.29, 1268.88, 1193.07, 768.87, 734.65. HRMS (ESI) ($M+Na^+$) Calcd. for $C18H15NNaO3S^+$: 348.0665, found 348.0666.

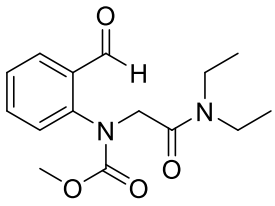


methyl butyl(2-formylphenyl)carbamate s3-r Yield: 50%. Hexanes/ethyl acetate = 5/1. ^{1}H NMR (600 MHz, $CDCl_3$) δ 10.10 (s, 1H), 7.92 (d, *J* = 7.7 Hz, 1H), 7.63 (t, *J* = 7.4 Hz, 1H), 7.44 (t, *J* = 7.5 Hz, 1H), 7.25 (s, 1H), 3.70 and 3.61 (br, 5H), 1.55 – 1.52 (m, 2H), 1.34 – 1.27 (m, 2H), 0.89 (t, *J* = 7.4 Hz, 3H). ^{13}C NMR (150 MHz, $CDCl_3$) δ 189.82, 155.81, 143.92, 134.83, 132.79, 129.22, 128.63, 127.72, 53.12, 51.21, 30.13, 19.98, 13.70. IR (neat, cm^{-1}): 2958.28, 2934.23, 2863.57, 1715.49, 1599.16, 1459.51, 1388.38, 1305.41, 1193.94, 772.69, 739.25. HRMS (ESI) ($M+H^+$) Calcd. for $C13H18NO3^+$: 236.1281, found 236.1283.

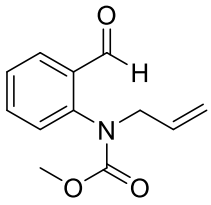


methyl *N*-(2-formylphenyl)-*N*-(methoxycarbonyl)glycinate s3-s Yield: 63%.

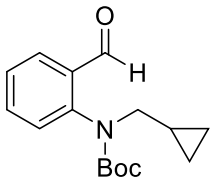
Hexanes/ethyl acetate = 2/1. ^{1}H NMR (600 MHz, $CDCl_3$) δ 10.27 (s, 1H), 7.92 (d, J = 7.8 Hz, 1H), 7.62 (t, J = 7.7, 1H), 7.47 – 7.43 (m, 2H), 4.53 (d, J = 17.6 Hz, 1H), 4.32 (d, J = 17.6 Hz, 1H), 3.75 (s, 3H), 3.81 and 3.67 (s, 3H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 190.20, 169.60, 155.86, 143.16, 134.86, 132.55, 130.12, 129.51, 128.18, 53.65, 52.32, 52.22. IR (neat, cm^{-1}): 2955.86, 2854.05, 1749.40, 1714.17, 1694.29, 1599.00, 1486.45, 1447.36, 1375.33, 1271.26, 1214.81, 776.76. HRMS (ESI) ($M+H^+$) Calcd. for $C12H14NO5^+$: 252.0866, found 252.0868.



methyl (2-(diethylamino)-2-oxoethyl)(2-formylphenyl)carbamate s3-t Yield: 78%. Hexanes/ethyl acetate = 2/1. ^{1}H NMR (500 MHz, $CDCl_3$) δ 10.30 (s, 1H), 7.87 (d, *J* = 7.5 Hz, 1H), 7.60 – 7.59 (m, 2H), 7.42 – 7.39 (m, 1H), 4.67 (d, *J* = 16.2 Hz, 1H), 4.21 (d, *J* = 16.2 Hz, 1H), 3.63 (s, 3H), 3.43 – 3.33 (br, m, 2H), 3.32 – 3.24 (br, m, 2H), 1.21 (t, *J* = 7.1 Hz, 3H), 1.10 (t, *J* = 7.1 Hz, 3H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 190.61, 166.65, 155.98, 143.86, 134.67, 132.48, 129.23, 128.56, 127.70, 53.39, 52.20, 41.18, 40.63, 14.14, 12.97. IR (neat, cm^{-1}): 2975.98, 2934.23, 1712.52, 1693.54, 1654.68, 1485.49, 1459.82, 1379.82, 1265.69, 1195.86, 760.72. HRMS (ESI) ($M+H^+$) Calcd. for $C15H21N2O4^+$: 293.1496, found 293.1498.

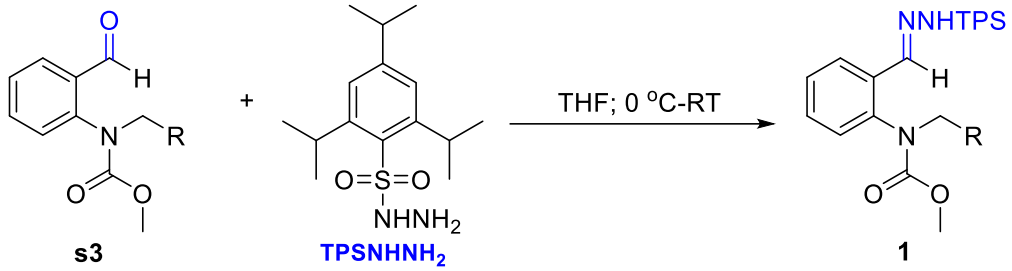


methyl allyl(2-formylphenyl)carbamate s3-u Yield: 99%. Hexanes/ethyl acetate = 5/1. ^{1}H NMR (600 MHz, $CDCl_3$) δ 10.06 (s, 1H), 7.88 (dd, *J* = 7.7, 1.6 Hz, 1H), 7.60 (t, *J* = 7.7, 1H), 7.42 (t, *J* = 7.5 Hz, 1H), 7.26 – 7.24 (m, 1H), 5.89 (ddt, *J* = 16.8, 10.2, 6.6 Hz, 1H), 5.12 – 5.07 (m, 2H), 4.28 (br, 2H), 3.61 (br, 3H). ^{13}C NMR (150 MHz, $CDCl_3$) δ 189.84, 155.72, 143.38, 134.78, 132.67, 132.49, 129.44, 128.67, 127.86, 119.14, 54.01, 53.26. IR (neat, cm^{-1}): 2969.80, 1721.22, 1599.12, 1456.18, 1375.60, 1229.57. HRMS (ESI) ($M+H^+$) Calcd. for $C12H14NO3^+$: 220.0968, found 220.0974.

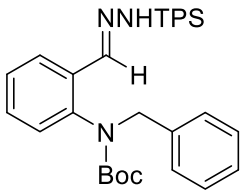


***tert*-butyl (cyclopropylmethyl)(2-formylphenyl)carbamate s3-v** Yield: 45%. Hexanes/ethyl acetate = 6/1. ^{1}H NMR (600 MHz, $CDCl_3$) δ 10.22 (s, 1H), 7.91 (d, *J* = 7.5 Hz, 1H), 7.62 – 7.60 (m, 1H), 7.40 (t, *J* = 7.5 Hz, 1H), 7.26 (br, 1H), 3.81 (s, 1H), 3.38 (s, 1H), 1.59 – 1.31 (m, 9H), 0.97 (br, 1H), 0.43 (br, 2H), 0.15 (s, 1H), 0.03 (s, 1H). ^{13}C NMR (150 MHz, $CDCl_3$) major rotamer: δ 190.46, 154.43, 145.09, 134.70, 133.27, 129.82, 128.31, 127.31, 80.94, 54.87, 29.70, 28.15, 9.96, 3.73. IR (neat, cm^{-1}): 2979.07, 2929.87, 2855.13, 2760.04, 1684.56, 1596.35, 1429.78, 1369.43, 1291.66, 1149.82, 1129.13, 974.20, 760.89. HRMS (ESI) ($M+Na^+$) Calcd. for $C16H21NNaO3^+$: 298.1414, found 298.1413.

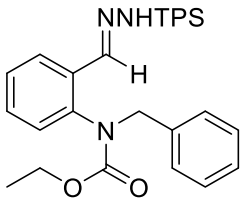
IX. The Synthetic Procedure for Triisopropyl Sulfonylhydrazone Derivatives 1



To a stirred solution of pure 2,4,6-triisopropylbenzenesulfonohydrazide ($TPSNHNH_2$, 2 mmol) in THF (10.0 mL) at 0 °C, aldehyde **s3** (1 equiv.) was added dropwise (or portionwise if solid). The reaction was monitored by TLC. After the reaction was completed, the solvent was removed directly under reduced pressure, and the crude solid was further purified by flash column chromatography.

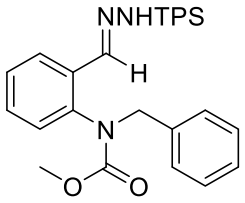


***tert*-butyl benzyl(2-((2-((2,4,6-triisopropylphenyl)sulfonyl)hydrazono) methyl)phenyl)carbamate 1-a** Yield: 70%. Hexanes/ethyl acetate = 6/1. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.98 (s, 1H), 7.80 (d, *J* = 7.6 Hz, 1H), 7.47 (s, 1H), 7.33 (br, 1H), 7.24 – 7.14 (m, 8H), 6.89 (s, 1H), 4.76 (d, *J* = 14.7 Hz, 1H), 4.57 (d, *J* = 14.7 Hz, 1H), 4.26 (hept, *J* = 6.7 Hz, 2H), 2.90 (hept, *J* = 6.9 Hz, 1H), 1.33 – 1.25 (m, 27H).^{13}C NMR (125 MHz, $CDCl_3$) δ 154.82, 153.38, 151.36, 142.47, 141.10, 137.24, 131.38, 131.01, 130.54, 128.84, 128.48, 127.75, 127.49, 127.27, 126.38, 123.82, 80.86, 54.10, 34.17, 30.01, 28.20, 24.89, 23.53. IR (neat, cm^{-1}): 3178.54, 2963.62, 1669.48, 1600.75, 1399.42, 1315.89, 1157.20, 1071.86, 855.44, 757.16, 731.40. HRMS (ESI) ($M+H^+$) Calcd. for $C_{34}H_{46}N_3O_4S^+$: 592.3249, found 592.3274.

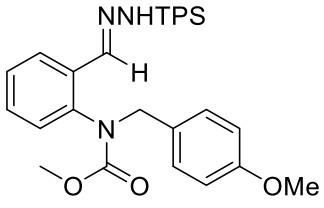


ethyl benzyl(2-((2-((2,4,6-triisopropylphenyl)sulfonyl)hydrazono) methyl) phenyl) carbamate 1-b Yield: 86%. Hexanes/ethyl acetate = 4/1. ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.10 (s, 1H), 7.82 (dd, *J* = 7.8, 1.4 Hz, 1H), 7.45 (s, 1H), 7.29 – 7.20 (m, 5H), 7.17 – 7.14 (m, 4H),

6.91 (s, 1H), 4.79 and 4.76 (br, 1H), 4.63 and 4.61 (br, 1H), 4.25 (hept, J = 6.7 Hz, 2H), 4.03 (br, 2H), 2.89 (hept, J = 6.9 Hz, 1H), 1.30 – 1.27 (m, 12H), 1.25 (d, J = 6.9 Hz, 6H), 0.99 (br, 3H). ^{13}C NMR (150 MHz, $CDCl_3$) δ 155.87, 153.38, 151.34, 141.87, 140.22, 136.72, 131.42, 131.14, 130.62, 129.05, 128.53, 127.94, 127.66, 126.52, 123.99, 123.81, 62.12, 54.74, 34.17, 30.00, 24.88, 23.52, 14.46. IR (neat, cm^{-1}): 2960.77, 2869.70, 1673.95, 1600.11, 1455.15, 1319.36, 1297.65, 1166.56, 1037.84, 942.18, 743.78, 657.99. HRMS (ESI) ($M+H^+$) Calcd. for $C_{32}H_{42}N_3O_4S^+$: 564.2891, found 564.2876.

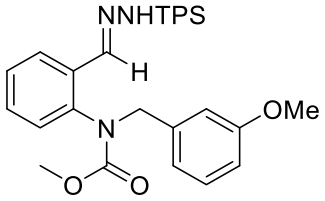


methyl benzyl(2-((2-((2,4,6-triisopropylphenyl)sulfonyl)hydrazono) methyl)phenyl) carbamate 1-c Yield: 90%. Hexanes/ethyl acetate = 4/1. ^{1}H NMR (600 MHz, $CDCl_3$) δ 8.13 (s, 1H), 7.82 (d, J = 7.8 Hz, 1H), 7.46 (s, 1H), 7.28 (td, J = 7.6, 1.4 Hz, 1H), 7.25 – 7.22 (m, 4H), 7.18 (s, 2H), 7.15 (br, 2H), 6.91 (d, J = 6.5 Hz, 1H), 4.75 (d, J = 14.5 Hz, 1H), 4.65 (d, J = 14.5 Hz, 1H), 4.24 (hept, J = 6.7 Hz, 2H), 3.54 (s, 3H), 2.90 (hept, J = 6.9 Hz, 1H), 1.30 – 1.28 (m, 12H), 1.25 (d, J = 6.9 Hz, 6H). ^{13}C NMR (150 MHz, $CDCl_3$) δ 156.31, 153.41, 151.33, 141.80, 140.04, 136.54, 131.36, 131.18, 130.71, 129.12, 128.67, 128.56, 128.03, 127.83, 126.58, 123.82, 54.93, 53.26, 34.17, 30.00, 24.87, 23.52. IR (neat, cm^{-1}): 3213.94, 2958.2, 2869.64, 1696.82, 1601.14, 1321.40, 1166.54, 1152.04. HRMS (ESI) ($M+H^+$) Calcd. for $C_{31}H_{40}N_3O_4S^+$: 550.2734, found 550.2740.

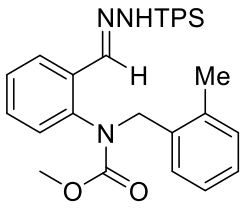


methyl (4-methoxybenzyl)(2-((2-((2,4,6-triisopropylphenyl) sulfonyl)hydrazono)methyl)phenyl)carbamate 1-d Yield: 87%. Hexanes/ethyl acetate = 3/1. ^{1}H NMR (600 MHz, $CDCl_3$) δ 8.24 (s, 1H), 7.82 (d, J = 7.8 Hz, 1H), 7.51 (s, 1H), 7.31 – 7.27 (m, 1H), 7.24 – 7.20 (m, 1H), 7.18 (s, 2H), 7.07 (d, J = 7.5 Hz, 2H), 6.90 – 6.86 (m, 1H), 6.78 (d, J = 8.5 Hz, 2H), 4.75 (d, J = 14.3 Hz, 1H), 4.51 (d, J = 14.3 Hz, 1H), 4.25 (hept, J = 6.7 Hz, 2H), 3.77 (s, 3H), 3.52 (s, 3H), 2.89 (hept, J = 6.9 Hz, 1H), 1.29 (d, J = 6.7 Hz, 12H), 1.25 (d, J = 6.9 Hz, 6H). ^{13}C NMR (150 MHz, $CDCl_3$) δ 159.30, 156.29, 153.38, 151.33, 141.79, 140.01, 131.38,

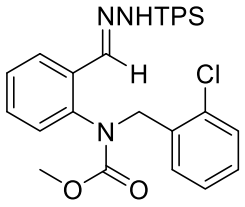
131.15, 130.61, 130.44, 128.82, 128.64, 127.77, 126.52, 123.80, 113.87, 55.19, 54.29, 53.20, 34.16, 29.99, 24.85, 23.52. IR (neat, cm^{-1}): 3161.77, 2957.90, 2868.98, 2359.85, 2342.17, 1704.86, 1681.69, 1456.72. HRMS (ESI) ($M+H^+$) Calcd. for $C_{32}H_{42}N_3O_5S^+$: 580.2840, found 580.2839.



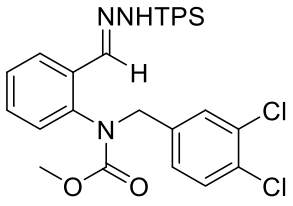
methyl (3-methoxybenzyl)(2-((2-((2,4,6-triisopropylphenyl) sulfonyl) hydrazono)methyl)phenyl)carbamate 1-e Yield: 94%. Hexanes/ethyl acetate = 4/1. ^1H NMR (600 MHz, $CDCl_3$) δ 8.18 (s, 1H), 7.82 (dd, *J* = 7.8, 1.5 Hz, 1H), 7.50 (s, 1H), 7.27 (ddd, *J* = 5.8, 4.9, 1.6 Hz, 1H), 7.23 (td, *J* = 7.6, 0.9 Hz, 1H), 7.18 – 7.15 (m, 3H), 6.91 (br, 1H), 6.79 (dd, *J* = 8.2, 2.3 Hz, 1H), 6.73 (d, *J* = 7.0 Hz, 1H), 6.69 (s, 1H), 4.76 (d, *J* = 14.5 Hz, 1H), 4.56 (d, *J* = 14.5 Hz, 1H), 4.24 (hept, *J* = 6.7 Hz, 2H), 3.72 (s, 3H), 3.53 (s, 3H), 2.90 (hept, *J* = 6.9 Hz, 1H), 1.28 (d, *J* = 6.7 Hz, 12H), 1.24 (d, *J* = 6.9 Hz, 6H). ^{13}C NMR (125MHz, $CDCl_3$) δ 159.62, 156.32, 153.40, 151.33, 141.80, 140.03, 138.04, 131.40, 131.17, 130.67, 129.58, 128.68, 127.84, 126.56, 123.81, 121.30, 114.79, 113.31, 55.19, 54.85, 53.27, 34.17, 30.00, 24.86, 23.52. IR (neat, cm^{-1}): 3182.56, 2958.18, 2932.27, 2869.32, 2359.69, 2343.66, 1706.33, 1679.61, 1454.13, 1263.22, 1153.49, 735.74. HRMS (ESI) ($M+H^+$) Calcd. for $C_{32}H_{42}N_3O_5S^+$: 580.2840, found 580.2839.



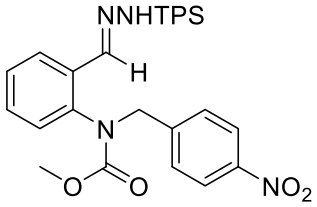
methyl (2-methylbenzyl)(2-((2-((2,4,6-triisopropylphenyl)sulfonyl) hydrazono)methyl)phenyl)carbamate 1-f Yield: 94%. Hexanes/ethyl acetate = 5/1. ^1H NMR (500 MHz, $CDCl_3$) δ 8.06 (br, 1H), 7.80 (dd, *J* = 7.4, 2.0 Hz, 1H), 7.47 (s, 1H), 7.23 (td, *J* = 7.6, 1.5 Hz, 2H), 7.18 (s, 2H), 7.10 (t, *J* = 7.3 Hz, 2H), 7.04 (t, *J* = 7.6 Hz, 2H), 6.87 (br, 1H), 4.87 (d, *J* = 14.4 Hz, 1H), 4.69 (d, *J* = 14.4 Hz, 1H), 4.24 (hept, *J* = 6.7 Hz, 2H), 3.56 (br, 3H), 2.90 (hept, *J* = 6.9 Hz, 1H), 2.01 (s, 3H), 1.28 (br, 12H), 1.25 (d, *J* = 6.9 Hz, 6H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 158.95, 156.05, 153.99, 144.33, 142.49, 139.24, 137.15, 134.05, 133.29, 133.11, 132.80, 131.38, 130.74, 130.50, 129.21, 128.72, 126.66, 126.48, 55.95, 54.43, 36.83, 32.66, 27.53, 26.19, 21.59. IR (neat, cm^{-1}): 3184.54, 2957.29, 2868.69, 2360.21, 2342.60, 1706.76, 1676.98, 1456.36, 1152.06, 746.98, 658.54. HRMS (ESI) ($M+H^+$) Calcd. for $C_{32}H_{42}N_3O_4S^+$: 564.2891, found 564.2895.



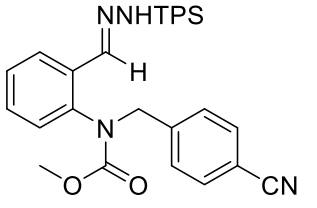
methyl (2-chlorobenzyl)(2-((2-((2,4,6-triisopropylphenyl)sulfonyl) hydrazono)methyl)phenyl)carbamate 1-g Yield: 85%. Hexanes/ethyl acetate = 5/1. ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.96 (s, 1H), 7.77 (d, *J* = 7.8 Hz, 1H), 7.60 (s, 1H), 7.28 – 7.25 (m, 1H), 7.21 (t, *J* = 6.9 Hz, 3H), 7.17 (s, 2H), 7.13 – 7.10 (m, 2H), 6.98 (br, 1H), 4.90 and 4.88 (br, 2H), 4.23 (hept, *J* = 6.6 Hz, 2H), 3.57 (s, 3H), 2.90 (hept, *J* = 6.7 Hz, 1H), 1.29 (br, 12H), 1.24 (d, *J* = 6.9 Hz, 6H). ^{13}C NMR (150 MHz, $CDCl_3$) δ 156.27, 153.36, 151.31, 141.58, 139.60, 133.99, 133.80, 131.44, 131.40, 131.24, 130.55, 129.54, 129.36, 128.31, 127.84, 126.88, 126.53, 123.82, 53.41, 51.72, 34.17, 30.00, 24.86, 23.52. IR (neat, cm^{-1}): 3162.51, 2958.56, 2929.17, 2868.95, 2360.16, 2342.85, 1684.03, 1599.56, 1456.61, 1383.67, 1166.61, 739.94. HRMS (ESI) ($M+H^+$) Calcd. for $C31H39ClN3O4S^+$: 584.2344, found 584.2347.



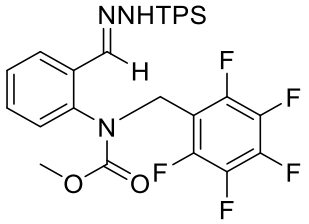
methyl (3,4-dichlorobenzyl)(2-((2-((2,4,6-triisopropylphenyl)sulfonyl) hydrazono)methyl)phenyl)carbamate 1-h Yield: 85%. Hexanes/ethyl acetate = 4/1. ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.99 (s, 1H), 7.83 (d, *J* = 7.0 Hz, 1H), 7.62 (s, 1H), 7.32 (d, *J* = 8.2 Hz, 1H), 7.30 – 7.27 (m, 3H), 7.19 (s, 2H), 6.98 (d, *J* = 7.0 Hz, 1H), 6.82 (br, 1H), 4.92 (d, *J* = 14.3 Hz, 1H), 4.38 – 4.22 (m, 3H), 3.56 (br, 3H), 2.90 (hept, *J* = 6.9 Hz, 1H), 1.29 (d, *J* = 6.5 Hz, 12H), 1.24 (d, *J* = 6.9 Hz, 6H). ^{13}C NMR (125MHz, $CDCl_3$) δ 156.38, 153.53, 151.35, 141.26, 139.61, 136.89, 132.58, 132.10, 131.28, 130.83, 130.69, 130.53, 128.72, 128.21, 128.12, 127.11, 123.87, 53.69, 53.45, 34.18, 30.03, 24.85, 23.51. IR (neat, cm^{-1}): 3182.21, 2959.02, 2869.36, 1708.61, 1680.94, 1455.62, 1374.93, 1151.17, 1033.78. HRMS (ESI) ($M+H^+$) Calcd. for $C31H38Cl2N3O4S^+$: 618.1955, found 618.1960.



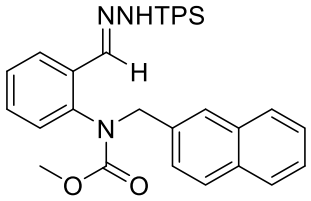
methyl (4-nitrobenzyl)(2-((2-((2,4,6-triisopropylphenyl)sulfonyl) hydrazono)methyl)phenyl)carbamate 1-i Yield: 50%. Hexanes/ethyl acetate = 4/1. ^{1}H NMR (600 MHz, $CDCl_3$) δ 8.46 (s, 1H), 8.12 (d, *J* = 8.5 Hz, 2H), 7.83 (br, 1H), 7.72 (s, 1H), 7.35 (d, *J* = 8.0 Hz, 2H), 7.27 – 7.25 (m, 2H), 7.19 (s, 2H), 6.80 (s, 1H), 5.10 (d, *J* = 14.4 Hz, 1H), 4.39 (d, *J* = 14.4 Hz, 1H), 4.25 (hept, *J* = 6.7 Hz, 2H), 3.58 (br, 3H), 2.90 (hept, *J* = 6.9 Hz, 1H), 1.29 (d, *J* = 6.7 Hz, 12H), 1.25 (d, *J* = 6.9 Hz, 6H). ^{13}C NMR (150 MHz, $CDCl_3$) δ 156.38, 153.63, 151.34, 147.57, 143.91, 141.15, 139.57, 131.17, 130.77, 130.75, 129.68, 128.59, 128.21, 127.23, 123.89, 123.80, 54.07, 53.53, 34.18, 30.02, 24.83, 23.51. IR (neat, cm^{-1}): 3218.81, 2960.23, 2359.80, 1670.43, 1519.94, 1346.84, 738.21. HRMS (ESI) ($M+H^+$) Calcd. for $C31H39N4O6S^+$: 595.2585, found 595.2587.



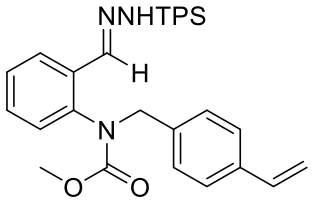
methyl (4-cyanobenzyl)(2-((2-((2,4,6-triisopropylphenyl)sulfonyl) hydrazono)methyl)phenyl)carbamate 1-j Yield: 80%. Hexanes/ethyl acetate = 3/1. ^{1}H NMR (600 MHz, CDCl3) 8.44 (br, 1H), 7.82 (br, 1H), 7.71 (s, 1H), 7.55 (d, *J* = 8.3 Hz, 2H), 7.29 – 7.24 (m, 4H), 7.19 (s, 2H), 6.79 (br, 1H), 5.05 (d, *J* = 14.6 Hz, 1H), 4.35 (d, *J* = 14.6 Hz, 1H), 4.26 (hept, *J* = 6.7 Hz, 2H), 3.57 (br, 3H), 2.91 (hept, *J* = 6.9 Hz, 1H), 1.30 (d, *J* = 6.7 Hz, 12H), 1.25 (d, *J* = 6.9 Hz, 6H). ^{13}C NMR (150 MHz, $CDCl_3$) δ 156.42, 153.60, 151.34, 141.92, 141.17, 139.60, 132.37, 131.23, 130.81, 130.68, 129.51, 128.56, 128.15, 127.17, 123.88, 118.44, 111.87, 54.37, 53.50, 34.17, 30.02, 24.85, 23.51. IR (neat, cm^{-1}): 3190.41, 2959.79, 2870.26, 2360.11, 2343.40, 1706.75, 1684.15, 1265.40, 742.06. HRMS (ESI) ($M+H^+$) Calcd. for $C32H39N4O4S^+$: 575.2687, found 575.2691.



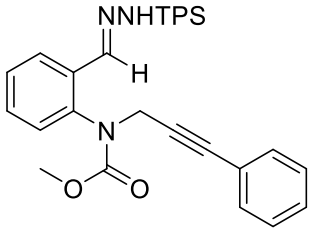
methyl ((perfluorophenyl)methyl)(2-((2-((2,4,6-triisopropylphenyl) sulfonyl)hydrazono)methyl)phenyl)carbamate 1-k Yield: 92%. Hexanes/ethyl acetate = 4/1. ^{1}H NMR (600 MHz, $CDCl_3$) δ 8.28 (s, 1H), 7.80 (d, *J* = 7.6 Hz, 1H), 7.71 (s, 1H), 7.31 (td, *J* = 7.6, 1.4 Hz, 1H), 7.27 – 7.25 (m, 1H), 7.18 (s, 2H), 6.98 (d, *J* = 7.8 Hz, 1H), 4.96 (d, *J* = 14.3 Hz, 1H), 4.84 (d, *J* = 14.3 Hz, 1H), 4.24 (hept, *J* = 6.7 Hz, 2H), 3.58 (br, 3H), 2.90 (hept, *J* = 6.9 Hz, 1H), 1.29 (d, *J* = 6.7 Hz, 12H), 1.25 (d, *J* = 6.9 Hz, 6H). ^{13}C NMR (150 MHz, $CDCl_3$) δ 155.78, 153.51, 151.36, 145.39 (md, *J* = 253.0 Hz), 141.12 (md, *J* = 251.6 Hz), 140.78, 138.56, 137.31 (md, *J* = 253.7 Hz), 131.32, 131.22, 130.73, 128.35, 128.04, 127.05, 123.85, 109.61, 53.61, 41.62, 34.18, 30.02, 24.80, 23.50. IR (neat, cm^{-1}): 3170.74, 2959.60, 2869.98, 1714.25, 1687.55, 1504.90, 1036.45, 738.86. HRMS (ESI) ($M+H^+$) Calcd. for $C_{31}H_{35}F_5N_3O_4S^+$: 640.2263, found 640.2264.



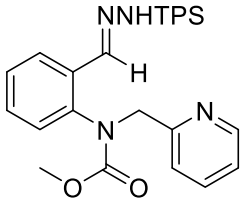
methyl (naphthalen-2-ylmethyl)(2-((2-((2,4,6-triisopropylphenyl) sulfonyl)hydrazono)methyl)phenyl)carbamate 1-l Yield: 96%. Hexanes/ethyl acetate = 5/1. ^{1}H NMR (600 MHz, $CDCl_3$) δ 8.04 (br, 1H), 7.83 – 7.82 (m, 1H), 7.80 – 7.79 (m, 1H), 7.76 (d, *J* = 8.4 Hz, 1H), 7.73 – 7.71 (m, 1H), 7.61 (s, 1H), 7.55 (s, 1H), 7.45 (p, *J* = 7.6 Hz, 2H), 7.34 (d, *J* = 7.5 Hz, 1H), 7.24 – 7.20 (m, 2H), 7.17 (s, 2H), 6.84 (br, 1H), 5.09 (d, *J* = 14.6 Hz, 1H), 4.59 (d, *J* = 14.6 Hz, 1H), 4.23 (hept, *J* = 6.6 Hz, 2H), 3.55 (br, 3H), 2.89 (hept, *J* = 6.9 Hz, 1H), 1.27 (d, *J* = 6.6 Hz, 12H), 1.24 (d, *J* = 6.9 Hz, 6H).^{13}C NMR (150 MHz, $CDCl_3$) δ 156.53, 153.38, 151.32, 141.58, 139.98, 134.01, 133.12, 132.83, 131.40, 131.00, 130.56, 128.77, 128.40, 128.03, 127.84, 127.66, 126.66, 126.25, 126.14, 123.81, 54.99, 53.34, 34.16, 29.99, 24.84, 23.52. IR (neat, cm^{-1}): 3169.11, 2956.97, 2867.76, 2359.96, 1679.88, 1600.27, 1152.06, 751.50. HRMS (ESI) ($M+H^+$) Calcd. for $C_{35}H_{42}N_3O_4S^+$: 600.2891, found 600.2891.



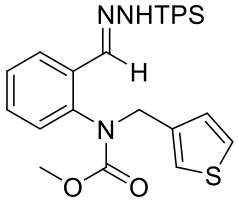
methyl (2-((2-((2,4,6-triisopropylphenyl)sulfonyl)hydrazono)methyl) phenyl)(4-vinylbenzyl)carbamate 1-m Yield: 85%. Hexanes/ethyl acetate = 4/1. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.90 (s, 1H), 7.82 (dd, *J* = 7.7, 1.7 Hz, 1H), 7.50 (s, 1H), 7.30 (d, *J* = 8.0 Hz, 2H), 7.27 – 7.22 (m, 2H), 7.17 (s, 2H), 7.11 (d, *J* = 7.7 Hz, 2H), 6.88 (d, *J* = 7.5 Hz, 1H), 6.67 (dd, *J* = 17.6, 10.9 Hz, 1H), 5.72 (d, *J* = 17.6 Hz, 1H), 5.25 (d, *J* = 10.9 Hz, 1H), 4.82 (d, *J* = 14.5 Hz, 1H), 4.51 (d, *J* = 14.5 Hz, 1H), 4.23 (hept, *J* = 6.7 Hz, 2H), 3.53 (br, 3H), 2.89 (hept, *J* = 6.9 Hz, 1H), 1.28 (d, *J* = 6.7 Hz, 12H), 1.24 (d, *J* = 6.9 Hz, 6H). ^{13}C NMR (150 MHz, $CDCl_3$) δ 156.37, 153.40, 151.79, 151.33, 141.65, 139.99, 137.26, 136.20, 131.37, 131.03, 130.60, 129.23, 128.73, 126.66, 126.37, 123.99, 123.81, 114.27, 54.58, 53.28, 34.16, 29.99, 24.85, 23.52. IR (neat, cm^{-1}): 3182.58, 2959.56, 2869.56, 1682.03, 1456.25, 1384.34, 1265.36, 742.69. HRMS (ESI) ($M+H^+$) Calcd. for $C33H42N3O4S^+$: 576.2891, found 576.2895.



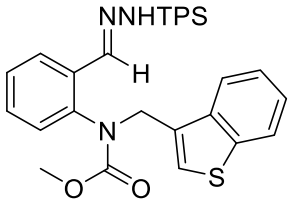
methyl (3-phenylprop-2-yn-1-yl)(2-((2-((2,4,6-triisopropylphenyl) sulfonyl)hydrazono)methyl)phenyl)carbamate 1-n Yield: 76%. Hexanes/ethyl acetate = 5/1. ^{1}H NMR (600 MHz, $CDCl_3$) δ 8.50 (s, 1H), 7.99 (s, 1H), 7.94 (d, *J* = 7.4 Hz, 1H), 7.40 (t, *J* = 7.5 Hz, 1H), 7.33 – 7.28 (m, 5H), 7.23 (d, *J* = 6.5 Hz, 2H), 7.18 (s, 2H), 4.60 (s, 2H), 4.27 (hept, *J* = 6.7 Hz, 2H), 3.56 (s, 3H), 2.91 (hept, *J* = 6.9 Hz, 1H), 1.29 (d, *J* = 6.7 Hz, 12H), 1.26 (d, *J* = 6.9 Hz, 6H). ^{13}C NMR (150 MHz, $CDCl_3$) δ 155.83, 153.36, 151.34, 141.74, 139.40, 131.70, 131.51, 130.78, 128.48, 128.39, 128.32, 128.27, 126.54, 123.83, 122.16, 85.33, 83.48, 53.52, 41.22, 34.18, 30.02, 24.82, 23.53. IR (neat, cm^{-1}): 3188.68, 2960.16, 2868.25, 1682.88, 1600.66, 1454.10, 1280.56, 1167.12, 755.85. HRMS (ESI) ($M+H^+$) Calcd. for $C33H40N3O4S^+$: 574.2734, found 574.2732.



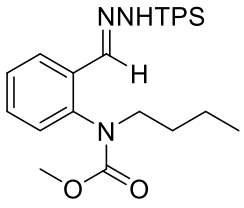
methyl (pyridin-2-ylmethyl)(2-((2-((2,4,6-triisopropylphenyl)sulfonyl) hydrazono)methyl)phenyl)carbamate 1-o Yield: 76%. Hexanes/ethyl acetate = 2/1. ^{1}H NMR (600 MHz, $CDCl_3$) δ 8.48 (s, 1H), 8.33 (s, 1H), 7.89 (s, 1H), 7.81 (d, *J* = 7.8 Hz, 1H), 7.57 (td, *J* = 7.7, 1.4 Hz, 1H), 7.28 (t, *J* = 7.6 Hz, 1H), 7.21 (t, *J* = 7.6 Hz, 2H), 7.17 (s, 2H), 7.13 – 7.08 (m, 2H), 4.86 (br, 2H), 4.27 (hept, *J* = 6.7 Hz, 2H), 3.55 (s, 3H), 2.89 (hept, *J* = 6.9 Hz, 1H), 1.29 (d, *J* = 6.7 Hz, 12H), 1.25 (d, *J* = 6.9 Hz, 6H). ^{13}C NMR (150 MHz, $CDCl_3$) δ 156.30, 153.31, 151.31, 149.35, 142.27, 140.43, 136.62, 131.53, 131.26, 130.63, 128.09, 127.73, 126.56, 123.80, 123.68, 122.98, 122.65, 56.27, 53.34, 34.17, 30.00, 24.88, 23.53. IR (neat, cm^{-1}): 3208.37, 2957.41, 2867.81, 2359.66, 2341.70, 1683.12, 1596.95, 1445.71, 1375.62, 1164.31, 737.28. HRMS (ESI) ($M+H^+$) Calcd. for $C30H39N4O4S^+$: 551.2687, found 551.2693.



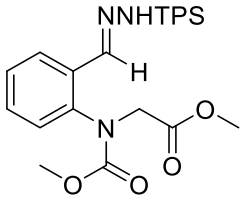
methyl (thiophen-3-ylmethyl)(2-((2-((2,4,6-triisopropylphenyl)sulfonyl) hydrazono)methyl)phenyl)carbamate 1-p Yield: 85%. Hexanes/ethyl acetate = 4/1. ^{1}H NMR (600 MHz, $CDCl_3$) δ 8.04 (s, 1H), 7.84 (d, *J* = 7.7 Hz, 1H), 7.39 (s, 1H), 7.31 (td, *J* = 7.6, 1.3 Hz, 1H), 7.26 – 7.24 (m, 1H), 7.21 – 7.20 (m, 1H), 7.18 (s, 2H), 7.00 – 6.92 (m, 3H), 4.73 – 4.65 (m, 2H), 4.25 (hept, *J* = 6.7 Hz, 2H), 3.53 (s, 3H), 2.90 (hept, *J* = 6.9 Hz, 1H), 1.29 (d, *J* = 6.7 Hz, 12H), 1.25 (d, *J* = 6.9 Hz, 6H). ^{13}C NMR (150 MHz, $CDCl_3$) δ 156.12, 153.42, 151.35, 141.73, 139.99, 137.08, 131.39, 131.30, 130.75, 128.60, 128.30, 127.90, 126.53, 126.25, 124.57, 123.83, 53.24, 49.37, 34.18, 30.02, 24.89, 23.53. IR (neat, cm^{-1}): 3196.36, 2957.33, 2868.55, 1681.89, 1454.44, 1374.11, 1153.88, 768.53, 588.75. HRMS (ESI) ($M+H^+$) Calcd. for $C29H38N3O4S2^+$: 556.2298, found 556.2301.



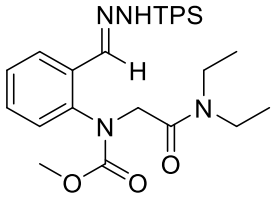
methyl (benzo[*b*]thiophen-3-ylmethyl)(2-((2-((2,4,6-triisopropyl phenyl) sulfonyl)hydrazono)methyl)phenyl)carbamate 1-q Yield: 80%. Hexanes/ethyl acetate = 5/1. ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.91 (br, 1H), 7.87 – 7.86 (m, 1H), 7.72 (d, *J* = 7.8 Hz, 1H), 7.40 (p, *J* = 6.5 Hz, 2H), 7.29 – 7.26 (m, 1H), 7.22 (t, *J* = 7.5 Hz, 1H), 7.15 (s, 2H), 7.08 (br, 2H), 6.94 (s, 1H), 6.91 (d, *J* = 7.3 Hz, 1H), 5.21 (d, *J* = 14.7Hz, 1H), 4.82 (d, *J* = 14.7 Hz, 1H), 4.14 (hept, *J* = 6.7 Hz, 2H), 3.54 (br, 3H), 2.88 (hept, *J* = 6.9 Hz, 1H), 1.28 – 1.23 (m, 18H). ^{13}C NMR (150 MHz, $CDCl_3$) δ 156.25, 153.32, 151.27, 141.60, 140.33, 139.41, 138.01, 131.55, 131.36, 131.01, 130.62, 128.67, 127.94, 127.09, 126.35, 124.58, 124.41, 123.75, 123.11, 122.07, 53.35, 47.86, 34.13, 29.93, 24.85, 23.50. IR (neat, cm^{-1}): 3170.74, 2959.60, 2869.98, 1714.25, 1687.55, 1521.47, 1504.90, 1122.65, 944.15, 738.86. HRMS (ESI) ($M+H^+$) Calcd. for $C33H40N3O4S2^+$: 606.2455, found 606.2456.



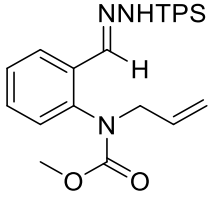
methyl butyl(2-((2-((2,4,6-triisopropylphenyl)sulfonyl)hydrazono)methyl) phenyl)carbamate 1-r Yield: 67%. Hexanes/ethyl acetate = 5/1. ^{1}H NMR (600 MHz, $CDCl_3$) δ 8.91 (s, 1H), 7.90 (d, *J* = 8.0 Hz, 1H), 7.88 (s, 1H), 7.37 – 7.35 (m, 1H), 7.28 – 7.26 (m, 1H), 7.18 (s, 2H), 7.10 (d, *J* = 7.8 Hz, 1H), 4.30 (hept, *J* = 6.7 Hz, 2H), 3.67 (br, 1H), 3.54 (s, 3H), 3.41 (br, 1H), 2.90 (hept, *J* = 6.9 Hz, 1H), 1.48 – 1.43 (m, 2H), 1.30 (d, *J* = 6.7 Hz, 12H), 1.29 – 1.27 (m, 2H), 1.25 (d, *J* = 6.9 Hz, 6H), 0.84 (t, *J* = 7.4 Hz, 3H). ^{13}C NMR (150 MHz, $CDCl_3$) δ 156.18, 153.30, 151.27, 141.28, 140.26, 131.56, 131.18, 130.61, 128.50, 127.69, 126.48, 123.79, 53.14, 51.01, 34.17, 29.98, 29.78, 24.87, 23.52, 19.92, 13.68. IR (neat, cm^{-1}): 3155.25, 2956.43, 2868.74, 2359.98, 2342.85, 1682.72, 1601.37, 1456.82, 1315.52, 1152.59, 555.64. HRMS (ESI) ($M+H^+$) Calcd. for $C28H42N3O4S^+$: 516.2891, found 516.2898.



methyl *N*-(methoxycarbonyl)-*N*-(2-((2-((2,4,6-triisopropylphenyl) sulfonyl) hydrazono)methyl)phenyl)glycinate 1-s Yield: 90%. Hexanes/ethyl acetate = 3/1. ^{1}H NMR (600 MHz, $CDCl_3$) δ 8.34(s, 1H), 8.17 (s, 1H), 7.88 (d, *J* = 7.8 Hz, 1H), 7.35 (td, *J* = 7.5, 1.1 Hz, 1H), 7.28 – 7.24 (m, 2H), 7.18 (s, 2H), 4.31 – 4.18 (m, 3H), 3.78 (d, *J* = 11.2 Hz, 1H), 3.70 (s, 3H), 3.56 (s, 3H), 2.89 (hept, *J* = 6.9 Hz, 1H), 1.30 (d, *J* = 6.7 Hz, 12H), 1.24 (d, *J* = 6.9 Hz, 6H). ^{13}C NMR (150 MHz, $CDCl_3$) δ 169.56, 156.15, 153.32, 151.30, 142.46, 140.38, 131.52, 131.32, 130.83, 128.07, 127.84, 126.70, 123.80, 53.57, 52.28, 52.24, 34.16, 30.00, 24.85, 23.52. IR (neat, cm^{-1}): 3176.72, 3057.43, 2958.72, 1752.67, 1692.37, 1599.82, 1264.81, 1165.49, 734.33, 703.22. HRMS (ESI) ($M+H^+$) Calcd. for $C_{27}H_{38}N_3O_6S^+$: 532.2476, found 532.2479.

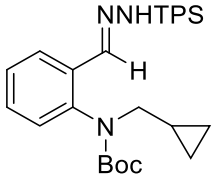


methyl (2-(diethylamino)-2-oxoethyl)(2-((2-((2,4,6-triisopropylphenyl) sulfonyl)hydrazono)methyl)phenyl)carbamate 1-t Yield: 78%. Hexanes/ethyl acetate = 1/1. ^{1}H NMR (600 MHz, $CDCl_3$) δ 8.46 (s, 1H), 8.38 (s, 1H), 7.87 (d, *J* = 7.8 Hz, 1H), 7.38 – 7.32 (m, 2H), 7.24 – 7.23 (m, 1H), 7.17 (s, 2H), 4.36 – 4.28 (m, 4H), 3.52 (s, 3H), 3.38 – 3.30 (m, 2H), 3.26 – 3.18 (m, 2H), 2.89 (hept, *J* = 6.9 Hz, 1H), 1.30 (t, *J* = 6.7 Hz, 12H), 1.24 (d, *J* = 6.9 Hz, 6H), 1.16 (t, *J* = 7.1 Hz, 3H), 1.08 (t, *J* = 7.1 Hz, 3H). ^{13}C NMR (150 MHz, $CDCl_3$) δ 166.43, 156.25, 153.19, 151.29, 143.43, 141.16, 131.68, 131.51, 130.64, 128.11, 127.67, 126.48, 123.76, 53.40, 52.33, 41.12, 40.67, 34.15, 29.98, 24.88, 23.52, 14.10, 12.99. IR (neat, cm^{-1}): 3159.61, 2958.54, 2869.40, 1694.41, 1651.06, 1455.46, 1153.45, 1036.92, 944.22, 757.98. HRMS (ESI) ($M+H^+$) Calcd. for $C_{30}H_{45}N_4O_5S^+$: 573.3105, found 573.3108.



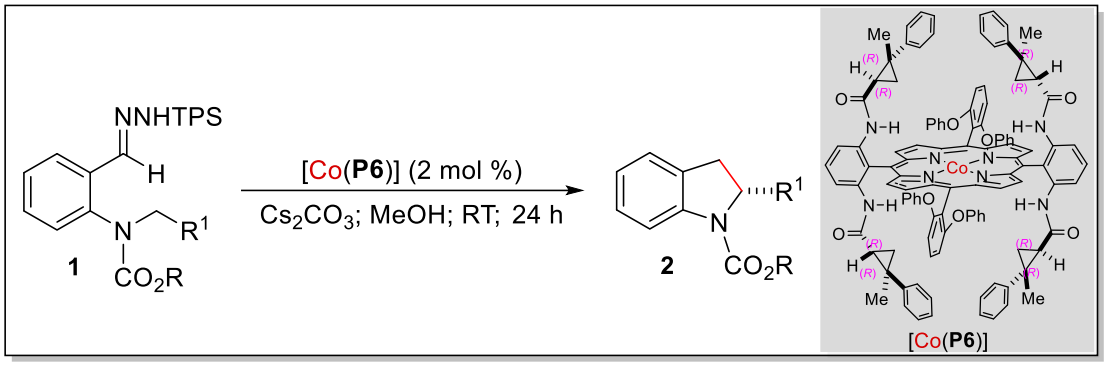
methyl allyl(2-((2-((2,4,6-triisopropylphenyl)sulfonyl)hydrazono)methyl) phenyl)carbamate 1-u Yield: 90%. Hexanes/ethyl acetate = 5/1. ^{1}H NMR (500 MHz, $CDCl_3$) δ

8.07 (br, 1H), 7.87 (d, *J* = 7.9 Hz, 1H), 7.76 (s, 1H), 7.35 (td, *J* = 7.7, 1.6 Hz, 1H), 7.28 – 7.25 (m, 1H), 7.18 (s, 2H), 7.09 (d, *J* = 7.9 Hz, 1H), 5.80 (ddt, *J* = 16.8, 10.1, 6.6 Hz, 1H), 5.08 – 5.02 (m, 2H), 4.30 – 4.21 (m, 3H), 4.02 (br, 1H), 3.54 (br, 3H), 2.90 (hept, *J* = 6.9 Hz, 1H), 1.30 (d, *J* = 6.7 Hz, 12H), 1.25 (d, *J* = 6.9 Hz, 6H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 156.04, 153.37, 151.31, 141.63, 132.18, 131.48, 131.17, 130.64, 128.51, 127.82, 126.60, 124.00, 123.81, 119.20, 53.83, 53.23, 34.18, 30.00, 24.86, 23.52. IR (neat, cm^{-1}): 3146.27, 2959.08, 2869.15, 1675.25, 1601.09, 1455.41, 1376.42, 1278.58, 1058.74, 1038.32, 937.35, 751.55. HRMS (ESI) (M+H$^+$) Calcd. for $C_{27}H_{38}N_3O_4S^+$: 500.2578, found 500.2583.

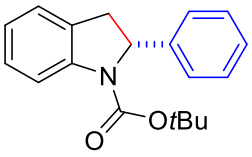


***tert*-butyl (cyclopropylmethyl)(2-((2-((2,4,6-triisopropylphenyl)sulfonyl) hydrazono)methyl)phenyl)carbamate 1-v** Yield: 83%. Hexanes/ethyl acetate = 5/1. ^{1}H NMR (600 MHz, $CDCl_3$) δ 8.01 (s, 1H), 7.88 (s, 1H), 7.86 (d, J = 7.9 Hz, 1H), 7.34 (t, J = 7.3 Hz, 1H), 7.22 (t, J = 7.6 Hz, 1H), 7.17 (s, 2H), 7.12 (s, 1H), 4.31 – 4.25 (m, 2H), 3.37 – 3.34 (m, 2H), 2.93 – 2.86 (m, 1H), 1.48 (br, 2H), 1.30 – 1.24 (m, 25H), 0.86 (s, 1H), 0.35 and 0.29 (br, 2H), 0.10 – 0.03 (m, 1H), 0.01 – -0.05 (m, 1H). ^{13}C NMR (150 MHz, $CDCl_3$) δ 154.74, 153.32, 151.34, 142.51, 141.47, 131.43, 131.29, 130.41, 128.46, 127.12, 126.18, 123.78, 80.47, 54.69, 34.16, 29.99, 28.20, 24.87, 23.52, 9.90, 3.76, 3.44. IR (neat, cm^{-1}): 3163.62, 2961.96, 2867.56, 2359.83, 1670.21, 1601.26, 1154.31, 757.50, 590.96. HRMS (ESI) (M+Na$^+$) Calcd. for $C_{31}H_{45}N_3NaO_4S^+$: 578.3023, found 578.3021.

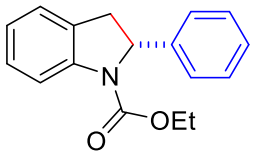
X. General Procedure for [Co(P6)]-Catalyzed Enantioselective Radical C–H Alkylation



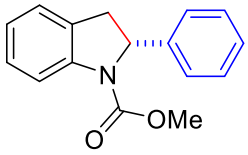
An oven-dried Schlenk tube was charged with sulfonyl hydrazone **1** (0.1 mmol), [Co(**P6**)] (2 mol %) and Cs_2CO_3 (0.2 mmol). The Schlenk tube was then evacuated and back filled with nitrogen for 3 times. The Teflon screw cap was replaced with a rubber septum, methanol (1.0 mL) was added via a gastight syringe. The Schlenk tube was then purged with nitrogen for 30 s and the rubber septum was replaced with a Teflon screw cap. The mixture was then stirred at RT. After 24 h, the reaction mixture was filtrated through a short pad of silica gel, concentrated under vacuum and purified by flash column chromatography. The fractions containing product were collected and concentrated under vacuum to afford the desired compound **2**.



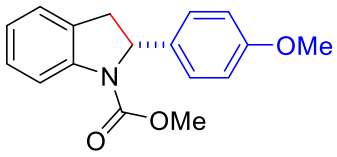
***tert*-butyl (*R*)-2-phenylindoline-1-carboxylate 2a** Yield: 82%. *ee*: 66%. Hexanes/ethyl acetate = 9/1, R_f = 0.50. ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.93 (s, 1H), 7.28 – 7.17 (m, 6H), 7.11 (d, *J* = 7.3 Hz, 1H), 6.97 (t, *J* = 7.4 Hz, 1H), 5.36 (br, 1H), 3.66 (dd, *J* = 16.2, 10.7 Hz, 1H), 2.95 (d, *J* = 16.2 Hz, 1H), 1.30 (br, 9H). ^{13}C NMR (150 MHz, $CDCl_3$) δ 152.34, 144.60, 143.00, 129.10, 128.47, 127.58, 127.10, 125.24, 124.75, 122.52, 114.62, 80.73, 62.58, 37.76, 28.13. IR (neat, cm^{-1}): 2977.25, 2929.13, 1693.35, 1482.59, 1387.15, 1139.31, 1015.13, 760.13, 701.62. HPLC analysis: *ee* = 66%. IA (99.7% hexanes: 0.3% isopropanol, 0.8 mL/min): t_{major} = 17.03 min, t_{minor} = 14.45 min. $[\alpha]^{20}_D$ = 29.6 (*c* = 0.5, $CHCl_3$) . HRMS (ESI) ($M+H^+$) Calcd. for $C19H22NO2^+$: 296.1645, found 296.1648.



ethyl (*R*)-2-phenylindoline-1-carboxylate 2b Yield: 92%. *ee*: 86%. Hexanes/ethyl acetate = 8/1, R_f = 0.55. ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.91 (s, 1H), 7.28 – 7.18 (m, 6H), 7.14 (d, *J* = 7.3 Hz, 1H), 7.00 (t, *J* = 7.4 Hz, 1H), 5.43 (br, 1H), 4.13 (br, 2H), 3.70 (dd, *J* = 16.2, 10.6 Hz, 1H), 2.99 (d, *J* = 16.2 Hz, 1H), 1.08 (br, 3H). ^{13}C NMR (150 MHz, $CDCl_3$) δ 153.28, 143.98, 129.25, 128.54, 127.67, 127.27, 125.36, 124.82, 122.88, 114.85, 62.36, 61.36, 37.84, 14.32. IR (neat, cm^{-1}): 2979.21, 2928.36, 1709.64, 1486.22, 1407.35, 1382.31, 1274.04, 1055.34, 755.99. HPLC analysis: *ee* = 86%. IA (99% hexanes: 1% isopropanol, 0.8 mL/min): t_{major} = 17.07 min, t_{minor} = 12.02 min. $[\alpha]^{20}{}_D$ = 50.8 (*c* = 0.5, $CHCl_3$). HRMS (ESI) ($M+H^+$) Calcd. for $C17H18NO2^+$: 268.1338, found 268.1342.

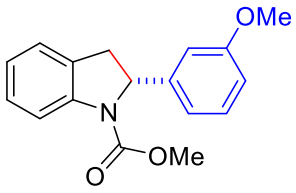


methyl (*R*)-2-phenylindoline-1-carboxylate 2c Yield: 92%. *ee*: 94%. Hexanes/ethyl acetate = 8/1, R_f = 0.50. ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.92 (s, 1H), 7.29 – 7.18 (m, 6H), 7.14 (d, *J* = 7.3 Hz, 1H), 7.00 (t, *J* = 7.4 Hz, 1H), 5.46 and 5.44 (br, 1H), 3.71 (dd, *J* = 16.2, 10.5 Hz, 1H), 3.70 (br, 3H), 2.98 (dd, *J* = 16.2, 2.7 Hz, 1H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 153.72, 143.74, 142.55, 129.42, 128.60, 127.42, 127.30, 125.23, 124.86, 123.00, 114.88, 62.30, 52.51, 37.92. IR (neat, cm^{-1}): 2952.64, 2920.54, 1705.53, 1483.87, 1440.85, 1386.08, 1274.07, 1055.93, 753.68. HPLC analysis: *ee* = 94%. IA (99.5% hexanes: 0.5% isopropanol, 0.8 mL/min): t_{major} = 26.13 min, t_{minor} = 19.12 min. $[\alpha]^{20}{}_D$ = 52.8 (*c* = 0.5, $CHCl_3$). HRMS (ESI) ($M+H^+$) Calcd. for $C16H16NO2^+$: 254.1176, found 254.1181.

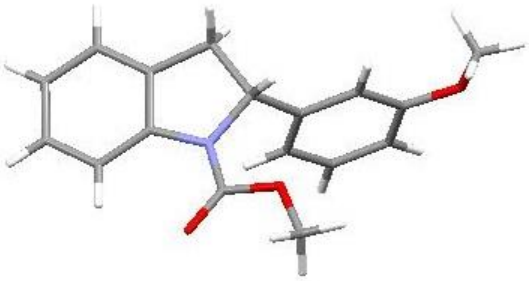


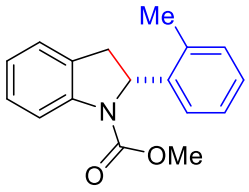
methyl (*R*)-2-(4-methoxyphenyl)indoline-1-carboxylate 2d Yield: 98%. *ee*: 94%. Hexanes/ethyl acetate = 5/1, R_f = 0.48. ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.89 (s, 1H), 7.24 (t, *J* = 7.7 Hz, 1H), 7.16 – 7.12 (m, 3H), 7.01 (t, *J* = 7.4 Hz, 1H), 6.81 (d, *J* = 8.5 Hz, 2H), 5.42 and 5.40 (br, 1H), 3.77 (s, 3H), 3.71 (br, 3H), 3.69 (dd, *J* = 16.2, 10.4 Hz, 1H), 2.97 (dd, *J* = 16.2, 2.1 Hz, 1H). ^{13}C NMR (150 MHz, $CDCl_3$) δ 158.81, 153.76, 142.51, 135.98, 129.91, 127.64,

126.56, 124.85, 122.95, 114.92, 113.92, 61.84, 55.21, 52.52, 37.93. IR (neat, cm^{-1}): 2951.72, 1704.02, 1612.04, 1512.83, 1459.71, 1247.99, 1051.36, 1025.76, 846.10, 742.09. HPLC analysis: *ee* = 94%. IA (99% hexanes: 1% isopropanol, 1.0 mL/min): t_{major} = 24.97 min, t_{minor} = 19.37 min. $[\alpha]^{20}{}_{D}$ = 72.8 (*c* = 0.5, $CHCl_3$). HRMS (ESI) ($M+H^+$) Calcd. for $C17H18NO3^+$: 284.1281, found 284.1278.



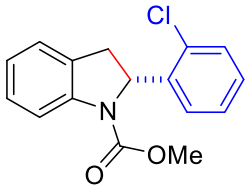
methyl (*R*)-2-(3-methoxyphenyl)indoline-1-carboxylate 2e Yield: 98%. *ee*: 90%. Hexanes/ethyl acetate = 5/1, R_f = 0.45. ^1H NMR (600 MHz, $CDCl_3$) δ 7.91 (s, 1H), 7.26 – 7.23 (m, 1H), 7.20 (t, *J* = 7.9 Hz, 1H), 7.13 (d, *J* = 7.3 Hz, 1H), 7.00 (t, *J* = 7.4 Hz, 1H), 6.78 – 6.76 (m, 2H), 6.73 (s, 1H), 5.43 and 5.42 (br, 1H), 3.75 – 3.68 (m, 7H), 2.97 (dd, *J* = 16.2, 2.7 Hz, 1H). ^{13}C NMR (150 MHz, $CDCl_3$) δ 159.77, 153.74, 145.48, 129.73, 127.69, 124.88, 123.03, 117.70, 117.48, 114.90, 113.20, 112.26, 111.26, 62.27, 55.14, 52.59, 37.93. IR (neat, cm^{-1}): 2955.13, 1701.26, 1599.57, 1483.93, 1440.81, 1385.47, 1264.11, 1134.70, 1056.34, 733.23. HPLC analysis: *ee* = 90%. IA (99% hexanes: 1% isopropanol, 1.0 mL/min): t_{major} = 24.61 min, t_{minor} = 15.78 min. $[\alpha]^{20}{}_{D}$ = 47.2 (*c* = 0.5, $CHCl_3$). HRMS (ESI) ($M+H^+$) Calcd. for $C17H18NO3^+$: 284.1281, found 284.1282.



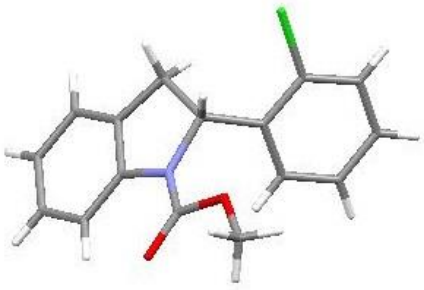


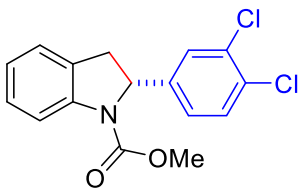
methyl (*R*)-2-(o-tolyl)indoline-1-carboxylate 2f Yield: 96%. *ee*: 94%. Hexanes/ethyl acetate = 6/1, R_f = 0.5. ^1H NMR (600 MHz, $CDCl_3$) δ 7.99 (s, 1H), 7.27 – 7.25 (m, 1H), 7.17 (d, *J* = 7.4 Hz, 1H), 7.14 – 7.10 (m, 2H), 7.06 (t, *J* = 7.5 Hz, 1H), 7.01 – 6.99 (m, 2H),

5.67 and 5.66 (br, 1H), 3.73 (dd, J = 16.0, 10.6 Hz, 1H), 3.67 (br, 3H), 2.84 (dd, J = 16.0, 3.0 Hz, 1H), 2.42 (s, 3H). ^{13}C NMR (150 MHz, $CDCl_3$) δ 153.74, 142.81, 141.90, 133.67, 130.50, 129.33, 127.71, 126.97, 126.38, 125.07, 123.53, 123.03, 114.87, 59.16, 52.63, 37.03, 19.28. IR (neat, cm^{-1}): 2954.05, 1702.15, 1599.16, 1485.45, 1440.67, 1265.75, 1191.94, 1054.33, 737.90. HPLC analysis: *ee* = 94%. IA (99.5% hexanes: 0.5% isopropanol, 0.8 mL/min): t_{major} = 25.25 min, t_{minor} = 18.29 min. $[\alpha]^{20}{}_D$ = 167.2 (c = 0.5, $CHCl_3$). HRMS (ESI) ($M+H^+$) Calcd. for $C17H18NO2^+$: 268.1332, found 268.1336.



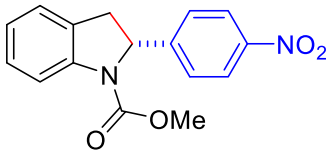
methyl (*R*)-2-(2-chlorophenyl)indoline-1-carboxylate 2g Yield: 92%. *ee*: 93%. Hexanes/ethyl acetate = 6/1, R_f = 0.55. ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.99 (s, 1H), 7.39 (d, J = 7.8 Hz, 1H), 7.28 – 7.25 (m, 1H), 7.17 (t, J = 7.4 Hz, 1H), 7.15 – 7.11 (m, 2H), 7.06 (d, J = 7.6 Hz, 1H), 7.01 (t, J = 7.4 Hz, 1H), 5.86 and 5.84 (br, 1H), 3.79 (dd, J = 16.3, 10.5 Hz, 1H), 3.69 (s, 3H), 2.89 (dd, J = 16.3, 2.3 Hz, 1H). ^{13}C NMR (150 MHz, $CDCl_3$) δ 153.60, 142.64, 140.88, 131.50, 129.73, 129.00, 128.22, 127.75, 127.10, 125.29, 125.08, 123.23, 114.93, 59.53, 52.70, 36.92. IR (neat, cm^{-1}): 2954.71, 1701.97, 1601.19, 1484.01, 1439.31, 1385.42, 1056.01, 744.34, 628.11. HPLC analysis: *ee* = 93%. IA (99% hexanes: 1% isopropanol, 0.8 mL/min): t_{major} = 15.71 min, t_{minor} = 12.04 min. $[\alpha]^{20}{}_D$ = 77.6 (c = 0.5, $CHCl_3$). HRMS (ESI) ($M+H^+$) Calcd. for $C16H15ClNO2^+$: 288.0786, found 288.0792.



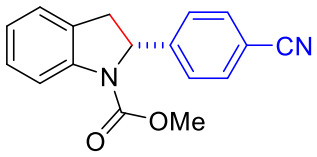


methyl (*R*)-2-(3,4-dichlorophenyl)indoline-1-carboxylate 2h Yield:

85%. *ee*: 96%. Hexanes/ethyl acetate = 6/1, R_f = 0.55. ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.91 (s, 1H), 7.35 (d, *J* = 8.3 Hz, 1H), 7.29 – 7.25 (m, 2H), 7.15 (d, *J* = 7.3 Hz, 1H), 7.03 (t, *J* = 7.4 Hz, 2H), 5.40 and 5.38 (br, 1H), 3.73 (dd, *J* = 16.3, 10.6 Hz, 1H), 3.71 (br, 3H), 2.93 (dd, *J* = 16.3, 3.0 Hz, 1H). ^{13}C NMR (150 MHz, $CDCl_3$) δ 153.44, 143.98, 132.69, 131.36, 130.72, 127.98, 127.46, 124.98, 124.71, 123.36, 115.00, 61.38, 52.77, 37.73. IR (neat, cm^{-1}): 2953.91, 1705.31, 1601.07, 1484.60, 1441.48, 1383.34, 1274.30, 1057.23, 1030.58, 738.80. HPLC analysis: *ee* = 96%. IB (99.5% hexanes: 0.5% isopropanol, 0.8 mL/min): t_{major} = 18.57 min, t_{minor} = 23.24 min. $[\alpha]^{20}{}_D$ = 58.0 (*c* = 0.5, $CHCl_3$). HRMS (ESI) ($M+H^+$) Calcd. for C16H14Cl2NO2$^+$: 322.0396, found 322.0393.

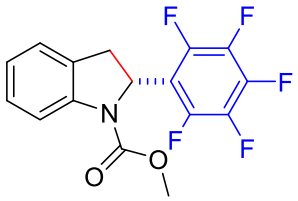


methyl (*R*)-2-(4-nitrophenyl)indoline-1-carboxylate 2i Yield: 96%. *ee*: 87%. Hexanes/ethyl acetate = 4/1, R_f = 0.38. ^{1}H NMR (600 MHz, $CDCl_3$) δ 8.16 (d, *J* = 8.6 Hz, 2H), 7.95 (br, 1H), 7.37 (d, *J* = 7.8 Hz, 2H), 7.28 (t, *J* = 7.8 Hz, 1H), 7.16 (d, *J* = 7.3 Hz, 1H), 7.04 (t, *J* = 7.5 Hz, 1H), 5.55 and 5.53 (br, 1H), 3.77 (dd, *J* = 16.3, 10.7 Hz, 1H), 3.71 (br, 3H), 2.95 (dd, *J* = 16.3, 3.0 Hz, 1H). ^{13}C NMR (150 MHz, $CDCl_3$) δ 150.86, 147.28, 128.10, 126.20, 125.03, 124.13, 123.49, 115.00, 61.78, 52.82, 37.60. IR (neat, cm^{-1}): 2961.51, 2940.10, 1714.04, 1595.41, 1509.88, 1483.70, 1382.51, 1141.76, 1055.66, 762.06. HPLC analysis: *ee* = 87%. IA (98% hexanes: 2% isopropanol, 1.0 mL/min): t_{major} = 27.95 min, t_{minor} = 34.67 min. $[\alpha]^{20}{}_D$ = 43.2 (*c* = 0.5, $CHCl_3$). HRMS (ESI) ($M+H^+$) Calcd. for C16H15N2O4$^+$: 299.1026, found 299.1026.

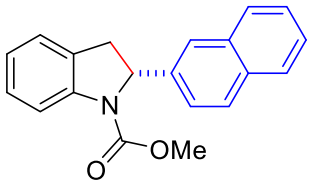


methyl (*R*)-2-(4-cyanophenyl)indoline-1-carboxylate 2j Yield: 97%. *ee*: 94%. Hexanes/ethyl acetate = 4/1, R_f = 0.40. ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.94 (s, 1H), 7.59 (d, *J* = 8.2 Hz, 2H), 7.31 – 7.25 (m, 3H), 7.15 (d, *J* = 7.3 Hz, 1H), 7.03 (t, *J* = 7.4 Hz, 1H), 5.49 and 5.48 (br, 1H), 3.75 (dd, *J* = 16.3, 10.7 Hz, 1H), 3.71 (br, 3H), 2.93 (dd, *J* = 16.3, 3.0 Hz, 1H). ^{13}C NMR (150 MHz, $CDCl_3$) δ 153.47, 148.89, 132.64, 128.57, 128.03, 126.09, 125.00, 123.42, 118.63, 114.96, 111.34, 61.96, 52.77, 37.63. IR (neat, cm^{-1}): 2954.50, 2228.06, 1701.14, 1607.61, 1483.90, 1463.55, 1384.31, 1272.02, 1055.25, 749.31. HPLC analysis: *ee* = 94%. IA (98% hexanes:

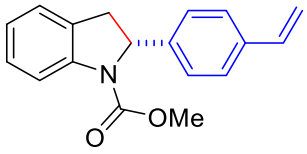
2% isopropanol, 1.0 mL/min): t_{major} = 28.18 min, t_{minor} = 32.55 min. $[\alpha]^{20}{}_D$ = 114.4 (c = 0.5, $CHCl_3$). HRMS (ESI) ($M+H^+$) Calcd. for $C17H15N2O2^+$: 279.1128, found 279.1132.



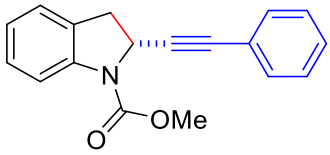
methyl (*R*)-2-(perfluorophenyl)indoline-1-carboxylate 2k Yield: 90%. *ee*: 95%. Hexanes/ethyl acetate = 7/1, R_f = 0.6. ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.89 and 7.51 (br, 1H), 7.26 – 7.24 (m, 1H), 7.17 (d, J = 7.4 Hz, 1H), 7.04 (t, J = 7.4 Hz, 1H), 5.81 (br, 1H), 3.85 (br, 3H), 3.76 (dd, J = 16.5, 11.4 Hz, 1H), 3.12 (dd, J = 16.5, 4.8 Hz, 1H). ^{13}C NMR (150 MHz, $CDCl_3$) δ 152.80, 144.97 (md, J = 253.6 Hz), 141.89, 140.94, 140.59 (md, J = 254.0 Hz), 137.51 (md, J = 251.4 Hz), 128.46, 127.92, 124.14, 123.26, 116.47, 52.84, 35.78, 35.20. IR (neat, cm^{-1}): 2961.76, 1713.96, 1503.70, 1484.98, 1602.15, 1442.06, 1387.29, 1282.30, 1193.43, 1123.53, 1011.90, 752.80. HPLC analysis: *ee* = 95%. IA (99.5% hexanes: 0.5% isopropanol, 0.8 mL/min): t_{major} = 33.47 min, t_{minor} = 14.21 min. $[\alpha]^{20}{}_D$ = -54.4 (c = 0.5, $CHCl_3$). HRMS (ESI) ($M+H^+$) Calcd. for $C16H11F5NO2^+$: 344.0704, found 344.0708.



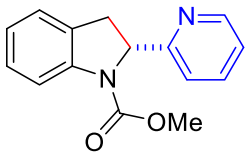
methyl (*R*)-2-(naphthalen-2-yl)indoline-1-carboxylate 2l Yield: 90%. *ee*: 94%. Hexanes/ethyl acetate = 7/1, R_f = 0.58. ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.99 (s, 1H), 7.80 – 7.77 (m, 3H), 7.64 (br, 1H), 7.46 – 7.43 (m, 2H), 7.31 – 7.28 (m, 2H), 7.16 (d, J = 7.2 Hz, 1H), 7.03 (td, J = 7.5, 0.7 Hz, 1H), 5.63 and 5.62 (br, 1H), 3.78 (dd, J = 16.3, 10.6 Hz, 1H), 3.68 (s, 3H), 3.05 (dd, J = 16.3, 2.5 Hz, 1H). ^{13}C NMR (150 MHz, $CDCl_3$) δ 153.82, 141.06, 133.28, 132.79, 128.72, 127.92, 127.77, 127.61, 126.14, 125.74, 124.94, 123.77, 123.52, 123.10, 114.95, 62.48, 52.60, 37.99. IR (neat, cm^{-1}): 2917.63, 2849.15, 1701.58, 1598.18, 1485.44, 1440.01, 1274.59, 1152.34, 1054.00, 748.53. HPLC analysis: *ee* = 94%. IA (99% hexanes: 1% isopropanol, 0.8 mL/min): t_{major} = 27.96 min, t_{minor} = 20.63 min. $[\alpha]^{20}{}_D$ = 83.2 (c = 0.5, $CHCl_3$). HRMS (ESI) ($M+H^+$) Calcd. for $C20H18NO2^+$: 304.1332, found 304.1337.



methyl (*R*)-2-(4-vinylphenyl)indoline-1-carboxylate 2m Yield: 57%. *ee*: 95%. Hexanes/ethyl acetate = 6/1, R_f = 0.50. ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.92 (s, 1H), 7.32 (d, *J* = 8.1 Hz, 2H), 7.26 – 7.24 (m, 1H), 7.15 – 7.10 (m, 3H), 7.01 (t, *J* = 7.4 Hz, 1H), 6.67 (dd, *J* = 17.6, 10.9 Hz, 1H), 5.70 (d, *J* = 17.6 Hz, 1H), 5.45 and 5.43 (br, 1H), 5.21 (d, *J* = 10.9 Hz, 1H), 3.72 (br, 3H), 3.71 (dd, *J* = 16.1, 10.4 Hz, 1H), 2.97 (dd, *J* = 16.2, 2.6 Hz, 1H). ^{13}C NMR (150 MHz, $CDCl_3$) δ 153.70, 145.14, 143.34, 136.76, 136.38, 128.06, 127.72, 126.50, 125.48, 124.89, 123.04, 114.91, 113.74, 62.12, 52.57, 37.94. IR (neat, cm^{-1}): 2952.07, 1705.42, 1483.20, 1439.26, 1382.70, 1271.60, 1136.01, 1053.63, 733.20. HPLC analysis: *ee* = 95%. IA (99.5% hexanes: 0.5% isopropanol, 0.8 mL/min): t_{major} = 30.74 min, t_{minor} = 22.49 min. $[\alpha]^{20}{}_D$ = 40.8 (*c* = 0.5, $CHCl_3$). HRMS (ESI) ($M+H^+$) Calcd. for C18H18NO2$^+$: 280.1332, found 280.1336.

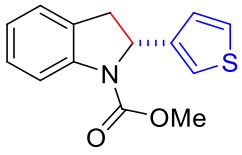


methyl (*R*)-2-(phenylethynyl)indoline-1-carboxylate 2n Yield: 50%. *ee*: 87%. Hexanes/ethyl acetate = 7/1, R_f = 0.60. ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.82 (s, 1H), 7.38 (d, *J* = 7.6 Hz, 2H), 7.30 – 7.19 (m, 5H), 7.01 (t, *J* = 7.4 Hz, 1H), 5.35 and 5.33 (br, 1H), 3.91 (s, 3H), 3.58 (dd, *J* = 15.9, 10.2 Hz, 1H), 3.27 (dd, *J* = 15.9, 2.1 Hz, 1H). ^{13}C NMR (150 MHz, $CDCl_3$) δ 153.38, 141.12, 131.77, 129.13, 128.31, 128.15, 127.76, 124.78, 122.96, 122.56, 115.21, 88.74, 82.65, 52.83, 50.74, 36.47. IR (neat, cm^{-1}): 2916.84, 2848.92, 1708.75, 1598.12, 1485.02, 1441.52, 1385.53, 1269.92, 1192.02, 1130.13, 1056.18, 755.55, 691.55. HPLC analysis: *ee* = 87%. IC (99% hexanes: 1% isopropanol, 0.8 mL/min): t_{major} = 30.67 min, t_{minor} = 34.95 min. $[\alpha]^{20}{}_D$ = 18.0 (*c* = 0.5, $CHCl_3$). HRMS (ESI) ($M+H^+$) Calcd. for C18H16NO2$^+$: 278.1176, found 278.1180.

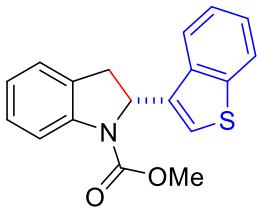


methyl (*R*)-2-(pyridin-2-yl)indoline-1-carboxylate 2o Yield: 93%. *ee*: 90%. Hexanes/ethyl acetate = 2/1, R_f = 0.40. ^{1}H NMR (600 MHz, $CDCl_3$) δ 8.57 (d, *J* = 4.7 Hz, 1H), 7.97 (s, 1H), 7.59 (t, *J* = 7.7 Hz, 1H), 7.26 – 7.23 (m, 1H), 7.16 – 7.12 (m, 3H), 7.00 (t, *J* = 7.4 Hz, 1H), 5.60 and 5.58 (br, 1H), 3.74 (dd, *J* = 16.1, 10.9 Hz, 1H), 3.69 (s, 3H), 3.14 and 3.12 (br, 1H).

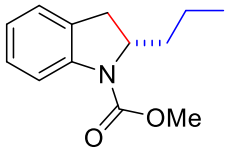
^{13}C NMR (150 MHz, $CDCl_3$) δ 162.18, 153.83, 149.53, 136.90, 129.08, 127.68, 124.93, 123.20, 122.21, 118.94, 118.83, 115.00, 63.64, 52.65, 36.55. IR (neat, cm^{-1}): 2922.76, 1709.13, 1591.33, 1484.77, 1465.88, 1265.66, 1058.53, 738.98. HPLC analysis: *ee* = 90%. IC (95% hexanes: 5% isopropanol, 1.0 mL/min): t_{major} = 36.35 min, t_{minor} = 29.75 min. $[\alpha]^{20}{}_D$ = 130.4 (*c* = 0.5, $CHCl_3$). HRMS (ESI) ($M+H^+$) Calcd. for $C15H15N2O2^+$: 255.1128, found 255.1133.



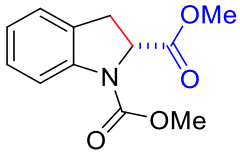
methyl (*R*)-2-(thiophen-3-yl)indoline-1-carboxylate 2p Yield: 97%. *ee*: 94%. Hexanes/ethyl acetate = 6/1, R_f = 0.60. ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.86 (br, 1H), 7.25 – 7.21 (m, 2H), 7.17 (d, *J* = 7.4 Hz, 1H), 7.08 (br, 1H), 7.01 (t, *J* = 7.4 Hz, 1H), 6.90 (d, *J* = 4.7 Hz, 1H), 5.58 and 5.57 (br, 1H), 3.77 (br, 3H), 3.64 (dd, *J* = 16.0, 10.1 Hz, 1H), 3.01 (dd, *J* = 16.0, 2.1 Hz, 1H). ^{13}C NMR (150 MHz, $CDCl_3$) δ 153.76, 144.00, 141.89, 129.65, 127.68, 126.15, 125.36, 124.85, 123.02, 120.64, 115.19, 58.36, 52.56, 36.96. IR (neat, cm^{-1}): 2952.85, 1700.19, 1601.40, 1482.94, 1440.10, 1382.32, 1273.15, 1054.83, 736.24. HPLC analysis: *ee* = 94%. IA (99.5% hexanes: 0.5% isopropanol, 0.8 mL/min): t_{major} = 34.25 min, t_{minor} = 23.41 min. $[\alpha]^{20}{}_D$ = 63.2 (*c* = 0.5, $CHCl_3$). HRMS (ESI) ($M+H^+$) Calcd. for $C14H14NO2S^+$: 260.0740, found 260.0741.



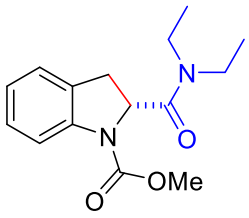
methyl (*R*)-2-(benzo[*b*]thiophen-3-yl)indoline-1-carboxylate 2q Yield: 97%. *ee*: 94%. Hexanes/ethyl acetate = 6/1, R_f = 0.40. ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.95 (s, 1H), 7.86 (d, *J* = 6.9 Hz, 1H), 7.69 (d, *J* = 6.9 Hz, 1H), 7.38 – 7.36 (m, 2H), 7.28 (t, *J* = 7.6 Hz, 1H), 7.13 (d, *J* = 7.3 Hz, 1H), 7.10 (s, 1H), 7.02 (t, *J* = 7.4 Hz, 1H), 5.89 and 5.87 (br, 1H), 3.75 (dd, *J* = 16.0, 10.4 Hz, 1H), 3.73 (br, 3H), 3.07 (dd, *J* = 16.0, 2.4 Hz, 1H). ^{13}C NMR (150 MHz, $CDCl_3$) δ 153.73, 141.16, 137.54, 136.61, 127.81, 125.07, 124.41, 124.10, 123.21, 123.12, 121.53, 121.31, 115.21, 58.03, 52.70, 36.15. IR (neat, cm^{-1}): 2917.57, 2849.54, 1706.67, 1600.89, 1484.33, 1441.52, 1388.37, 1273.64, 761.73. HPLC analysis: *ee* = 92%. IA (99% hexanes: 1% isopropanol, 0.8 mL/min): t_{major} = 27.22 min, t_{minor} = 21.61 min. $[\alpha]^{20}{}_D$ = 127.2 (*c* = 0.5, $CHCl_3$). HRMS (ESI) ($M+H^+$) Calcd. for $C18H16NO2S^+$: 310.0896, found 310.0896.



methyl (*S*)-2-propylindoline-1-carboxylate 2r Yield: 65%. *ee*: 87%. Hexanes/ethyl acetate = 6/1, R_f = 0.65. ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.78 (br, 1H), 7.17 (t, *J* = 7.7 Hz, 1H), 7.14 (d, *J* = 7.4 Hz, 1H), 6.96 (t, *J* = 7.4 Hz, 1H), 4.45 (br, 1H), 3.84 (s, 3H), 3.29 (dd, *J* = 16.0, 9.5 Hz, 1H), 2.75 (d, *J* = 16.0 Hz, 1H), 1.71 (br, 1H), 1.54 – 1.52 (m, 1H), 1.35 – 1.31 (m, 2H), 0.92 (t, *J* = 7.3 Hz, 3H). ^{13}C NMR (150 MHz, $CDCl_3$) δ 153.77, 130.42, 127.33, 124.83, 124.66, 122.68, 115.34, 59.33, 52.42, 36.75, 33.42, 18.10, 13.98. IR (neat, cm^{-1}): 2956.18, 2871.65, 2359.34, 2341.70, 1711.07, 1602.79, 1487.15, 1443.56, 1393.64, 1291.74, 765.20. HPLC analysis: *ee* = 87%. IA (99.8% hexanes: 0.2% isopropanol, 0.8 mL/min): t_{major} = 25.18 min, t_{minor} = 18.77 min. $[\alpha]^{20}{}_D$ = 27.2 (*c* = 0.5, $CHCl_3$). HRMS (ESI) ($M+H^+$) Calcd. for $C13H18NO2^+$: 220.1332, found 220.1332.



dimethyl (*R*)-indoline-1,2-dicarboxylate 2s Yield: 49%. *ee*: 81%. Hexanes/ethyl acetate = 5/1, R_f = 0.35. ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.92 and 7.51 (s, br, 1H), 7.22 (br, 1H), 7.13 (d, *J* = 7.4 Hz, 1H), 6.98 (t, *J* = 7.4 Hz, 1H), 4.94 (br, 1H), 3.93 and 3.80 (br, 3H), 3.75 (s, 3H), 3.58 – 3.53 (m, 1H), 3.15 (d, *J* = 16.4 Hz, 1H). ^{13}C NMR (150 MHz, $CDCl_3$) δ 172.06, 152.93, 142.21, 127.98, 124.78, 124.36, 122.99, 114.79, 59.99, 52.76, 52.53, 32.96 and 32.11 (a pair of s). IR (neat, cm^{-1}): 2956.69, 1749.89, 1698.64, 1484.85, 1433.36, 1049.66, 1001.19, 751.73. HPLC analysis: *ee* = 81%. IA (95% hexanes: 5% isopropanol, 0.8 mL/min): t_{major} = 17.40 min, t_{minor} = 13.67 min. $[\alpha]^{20}{}_D$ = 20.0 (*c* = 0.5, $CHCl_3$). HRMS (ESI) ($M+H^+$) Calcd. for $C12H14NO4^+$: 236.0917, found 236.0925.

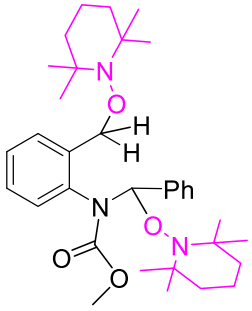


methyl (*R*)-2-(diethylcarbamoyl)indoline-1-carboxylate 2t Yield: 92%. *ee*: 68%. Hexanes/ethyl acetate = 1/1, R_f = 0.35. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.93 and 7.51 (d, *J* = 6.5 Hz, 1H), 7.20 – 7.16 (m, 1H), 7.09 (d, *J* = 7.1 Hz, 1H), 6.95 – 6.93 (m, 1H), 5.18 and 5.09 (d,

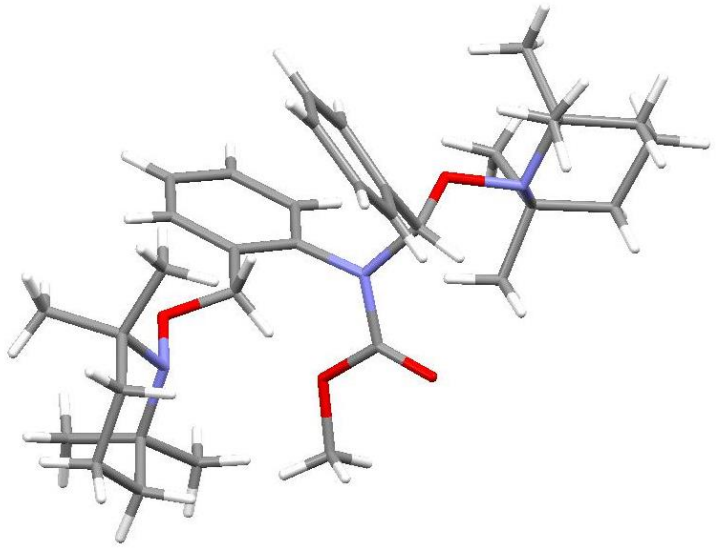
J = 8.4 Hz, 1H), 3.90 (s, 1H), 3.75 – 3.34 (m, 7H), 3.01 (d, *J* = 11.4 Hz, 1H), 1.33 – 1.25 (m, 3H), 1.13 (t, *J* = 7.1 Hz, 3H). ^{13}C NMR (150 MHz, $CDCl_3$) major rotamer: δ 170.42, 152.80, 143.04, 128.95, 127.89, 124.27, 122.60, 114.66, 57.94, 52.43, 41.58, 40.71, 33.43, 14.44, 12.91. IR (neat, cm^{-1}): 2976.38, 2936.18, 1715.66, 1652.06, 1488.71, 1445.25, 1392.19, 1266.04, 1138.22, 1063.88, 740.66. HPLC analysis: *ee* = 68%. IA (92% hexanes: 8% isopropanol, 0.8 mL/min): t_{major} = 38.78 min, t_{minor} = 36.22 min. $[\alpha]^{20}_{D}$ = 91.6 (*c* = 0.5, $CHCl_3$). HRMS (ESI) ($M+H^+$) Calcd. for $C15H21N2O3^+$: 277.1547, found 277.1550.

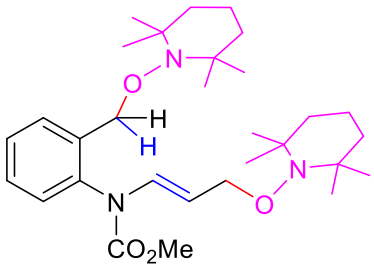
XI. General Procedure for TEMPO Trapping Reactions

An oven-dried Schlenk tube was charged with 1.0 equivalent of sulfonyl hydrazone **1** (0.1 mmol), [Co(Por)] (2 mol %) and Cs_2CO_3 (0.2 mmol). The Schlenk tube was then evacuated and back filled with nitrogen for 3 times. The Teflon screw cap was replaced with a rubber septum, TEMPO (2.5 equiv.) was added under nitrogen flow and methanol (1.0 mL) was added via a gastight syringe. The Schlenk tube was then purged with nitrogen for 10 s and the rubber septum was replaced with a Teflon screw cap. The mixture was then stirred at RT or 40 °C. After 24 h, the reaction mixture was filtrated through a short pad of silica, concentrated under vacuum and purified by flash column chromatography. The fractions containing product were collected and concentrated under vacuum to afford the desired compound.

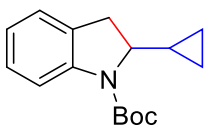


methyl (phenyl((2,2,6,6-tetramethylpiperidin-1-yl)oxy)methyl)(2-(((2,2,6,6-tetramethylpiperidin-1-yl)oxy)methyl)phenyl)carbamate 3c Yield: 90%. *ee*: 93%. Hexanes/ethyl acetate = 8/1, R_f = 0.70. ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.83 (d, *J* = 7.6 Hz, 1H), 7.41 (d, *J* = 7.3 Hz, 1H), 7.32 – 7.28 (m, 2H), 7.17 (s, 1H), 7.08 – 7.05 (m, 5H), 4.30 (d, *J* = 13.7 Hz, 1H), 3.66 (d, *J* = 13.7 Hz, 1H), 3.87 and 3.64 (s, 3H), 1.65 – 1.34 (m, 18H), 1.12 – 1.01 (m, 12H), 0.89 – 0.77 (m, 6H). ^{13}C NMR (150 MHz, $CDCl_3$) δ 155.40, 138.01, 137.54, 133.91, 128.13, 127.83, 126.97, 126.81, 126.51, 125.80, 125.48, 93.20, 72.79, 60.11, 59.31, 59.13, 52.60, 39.91, 39.64, 39.05, 32.98, 32.63, 32.49, 31.87, 20.17, 19.85, 19.53, 16.49. IR (neat, cm^{-1}): 2974.31, 2932.90, 1711.46, 1439.23, 1301.16, 1026.18, 732.55, 700.86. HPLC analysis: *ee* = 93%. IA (99.7% hexanes: 0.3% isopropanol, 0.8 mL/min): t_{major} = 8.05 min, t_{minor} = 9.15 min. $[\alpha]^{20}_D$ = -116.4 (*c* = 0.5, $CHCl_3$). HRMS (ESI) ($M+H^+$) Calcd. for $C_{34}H_{52}N_3O_4^+$: 566.3952, found 566.3959.



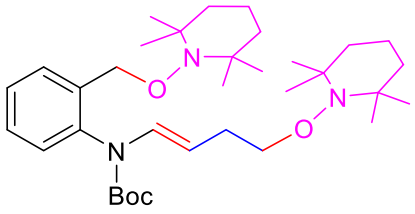


methyl (2-(((2,2,6,6-tetramethylpiperidin-1-yl)oxy)methyl) phenyl)(3-((2,2,6,6-tetramethylpiperidin-1-yl)oxy)prop-1-en-1-yl)carbamate 3u Yield: 72%. Hexanes/ethyl acetate = 8/1, R_f = 0.70. ^{1}H NMR (600 MHz, $CDCl_3$) (Contains both E/Z isomers of enamine) δ 7.67 and 7.62 (d, *J* = 7.6 Hz, 1H), 7.44 – 7.29 (m, 2.5 H), 7.18 and 7.12 (d, *J* = 7.6 Hz, and d, *J* = 6.6 Hz, 1H), 6.75 (br, 0.5 H), 4.82 – 4.78 (m, 0.5 H), 4.79 – 4.68 (m, 2H), 4.41 – 4.37 (m, 0.5 H), 4.17 (br, 1H), 3.67 (br, 3H), 3.47 (br, 1H), 1.61 – 0.86 (m, 36H). ^{13}C NMR (150 MHz, $CDCl_3$) (Contains both E/Z isomers of enamine) δ 154.29, 136.83, 136.34, 136.12, 134.86, 131.00, 128.65, 128.48, 128.37, 128.20, 128.12, 128.02, 127.92, 127.65, 125.07, 109.41, 106.26, 75.54, 73.98, 73.94, 71.71, 59.69, 59.62, 59.26, 58.90, 53.11, 53.06, 39.37, 39.28, 39.16, 32.64, 32.56, 32.45, 29.37, 20.03, 19.82, 19.60, 16.81, 16.76. IR (neat, cm^{-1}): 2977.25, 2930.79, 1715.52, 1661.25, 1442.54, 1314.88, 1215.86, 766.94. HRMS (ESI) ($M+H^+$) Calcd. for $C30H50N3O4^+$: 516.3796, found 516.3807.



***tert*-butyl 2-cyclopropylindoline-1-carboxylate 2v** Yield: 50%. Hexanes/ethyl

acetate = 8/1, R_f = 0.60. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.68 (s, 1H), 7.18 – 7.14 (m, 2H), 6.94 (t, *J* = 7.4 Hz, 1H), 4.00 (br, 1H), 3.29 (dd, *J* = 15.8, 9.4 Hz, 1H), 2.79 (d, *J* = 15.8 Hz, 1H), 1.57 (s, 9H), 1.10 – 1.04 (m, 1H), 0.65 – 0.60 (m, 1H), 0.53 – 0.48 (m, 1H), 0.44 – 0.38 (m, 1H), 0.23 – 0.18 (m, 1H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 152.78, 142.28, 130.63, 127.20, 124.67, 122.32, 115.62, 80.67, 62.93, 34.14, 28.49, 16.43, 4.22, 1.29. IR (neat, cm^{-1}): 2976.54, 1695.12, 1603.06, 1482.27, 1387.98, 1167.23, 1138.53, 1012.96, 740.25. HRMS ($Dart^+$) Calcd. for $C16H22NO2^+$: 260.1645, found 260.1651.



***tert*-butyl (4-((2,2,6,6-tetramethylpiperidin-1-yl)oxy)but-1-en-1-yl)(2-(((2,2,6,6-tetramethylpiperidin-1-yl)oxy)methyl)phenyl)carbamate 3v** Yield: 40%. Hexanes/ethyl acetate = 9/1, R_f = 0.70. ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.63 (d, *J* = 7.5 Hz, 1H), 7.34 (t, *J* = 7.4 Hz, 1H), 7.31 – 7.27 (m, 1H), 7.10 (d, *J* = 13.3 Hz, 1H), 6.99 (d, *J* = 6.6 Hz, 1H), 4.72 – 4.67 (m, 2H), 4.25 (dt, *J* = 14.4, 7.3 Hz, 1H), 3.59 (t, *J* = 7.0 Hz, 2H), 2.13 (br, 2H), 1.48 – 1.00 (m, 45H). ^{13}C NMR (150 MHz, $CDCl_3$) δ 152.41, 136.60, 135.91, 129.22, 128.69, 128.01, 127.73, 127.62, 107.54, 80.70, 74.11, 59.93, 59.57, 39.64, 39.59, 33.06, 32.89, 29.22, 28.08, 20.35, 20.00, 17.09. IR (neat, cm^{-1}): 2975.35, 2931.05, 1710.91, 1662.69, 1454.51, 1373.32, 1320.14, 1263.84, 1169.25, 1048.75, 741.49. HRMS (ESI) ($M+H^+$) Calcd. for $C34H58N3O4^+$: 572.4422, found 572.4424.

XII. References

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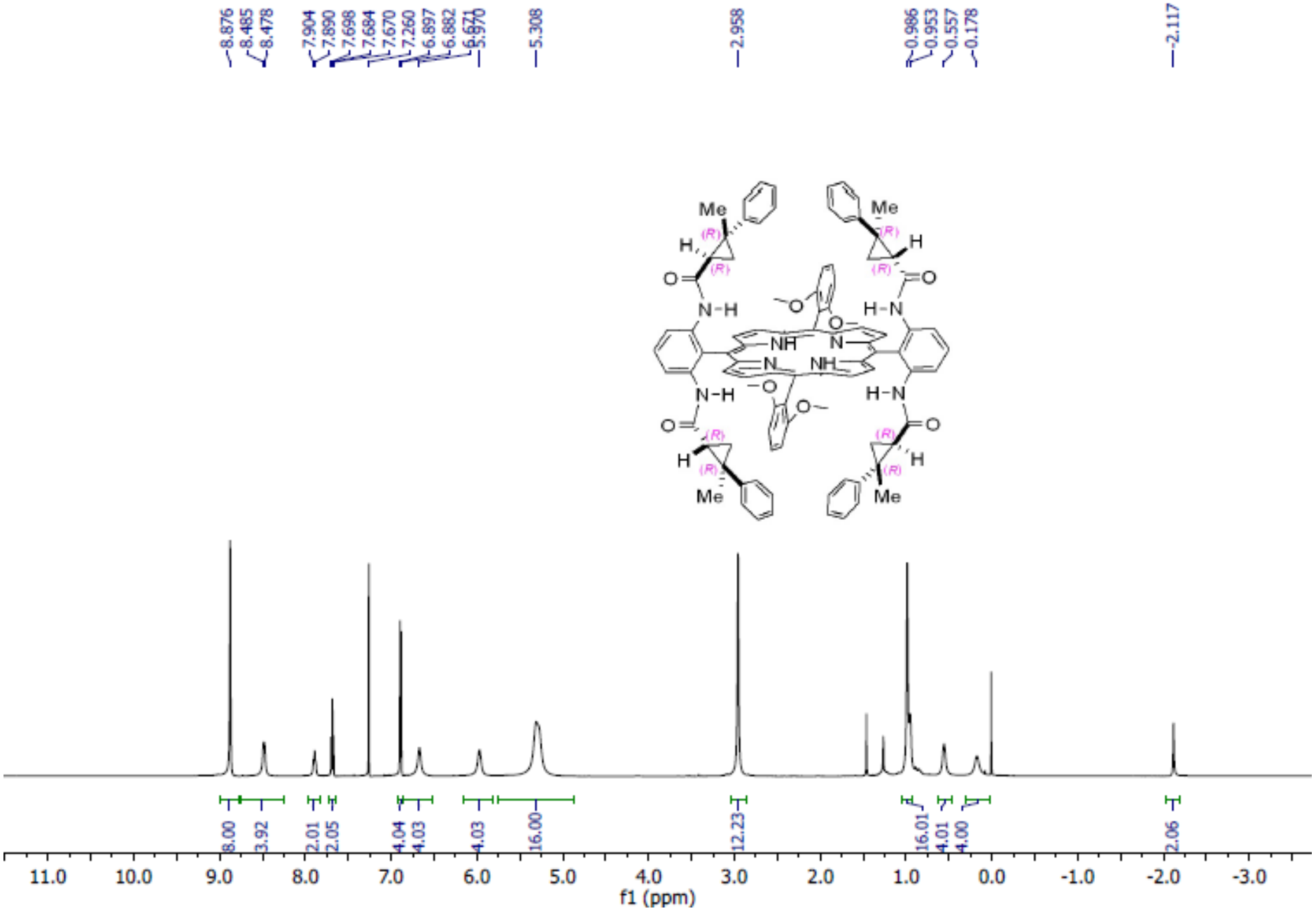
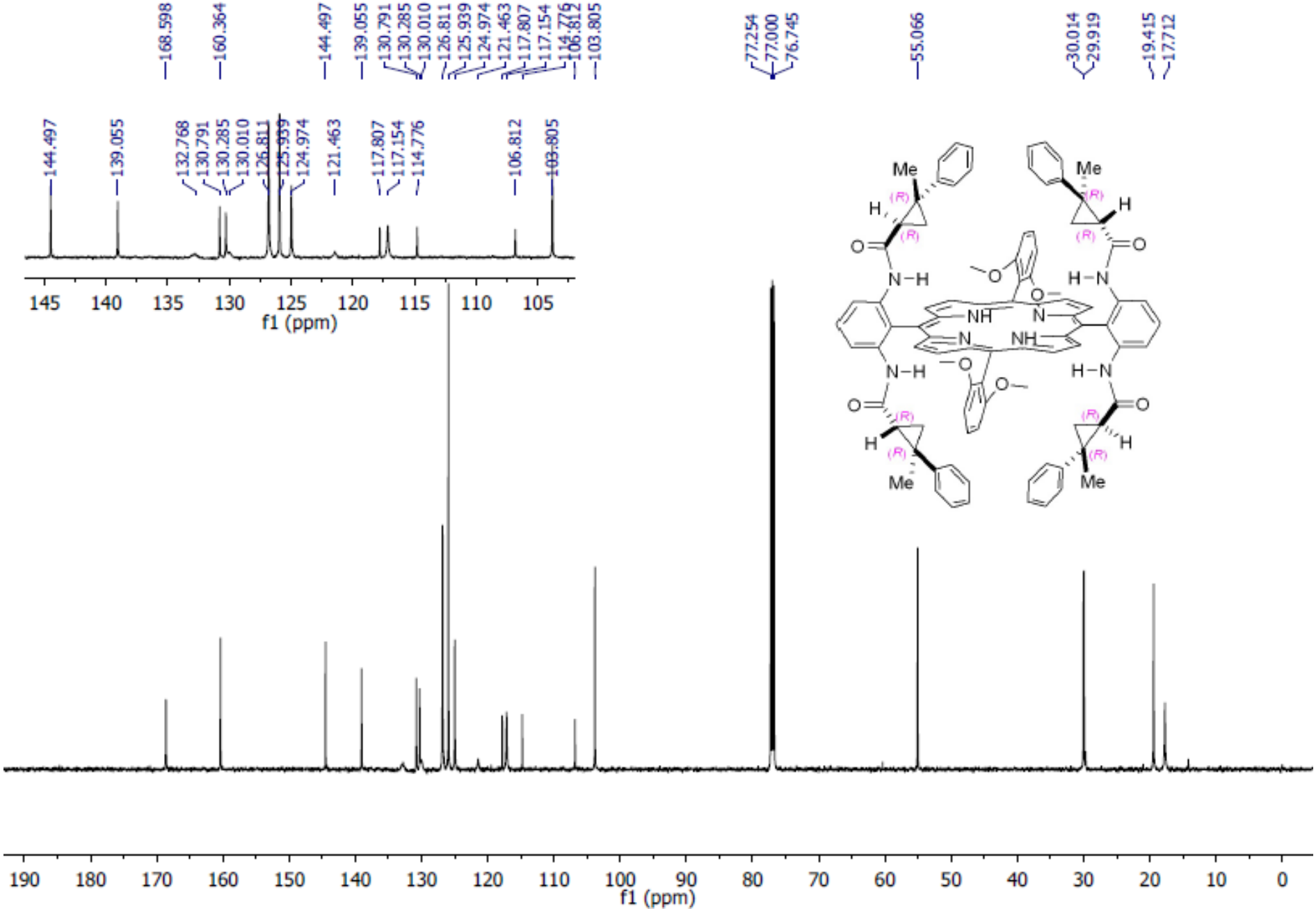
[7] P. Y. Chong, S. Z. Janicki, P. A. Petillo, *J. Org. Chem.* **1998**, *63*, 8515-8521.

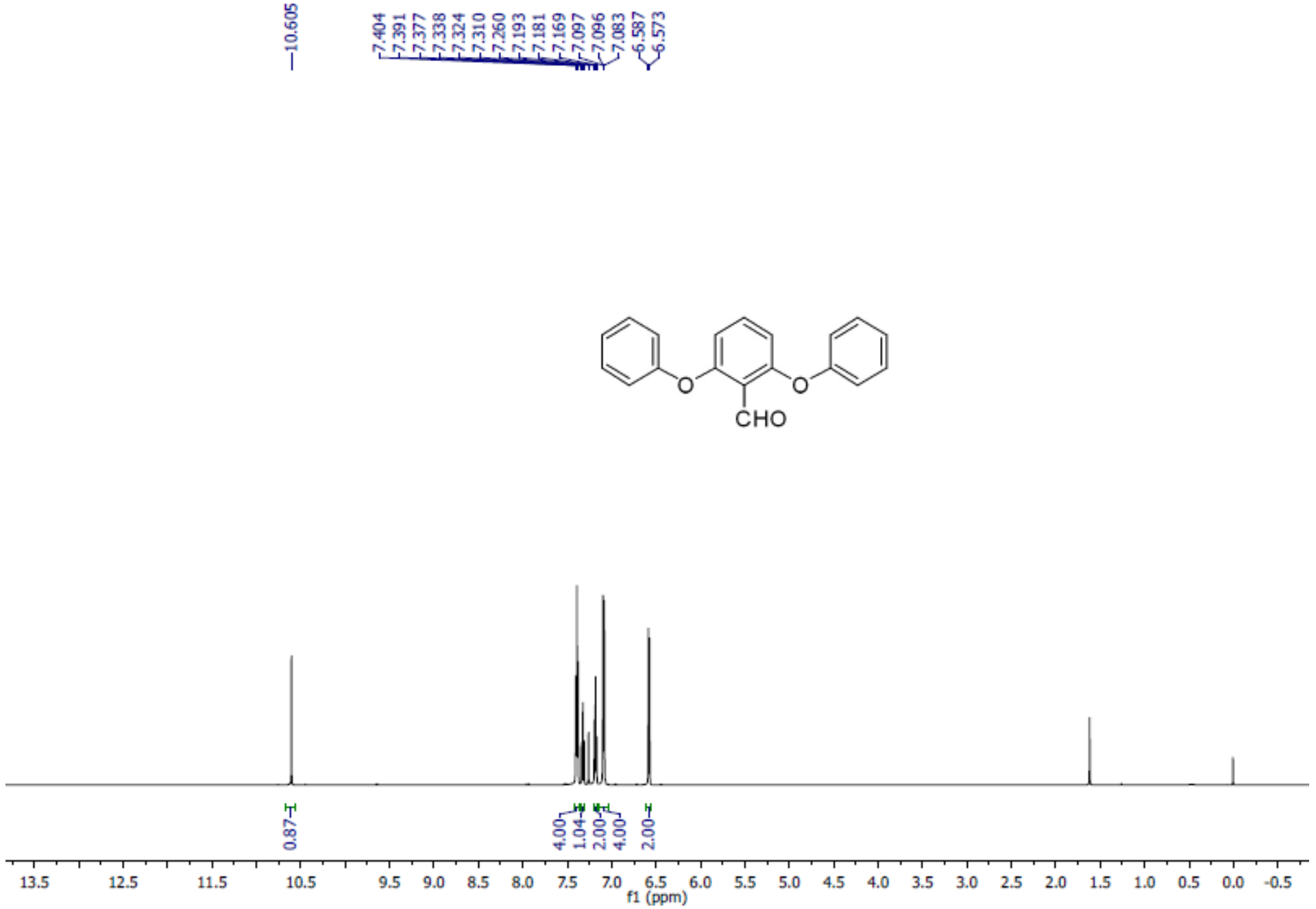
[H_2(2,6-DiMeO-QingPhyrin)] ([H_2(P5)])



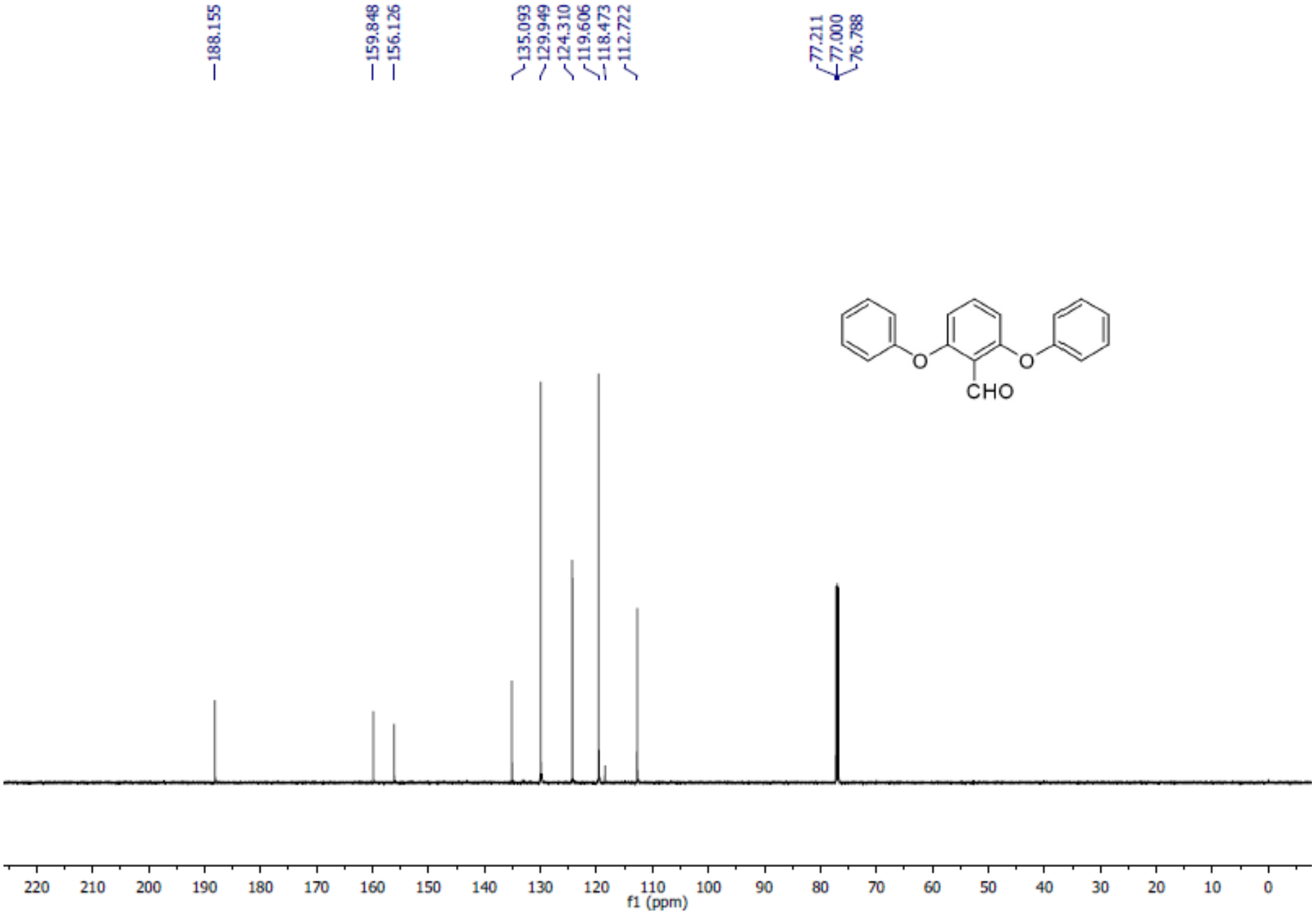
[H_2(2,6-DiMeO-QingPhyrin)] ([H_2(P5)])



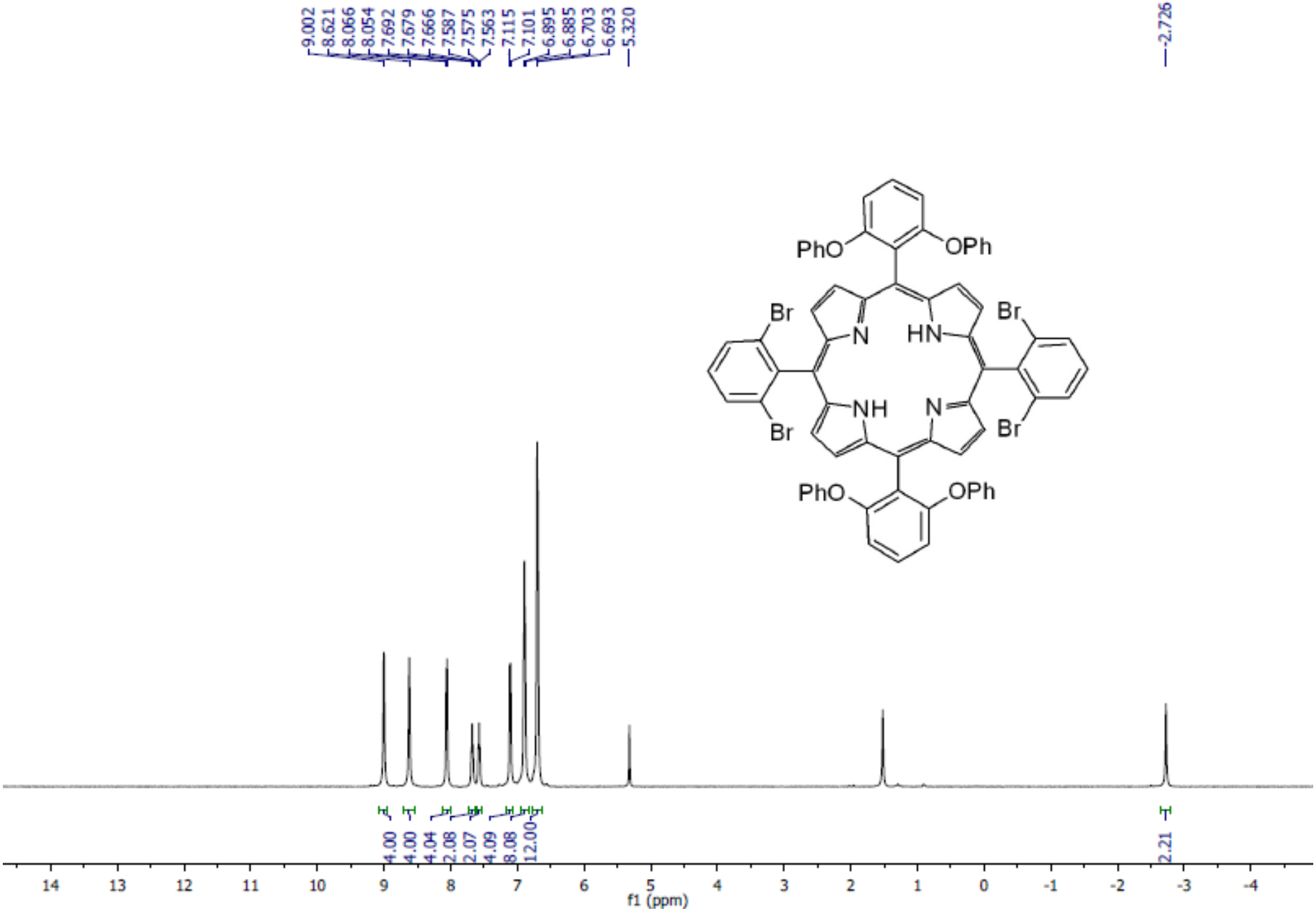
2,6-diphenoxybenzaldehyde



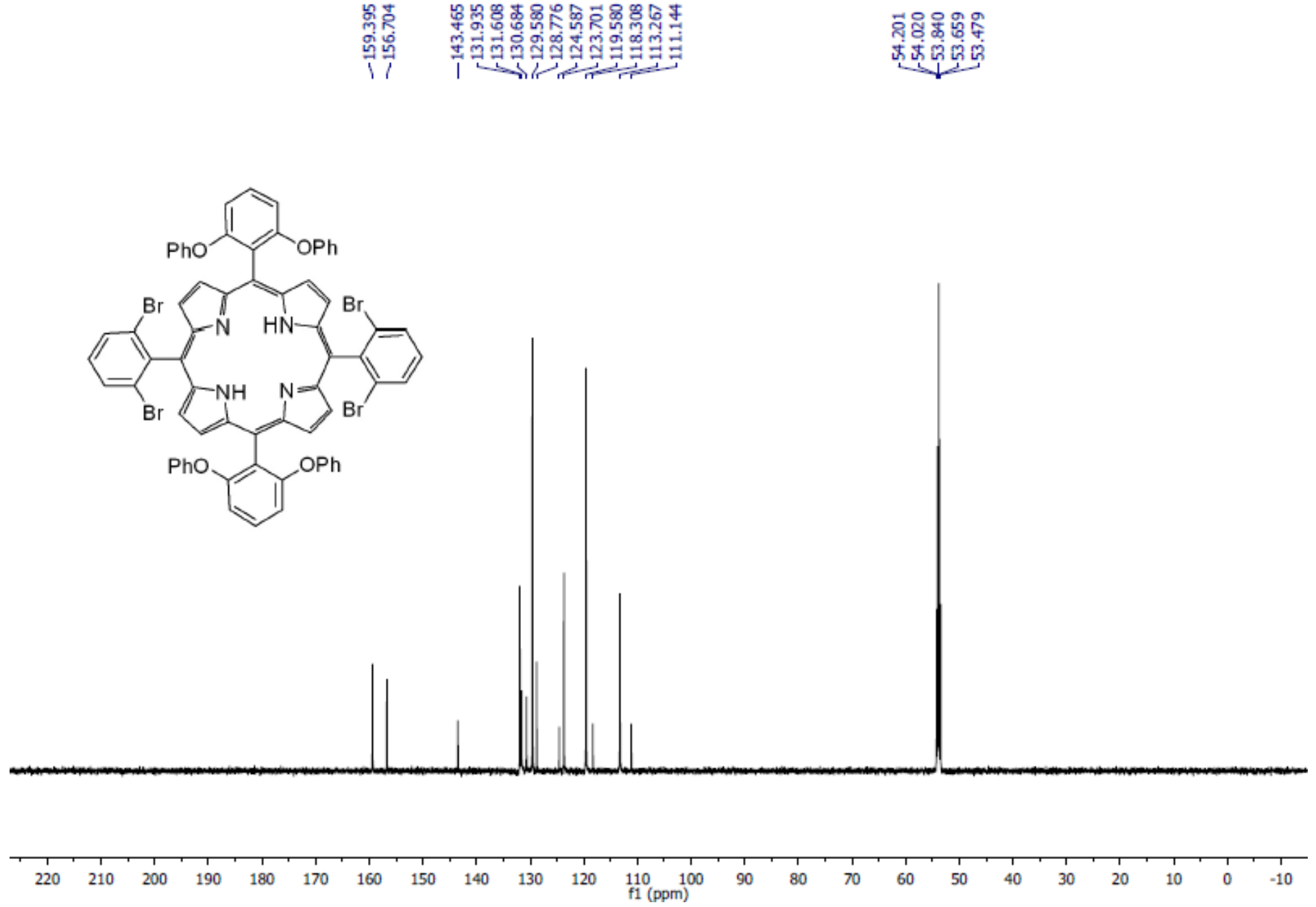
2,6-diphenoxybenzaldehyde



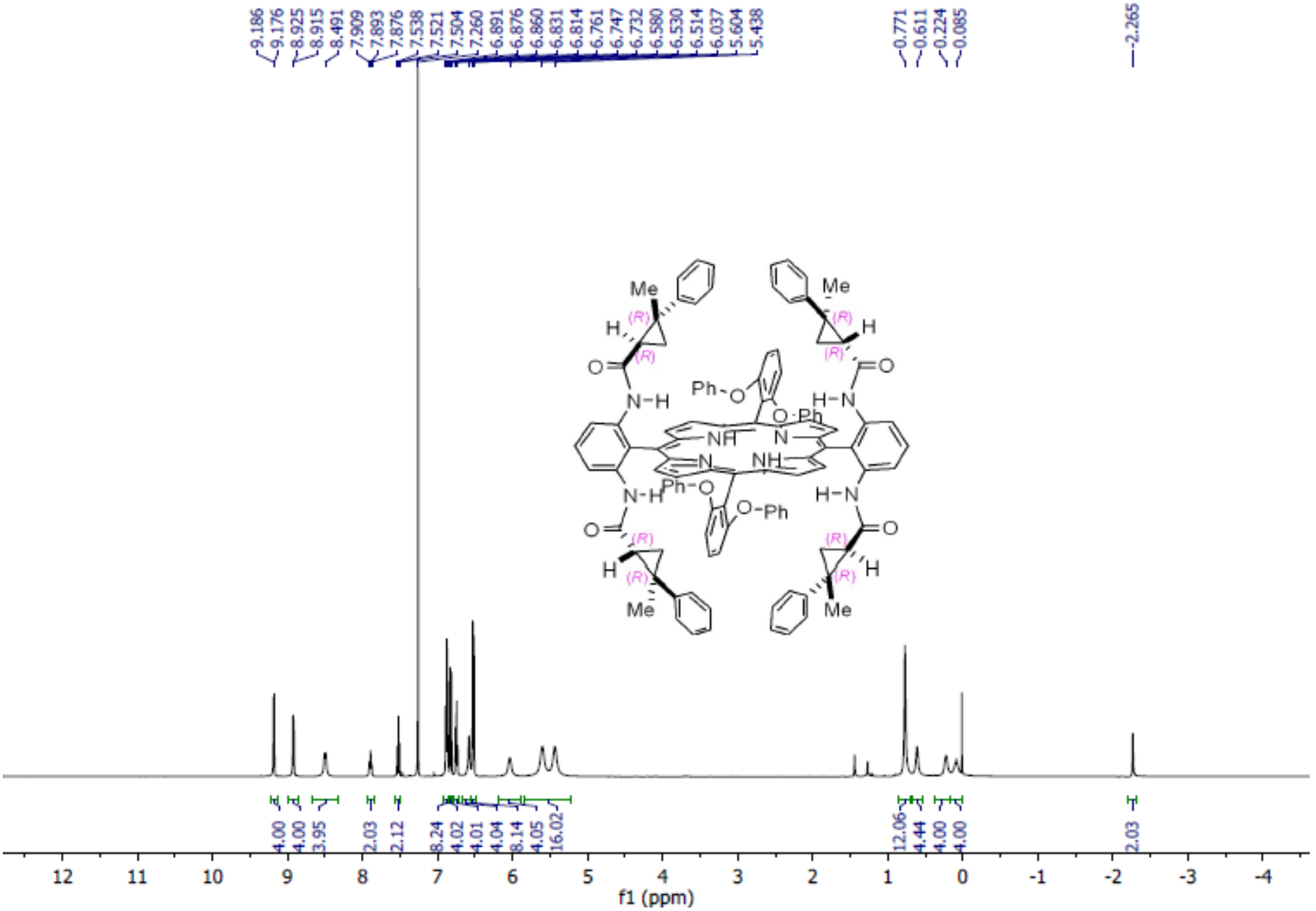
5,15-bis(2,6-dibromophenyl)-10,20-bis(2,6-diphenoxyphenyl)porphyrin in CD_2Cl_2



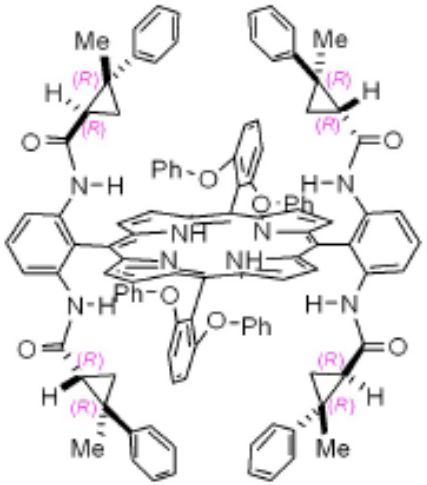
5,15-bis(2,6-dibromophenyl)-10,20-bis(2,6-diphenoxyphenyl)porphyrin in CD_2Cl_2



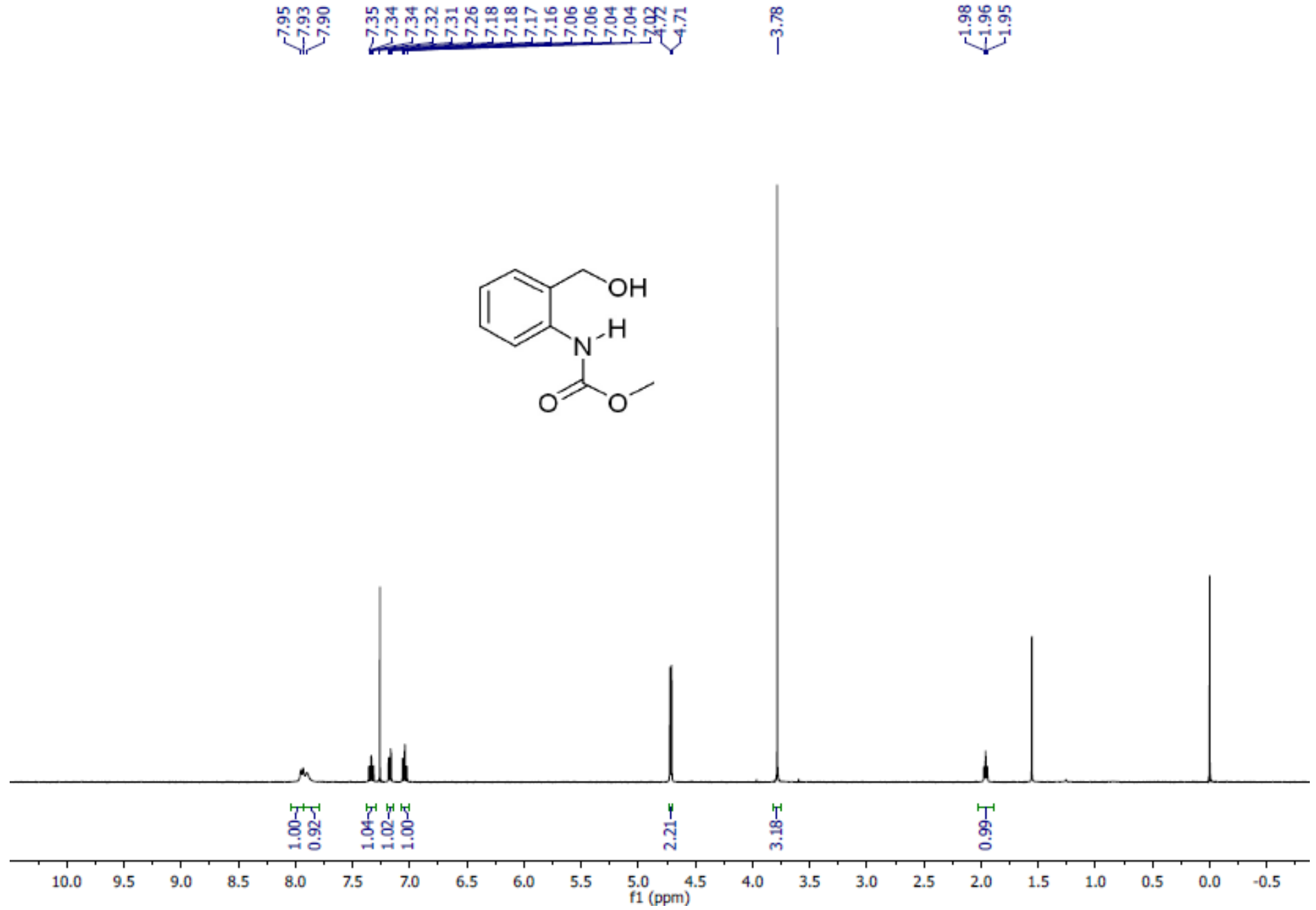
[H_2(2,6-DiPhO-QingPhyrin)] ([H_2(P6)])



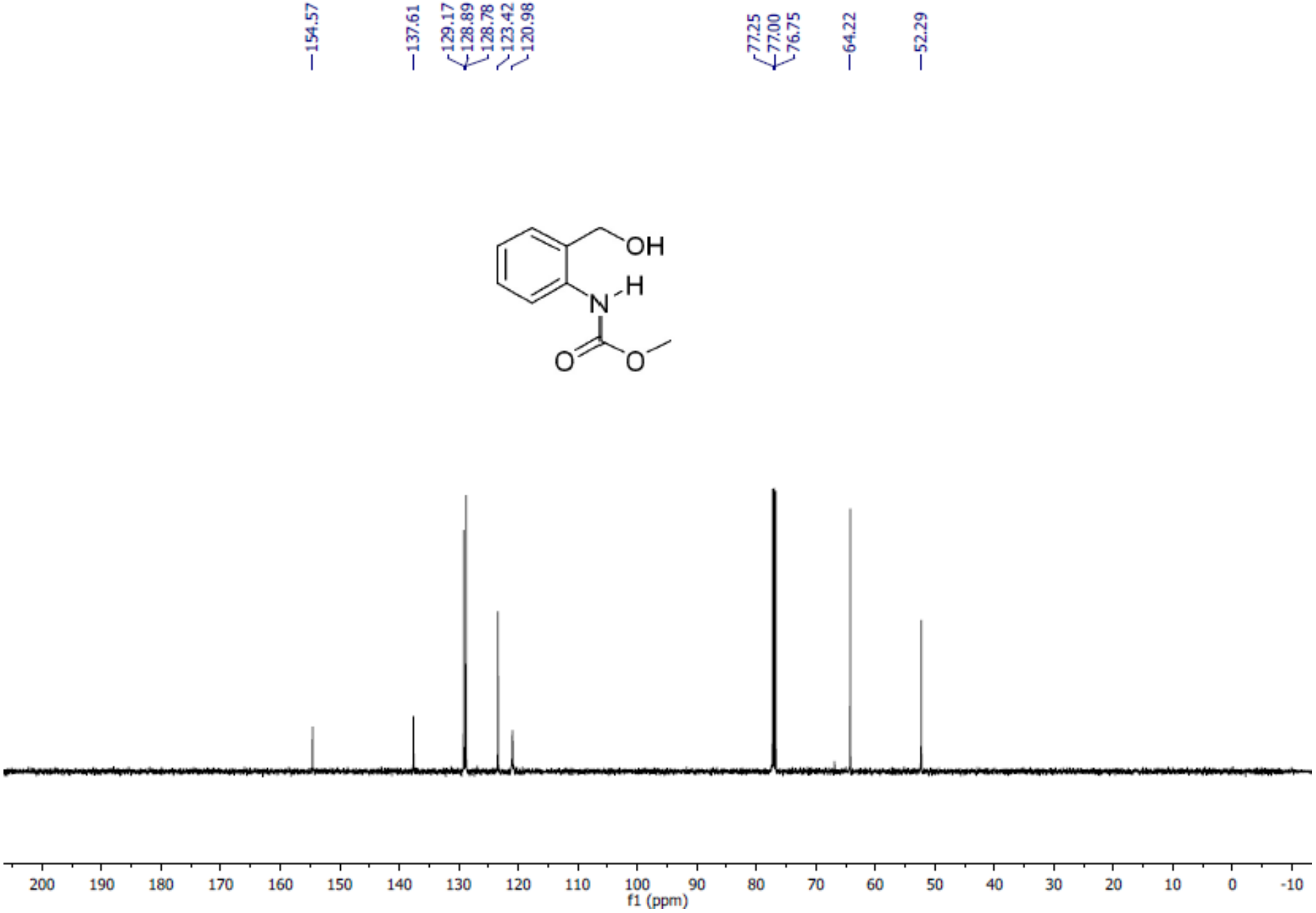
[H_2(2,6-DiPhO-QingPhyrin)] ([H_2(P6)])



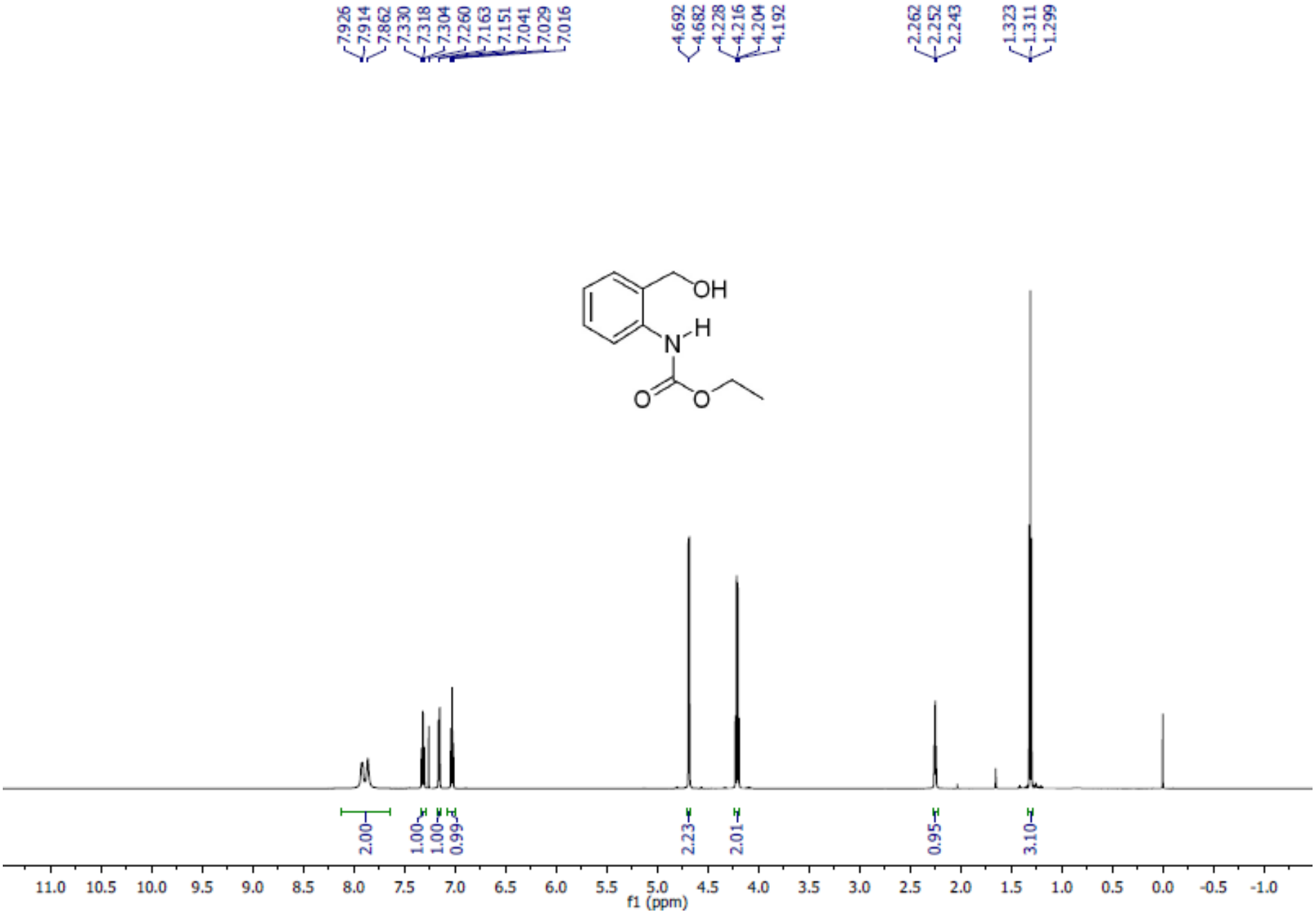
methyl (2-(hydroxymethyl)phenyl)carbamate s1-a



methyl (2-(hydroxymethyl)phenyl)carbamate s1-a



ethyl (2-(hydroxymethyl)phenyl)carbamate s1-b



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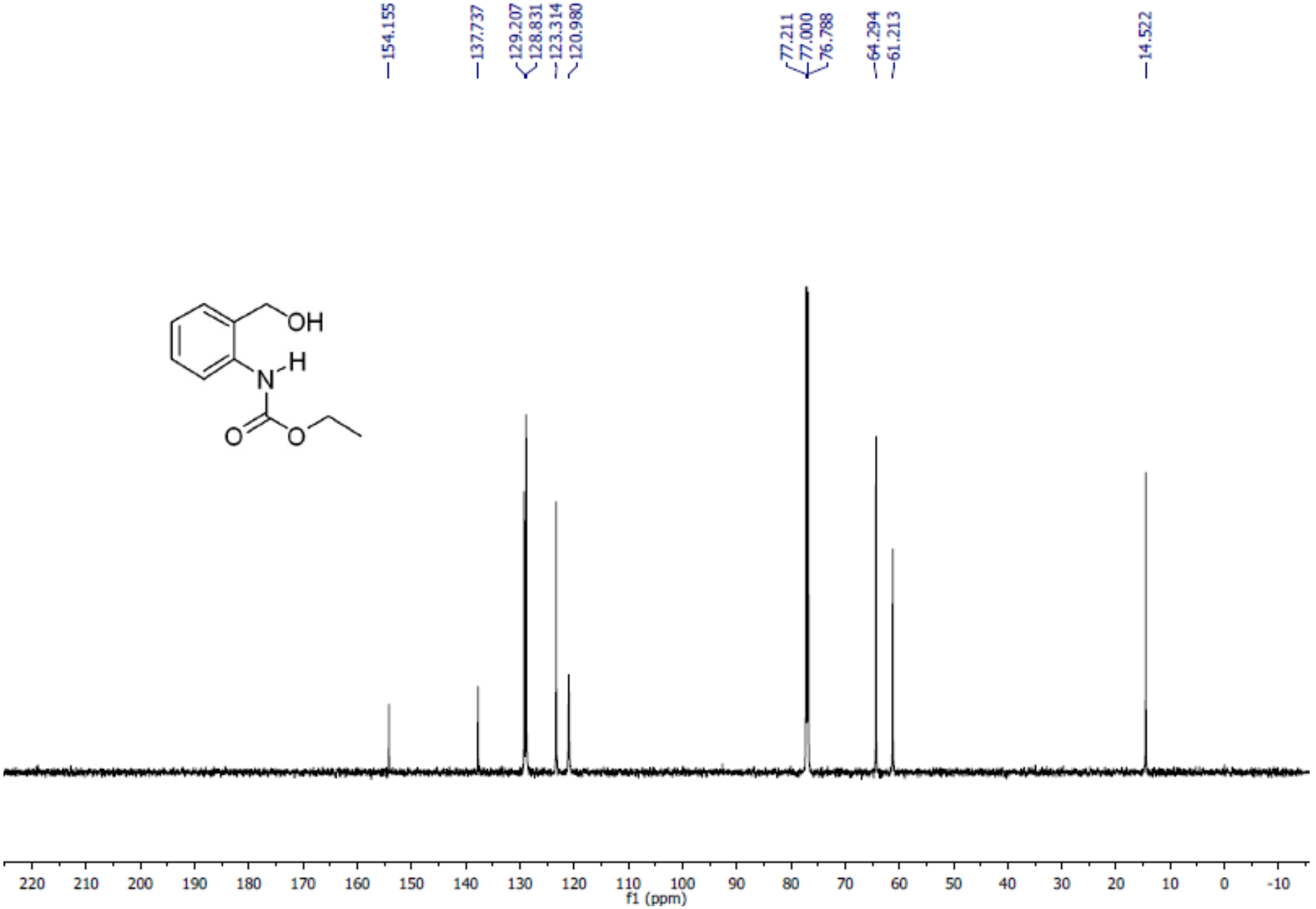
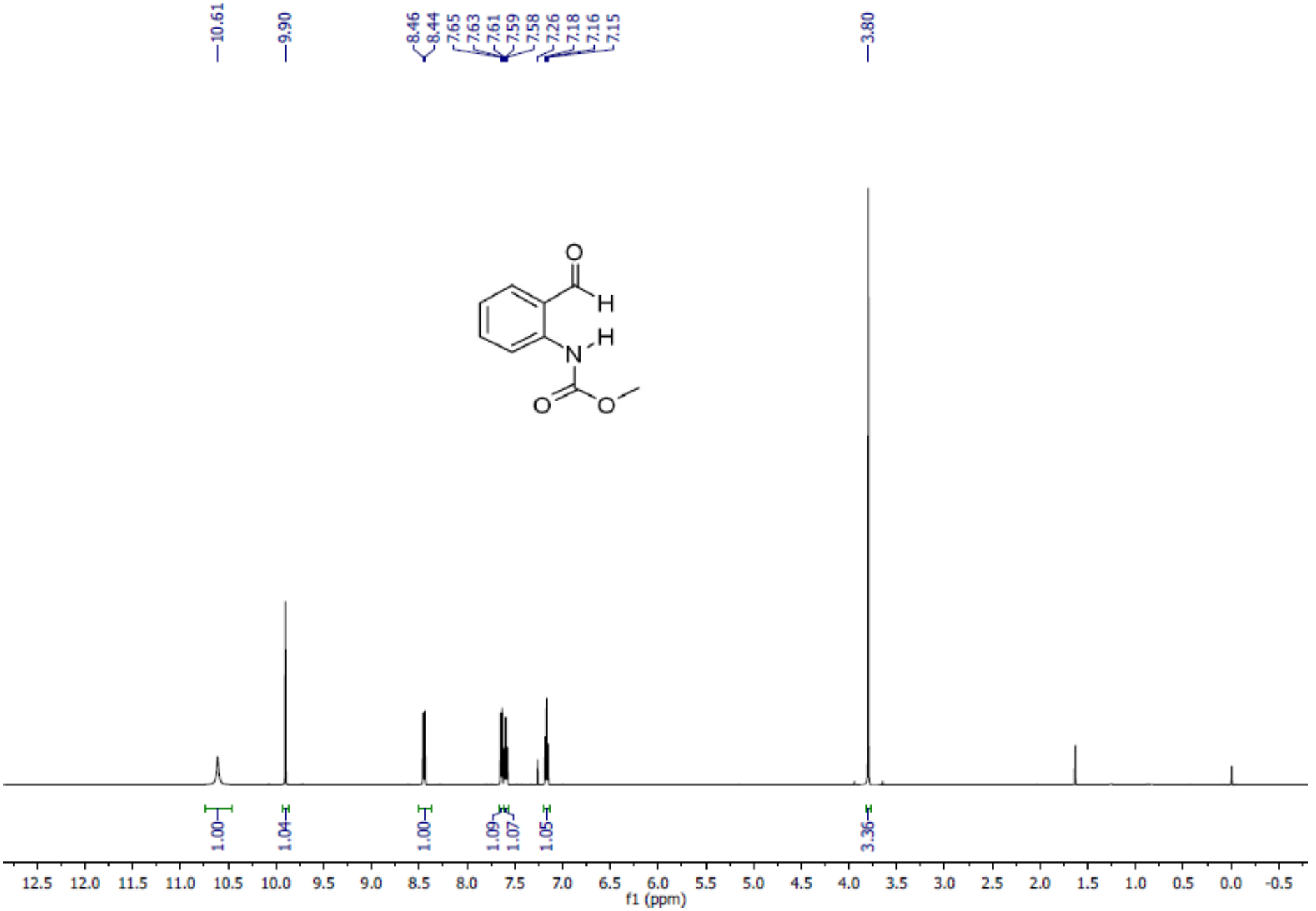


methyl (2-formylphenyl)carbamate s2-a



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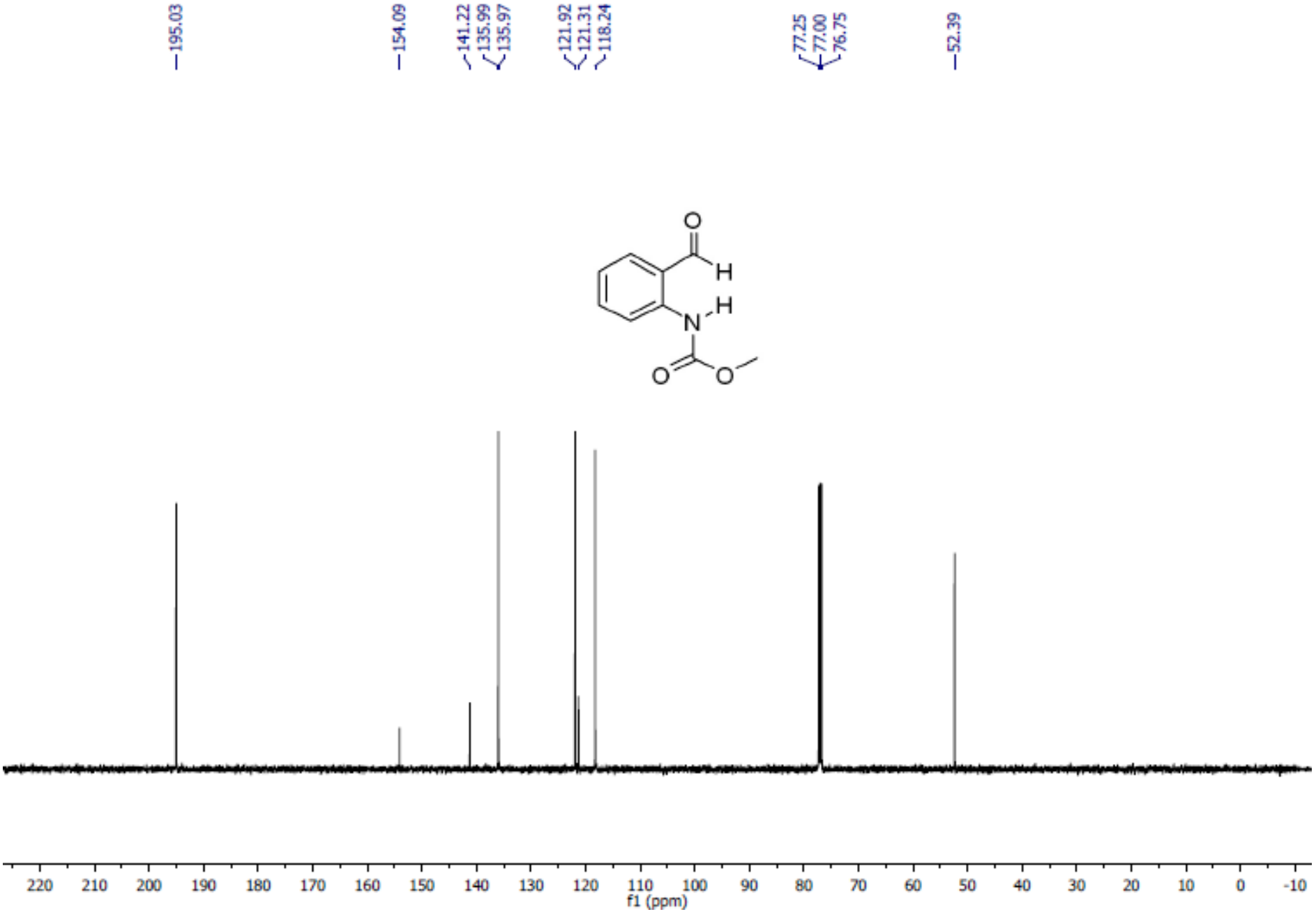

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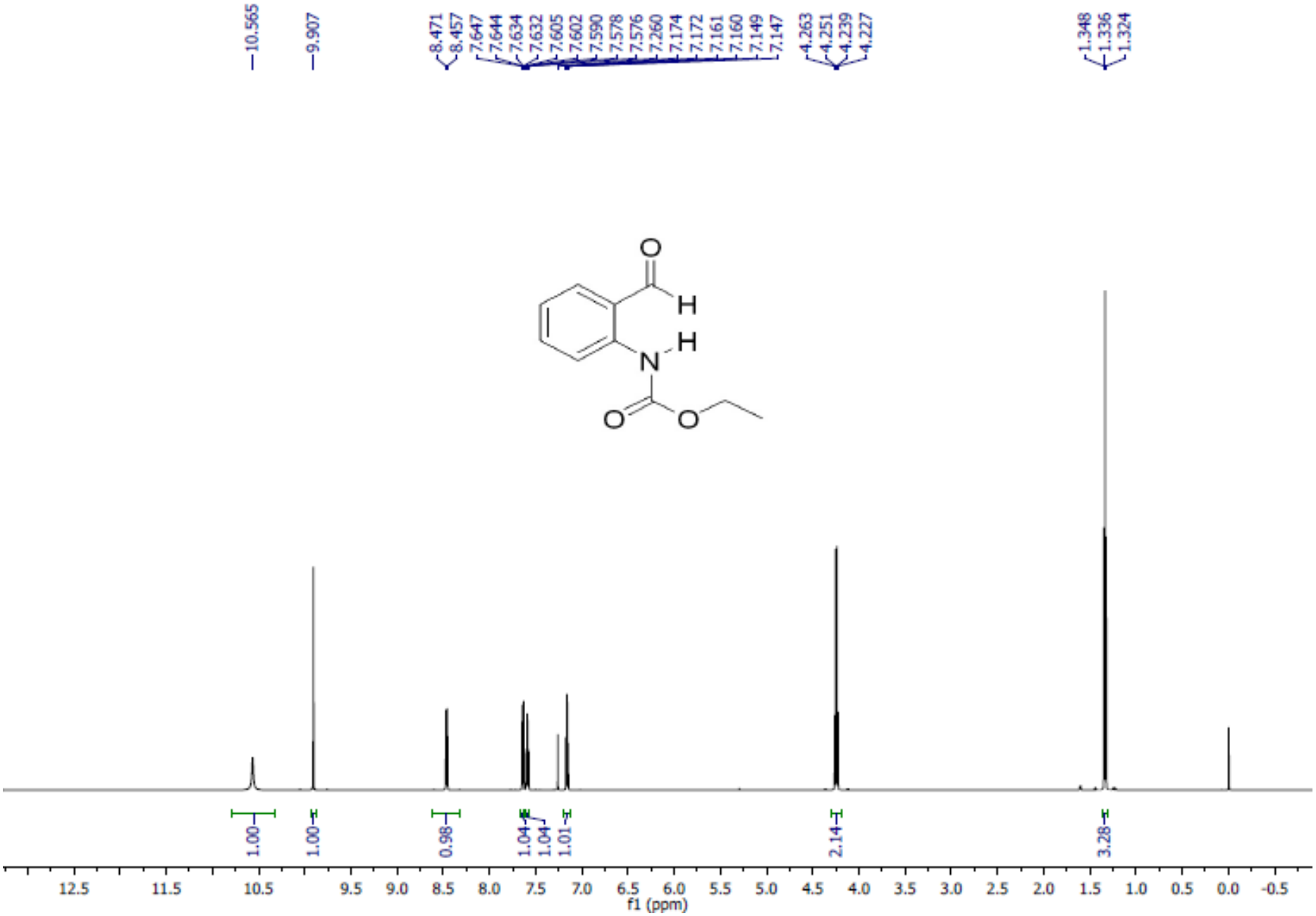
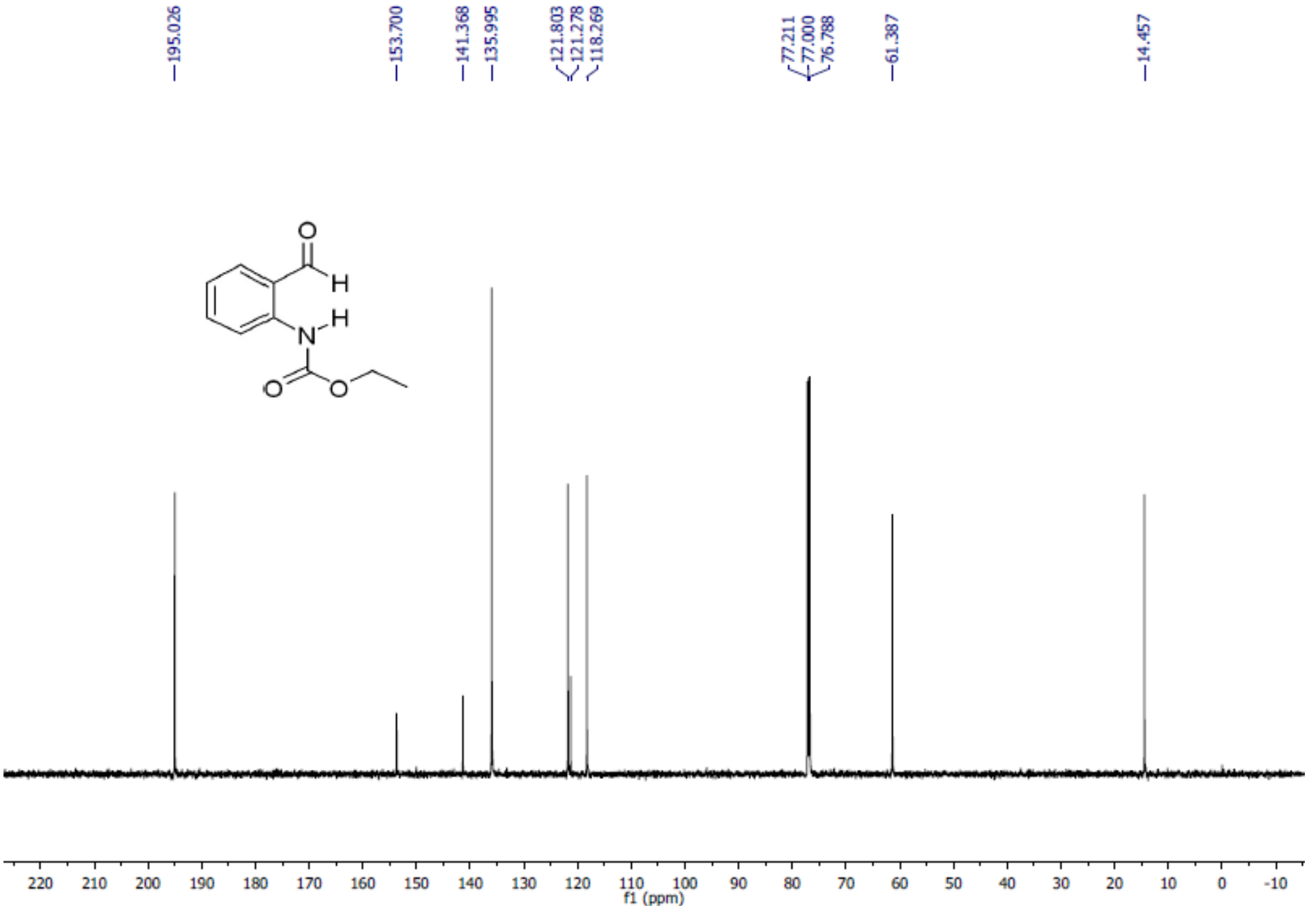
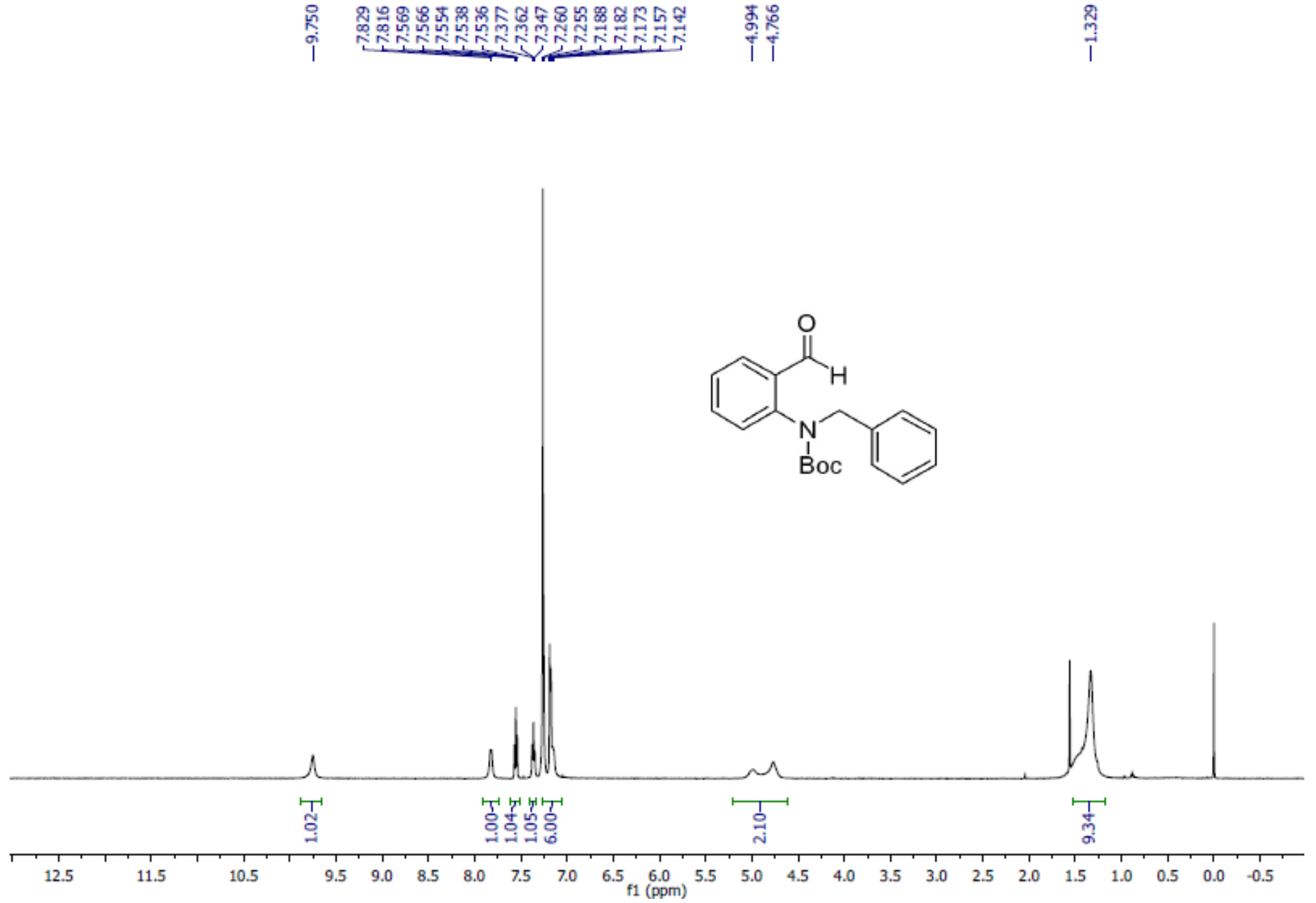


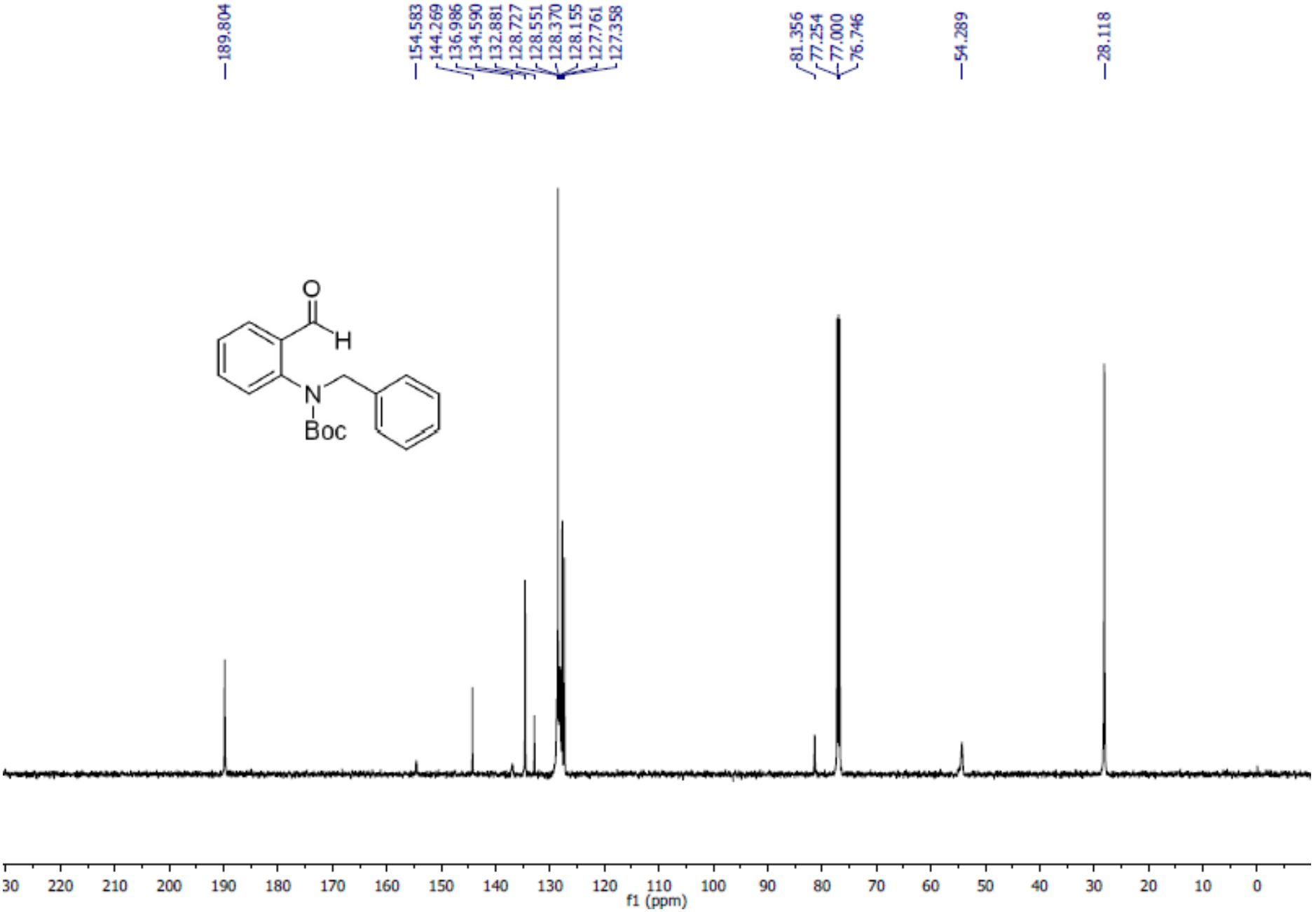
ethyl (2-formylphenyl)carbamate s2-b



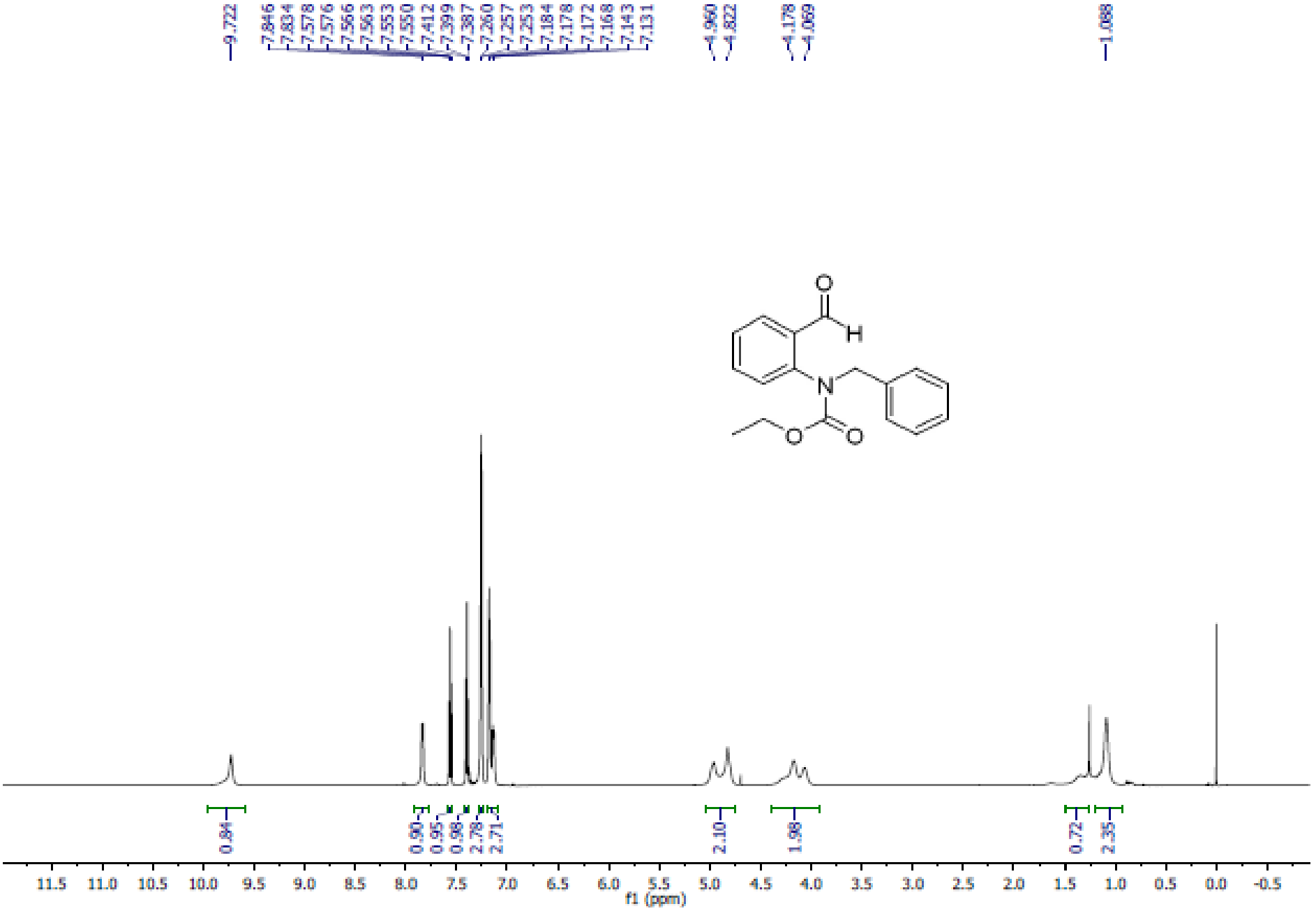
tert-butyl benzyl(2-formylphenyl)carbamate s3-a



tert-butyl benzyl(2-formylphenyl)carbamate s3-a



ethyl benzyl(2-formylphenyl)carbamate s3-b



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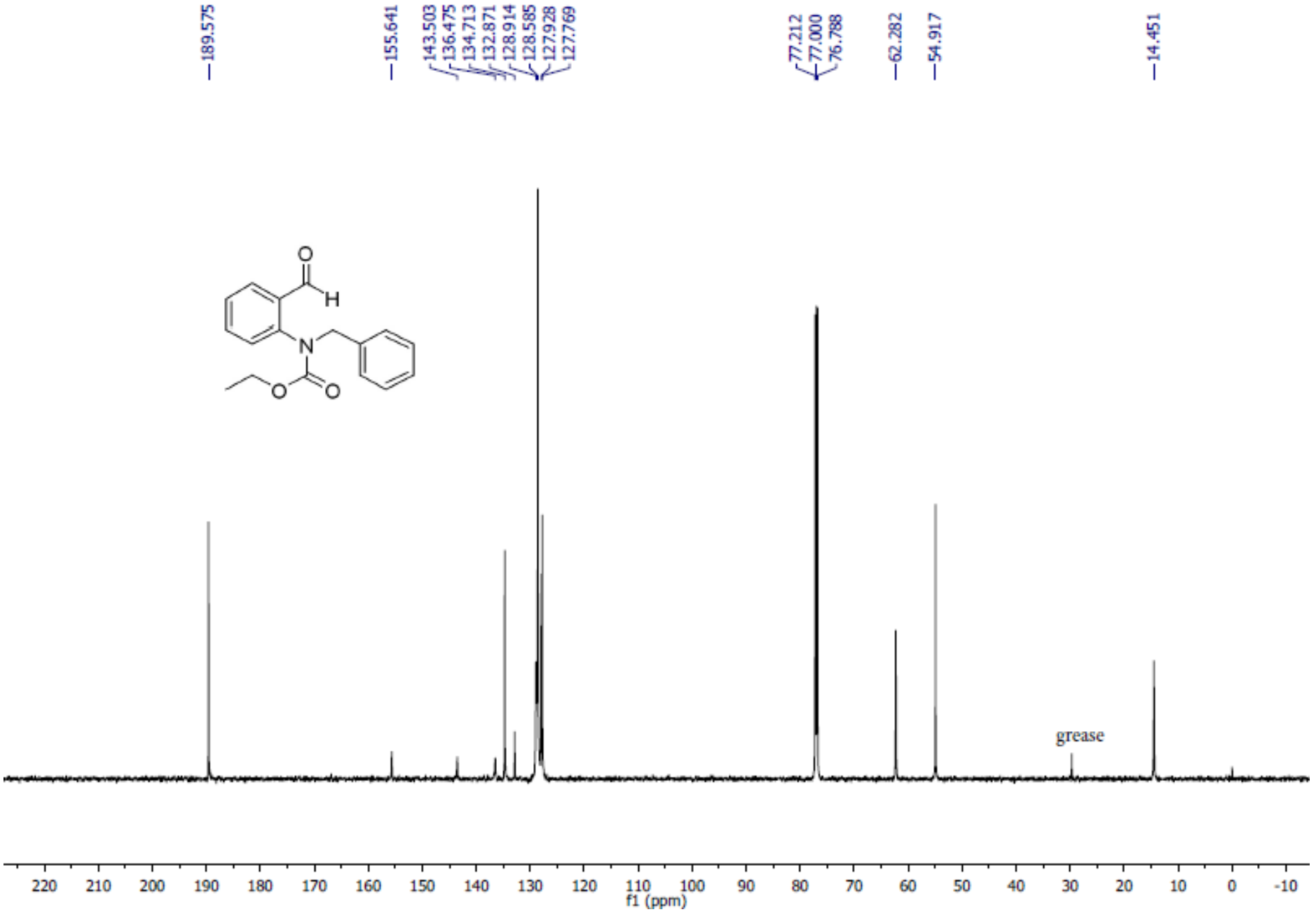
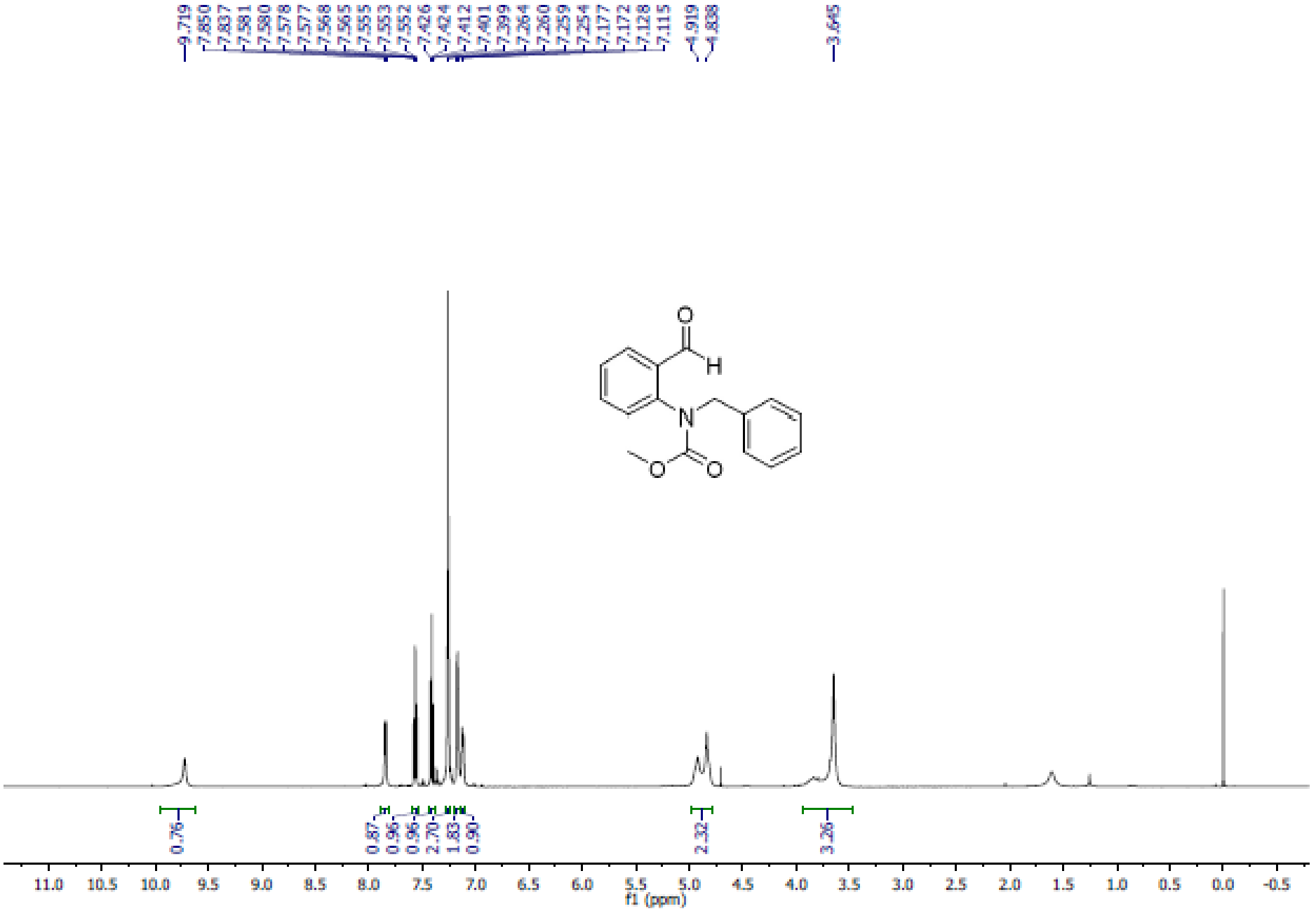
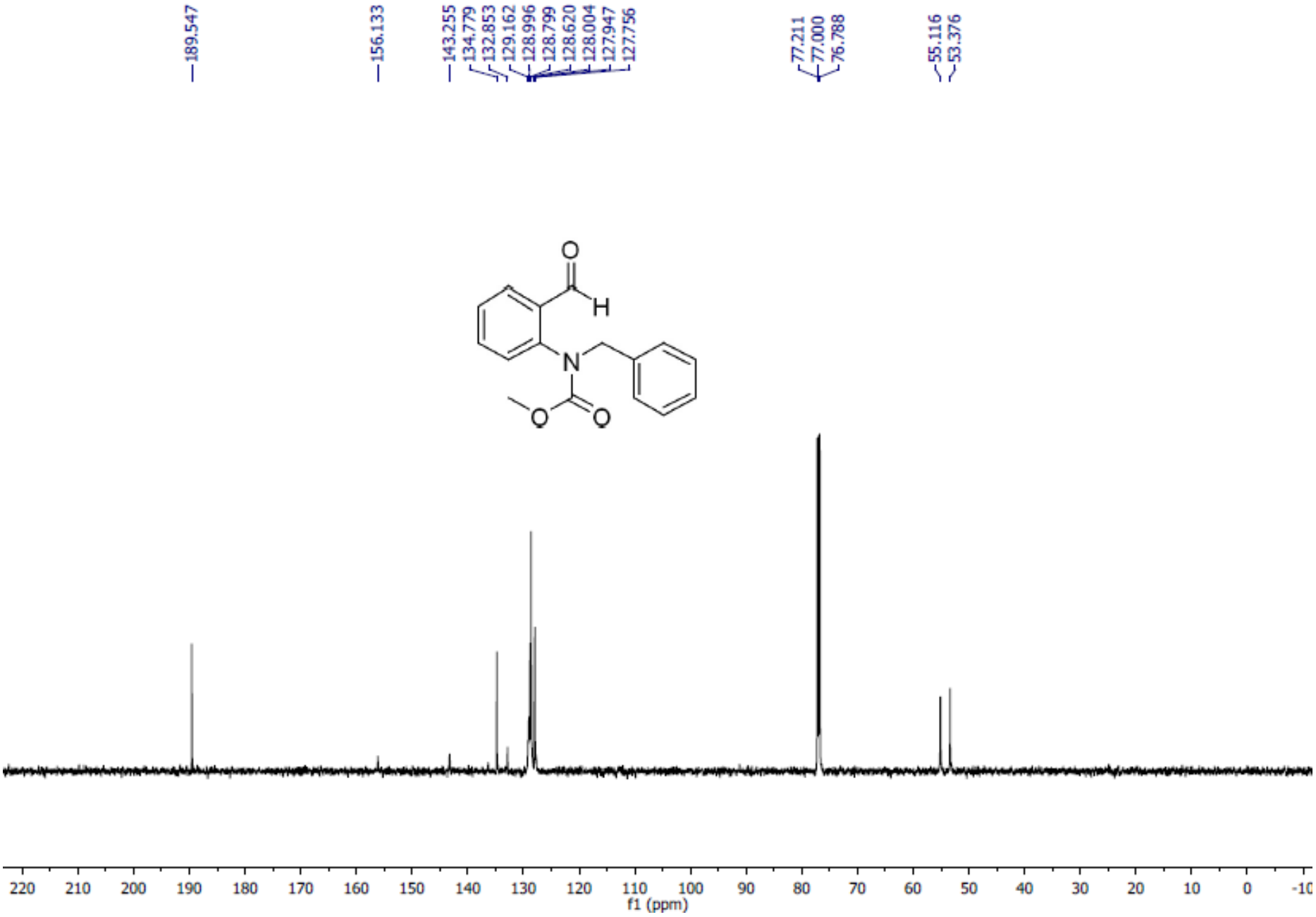


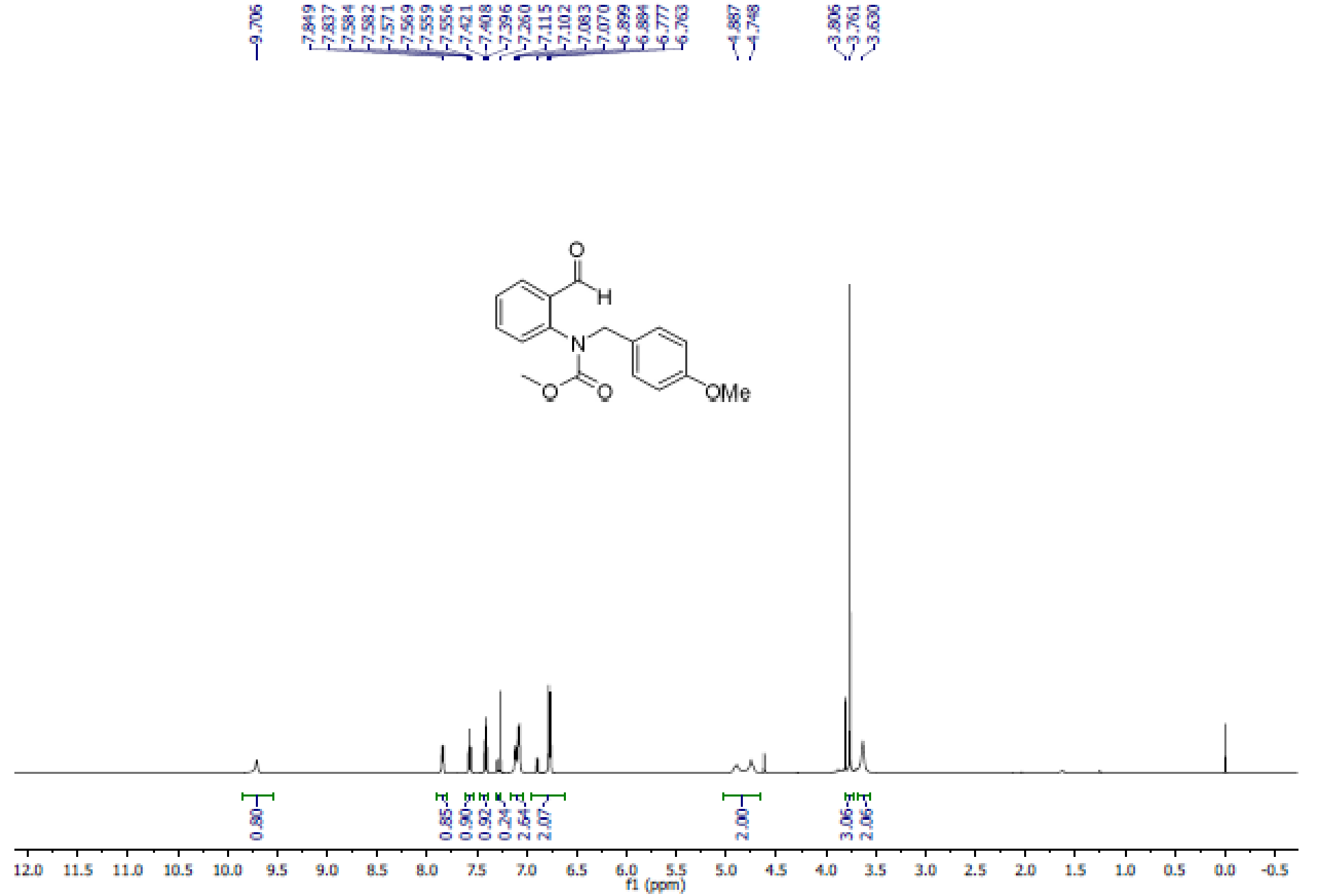
methyl benzyl(2-formylphenyl)carbamate s3-c



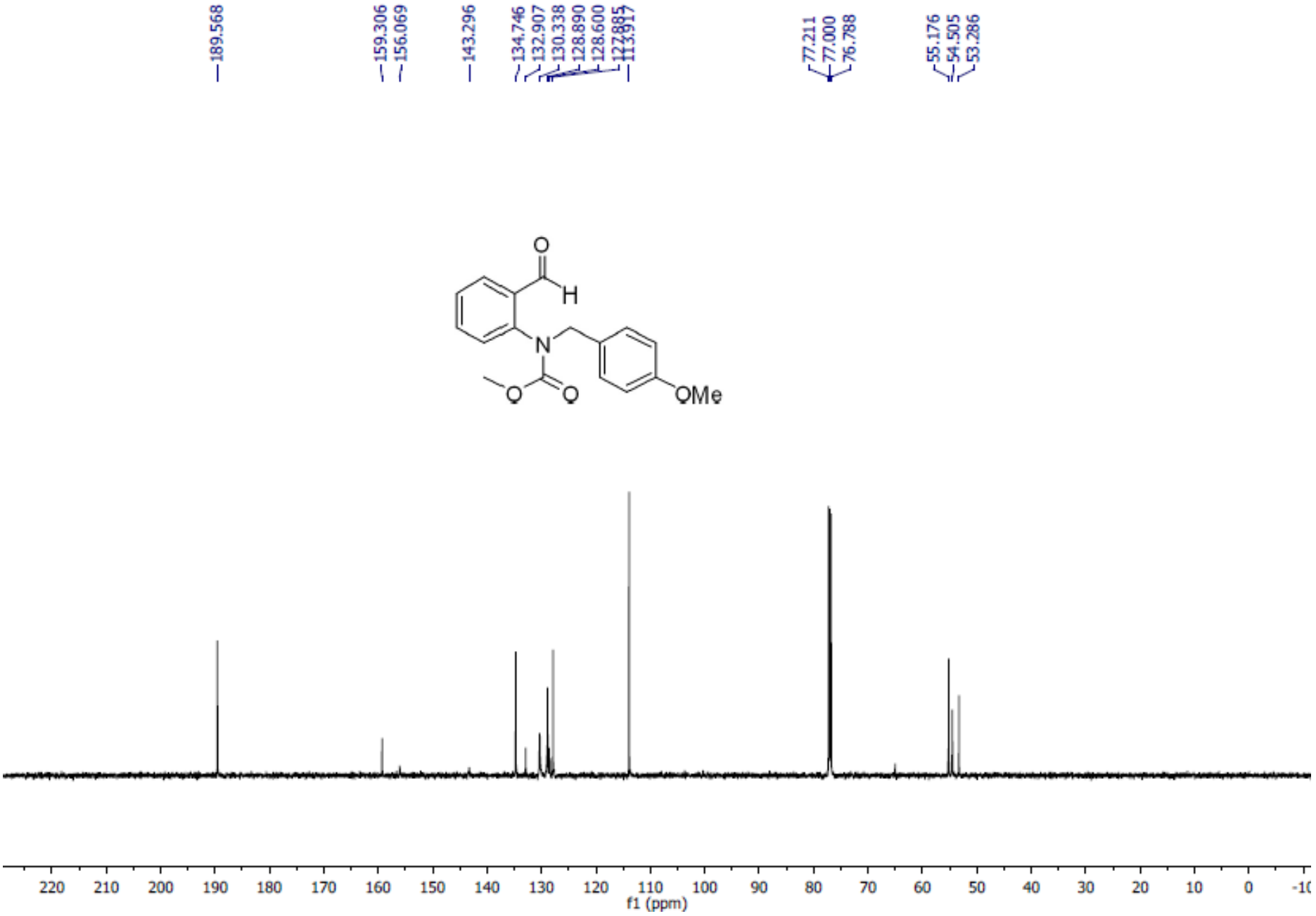
methyl benzyl(2-formylphenyl)carbamate s3-c



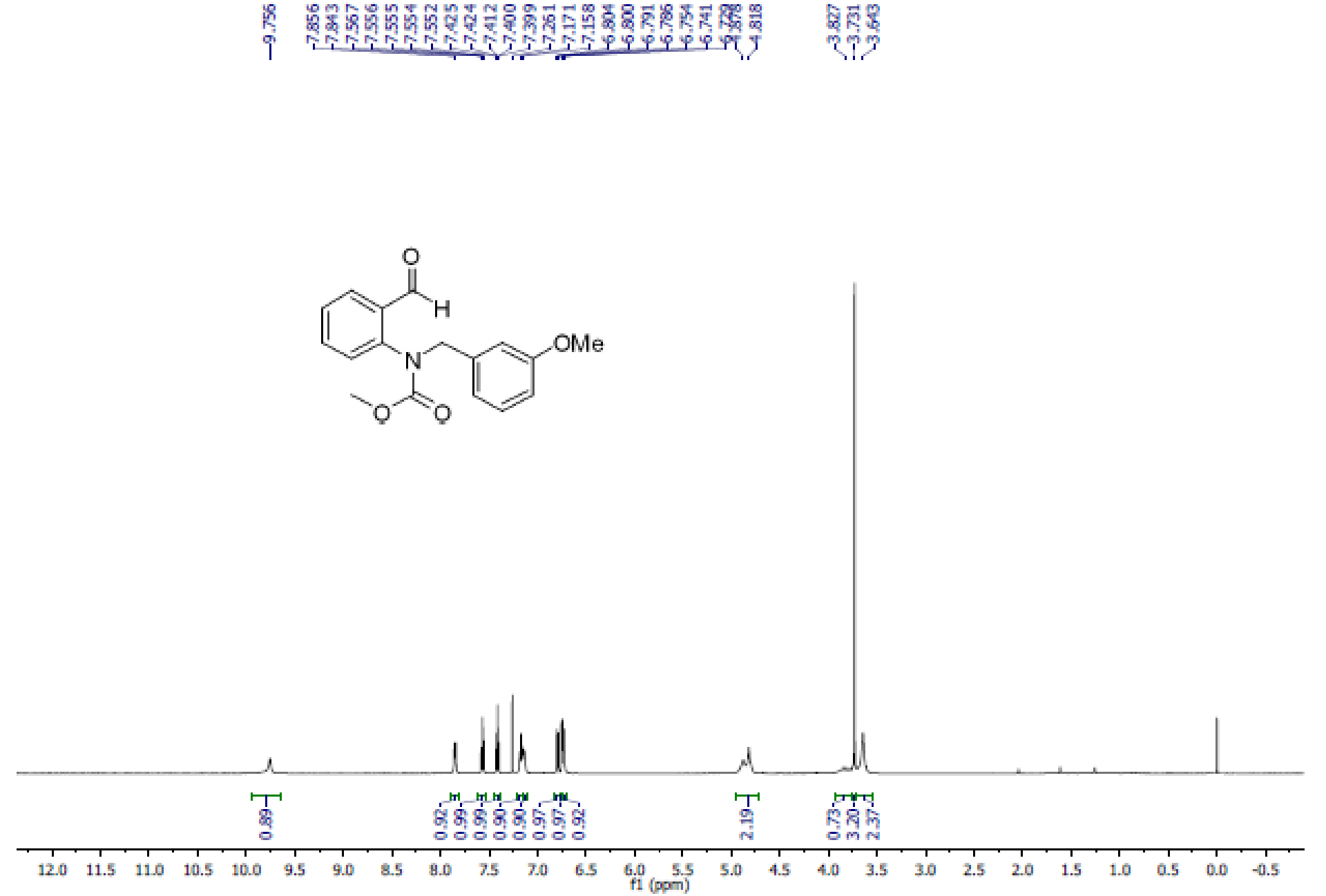
methyl (2-formylphenyl)(4-methoxybenzyl)carbamate s3-d



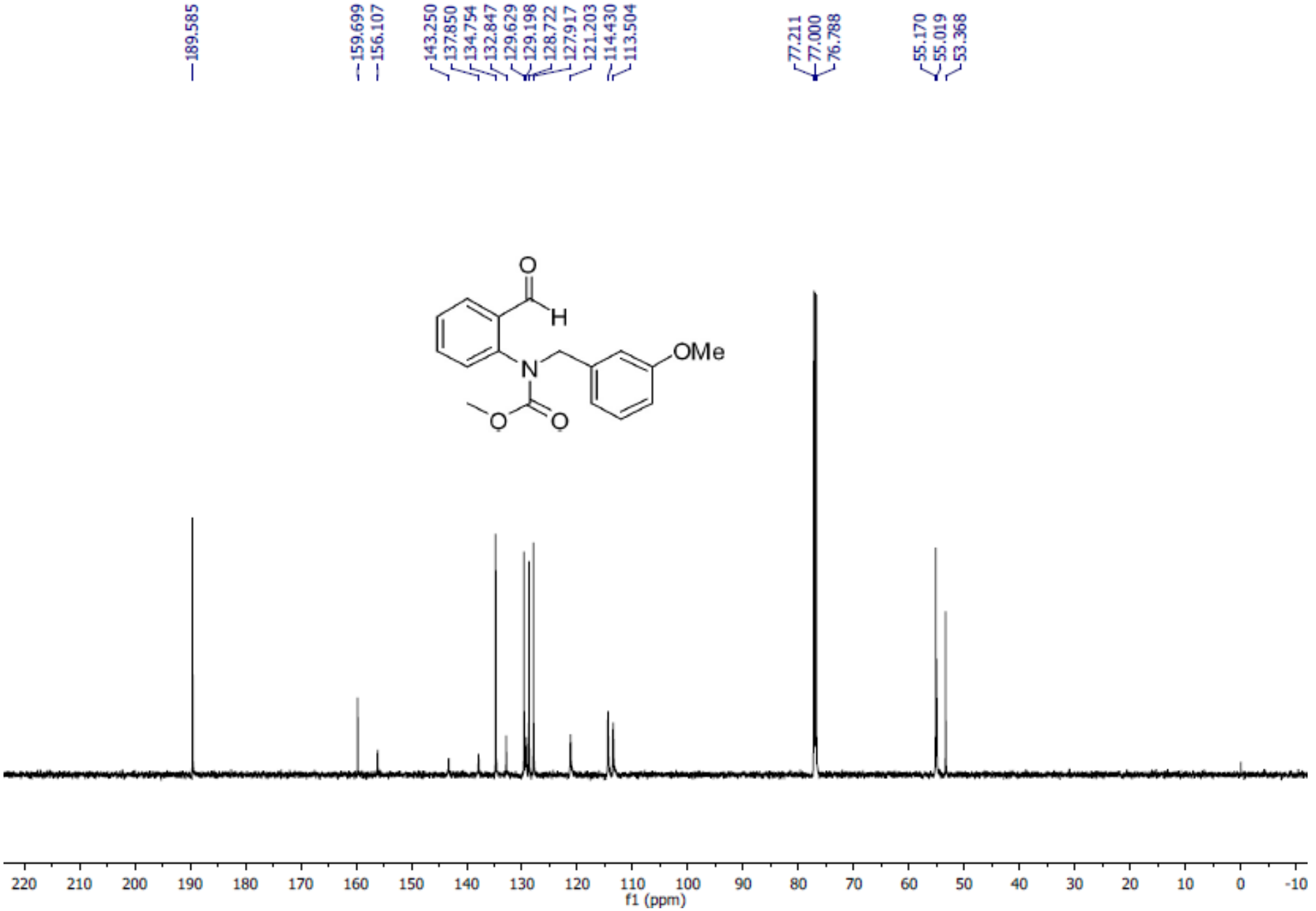
methyl (2-formylphenyl)(4-methoxybenzyl)carbamate s3-d



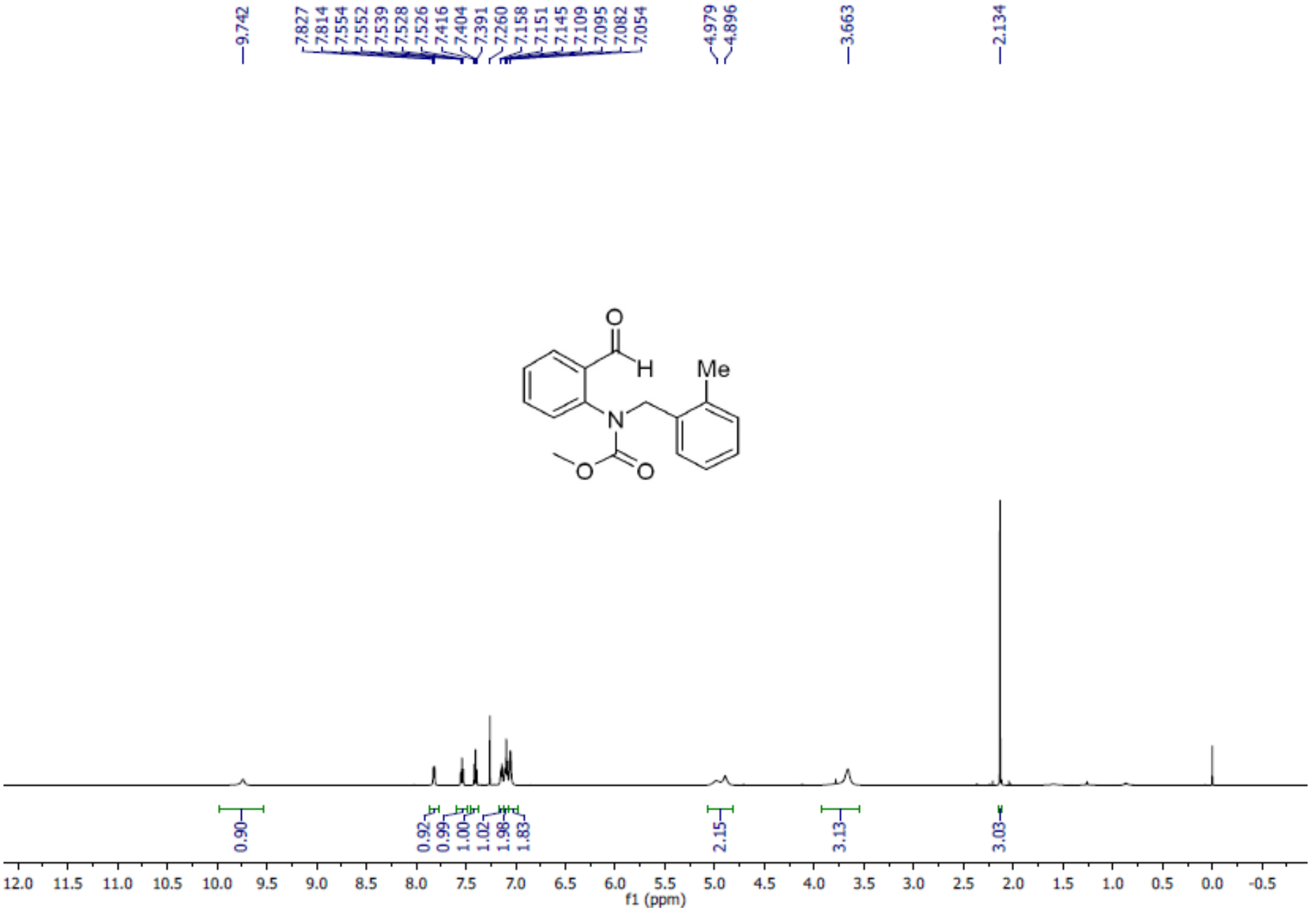
methyl (2-formylphenyl)(3-methoxybenzyl)carbamate s3-e



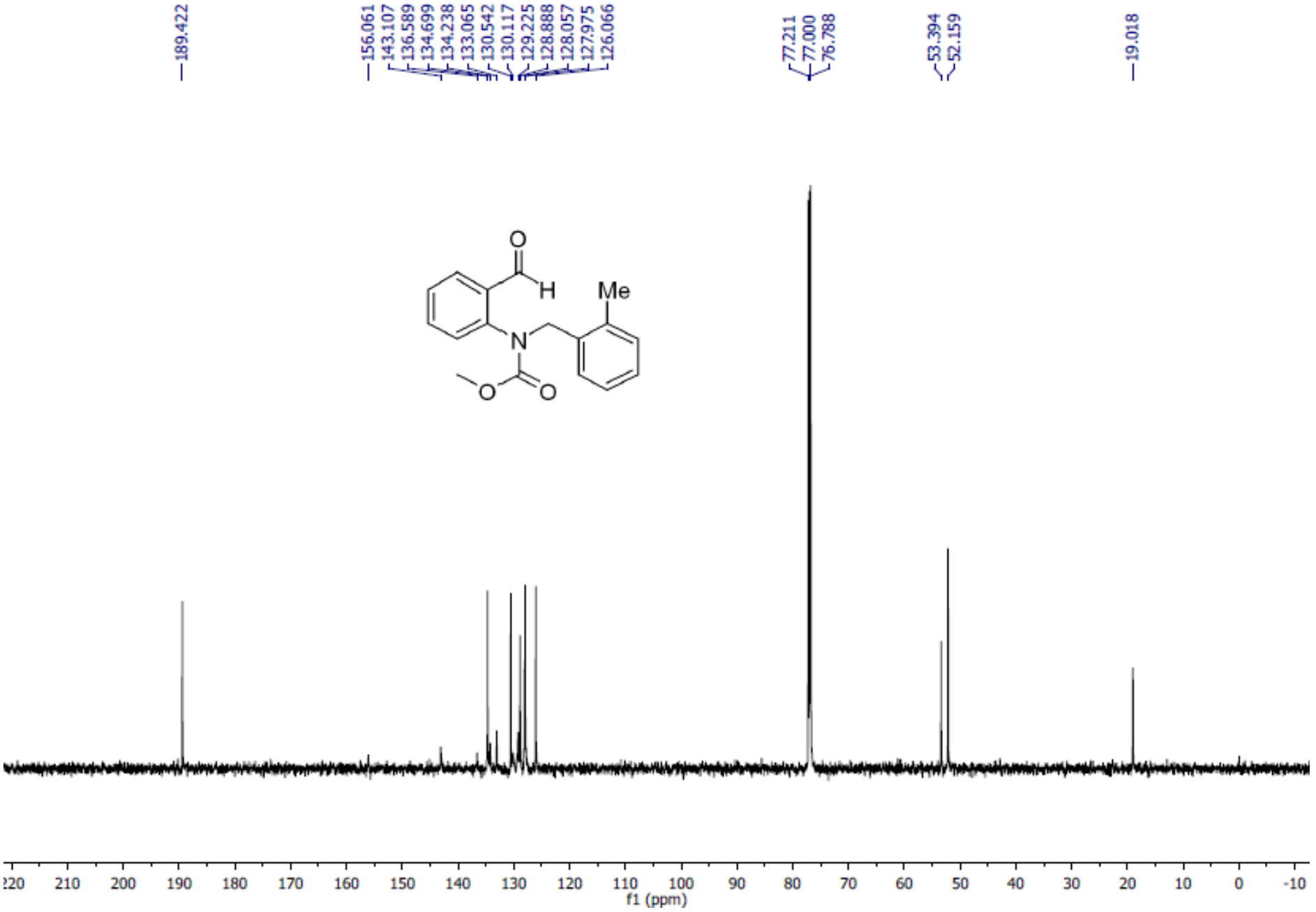
methyl (2-formylphenyl)(3-methoxybenzyl)carbamate s3-e



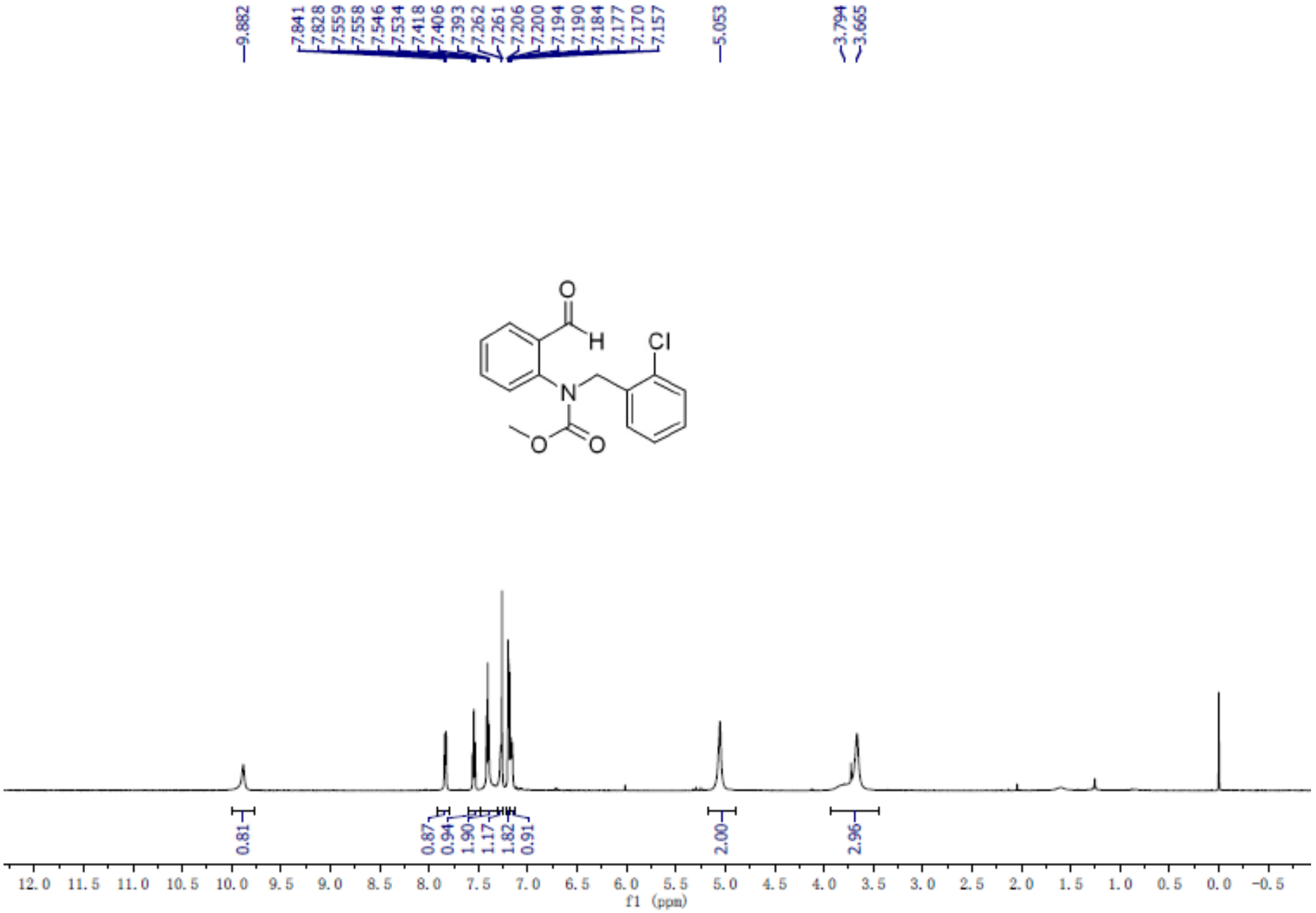
methyl (2-formylphenyl)(2-methylbenzyl)carbamate s3-f



methyl (2-formylphenyl)(2-methylbenzyl)carbamate s3-f



methyl (2-chlorobenzyl)(2-formylphenyl)carbamate s3-g



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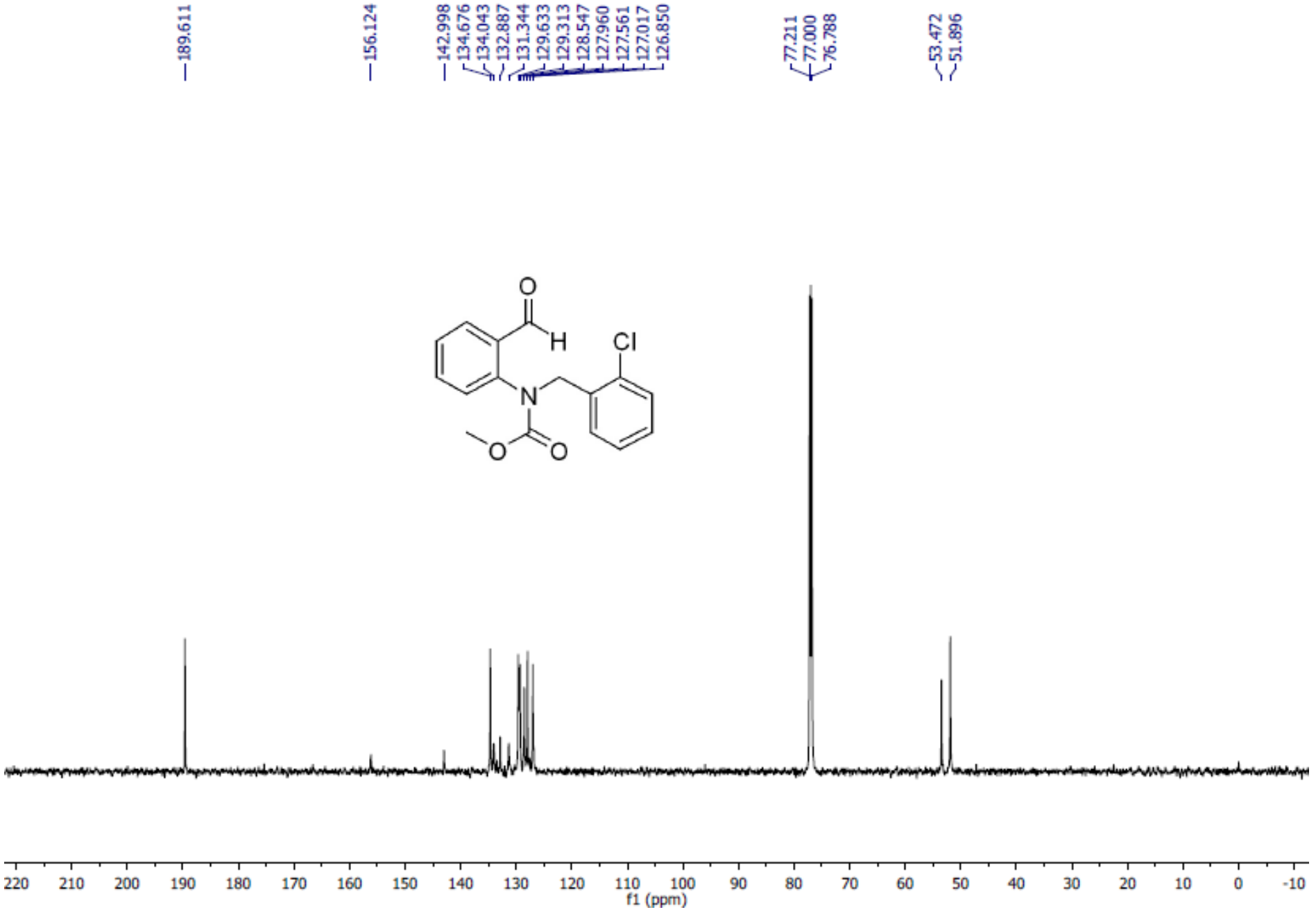
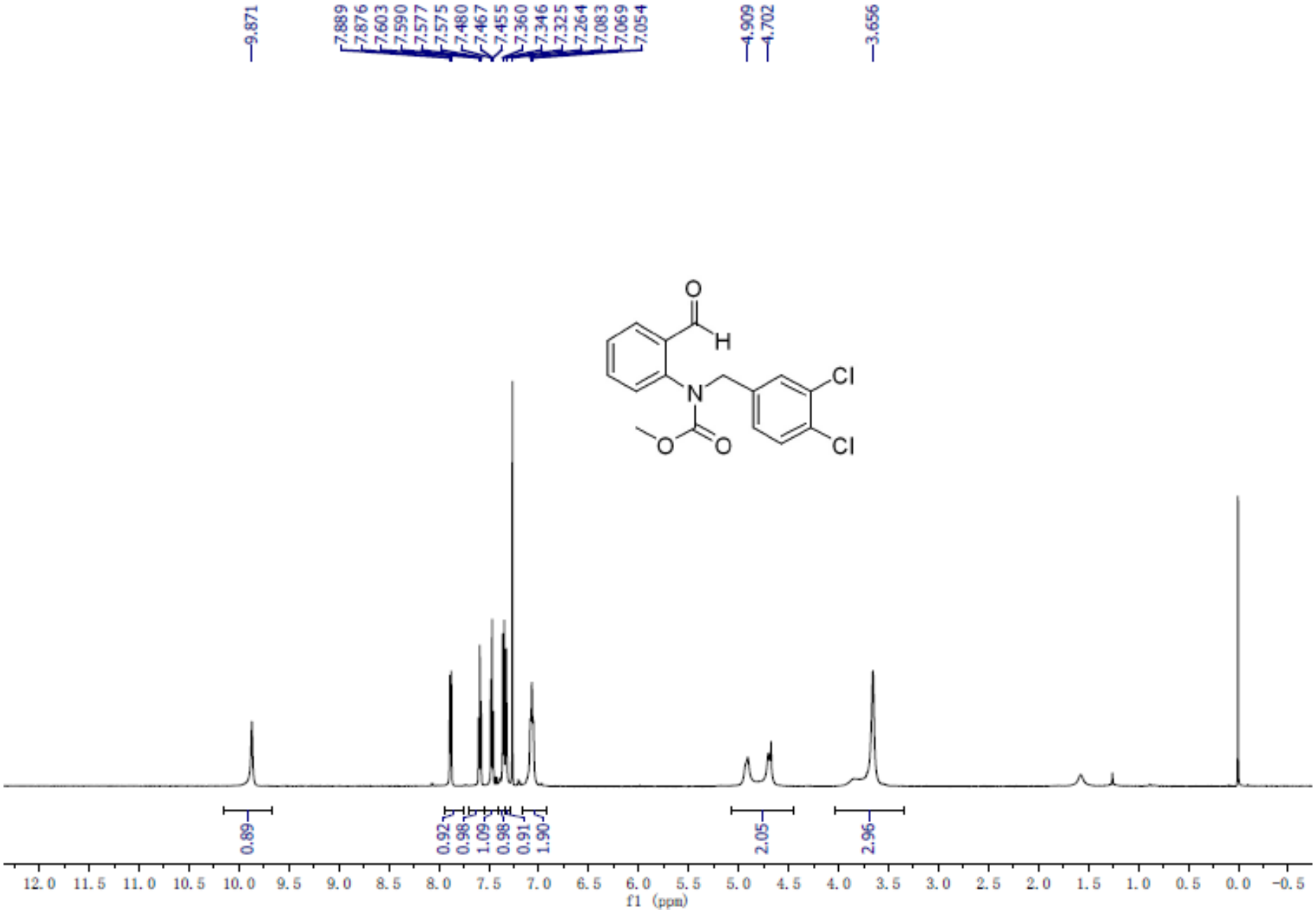
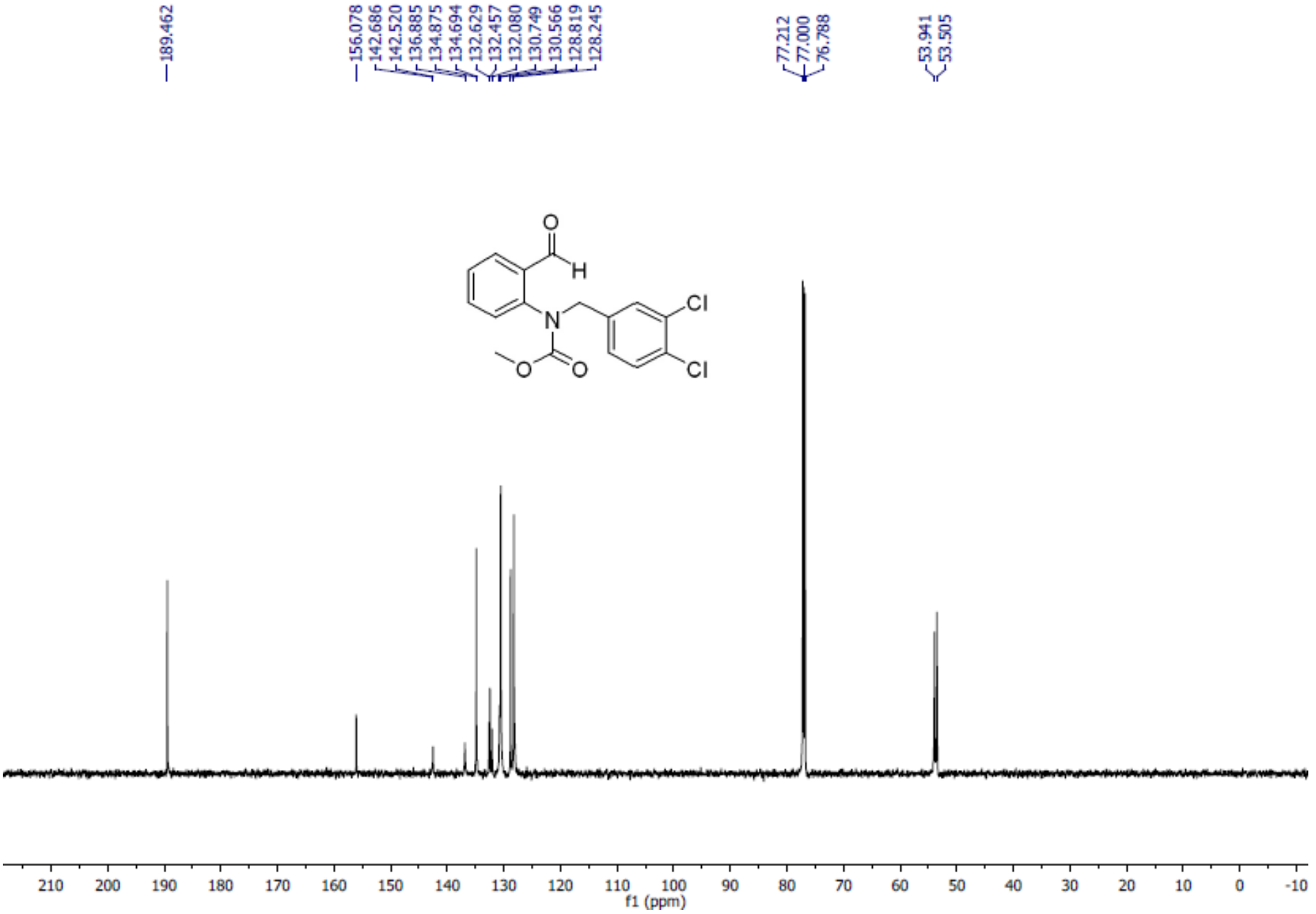


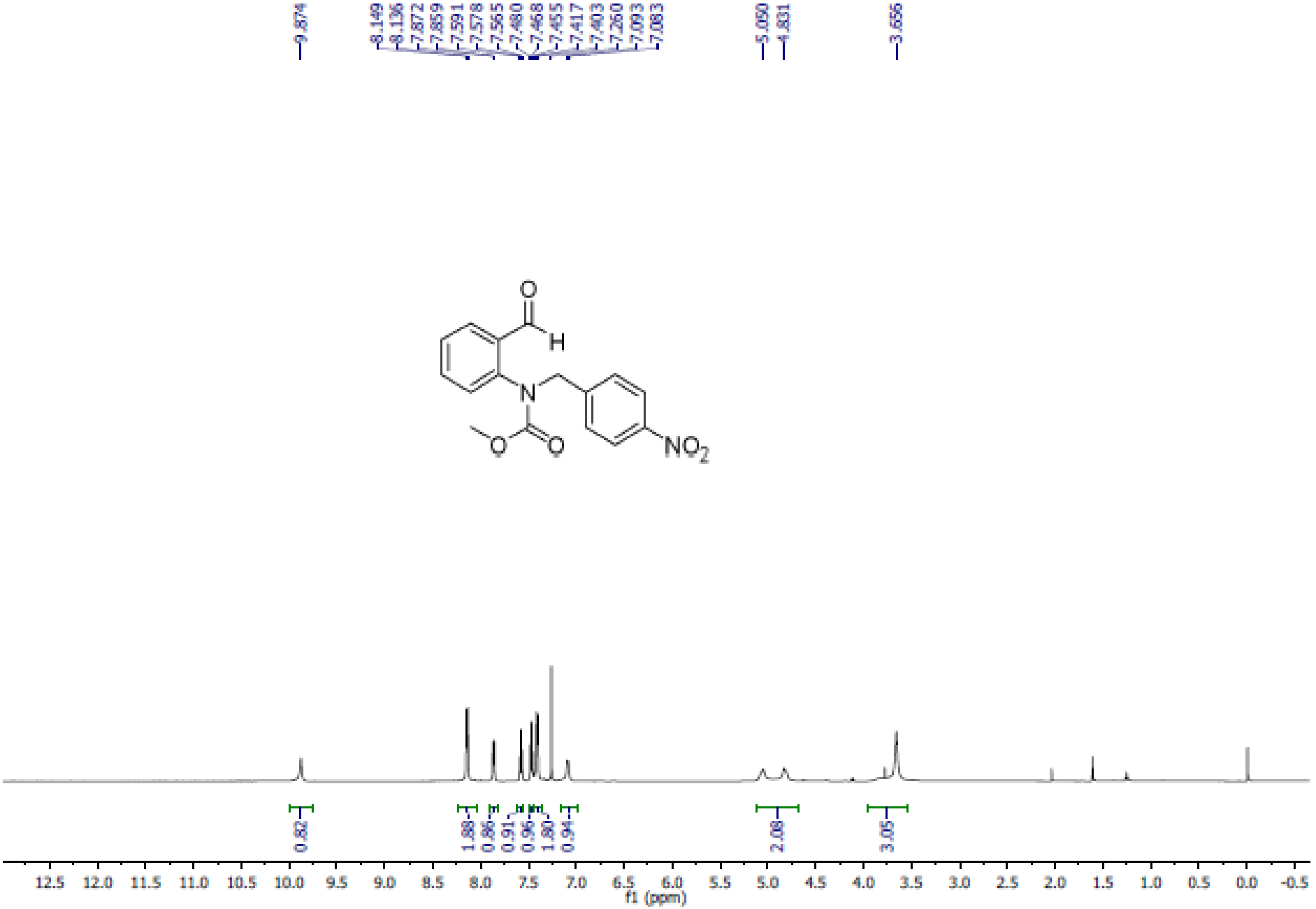
methyl (3,4-dichlorobenzyl)(2-formylphenyl)carbamate s3-h



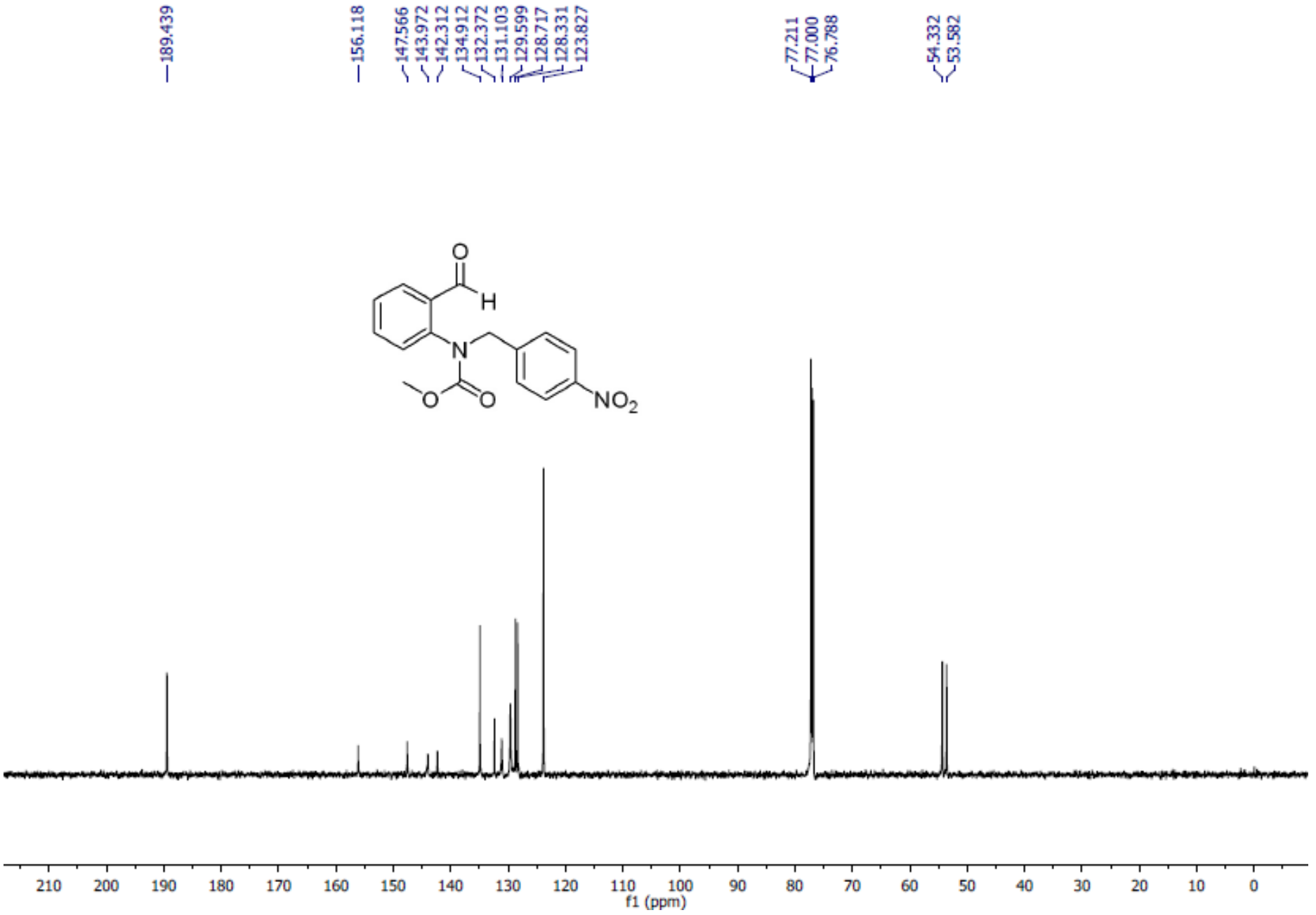
methyl (3,4-dichlorobenzyl)(2-formylphenyl)carbamate s3-h



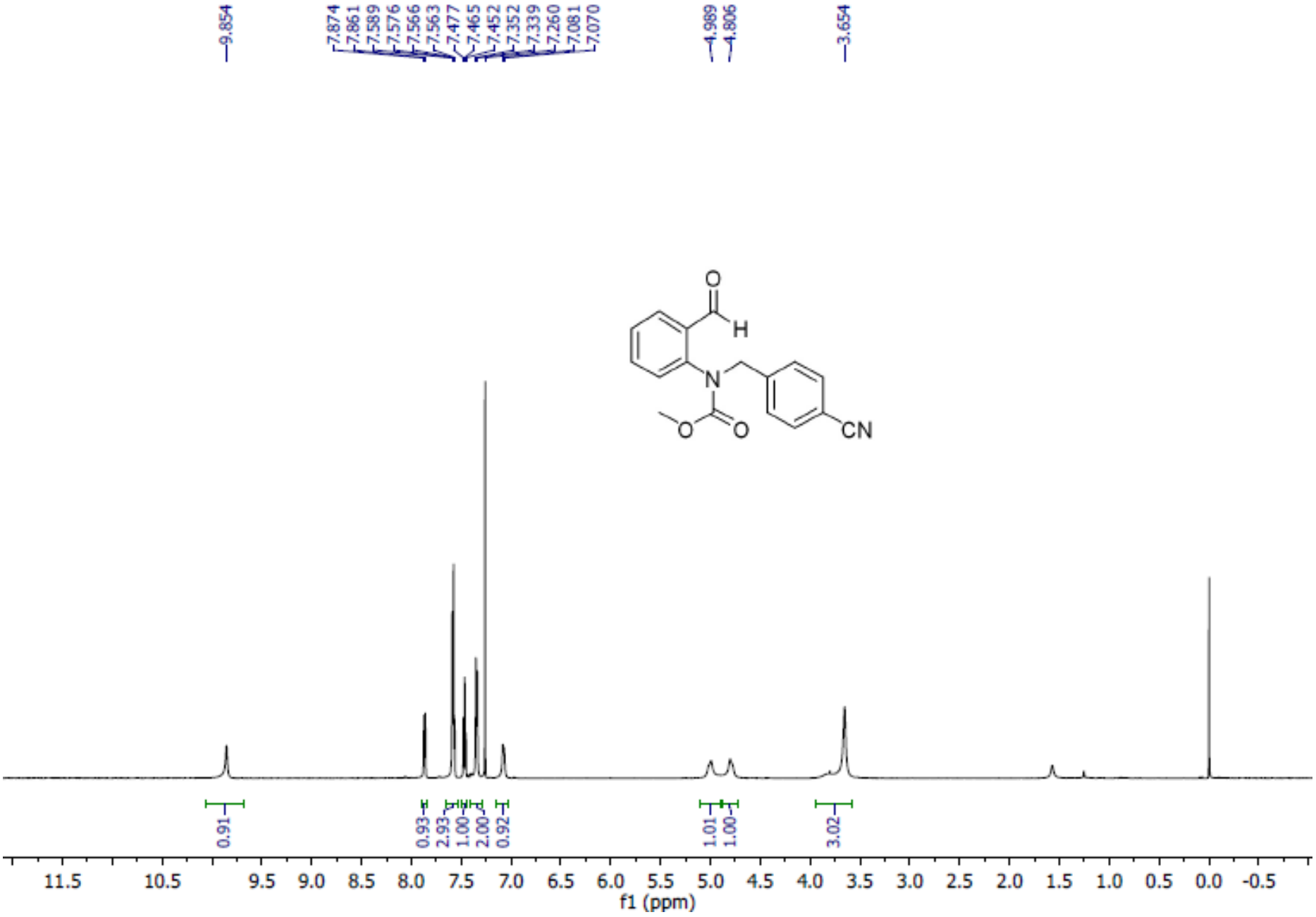
methyl (2-formylphenyl)(4-nitrobenzyl)carbamate s3-i



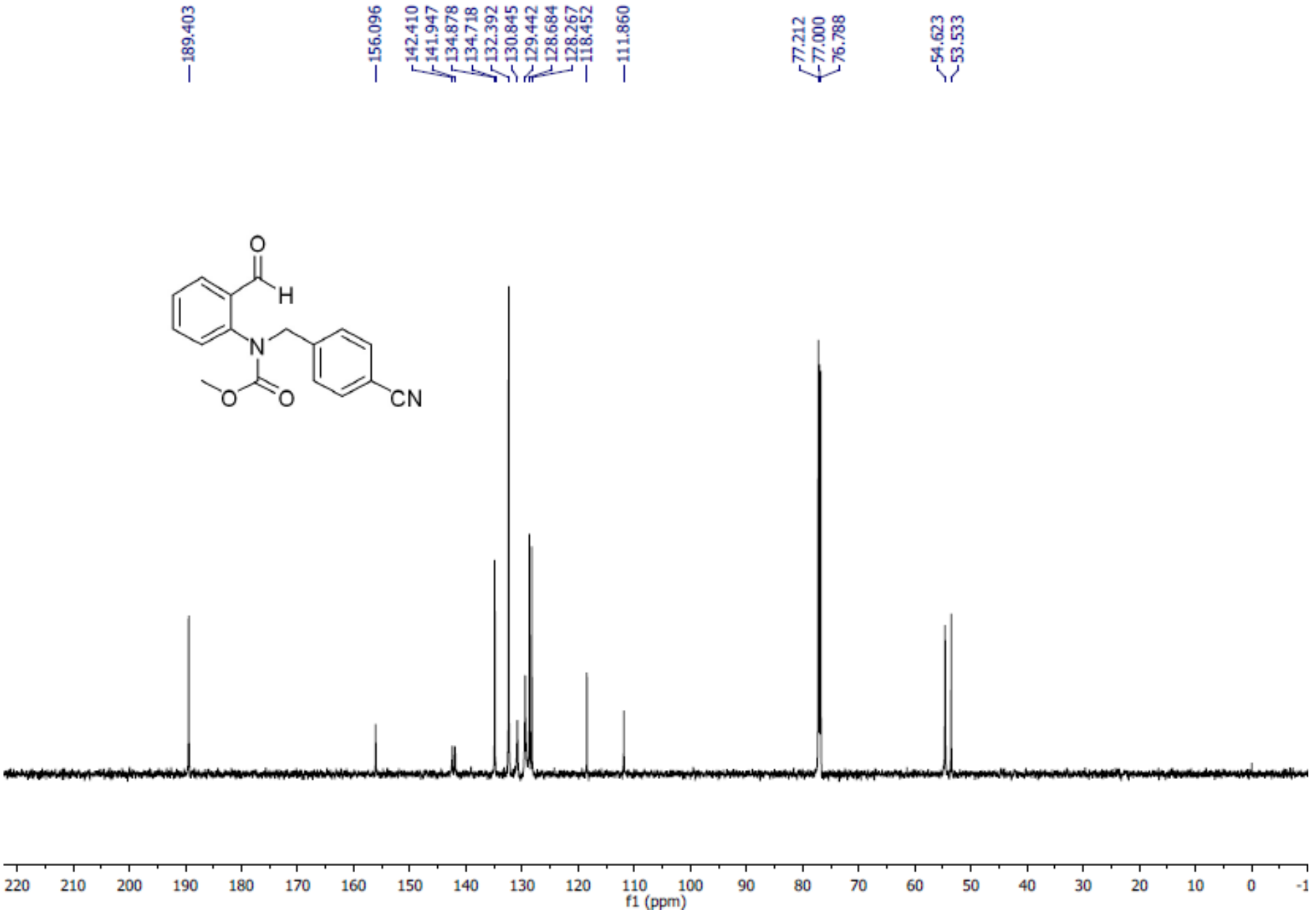
methyl (2-formylphenyl)(4-nitrobenzyl)carbamate s3-i



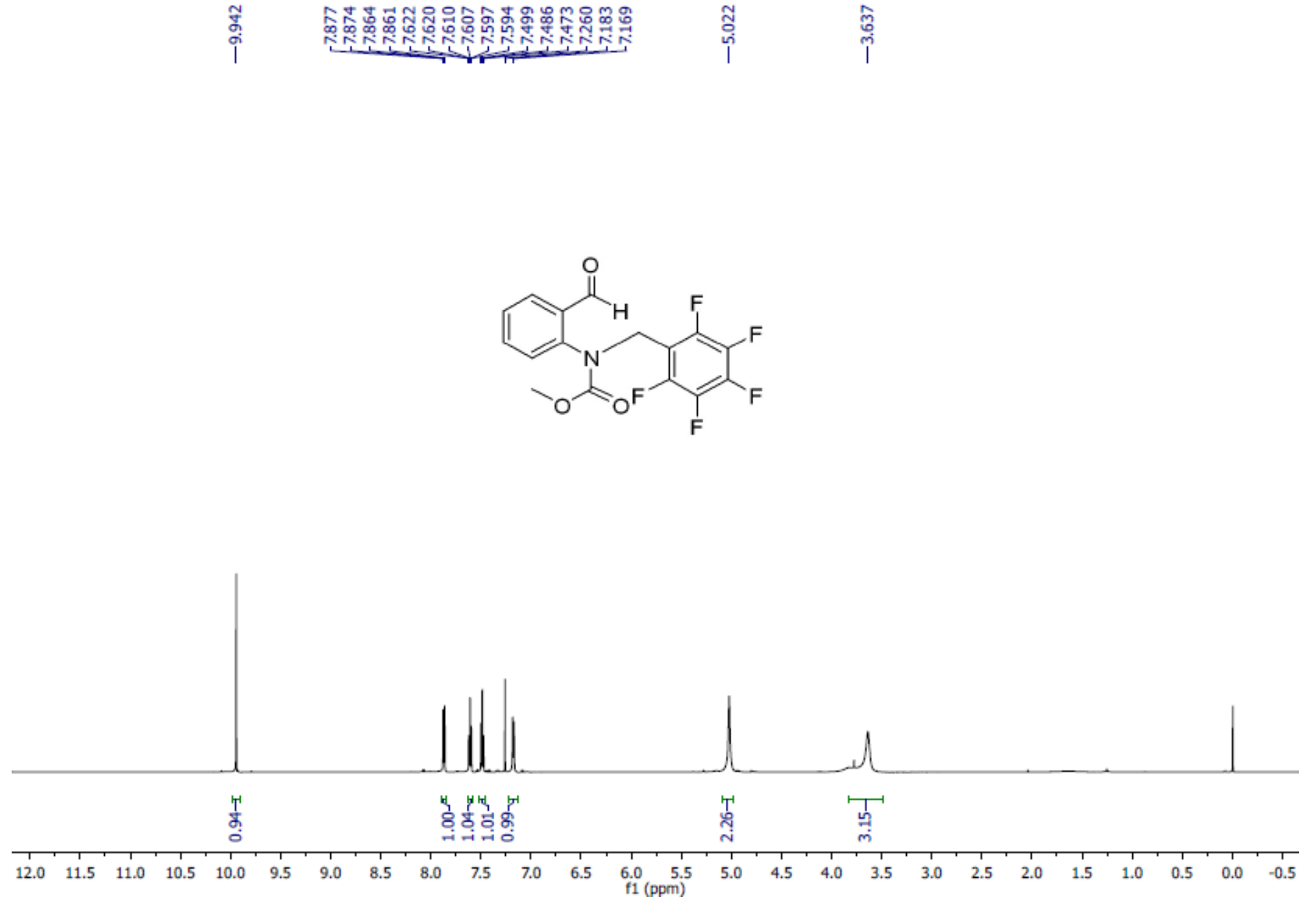
methyl (4-cyanobenzyl)(2-formylphenyl)carbamate s3-j



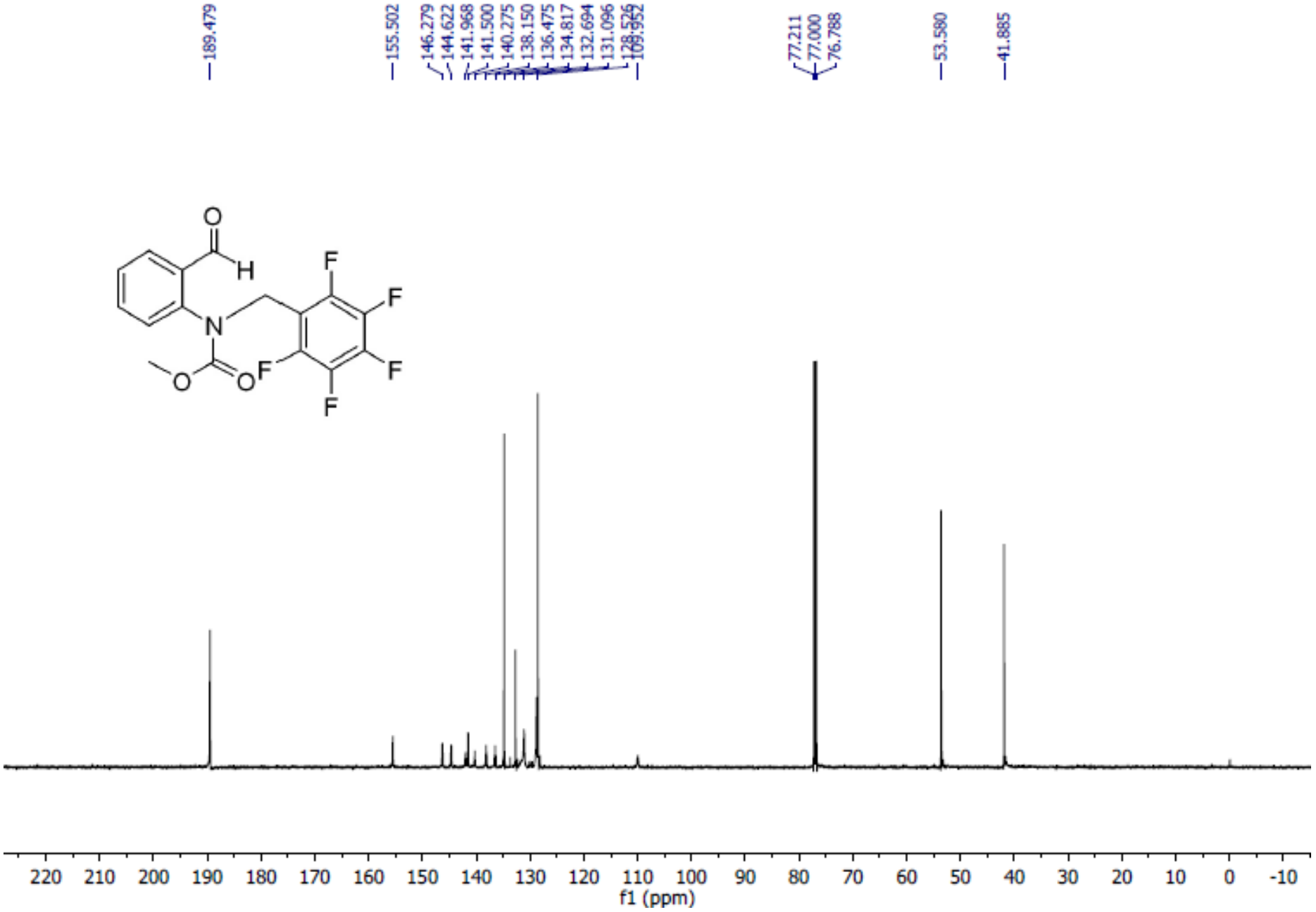
methyl (4-cyanobenzyl)(2-formylphenyl)carbamate s3-j



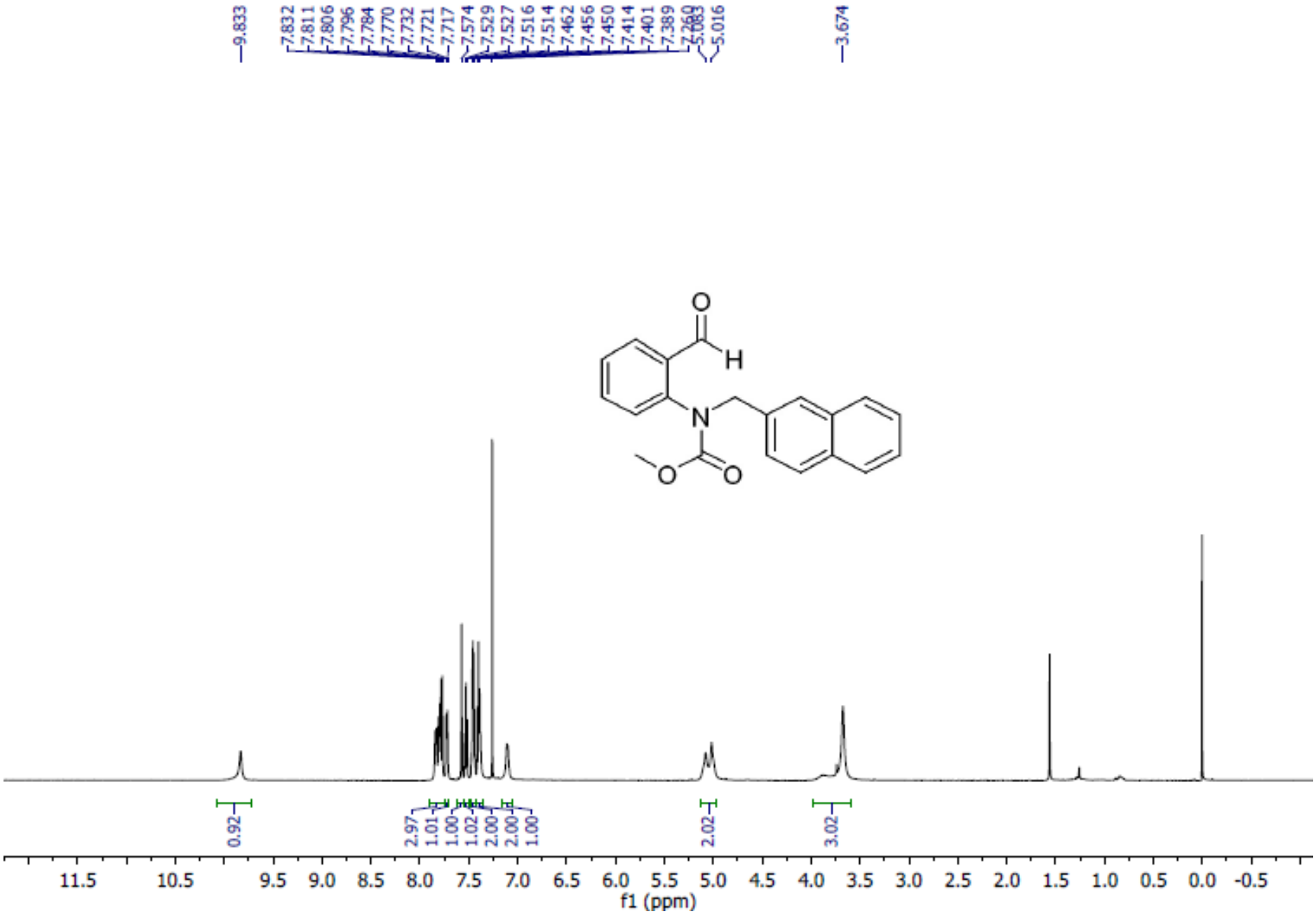
methyl (2-formylphenyl)((perfluorophenyl)methyl)carbamate s3-k



methyl (2-formylphenyl)((perfluorophenyl)methyl)carbamate s3-k



methyl (2-formylphenyl)(naphthalen-2-ylmethyl)carbamate s3-l



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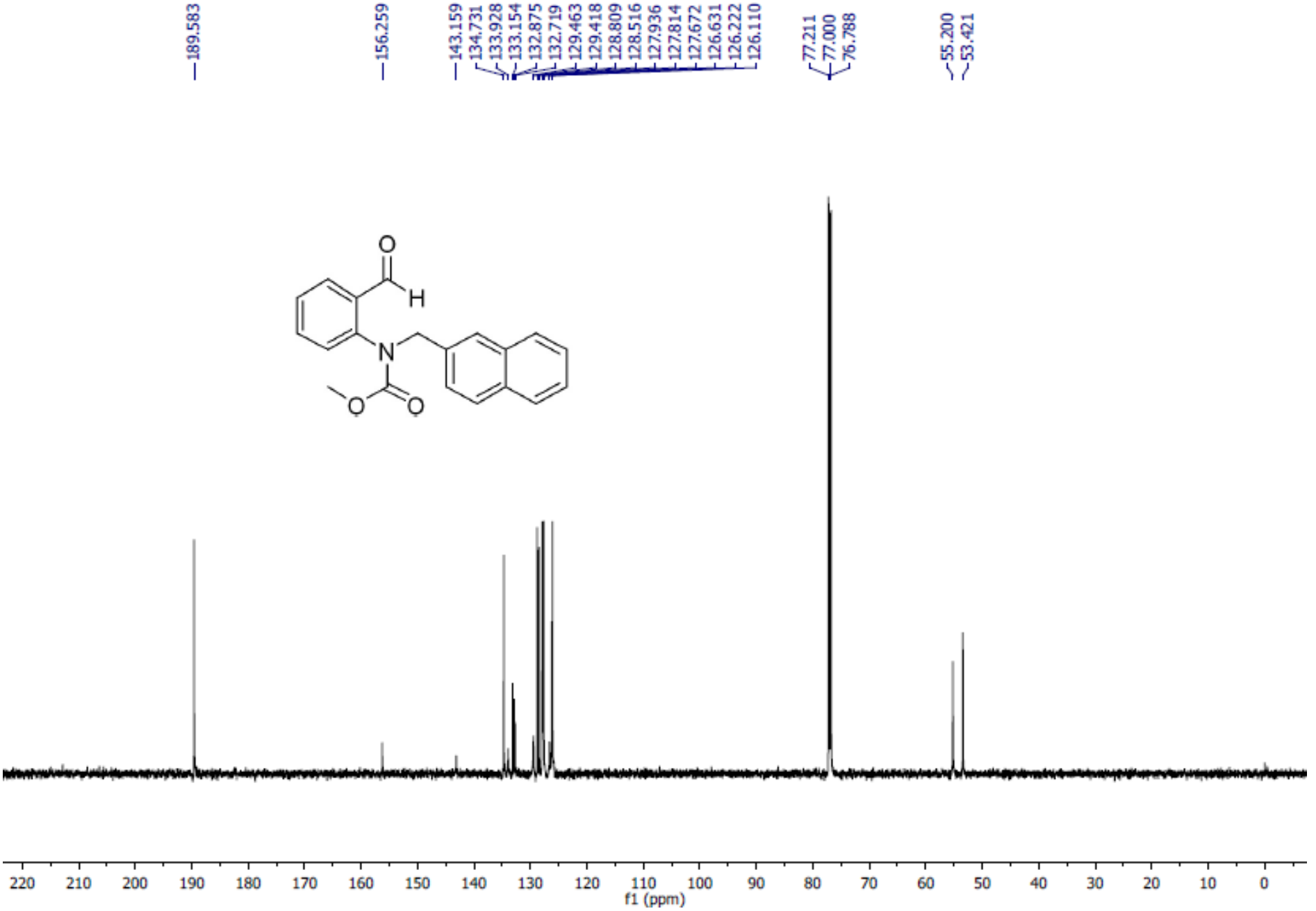
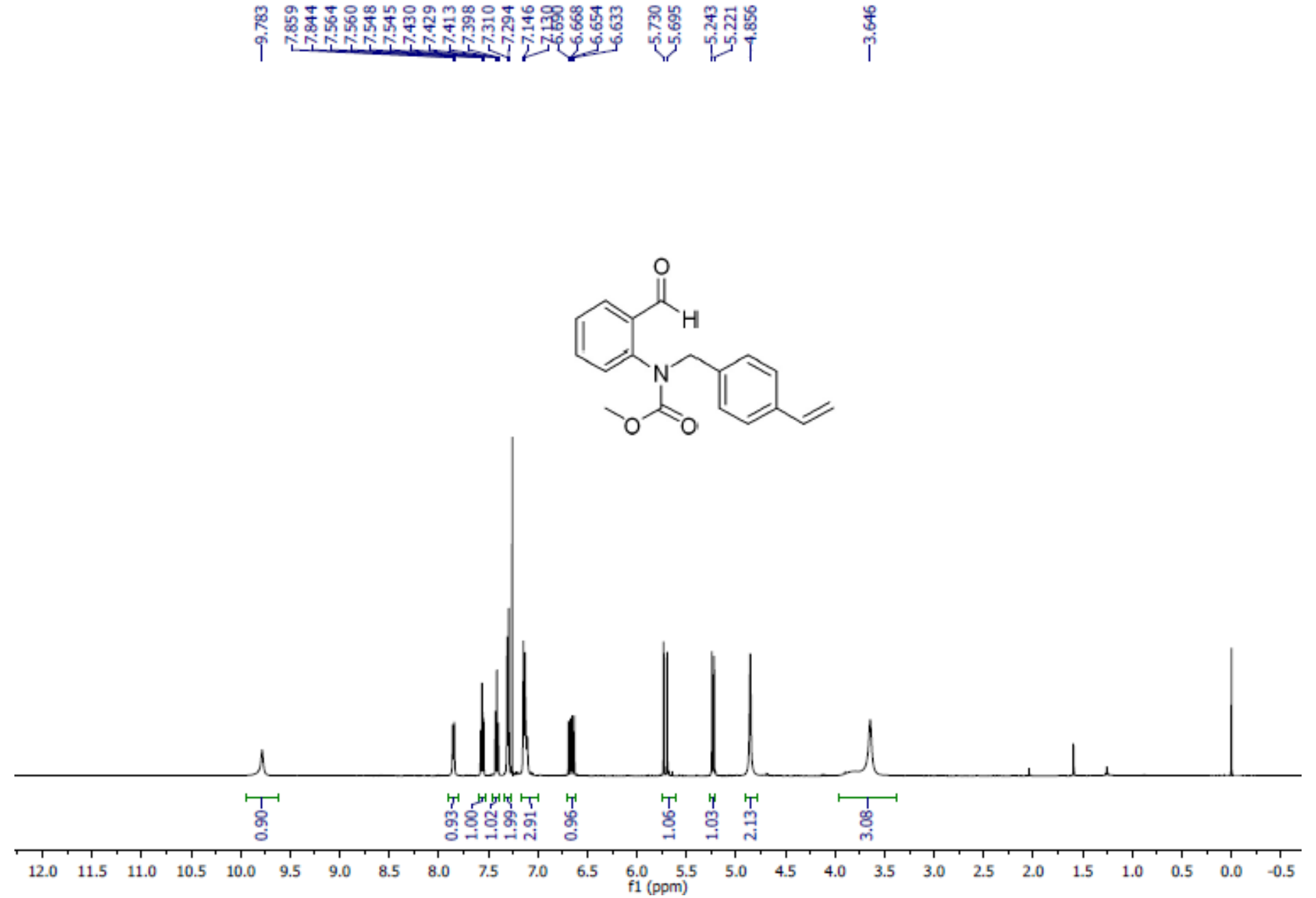
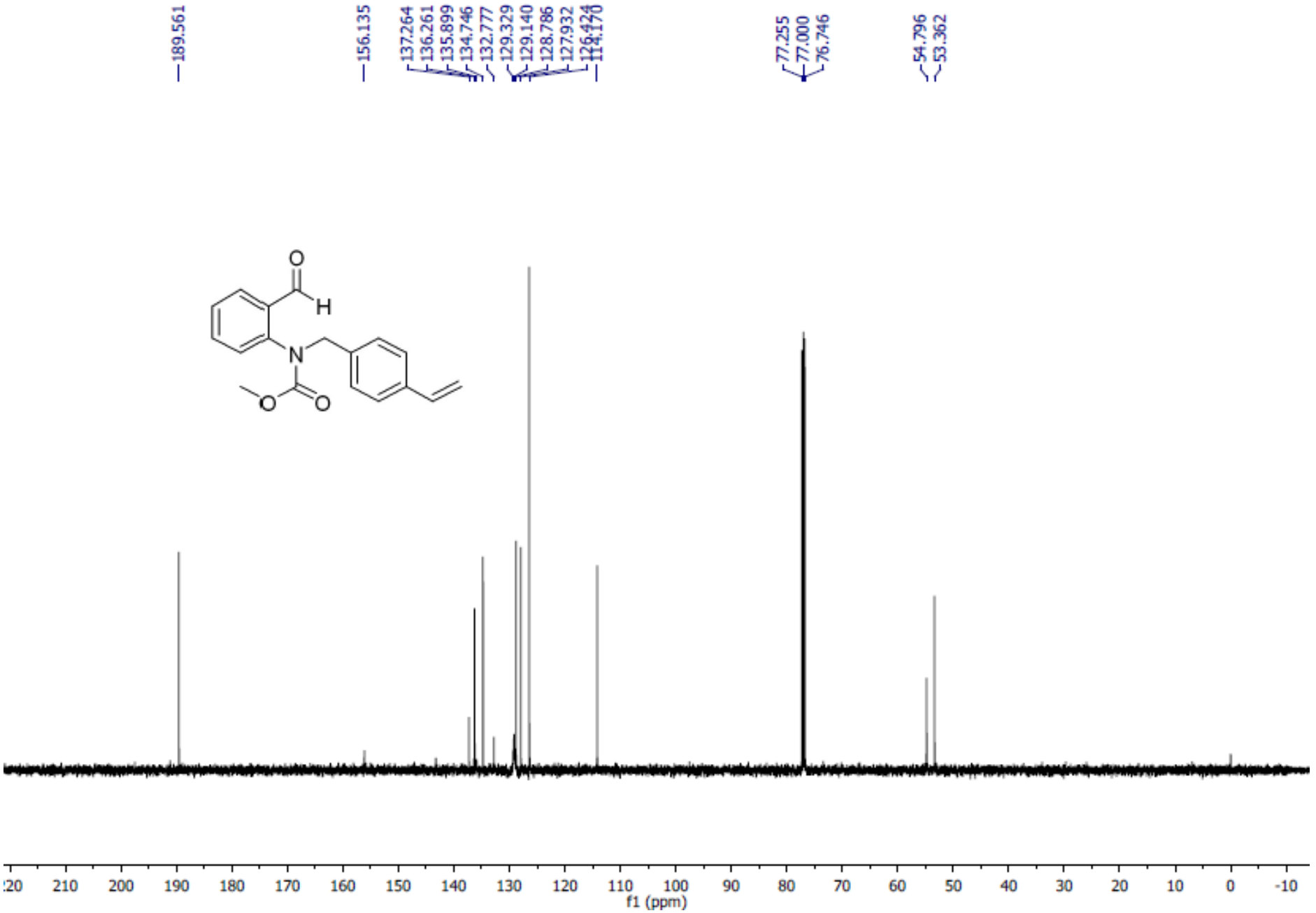


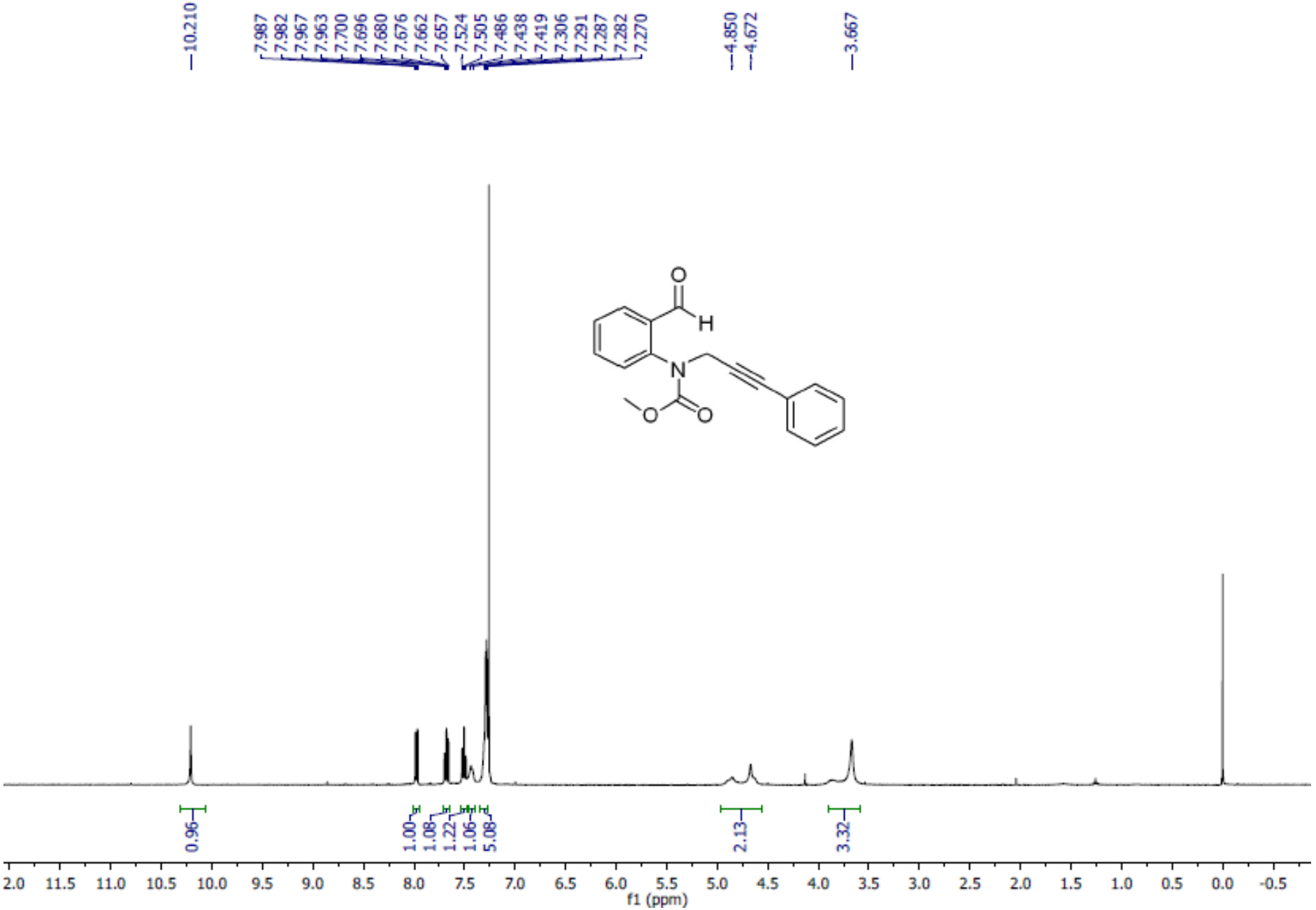
methyl (2-formylphenyl)(4-vinylbenzyl)carbamate s3-m



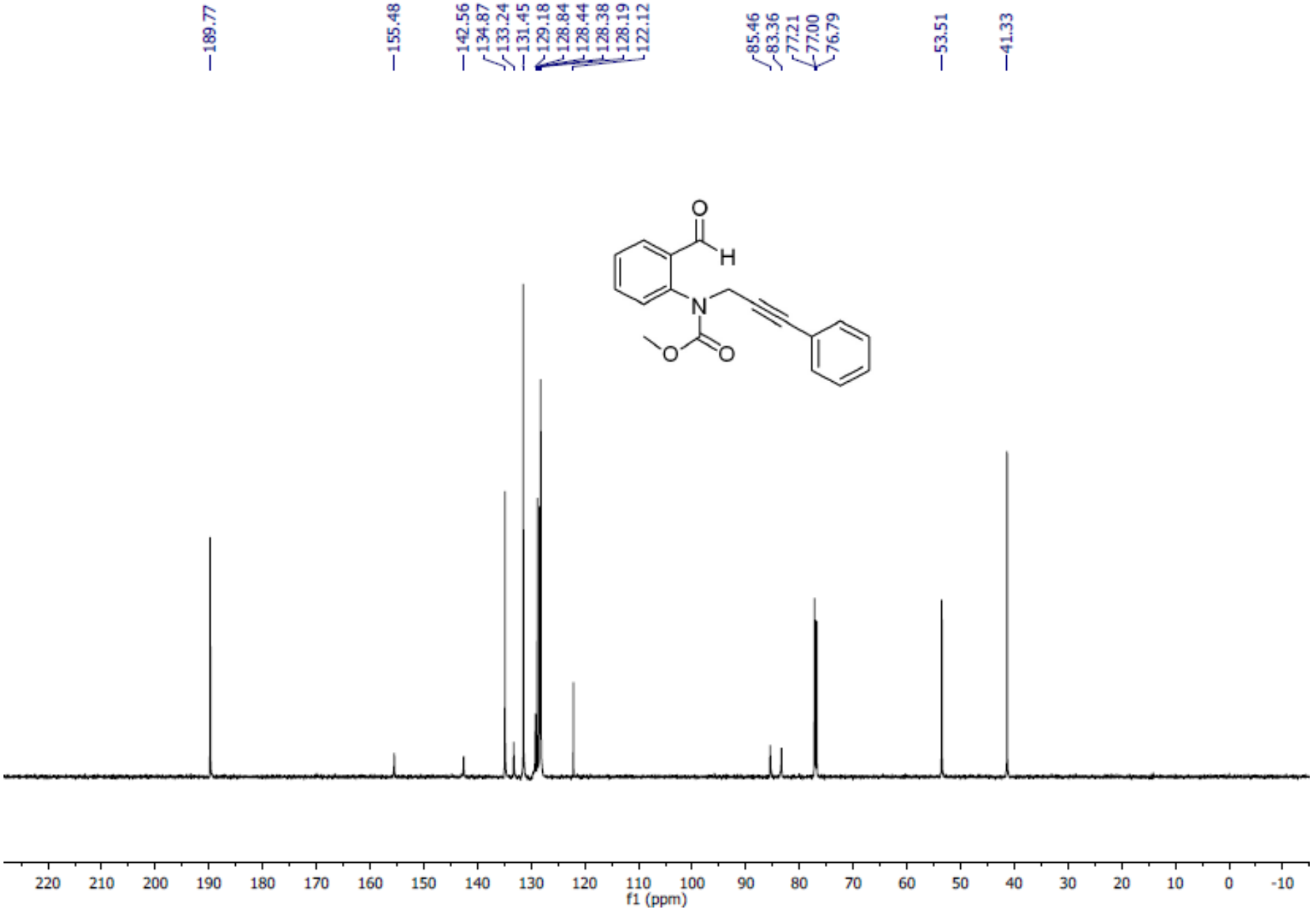
methyl (2-formylphenyl)(4-vinylbenzyl)carbamate s3-m



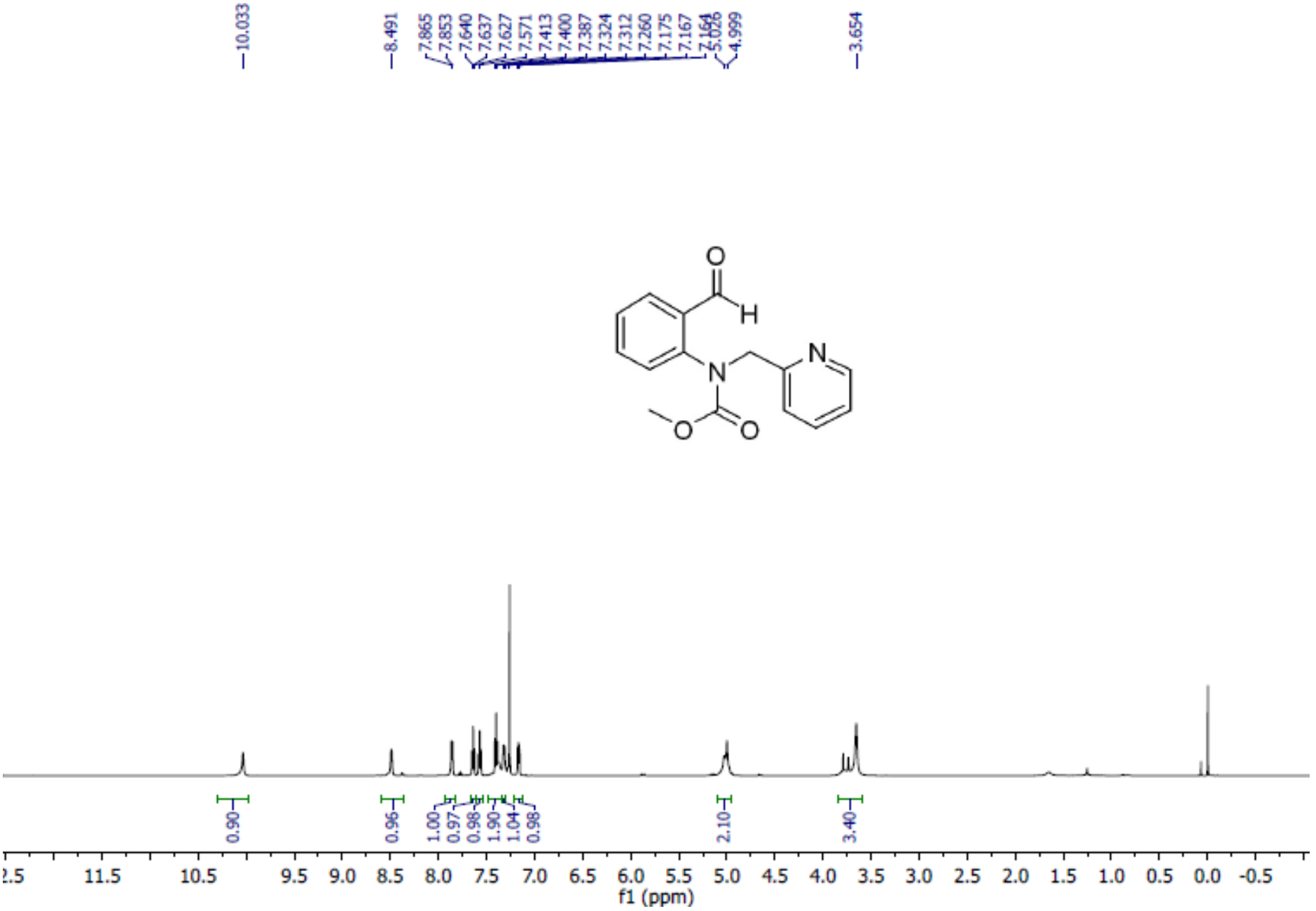
methyl (2-formylphenyl)(3-phenylprop-2-yn-1-yl)carbamate s3-n



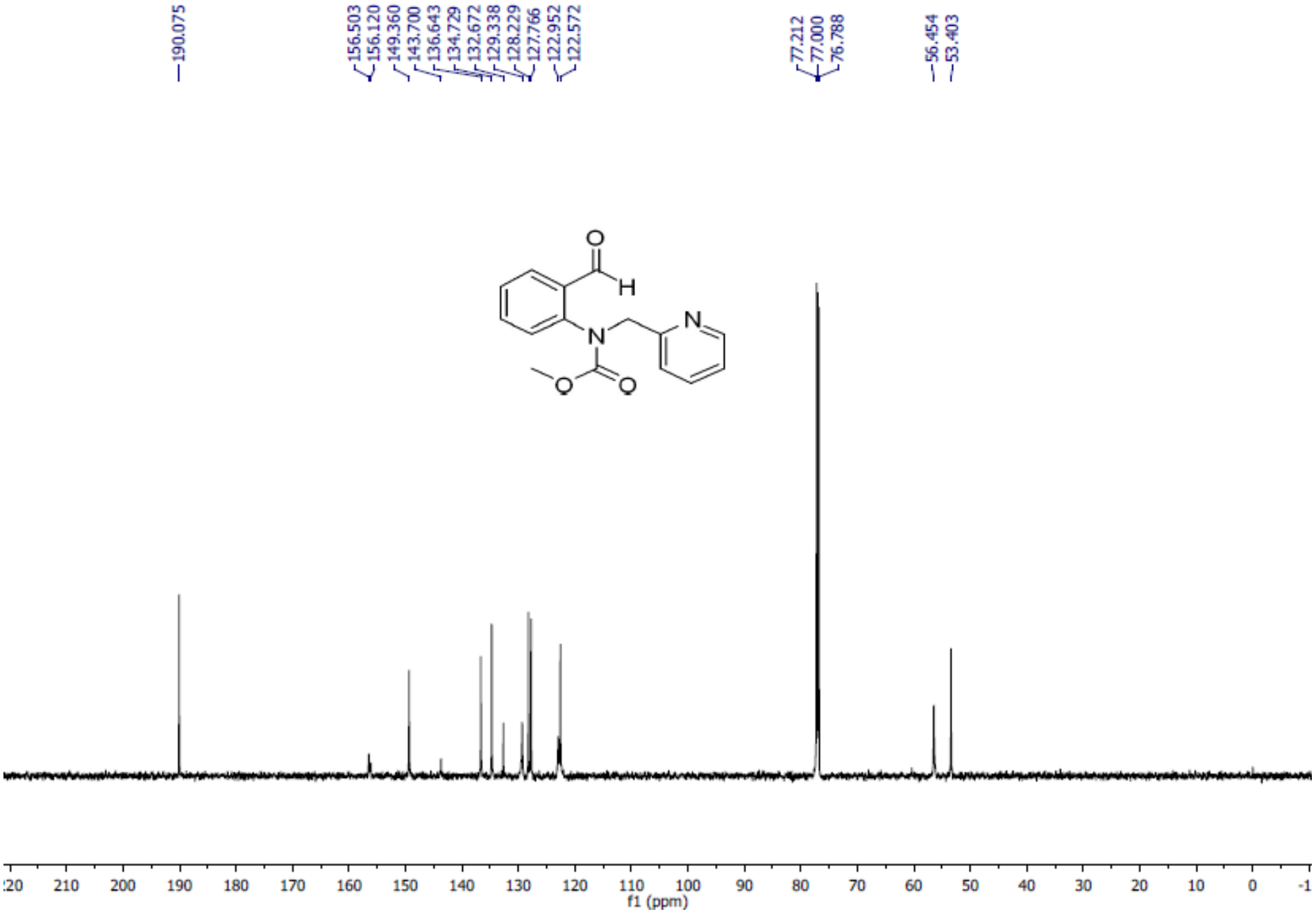
methyl (2-formylphenyl)(3-phenylprop-2-yn-1-yl)carbamate s3-n



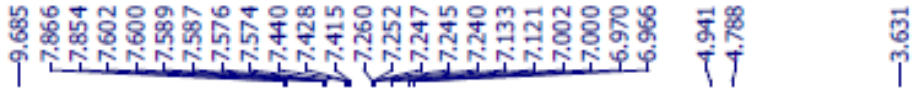
methyl (2-formylphenyl)(pyridin-2-ylmethyl)carbamate s3-o

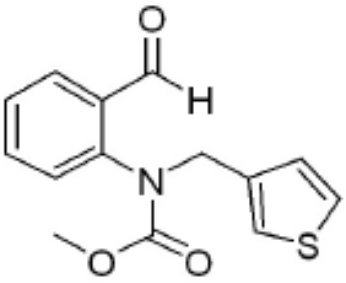


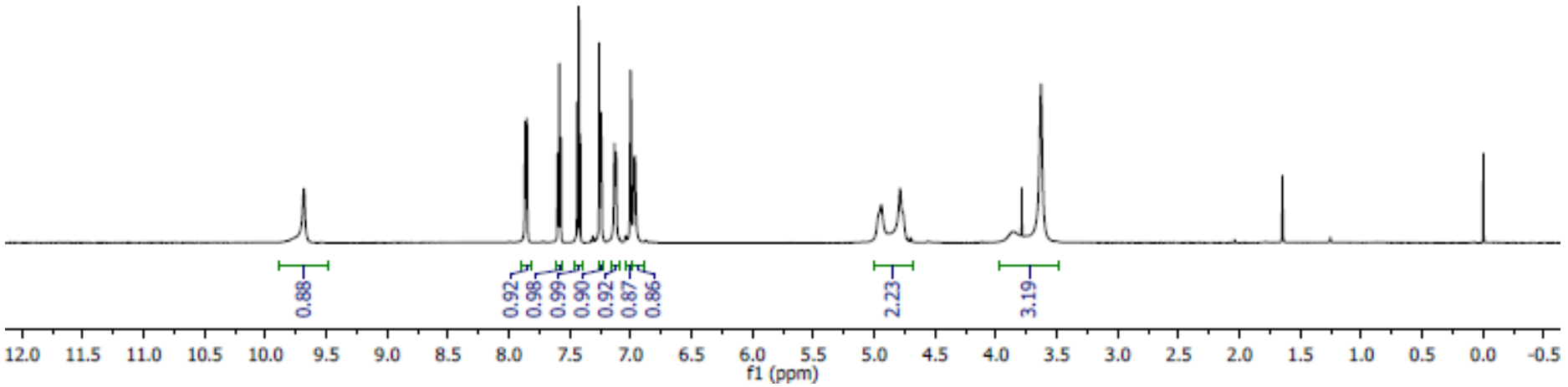
methyl (2-formylphenyl)(pyridin-2-ylmethyl)carbamate s3-o



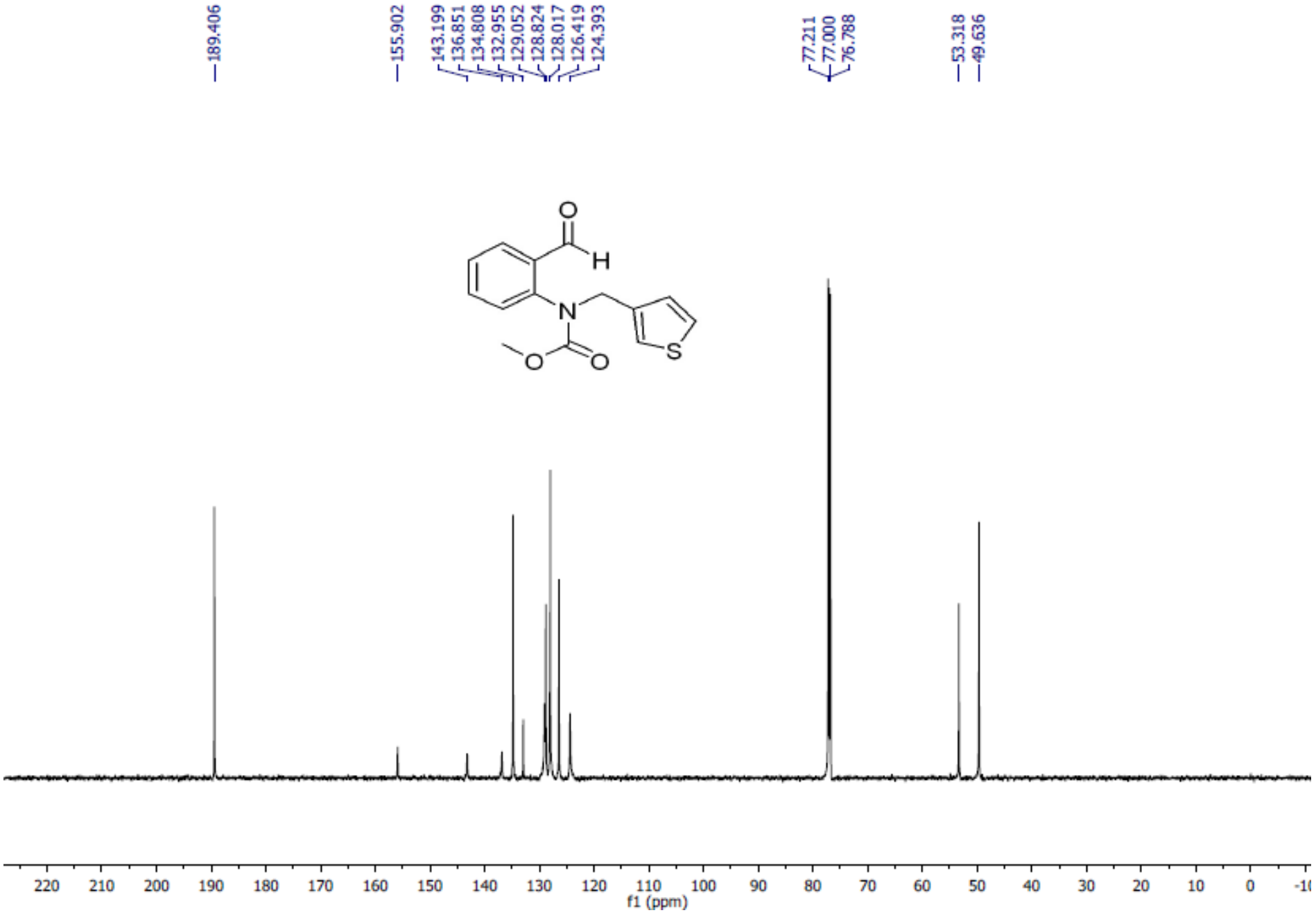
methyl (2-formylphenyl)(thiophen-3-ylmethyl)carbamate s3-p



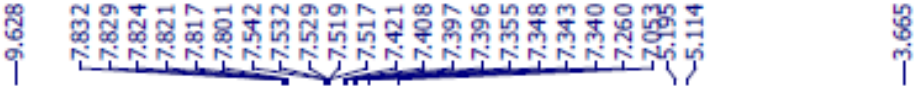


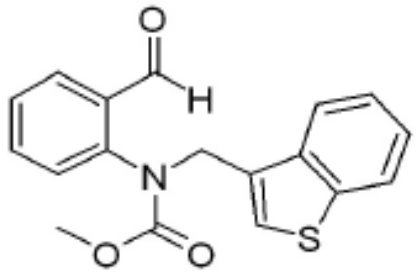


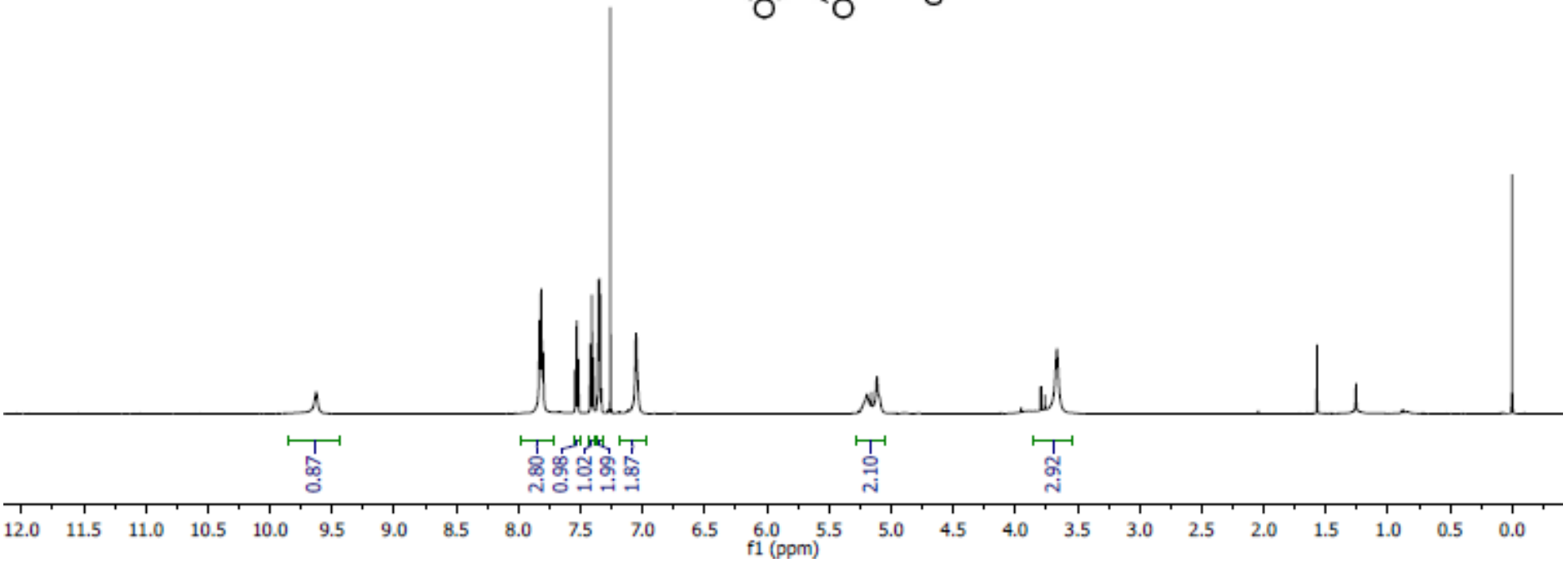
methyl (2-formylphenyl)(thiophen-3-ylmethyl)carbamate s3-p



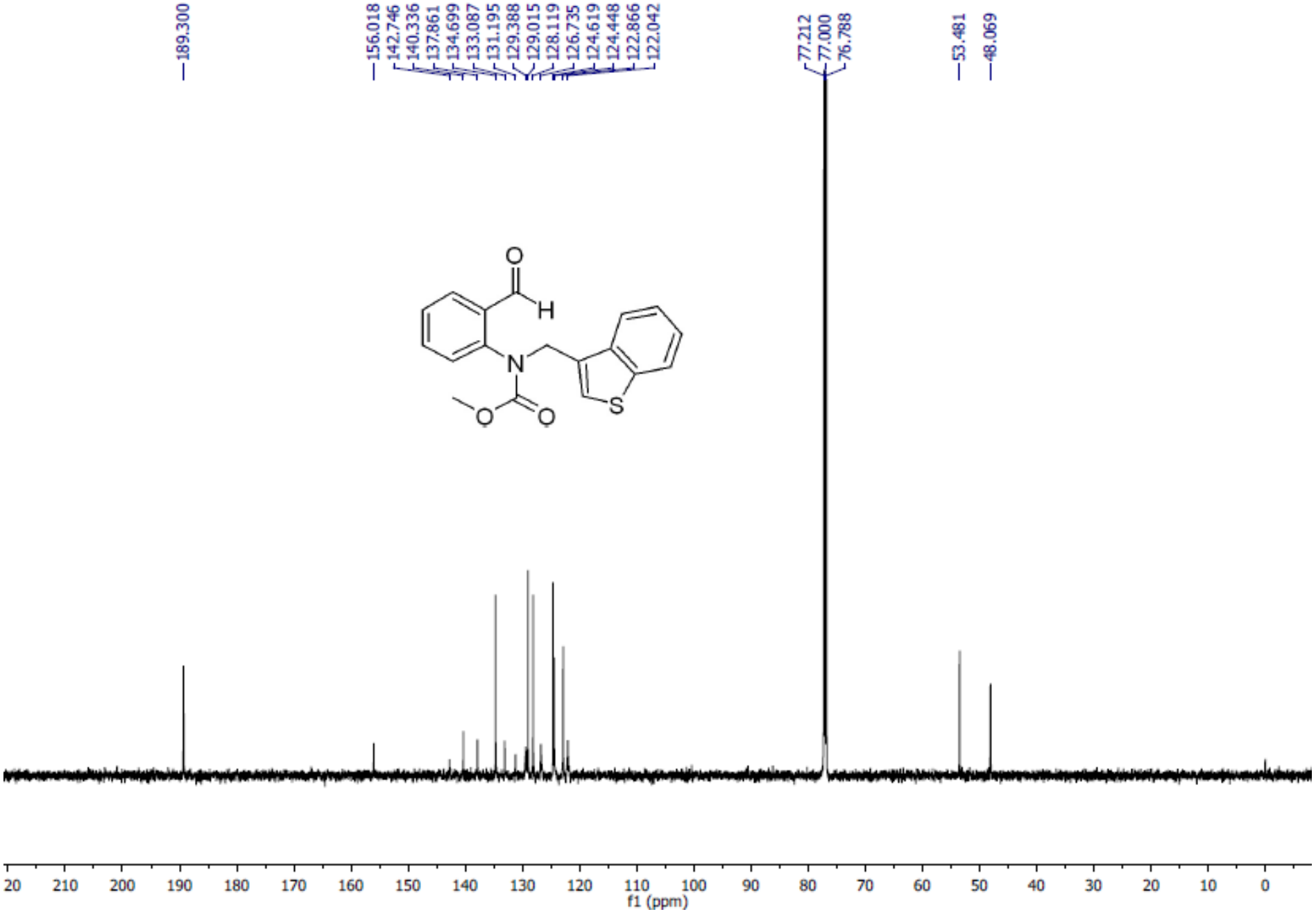
methyl (benzo[*b*]thiophen-3-ylmethyl)(2-formylphenyl)carbamate s3-q



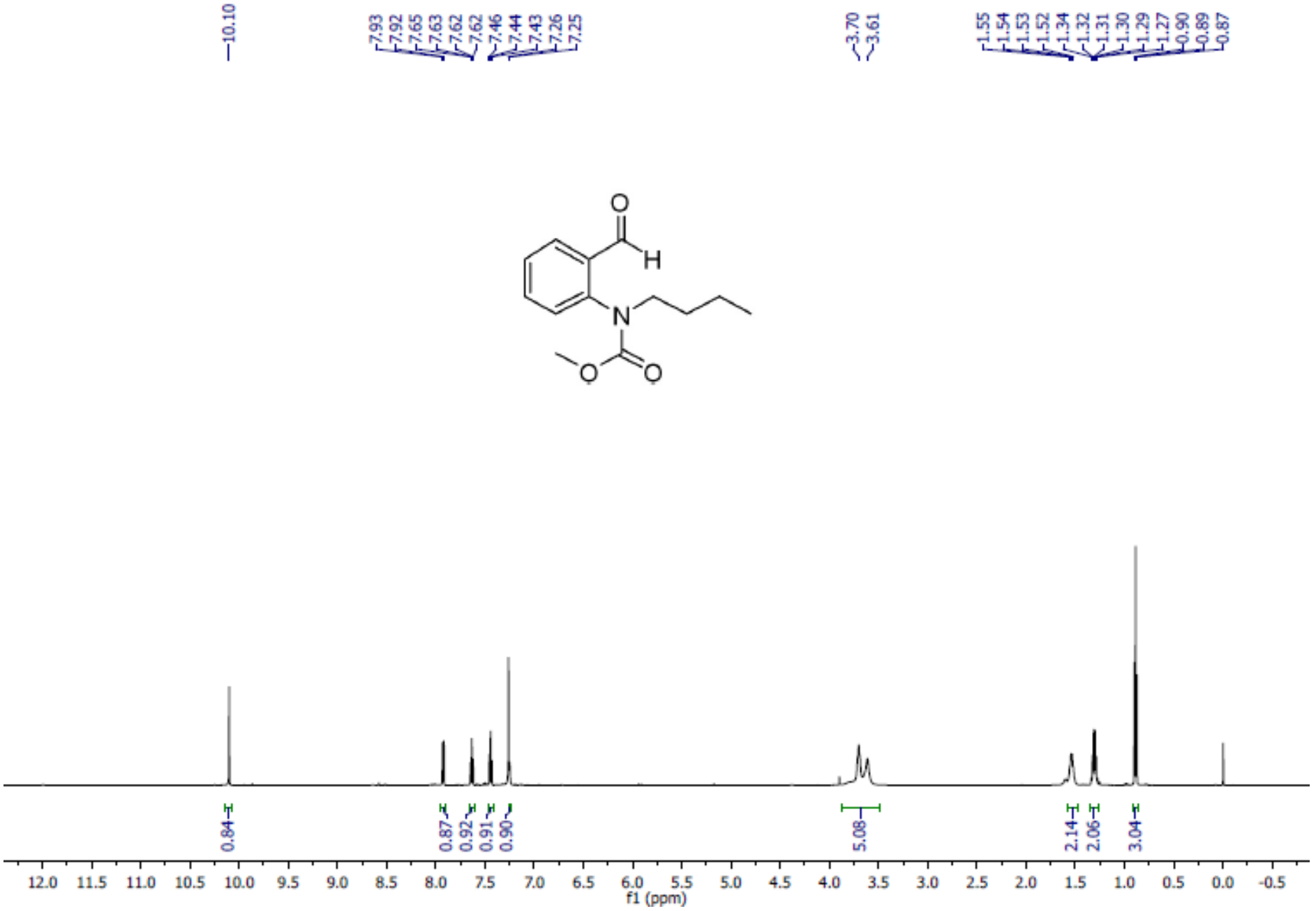




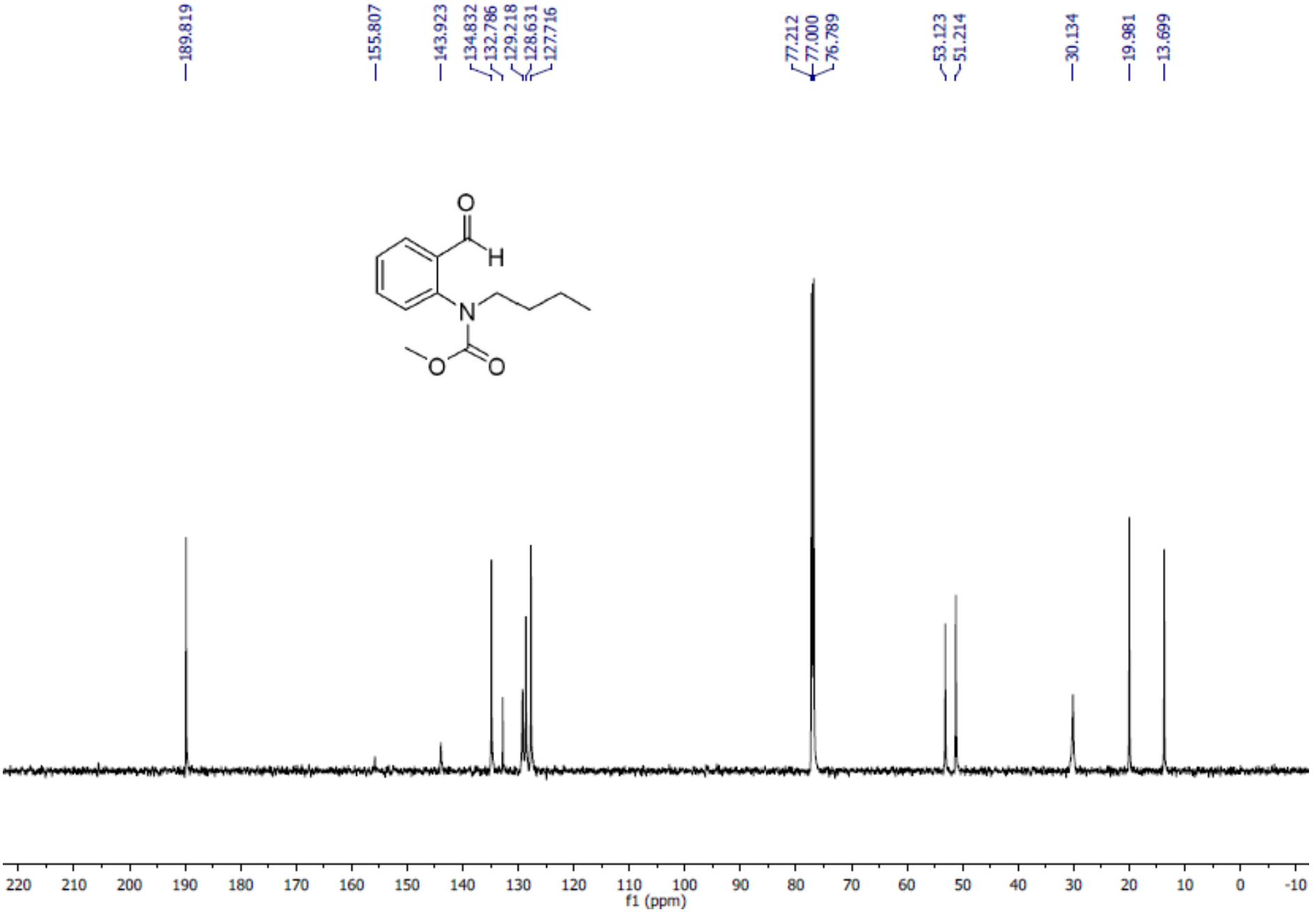
methyl (benzo[*b*]thiophen-3-ylmethyl)(2-formylphenyl)carbamate s3-q



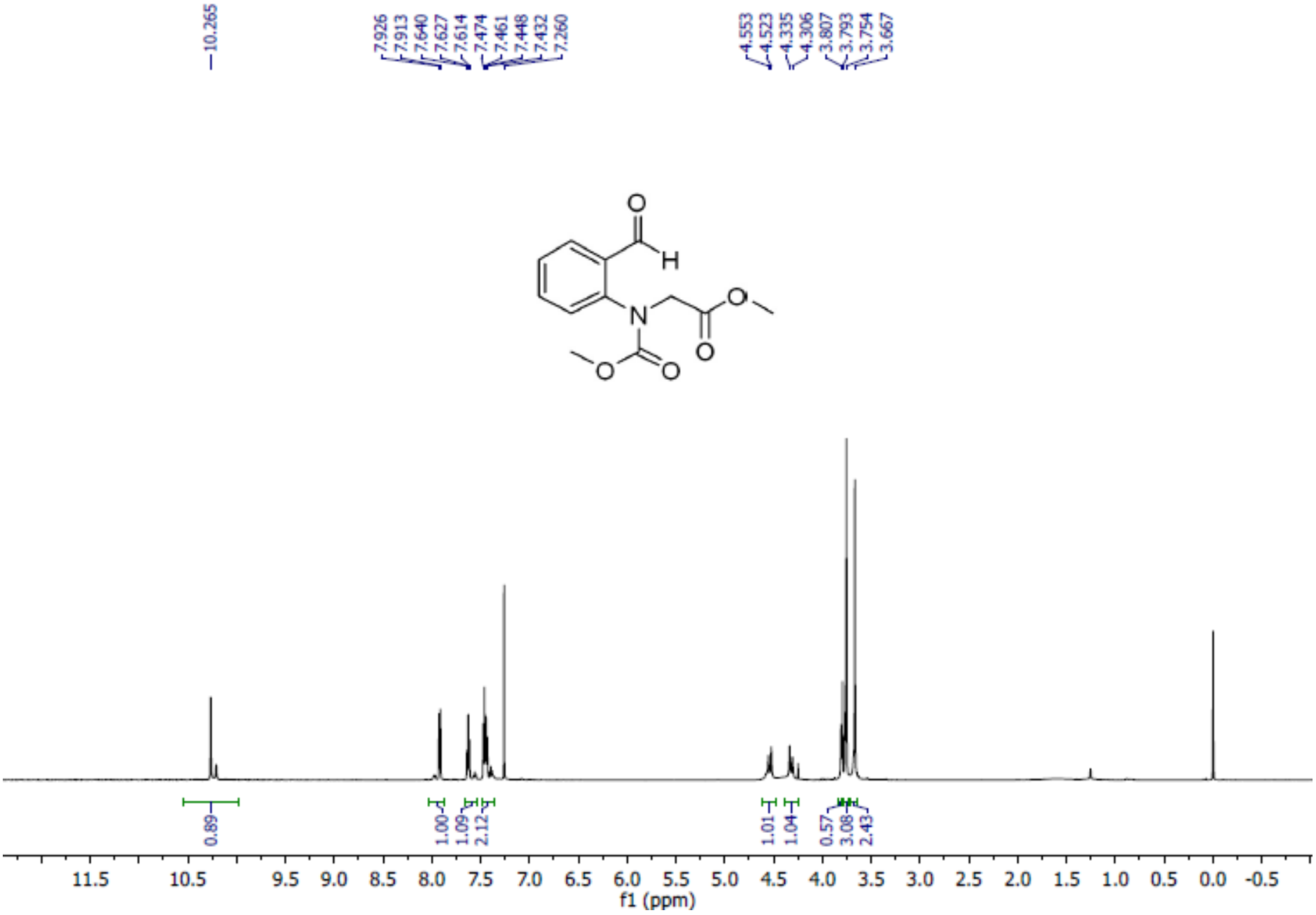
methyl butyl(2-formylphenyl)carbamate s3-r



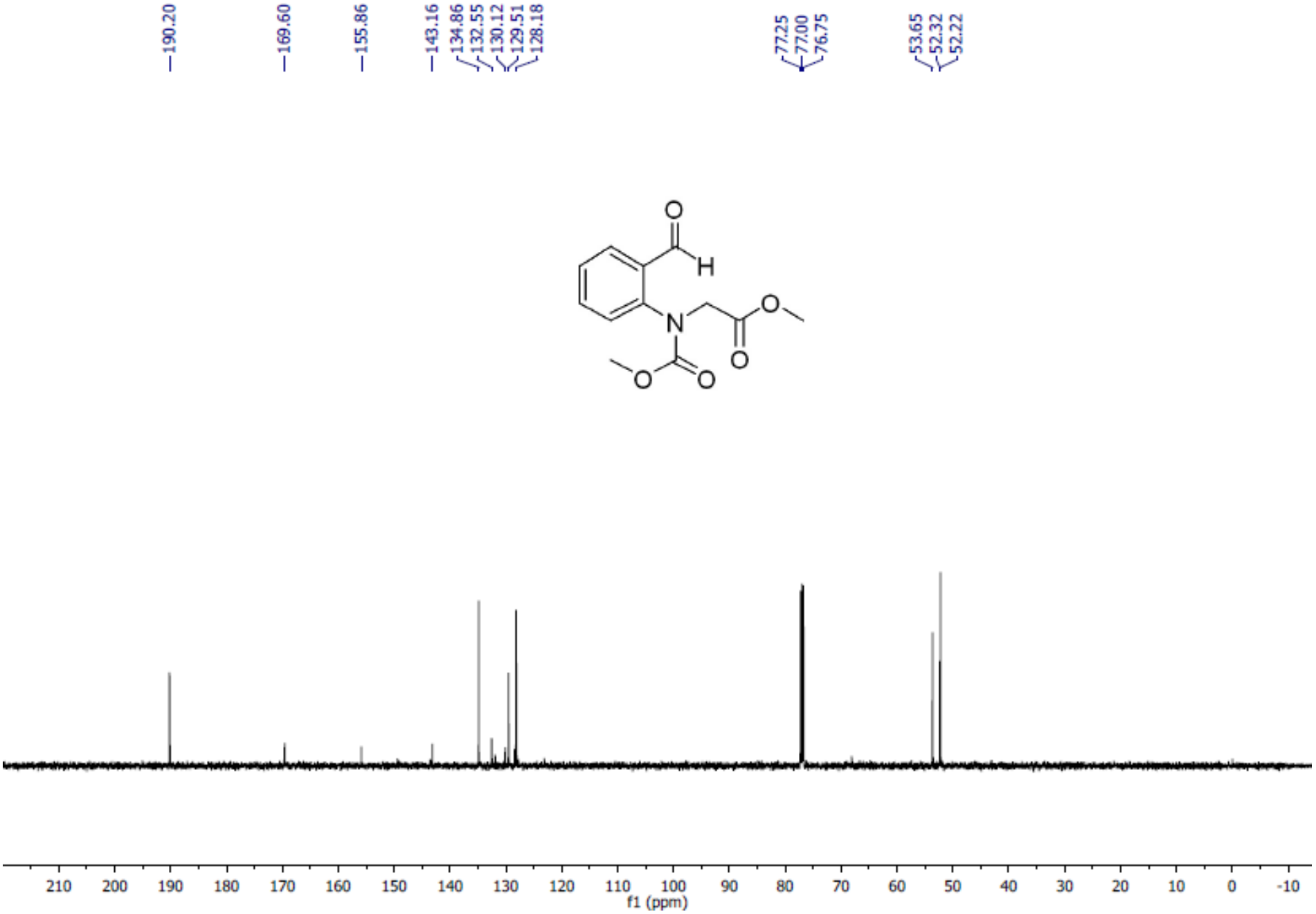
methyl butyl(2-formylphenyl)carbamate s3-r



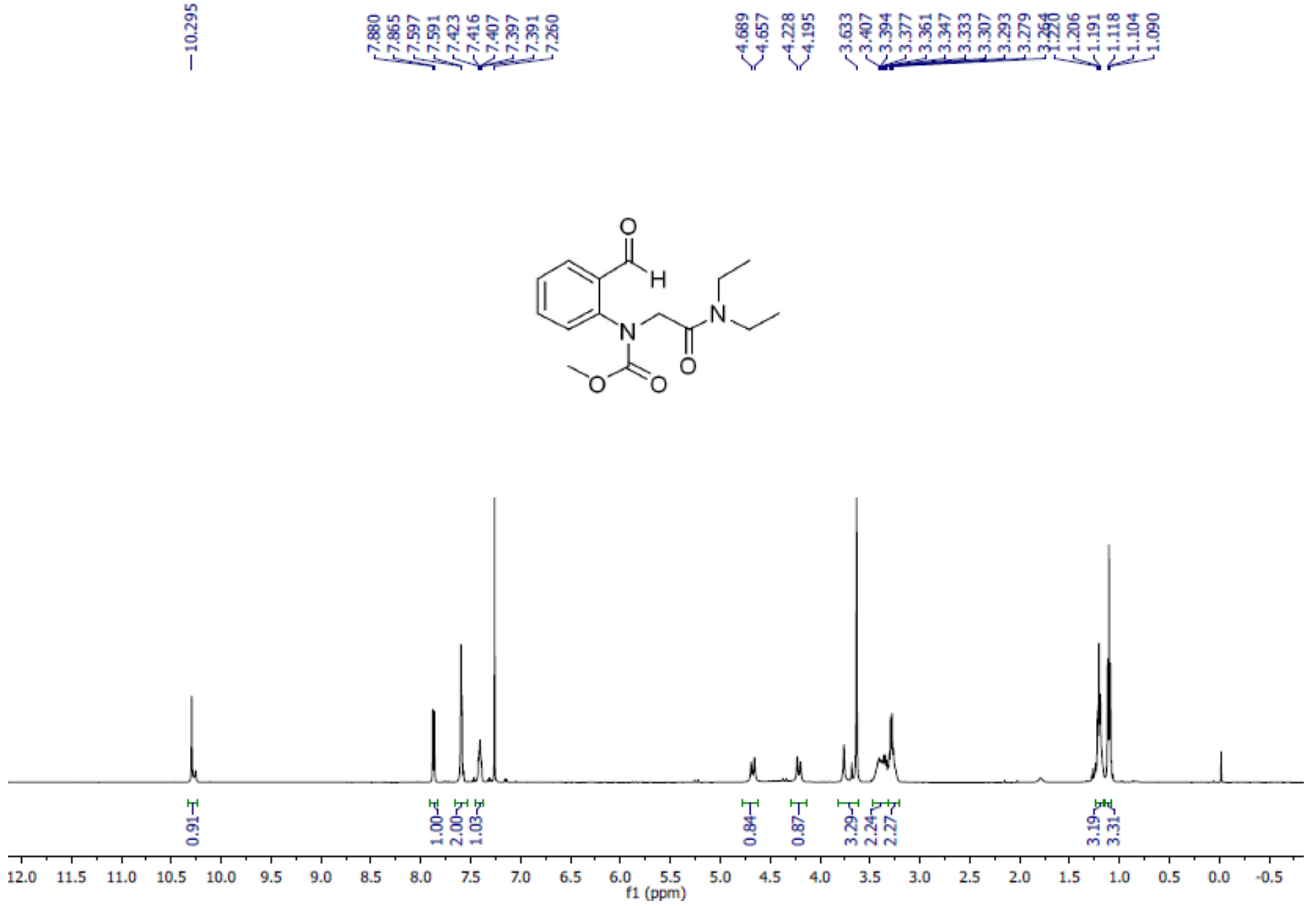
methyl *N*-(2-formylphenyl)-*N*-(methoxycarbonyl)glycinate s3-s



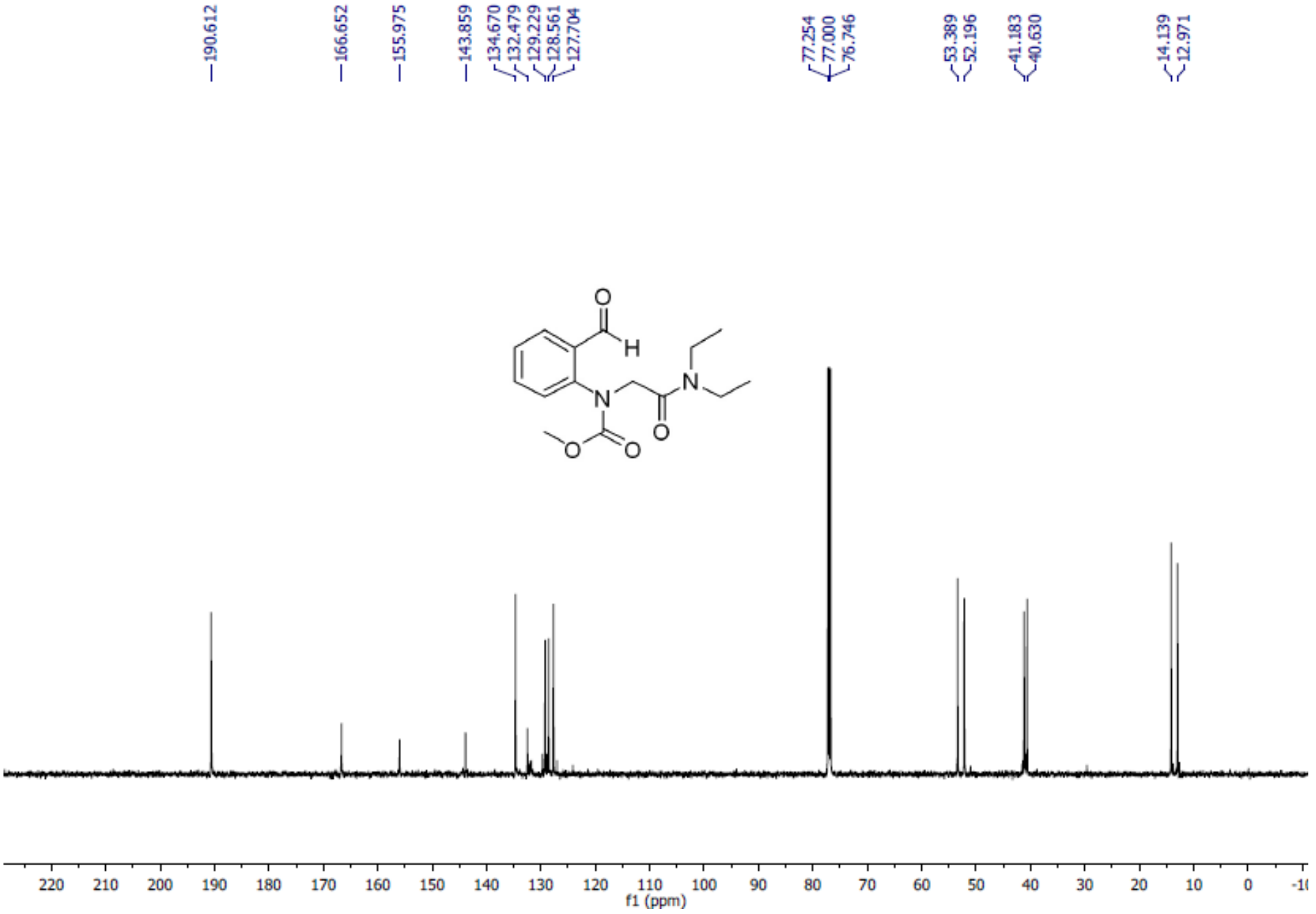
methyl *N*-(2-formylphenyl)-*N*-(methoxycarbonyl)glycinate s3-s



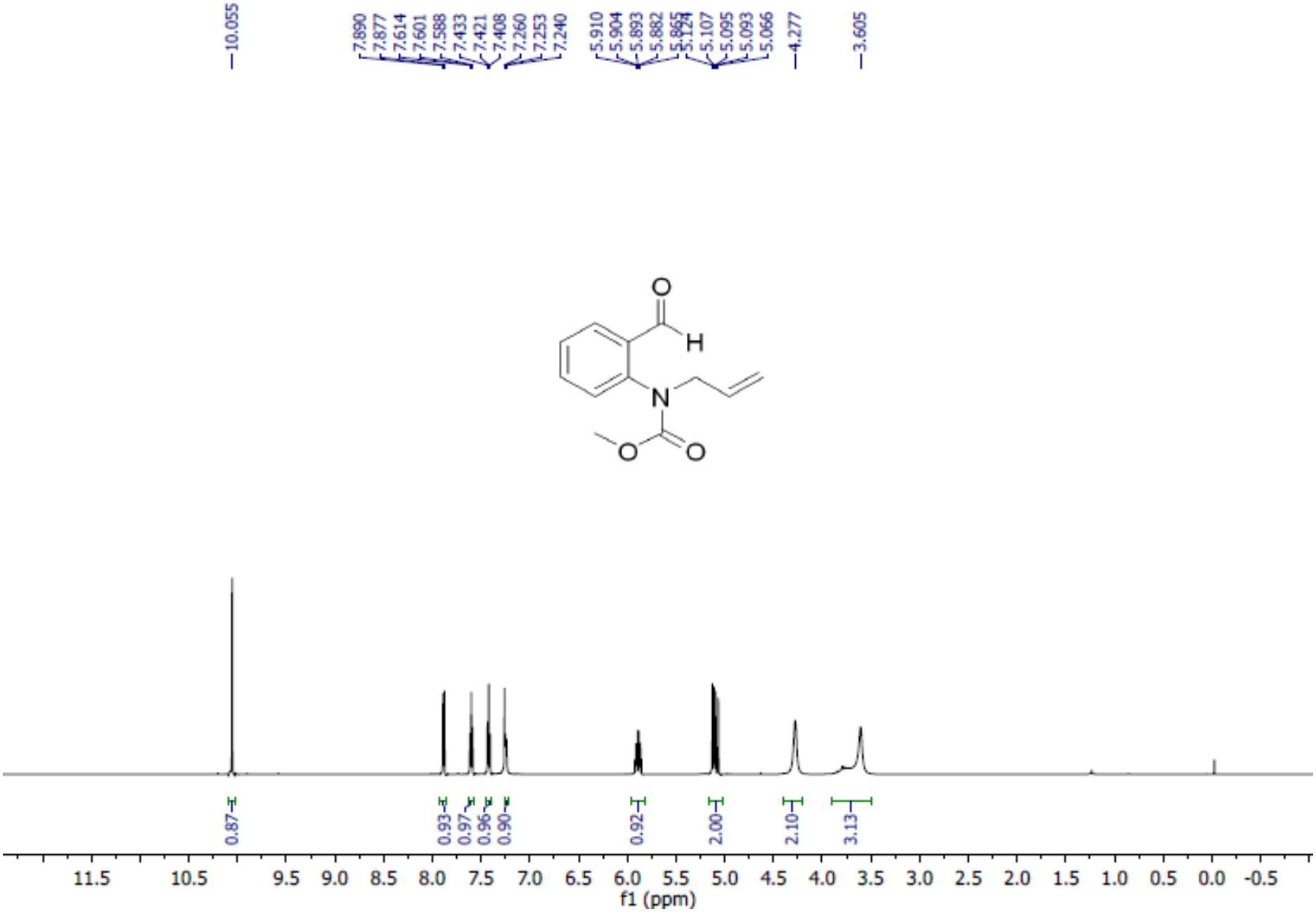
methyl (2-(diethylamino)-2-oxoethyl)(2-formylphenyl)carbamate s3-t



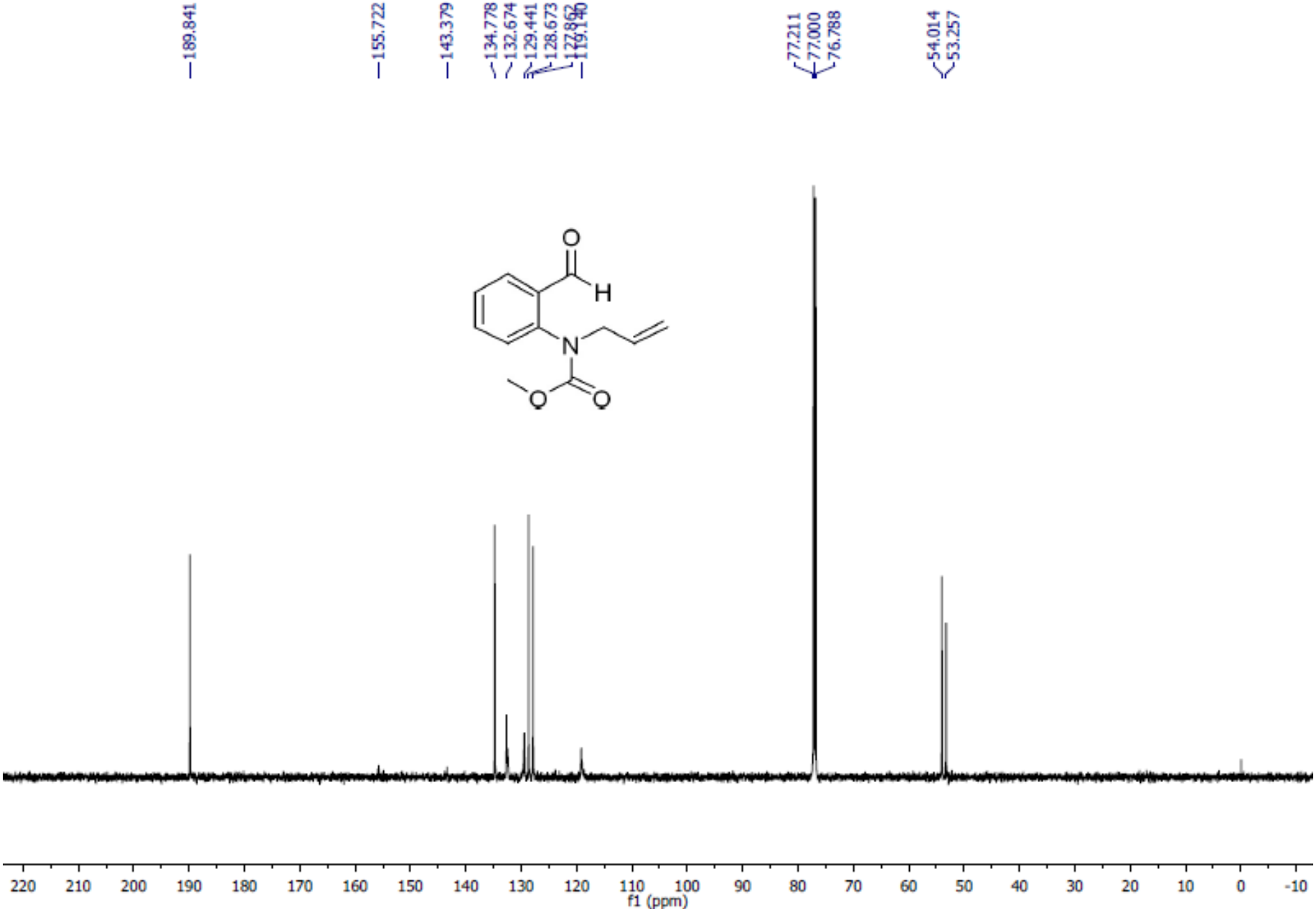
methyl (2-(diethylamino)-2-oxoethyl)(2-formylphenyl)carbamate s3-t



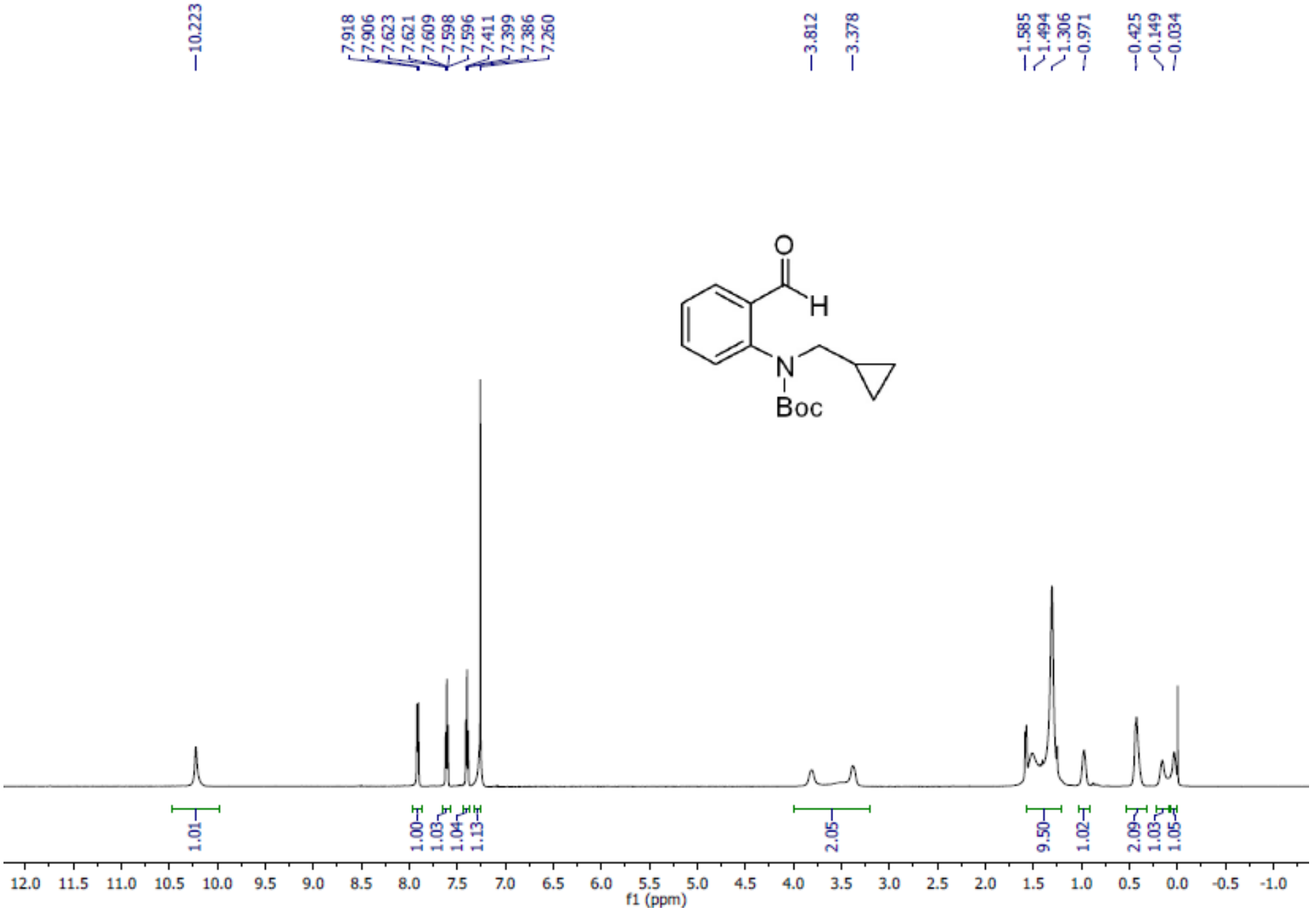
methyl allyl(2-formylphenyl)carbamate s3-u



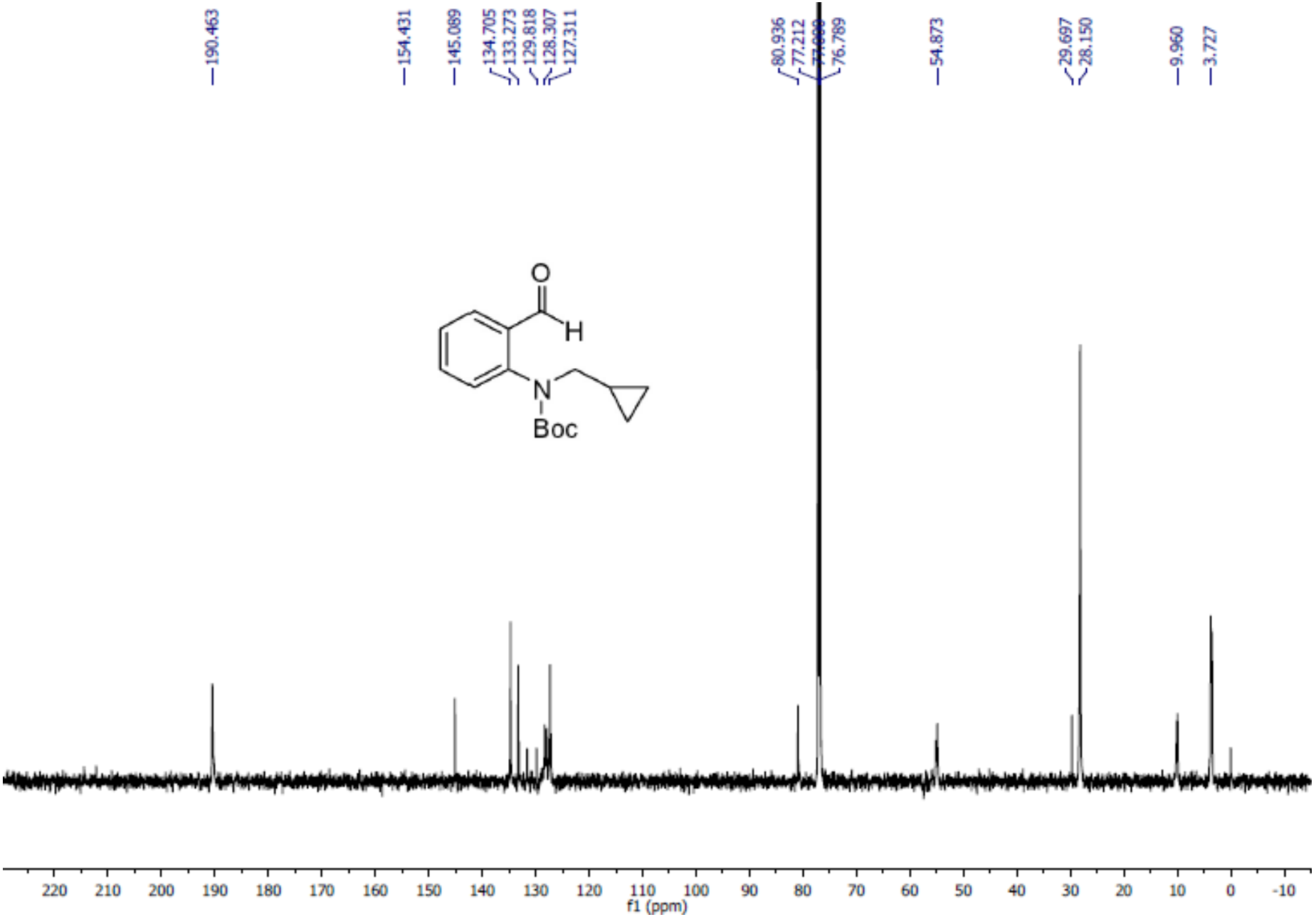
methyl allyl(2-formylphenyl)carbamate s3-u



tert-butyl (cyclopropylmethyl)(2-formylphenyl)carbamate s3-v

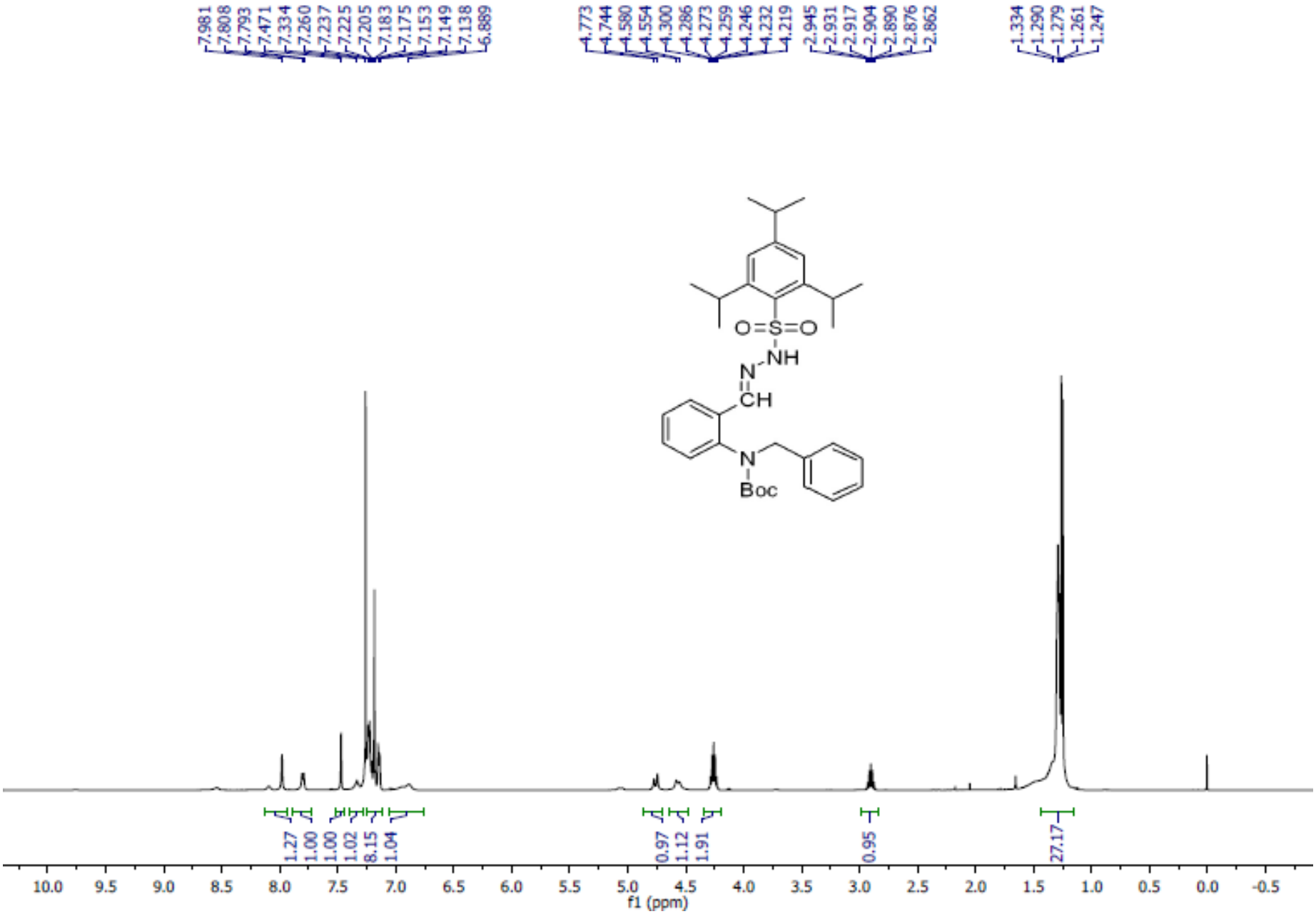


tert-butyl (cyclopropylmethyl)(2-formylphenyl)carbamate s3-v

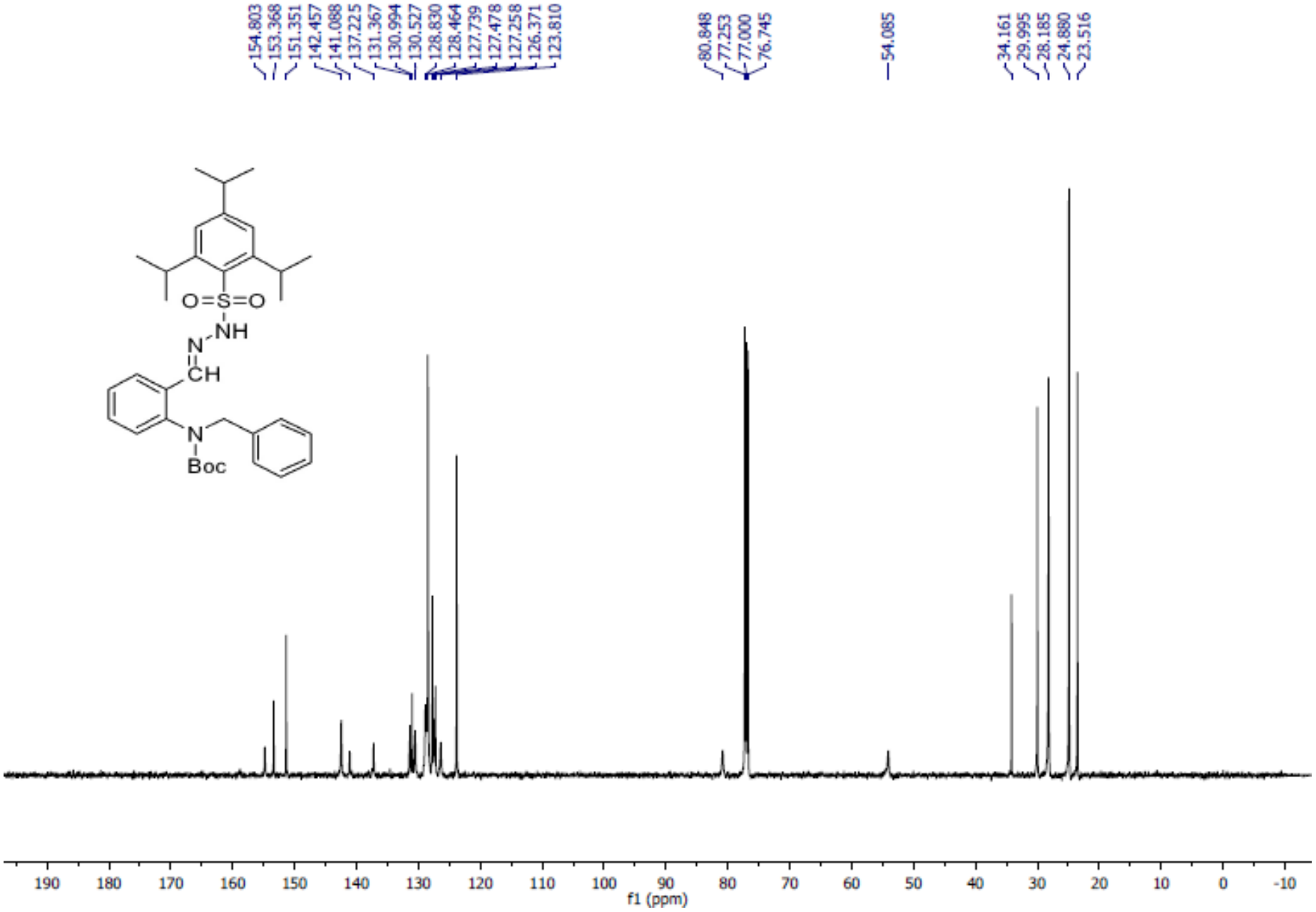


Note: The spectrum contains a mixture of amide rotamers and the major rotamer was labelled.

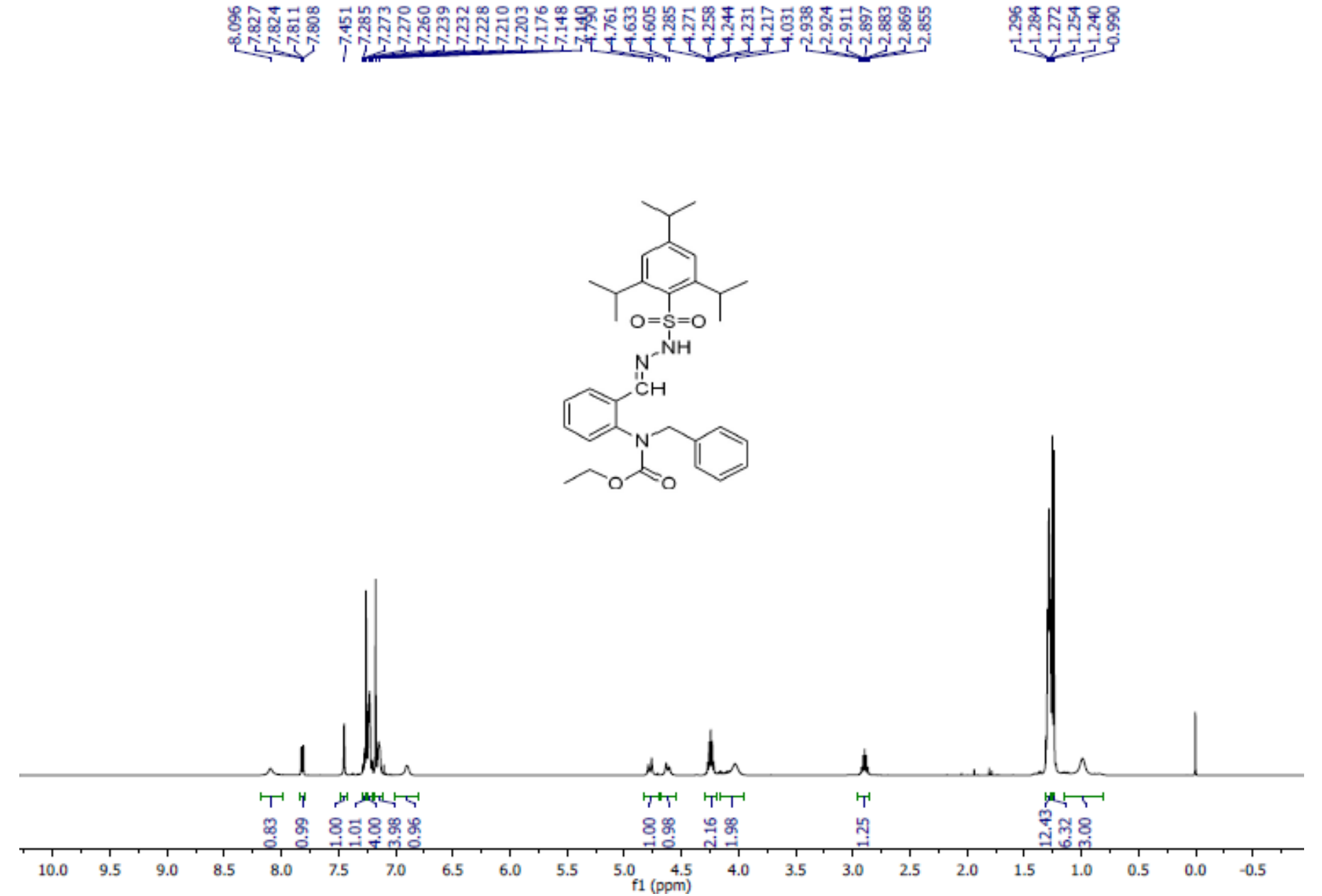
tert-butyl benzyl(2-((2-((2,4,6-triisopropylphenyl)sulfonyl)hydrazono) methyl)phenyl)carbamate 1-a



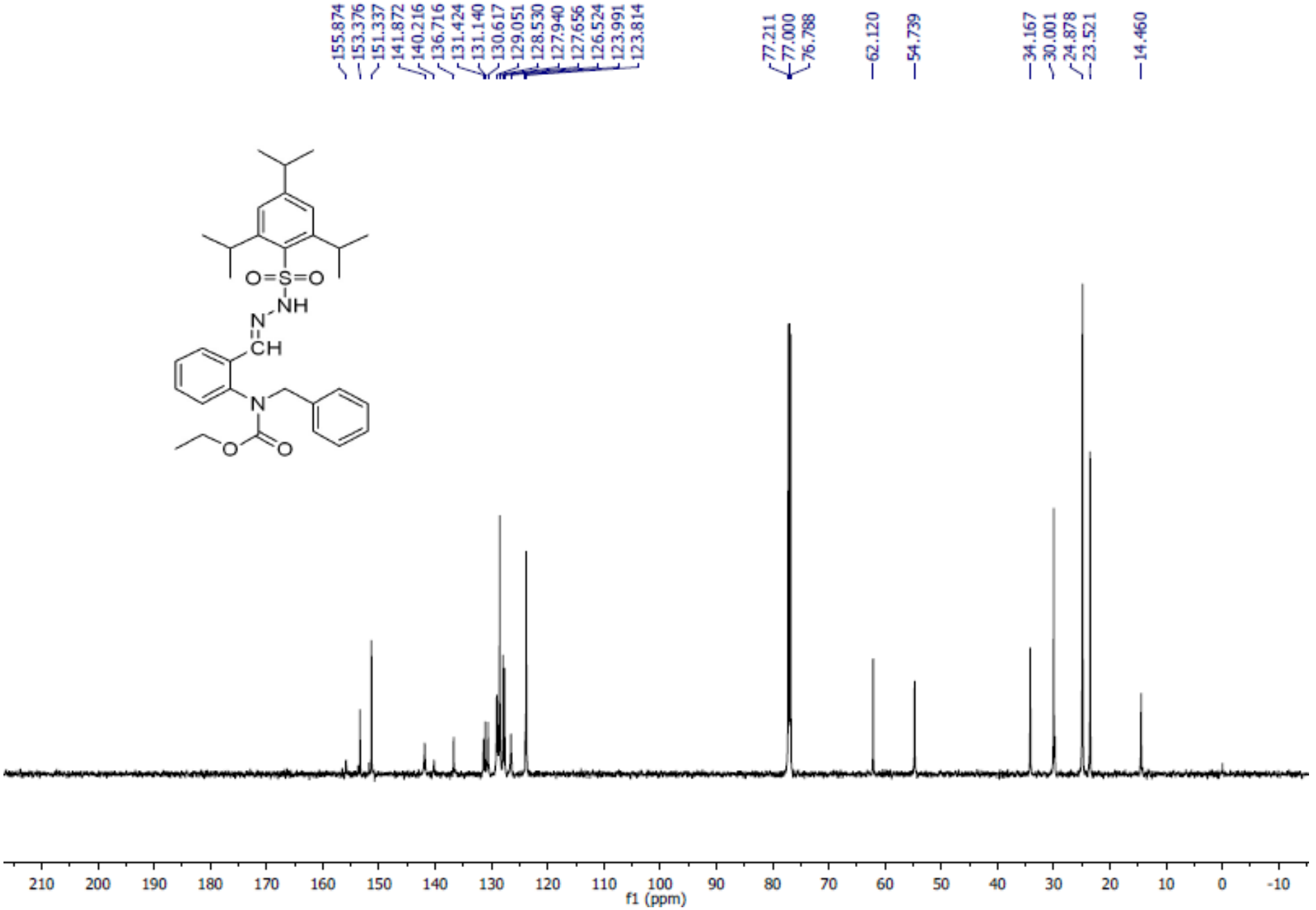
tert-butyl benzyl(2-((2-((2,4,6-triisopropylphenyl)sulfonyl)hydrazono)methyl)phenyl)carbamate 1-a



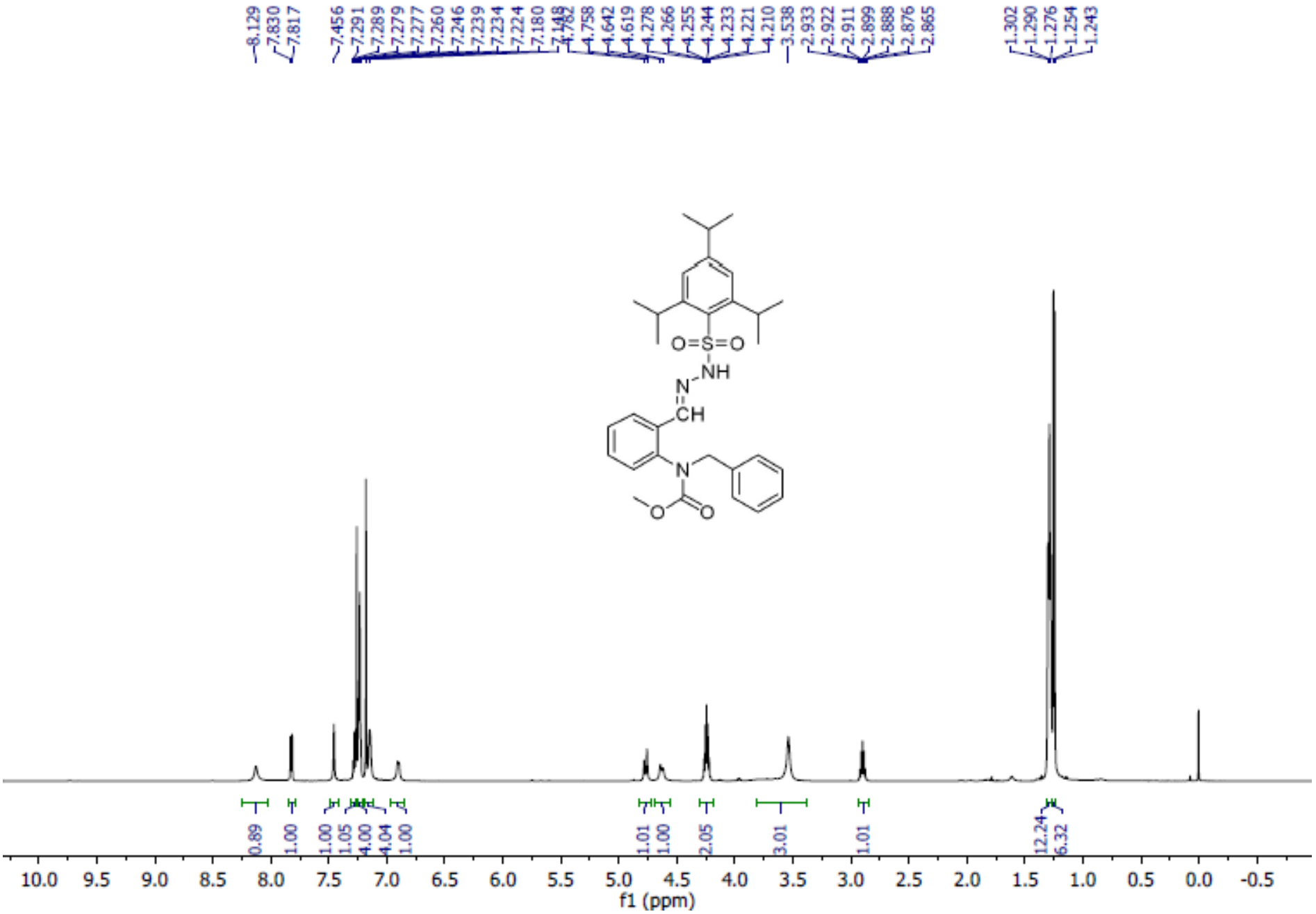
ethyl benzyl(2-((2-((2,4,6-triisopropylphenyl)sulfonyl)hydrazono)methyl)phenyl) carbamate 1-b



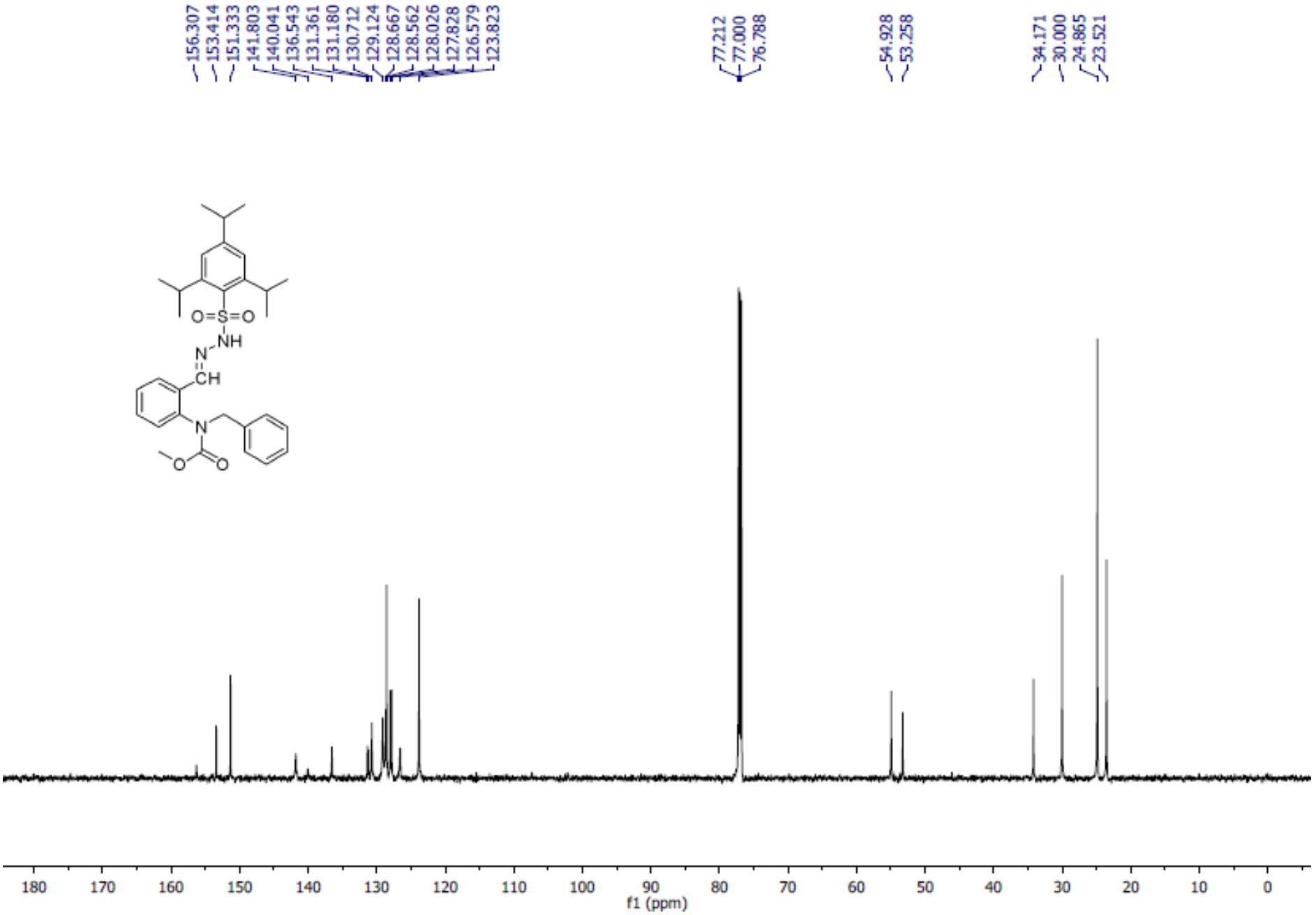
ethyl benzyl(2-((2-((2,4,6-triisopropylphenyl)sulfonyl)hydrazono)methyl)phenyl) carbamate 1-b



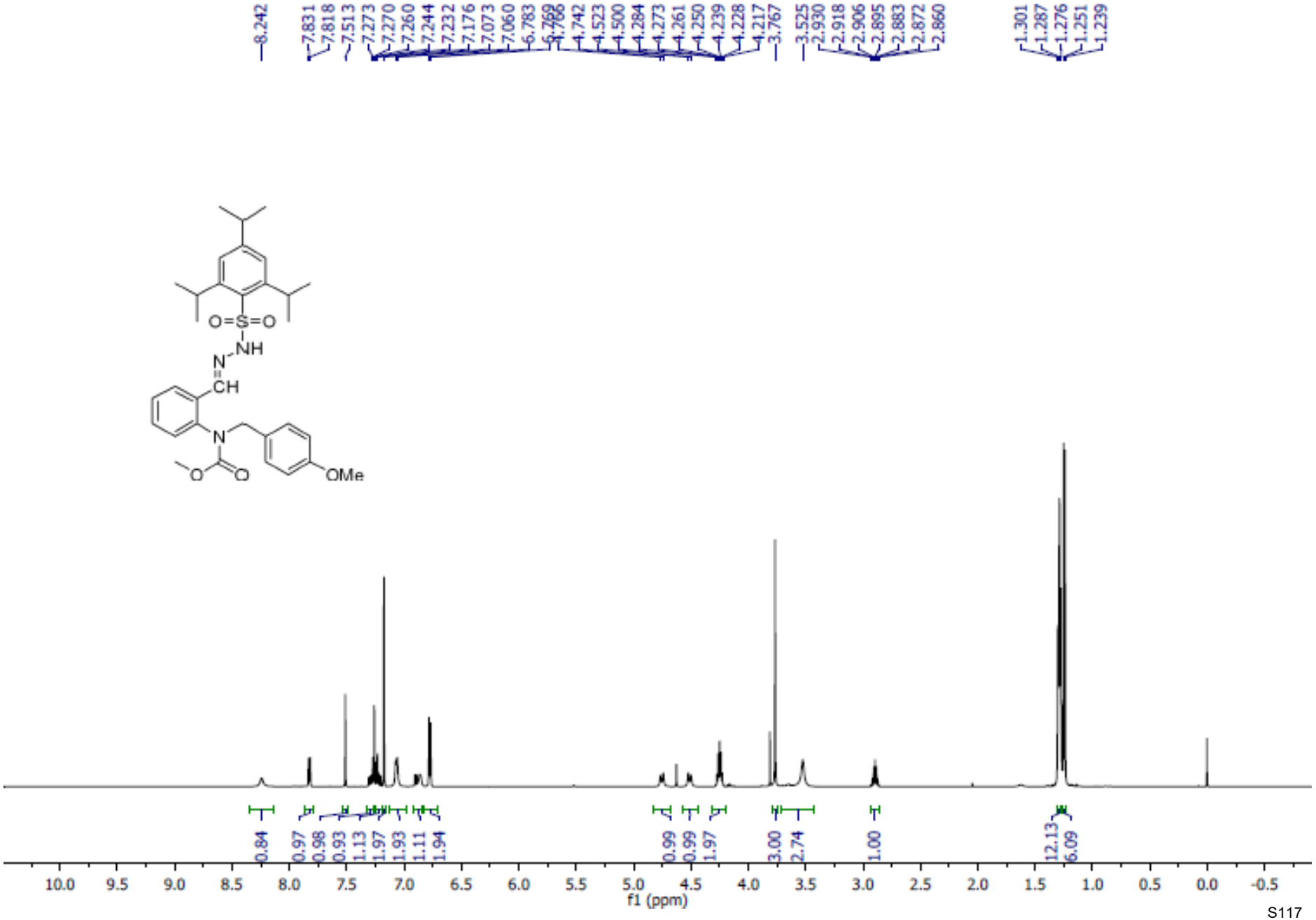
methyl benzyl(2-((2-((2,4,6-triisopropylphenyl)sulfonyl)hydrazono)methyl)phenyl)carbamate 1-c



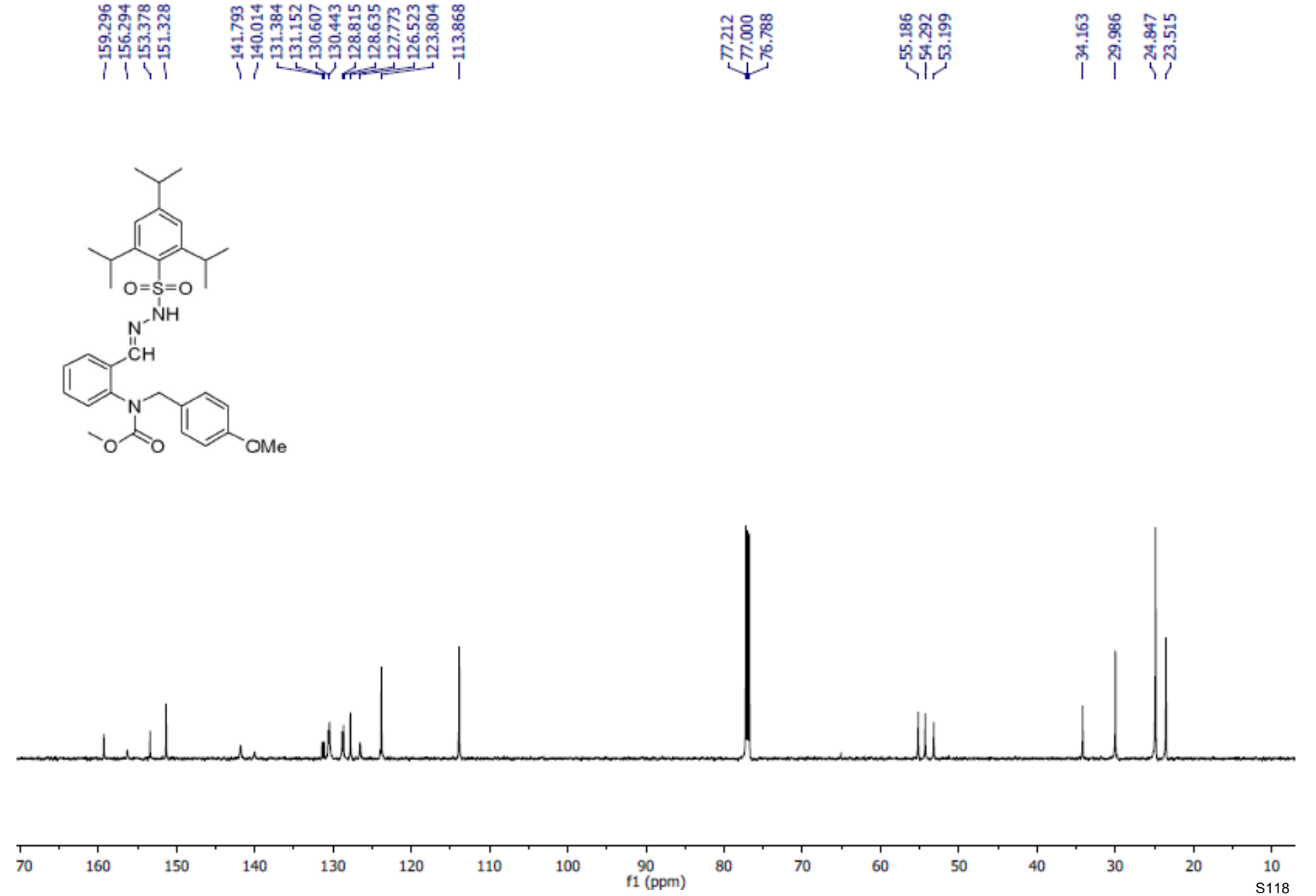
methyl benzyl(2-((2-((2,4,6-triisopropylphenyl)sulfonyl)hydrazono)methyl)phenyl)carbamate 1-c



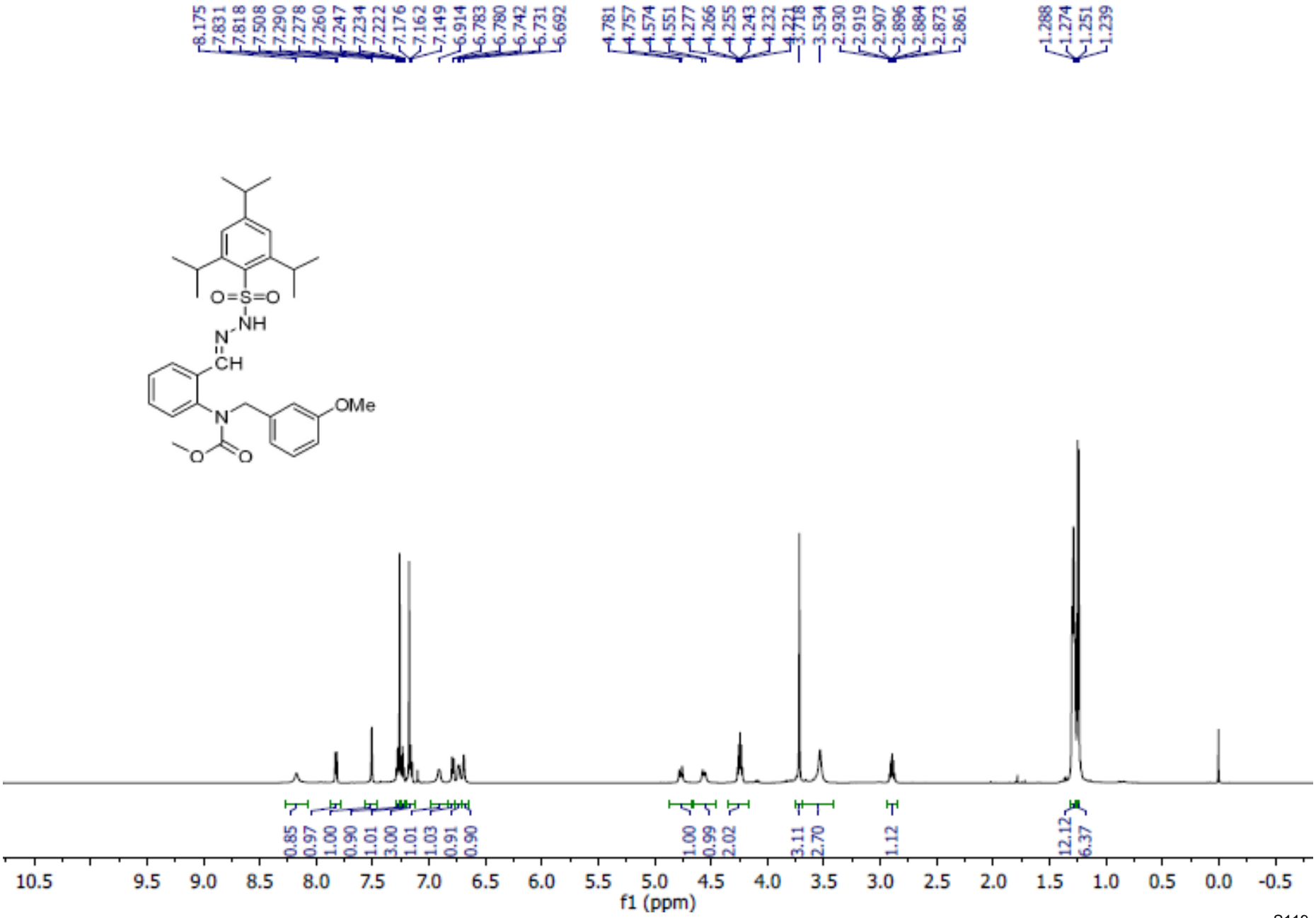
methyl (4-methoxybenzyl)(2-((2-((2,4,6-triisopropylphenyl) sulfonyl)hydrazono)methyl)phenyl)carbamate 1-d



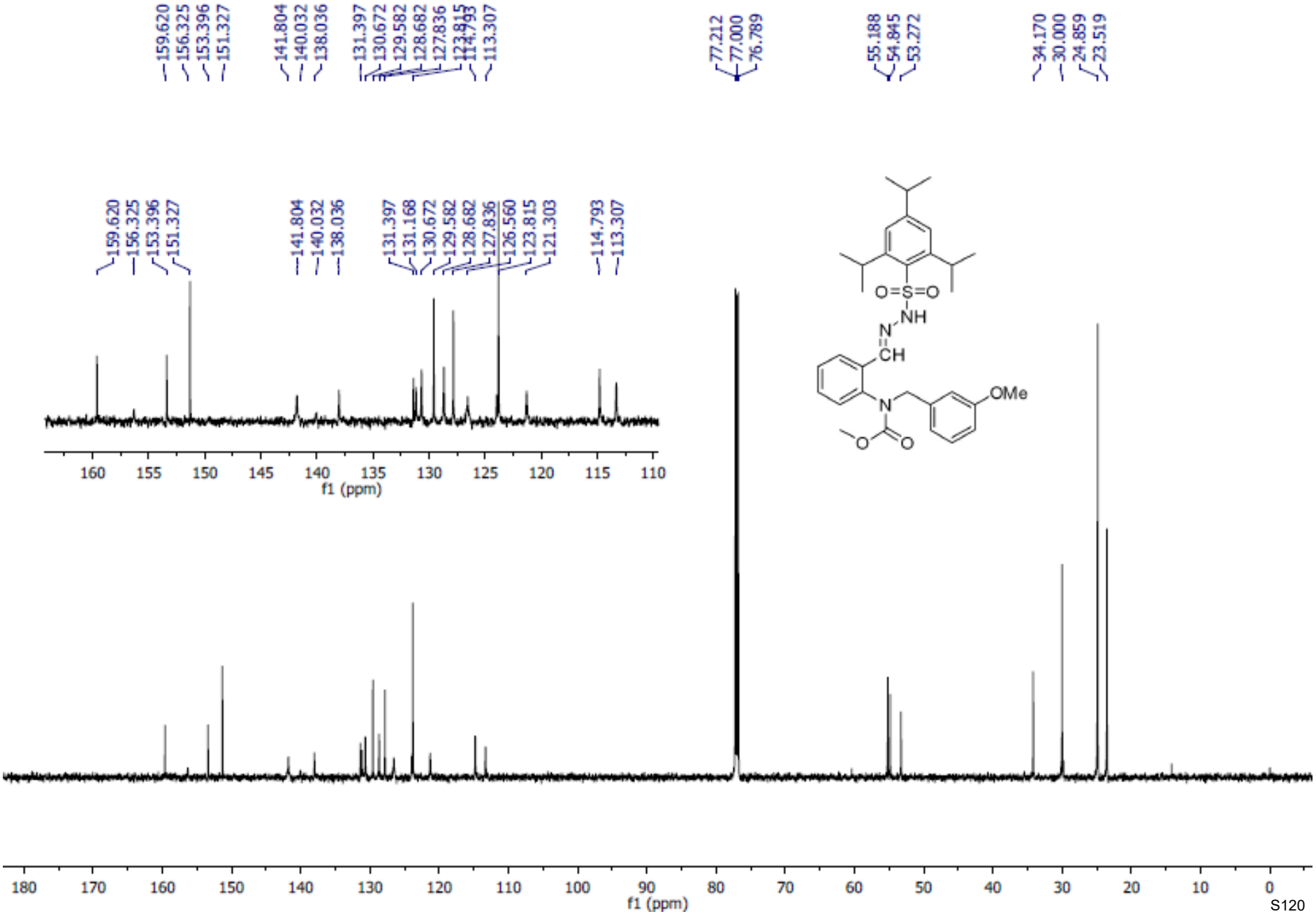
methyl (4-methoxybenzyl)(2-((2-((2,4,6-triisopropylphenyl) sulfonyl)hydrazono)methyl)phenyl)carbamate 1-d



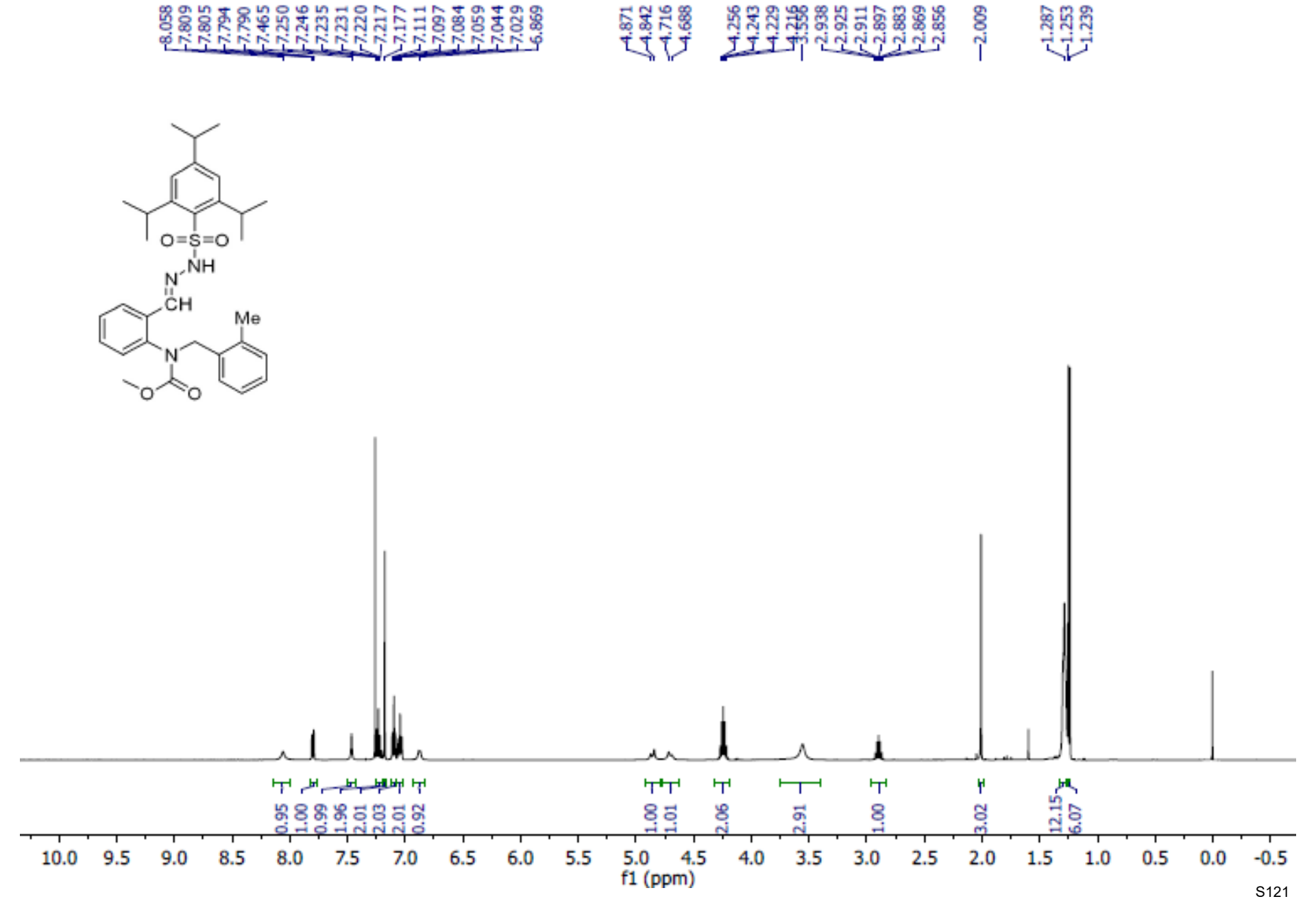
methyl (3-methoxybenzyl)(2-((2-((2,4,6-triisopropylphenyl) sulfonyl) hydrazono)methyl)phenyl)carbamate 1-e



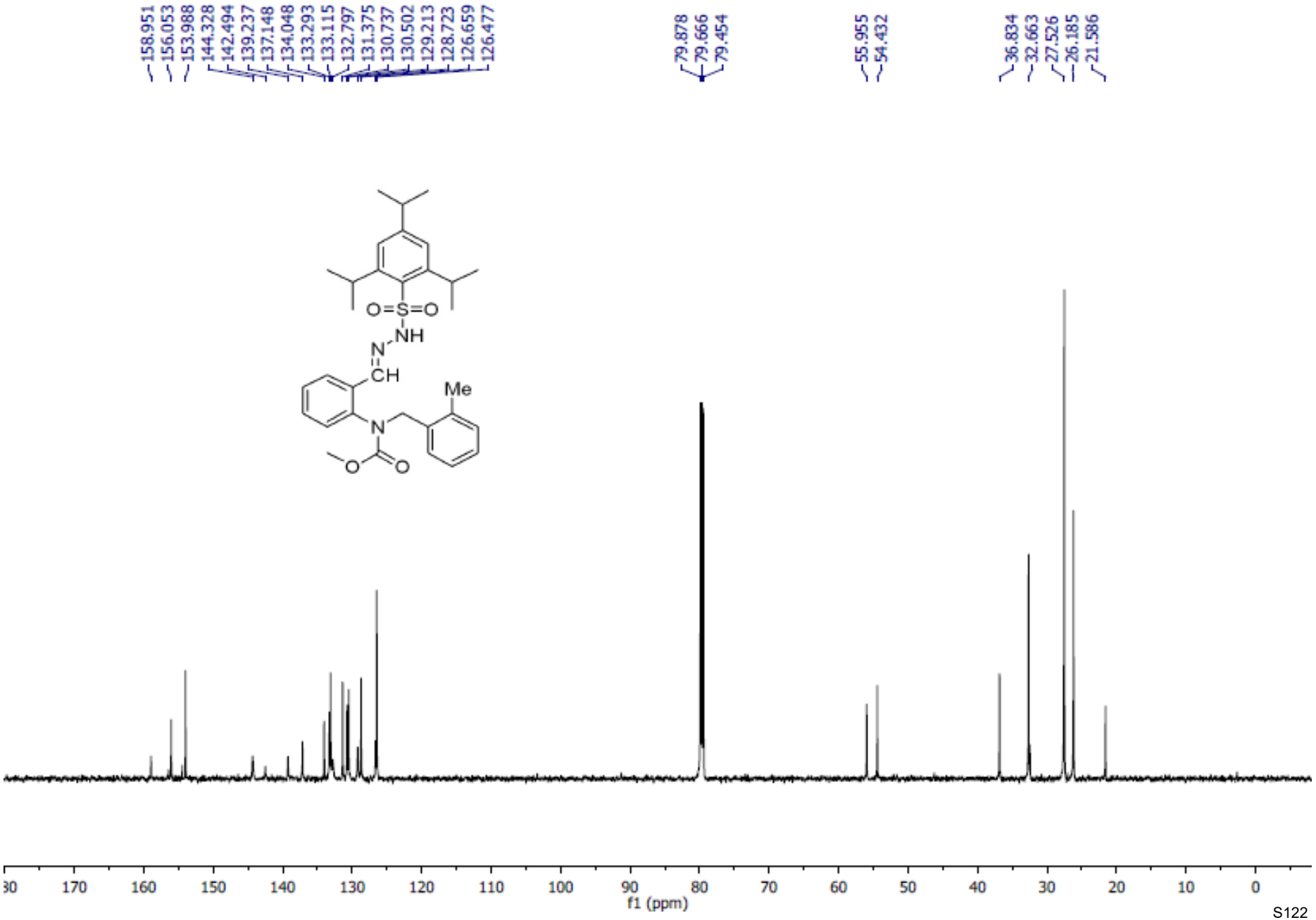
methyl (3-methoxybenzyl)(2-((2-((2,4,6-triisopropylphenyl) sulfonyl) hydrazono)methyl)phenyl)carbamate 1-e



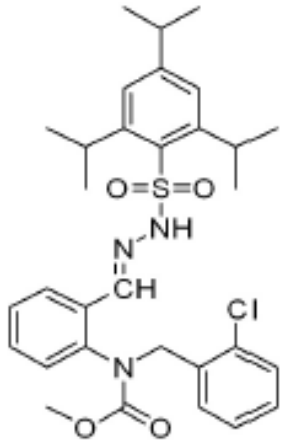
methyl (2-methylbenzyl)(2-((2-((2,4,6-triisopropylphenyl)sulfonyl) hydrazono)methyl)phenyl)carbamate 1-f

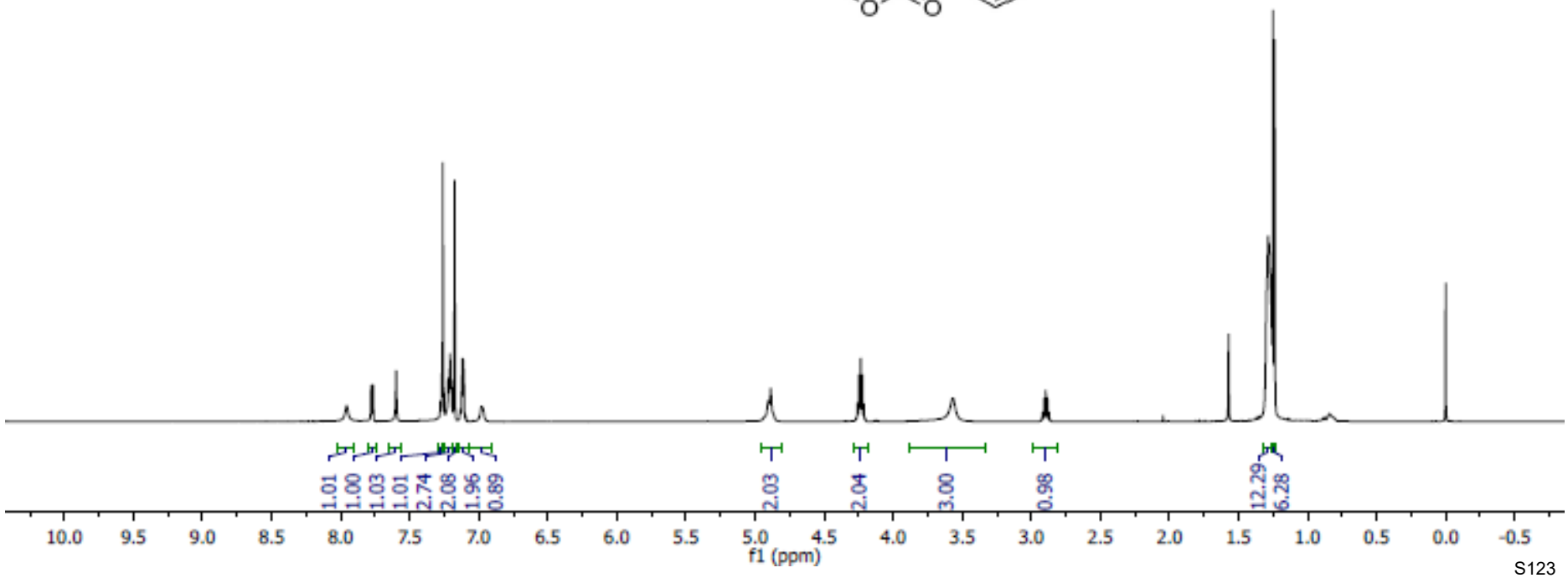


methyl (2-methylbenzyl)(2-((2-((2,4,6-triisopropylphenyl)sulfonyl) hydrazono)methyl)phenyl)carbamate 1-f

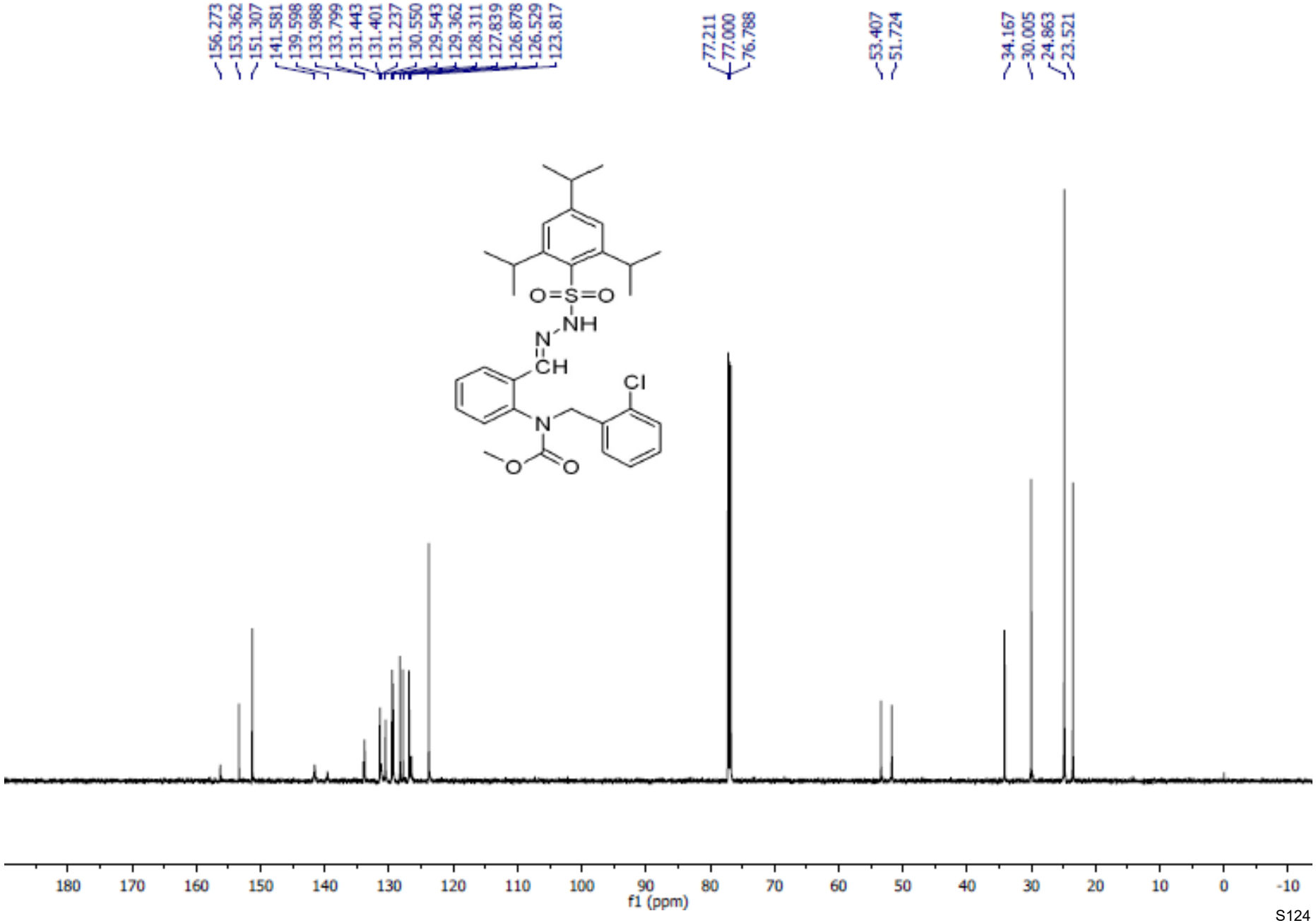


methyl (2-chlorobenzyl)(2-((2-((2,4,6-triisopropylphenyl)sulfonyl) hydrazono)methyl)phenyl)carbamate 1-g

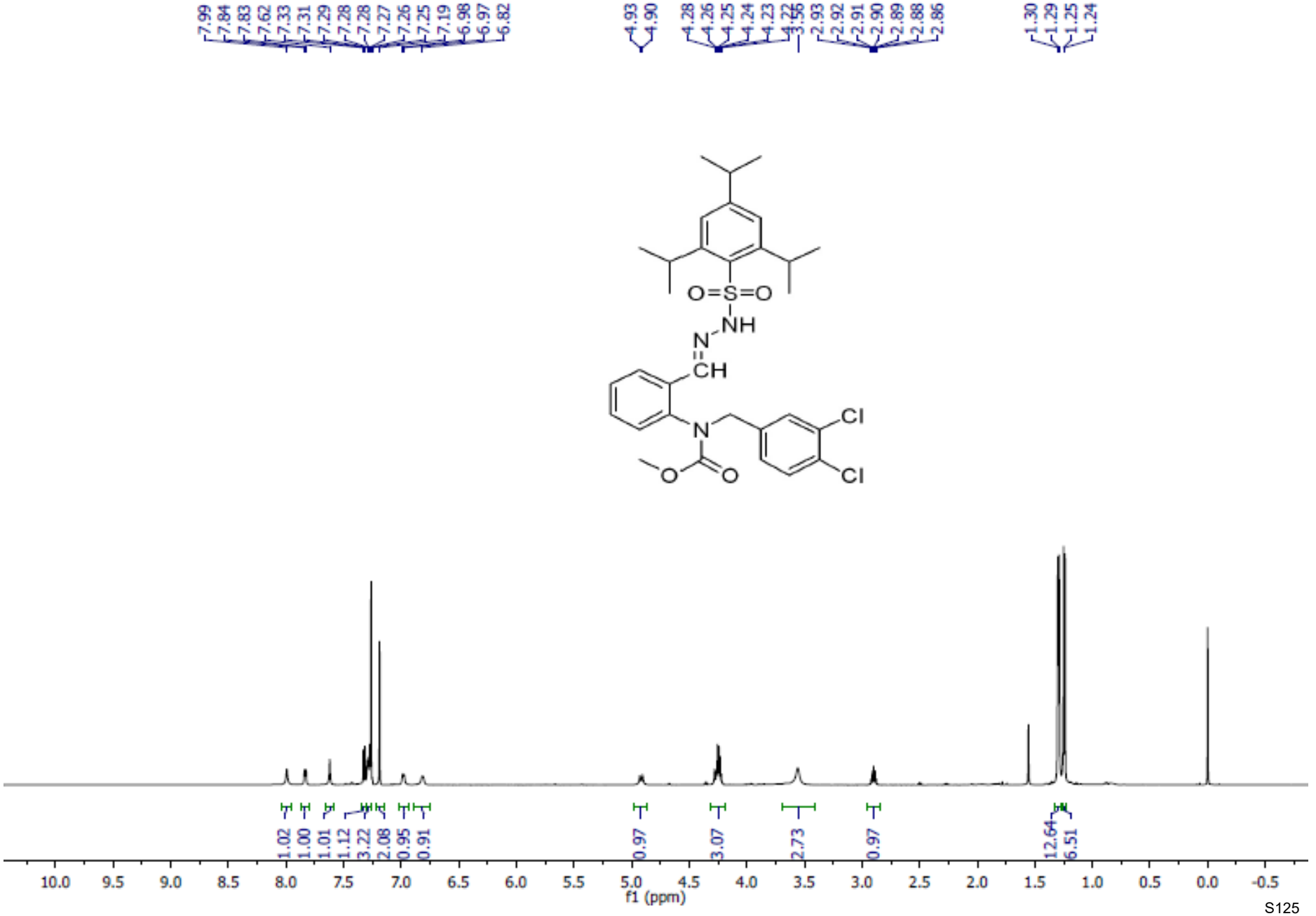




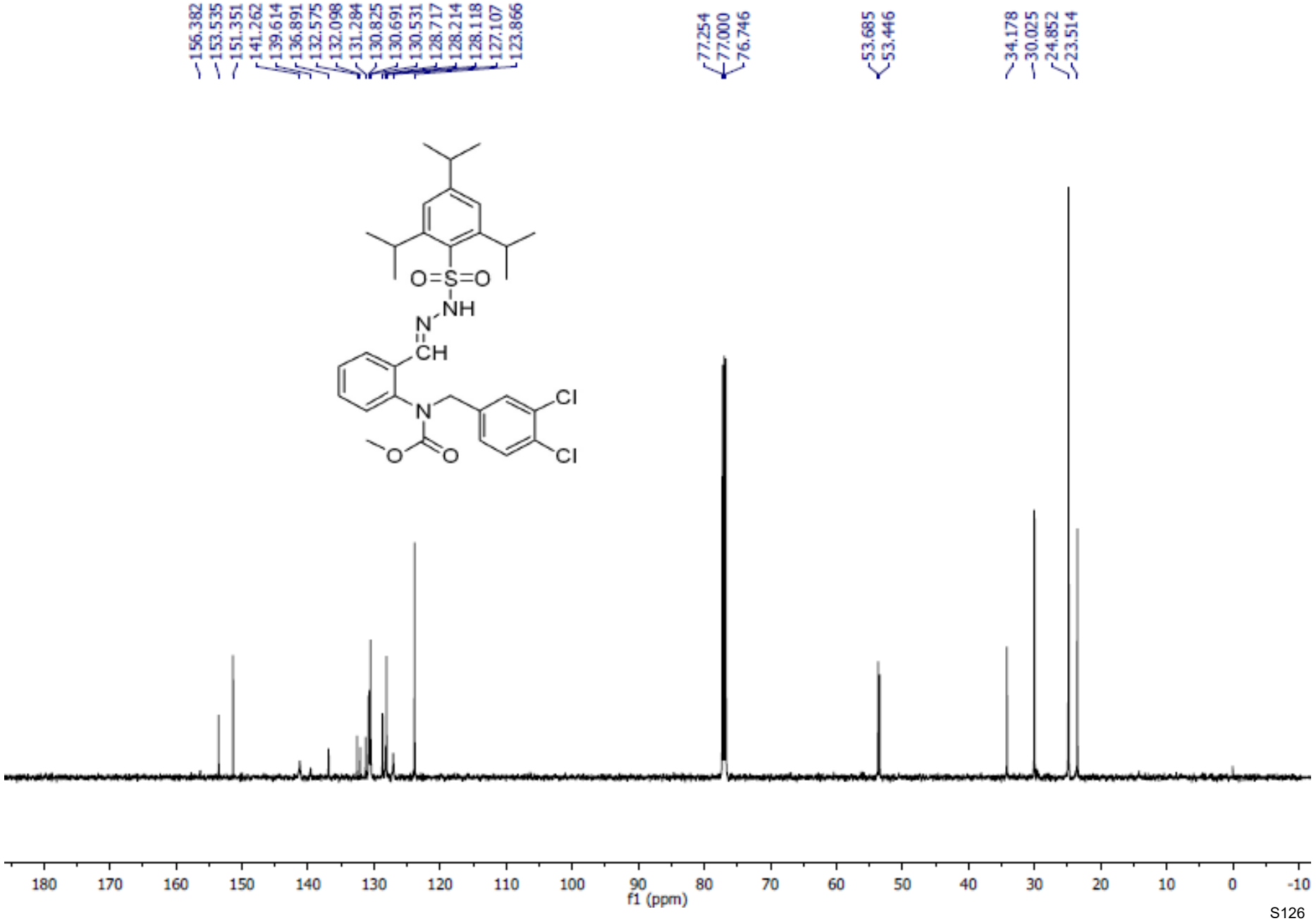
methyl (2-chlorobenzyl)(2-((2-((2,4,6-triisopropylphenyl)sulfonyl) hydrazono)methyl)phenyl)carbamate 1-g



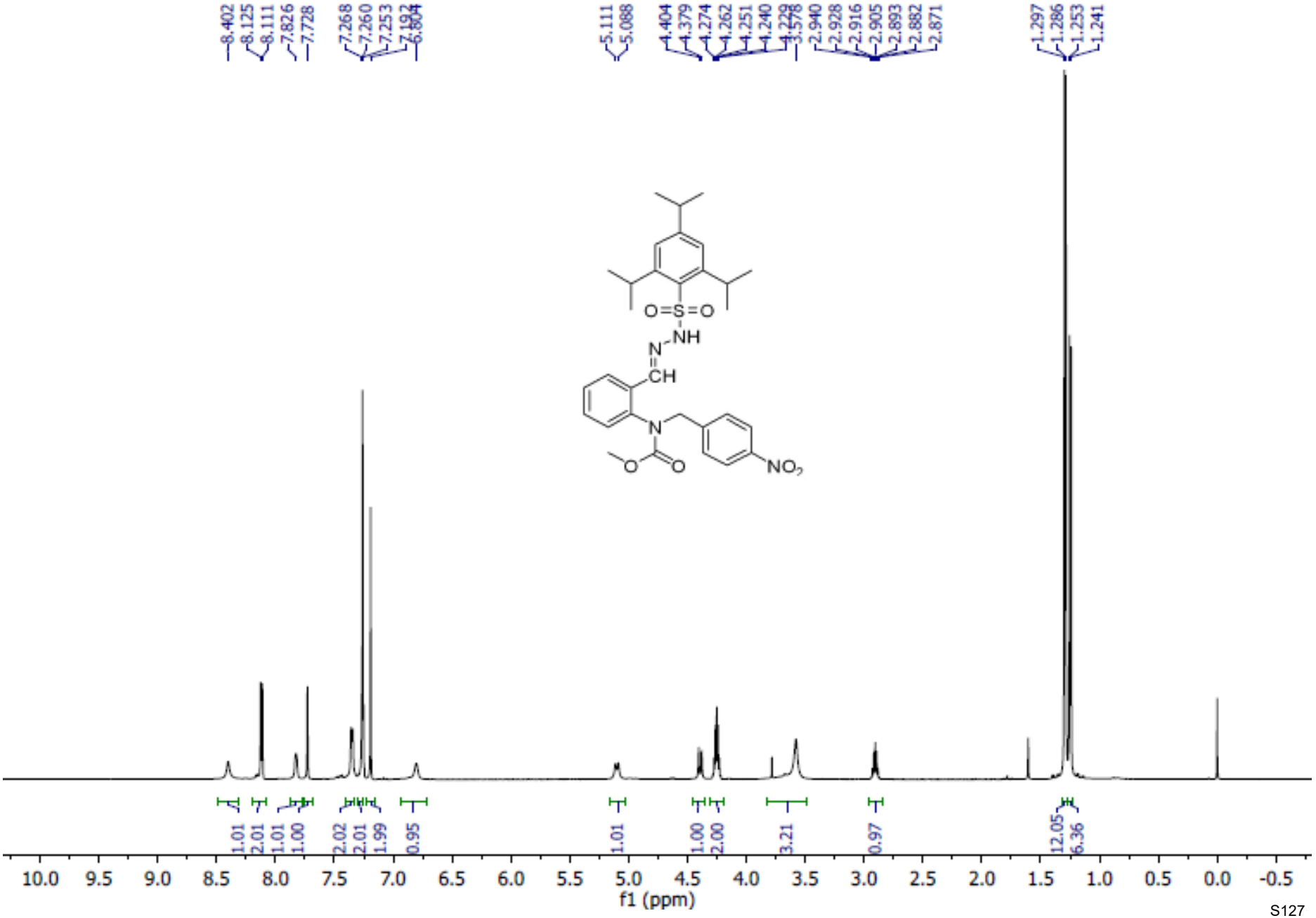
methyl (3,4-dichlorobenzyl)(2-((2-((2,4,6-triisopropylphenyl)sulfonyl) hydrazono)methyl)phenyl)carbamate 1-h



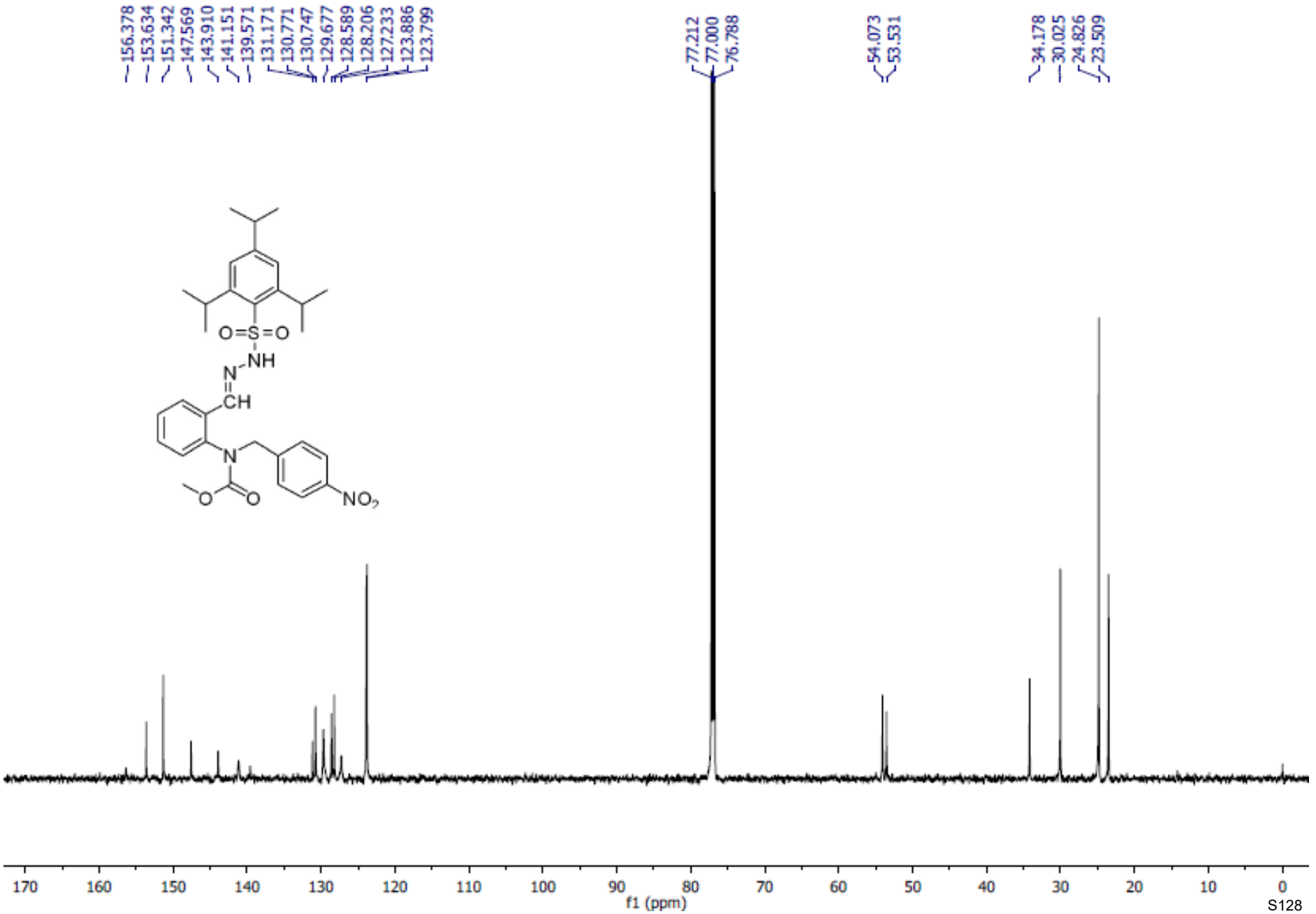
methyl (3,4-dichlorobenzyl)(2-((2-((2,4,6-triisopropylphenyl)sulfonyl) hydrazono)methyl)phenyl)carbamate 1-h



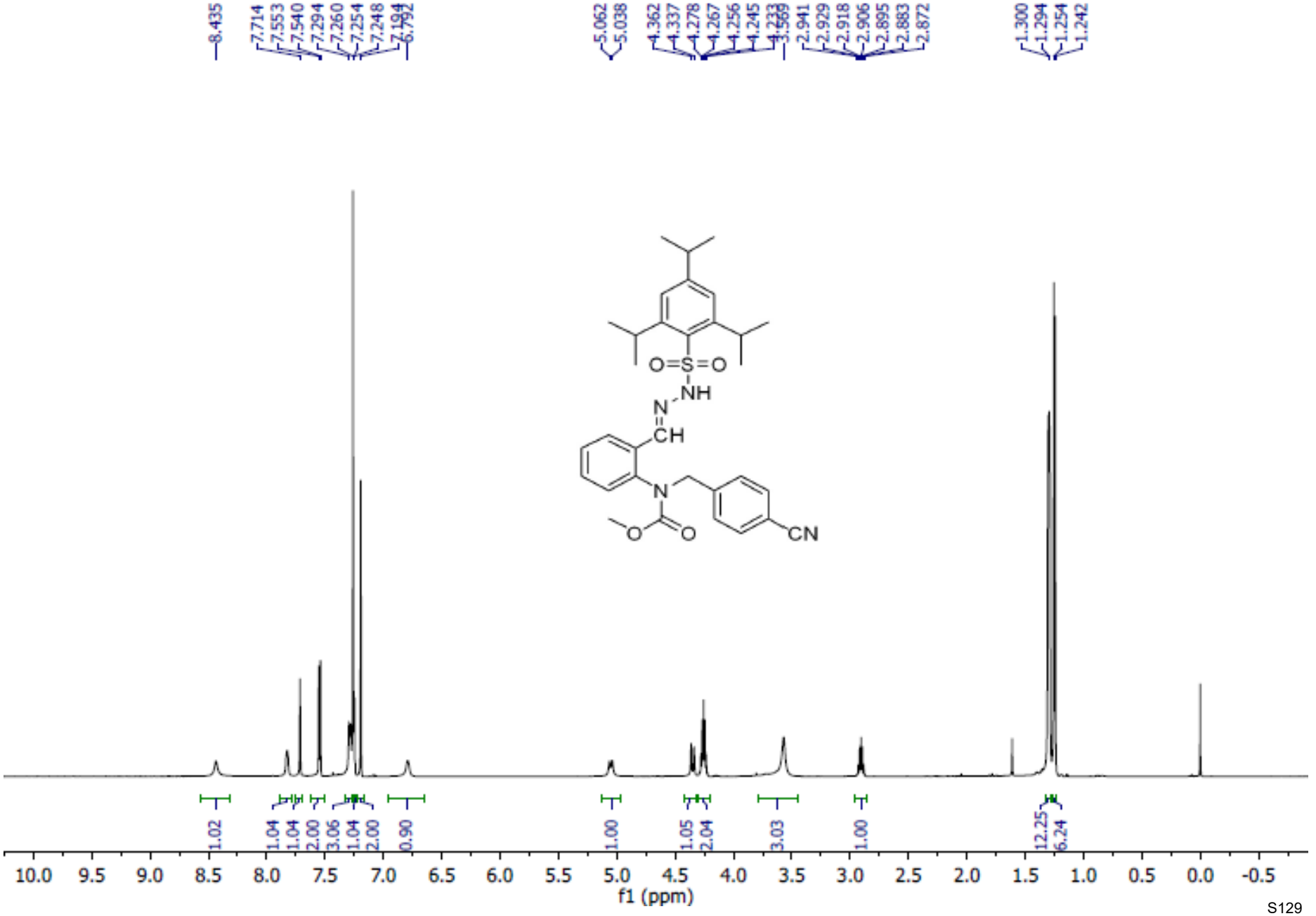
methyl (4-nitrobenzyl)(2-((2-((2,4,6-triisopropylphenyl)sulfonyl) hydrazono)methyl)phenyl)carbamate 1-i



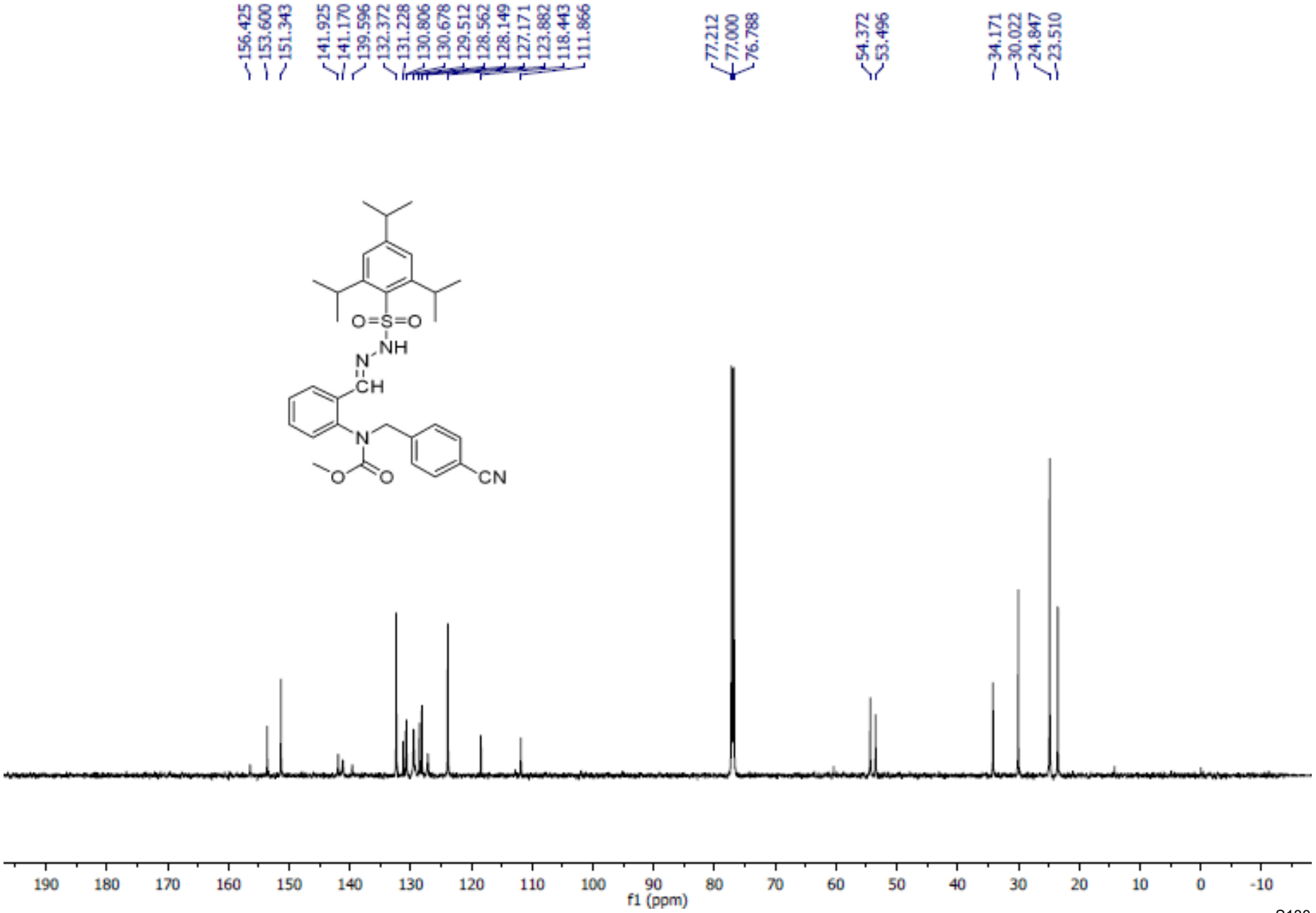
methyl (4-nitrobenzyl)(2-((2-((2,4,6-triisopropylphenyl)sulfonyl) hydrazono)methyl)phenyl)carbamate 1-i



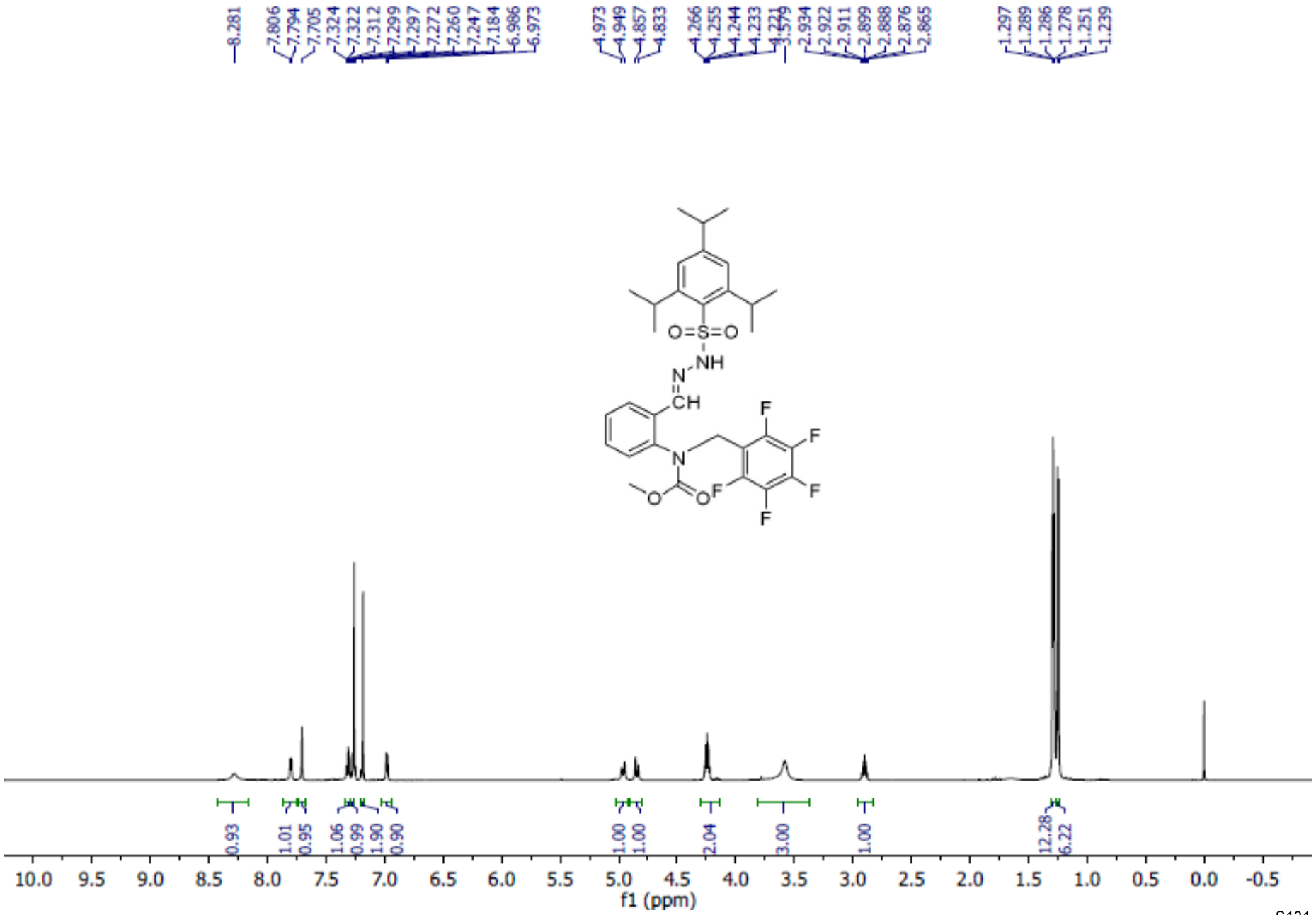
methyl (4-cyanobenzyl)(2-((2-((2,4,6-triisopropylphenyl)sulfonyl) hydrazono)methyl)phenyl)carbamate 1-j



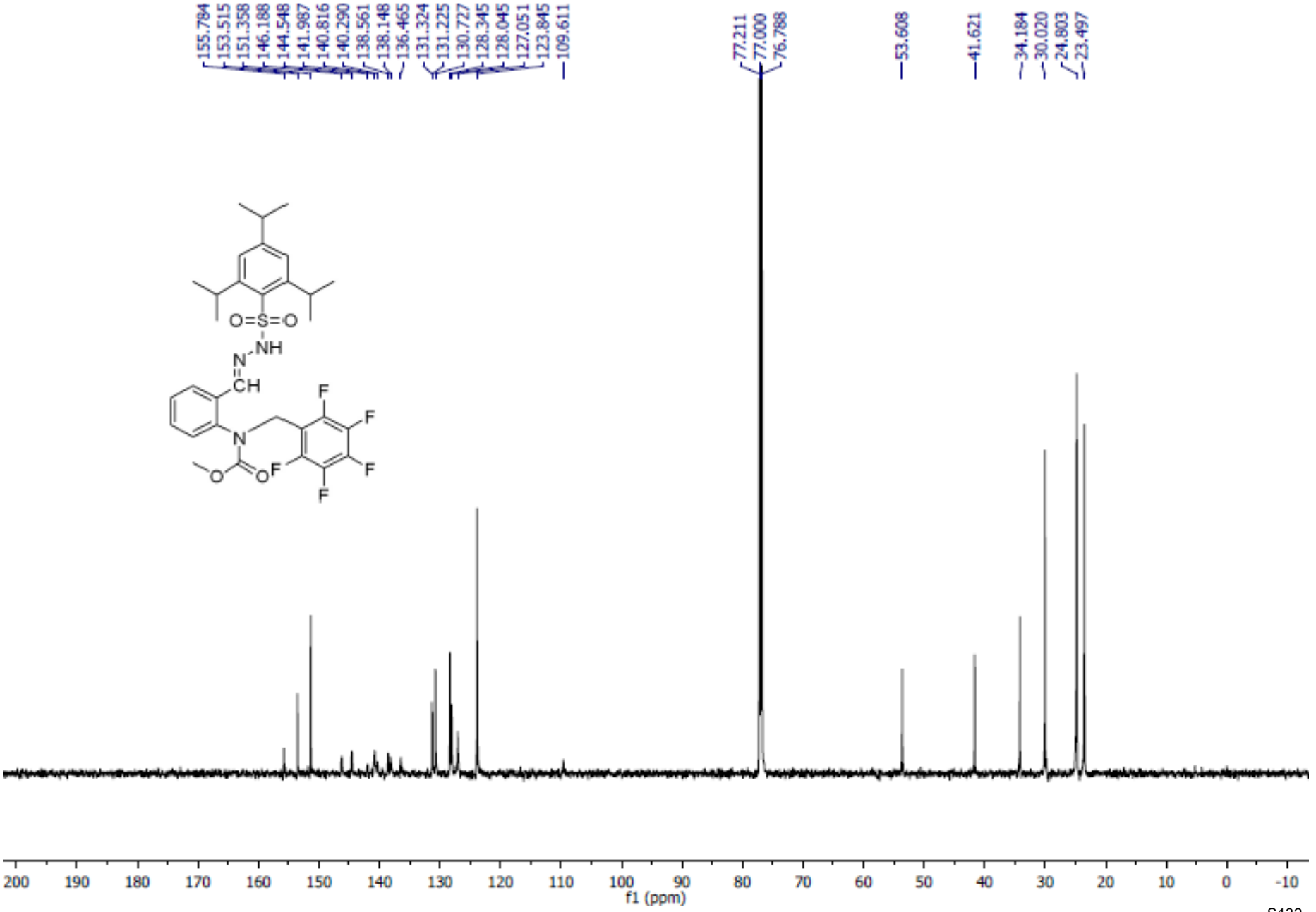
methyl (4-cyanobenzyl)(2-((2-((2,4,6-triisopropylphenyl)sulfonyl) hydrazono)methyl)phenyl)carbamate 1-j



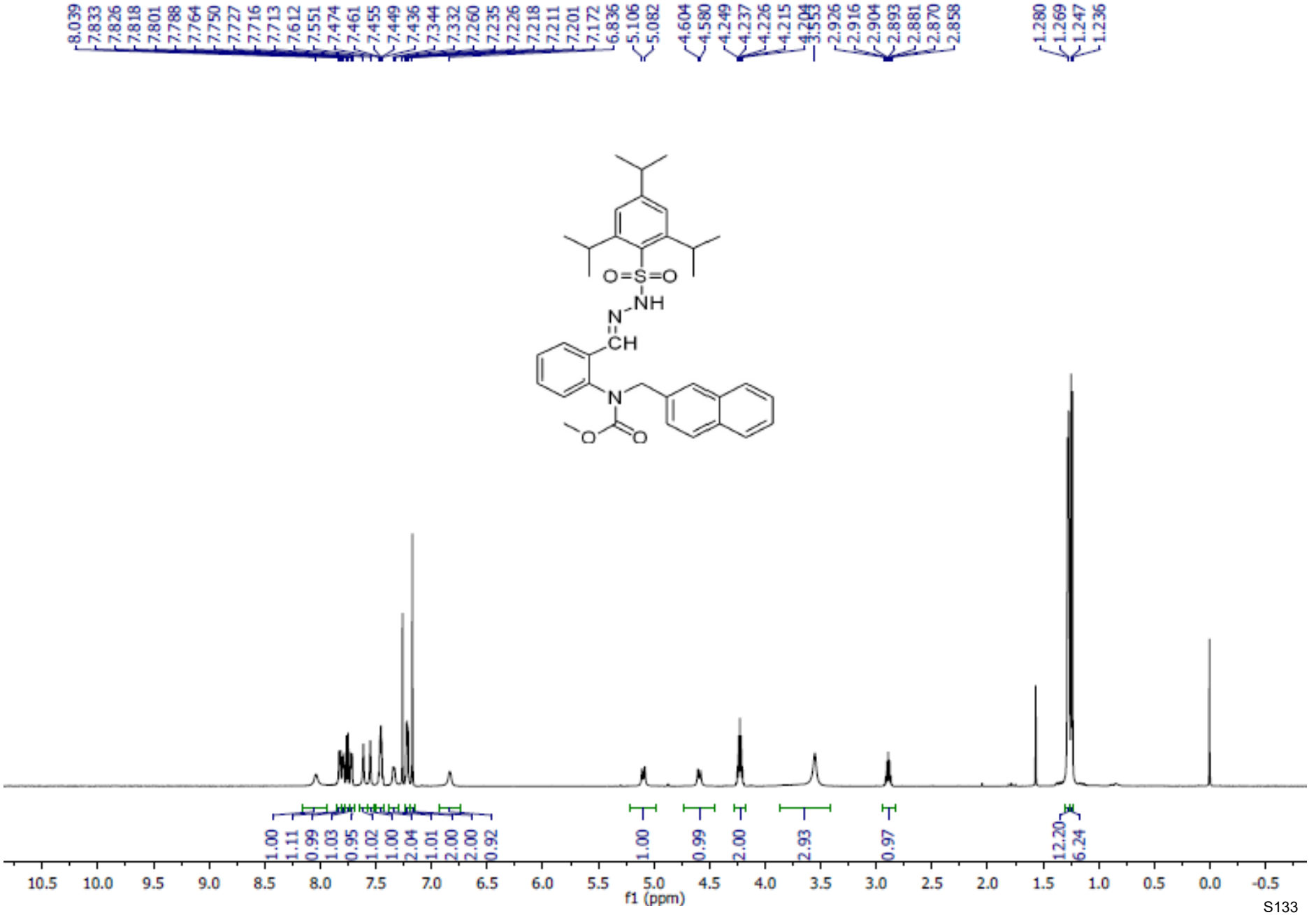
methyl ((perfluorophenyl)methyl)(2-((2-((2,4,6-triisopropylphenyl) sulfonyl)hydrazono)methyl)phenyl)carbamate 1-k



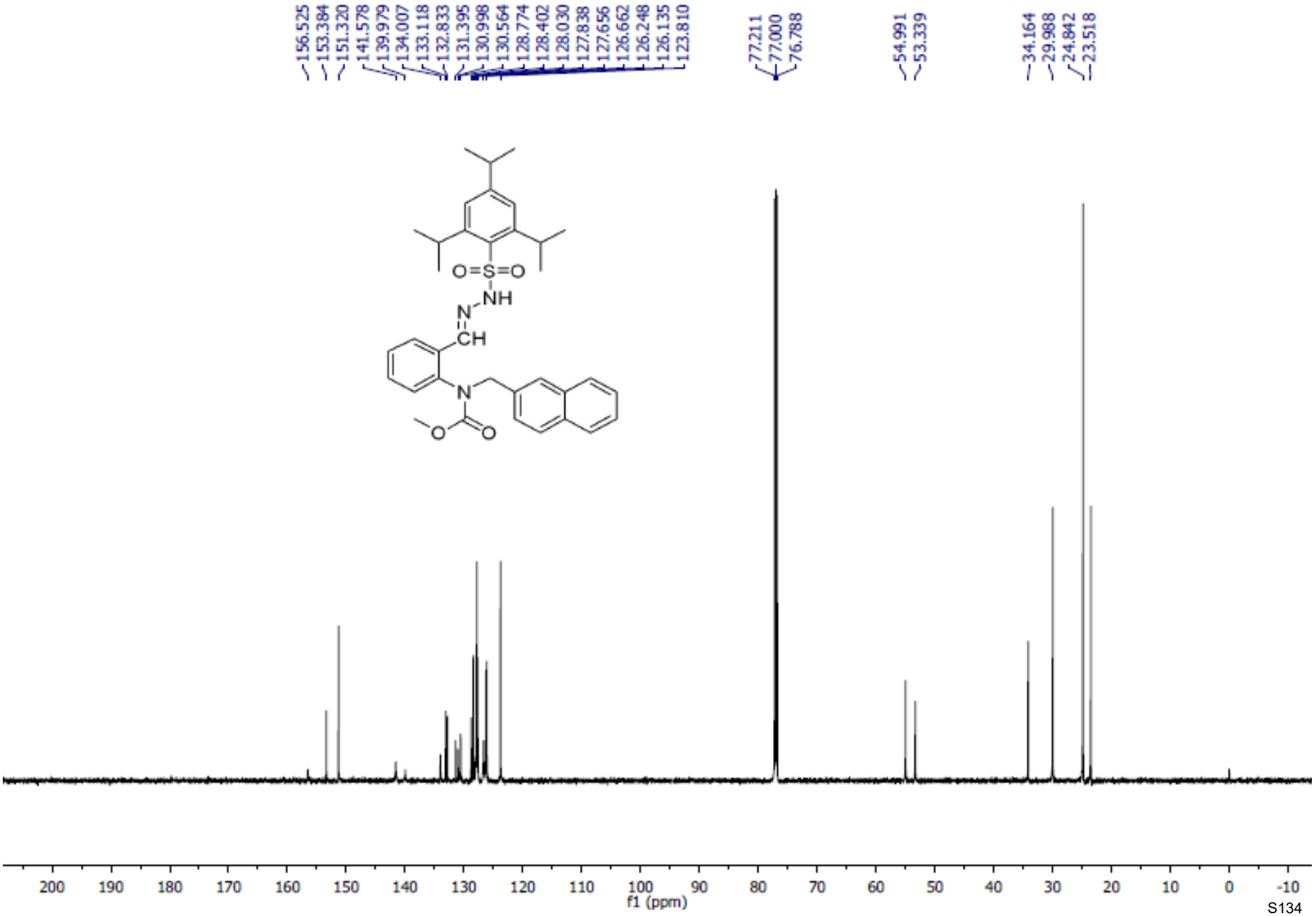
methyl ((perfluorophenyl)methyl)(2-((2-((2,4,6-triisopropylphenyl) sulfonyl)hydrazono)methyl)phenyl)carbamate 1-k



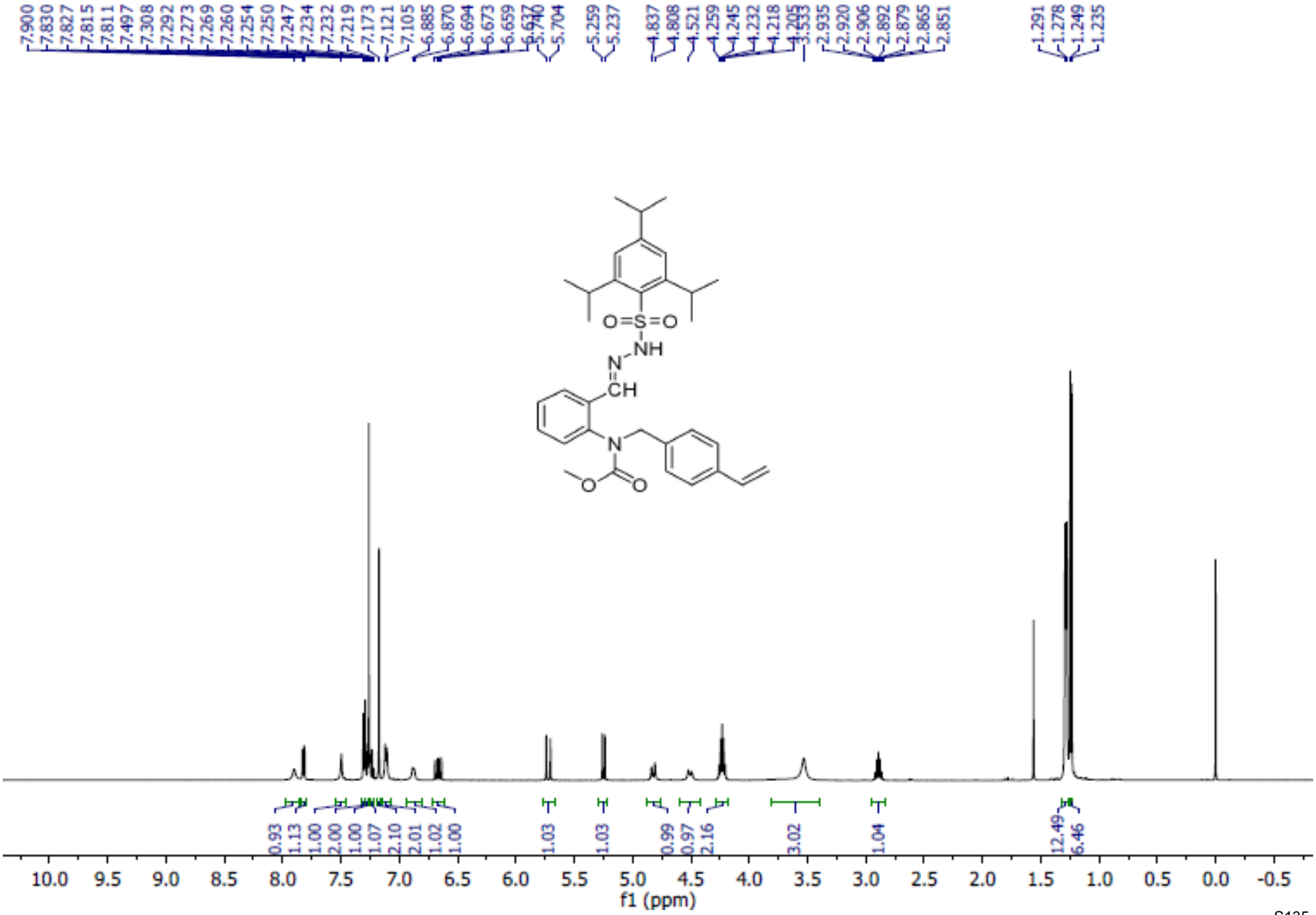
methyl (naphthalen-2-ylmethyl)(2-((2-((2,4,6-triisopropylphenyl) sulfonyl)hydrazono)methyl)phenyl)carbamate 1-l



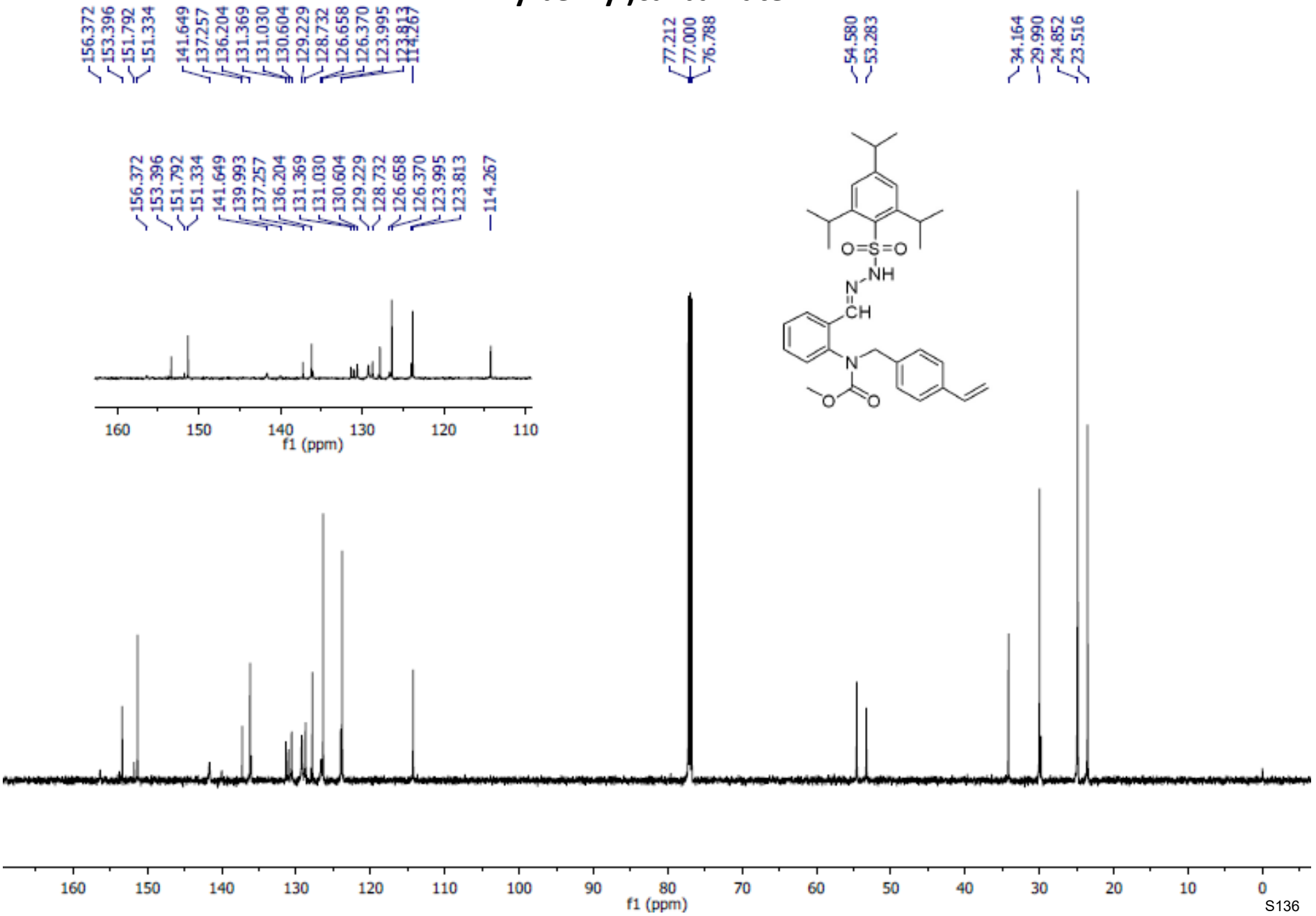
methyl (naphthalen-2-ylmethyl)(2-((2-((2,4,6-triisopropylphenyl) sulfonyl)hydrazono)methyl)phenyl)carbamate 1-l



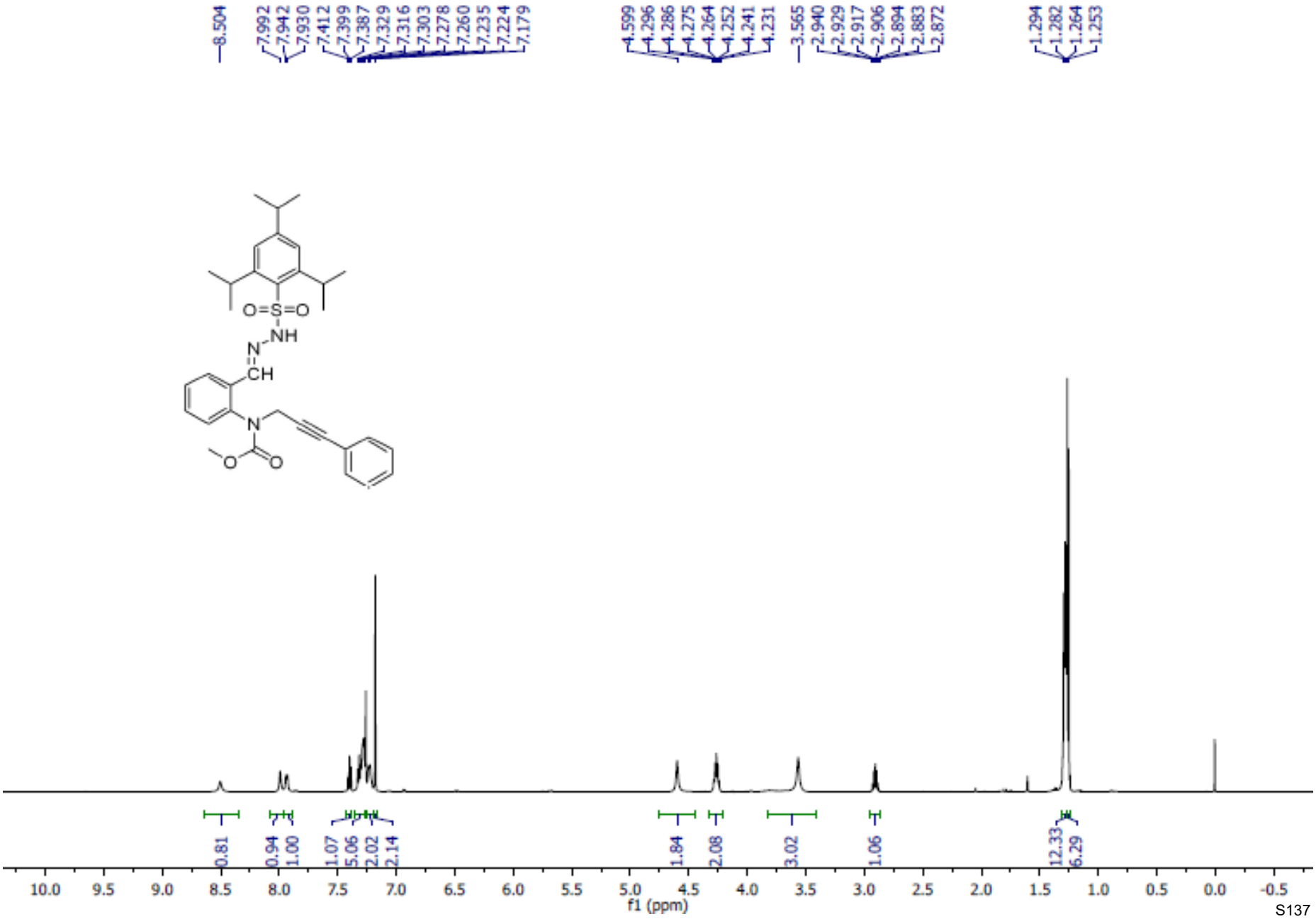
methyl (2-((2-((2,4,6-triisopropylphenyl)sulfonyl)hydrazono)methyl) phenyl)(4-vinylbenzyl)carbamate 1-m



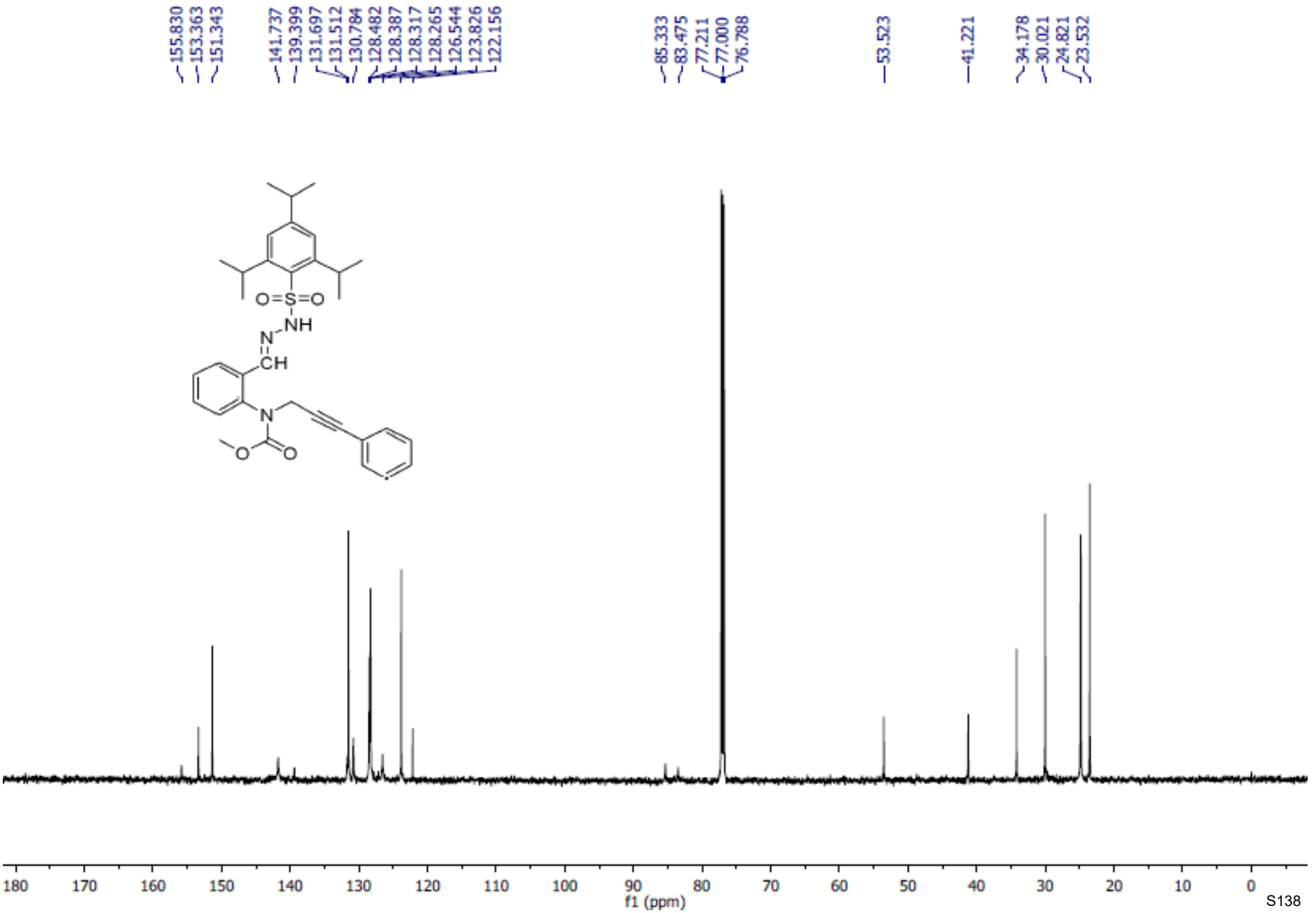
methyl (2-((2-((2,4,6-triisopropylphenyl)sulfonyl)hydrazono)methyl) phenyl)(4-vinylbenzyl)carbamate 1-m



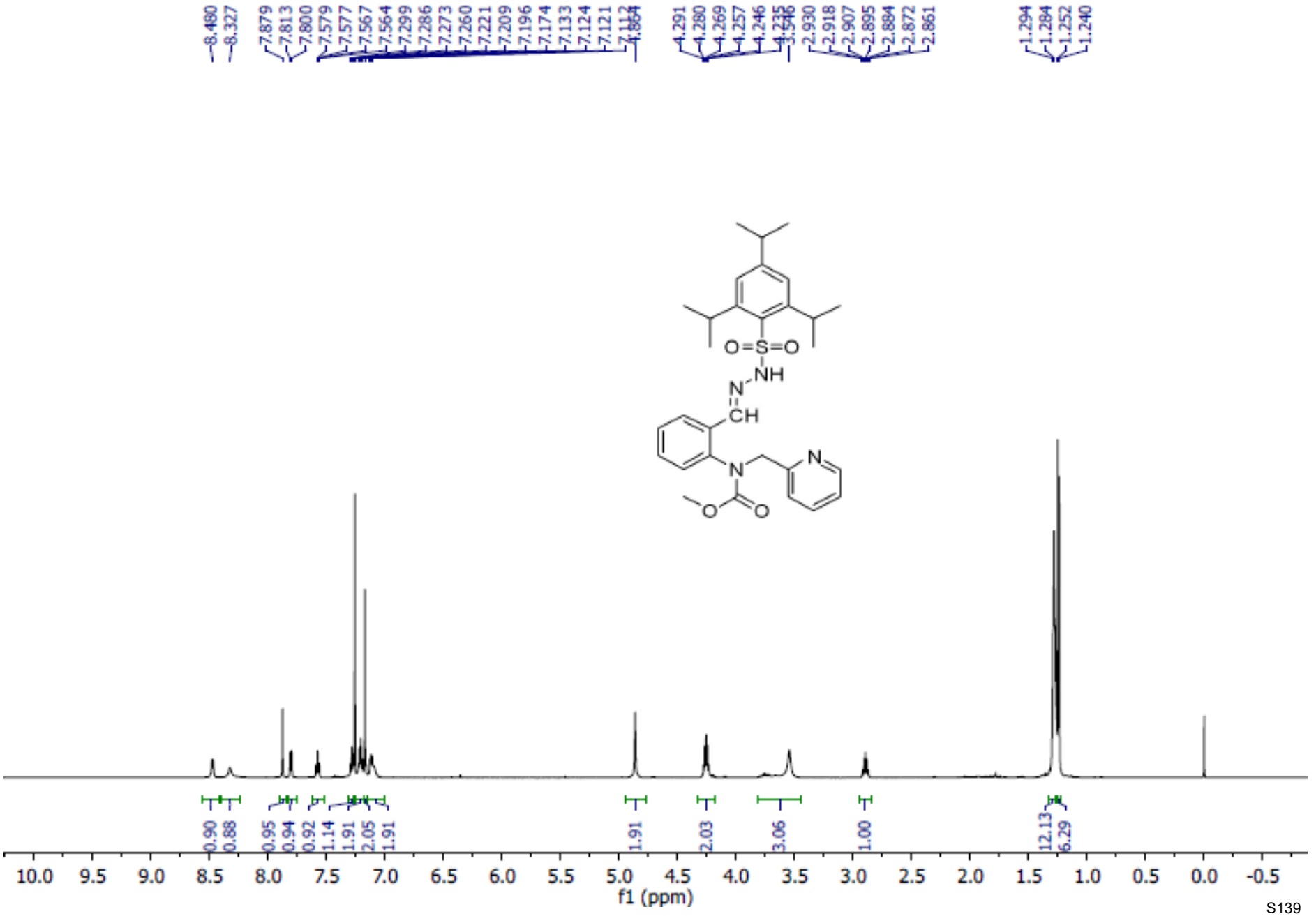
methyl (3-phenylprop-2-yn-1-yl)(2-((2-((2,4,6-triisopropylphenyl) sulfonyl)hydrazono)methyl)phenyl)carbamate 1-n



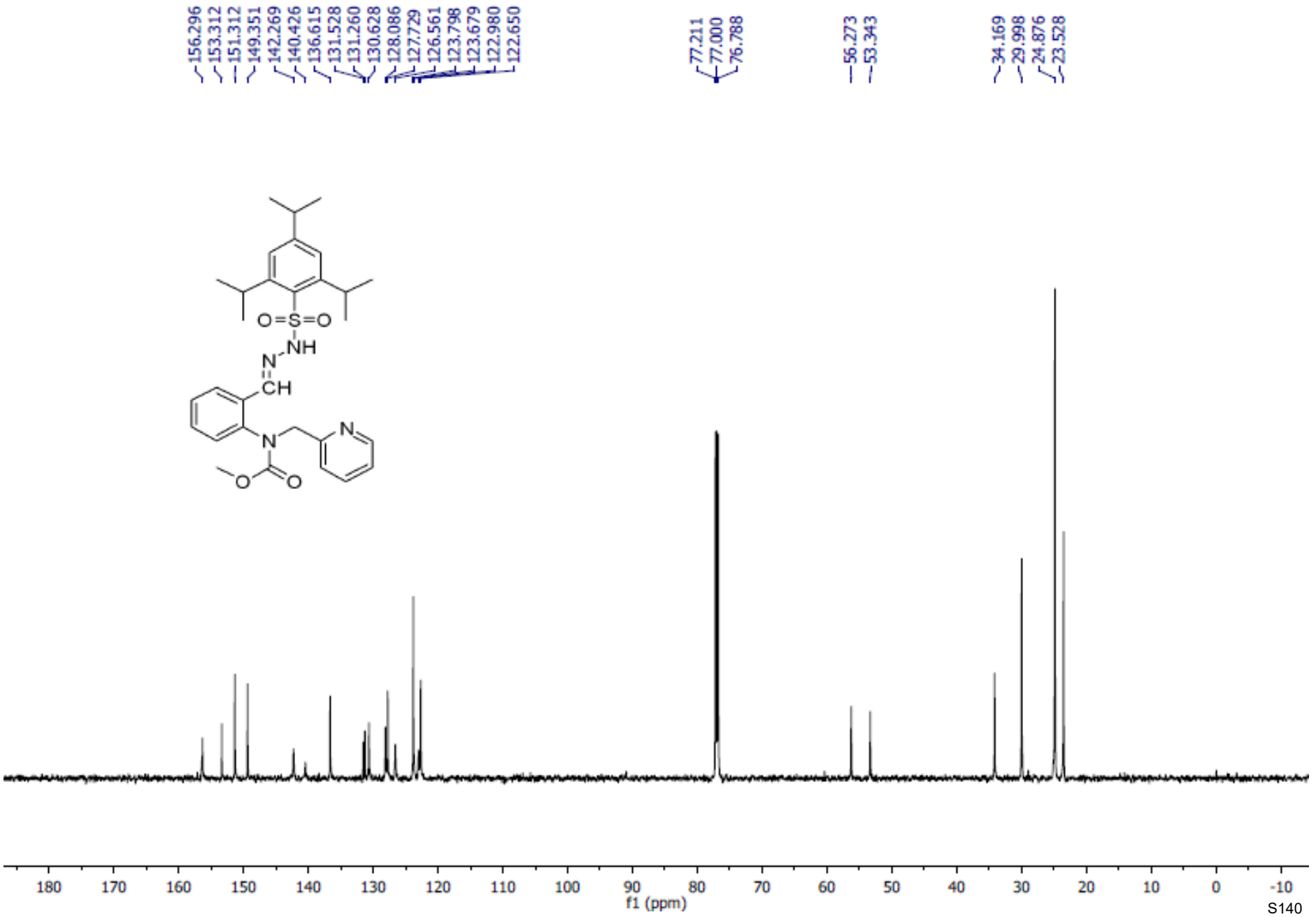
methyl (3-phenylprop-2-yn-1-yl)(2-((2-((2,4,6-triisopropylphenyl) sulfonyl)hydrazono)methyl)phenyl)carbamate 1-n



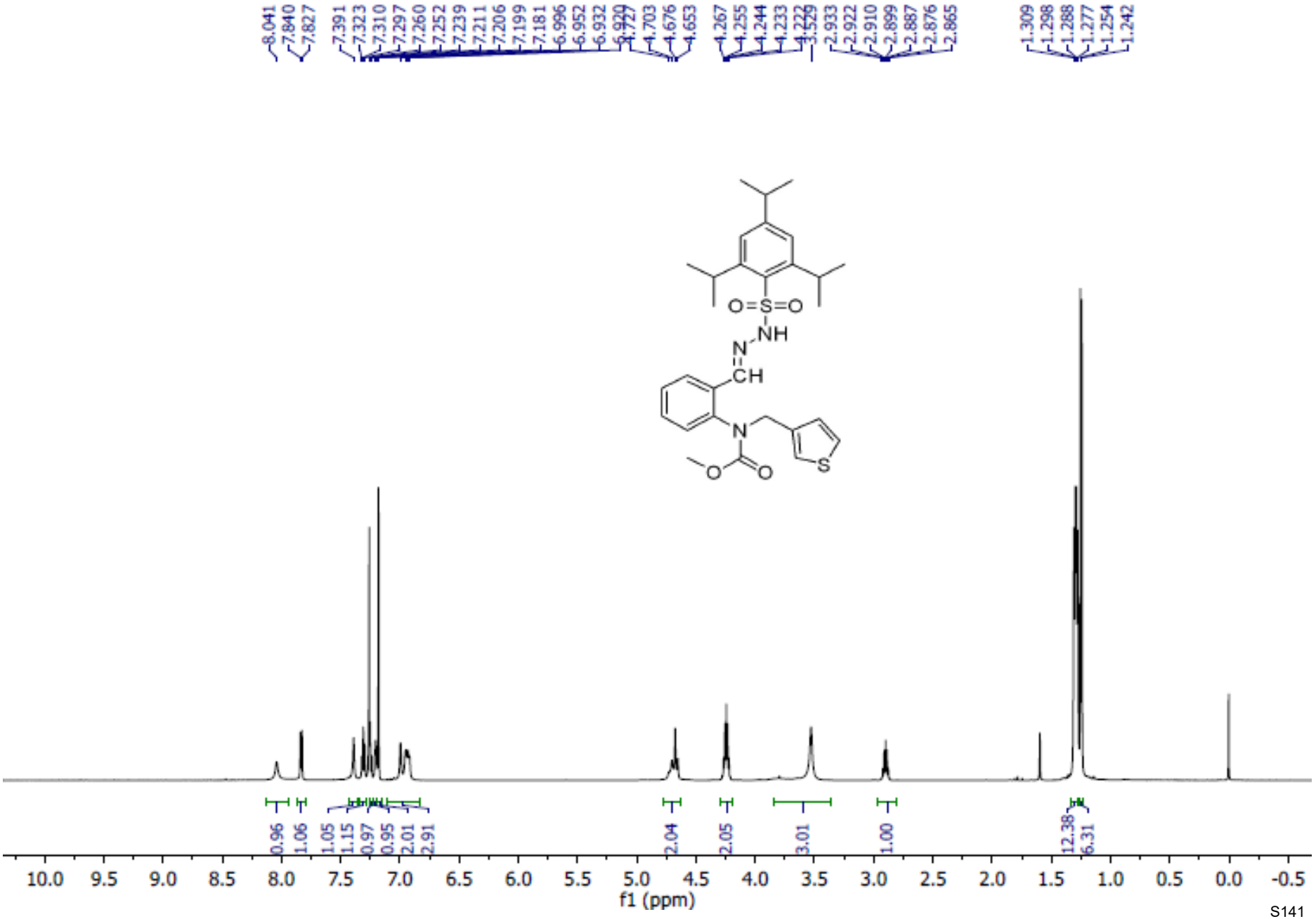
methyl (pyridin-2-ylmethyl)(2-((2-((2,4,6-triisopropylphenyl)sulfonyl) hydrazono)methyl)phenyl)carbamate 1-o



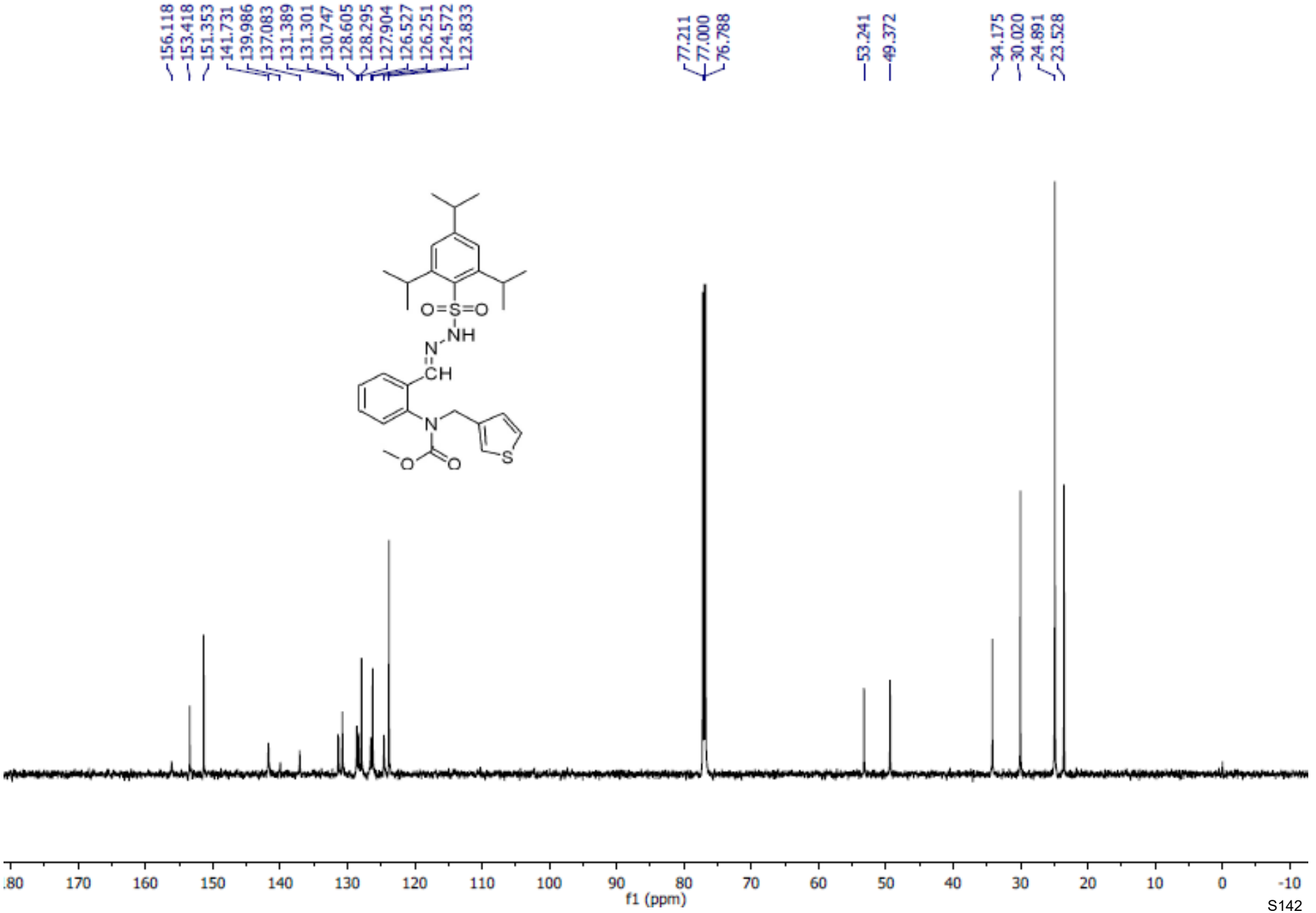
methyl (pyridin-2-ylmethyl)(2-((2-((2,4,6-triisopropylphenyl)sulfonyl) hydrazono)methyl)phenyl)carbamate 1-o



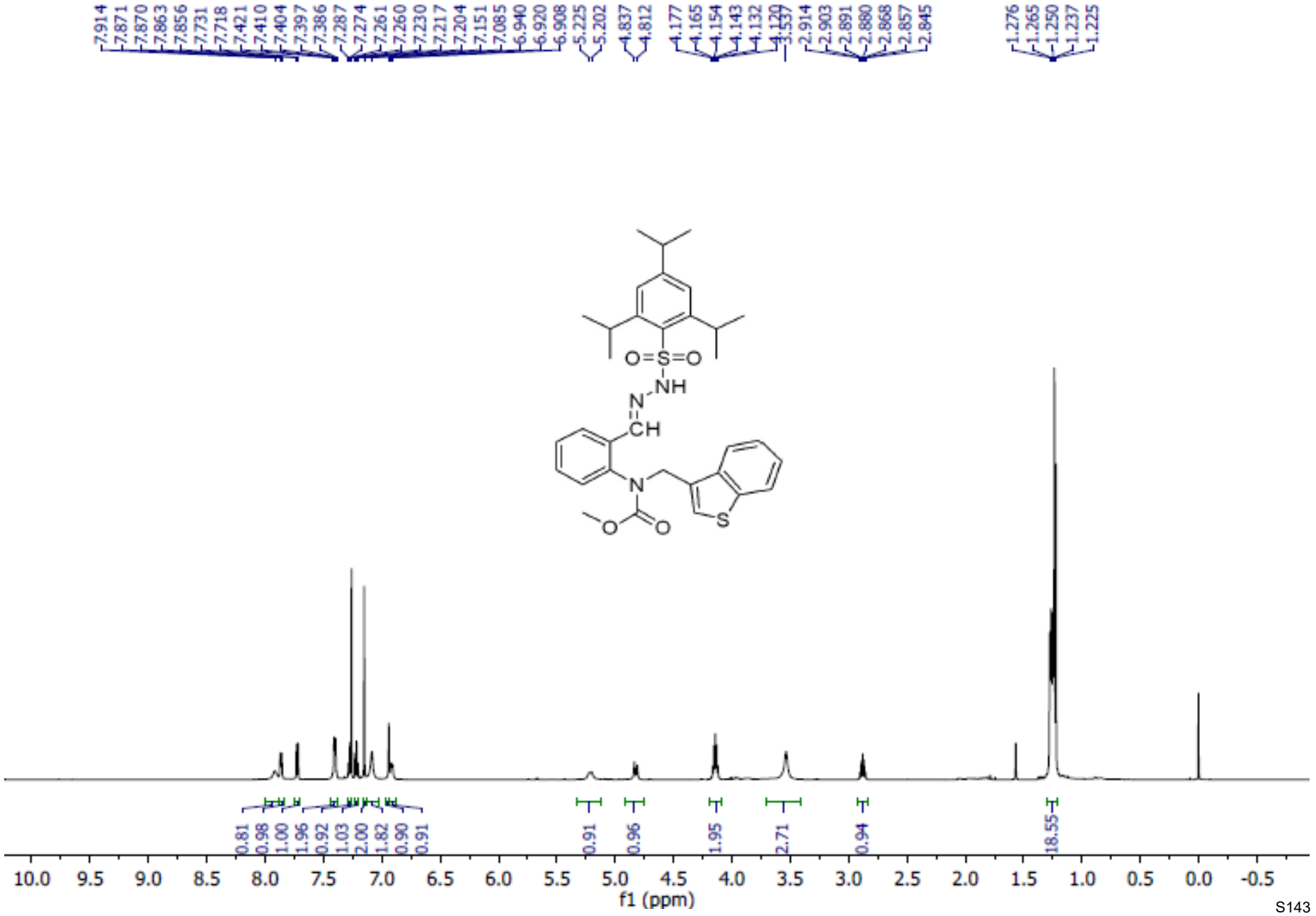
methyl (thiophen-3-ylmethyl)(2-((2-((2,4,6-triisopropylphenyl)sulfonyl) hydrazono)methyl)phenyl)carbamate 1-p



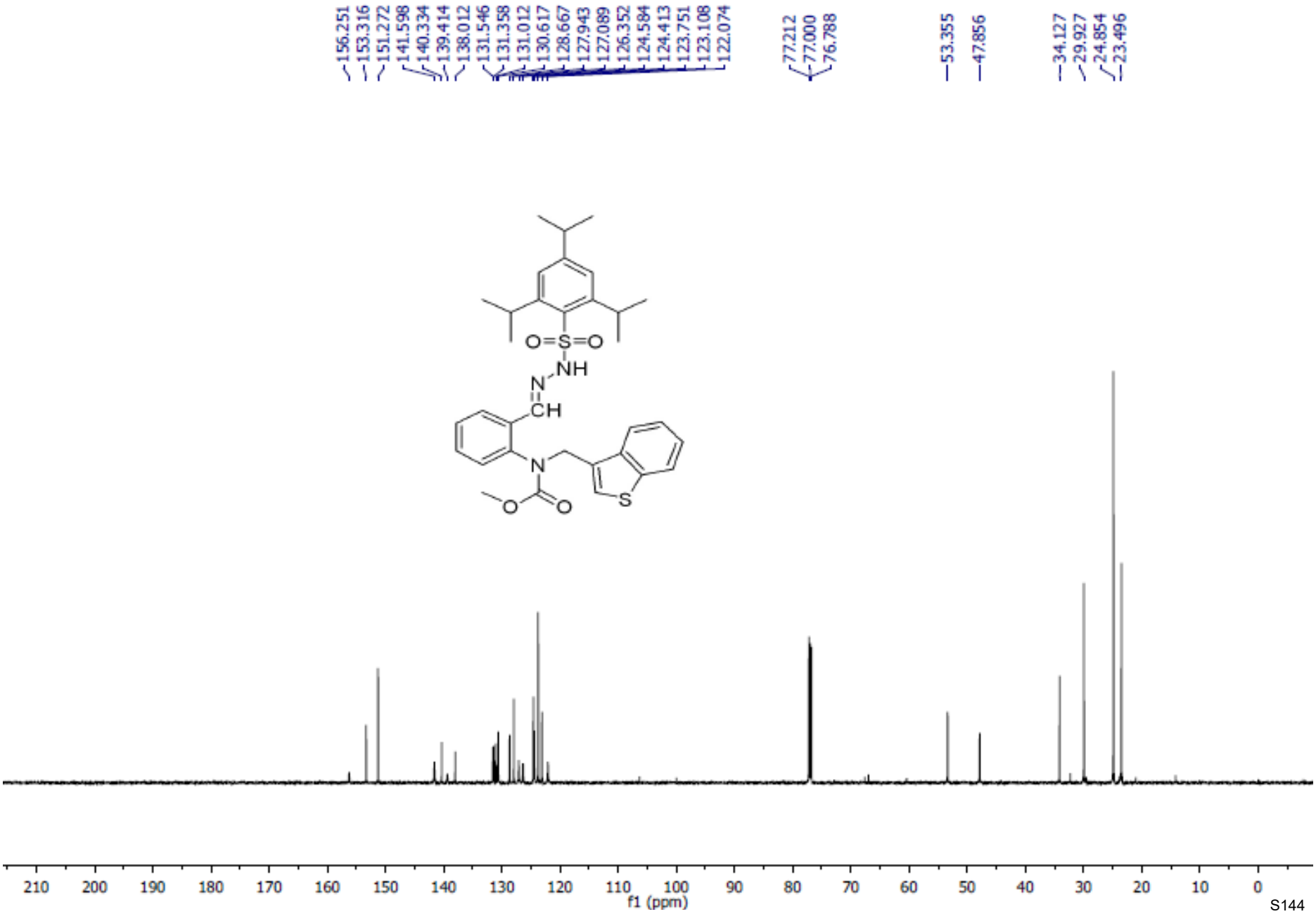
methyl (thiophen-3-ylmethyl)(2-((2-((2,4,6-triisopropylphenyl)sulfonyl) hydrazono)methyl)phenyl)carbamate 1-p



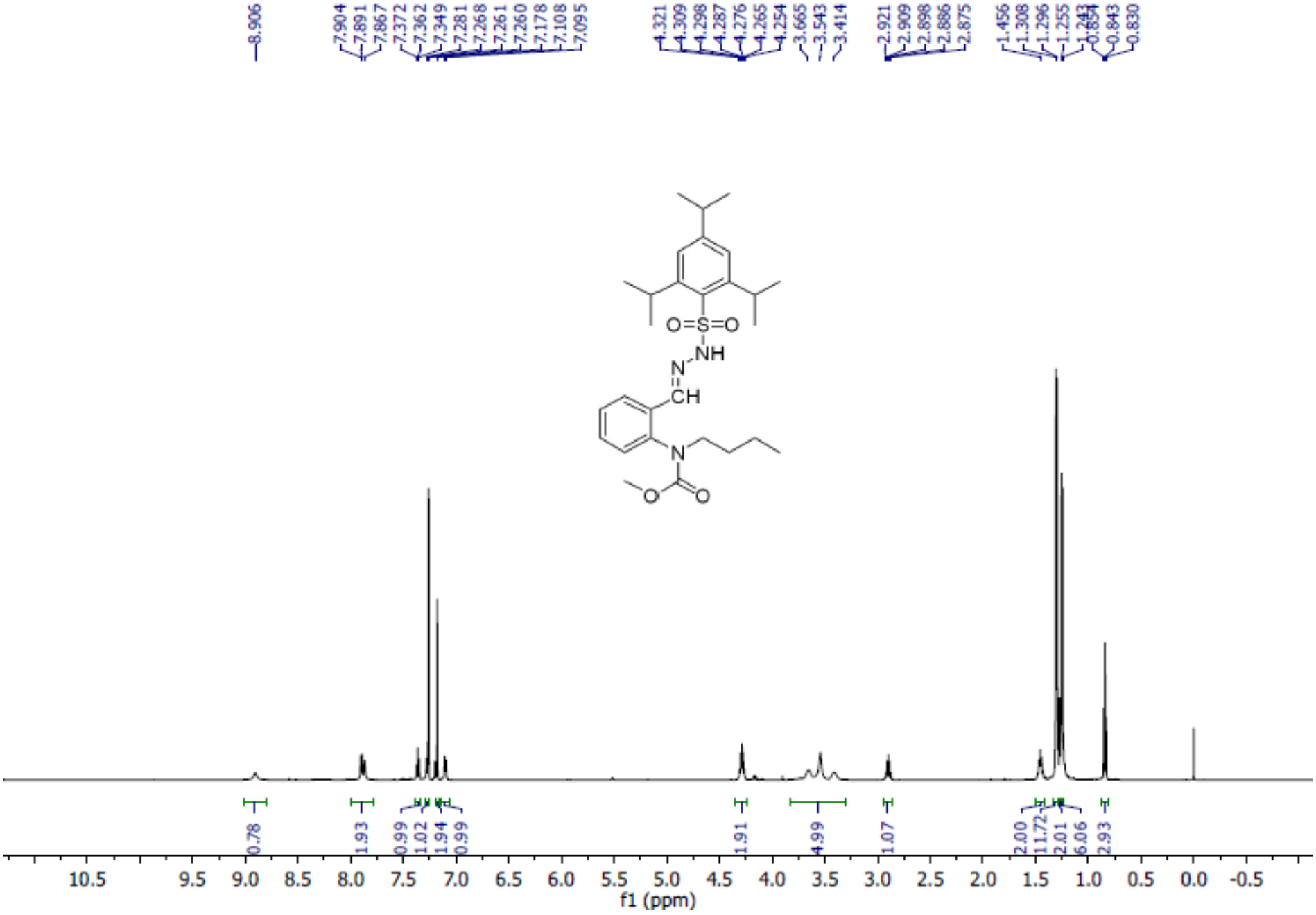
methyl (benzo[*b*]thiophen-3-ylmethyl)(2-((2-((2,4,6-triisopropyl phenyl) sulfonyl)hydrazono)methyl)phenyl)carbamate 1-q



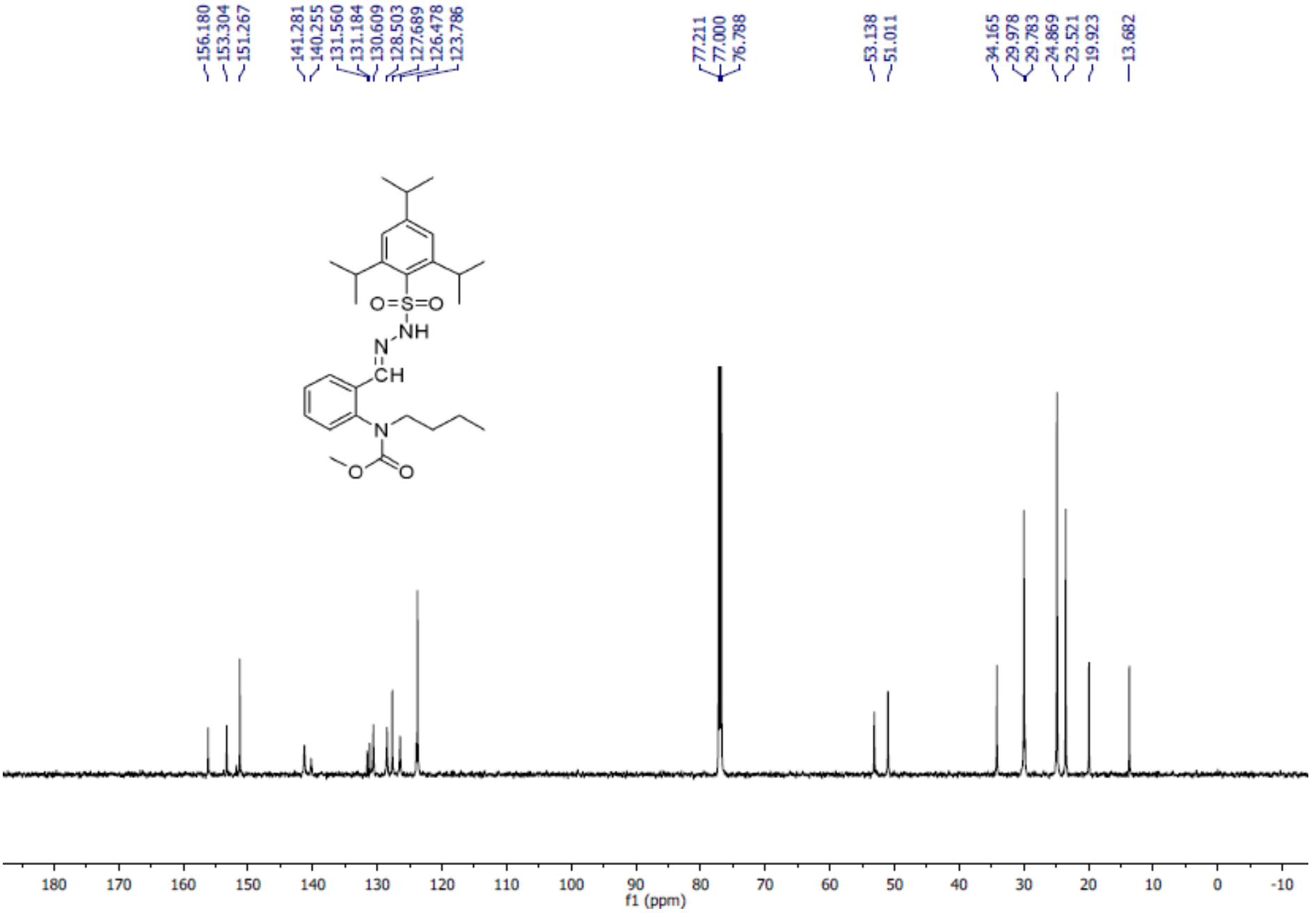
methyl (benzo[*b*]thiophen-3-ylmethyl)(2-((2-((2,4,6-triisopropyl phenyl) sulfonyl)hydrazono)methyl)phenyl)carbamate 1-q



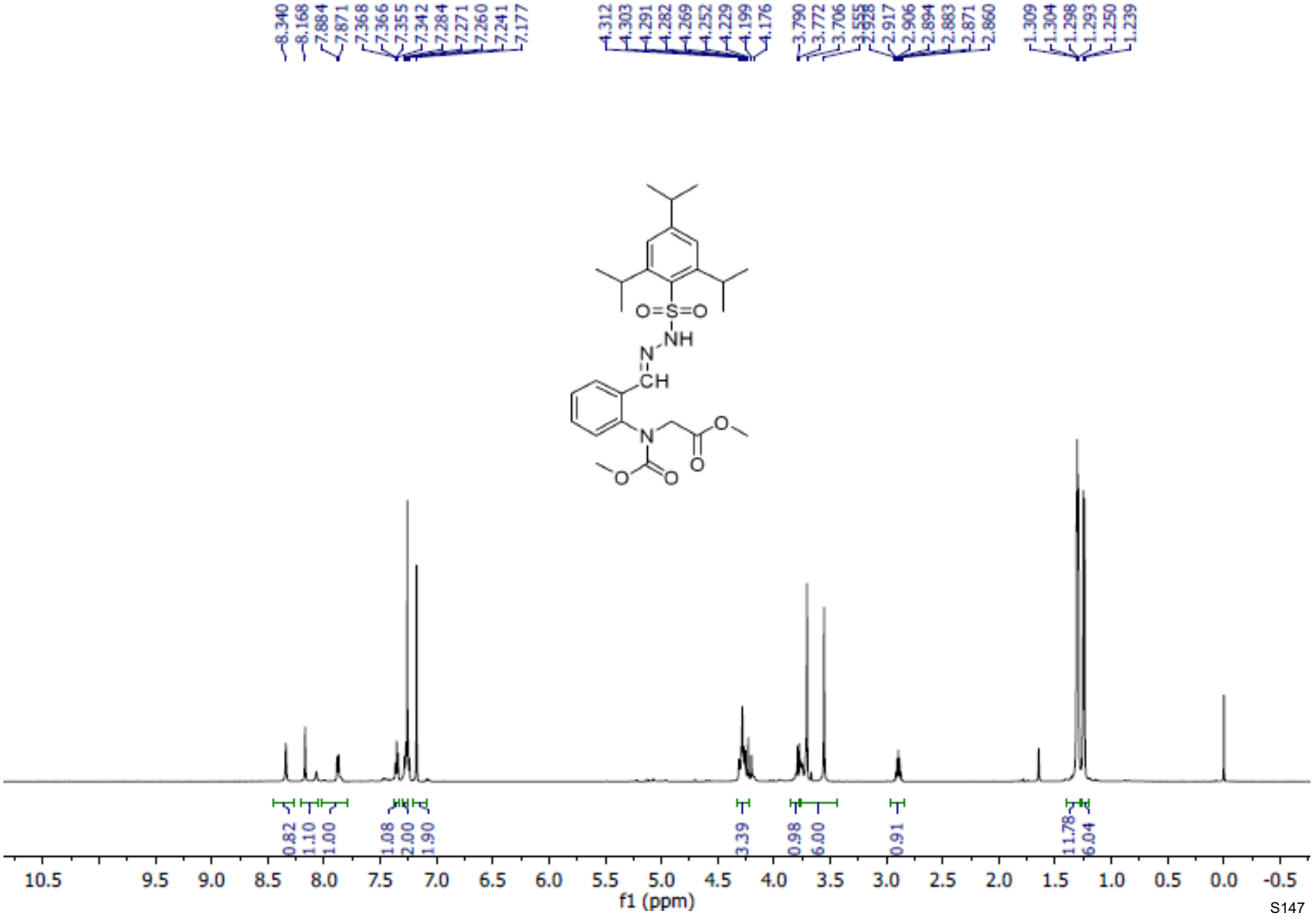
methyl butyl(2-((2-((2,4,6-triisopropylphenyl)sulfonyl)hydrazono)methyl)phenyl)carbamate 1-r



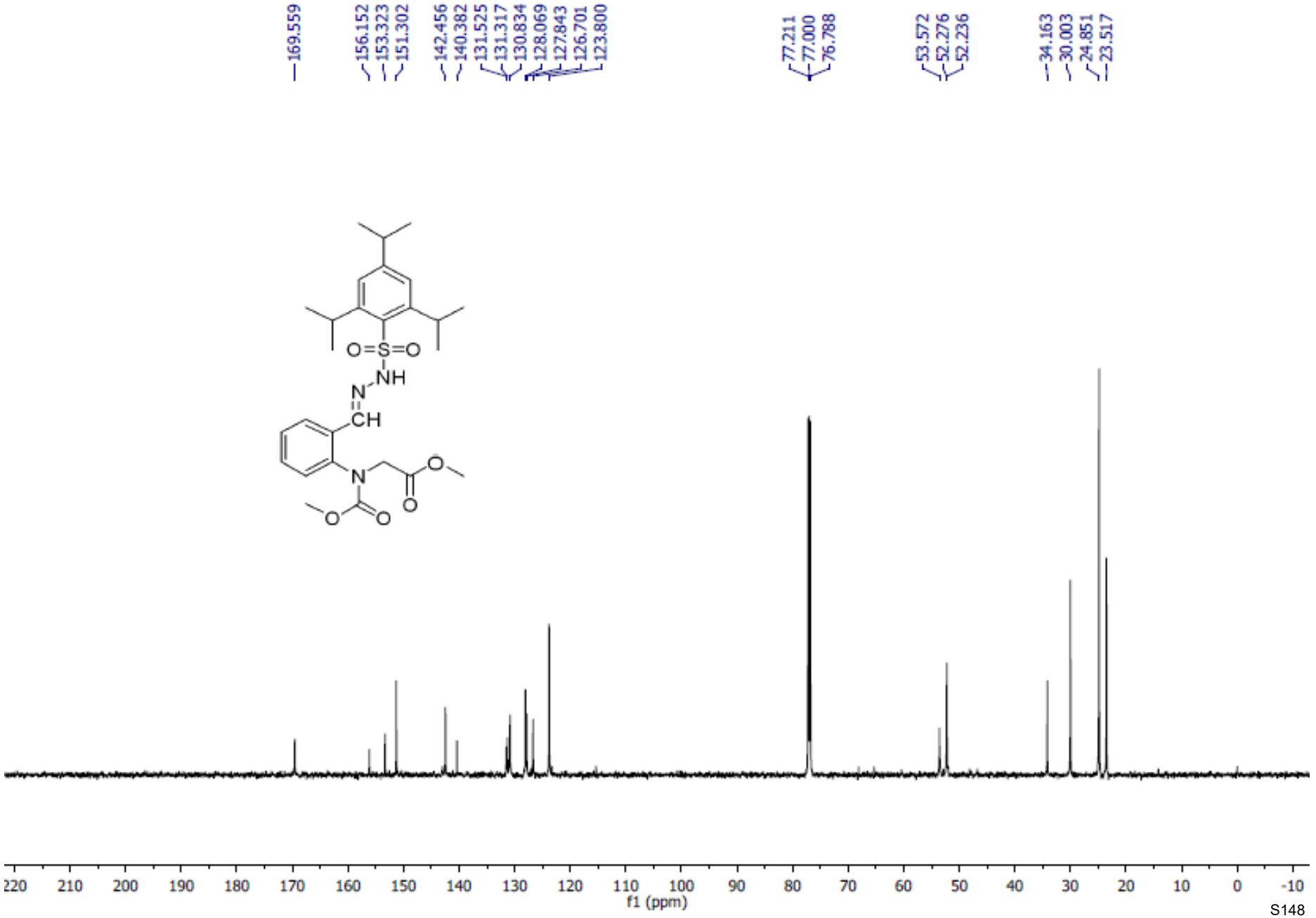
methyl butyl(2-((2-((2,4,6-triisopropylphenyl)sulfonyl)hydrazono)methyl)phenyl)carbamate 1-r



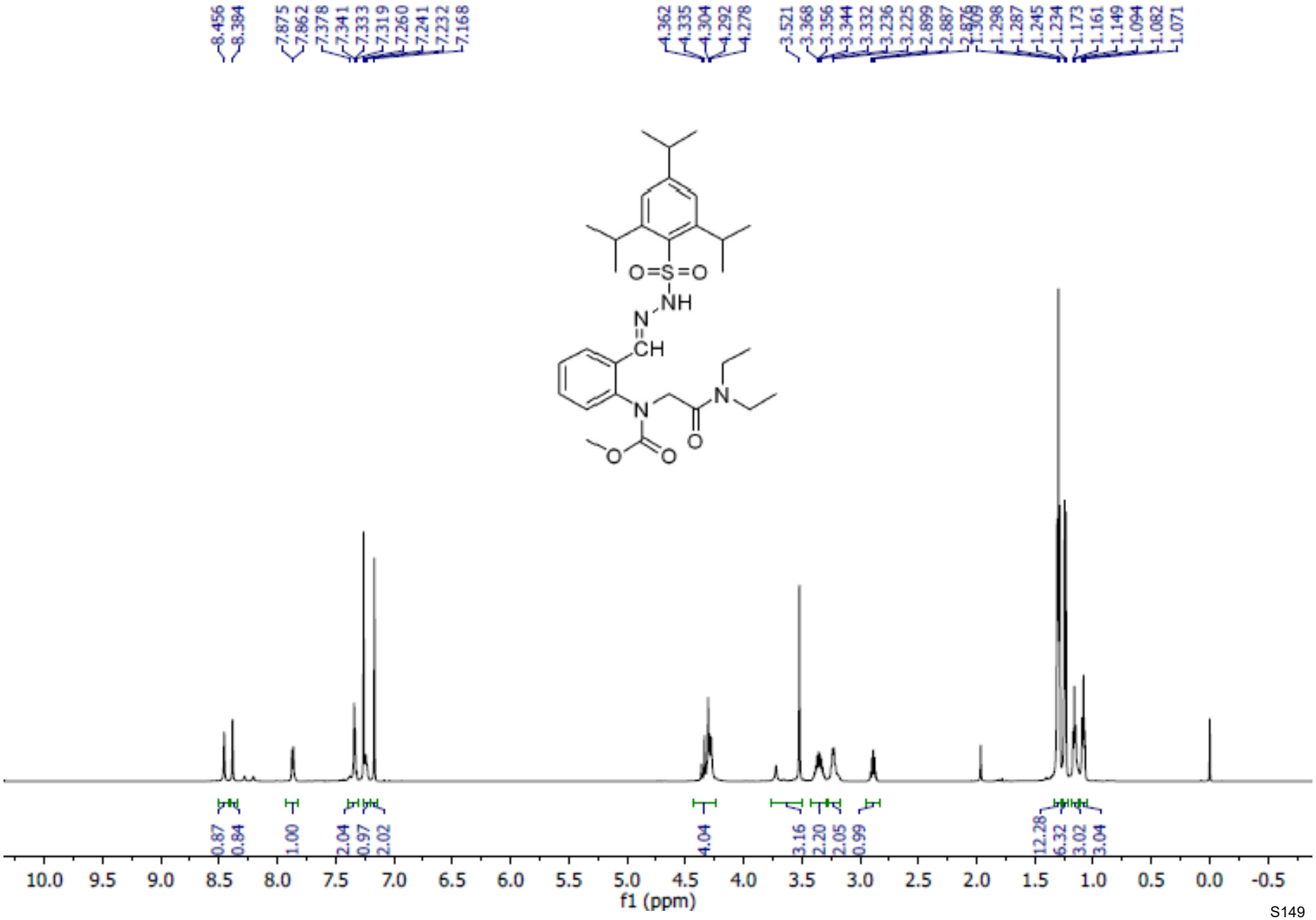
methyl *N*-(methoxycarbonyl)-*N*-(2-((2-((2,4,6-triisopropylphenyl) sulfonyl) hydrazono)methyl)phenyl)glycinate 1-s



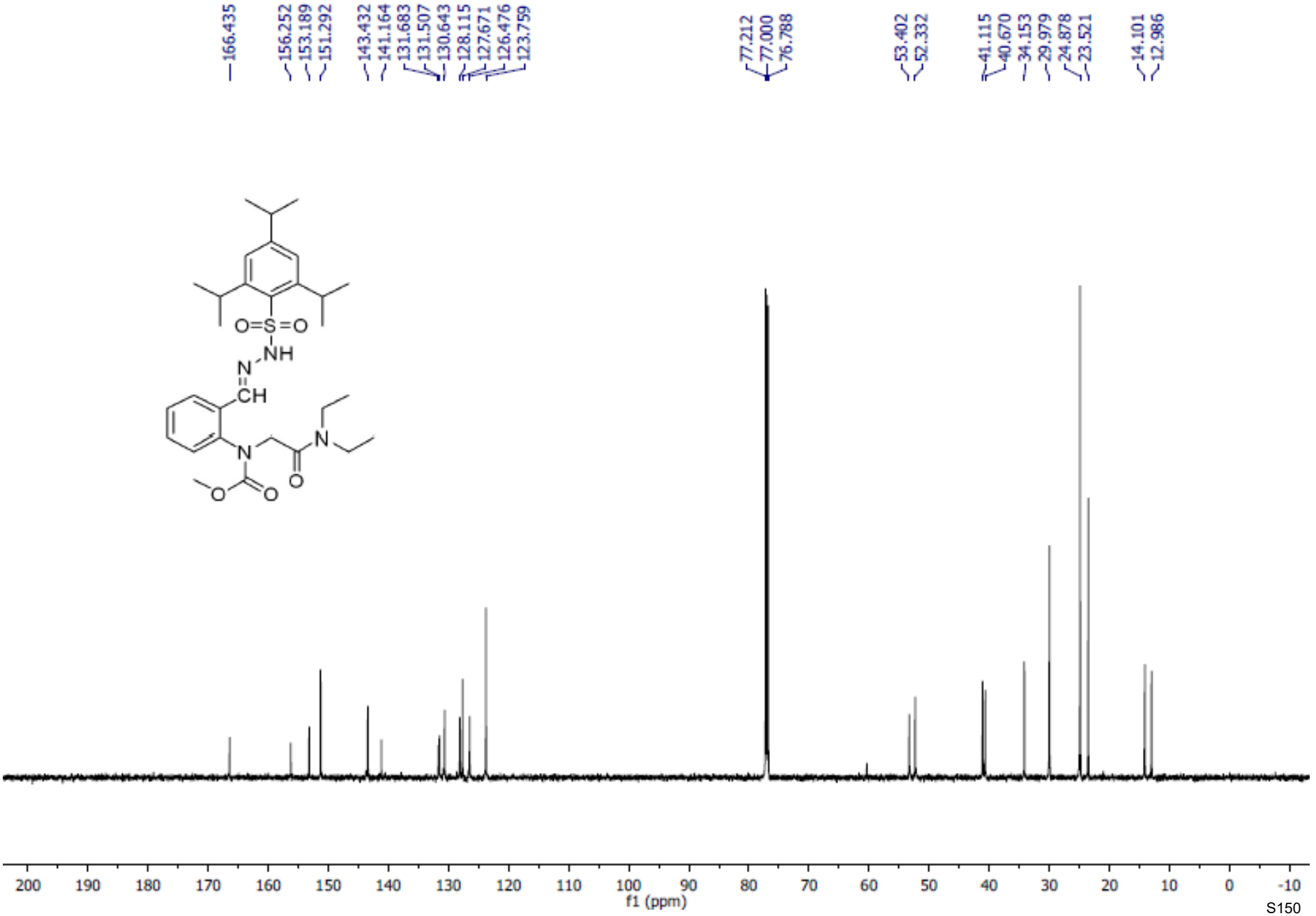
methyl *N*-(methoxycarbonyl)-*N*-(2-((2-((2,4,6-triisopropylphenyl) sulfonyl) hydrazono)methyl)phenyl)glycinate 1-s



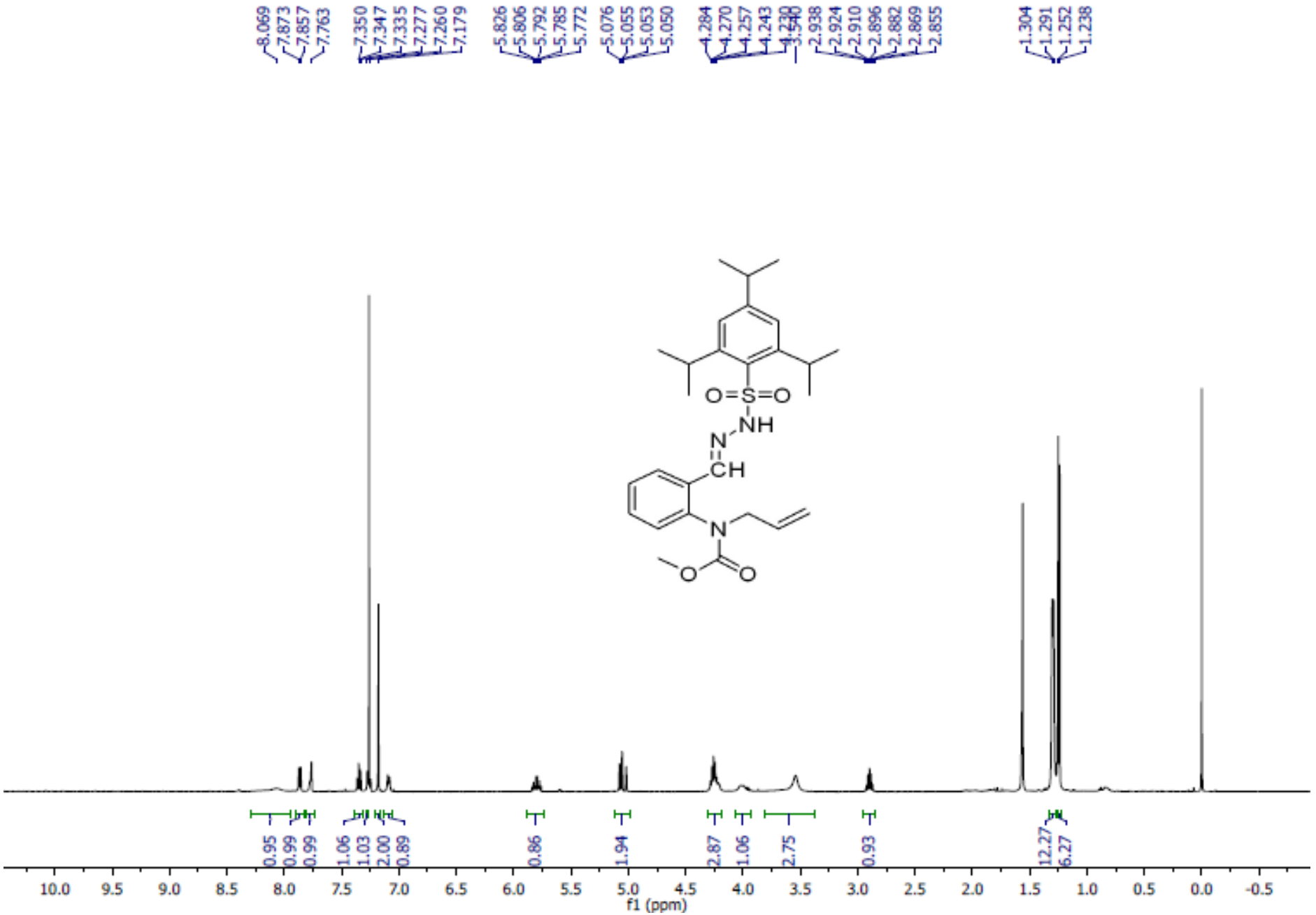
methyl (2-(diethylamino)-2-oxoethyl)(2-((2-((2,4,6-triisopropylphenyl) sulfonyl)hydrazono)methyl)phenyl)carbamate 1-t



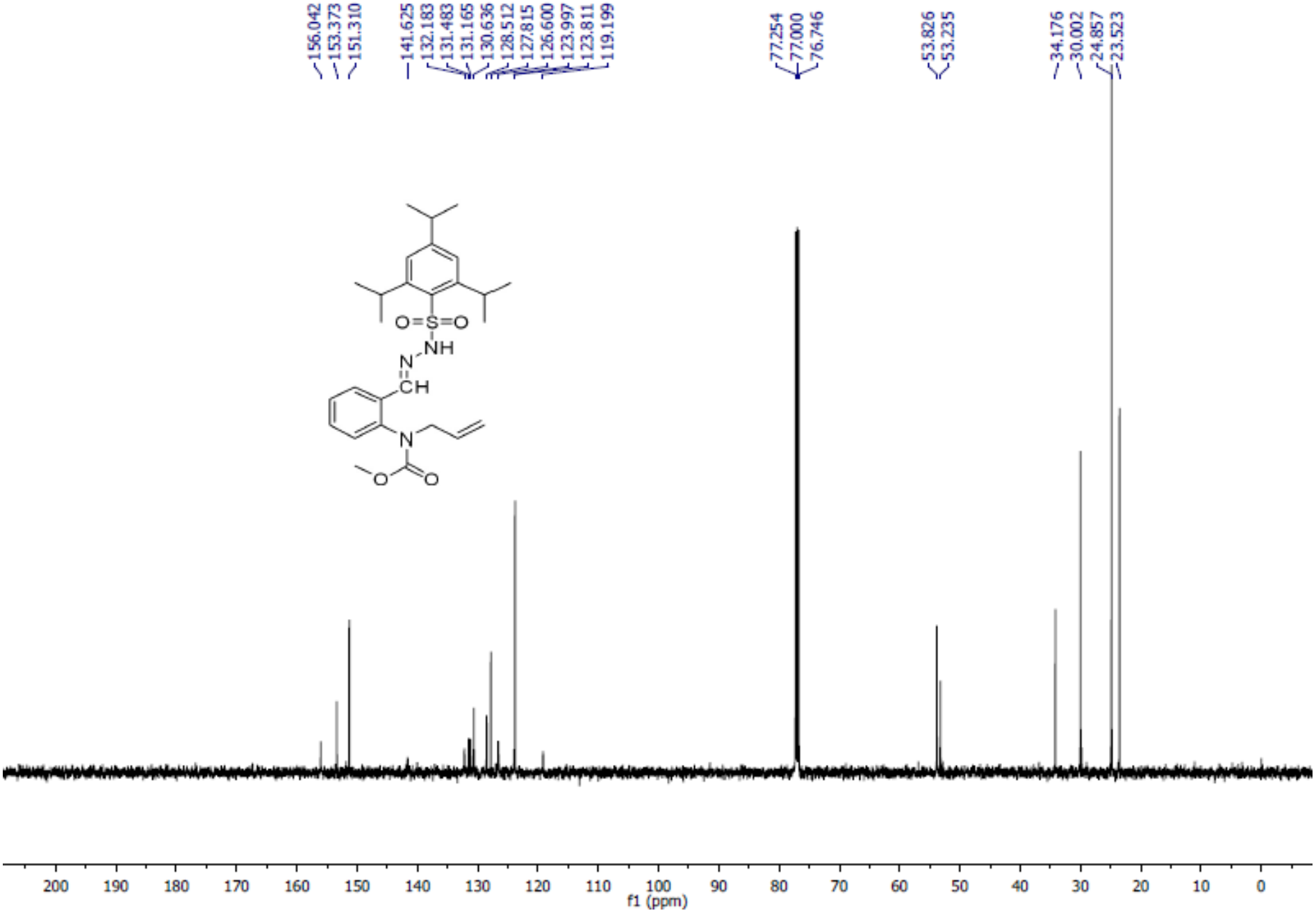
methyl (2-(diethylamino)-2-oxoethyl)(2-((2-((2,4,6-triisopropylphenyl) sulfonyl)hydrazono)methyl)phenyl)carbamate 1-t



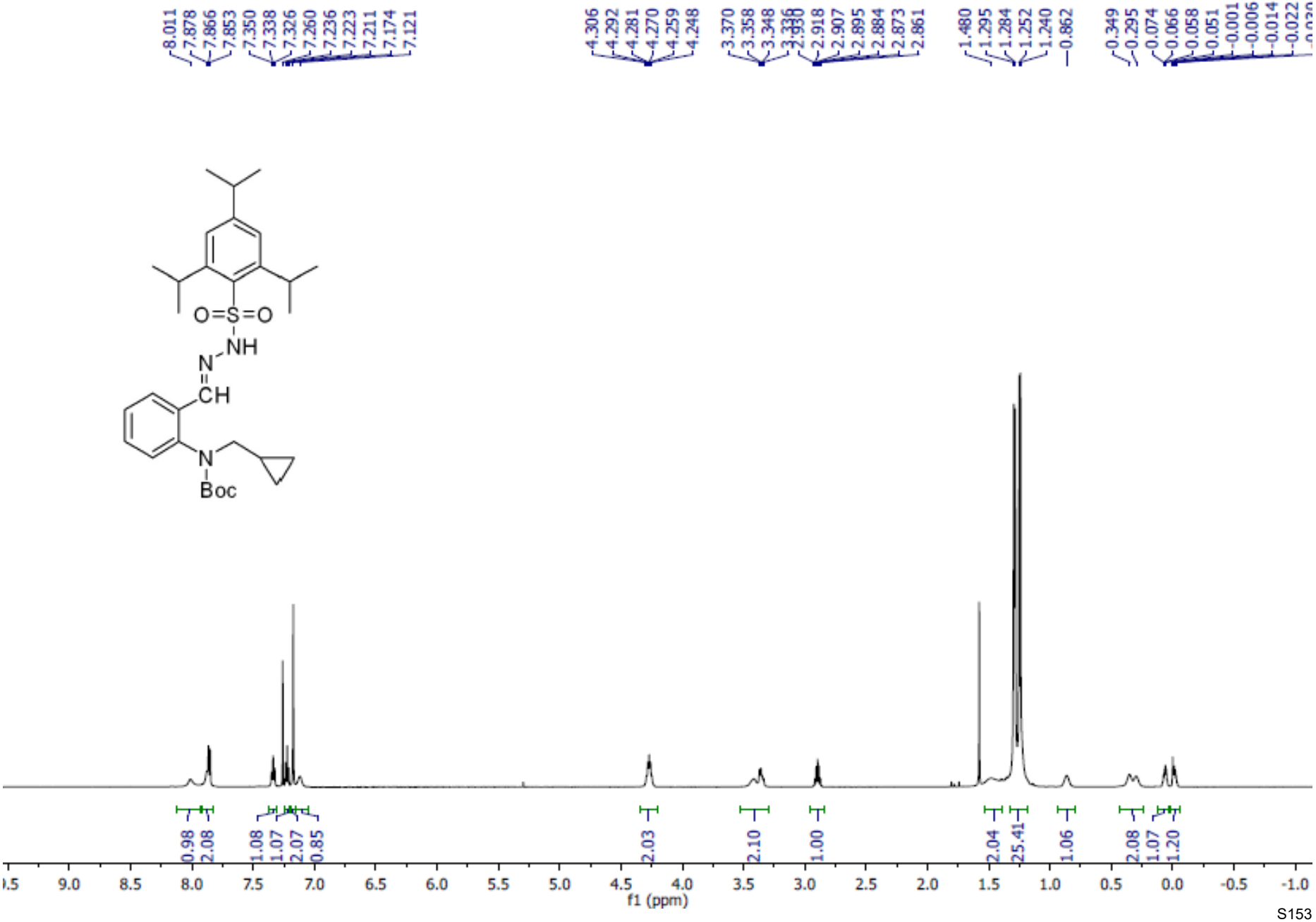
methyl allyl(2-((2-((2,4,6-triisopropylphenyl)sulfonyl)hydrazono)methyl)phenyl)carbamate 1-u



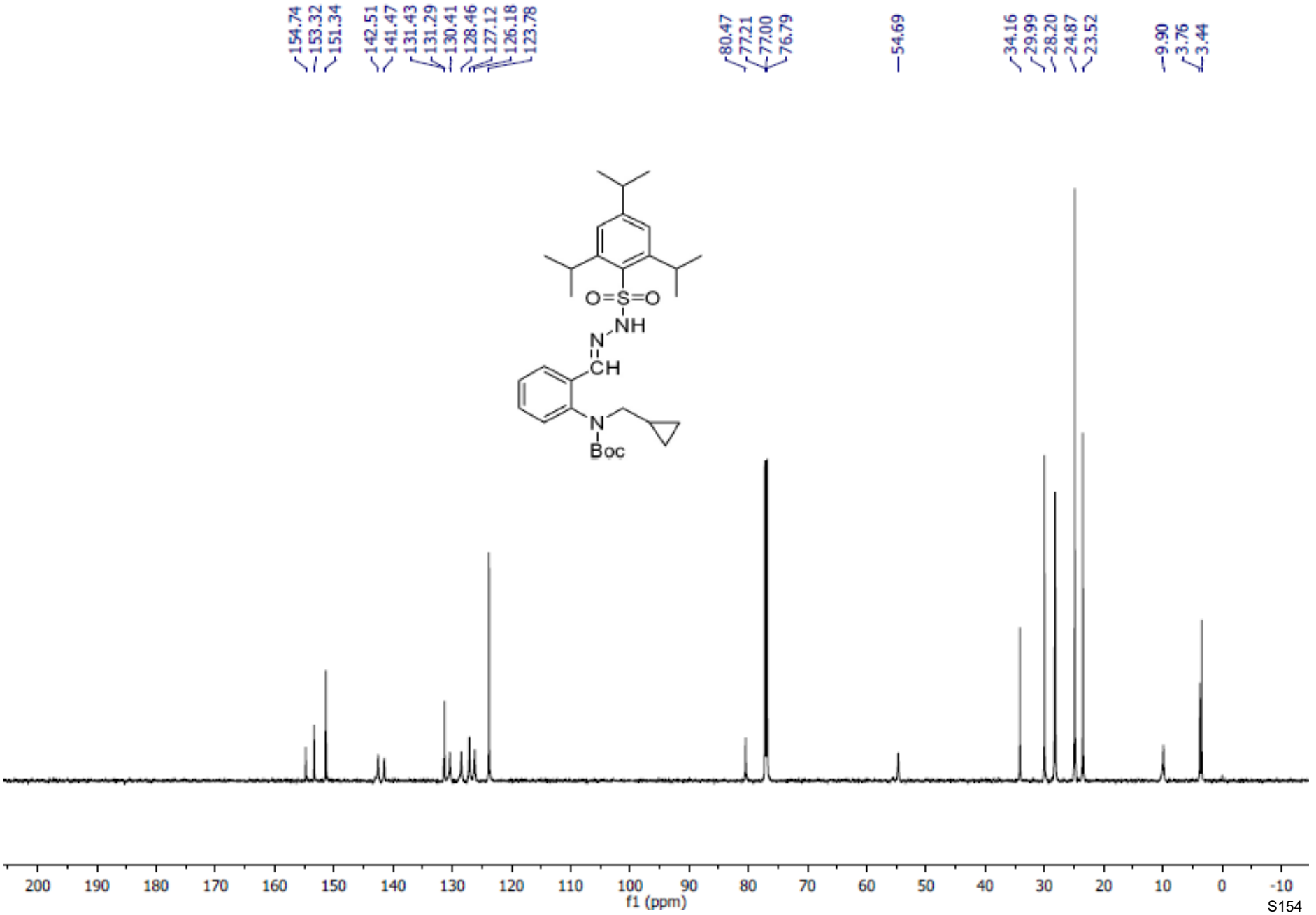
methyl allyl(2-((2-((2,4,6-triisopropylphenyl)sulfonyl)hydrazono)methyl)phenyl)carbamate 1-u



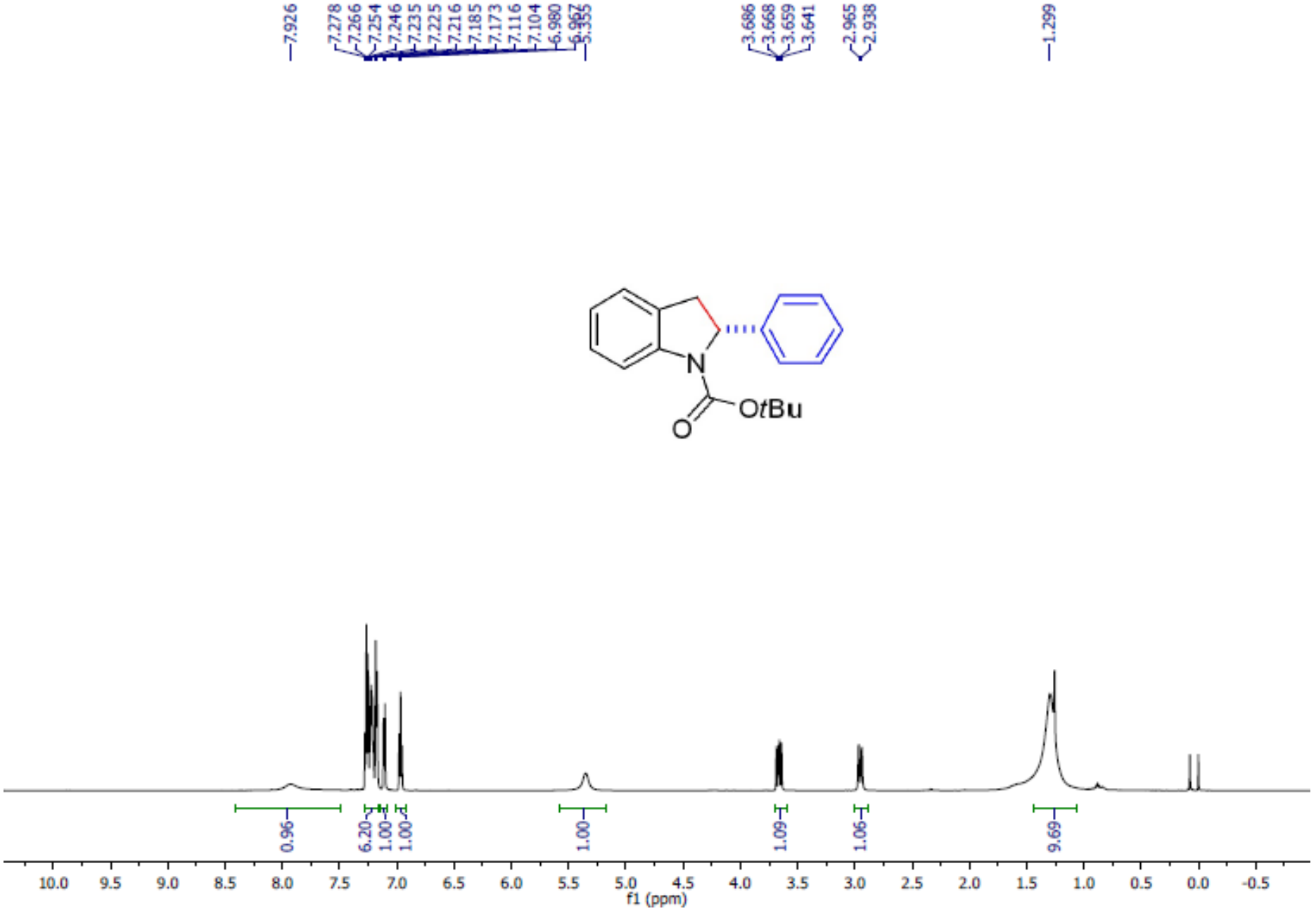
tert-butyl (cyclopropylmethyl)(2-((2-((2,4,6-triisopropylphenyl)sulfonyl) hydrazono)methyl)phenyl)carbamate 1-v



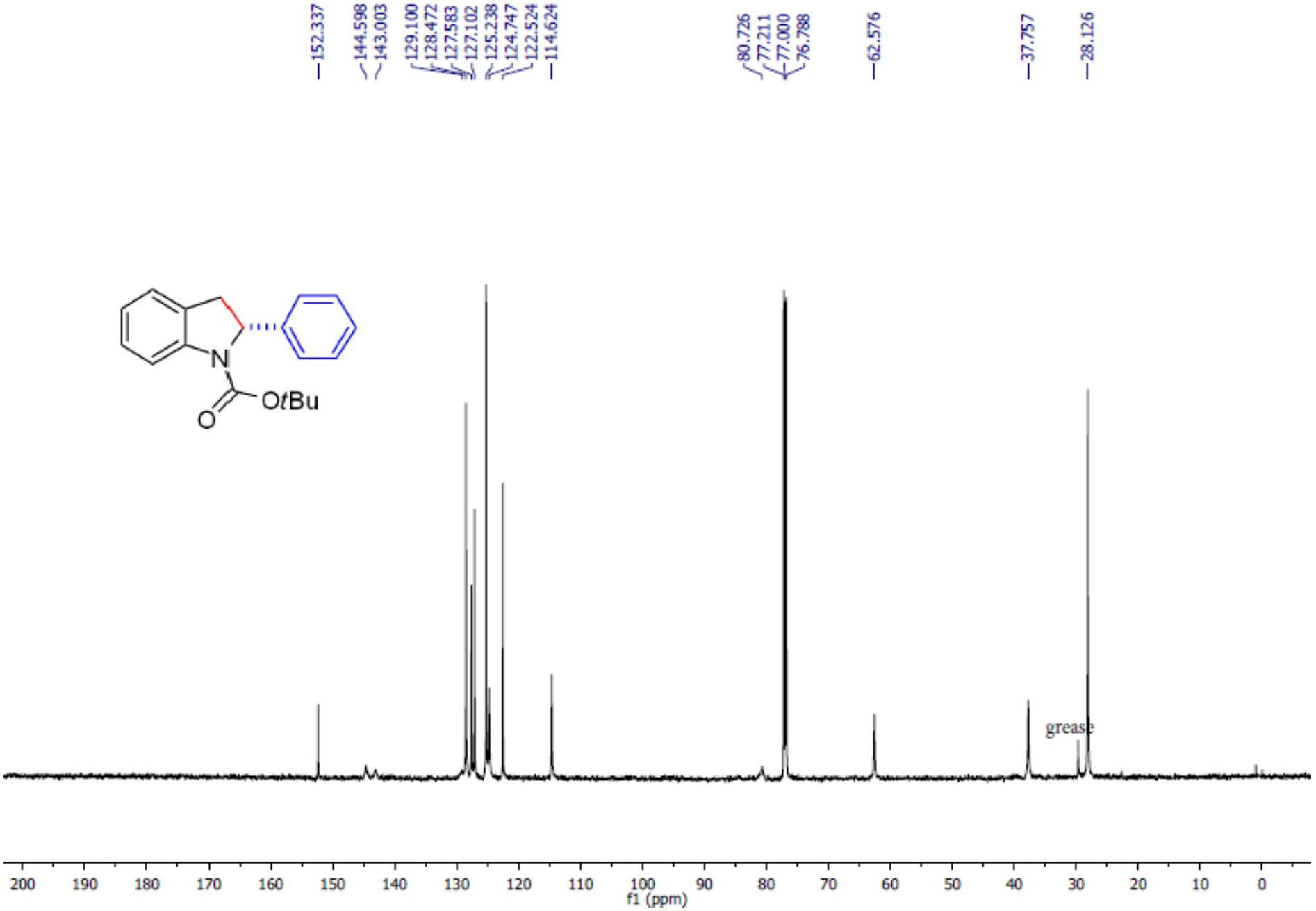
tert-butyl (cyclopropylmethyl)(2-((2-((2,4,6-triisopropylphenyl)sulfonyl) hydrazono)methyl)phenyl)carbamate 1-v



tert-butyl (*R*)-2-phenylindoline-1-carboxylate 2a



tert-butyl (*R*)-2-phenylindoline-1-carboxylate 2a

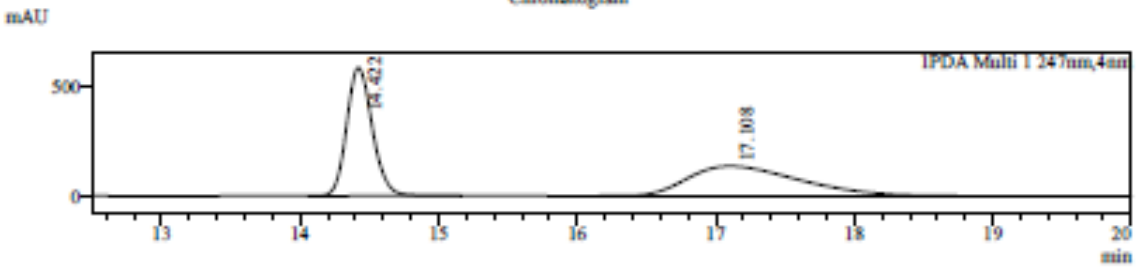


tert-butyl (*R*)-2-phenylindoline-1-carboxylate 2a

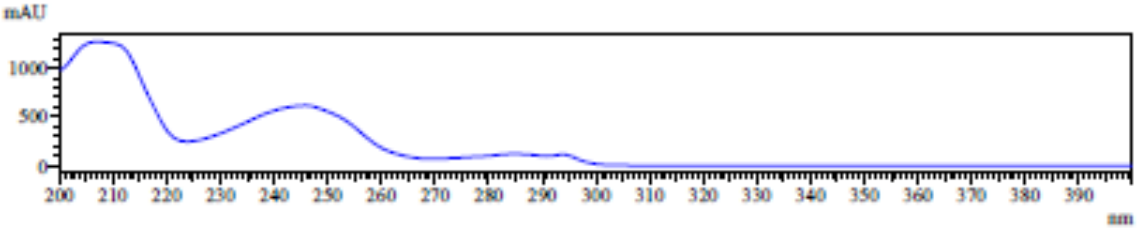
Sample Information

Sample Name	: XW-IV-8-IA-0.3%0.8mL-1
Sample ID	: XW-IV-8-IA-0.3%0.8mL-1
Data File	: XW-IV-8-IA-0.3%0.8mL-1.lcd
Method File	: XW-0.3%-0.8ml.lcm

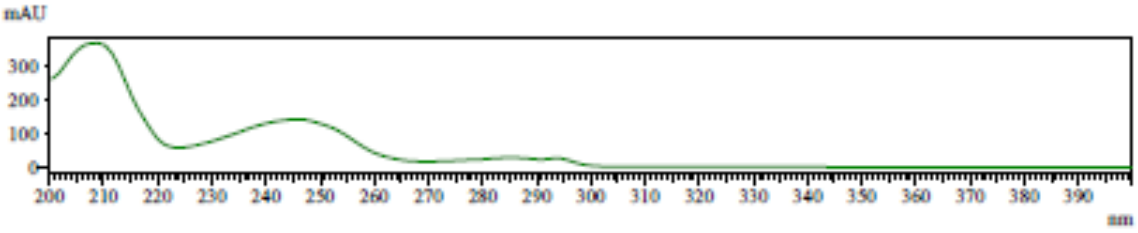
Chromatogram



UV Spectrum
Retention time = 14.422



UV Spectrum
Retention time = 17.108



Peak Table

PDA Ch1 247nm

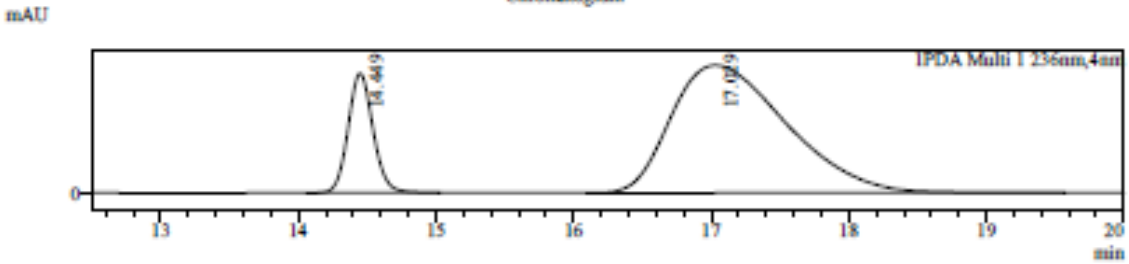
Peak#	Ret. Time	Area	Area%
1	14.422	7635869	49.725
2	17.108	7720233	50.275
Total		15356102	100.000

tert-butyl (*R*)-2-phenylindoline-1-carboxylate 2a

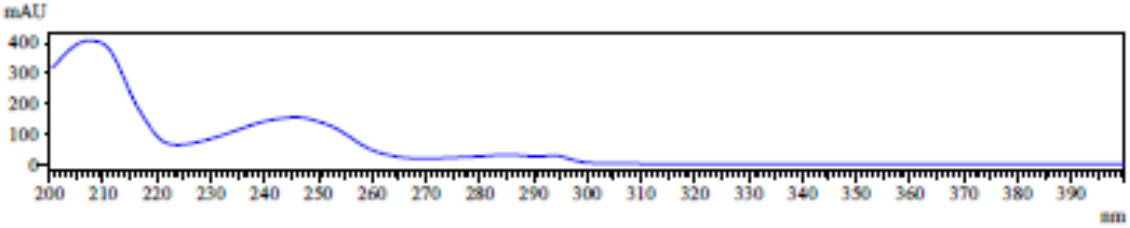
Sample Information

Sample Name	: XW-IV-18-IA-0.3%0.8mL-1
Sample ID	: XW-IV-18-IA-0.3%0.8mL-1
Data File	: XW-IV-18-IA-0.3%0.8mL-1.lcd
Method File	: XW-0.3%-0.8ml.lcm

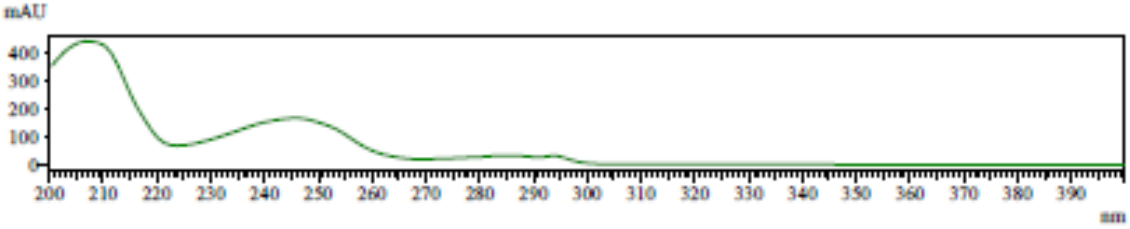
Chromatogram



UV Spectrum
Retention time = 14.449



UV Spectrum
Retention time = 17.029

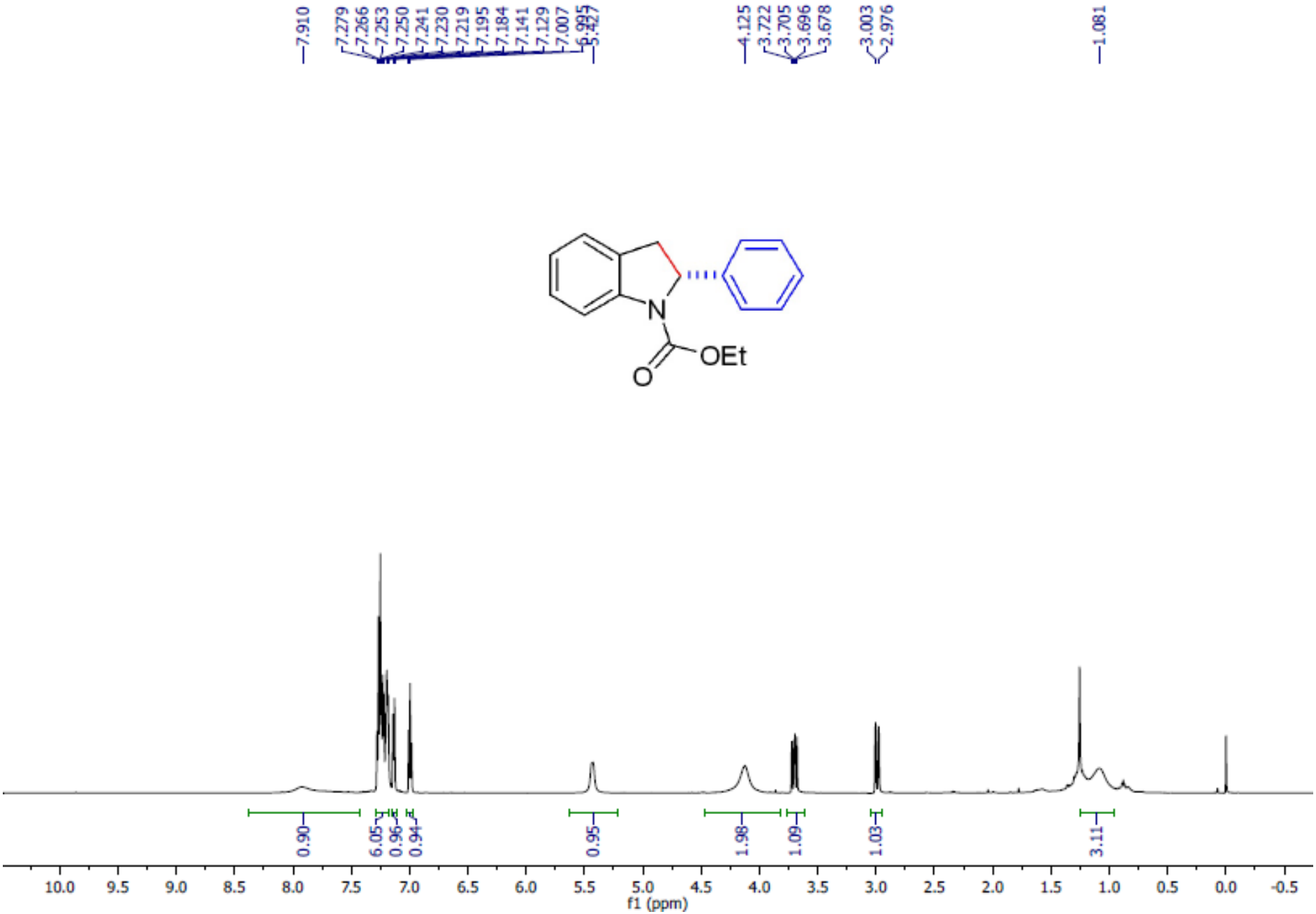


Peak Table

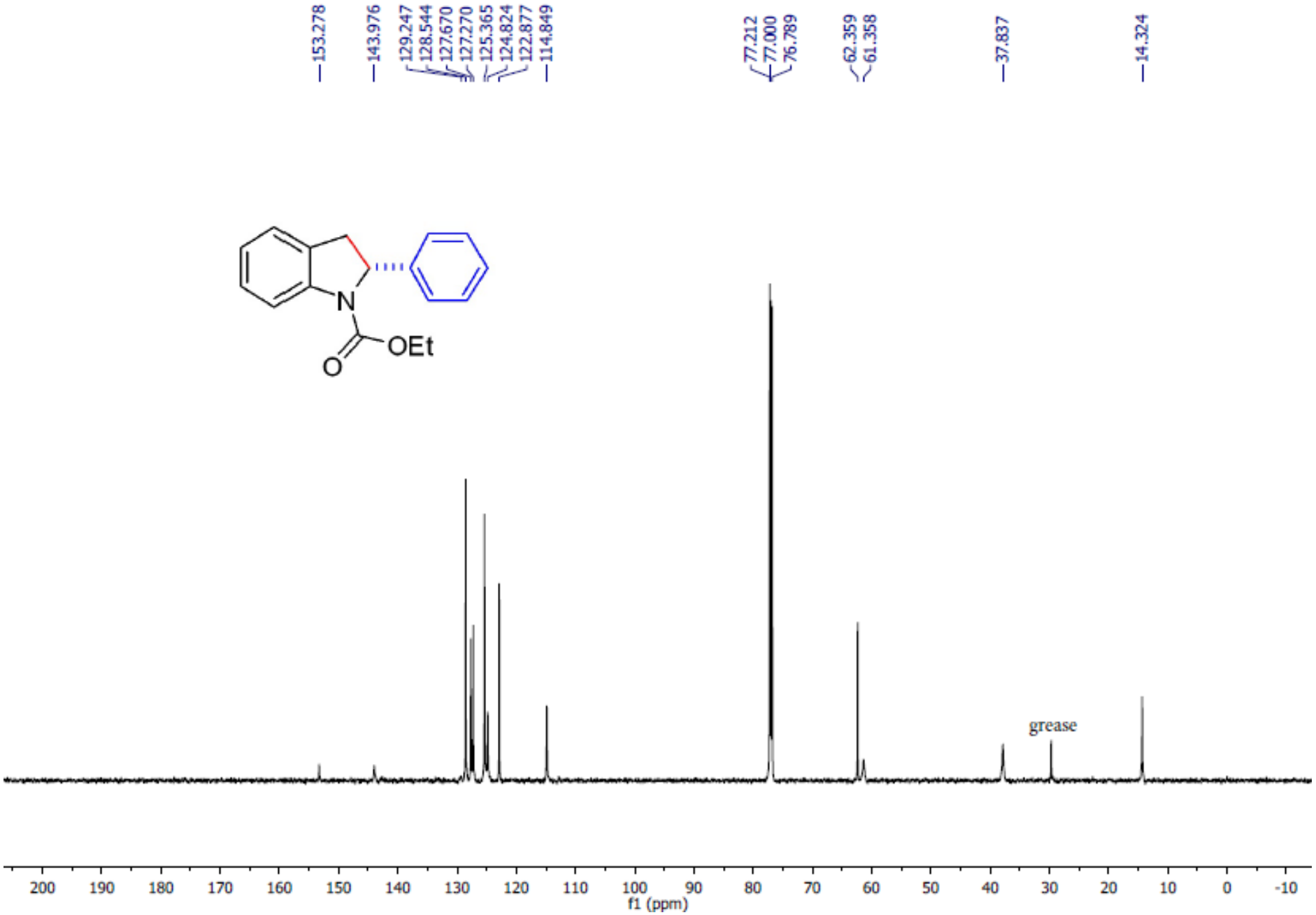
PDA Ch1 236nm

Peak#	Ret. Time	Area	Area%
1	14.449	1506306	16.837
2	17.029	7440359	83.163
Total		8946665	100.000

ethyl (*R*)-2-phenylindoline-1-carboxylate 2b



ethyl (*R*)-2-phenylindoline-1-carboxylate 2b

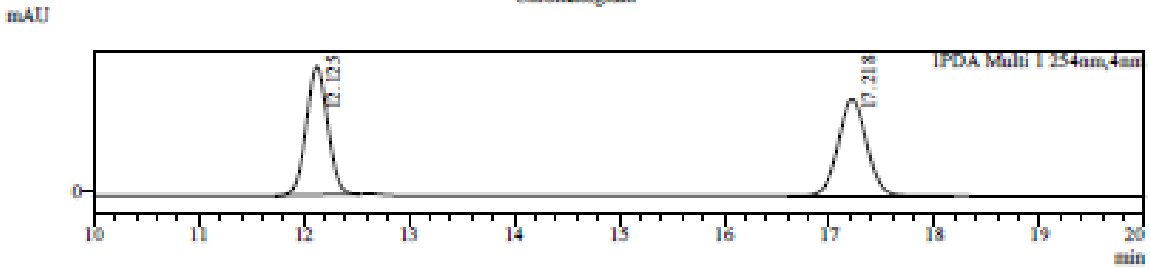


ethyl (*R*)-2-phenylindoline-1-carboxylate 2b

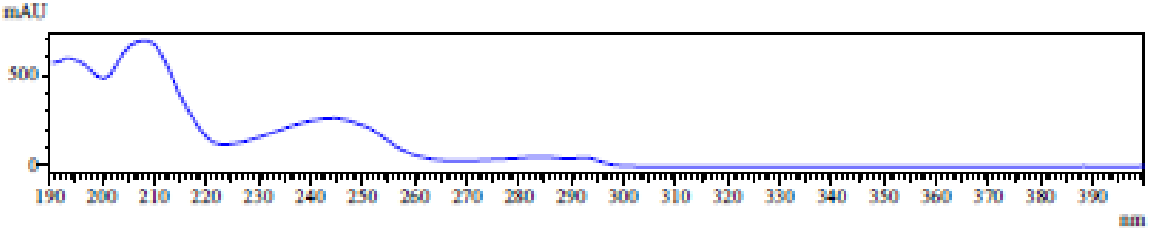
Sample Information

Sample Name	: XW-IV-268-IA-2-1%0.8mL
Sample ID	: XW-IV-268-IA-2-1%0.8mL
Data File	: XW-IV-268-IA-2-1%0.8mL.lcd
Method File	: XW-1%-0.8ml.lcm

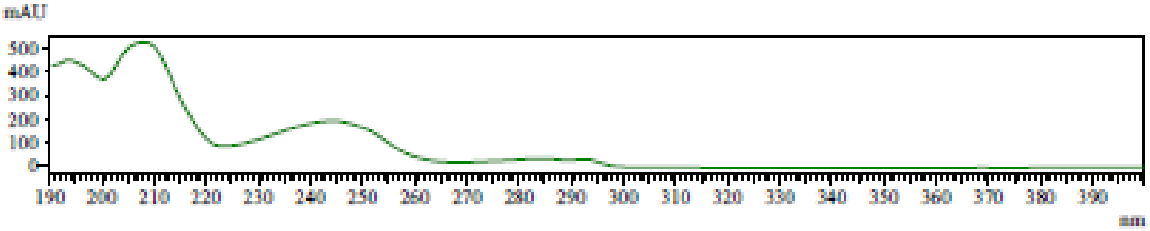
Chromatogram



UV Spectrum
Retention time = 12.125



UV Spectrum
Retention time = 17.218



Peak Table

PDA Ch1 254nm

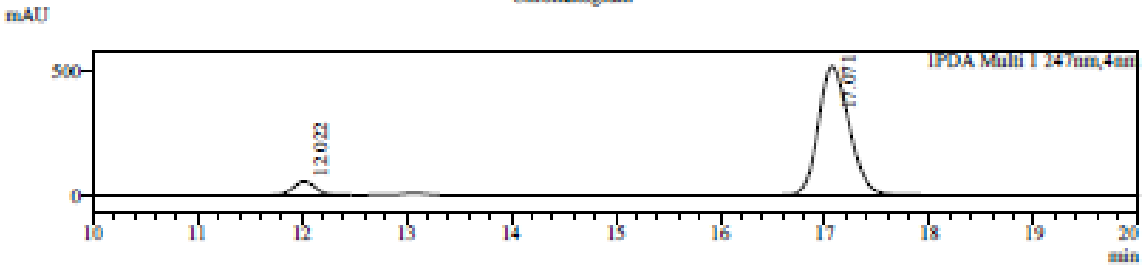
Peak#	Ret. Time	Area	Area%
1	12.125	2254333	49.762
2	17.218	2275872	50.238
Total		4530205	100.000

ethyl (*R*)-2-phenylindoline-1-carboxylate 2b

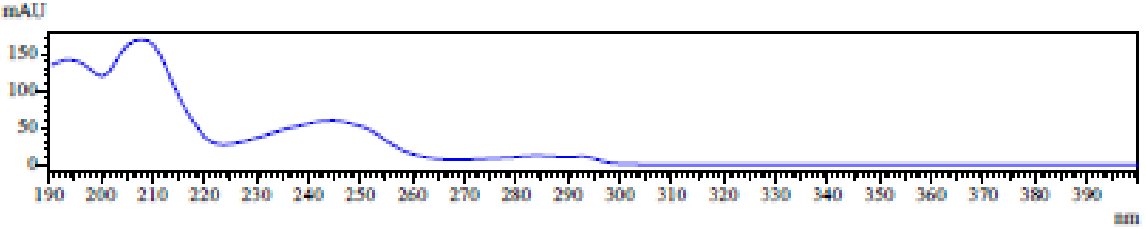
Sample Information

Sample Name	: XW-IV-269-IA-1%0.8mL
Sample ID	: XW-IV-269-IA-1%0.8mL
Data File	: XW-IV-269-IA-1%0.8mL.lcd
Method File	: XW-1%-0.8ml.lcm

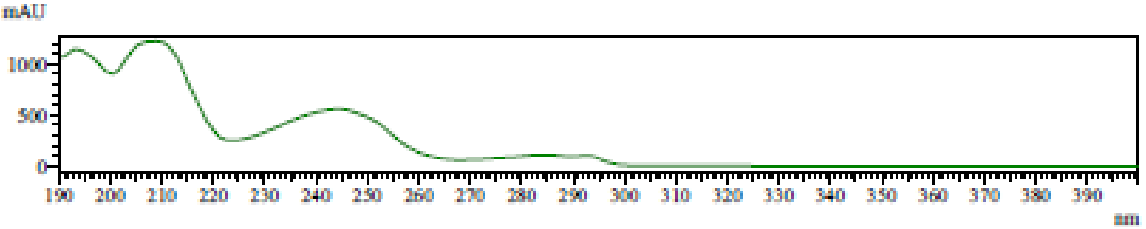
Chromatogram



UV Spectrum
Retention time = 12.022



UV Spectrum
Retention time = 17.071

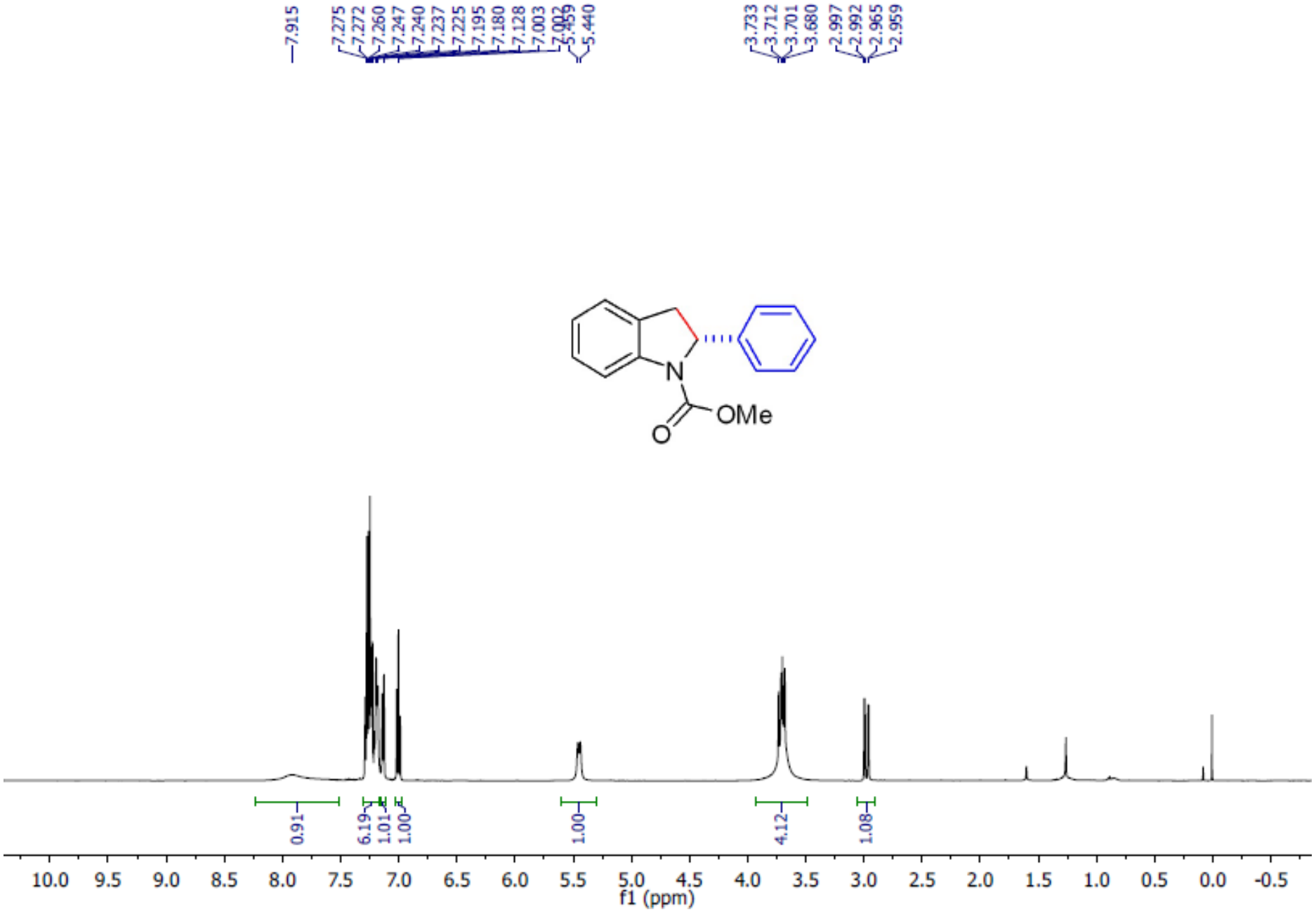


Peak Table

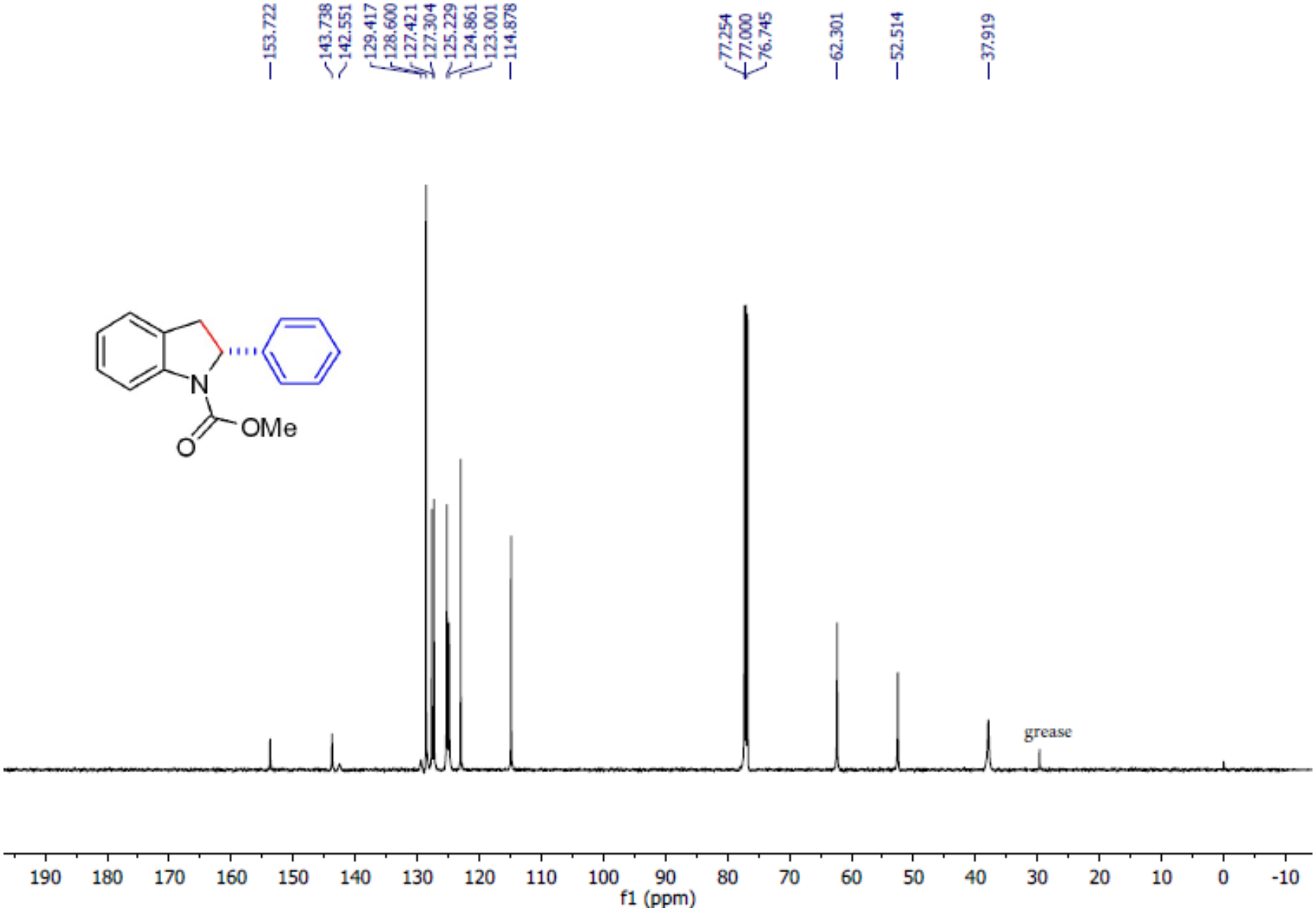
PDA Ch1 247nm

Peak#	Ret. Time	Area	Area%
1	12.022	837835	7.273
2	17.071	10682085	92.727
Total		11519920	100.000

methyl (*R*)-2-phenylindoline-1-carboxylate 2c



methyl (*R*)-2-phenylindoline-1-carboxylate 2c

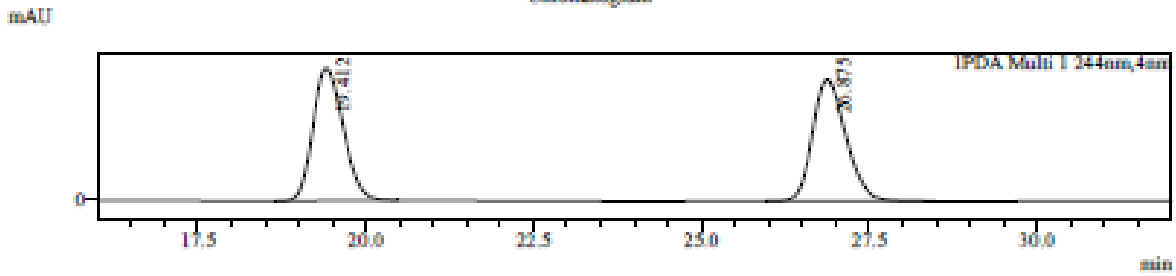


methyl (*R*)-2-phenylindoline-1-carboxylate 2c

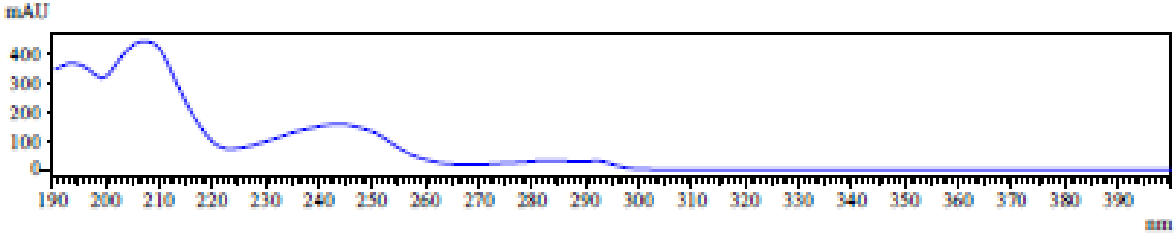
Sample Information

Sample Name	: XW-IV-246-IA-0.5%0.8mL
Sample ID	: XW-IV-246-IA-0.5%0.8mL
Data File	: XW-IV-246-IA-0.5%0.8mL.lcd
Method File	: XW-0.5%-0.8ml.lcm

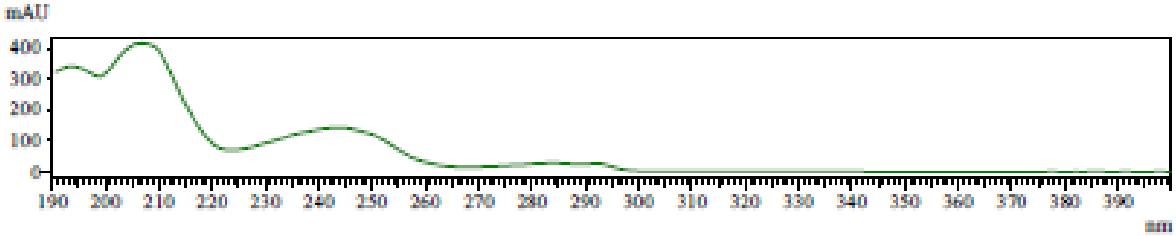
Chromatogram



UV Spectrum
Retention time = 19.412



UV Spectrum
Retention time = 26.875



Peak Table

PDA Ch1 244nm

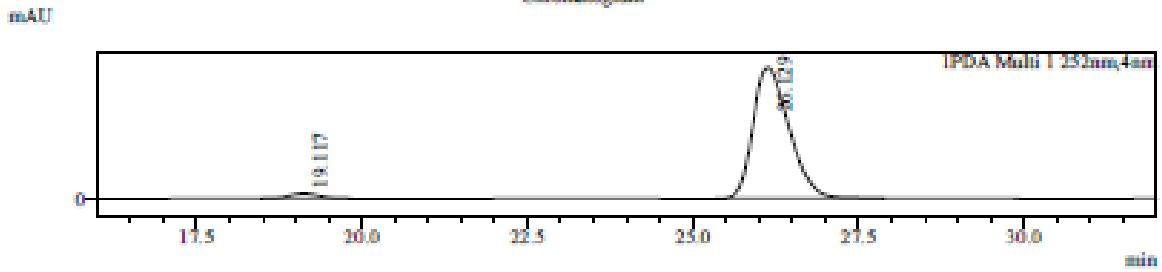
Peak#	Ret. Time	Area	Area%
1	19.412	4857504	49.571
2	26.875	4941667	50.429
Total		9799171	100.000

methyl (*R*)-2-phenylindoline-1-carboxylate 2c

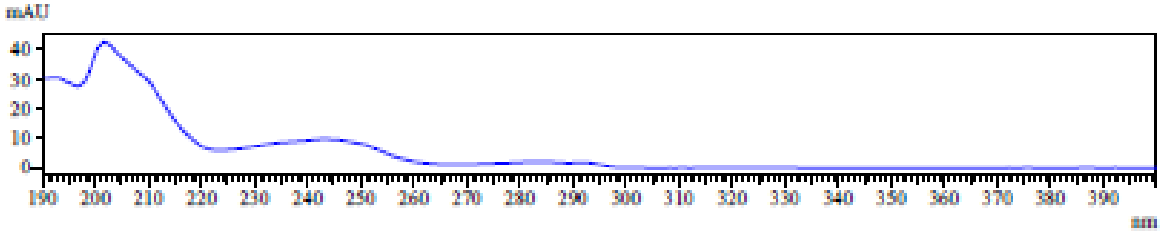
Sample Information

Sample Name	: XW-IV-283-MeOH-IA-0.5%0.8mL
Sample ID	: XW-IV-283-MeOH-IA-0.5%0.8mL
Data File	: XW-IV-283-MeOH-IA-0.5%0.8mL.lcd
Method File	: XW-0.5%-0.8ml.lcm

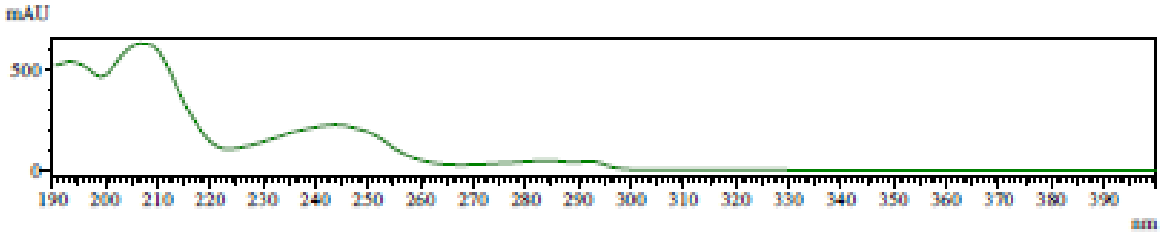
Chromatogram



UV Spectrum
Retention time = 19.117



UV Spectrum
Retention time = 26.129

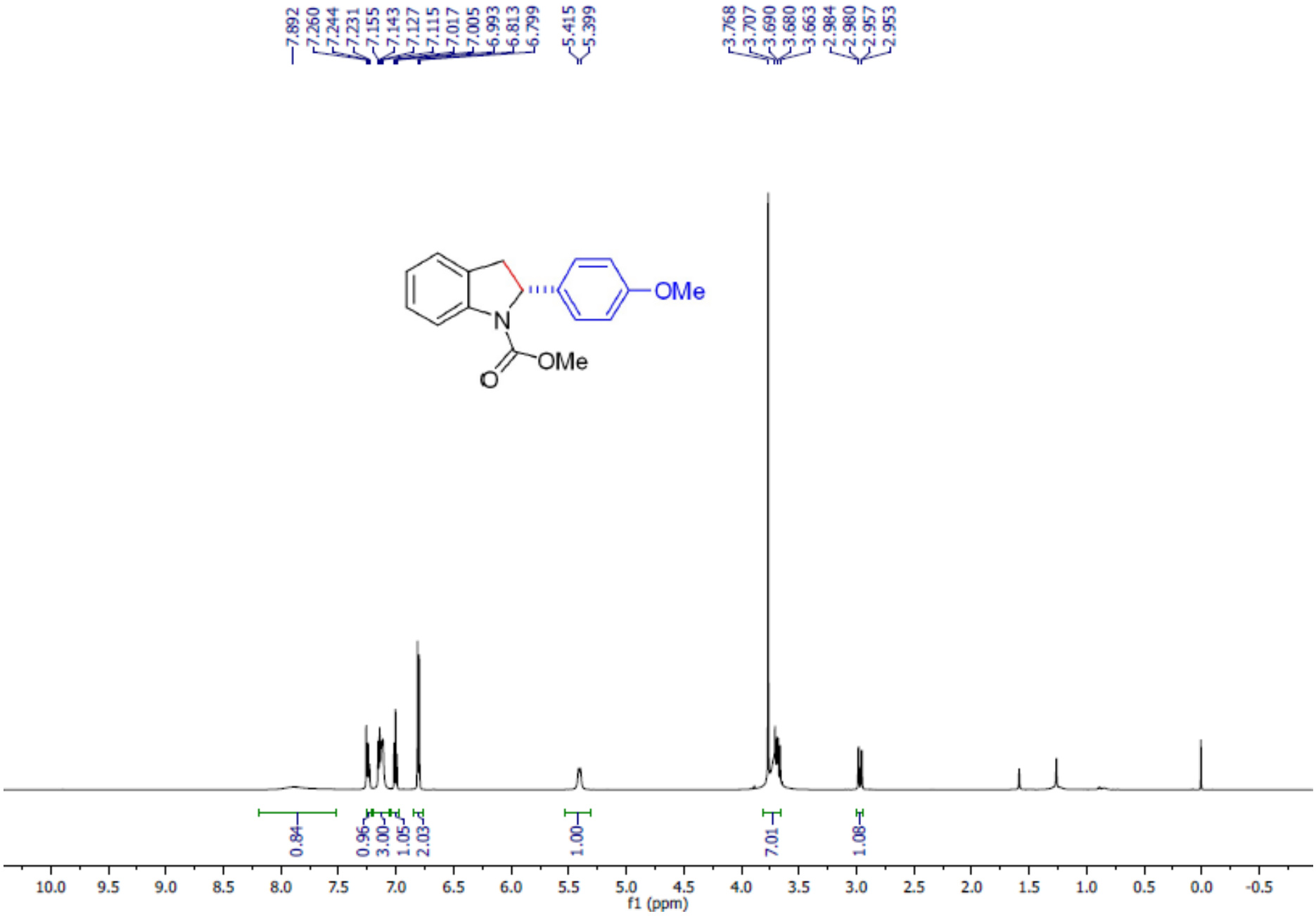


Peak Table

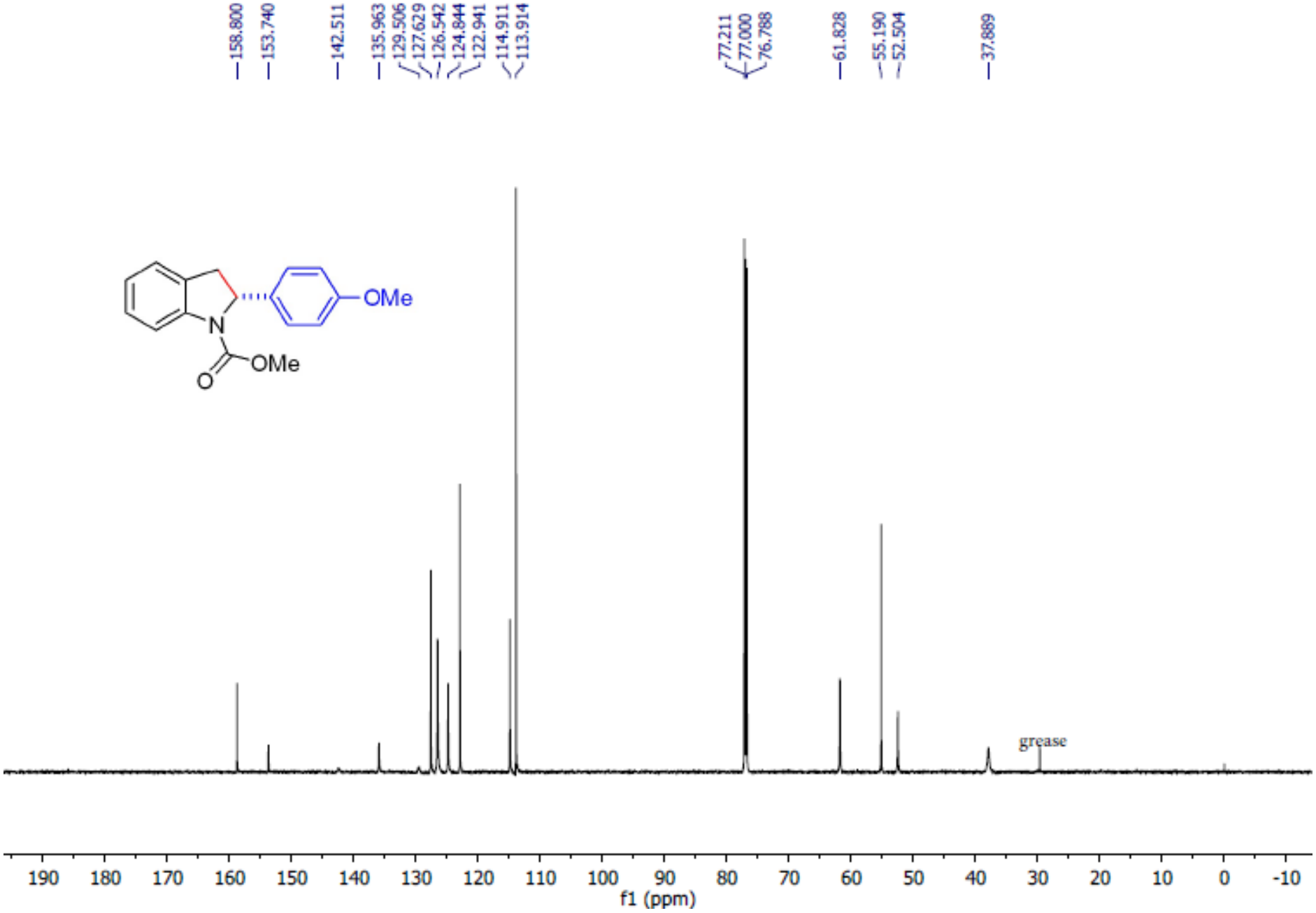
PDA Ch1 252nm

Peak#	Ret. Time	Area	Area%
1	19.117	190423	3.140
2	26.129	5873582	96.860
Total		6064005	100.000

methyl (*R*)-2-(4-methoxyphenyl)indoline-1-carboxylate 2d



methyl (*R*)-2-(4-methoxyphenyl)indoline-1-carboxylate 2d

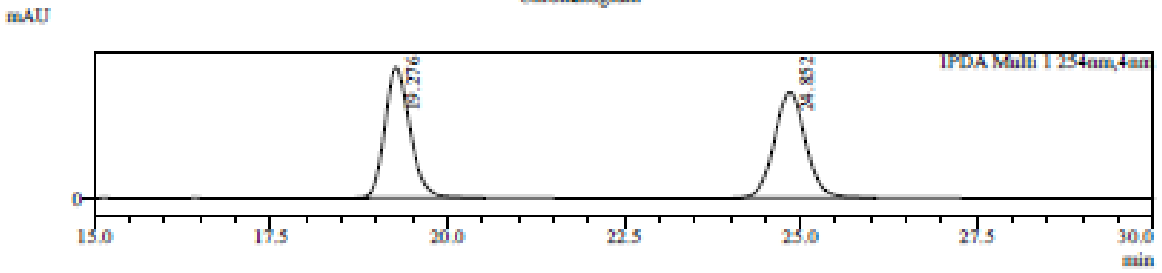


methyl (*R*)-2-(4-methoxyphenyl)indoline-1-carboxylate 2d

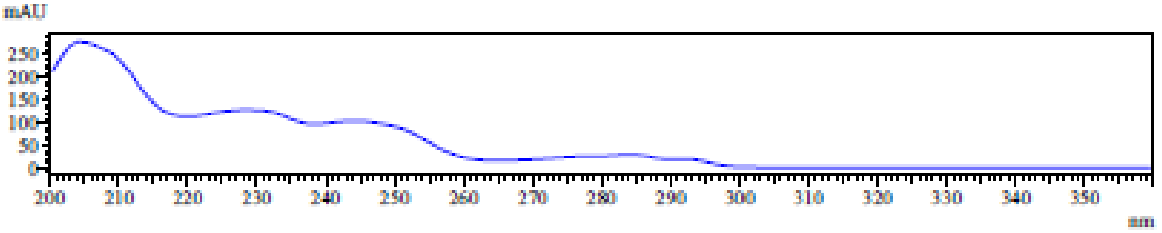
Sample Information

Sample Name	: XW-V-17-2-IA-1%1.0mL
Sample ID	: XW-V-17-2-IA-1%1.0mL
Data File	: XW-V-17-2-IA-1%1.0mL.lcd
Method File	: XW-1%-1.0ml.lcm

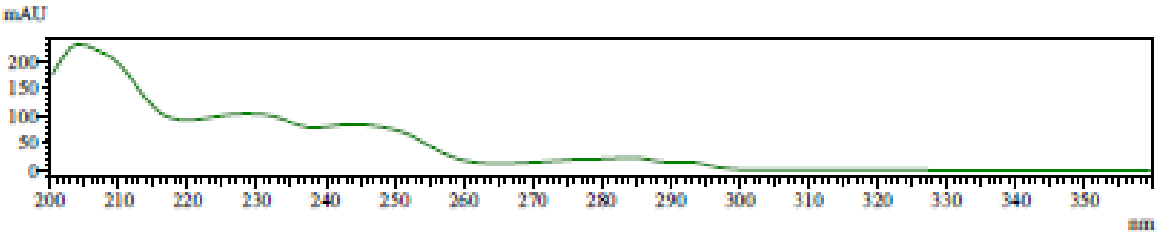
Chromatogram



UV Spectrum
Retention time = 19.276



UV Spectrum
Retention time = 24.852



Peak Table

PDA Ch1 254nm

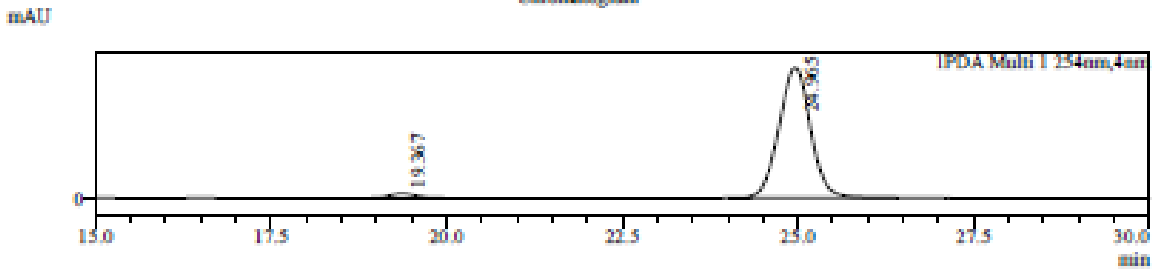
Peak#	Ret. Time	Area	Area%
1	19.276	1594697	49.760
2	24.852	1610066	50.240
Total		3204763	100.000

methyl (*R*)-2-(4-methoxyphenyl)indoline-1-carboxylate 2d

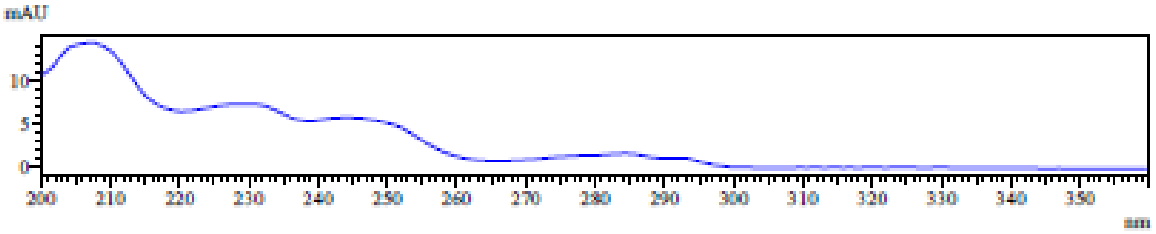
Sample Information

Sample Name	: XW-V-18-new-IA-1%1.0mL
Sample ID	: XW-V-18-new-IA-1%1.0mL
Data File	: XW-V-18-new-IA-1%1.0mL.lcd
Method File	: XW-1%-1.0ml.lcm

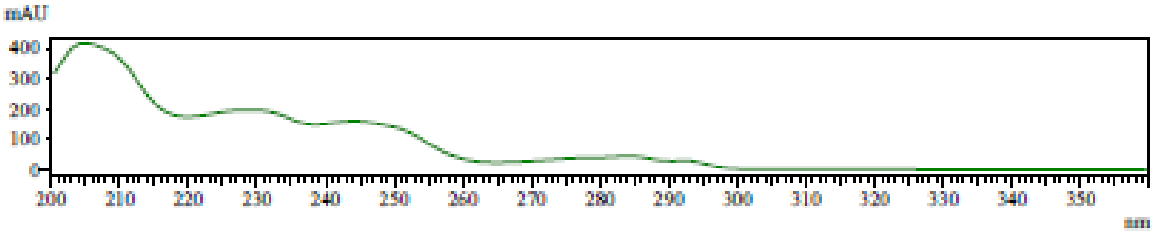
Chromatogram



UV Spectrum
Retention time = 19.367



UV Spectrum
Retention time = 24.965

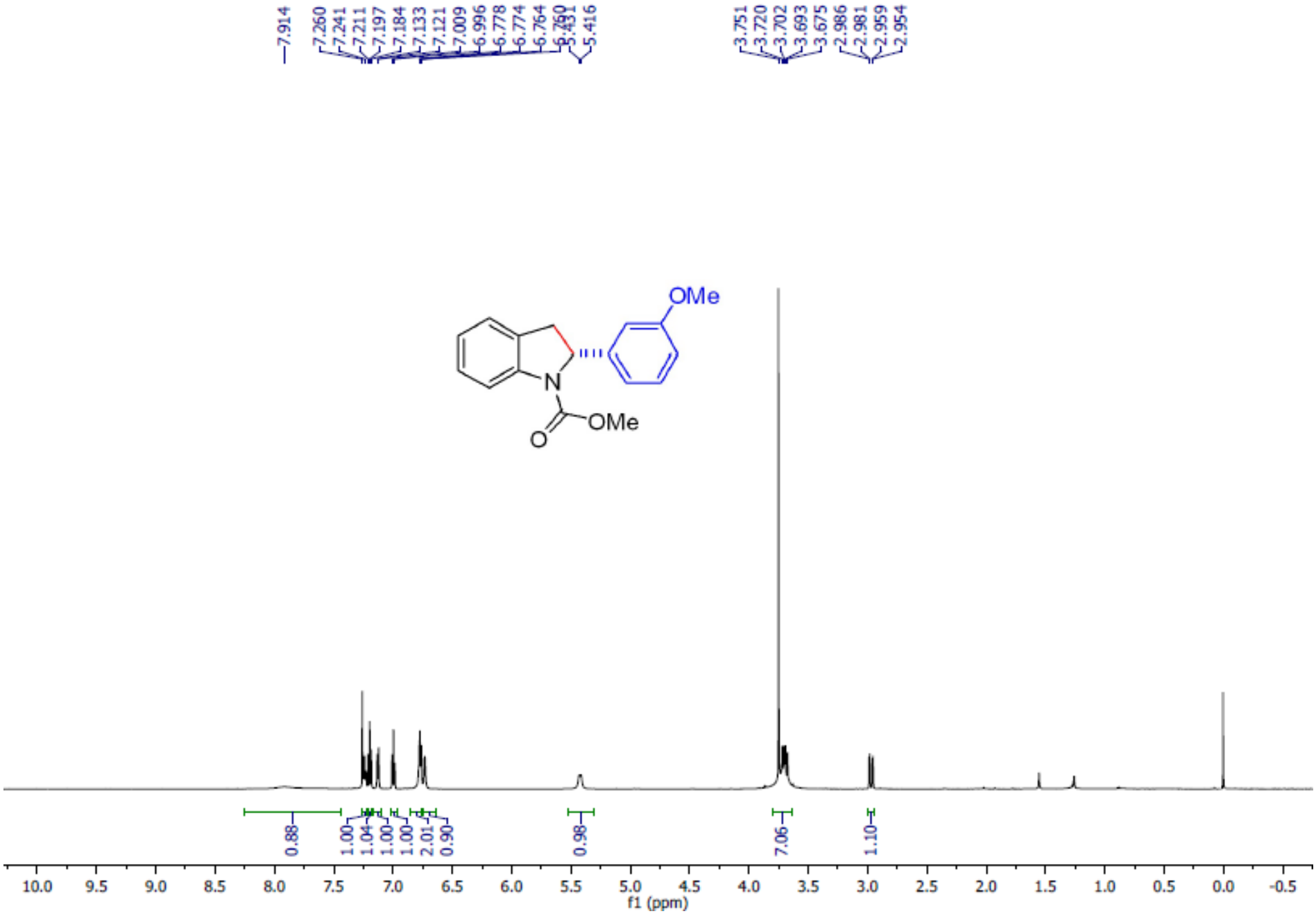


Peak Table

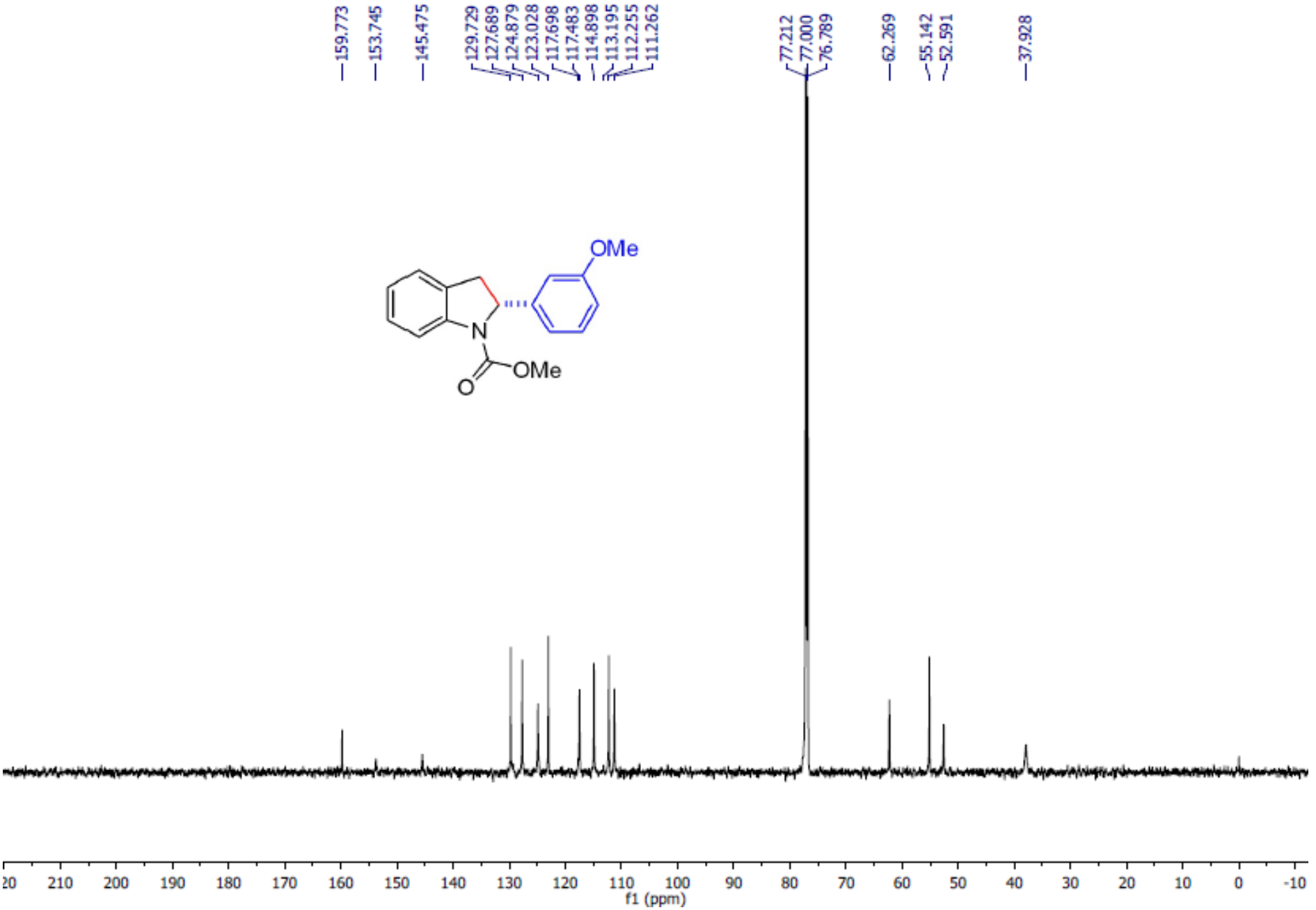
PDA Ch1 254nm

Peak#	Ret. Time	Area	Area%
1	19.367	89446	2.820
2	24.965	3082787	97.180
Total		3172233	100.000

methyl (*R*)-2-(3-methoxyphenyl)indoline-1-carboxylate 2e



methyl (*R*)-2-(3-methoxyphenyl)indoline-1-carboxylate 2e

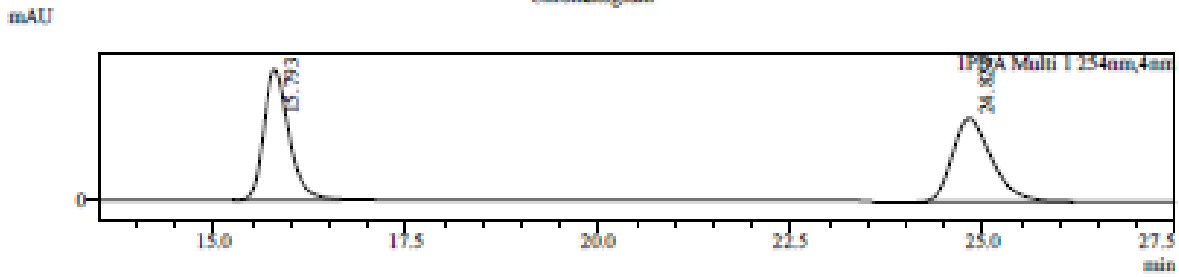


methyl (*R*)-2-(3-methoxyphenyl)indoline-1-carboxylate 2e

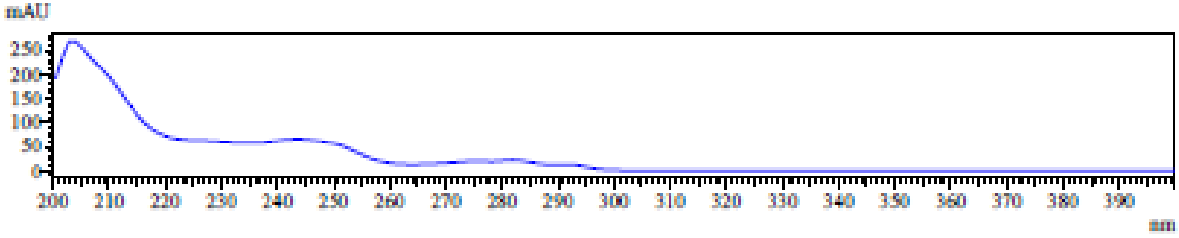
Sample Information

Sample Name	: XW-V-7-IA-1%1.0mL
Sample ID	: XW-V-7-IA-1%1.0mL
Data File	: XW-V-7-IA-1%1.0mL.lcd
Method File	: XW-1%-1.0ml.lcm

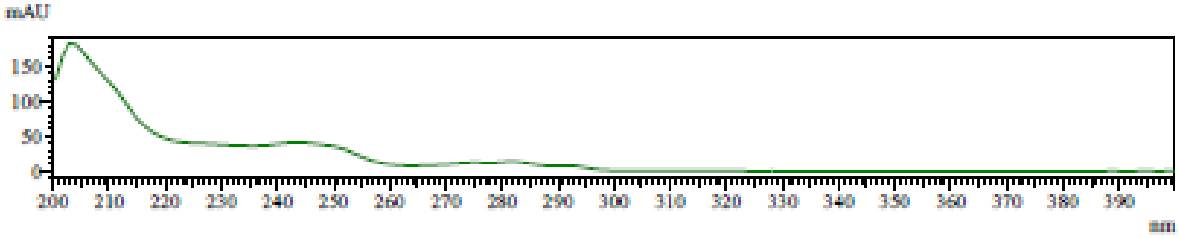
Chromatogram



UV Spectrum
Retention time = 15.793



UV Spectrum
Retention time = 24.824



Peak Table

PDA Ch1 254nm

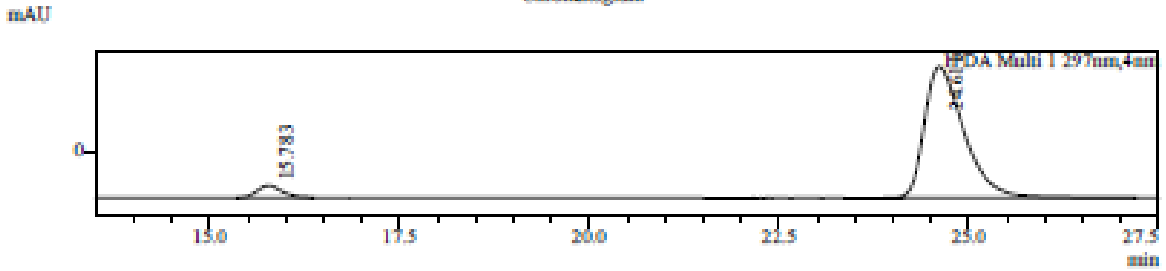
Peak#	Ret. Time	Area	Area%
1	15.793	875771	50.011
2	24.824	875394	49.989
Total		1751165	100.000

methyl (*R*)-2-(3-methoxyphenyl)indoline-1-carboxylate 2e

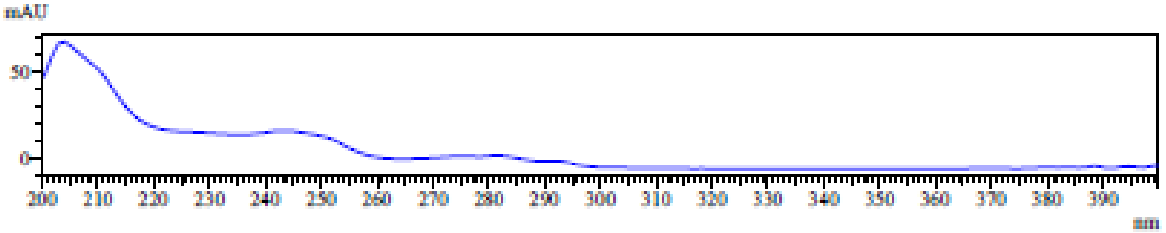
Sample Information

Sample Name	: XW-V-8A-IA-1%1.0mL
Sample ID	: XW-V-8A-IA-1%1.0mL
Data File	: XW-V-8A-MeOH-IA-1%1.0mL.lcd
Method File	: XW-1%-1.0ml.lcm

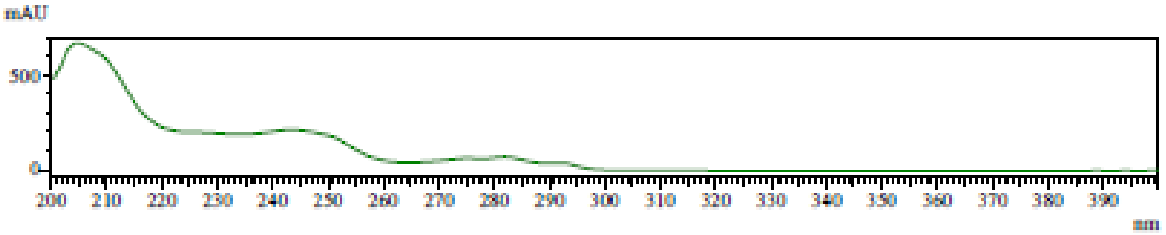
Chromatogram



UV Spectrum
Retention time = 15.783



UV Spectrum
Retention time = 24.611

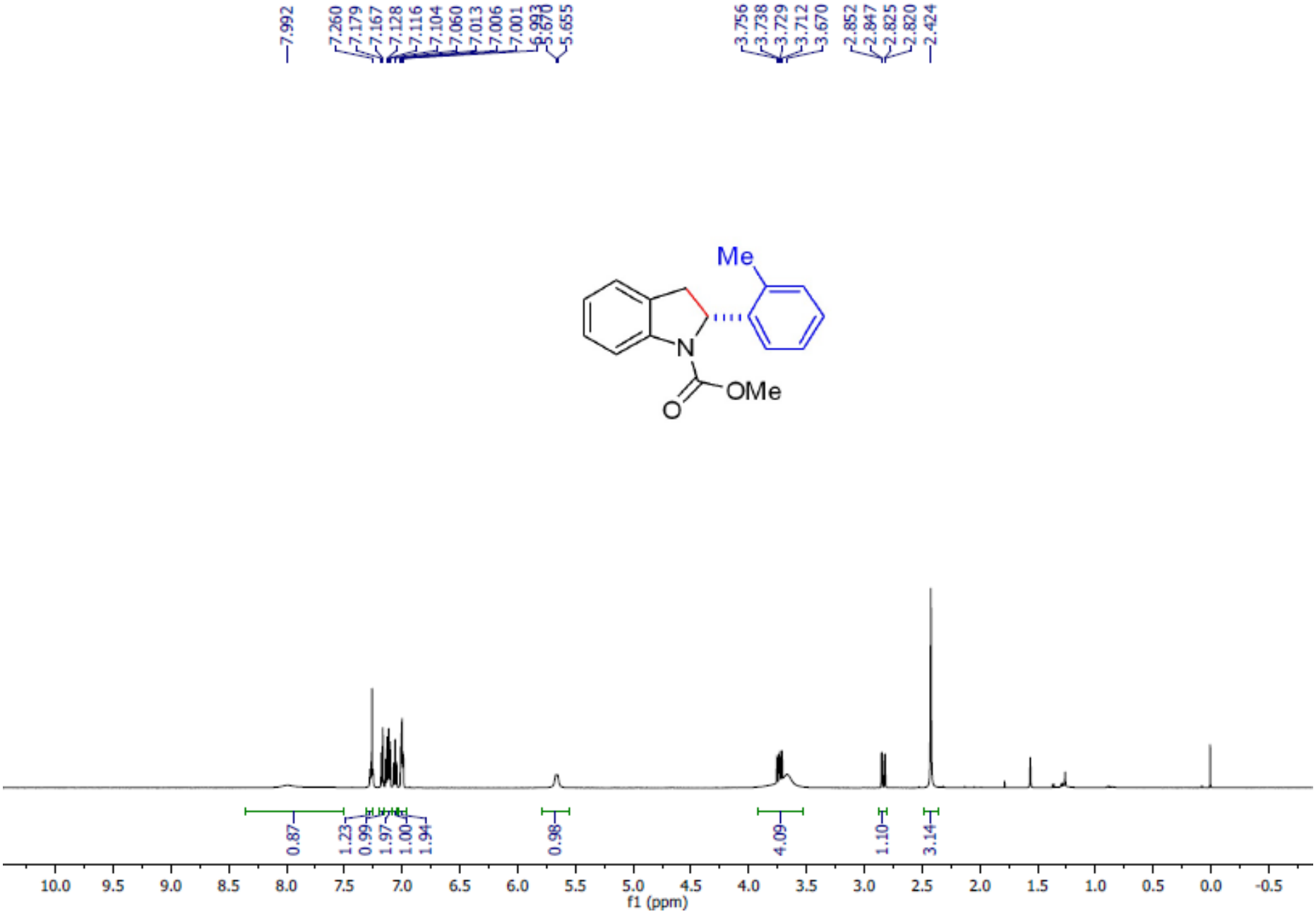


Peak Table

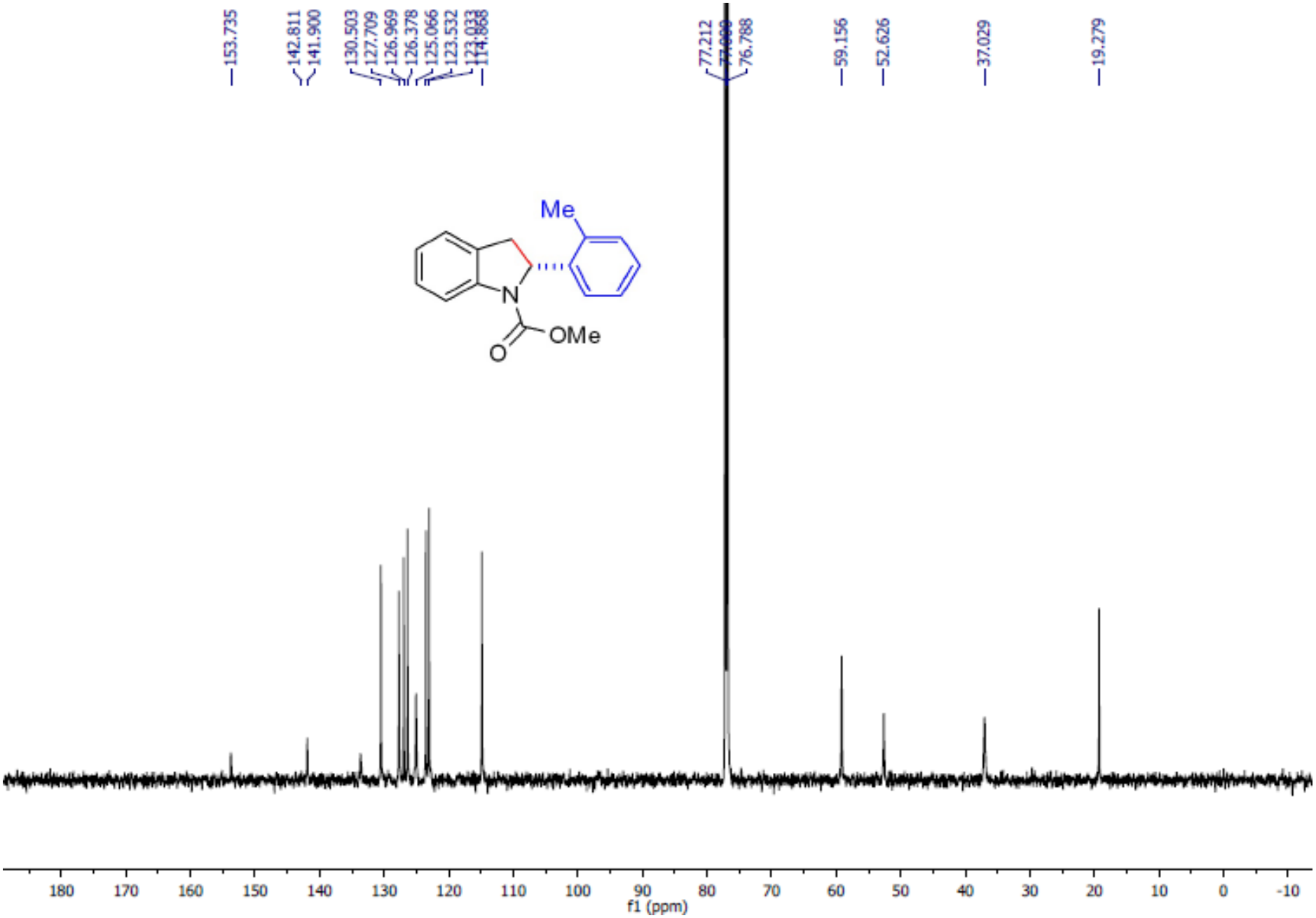
PDA Ch1 297nm

Peak#	Ret. Time	Area	Area%
1	15.783	34284	5.273
2	24.611	615910	94.727
Total		650195	100.000

methyl (*R*)-2-(o-tolyl)indoline-1-carboxylate 2f



methyl (*R*)-2-(o-tolyl)indoline-1-carboxylate 2f

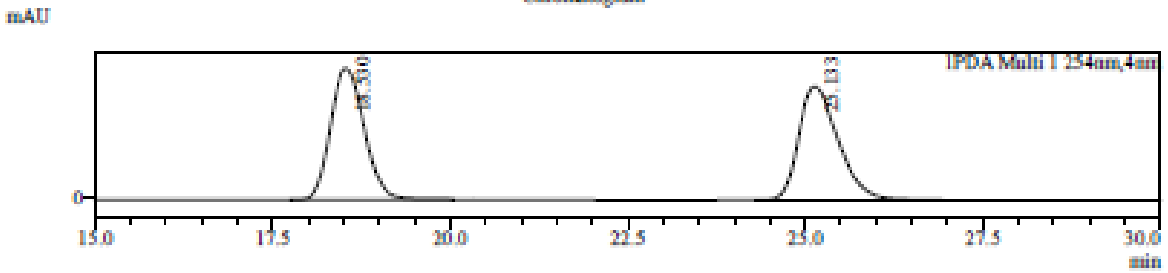


methyl (*R*)-2-(o-tolyl)indoline-1-carboxylate 2f

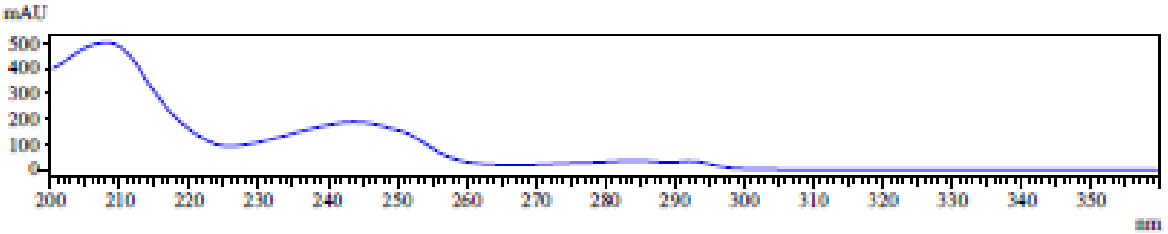
Sample Information

Sample Name	: XW-V-46-IA-0.5%0.8mL
Sample ID	: XW-V-46-IA-0.5%0.8mL
Data File	: XW-V-46-IA-0.5%0.8mL.lcd
Method File	: XW-0.5%-0.8ml.lcm

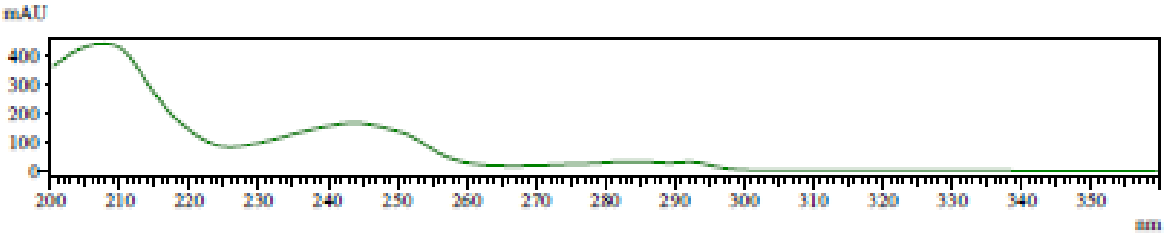
Chromatogram



UV Spectrum
Retention time = 18.530



UV Spectrum
Retention time = 25.133



Peak Table

PDA Ch1 254nm

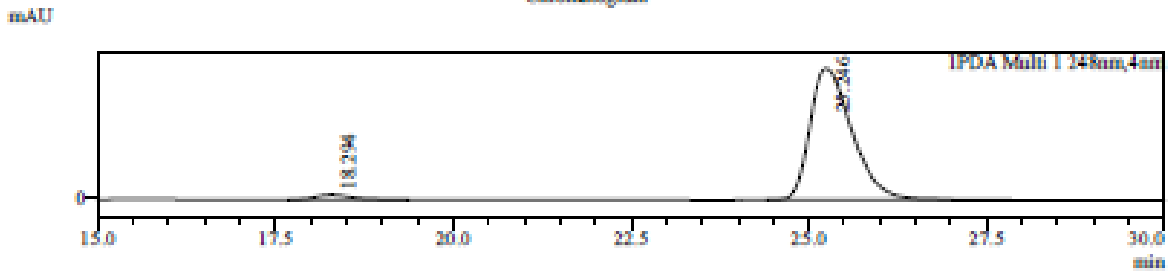
Peak#	Ret. Time	Area	Area%
1	18.530	3403363	49.929
2	25.133	3413071	50.071
Total		6816434	100.000

methyl (*R*)-2-(o-tolyl)indoline-1-carboxylate 2f

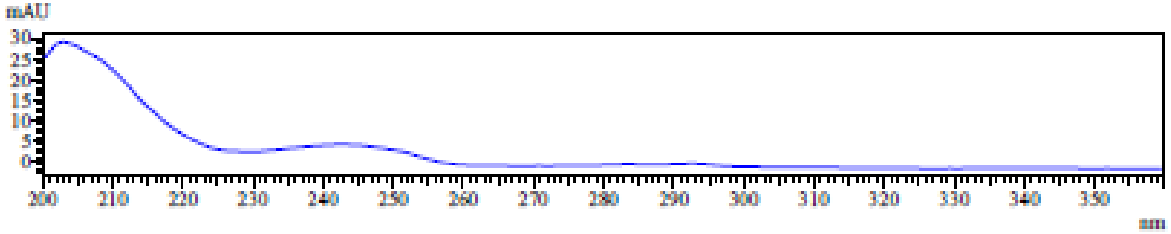
Sample Information

Sample Name	: XW-V-47A-LA-0.5%0.8mL
Sample ID	: XW-V-47A-LA-0.5%0.8mL
Data File	: XW-V-47A-LA-0.5%0.8mL.lcd
Method File	: XW-0.5%-0.8ml.lcm

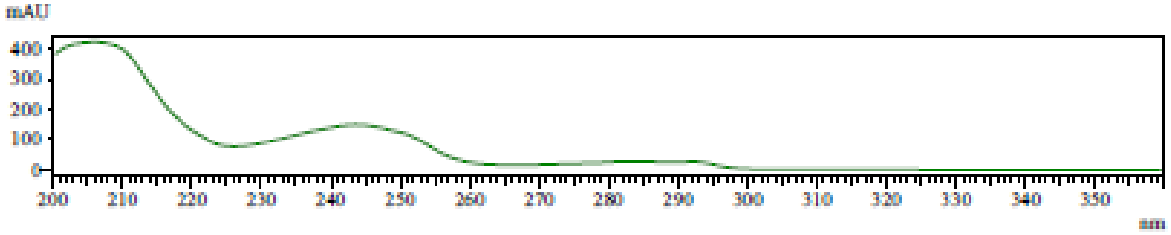
Chromatogram



UV Spectrum
Retention time = 18.294



UV Spectrum
Retention time = 25.246

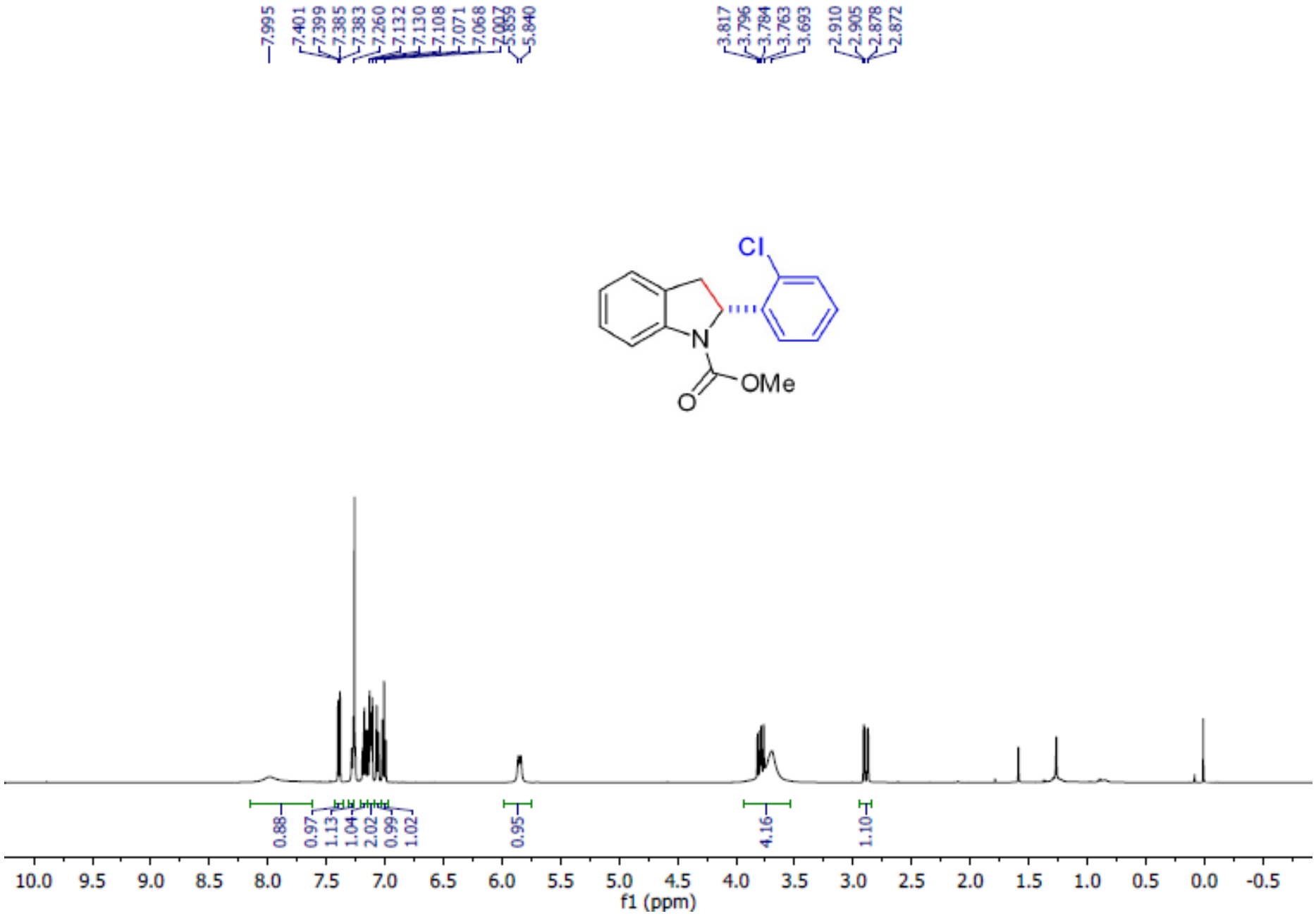


Peak Table

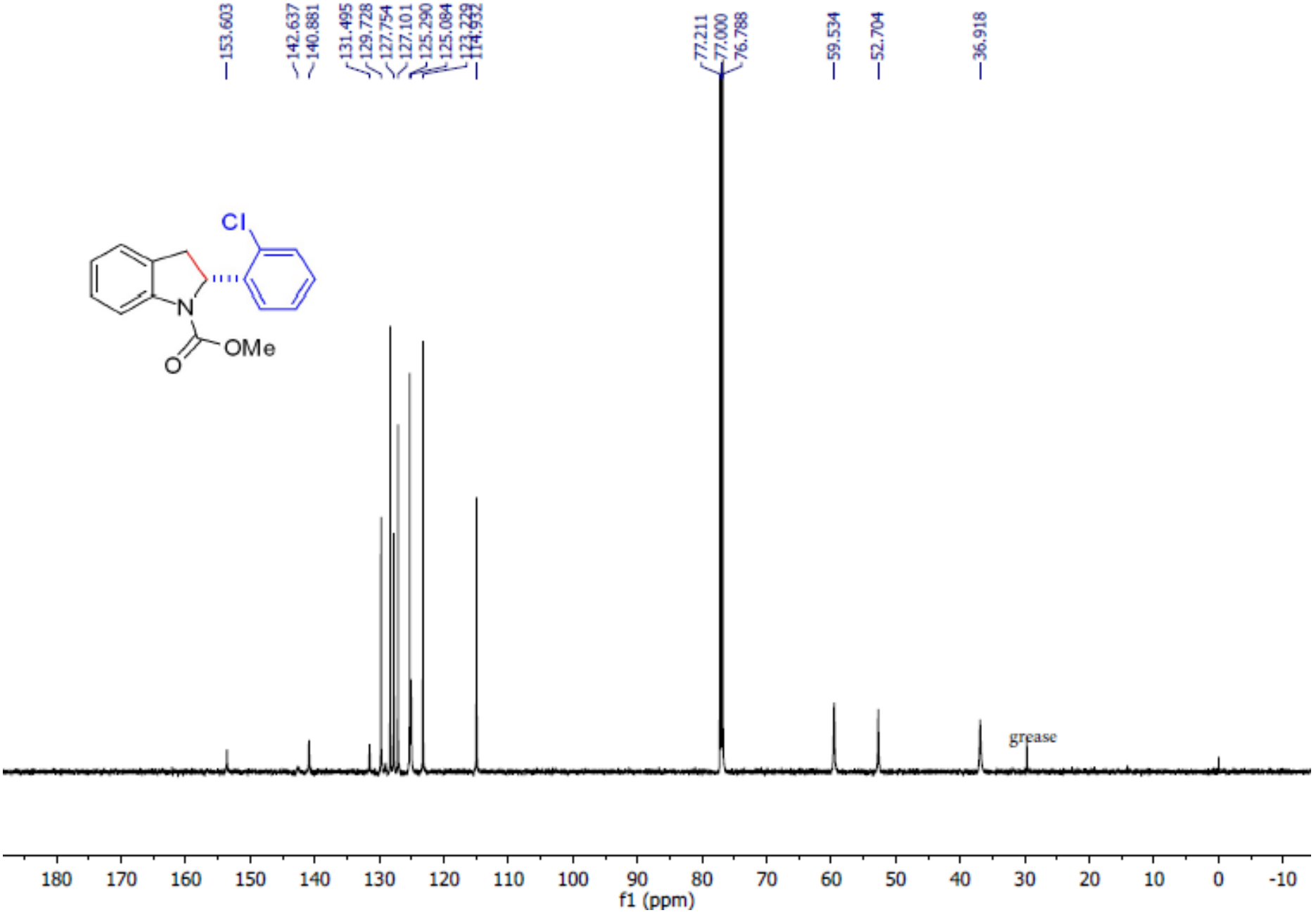
PDA Ch1 248nm

Peak#	Ret. Time	Area	Area%
1	18.294	164441	2.886
2	25.246	5533370	97.114
Total		5697811	100.000

methyl (*R*)-2-(2-chlorophenyl)indoline-1-carboxylate 2g



methyl (*R*)-2-(2-chlorophenyl)indoline-1-carboxylate 2g

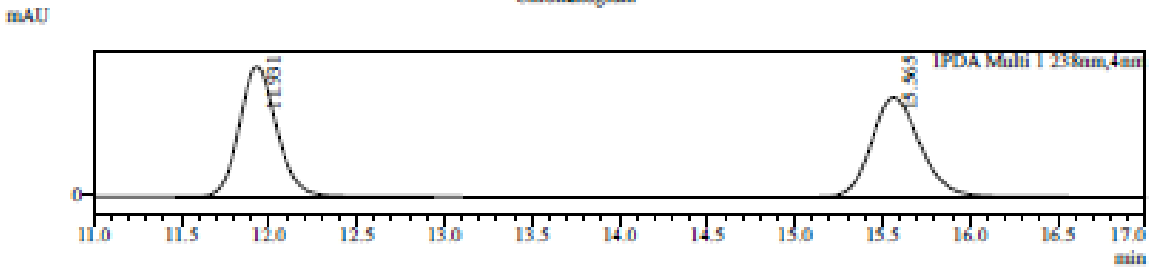


methyl (*R*)-2-(2-chlorophenyl)indoline-1-carboxylate 2g

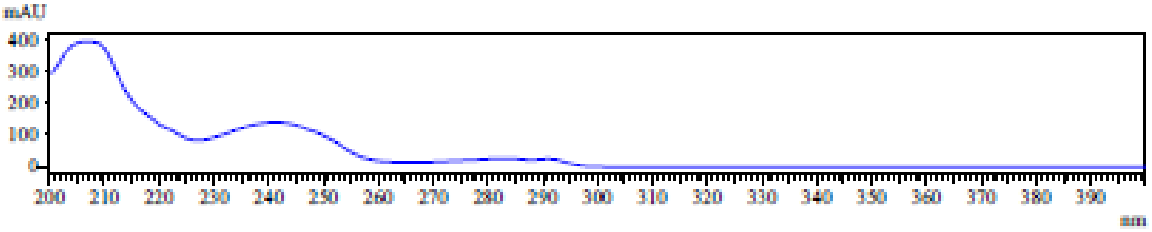
Sample Information

Sample Name	: XW-V-13-IA-1%0.8.0mL
Sample ID	: XW-V-13-IA-1%0.8.0mL
Data File	: XW-V-13-IA-1%0.8.0mL.lcd
Method File	: XW-1%-0.8ml.lcm

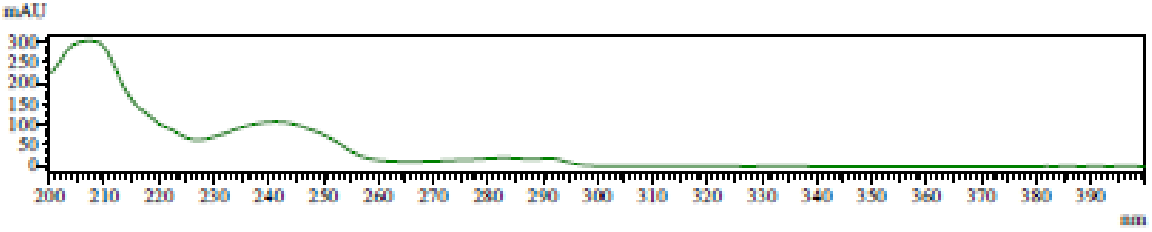
Chromatogram



UV Spectrum
Retention time = 11.931



UV Spectrum
Retention time = 15.565



Peak Table

PDA Ch1 238nm

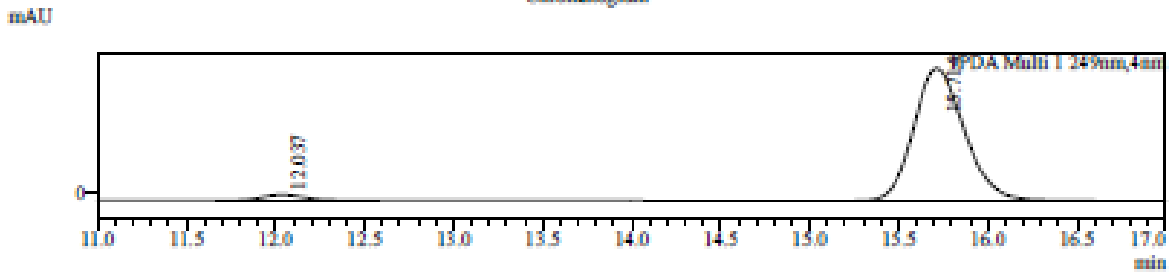
Peak#	Ret. Time	Area	Area%
1	11.931	2006931	50.175
2	15.565	1992922	49.825
Total		3999853	100.000

methyl (*R*)-2-(2-chlorophenyl)indoline-1-carboxylate 2g

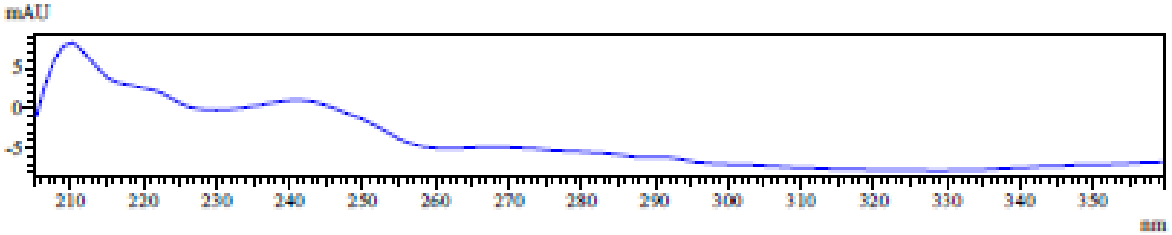
Sample Information

Sample Name	: XW-V-11-MeOH-2-IA-1%0.8mL
Sample ID	: XW-V-11-MeOH-2-IA-1%0.8mL
Data File	: XW-V-11-MeOH-2-IA-1%0.8mL.lcd
Method File	: XW-1%-0.8ml.lcm

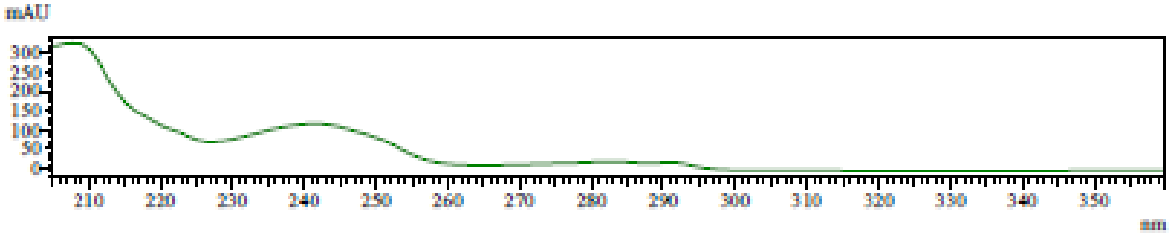
Chromatogram



UV Spectrum
Retention time = 12.037



UV Spectrum
Retention time = 15.714

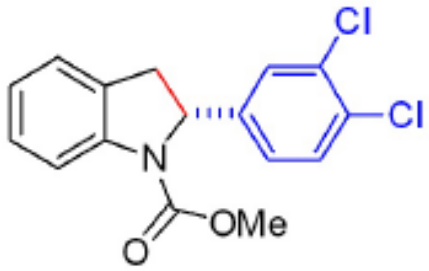


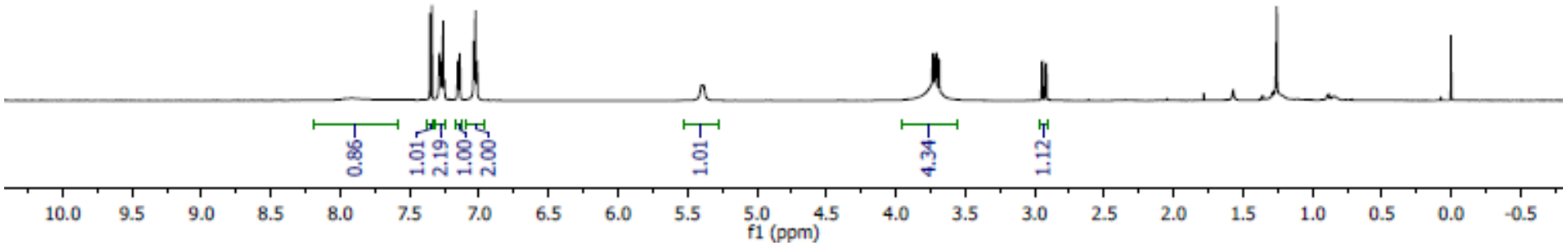
Peak Table

PDA Ch1 249nm

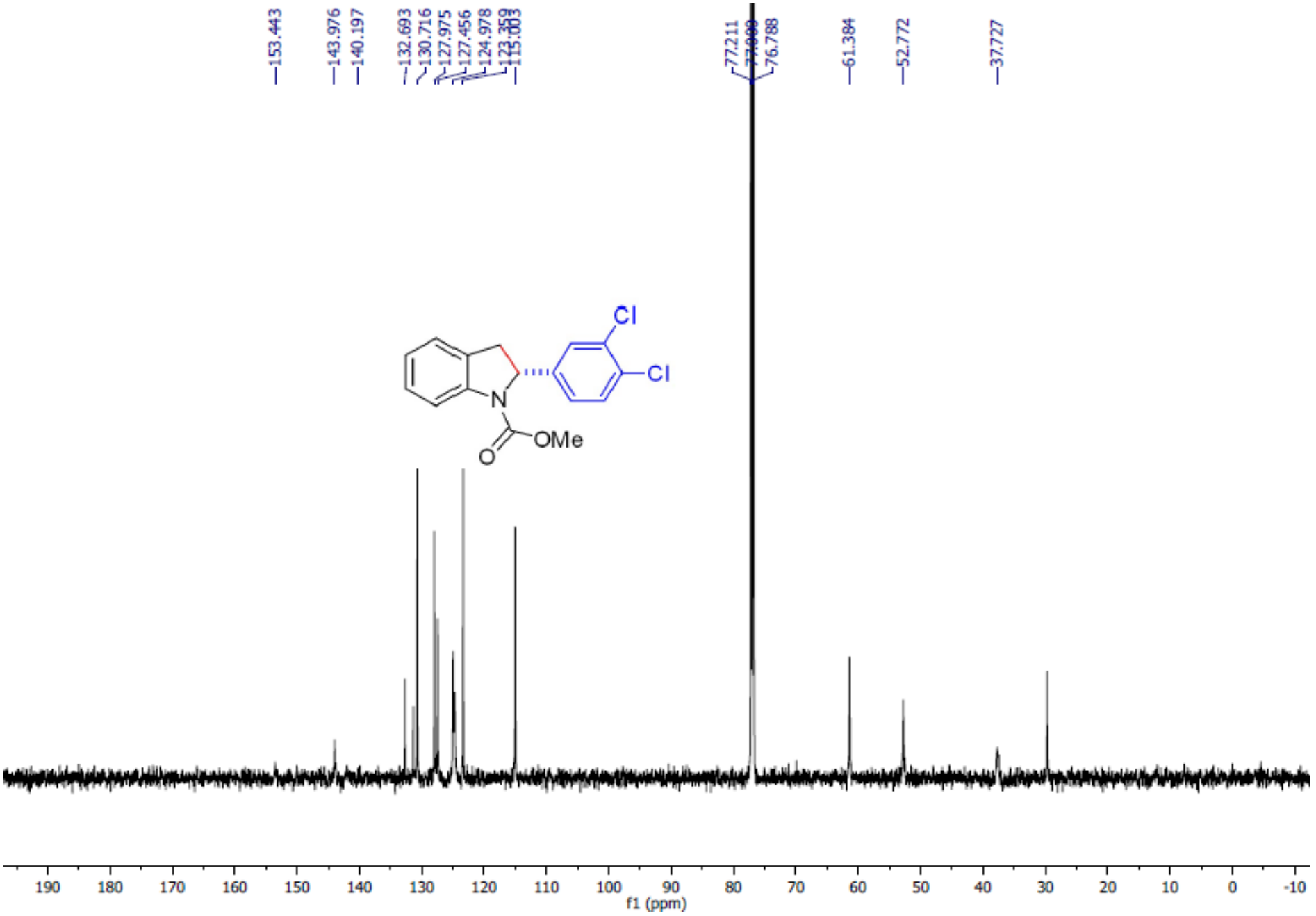
Peak#	Ret. Time	Area	Area%
1	12.037	64130	3.500
2	15.714	1768249	96.500
Total		1832379	100.000

methyl (*R*)-2-(3,4-dichlorophenyl)indoline-1-carboxylate 2h





methyl (*R*)-2-(3,4-dichlorophenyl)indoline-1-carboxylate 2h

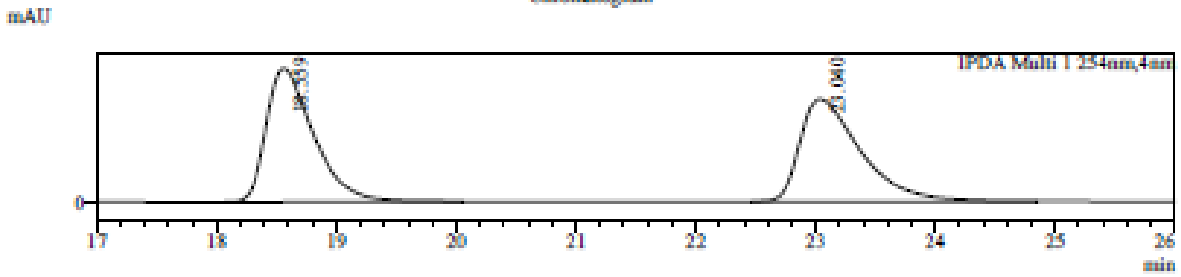


methyl (*R*)-2-(3,4-dichlorophenyl)indoline-1-carboxylate 2h

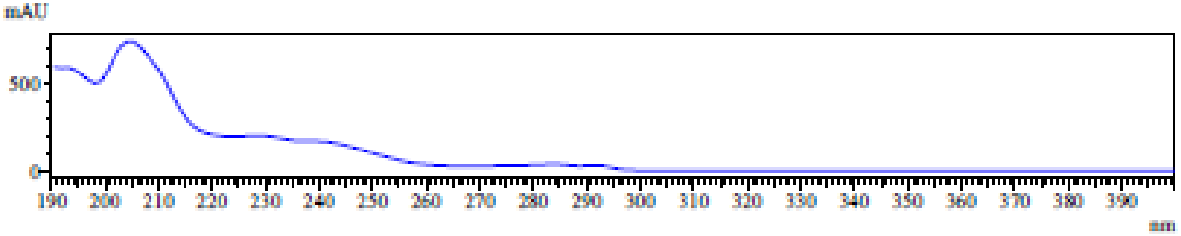
Sample Information

Sample Name	: XW-IV-262-2-IB-0.5%0.8mL
Sample ID	: XW-IV-262-2-IB-0.5%0.8mL
Data File	: XW-IV-262-2-IB-0.5%0.8mL.lcd
Method File	: XW-0.5%-0.8ml.lcm

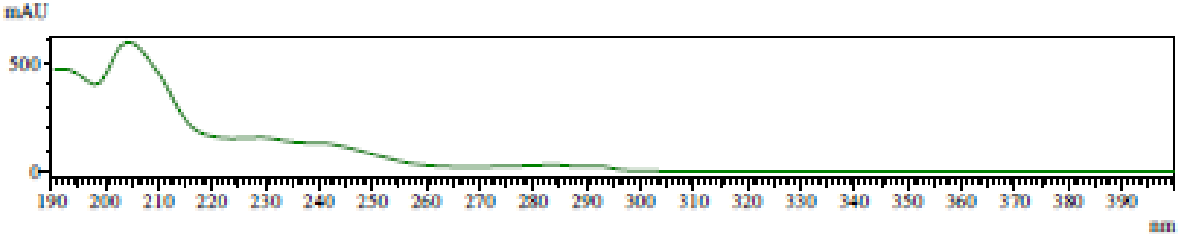
Chromatogram



UV Spectrum
Retention time = 18.559



UV Spectrum
Retention time = 23.040



Peak Table

PDA Ch1 254nm

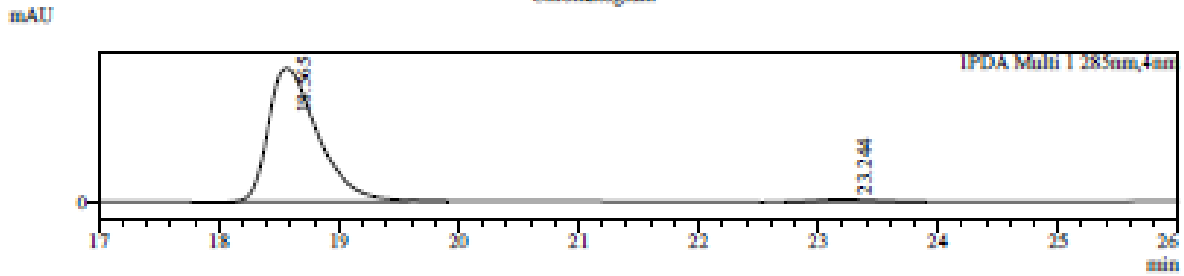
Peak#	Ret. Time	Area	Area%
1	18.559	1880586	50.371
2	23.040	1852905	49.629
Total		3733491	100.000

methyl (*R*)-2-(3,4-dichlorophenyl)indoline-1-carboxylate 2h

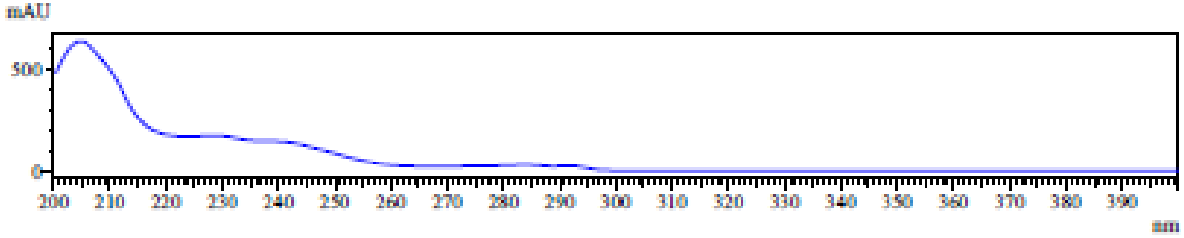
Sample Information

Sample Name : XW-IV-292B-MeOH-1-IB-0.5%0.8mL
Sample ID : XW-IV-292B-MeOH-1-IB-0.5%0.8mL
Data File : XW-IV-292B-MeOH-1-IB-0.5%0.8mL.lcd
Method File : XW-0.5%-0.8ml.lcm

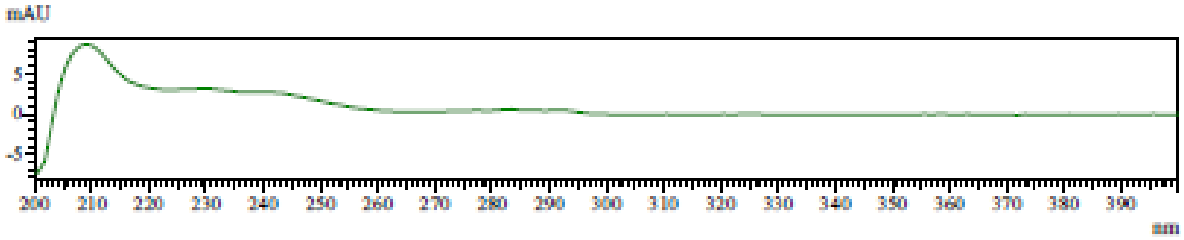
Chromatogram



UV Spectrum
Retention time = 18.565



UV Spectrum
Retention time = 23.244

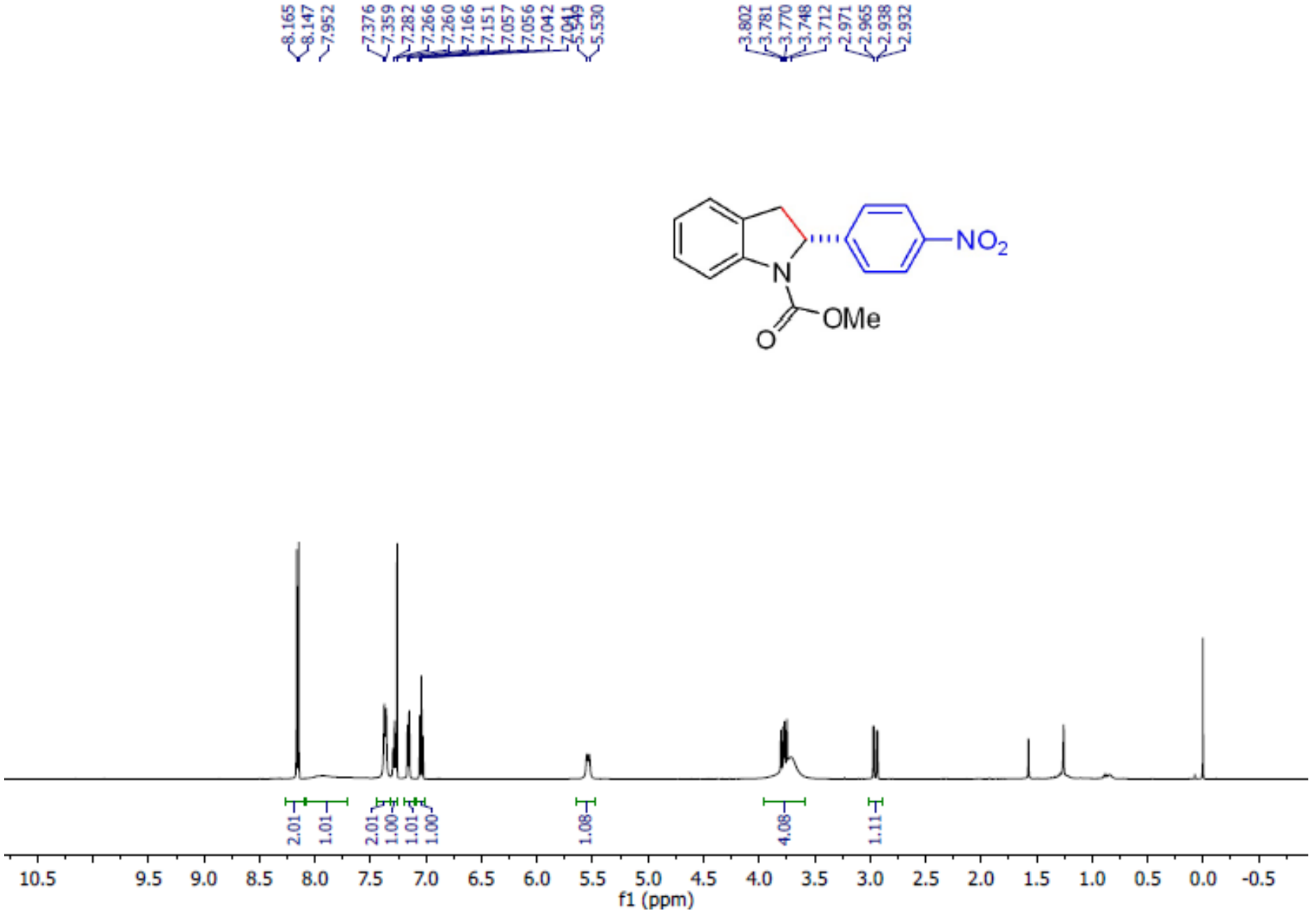


Peak Table

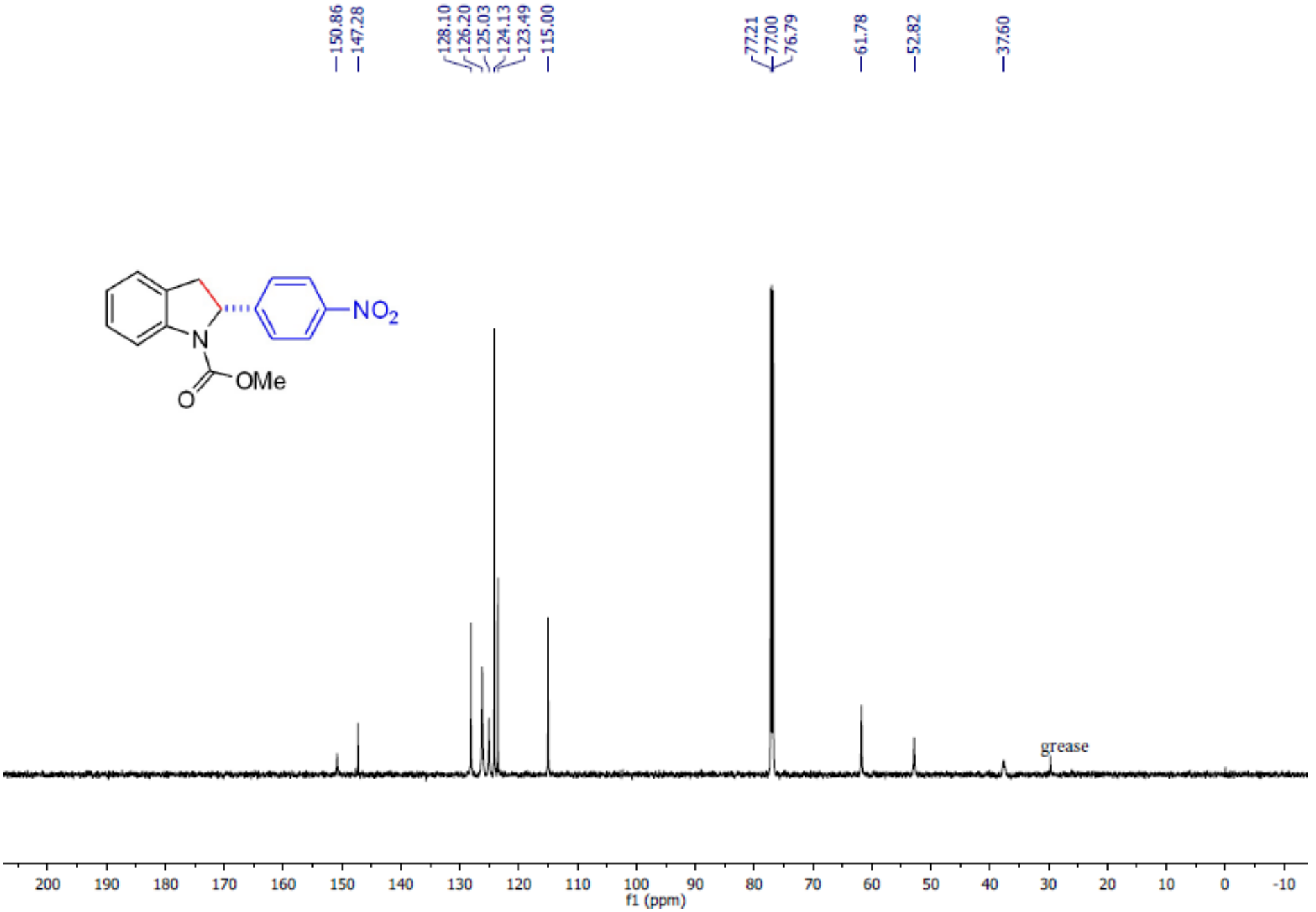
PDA Ch1 285nm

Peak#	Ret. Time	Area	Area%
1	18.565	849351	98.008
2	23.244	17267	1.992
Total		866618	100.000

methyl (*R*)-2-(4-nitrophenyl)indoline-1-carboxylate 2i



methyl (*R*)-2-(4-nitrophenyl)indoline-1-carboxylate 2i

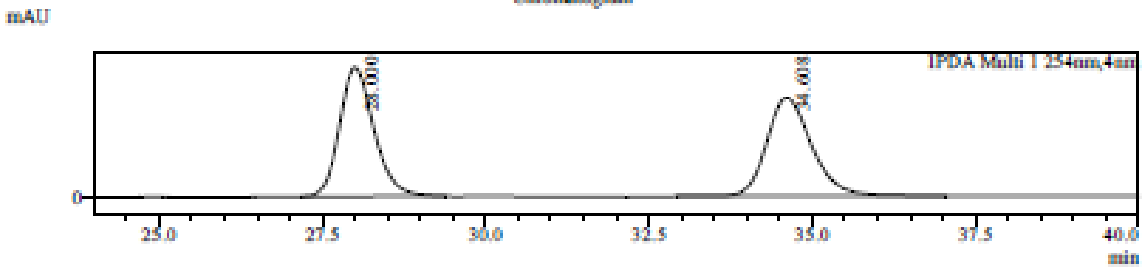


methyl (*R*)-2-(4-nitrophenyl)indoline-1-carboxylate 2i

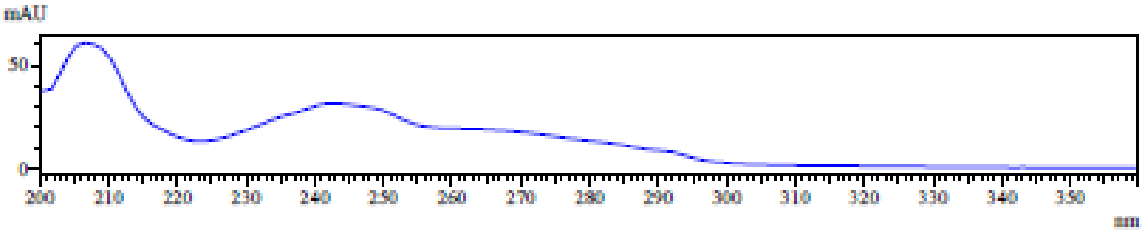
Sample Information

Sample Name	: XW-V-31-2-IA-2%1.0mL
Sample ID	: XW-V-31-2-IA-2%1.0mL
Data File	: XW-V-31-2-IA-2%1.0mL.lcd
Method File	: XW-2%-1ml.lcm

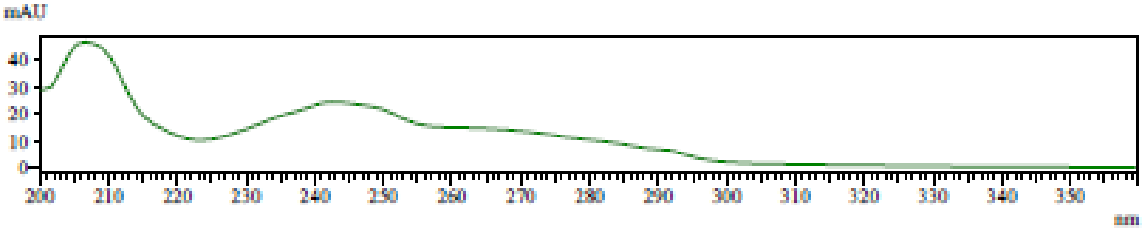
Chromatogram



UV Spectrum
Retention time = 28.000



UV Spectrum
Retention time = 34.608



Peak Table

PDA Ch1 254nm

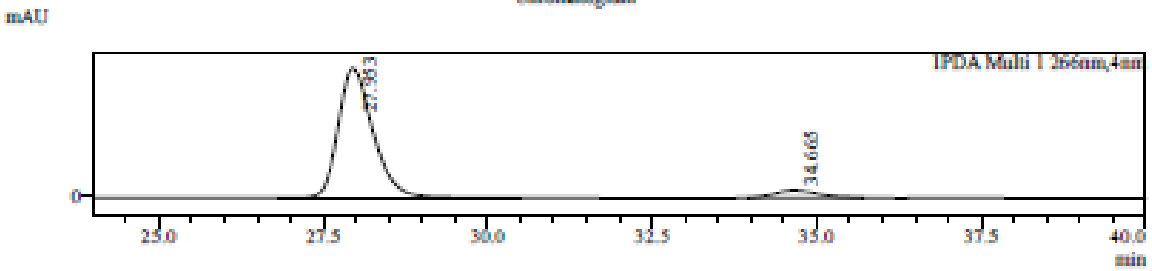
Peak#	Ret. Time	Area	Area%
1	28.000	824550	50.067
2	34.608	822338	49.933
Total		1646888	100.000

methyl (*R*)-2-(4-nitrophenyl)indoline-1-carboxylate 2i

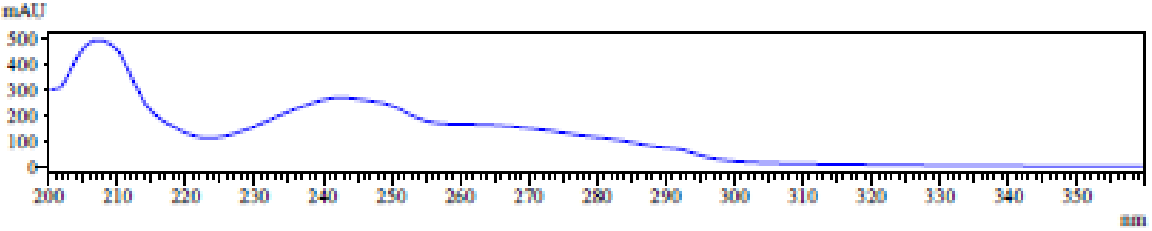
Sample Information

Sample Name	: XW-V-32A-IA-2%1.0mL
Sample ID	: XW-V-32A-IA-2%1.0mL
Data File	: XW-V-32A-IA-2%1.0mL.lcd
Method File	: XW-2%-1ml.lcm

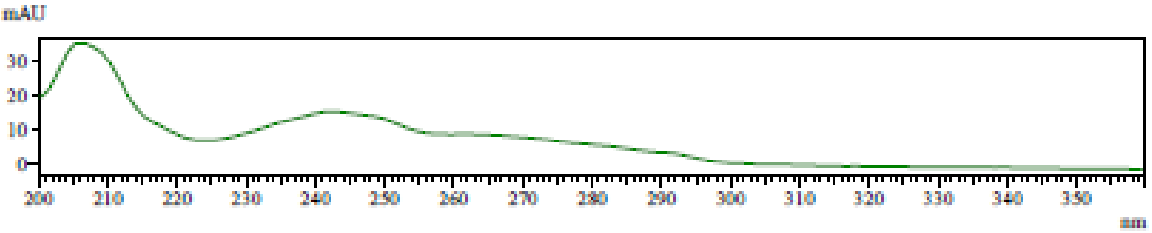
Chromatogram



UV Spectrum
Retention time = 27.953



UV Spectrum
Retention time = 34.665

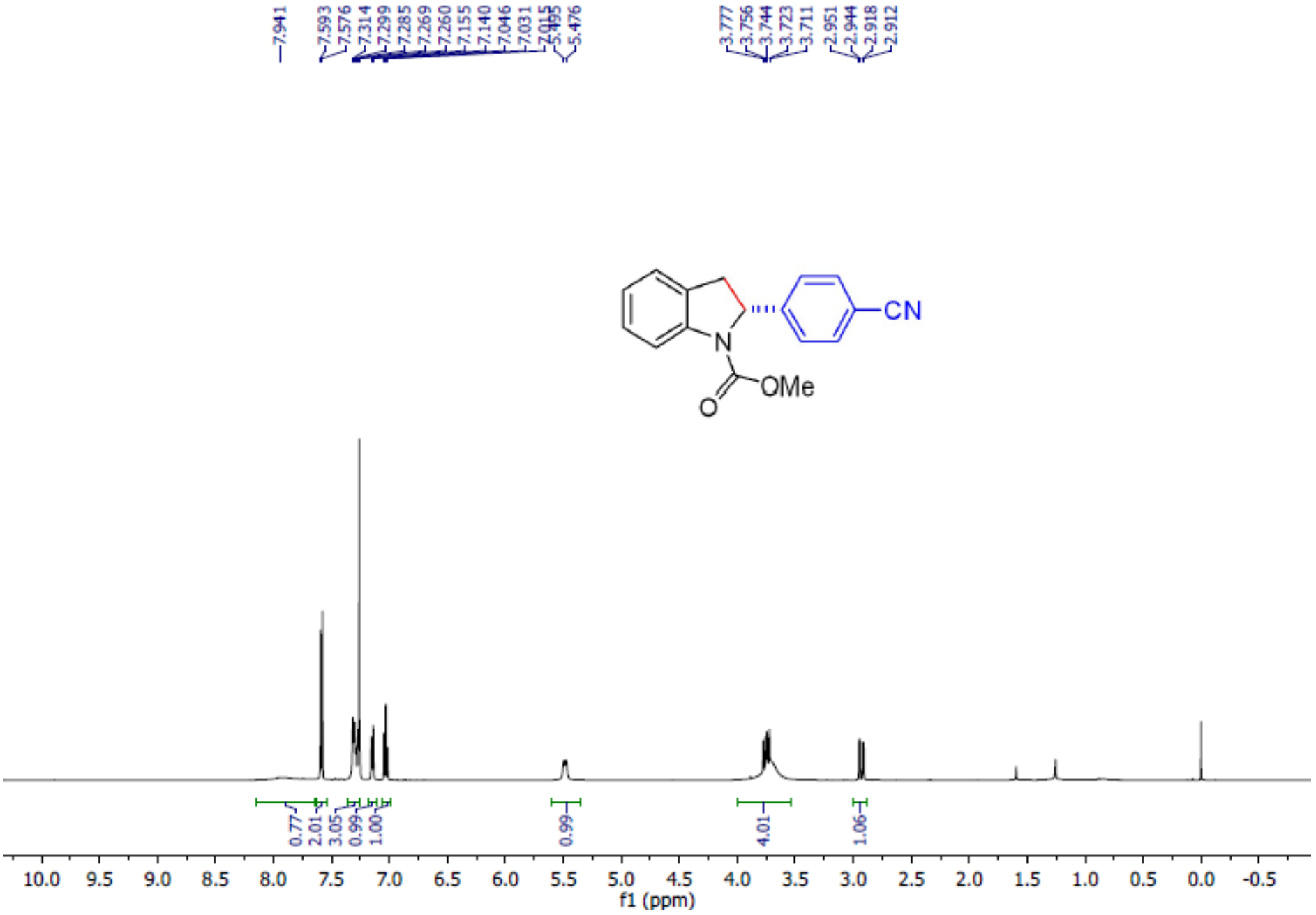


Peak Table

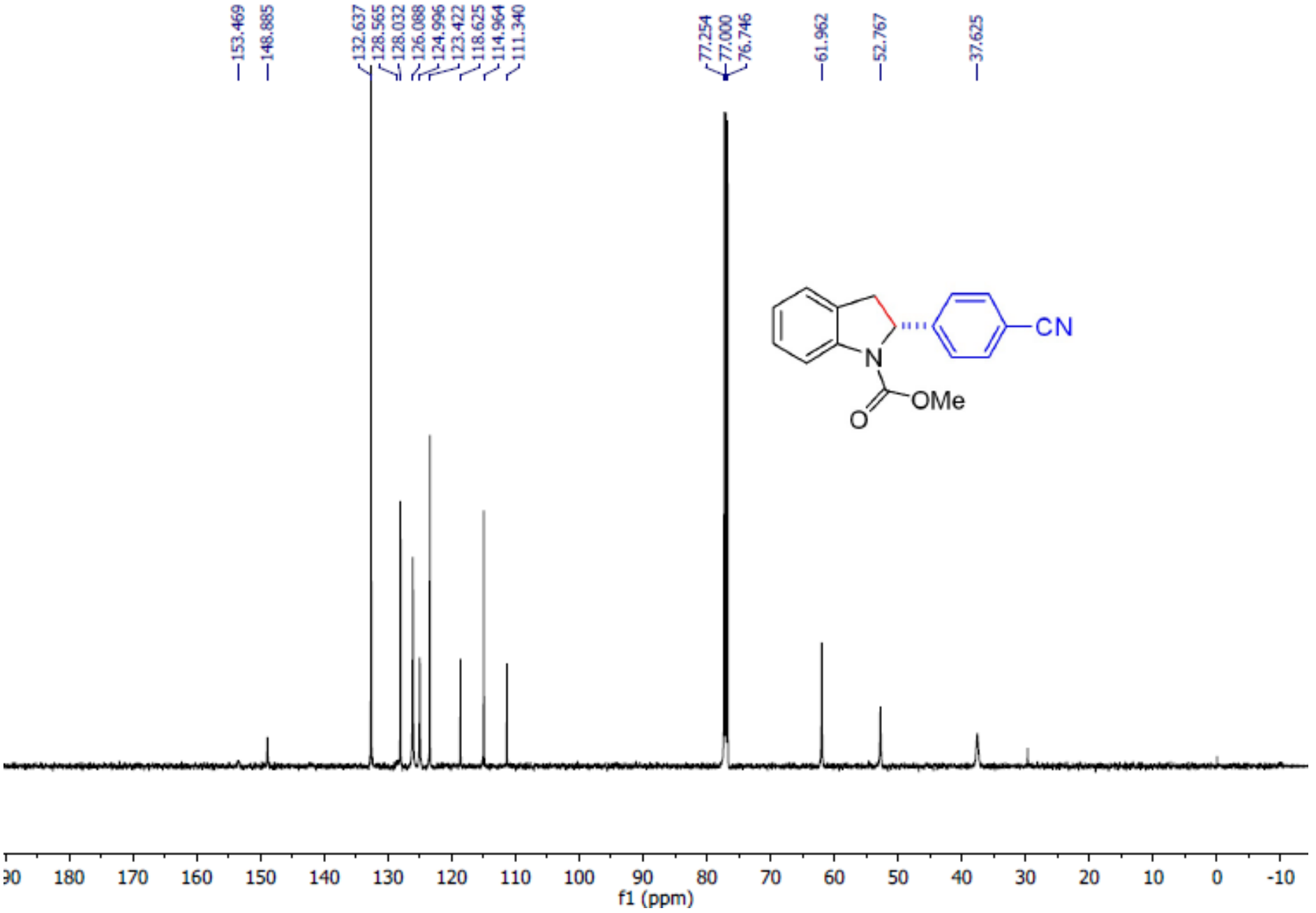
PDA Ch1 266nm

Peak#	Ret. Time	Area	Area%
1	27.953	5923290	93.377
2	34.665	420091	6.623
Total		6343381	100.000

methyl (*R*)-2-(4-cyanophenyl)indoline-1-carboxylate 2j



methyl (*R*)-2-(4-cyanophenyl)indoline-1-carboxylate 2j

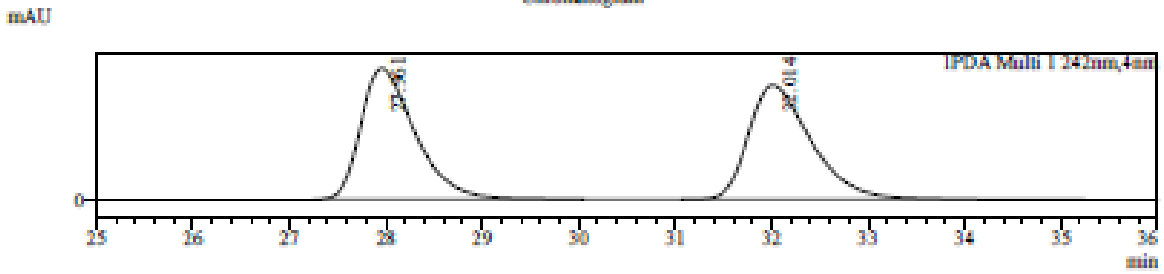


methyl (*R*)-2-(4-cyanophenyl)indoline-1-carboxylate 2j

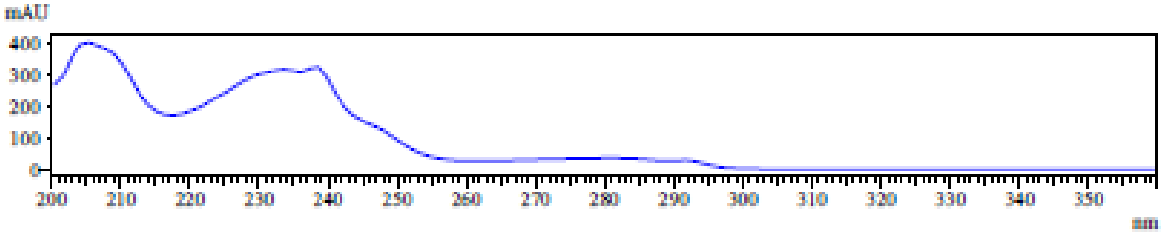
Sample Information

Sample Name	: XW-V-19-IA-2%1.0mL
Sample ID	: XW-V-19-IA-2%1.0mL
Data File	: XW-V-19-IA-2%1.0mL.lcd
Method File	: XW-2%-1ml.lcm

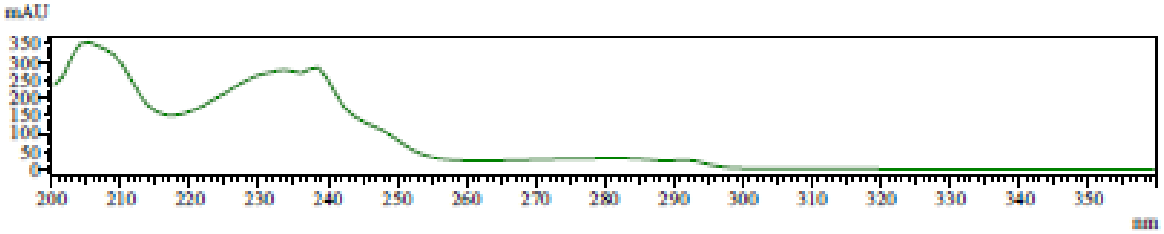
Chromatogram



UV Spectrum
Retention time = 27.961



UV Spectrum
Retention time = 32.014



Peak Table

PDA Ch1 242nm

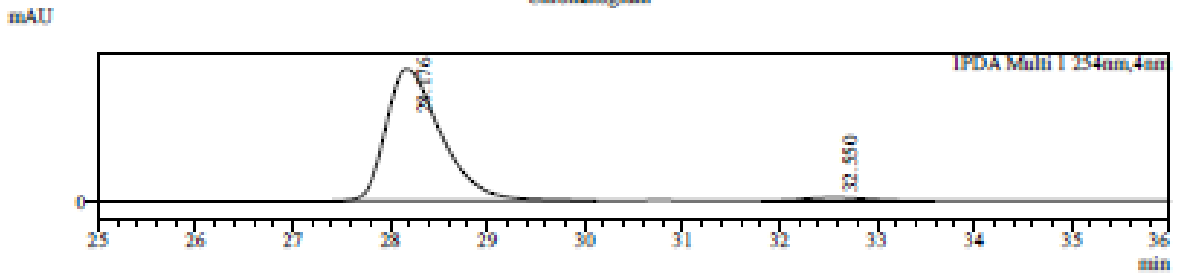
Peak#	Ret. Time	Area	Area%
1	27.961	9127467	49.823
2	32.014	9192143	50.177
Total		18319611	100.000

methyl (*R*)-2-(4-cyanophenyl)indoline-1-carboxylate 2j

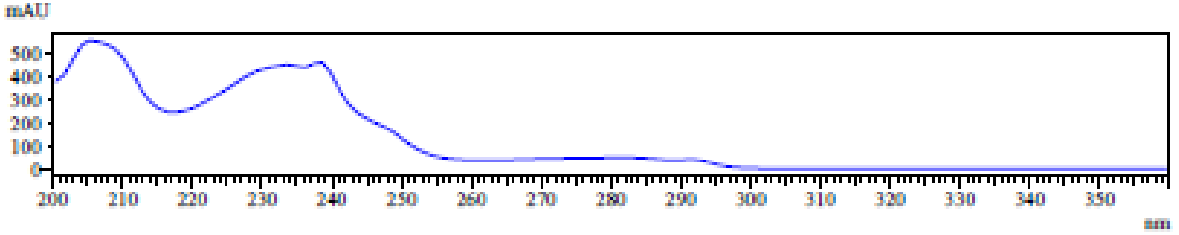
Sample Information

Sample Name	: XW-V-20-new-IA-2%1.0mL
Sample ID	: XW-V-20-new-IA-2%1.0mL
Data File	: XW-V-20-new-IA-2%1.0mL.lcd
Method File	: XW-2%-1ml.lcm

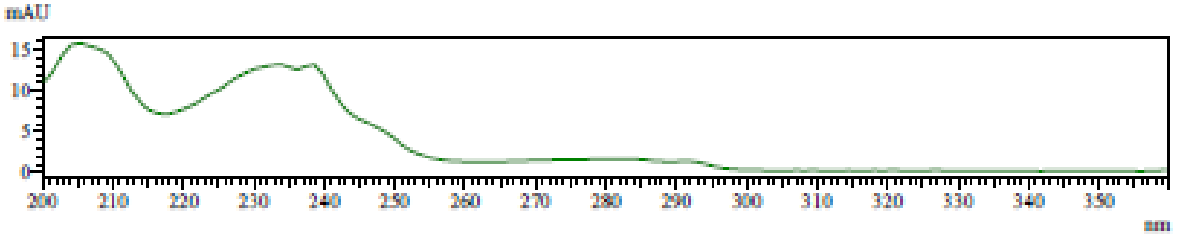
Chromatogram



UV Spectrum
Retention time = 28.176



UV Spectrum
Retention time = 32.550

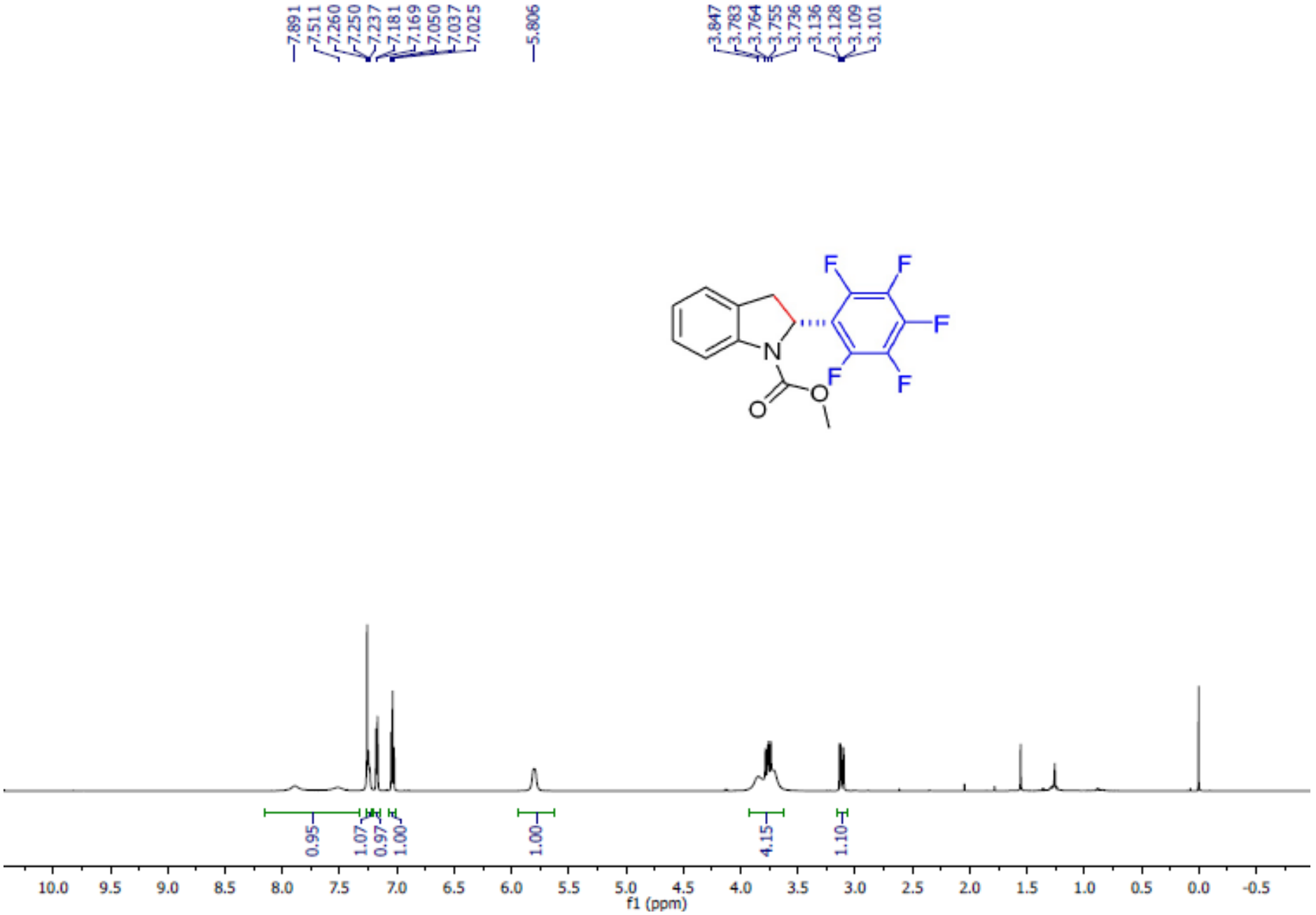


Peak Table

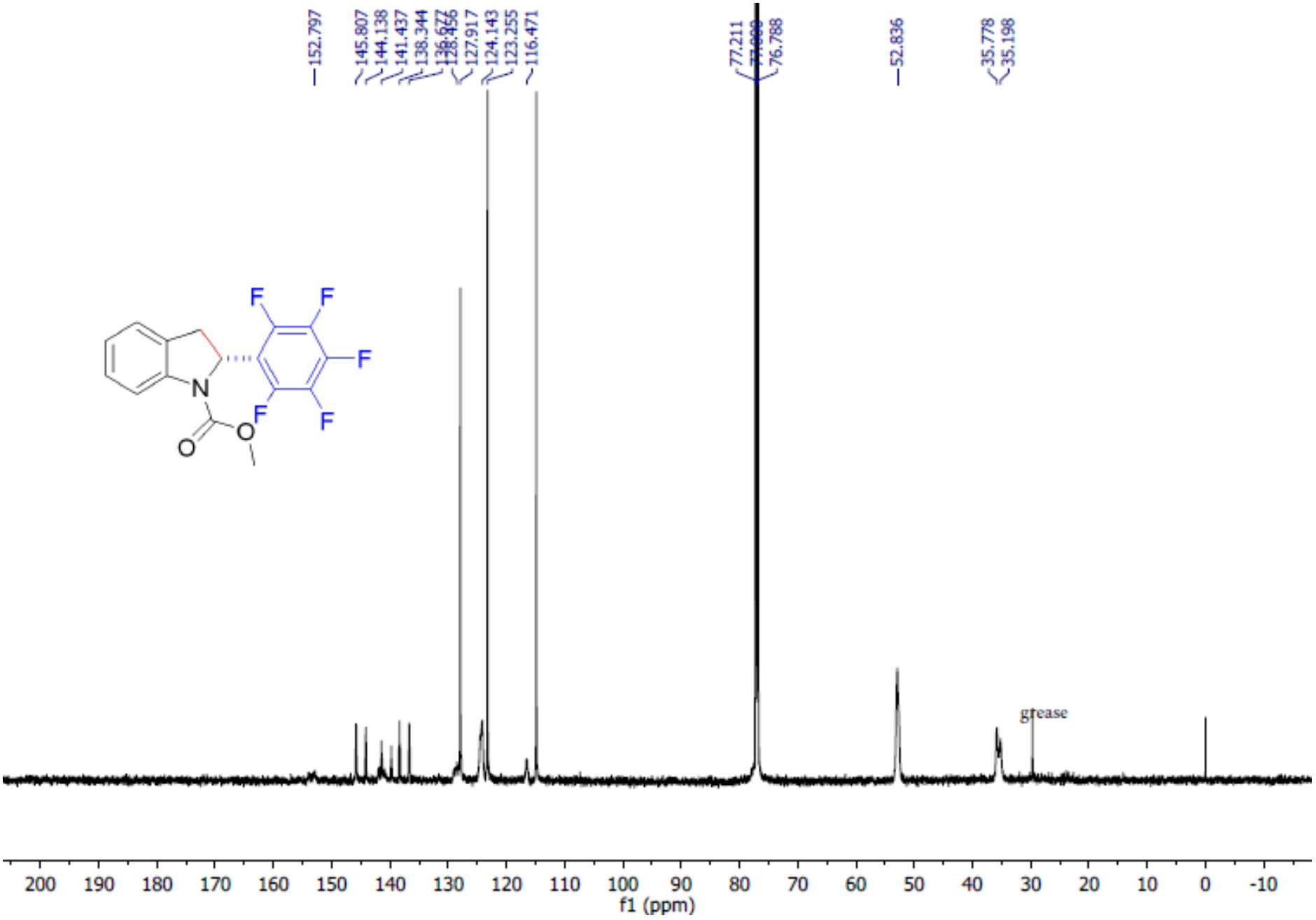
PDA Ch1 254nm

Peak#	Ret. Time	Area	Area%
1	28.176	2751841	97.097
2	32.550	82286	2.903
Total		2834127	100.000

methyl (*R*)-2-(perfluorophenyl)indoline-1-carboxylate 2k



methyl (*R*)-2-(perfluorophenyl)indoline-1-carboxylate 2k

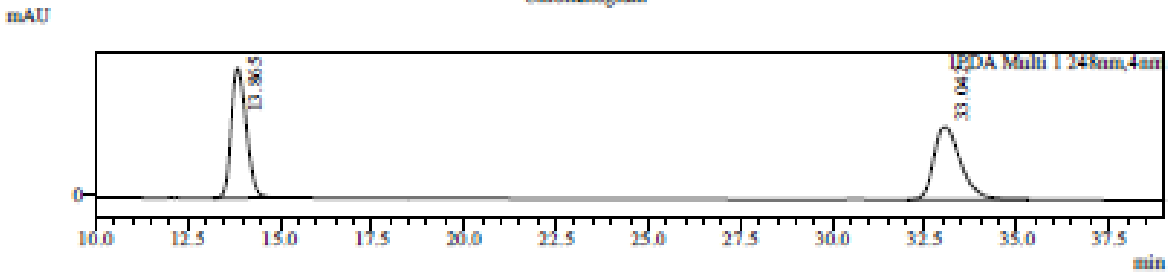


methyl (*R*)-2-(perfluorophenyl)indoline-1-carboxylate 2k

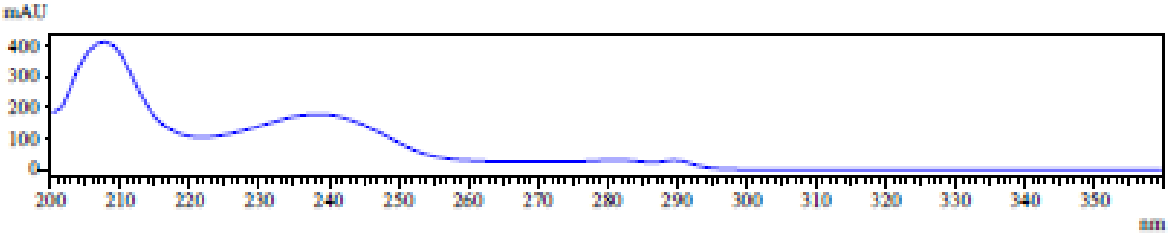
Sample Information

Sample Name	: XW-V-103-IA-0.5%0.8mL
Sample ID	: XW-V-103-IA-0.5%0.8mL
Data File	: XW-V-103-IA-0.5%0.8mL.lcd
Method File	: XW-0.5%-0.8ml.lcm

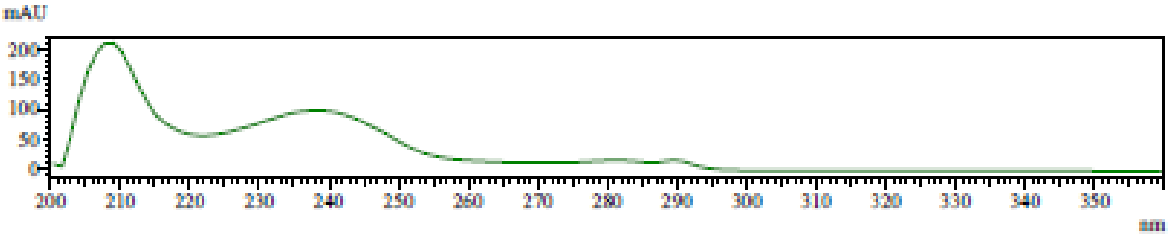
Chromatogram



UV Spectrum
Retention time = 13.865



UV Spectrum
Retention time = 33.042



Peak Table

PDA Ch1 248nm

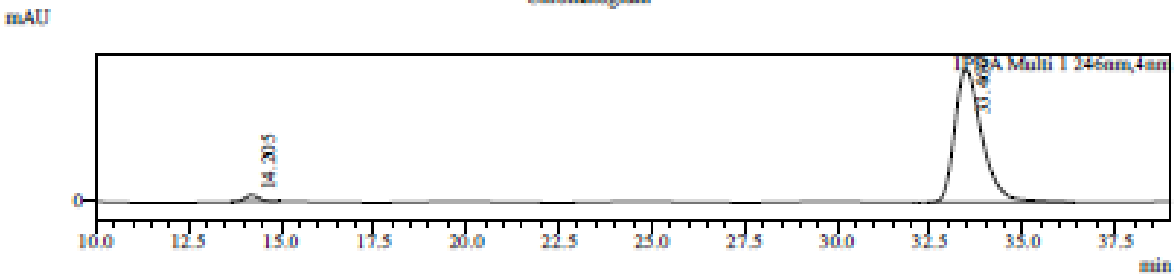
Peak#	Ret. Time	Area	Area%
1	13.865	3220132	50.366
2	33.042	3173293	49.634
Total		6393424	100.000

methyl (*R*)-2-(perfluorophenyl)indoline-1-carboxylate 2k

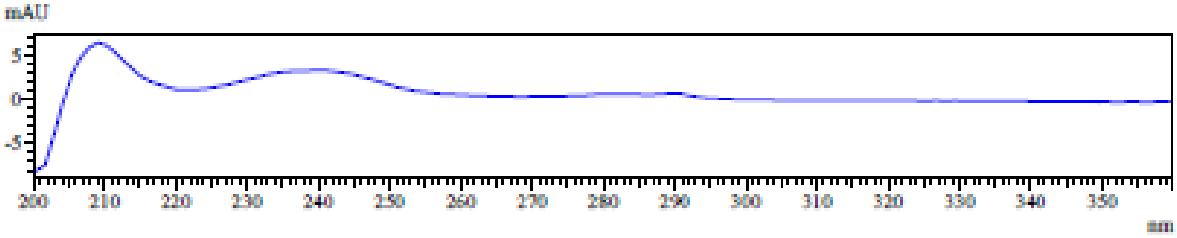
Sample Information

Sample Name	: XW-V-104-old-IA-0.5%0.8mL
Sample ID	: XW-V-104-old-IA-0.5%0.8mL
Data File	: XW-V-104-old-IA-0.5%0.8mL.lcd
Method File	: XW-0.5%-0.8ml.lcm

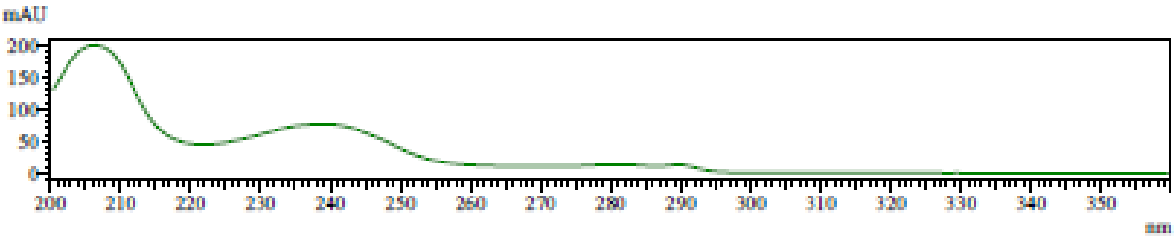
Chromatogram



UV Spectrum
Retention time = 14.205



UV Spectrum
Retention time = 33.466

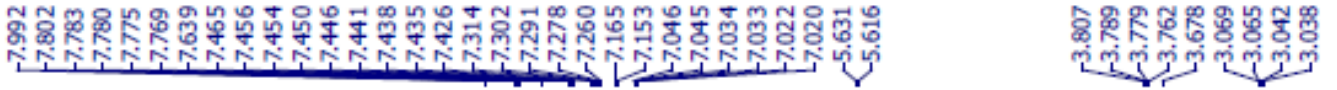


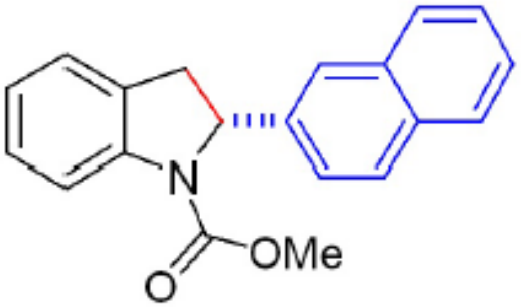
Peak Table

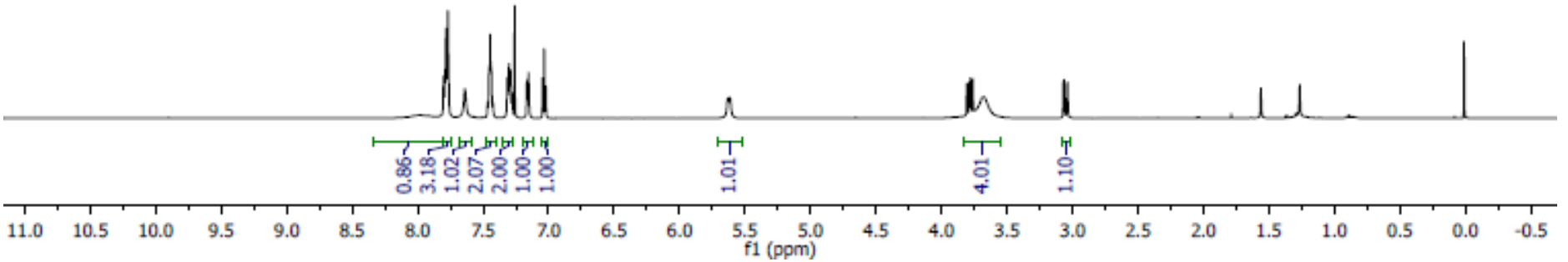
PDA Ch1 246nm

Peak#	Ret. Time	Area	Area%
1	14.205	79148	2.588
2	33.466	2979528	97.412
Total		3058675	100.000

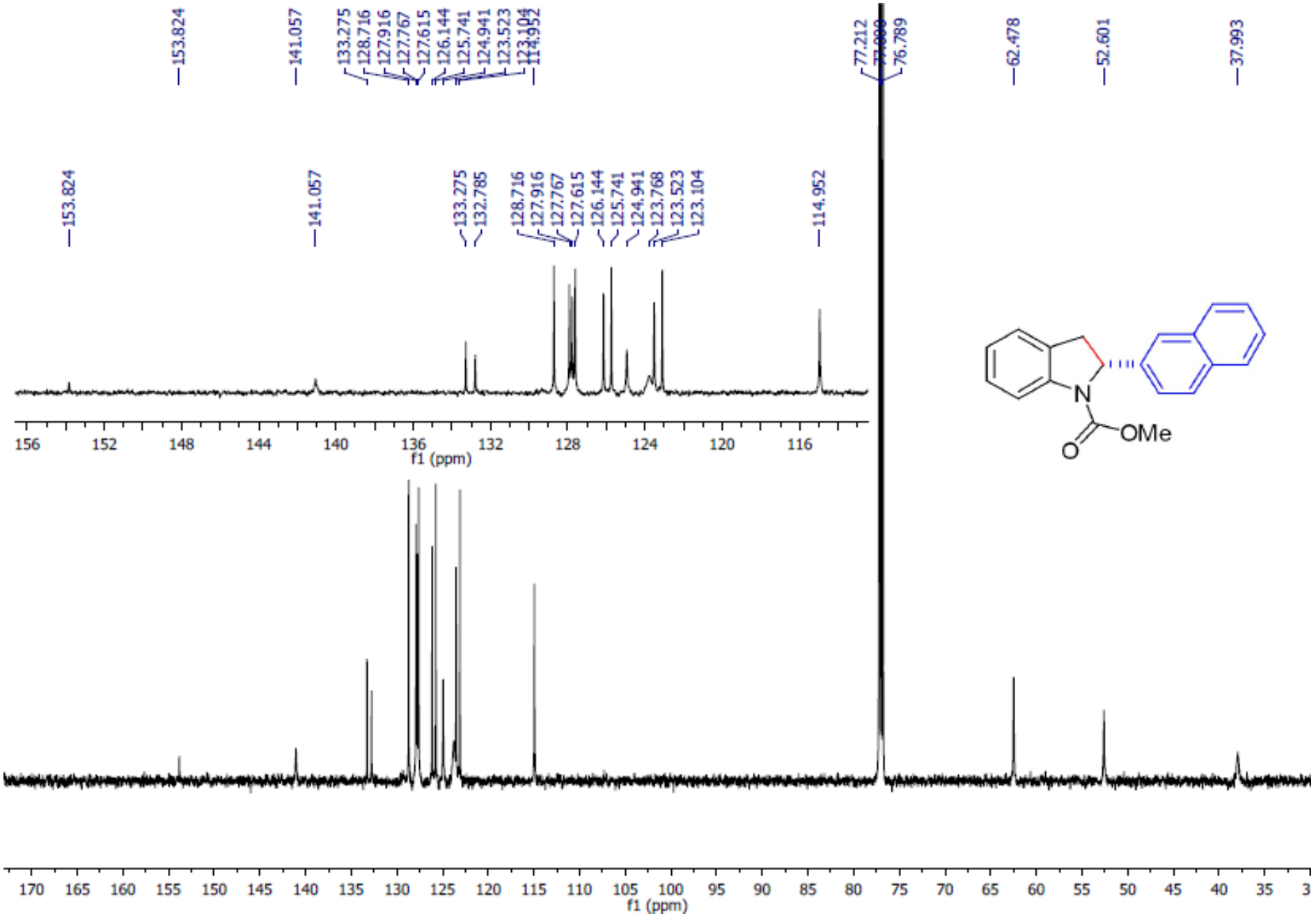
methyl (*R*)-2-(naphthalen-2-yl)indoline-1-carboxylate 2l







methyl (*R*)-2-(naphthalen-2-yl)indoline-1-carboxylate 2l

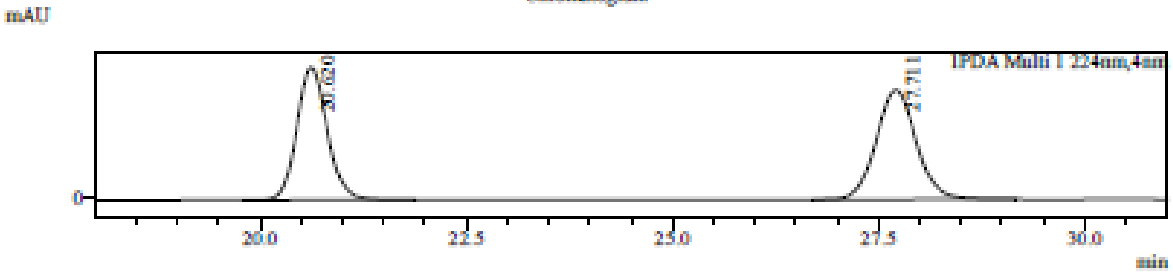


methyl (*R*)-2-(naphthalen-2-yl)indoline-1-carboxylate 2l

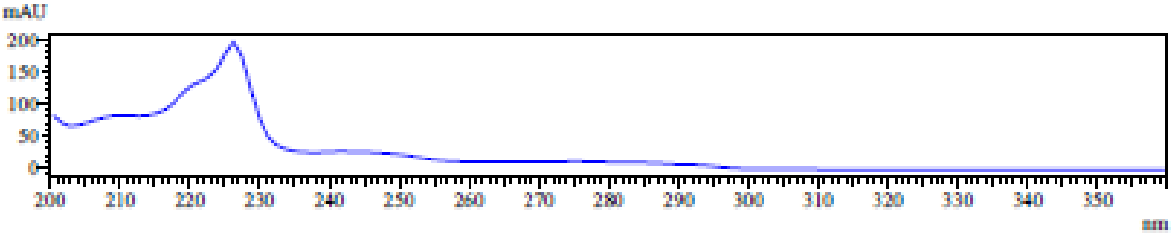
Sample Information

Sample Name	: XW-V-98-1-IA-1%0.8mL
Sample ID	: XW-V-98-1-IA-1%0.8mL
Data File	: XW-V-98-1-IA-1%0.8mL.lcd
Method File	: XW-1%-0.8ml.lcm

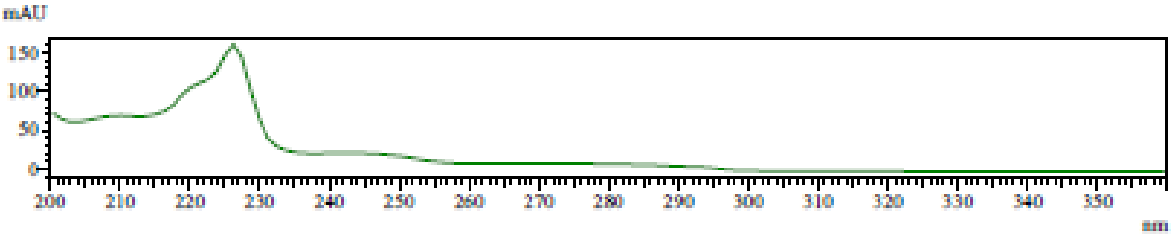
Chromatogram



UV Spectrum
Retention time = 20.620



UV Spectrum
Retention time = 27.711



Peak Table

PDA Ch1 224nm

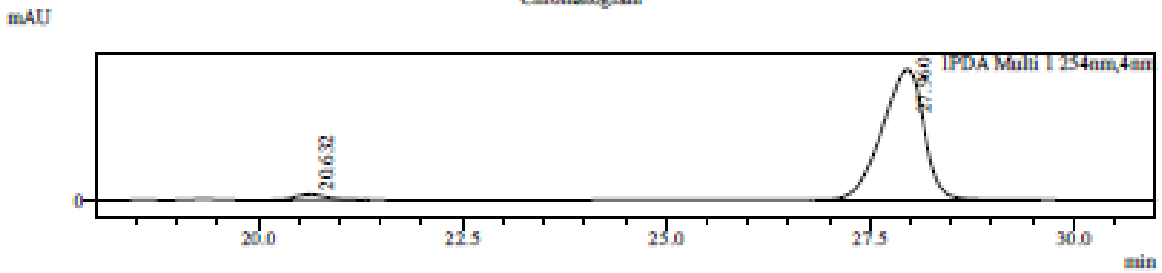
Peak#	Ret. Time	Area	Area%
1	20.620	4098843	48.021
2	27.711	4436621	51.979
Total		8535464	100.000

methyl (*R*)-2-(naphthalen-2-yl)indoline-1-carboxylate 2l

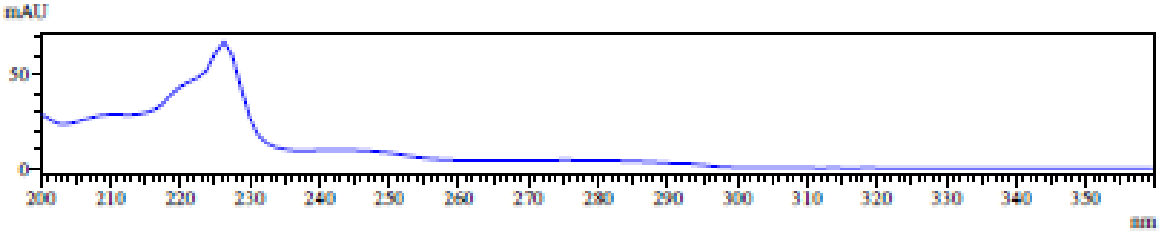
Sample Information

Sample Name	: XW-V-99-Old-IA-1%0.8mL
Sample ID	: XW-V-99-Old-IA-1%0.8mL
Data File	: XW-V-99-Old-IA-1%0.8mL.lcd
Method File	: XW-1%-0.8ml.lcm

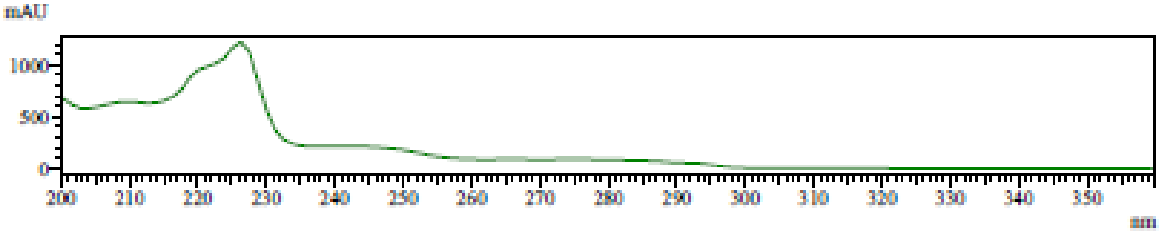
Chromatogram



UV Spectrum
Retention time = 20.632



UV Spectrum
Retention time = 27.960

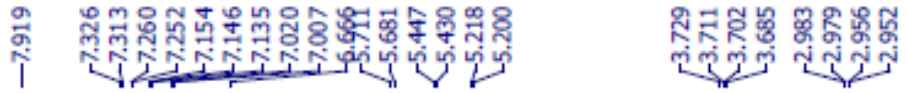


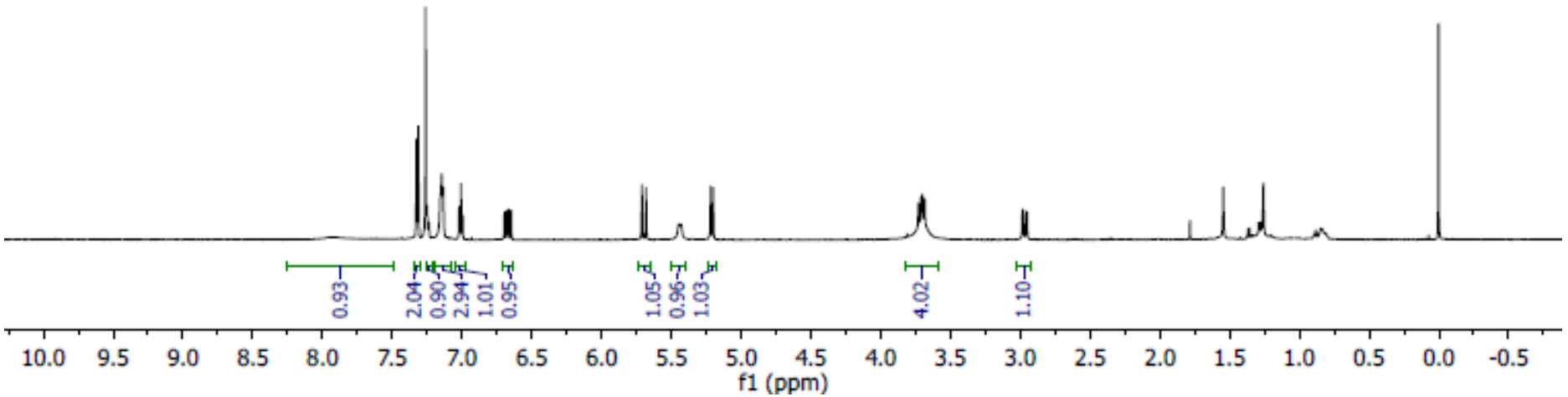
Peak Table

PDA Ch1 254nm

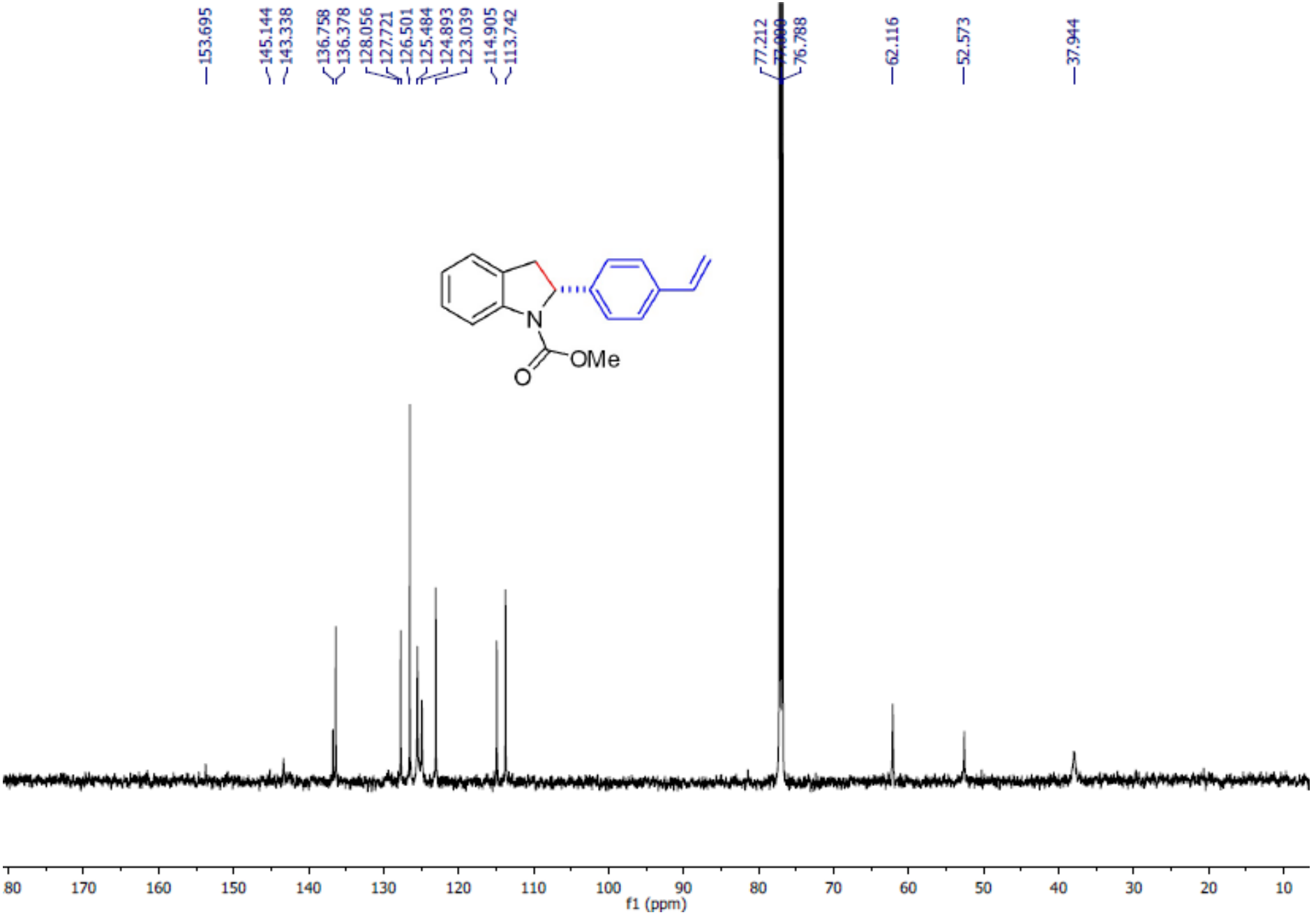
Peak#	Ret. Time	Area	Area%
1	20.632	145509	2.999
2	27.960	4706561	97.001
Total		4852070	100.000

methyl (*R*)-2-(4-vinylphenyl)indoline-1-carboxylate 2m





methyl (*R*)-2-(4-vinylphenyl)indoline-1-carboxylate 2m

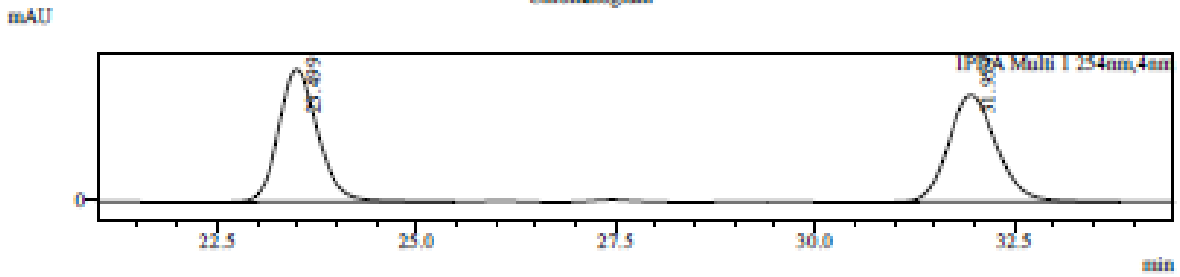


methyl (*R*)-2-(4-vinylphenyl)indoline-1-carboxylate 2m

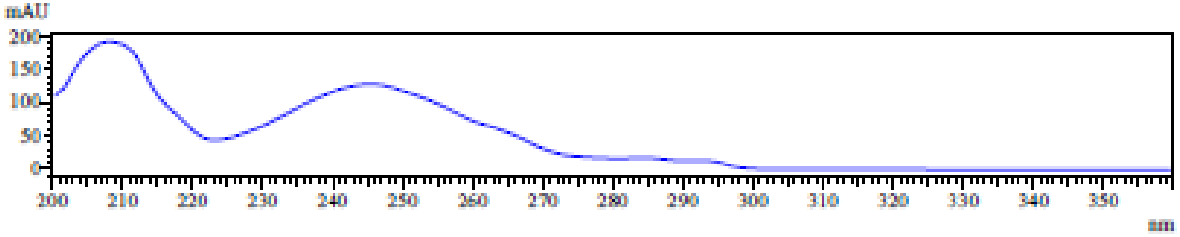
Sample Information

Sample Name	: XW-V-88-IA-0.5%0.8mL
Sample ID	: XW-V-88-IA-0.5%0.8mL
Data File	: XW-V-88-IA-0.5%0.8mL.lcd
Method File	: XW-0.5%-0.8ml.lcm

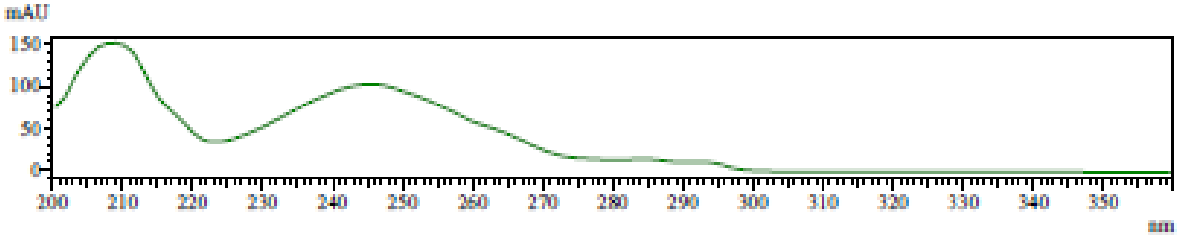
Chromatogram



UV Spectrum
Retention time = 23.499



UV Spectrum
Retention time = 31.959



Peak Table

PDA Ch1 254nm

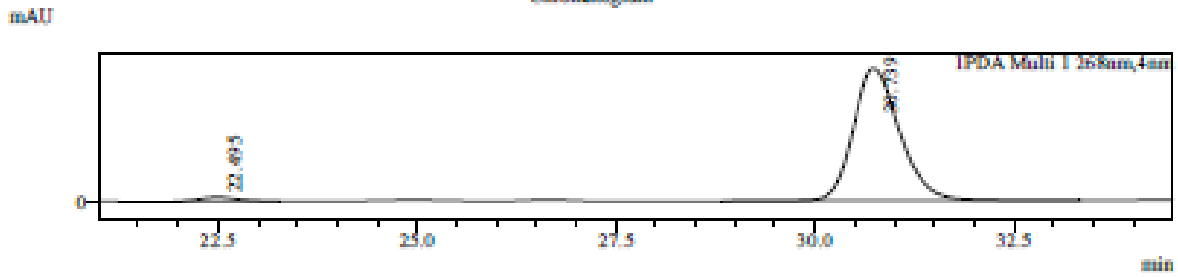
Peak#	Ret. Time	Area	Area%
1	23.499	3519407	50.275
2	31.959	3480837	49.725
Total		7000243	100.000

methyl (*R*)-2-(4-vinylphenyl)indoline-1-carboxylate 2m

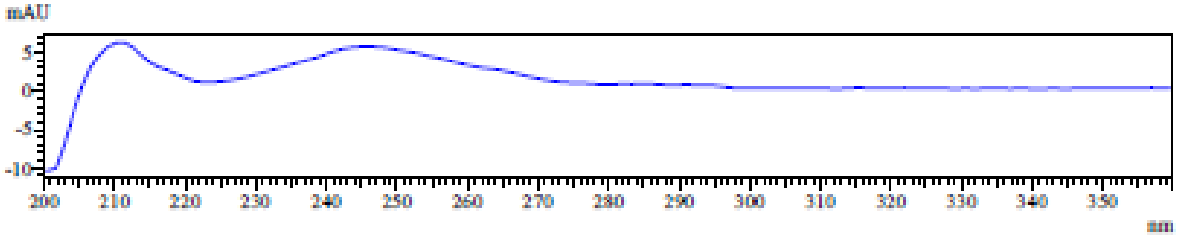
Sample Information

Sample Name	: XW-V-89-New-batch-IA-0.5%0.8mL
Sample ID	: XW-V-89-New-batch-IA-0.5%0.8mL
Data File	: XW-V-89-New-batch-IA-0.5%0.8mL.lcd
Method File	: XW-0.5%-0.8ml.lcm

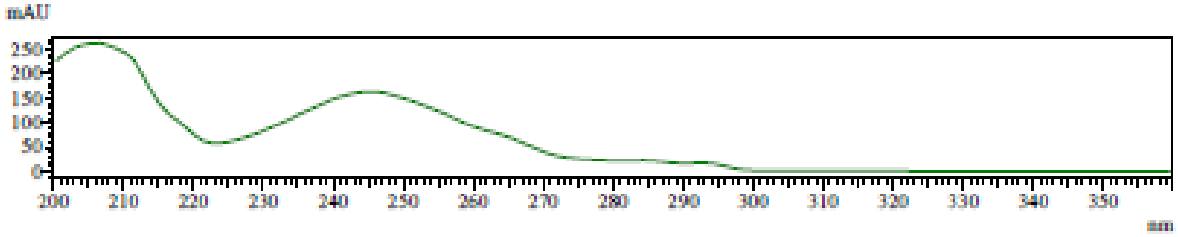
Chromatogram



UV Spectrum
Retention time = 22.495



UV Spectrum
Retention time = 30.739

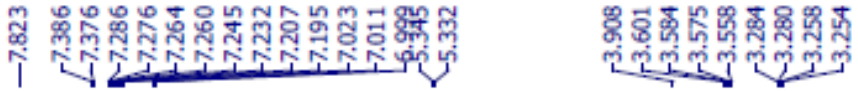


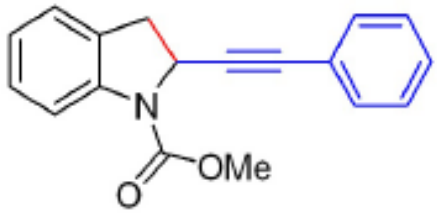
Peak Table

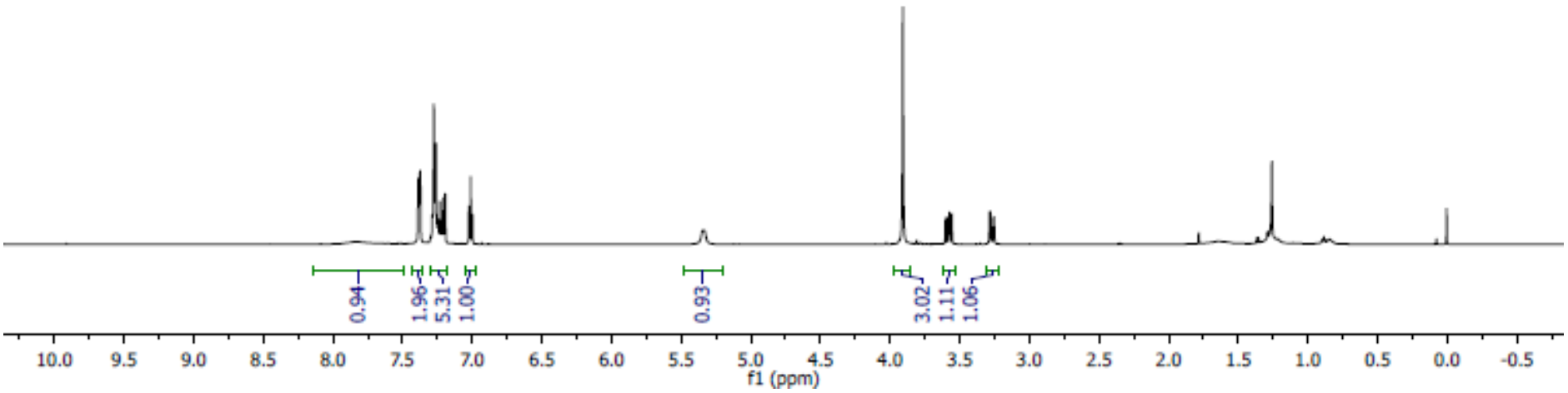
PDA Ch1 268nm

Peak#	Ret. Time	Area	Area%
1	22.495	62384	2.698
2	30.739	2250109	97.302
Total		2312493	100.000

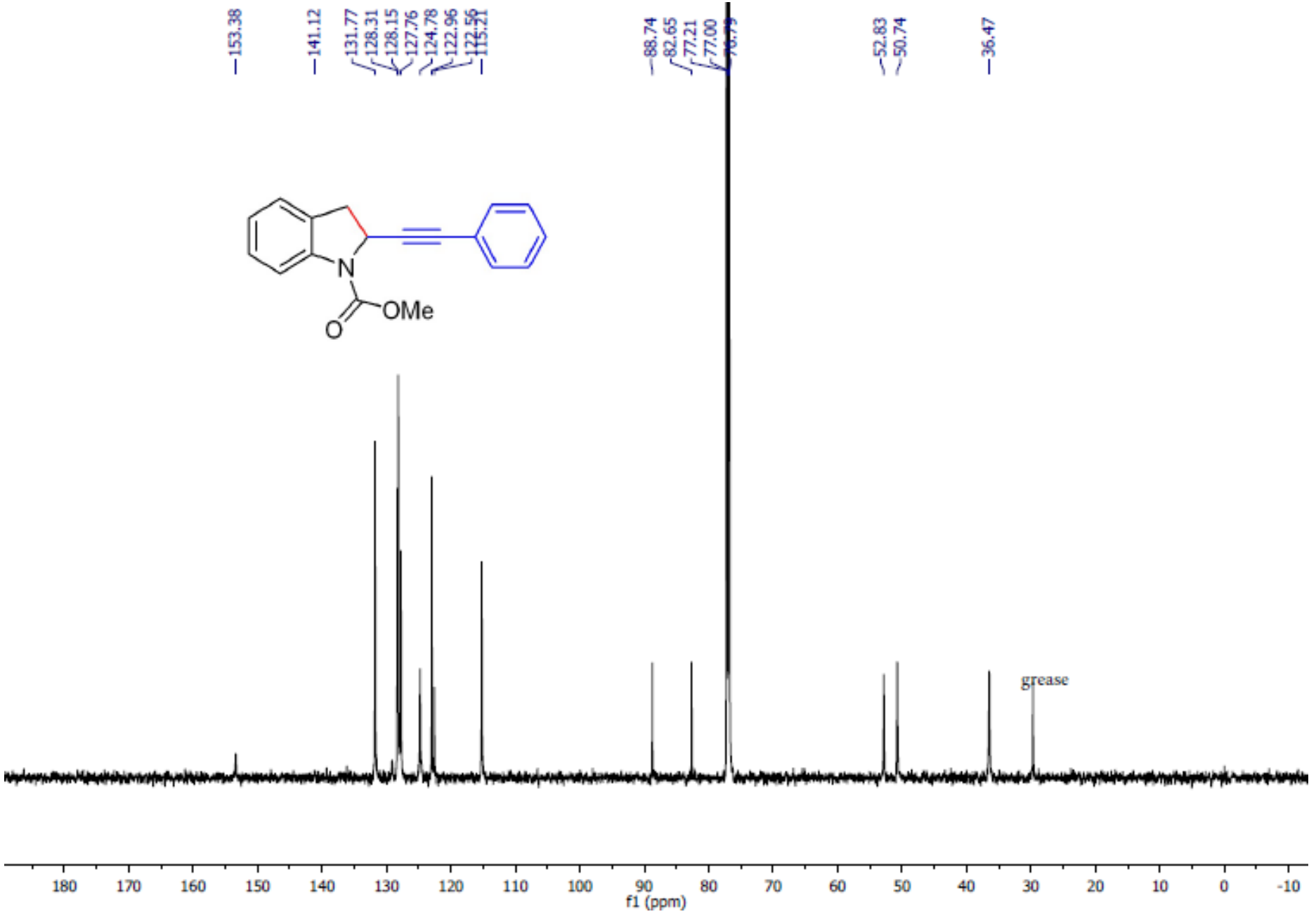
methyl (R)-2-(phenylethynyl)indoline-1-carboxylate 2n







methyl (R)-2-(phenylethynyl)indoline-1-carboxylate 2n

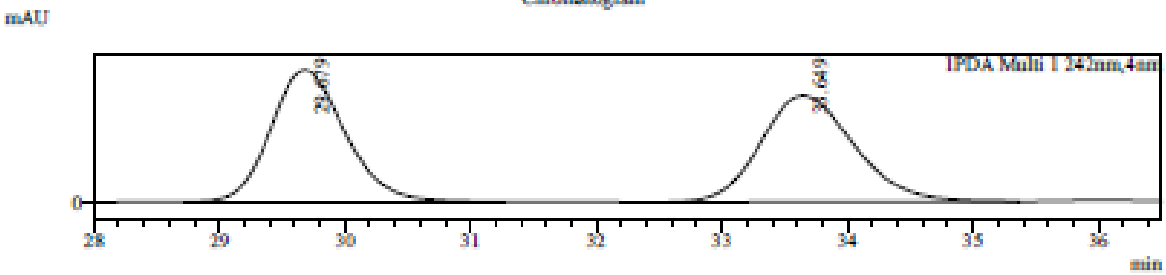


methyl (R)-2-(phenylethynyl)indoline-1-carboxylate 2n

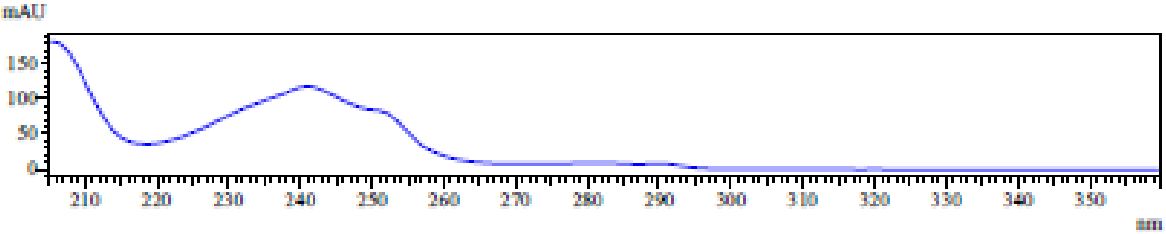
Sample Information

Sample Name	: XW-V-132-IC-1%0.8mL
Sample ID	: XW-V-132-IC-1%0.8mL
Data File	: XW-V-132-IC-1%0.8mL.lcd
Method File	: XW-1%-0.8ml.lcm

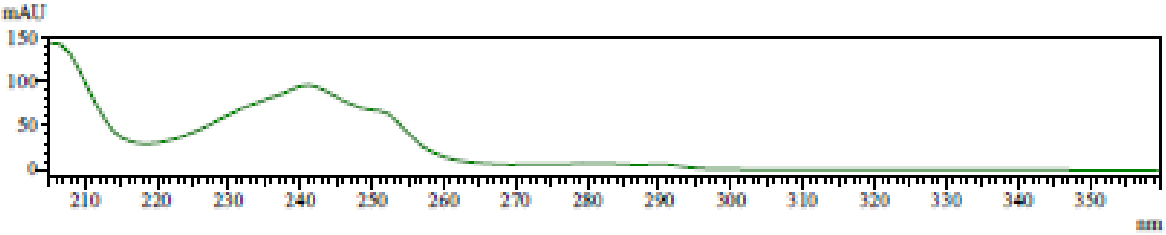
Chromatogram



UV Spectrum
Retention time = 29.679



UV Spectrum
Retention time = 33.649



Peak Table

PDA Ch1 242nm

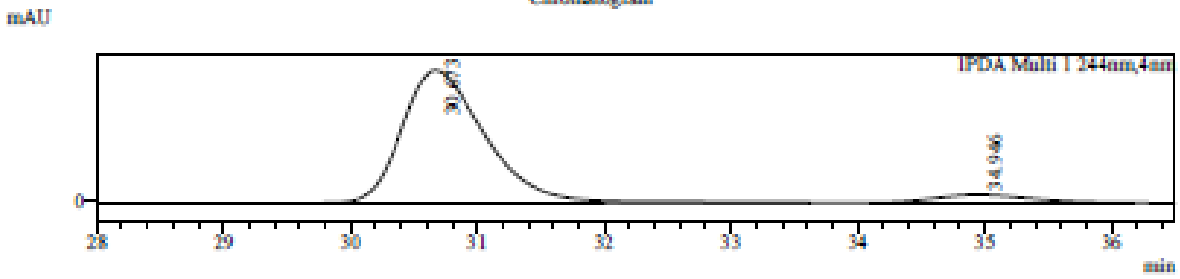
Peak#	Ret. Time	Area	Area%
1	29.679	4639557	49.555
2	33.649	4722845	50.445
Total		9362402	100.000

methyl (R)-2-(phenylethynyl)indoline-1-carboxylate 2n

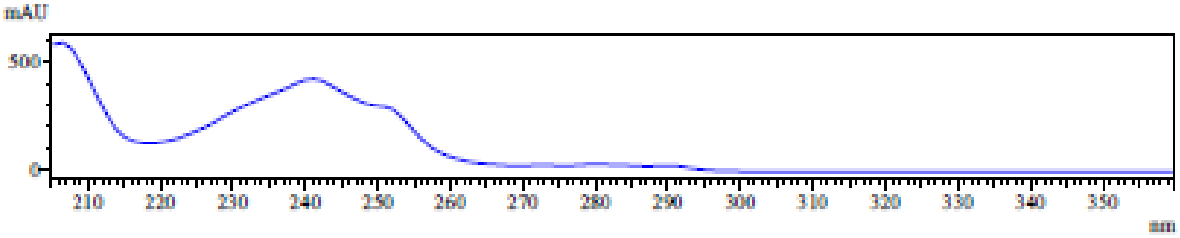
Sample Information

Sample Name	: XW-V-129B-IC-1%0.8mL
Sample ID	: XW-V-129B-IC-1%0.8mL
Data File	: XW-V-129B-IC-1%0.8mL.lcd
Method File	: XW-1%-0.8ml.lcm

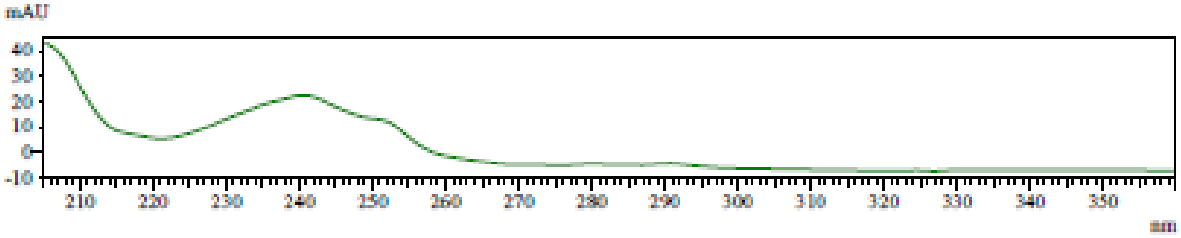
Chromatogram



UV Spectrum
Retention time = 30.673



UV Spectrum
Retention time = 34.946

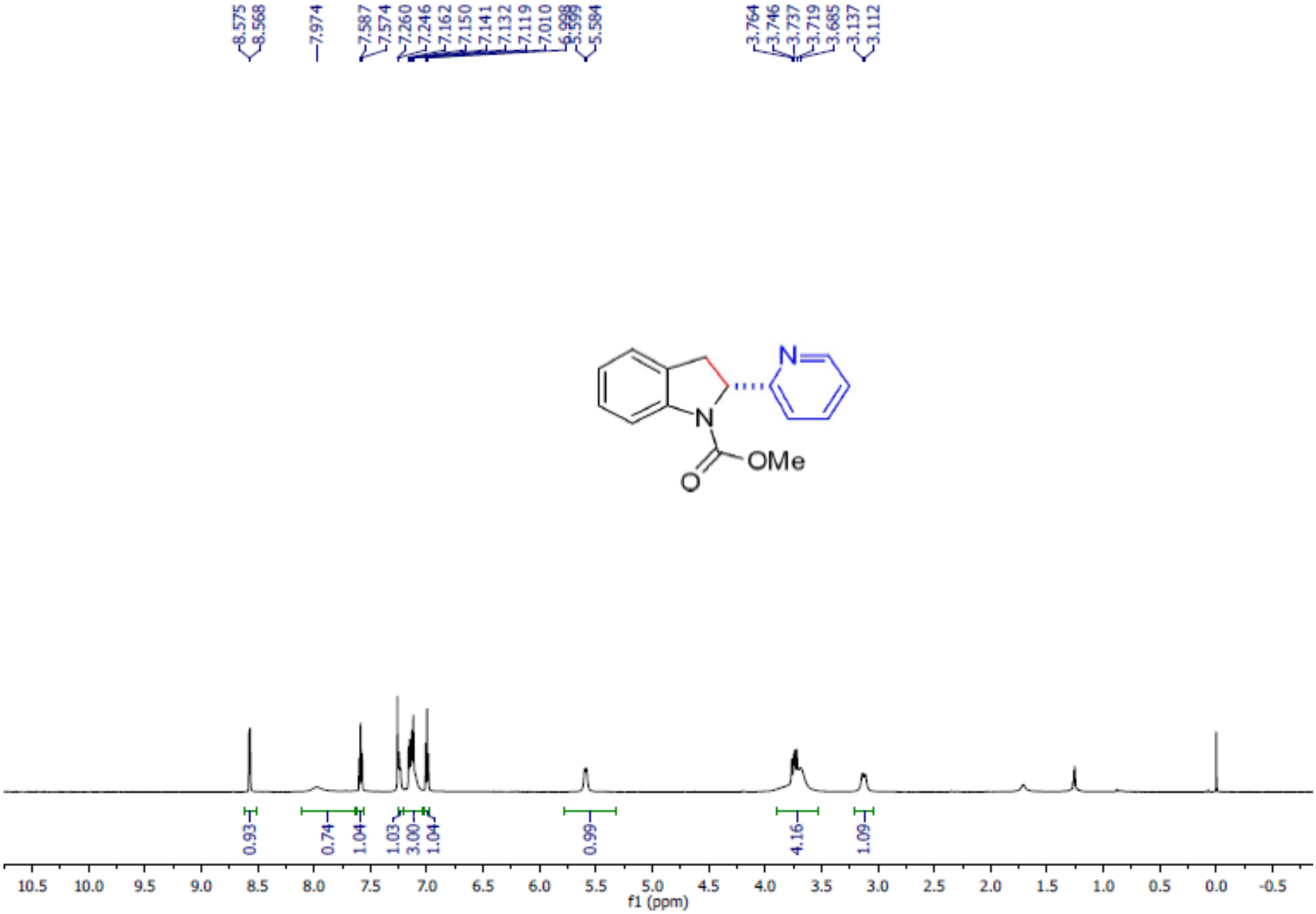


Peak Table

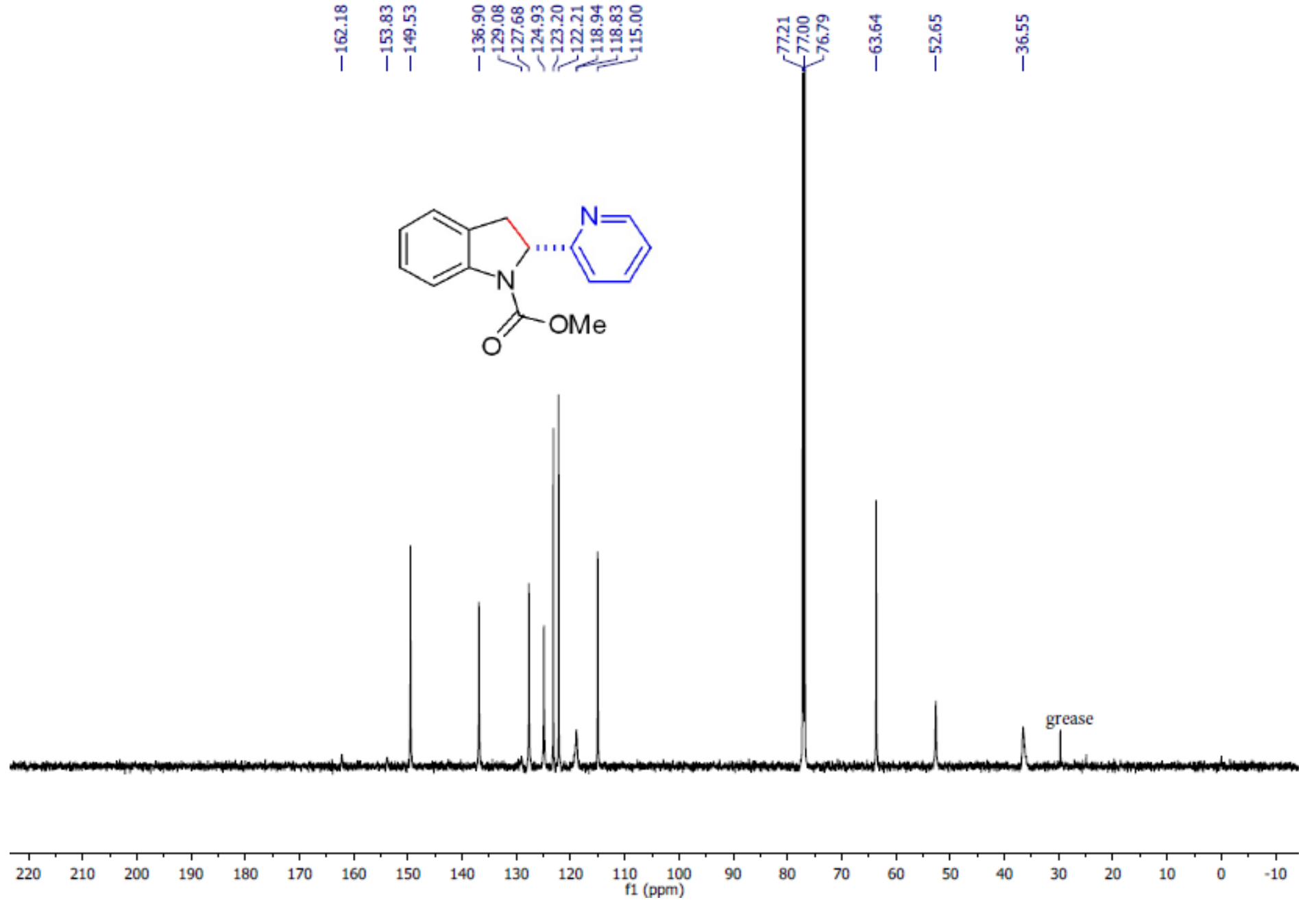
PDA Ch1 244nm

Peak#	Ret. Time	Area	Area%
1	30.673	17400671	93.483
2	34.946	1213004	6.517
Total		18613675	100.000

methyl (R)-2-(pyridin-2-yl)indoline-1-carboxylate 2o



methyl (R)-2-(pyridin-2-yl)indoline-1-carboxylate 2o

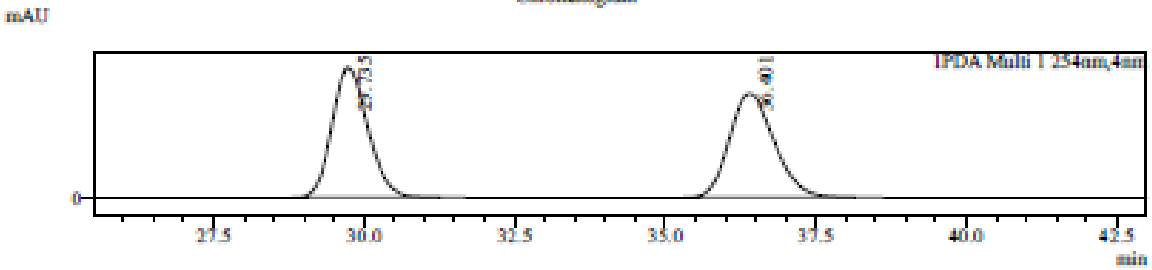


methyl (R)-2-(pyridin-2-yl)indoline-1-carboxylate 2o

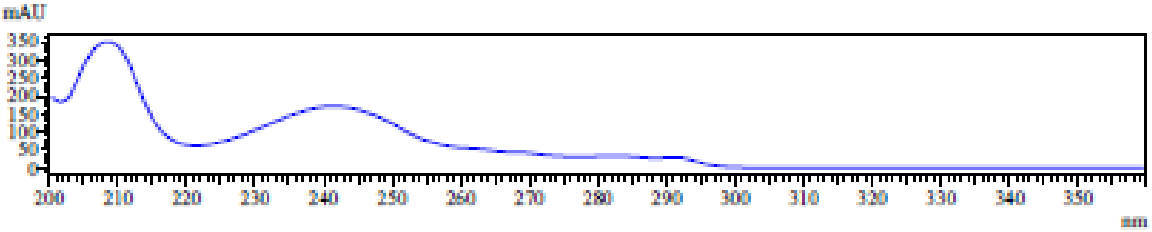
Sample Information

Sample Name	: XW-V-44-IC-5%1mL
Sample ID	: XW-V-44-IC-5%1mL
Data File	: XW-V-44-IC-5%1mL.lcd
Method File	: XW-5%-1ml.lcm

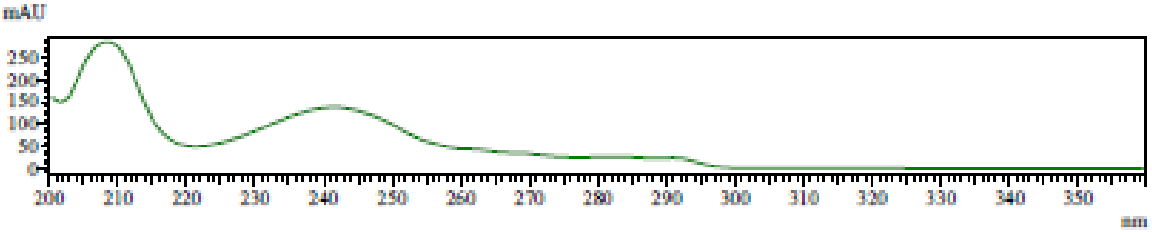
Chromatogram



UV Spectrum
Retention time = 29.735



UV Spectrum
Retention time = 36.401



Peak Table

PDA Ch1 254nm

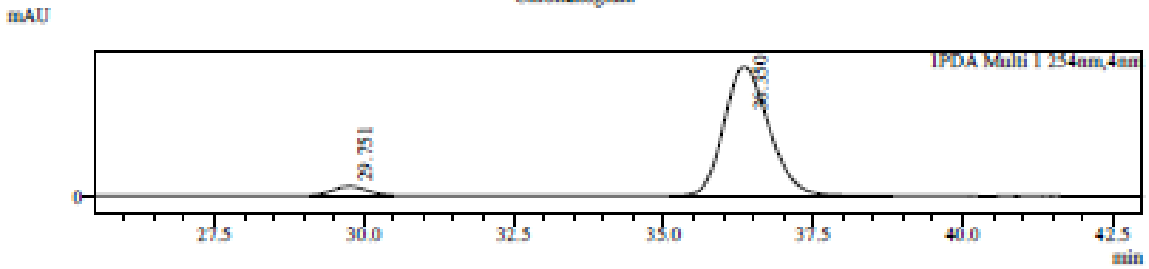
Peak#	Ret. Time	Area	Area%
1	29.735	3562726	50.034
2	36.401	3557929	49.966
Total		7120655	100.000

methyl (R)-2-(pyridin-2-yl)indoline-1-carboxylate 2o

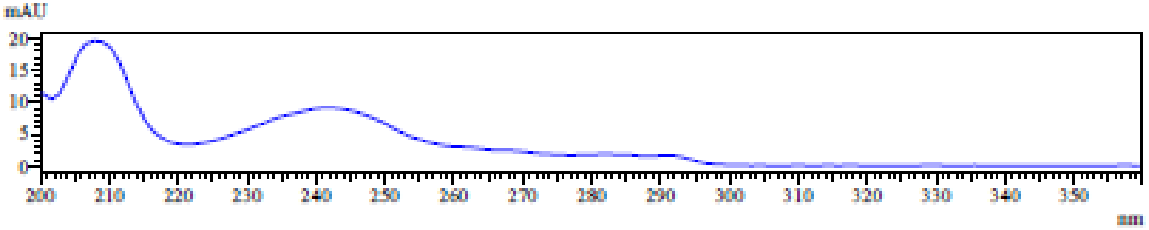
Sample Information

Sample Name	: XW-V-45B-IC-5%1mL
Sample ID	: XW-V-45B-IC-5%1mL
Data File	: XW-V-45B-IC-5%1mL.lcd
Method File	: XW-5%-1ml.lcm

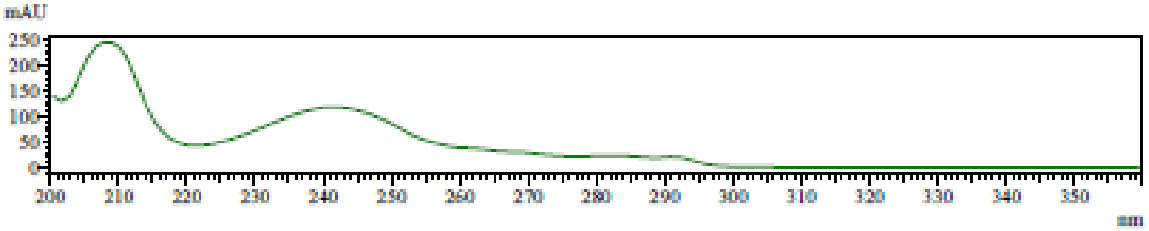
Chromatogram



UV Spectrum
Retention time = 29.751



UV Spectrum
Retention time = 36.350

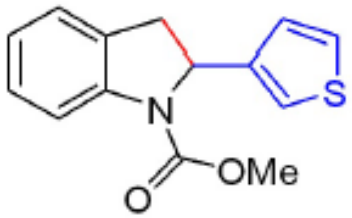


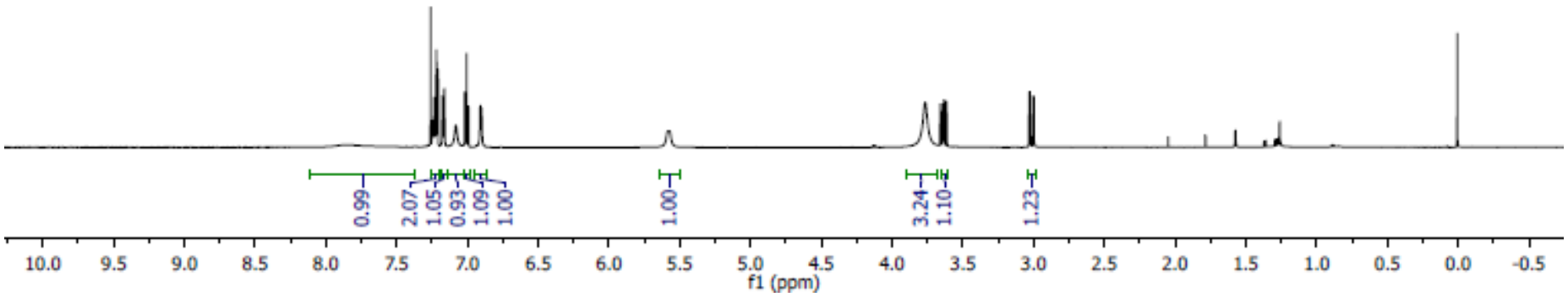
Peak Table

PDA Ch1 254nm

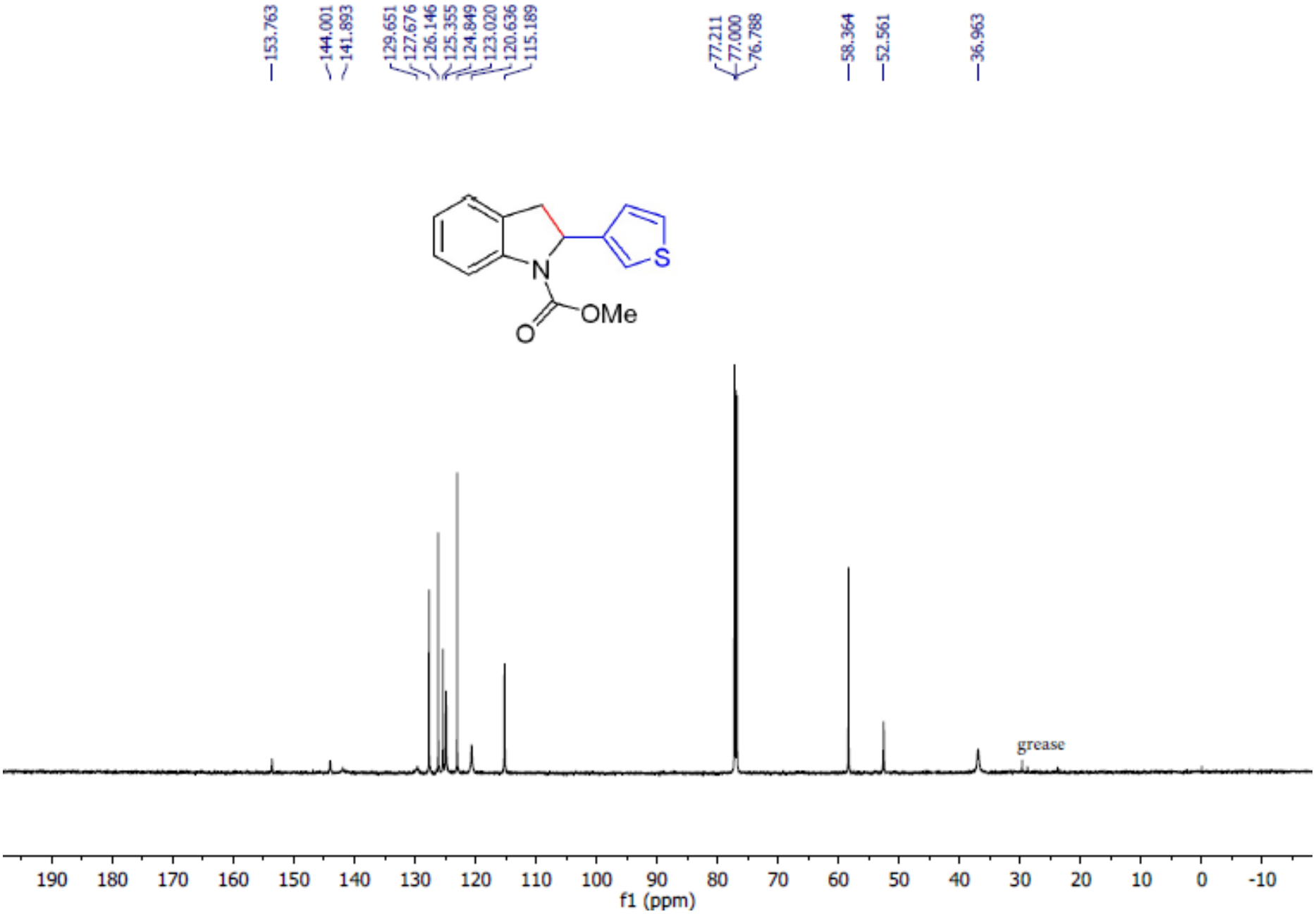
Peak#	Ret. Time	Area	Area%
1	29.751	165124	5.058
2	36.350	3099441	94.942
Total		3264565	100.000

methyl (*R*)-2-(thiophen-3-yl)indoline-1-carboxylate 2p





methyl (*R*)-2-(thiophen-3-yl)indoline-1-carboxylate 2p

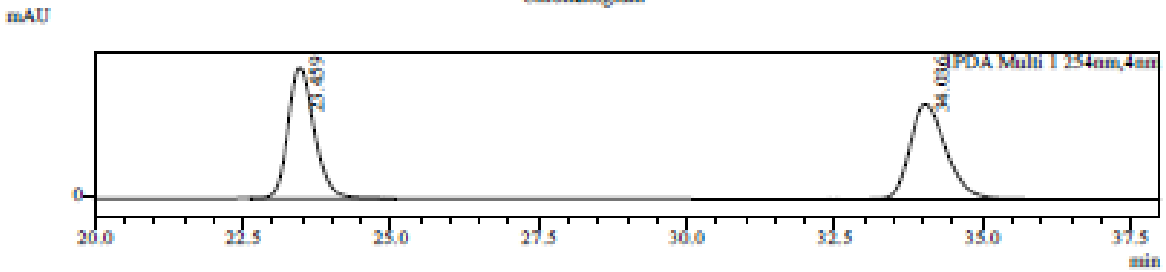


methyl (*R*)-2-(thiophen-3-yl)indoline-1-carboxylate 2p

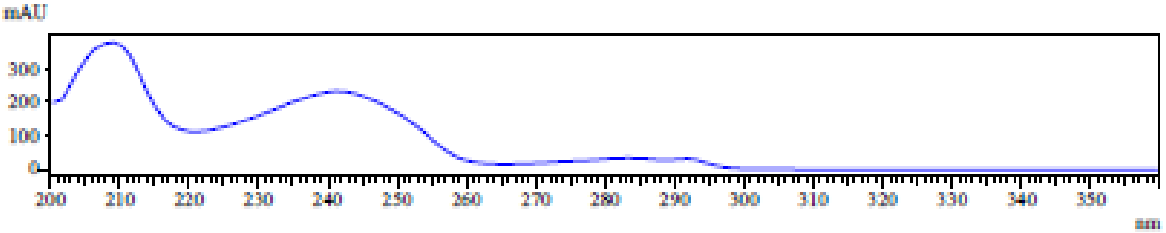
Sample Information

Sample Name	: XW-V-68-IA-0.5%0.8mL
Sample ID	: XW-V-68-IA-0.5%0.8mL
Data File	: XW-V-68-IA-0.5%0.8mL.lcd
Method File	: XW-0.5%-0.8ml.lcm

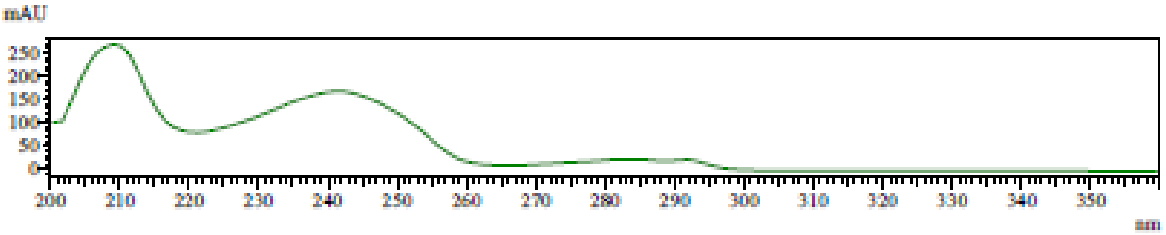
Chromatogram



UV Spectrum
Retention time = 23.459



UV Spectrum
Retention time = 34.036



Peak Table

PDA Ch1 254nm

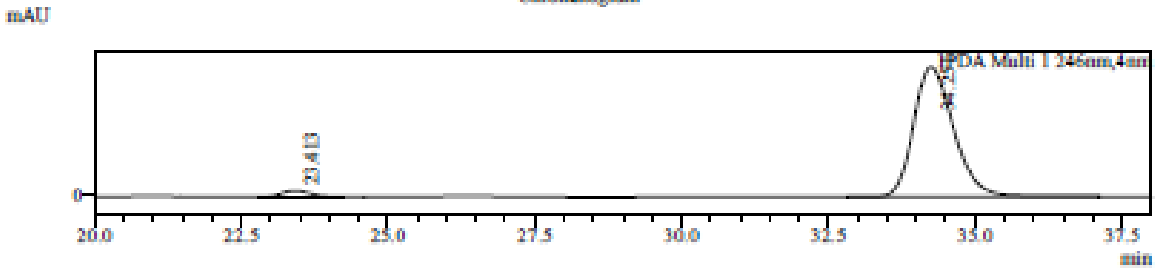
Peak#	Ret. Time	Area	Area%
1	23.459	3317129	50.079
2	34.036	3306717	49.921
Total		6623845	100.000

methyl (*R*)-2-(thiophen-3-yl)indoline-1-carboxylate 2p

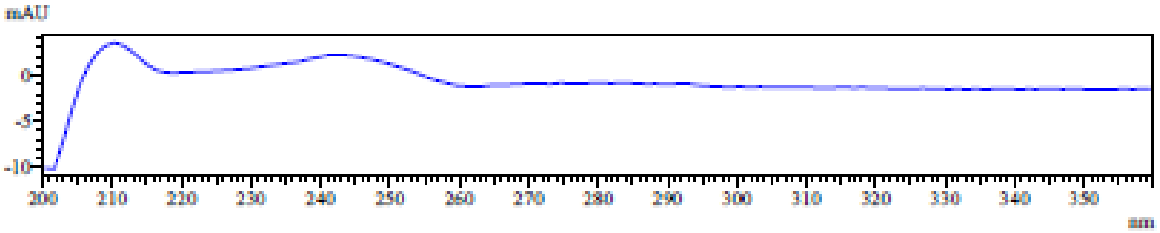
Sample Information

Sample Name	: XW-V-69B-LA-0.5%0.8mL
Sample ID	: XW-V-69B-LA-0.5%0.8mL
Data File	: XW-V-69B-LA-0.5%0.8mL.lcd
Method File	: XW-0.5%-0.8ml.lcm

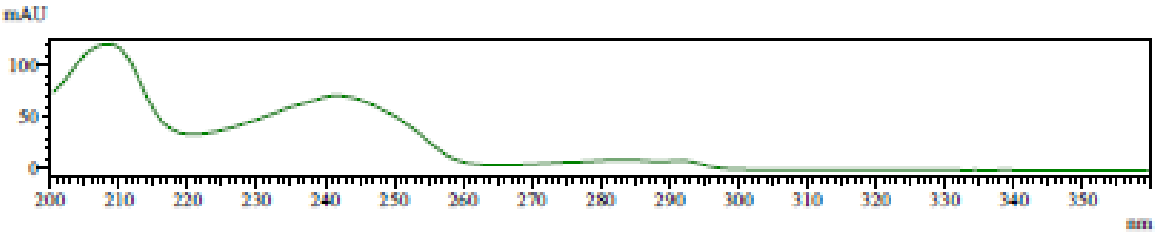
Chromatogram



UV Spectrum
Retention time = 23.413



UV Spectrum
Retention time = 34.251

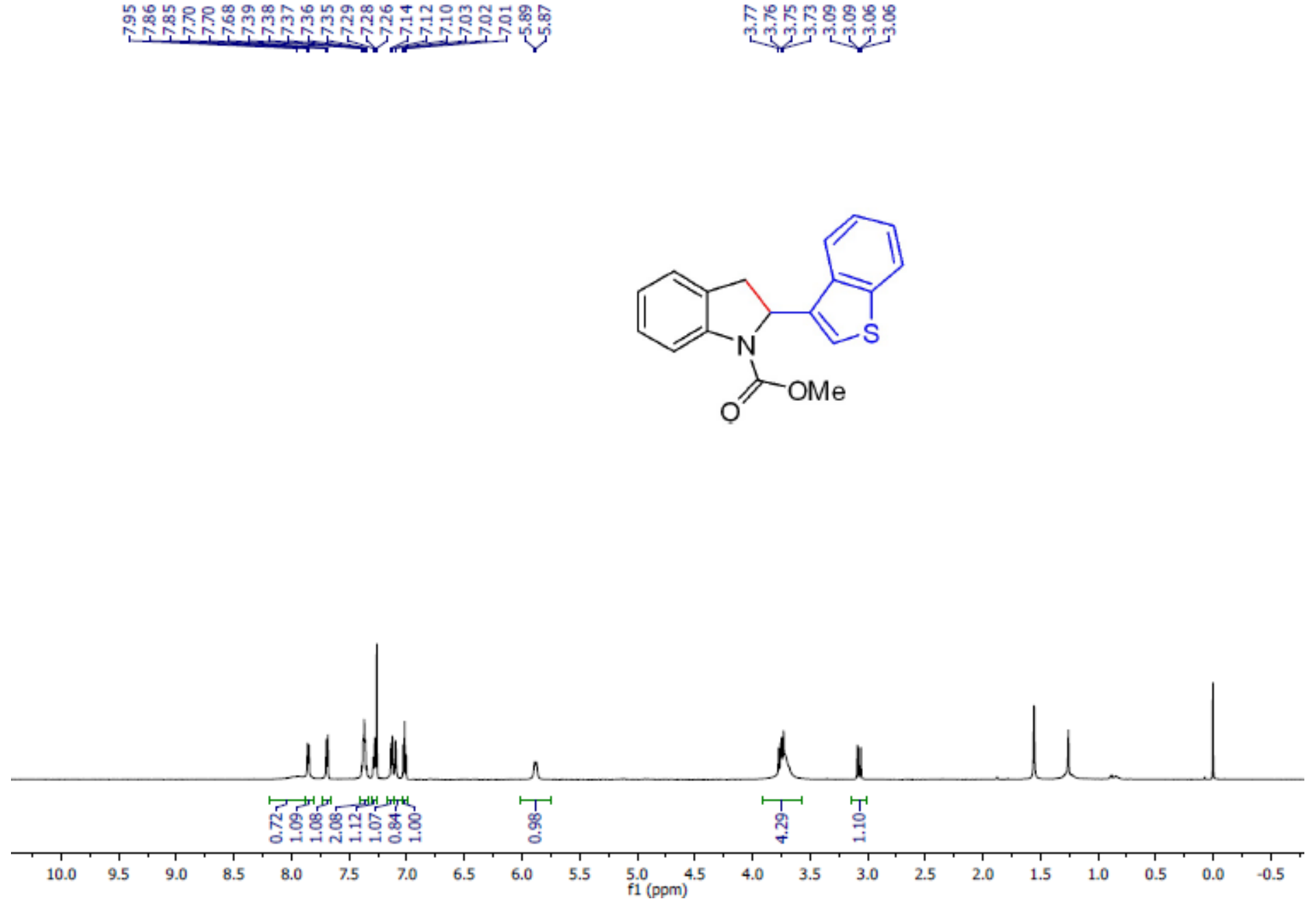


Peak Table

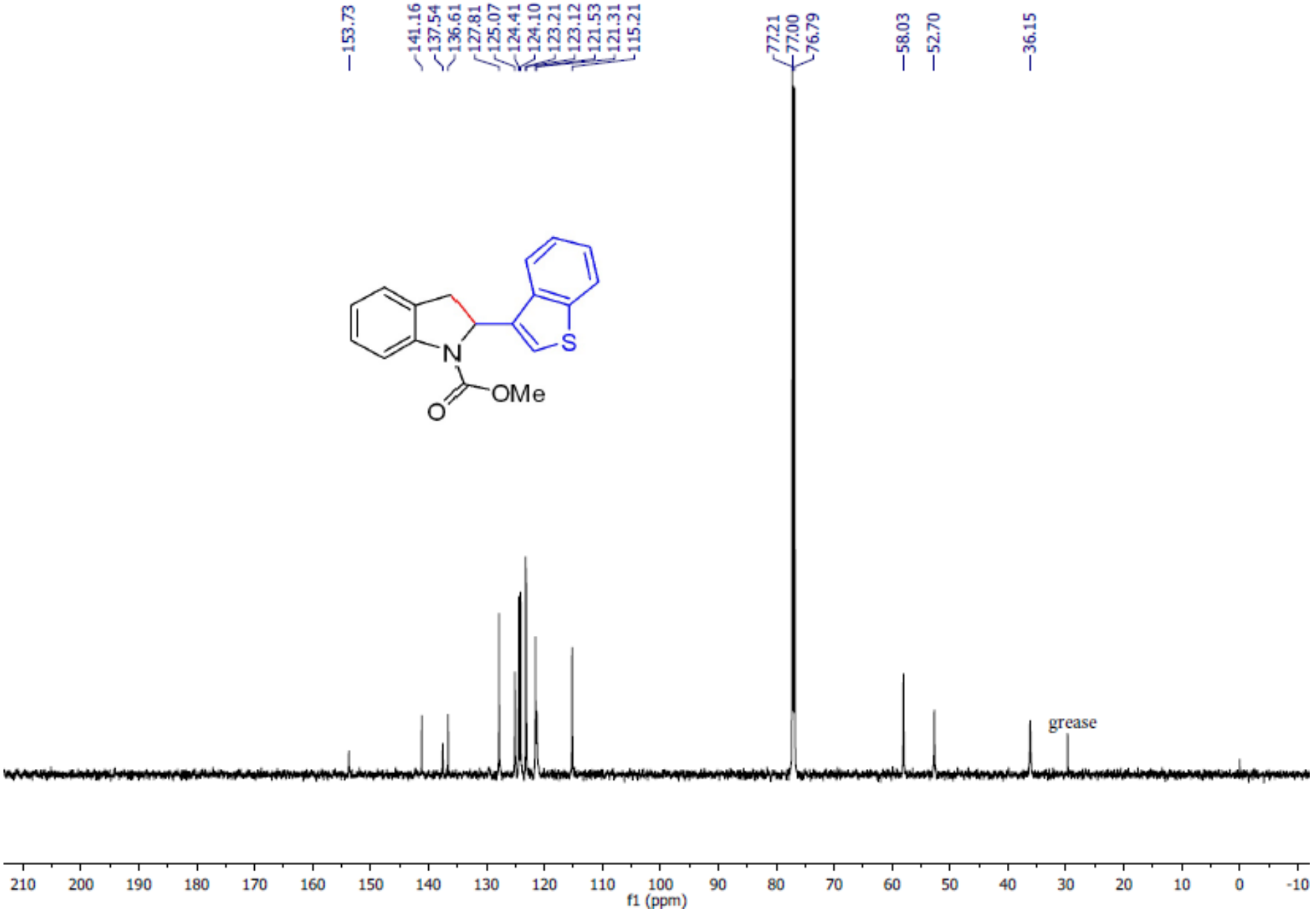
PDA Ch1 246nm

Peak#	Ret. Time	Area	Area%
1	23.413	99299	3.163
2	34.251	3040058	96.837
Total		3139357	100.000

methyl (*R*)-2-(benzo[*b*]thiophen-3-yl)indoline-1-carboxylate 2q



methyl (*R*)-2-(benzo[*b*]thiophen-3-yl)indoline-1-carboxylate 2q

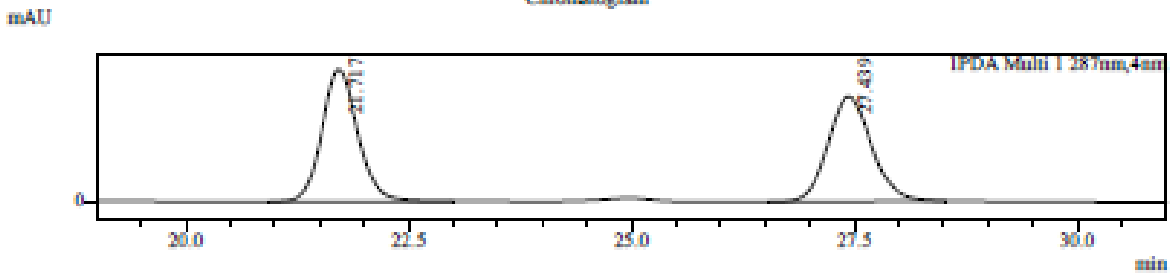


methyl (*R*)-2-(benzo[*b*]thiophen-3-yl)indoline-1-carboxylate 2q

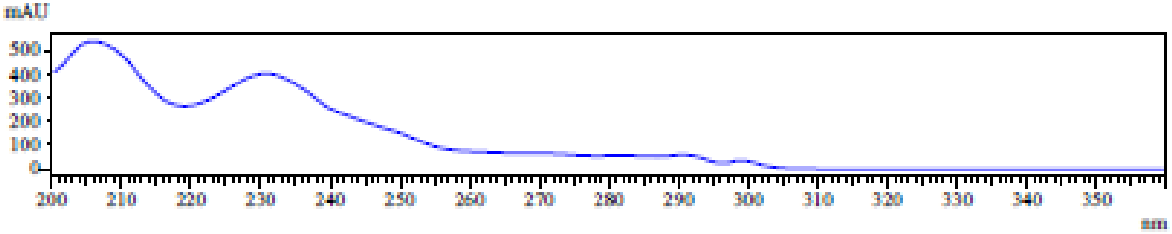
Sample Information

Sample Name	: XW-V-116-LA-1%0.8mL
Sample ID	: XW-V-116-LA-1%0.8mL
Data File	: XW-V-116-LA-1%0.8mL.lcd
Method File	: XW-1%-0.8ml.lcm

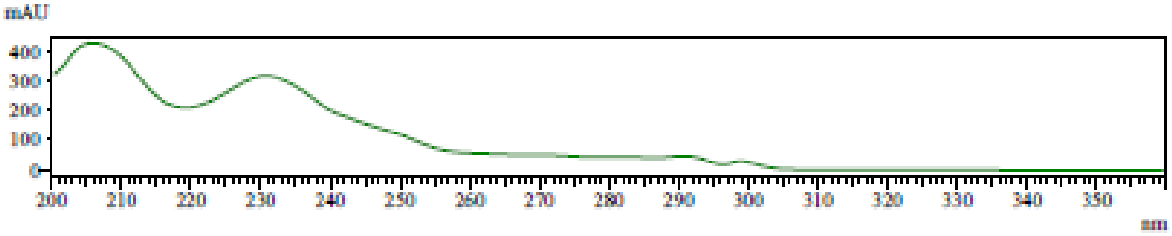
Chromatogram



UV Spectrum
Retention time = 21.717



UV Spectrum
Retention time = 27.439



Peak Table

PDA Ch1 287nm

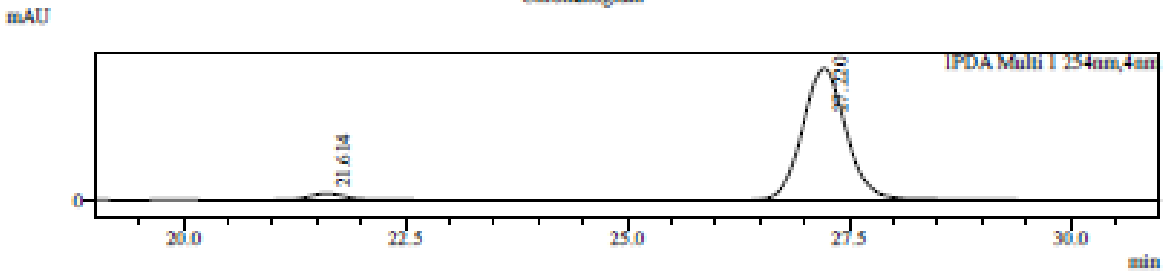
Peak#	Ret. Time	Area	Area%
1	21.717	1545390	50.918
2	27.439	1489687	49.082
Total		3035077	100.000

methyl (*R*)-2-(benzo[*b*]thiophen-3-yl)indoline-1-carboxylate 2q

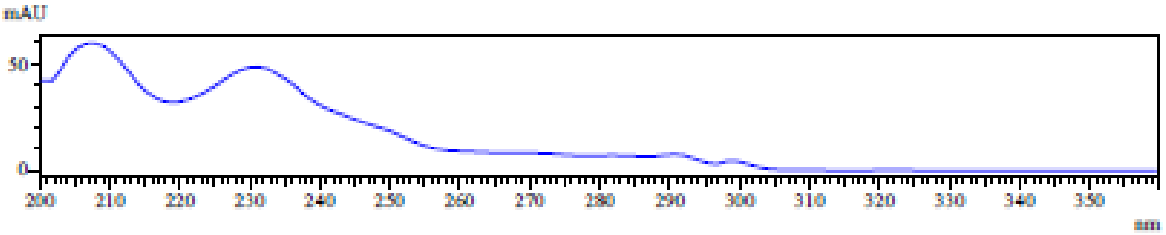
Sample Information

Sample Name	: XW-V-117-old-IA-1%0.8mL
Sample ID	: XW-V-117-old-IA-1%0.8mL
Data File	: XW-V-117-old-IA-1%0.8mL.lcd
Method File	: XW-1%-0.8ml.lcm

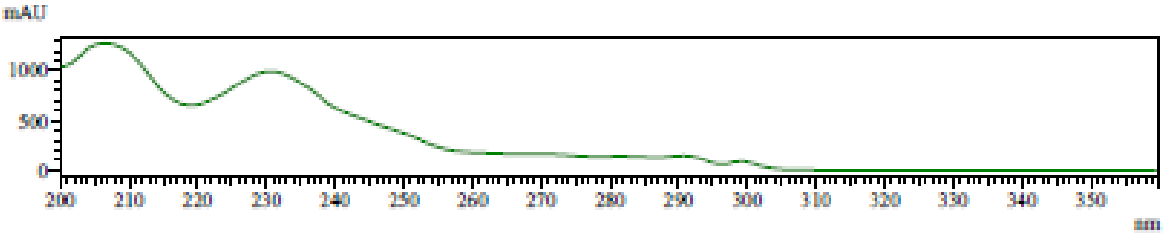
Chromatogram



UV Spectrum
Retention time = 21.614



UV Spectrum
Retention time = 27.220

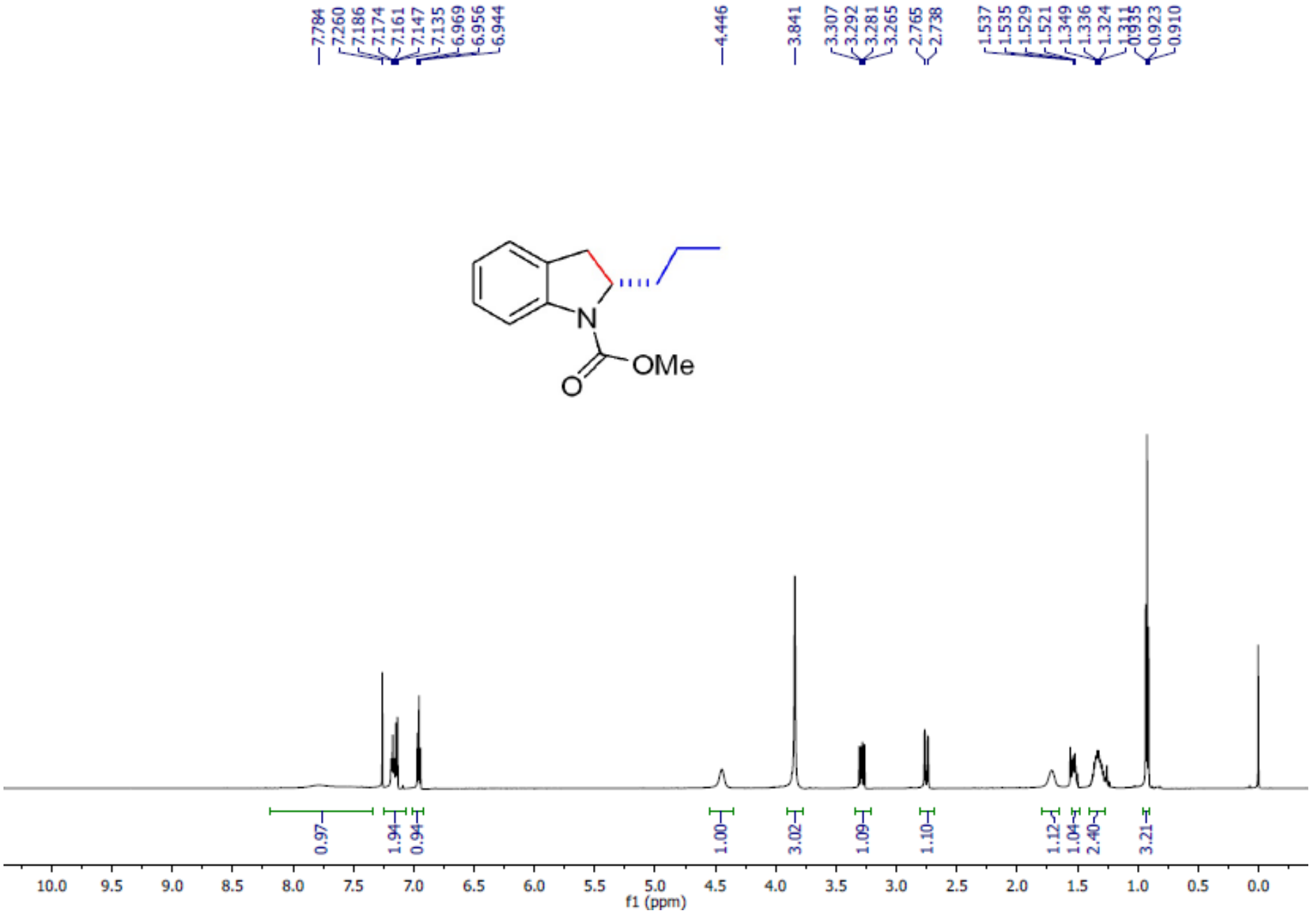


Peak Table

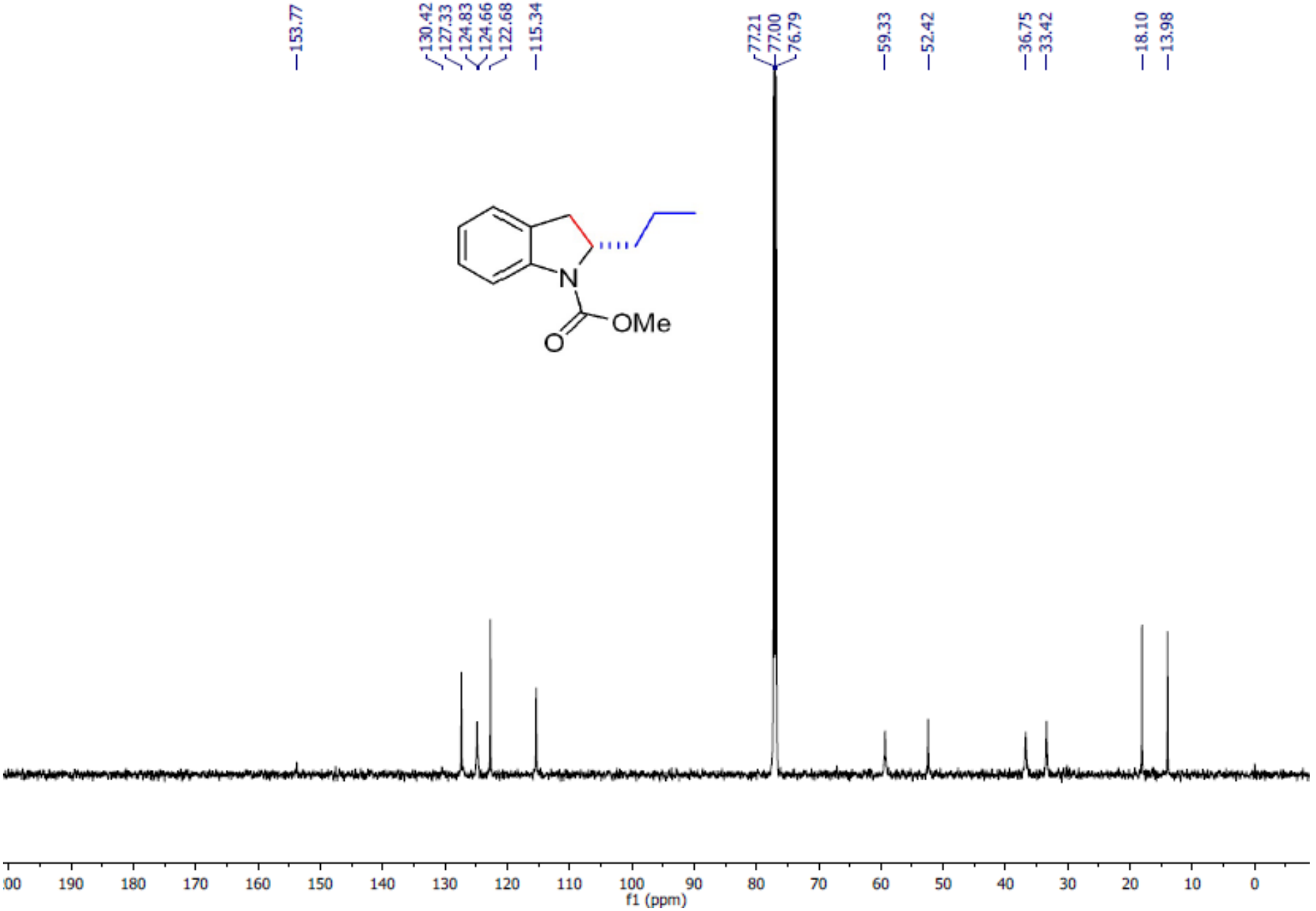
PDA Ch1 254nm

Peak#	Ret. Time	Area	Area%
1	21.614	376448	3.980
2	27.220	9082278	96.020
Total		9458726	100.000

methyl (*S*)-2-propylindoline-1-carboxylate 2r



methyl (*S*)-2-propylindoline-1-carboxylate 2r

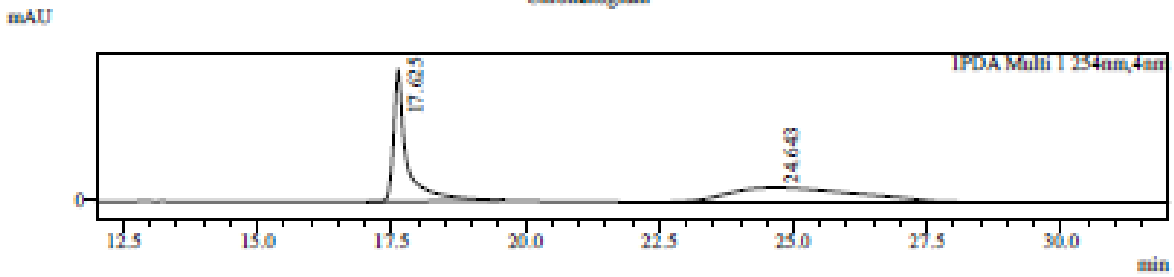


methyl (*S*)-2-propylindoline-1-carboxylate 2r

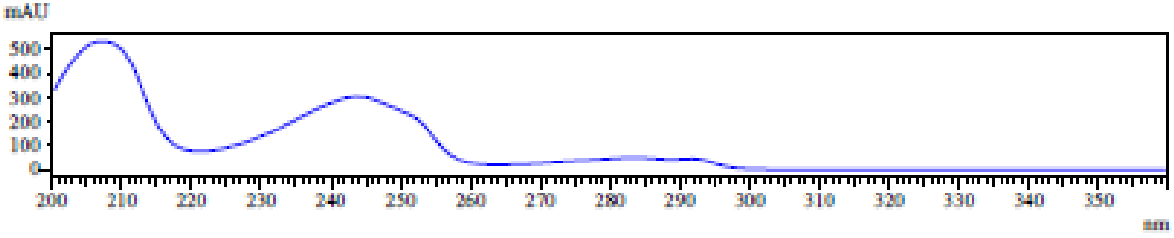
Sample Information

Sample Name	: XW-V-52--2-IA-0.2%0.8mL
Sample ID	: XW-V-52--2-IA-0.2%0.8mL
Data File	: XW-V-52--2-IA-0.2%0.8mL.lcd
Method File	: XW-0.2%-0.8ml.lcm

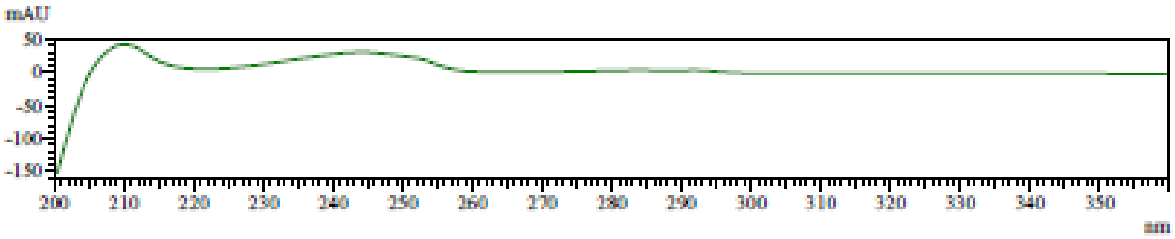
Chromatogram



UV Spectrum
Retention time = 17.625



UV Spectrum
Retention time = 24.643



Peak Table

PDA Ch1 254nm

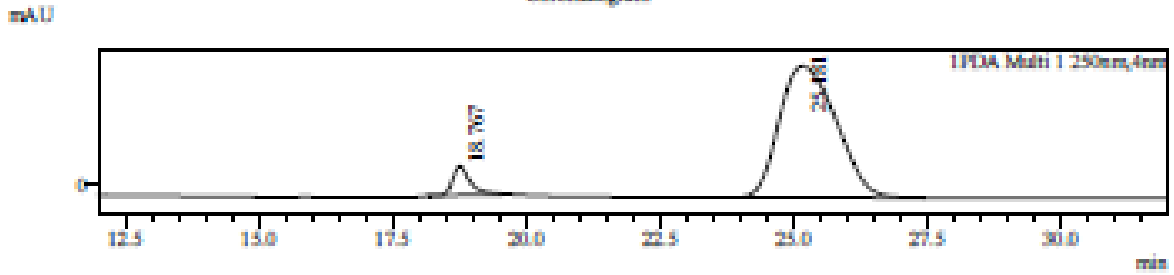
Peak#	Ret. Time	Area	Area%
1	17.625	2722739	50.278
2	24.643	2692668	49.722
Total		5415407	100.000

methyl (*S*)-2-propylindoline-1-carboxylate 2r

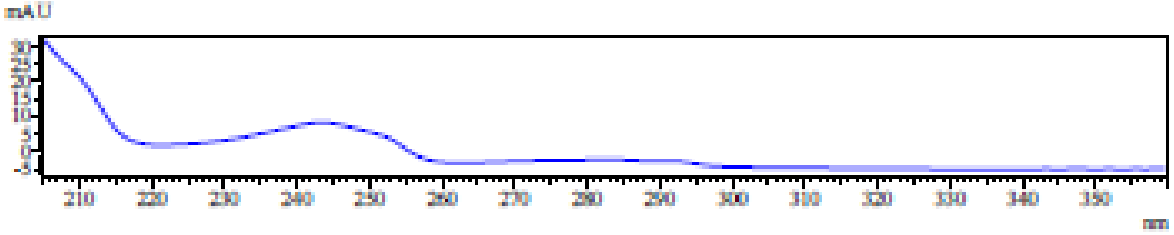
Sample Information

Sample Name	: XW-V-53-60oC-2-IA-0.2%0.8mL
Sample ID	: XW-V-53-60oC-2-IA-0.2%0.8mL
Data File	: XW-V-53-60oC-2-IA-0.2%0.8mL.lcd
Method File	: XW-0.2%-0.8ml.lcm

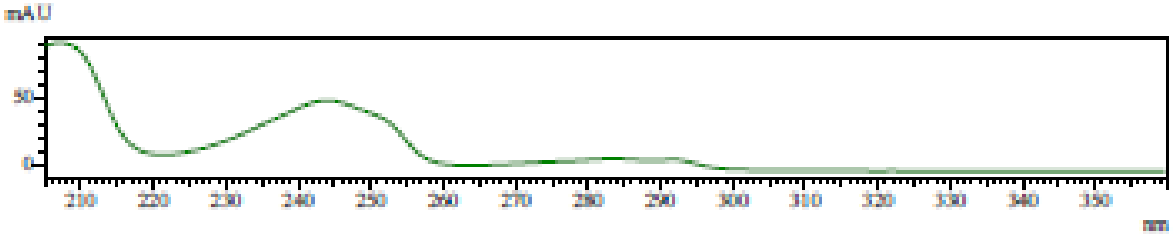
Chromatogram



UV Spectrum
Retention time = 18.767



UV Spectrum
Retention time = 25.181

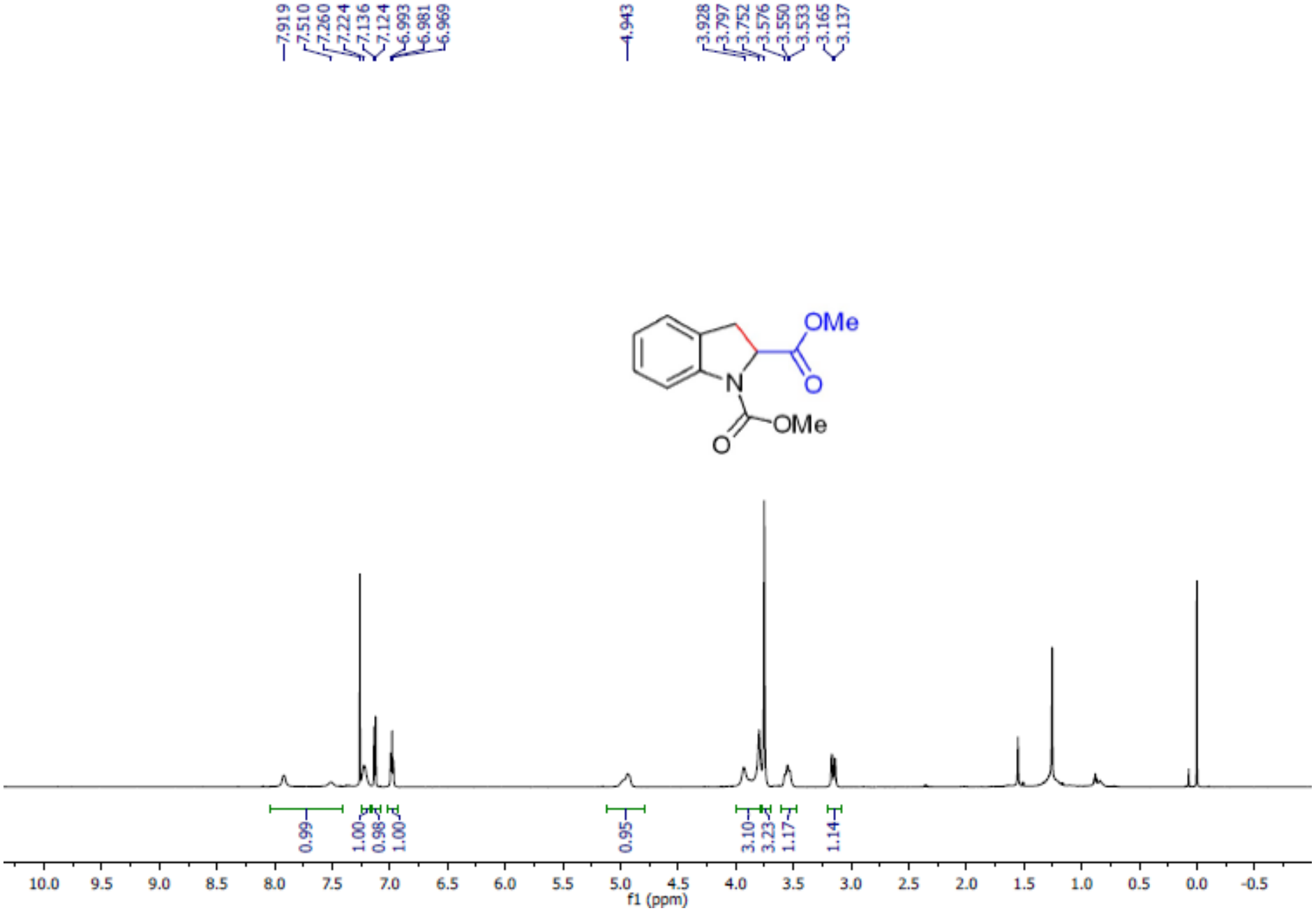


Peak Table

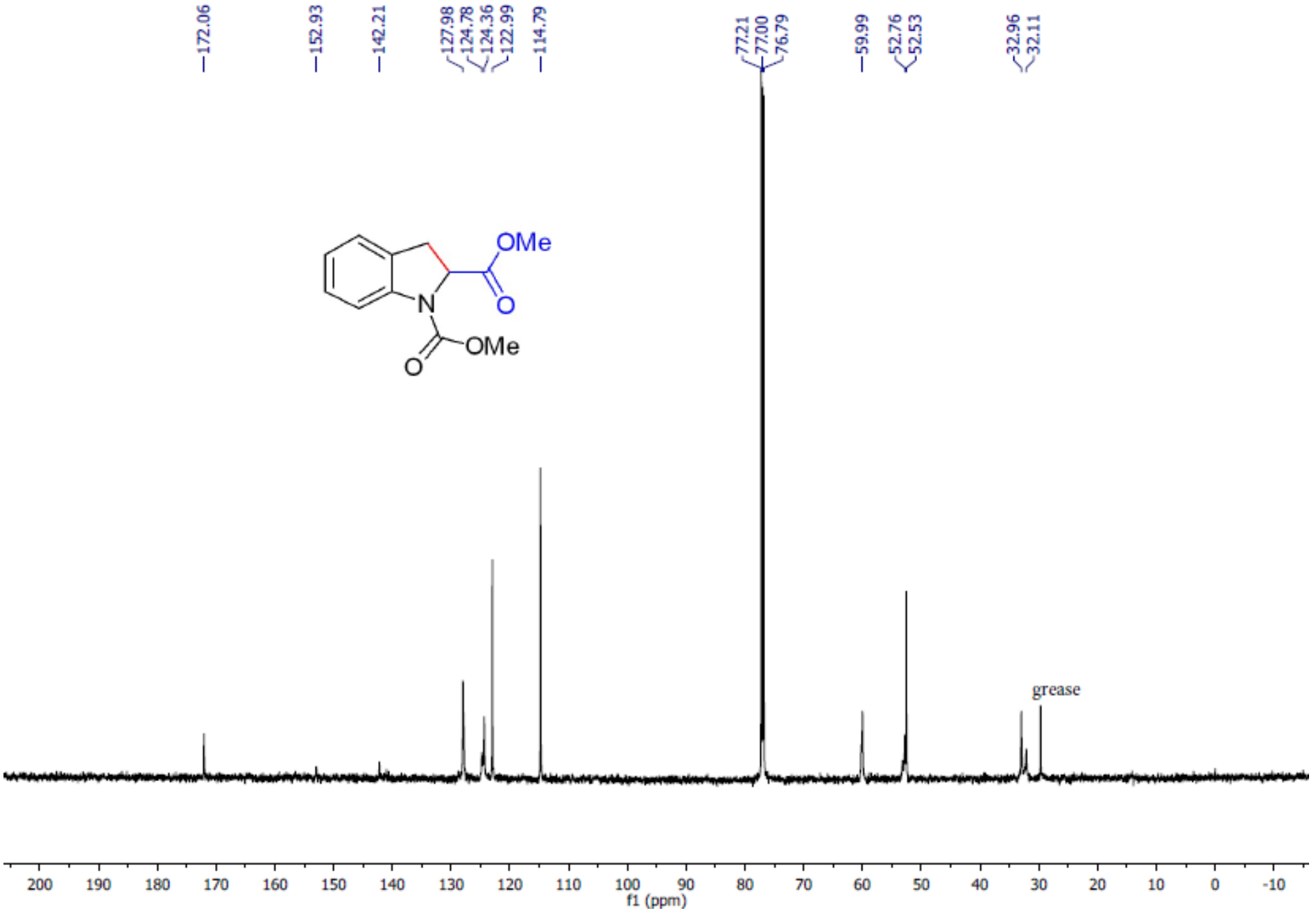
PDA Ch1 250nm

Peak#	Ret. Time	Area	Area%
1	18.767	213047	6.350
2	25.181	3142094	93.650
Total		3355141	100.000

dimethyl (*R*)-indoline-1,2-dicarboxylate 2s



dimethyl (*R*)-indoline-1,2-dicarboxylate 2s

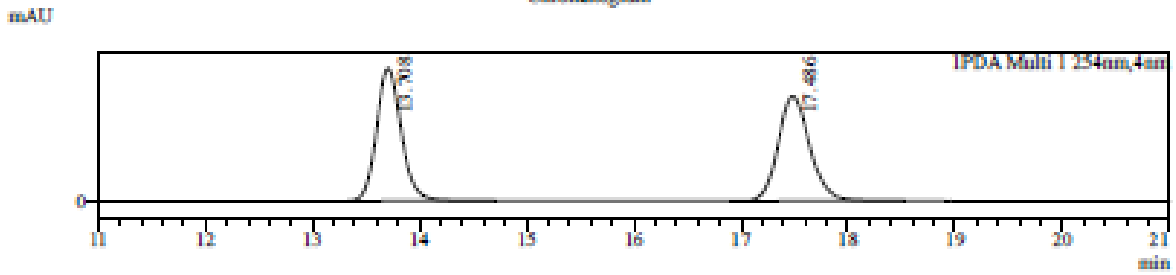


dimethyl (*R*)-indoline-1,2-dicarboxylate 2s

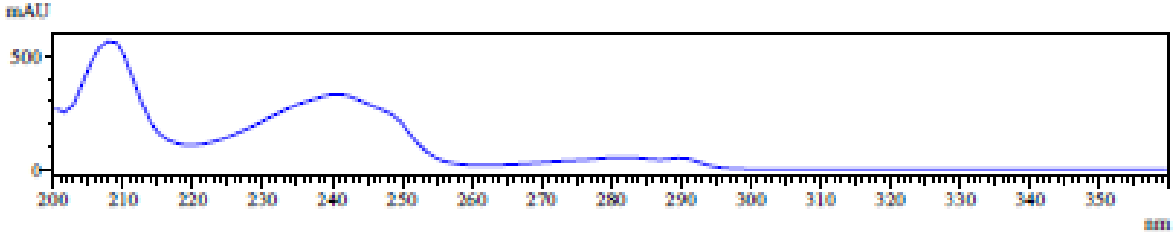
Sample Information

Sample Name	: XW-V-86-2-LA-5%0.8mL
Sample ID	: XW-V-86-2-LA-5%0.8mL
Data File	: XW-V-86-2-LA-5%0.8mL.lcd
Method File	: XW-5%-0.8ml.lcm

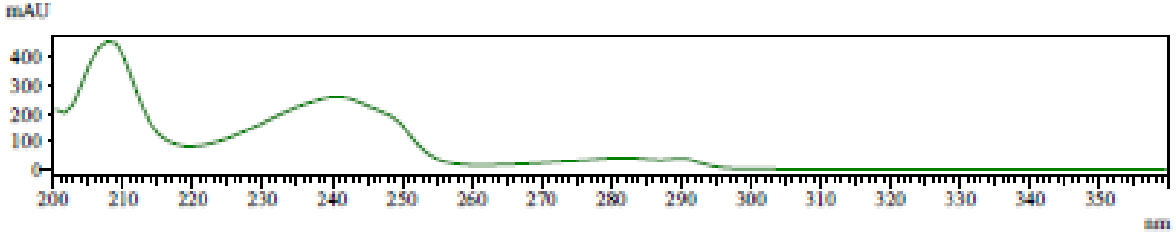
Chromatogram



UV Spectrum
Retention time = 13.708



UV Spectrum
Retention time = 17.486



Peak Table

PDA Ch1 254nm

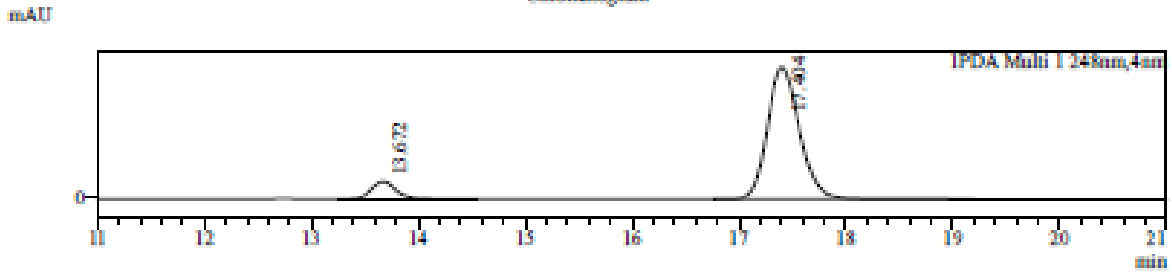
Peak#	Ret. Time	Area	Area%
1	13.708	1284010	49.832
2	17.486	1292669	50.168
Total		2576679	100.000

dimethyl (*R*)-indoline-1,2-dicarboxylate 2s

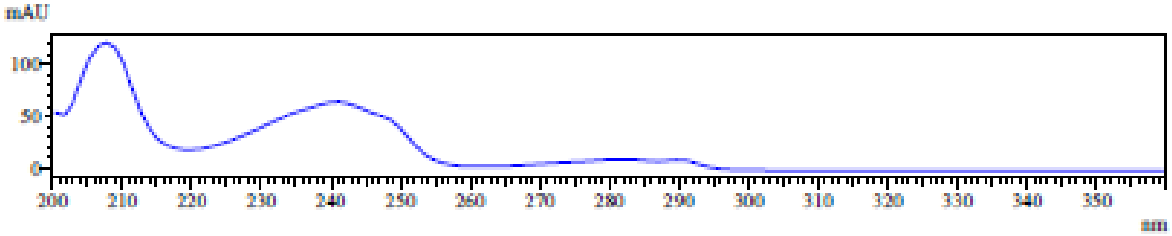
Sample Information

Sample Name	: XW-V-87-40C-IA-5%0.8mL
Sample ID	: XW-V-87-40C-IA-5%0.8mL
Data File	: XW-V-87-40C-IA-5%0.8mL.lcd
Method File	: XW-5%-0.8ml.lcm

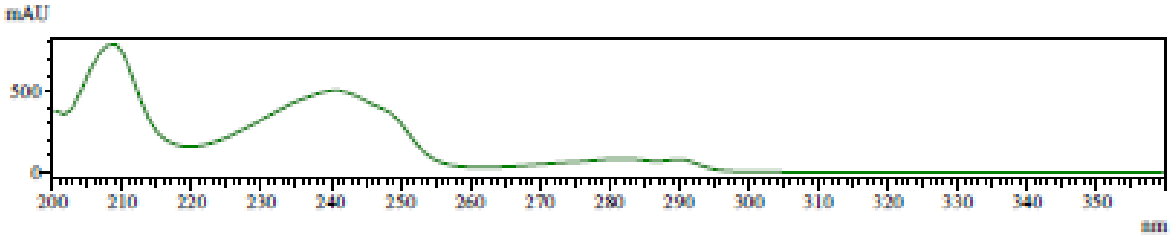
Chromatogram



UV Spectrum
Retention time = 13.672



UV Spectrum
Retention time = 17.404

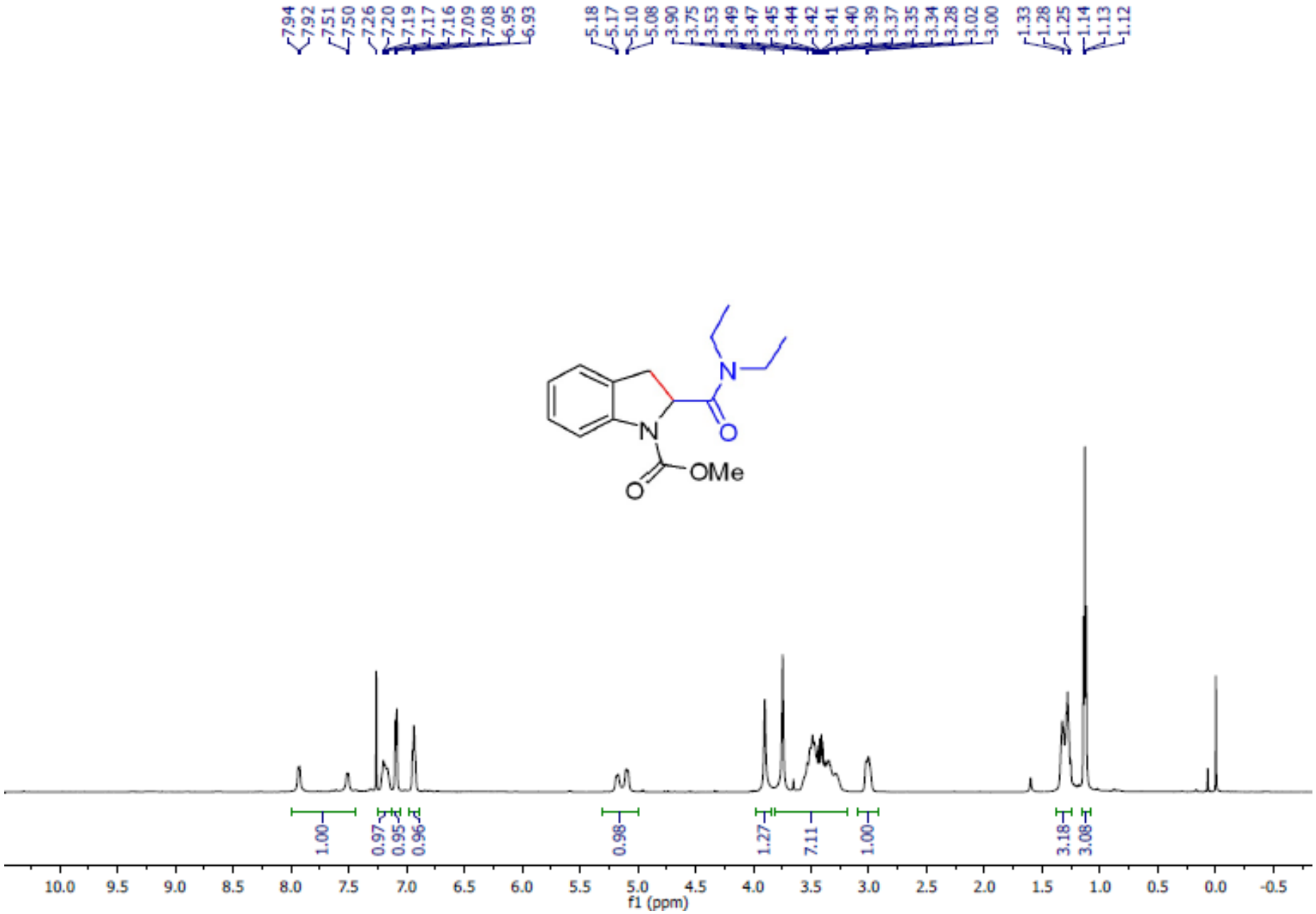


Peak Table

PDA Ch1 248nm

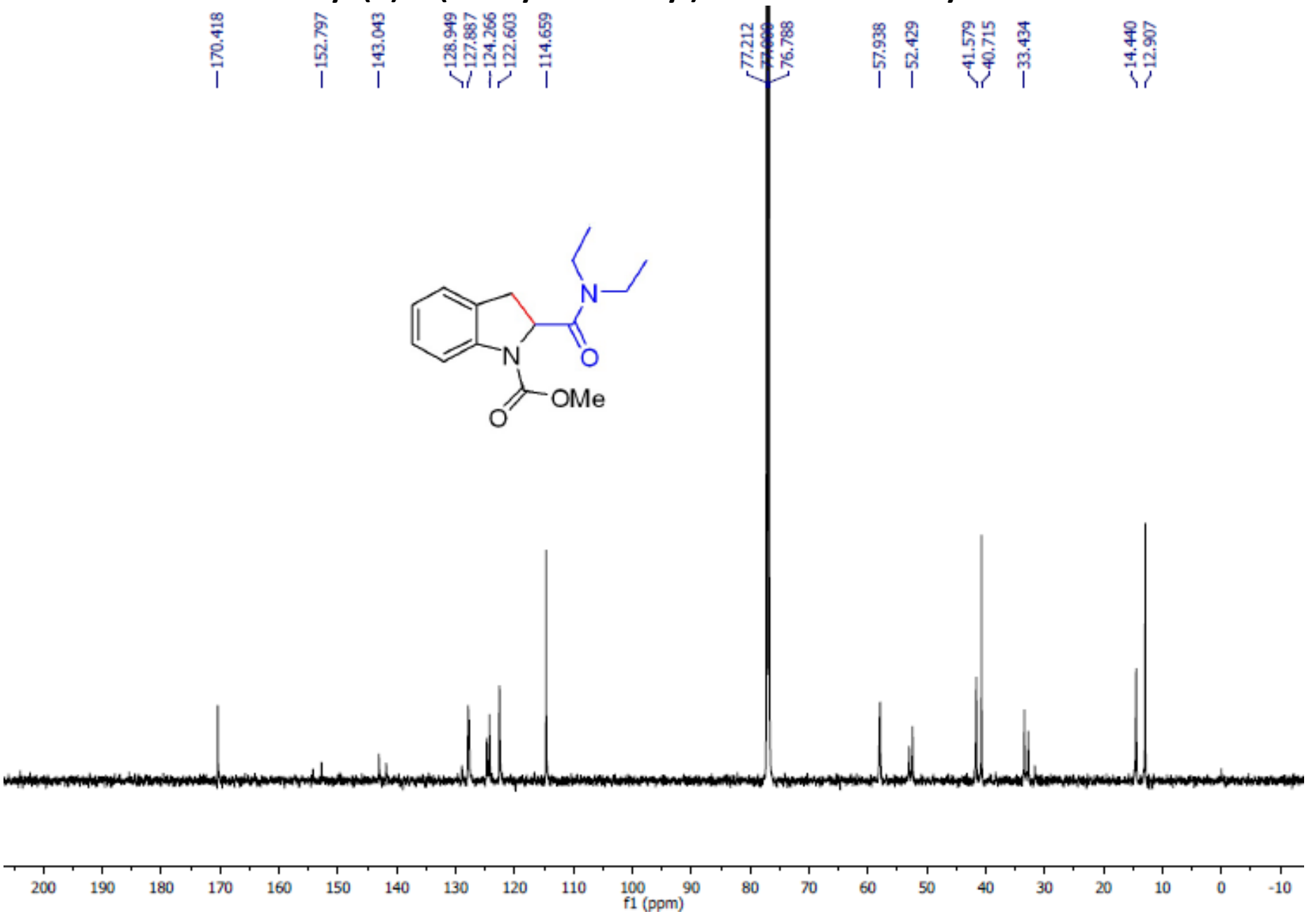
Peak#	Ret. Time	Area	Area%
1	13.672	799187	9.368
2	17.404	7731407	90.632
Total		8530593	100.000

methyl (*R*)-2-(diethylcarbamoyl)indoline-1-carboxylate 2t



Note: The broad spectrum was due to the amide rotamers

methyl (*R*)-2-(diethylcarbamoyl)indoline-1-carboxylate 2t



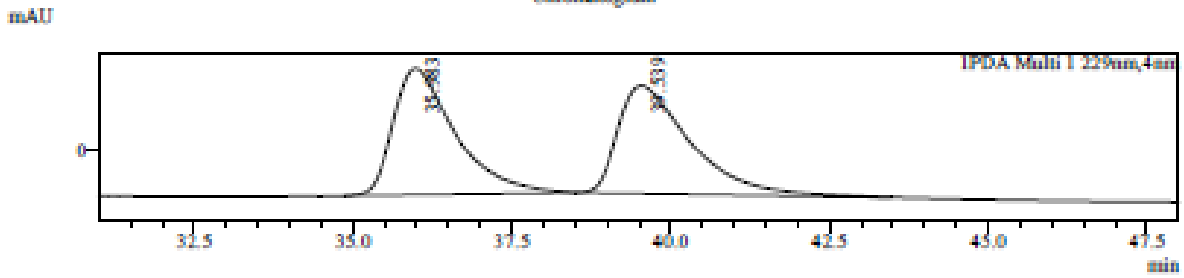
Note: The spectrum contains a mixture of amide rotamers and the major rotamer was labelled.

methyl (*R*)-2-(diethylcarbamoyl)indoline-1-carboxylate 2t

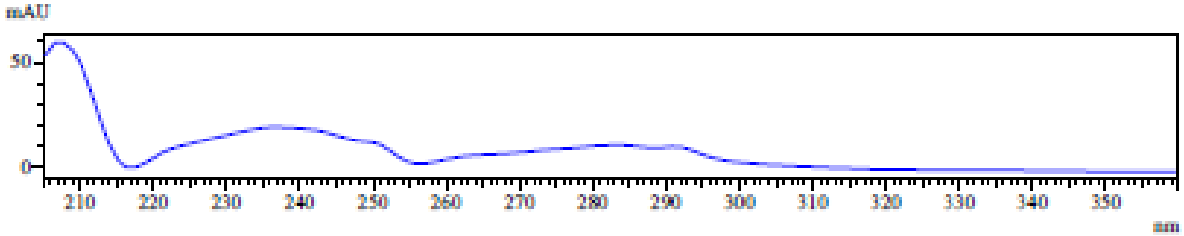
Sample Information

Sample Name	: XW-V-111-IA-8%0.8mL
Sample ID	: XW-V-111-IA-8%0.8mL
Data File	: XW-V-111-IA-8%0.8mL001.lcd
Method File	: XW-8%-0.8ml.lcm

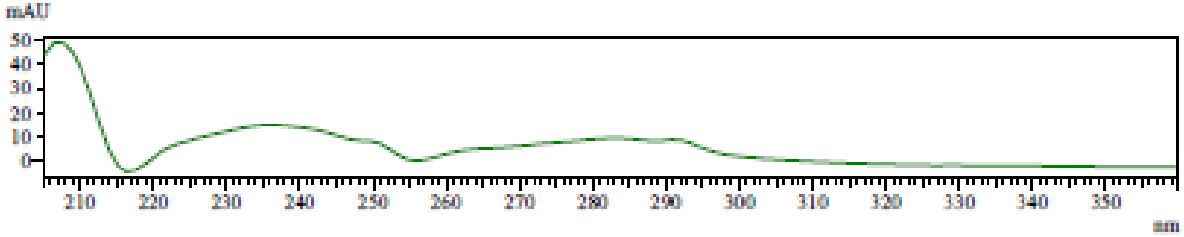
Chromatogram



UV Spectrum
Retention time = 35.983



UV Spectrum
Retention time = 39.539



Peak Table

PDA Ch1 229nm

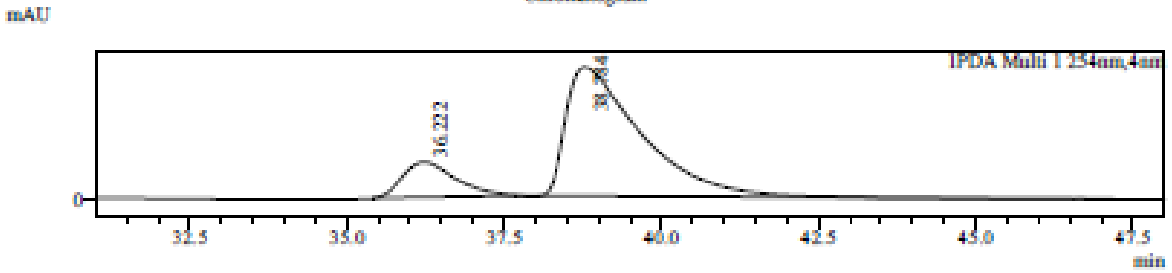
Peak#	Ret. Time	Area	Area%
1	35.983	1476682	49.895
2	39.539	1482878	50.105
Total		2959560	100.000

methyl (*R*)-2-(diethylcarbamoyl)indoline-1-carboxylate 2t

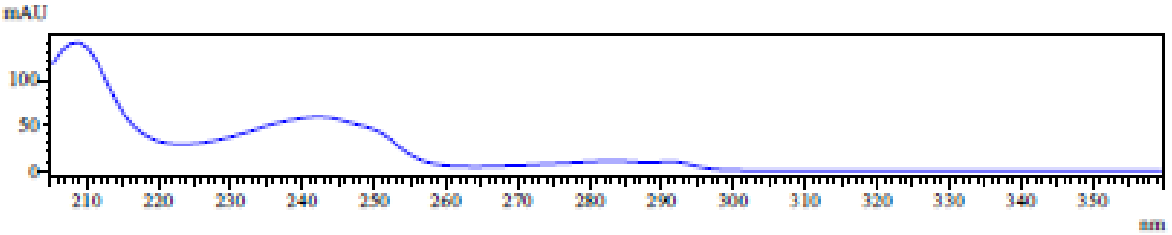
Sample Information

Sample Name	: XW-V-113-40C-IA-8%0.8mL
Sample ID	: XW-V-113-40C-IA-8%0.8mL
Data File	: XW-V-113-40C-IA-8%0.8mL.lcd
Method File	: XW-8%-0.8ml.lcm

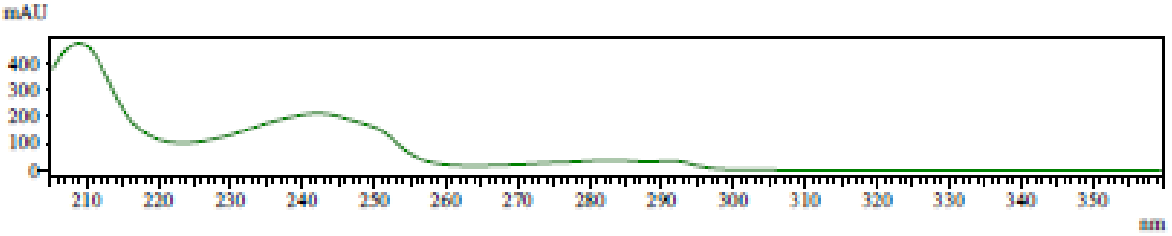
Chromatogram



UV Spectrum
Retention time = 36.222



UV Spectrum
Retention time = 38.784

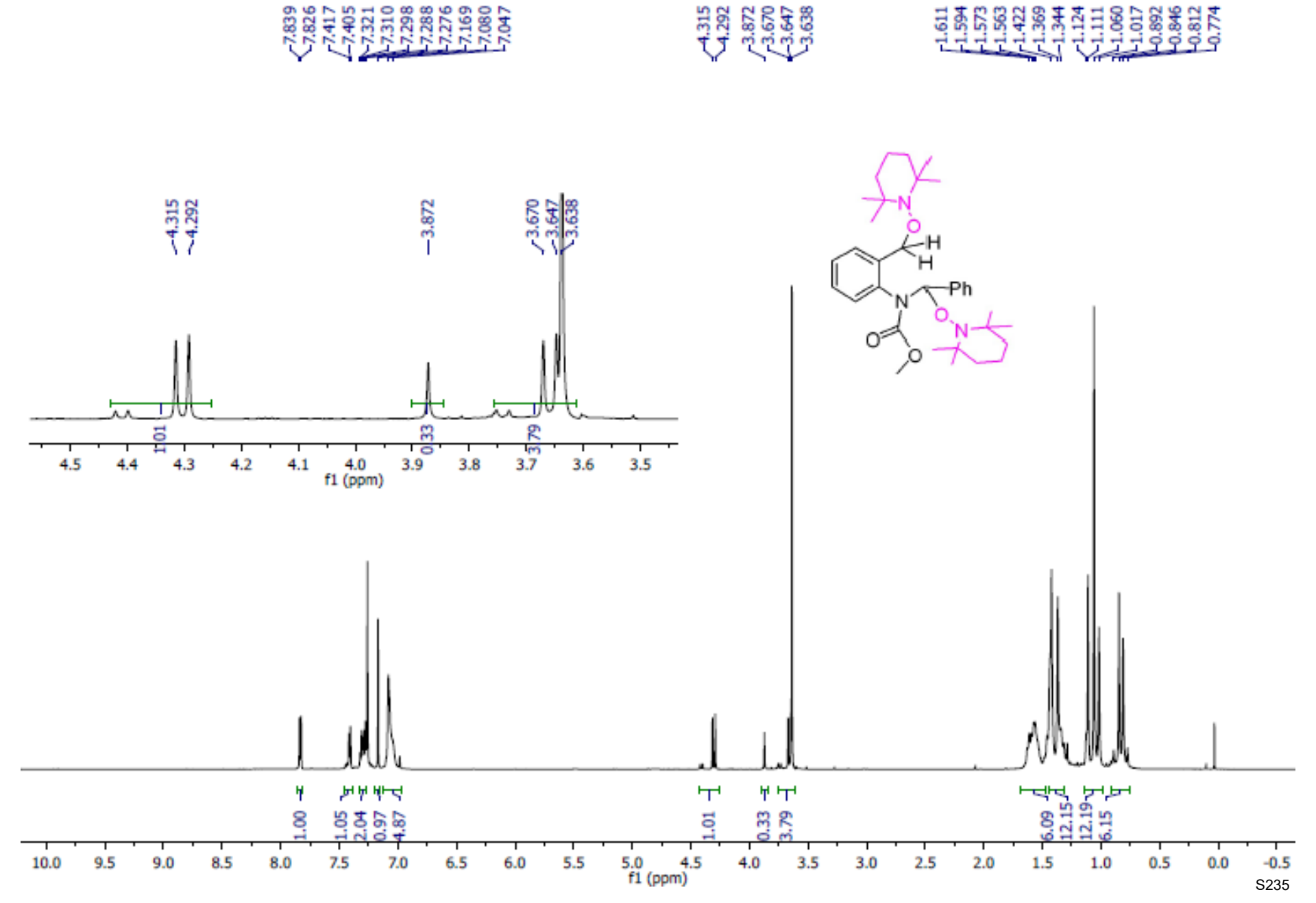


Peak Table

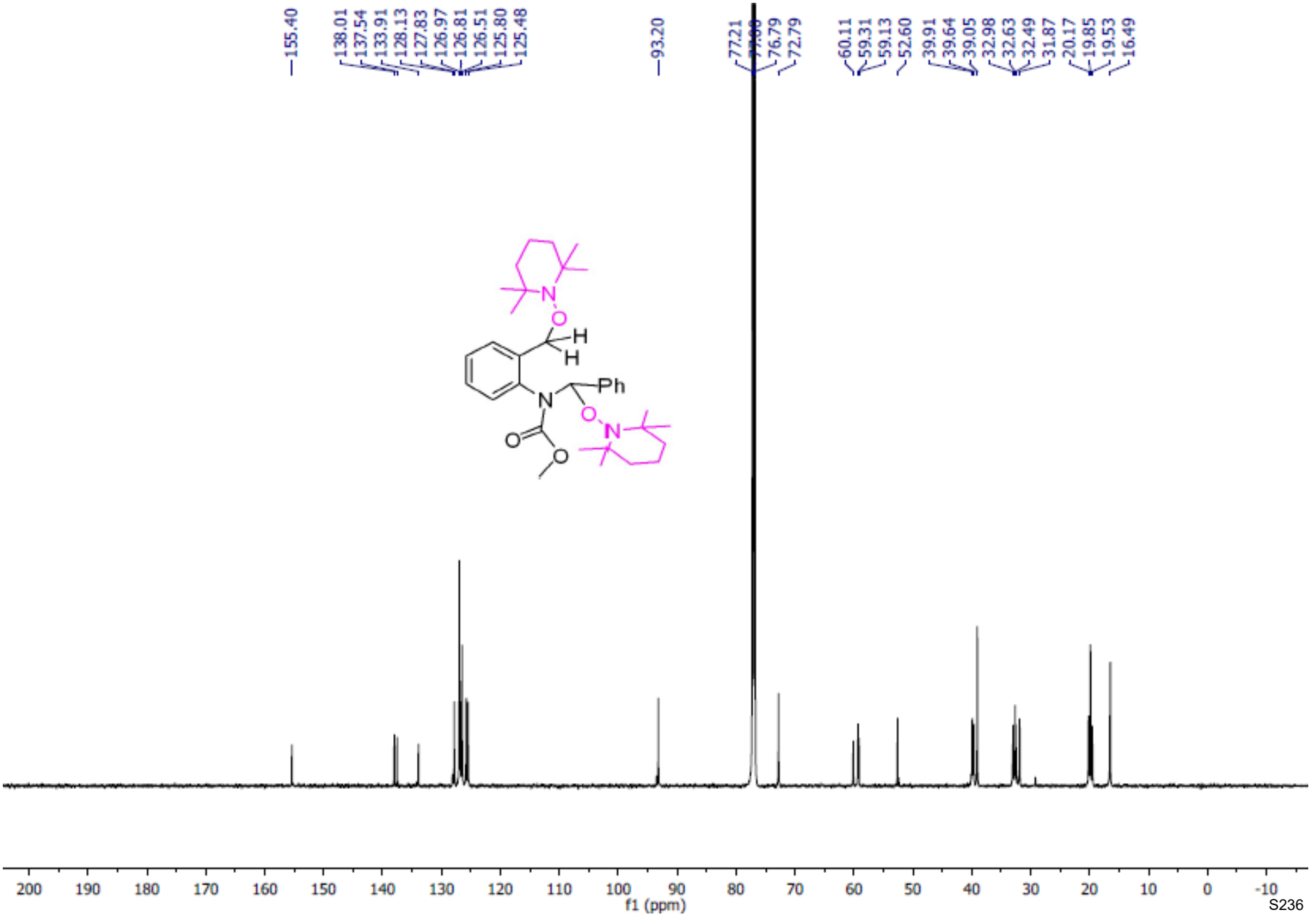
PDA Ch1 254nm

Peak#	Ret. Time	Area	Area%
1	36.222	1352139	16.203
2	38.784	6992618	83.797
Total		8344758	100.000

methyl (phenyl((2,2,6,6-tetramethylpiperidin-1-yl)oxy)methyl)(2-(((2,2,6,6-tetramethylpiperidin-1-yl)oxy)methyl)phenyl)carbamate 3c



methyl (phenyl((2,2,6,6-tetramethylpiperidin-1-yl)oxy)methyl)(2-(((2,2,6,6-tetramethylpiperidin-1-yl)oxy)methyl)phenyl)carbamate 3c

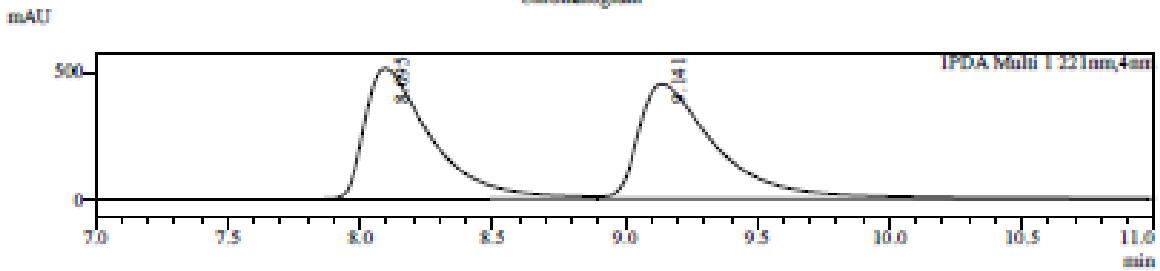


methyl (phenyl((2,2,6,6-tetramethylpiperidin-1-yl)oxy)methyl)(2-(((2,2,6,6-tetramethylpiperidin-1-yl)oxy)methyl)phenyl)carbamate 3c

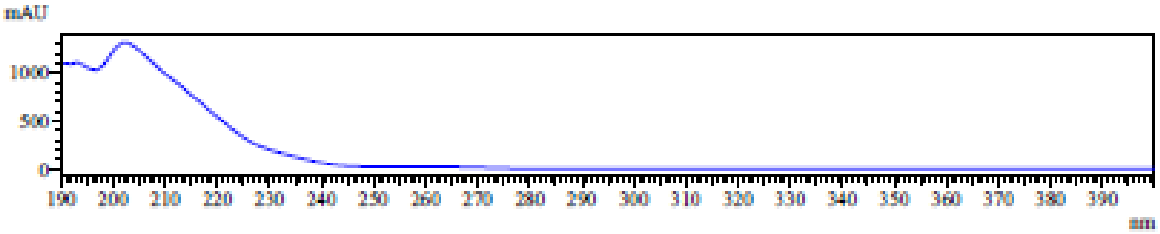
Sample Information

Sample Name	: XW-V-127-IA-0.3%0.8mL-1
Sample ID	: XW-V-127-IA-0.3%0.8mL-1
Data File	: XW-V-127-IA-0.3%0.8mL-1.lcd
Method File	: XW-0.3%-0.8ml.lcm

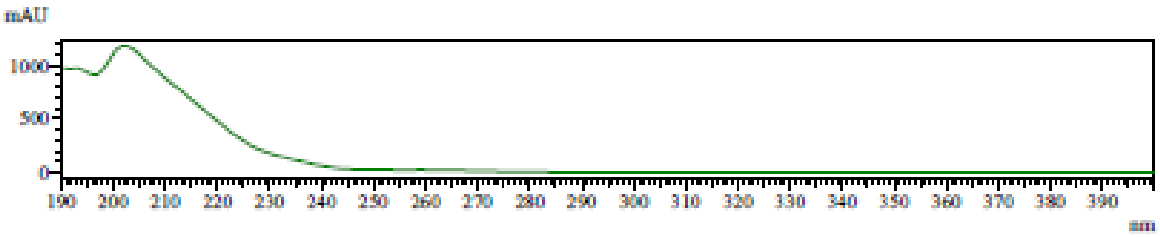
Chromatogram



UV Spectrum
Retention time = 8.095



UV Spectrum
Retention time = 9.141



Peak Table

PDA Ch1 221nm

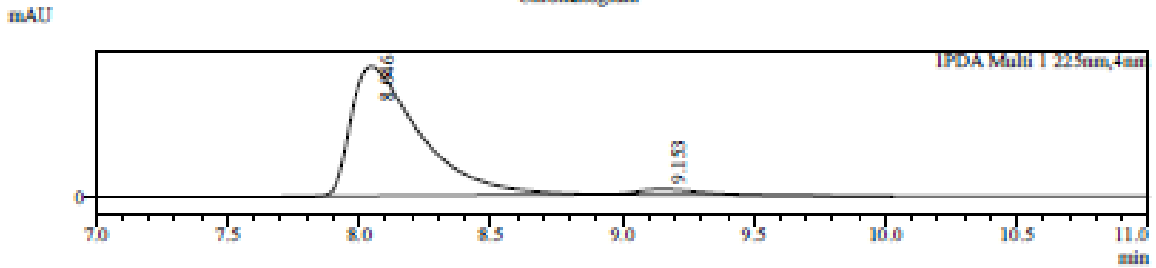
Peak#	Ret. Time	Area	Area%
1	8.095	9013766	49.278
2	9.141	9277793	50.722
Total		18291559	100.000

methyl (phenyl((2,2,6,6-tetramethylpiperidin-1-yl)oxy)methyl)(2-(((2,2,6,6-tetramethylpiperidin-1-yl)oxy)methyl)phenyl)carbamate 3c

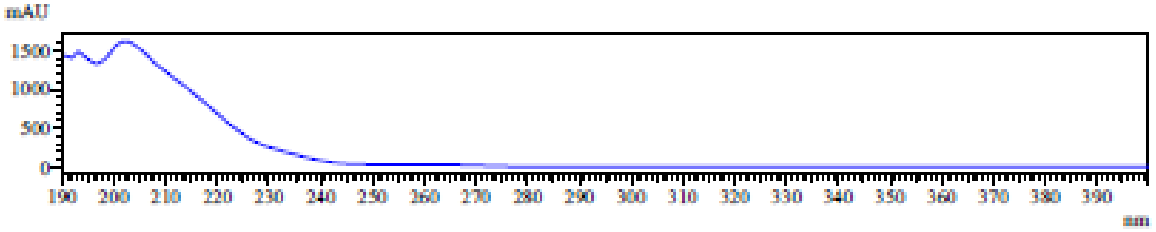
Sample Information

Sample Name	: XW-V-137-LA-0.3%0.8mL-1
Sample ID	: XW-V-137-LA-0.3%0.8mL-1
Data File	: XW-V-137-LA-0.3%0.8mL-1.lcd
Method File	: XW-0.3%-0.8ml.lcm

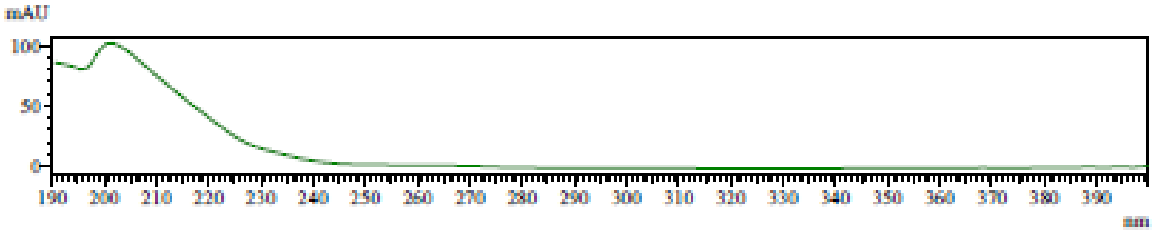
Chromatogram



UV Spectrum
Retention time = 8.046



UV Spectrum
Retention time = 9.153

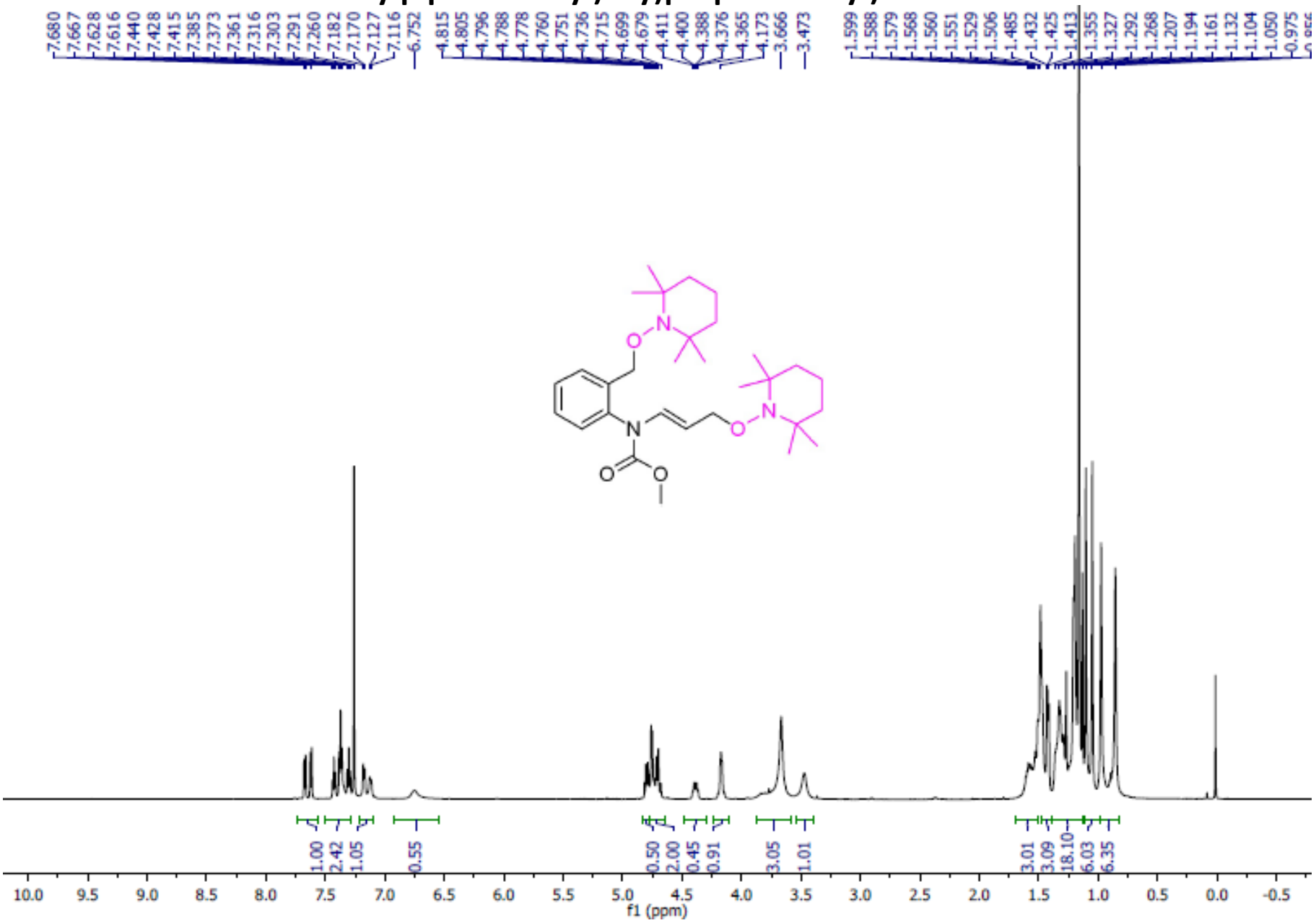


Peak Table

PDA Ch1 225nm

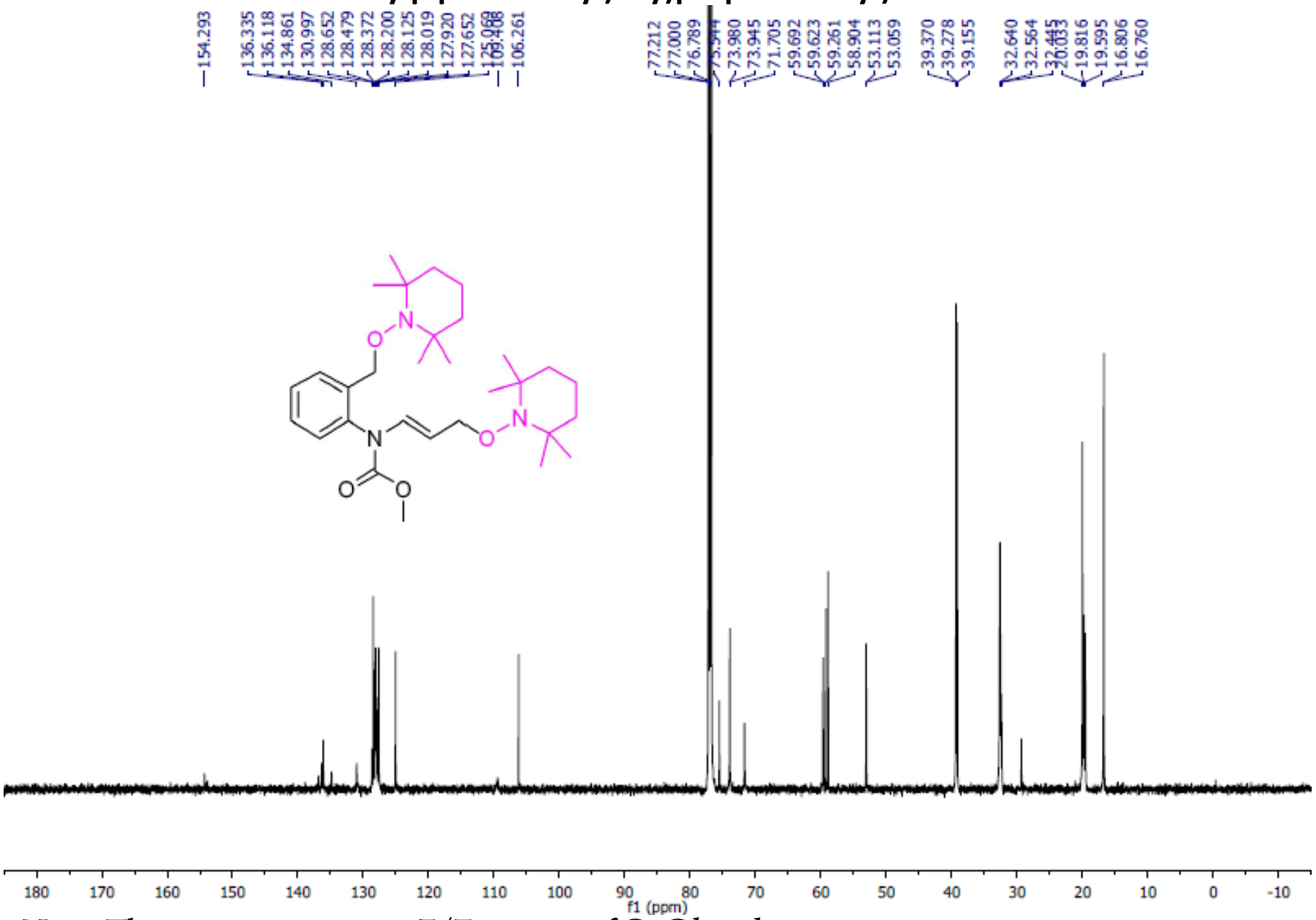
Peak#	Ret. Time	Area	Area%
1	8.046	7823322	96.446
2	9.153	288260	3.554
Total		8111582	100.000

methyl (2-(((2,2,6,6-tetramethylpiperidin-1-yl)oxy)methyl) phenyl)(3-((2,2,6,6-tetramethylpiperidin-1-yl)oxy)prop-1-en-1-yl)carbamate 3u



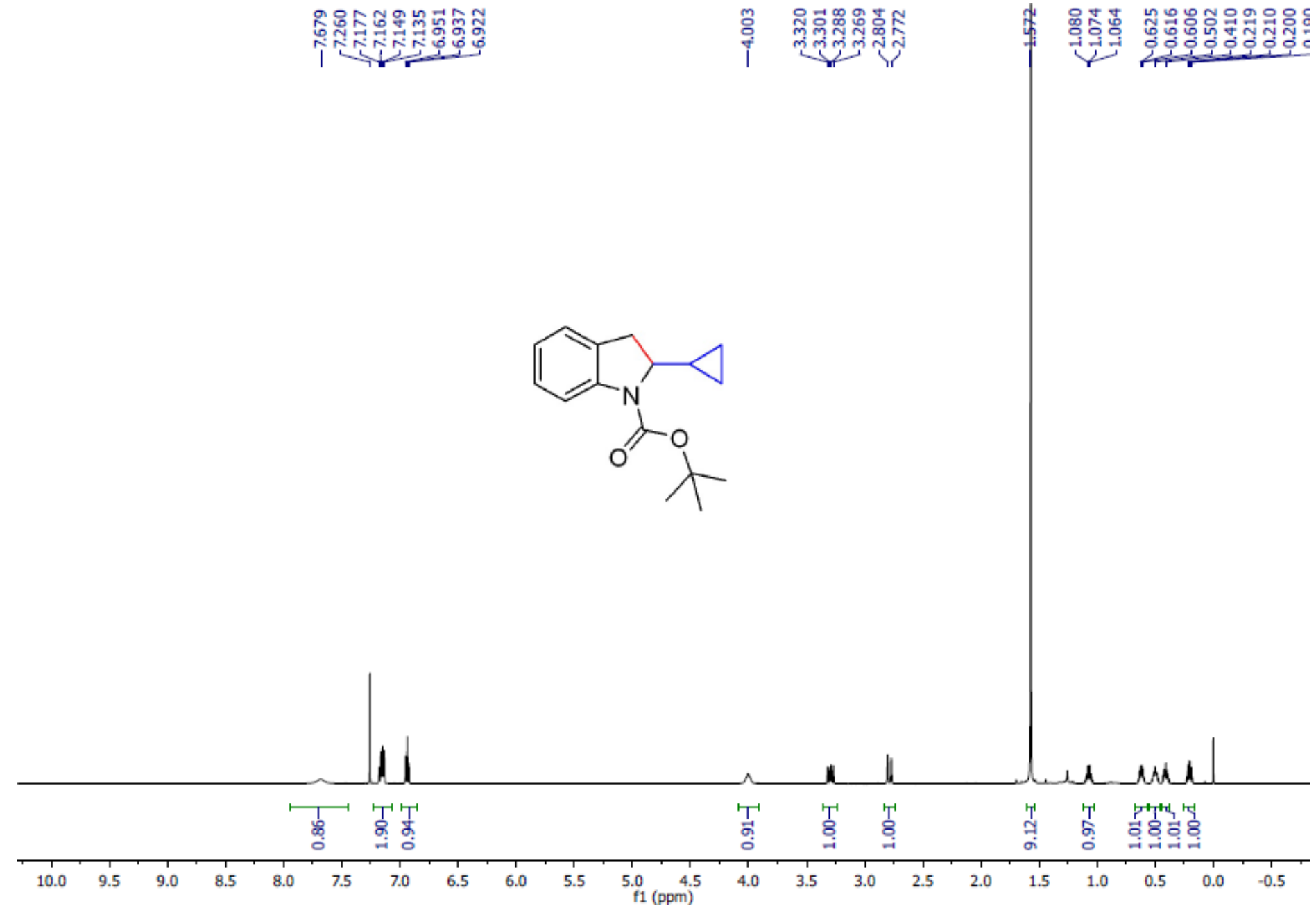
Note: The spectrum contains E/Z isomers of C=C bond.

methyl (2-(((2,2,6,6-tetramethylpiperidin-1-yl)oxy)methyl) phenyl)(3-((2,2,6,6-tetramethylpiperidin-1-yl)oxy)prop-1-en-1-yl)carbamate 3u

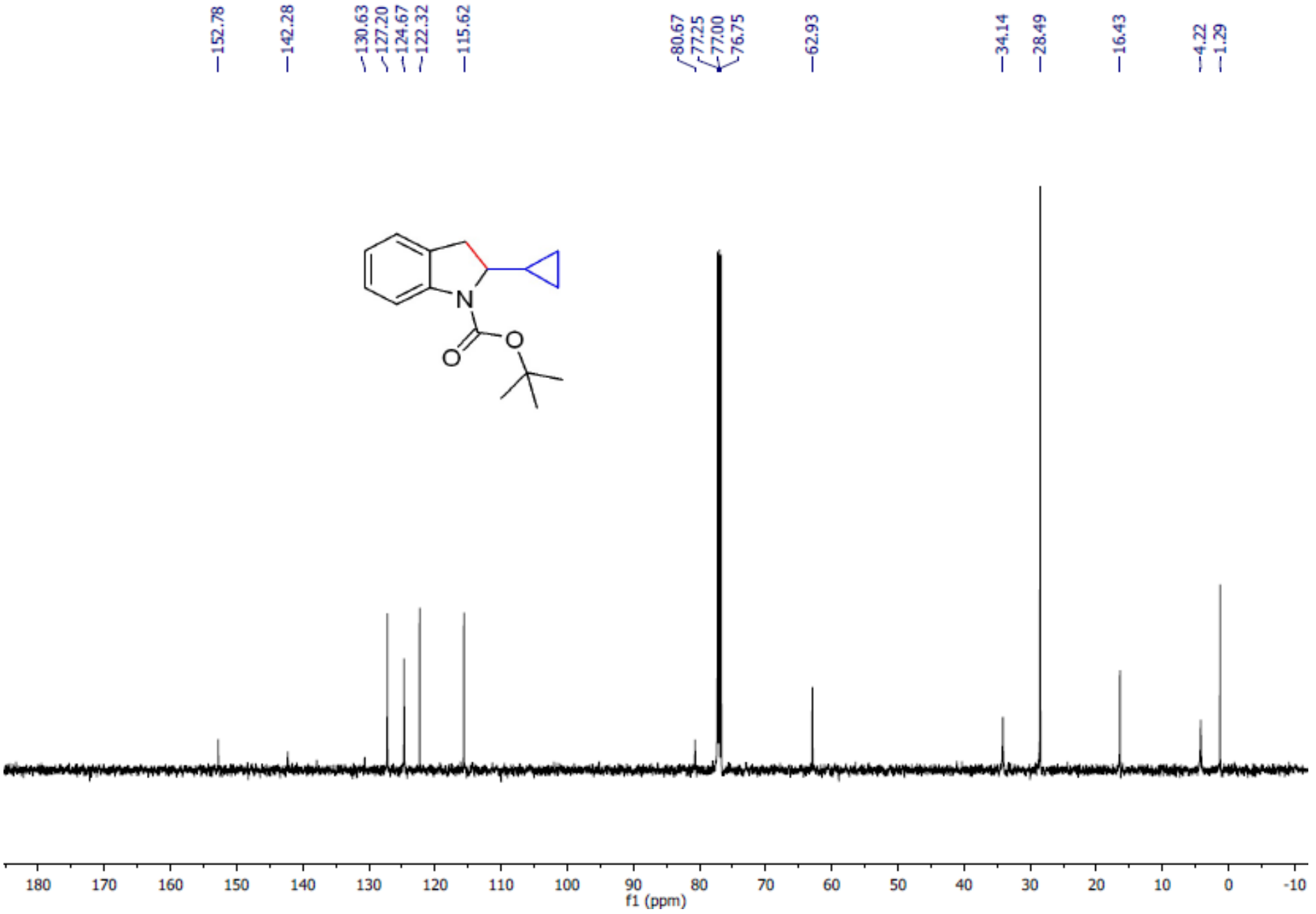


Note: The spectrum contains E/Z isomers of C=C bond.

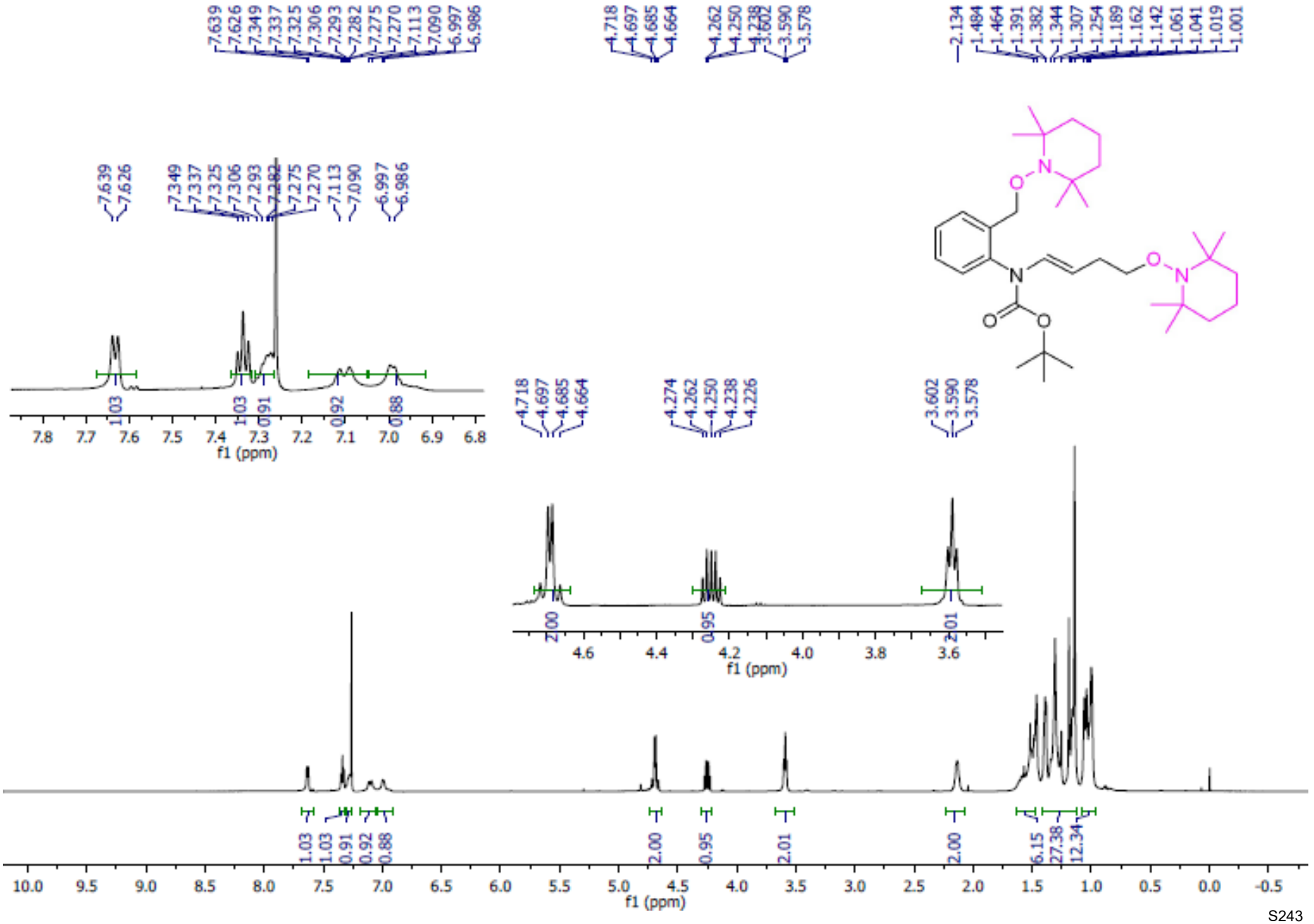
tert-butyl 2-cyclopropylindoline-1-carboxylate 2v



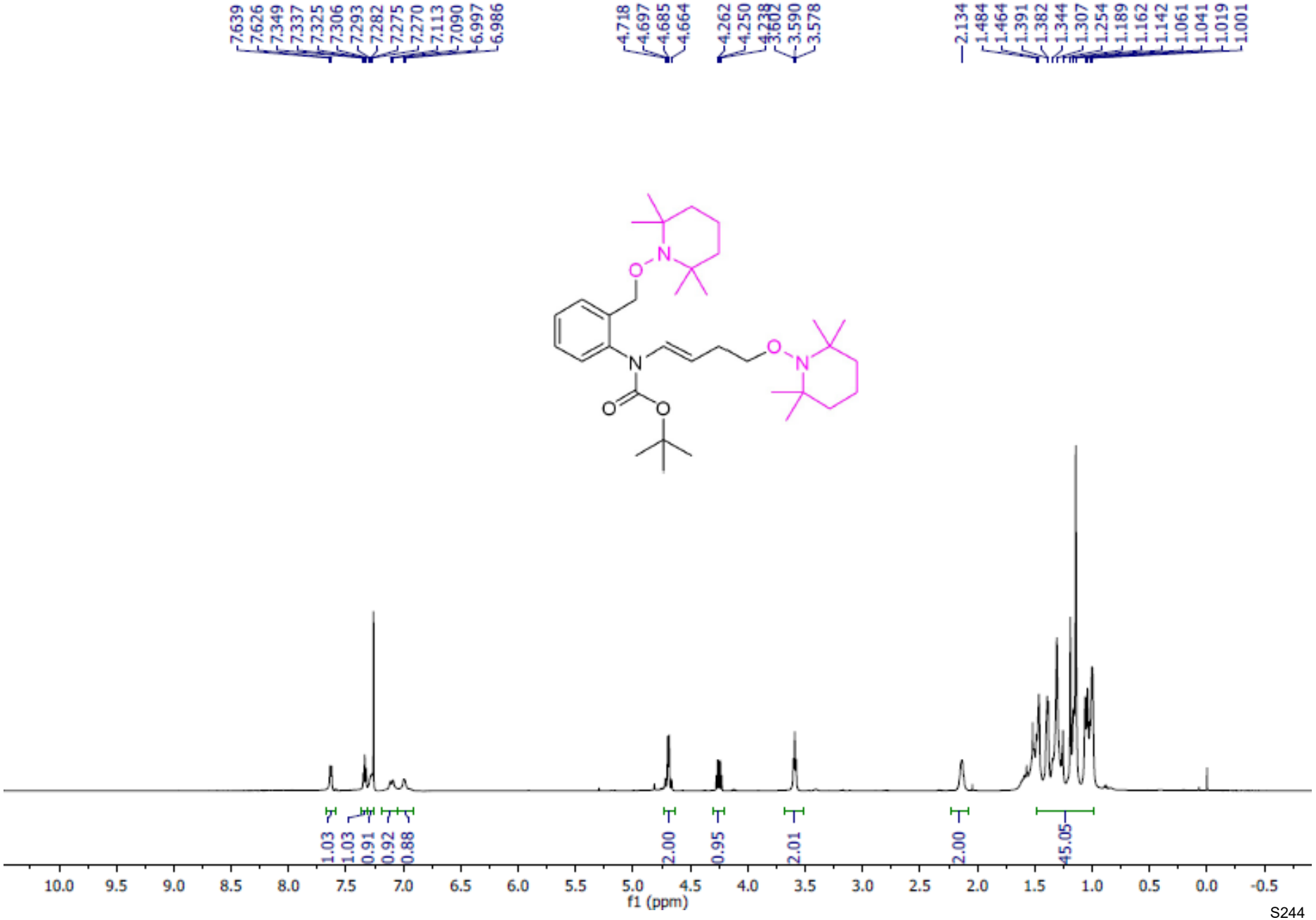
tert-butyl 2-cyclopropylindoline-1-carboxylate 2v



tert-butyl (4-((2,2,6,6-tetramethylpiperidin-1-yl)oxy)but-1-en-1-yl)(2-(((2,2,6,6-tetramethylpiperidin-1-yl)oxy)methyl)phenyl)carbamate 3v



tert-butyl (4-((2,2,6,6-tetramethylpiperidin-1-yl)oxy)but-1-en-1-yl)(2-(((2,2,6,6-tetramethylpiperidin-1-yl)oxy)methyl)phenyl)carbamate 3v



tert-butyl (4-((2,2,6,6-tetramethylpiperidin-1-yl)oxy)but-1-en-1-yl)(2-(((2,2,6,6-tetramethylpiperidin-1-yl)oxy)methyl)phenyl)carbamate 3v