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SUPPLEMENTARY INFORMATION

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Photochemical generation of radicals from alkyl electrophiles using a nucleophilic organic catalyst

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Supplementary Information for

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A. General Information

The NMR spectra were recorded at 400 MHz and 500 MHz for ^{1}H and 100 or 125 MHz for ^{13}C. The chemical shift (δ) for ^{1}H and ^{13}C are given in ppm relative to residual signals of the solvents ($CHCl_3$ @ 7.26 ppm ^{1}H NMR and 77.16 ppm ^{13}C NMR, and tetramethylsilane @ 0 ppm). Coupling constants are given in Hertz. The following abbreviations are used to indicate the multiplicity: s, singlet; d, doublet; q, quartet; m, multiplet; bs, broad signal; app, apparent.
Infrared (IR) spectra were obtained using a Bruker Alpha FT-IR spectrometer.
High resolution mass spectra (HRMS) were obtained from the ICIQ HRMS unit on MicroTOF Focus and Maxis Impact (Bruker Daltonics) with electrospray ionization. (ESI). X-ray data were obtained from the ICIQ X-Ray unit using a Brucker-Nonius diffractometer equipped with an APPEX 2 4K CCD area detector.
UV-vis measurements were carried out on a Shimadzu UV-2401PC spectrophotometer equipped with photomultiplier detector, double beam optics and D_2 and W light sources or an Agilent Cary60 spectrophotometer. Emission spectra of light sources were recorded on Ocean Optics USB4000 fiber optic spectrometer.
Isolated yields refer to materials of >95% purity as determined by ^{1}H NMR.

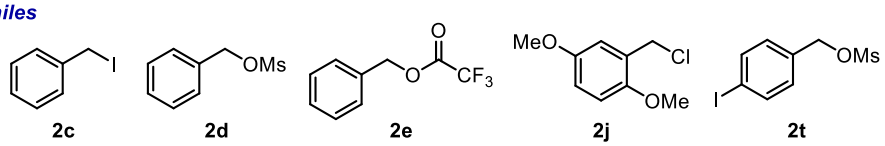
The authors are indebted to the team of the Research Support Area at ICIQ, particularly to the NMR, the X-ray Diffraction, and the High Resolution Mass Spectrometry Units. Grace Fox is thanked for proofreading the manuscript.

General Procedures. All reactions were set up under an argon atmosphere in oven-dried glassware using standard Schlenk techniques, unless otherwise stated. Synthesis grade solvents were used as purchased; anhydrous solvents were taken from a commercial SPS solvent dispenser. Chromatographic purification of products was accomplished using forced-flow chromatography (FC) on silica gel (35-70 mesh). For thin layer chromatography (TLC) analysis throughout this work, Merck pre-coated TLC plates (silica gel 60 GF_{254}, 0.25 mm) were employed, using UV light as the visualizing agent and an acidic mixture of vanillin or basic aqueous potassium permanganate ($KMnO_4$) stain solutions, and heat as developing agents. Organic solutions were concentrated under reduced pressure on a Büchi rotatory evaporator.

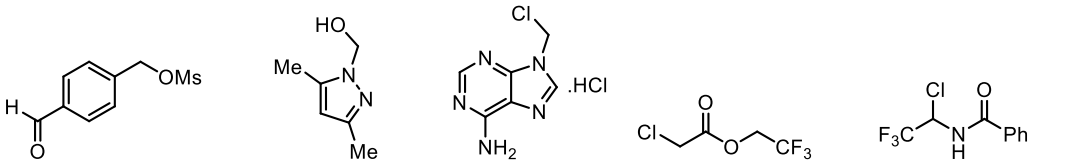
Materials. Most of the starting materials used in this study are commercial and were purchased in the highest purity available from Sigma-Aldrich, Fluka, Alfa Aesar, Fluorochem, and used as received, without further purifications.

The substrates reported in Supplementary Figure 1 below were synthesized according to procedures reported in the literature.

Electrophiles

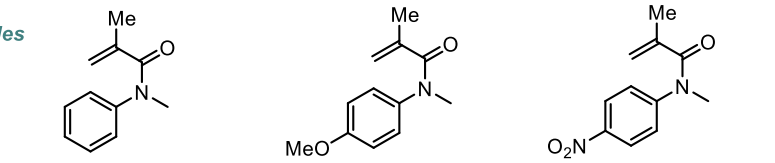


2c (Reference 1) | **2d** (Reference 2) | **2e** (Reference 3) | **2j** (Reference 4) | **2t** (Reference 5)

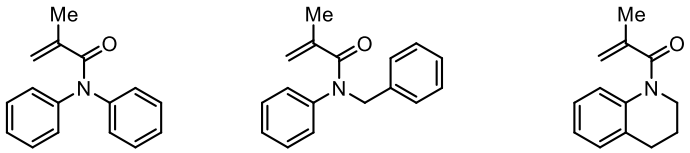


2w (Reference 6) | **(3,5-dimethyl-1*H*-pyrazol-1-yl)methanol** (Reference 7) | **16** (Reference 8) | **2,2,2-Trifluoro Chloroacetate** (Reference 9) | ***N*-(1-chloro-2,2,2-trifluoroethyl)benzamide** (Reference 10)

Methacrylamides



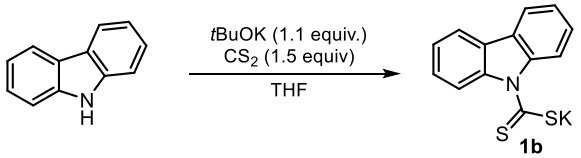
***N*-methyl-*N*-phenylmethacrylamide** (Reference 11) | ***N*-(4-methoxyphenyl)-*N*-methylmethacrylamide** (Reference 11) | ***N*-methyl-*N*-(4-nitrophenyl)methacrylamide** (Reference 11)



***N*,*N*-diphenylmethacrylamide** (Reference 12) | ***N*-benzyl-*N*-phenylmethacrylamide** (Reference 11) | **1-(3,4-dihydroquinolin-1(2*H*)-yl)-2-methylprop-2-en-1-one** (Reference 11)

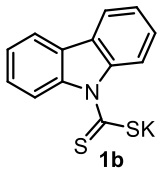
Supplementary Figure 1: Starting materials synthesized according to literature precedents and corresponding references.

B. Synthesis of the Dithiocarbamate Anion Catalysts 1
Potassium 9H-carbazole-9-carbodithioate (1b)



In an oven dried round bottom flask under an atmosphere of argon, carbazole (3 g, 17.94 mmol, 1 equiv.) was dissolved in a minimal amount of THF (30 mL, commercial synthesis grade). The flask was cooled to 0 ºC in an ice-water bath and potassium *tert*-butoxide (2.1 g, 1.1 equiv.) was added portionwise. The mixture turned a slight yellow/orange color and was left to stir for 30 minutes. Still at 0 ºC, carbon disulfide (1.6 mL, 1.5 equiv.) was added dropwise *via* syringe addition. The mixture immediately turned a bright orange color and was left to stir for one hour at 0 ºC. After warming up to ambient temperature, THF was evaporated carefully on a rotary evaporator to a thick syrupy consistency. Diethyl ether (50 mL) was added and the resulting suspension stirred vigorously to free the solid. The yellow solid was then filtered under a flow of argon, washed twice with a small amount of ether and further dried overnight under high vacuum to obtain the desired product as a bright yellow free flowing powder (4.9 g, 97% yield).

Note: Once isolated, **1b** is slightly hygroscopic and should therefore be protected from ambient humidity. It was typically stored in brown glass bottles within a desiccator. Only minor degradation to the parent carbazole was observed over a 3-month period, as indicated by NMR analysis. **1b** could be readily purified again by washing the solid with diethyl ether to remove the carbazole from the yellow solid.



Potassium 9H-carbazole-9-carbodithioate (1b)

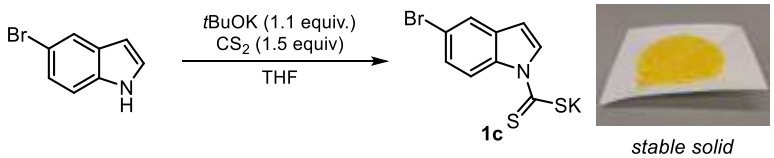
^{1}H NMR (400 MHz, d6-DMSO) δ 8.49 (d, *J*=8.3 Hz, 2H); 8.08 (d, *J*=7.6 Hz, 2H); 7.37 (app t, *J*=7.9 Hz, 2H); 7.19 (app t, *J*=7.9 Hz, 2H);

^{13}C NMR (101 MHz, d6-DMSO) δ 221.0 (C), 139.7 (C), 125.3 (CH); 123.1 (C); 120.0 (CH); 119.6 (CH); 115.3 (CH).

HRMS (ESI negative): calculated for $C_{13}H_8NS_2$ (M): 242.0104 found 242.0108.

Using the catalog prices of the suppliers the chemicals were obtained from, a cost of 0.54 €·g^{-1} or 150 €·mol^{-1} in materials (excluding labor) was calculated.

Potassium 5-bromo-1*H*-indole-1-carbodithioate (1c):



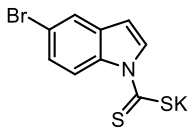
stable solid

In an oven dried round bottom flask under an atmosphere of argon, *5*-bromoindole (1 equiv.) was dissolved in a minimal amount of THF (commercial synthesis grade; 10 mL·g^{-1}). The flask was cooled to 0 ºC in an ice-water bath and potassium *tert*-butoxide (1.1 equiv.) was added portionwise. The mixture turned a slight yellow/orange color and was left to stir for 30 minutes. Still at 0 ºC, carbon disulfide (1.5 equiv.) was added dropwise *via* syringe addition. The mixture immediately turned a bright orange color and was left to stir for one hour at 0 ºC. After warming up to ambient temperature, THF was

evaporated carefully on a rotary evaporator to a thick syrupy consistence. Toluene was added and evaporated to obtain a yellow solid residue. The residue was suspended in a 1:1 mixture of pentane and diethyl ether and stirred vigorously to obtain a fine suspension. The yellow solid was then filtered under a flow of argon, washed twice with a small amount of a 1:1 mixture of pentane and diethyl ether and further dried overnight under high vacuum to obtain the desired product as a yellow free flowing powder.

This reaction has been performed on various scales (5 to 55 mmol of starting *5*-bromoindole) with isolated yields ranging from 88 to 95%.

Note: Once isolated, **1c** is slightly hygroscopic and should therefore be protected from ambient humidity. It was typically stored in brown glass bottles in a desiccator. No degradation was detected over a 3-month period, as indicated by NMR analysis.



Potassium 5-bromo-1*H*-indole-1-carbodithioate (1c)

^{1}H NMR (400 MHz, d6-DMSO) δ 6.39 (d, *J*=3.5 Hz, 1H), 7.25 (dd, J= 9, 2.1 Hz, 1H), 7.68 (d, J=2.1 Hz, 1H), 8.69 (d, J= 3.5 Hz, 1H), 9.28 (d, J= 9 Hz, 1H)

^{13}C NMR (101 MHz, d6-DMSO) δ 218.7 (C), 134.9 (C), 133.9 (C), 132.2 (CH), 124.9 (CH), 122.7 (CH), 120.0 (CH), 113.5 (C), 102.0 (CH)

IR (thin film) ν 1744, 1438, 1354, 1292, 1204, 1174, 1065, 1000, 829, 724 cm^{-1}.

HRMS (ESI negative): calculated for $C_9H_5{}^{79}BrNS_2$ (M): 269.9052, found 269.9044.

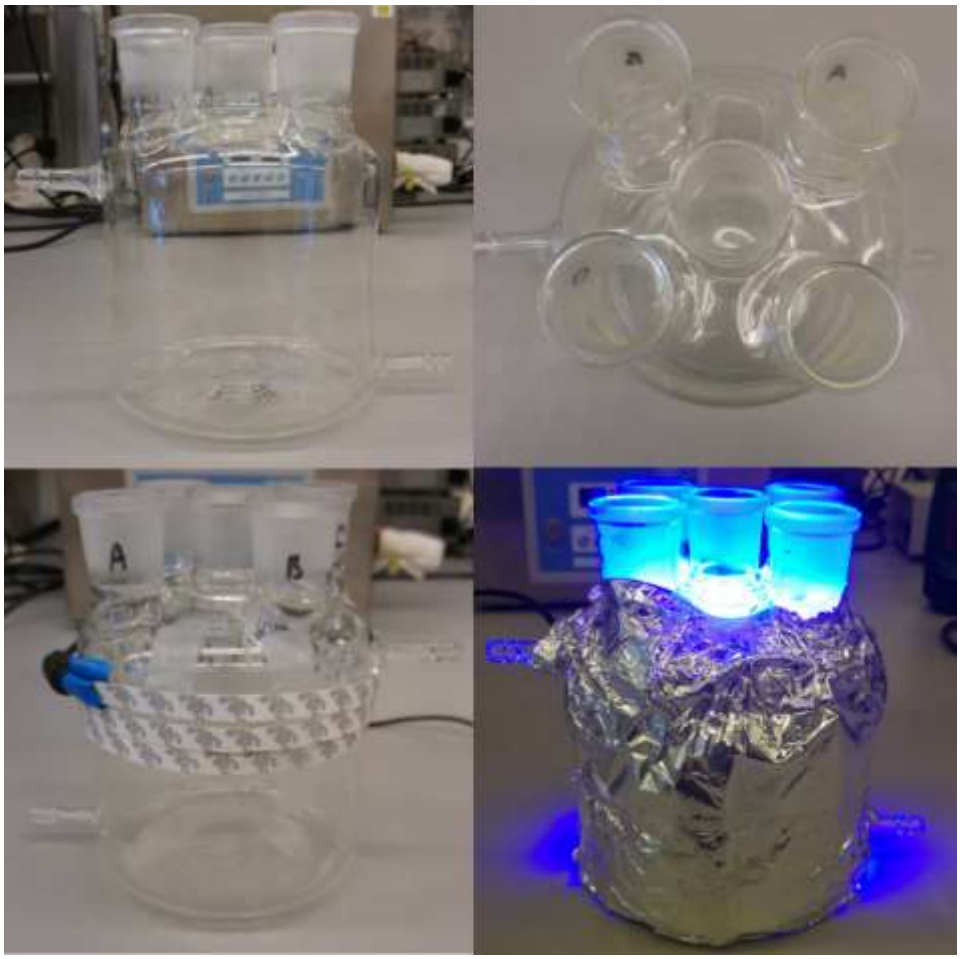
Using the catalog prices of the suppliers the chemicals were obtained from, a cost of 0.63 €·g^{-1} or 192 €·mol^{-1} in materials (excluding labor) was calculated.

We also synthesized the simple indole-containing dithiocarbamate catalyst (potassium 1*H*-indole-1-carbodithioate). However, this compound is highly hygroscopic, which prevented its convenient handling and use under an open atmosphere. After screening of several substitution patterns on the indole scaffold, 5-bromoindole derivative **1c** proved optimal in terms of stability and handling while giving excellent results in the catalytic reactions.

C. Experimental Setup

C.1. Temperature-controlled photoreactor.

The photoreactor consisted of a 12.5 cm diameter jar, fitted with 4 standard 29 sized ground glass joints arranged in a square and a central 29 sized joint. A commercial 1 meter LED strip was wrapped around the jar, followed by a layer of aluminium foil and cotton for insulation (Supplementary Supplementary Figure 2).



Supplementary Figure 2: Photoreactor used in this study - pictures taken at different stages of construction.

Each of the joints could be used to fit a standard 16 mm or 25 mm diameter Schlenk tube with a Teflon adaptor (Supplementary Supplementary Figure 3).

Supplementary Figure 3: Teflon adaptors used to accommodate Schlenk tubes in the photoreactor.

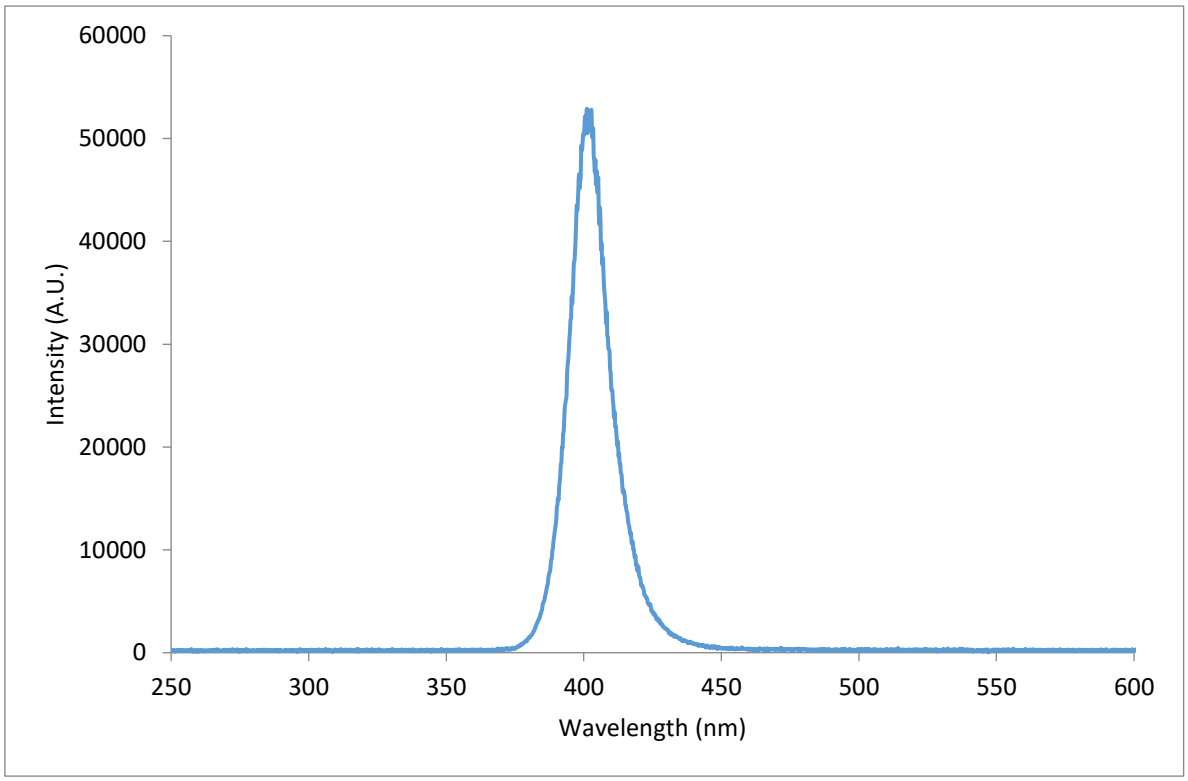
An inlet and an outlet allow the circulation of liquid from a Huber Minichiller 300 inside the jar. This setup allows to perform reactions at temperatures ranging from -20 ºC to 80 ºC with accurate control of the reaction temperature (± 1ºC) (Supplementary Supplementary Figure 4).

Supplementary Figure 4: Fully assembled photoreactor in operation.

To maintain a consistent illumination during different experiments, only the four external positions were used to perform reactions while the central one was used to monitor the temperature inside a Schlenk tube identical to those used to perform reactions.

C.2. Emission spectra of the light sources used in this study

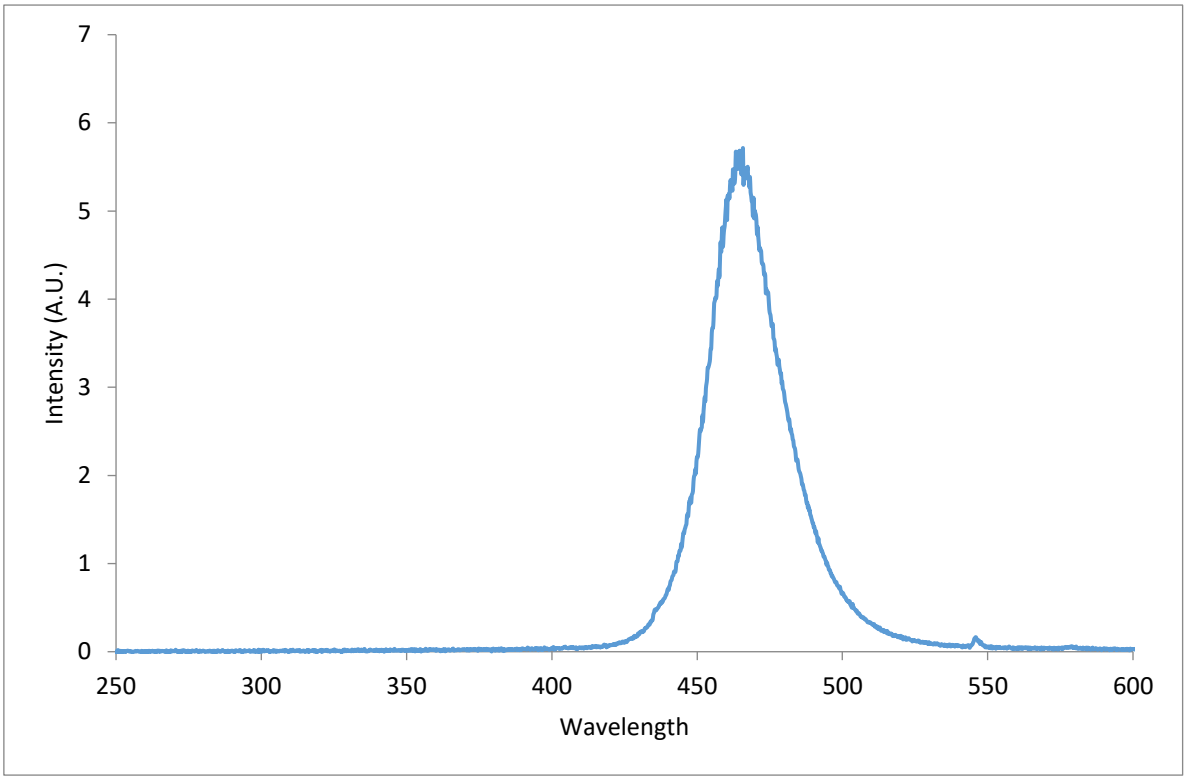
Experiments at *400 nm* were conducted using a 1m strip, 7.2W "Paulmann Function BlackLight LED Strip" purchased from Amazon. The emission spectrum of these LEDs was recorded (Supplementary Supplementary Figure 5).



Supplementary Figure 5: Emission spectrum of the 400 nm LED strip used in this study.

The emission maximum was determined as 400 nm with a spectral width of 20 nm (390-410 nm) at half peak intensity and a total spectral width of 80 nm (370-450 nm).

Experiments at *465 nm* were conducted using a 1m strip, 14.4W 'LEDXON MODULAR 9009083 LED, SINGLE 5050' purchased from Farnell, catalog number 9009083. The emission spectrum of these LEDs was recorded (Supplementary Supplementary Figure 6).

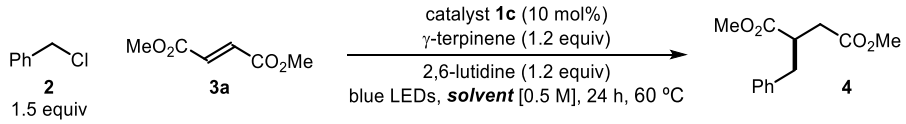


Supplementary Figure 6: Emission spectrum of the 465 nm *blue LED strip* used in this study.

The emission maximum was determined as 465 nm with a spectral width of 30 nm (450-480 nm) at half peak intensity and a total spectral width of 120 nm (420-540 nm).

D. Dithiocarbamate Anion Catalyzed Conjugate Radical Addition

Supplementary Table 1: solvent screen

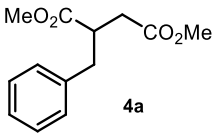


entry	solvent	NMR yield (%)
1	MeCN	90
2	AcOEt	88
3	DCE	86
4	MTBE	90
5	Toluene	87

All reaction performed on a 0.5 mmol scale, yield determined by ^{1}H NMR analysis of the crude reaction mixture by comparison with trichloroethylene as internal standard.

D.1. General Procedure

In an oven dried Schlenk tube, catalyst **1c** (15.5 mg, 0.05 mmol, 0.1 equiv.) was dissolved in acetonitrile (1 mL, HPLC grade), then the alkyl electrophile **2** (0.75 mmol, 1.5 equiv.) was added followed by 2,6-lutidine (70 μL, 0.6 mmol, 1.2 equiv.), γ-terpinene (96 μL, 0.6 mmol, 1.2 equiv.), and the Michael acceptor **3** (0.5 mmol, 1 equiv.). The resulting yellow mixture was degassed via three cycles of freeze-pump-thaw. The Schlenk tube was then placed in the irradiation setup (see section C.1), maintained at a temperature of 60 ºC (60-61ºC measured in the central well), and the reaction was stirred for 24 hours under continuous irradiation. After cooling to ambient temperature, the solvent was evaporated and the residue purified by column chromatography to afford the corresponding product **4** in the stated yield with >95% purity according to ^{1}H NMR analysis. The exact conditions for chromatography are reported for each compound.



Dimethyl 2-benzylsuccinate (4a): Synthesized according to the general procedure using benzyl chloride **2a** (60 μL, 0.75 mmol, 1.5 equiv.) and dimethyl fumarate **3a** (72 mg, 0.5 mmol, 1 equiv.). Chromatography on silica gel (gradient from pure toluene to 2% AcOEt in toluene as eluent): 92 mg, 78% yield, clear oil.

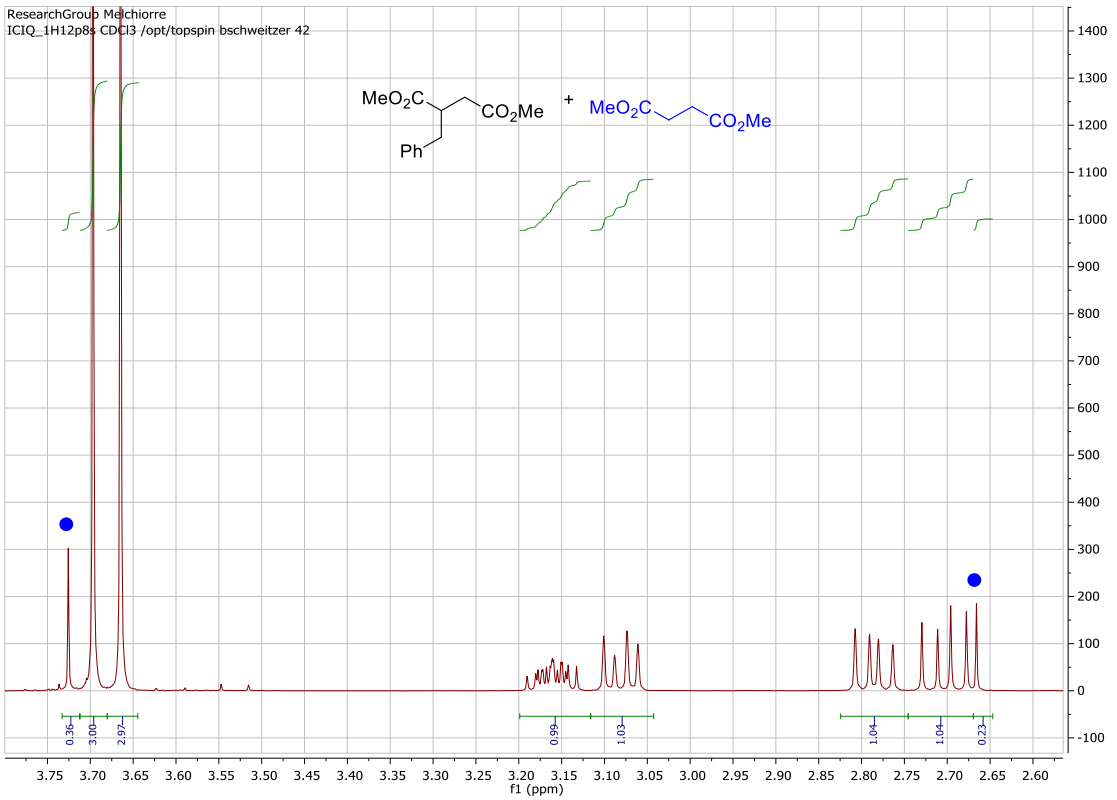
^{1}H NMR (500 MHz, $CDCl_3$) δ 7.34 – 7.27 (m, 2H), 7.26 – 7.21 (m, 1H), 7.19 – 7.14 (m, 2H), 3.68 (s, 3H), 3.66 (s, 3H), 3.19 – 3.11 (m, 1H), 3.07 (dd, *J* = 13.5, 6.3 Hz, 1H), 2.78 (dd, *J* = 13.5, 8.5 Hz, 1H), 2.70 (dd, *J* = 16.8, 9.2 Hz, 1H), 2.43 (dd, *J* = 16.8, 5 Hz, 1H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 174.6 (C), 172.2 (C), 138.1 (C), 129.0 (CH), 128.5 (CH), 126.7 (CH), 51.9 (CH_3), 51.7 (CH_3), 43.0 (CH), 37.7 (CH_2), 34.9 (CH_2).

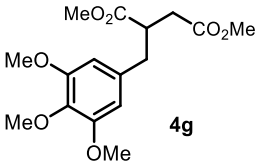
Matching reported literature data[13].

Note: in the reported optimization experiments, dimethyl succinate is formed in amounts ranging from 5 to 10% according to ^{1}H NMR analysis of the crude mixture and cannot be easily separated from the desired benzylated product. Therefore, isolated samples of product **3** commonly contain a small amount (2 to 5%) of dimethyl succinate (see Supplementary Supplementary Figure 7) if the chromatography is not performed carefully. In order to remove those impurities, long chromatographic purification is

required, which still gives mixtures in the initial fractions, resulting in losses of yield and explaining the significant difference between NMR and isolated yield of product **4a**.



Supplementary Figure 7: Example of an isolated product **4a** containing 5% of dimethylsuccinate impurity.



4g

Dimethyl 2-(3,4,5-trimethoxybenzyl)succinate (4g): Synthesized according to the general procedure using 3,4,5-trimethoxybenzyl chloride **2g** (162 mg, 0.75 mmol, 1.5 equiv.) and dimethyl fumarate **3a** (72 mg, 0.5 mmol, 1 equiv.). Chromatography on silica gel (gradient from 20% to 30% AcOEt in hexanes as eluent): 139 mg slowly crystallizing, slightly yellow solid, 85% yield.

^{1}H NMR (500 MHz, $CDCl_3$) δ 6.34 (s, 2H); 3.81 (s, 6H); 3.79 (s, 3H); 366 (S, 3H); 3.63 (s, 3H); 3.14-3.05 (m, 1H); 2.98 (dd, *J* = 13.6, 6.4 Hz, 1H); 2.71-2.61 (m, 2H); 2.41 (dd, *J* = 16.6, 5.1 Hz, 1H).

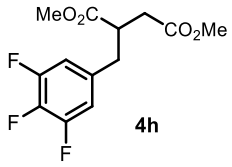
^{13}C NMR (126 MHz, $CDCl_3$) δ 174.5 (C); 172.2 (C); 153.2 (C); 136.7 (C); 133.8 (C); 105.9 (CH); 60.7 (CH_3); 56.0 (CH_3); 51.9 (CH_3); 51.7 (CH_3); 43.0 (CH), 38.0 (CH_2); 34.9 (CH_2).

IR (thin film) ν 2919, 1735, 1590, 1508, 1437, 1338, 1239, 1157, 1127, 1008 cm^{-1}.

HRMS (ESI pos): calculated for $C_{16}H_{22}NaO_7$ ($M+Na^+$): 349.1258, found 349.1267.

The same reaction was performed on *7.5 mmol scale*: to an oven dried 25 mL Schlenk tube, **1c** (233 mg, 0.75 mmol, 0.1 equiv.) was dissolved in acetonitrile (15 mL), then 3,4,5-trimethoxybenzyl chloride **2g** (2.437 g, 11.25 mmol, 1.5 equiv.) was added followed by 2,6-lutidine (1,05 mL, 9 mmol, 1.2 equiv.), γ-terpinene (1.44 mL, 9 mmol, 1.2 equiv.) and dimethyl fumarate **3a** (1.081 g, 7.5 mmol, 1 equiv.). The resulting yellow

mixture was degassed via three cycles of freeze-pump-thaw. The Schlenk tube was then placed in the irradiation setup set at a temperature of 60 ºC and irradiated for 24 hours. After cooling to ambient temperature, the reaction mixture was transferred to an extraction funnel containing 25 mL of distilled water and 25 mL of AcOEt. The phases were separated and the aqueous phase extracted twice with AcOEt (25 mL each). The combined organic fractions were combined, washed with brine, dried with $MgSO_4$ and concentrated to dryness. The resulting yellow oil was purified by column chromatography on silica gel (100 g silica, gradient from 20% to 30% AcOEt in hexanes as eluent, R_f = 0,2 @ 20% AcOEt in hexanes, staining red with vanillin stain solution) to afford **4g** as a slowly crystalizing slightly yellow oil: 2.13 g (87% yield).



4h

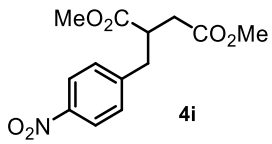
Dimethyl 2-(3,4,5-trifluorobenzyl)succinate (4h): Synthesized according to the general procedure using 5-(bromomethyl)-1,2,3-trifluorobenzene **2h** (169 mg, 0.75 mmol, 1.5 equiv.) and dimethyl fumarate **3a** (72 mg, 0.5 mmol, 1 equiv.). Chromatography on silica gel (gradient from toluene to 2% AcOEt in toluene as eluent): 116 mg slightly yellow oil, 80% yield.

^{1}H NMR (500 MHz, $CDCl_3$) δ 6.86-6.74 (m, 2H); 3.69 (s, 3H); 3.68 (s, 3H); 3.14-3.05 (m, 1H); 2.96 (dd, *J* = 13.7, 7.4 Hz, 1H); 2.77 (dd, *J* = 13.7, 7.2 Hz, 1H); 2.70 (dd, *J* = 16.7, 8.2 Hz, 1H); 2.44 (dd, *J* = 16.7, 5.9 Hz, 1H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 172. (d, *J*=206.4 Hz, C); 151.1 (ddd; *J*=250.2, 9.8, 4 Hz, C); 140.1 (td, *J*=250.2, 15.4 Hz, C); 134.9-134.4 (m, C);112.9 (dd, *J*=15.4, 5.4 Hz, C); 52.1 (CH_3); 51.9 (CH_3); 42.6 (CH), 36.7 (CH_2); 34.9 (CH_2).

^{19}F NMR (376 MHz, $CDCl_3$, proton decoupled) δ -134.03 (d, J=20.6 Hz, 2F); -162.67 (t, J=20.6 Hz), 1F).

HRMS (ESI pos): calculated for $C_{13}H_{13}F_3NaO_4$ ($M+Na^+$): 313.0658, found 313.0666.



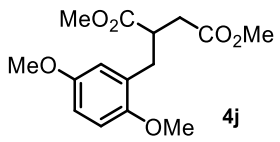
4i

Dimethyl 2-(4-nitrobenzyl)succinate (4i): Synthesized according to a modification of the general procedure: in an oven dried Schlenk tube, catalyst **1c** (15.5 mg, 0.1 equiv.) was dissolved in acetonitrile (1 mL, HPLC grade), then 4-nitrobenzyl bromide **2i** (216 mg, 1 mmol, 2 equiv.) was added followed by 2,6-lutidine (116 μL, 1 mmol, 2 equiv.), γ-terpinene (160 μL, 2 mmol, 2 equiv.), and dimethyl fumarate **3a** (72 mg, 0.5 mmol, 1 equiv.). The resulting yellow mixture was degassed via three cycles of freeze-pump-thaw. The Schlenk tube was then placed in the irradiation setup set at a temperature of 60ºC (60-61ºC measured in the central well) and irradiated for 24 hours. After cooling to ambient temperature, the solvent was evaporated and the residue purified by column chromatography (gradient from 15% to 30% AcOEt in hexanes as eluent): 112 mg slightly yellow oil, 80% yield.

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.18 (app d, *J*= 8.5 Hz, 2H), 7.37 (app d, *J*= 8.5 Hz, 2H), 3.69 (s, 3H), 3.67 (s, 3H), 3.24 – 3.06 (m, 2H), 2.95 (dd, *J* = 13.0, 6.6 Hz, 1H), 2.73 (dd, *J* = 16.6, 7.6 Hz, 1H), 2.46 (dd, *J* = 16.6, 5.4 Hz, 1H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 173.8 (C), 171.7 (C), 146.0 (C), 129.8 (CH), 123.7 (CH), 52.1 (CH_3), 51.9 (CH_3), 42.6 (CH), 37.3 (CH_2), 35.1 (CH_2).

IR (thin film) ν 2954, 1731, 1604, 1518, 1437, 1345, 1199, 1164, 1100, 855 cm^{-1}.
HRMS (ESI pos): calculated for $C_{13}H_{15}NaO_6$ ($M+Na^+$): 304.0792, found 304.0805.



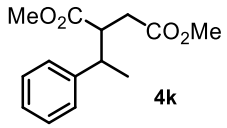
Dimethyl 2-(2,5-dimethoxybenzyl)succinate (4j): Synthesized according to the general procedure using 2-(chloromethyl)-1,4-dimethoxybenzene **2j** (140 mg, 0.75 mmol, 1.5 equiv.) and dimethyl fumarate **3a** (72 mg, 0.5 mmol, 1 equiv.). Chromatography on silica gel (20% AcOEt in hexanes as eluent): 127 mg slightly yellow oil, 86% yield.

^{1}H NMR (400 MHz, $CDCl_3$) δ 6.8-6.71 (m, 2H); 6.67 (d, *J*=2.8 Hz, 1H); 3.78 (s, 3H); 3.76 (s, 3H); 3.69 (s, 3H); 3.64 (s, 3H); 3.26-3.16 (m, 1H); 3.03 (dd, *J*=13.2, 6.2 Hz, 1H); 2.78 (dd, *J*=13.2, 8.6 Hz, 1H); 2.68 (dd, *J*=16.9, 9.6 Hz, 1H); 2.42 (dd, *J*=16.9, 4.6 Hz, 1H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 175.0 (C); 172.5 (C); 153.3 (C); 151.9 (C); 127.6 (C); 117.0 (C); 112.2 (C); 111.1 (C); 55.7 (CH_3); 55.7 (CH_3); 51.8 (CH_3); 51.6 (CH_3); 41.3 (CH); 34.9 (CH_2); 32.5 (CH_2).

IR (thin film) ν 2951, 2835, 1731, 1500, 1436, 1222, 1156, 1045, 804, 710 cm^{-1}.

HRMS (ESI pos): calculated for $C_{15}H_{20}NaO_6$ ($M+Na^+$): 31.1152, found 319.1140.

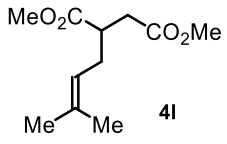


dimethyl 2-(4-fluorobenzyl)succinate (4k): Synthesized according to the general procedure using (1-bromoethyl)benzene **2k** (102 μL, 1.5 equiv.) and dimethyl fumarate **3a** (72 mg, 1 equiv.). The diastereomeric ratio was determined by ^{1}H spectroscopic analysis of the crude reaction mixture by comparison of the resonances at δ 2.34 (minor diastereomer) and δ 2.20 (major diastereomer). Chromatography on silica gel (gradient from pure toluene to 5% AcOEt in toluene as eluent): 61 mg yellow oil, 49% yield and 1:1.2 d.r.

^{1}H NMR for diastereomeric mixture (500 MHz, $CDCl_3$) δ 7.32-7.28 (m, 2H), 7.24-7.15 (m, 3H), 3.74 (s, 3H-major), 3.61 (s, 3H-minor), 3.56 (s, 3H), 3.25-3.20 (m, 1H-minor), 3.14-3.10 (m, 1H-minor), 3.02-2.97 (m, 1H-major), 2.93-2.87 (m, 1H-major), 2.74 (dd, *J* = 16.8, 10.9 Hz, 1H-minor), 2.59 (dd, *J* = 16.9, 11.0 Hz, 1H-major), 2.34 (dd, *J* = 16.8, 3.8 Hz, 1H-minor), 2.20 (dd, *J* = 16.9, 3.8 Hz, 1H-major), 1.28 (d, *J* = 2.3 Hz, 3H-major), 1.27 (d, *J* = 2.1 Hz, 3H-minor).

^{13}C NMR (126 MHz, $CDCl_3$) δ 175.4, 174.6, 173.0, 172.7, 143.9, 143.5, 129.1, 128.8, 127.8, 127.7, 127.3, 127.1, 52.1, 52.1, 52.0, 52.0, 48.7, 48.5, 42.8, 41.1, 35.7, 32.7, 20.6, 16.7.

HRMS (ESI pos): calculated for $C_{14}H_{18}NaO_4^+$ ($M+Na^+$): 273.1097, found 273.1103. Matching reported data[13].



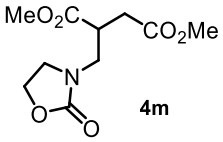
Dimethyl 2-(3-methylbut-2-en-1-yl)succinate (4l): synthesized according to a modification of the general procedure using prenyl chloride **2l** (169 μL, 3 equiv.), 2,6-lutidine (175 μL, 3 equiv.), γ-terpinene (240 μL, 3 equiv.) and dimethyl fumarate **3a** (72 mg, 1 equiv.). Chromatography on silica gel (slow gradient from 1% to 3% AcOEt in hexanes as eluent): 60 mg clear oil, 56% yield.

^{1}H NMR (500 MHz, $CDCl_3$) δ 5.07 (app t; *J*=7.5 Hz, 1H); 3.71 (s, 3H); 3068 (s, 3H); 2.93-2.85 (m, 1H); 2.70 (dd, *J*= 16.7, 9.2 Hz, 1H); 2.45 (dd, *J*=16.7, 5.2 Hz, 1H); 2.40-2.32 (m, 1H); 2.31-2.22 (m, 1H); 1.71 (s, 3H); 1.61 (s, 3H).

^{13}C NMR (125 MHz, $CDCl_3$) δ 175.0 (C); 172.6 (C); 134.9 (C); 120.0 (CH); 51.8 (CH_3); 51.7 (CH_3); 41.4 (CH); 34.9 (CH_2); 30.1 (CH_2); 25.7 (CH_3); 17.7 (CH_3).

IR (thin film) ν 2954, 2918, 1733, 1436, 1348, 1308, 1196, 1159, 1032, 1006 cm^{-1}.

HRMS (ESI pos): calculated for $C_{11}H_{18}NaO_4$ ($M+Na^+$): 237.1097, found 237.1092.



Dimethyl 2-(thiazol-4-ylmethyl)succinate (4m): Synthesized by a two-step one-pot procedure. Following a reported procedure[14], in an oven dried Schlenk tube, oxazolidin-2-one (65 mg, 0.75 mmol, 1.5 equiv.) and paraformaldehyde (23 mg, 0.75 mmol, 1.5 equiv.) were dissolved in acetonitrile (0.75 mL), TMSCl (479 µL, 3.75 mmol, 7.5 equiv.) was added dropwise and the reaction was refluxed for 16 h. Solvent was removed under vacuum at 25 ºC to obtain the crude 3-(chloromethyl)oxazolidin-2-one **2m** as a white solid that was used without further purification.

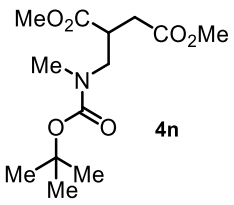
In the same Schlenk tube, **1c** (15.5 mg, 0.1 equiv.) was dissolved in acetonitrile (1 mL, HPLC grade), followed by addition of 2,6-lutidine (70 µL, 0.6 mmol, 1.2 equiv.), γ-terpinene (96 µL, 0.6 mmol, 1.2 equiv.) and dimethyl fumarate (72 mg, 0.5 mmol, 1 equiv.). The resulting yellow mixture was degassed via three cycles of freeze-pump-thaw. The Schlenk tube was then placed in the irradiation setup set at a temperature of 60 ºC and irradiated for 24 hours. After cooling to ambient temperature, the solvent was evaporated and the residue purified by column chromatography (50% acetone in hexanes as eluent): 100 mg yellow oil, 82% yield.

^{1}H NMR (500 MHz, $CDCl_3$) δ 4.32-4.22 (m, 2H), 3.68 (s, 3H); 3.64 (s, 3H); 3.59-3.50 (m, 3H), 3.44 (dd, *J*=6.4, 14.2 Hz, 1H) 3.16-3.06 (m, 1H); 2.75 (dd, *J*=7.9, 17.1 Hz, 1H); 2.52 (dd, *J*=5.7, 17.1 Hz, 1H)

^{13}C NMR (125 MHz, $CDCl_3$) δ 173 (C); 171.9 (C); 158.7 (C); 62.0 (CH_2); 52.4 (CH_3); 52.0 (CH_3); 45.7 (CH_2); 45.2 (CH_2); 40.0 (CH); 33.4 (CH_2).

IR (thin film) ν 3601, 3502, 2993, 1727, 1486, 1372, 1201, 1100, 1043, 891 cm^{-1}.

HRMS (ESI pos): calculated for $C_{10}H_{15}NNaO_6$ ($M+Na^+$): 268.0792, found 268.0796.



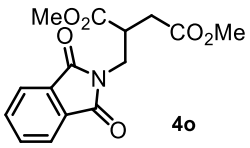
Dimethyl 2-(((*tert*-butoxycarbonyl)(methyl)amino)methyl)succinate (4n): Synthesized by a two-step procedure. Following a reported procedure[15], in a round bottom flask, *tert*-butyl methylcarbamate (131 mg, 1 mmol, 1 equiv.) and paraformaldehyde (48 mg, 1.6 mmol, 1.6 equiv.) was dissolved in dry DCM (4 mL). TMSCl (383 µL, 3 mmol, 3 equiv.) was added dropwise and the reaction was left stirring at ambient temperature for 2 hours. Solvent was removed under vacuum at 25 ºC to obtain the crude *tert*-butyl (chloromethyl)(methyl)carbamate **2n** as a colorless oil that was used without further purification. The crude product **2n** was dissolved in acetonitrile (HPLC grade) giving a stock solution 0.75 M. The crude *tert*-butyl (chloromethyl)(methyl)carbamate **2n** (0.75 M, 0.75 mmol, 1.5 eq.) was placed in an oven dried Schlenk tube, dissolved in 1 mL of acetonitrile (HPLC grade) followed by the addition of **1c** (15.5 mg, 0.1 equiv.), 2,6-lutidine (70 µL, 0.6 mmol, 1.2 equiv.), γ-terpinene (96 µL, 0.6 mmol, 1.2 equiv.), and dimethyl fumarate (72 mg, 0.5 mmol, 1 equiv.). The resulting yellow mixture was degassed via three cycles of freeze-pump-thaw. The Schlenk tube was then placed in the

irradiation setup set at a temperature of 60 ºC and irradiated for 24 hours. After cooling to ambient temperature, the solvent was evaporated and the residue purified by column chromatography (25% AcOEt in hexanes as eluent): 104 mg yellow oil, 72% yield.

^{1}H NMR (500 MHz, $CDCl_3$) δ 3.67 (s, 3H); 3.64 (s, 3H); 3.43 (app d, *J* = 6.9 Hz, 2H) 3.15-3.07 (m, 1H); 3.81 (s, 3H); 2.75-2.63 (m, 1H); 2.45 (dd, *J*=5.2, 17 Hz, 1H); 1.42 (s, 9H).

^{13}C NMR (125 MHz, $CDCl_3$) δ 173.7 (C); 172.2 (rotamer); 172.0 (C, rotamer); 155.9 (C, rotamer); 155.5 (C, rotamer); 80.1 (C, rotamer); 79.8 (C, rotamers); 52.2 (CH_3); 51.9 (CH_3); 50.4-50.2 (CH_2, rotamers); 40.6 (CH, rotamer); 40.4 (CH, rotamer); 35.2 (CH_3, rotamer); 34.7 (CH_3, rotamer); 33.4 (CH_2); 28.4 (CH_3).

HRMS (ESI pos): calculated for $C_{13}H_{23}NNaO_6$ ($M+Na^+$): 312.1418, found 312.1416.



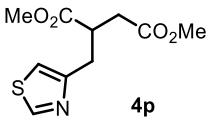
Dimethyl 2-((1,3-dioxoisoindolin-2-yl)methyl)succinate (4o): Synthesized according to the general procedure using 2-(chloromethyl)isoindoline-1,3-dione **2o** (147 mg, 1.5 equiv.) and dimethyl fumarate **3a** (72 mg, 1 equiv.). Chromatography on silica gel (10% AcOEt in toluene as eluent): 127 mg white solid, 83% yield.

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.89-7.83 (m, 2H); 7.77-7.71 (m, 2H); 4.05 (dd, *J*= 14; 6.7 Hz, 1H); 3.94 (dd, *J*= 14; 6.4 Hz, 1H); 3.71 (s, 3H); 3.66 (s, 3H); 3.35-3.26 (m, 1H); 2.79 (dd, *J*= 17.2; 8.8 Hz, 1H); 2.59 (dd, *J*= 17.2; 5.2 Hz, 1H).

^{13}C NMR (125 MHz, $CDCl_3$) δ 172.5 (C); 171.6 (C); 168.0 (C); 134.1 (CH); 131.8 (C); 123.4 (CH); 52.4 (CH_3); 51.9 (CH_3); 40.4 (CH); 38.6 (CH_2); 33.3 (CH_2).

IR (thin film) ν 2954, 1774, 1709, 1437, 1396, 1366, 1267, 1168, 1007, 720 cm^{-1}.

HRMS (ESI pos): calculated for $C_{15}H_{15}NaO_6$ ($M+Na^+$): 328.0792, found 328.0801.



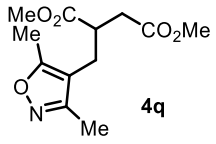
Dimethyl 2-(thiazol-4-ylmethyl)succinate (4p): Synthesized according to a modification of the general procedure: in an oven dried Schlenk tube, 4-(chloromethyl)thiazole hydrochloride **2p** (128 mg, 0.75 mmol, 1.5 equiv.) was dissolved in acetonitrile (1 mL, HPLC grade), then 2,6-lutidine (157 μL, 1.35 mmol, 2.7 equiv.) was added followed by **1c** (15.5 mg, 0.1 equiv.), γ-terpinene (96 μL, 0.6 mmol, 1.2 equiv.), and dimethyl fumarate **3a** (72 mg, 0.5 mmol, 1 equiv.). The resulting yellow mixture was degassed via three cycles of freeze-pump-thaw. The Schlenk tube was then placed in the irradiation setup set at a temperature of 60 ºC and irradiated for 24 hours. After cooling to ambient temperature, the solvent was evaporated and the residue purified by column chromatography (15% acetone in hexanes as eluent): 74 mg yellow oil, 61% yield.

^{1}H NMR (500 MHz, $CDCl_3$) δ 8.72 (d, 1H); 7.01-6.99 (m, 1H); (s, 1H); 3.65 (s, 3H); 3.62 (s, 3H); 3.36-3.28 (m, 1H); 3.22 (dd, *J*=6.3, 14.4 Hz, 1H); 3.04 (dd, *J*=7.7, 14.4 Hz, 1H); 2.69 (dd, *J*=8.7, 16.7 Hz, 1H); 2.50 (dd, *J*=5.1, 16.7 Hz, 1H).

^{13}C NMR (125 MHz, $CDCl_3$) δ 174.5 (C); 172.2 (C); 154.3 (C); 152.8 (CH); 114.9 (CH); 52.1 (CH_3); 51.8 (CH_3); 41.3 (CH); 35.1 (CH_2); 32.9 (CH_2).

IR (thin film) ν 3310, 2953, 1727, 1435, 1358, 1261, 1194, 1095, 1060, 939 cm^{-1}.

HRMS (ESI pos): calculated for $C_{10}H_{13}NNaO_4S$ ($M+Na^+$): 266.0457, found 266.0457.



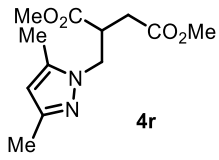
Dimethyl 2-((3,5-dimethylisoxazol-4-yl)methyl)succinate (4q): Synthesized according to the general procedure using 4-(chloromethyl)-3,5-dimethylisoxazole **2q** (93 μL, 0.75 mmol, 1.5 equiv.) and dimethyl fumarate **3a** (72 mg, 0.5 mmol, 1 equiv.). The resulting yellow mixture was degassed via three cycles of freeze-pump-thaw. The Schlenk tube was then placed in the irradiation setup at a temperature of 60 ºC and irradiated for 24 hours. After cooling to ambient temperature, the solvent was evaporated and the residue purified by column chromatography (30% AcOEt in hexanes as eluent): 100 mg yellow oil, 78% yield.

^{1}H NMR (500 MHz, $CDCl_3$) δ 3.62 (s, 3H); 3.62 (s, 3H); 2.97-2.90 (m, 1H); 2.72-2.60 (m, 2H); 2.46 (dd, *J*=7.7, 14.7 Hz, 1H); 2.39 (dd, *J*=6.2, 17.7 Hz, 1H); 2.27 (s, 3H); 2.18 (s, 3H).

^{13}C NMR (125 MHz, $CDCl_3$) δ 174.3 (C); 171.8 (C); 166.1 (C); 159.6 (C); 110.4 (C); 52.2 (CH_3); 51.9 (CH_3); 41.3 (CH); 35.4 (CH_2); 24.5 (CH_2); 11.0 (CH_3); 10.2 (CH_3).

IR (thin film) ν 2955, 1731, 1637, 1436, 1335, 1240, 1196, 1107, 1038, 890 cm^{-1}.

HRMS: calculated for $C_{12}H_{17}NNaO_5$ ($M+Na^+$): 278.0999, found 278.1009.



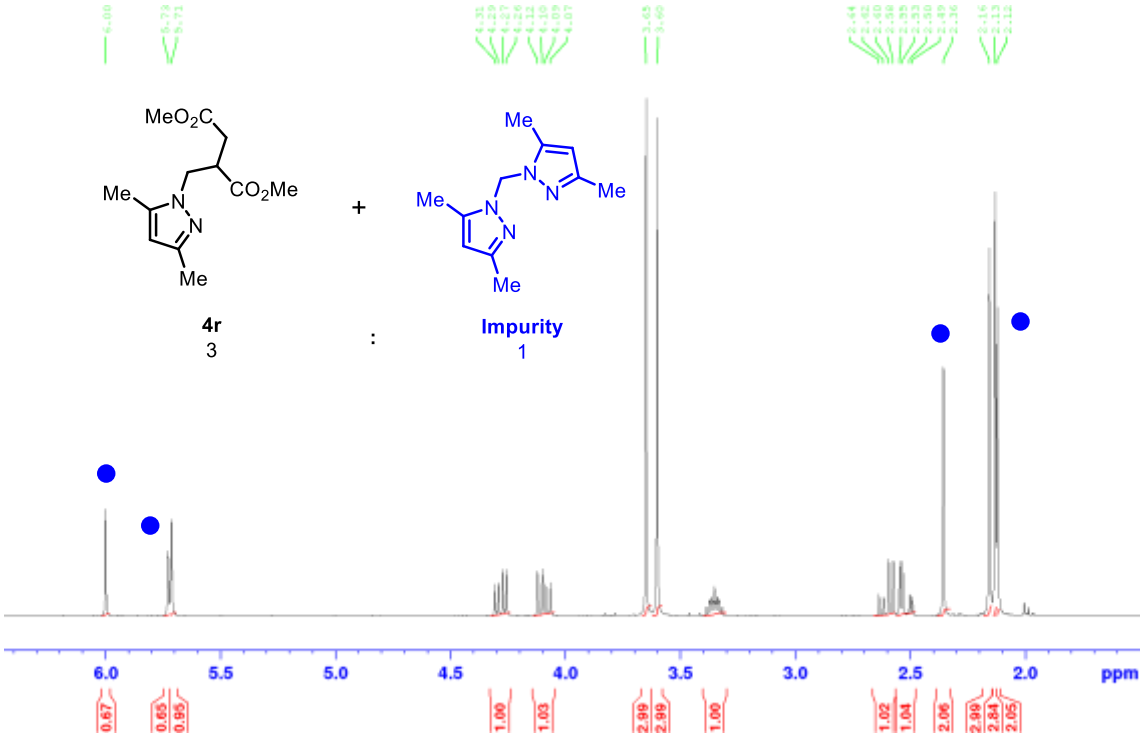
Dimethyl 2-((3,5-dimethyl-1*H*-pyrazol-1-yl)methyl)succinate (4r):
Synthesized by a two-step procedure. Through a variation of a reported procedure[26], in a round bottom flask, (3,5-dimethyl-1*H*-pyrazol-1-yl)methanol (126 mg, 1 mmol, 1 equiv.) was dissolved in dry chloroform (5 mL) and cooled at 0 ºC. Thionyl chloride (88 μL, 1.2 mmol, 1.2 equiv.) was added dropwise and the reaction was left stirring at ambient temperature for 30 min. Solvent was removed under vacuum at 25 ºC, diethyl ether (5 mL) was added and dried (this was repeated twice) to obtain the crude 1-(chloromethyl)-3,5-dimethyl-1*H*-pyrazole hydrochloride **2r** as a white solid that was used without further purification. The crude product **2r** was dissolved in acetonitrile (HPLC grade) giving a stock solution 0.75 M. 1-(chloromethyl)-3,5-dimethyl-1*H*-pyrazole hydrochloride **2r** (0.75 M, 0.75 mmol, 1.5 equiv.) was added **in** an oven dried Schlenk tube and diluted with acetonitrile (1 mL, HPLC grade), then catalyst **1c** (15.5 mg, 0.1 equiv.) was added followed by 2,6-lutidine (157 μL, 1.35 mmol, 2.7 equiv.), γ-terpinene (96 μL, 0.6 mmol, 1.2 equiv.), and dimethyl fumarate **3a** (72 mg, 0.5 mmol, 1 equiv.). The resulting yellow mixture was degassed via three cycles of freeze-pump-thaw. The Schlenk tube was then placed in the irradiation setup at a temperature of 60 ºC and irradiated for 24 hours. After cooling to ambient temperature, the solvent was evaporated and the residue purified by column chromatography (30% AcOEt in hexanes as eluent): 105 mg yellow oil: mixture containing product and reported impurity[14] in a proportion of 3:1.

A purity of 78% weight was determined ^{1}H NMR analysis. Corrected yield: 65%.

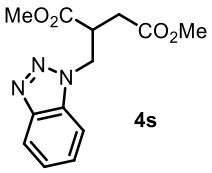
^{1}H NMR (500 MHz, $CDCl_3$) δ 5.71 (s, 1H); 4.28 (dd, *J*=6.1, 14.1 Hz, 1H); 4.09 (dd, *J*=8.2, 14.1z Hz, 1H); 3.65 (s, 3H); 3.60 (s, 3H); 3.40-3.31 (m, 1H); 2.61 (dd, *J*=7.7, 17.1 Hz, 1H); 2.52 (dd, *J*=5.3, 17.1 Hz, 1H), 2.16 (s, 3H); 2.13 (s, 3H).

^{13}C NMR (125 MHz, $CDCl_3$) δ 172.7 (C); 171.8 (C); 148 (C); 139.3 (C); 105.2 (CH); 52.2 (CH_3); 51.8 (CH_3); 48.4 (CH_2); 42.1 (CH); 33.0 (CH_2); 13.4 (CH_3); 10.8 (CH_3).

HRMS (ESI pos): calculated for $C_{12}H_{18}N_2O_4$ ($M+H^+$): 255.1339, found 255.1334.



Supplementary Figure 8: NMR spectrum of the mixture of product **4r** and impurity for ratio determination.



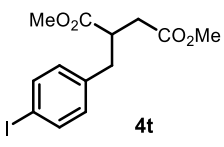
Dimethyl 2-((1*H*-benzo[*d*][1,2,3]triazol-1-yl)methyl)succinate (4s): Synthesized according to the general procedure using 1-(chloromethyl)-1*H*-benzo[*d*][1,2,3]triazole **2s** (120 mg, 0.75 mmol, 1.5 equiv.) and dimethyl fumarate **3a** (72 mg, 0.5 mmol, 1 equiv.). The resulting yellow mixture was degassed via three cycles of freeze-pump-thaw. The Schlenk tube was then placed in the irradiation setup at a temperature of 60 ºC and irradiated for 24 hours. After cooling to ambient temperature, the solvent was evaporated and the residue purified by column chromatography (20% AcOEt in toluene as eluent): 120 mg yellow oil, 87% yield.

^{1}H NMR (500 MHz, $CDCl_3$) δ 8.01 (d, *J*=8.4 Hz, 1H); 7.53 (d, *J*=8.4 Hz, 1H); 7.47 (ddd, *J*=1, 6.8, 8.4 Hz, 1H); 7.34 (ddd, *J*=1, 6.9, 8.4 Hz, 1H); 4.98 (dd, *J*=6.3, 14.4 Hz, 1H); 4.90 (dd, *J*=6.9, 14.4 Hz, 1H); 3.62 (s, 3H); 3.61 (s, 3H); 3.56-3.49 (m, 1H); 2.70 (dd, *J*=7, 17.3 Hz, 1H); 2.62 (dd, *J*=6, 17.4 Hz, 1H).

^{13}C NMR (125 MHz, $CDCl_3$) δ 172 (C); 171.5 (C); 145.9 (C); 133.3 (C); 127.7 (CH); 124.1 (CH); 120.1 (CH); 109.3 (CH); 52.6 (CH_3); 52.1 (CH_3); 48.0 (CH_2); 41.7 (CH); 33.0 (CH_2).

IR (thin film) ν 3003, 2954, 1729, 1615, 1496, 1437, 1372, 1163, 1090, 891 cm^{-1}.

HRMS (ESI pos): calculated for $C_{13}H_{15}N_3NaO_4$ ($M+Na^+$): 300.0955, found 300.0956.

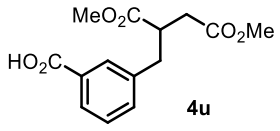


Dimethyl 2-(4-iodobenzyl)succinate (4t): Synthesized according to the general procedure using 4-iodobenzyl methanesulfonate **2t** (234 mg, 0.75 mmol, 1.5 equiv.) and dimethyl fumarate **3a** (72 mg, 0.5 mmol, 1 equiv.). Chromatography on silica gel (gradient from pure toluene to 1% AcOEt in toluene as eluent): 147 mg clear oil, 81% yield.

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.66 – 7.58 (m, 2H), 6.96 – 6.89 (m, 2H), 3.68 (s, *3*H), 3.66 (s, *3*H), 3.16 – 3.06 (m, *1*H), 2.99 (dd, *J* = 13.6, 6.7 Hz, 1H), 2.74 (dd, *J* = 13.6, 8.0 Hz, 1H), 2.68 (dd, *J* = 16.7, 8.8 Hz, 1H), 2.41 (dd, *J* = 16.7, 5.4 Hz, 1H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 174.3 (C), 172.0 (C), 137.8 (C), 137.6 (CH), 131.0 (CH), 92.0 (C), 51.9 (CH_3), 51.8 (CH_3), 42.7 (CH), 37.1 (CH_2), 34.9 (CH_2).

Matching reported data[13].



4u

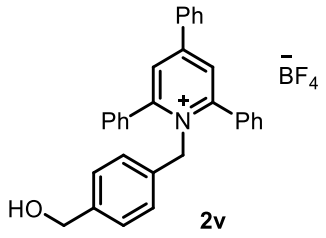
3-(4-Methoxy-2-(methoxycarbonyl)-4-oxobutyl)benzoic acid (4u): Synthesized according to a modification of the general procedure: in an oven dried Schlenk tube, 3-(chloromethyl)benzoic acid **2u** (128 mg, 0.75 mmol, 1.5 equiv.) was dissolved in acetonitrile (1 mL, HPLC grade), then 2,6-lutidine (157 μL, 1.35 mmol, 2.7 equiv.) was added followed by **1c** (15.5 mg, 0.1 equiv.), γ-terpinene (96 μL, 0.6 mmol, 1.2 equiv.) and dimethyl fumarate **3a** (72 mg, 0.5 mmol, 1 equiv.). The resulting yellow mixture was degassed via three cycles of freeze-pump-thaw. The Schlenk tube was then placed in the irradiation setup at a temperature of 60 ºC and irradiated for 24 hours. After cooling to ambient temperature, the solvent was evaporated and the residue was diluted with AcOEt and NH_4Cl sat. aqueous solution, the layers were separated and extracted with AcOEt (3x15 mL). Organic layer was purified by column chromatography (30% acetone in hexanes with 7% vol. acetic acid as eluent): 94 mg light orange solid, 67% yield.

^{1}H NMR (500 MHz, $CDCl_3$) δ 11.61 (br s, 1H); 7.99-7.95 (m, 1H); 7.90 (s, 1H); 7.41-7.37 (m, 2H); 3.66 (s, 3H); 3.64 (s, 3H); 3.20-3.12 (m, 1H); 3.08 (dd, *J*=6.8, 13.6 Hz, 1H); 2.86 (dd, *J*=7.8, 13.6 Hz, 1H); 2.69 (dd, *J*=8.9, 16.8 Hz, 1H); 2.42 (dd, *J*=5.2, 16.8 Hz, 1H).

^{13}C NMR (125 MHz, $CDCl_3$) δ 174.5 (C); 172.2 (C); 171.9 (C); 138.8 (C); 134.4 (CH); 130.7 (CH); 130.0 (C); 128.8 (CH); 128.7 (CH); 52.1 (CH_3); 51.9 (CH_3); 43 (CH); 37.5 (CH_2); 35.1 (CH_2).

IR (thin film) ν 3650, 3004, 2953, 2666, 1726, 1608, 1589, 1437, 1278, 1194 cm^{-1}.

HRMS (ESI pos): calculated for $C_{14}H_{16}NaO_6$ ($M+Na^+$): 303.0839, found 303.0846.



2v

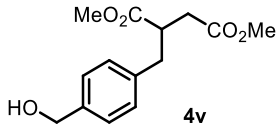
1-(4-(Hydroxymethyl)benzyl)-2,4,6-triphenylpyridin-1-ium tetrafluoroborate (2v): Substrate **2v** was synthesized according to an adaptation of a reported procedure[18]. In a Schleck tube, (4-(aminomethyl)phenyl)methanol (644 mg, 4.70 mmol, 1.2 equiv.) and 2,4,6-triphenylpyrylium tetrafluoroborate (1.55 g, 3.91 mmol, 1 equiv.) were dissolved in ethanol (3.5 mL) and heated at 85 ºC for 4 hours. The solvent was evaporated, and the product recrystallized by a mixture of DCM and diethyl ether. The precipitate was filtered and washed with diethyl ether to obtain product **2v** as a white solid: 2.016 g, 95% yield.

^{1}H NMR (500 MHz, d6-DMSO) δ 8.54 (s, 2H), 8.31 (d, *J* = 7.2 Hz, 2H), 7.78 – 7.51 (m, 13H), 7.06 (d, *J* = 7.8 Hz, 2H), 6.54 (d, *J* = 7.8 Hz, 2H), 5.64 (s, 2H), 5.15 (t, *J* = 5.6 Hz, OH, 1H), 4.39 (d, *J* = 5.6 Hz, 1H).

^{13}C NMR (125 MHz, d6-DMSO) δ 156.6 (C), 154.9 (C), 142.4 (C), 133.1 (C), 133.0 (C), 132.6 (CH), 132.2 (C), 130.9 (CH), 129.6 (CH), 129.2 (CH), 128.9 (CH), 126.4 (CH), 126.3 (CH), 125.9 (CH), 62.2 (CH_2), 57.6 (CH_2).

^{19}F NMR (470 MHz, DMSO-d_6) δ -148.35 (minor, $^{11}BF_4$), -148.41 (major, $^{10}BF_4$).

HRMS (ESI pos): calculated for $C_{31}H_{26}NO$ (M^+): 428.2009, found 428.2012.



4v

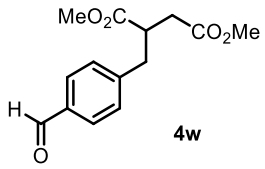
Dimethyl 2-(4-(hydroxymethyl)phenyl)succinate (4v): Synthesized according to the general procedure using 1-(4-(hydroxymethyl)benzyl)-2,4,6-triphenylpyridin-1-ium tetrafluoroborate **2v** (387 mg, 0.75 mmol, 1.5 equiv.) and dimethyl fumarate **3a** (72 mg, 0.5 mmol, 1 equiv.). The resulting yellow mixture was degassed via three cycles of freeze-pump-thaw. The Schlenk tube was then placed in the irradiation setup at a temperature of 60 ºC and irradiated for 24 hours. After cooling to ambient temperature, the solvent was evaporated and the residue purified by column chromatography (30% AcOEt in hexanes as eluent): 72 mg yellow oil, 54% yield.

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.30 (d, *J*= 8.1 Hz, 2H); 7.15 (d, *J*= 8.1 Hz, 2H); 4.66 (s, 2H); 3.68 (s, 3H); 3.65 (s, 3H); 3.17-3.10 (m, 1H); 3.06 (dd, *J*=6.4, 13.6 Hz, 1H); 2.77 (dd, *J*=8.2, 13.6 Hz, 1H); 2.68 (dd, *J*=9.2, 16.8 Hz, 1H); 2.41 (dd, *J*=5.0, 16.8 Hz, 1H); 2.19-2.02 (br s, 1H).

^{13}C NMR (125 MHz, $CDCl_3$) δ 174.3 (C); 172.4 (C); 139.5 (C); 137.6 (C); 129.3 (CH); 127.3 (CH); 65.0 (CH_2); 52.0 (CH_3); 51.9 (CH_3); 43.1 (CH); 37.4 (CH_2); 35.0 (CH_2).

IR (thin film) ν 3457, 2953, 2870, 1730, 1437, 1332, 1198, 1094, 893, 810 cm^{-1}.

HRMS (ESI pos): calculated for $C_{14}H_{18}NaO_5$ ($M+Na^+$): 289.1046, found 289.1059.



4w

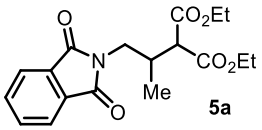
Dimethyl 2-(4-formylbenzyl)succinate (4w): Synthesized according to a modification of the general procedure: in an oven dried Schlenk tube, **1c** (15.5 mg, 0.1 equiv.) was dissolved in acetonitrile (1 mL, HPLC grade), then 4-formylbenzyl methanesulfonate **2w** (214 mg, 1 mmol, 2 equiv.) was added followed by 2,6-lutidine (116 μL, 1 mmol, 2 equiv.), γ-terpinene (160 μL, 2 mmol, 2 equiv.), and dimethyl fumarate **3a** (72 mg, 0.5 mmol, 1 equiv.). The resulting yellow mixture was degassed via three cycles of freeze-pump-thaw. The Schlenk tube was then placed in the irradiation setup at a temperature of 60 ºC and irradiated for 24 hours. After cooling to ambient temperature, the solvent was evaporated and the residue purified by column chromatography (15% AcOEt in hexanes as eluent, two consecutive purifications): 105 mg clear oil, 79% yield.

^{1}H NMR (400 MHz, $CDCl_3$) δ 9.97 (s, 1H); 7.82-7.77 (m, 2H); 7.36-7.29 (m, 2H); 3.64 (app s, 2x3H); 3.21-3.11 (m, 1H); 3.09 (dd, *J*=7, 13.5 Hz, 1H); 2.87 (dd, *J*=7.5, 13.5 Hz, 1H); 2.69 (dd, *J*=8.5, 16.7 Hz, 1H); 2.42 (dd, *J*=5.4, 16.7 Hz, 1H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 191.7 (CH); 174.1 (C); 171.9 (C); 145.5 (C); 135.1 (C); 130.0 (CH); 129.7 (CH); 52.0 (CH_3); 51.8 (CH_3); 42.6 (CH); 37.7 (CH_2); 35.0 (CH_2).

IR (thin film) ν 2953, 1731, 1697, 1607, 1437, 1364, 1212, 1168, 1006, 844 cm^{-1}.

HRMS (ESI pos): calculated for $C_{14}H_{16}NaO_5$ ($M+Na^+$): 287.0890, found 287.0895.

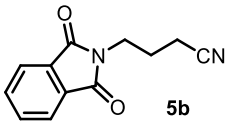


Diethyl 2-(1-(1,3-dioxoisoindolin-2-yl)propan-2-yl)malonate (5a): Synthesized according to the general procedure using 2-(chloromethyl)isoindoline-1,3-dione **2o** (147 mg, 0.75 mmol, 1.5 equiv.), 2,6-lutidine (70 µL, 0.6 mmol, 1.2 equiv.), γ-terpinene (96 µL, 0.6 mmol, 1.2 equiv.) and diethyl 2-ethylidenemalonate (91 µL, 0.5 mmol, 1 equiv.). Chromatography on silica gel (gradient from 5% to 25% AcOEt in hexanes as eluent): 69 mg, yellow oil, 40% yield.

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.85-7.81 (m, 2H), 7.72-7.69 (m, 2H), 4.23-4.14 (m, 4H), 3.78 (d, *J* = 6.5 Hz, 2H), 3.34 (d, *J* = 7.5 Hz, 1H), 2.79-2.71 (m, 1H), 1.26 (q, *J* = 7.1 Hz, 6H), 1.03 (d, *J* = 7.0 Hz, 3H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 168.8, 168.6, 168.5, 134.4, 132.3, 123.6, 61.8, 61.8, 55.6, 42.0, 33.3, 15.6, 14.4, 14.4.

HRMS (ESI pos): calculated for $C_{18}H_{21}NNaO_6$ ($M+Na^+$): 370.1261, found 370.1256.



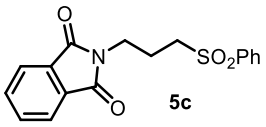
4-(1,3-Dioxoisoindolin-2-yl)butanenitrile (5b): Synthesized according to a modification of the general procedure using 2-(chloromethyl)isoindoline-1,3-dione **2o** (147 mg, 1,5 equiv.), 2,6-lutidine (140 µL, 2 equiv.), γ-terpinene (192 µL, 2 equiv.) and dimethyl fumarate (72 mg, 1 equiv.). Chromatography on silica gel (15% AcOEt in hexanes, then a second chromatography with 15% AcOEt in toluene as eluent): 70 mg, slightly yellow solid, 65% yield.

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.87-7.81 (m, 2H); 7.75-7.69 (m, 2H); 3.80 (t, J= 6.7 Hz, 2H); 2.42 (t, J= 7.3 Hz, 2H); 2.06 (m, J= 7.1 Hz, 2H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 168.3 (C); 134.3 (CH); 131.9 (C); 123.5 (CH); 118.8 (C); 36.7 (CH_2); 24.8 (CH_2); 15.2 (CH_2).

IR (thin film) ν 2942, 2247, 1773, 1706, 1467, 1397, 1122, 1027, 866, 719 cm^{-1}.

HRMS (ESI pos): calculated for $C_{12}H_{10}N_2NaO_2$ ($M+Na^+$): 327.0634, found 327.0629.



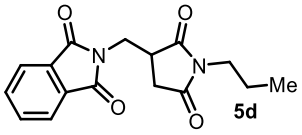
2-(3-(phenylsulfonyl)propyl)isoindoline-1,3-dione (5c): Synthesized according to a modification of the general procedure using 2-(chloromethyl)isoindoline-1,3-dione **2o** (147 mg, 1,5 equiv.), 2,6-lutidine (140 µL, 2 equiv.), γ-terpinene (192 µL, 2 equiv.) and dimethyl fumarate (72 mg, 1 equiv.). Chromatography on silica gel (10% AcOEt in toluene, then a second chromatography with 40% AcOEt in hexane as eluent): 70 mg, slightly yellow solid, 68% yield.

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.90-7.85 (m, 2H); 7.83-7.76 (m, 2H); 7.73-7.67 (m, 2H); 7.65-7.59 (m, 1H); 7.56-7.50 (m, 2H); 3.74 (t, J= 6.7 Hz, 2H); 3.20-3.12 (m, 2H); 2.13-2.03 (m, 2H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 168.2 (C); 138.8 (C); 134.3 (CH); 133.9 (CH); 131.9 (C); 129.4 (CH); 128.2 (CH); 123.5 (CH); 54.0 (CH_2); 36.3 (CH_2); 22.4 (CH_2).

IR (thin film) ν 2923, 2853, 1771, 1705, 1446, 1397, 1306, 1145, 1015, 720 cm^{-1}.

HRMS (ESI pos): calculated for $C_{17}H_{15}NNaO_4S$ ($M+Na^+$): 352.0614, found 327.0613.

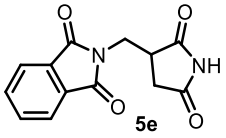


2-((2,5-Dioxo-1-propylpyrrolidin-3-yl)methyl)isoindoline-1,3-dione (5d): Synthesized according to the general procedure using 2-(chloromethyl)isoindoline-1,3-dione **2o** (147 mg, 0.75 mmol, 1.5 equiv.), 2,6-lutidine (70 µL, 0.6 mmol, 1.2 equiv.), γ-terpinene (96 µL, 0.6 mmol, 1.2 equiv.), and *N*-propylmaleimide (63 µL, 0.5 mmol, 1 equiv.). Chromatography on silica gel (gradient from 5% AcOEt in hexanes to pure AcOEt as eluent): 130 mg, yellow oil, 86% yield.

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.84-7.80 (m, 2H), 7.73-7.69 (m, 2H), 4.08 (dd, *J* = 14.0, 7.3 Hz, 1H), 3.86 (dd, *J* = 14.0, 8.3 Hz, 1H), 3.40 (t, *J* = 7.5 Hz, 2H), 3.35-3.29 (m, 1H), 2.78 (dd, *J* = 18.3, 9.2 Hz, 1H), 2.55 (dd, *J* = 18.3, 4.7 Hz, 1H), 1.57-1.50 (m, 2H), 0.84 (t, *J* = 7.5 Hz, 3H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 177.3, 175.9, 168.4, 134.6, 132.1, 123.9, 40.9, 38.9, 38.8, 33.1, 21.3, 11.6.

HRMS: calculated for $C_{16}H_{16}N_2NaO_4$ ($M+Na^+$): 323.1002, found 323.1014.



2-((2,5-Dioxopyrrolidin-3-yl)methyl)isoindoline-1,3-dione (5e): Synthesized according to the general procedure using 2-(chloromethyl)isoindoline-1,3-dione **2o** (147 mg, 0.75 mmol, 1.5 equiv.), 2,6-lutidine (70 µL, 0.6 mmol, 1.2 equiv.), γ-terpinene (96 µL, 0.6 mmol, 1.2 equiv.) and maleimide (49 mg, 0.5 mmol, 1 equiv.). Chromatography on silica gel (gradient from 5% AcOEt in hexanes to pure AcOEt as eluent): 115 mg, light yellow solid, 89% yield. Tautomer observed by ^{1}H NMR.

^{1}H NMR (500 MHz, $CDCl_3$) δ 9.06 (bs, 1H), 7.84-7.82 (m, 2H), 7.72-7.70 (m, 2H), 4.12 (dd, *J* = 14.0, 7.0 Hz, 1H), 3.90 (dd, *J* = 14.0, 8.4 Hz, 1H), 3.40-3.34 (m, 1H), 2.83 (dd, *J* = 18.4, 9.2 Hz, 1H), 2.63 (dd, *J* = 18.4, 5.0 Hz, 1H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 178.1, 176.8, 168.6, 134.7, 132.1, 124.0, 40.4, 38.7, 34.3.

IR (thin film) ν 3235, 3084, 2925, 1773, 1707, 1399, 1337, 1182, 720 cm^{-1}.

HRMS (ESI pos): calculated for $C_{13}H_{10}N_2NaO_4$ ($M+Na^+$): 281.0553, found 281.0531.

D.2. 50 mmol scale addition of benzyl chloride to dimethyl fumarate

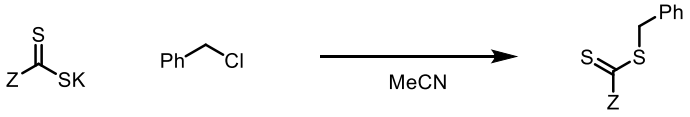
In a 150 mL capacity reaction tube, potassium 5-bromo-1*H*-indole-1-carbodithioate **1c** (1.55 g, 5 mmol, 0.1 equiv.) was dissolved in degassed acetonitrile (20 mL, degassed *via* nitrogen sparging for 30 minutes prior to the reaction), then benzyl chloride **2a** (8.6 mL, 75 mmol, 1.5 equiv.) was added and the mixture stirred for five minutes. 2,6-Lutidine (7 mL, 60 mmol, 1.2 equiv.), γ-terpinene (9.6 mL, 60 mmol, 1.2 equiv.) and dimethyl fumarate **3a** (7.21 g, 50 mmol, 1 equiv.). Acetonitrile (62 mL, degassed *via* nitrogen sparging for 30 minutes prior to the reaction) and distilled water (18 mL) were added. The reaction tube was then immersed in a 1L beaker containing silicon oil heated to 60 ºC and wrapped with a one meter 14W blue LED strip and further wrapped with aluminium foil.

The reaction mixture was further degassed via nitrogen sparging for 5 minutes at this temperature then irradiated for 48 hours. After cooling down to ambient temperature, the mixture was transferred to a round bottom flask and acetonitrile evaporated. The yellow oil was transferred to an extraction funnel, diluted with ethyl acetate (100 mL) and washed with 1M HCl (30 mL), saturated $NaHCO_3$ (30 mL), and brine. The organic phase was dried ($MgSO_4$) and concentrated to dryness. Trichloroethylene (0.9 mL, 0.2 equiv.) was added and an aliquot of the resulting mixture analysed by ^{1}H NMR to determine the yield of the reaction (79% NMR yield, 90% conversion based on dimethyl fumarate **3a**). The product was then distilled under reduced pressure (2 mbar pressure, fraction collected between 129 ºC and 135ºC) to afford 6.924 g of clean product **4a** as a slightly yellow iridescent liquid (29.33 mmol). The distillation apparatus was dismantled and rinsed back into the boiling flask. The orange residue was then purified by chromatography on silica gel to afford an additional 1.706 g of product **4a** as a yellow oil of slightly lower purity (7.22 mmol). The combined isolated yield of the reaction is therefore 8.63 g (36.56 mmol, 73%) of product of >95% purity as determined by ^{1}H NMR analysis.

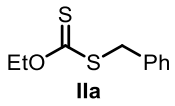
E. Mechanistic Aspects

E.1. Characterization of Dithiocarbamate Intermediates II

General procedure for the synthesis of benzyl dithiocarbamate intermediates



In a round bottom flask, the dithiocarbamate anion salt **1a-c** (1 equiv.) was dissolved in acetonitrile (HPLC grade, 0.1 M). Then benzyl chloride **2a** (1 equiv.) was added and the resulting mixture stirred for one hour at ambient temperature. Solvent was evaporated, then the residue dissolved in ethyl acetate and transferred to an extraction funnel containing water and ethyl acetate, the phases separated and the organic phase washed with distilled water and brine. The combined organic fractions were dried ($MgSO_4$) and concentrated to dryness to obtain the desired product in >95% purity.



IIa

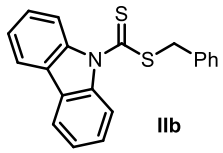
_S_-Benzyl _O_-ethyl carbonodithioate (IIa)

Prepared using potassium ethyl xanthogenate **1a** (2 mmol, 321 mg) and benzyl chloride **2a** (2 mmol, 230 μL) in 20mL acetonitrile. After work-up, product **IIa** was obtained as a slightly yellow oil (416 mg, 98% yield)

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.40-7.27 (m, 5H); 4.69 (q, *J*=7 Hz, 2 H); 4.40 (s, 2H); 1.45 (t, *J*= 7Hz, 3 H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 214.0 (C); 135.7 (C); 129.1 (CH); 128.6 (CH); 127.5 (CH); 70.0 (CH_2); 40.4 (CH_2); 13.8 (CH_3).

Matching reported data[19].



IIb

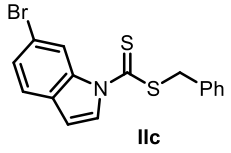
Benzyl 9*H*-carbazole-9-carbodithioate (IIb)

Prepared using potassium 9*H*-carbazole-9-carbodithioate **1b** (2 mmol, 563 mg) and benzyl chloride **2a** (2 mmol, 230 μL) in 20mL acetonitrile. After work-up, product **IIb** was obtained as a yellow solid (572 mg, 86% yield)

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.50 (td, *J*= 8.4, 0.7 Hz, 2H); 8.03 (app d, *J*=7.6 Hz, 2H); .7.55-7.33 (m, 9 H); 4.77 (s, 2H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 201.7 (C); 139.8 (C); 134.5 (C); 129.5 (CH); 128.8 (CH); 127.9 (CH); 126.7 (CH); 125.8 (C); 123.4 (CH); 119.8 (CH); 115.2 (CH); 42.6 (CH_2).

Matching reported data[20].



IIc

Benzyl 5-bromo-1*H*-indole-1-carbodithioate (IIc)

Prepared according to the general procedure using potassium 5-bromo-1*H*-indole-1-carbodithioate **1c** (2 mmol, 621 mg) and benzyl chloride **2a** (2 mmol, 230 μL) in 20mL acetonitrile. After work-up, product **IIc** was obtained as a yellow solid (695 mg, 96% yield).

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.88 (d, *J*= 9 Hz, 1H); 8.10 (d, *J*= 3.8 Hz, 1H); 7.70 (d, *J*= 2 Hz, 1H); 7.48-7.42 (m, 3H); 7.41-7.31 (m, 3H); 6.62 (dd, *J*= 3.8, 0.5 Hz, 1H); 4.65 (s, 2H).

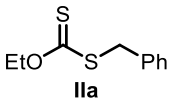
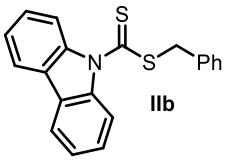
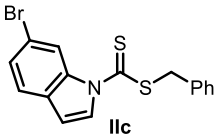
^{13}C NMR (101 MHz, $CDCl_3$) δ 197.8 (C); 135.7 (C); 134.2 (C); 133.7 (C); 129.5 (CH); 128.8 (CH); 128.1 (CH); 128.0 (CH); 127.9 (CH); 123.9 (CH); 118.2 (CH); 117.3 (C); 108.7 (CH); 41.9 (CH_2).

IR (thin film) ν 1439, 1366, 1301, 1272, 1224, 1185, 1100, 1038, 808, 709 cm^{-1}.

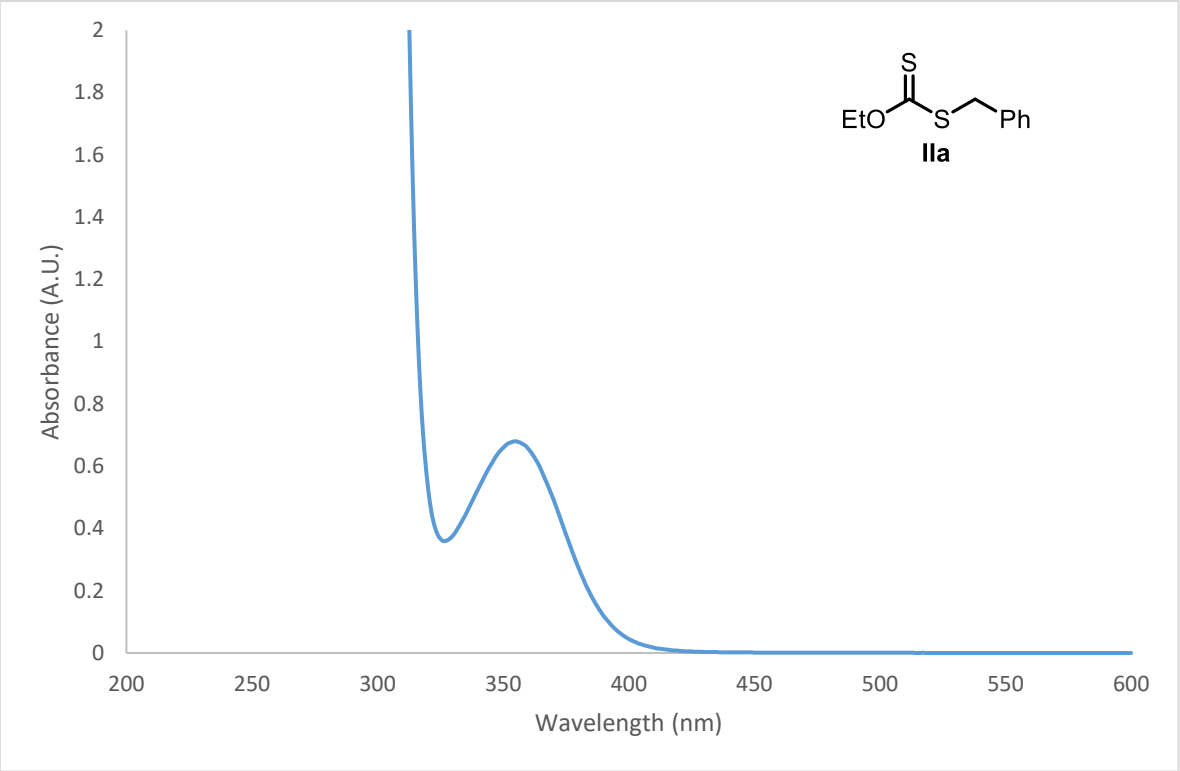
HRMS (APCI pos): calculated for $C_{16}H_{13}{}^{79}BrNS_2$ ($M+H^+$): 361.9667, found 361.9665.

E.2. UV-Vis Characterization of Dithiocarbamate Intermediates.

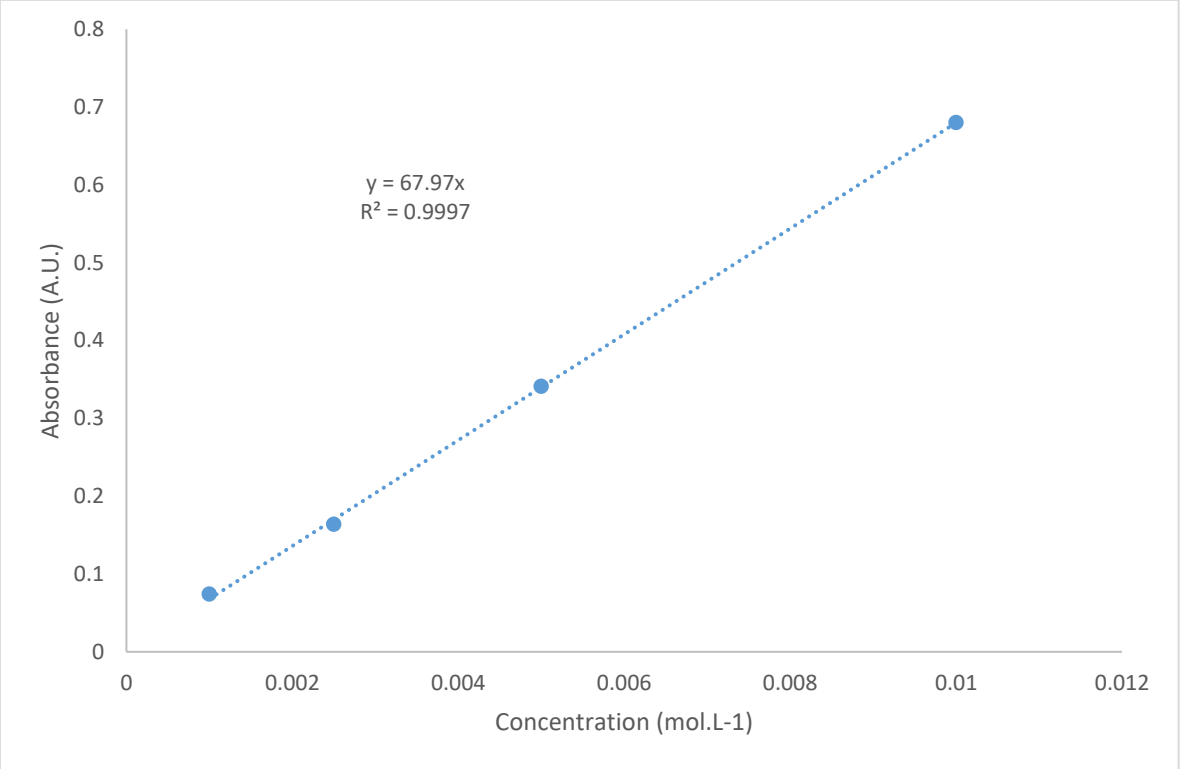
Supplementary Table 2: Spectroscopic characteristics of **IIa-c**

Dithiocarbamate intermediate	λ_{max} (extinction coefficient)	tail of absorption at 0.05M
 **IIa**	355 nm (68)	415 nm
 **IIb**	365 nm (8.938)	537 nm
 **IIc**	344 nm (18.408)	655 nm

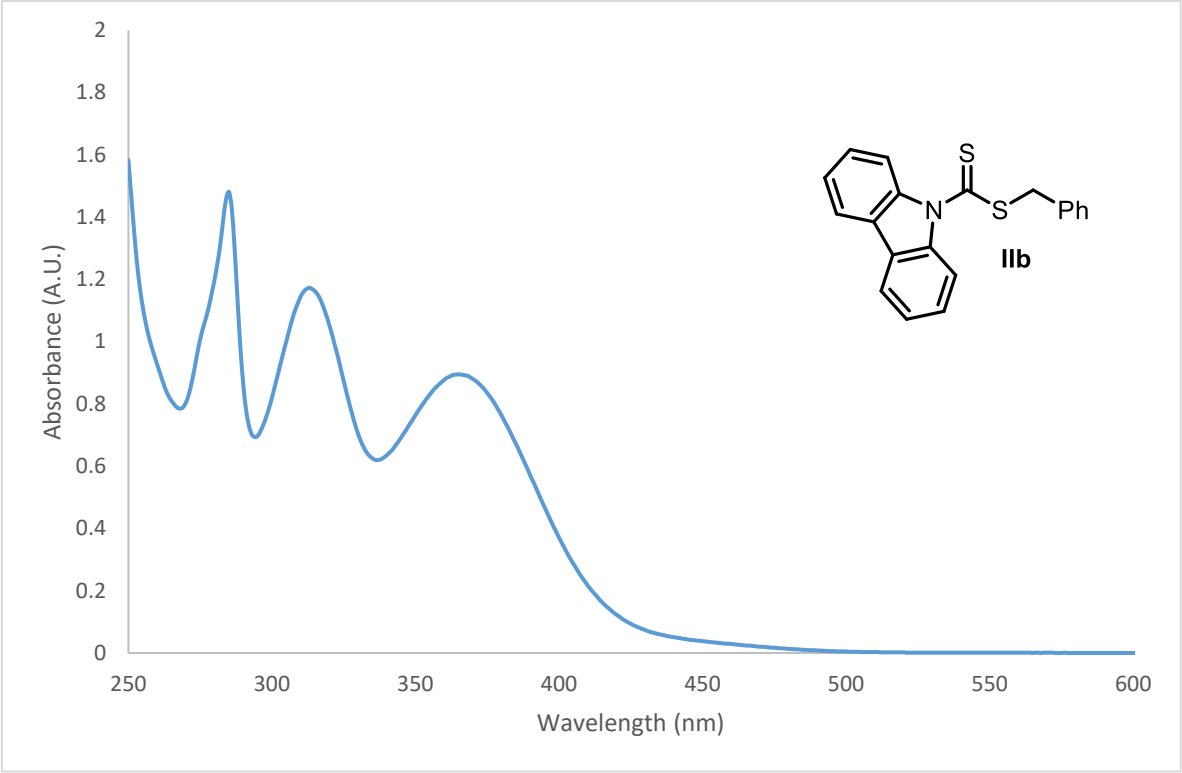
After isolation, the UV-Vis spectra of the intermediates **II** were recorded to determine the wavelength of maximal absorption in acetonitrile. Then, the molar absorption coefficient at this wavelength was determined according to Beer-Lambert's law at different concentrations to confirm linearity.



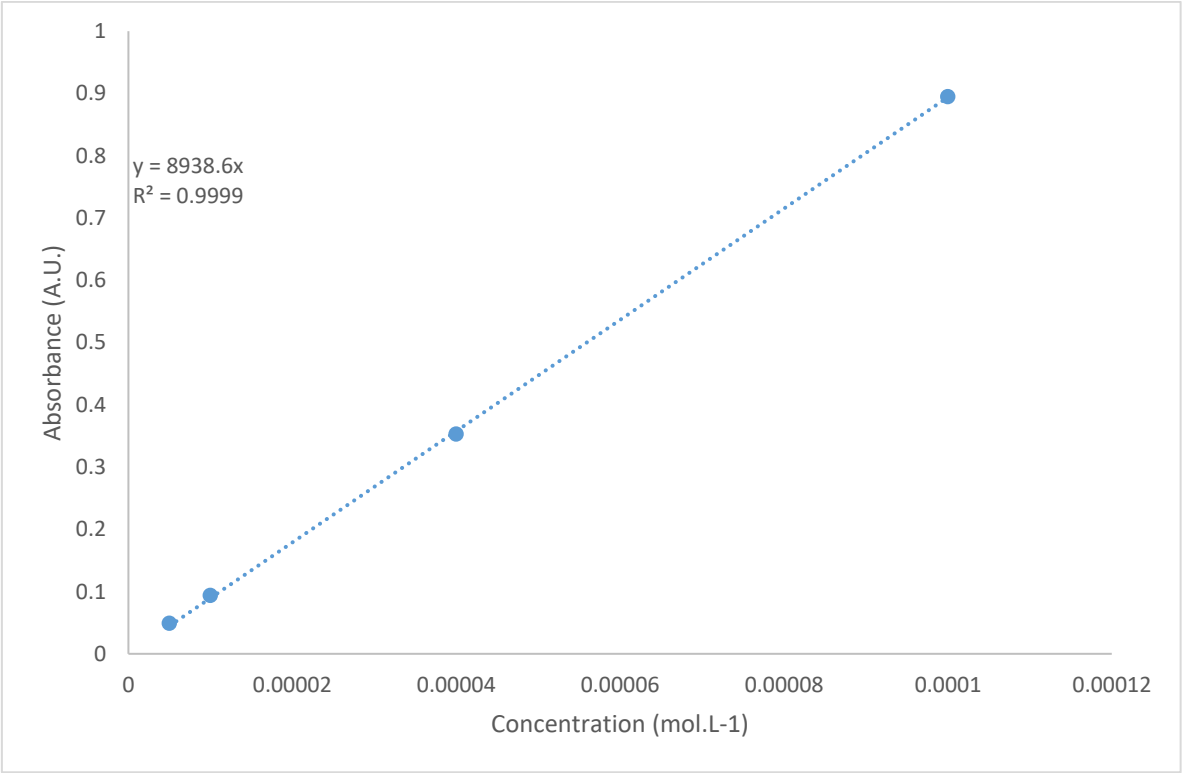
Supplementary Figure 9: UV-Vis absorption spectrum of **IIa** recorded at 10^{-2}M concentration in acetonitrile



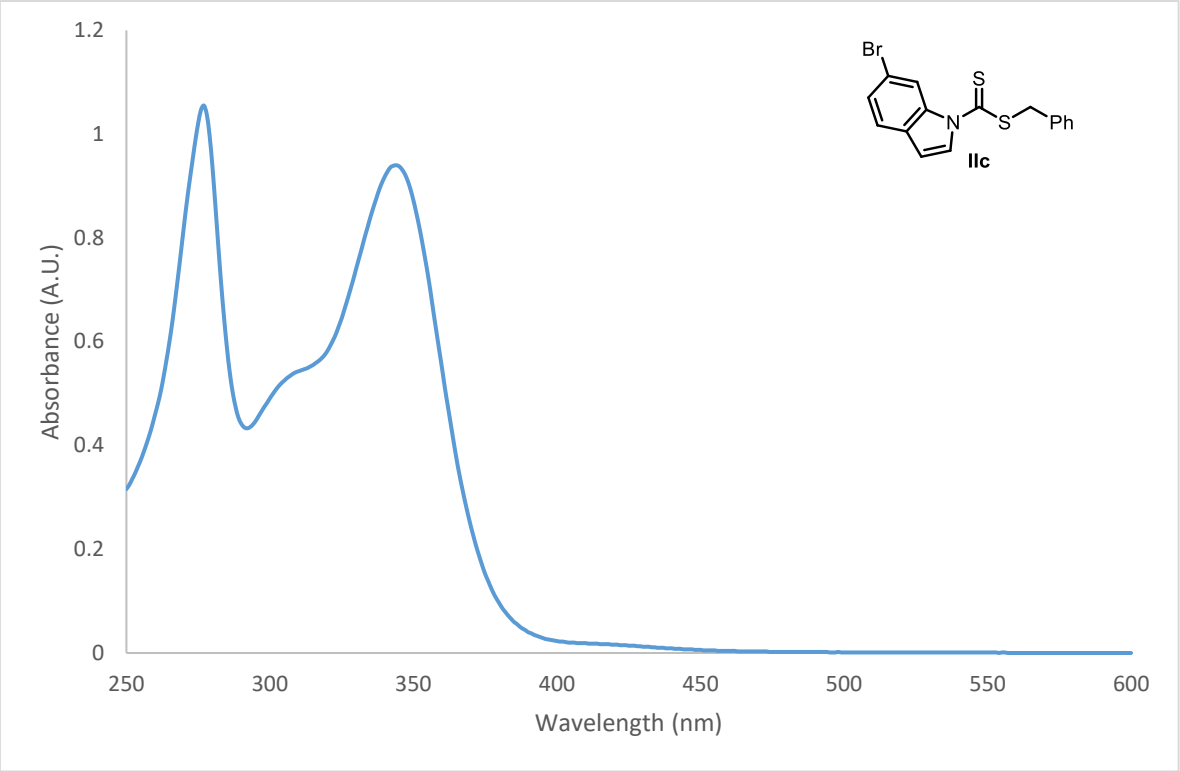
Supplementary Figure 10: Molar absorption coefficient determination of **IIa** at 355 nm.



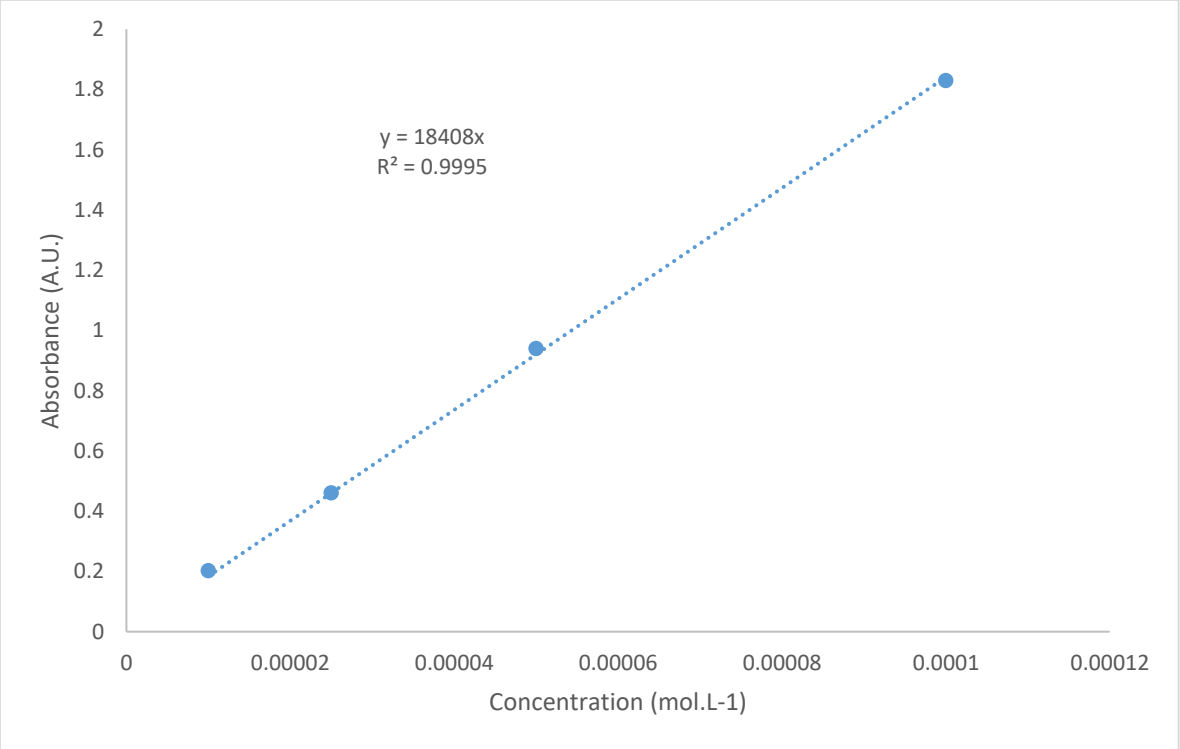
Supplementary Figure 11: UV-Vis absorption spectrum of **IIb** recorded at 10^{-4}M concentration in acetonitrile.



Supplementary Figure 12: Molar absorption coefficient determination of **IIb** at 365 nm.

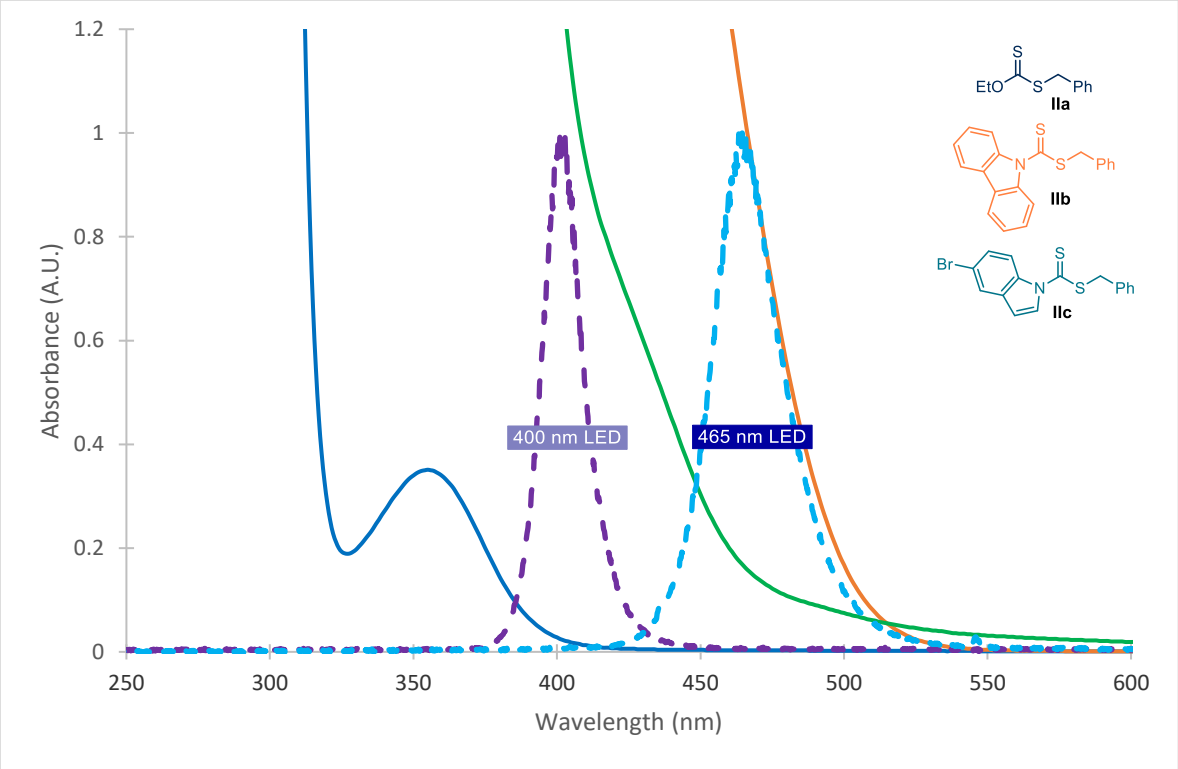


Supplementary Figure 13: UV-Vis absorption spectrum of **IIc** recorded at 5.10^{-5} M concentration in acetonitrile.



Supplementary Figure 14: Molar absorption coefficient determination of **IIc** at 345 nm.

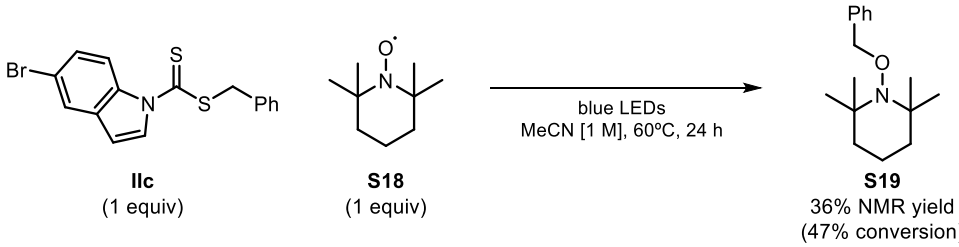
The UV-Vis spectra of the different intermediates were then recorded in a 1mm optical path cuvette at a concentration of 0.05 M, identical to the concentration of the intermediates under the reaction conditions at the beginning of irradiation (10 mol% of catalyst). The maximum wavelength of absorption at this concentration was then determined considering an absorbance of less than 0.01 arbitrary unit.



Supplementary Figure 15: Maximum wavelength of absorption of the different benzyl dithiocarbamate intermediates **II**. Spectra recorded at 0.05 M concentration in acetonitrile in a 1mm optical path cuvette. Dashed lines represent the normalized emission spectra of the LED light sources used in this study.

E.3. TEMPO trapping experiment of the benzyl radical.

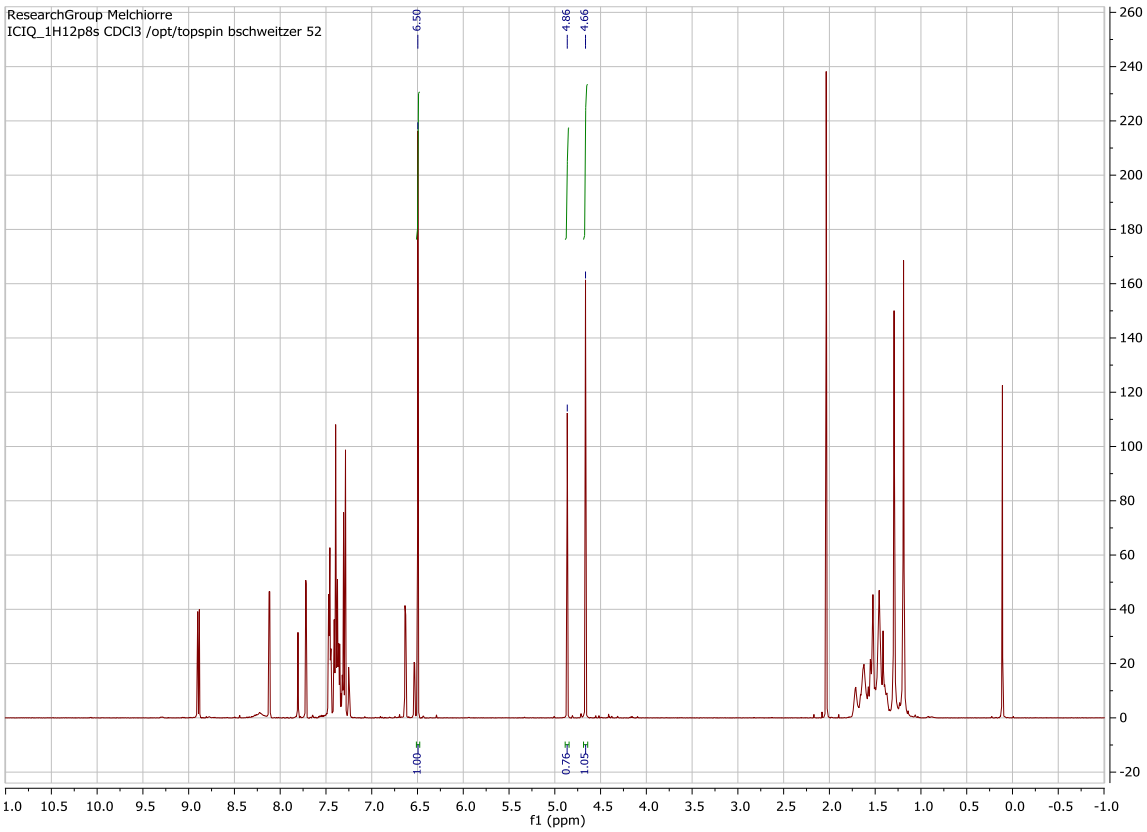
Stoichiometric reaction



IIc (1 equiv) | **S18** (1 equiv) | **S19** 36% NMR yield (47% conversion)

In an oven-dried Schlenk tube, benzyl 5-bromo-1*H*-indole-1-carbodithioate **IIc** (72 mg, 0.2 mmol, 1 equiv.) and 2,2,6,6-tetramethylpiperidine 1-oxyl (TEMPO) **18** (31 mg, 0.2 mmol, 1 equiv.) were dissolved in acetonitrile (2 mL). The resulting mixture was degassed via three cycles of freeze-pump-thaw. The Schlenk tube was then placed in the irradiation setup at a temperature of 60 ºC and irradiated for 24 hours.

After cooling to ambient temperature, the solvent was evaporated, trichloroethylene (18 µl, 1 equiv.) added as internal standard, the residue diluted with $CDCl_3$ and an aliquot analyzed by ^{1}H NMR spectroscopy to determine conversion and yield of the trapping product S**19** (Supplementary Supplementary Figure 16).



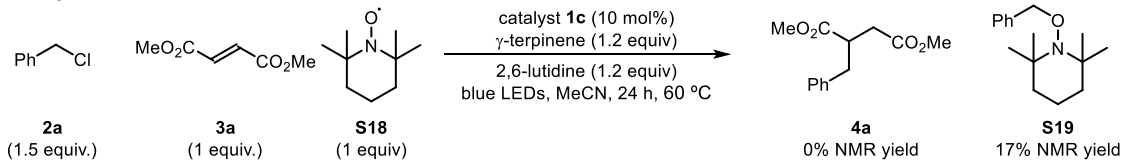
Supplementary Figure 16: ^{1}H NMR of the crude mixture for the trapping of benzyl radical with TEMPO.

Using the peak of trichloroethylene (6.50 ppm, s, 1H) as reference, a conversion of 47% (53% remaining benzyl 5-bromo-1*H*-indole-1-carbodithioate **IIc**) was determined (peak

at 4.66 ppm, s, 2H). The trapping product S**19** was detected at 4.86 ppm (s, 2H) and a 38% yield determined by integration of this peak[21].

A control experiment in the absence of light showed no conversion into the trapping product.

Catalytic reaction:



2a (1.5 equiv.) | **3a** (1 equiv.) | **S18** (1 equiv) | **4a** 0% NMR yield | **S19** 17% NMR yield

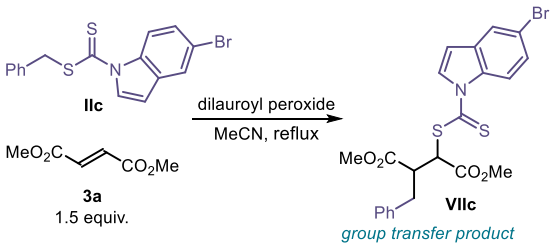
In an oven dried Schlenk tube, **1c** (15.5 mg, 0.05 mmol, 0,1 equiv.) was dissolved in acetonitrile (1 mL), then benzyl chloride **2a** (60 µL, 0.5 mmol, 1 equiv.) was added followed by 2,6-lutidine (70 µL, 0.6 mmol, 1.2 equiv.), γ-terpinene (96 µL, 0.6 mmol, 1.2 equiv.), dimethyl fumarate **3a** (72 mg, 0.5 mmol, 1 equiv.) and TEMPO S**18** (78 mg, 0.5 mmol, 1 equiv.). The resulting yellow mixture was degassed via three cycles of freeze-pump-thaw. The Schlenk tube was then placed in the irradiation setup at a temperature of 60 °C and irradiated for 24 hours. After cooling to ambient temperature, the solvent was evaporated, trichloroethylene (45 µl, 1 equiv.) added as internal standard, the residue diluted with $CDCl_3$ and an aliquot analyzed by ^1H NMR spectroscopy to determine conversion and yield.

Product **4a** was not detected by NMR while the trapping product S**19** was formed in 17% yield according to the internal standard.

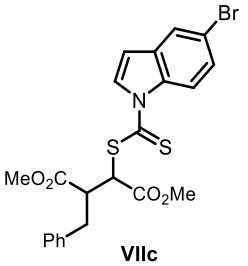
A control experiment in the absence of light showed no conversion into the trapping product.

E.4. Possible involvement of group transfer product VII in the catalytic reaction

*Independent synthesis of the group transfer product **VIIc***



In an oven-dried two necked flask equipped with a reflux condenser and under argon, compound **IIc** (362 mg, 1 mmol, 1 equiv.) and dimethyl fumarate **3a** (216 mg, 1.5 mmol, 1.5 equiv.) were dissolved in acetonitrile (10 mL). The mixture was brought to reflux under an argon atmosphere and dilauroyl peroxide was added portionwise (20 mg, 0.05 mmol, 0.05 equiv. portions; 3 portions in total) every 30 minutes until TLC analysis showed complete conversion of the starting material. The mixture was then transferred to an extraction funnel, diluted with AcOEt and washed with water and brine, dried ($MgSO_4$) and concentrated to a yellow oil. Column chromatography (10% AcOEt in hexanes as eluent) afforded the product as a yellow oil in 36% yield (183 mg) as a mixture of diastereomers (1:1.3 ratio).



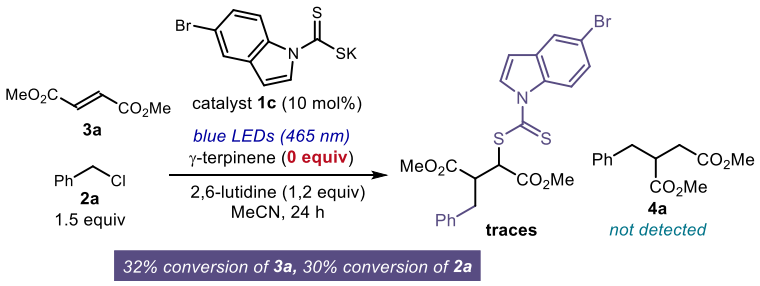
dimethyl 2-benzyl-3-((5-bromo-1H-indole-1-carbonothioyl)thio)succinate (VIIc):

^{1}H NMR for a 1:1.3 diastereomeric mixture (400 MHz, $CDCl_3$) δ 8.85 (d, J= 9.2 Hz, 1H, *major*); 8.82 (d, *J*= 9.2 Hz, 1H, *minor*); 8.17 (d, *J*= 3.8 Hz, 1H, *major*); 8.08 (d, *J*= 3.8 Hz, 1H, *minor)*; 7.72 (d, *J*= 2.0 Hz, 1H, *major*); 7.71 (d, *J*= 2.0 Hz, 1H, *minor*); 7.49-7.43 (m, 2 H, *minor* + *major*); 7.36-7.17 (m, 5H, *minor* + *major*); 6.68 (dd, *J*= 3.8, 0.5 Hz, 1H, *major*); 6.65 (dd, *J*= 3.8, 0.5 Hz, 1H, *minor*); 5.34 (d, *J*= 5.6 Hz, 1H, *minor*); 5.22 (d, *J*= 4.7 Hz, 1H, *major*); 3.83-3.77 (m, 1H, *minor* + *major*); 3.78 (s, 3H, *minor*); 3.76 (s, 3H, *major*); 3.73-3.65 (m, 1H, *major*); 3.72 (s, 3H, *major*); 3.68 (s, 3H, *minor*); 3.51-3.44 (m, 1H; *minor*); 3.31-3.22 (m, 1H, *minor* + *major*); 3.17 (dd, *J*= 13.9, 6.8 Hz, 1H, *minor*); 2.96 (dd, *J*= 14.0, 8.4 Hz, 1H, *major*).

^{13}C NMR (100 MHz, $CDCl_3$) δ 195.72; 194.65; 172.85; 172.40; 169.87; 169.48; 137.88; 137.46; 136.09; 136.05; 133.70; 129.20; 129.01; 128.74; 128.59; 128.22; 128.17; 128.05; 127.97; 126.99; 126.90; 124.05; 124.02; 118.30; 118.28; 117.69; 117.61; 109.30; 109.17; 53.83; 53.81; 53.25; 53.18; 52.41; 52.30; 49.53; 48.56; 36.11; 35.65.

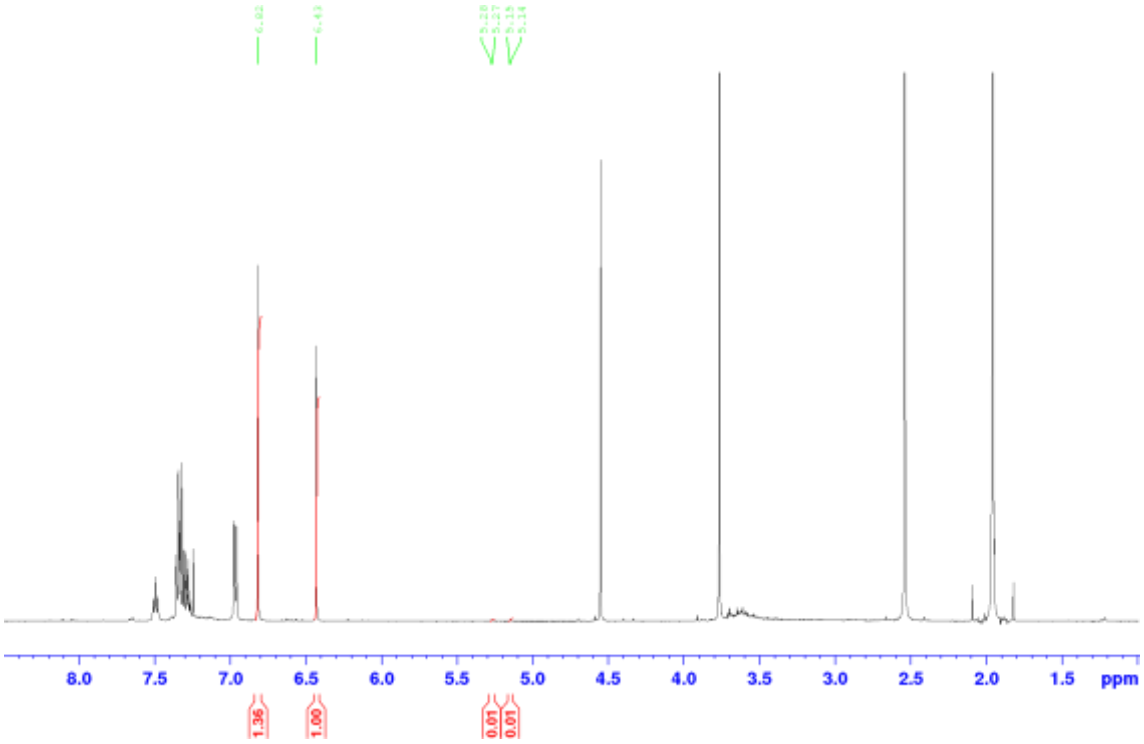
HRMS (ESI pos): calculated for $C_{22}H_{20}{}^{79}BrNNaO_4S_2$ ($M+Na^+$): 527.9909, found 527.9906.

Conjugate addition reaction in the absence of γ-terpinene:



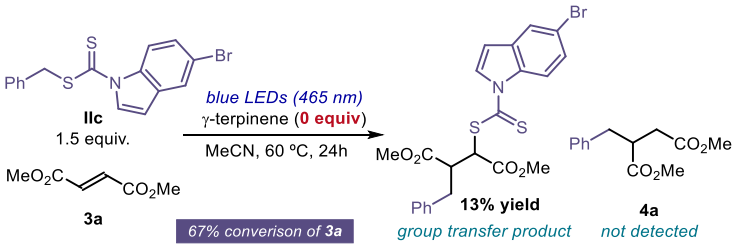
In an oven-dried Schlenk tube, **1c** (16 mg, 0.05 mmol, 0.1 equiv) was dissolved in acetonitrile (1 mL) and benzyl chloride **2a** (86µL, 0.75 mmol, 1.5 equiv.) was added, followed by 2,6-lutidine (70 µL, 0.6 mmol, 1.2 equiv.) and dimethyl fumarate **3a** (72 mg, 0.5 mmol, 1 equiv.). The mixture was then degassed (freeze-pump-thaw, three cycles) and irradiated at 60 ºC for 24 hours. The majority of volatiles were evaporated under a stream of nitrogen, trichloroethylene (18 µl, 0.2 mmol, 1 equiv.) was added, the residue dissolved in $CDCl_3$ and analyzed by ^1H NMR to determine conversion and yield.

Using the peaks at 5.3 ppm (1H) and 5.2 ppm (1H), trace amounts of **VIIc** were observed. Using the peak at 6.8 ppm (2H), a conversion of dimethyl fumarate **3a** of 32% was inferred (Supplementary Supplementary Figure 17)



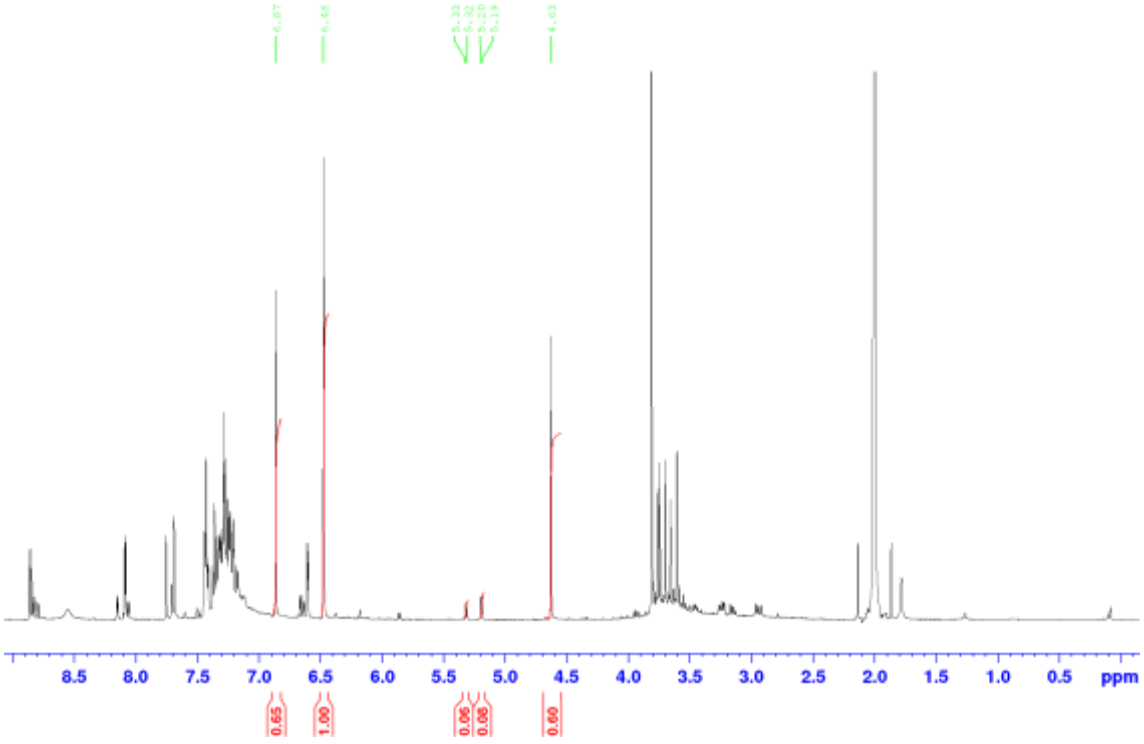
Supplementary Figure 17: ^1H NMR of the crude mixture of the Giese-type radical addition in the absence of γ-terpinene.

*Formation of **VIIc** from **IIc** under irradiation:*



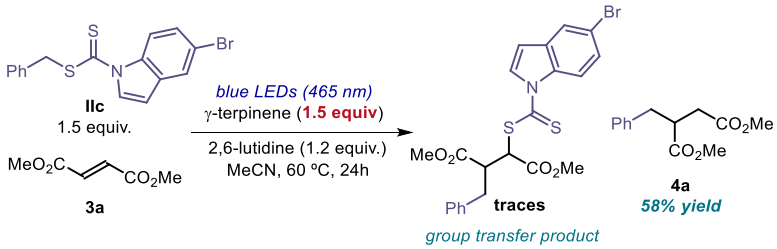
In an oven-dried Schlenk tube, **IIc** (109 mg, 0.3 mmol, 1.5 equiv) and dimethyl fumarate **3a** (29 mg, 0.2 mmol, 1 equiv.) were dissolved in acetonitrile (0.4 mL). The mixture was then degassed (freeze-pump-thaw, three cycles) and irradiated at 60 ºC for 24 hours. The majority of volatiles were evaporated under a stream of nitrogen, trichloroethylene (18 µl, 0.2 mmol, 1 equiv.) was added, the residue dissolved in $CDCl_3$ and analyzed by ^1H NMR to determine conversion and yield.

Using the peaks at 5.3 ppm (1H) and 5.2 ppm (1H), a yield of **VIIc** of 13% was determined. Using the peak at 6.8 ppm (2H), a conversion of dimethyl fumarate **3a** of 67% was inferred (Supplementary Supplementary Figure 18). The appearance of broad signals in the 3.8-3.4 ppm region may suggest the formation of polymers.



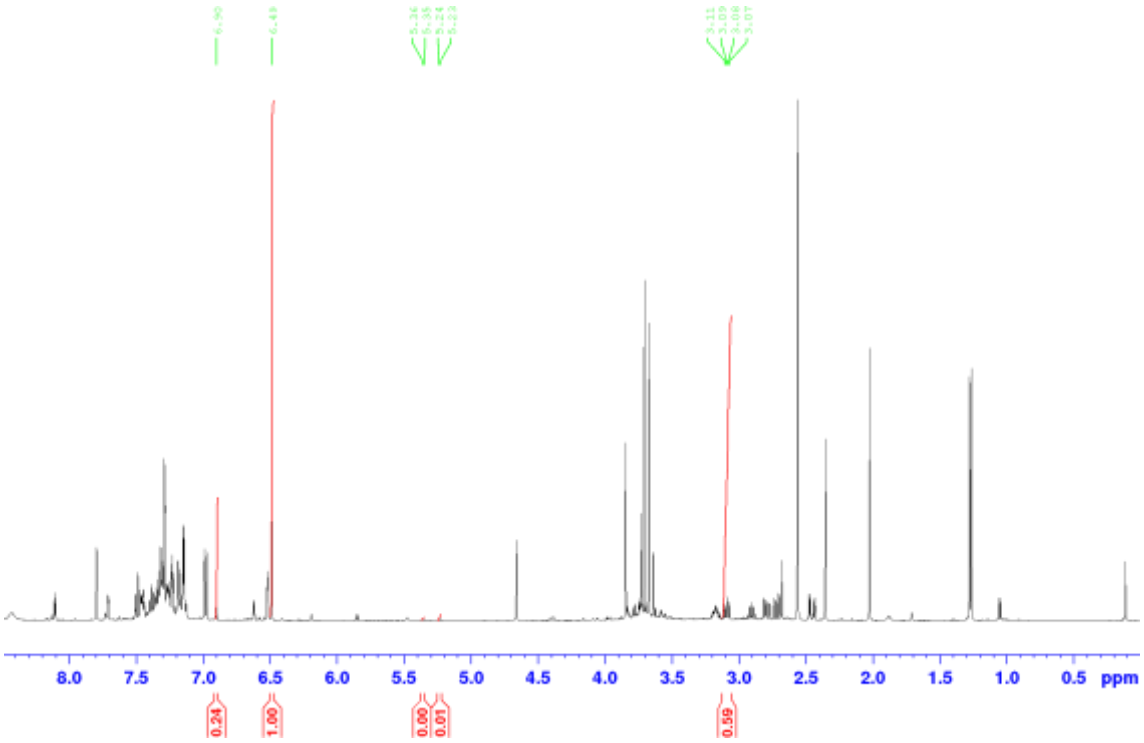
Supplementary Figure 18: ^1H NMR of the crude mixture for the formation of **VIIc** under irradiation.

*Formation of **4a** from **IIc** under stoichiometric reaction conditions:*



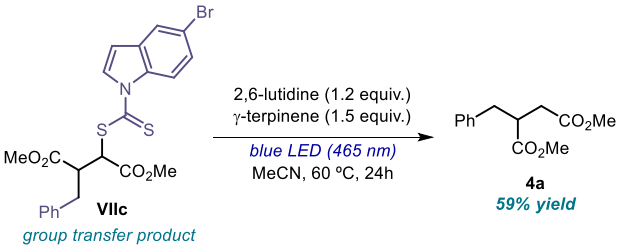
In an oven-dried Schlenk tube, **IIc** (109 mg, 0.3 mmol, 1.5 equiv.) and dimethyl fumarate **3a** (29 mg, 0.2 mmol, 1 equiv.) were dissolved in acetonitrile (0.4 mL) and γ-terpinene (948 µL, 0.3 mmol, 1.5 equiv.) and 2,6-lutidine (28 µL, 0.24 mmol, 1.5 equiv.). The mixture was then degassed (freeze-pump-thaw, three cycles) and irradiated at 60 ºC for 24 hours. The majority of volatiles were evaporated under a stream of nitrogen, trichloroethylene (18 µl, 0.2 mmol, 1 equiv.) was added, the residue dissolved in $CDCl_3$ and analysed by ^1H NMR to determine conversion and yield.

Using the peaks at 5.3ppm (1H) and 5.2 ppm (1H), traces of **VIIc** were detected. Using the peak at 3.11 ppm (m, 1H), a 58% yield of **4a** was inferred (Supplementary Supplementary Figure 19). The presence of γ–terpinene exclusively triggered the Giese-type addition manifold.



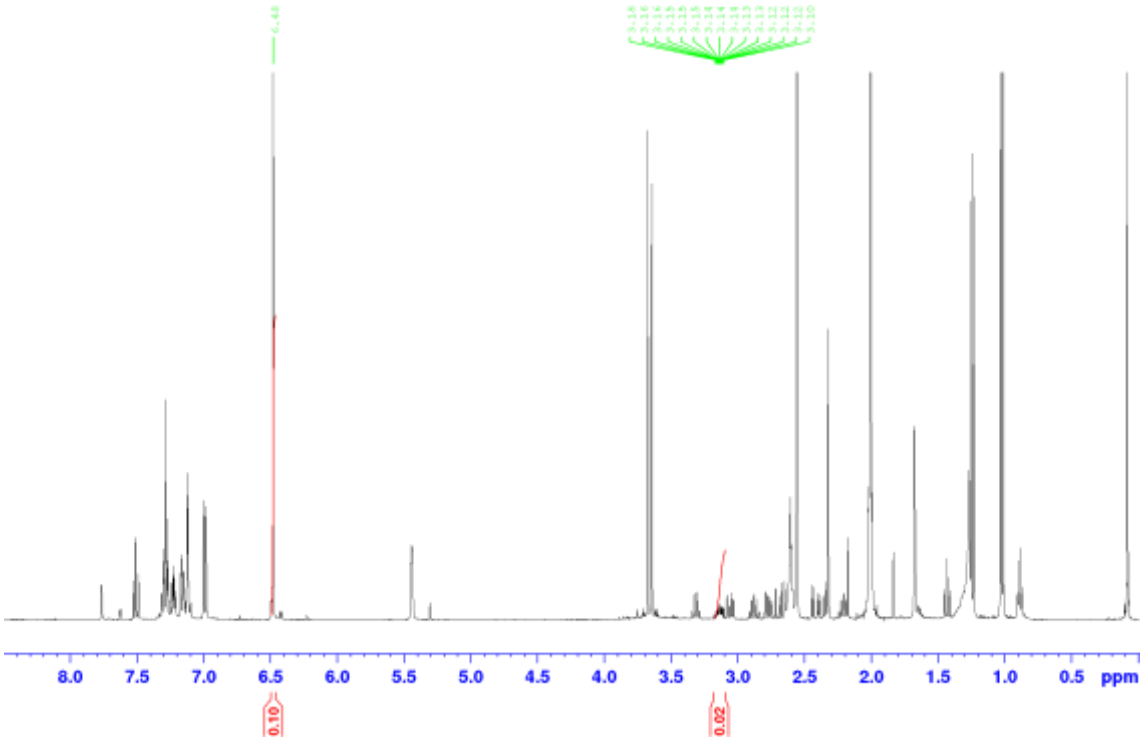
Supplementary Figure 19: ^1H NMR of the crude mixture for the formation of **4a** from **IIc** under irradiation in the presence of γ-terpinene.

*Formation of **4a** upon irradiation of **VIIc**:*



In an oven-dried Schlenk tube, **VIIc** (20 mg, 0.039 mmol, 1.5 equiv), γ-terpinene (9.5 µL, 0.06 mmol, 1.5 equiv.) and 2,6-lutidine (5.5 µL, 0.06 mmol, 1.5 equiv.) were dissolved in acetonitrile (80 µL). The mixture was then degassed (freeze-pump-thaw, three cycles) and irradiated at 60 ºC for 24 hours. The majority of volatiles were evaporated under a stream of nitrogen, trichloroethylene (9 µl, 0.1 mmol, 2.53 equiv.) was added, the residue dissolved in $CDCl_3$ and analyzed by ^1H NMR to determine conversion and yield.

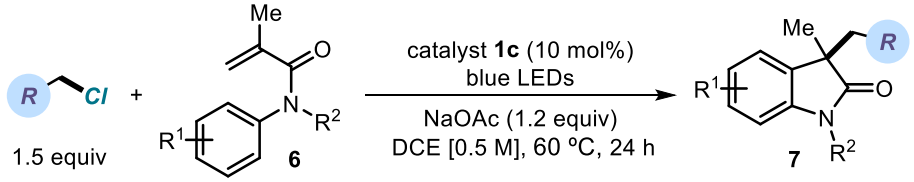
Using the peaks at 5.3ppm (1H) and 5.2 ppm (1H), full conversion of **VIIc** was determined. Using the peak at 3.14 ppm (m, 1H), a yield of 59% was inferred for the benzylation product **4a** (Supplementary Supplementary Figure 20).



Supplementary Figure 20: ^1H NMR of the crude mixture for the conversion of **VIIc** to **4a** under irradiation.

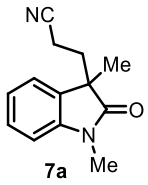
Based on these experiments, we can draw the following conclusions: *i*) In consonance with the RAFT polymerisation literature (such as reference 16 of the main manuscript), it appears that intermediate **IIc** might act as a competent chain transfer agent. The conversion of fumarate in the stoichiometric control experiment conducted in the absence of H-donor and the appearance of broad signals in the NMR analysis may indicate the formation of polymers. *ii*) The presence of γ–terpinene favours the Giese-type addition manifold under catalytic conditions and prevents any efficient polymerisation, as shown by the generally high yields of product **4** obtained in the experiments conducted with a H donor. *iii)* **VIIc** , if formed under reaction conditions is a competent precursor to radicals **IV** and **V**, that can reenter the catalytic cycle to products **4**.

F. Tandem radical addition-cyclization of aromatic acrylamides:



F.1. General Procedure:

An oven dried Schlenk tube was sequentially charged with the electrophile (0,75 mmol, 1.5 equiv.), dichloroethane (1 mL, reagent grade), and then the catalyst **1c** (15.5 mg, 0.05 mmol, 0.1 equiv.). The aromatic acrylamide **6** (0.50 mmol, 1.0 equiv.) was added, followed by sodium acetate (49.2 mg, 0.60 mmol, 1.2 equiv.). The resulting yellow mixture was degassed via three cycles of freeze-pump-thaw. The Schlenk tube was then placed in the irradiation setup (see section C.1.) at a temperature of 60 ºC (60-61 ºC measured in the central well) and the reaction mixture was stirred under irradiation for 24 hours. After cooling to ambient temperature, the solvent was evaporated and the residue purified by column chromatography to afford the corresponding product in the stated yield with >95% purity according to ^{1}H NMR analysis. The exact conditions for chromatography are reported for each compound.



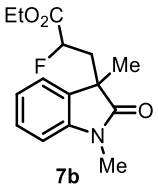
3-(1,3-Dimethyl-2-oxoindolin-3-yl)propanenitrile (7a). Synthesized according to the general procedure using chloroacetonitrile (48 μL, 0.75 mmol, 1.5 equiv.) and *N*-methyl-*N*-phenylmethacrylamide (87.6 mg, 0.5 mmol, 1 equiv.). Chromatography on silica gel (gradient from 10% to 40% AcOEt in hexanes as eluent): 102 mg brown oil, 95% yield.

Matching reported data[22]:

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.34-7.30 (m, 1H); 7.21-7.19 (m, 1H), 7.13-7.10 (m, 1H), 6.89 (d, *J* = 7.8 Hz, 1H), 3.22 (s, 3H), 2.36-2.30 (m, 1H), 2.13-1.97 (m, 3H), 1.40 (s, 3H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 178.9, 143.2, 131.7, 128.7, 123.0, 122.6, 118.8, 108.5, 47.3, 33.4, 26.3, 23.5, 12.8.

HRMS (ESI pos): calculated for $C_{13}H_{14}N_2NaO^+$ ($M+Na^+$): 237.0998, found 237.0997.



Ethyl 3-(1,3-dimethyl-2-oxoindolin-3-yl)-2-fluoropropanoate (7b) Synthesized according to the general procedure using ethyl bromofluoroacetate (89 μL, 0.75 mmol, 1.5 equiv.) and *N*-methyl-*N*-phenylmethacrylamide (87.6 mg, 0.5 mmol, 1 equiv.). The diastereomeric ratio was determined by ^{1}H NMR analysis of the crude reaction mixture by comparison of the resonances at δ 3.20 (minor diastereomer) and δ 3.17 (major diastereomer). Chromatography on silica gel (gradient from 15% to 35% AcOEt in hexanes as eluent): 135 mg brown oil, 97% yield (1.2:1 dr).

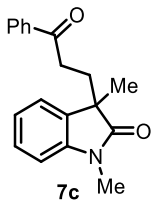
^{1}H NMR for diastereomeric mixture (500 MHz, $CDCl_3$) δ 7.29-7.23 (m, 1H), 7.20-7.17 (m, 1H), 7.06-7.01 (m, 1H), 6.84-6.82 (m, 1H), 4.76 (ddd, *J* = 48.7, 7.4, 4.4 Hz, 1H-minor), 4.61 (ddd, *J* = 49.3, 10.9, 2.6 Hz, 1H-major), 4.16-4.07 (m, 2H), 4.06-3.97 (m, 2H), 3.20 (s, 3H-minor), 3.17 (s, 3H-major), 2.56-2.38 (m, 3H), 2.28 (ddd, *J* = 39.3, 15.0, 2.6 Hz, 1H-major), 1.39 (s, 3H-major), 1.38 (s, 3H-minor), 1.21 (t, *J* = 7.1 Hz, 3H-major), 1.17 (t, *J* = 7.2 Hz-minor).

^{13}C NMR for diastereomeric mixture (126 MHz, $CDCl_3$) δ 179.6, 179.4, 169.2 (d, *J* = 18.5 Hz), 169.0 (d, *J* = 19.2 Hz), 143.5, 143.2, 132.2, 131.8, 128.5, 128.2, 123.4, 122.9, 122.5, 122.4, 108.4, 108.3, 87.4 (d, *J* = 37.7 Hz), 85.9 (d, *J* = 36.3 Hz), 61.6 (diastereomeric signals overlapping), 46.0, 45.7, 39.9 (d, *J* = 20.3 Hz), 39.3 (d, *J* = 19.8 Hz), 26.3 (diastereomeric signals overlapping), 24.8, 24.2, 14.0, 13.9.

^{19}F NMR for diastereomeric mixture (376 MHz, $CDCl_3$) δ -190.5, -191.0.

IR (thin film) ν 2972, 1710, 1613, 1494, 1471, 1375, 1348, 1298, 1031, 755 cm^{-1}.

HRMS (ESI pos): calculated for $C_{15}H_{19}FNO_3^+$ ($M+H^+$): 280.1343, found 280.1354.



7c

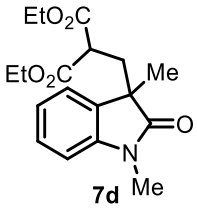
1,3-dimethyl-3-(3-oxo-3-phenylpropyl)indolin-2-one (7c) Synthesized according to the general procedure using 2-chloro-1-phenylethan-1-one (115.9 mg, 0.75 mmol, 1.5 equiv.) and *N*-methyl-*N*-phenylmethacrylamide (87.6 mg, 0.5 mmol, 1 equiv.). Chromatography on silica gel (gradient from 10% to 25% AcOEt in hexanes as eluent): 100 mg orange oil, 68% yield.

Matching reported data[23].

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.80-7.78 (m, 2H), 7.51 (t, *J* = 7.4 Hz, 1H), 7.39 (t, *J* = 7.9 Hz, 2H), 7.30-7.26 (m, 1H), 7.22 (d, *J* = 7.3 Hz, 1H), 7.10-7.07 (m, 1H), 6.87 (d, *J* = 7.8 Hz, 1H), 3.26 (s, 3H), 2.84-2.78 (m, 1H), 2.55-2.48 (m, 1H), 2.38-2.33 (m, 1H), 2.28-2.22 (m, 1H), 1.44 (s, 3H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 199.3, 180.2, 143.2, 136.7, 133.4, 133.0, 128.5, 128.1, 128.0, 122.8, 122.7, 108.1, 47.6, 33.6, 32.5, 26.2, 23.8.

HRMS (ESI pos): calculated for $C_{19}H_{20}NO_2^+$ ($M+H^+$): 294.1489, found 294.1498.



7d

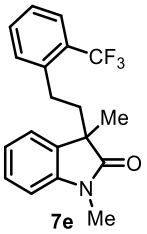
Diethyl 2-((1,3-dimethyl-2-oxoindolin-3-yl)methyl)malonate (7d) Synthesized according to the general procedure using diethyl 2-chloromalonate (121 μL, 0.75 mmol, 1.5 equiv.) and *N*-methyl-*N*-phenylmethacrylamide (87.6 mg, 0.5 mmol, 1 equiv.). Chromatography on silica gel was not able to remove residual methacrylamide from the reaction mixture, therefore an NMR yield is reported (46%). Semi-preparative HPLC (IA column, 70:30 Hexane/CH_2Cl_2, 1 mL/min) was performed to obtain an analytical amount of the isolated product as a yellow oil.

Matching reported data[24]. Previously reported data was missing the methylene peaks in the ^{1}H NMR peak data tabulation, but the peaks were present in the ^{1}H NMR copy.

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.27-7.23 (m, 1H), 7.14 (d, *J* = 6.8 Hz, 1H), 7.04-7.01 (m, 1H), 6.82 (d, *J* = 7.8 Hz, 1H), 4.11 (q, *J* = 7.2 Hz, 2H), 3.85-3.79 (m, 1H), 3.70-3.63 (m, 1H), 3.20 (s, 3H), 3.01 (dd, *J* = 8.1, 5.5 Hz, 1H), 2.55 (dd, *J* = 14.4, 5.5 Hz, 1H), 2.49 (dd, *J* = 14.3, 8.1 Hz, 1H), 1.37 (s, 3H), 1.20 (t, 7.1 Hz, 3H), 1.05 (t, 7.2 Hz, 3H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 179.7, 169.2, 169.1, 143.8, 132.5, 128.6, 123.8, 122.8, 108.5, 61.9, 61.7, 49.0, 47.4, 36.2, 26.6, 24.8, 14.3, 14.1.

HRMS (ESI pos): calculated for $C_{18}H_{24}NO_5^+$ ($M+H^+$): 334.1649, found 334.1648.

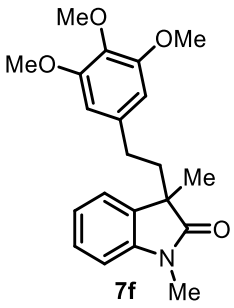


1,3-dimethyl-3-(2-(trifluoromethyl)phenethyl)indolin-2-one (7e) Synthesized according to the general procedure using 1-(chloromethyl)-2-(trifluoromethyl)benzene (109 μL, 0.75 mmol, 1.5 equiv.) and *N*-methyl-*N*-phenylmethacrylamide (87.6 mg, 0.5 mmol, 1 equiv.). Chromatography on silica gel (gradient from 10% to 30% AcOEt in hexanes as eluent; mixed fractions were re-purified in the same manner): 71 mg yellow oil, 43% yield.

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.53 (d, *J* = 7.7 Hz, 1H), 7.43-7.38 (m, 1H), 7.32-7.29 (m, 1H), 7.25-7.22 (m, 3H9, 7.14-7.11 (m, 1H), 6.88 (d, *J* = 7.8 Hz, 1H), 3.26 (s, 3H), 2.43-2.36 (m, 2H), 2.22-2.15 (m, 1H), 2.07-1.99 (m, 1H), 1.40 (s, 3H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 180.6, 143.7, 140.7, 133.7, 132.1, 131.8, 128.6 (d, *J* = 29.7 Hz), 128.3, 126.4, 126.2 (q, *J* = 5.6 Hz), 125.9, 123.7, 123.1 (d, *J* = 4.7 Hz), 108.4, 48.8, 40.8, 28.3, 26.6, 24.1. ^{19}F NMR (376 MHz, $CDCl_3$) δ -59.8.

IR (thin film) ν 2966, 1709, 1613, 1471, 1347, 1313, 1114, 905, 766, 718 cm^{-1}.
HRMS (ESI pos): calculated for $C_{19}H_{18}F_3NNaO^+$ ($M+Na^+$): 356.1233, found 356.1230.



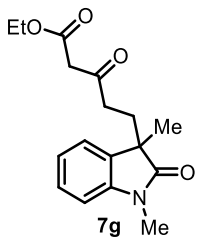
1,3-dimethyl-3-(3,4,5-trimethoxyphenethyl)indolin-2-one (7f) Synthesized according to the general procedure using 5-(chloromethyl)-1,2,3-trimethoxybenzene (162.5 mg, 0.75 mmol, 1.5 equiv.) and *N*-methyl-*N*-phenylmethacrylamide (87.6 mg, 0.5 mmol, 1 equiv.). Chromatography on silica gel (gradient from 20% to 50% AcOEt in hexanes as eluent): 49 mg brown oil, 28% yield.

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.34-7.31 (m, 1H), 7.26 (d, *J* = 7.2 Hz, 1H), 7.14 (t, *J* = 7.5 Hz, 1H), 6.88 (d, *J* = 7.7 Hz, 1H), 6.23 (s, 2H), 3.81 (s, 9H), 3.22 (s, 3H), 2.34-2.25 (m, 2H), 2.15-2.00 (m, 2H), 1.42 (s, 3H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 180.4, 153.0, 143.4, 137.0, 136.0, 133.7, 127.9, 122.6, 122.6, 108.0, 105.2, 60.8, 56.0, 48.3, 40.0, 31.5, 26.1, 24.1.

IR (thin film) ν 2935, 1707, 1612, 1589, 1493, 1455, 1331, 1238, 1009, 751 cm^{-1}.

HRMS (ESI pos): calculated for $C_{21}H_{25}NNaO_4^+$ ($M+Na^+$): 378.1676, found 378.1675.



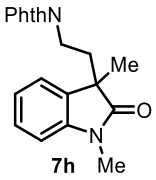
Ethyl 5-(1,3-dimethyl-2-oxoindolin-3-yl)-3-oxopentanoate (7g). Synthesized according to the general procedure using ethyl 4-chloro-3-oxobutanoate (101 μL, 0.75 mmol, 1.5 equiv.) and *N*-methyl-*N*-phenylmethacrylamide (87.6 mg, 0.5 mmol, 1 equiv.). Chromatography on silica gel (gradient from 10% to 40% AcOEt in hexanes as eluent): 103 mg yellow oil, 68% yield. Isolated product sits as ketone:enol mixture (95:5).

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.29-7.26 (m, 1H), 7.15 (d, *J* = 7.3 Hz, 1H), 7.08—7.05 (m, 1H), 6.86 (d, *J* = 7.8 Hz, 1H), 4.11 (q, *J* = 7.2 Hz, 2H), 3.25 (d, *J* = 3.5 Hz, 2H), 3.21 (s, 3H), 2.35-2.28 (m, 1H), 2.20-2.06 (m, 3H), 1.37 (s, 3H), 1.20 (t, *J* = 7.2 Hz, 3H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 201.7, 179.9, 166.9, 143.1, 133.1, 128.1, 122.8, 122.7, 108.1, 61.3, 49.2, 47.2, 38.0, 31.4, 26.2, 23.6, 14.0.

IR (thin film) ν 2970, 1704, 1612, 1493, 1471, 1375, 1349, 1254, 1031, 756 cm^{-1}.

HRMS (ESI pos): calculated for $C_{17}H_{21}NNaO_4^+$ ($M+Na^+$): 326.1363, found 326.1371.



7h

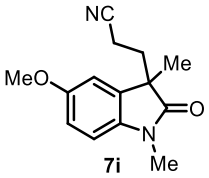
2-(2-(1,3-dimethyl-2-oxoindolin-3-yl)ethyl)isoindoline-1,3-dione (7h). Synthesized according to the general procedure using *N*-(chloromethyl)phthalimide (146.7 mg, 0.75 mmol, 1.5 equiv.) and *N*-methyl-*N*-phenylmethacrylamide (87.6 mg, 0.5 mmol, 1 equiv.). Chromatography on silica gel could not remove a residual byproduct from the reaction mixture, therefore an NMR yield is reported (65%). Semi-preparative HPLC (IA column, 70:30:0.1 hexane/CH_2Cl_2/diethylamine, 1 mL/min) was performed to obtain an analytical amount of the isolated product as a clear oil.

Matching reported data[24]:

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.66-7.60 (m, 4H), 7.09-7.07 (m, 2H), 6.96-6.93 (m, 1H), 6.74-6.69 (m, 2H), 3.62-3.56 (m, 1H), 3.47-3.42 (m, 1H), 3.19 (s, 3H), 2.48-2.42 (m, 1H), 2.33-2.28 (m, 1H), 1.35 (s, 3H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 179.9, 168.1, 143.5, 133.9, 133.3, 132.3, 127.9, 123.2, 122.7, 122.4, 108.7, 47.3, 35.0, 34.9, 26.6, 25.4.

HRMS (ESI pos): calculated for $C_{20}H_{18}N_2NaO_3^+$ ($M+Na^+$): 357.1210, found 357.1224.



7i

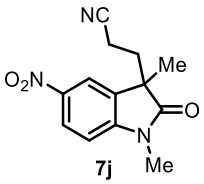
3-(5-Methoxy-1,3-dimethyl-2-oxoindolin-3-yl)propanenitrile (7i). Synthesized according to the general procedure using chloroacetonitrile (48 μL, 0.75 mmol, 1.5 equiv.) and *N*-(4-methoxyphenyl)-*N*-methylmethacrylamide (102.6 mg, 0.5 mmol, 1 equiv.). Chromatography on silica gel (gradient from 10% to 50% AcOEt in hexanes as eluent): 109 mg brown oil, 89% yield.

Matching reported data[22]:

^{1}H NMR (500 MHz, $CDCl_3$) δ 6.84-6.77 (m, 3H), 3.80 (s, 3H), 3.19 (s, 3H), 2.35-2.27 (m, 1H), 2.12-1.97 (m, 3H), 1.38 (s, 3H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 178.5, 156.4, 136.6, 133.0, 118.9, 112.6, 110.3, 108.9, 55.8, 47.7, 33.5, 26.4, 23.5, 12.8.

HRMS (ESI pos): calculated for $C_{14}H_{17}N_2O_2^+$ ($M+H^+$): 245.1285, found 245.1289.



7j

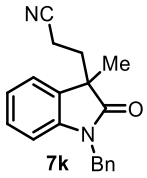
3-(5-Methoxy-1,3-dimethyl-2-oxoindolin-3-yl)propanenitrile (7j). Synthesized according to the general procedure using chloroacetonitrile (48 μL, 0.75 mmol, 1.5 equiv.) and *N*-methyl-*N*-(4-nitrophenyl)methacrylamide (110.1 mg, 0.5 mmol, 1 equiv.). Chromatography on silica gel (gradient from 30% to 50% AcOEt in hexanes as eluent): 71 mg orange oil, 55% yield.

Matching reported data[22]:

^{1}H NMR (500 MHz, $CDCl_3$) δ 8.33 (dd, J = 8.6, 2.2 Hz, 1H), 8.13 (d, J = 2.2 Hz, 1H), 7.01 (d, J = 8.7 Hz, 1H), 3.32 (s, 3H), 2.44-2.36 (m, 1H), 2.20-2.14 (m, 3H), 1.49 (s, 3H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 179.0, 148.8, 143.7, 132.7, 126.0, 118.7, 118.1, 108.2, 47.3, 32.9, 26.8, 23.4, 12.9.

HRMS (ESI pos): calculated for $C_{13}H_{14}N_3O_3^+$ ($M+H^+$): 260.1030, found 260.1027.



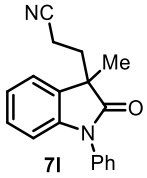
3-(1-benzyl-3-methyl-2-oxoindolin-3-yl)propanenitrile (7k) Synthesized according to the general procedure using chloroacetonitrile (48 μL, 0.75 mmol, 1.5 equiv.) and *N*-benzyl-*N*-phenylmethacrylamide (125.7 mg, 0.5 mmol, 1 equiv.). Chromatography on silica gel (gradient from 20% to 30% AcOEt in hexanes as eluent): 81 mg brown oil, 56% yield.

Matching reported data[22]:

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.37-7.28 (m, 5H), 7.25-7.22 (m, 2H), 7.12-7.09 (m, 1H), 6.82 (d, *J* = 7.7 Hz, 1H), 4.96 (d, *J* = 15.6 Hz, 1H), 4.91 (d, *J* = 15.6 Hz, 1H), 2.43-2.37 (m, 1H), 2.19-2.11 (m, 2H), 2.07-2.00 (m, 1H), 1.48 (s, 3H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 179.0, 142.3, 135.8, 131.7, 128.9, 128.6, 127.8, 127.3, 123.1, 122.7, 118.9, 109.6, 47.4, 43.9, 33.6, 23.8, 12.9.

HRMS (ESI pos): calculated for $C_{19}H_{18}N_2NaO^+$ ($M+Na^+$): 313.1311, found 313.1297.



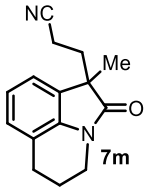
3-(3-Methyl-2-oxo-1-phenylindolin-3-yl)propanenitrile (7l). Synthesized according to the general procedure using chloroacetonitrile (48 μL, 0.75 mmol, 1.5 equiv.) and *N*,*N*-diphenylmethacrylamide (118.7 mg, 0.5 mmol, 1 equiv.). Chromatography on silica gel (gradient from 20% to 40% AcOEt in hexanes as eluent): 131 mg brown oil, 95% yield.

Matching reported data[22]:

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.57-7.54 (m, 2H), 7.46-7.43 (m, 3H), 7.30-7.26 (m, 2H), 7.19-7.16 (m, 1H), 6.90 (d, *J* = 7.8 Hz, 1H), 2.48-2.42 (m, 1H), 2.30-2.15 (m, 3H), 1.55 (s, 3H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 178.4, 143.1, 134.2, 131.5, 129.7, 128.6, 128.3, 126.5, 123.6, 123.0, 118.8, 109.9, 47.5, 33.7, 24.0, 13.0.

HRMS (ESI pos): calculated for $C_{18}H_{17}N_2O^+$ ($M+H^+$): 277.1335, found 277.1333.



3-(1-Methyl-2-oxo-1,2,5,6-tetrahydro-4*H*-pyrrolo[3,2,1-*ij*]quinolin-1-yl)propanenitrile (7m). Synthesized according to the general procedure using chloroacetonitrile (48 μL, 0.75 mmol, 1.5 equiv.) and 1-(3,4-dihydroquinolin-1(2*H*)-yl)-2-methylprop-2-en-1-one (100.6 mg, 0.5 mmol, 1 equiv.). Chromatography on silica gel (gradient from 20% to 45% AcOEt in hexanes as eluent): 76 mg brown oil, 63% yield.

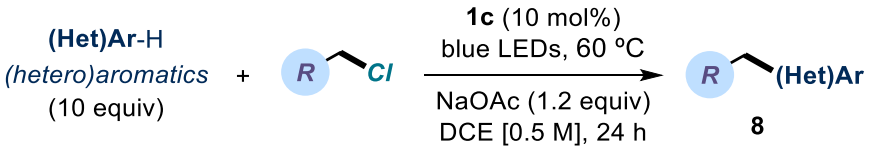
Matching reported data[25]:

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.09-6.99 (m, 3H), 3.78-3.69 (m, 2H), 2.81 (t, *J* = 6.0 Hz, 2H), 2.35-2.29 (m, 1H), 2.15-2.01 (m, 5H), 1.42 (s, 3H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 177.7, 138.9, 130.2, 127.4, 122.5, 120.7, 120.4, 118.9, 48.7, 38.9, 33.3, 24.5, 23.2, 21.2, 12.9.

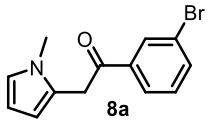
HRMS (ESI pos): calculated for $C_{15}H_{16}N_2NaO^+$ ($M+Na^+$): 263.1155, found 263.1157.

H. Radical addition to electron rich aromatic substrates



H.1. General procedure

In an oven dried Schlenk tube, catalyst **1c** (15.5 mg, 0.05 mmol, 0.1 equiv.) and sodium acetate (49 mg, 0.6 mmol, 1.2 equiv.) were suspended in 1,2-dichloroethane (1 mL), then the electrophile (0,5 mmol, 1 equiv.) was added followed by the (hetero)arene (5 mmol, 10 equiv.) substrate. The resulting yellow mixture was degassed via three cycles of freeze-pump-thaw. The Schlenk tube was then placed in the irradiation setup (see section C.1.) set at a temperature of 60 ºC (60-61ºC measured in the central well) and irradiated for 24 hours. After cooling to ambient temperature, the volatiles were evaporated and the residue purified by column chromatography to afford the corresponding product in the stated yield with >95% purity according to ^{1}H NMR analysis. The exact conditions for chromatography are reported for each compound.



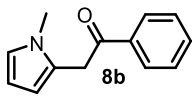
1-(3-Bromophenyl)-2-(1-methyl-1*H*-pyrrol-2-yl)ethan-1-one (8a). Synthesized according to the general procedure using 2-bromo-1-(3-bromophenyl)ethan-1-one (139 mg, 0.5 mmol, 1 equiv.) and N-methylpyrrole (0,444 mL, 5 mmol, 10 equiv.). Chromatography on silica gel (toluene as eluent): 90 mg off white solid, 65% yield.

^{1}H NMR (500 MHz, $CDCl_3$) δ 8.14 (t, *J*=1.7 Hz, 1H); 7.93 (dt, *J*= 7.8, 1 Hz, 1H); 7.70-7.66 (m, 1H); 7.34 (t, *J*= 7.8 Hz, 1H); 6.61 (t, *J*=2.3 Hz, 1H); 6.08 (t, *J*=3 Hz, 1H); 6.02-5.99 (m, 1H); 4.22 (s, 2H); 3.54 (s, 3H).

^{13}C NMR (125 MHz, $CDCl_3$) δ 195.4 (C); 138.4 (C); 136.5 (CH); 131.9 (CH); 130.6 (CH); 127.5 (CH); 125.1 (C); 123.4 (C); 123.1 (CH); 109.5 (CH); 107.6 (CH); 37.6 (CH_2); 34.4 (CH_3).

IR (thin film) ν 3064, 2944, 1686, 1566, 1494, 1417, 1299, 1206, 1088, 786 cm^{-1}.

HRMS (ESI pos): calculated for $C_{13}H_{13}{}^{79}BrNO$ (M^+): 278.0175, found 278.0188.

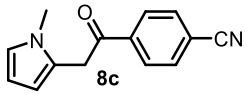


2-(1-Methyl-1*H*-pyrrol-2-yl)-1-phenylethan-1-one (8b). Synthesized according to the general procedure using phenacyl chloride (77 mg, 0.5 mmol, 1 equiv.) and *N*-methylpyrrole (0,444 mL, 5 mmol, 10 equiv.). Chromatography on silica gel (toluene as eluent): 70 mg off white solid, 70% yield.

Matching reported data[26]:

^{1}H NMR (300 MHz, $CDCl_3$) δ 8.12-8.01 (m, 2H); 7.66-7.56 (m, 1H); 6.65 (t, *J*= 2.2 Hz, 1H); 6.13 (t, *J*= 3 Hz, 1H); 7.56-7.44 (m, 2H); 6.08-6.03 (m, 1H); 4.30 (s, 2H); 3.59 (s, 3H).

^{13}C NMR (75 MHz, $CDCl_3$) δ 196.5 (C); 136.4 (C); 133.3 (CH); 128.7 (CH); 128.5 (CH); 125.4 (C); 122.6 (CH); 109.0 (CH); 107.1 (CH); 37.1 (CH_2); 34.1 (CH_3).



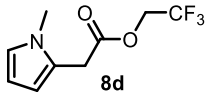
4-(2-(1-methyl-1*H*-pyrrol-2-yl)acetyl)benzonitrile (8c). Synthesized according to the general procedure using 4-(2-bromoacetyl)benzonitrile (112 mg, 0.5 mmol, 1 equiv.) and *N*-methylpyrrole (0,444 mL, 5 mmol, 10 equiv.). Chromatography on silica gel (1% AcOEt in toluene as eluent): 67 mg yellow solid, 60% yield.

^{1}H NMR (500 MHz, $CDCl_3$) δ 8.07 (t, *J*=8.6 Hz, 2H); 7.74 (t, *J*= 8.6 Hz, 2H); 6.60 (t, *J*=2.2 Hz, 1H); 6.06 (t, *J*= 3.1 Hz, 1H); 6.0-5.97 (m, 1H), 4.25 (s, 2H); 3.54 (s, 3H).

^{13}C NMR (125 MHz, $CDCl_3$) δ 195.5 (C); 139.6 (C); 132.9 (CH); 129.4 (CH); 124.6 (C); 123.4 (CH); 118.2 (C); 116.8 (C); 109.7 (CH); 107.7 (CH); 37.9 (CH_2); 34.4 (CH_3).

IR (thin film) ν 2923, 2851, 2229, 1691, 1493, 1402, 1298, 1210, 990, 716 cm^{-1}.

HRMS (ESI pos): calculated for $C_{14}H_{12}N_2NaO$ ($M+Na^+$): 247.0842, found 247.0840.



2,2,2-trifluoroethyl 2-(1-methyl-1*H*-pyrrol-2-yl)acetate (8d). Synthesized according to the general procedure using 2,2,2-trifluoroethyl 2-chloroacetate (88 mg, 0.5 mmol, 1 equiv.) and *N*-methylpyrrole (0,444 mL, 5 mmol, 10 equiv.). Chromatography on silica gel (toluene as eluent): 41 mg slightly yellow oil, 37% yield.

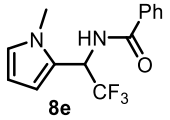
^{1}H NMR (500 MHz, $CDCl_3$) δ 6.60 (t, *J*= 2.2 Hz, 1H); 6.09-6.05 (m, 2H); 4.48 (q, *J*=8.5 Hz, 2H); 3.72 (s, 2H); 3.57 (s, 3H).

^{13}C NMR (125 MHz, $CDCl_3$) δ 169.3 (C); 123.8 (C); 123.3 (CH); 123.2 (q, *J*=277 Hz, C); 109.5 (CH); 107.5 (CH); 61.0 (q, *J*=36.8 Hz, CH_2); 34.1 (CH_3); 32.2 (CH_2);

^{19}F NMR (376 MHz, $CDCl_3$, proton decoupled) δ -73.85 (s, 3F).

IR (thin film) ν 2963, 1756, 1495, 1410, 1278, 1160, 1049, 977, 798, 711 cm^{-1}.

HRMS (ESI pos): calculated for $C_9H_{11}F_3NO2$ (M^+): 222.0736, found 222.0742.



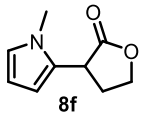
***N*-(2,2,2-trifluoro-1-(1-methyl-1*H*-pyrrol-2-yl)ethyl)benzamide (8e).** Synthesized according to the general procedure using *N*-(1-chloro-2,2,2-trifluoroethyl)benzamide (119 mg, 0.5 mmol, 1 equiv.) and *N*-methylpyrrole (0,444 mL, 5 mmol, 10 equiv.). Chromatography on silica gel (gradient from toluene to 5% AcOEt in toluene as eluent): 113 mg off-white solid, 80% yield.

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.84-7.66 (m, 2H); 7.60-7.53 (m, 1H); 7.51-7.43 (m, 2H); 6.70-6.67 (m, 1H); 6.48 (br d, *J*= 9 Hz, 1H); 6.41-6.36 (m, 1H); 6.16 (dd, *J*= 2.9, 3.7 Hz, 1H); 6.14-6.06 (m, 1H); 3.67 (s, 3H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 166.5 (C); 132.9 (C); 132.3 (CH); 128.7 (CH); 127.2 (CH); 124.5 (q, *J*=282 Hz, C); 124.0 (CH); 123.8 (CH); 108.5 (q, *J*=1.5 Hz, C); 107.7 (CH); 47.2 (q, *J*=33.1 Hz, CH); 34.1 (CH_3). ^{19}F NMR (376 MHz, $CDCl_3$, proton decoupled) δ -73.86 (s, 3F).

IR (thin film) ν 3287, 2934, 1642, 1517, 1440, 1267, 1171, 1109, 709, 693 cm^{-1}.

HRMS: calculated for $C_{14}H_{13}F_3N_2NaO$ ($M+Na^+$): 305.0872, found 305.0876.

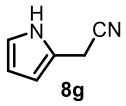


3-(1-Methyl-1*H*-pyrrol-2-yl)dihydrofuran-2(3*H*)-one (8f). Synthesized according to the general procedure using 3-*bromo*dihydrofuran-2(3*H*)-one (41 μL, 0.5 mmol, 1 equiv.) and *N*-methylpyrrole (0,444 mL, 5 mmol, 10 equiv.). Chromatography on silica gel (gradient from 10% AcOEt in hexanes to 40% AcOEt in hexanes as eluent): 76 mg brown oil, 91% yield.

^{1}H NMR (400 MHz, $CDCl_3$) δ 6.63 (app t, *J* = 4.5 Hz, 1H), 6.09 (app t, *J* = 3.6 Hz, 1H), 6.05-6.04 (m, 1H), 4.52-4.47 (m, 1H), 4.39-4.33 (m, 1H), 3.84 (t, *J* = 9.0 Hz, 1H), 3.69 (s, 3H), 2.69-2.61 (m, 1H), 2.57-2.48 (m, 1H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 176.0, 126.8, 123.4, 107.0, 106.4, 66.8, 37.7, 34.2, 29.2.

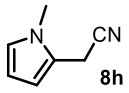
HRMS: calculated for $C_9H_{11}NNaO_2$ ($M+Na^+$): 188.0682, found 188.0680.



2-(1*H*-pyrrol-2-yl)acetonitrile (8g). Synthesized according to a modification of the general procedure: in an oven dried Schlenk tube, **1c** (31 mg, 0.1 mmol, 0.1 equiv.) and sodium acetate (93 mg, 1.2 mmol, 1.2 equiv.) were suspended in 1,2-dichloroethane (2 mL), then chloroacetonitrile (63 μL, 1 mmol, 1 equiv.) and pyrrole (0,699 mL, 10 mmol, 10 equiv.) were added. The resulting yellow mixture was degassed via three cycles of freeze-pump-thaw. The Schlenk tube was then placed in the irradiation setup at a temperature of 60 ºC and irradiated for 24 hours. After cooling to ambient temperature, the volatiles were evaporated and the residue purified by column chromatography to afford the corresponding product in the stated yield with >95% purity according to ^{1}H NMR analysis. Chromatography on silica gel (gradient from 15% AcOEt in hexanes to 30% AcOEt in hexanes as eluent) followed by a second chromatography (gradient from toluene to 50% AcOEt in toluene as eluent): 91 mg brownish oil, 86% yield.

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.4 (br s, 1H); 6.81-6.79 (m, 1H); 6.22-6.18 (m, 1H); 6.22-6.14 (m, 1H); 3.78 (s, 2H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 118.9 (CH); 118.6 (C); 117.2 (C); 109.2 (CH); 108.3 (CH); 16.9 (CH_2).
HRMS (ESI neg): calculated for $C_6H_5N_2$ (M^-): 105.0458, found 105.0454.

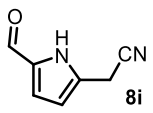


2-(1-Methyl-1*H*-pyrrol-2-yl)acetonitrile (8h). Synthesized according to the general procedure using chloroacetonitrile (32 μL, 0.5 mmol, 1 equiv.) and *N*-methylpyrrole (0.444 mL, 5 mmol, 10 equiv.). Chromatography on silica gel (toluene as eluent): 55 mg yellow oil, 92% yield.

Matching reported data[27]:

^{1}H NMR (400 MHz, $CDCl_3$) δ 6.66 (app t, *J*= 2.2 Hz, 1H); 6.17-6.12 (m, 1H); 6.10 (app t, *J*= 3.5 Hz, 1H); 3.69 (s, 2H); 3.64 (s, 3H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 123.6 (CH); 120.0 (C); 116.6 (C); 109.1 (CH); 107.4 (CH); 33.8 (CH_3); 15.9 (CH_2).

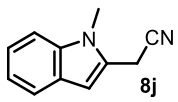


2-(5-Formyl-1*H*-pyrrol-2-yl)acetonitrile (8i). Synthesized according to the general procedure using chloroacetonitrile (32 μL, 0.5 mmol, 1 equiv.) and 1*H*-pyrrole-2-carbaldehyde (476 mg, 5 mmol, 10 equiv.). Chromatography on silica gel (15% acetone in hexanes as eluent): 28 mg slightly brown crystalline solid, 42% yield.

^{1}H NMR (400 MHz, $CDCl_3$) δ 9.40 (s, 1H);7.00-6.95 (m, 1H); 6.39-6.34 (m, 1H); 3.91 (s, 2H); NH proton not detected.

^{13}C NMR (100 MHz, $CDCl_3$) δ 179.7 (CH); 133.15 (C); 130.3 (C); 123.4 (CH); 115.9 (C); 111.2 (CH); 17.0 (CH_2).

HRMS (ESI negative): calculated for $C_7H_5N_2O$ (M^-): 133.0407, found 133.0401.



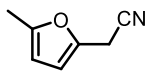
8j

2-(1-Methyl-1*H*-indol-2-yl)acetonitrile (8j). Synthesized according to the general procedure using chloroacetonitrile (32 μL, 0.5 mmol, 1 equiv.) and *N*-methylindole (0.444 mL, 5 mmol, 10 equiv.). Chromatography on silica gel (gradient from 10% AcOEt in hexanes to 15% AcOEt in hexanes as eluent): 67 mg yellow solid, 79% yield.

Matching reported data[28]:

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.59 (app d, 1H); 7.30-7.23 (m, 2H); 7.15 (dd, *J*= 6.6, 1.5 Hz, 1H); 6.50 (d, *J*= 0.7 Hz, 1H); 3.76 (s, 2H); 3.65 (s, 3H).

^{13}C NMR (125 MHz, $CDCl_3$) δ 138.3 (C); 128.2 (C); 127.5 (C); 122.6 (CH); 121.0 (CH); 120.5 (CH); 116.5 (C); 109.6 (CH); 102.5 (CH); 30.1 (CH_3); 16.9 (CH_2).



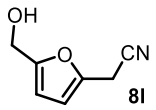
2-(5-Methylfuran-2-yl)acetonitrile (8k). Synthesized according to a modification of the general procedure: in an oven dried Schlenk tube, catalyst **1c** (31 mg, 0.1 mmol, 0.1 equiv.) and sodium acetate (93 mg, 1.2 mmol, 1.2 equiv.) were dissolved in 1.2-dichloroethane (2 mL), then chloroacetonitrile (63 μL, 1 mmol, 1 equiv.) and 2-methylfuran (0.902 mL, 10 mmol, 10 equiv.) were added. The resulting yellow mixture was degassed via three cycles of freeze-pump-thaw. The Schlenk tube was then placed in the irradiation setup at a temperature of 60 ºC and irradiated for 24 hours. After cooling to ambient temperature, the volatiles were evaporated and the residue purified by column chromatography to afford the corresponding product in the stated yield with >95% purity according to ^{1}H NMR analysis. Chromatography on silica gel (5% AcOEt in hexanes): 78 mg yellow oil, 64% yield.

Note: the product is volatile and care should be taken during the evaporation steps to avoid losses (no vacuum below 80 mbar was applied).

Matching reported data[28]:

^{1}H NMR (500 MHz, $CDCl_3$) δ 6.16 (app d, *J*= 3.1 Hz, 1H); 5.90 (dd, *J*= 3.1, 1 Hz, 1H); 3.68 (s, 2H); 2.25 (s, 3H).

^{13}C NMR (125 MHz, $CDCl_3$) δ 153.3 (C); 141.2 (C); 116.1 (C); 109.6 (CH); 107.0 (CH); 17.9 (CH_2); 13.8 (CH_3).



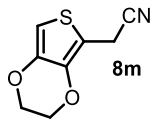
8l

2-(5-(Hydroxymethyl)furan-2-yl)acetonitrile (8l). Synthesized according to the general procedure using chloroacetonitrile (32 μL, 0.5 mmol, 1 equiv.) and furfuryl alcohol (0,434 mL, 5 mmol, 10 equiv.). Chromatography on silica gel (gradient from 20% AcOEt in hexanes to 40% AcOEt in hexanes): 35 mg yellow oil, 50% yield.

Matching reported data[28]:

^{1}H NMR (500 MHz, $CDCl_3$) δ 6.29-6.27 (m, 1H); 6.27-6.25 (m, 1H); 4.57 (s, 2H); 3.77 (s, 2H); 2.28 (br s, 1H).

^{13}C NMR (125 MHz, $CDCl_3$) δ 154.8 (C); 142.9 (C); 115.5 (C); 109.3 (CH); 108.9 (CH); 57.1 (CH_2); 17.57 (CH_2).



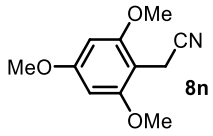
2-(2,3-Dihydrothieno[3,4-b][1,4]dioxin-5-yl)acetonitrile (8m). Synthesized according to the general procedure using chloroacetonitrile (32 μL, 0.5 mmol, 1 equiv.) and 2,3-dihydrothieno[3,4-*b*][1,4]dioxine (0,534 mL, 5 mmol, 10 equiv.). Chromatography on silica gel (gradient from 10% AcOEt in hexanes to 30% AcOEt in hexanes, then a second chromatography with toluene as eluent): 71 mg, slightly yellow oil, 78% yield.

^{1}H NMR (500 MHz, $CDCl_3$) δ 6.24 (s, 1H); 4.21-4.18 (m, 2H); 4.16-4.13 (m, 2H); 3.67 (s, 2H).

^{13}C NMR (125 MHz, $CDCl_3$) δ 141.4 (C); 149.8 (C); 116.7 (C); 103.6 (C); 98.4 (CH); 64.8 (CH_2); 64.5 (CH_2); 14.7 (CH_2).

IR (thin film) ν 3117, 2929, 2252, 1604, 1503, 1436, 1367, 1161, 1069, 919 cm^{-1}.

HRMS (ESI pos): calculated for $C_8H_7NnaO2S$ ($M+Na^+$): 204.0090, found 204.0086.



2-(2,4,6-trimethoxyphenyl)acetonitrile (8n). Synthesized according to the general procedure using chloroacetonitrile (32 μL, 0.5 mmol, 1 equiv.) and 1,3,5-trimethoxybenzene (841 mg, 5 mmol, 10 equiv.). Chromatography on silica gel (gradient from toluene to 5% AcOEt in toluene as eluent): 44 mg, off white solid, 42% yield.

^{1}H NMR (400 MHz, $CDCl_3$) δ 6.14 (s, 2H); 3.85 (s, 6H); 3.82 (s, 3H); 3.61 (s, 2H).

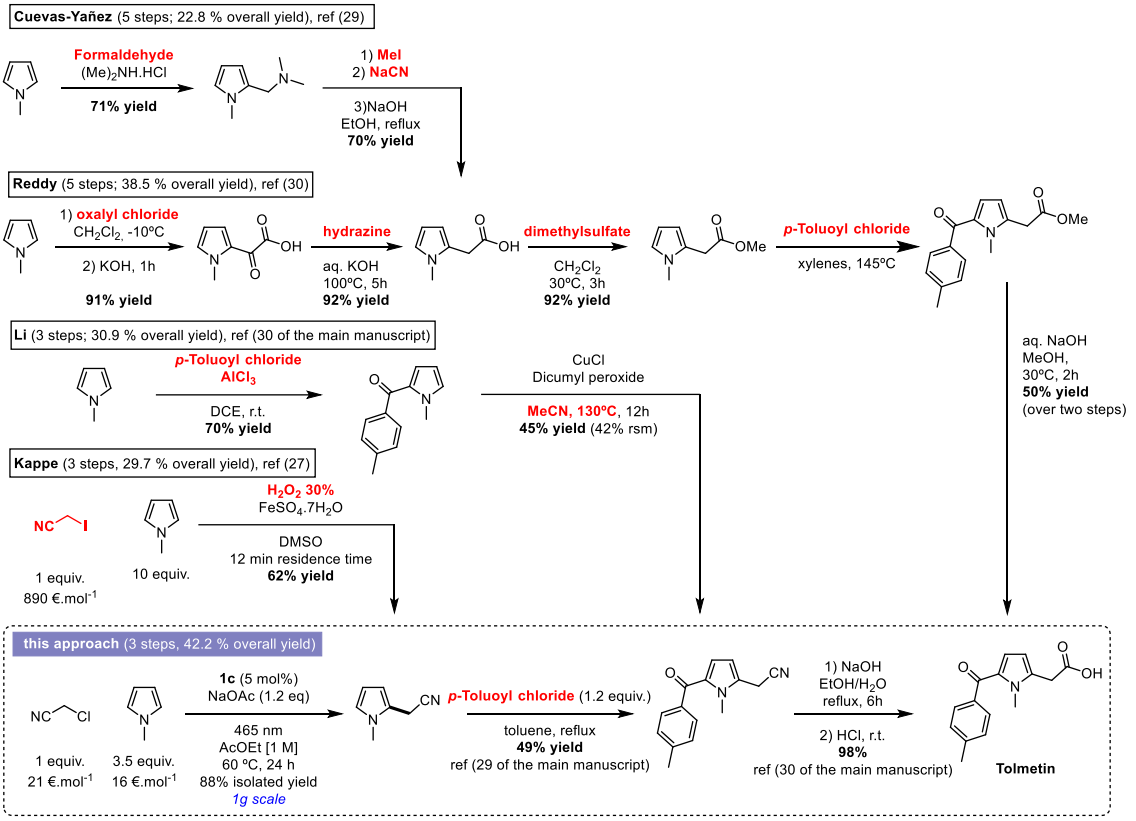
^{13}C NMR (100 MHz, $CDCl_3$) δ 161.4 (C); 158.6 (C); 118.8 (C); 99.3 (C); 90.4 (CH); 55.8 (CH_3); 55.4 (CH_3); 11.1 (CH_2).

IR (thin film) ν 2945, 2844, 2245, 1600, 1465, 1203, 1150, 1121, 1057, 1036 cm^{-1}.

HRMS (ESI pos): calculated for $C_{11}H_{14}NO_3$ ($M+H^+$): 208.0968, found 208.0971.

I. Streamlined Preparation of a Marketed Drug and Late-Stage Elaboration of Biorelevant Compounds

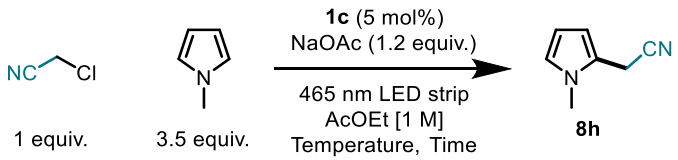
I.1. Analysis of Reported (Formal) Syntheses of Tolmetin and Comparison with Dithiocarbamate Anion Catalysis



Supplementary Figure 21: Formal synthesis of Tolmetin using dithiocarbamate anion catalysis and comparison with previously reported routes. All prices of reagents determined according to Sigma-Aldrich catalog prices in the highest quantity available; Hazardous reagents (classified as category 1 reagent in at least one category according to Regulation (EC) No 1272/2008) and conditions (reaction temperature significantly above the boiling point of the reaction solvent) are highlighted in red.

I.2. Synthesis of the precursor 8h: control experiments and scale up studies

Supplementary Table 3: Dithiocarbamate Anion-Catalyzed Alkylation of *N*-Methylpyrrole at different scales



Entry	Scale (mmol)	Temperature (ºC)	time (h)	Conversion (%)	Isolated yield (%)
1	0.5	60	24	100	91
2	10	60	24	100	88
3	175	75	116	45	39.5 (87)[a]

a) Yield based on conversion

Increasing the scale of the alkylation reaction to 10 mmol gave a 88% isolated yield of product in 24 hours (entry 2). Performing the multigram-scale preparation of adduct **8h** required the use of a large reaction vessel, which greatly decreased the efficiency of irradiation. The situation was further complicated by the fact that the reaction mixture developed a dark colour after few hours, probably as a consequence of partial polymerization of the *N*-methylpyrrole substrate. This further hampered an efficient irradiation of the solution. Therefore, at a 0.175 mol scale, the conversion stalled at about 40% after 48 hours of irradiation and little or no conversion was observed past this point. We decided to stop the reaction after 116 hours to obtain the product **8h** in 39.5% isolated yield at 45% conversion (entry 3).

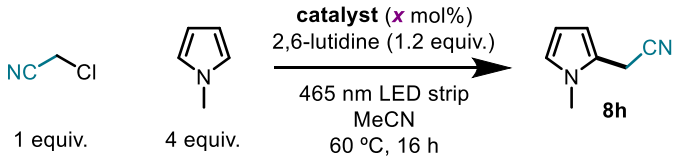
Procedure for the 0.175 mmol reaction: In an oven dried 250 mL capacity Schlenk tube, catalyst **1c** (2.71 g, 8.75 mmol, 0.05 equiv.) and sodium acetate (17.23 g, 0.21 mol, 1.2 equiv.) were suspended in ethyl acetate (175 mL, degassed *via* nitrogen sparging for 30 minutes prior to the reaction), then chloroacetonitrile (11.07 mL, 0.175 mol, 1 equiv.) was added followed by *N*-methylpyrrole (54.4 mL, 0.613 mol, 3.5 equiv.) substrate. The reaction tube was then immersed in a 1L beaker containing silicon oil heated to 60 ºC and wrapped with a one meter 14W blue LED strip and further wrapped with aluminium foil. The reaction mixture was further degassed via nitrogen sparging for 5 minutes at this temperature then irradiated for 116 hours under strong stirring. NMR analysis of an aliquot at this time showed a 45% conversion of chloroacetonitrile.

The mixture was transferred to an extraction funnel and washed with distilled water twice, followed by a saturated solution of $NaHCO_3$. The combined aqueous layers were back extracted with AcOEt and the combined organic layers were dried ($MgSO_4$) and concentrated to a brown oil (14.3 g). A yield of **9** of 42% was determined by ^1H NMR analysis of the residue after addition of an internal standard. Vacuum distillation (1.4 mbar, 85-91ºC) afforded 7.631g of **9** as a slightly yellow iridescent liquid (63.525 mmol, 36.3% yield). After rinsing the distillation apparatus back into the boiling flask, an additional 0.67 g of **9** was obtained after column chromatography as a red oil.

The combined product obtained corresponds to 8.301 g (69.075 mmol, 39.5 % yield)

Control experiments:

Supplementary Table 4: *N*-Methylpyrrole alkylation with chloroacetonitrile



entry	catalyst	catalyst loading	yield (%)	Estimated product cost[a]
1	**1c**	5 (mol%)	90	144 (€·mol^{-1})
2	$Ru(bpy)_3^{2+}$	1 (mol%)	-	-
3	$Ir(ppy)_3$	1 (mol%)	69[b]	4780 (€·mol^{-1})

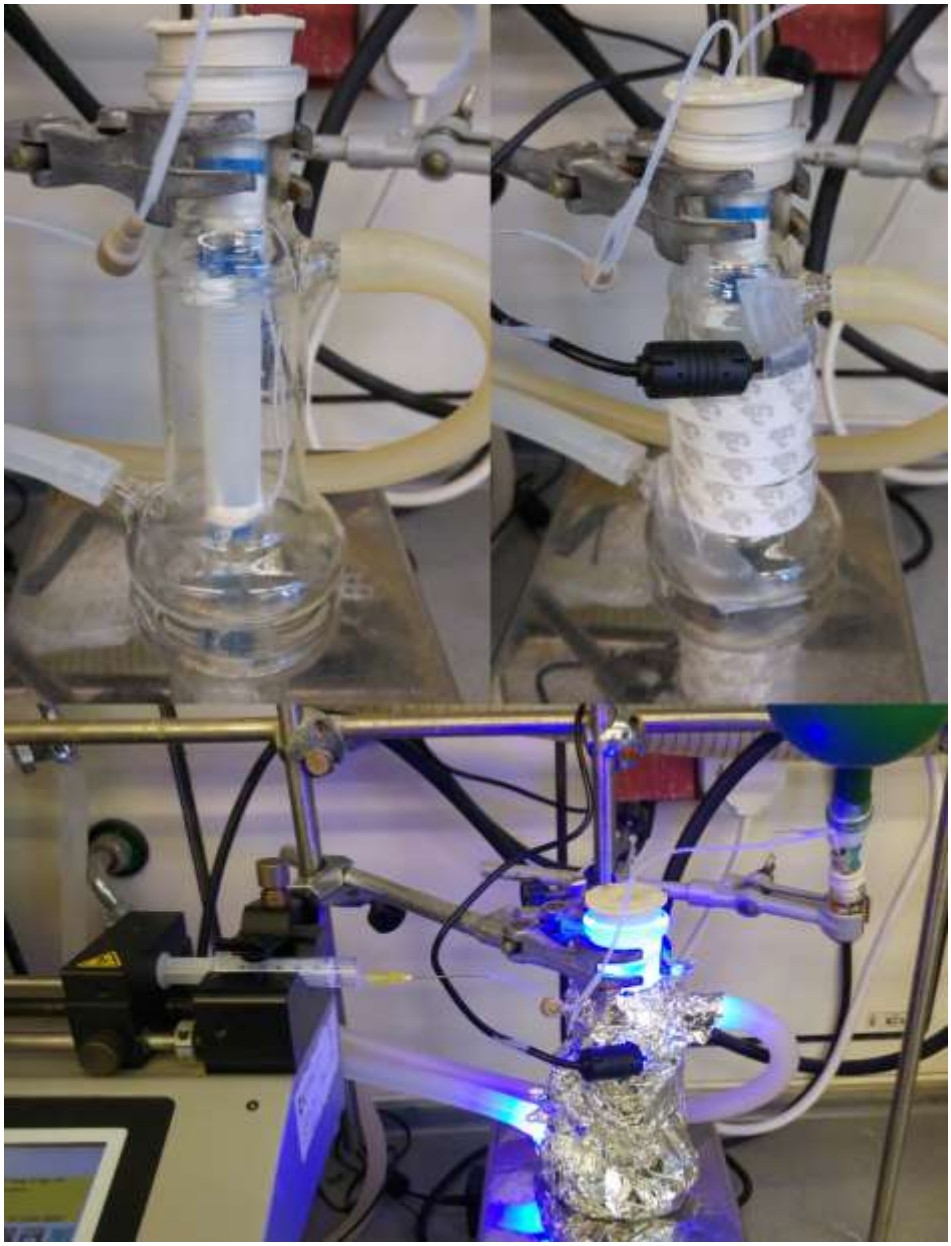
Reactions performed on a 0.5 mmol scale at 60 °C over 16 hours. [a]Cost estimation using Sigma-Aldrich catalogue prices of all the reaction components (solvent excluded) in their highest available quantity weighed through the yield of reaction. [b]NMR yield determined by comparison with an internal standard on the crude reaction mixture.

We explored the possibility to use photoredox catalysis to perform the alkylation of *N*-methylpyrrole with chloroacetonitrile (which has a reported E_{red} of -1.96 V vs SCE).[31] $Ru(bpy)_3$ (E(Ru(II)*/Ru(III))= -0.81 V vs SCE) did not give any product. The more reducing $Ir(ppy)_3$ (E(Ir(III)*/Ir(VI))= -1.73 V vs SCE) afforded product **8h** in a moderate yield of 67% (entry 3). The dithiocarbamate-based catalyst **1c** afforded **8h** in 90% yield under the same conditions (entry 1). The high cost of the iridium complex means that the iridium-based photoredox system is 30 times more expensive than the presented method.

I.3. Continuous-flow photochemistry studies

The continuous-flow photoreactor:

The flow photoreactor was assembled using a variation of the temperature-controlled photoreactor used for batch reactions. It consisted of a smaller 4 cm diameter jar fitted with a standard 29-sized ground glass joint. A commercial 1-meter LED strip was wrapped around the jar, followed by a layer of aluminium foil and cotton for insulation. The reactor itself consisted of a length of standard HPLC tubing (0.78 mm ID, PTFE) wrapped around a 14 mm diameter test tube to allow an irradiated volume of 1 mL and fitted inside the jar. A syringe pump was used to circulate the reaction mixture at a defined rate (Supplementary Supplementary Figure)

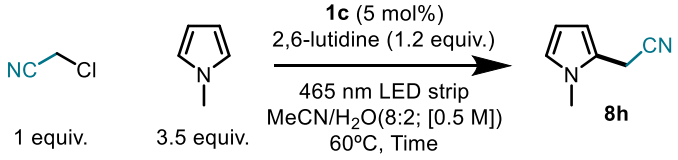


Supplementary Figure 22: Flow photoreactor at different stage of construction and under operation.

Flow conversion studies

To provide homogeneous conditions throughout the progress of the reaction and avoid clogging of the flow reactor, the solvent and base had to be replaced by acetonitrile/water mixtures and 2,6-lutidine, respectively. Using sodium acetate as the base gave phase separation between acetonitrile and water before the start of the reaction without coalescence upon heating to 60 ºC.

Supplementary Table 5: Dithiocarbamate Anion-Catalyzed Alkylation of *N*-Methylpyrrole under batch and flow conditions

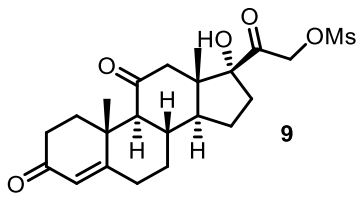


Entry	Concentration	Time	NMR yield (%)
1	[0.5]	6 h (batch)	50
2	[0.5]	16 h (batch)	92
3	[0.5]	20 min (flow)[a]	22
4	[0.5]	40 min (flow)[a]	43
5	[0.5]	2 h (flow)[a]	75
6	[1]	2 h (flow)[a]	82[b]
7	[1.5]	2 h (flow)[a]	87[c]
8	[2]	2 h (flow)[a]	75[d]

All reactions performed on a 0.5 mmol scale, NMR yield determined by comparison with an internal standard on the crude reaction mixture. a) Theoretical residence time calculated according to the flow rate programmed. b) 1 mmol scale. c) 1.5 mmol scale. d) 2 mmol scale.

To accurately benchmark the batch and flow systems, we compared similar scales of reaction leading to the adduct **8h**. On a 0.5 mmol scale and under batch conditions, the productivity was 0.028 mmol·h^{-1} (result refers to the formation of product **8h** reported in Figure 3d of the manuscript, or Supplementary Table 5, entry 2). Under flow conditions, the productivity increased fourteen fold to 0.40 mmol·h^{-1} (calculated under continuous operation, result refers to the formation of product **8h** reported in Figure 4a, or Supplementary Table 5, entry 7).

I.4. Synthesis of cortisone analogues.



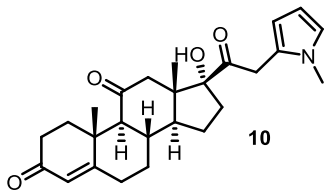
9

2-((8*S*,9*S*,10*R*,13*S*,14*S*,17*R*)-17-hydroxy-10,13-dimethyl-3,11-dioxo-2,3,6,7,8,9,10,11,12,13,14,15,16,17-tetradecahydro-1*H*-cyclopenta[*a*]phenanthren-17-yl)-2-oxoethyl methanesulfonate (cortisone mesylate) (9). Synthesized according to a procedure adapted from a patent by The Upjohn Company - EP263213, 1988, A1. A round-bottomed flask was charged with cortisone (500 mg, 1.39 mmol, 1.0 equiv.), followed by pyridine (5 mL). The flask was cooled in an ice bath and methanesulfonyl chloride (113 μL, 1.46 mmol, 1.05 equiv.) was added. The reaction was allowed to warm to ambient temperature as the reaction proceeded for a total reaction time of 1 h. The crude reaction was concentrated to remove pyridine, then the residue was dissolved in CH_2Cl_2 and washed with 1M HCl twice. The organic layer was dried with sodium sulfate and concentrated in vacuo. Chromatography on silica gel (gradient from 40% to 70% AcOEt in hexanes as eluent): 307.0 mg, 51% yield.

^{1}H NMR (500 MHz, $CDCl_3$) δ 5.72 (s, 1H), 5.32 (d, *J* = 18.1 Hz, 1H), 4.89 (d, *J* = 18.1 Hz), 3.20 (s, 3H), 2.97 (bs, 1H), 2.89 (d, *J* = 12.4 Hz, 1H), 2.83-2.73 (m, 2H), 2.51-2.27 (m, 5H), 2.17 (d, J = 12.4 Hz, 1H), 2.00-1.91 (m, 4H), 1.75-1.60 (m, 2H), 1.52-1.44 (m, 1H), 1.40 (s, 3H), 1.34-1.25 (m, 1H), 0.67 (s, 3H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 208.6, 203.7, 200.1, 168.9, 124.6, 89.0, 72.1, 62.5, 51.5, 49.9, 49.8, 39.3, 38.2, 36.5, 35.4, 34.7, 33.7, 32.3, 32.2, 23.2, 17.2, 16.0.

HRMS (ESI pos): calculated for $C_{22}H_{30}NaO_7S^+$ ($M+Na^+$): 461.1604, found 461.1604.



10

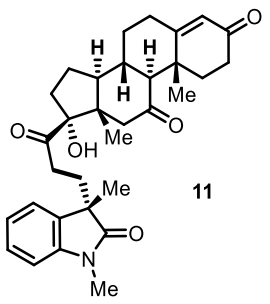
(8*S*,9*S*,10*R*,13*S*,14*S*,17*R*)-17-hydroxy-10,13-dimethyl-17-(2-(1-methyl-1*H*-pyrrol-2-yl)acetyl)-1,6,7,8,9,10,12,13,14,15,16,17-dodecahydro-3*H*-cyclopenta[*a*]phenanthrene-3,11(2*H*)-dione (10). Synthesized according to the general procedure using cortisone mesylate **9** (219 mg, 0.5 mmol, 1 equiv.) and *N*-methylpyrrole (0,444 mL, 5 mmol, 10 equiv.). 20 mol % catalyst was used: **1c** (31.0 mg, 0.10 mmol, 0.2 equiv.). Chromatography on silica gel (gradient from 40% AcOEt in hexanes to 55% AcOEt in hexanes; three rounds of chromatography were required): 53 mg pink foam, 25% yield.

^{1}H NMR (500 MHz, $CDCl_3$) δ 6.58-6.57 (m, 1H), 6.04-6.03 (m, 1H), 5.91-5.90 (m, 1H), 5.70 (s, 1H), 3.97 (d, *J* = 16.7 Hz, 1H), 3.67 (d, *J* = 16.7 Hz, 1H), 3.50 (s, 3H), 2.95 (br s, 1H), 2.81-2.71 (m, 3H), 2.49-2.24 (m, 5H), 1.98-1.89 (m, 5H), 1.72-1.65 (m, 1H), 1.65-1.58 (m, 1H), 1.47-1.43 (m, 1H), 1.38 (s, 3H), 1.32-1.24 (m, 1H), 0.69 (s, 3H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 209.8, 208.8, 200.2, 169.1, 125.0, 124.9, 123.2, 108.9, 107.6, 89.8, 62.9, 52.0, 50.5, 50.0, 38.6, 38.1, 36.9, 35.2, 35.1, 34.5, 34.1, 32.7, 32.6, 23.8, 17.6, 16.5.

IR (thin film) ν 3433, 2942, 1703, 1656, 1494, 1442, 1270, 1221, 1053, 734 cm^{-1}.

HRMS: calculated for $C_{26}H_{33}NNaO_4$ ($M+Na^+$): 446.2302, found 446.2306.



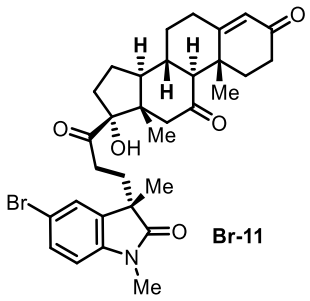
11

(8*S*,9*S*,10*R*,13*S*,14*S*,17*R*)-17-(3-(1,3-dimethyl-2-oxoindolin-3-yl)propanoyl)-17-hydroxy-10,13-dimethyl-1,6,7,8,9,10,12,13,14,15,16,17-dodecahydro-3*H*-cyclopenta[a]phenanthrene-3,11(2*H*)-dione (11). Synthesized according to the general procedure using cortisone mesylate **9** (219.3 mg, 0.5 mmol, 1.0 equiv.) and *N*-methyl-*N*-phenylmethacrylamide (131.4 mg, 0.75 mmol, 1.5 equiv.). 20 mol % catalyst was used: **1c** (31.0 mg, 0.10 mmol, 0.2 equiv.). The diastereomeric ratio was determined by ^{1}H NMR spectroscopic analysis of the crude reaction mixture by comparison of the resonances at δ 3.17 (minor diastereomer) and δ 3.14 (major diastereomer). Chromatography on silica gel (gradient from 40% to 100% AcOEt in hexanes as eluent): 142 mg major diastereomer as an off-white solid and 62 mg minor diastereomer (isolated as 2.2:1 dr mixture favoring the minor diastereomer, off-white solid), 79% combined yield.

^{1}H NMR for major diastereomer (500 MHz, $CDCl_3$) δ 7.31-7.28 (m, 1H), 7.16 (d, *J* = 6.6 Hz, 1H), 7.10-7.07 (m, 1H), 6.87 (d, *J* = 7.8 Hz, 1H), 5.73 (s, 1H), 3.19 (s, 3H), 2.96-2.91 (m, 1H), 2.88 (d, *J* = 12.1 Hz, 1H), 2.78-2.66 (m, 2H), 2.51-2.38 (m, 3H), 2.32-2.27 (m, 2H), 2.21-2.09 (m, 2H), 2.01 (d, *J* = 12.5 Hz, 1H), 1.99-1.87 (m, 5H), 1.67-1.57 (m, 2H) 1.39 (s, 3H), 1.38 (s, 3H), 1.36-1.25 (m, 2H), 0.49 (s, 3H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 211.1, 209.8, 199.8, 180.6, 168.8, 142.7, 134.2, 128.1, 124.5, 122.9, 122.7, 108.3, 89.1, 62.6, 50.5, 50.3, 49.7, 47.0, 38.2, 36.6, 34.7, 33.7, 33.5, 33.2, 32.5, 32.3, 32.3, 26.3, 23.3, 22.6, 17.2, 15.8.
HRMS (ESI pos): calculated for $C_{32}H_{40}NO_5^+$ ($M+H^+$): 518.2901, found 518.2904.

Stereochemistry of the major diastereomer was determined by synthesis of the brominated derivative below, which was purified (the major diastereomer was isolated) and analyzed by x-ray diffraction.



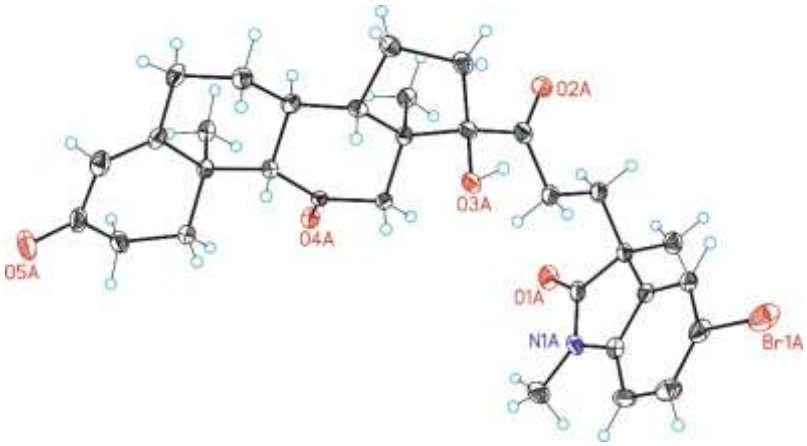
Br-11

(8*S*,9*S*,10*R*,13*S*,14*S*,17*R*)-17-(3-((*S*)-5-bromo-1,3-dimethyl-2-oxoindolin-3-yl)propanoyl)-17-hydroxy-10,13-dimethyl-1,6,7,8,9,10,12,13,14,15,16,17-dodecahydro-3*H*-cyclopenta[*a*]phenanthrene-3,11(2*H*)-dione (Br-11) Synthesized according to the general procedure using cortisone mesylate (219.3 mg, 0.5 mmol, 1.0 equiv.) and *N*-(4-bromophenyl)-*N*-methylmethacrylamide (190.6 mg, 0.75 mmol, 1.5 equiv.). 20 mol % catalyst was used: **1c** (31.0 mg, 0.10 mmol, 0.2 equiv.). The diastereomeric ratio was determined by ^{1}H spectroscopic analysis of the crude reaction mixture by comparison of the resonances at δ 3.12 (minor diastereomer) and δ 3.09 (major diastereomer). Chromatography on silica gel (gradient from 40% to 100% AcOEt in hexanes as eluent): 98 mg major diastereomer (33% yield; only collected pure fractions of major diastereomer) as a white solid. Recrystallization from heptane and ethyl acetate provided a crystal, which was analyzed by x-ray diffraction.

^{1}H NMR for major diastereomer (500 MHz, $CDCl_3$) δ 7.41 (dd, *J* = 8.3, 1.9 Hz, 1H), 7.27 (d, *J* = 2.1 Hz, 1H), 6.74 (d, *J* = 8.3 Hz, 1H), 5.72 (s, 1H), 3.71 (bs, 1H), 3.15 (s, 3H), 3.03-2.97 (m, 1H), 2.89 (d, *J* = 12.5 Hz, 1H), 2.77-2.69 (m, 2H), 2.51-2.38 (m, 3H), 2.31-2.27 (m, 2H), 2.17-2.06 (m, 2H), 2.02 (d, *J* = 12.5 Hz, 1H), 1.99-1.86 (m, 5H), 1.67-1.59 (m, 2H), 1.39 (s, 3H), 1.37 (s, 3H), 1.35-1.25 (m, 2H), 0.51 (s, 3H).

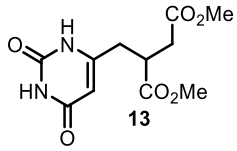
^{13}C NMR for major diastereomer (126 MHz, $CDCl_3$) δ 211.1, 210.0, 200.0, 180.4, 168.9, 142.1, 136.8, 131.3, 126.3, 125.0, 116.0, 110.1, 89.5, 63.0, 50.8, 50.7, 50.1, 47.5, 38.5, 37.0, 35.1, 34.1, 33.4, 32.9, 32.7, 32.6, 31.3, 26.7, 23.6, 22.8, 17.6, 16.2.

IR (thin film) ν 3440, 2938, 1703, 1668, 1607, 1488, 1346, 1270, 1131, 735 cm^{-1}.
HRMS (ESI pos): calculated for $C_{32}H_{38}{}^{79}BrNNaO_5^+$ ($M+Na^+$): 618.1826, found 618.1831.



Supplementary Figure 23: ORTEP Diagram of **Br-11** (CCDC 1839232).

I.5. Synthesis of nucleobase analogues



13

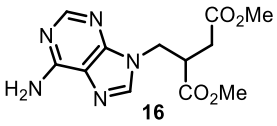
Dimethyl 2-((2,6-dioxo-1,2,3,6-tetrahydropyrimidin-4-yl)methyl)succinate (13): In an oven dried Schlenk tube, 6-(chloromethyl)pyrimidine-2,4(1*H*,3*H*)-dione **12** (126 mg, 0.75 mmol, 1.5 equiv.) was dissolved in DMF (1 mL), then catalyst **1c** (15.5 mg, 0.1 equiv.) was added followed by 2,6-lutidine (70 μL, 0.6 mmol, 1.2 equiv.), γ-terpinene (96 μL, 0.6 mmol, 1.2 equiv.), and dimethyl fumarate **3a** (72 mg, 0.5 mmol, 1 equiv.). The resulting yellow mixture was degassed via three cycles of freeze-pump-thaw. The Schlenk tube was then placed in the irradiation setup (section C.1.) at a temperature of 60 ºC and irradiated for 24 hours. After cooling to ambient temperature, the solvent was evaporated and the residue was diluted with AcOEt and water, the layers were separated and extracted with AcOEt (2x15 mL) and DCM (1x15 mL). Organic layer was purified by column chromatography (AcOEt/Acetone/Hexane 1:1:1 as eluent): 60 mg orange oil, 44% yield.

^{1}H NMR (500 MHz, $CDCl_3$) δ 10.52 (s, 1H, NH); 10.15 (s, 1H, NH); 5.53 (s, 1H); 3.70 (s, 3H); 3.67 (s, 3H); 3.25-3.18 (m, 1H); 2.87 (dd, *J*=7.4, 16.9 Hz, 1H); 2.74 (dd, *J*=7.4, 16.9 Hz, 1H); 2.68-2.59 (m, 2H).

^{13}C NMR (125 MHz, $CDCl_3$) δ 173.6 (C); 171.8 (C); 164.9 (C); 153.6 (C); 152.6 (C); 101 (CH); 52.7 (CH_3); 52.2 (CH_3); 39.5 (CH); 35.1 (CH_2); 34.2 (CH_2).

IR (thin film) ν 3494, 3113, 2851, 1716, 1664, 1438, 1169, 1098, 1025, 964 cm^{-1}.

HRMS (ESI pos): calculated for $C_{11}H_{14}N_2NaO_6$ ($M+Na^+$): 293.0744, found 293.0741.

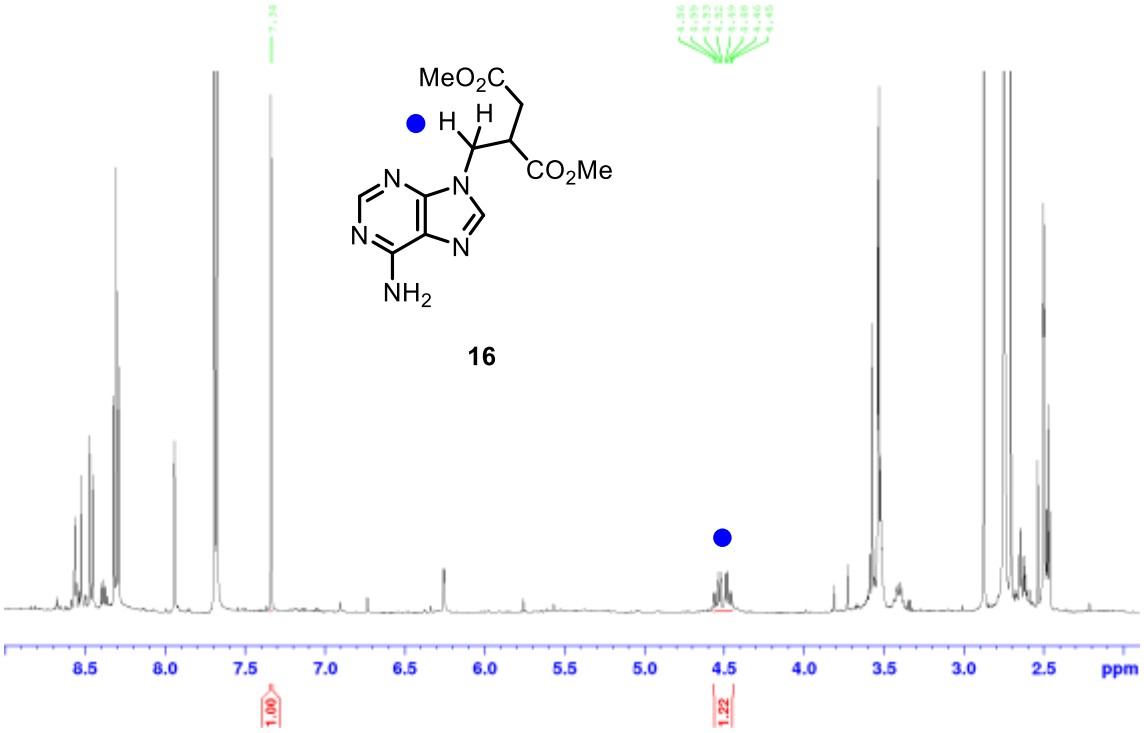


Dimethyl 2-((6-amino-9*H*-purin-9-yl)methyl)succinate (16): In an oven dried Schlenk tube, 9-(chloromethyl)-9*H*-purin-6-amine hydrochloride **15** (83 mg, 0.375 mmol, 1.5 equiv.) was dissolved in DMF (2.5 mL), then catalyst **1c** (7.76 mg, 0.1 equiv.) was added followed by 2,6-lutidine (79 µL, 0.675 mmol, 2.7 equiv.), γ-terpinene (48 µL, 0.3 mmol, 1.2 equiv.), and dimethyl fumarate **3a** (36 mg, 0.25 mmol, 1 equiv.). The resulting yellow mixture was degassed via three cycles of freeze-pump-thaw. The Schlenk tube was then placed in the irradiation setup at a temperature of 60 ºC and irradiated for 24 hours. After cooling to ambient temperature, the solvent was evaporated. Chromatography on silica gel could not remove a residual byproduct from the reaction mixture, therefore an NMR yield is reported (55%) by comparison of the resonance peaks at 4.5 ppm with trichloroethylene (1 equiv.) as internal standard. The residue was purified by column chromatography (AcOEt/Water/MeOH 7:2:1 as eluent) followed by preparative TLC (8% MeOH in DCM as eluent) to obtain an analytical amount of the isolated product **16** as a white solid.

^{1}H NMR (400 MHz, d6-DMSO) δ 8.13 (s, 1H); 8.06 (s, 1H); 7.22 (s, 2H, NH); 4.44 (dd, *J*=6.9, 14.2 Hz, 1H); 4.37 (dd, *J*=6.6, 14.2 Hz, 1H); 3.54 (s, 3H); 3.54 (s, 3H); 3.43-3.36 (m, 1H); 2.60 (d, *J*=6.9 Hz, 2H)

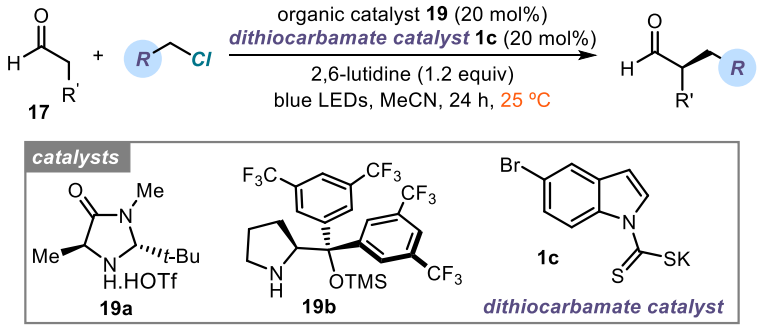
^{13}C NMR (100 MHz, d6-DMSO) δ 172.2 (C); 171.2 (C); 156 (C); 152.5 (CH); 149.7 (C); 141 (CH); 118.5 (C); 52 (CH_3); 51.6 (CH_3); 43.6 (CH_2); 41.2 (CH); 33 (CH_2).

HRMS (ESI pos): calculated for $C_{12}H_{16}N_5O_4$ ($M+H^+$): 294.1197, found 294.1189.



Supplementary Figure 24: NMR yield determination of **16**.

I.6. Asymmetric catalytic α-alkylation of aldehydes

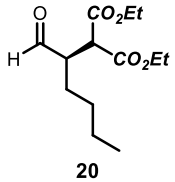


General Procedure

In an oven-dried 10 mL Schlenk tube, the chiral secondary amine catalyst **19** (0.04 mmol, 0.2 equiv.) was dissolved in acetonitrile (0.4 mL, synthesis grade) then freshly distilled aldehyde **17** (0.6 mmol, 3 equiv.) was added followed by freshly distilled 2,6-lutidine (24 µL, 0.24 mmol, 1.2 equiv.), catalyst **1c** (12 mg, 0.04 mmol, 0.2 equiv.) and the alkyl chloride (0.2 mmol, 1 equiv.). The reaction mixture was then degassed via three cycles of Freeze-Pump-Thaw. The Schlenk tube was then placed in the irradiation setup at a temperature of 25 ºC and irradiated for 20 hours. After the end of irradiation, water and ethyl acetate were added and the aqueous layer extracted two more times with ethyl acetate. The combined organic fraction were dried ($MgSO_4$) and concentrated to a yellow oil. Trichloroethylene (18 µL, 0.2 mmol, 1 equiv.) was added and the sample diluted with $CDCl_3$. An aliquot was then analyzed to determine the yield of reaction using ^{1}H NMR analysis.

Note: Since epimerization of the reaction product was commonly observed upon purification on silica gel chromatography (typical loss of 2 to 15 points of e.e. for products that stayed in contact with silica gel for 3 to 30 minutes), the enantiomeric excess of the alkylated aldehyde was determined directly on the crude reaction mixtures or after derivatisation on the crude reaction mixture. Exact conditions are reported for each entry.

An analytical amount of the alkylated aldehyde product was isolated by column chromatography for characterization purposes of unknown products.



(R)-diethyl-2-(1-oxohexan-2-yl)malonate (20)

Prepared according to the general procedure using aminocatalyst **19b** (24 mg, 0.04 mmol, 0.2 equiv.), hexanal (74 µL, 0.6 mmol, 3 equiv.) and diethylchloromalonate (33 µL, 0.2 mmol, 1 equiv.). Yield determined by ^{1}H NMR as 77%. Chromatography on silica gel (gradient from 5% to 10% AcOEt in hexanes as eluent) for characterisation.

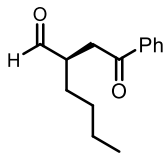
^{1}H NMR (400 MHz, $CDCl_3$) δ 9.78 (d, J=0.5 Hz, 1 H), 4.20-4.11 (m, 4 H); 3.75 (d, J=8.6 Hz, 1 H), 3.15-3.07 (m, 1 H), 1.77-1.68 (m, 1 H), 1.65-1.56 (m, 1 H), 1.43-1.25 (m, 10 H), 0.90 (t, J=7.1 Hz, 3 H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 201.5 (CH), 168.12 (C), 168.0 (C), 61.8 (CH_2), 61.7 (CH_2), 51.8 (CH), 50.2 (CH), 28.6 (CH_2), 26.7 (CH_2), 22.6 (CH_2), 14.0 (CH_3); 13.9 (CH_3), 13.7 (CH_3).

HRMS: calculated for $C_{13}H_{22}NaO_5$ ($M+Na^+$): 281.1359, found 281.1363.

The enantiomeric excess was determined as follows: to an aliquot of the $CDCl_3$ solution prepared as described in the general procedure, methanol and *p*-toluenesulfonyl hydrazide were added and the resulting mixture stirred for 15 minutes. The hydrazone was purified via preparative TLC (6:4 hexanes/AcOEt mixture as eluent, R_f = 0.45) and the enantiomeric excess determined by UPC[2] analysis on an Acquity Trefoil AMY1 column: gradient from 100% CO_2 to 60:40 CO_2/EtOH over 5 minutes, curve 6, flow rate 3.00 mL/min, λ = 230 nm; τ_{Major} = 4.38 min, τ_{Minor} = 4.49 min; e.r.= 87.1/12.9; e.e. = 74%

Absolute configuration assigned by correlation with our previously reported data.[32]



21

(R)-2-(2-oxo-2-phenylethyl)hexanal (21)

Prepared according to the general procedure using aminocatalyst **19b** (24 mg, 0.04 mmol, 0.2 equiv.), hexanal (74 μL, 0.6 mmol, 3 equiv.) and phenacyl chloride (31 mg, 0.2 mmol, 1 equiv.). Yield determined by ^{1}H NMR as 57%. Chromatography on silica gel (gradient from 2% to 5% AcOEt in hexanes as eluent).

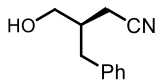
^{1}H NMR (400 MHz, $CDCl_3$) δ 9.85 (d, J=0.9 Hz, 1 H), 8.02-7.97 (m, 2H), 7.62-7.57 (m, 1 H), 7.51-7.46 (m, 2 H), 3.50 (dd, J= 7.7, 17.7 Hz, 1 H), 3.16-3.08 (m, 1 H), 3.05 (dd, J= 4.8, 17.7 Hz, 1 H), 1.87-1.77 (m, 1 H), 1.62-1.51 (m, 1 H), 1,44-1,31 (m, 4 H), 0.97-0.86 (m, 3 H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 203.6 (CH), 198.0 (C), 136.6 (C), 133.2 (CH), 128.6 (2xCH), 128.1 (2xCH), 46.7 (CH), 37.6 (CH_2), 29.2 (CH_2), 28.5 (CH_2), 22.7 (CH_2), 13.8 (CH_3).

HRMS: calculated for $C_{14}H_{18}NaO_2$ ($M+Na^+$): 241.1199, found 241.1197.

The enantiomeric excess of the alkylated aldehyde was determined directly on the crude reaction mixture after isolation of a few milligrams by a short preparative TLC (toluene as eluent, R_f = 0.2, staining purple with vanillin; typical elution time of 2 to 3 minutes). The aldehyde was then analyzed by HPLC analysis on a Daicel Chiralpak IA: 20 ºC, 98:2 hexane/*i*PrOH, flow rate 0.8 mL/min, λ = 254 nm; τ_{Minor} = 13.4 min, τ_{Major} = 15.9 min; e.r.= 89.8/10.2; e.e.= 79%

Absolute configuration assigned by analogy with our previously reported data.[33]



22

(R)-3-benzyl-4-hydroxybutanenitrile (S22)

Prepared according to a modification of the general procedure. In an oven-dried 10 mL Schlenk tube, the chiral secondary amine catalyst **19a** (12 mg, 0.04 mmol, 0.2 equiv.) was dissolved in acetonitrile (0.4 mL, synthesis grade) then freshly distilled hydrocinnamaldehyde (79 μL, 0.6 mmol, 3 equiv.) was added followed by freshly distilled 2,6-lutidine (24 μL, 0.24 mmol, 1.2 equiv.), catalyst **1c** (12 mg, 0.04 mmol, 0.2 equiv.) and chloroacetonitrile (13 μL, 0.2 mmol, 1 equiv.). The reaction mixture was then degassed via three cycles of Freeze-Pump-Thaw. The Schlenk tube was then placed in the irradiation setup at a temperature of 25 ºC and irradiated for 20 hours. After the end of irradiation, the reaction mixture was diluted in CH_2Cl_2 and MeOH and an excess of $NaBH_4$ added. After quenching the mixture with a saturated solution of NH_4Cl, the mixture was extracted with AcOEt, three times. The combined

organic fraction were dried ($MgSO_4$) and concentrated to a yellow oil. Trichloroethylene (18 μL, 0.2 mmol, 1 equiv.) was added and the sample diluted with $CDCl_3$. An aliquot was then analysed to determine the yield of reaction. Yield determined by ^{1}H NMR as 60%. Product. Chromatography on silica gel (gradient from 20% to 30% AcOEt in hexanes as eluent).

^{1}H NMR (300 MHz, $CDCl_3$) δ 7.38-7.26 (m, 2H); 7.29-7.24 (m, 1H); 7.24-7.17 (m, 2H); 3.77 (dd, *J*= 4.6, 10.7 Hz, 1H); 3.64 (dd, *J*= 6.9, 10.7 Hz, 1H); 2.82 (dd, *J*= 6.9, 13.7 Hz, 1H); 2.69 (dd, *J*= 7.0, 13.7 Hz, 1H); 2.49 (dd, *J*= 5.7, 16.9 Hz, 1H); 2.40 (dd, *J*= 6.0, 16.9 Hz, 1H); 2.29-2.13 (m, 1H)

Analytical data in accordance with literature reports.[34]

The alcohol was analysed by to be 90% by UPC^2 on Acquity Trefoil ID3 column: gradient from 100% CO_2 to 60:40 CO_2/iPrOH over 5 minutes, curve 6, flow rate 3.00 mL/min, λ = 230 nm; τ_{Major} = 3.18 min, τ_{Minor} = 3.31 min; e.r.= 95.3/4.6; e.e.= 90%.

Absolute configuration assigned according to reported data.[34]

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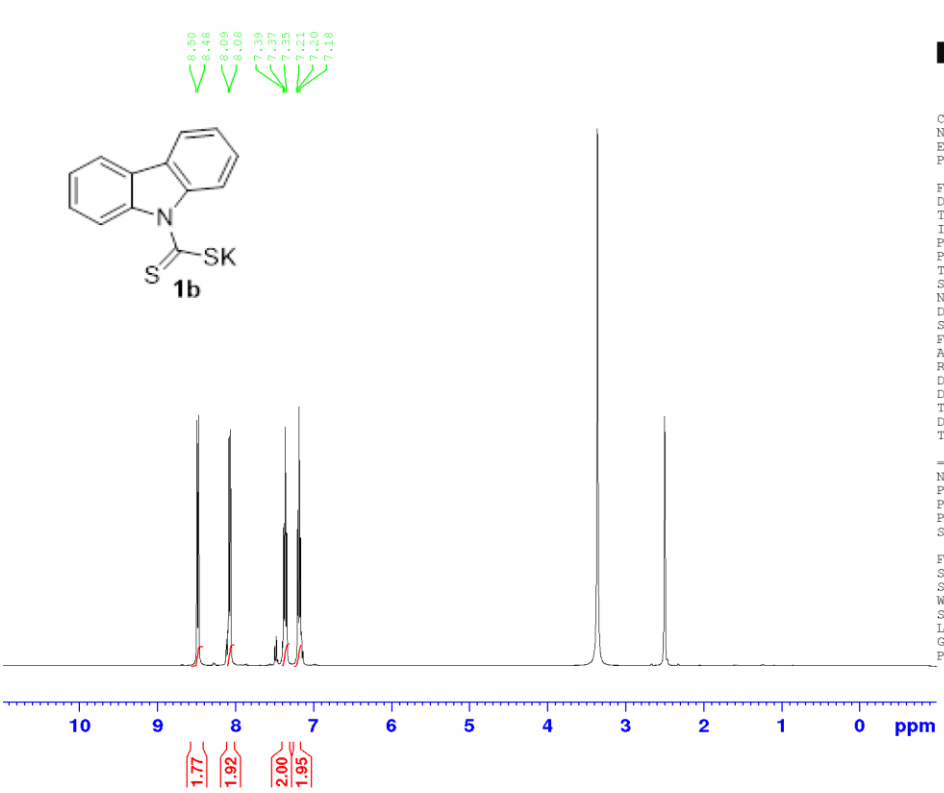
K. NMR Spectra and HPLC traces

Catalysts

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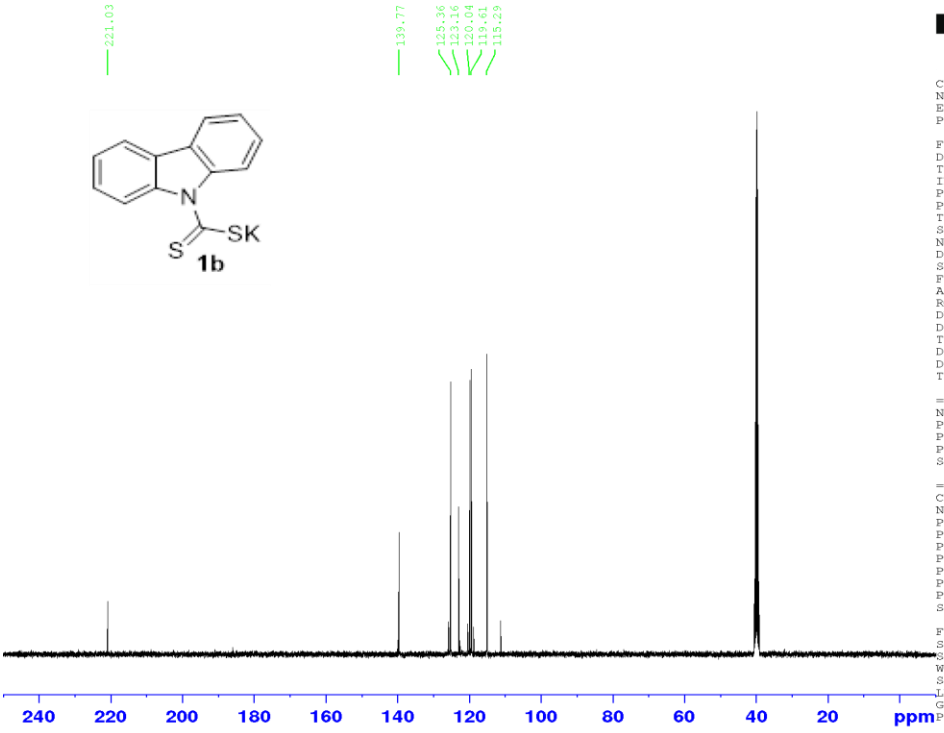


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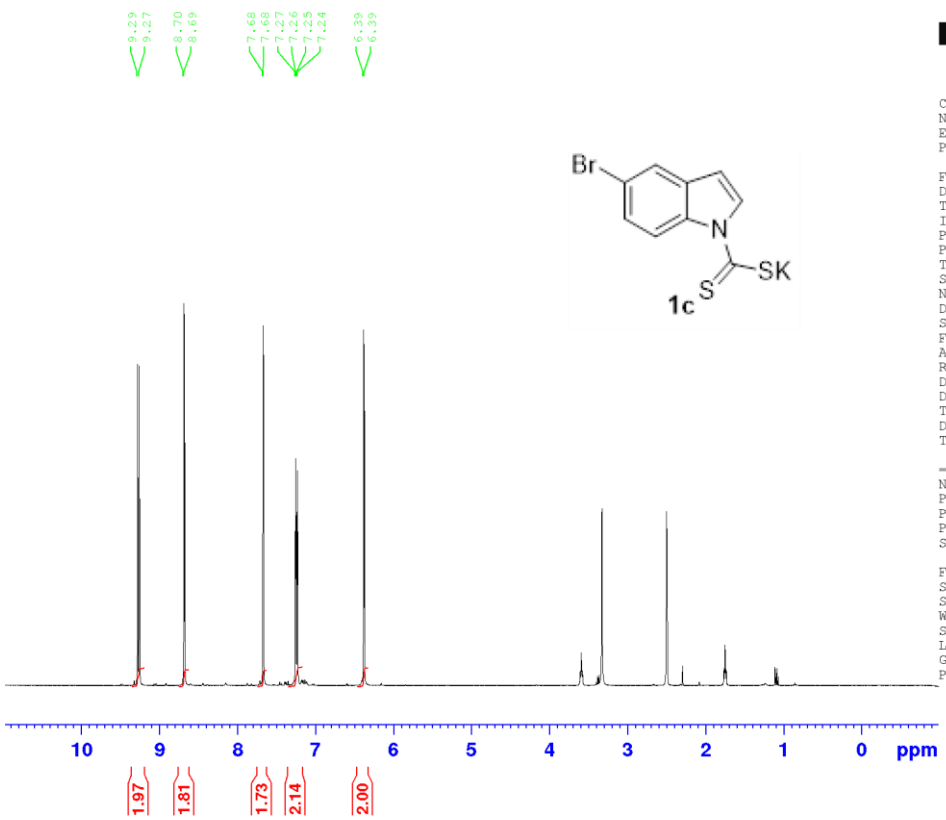


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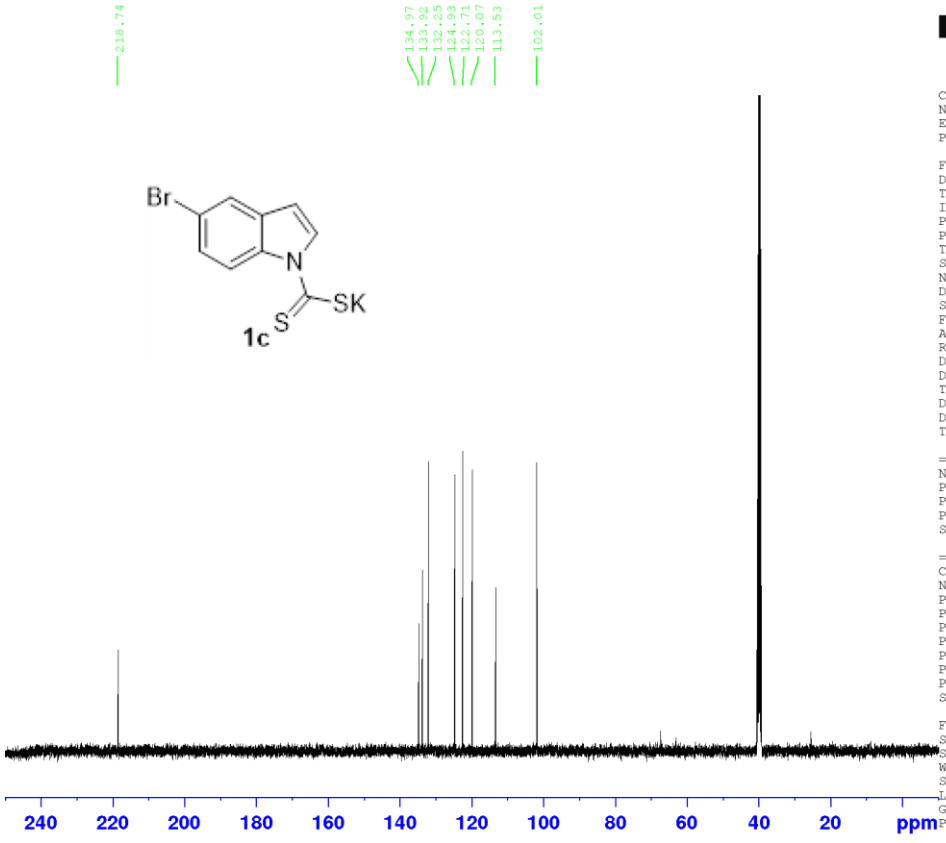


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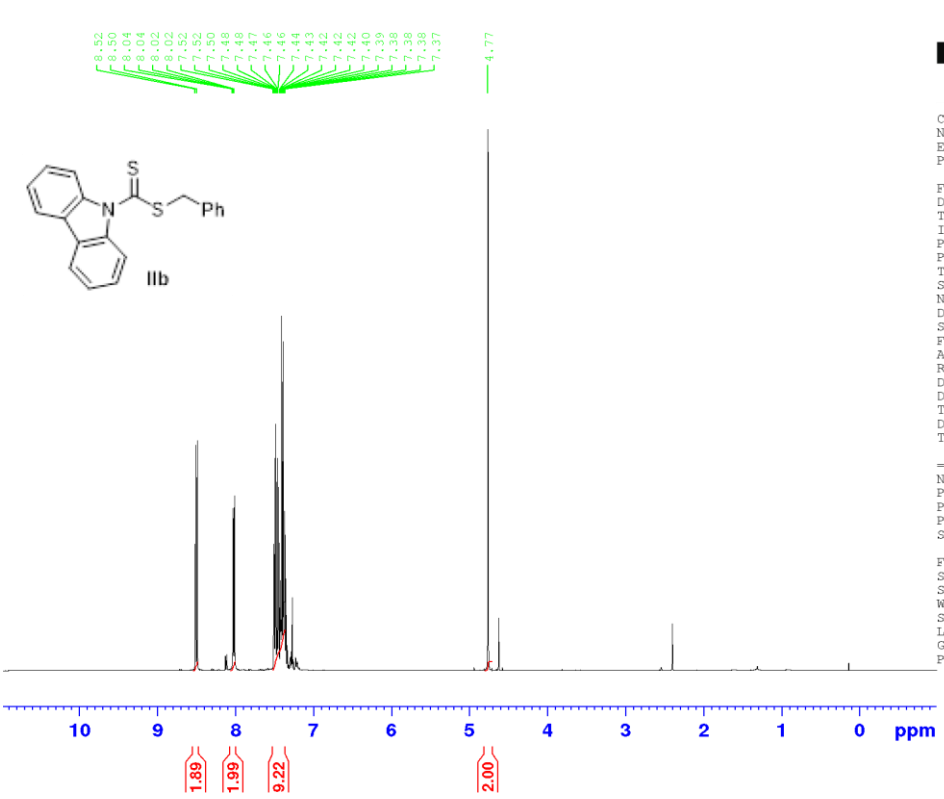
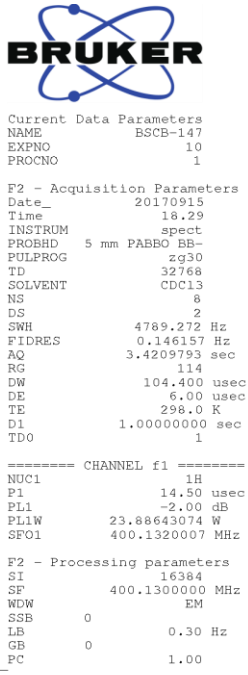
Characterisation of intermediates





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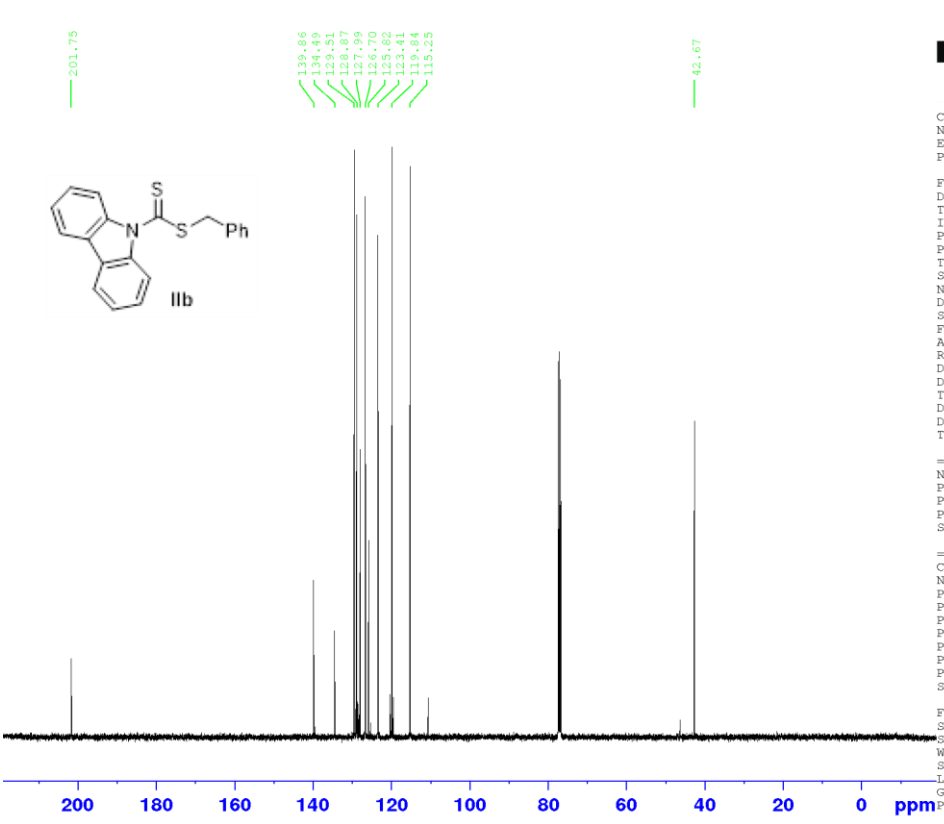

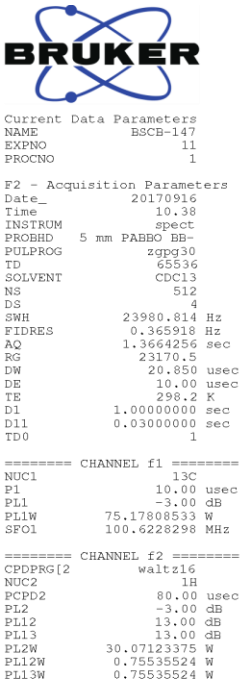

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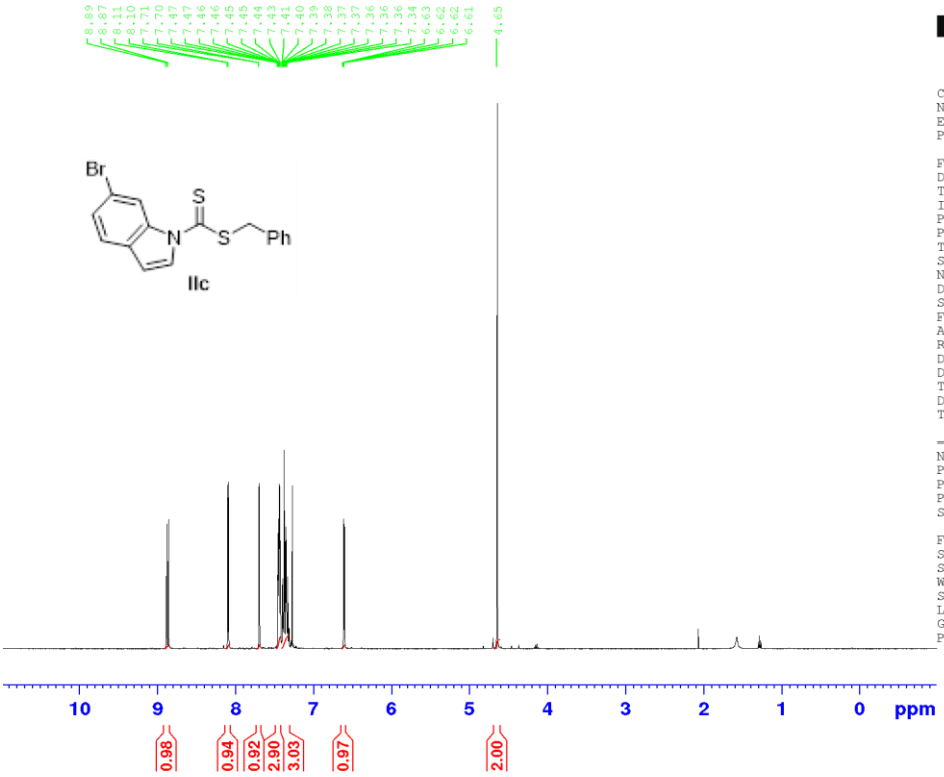

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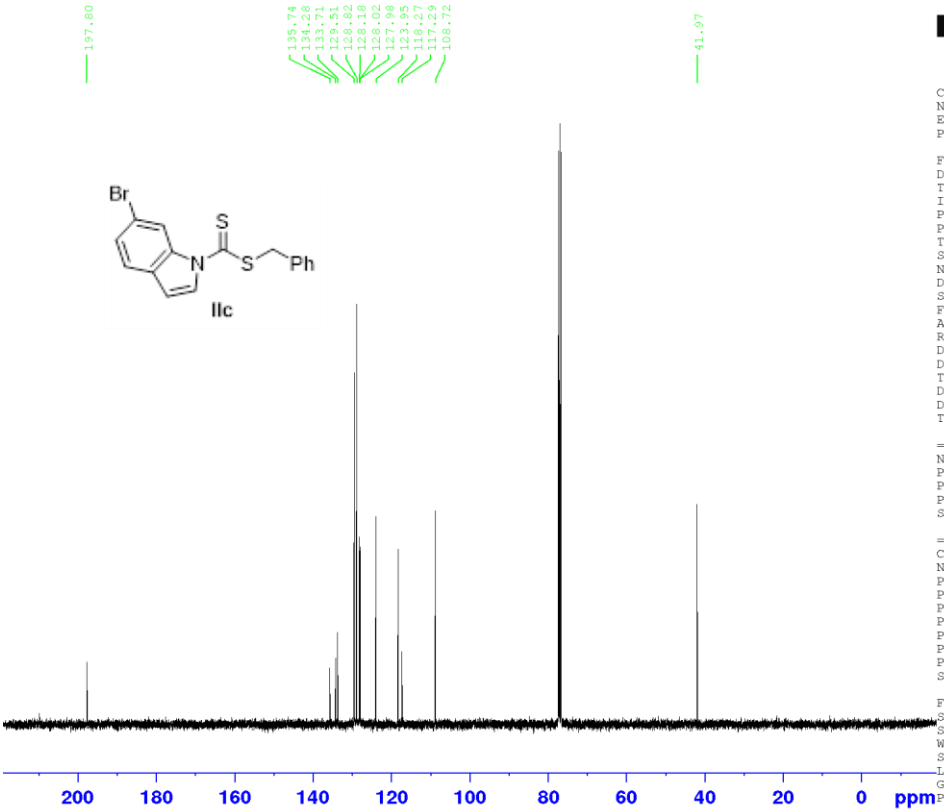

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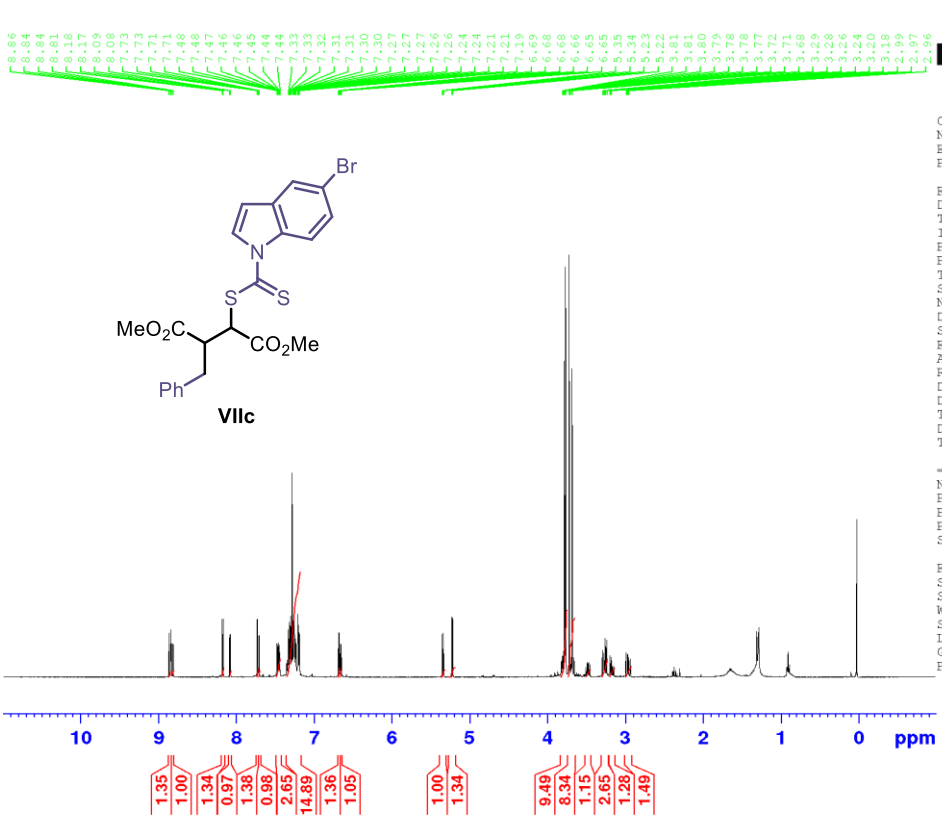


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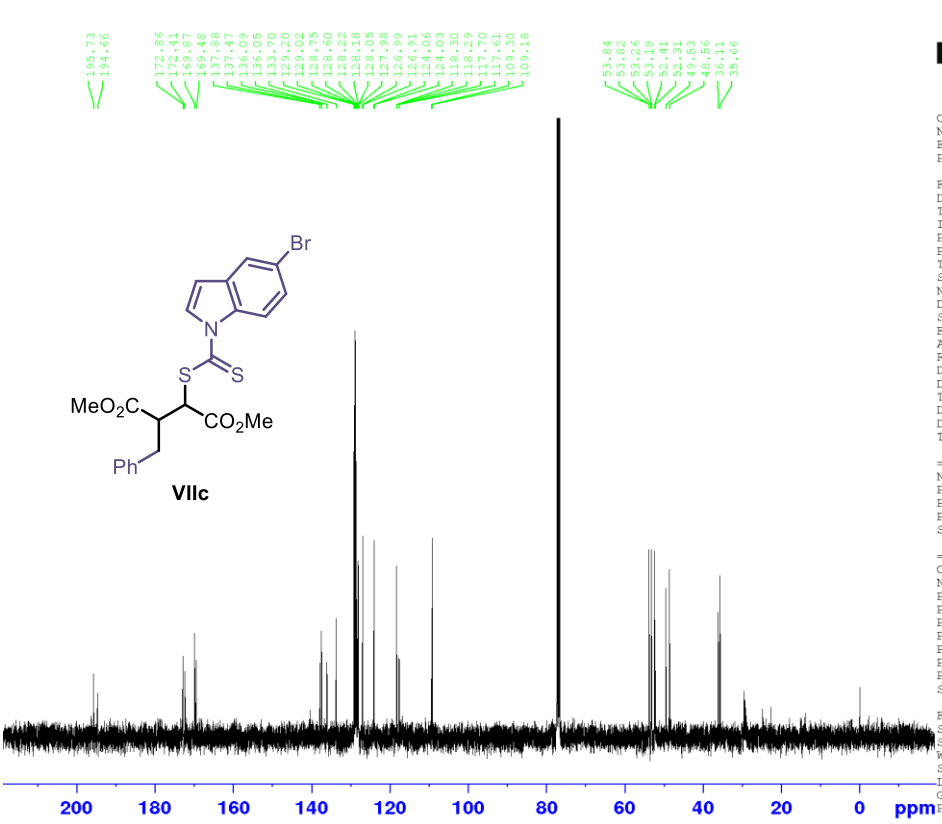
Time                13.13
INSTRUM             spect
PROBHD    5 mm PABBO BB-
PULPROG              zg30
TD                  32768
SOLVENT             CDCl3
NS                      8
DS                      2
SWH              4789.272 Hz
FIDRES           0.146157 Hz
AQ               3.4209793 sec
RG                  228.1
DW                104.400 usec
DE                  6.00 usec
TE                298.0 K
D1              1.00000000 sec
TD0                     1

======== CHANNEL f1 ========
NUC1                   1H
P1                  14.50 usec
PL1                 -2.00 dB
PL1W           23.88643074 W
SFO1           400.1320007 MHz

F2 - Processing parameters
SI                  16384
SF             400.1300000 MHz
WDW                    EM
SSB       0
LB                  0.30 Hz
GB        0
PC                  1.00
```




```
Current Data Parameters
NAME       BSCB-416 char
EXPNO                11
PROCNO                1

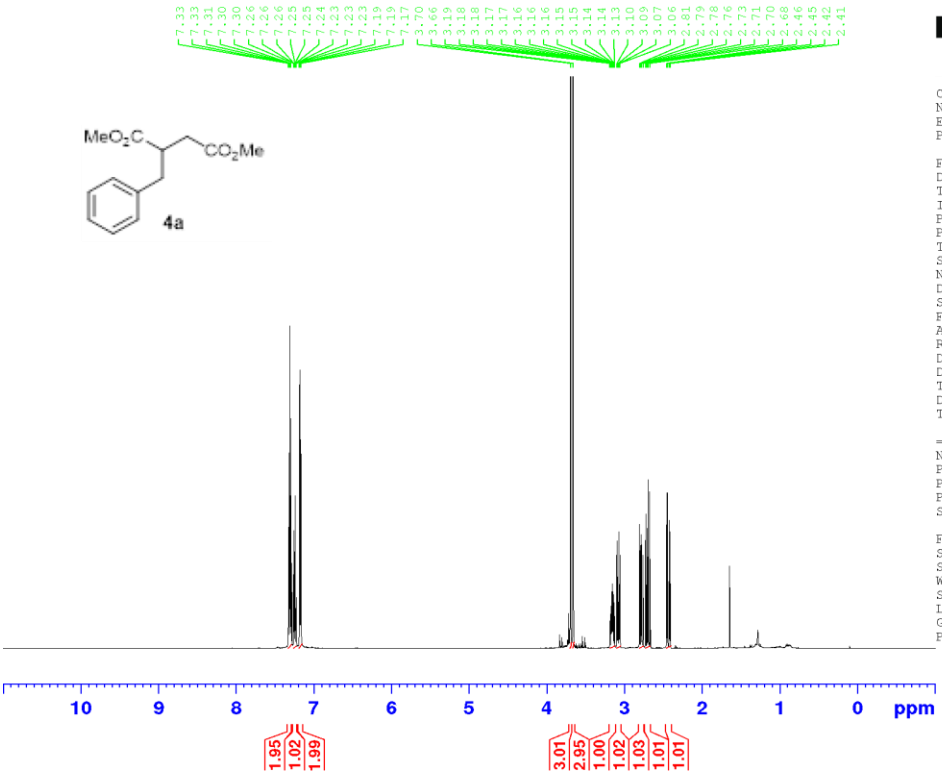
F2 - Acquisition Parameters
Date_             20180807
Time                15.06
INSTRUM             spect
PROBHD    5 mm PABBO BB-
PULPROG            zgpg30
TD                  65536
SOLVENT             CDCl3
NS                    512
DS                      4
SWH             23980.814 Hz
FIDRES           0.365918 Hz
AQ               1.3664256 sec
RG                  23170.5
DW                  20.850 usec
DE                  10.00 usec
TE                298.0 K
D1              1.00000000 sec
D11             0.03000000 sec
TD0                     1

======== CHANNEL f1 ========
NUC1                  13C
P1                  10.00 usec
PL1                 -3.00 dB
PL1W           75.17808533 W
SFO1           100.6228298 MHz

======== CHANNEL f2 ========
CPDPRG[2          waltz16
NUC2                   1H
PCPD2               80.00 usec
PL2                 -3.00 dB
PL12                13.00 dB
PL13                13.00 dB
PL2W           30.07123375 W
PL12W           0.75535524 W
PL13W           0.75535524 W
SFO2           400.1316005 MHz

F2 - Processing parameters
SI                  32768
SF             100.6127690 MHz
WDW                    EM
SSB       0
LB                  1.00 Hz
GB        0
PC                  1.40
```

Giese Addition

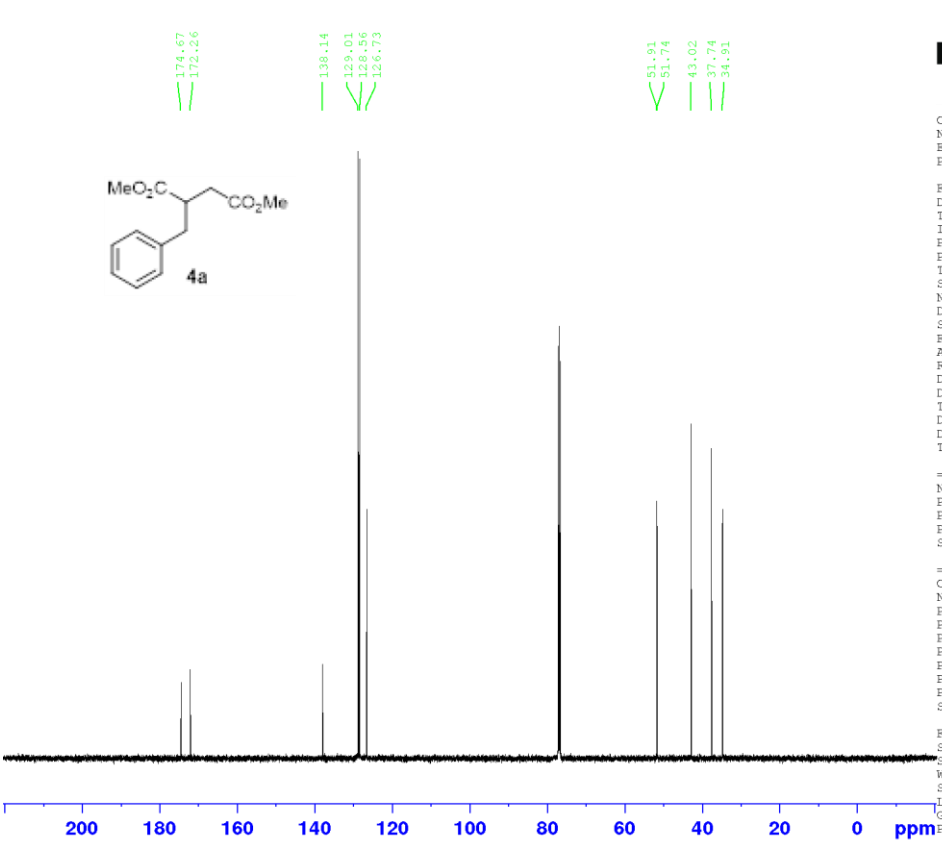



```
Current Data Parameters
NAME        BSCB-065 col
EXPNO                 10
PROCNO                 1

F2 - Acquisition Parameters
Date_           20170629
Time               23.08
INSTRUM            spect
PROBHD    5 mm PABBO BB-
PULPROG            zg30
TD                32768
SOLVENT           CDCl3
NS                   32
DS                    2
SWH            6009.615 Hz
FIDRES        0.183399 Hz
AQ            2.7262976 sec
RG                  101
DW               83.200 usec
DE               10.00 usec
TE                298.0 K
D1           1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                1H
P1               15.30 usec
PL1               3.00 dB
PL1W        12.58925438 W
SFO1       500.1325007 MHz

F2 - Processing parameters
SI                 16384
SF          500.1300000 MHz
WDW                   EM
SSB       0
LB                0.30 Hz
GB        0
PC                1.00
```



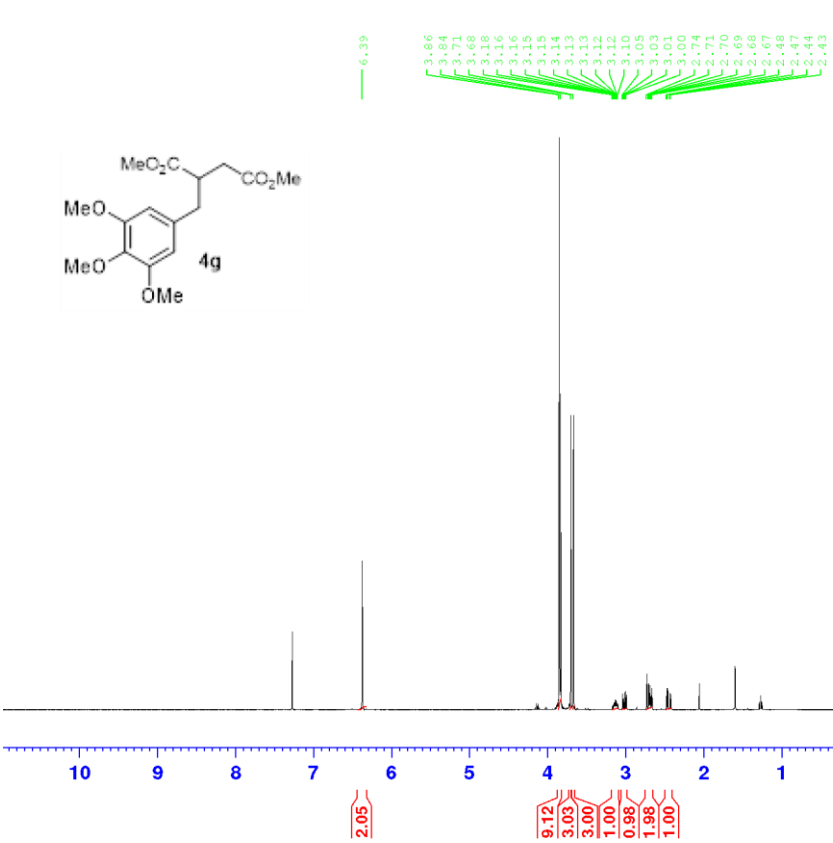

```
Current Data Parameters
NAME        BSCB-065 col
EXPNO                 11
PROCNO                 1

F2 - Acquisition Parameters
Date_           20170629
Time               23.27
INSTRUM            spect
PROBHD    5 mm PABBO BB-
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                  512
DS                    4
SWH           30303.031 Hz
FIDRES        0.462388 Hz
AQ            1.0813440 sec
RG                16400
DW               16.500 usec
DE               10.00 usec
TE                299.2 K
D1           1.00000000 sec
D11          0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1               13C
P1               10.30 usec
PL1          0 dB
PL1W        75.35659027 W
SFO1       125.7703648 MHz

======== CHANNEL f2 ========
CPDPRG[2         waltz16
NUC2                1H
PCPD2            80.00 usec
PL2               3.00 dB
PL12             17.54 dB
PL13             17.54 dB
PL2W        12.58925438 W
PL12W        0.44258827 W
PL13W        0.44258827 W
SFO2       500.1320005 MHz

F2 - Processing parameters
SI                 32768
SF          125.7577890 MHz
WDW                   EM
SSB       0
LB                1.00 Hz
GB        0
PC                1.40
```

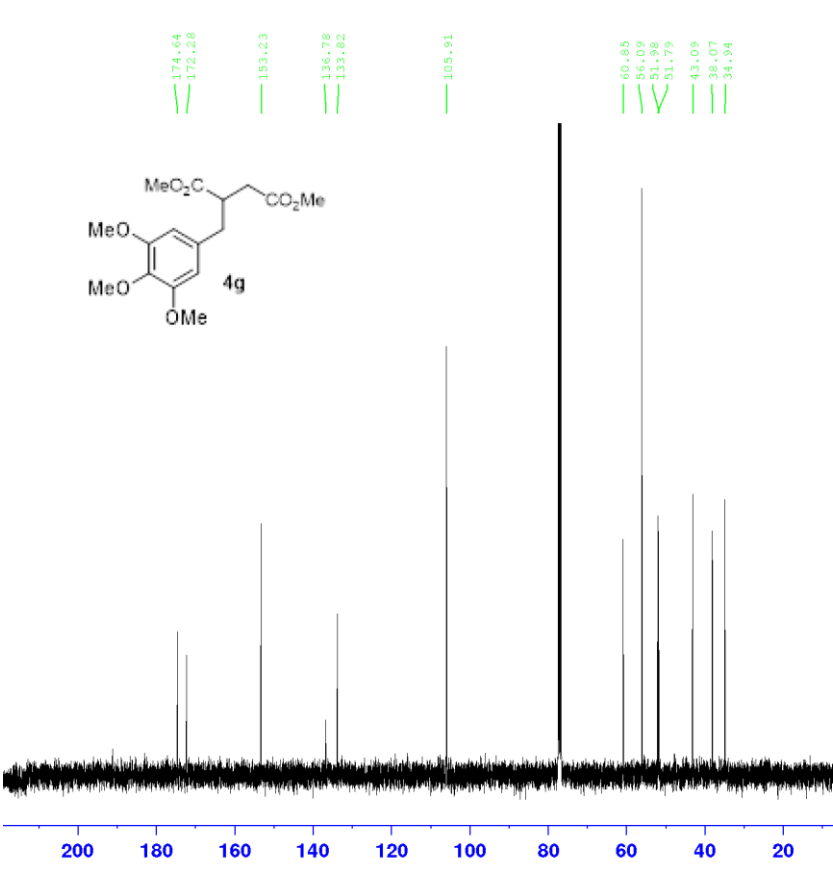


```
Current Data Parameters
NAME       BSCB-133 char
EXPNO                10
PROCNO                1

F2 - Acquisition Parameters
Date_          20170908
Time               8.15
INSTRUM           spect
PROBHD   5 mm PABBO BB-
PULPROG            zg30
TD                32768
SOLVENT           CDCl3
NS                    8
DS                    2
SWH            4789.272 Hz
FIDRES        0.146157 Hz
AQ            3.4209793 sec
RG                  256
DW              104.400 usec
DE                6.00 usec
TE                298.0 K
D1           1.00000000 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                 1H
P1                14.50 usec
PL1               -2.00 dB
PL1W        23.88643074 W
SFO1        400.1320007 MHz

F2 - Processing parameters
SI                16384
SF          400.1300000 MHz
WDW                  EM
SSB       0
LB                0.30 Hz
GB        0
PC                1.00
```



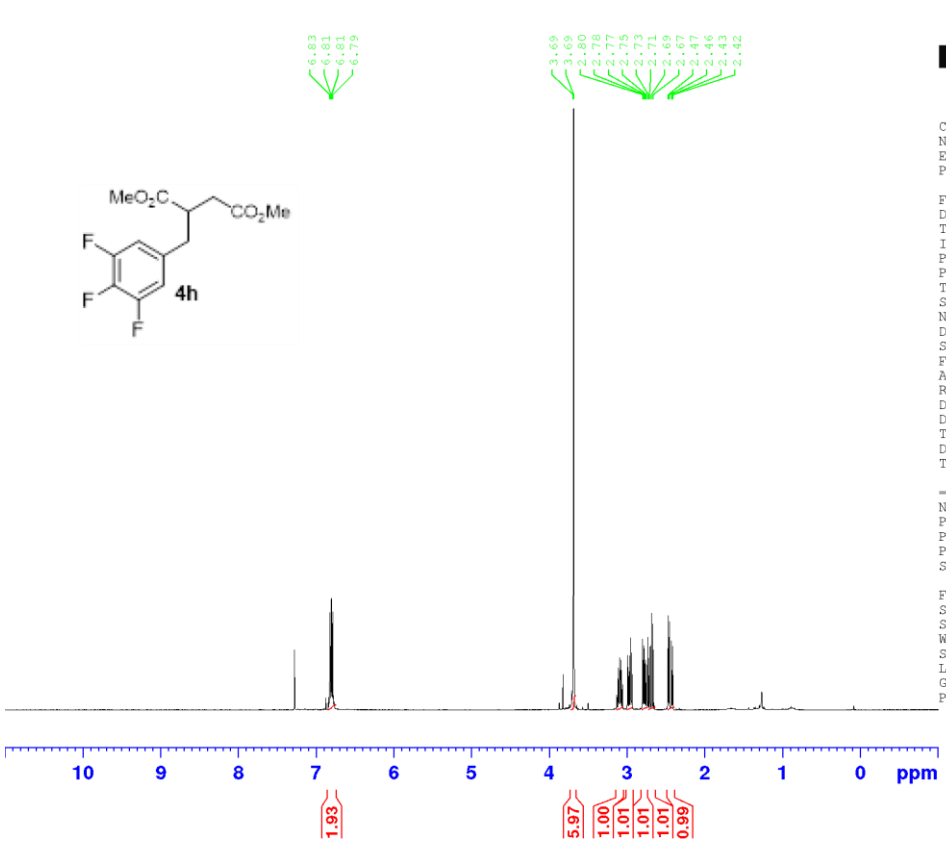
```
Current Data Parameters
NAME       BSCB-133 char
EXPNO                11
PROCNO                1

F2 - Acquisition Parameters
Date_          20170908
Time               8.44
INSTRUM           spect
PROBHD   5 mm PABBO BB-
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                  512
DS                    4
SWH           23980.814 Hz
FIDRES        0.365918 Hz
AQ            1.3664256 sec
RG                32768
DW              20.850 usec
DE                10.00 usec
TE                298.0 K
D1           1.00000000 sec
D11          0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                13C
P1                10.00 usec
PL1               -3.00 dB
PL1W        75.17808533 W
SFO1        100.6228298 MHz

======== CHANNEL f2 ========
CPDPRG[2         waltz16
NUC2                 1H
PCPD2             80.00 usec
PL2               -3.00 dB
PL12              13.00 dB
PL13              13.00 dB
PL2W        30.07123375 W
PL12W        0.75535524 W
PL13W        0.75535524 W
SFO2        400.1316005 MHz

F2 - Processing parameters
SI                32768
SF          100.6127690 MHz
WDW                  EM
SSB       0
LB                1.00 Hz
GB        0
PC                1.40
```

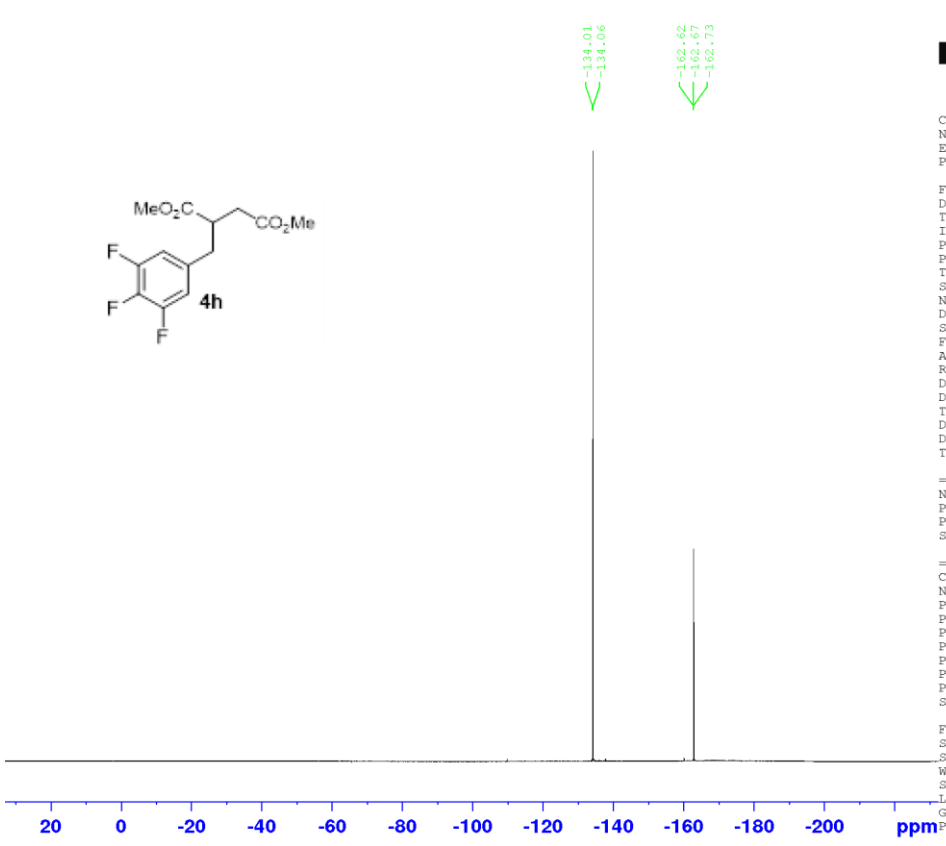



```
Current Data Parameters
NAME        BSCB-143 col
EXPNO                10
PROCNO                1

F2 - Acquisition Parameters
Date_          20170914
Time              17.07
INSTRUM           spect
PROBHD   5 mm PABBO BB-
PULPROG            zg30
TD                32768
SOLVENT           CDCl3
NS                    8
DS                    2
SWH            4807.692 Hz
FIDRES        0.146719 Hz
AQ            3.4078720 sec
RG                  128
DW              104.000 usec
DE               10.00 usec
TE               298.0 K
D1            1.00000000 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                 1H
P1                13.00 usec
PL1               -3.00 dB
PL1W        30.07123375 W
SFO1        400.1620008 MHz

F2 - Processing parameters
SI                16384
SF          400.1600000 MHz
WDW                  EM
SSB       0
LB                0.30 Hz
GB        0
PC                1.00
```



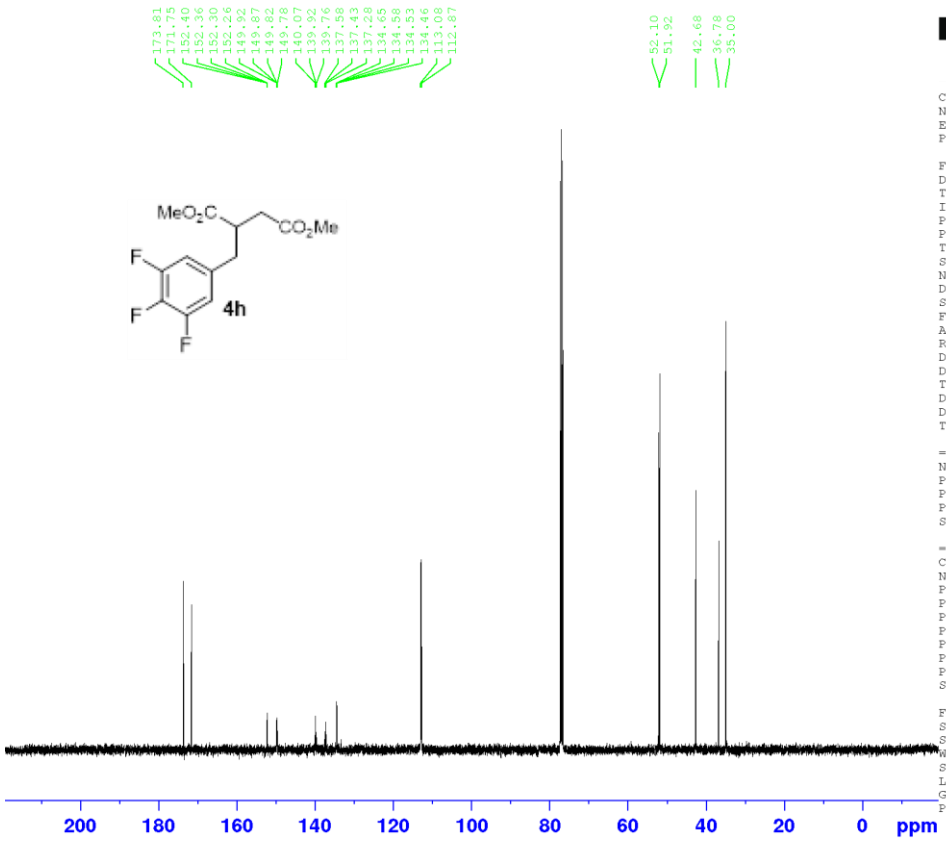

```
Current Data Parameters
NAME        BSCB-143 col
EXPNO                11
PROCNO                1

F2 - Acquisition Parameters
Date_          20170914
Time              17.11
INSTRUM           spect
PROBHD   5 mm PABBO BB-
PULPROG          zgpg30
TD               135168
SOLVENT           CDCl3
NS                    8
DS                    2
SWH          100000.000 Hz
FIDRES         0.739820 Hz
AQ             0.6758400 sec
RG                1290.2
DW                5.000 usec
DE               10.00 usec
TE               298.3 K
D1            5.00000000 sec
D11           0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                19F
P1                14.50 usec
PL1               -3.00 dB
SFO1        376.4889413 MHz

======== CHANNEL f2 ========
CPDPRG[2         waltz16
NUC2                 1H
PCPD2             80.00 usec
PL2               -3.00 dB
PL12              12.78 dB
PL13              12.78 dB
PL2W        30.07123375 W
PL12W        0.79460496 W
PL13W        0.79460496 W
SFO2        400.1616006 MHz

F2 - Processing parameters
SI                65536
SF          376.5264850 MHz
WDW                  EM
SSB       0
LB             1.00 Hz
GB        0
PC                1.40
```



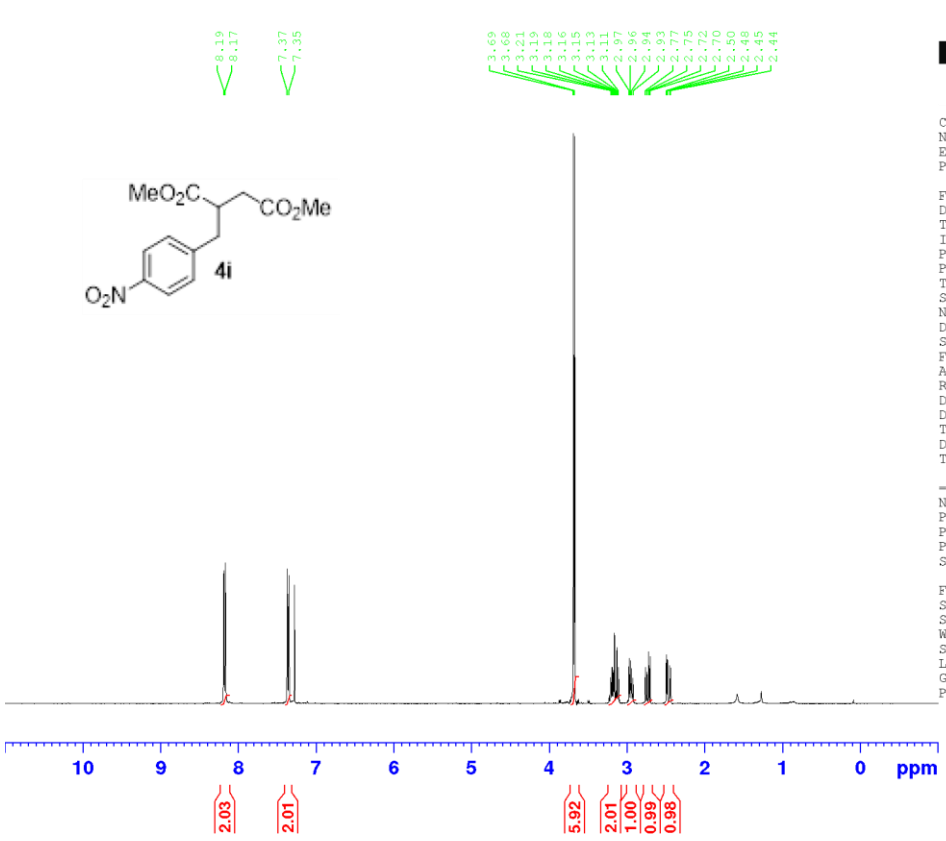
Current Data Parameters
NAME BSCB-143 col
EXPNO 12
PROCNO 1

F2 - Acquisition Parameters
Date_ 20170915
Time 0.08
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 512
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631488 sec
RG 26008
DW 20.800 usec
DE 10.00 usec
TE 298.1 K
D1 1.00000000 sec
D11 0.03000000 sec
TD0 1

======== CHANNEL f1 ========
NUC1 13C
P1 9.25 usec
PL1 -3.00 dB
PL1W 75.17808533 W
SFO1 100.6303736 MHz

======== CHANNEL f2 ========
CPDPRG[2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -3.00 dB
PL12 12.78 dB
PL13 12.78 dB
PL2W 30.07123375 W
PL12W 0.79460496 W
PL13W 0.79460496 W
SFO2 400.1616006 MHz

F2 - Processing parameters
SI 32768
SF 100.6203120 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

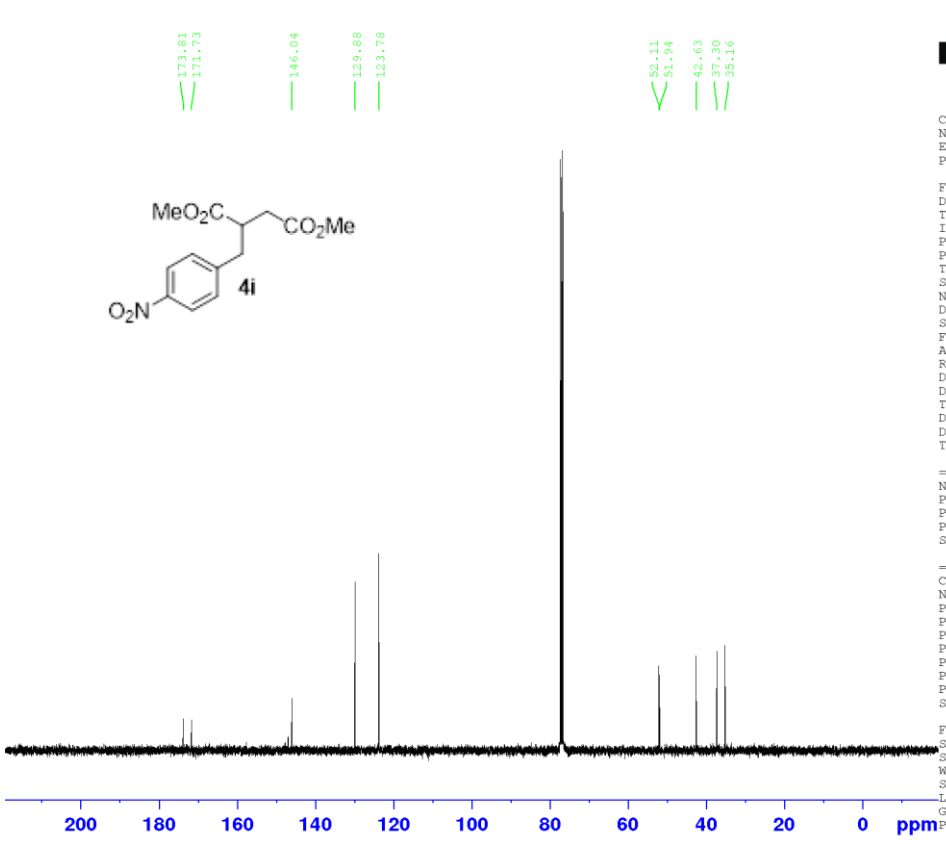


Current Data Parameters
NAME BSCB-207 char
EXPNO 10
PROCNO 1

F2 - Acquisition Parameters
Date_ 20171114
Time 20.04
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 8
DS 2
SWH 4807.692 Hz
FIDRES 0.146719 Hz
AQ 3.4078720 sec
RG 256
DW 104.000 usec
DE 10.00 usec
TE 297.7 K
D1 1.00000000 sec
TD0 1

======== CHANNEL f1 ========
NUC1 1H
P1 13.00 usec
PL1 -3.00 dB
PL1W 30.07123375 W
SFO1 400.1620008 MHz

F2 - Processing parameters
SI 16384
SF 400.1600000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



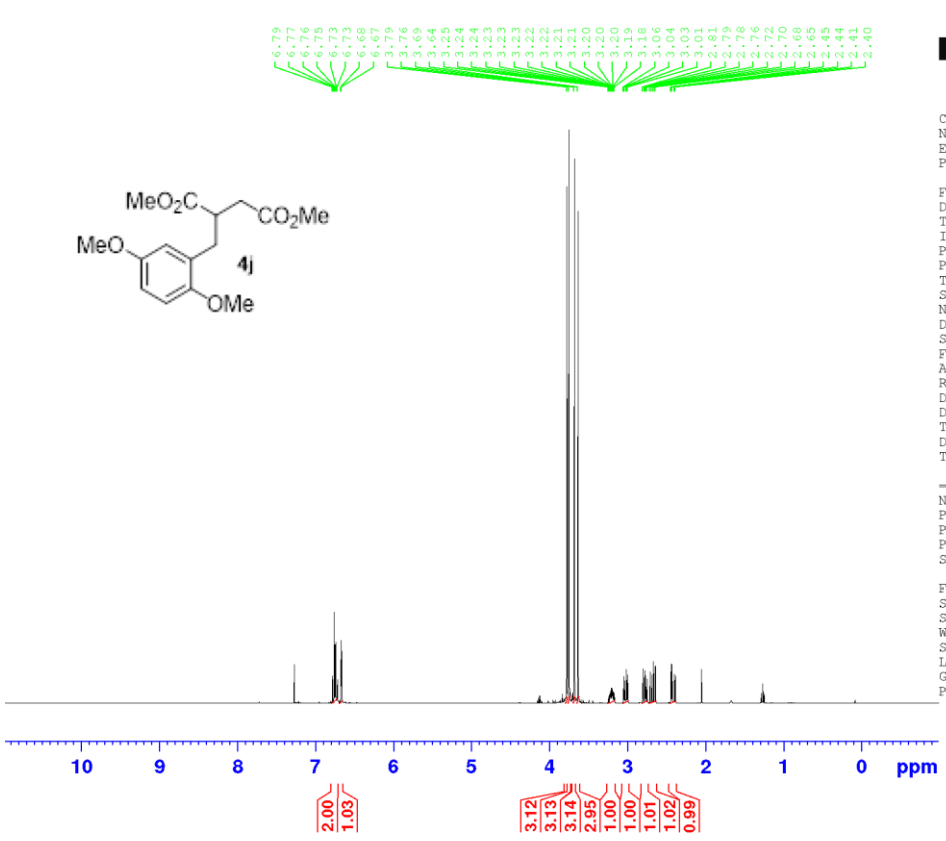

```
Current Data Parameters
NAME        BSCB-207 char
EXPNO                 11
PROCNO                 1

F2 - Acquisition Parameters
Date_           20171114
Time               20.25
INSTRUM            spect
PROBHD    5 mm PABBO BB-
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                  512
DS                    4
SWH           24038.461 Hz
FIDRES         0.366798 Hz
AQ             1.3631488 sec
RG                32768
DW               20.800 usec
DE               10.00 usec
TE                298.3 K
D1            1.00000000 sec
D11           0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  13C
P1                   9.25 usec
PL1                 -3.00 dB
PL1W          75.17808533 W
SFO1          100.6303736 MHz

======== CHANNEL f2 ========
CPDPRG[2          waltz16
NUC2                  1H
PCPD2              80.00 usec
PL2                -3.00 dB
PL12               12.78 dB
PL13               12.78 dB
PL2W          30.07123375 W
PL12W          0.79460496 W
PL13W          0.79460496 W
SFO2          400.1616006 MHz

F2 - Processing parameters
SI                 32768
SF            100.6203120 MHz
WDW                   EM
SSB       0
LB                 1.00 Hz
GB        0
PC                 1.40
```

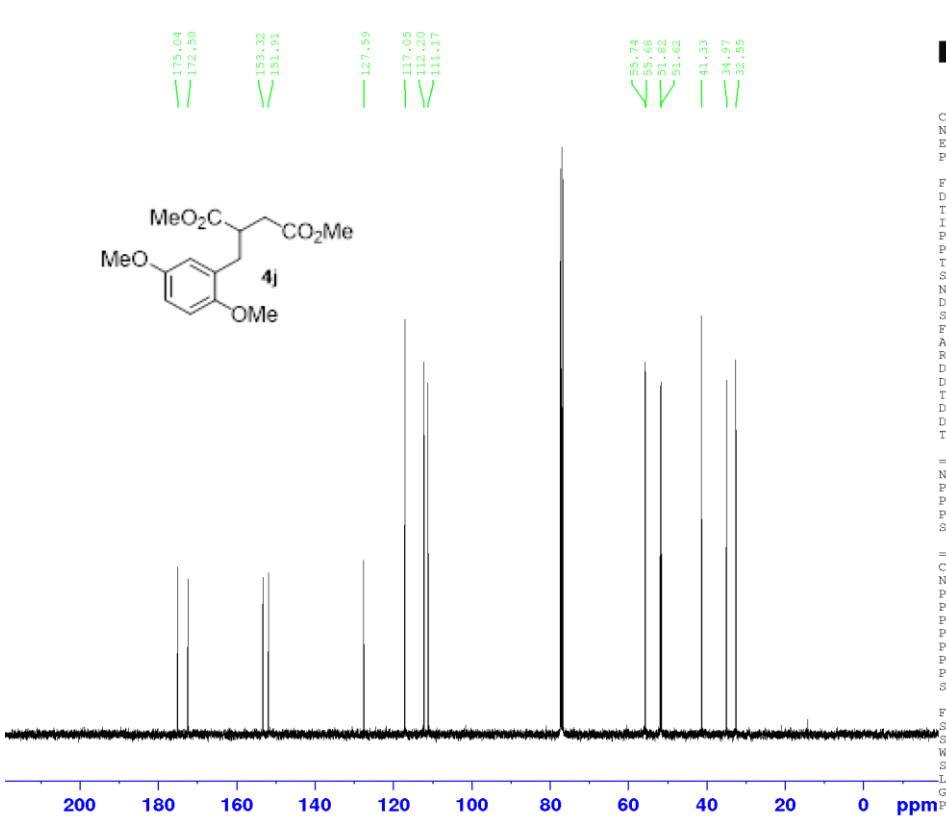



```
Current Data Parameters
NAME        BSCB-377 col
EXPNO                 10
PROCNO                 1

F2 - Acquisition Parameters
Date_           20180329
Time               18.00
INSTRUM            spect
PROBHD    5 mm PABBO BB-
PULPROG             zg30
TD                32768
SOLVENT           CDCl3
NS                    8
DS                    2
SWH            4789.272 Hz
FIDRES         0.146157 Hz
AQ             3.4209793 sec
RG                  114
DW               104.400 usec
DE                 6.00 usec
TE                298.0 K
D1            1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                14.50 usec
PL1               -2.00 dB
PL1W          23.88643074 W
SFO1          400.1320007 MHz

F2 - Processing parameters
SI                 16384
SF            400.1300000 MHz
WDW                   EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```



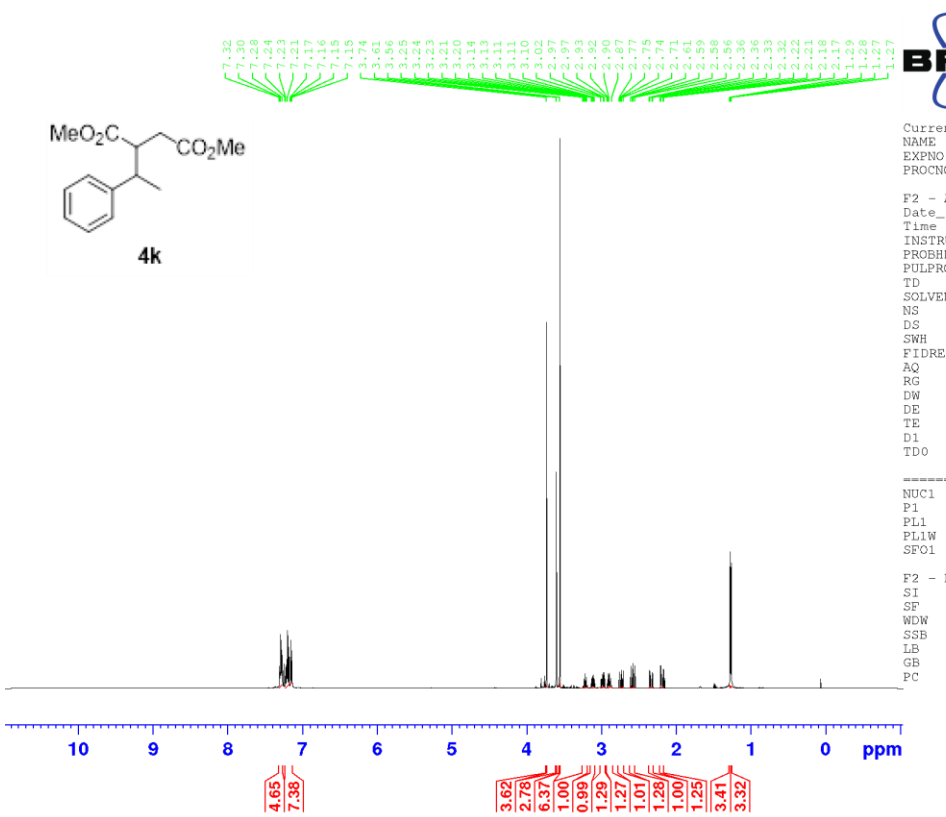

```
Current Data Parameters
NAME        BSCB-377 col
EXPNO                11
PROCNO                1

F2 - Acquisition Parameters
Date_           20180329
Time               20.15
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                  512
DS                    4
SWH           23980.814 Hz
FIDRES         0.365918 Hz
AQ             1.3664256 sec
RG                16384
DW               20.850 usec
DE               10.00 usec
TE               298.6 K
D1            1.00000000 sec
D11           0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                 13C
P1                 10.00 usec
PL1                -3.00 dB
PL1W         75.17808533 W
SFO1        100.6228298 MHz

======== CHANNEL f2 ========
CPDPRG[2          waltz16
NUC2                 1H
PCPD2              80.00 usec
PL2                -3.00 dB
PL12               13.00 dB
PL13               13.00 dB
PL2W         30.07123375 W
PL12W         0.75535524 W
PL13W         0.75535524 W
SFO2        400.1316005 MHz

F2 - Processing parameters
SI                 32768
SF          100.6127690 MHz
WDW                   EM
SSB       0
LB                 1.00 Hz
GB        0
PC                 1.40
```

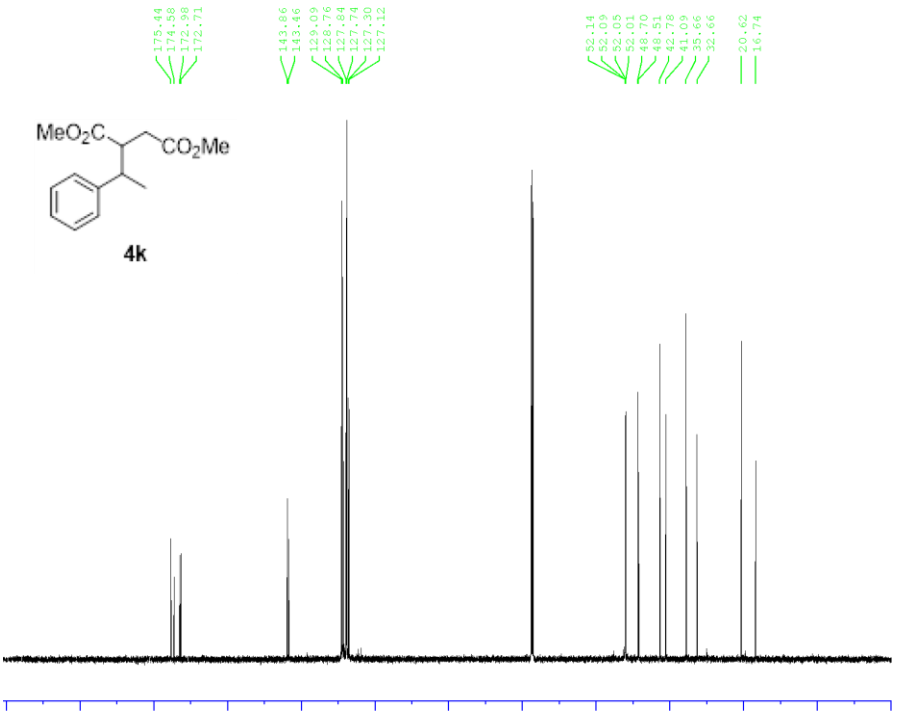



```
Current Data Parameters
NAME         mah-147-1H
EXPNO                10
PROCNO                1

F2 - Acquisition Parameters
Date_           20180313
Time               22.38
INSTRUM            spect
PROBHD   5 mm PATBO BB-
PULPROG             zg30
TD                32768
SOLVENT           CDCl3
NS                  128
DS                    2
SWH            6009.615 Hz
FIDRES         0.183399 Hz
AQ             2.7262976 sec
RG                 71.8
DW               83.200 usec
DE               10.00 usec
TE               298.1 K
D1            1.00000000 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                 1H
P1                 14.90 usec
PL1                -3.00 dB
PL1W         50.11872482 W
SFO1        500.1325007 MHz

F2 - Processing parameters
SI                 16384
SF          500.1300132 MHz
WDW                   EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```



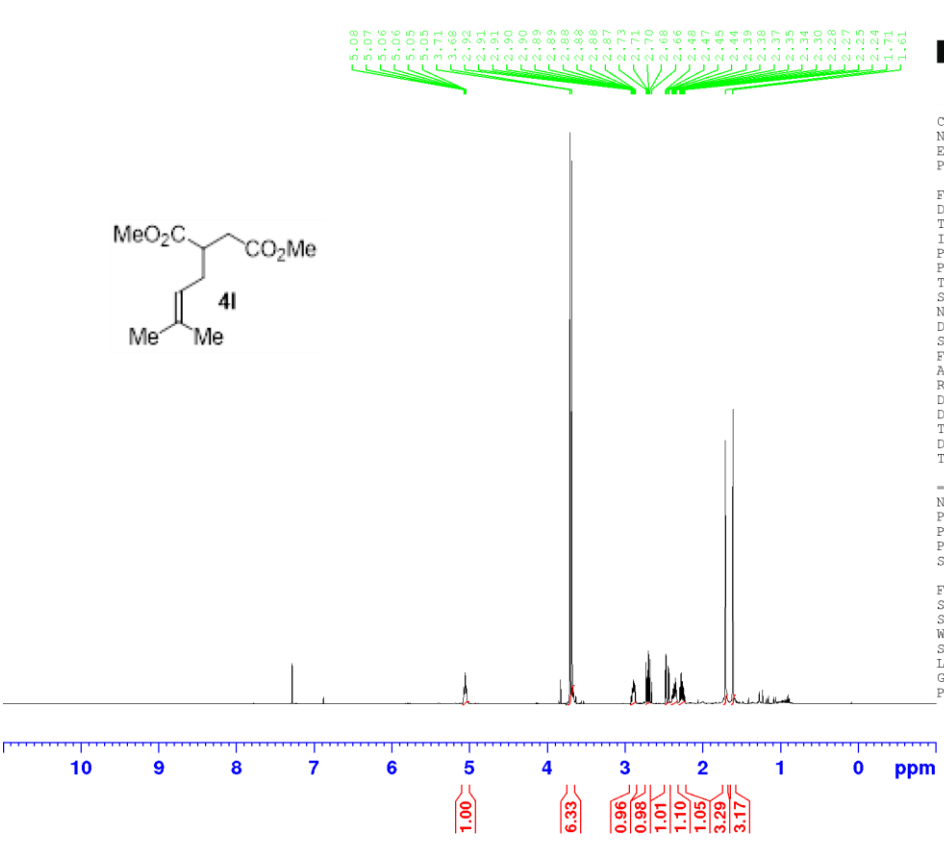
Current Data Parameters
NAME mah-147-13C
EXPNO 10
PROCNO 1

F2 - Acquisition Parameters
Date_ 20180313
Time 22.56
INSTRUM spect
PROBHD 5 mm PATBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 512
DS 4
SWH 30303.031 Hz
FIDRES 0.462388 Hz
AQ 1.0813440 sec
RG 13000
DW 16.500 usec
DE 10.00 usec
TE 299.6 K
D1 1.00000000 sec
D11 0.03000000 sec
TD0 1

======== CHANNEL f1 ========
NUC1 13C
P1 9.75 usec
PL1 -1.00 dB
PL1W 94.86833191 W
SFO1 125.7703648 MHz

======== CHANNEL f2 ========
CPDPRG[2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -3.00 dB
PL12 11.96 dB
PL13 11.96 dB
PL2W 50.11872482 W
PL12W 1.59955800 W
PL13W 1.59955800 W
SFO2 500.1320005 MHz

F2 - Processing parameters
SI 32768
SF 125.7577441 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

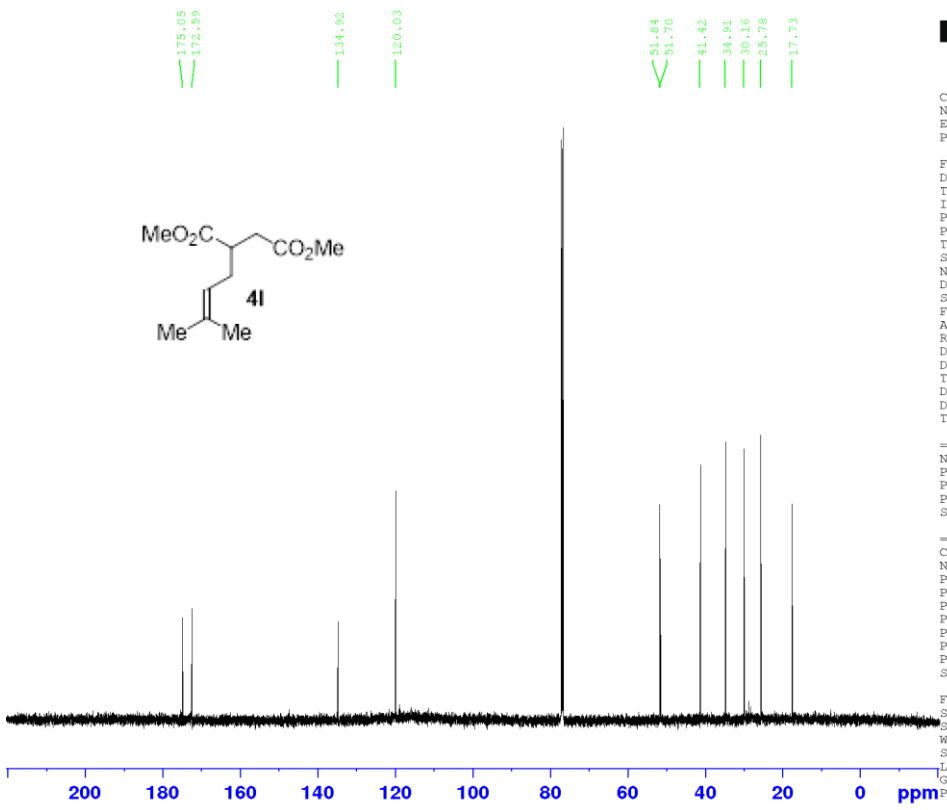


Current Data Parameters
NAME BSCB-181-01 col
EXPNO 10
PROCNO 1

F2 - Acquisition Parameters
Date_ 20171013
Time 19.56
INSTRUM spect
PROBHD 5 mm PABBI 1H/
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 8
DS 2
SWH 6009.615 Hz
FIDRES 0.183399 Hz
AQ 2.7262976 sec
RG 50.8
DW 83.200 usec
DE 10.00 usec
TE 300.0 K
D1 1.00000000 sec
TD0 1

======== CHANNEL f1 ========
NUC1 1H
P1 6.70 usec
PL1 1.00 dB
PL1W 19.95262337 W
SFO1 500.1325007 MHz

F2 - Processing parameters
SI 16384
SF 500.1300000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



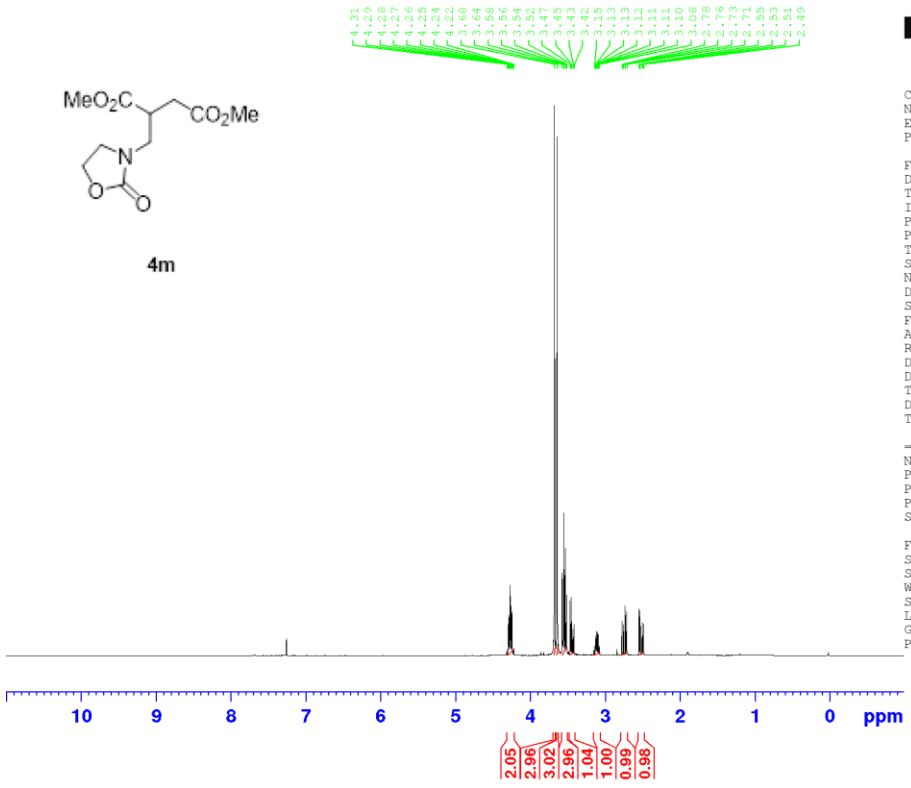

```
Current Data Parameters
NAME      BSCB-181-01 col
EXPNO                 11
PROCNO                 1

F2 - Acquisition Parameters
Date_           20171014
Time                2.17
INSTRUM            spect
PROBHD   5 mm PABBI 1H/
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                  512
DS                    4
SWH           30303.031 Hz
FIDRES         0.462388 Hz
AQ             1.0813440 sec
RG                14600
DW               16.500 usec
DE               10.00 usec
TE               298.5 K
D1            1.00000000 sec
D11           0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                 13C
P1                 18.50 usec
PL1                -0.50 dB
PL1W         84.55149078 W
SFO1        125.7703648 MHz

======== CHANNEL f2 ========
CPDPRG[2          waltz16
NUC2                 1H
PCPD2              80.00 usec
PL2                 1.00 dB
PL12               22.54 dB
PL13               22.54 dB
PL2W         19.95262337 W
PL12W         0.13995871 W
PL13W         0.13995871 W
SFO2        500.1320005 MHz

F2 - Processing parameters
SI                 32768
SF          125.7577890 MHz
WDW                   EM
SSB       0
LB                 1.00 Hz
GB        0
PC                 1.40
```

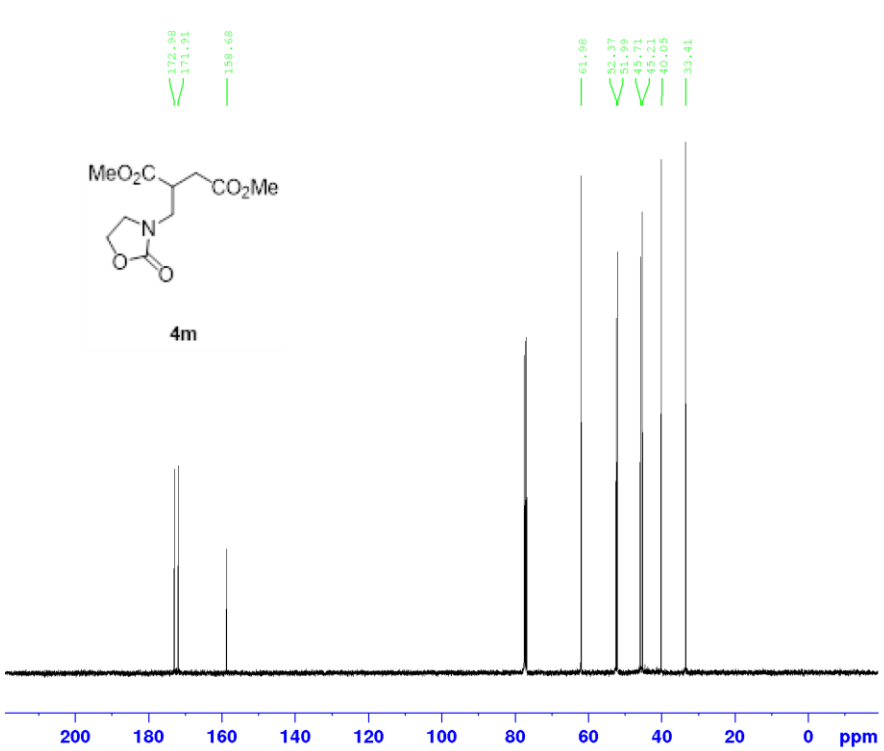



```
Current Data Parameters
NAME      epb274-prod_10
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20180410
Time                5.58
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG             zg30
TD                65536
SOLVENT           CDCl3
NS                    8
DS                    2
SWH           8278.146 Hz
FIDRES         0.126314 Hz
AQ             3.9583745 sec
RG                   64
DW               60.400 usec
DE                6.00 usec
TE               298.1 K
D1            1.00000000 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                 1H
P1                 14.50 usec
PL1                -2.00 dB
PL1W         23.88643074 W
SFO1        400.1324008 MHz

F2 - Processing parameters
SI                 32768
SF          400.1300094 MHz
WDW                   EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```



4m

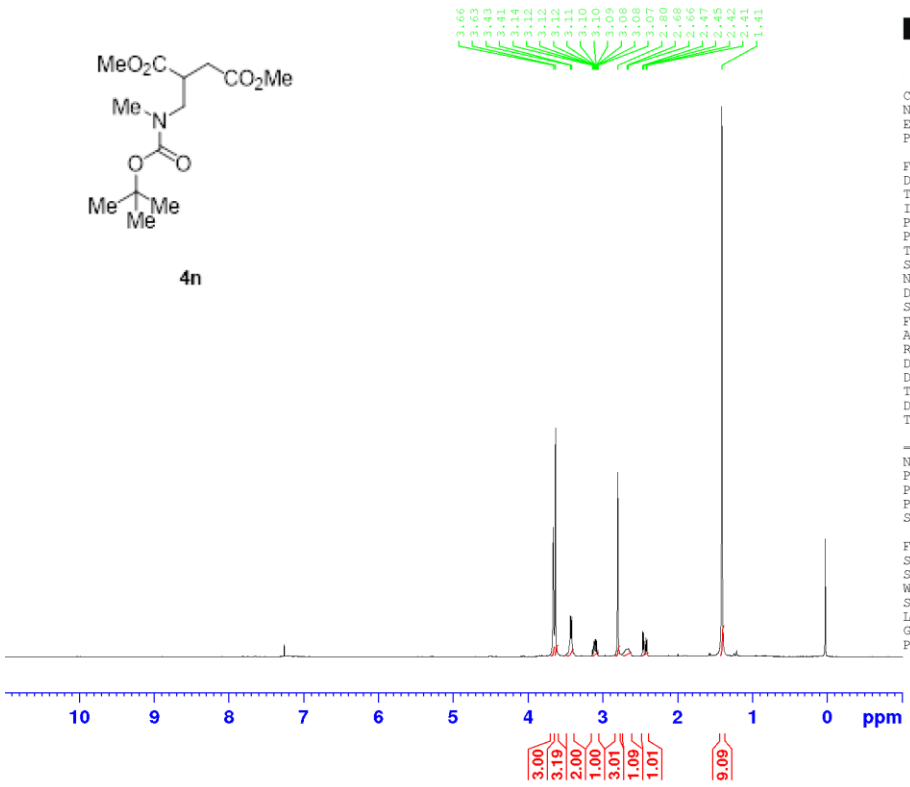
```
Current Data Parameters
NAME      epb274-prod-13C_10
EXPNO                      1
PROCNO                     1

F2 - Acquisition Parameters
Date_            20180410
Time                6.19
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG          zgpg30
TD                65536
SOLVENT             CDC13
NS                    512
DS                      4
SWH             23980.814 Hz
FIDRES          0.365918 Hz
AQ              1.3664256 sec
RG                  32768
DW                20.850 usec
DE                10.00 usec
TE                298.0 K
D1           1.00000000 sec
D11          0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  13C
P1                  10.00 usec
PL1                 -3.00 dB
PL1W         75.17808533 W
SFO1         100.6228298 MHz

======== CHANNEL f2 ========
CPDPRG[2          waltz16
NUC2                  1H
PCPD2               80.00 usec
PL2                 -3.00 dB
PL12                13.00 dB
PL13                13.00 dB
PL2W         30.07123375 W
PL12W         0.75535524 W
PL13W         0.75535524 W
SFO2         400.1316005 MHz

F2 - Processing parameters
SI                  32768
SF           100.6127640 MHz
WDW                    EM
SSB       0
LB                  1.00 Hz
GB        0
PC                  1.40
```



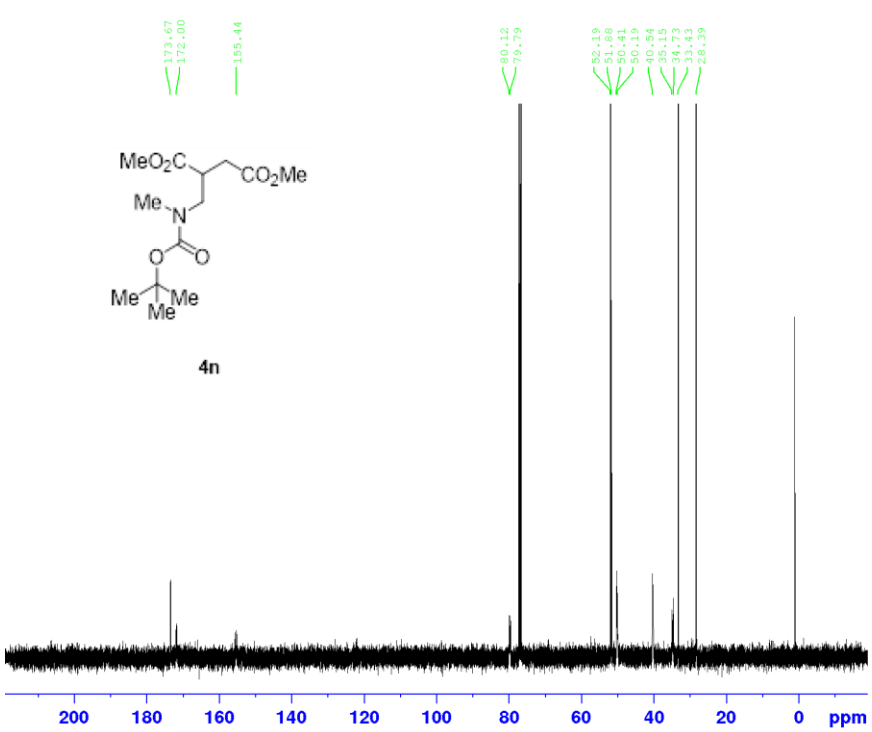
4n

```
Current Data Parameters
NAME      epb275-prod_10
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_            20180410
Time                6.29
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG             zg30
TD                65536
SOLVENT             CDC13
NS                      8
DS                      2
SWH              8278.146 Hz
FIDRES          0.126314 Hz
AQ              3.9583745 sec
RG                   40.3
DW                60.400 usec
DE                 6.00 usec
TE                298.1 K
D1           1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                   1H
P1                 14.50 usec
PL1                -2.00 dB
PL1W         23.88643074 W
SFO1         400.1324008 MHz

F2 - Processing parameters
SI                  32768
SF           400.1300094 MHz
WDW                   no
SSB       0
LB        0 Hz
GB        0
PC                  1.00
```



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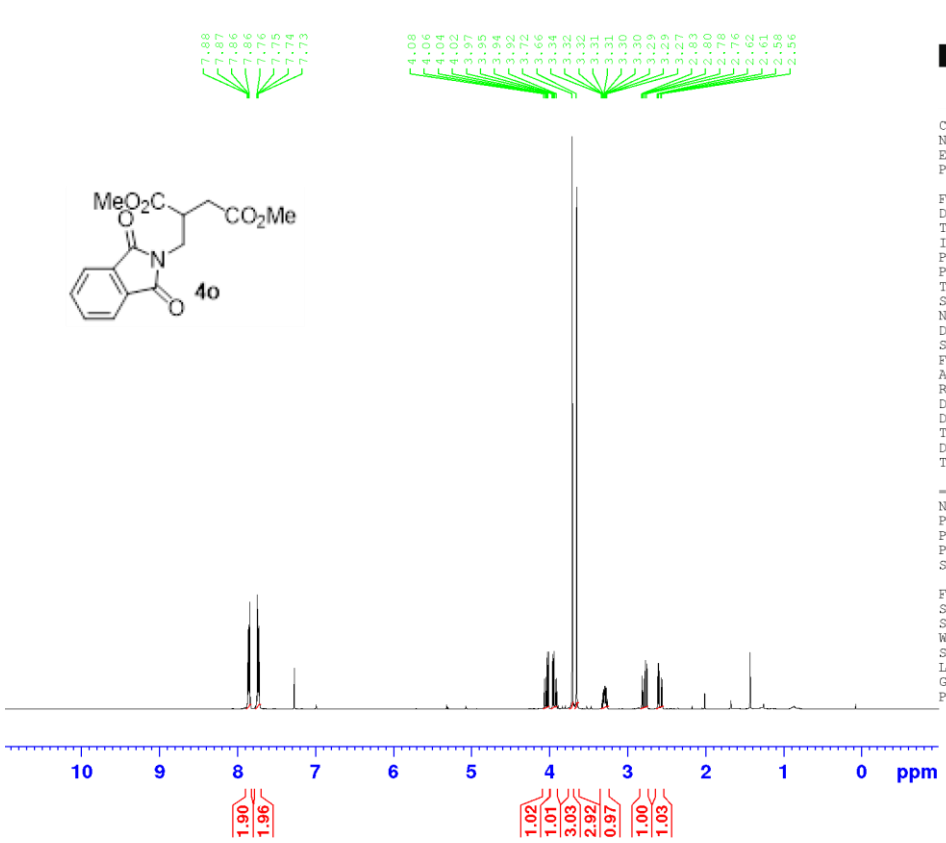
```
Current Data Parameters
NAME      epb275-prod-13C_10
EXPNO                      1
PROCNO                     1

F2 - Acquisition Parameters
Date_            20180410
Time                6.51
INSTRUM            spect
PROBHD    5 mm PABBO BB-
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                  512
DS                    4
SWH           23980.814 Hz
FIDRES         0.365918 Hz
AQ             1.3664256 sec
RG               20642.5
DW                20.850 usec
DE                10.00 usec
TE                298.1 K
D1            1.00000000 sec
D11           0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  13C
P1                 10.00 usec
PL1                -3.00 dB
PL1W         75.17808533 W
SFO1         100.6228298 MHz

======== CHANNEL f2 ========
CPDPRG[2           waltz16
NUC2                  1H
PCPD2              80.00 usec
PL2                -3.00 dB
PL12               13.00 dB
PL13               13.00 dB
PL2W         30.07123375 W
PL12W         0.75535524 W
PL13W         0.75535524 W
SFO2         400.1316005 MHz

F2 - Processing parameters
SI                 32768
SF           100.6127598 MHz
WDW                   no
SSB        0
LB         0 Hz
GB         0
PC                  1.40
```



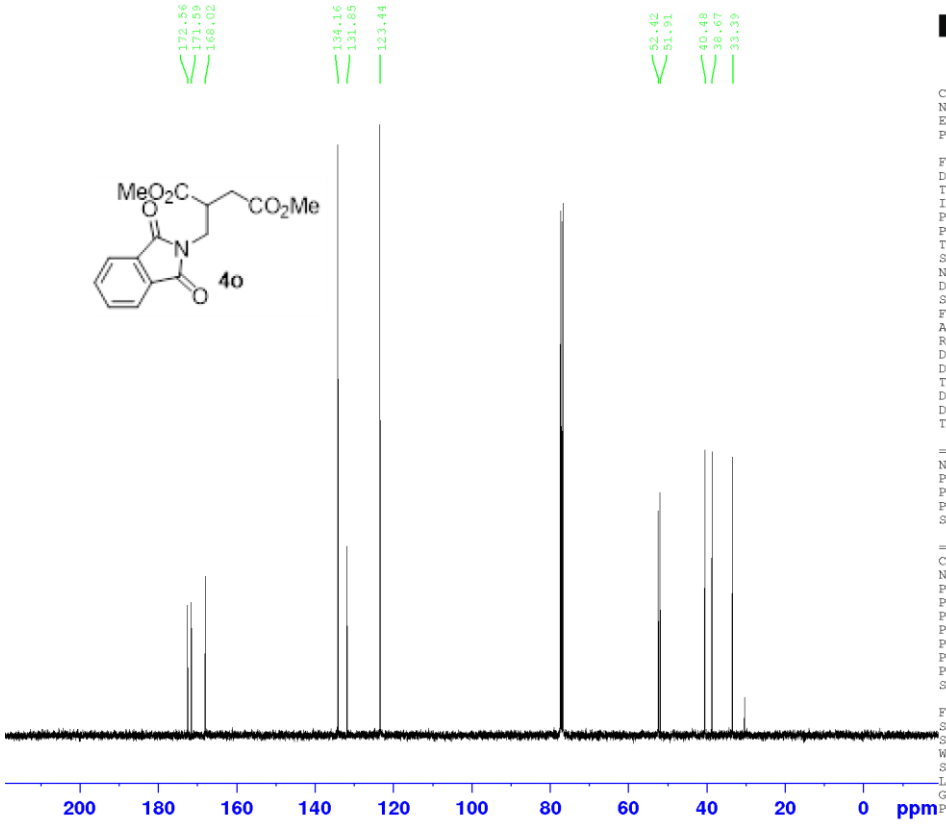
BRUKER

```
Current Data Parameters
NAME        BSCB-230 tol
EXPNO                 10
PROCNO                 1

F2 - Acquisition Parameters
Date_            20171130
Time                19.08
INSTRUM            spect
PROBHD    5 mm PABBO BB-
PULPROG             zg30
TD                 32768
SOLVENT           CDCl3
NS                     8
DS                     2
SWH             4789.272 Hz
FIDRES          0.146157 Hz
AQ             3.4209793 sec
RG                   128
DW                104.400 usec
DE                  6.00 usec
TE                298.0 K
D1            1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                 14.50 usec
PL1                -2.00 dB
PL1W         23.88643074 W
SFO1         400.1320007 MHz

F2 - Processing parameters
SI                 16384
SF           400.1300000 MHz
WDW                   EM
SSB        0
LB                 0.30 Hz
GB         0
PC                  1.00
```



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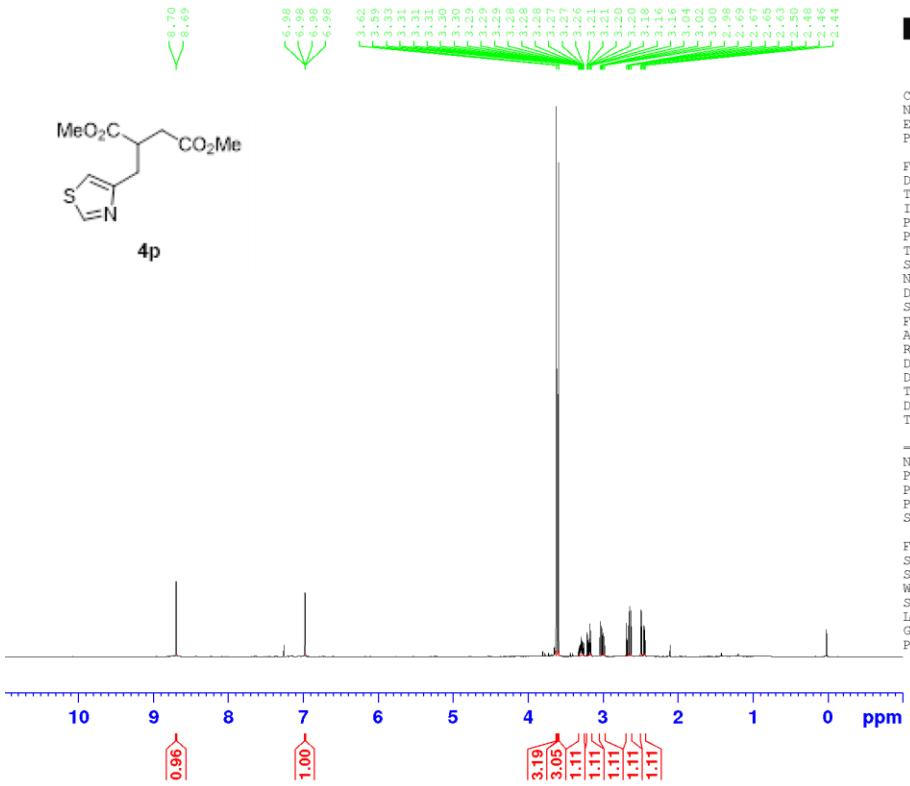
```
Current Data Parameters
NAME         BSCB-230 tol
EXPNO                 11
PROCNO                 1

F2 - Acquisition Parameters
Date_           20171201
Time                2.48
INSTRUM            spect
PROBHD    5 mm PABBO BB-
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                  512
DS                    4
SWH           23980.814 Hz
FIDRES         0.365918 Hz
AQ             1.3664256 sec
RG               20642.5
DW                20.850 usec
DE                10.00 usec
TE                298.1 K
D1             1.00000000 sec
D11            0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                 13C
P1                 10.00 usec
PL1                -3.00 dB
PL1W         75.17808533 W
SFO1        100.6228298 MHz

======== CHANNEL f2 ========
CPDPRG[2          waltz16
NUC2                 1H
PCPD2              80.00 usec
PL2                -3.00 dB
PL12               13.00 dB
PL13               13.00 dB
PL2W         30.07123375 W
PL12W         0.75535524 W
PL13W         0.75535524 W
SFO2        400.1316005 MHz

F2 - Processing parameters
SI                 32768
SF          100.6127690 MHz
WDW                   EM
SSB       0
LB                 1.00 Hz
GB        0
PC                 1.40
```



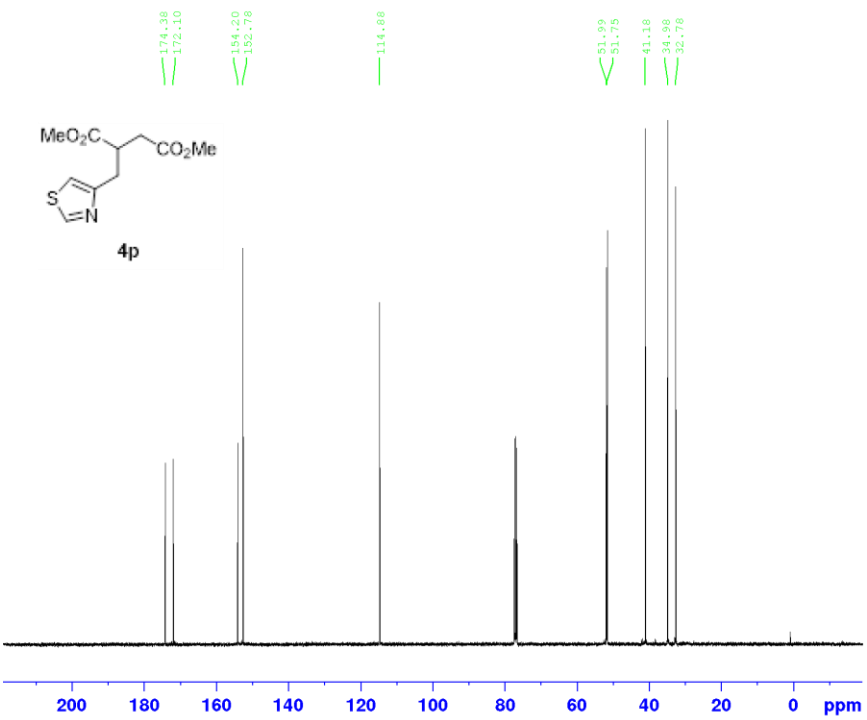
BRUKER

```
Current Data Parameters
NAME      epb220-prod_10
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20180410
Time                5.05
INSTRUM            spect
PROBHD    5 mm PABBO BB-
PULPROG             zg30
TD                65536
SOLVENT           CDCl3
NS                    8
DS                    2
SWH           8278.146 Hz
FIDRES         0.126314 Hz
AQ             3.9583745 sec
RG                   64
DW                60.400 usec
DE                 6.00 usec
TE                298.0 K
D1             1.00000000 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                 1H
P1                 14.50 usec
PL1                -2.00 dB
PL1W         23.88643074 W
SFO1        400.1324008 MHz

F2 - Processing parameters
SI                 32768
SF          400.1300097 MHz
WDW                   no
SSB       0
LB        0 Hz
GB        0
PC                 1.00
```



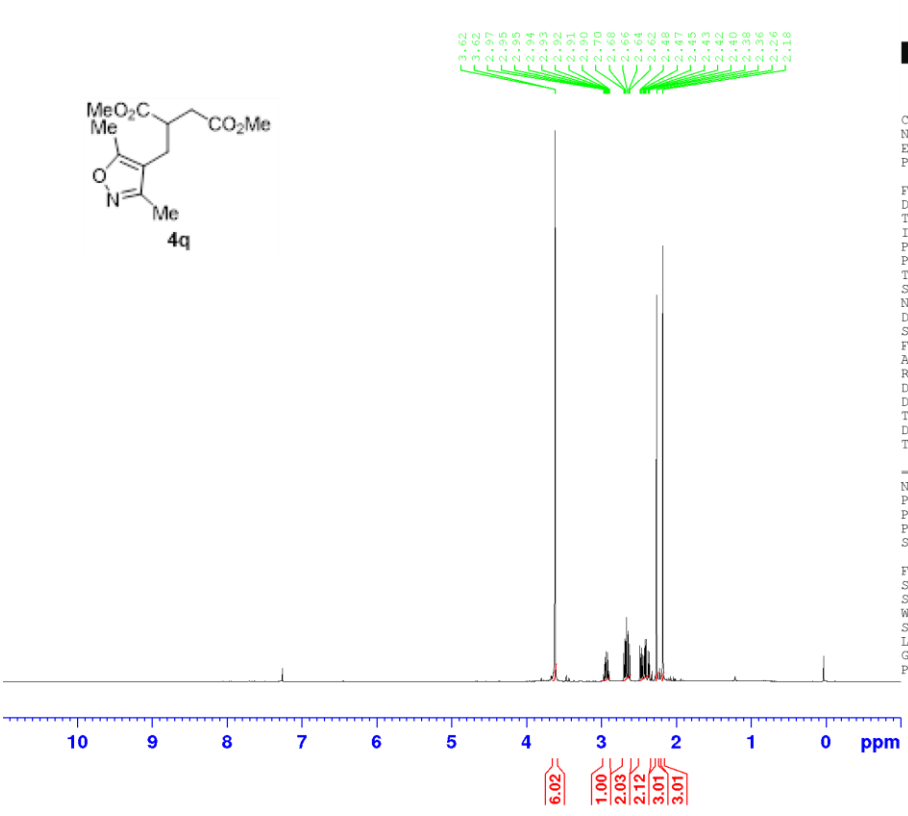
```
Current Data Parameters
NAME      epb220-prod-13C_10
EXPNO                      3
PROCNO                     1

F2 - Acquisition Parameters
Date_            20180410
Time                 5.27
INSTRUM             spect
PROBHD    5 mm PABBO BB-
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                  512
DS                    4
SWH           23980.814 Hz
FIDRES         0.365918 Hz
AQ             1.3664256 sec
RG                32768
DW               20.850 usec
DE               10.00 usec
TE               298.1 K
D1            1.00000000 sec
D11           0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                 13C
P1                 10.00 usec
PL1                -3.00 dB
PL1W         75.17808533 W
SFO1        100.6228298 MHz

======== CHANNEL f2 ========
CPDPRG[2          waltz16
NUC2                 1H
PCPD2              80.00 usec
PL2                -3.00 dB
PL12               13.00 dB
PL13               13.00 dB
PL2W         30.07123375 W
PL12W         0.75535524 W
PL13W         0.75535524 W
SFO2         400.1316005 MHz

F2 - Processing parameters
SI                32768
SF           100.6127656 MHz
WDW                  EM
SSB       0
LB                 1.00 Hz
GB        0
PC                 1.40
```

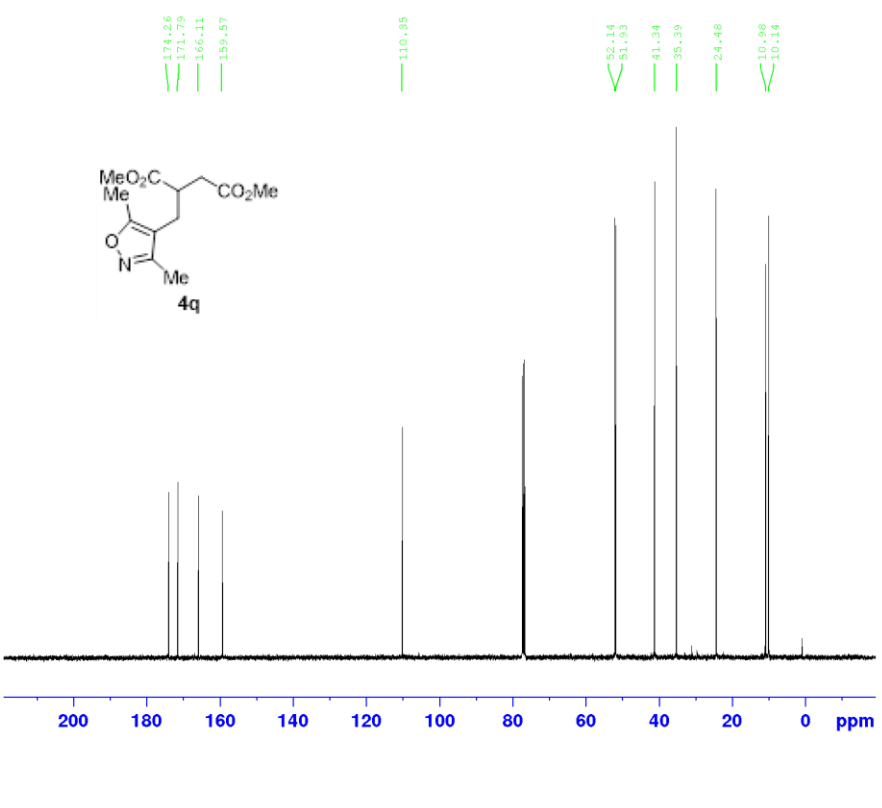


```
Current Data Parameters
NAME      epb214-prod_10
EXPNO                   1
PROCNO                  1

F2 - Acquisition Parameters
Date_            20180410
Time                 4.40
INSTRUM             spect
PROBHD    5 mm PABBO BB-
PULPROG             zg30
TD                65536
SOLVENT           CDCl3
NS                    8
DS                    2
SWH           8278.146 Hz
FIDRES         0.126314 Hz
AQ             3.9583745 sec
RG                   64
DW               60.400 usec
DE                6.00 usec
TE               297.9 K
D1            1.00000000 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                 1H
P1                 14.50 usec
PL1                -2.00 dB
PL1W         23.88643074 W
SFO1         400.1324008 MHz

F2 - Processing parameters
SI                32768
SF           400.1300094 MHz
WDW                  EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```



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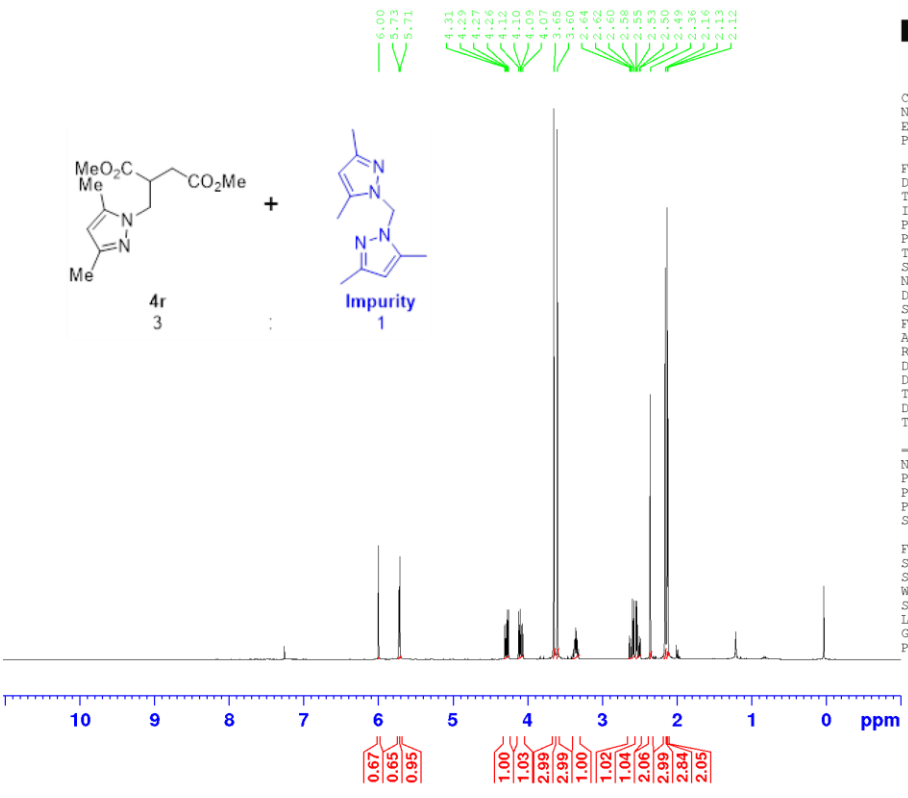
```
Current Data Parameters
NAME      epb214-prod-13C_10
EXPNO                      1
PROCNO                     1

F2 - Acquisition Parameters
Date_            20180410
Time                 5.01
INSTRUM             spect
PROBHD   5 mm PABBO BB-
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                  512
DS                    4
SWH           23980.814 Hz
FIDRES         0.365918 Hz
AQ             1.3664256 sec
RG                32768
DW               20.850 usec
DE               10.00 usec
TE               298.0 K
D1            1.00000000 sec
D11           0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  13C
P1                  10.00 usec
PL1                 -3.00 dB
PL1W          75.17808533 W
SFO1         100.6228298 MHz

======== CHANNEL f2 ========
CPDPRG[2           waltz16
NUC2                  1H
PCPD2               80.00 usec
PL2                 -3.00 dB
PL12                13.00 dB
PL13                13.00 dB
PL2W          30.07123375 W
PL12W          0.75535524 W
PL13W          0.75535524 W
SFO2         400.1316005 MHz

F2 - Processing parameters
SI                    32768
SF           100.6127619 MHz
WDW                      EM
SSB       0
LB                  1.00 Hz
GB        0
PC                  1.40
```



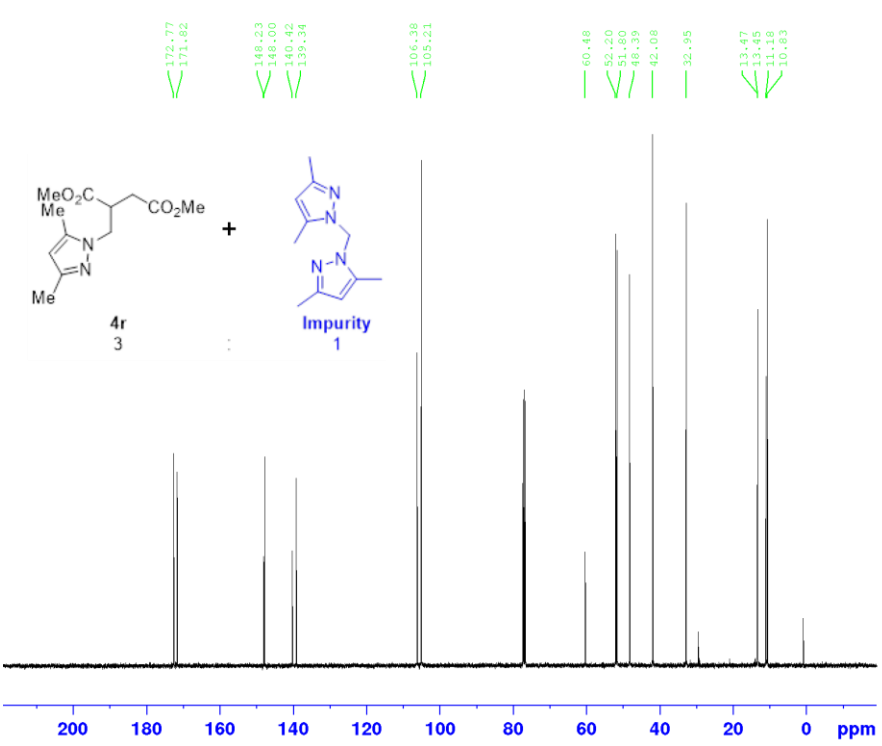
BRUKER

```
Current Data Parameters
NAME      epb298-prod_10
EXPNO                   2
PROCNO                  1

F2 - Acquisition Parameters
Date_            20180410
Time                 6.55
INSTRUM             spect
PROBHD   5 mm PABBO BB-
PULPROG             zg30
TD                65536
SOLVENT           CDCl3
NS                    8
DS                    2
SWH           8278.146 Hz
FIDRES         0.126314 Hz
AQ             3.9583745 sec
RG                 35.9
DW               60.400 usec
DE                6.00 usec
TE               298.0 K
D1            1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                 14.50 usec
PL1                -2.00 dB
PL1W          23.88643074 W
SFO1         400.1324008 MHz

F2 - Processing parameters
SI                    32768
SF           400.1300094 MHz
WDW                      EM
SSB       0
LB                  0.30 Hz
GB        0
PC                  1.00
```



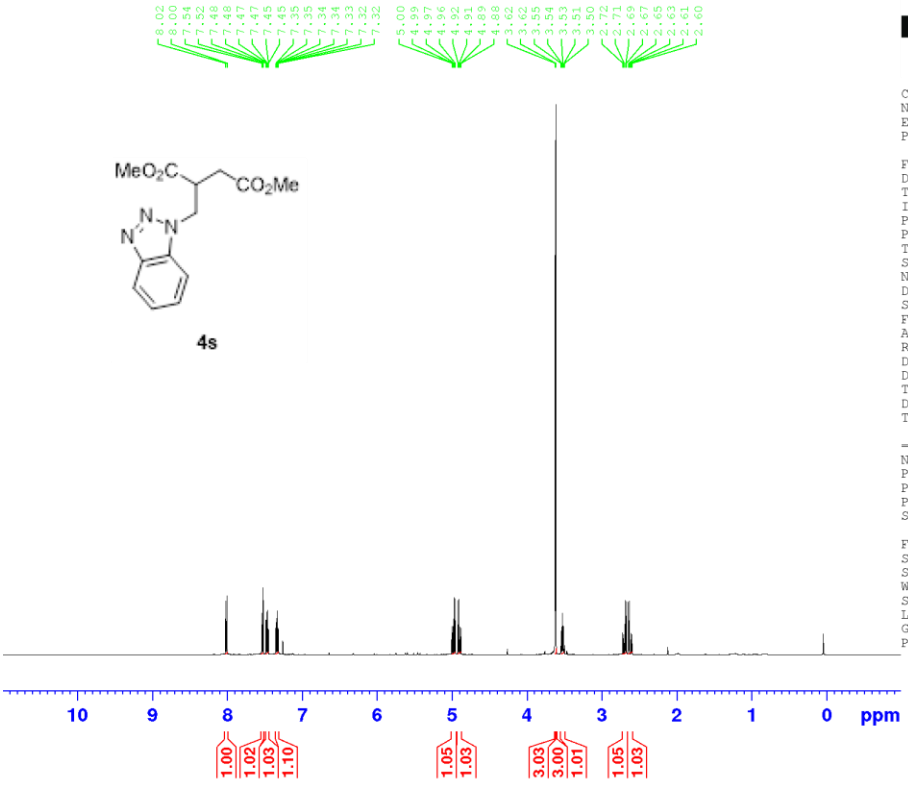

```
Current Data Parameters
NAME      epb298-prod-13C_10
EXPNO                      1
PROCNO                     1

F2 - Acquisition Parameters
Date_              20180410
Time                   7.17
INSTRUM               spect
PROBHD    5 mm PABBO BB-
PULPROG             zgpg30
TD                   65536
SOLVENT              CDCl3
NS                     512
DS                       4
SWH             23980.814 Hz
FIDRES           0.365918 Hz
AQ               1.3664256 sec
RG                   26008
DW                20.850 usec
DE                10.00 usec
TE                298.1 K
D1             1.00000000 sec
D11            0.03000000 sec
TD0                      1

======== CHANNEL f1 ========
NUC1                   13C
P1                  10.00 usec
PL1                 -3.00 dB
PL1W          75.17808533 W
SFO1         100.6228298 MHz

======== CHANNEL f2 ========
CPDPRG[2            waltz16
NUC2                   1H
PCPD2               80.00 usec
PL2                 -3.00 dB
PL12                13.00 dB
PL13                13.00 dB
PL2W          30.07123375 W
PL12W          0.75535524 W
PL13W          0.75535524 W
SFO2         400.1316005 MHz

F2 - Processing parameters
SI                     32768
SF           100.6127648 MHz
WDW                       EM
SSB        0
LB                  1.00 Hz
GB         0
PC                  1.40
```

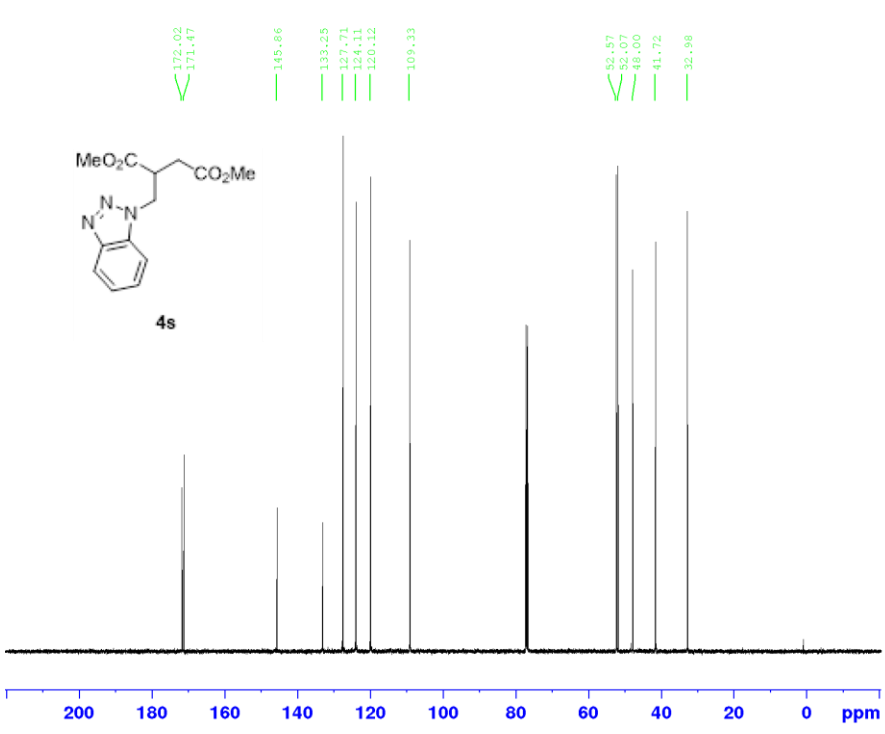



```
Current Data Parameters
NAME      epb252-prod_10
EXPNO                    1
PROCNO                   1

F2 - Acquisition Parameters
Date_              20180327
Time                   7.08
INSTRUM               spect
PROBHD    5 mm PATBO BB-
PULPROG              zg30
TD                   65536
SOLVENT              CDCl3
NS                       8
DS                       2
SWH            10000.000 Hz
FIDRES           0.152588 Hz
AQ               3.2767999 sec
RG                      64
DW                50.000 usec
DE                10.00 usec
TE                300.0 K
D1             1.00000000 sec
TD0                      1

======== CHANNEL f1 ========
NUC1                   1H
P1                  14.90 usec
PL1                 -3.00 dB
PL1W          50.11872482 W
SFO1         500.1330008 MHz

F2 - Processing parameters
SI                     32768
SF           500.1300131 MHz
WDW                       EM
SSB        0
LB                  0.30 Hz
GB         0
PC                  1.00
```



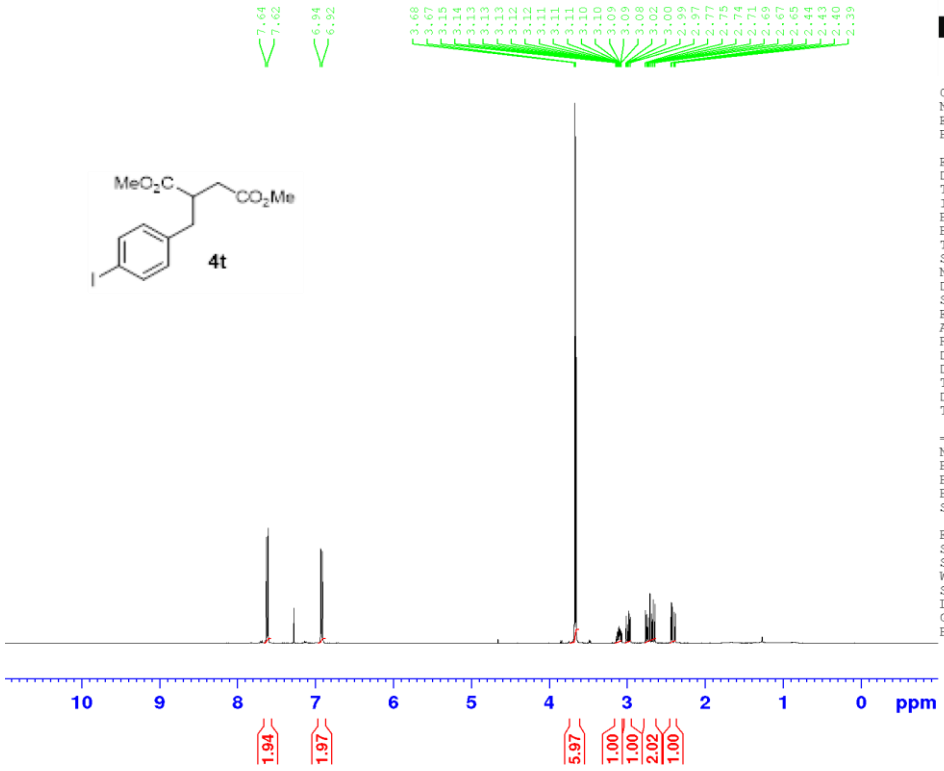

```
Current Data Parameters
NAME       epb252-prod-13C_10
EXPNO                       1
PROCNO                      1

F2 - Acquisition Parameters
Date_            20180327
Time                7.27
INSTRUM            spect
PROBHD   5 mm PATBO BB-
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                  512
DS                    4
SWH           30303.031 Hz
FIDRES         0.462388 Hz
AQ            1.0813440 sec
RG                11500
DW               16.500 usec
DE               10.00 usec
TE               298.4 K
D1            1.00000000 sec
D11           0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                 13C
P1                 9.75 usec
PL1               -1.00 dB
PL1W        94.86833191 W
SFO1       125.7703648 MHz

======== CHANNEL f2 ========
CPDPRG[2         waltz16
NUC2                 1H
PCPD2             80.00 usec
PL2               -3.00 dB
PL12              11.96 dB
PL13              11.96 dB
PL2W        50.11872482 W
PL12W        1.59955800 W
PL13W        1.59955800 W
SFO2       500.1320005 MHz

F2 - Processing parameters
SI                32768
SF           125.7577824 MHz
WDW                  EM
SSB       0
LB                1.00 Hz
GB        0
PC                1.40
```

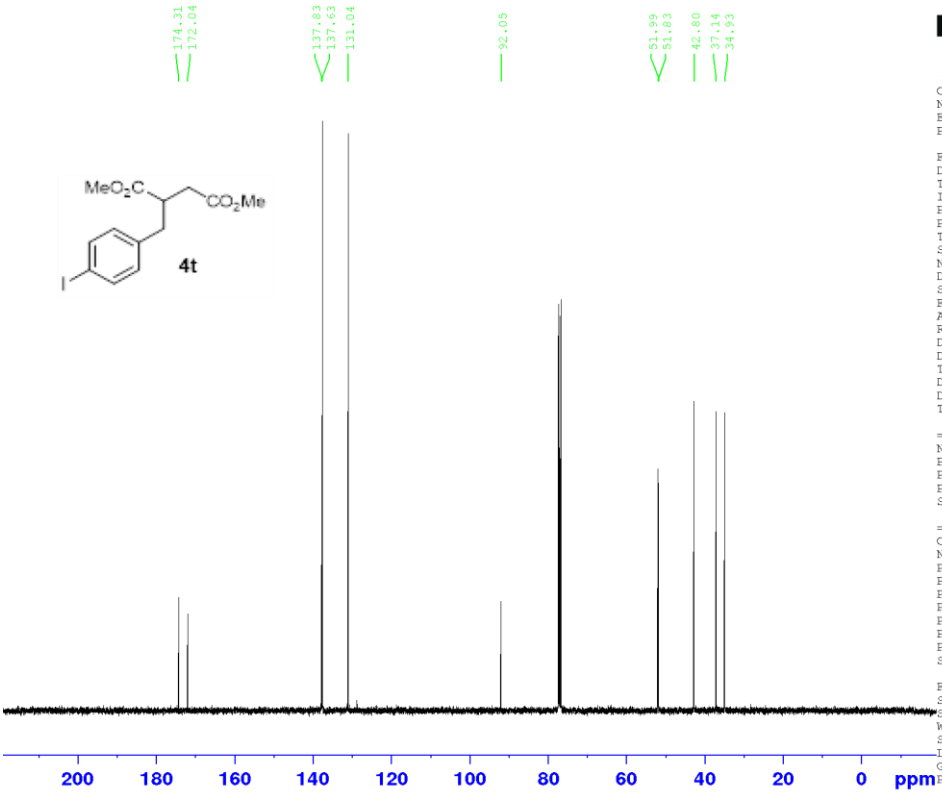



```
Current Data Parameters
NAME        BSCB-156 col
EXPNO                 10
PROCNO                 1

F2 - Acquisition Parameters
Date_            20170922
Time                14.13
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG             zg30
TD                 32768
SOLVENT           CDCl3
NS                     8
DS                     2
SWH             4789.272 Hz
FIDRES          0.146157 Hz
AQ              3.4209793 sec
RG                   128
DW                104.400 usec
DE                  6.00 usec
TE                298.1 K
D1            1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                14.50 usec
PL1               -2.00 dB
PL1W        23.88643074 W
SFO1       400.1320007 MHz

F2 - Processing parameters
SI                 16384
SF           400.1300000 MHz
WDW                   EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```



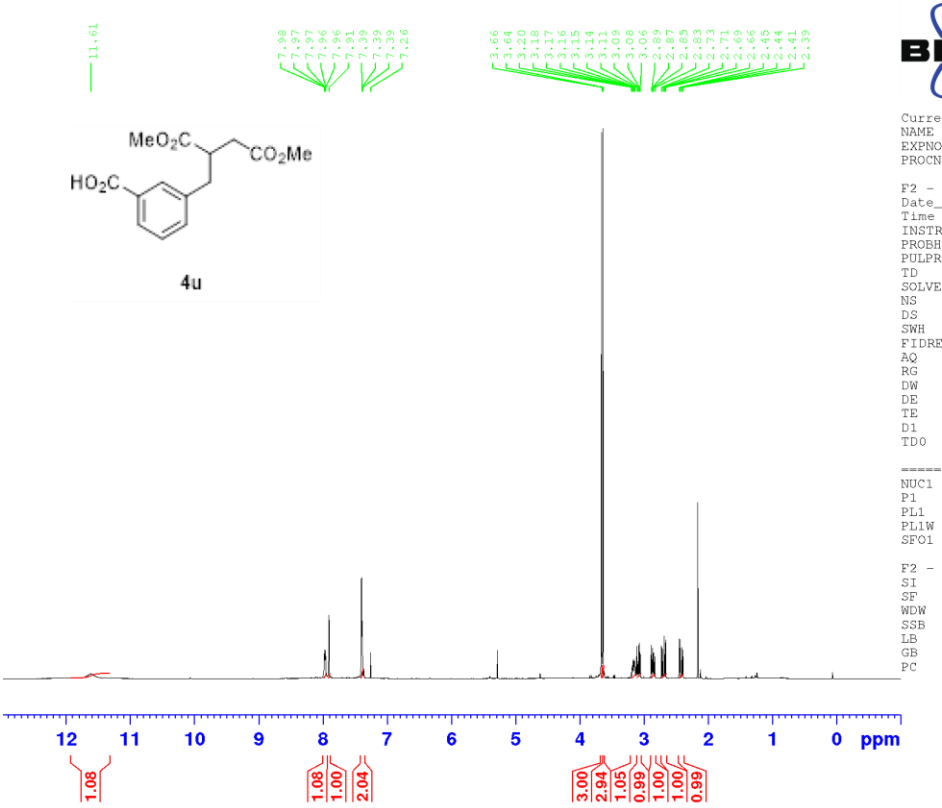

```
Current Data Parameters
NAME        BSCB-156 col
EXPNO                11
PROCNO                1

F2 - Acquisition Parameters
Date_           20170922
Time               15.38
INSTRUM            spect
PROBHD    5 mm PABBO BB-
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                  512
DS                    4
SWH           23980.814 Hz
FIDRES         0.365918 Hz
AQ             1.3664256 sec
RG                32768
DW               20.850 usec
DE               10.00 usec
TE               298.1 K
D1            1.00000000 sec
D11           0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                 13C
P1                 10.00 usec
PL1                -3.00 dB
PL1W         75.17808533 W
SFO1        100.6228298 MHz

======== CHANNEL f2 ========
CPDPRG[2          waltz16
NUC2                 1H
PCPD2              80.00 usec
PL2                -3.00 dB
PL12               13.00 dB
PL13               13.00 dB
PL2W         30.07123375 W
PL12W         0.75535524 W
PL13W         0.75535524 W
SFO2        400.1316005 MHz

F2 - Processing parameters
SI                32768
SF          100.6127690 MHz
WDW                  EM
SSB       0
LB                1.00 Hz
GB        0
PC                1.40
```

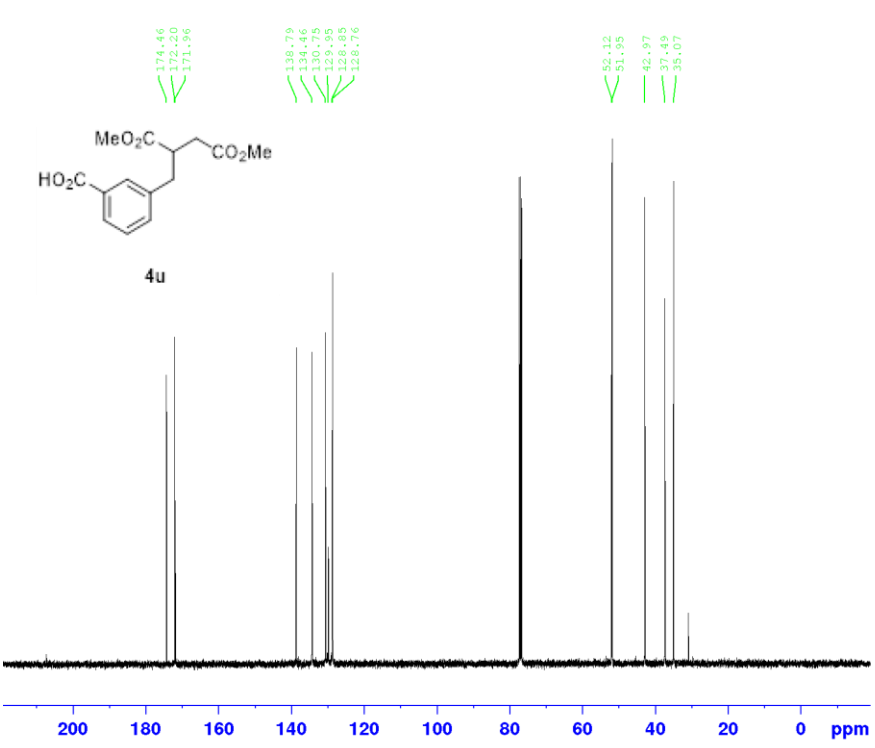



```
Current Data Parameters
NAME      epb212-prod_10
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20180410
Time               4.14
INSTRUM            spect
PROBHD    5 mm PABBO BB-
PULPROG             zg30
TD                65536
SOLVENT           CDCl3
NS                    8
DS                    2
SWH            8278.146 Hz
FIDRES         0.126314 Hz
AQ             3.9583745 sec
RG                   64
DW               60.400 usec
DE                6.00 usec
TE               298.0 K
D1            1.00000000 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                 1H
P1                14.50 usec
PL1               -2.00 dB
PL1W         23.88643074 W
SFO1        400.1324008 MHz

F2 - Processing parameters
SI                32768
SF          400.1300094 MHz
WDW                  EM
SSB       0
LB                0.30 Hz
GB        0
PC                1.00
```



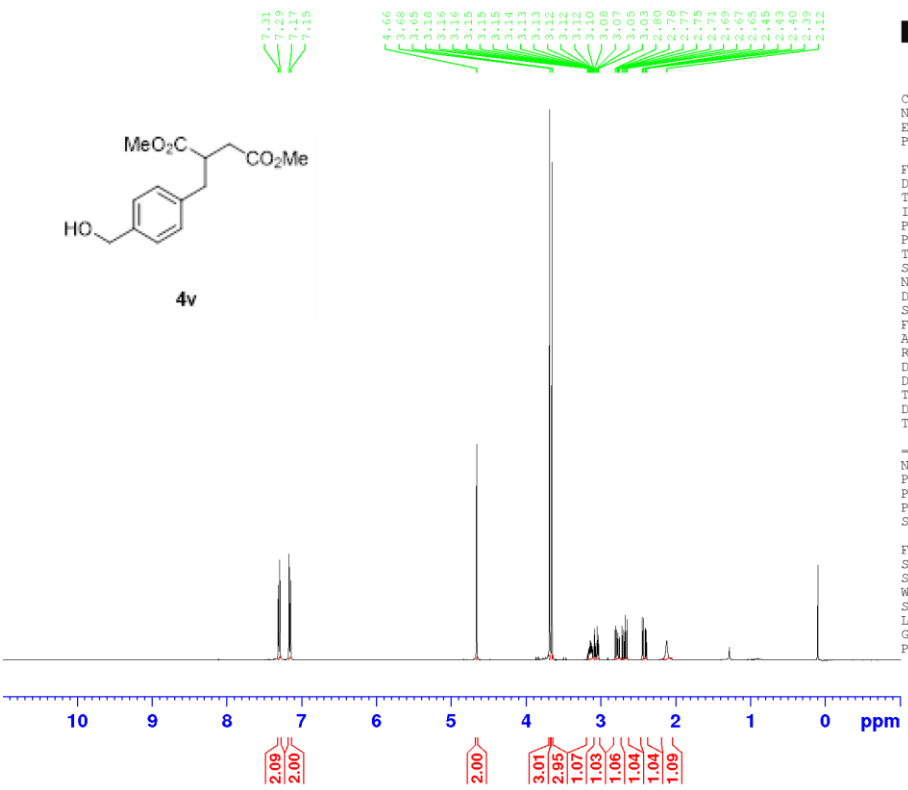

```
Current Data Parameters
NAME      epb212-prod-13C_10
EXPNO                      1
PROCNO                     1

F2 - Acquisition Parameters
Date_            20180410
Time                4.35
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                  512
DS                    4
SWH           23980.814 Hz
FIDRES         0.365918 Hz
AQ             1.3664256 sec
RG               23170.5
DW              20.850 usec
DE              10.00 usec
TE                298.0 K
D1           1.00000000 sec
D11          0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                 13C
P1                10.00 usec
PL1               -3.00 dB
PL1W        75.17808533 W
SFO1       100.6228298 MHz

======== CHANNEL f2 ========
CPDPRG[2          waltz16
NUC2                 1H
PCPD2             80.00 usec
PL2               -3.00 dB
PL12              13.00 dB
PL13              13.00 dB
PL2W        30.07123375 W
PL12W        0.75535524 W
PL13W        0.75535524 W
SFO2       400.1316005 MHz

F2 - Processing parameters
SI                32768
SF          100.6127591 MHz
WDW                  EM
SSB       0
LB                1.00 Hz
GB        0
PC                1.40
```

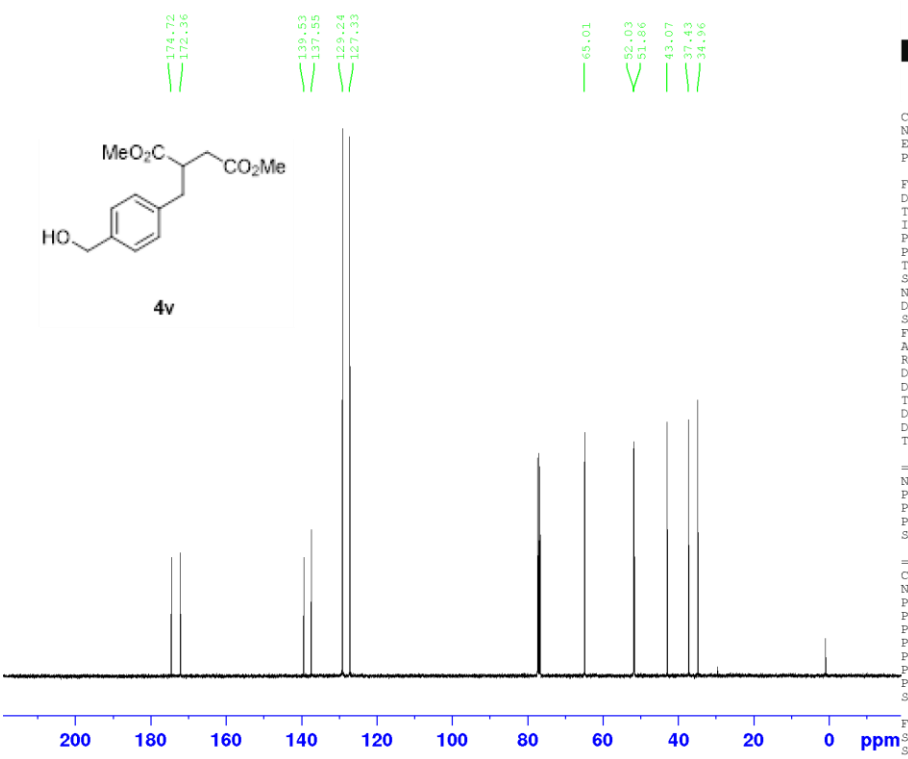



```
Current Data Parameters
NAME      epb240-prod_10
EXPNO                  3
PROCNO                 1

F2 - Acquisition Parameters
Date_            20180410
Time                5.31
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG            zg30
TD                65536
SOLVENT           CDCl3
NS                    8
DS                    2
SWH            8278.146 Hz
FIDRES         0.126314 Hz
AQ             3.9583745 sec
RG                   64
DW              60.400 usec
DE               6.00 usec
TE                298.0 K
D1           1.00000000 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                 1H
P1                14.50 usec
PL1               -2.00 dB
PL1W        23.88643074 W
SFO1       400.1324008 MHz

F2 - Processing parameters
SI                32768
SF          400.1299955 MHz
WDW                  EM
SSB       0
LB                0.30 Hz
GB        0
PC                1.00
```



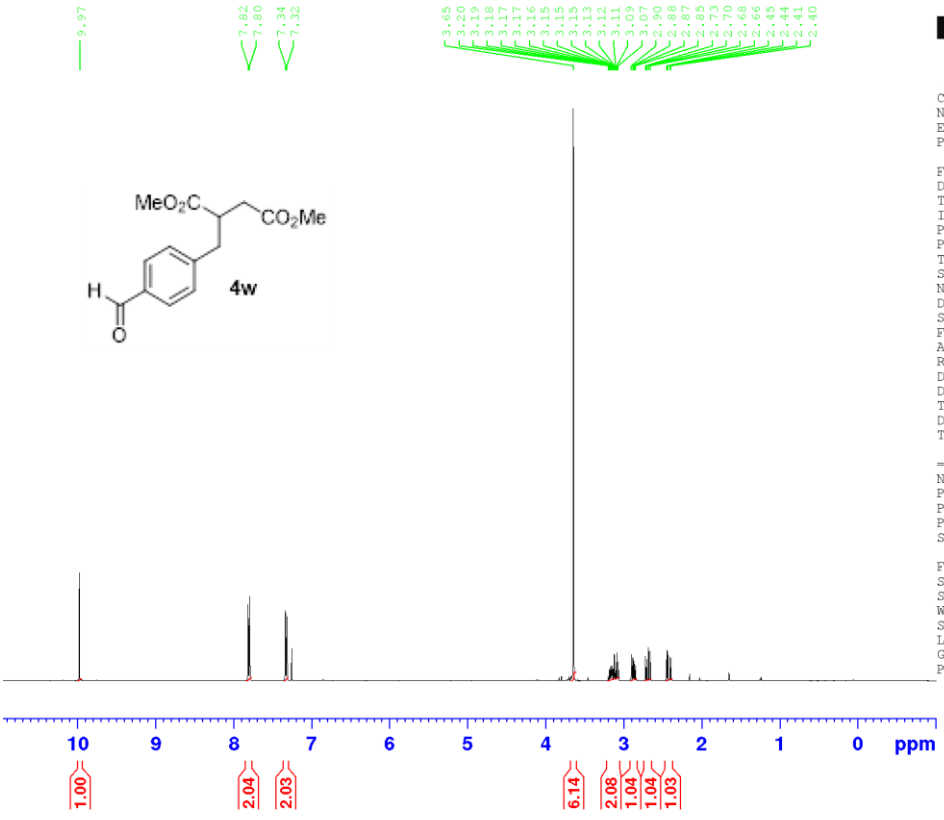

```
Current Data Parameters
NAME      epb240-prod-13C_10
EXPNO                      1
PROCNO                     1

F2 - Acquisition Parameters
Date_              20180410
Time                   5.53
INSTRUM               spect
PROBHD   5 mm PABBO BB-
PULPROG            zgpg30
TD                  65536
SOLVENT             CDCl3
NS                    512
DS                      4
SWH             23980.814 Hz
FIDRES          0.365918 Hz
AQ              1.3664256 sec
RG                23170.5
DW                20.850 usec
DE                10.00 usec
TE                298.0 K
D1              1.00000000 sec
D11             0.03000000 sec
TD0                        1

======== CHANNEL f1 ========
NUC1                     13C
P1                   10.00 usec
PL1                  -3.00 dB
PL1W          75.17808533 W
SFO1          100.6228298 MHz

======== CHANNEL f2 ========
CPDPRG[2            waltz16
NUC2                     1H
PCPD2                80.00 usec
PL2                  -3.00 dB
PL12                 13.00 dB
PL13                 13.00 dB
PL2W          30.07123375 W
PL12W          0.75535524 W
PL13W          0.75535524 W
SFO2          400.1316005 MHz

F2 - Processing parameters
SI                     32768
SF            100.6127606 MHz
WDW                       EM
SSB       0
LB                    1.00 Hz
GB        0
PC                    1.40
```

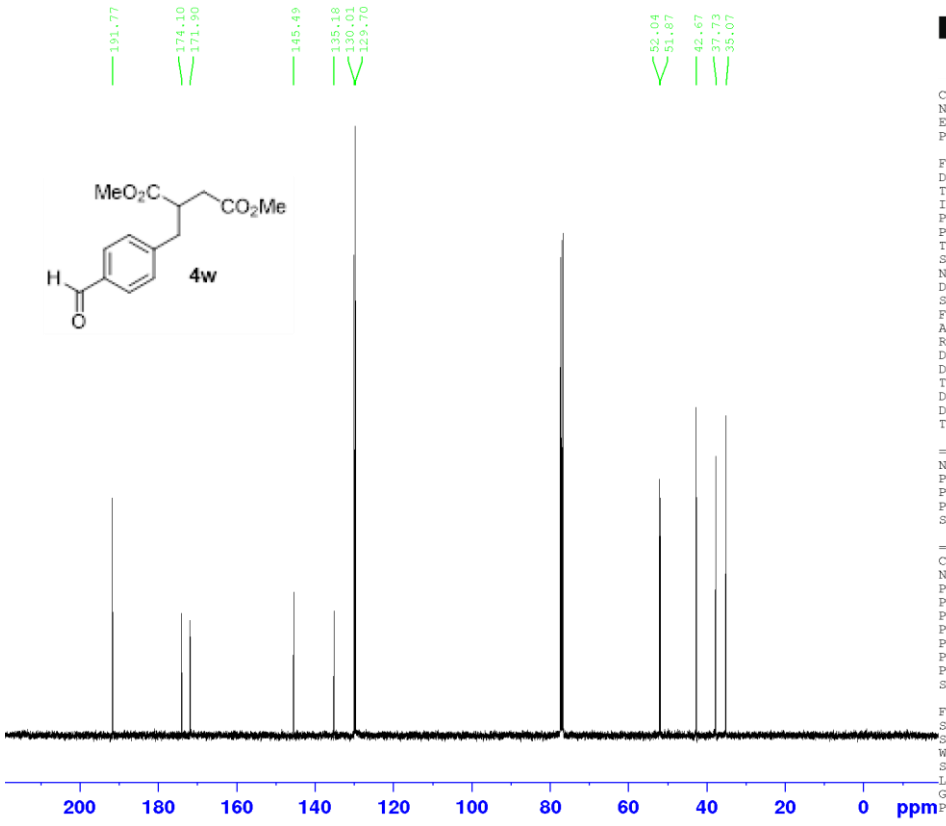



```
Current Data Parameters
NAME      BSCB-348-02 charac
EXPNO                     10
PROCNO                     1

F2 - Acquisition Parameters
Date_              20180314
Time                   7.10
INSTRUM               spect
PROBHD   5 mm PABBO BB-
PULPROG               zg30
TD                   32768
SOLVENT             CDCl3
NS                       8
DS                       2
SWH              4789.272 Hz
FIDRES          0.146157 Hz
AQ              3.4209793 sec
RG                   161.3
DW                104.400 usec
DE                  6.00 usec
TE                298.0 K
D1              1.00000000 sec
TD0                        1

======== CHANNEL f1 ========
NUC1                     1H
P1                   14.50 usec
PL1                  -2.00 dB
PL1W          23.88643074 W
SFO1          400.1320007 MHz

F2 - Processing parameters
SI                     16384
SF            400.1300095 MHz
WDW                       EM
SSB       0
LB                    0.30 Hz
GB        0
PC                    1.00
```



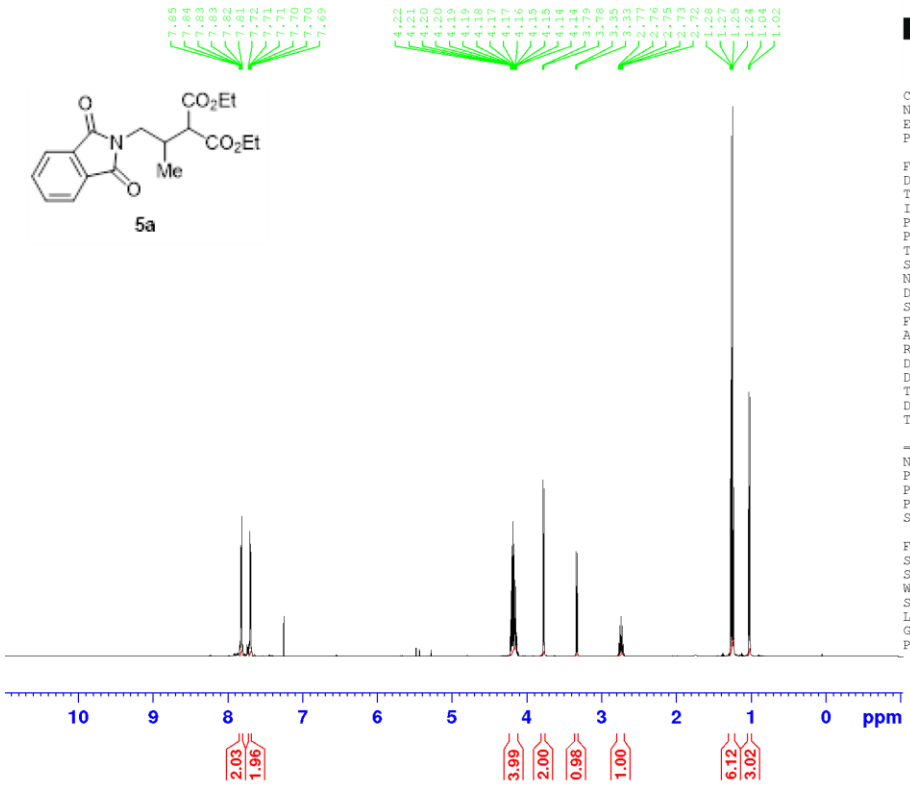

```
Current Data Parameters
NAME      BSCB-348-02 charac
EXPNO                 11
PROCNO                 1

F2 - Acquisition Parameters
Date_              20180314
Time                  7.32
INSTRUM              spect
PROBHD    5 mm PABBO BB-
PULPROG             zgpg30
TD                   65536
SOLVENT              CDCl3
NS                     512
DS                       4
SWH              23980.814 Hz
FIDRES           0.365918 Hz
AQ               1.3664256 sec
RG                   26008
DW                  20.850 usec
DE                  10.00 usec
TE                  298.0 K
D1             1.00000000 sec
D11            0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  13C
P1                  10.00 usec
PL1                 -3.00 dB
PL1W          75.17808533 W
SFO1         100.6228298 MHz

======== CHANNEL f2 ========
CPDPRG[2           waltz16
NUC2                  1H
PCPD2               80.00 usec
PL2                 -3.00 dB
PL12                13.00 dB
PL13                13.00 dB
PL2W          30.07123375 W
PL12W          0.75535524 W
PL13W          0.75535524 W
SFO2         400.1316005 MHz

F2 - Processing parameters
SI                   32768
SF           100.6127690 MHz
WDW                     EM
SSB       0
LB                   1.00 Hz
GB        0
PC                   1.40
```

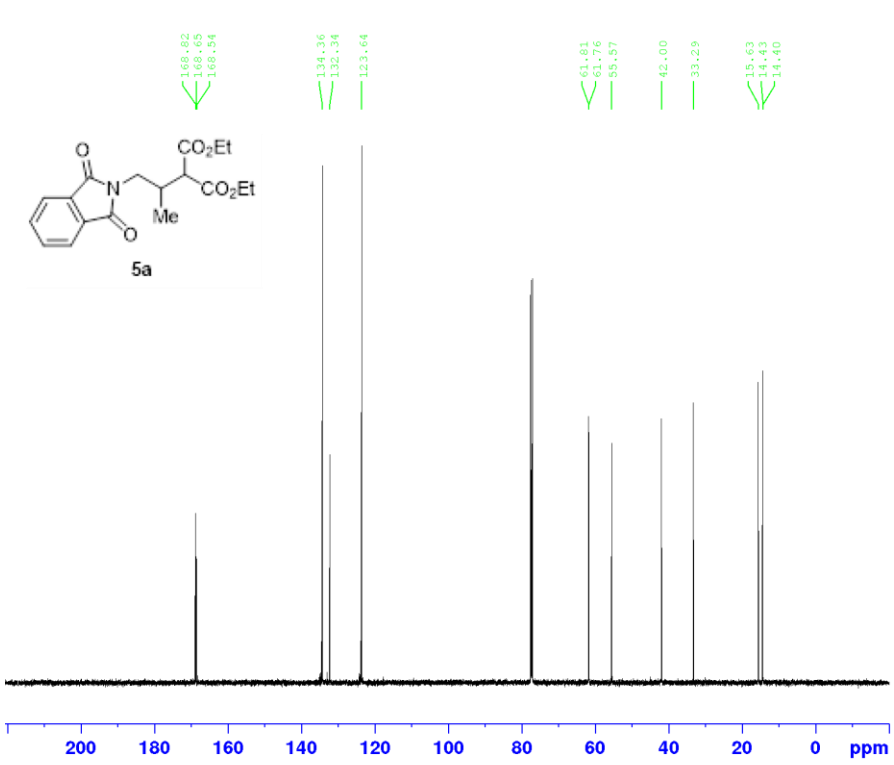



```
Current Data Parameters
NAME          mah-178-1H
EXPNO                 10
PROCNO                 1

F2 - Acquisition Parameters
Date_              20180404
Time                 15.38
INSTRUM              spect
PROBHD    5 mm PATBO BB-
PULPROG              zg30
TD                   32768
SOLVENT              CDCl3
NS                      32
DS                       2
SWH               6009.615 Hz
FIDRES           0.183399 Hz
AQ               2.7262976 sec
RG                    90.5
DW                  83.200 usec
DE                  10.00 usec
TE                  298.0 K
D1             1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                  14.90 usec
PL1                 -3.00 dB
PL1W          50.11872482 W
SFO1         500.1325007 MHz

F2 - Processing parameters
SI                   16384
SF           500.1300130 MHz
WDW                     EM
SSB       0
LB                   0.30 Hz
GB        0
PC                   1.00
```



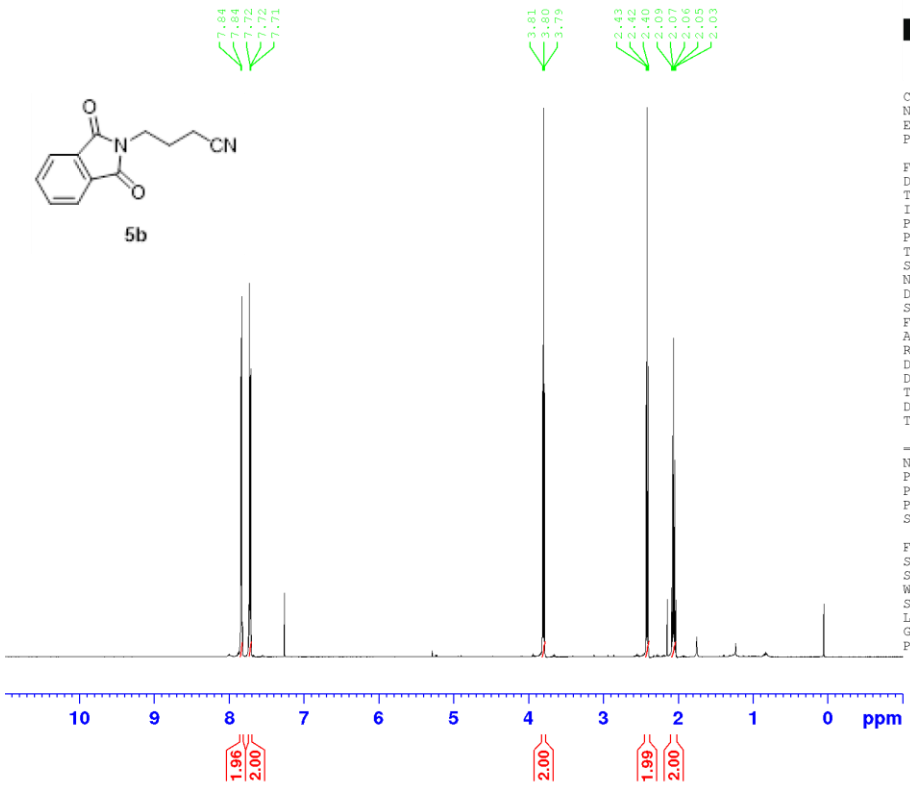

```
Current Data Parameters
NAME          mah-178-13C
EXPNO                  10
PROCNO                  1

F2 - Acquisition Parameters
Date_            20180404
Time                18.23
INSTRUM             spect
PROBHD   5 mm PATBO BB-
PULPROG           zgpg30
TD                 65536
SOLVENT            CDCl3
NS                   512
DS                     4
SWH           30303.031 Hz
FIDRES         0.462388 Hz
AQ             1.0813440 sec
RG                 13000
DW                16.500 usec
DE                10.00 usec
TE                299.9 K
D1            1.00000000 sec
D11           0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                 13C
P1                  9.75 usec
PL1                -1.00 dB
PL1W         94.86833191 W
SFO1        125.7703648 MHz

======== CHANNEL f2 ========
CPDPRG[2          waltz16
NUC2                 1H
PCPD2              80.00 usec
PL2                -3.00 dB
PL12               11.96 dB
PL13               11.96 dB
PL2W         50.11872482 W
PL12W         1.59955800 W
PL13W         1.59955800 W
SFO2        500.1320005 MHz

F2 - Processing parameters
SI                 32768
SF          125.7577441 MHz
WDW                   EM
SSB       0
LB                 1.00 Hz
GB        0
PC                 1.40
```

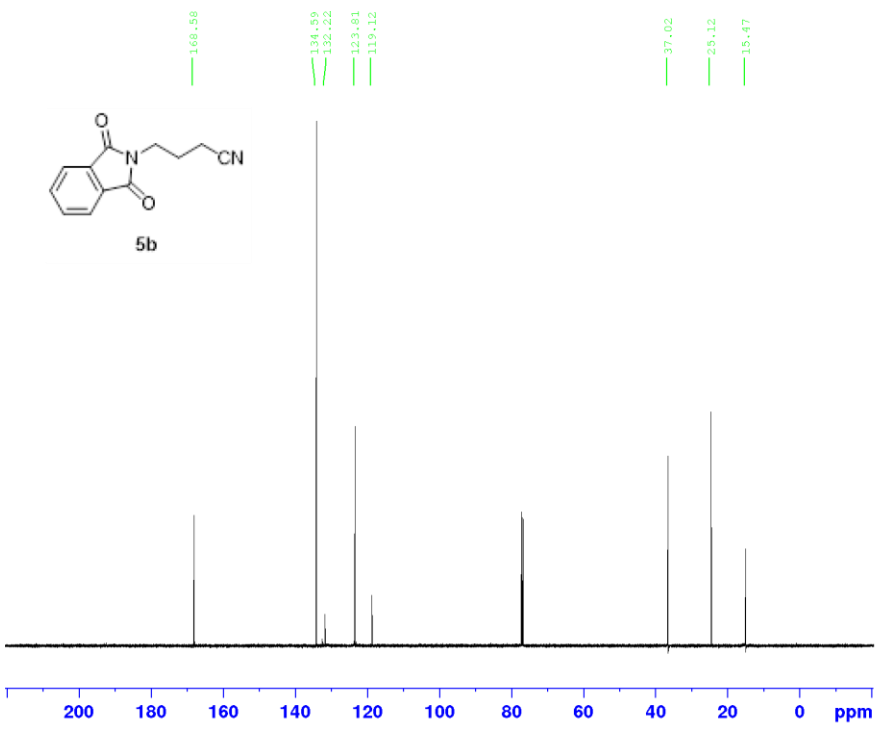



```
Current Data Parameters
NAME       epb246-prod_10
EXPNO                   1
PROCNO                  1

F2 - Acquisition Parameters
Date_            20180327
Time                6.44
INSTRUM             spect
PROBHD   5 mm PATBO BB-
PULPROG              zg30
TD                 65536
SOLVENT            CDCl3
NS                     8
DS                     2
SWH           10000.000 Hz
FIDRES         0.152588 Hz
AQ             3.2767999 sec
RG                   161
DW                50.000 usec
DE                10.00 usec
TE                300.0 K
D1            1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                 1H
P1                14.90 usec
PL1                -3.00 dB
PL1W         50.11872482 W
SFO1        500.1330008 MHz

F2 - Processing parameters
SI                 32768
SF          500.1300131 MHz
WDW                   EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```



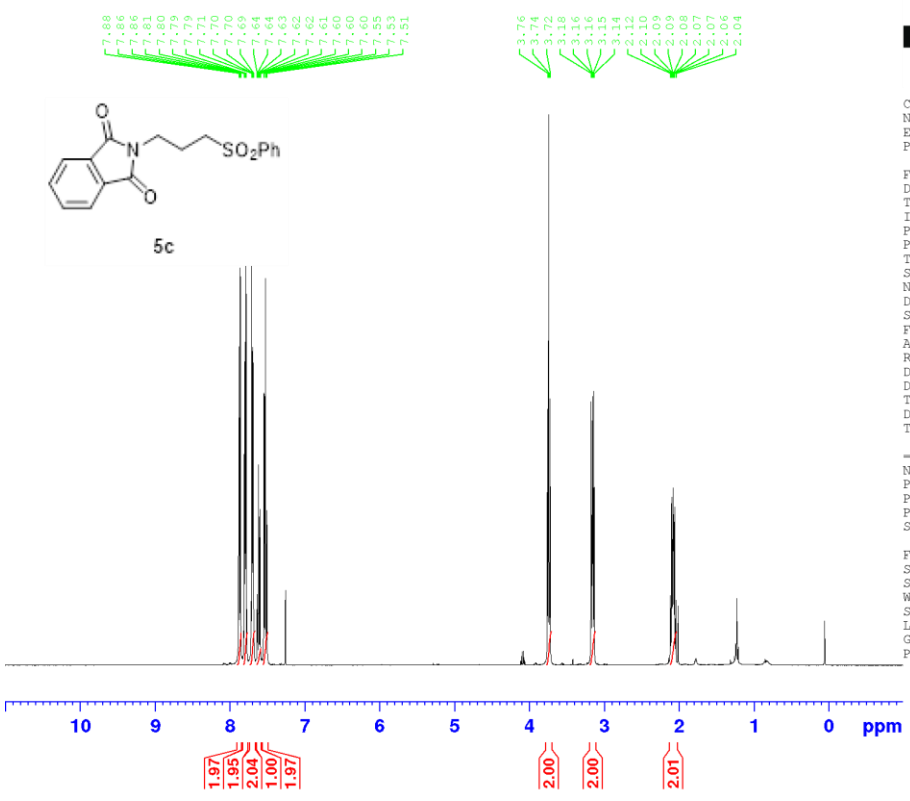

```
Current Data Parameters
NAME      epb246-prod-13C_10
EXPNO                      1
PROCNO                     1

F2 - Acquisition Parameters
Date_              20180327
Time                   7.03
INSTRUM               spect
PROBHD    5 mm PATBO BB-
PULPROG             zgpg30
TD                   65536
SOLVENT              CDCl3
NS                     512
DS                       4
SWH              30303.031 Hz
FIDRES           0.462388 Hz
AQ               1.0813440 sec
RG                   11500
DW                 16.500 usec
DE                 10.00 usec
TE                 298.5 K
D1              1.00000000 sec
D11             0.03000000 sec
TD0                      1

======== CHANNEL f1 ========
NUC1                   13C
P1                   9.75 usec
PL1                 -1.00 dB
PL1W          94.86833191 W
SFO1         125.7703648 MHz

======== CHANNEL f2 ========
CPDPRG[2           waltz16
NUC2                   1H
PCPD2               80.00 usec
PL2                 -3.00 dB
PL12                11.96 dB
PL13                11.96 dB
PL2W          50.11872482 W
PL12W          1.59955800 W
PL13W          1.59955800 W
SFO2         500.1320005 MHz

F2 - Processing parameters
SI                     32768
SF           125.7577796 MHz
WDW                       no
SSB       0
LB        0 Hz
GB        0
PC                     1.40
```

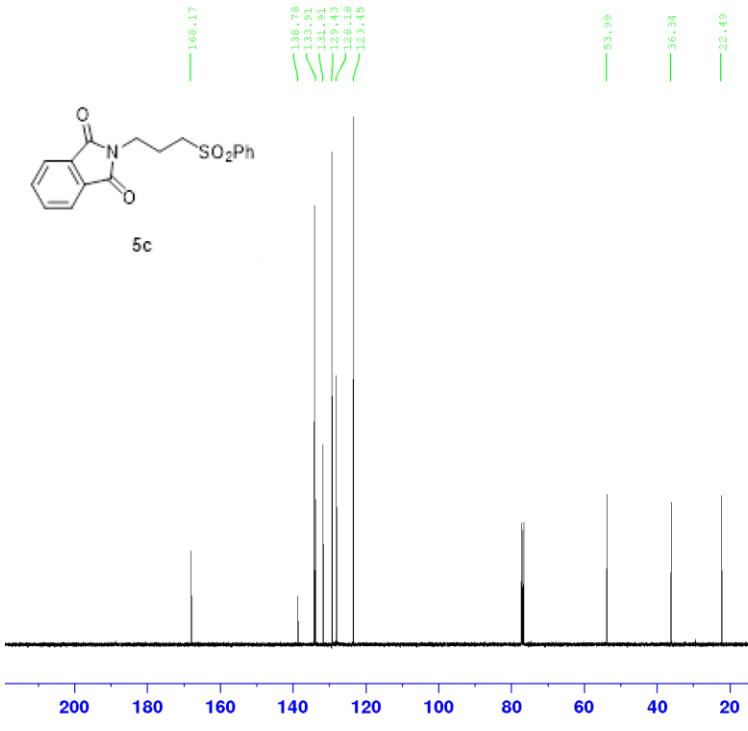



```
Current Data Parameters
NAME      epb180-prod_10
EXPNO                   3
PROCNO                  1

F2 - Acquisition Parameters
Date_              20180420
Time                   8.20
INSTRUM               spect
PROBHD    5 mm PABBO BB-
PULPROG               zg30
TD                   65536
SOLVENT              CDCl3
NS                       8
DS                       2
SWH              8278.146 Hz
FIDRES           0.126314 Hz
AQ               3.9583745 sec
RG                      64
DW                 60.400 usec
DE                  6.00 usec
TE                 298.0 K
D1              1.00000000 sec
TD0                      1

======== CHANNEL f1 ========
NUC1                   1H
P1                  14.50 usec
PL1                 -2.00 dB
PL1W          23.88643074 W
SFO1         400.1324008 MHz

F2 - Processing parameters
SI                     32768
SF           400.1300096 MHz
WDW                       EM
SSB       0
LB                  0.30 Hz
GB        0
PC                     1.00
```



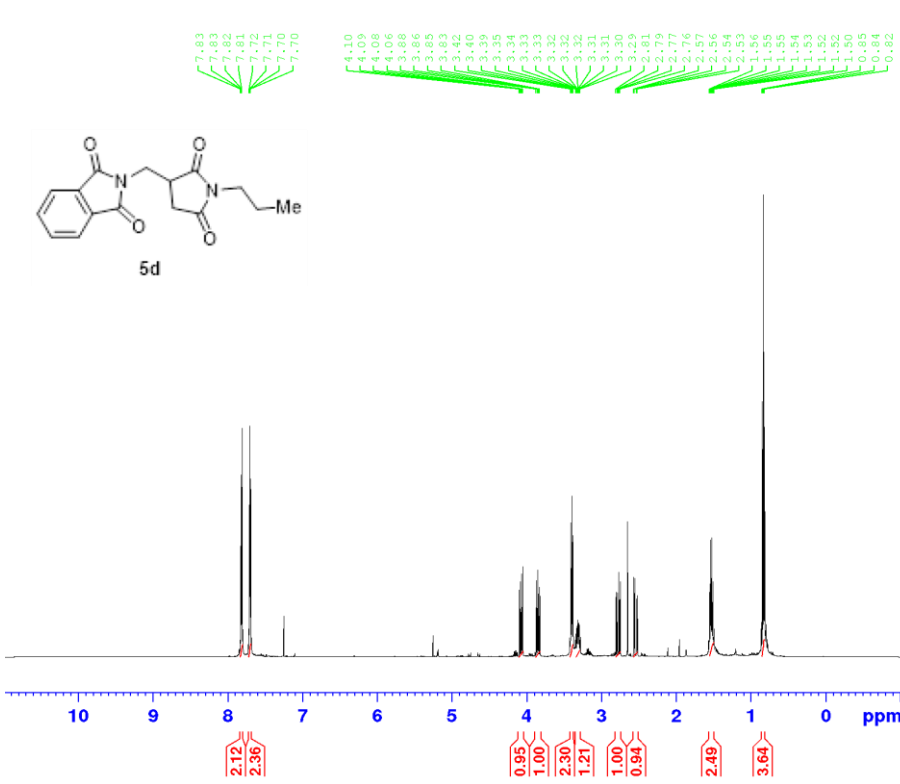

```
Current Data Parameters
NAME      epb180-prod-13C_10
EXPNO                      1
PROCNO                     1

F2 - Acquisition Parameters
Date_            20180420
Time                8.41
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                  512
DS                    4
SWH           23980.814 Hz
FIDRES         0.365918 Hz
AQ             1.3664256 sec
RG                32768
DW               20.850 usec
DE               10.00 usec
TE               298.3 K
D1            1.00000000 sec
D11           0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  13C
P1                 10.00 usec
PL1                -3.00 dB
PL1W         75.17808533 W
SFO1        100.6228298 MHz

======== CHANNEL f2 ========
CPDPRG[2          waltz16
NUC2                  1H
PCPD2              80.00 usec
PL2                -3.00 dB
PL12               13.00 dB
PL13               13.00 dB
PL2W         30.07123375 W
PL12W         0.75535524 W
PL13W         0.75535524 W
SFO2        400.1316005 MHz

F2 - Processing parameters
SI                 32768
SF          100.6127626 MHz
WDW                   no
SSB       0
LB        0 Hz
GB        0
PC                 1.40
```

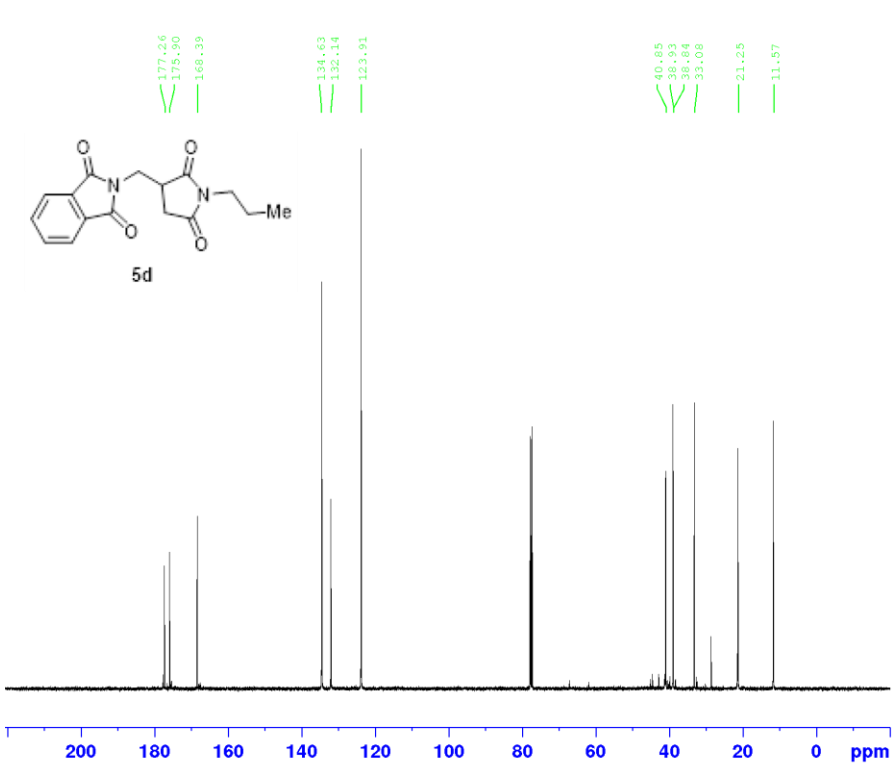



```
Current Data Parameters
NAME           mah-191-1H
EXPNO                  10
PROCNO                  1

F2 - Acquisition Parameters
Date_            20180410
Time                0.17
INSTRUM            spect
PROBHD   5 mm PATBO BB-
PULPROG             zg30
TD                32768
SOLVENT           CDCl3
NS                   32
DS                    2
SWH            6009.615 Hz
FIDRES         0.183399 Hz
AQ             2.7262976 sec
RG                   64
DW               83.200 usec
DE               10.00 usec
TE               299.2 K
D1            1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                 14.90 usec
PL1                -3.00 dB
PL1W         50.11872482 W
SFO1        500.1325007 MHz

F2 - Processing parameters
SI                 16384
SF          500.1300130 MHz
WDW                   EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```



5d

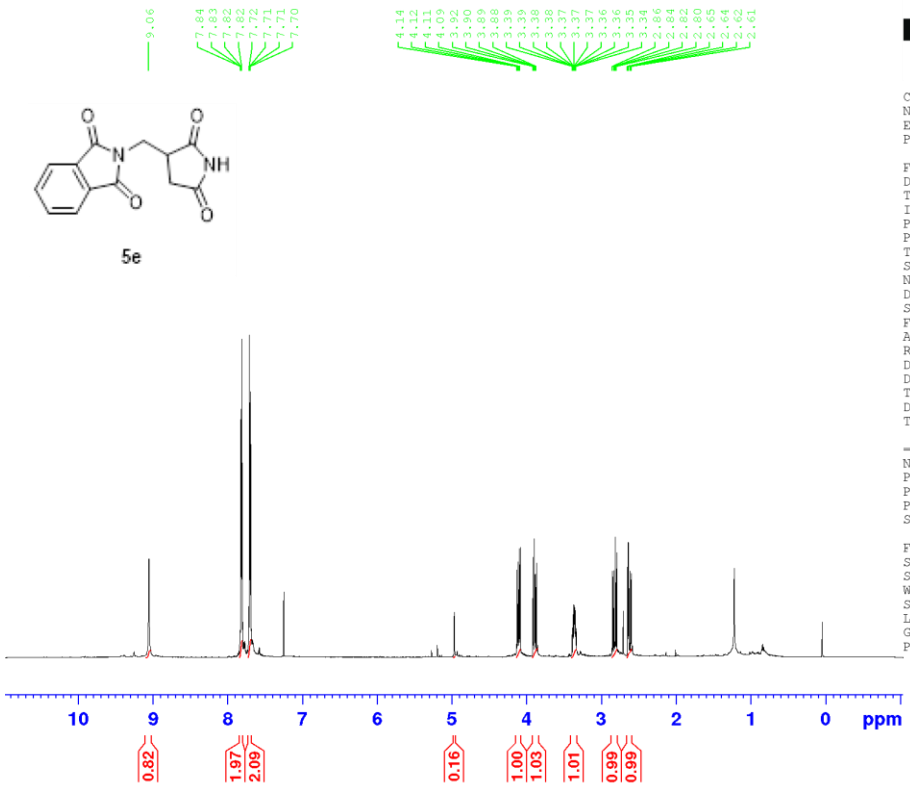
```
Current Data Parameters
NAME                  mah-191-13C
EXPNO                          10
PROCNO                          1

F2 - Acquisition Parameters
Date_                   20180410
Time                        0.36
INSTRUM                    spect
PROBHD     5 mm PATBO BB-
PULPROG                 zgpg30
TD                           65536
SOLVENT                     CDCl3
NS                             512
DS                               4
SWH                  30303.031 Hz
FIDRES                0.462388 Hz
AQ                    1.0813440 sec
RG                           11500
DW                        16.500 usec
DE                        10.00 usec
TE                        300.0 K
D1                    1.00000000 sec
D11                   0.03000000 sec
TD0                             1

======== CHANNEL f1 ========
NUC1                          13C
P1                           9.75 usec
PL1                         -1.00 dB
PL1W                94.86833191 W
SFO1               125.7703648 MHz

======== CHANNEL f2 ========
CPDPRG[2                 waltz16
NUC2                          1H
PCPD2                     80.00 usec
PL2                         -3.00 dB
PL12                        11.96 dB
PL13                        11.96 dB
PL2W                50.11872482 W
PL12W                1.59955800 W
PL13W                1.59955800 W
SFO2               500.1320005 MHz

F2 - Processing parameters
SI                           32768
SF                 125.7577441 MHz
WDW                             EM
SSB                0
LB                            1.00 Hz
GB                 0
PC                            1.40
```



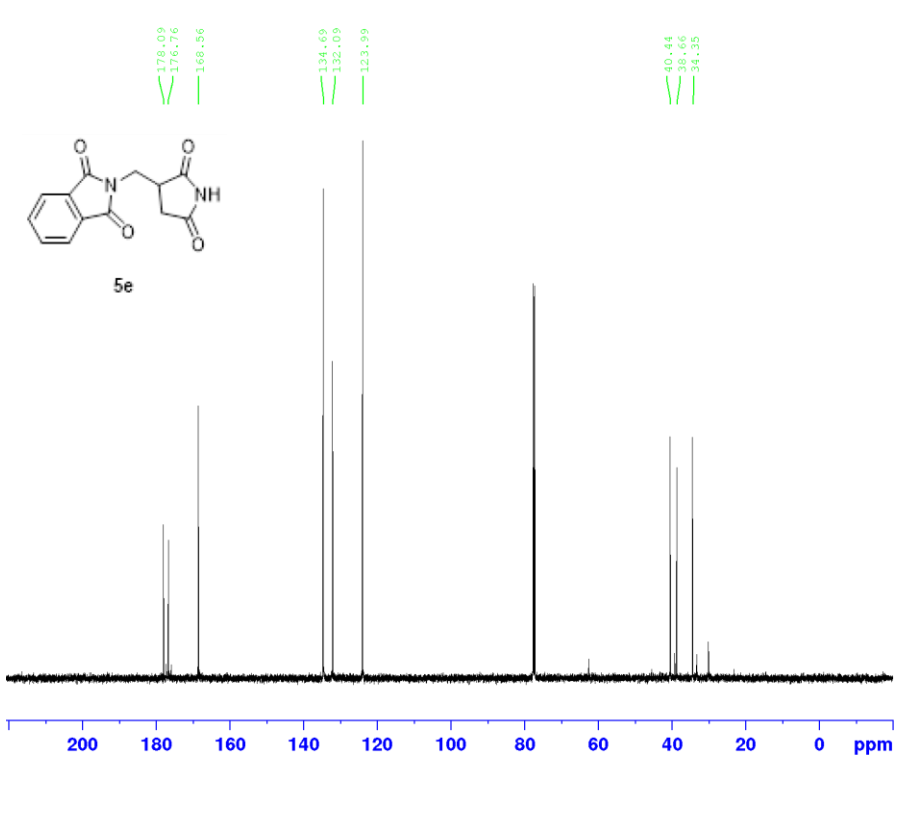
5e

```
Current Data Parameters
NAME                  mah-173-1H
EXPNO                          10
PROCNO                          1

F2 - Acquisition Parameters
Date_                   20180327
Time                       13.03
INSTRUM                    spect
PROBHD     5 mm PATBO BB-
PULPROG                    zg30
TD                           32768
SOLVENT                     CDCl3
NS                               8
DS                               2
SWH                   6009.615 Hz
FIDRES                0.183399 Hz
AQ                    2.7262976 sec
RG                            90.5
DW                        83.200 usec
DE                        10.00 usec
TE                        298.0 K
D1                    1.00000000 sec
TD0                             1

======== CHANNEL f1 ========
NUC1                          1H
P1                          14.90 usec
PL1                         -3.00 dB
PL1W                50.11872482 W
SFO1               500.1325007 MHz

F2 - Processing parameters
SI                           16384
SF                 500.1300136 MHz
WDW                             EM
SSB                0
LB                            0.30 Hz
GB                 0
PC                            1.00
```



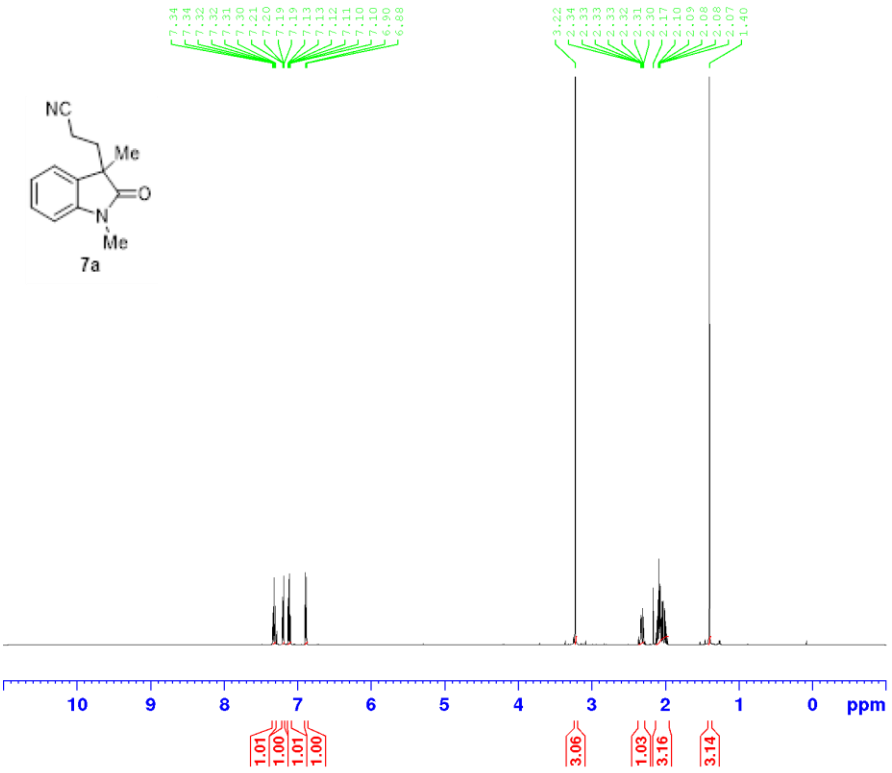
Current Data Parameters
NAME mah-173-13C
EXPNO 10
PROCNO 1

F2 - Acquisition Parameters
Date_ 20180327
Time 14.26
INSTRUM spect
PROBHD 5 mm PATBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 195
DS 4
SWH 30303.031 Hz
FIDRES 0.462388 Hz
AQ 1.0813440 sec
RG 11500
DW 16.500 usec
DE 10.00 usec
TE 298.8 K
D1 1.00000000 sec
D11 0.03000000 sec
TD0 1

======== CHANNEL f1 ========
NUC1 13C
P1 9.75 usec
PL1 -1.00 dB
PL1W 94.86833191 W
SFO1 125.7703648 MHz

======== CHANNEL f2 ========
CPDPRG[2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -3.00 dB
PL12 11.96 dB
PL13 11.96 dB
PL2W 50.11872482 W
PL12W 1.59955800 W
PL13W 1.59955800 W
SFO2 500.1320005 MHz

F2 - Processing parameters
SI 32768
SF 125.7577441 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40



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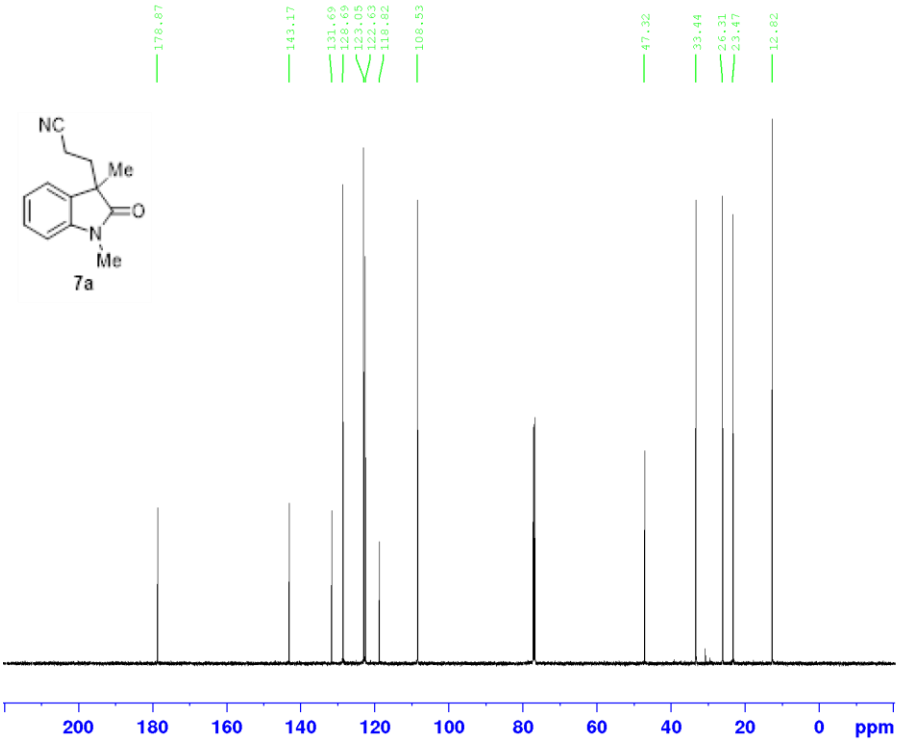
```
Current Data Parameters
NAME          mah-116-1H
EXPNO                 10
PROCNO                 1

F2 - Acquisition Parameters
Date_           20180224
Time              11.13
INSTRUM             spect
PROBHD   5 mm PABBO BB-
PULPROG             zg30
TD                 32768
SOLVENT            CDCl3
NS                    32
DS                     2
SWH            6009.615 Hz
FIDRES         0.183399 Hz
AQ            2.7262976 sec
RG                  50.8
DW                83.200 usec
DE                10.00 usec
TE                298.0 K
D1            1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                 15.30 usec
PL1                3.00 dB
PL1W        12.58925438 W
SFO1        500.1325007 MHz

F2 - Processing parameters
SI                 16384
SF          500.1300000 MHz
WDW                   EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```



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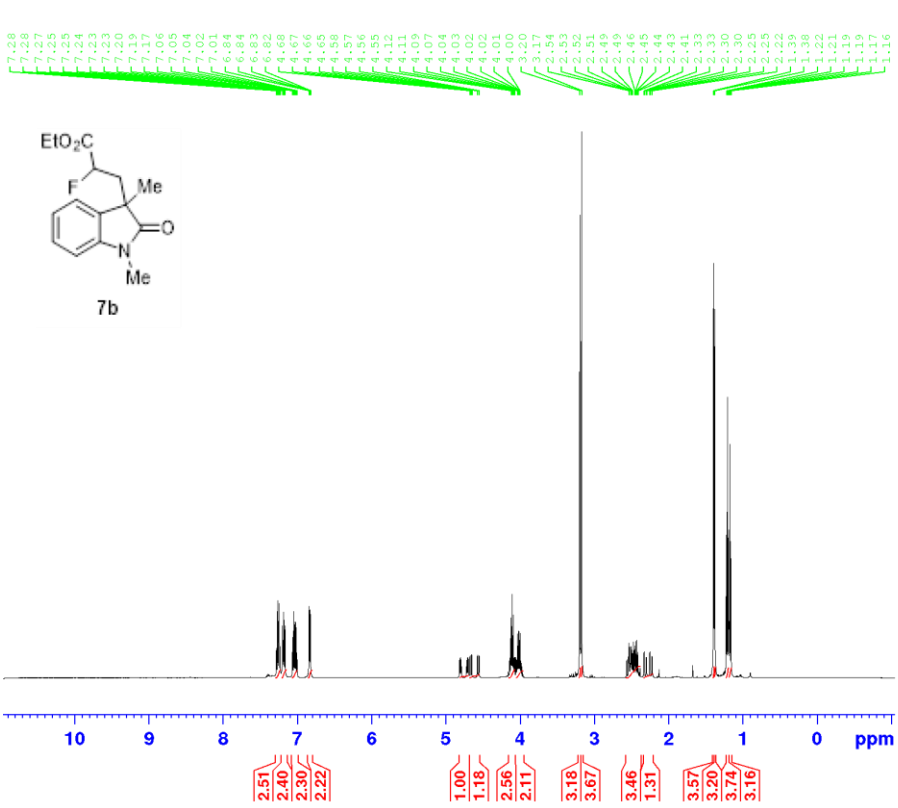
```
Current Data Parameters
NAME         mah-116-13C
EXPNO                 10
PROCNO                 1

F2 - Acquisition Parameters
Date_           20180224
Time              11.33
INSTRUM             spect
PROBHD   5 mm PABBO BB-
PULPROG          zgpg30
TD                 65536
SOLVENT            CDCl3
NS                   512
DS                     4
SWH           30303.031 Hz
FIDRES         0.462388 Hz
AQ            1.0813440 sec
RG                 13000
DW                16.500 usec
DE                10.00 usec
TE                300.0 K
D1            1.00000000 sec
D11           0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                 13C
P1                10.30 usec
PL1       0 dB
PL1W        75.35659027 W
SFO1        125.7703648 MHz

======== CHANNEL f2 ========
CPDPRG[2          waltz16
NUC2                  1H
PCPD2              80.00 usec
PL2                3.00 dB
PL12               17.54 dB
PL13               17.54 dB
PL2W        12.58925438 W
PL12W        0.44258827 W
PL13W        0.44258827 W
SFO2        500.1320005 MHz

F2 - Processing parameters
SI                 32768
SF          125.7577890 MHz
WDW                   EM
SSB       0
LB                 1.00 Hz
GB        0
PC                 1.40
```

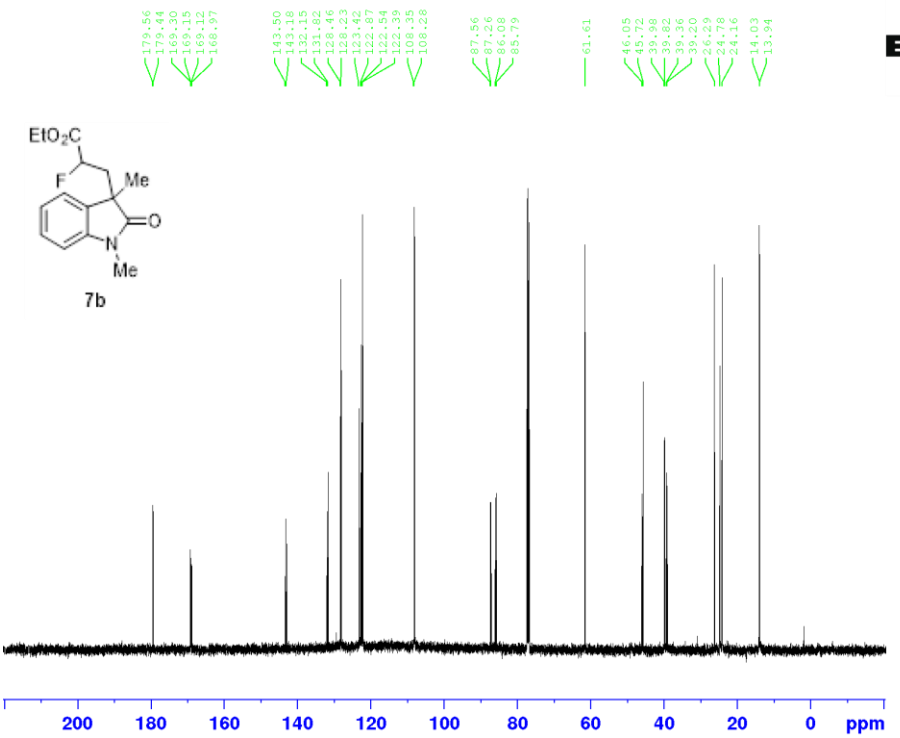


```
Current Data Parameters
NAME                  mah-136-1H
EXPNO                         10
PROCNO                         1

F2 - Acquisition Parameters
Date_                   20180302
Time                        6.32
INSTRUM                    spect
PROBHD    5 mm PATBO BB-
PULPROG                    zg30
TD                        32768
SOLVENT                   CDCl3
NS                          128
DS                            2
SWH                    6009.615 Hz
FIDRES                 0.183399 Hz
AQ                     2.7262976 sec
RG                           64
DW                       83.200 usec
DE                       10.00 usec
TE                       298.0 K
D1                    1.00000000 sec
TD0                            1

======== CHANNEL f1 ========
NUC1                         1H
P1                       14.90 usec
PL1                      -3.00 dB
PL1W                50.11872482 W
SFO1                500.1325007 MHz

F2 - Processing parameters
SI                           16384
SF                  500.1300210 MHz
WDW                             EM
SSB                0
LB                        0.30 Hz
GB                 0
PC                            1.00
```



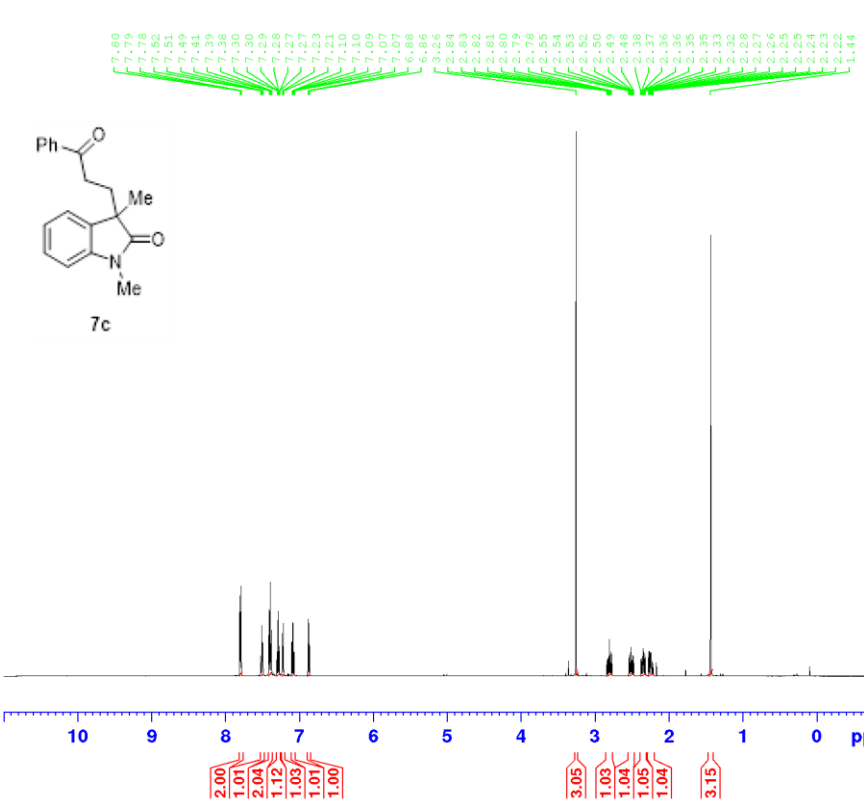
```
Current Data Parameters
NAME                  mah-136-13C
EXPNO                         10
PROCNO                         1

F2 - Acquisition Parameters
Date_                   20180301
Time                        7.54
INSTRUM                    spect
PROBHD    5 mm PABBI 1H/
PULPROG                 zgpg30
TD                        65536
SOLVENT                   CDCl3
NS                          476
DS                            4
SWH                   30303.031 Hz
FIDRES                 0.462388 Hz
AQ                     1.0813440 sec
RG                         14600
DW                       16.500 usec
DE                       10.00 usec
TE                       298.0 K
D1                    1.00000000 sec
D11                   0.03000000 sec
TD0                            1

======== CHANNEL f1 ========
NUC1                        13C
P1                       18.50 usec
PL1                      -0.50 dB
PL1W                84.55149078 W
SFO1                125.7703648 MHz

======== CHANNEL f2 ========
CPDPRG[2                waltz16
NUC2                         1H
PCPD2                    80.00 usec
PL2                       1.00 dB
PL12                     22.54 dB
PL13                     22.54 dB
PL2W                19.95262337 W
PL12W                0.13995871 W
PL13W                0.13995871 W
SFO2                500.1320005 MHz

F2 - Processing parameters
SI                           32768
SF                  125.7577890 MHz
WDW                             EM
SSB                0
LB                        1.00 Hz
GB                 0
PC                            1.40
```



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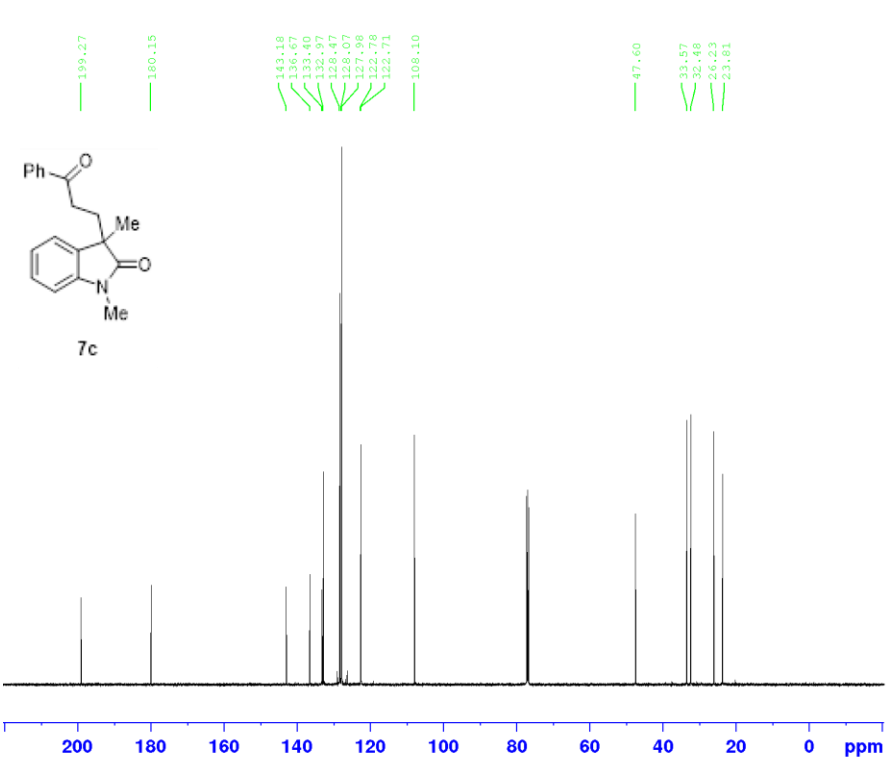
```
Current Data Parameters
NAME                  mah-110-1H
EXPNO                         10
PROCNO                         1

F2 - Acquisition Parameters
Date_                  20180224
Time                      10.24
INSTRUM                   spect
PROBHD    5 mm PABBO BB-
PULPROG                    zg30
TD                        32768
SOLVENT                   CDCl3
NS                           32
DS                            2
SWH                    6009.615 Hz
FIDRES                 0.183399 Hz
AQ                     2.7262976 sec
RG                         50.8
DW                       83.200 usec
DE                       10.00 usec
TE                       298.0 K
D1                    1.00000000 sec
TD0                            1

======== CHANNEL f1 ========
NUC1                          1H
P1                       15.30 usec
PL1                       3.00 dB
PL1W                 12.58925438 W
SFO1                 500.1325007 MHz

F2 - Processing parameters
SI                        16384
SF                   500.1300000 MHz
WDW                          EM
SSB                0
LB                        0.30 Hz
GB                 0
PC                        1.00
```



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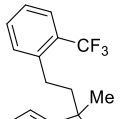
```
Current Data Parameters
NAME                  mah-110-13C
EXPNO                         10
PROCNO                         1

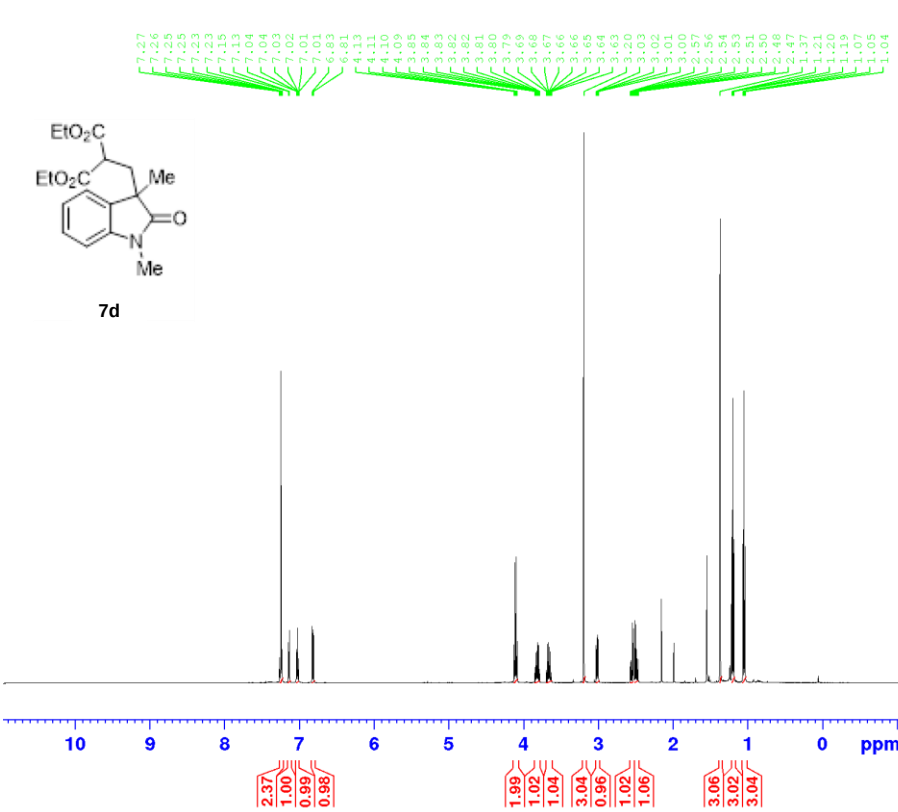
F2 - Acquisition Parameters
Date_                  20180224
Time                      10.43
INSTRUM                   spect
PROBHD    5 mm PABBO BB-
PULPROG                 zgpg30
TD                        65536
SOLVENT                   CDCl3
NS                          512
DS                            4
SWH                30303.031 Hz
FIDRES                 0.462388 Hz
AQ                     1.0813440 sec
RG                        13000
DW                       16.500 usec
DE                       10.00 usec
TE                       298.0 K
D1                    1.00000000 sec
D11                   0.03000000 sec
TD0                            1

======== CHANNEL f1 ========
NUC1                         13C
P1                       10.30 usec
PL1               0 dB
PL1W                 75.35659027 W
SFO1                 125.7703648 MHz

======== CHANNEL f2 ========
CPDPRG[2                waltz16
NUC2                          1H
PCPD2                    80.00 usec
PL2                       3.00 dB
PL12                     17.54 dB
PL13                     17.54 dB
PL2W                 12.58925438 W
PL12W                 0.44258827 W
PL13W                 0.44258827 W
SFO2                 500.1320005 MHz

F2 - Processing parameters
SI                        32768
SF                   125.7577890 MHz
WDW                          EM
SSB                0
LB                        1.00 Hz
GB                 0
PC                        1.40
```



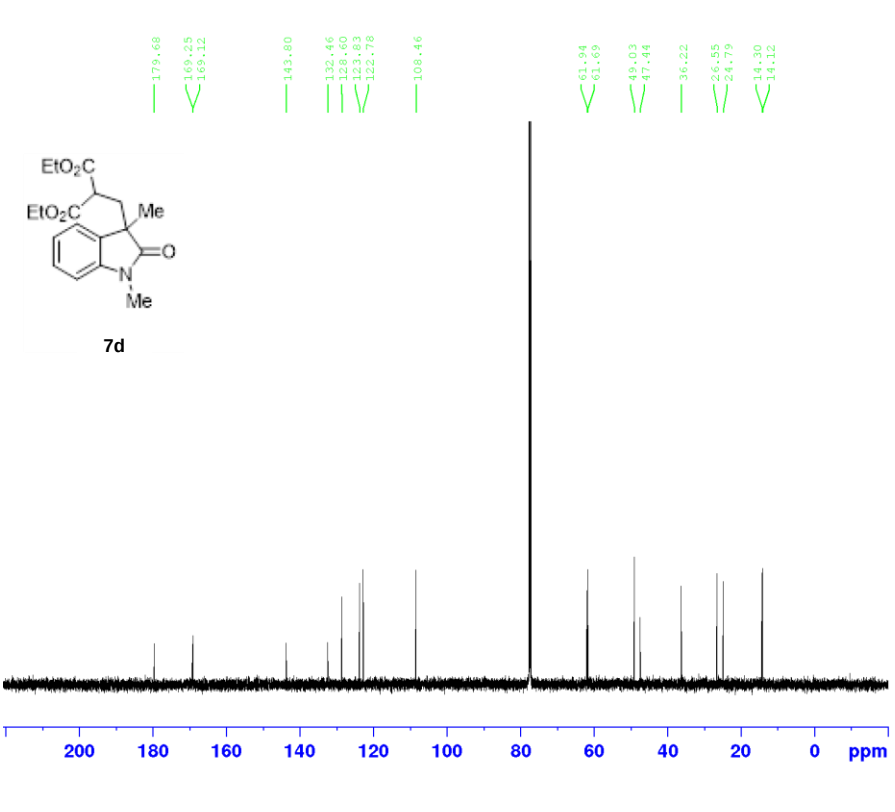
BRUKER

```
Current Data Parameters
NAME                  mah-122-1H
EXPNO                         10
PROCNO                         1

F2 - Acquisition Parameters
Date_                  20180302
Time                      20.04
INSTRUM                   spect
PROBHD    5 mm PATBO BB-
PULPROG                    zg30
TD                        32768
SOLVENT                   CDCl3
NS                          128
DS                            2
SWH                    6009.615 Hz
FIDRES                 0.183399 Hz
AQ                     2.7262976 sec
RG                          645
DW                       83.200 usec
DE                       10.00 usec
TE                       298.0 K
D1                    1.00000000 sec
TD0                            1

======== CHANNEL f1 ========
NUC1                         1H
P1                        14.90 usec
PL1                       -3.00 dB
PL1W                 50.11872482 W
SFO1                500.1325007 MHz

F2 - Processing parameters
SI                         16384
SF                  500.1300210 MHz
WDW                           EM
SSB                0
LB                        0.30 Hz
GB                 0
PC                        1.00
```



BRUKER

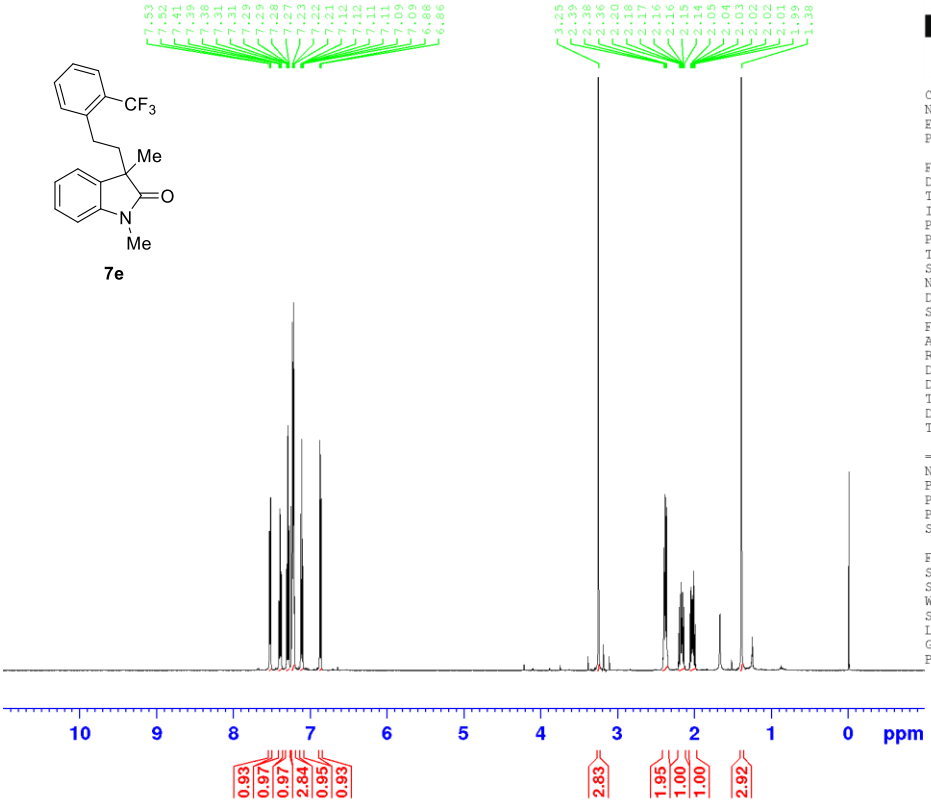
```
Current Data Parameters
NAME                  mah-122-13C
EXPNO                         10
PROCNO                         1

F2 - Acquisition Parameters
Date_                  20180302
Time                       6.20
INSTRUM                   spect
PROBHD    5 mm PATBO BB-
PULPROG                 zgpg30
TD                        65536
SOLVENT                   CDCl3
NS                          512
DS                            4
SWH                  30303.031 Hz
FIDRES                 0.462388 Hz
AQ                     1.0813440 sec
RG                         13000
DW                       16.500 usec
DE                       10.00 usec
TE                       298.5 K
D1                    1.00000000 sec
D11                   0.03000000 sec
TD0                            1

======== CHANNEL f1 ========
NUC1                        13C
P1                         9.75 usec
PL1                       -1.00 dB
PL1W                94.86833191 W
SFO1               125.7703648 MHz

======== CHANNEL f2 ========
CPDPRG[2                waltz16
NUC2                         1H
PCPD2                     80.00 usec
PL2                       -3.00 dB
PL12                      11.96 dB
PL13                      11.96 dB
PL2W                50.11872482 W
PL12W                1.59955800 W
PL13W                1.59955800 W
SFO2               500.1320005 MHz

F2 - Processing parameters
SI                         32768
SF                 125.7577441 MHz
WDW                           EM
SSB                0
LB                        1.00 Hz
GB                 0
PC                        1.40
```



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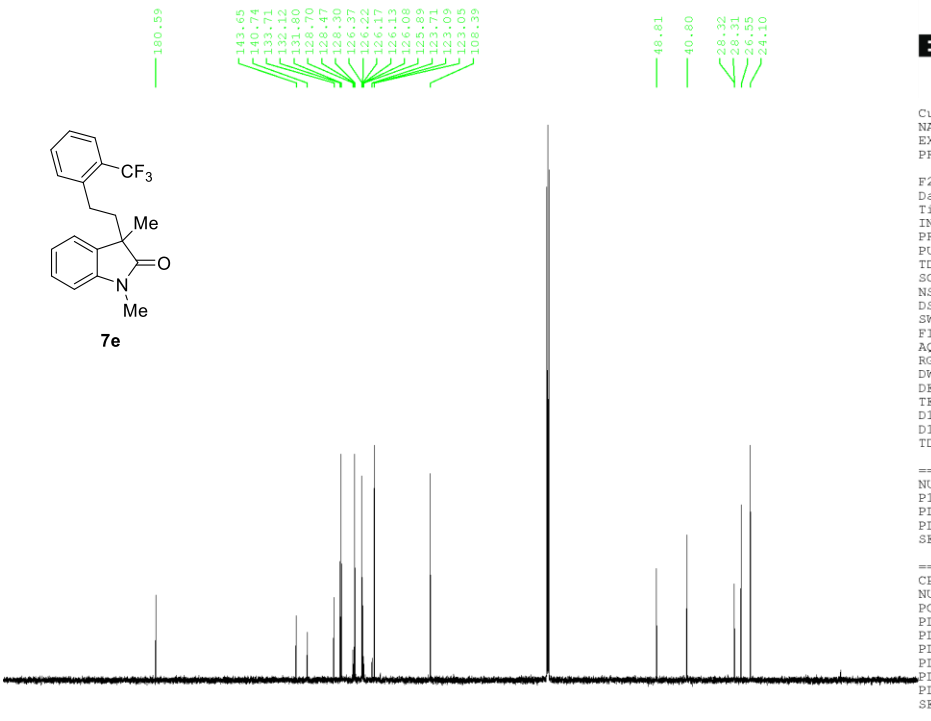
```
Current Data Parameters
NAME         mah-163r-1H
EXPNO                 10
PROCNO                 1

F2 - Acquisition Parameters
Date_           20180807
Time               15.48
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                     8
DS                     2
SWH           10000.000 Hz
FIDRES         0.152588 Hz
AQ            3.2767999 sec
RG                   161
DW              50.000 usec
DE              10.00 usec
TE                298.0 K
D1            1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                13.30 usec
PL1                3.00 dB
PL1W        12.58925438 W
SFO1        500.1330008 MHz

F2 - Processing parameters
SI                 32768
SF          500.1300210 MHz
WDW                   EM
SSB       0
LB                0.30 Hz
GB        0
PC                1.00
```



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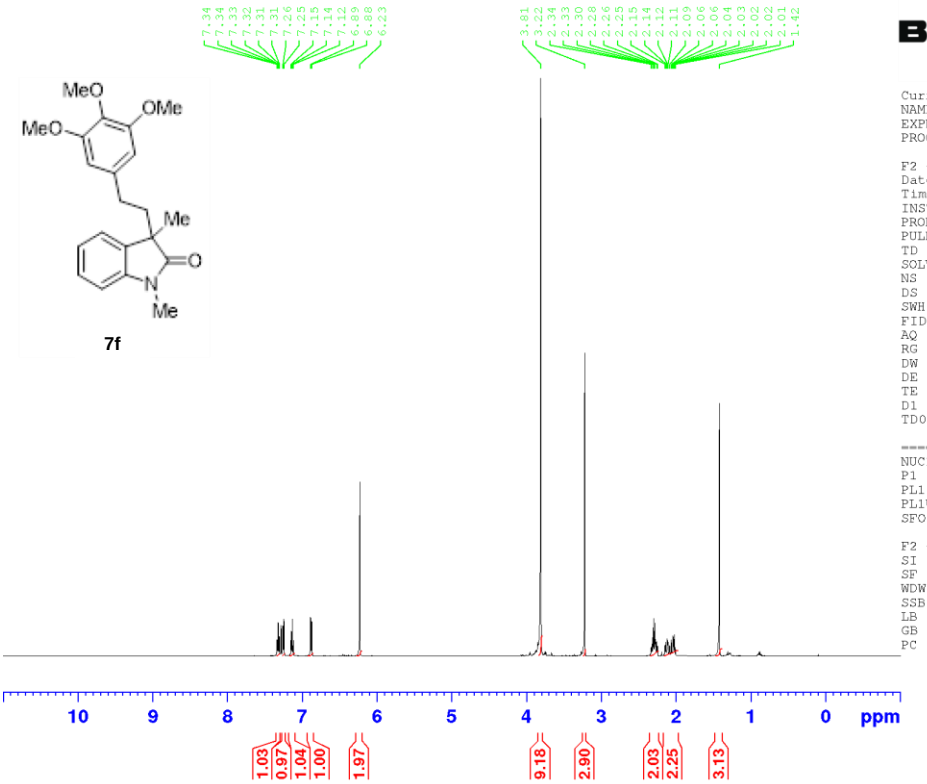
```
Current Data Parameters
NAME         mah-163r-13C
EXPNO                 10
PROCNO                 1

F2 - Acquisition Parameters
Date_           20180807
Time               16.44
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG          zgpg30
TD                 65536
SOLVENT            CDCl3
NS                   512
DS                     4
SWH           30303.031 Hz
FIDRES         0.462388 Hz
AQ            1.0813440 sec
RG                 13000
DW              16.500 usec
DE              10.00 usec
TE                300.0 K
D1            1.00000000 sec
D11           0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                 13C
P1                8.60 usec
PL1       0 dB
PL1W        63.80191422 W
SFO1       125.7703648 MHz

======== CHANNEL f2 ========
CPDPRG[2          waltz16
NUC2                  1H
PCPD2            80.00 usec
PL2               3.00 dB
PL12             18.58 dB
PL13             18.58 dB
PL2W        12.58925438 W
PL12W        0.34833732 W
PL13W        0.34833732 W
SFO2        500.1320005 MHz

F2 - Processing parameters
SI                 32768
SF          125.7577441 MHz
WDW                   EM
SSB       0
LB                1.00 Hz
GB        0
PC                1.40
```



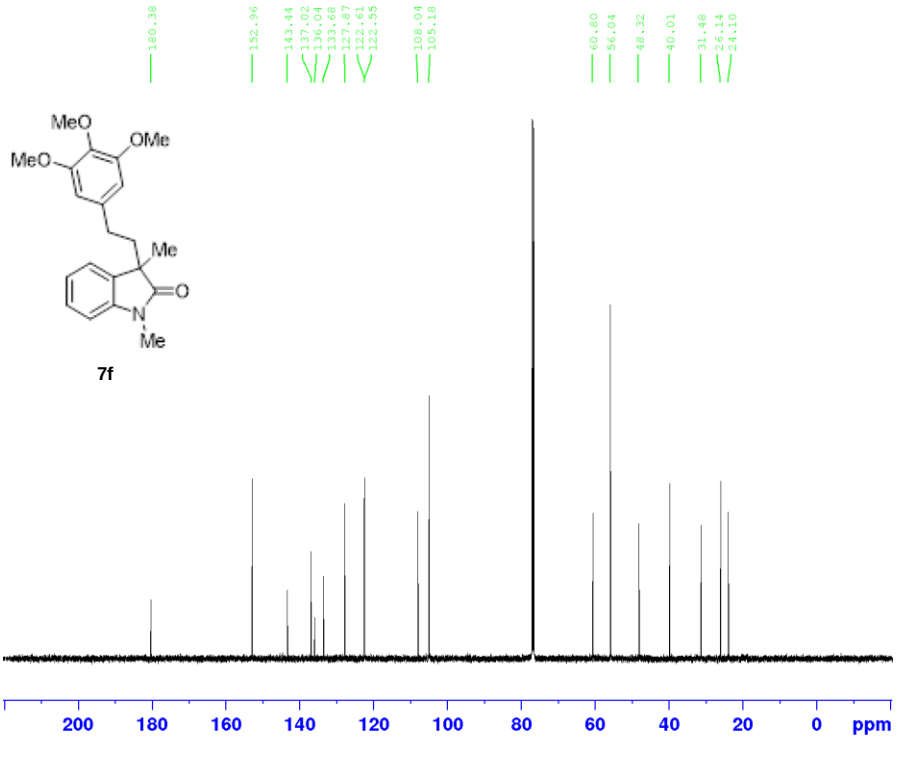
BRUKER

```
Current Data Parameters
NAME          mah-126-1H
EXPNO                 10
PROCNO                 1

F2 - Acquisition Parameters
Date_           20180225
Time               20.38
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG             zg30
TD                 32768
SOLVENT            CDCl3
NS                   128
DS                     2
SWH           6009.615 Hz
FIDRES        0.183399 Hz
AQ            2.7262976 sec
RG                   101
DW               83.200 usec
DE               10.00 usec
TE                295.6 K
D1           1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                15.30 usec
PL1                3.00 dB
PL1W        12.58925438 W
SFO1        500.1325007 MHz

F2 - Processing parameters
SI                 16384
SF          500.1300000 MHz
WDW                   EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```



BRUKER

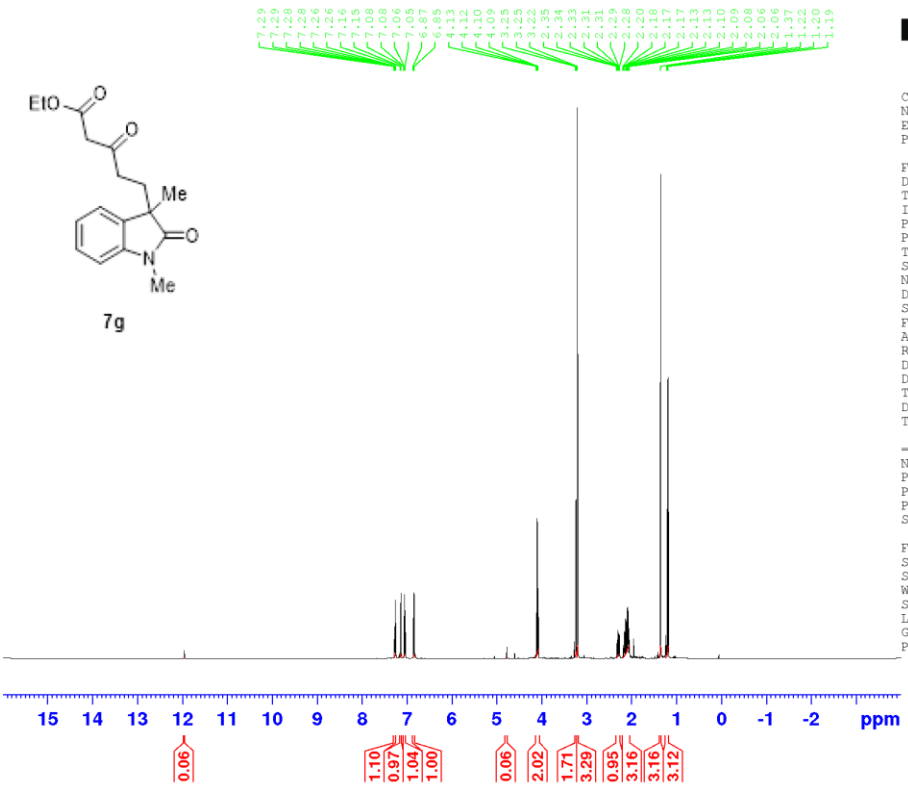
```
Current Data Parameters
NAME         mah-126-13C
EXPNO                 10
PROCNO                 1

F2 - Acquisition Parameters
Date_           20180225
Time               20.57
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG          zgpg30
TD                 65536
SOLVENT            CDCl3
NS                   512
DS                     4
SWH          30303.031 Hz
FIDRES         0.462388 Hz
AQ             1.0813440 sec
RG                 13000
DW               16.500 usec
DE               10.00 usec
TE                296.8 K
D1           1.00000000 sec
D11          0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                 13C
P1                10.30 usec
PL1        0 dB
PL1W        75.35659027 W
SFO1       125.7703648 MHz

======== CHANNEL f2 ========
CPDPRG[2          waltz16
NUC2                  1H
PCPD2             80.00 usec
PL2                3.00 dB
PL12              17.54 dB
PL13              17.54 dB
PL2W        12.58925438 W
PL12W        0.44258827 W
PL13W        0.44258827 W
SFO2        500.1320005 MHz

F2 - Processing parameters
SI                 32768
SF          125.7577890 MHz
WDW                   EM
SSB       0
LB                 1.00 Hz
GB        0
PC                 1.40
```



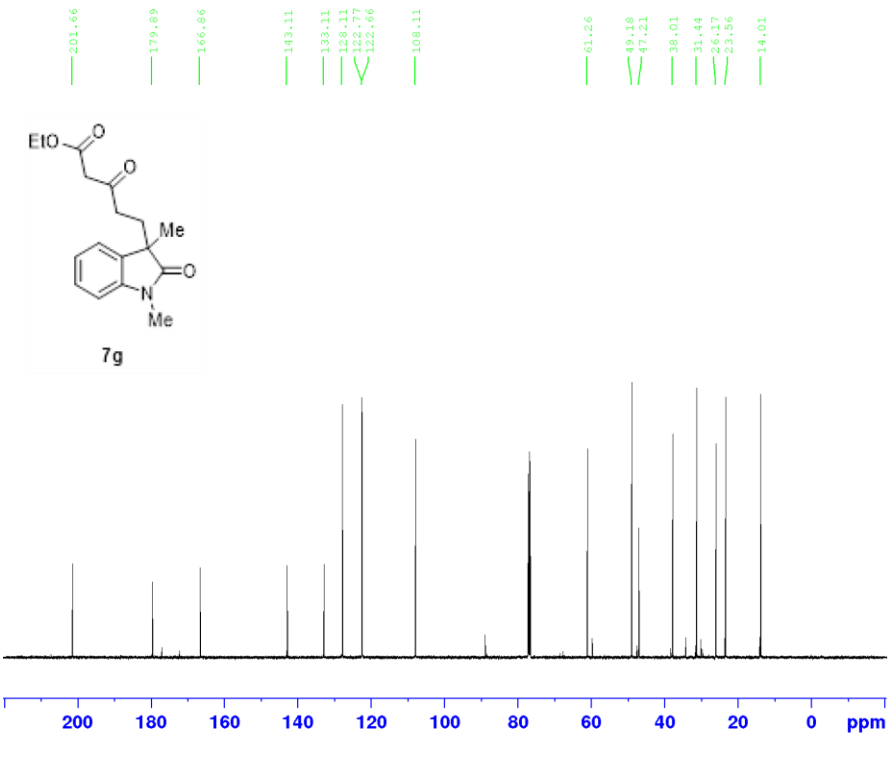
BRUKER

```
Current Data Parameters
NAME          mah-114-1H
EXPNO                 10
PROCNO                 1

F2 - Acquisition Parameters
Date_           20180224
Time               16.57
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                   128
DS                     2
SWH            10000.000 Hz
FIDRES             0.152588 Hz
AQ              3.2767999 sec
RG                  50.8
DW                 50.000 usec
DE                 10.00 usec
TE                 298.0 K
D1             1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                 15.30 usec
PL1                3.00 dB
PL1W          12.58925438 W
SFO1          500.1330008 MHz

F2 - Processing parameters
SI                 32768
SF            500.1300000 MHz
WDW                   EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```



BRUKER

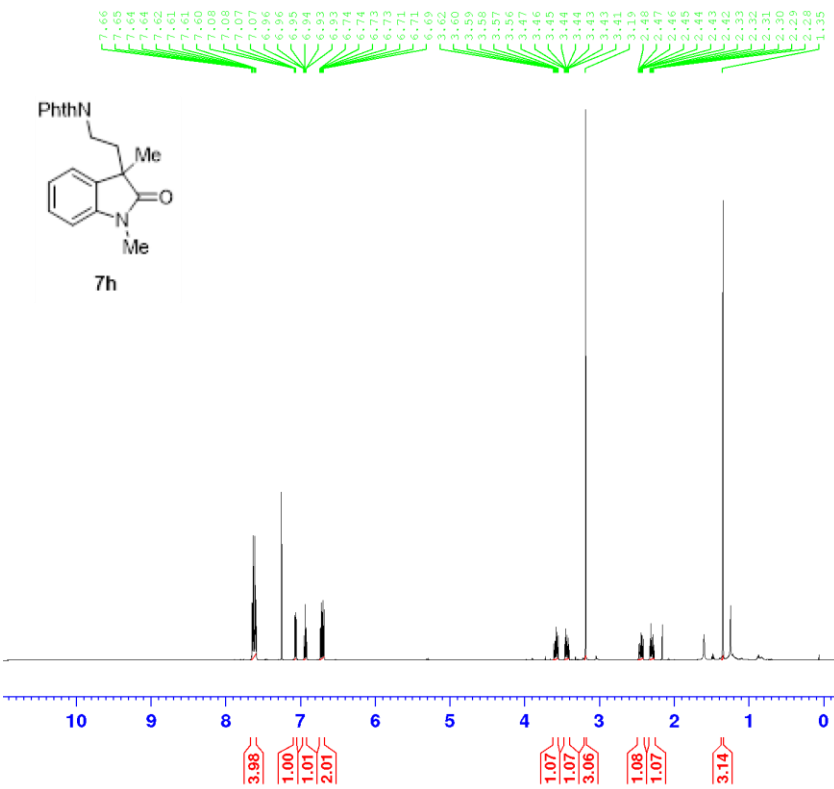
```
Current Data Parameters
NAME          mah-114-13C
EXPNO                 10
PROCNO                 1

F2 - Acquisition Parameters
Date_           20180224
Time               11.08
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG           zgpg30
TD                 65536
SOLVENT            CDCl3
NS                   512
DS                     4
SWH            30303.031 Hz
FIDRES             0.462388 Hz
AQ              1.0813440 sec
RG                 14600
DW                 16.500 usec
DE                 10.00 usec
TE                 298.0 K
D1             1.00000000 sec
D11            0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                 13C
P1                 10.30 usec
PL1       0 dB
PL1W          75.35659027 W
SFO1         125.7703648 MHz

======== CHANNEL f2 ========
CPDPRG[2          waltz16
NUC2                  1H
PCPD2              80.00 usec
PL2                3.00 dB
PL12               17.54 dB
PL13               17.54 dB
PL2W          12.58925438 W
PL12W          0.44258827 W
PL13W          0.44258827 W
SFO2          500.1320005 MHz

F2 - Processing parameters
SI                 32768
SF           125.7577890 MHz
WDW                   EM
SSB       0
LB                 1.00 Hz
GB        0
PC                 1.40
```



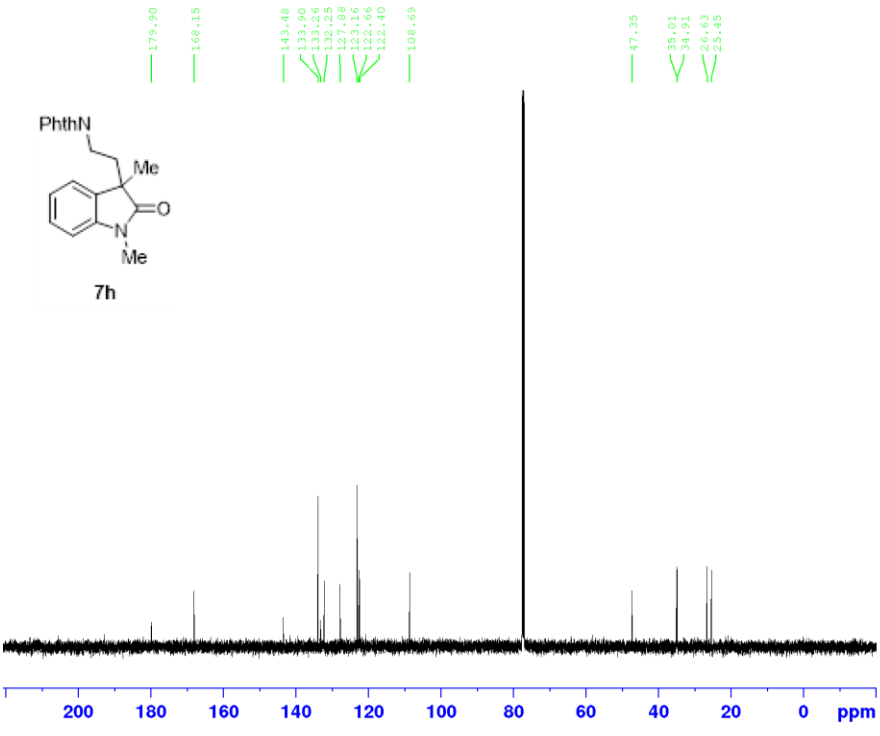
BRUKER

```
Current Data Parameters
NAME                  mah-125-1H
EXPNO                         20
PROCNO                         1

F2 - Acquisition Parameters
Date_                  20180317
Time                       8.47
INSTRUM                   spect
PROBHD    5 mm PATBO BB-
PULPROG                    zg30
TD                        32768
SOLVENT                   CDCl3
NS                          128
DS                            2
SWH                    6009.615 Hz
FIDRES                 0.183399 Hz
AQ                     2.7262976 sec
RG                          575
DW                     83.200 usec
DE                     10.00 usec
TE                     298.0 K
D1                     1.00000000 sec
TD0                            1

======== CHANNEL f1 ========
NUC1                         1H
P1                        14.90 usec
PL1                       -3.00 dB
PL1W                 50.11872482 W
SFO1                500.1325007 MHz

F2 - Processing parameters
SI                         16384
SF                  500.1300140 MHz
WDW                           EM
SSB                0
LB                         0.30 Hz
GB                 0
PC                         1.00
```



BRUKER

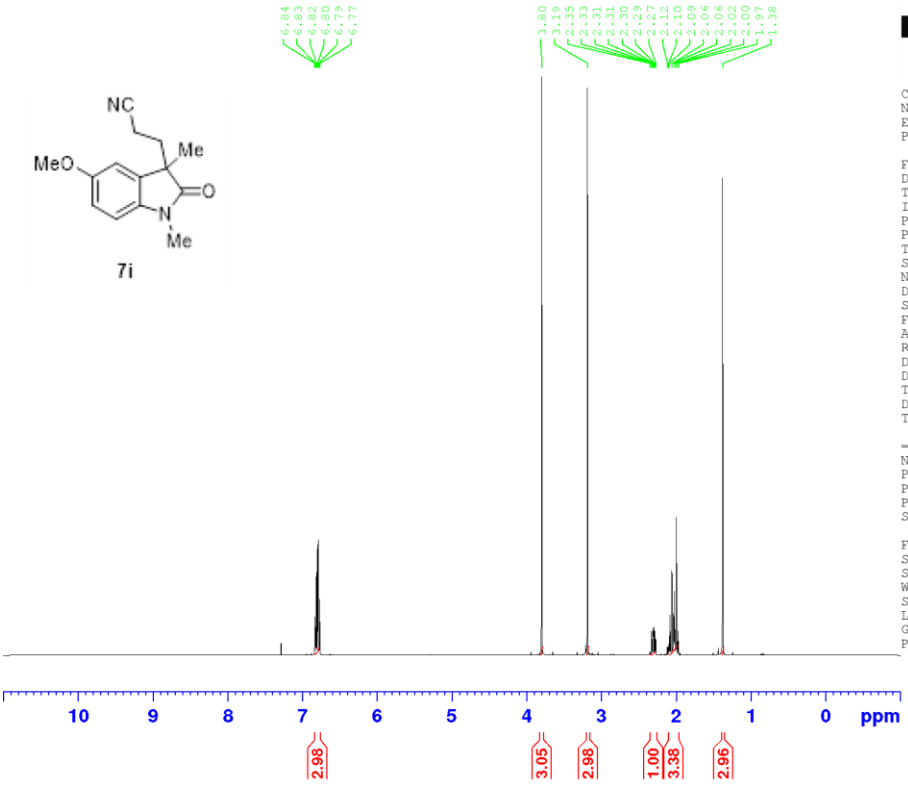
```
Current Data Parameters
NAME                  mah-125-13C
EXPNO                         20
PROCNO                         1

F2 - Acquisition Parameters
Date_                  20180317
Time                       9.06
INSTRUM                   spect
PROBHD    5 mm PATBO BB-
PULPROG                  zgpg30
TD                        65536
SOLVENT                   CDCl3
NS                          512
DS                            4
SWH                30303.031 Hz
FIDRES             0.462388 Hz
AQ                 1.0813440 sec
RG                        11500
DW                     16.500 usec
DE                     10.00 usec
TE                     299.0 K
D1                 1.00000000 sec
D11                0.03000000 sec
TD0                            1

======== CHANNEL f1 ========
NUC1                        13C
P1                         9.75 usec
PL1                       -1.00 dB
PL1W                94.86833191 W
SFO1               125.7703648 MHz

======== CHANNEL f2 ========
CPDPRG[2                waltz16
NUC2                         1H
PCPD2                     80.00 usec
PL2                       -3.00 dB
PL12                      11.96 dB
PL13                      11.96 dB
PL2W                50.11872482 W
PL12W                1.59955800 W
PL13W                1.59955800 W
SFO2                500.1320005 MHz

F2 - Processing parameters
SI                         32768
SF                  125.7577441 MHz
WDW                           EM
SSB                0
LB                         1.00 Hz
GB                 0
PC                         1.40
```



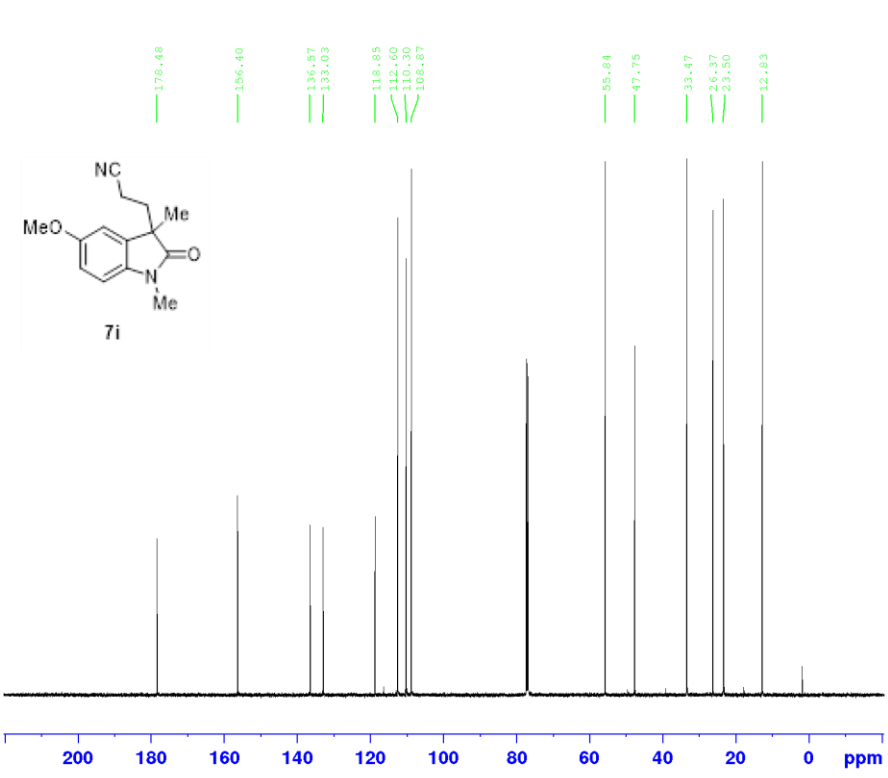
BRUKER

```
Current Data Parameters
NAME                  mah-118-1H
EXPNO                         10
PROCNO                         1

F2 - Acquisition Parameters
Date_                  20180225
Time                      19.37
INSTRUM                   spect
PROBHD    5 mm PABBO BB-
PULPROG                    zg30
TD                        32768
SOLVENT                   CDCl3
NS                          128
DS                            2
SWH                    6009.615 Hz
FIDRES                 0.183399 Hz
AQ                     2.7262976 sec
RG                         50.8
DW                       83.200 usec
DE                       10.00 usec
TE                       295.5 K
D1                    1.00000000 sec
TD0                            1

======== CHANNEL f1 ========
NUC1                          1H
P1                         15.30 usec
PL1                         3.00 dB
PL1W                 12.58925438 W
SFO1                500.1325007 MHz

F2 - Processing parameters
SI                         16384
SF                  500.1300000 MHz
WDW                           EM
SSB                0
LB                         0.30 Hz
GB                 0
PC                         1.00
```



BRUKER

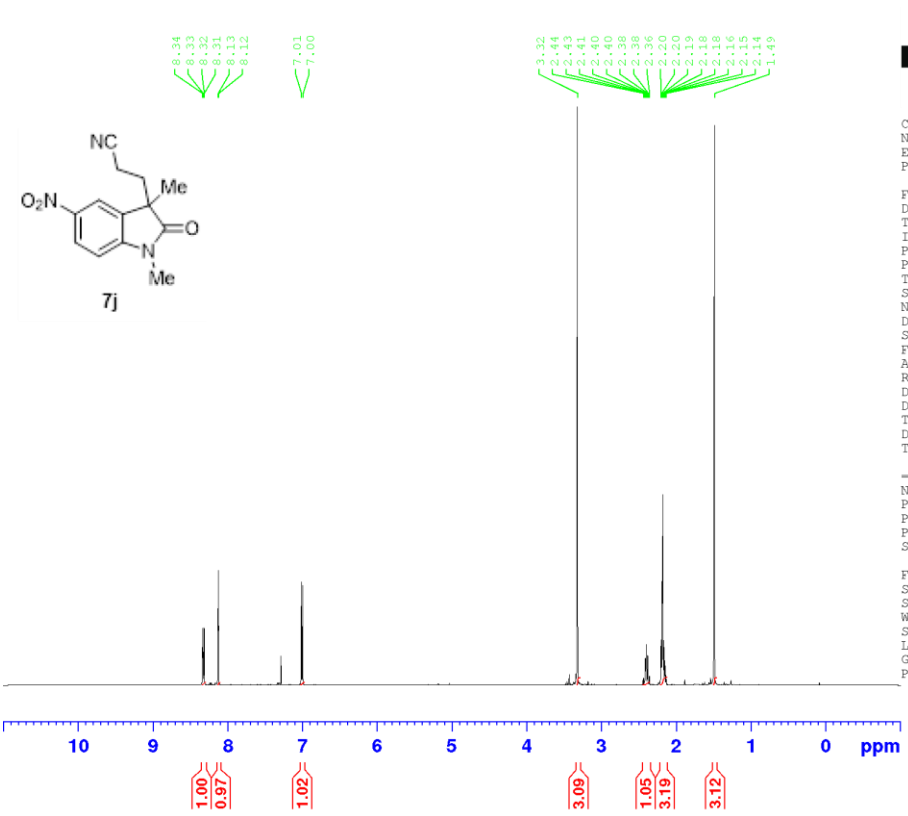
```
Current Data Parameters
NAME                  mah-118-13C
EXPNO                         10
PROCNO                         1

F2 - Acquisition Parameters
Date_                  20180225
Time                      19.56
INSTRUM                   spect
PROBHD    5 mm PABBO BB-
PULPROG                  zgpg30
TD                        65536
SOLVENT                   CDCl3
NS                          512
DS                            4
SWH                30303.031 Hz
FIDRES                 0.462388 Hz
AQ                     1.0813440 sec
RG                         14600
DW                       16.500 usec
DE                       10.00 usec
TE                       296.7 K
D1                    1.00000000 sec
D11                   0.03000000 sec
TD0                            1

======== CHANNEL f1 ========
NUC1                          13C
P1                         10.30 usec
PL1                 0 dB
PL1W                 75.35659027 W
SFO1                125.7703648 MHz

======== CHANNEL f2 ========
CPDPRG[2                waltz16
NUC2                          1H
PCPD2                      80.00 usec
PL2                         3.00 dB
PL12                       17.54 dB
PL13                       17.54 dB
PL2W                 12.58925438 W
PL12W                 0.44258827 W
PL13W                 0.44258827 W
SFO2                500.1320005 MHz

F2 - Processing parameters
SI                         32768
SF                  125.7577890 MHz
WDW                           EM
SSB                0
LB                         1.00 Hz
GB                 0
PC                         1.40
```



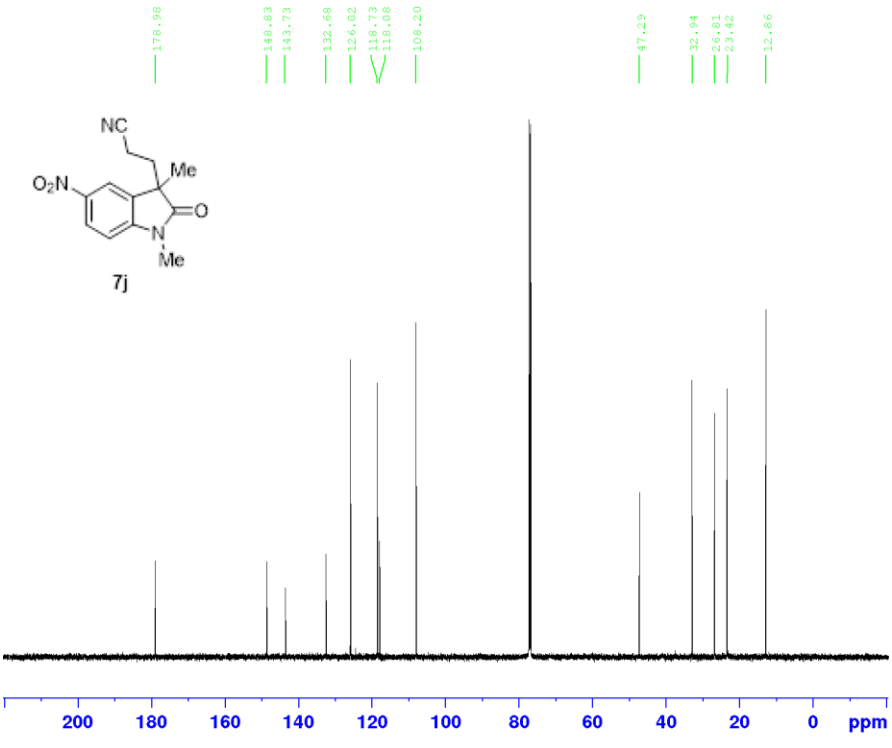
BRUKER

```
Current Data Parameters
NAME                  mah-117-1H
EXPNO                         10
PROCNO                         1

F2 - Acquisition Parameters
Date_                  20180224
Time                      11.38
INSTRUM                   spect
PROBHD     5 mm PABBO BB-
PULPROG                    zg30
TD                        32768
SOLVENT                   CDCl3
NS                           32
DS                            2
SWH                    6009.615 Hz
FIDRES                 0.183399 Hz
AQ                     2.7262976 sec
RG                          114
DW                       83.200 usec
DE                       10.00 usec
TE                       298.0 K
D1                     1.00000000 sec
TD0                            1

======== CHANNEL f1 ========
NUC1                          1H
P1                         15.30 usec
PL1                         3.00 dB
PL1W                 12.58925438 W
SFO1                500.1325007 MHz

F2 - Processing parameters
SI                         16384
SF                  500.1300000 MHz
WDW                           EM
SSB                0
LB                         0.30 Hz
GB                 0
PC                         1.00
```



BRUKER

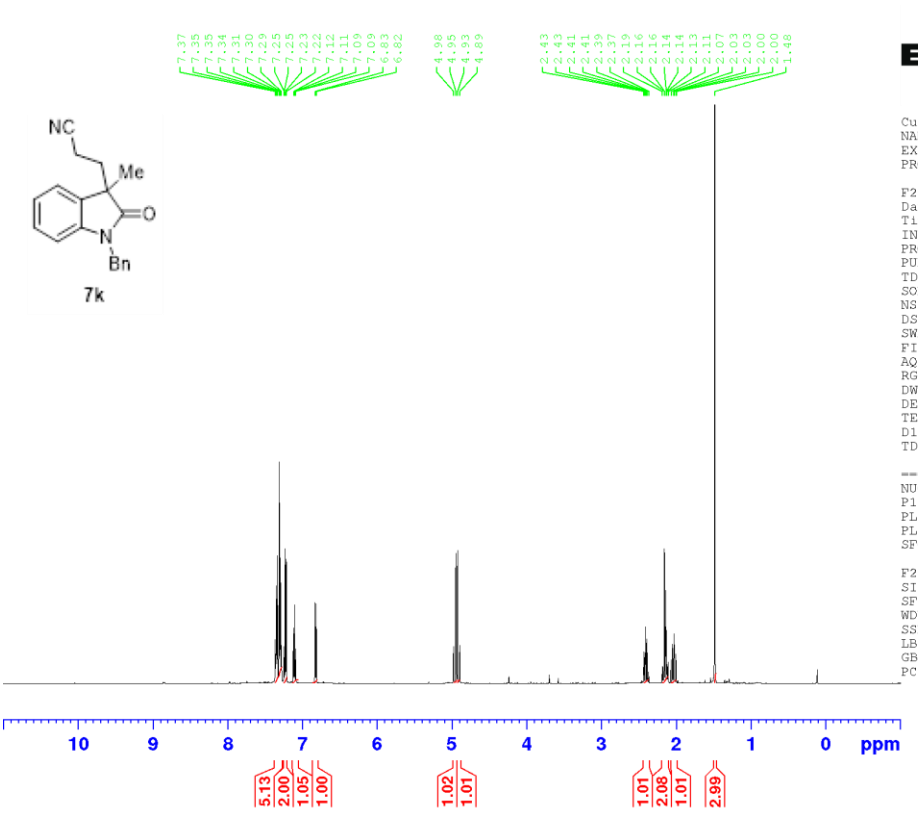
```
Current Data Parameters
NAME                  mah-117-13C
EXPNO                         10
PROCNO                         1

F2 - Acquisition Parameters
Date_                  20180224
Time                      11.57
INSTRUM                   spect
PROBHD     5 mm PABBO BB-
PULPROG                 zgpg30
TD                        65536
SOLVENT                   CDCl3
NS                          512
DS                            4
SWH                30303.031 Hz
FIDRES             0.462388 Hz
AQ                 1.0813440 sec
RG                        14600
DW                       16.500 usec
DE                       10.00 usec
TE                       297.9 K
D1                     1.00000000 sec
D11                    0.03000000 sec
TD0                            1

======== CHANNEL f1 ========
NUC1                         13C
P1                         10.30 usec
PL1                 0 dB
PL1W                75.35659027 W
SFO1               125.7703648 MHz

======== CHANNEL f2 ========
CPDPRG[2                waltz16
NUC2                          1H
PCPD2                      80.00 usec
PL2                         3.00 dB
PL12                       17.54 dB
PL13                       17.54 dB
PL2W                 12.58925438 W
PL12W                 0.44258827 W
PL13W                 0.44258827 W
SFO2                500.1320005 MHz

F2 - Processing parameters
SI                         32768
SF                  125.7577890 MHz
WDW                           EM
SSB                0
LB                         1.00 Hz
GB                 0
PC                         1.40
```



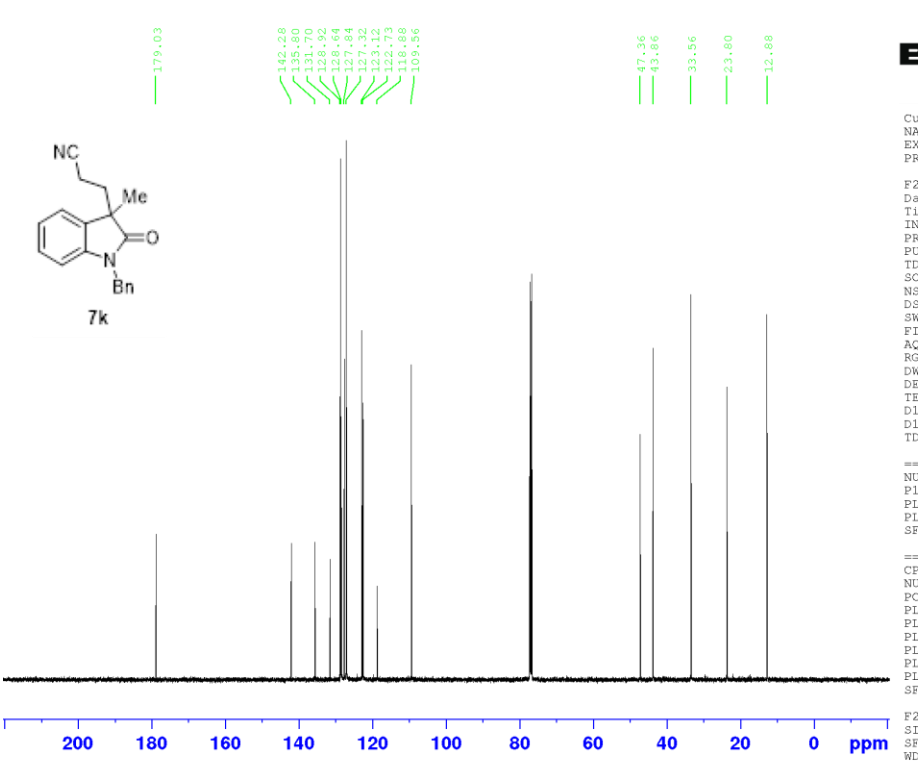
BRUKER

```
Current Data Parameters
NAME          mah-120-1H
EXPNO                 10
PROCNO                 1

F2 - Acquisition Parameters
Date_           20180224
Time               18.30
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG             zg30
TD                 32768
SOLVENT            CDCl3
NS                   128
DS                     2
SWH             6009.615 Hz
FIDRES          0.183399 Hz
AQ              2.7262976 sec
RG                  50.8
DW                83.200 usec
DE                10.00 usec
TE                298.0 K
D1             1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                 15.30 usec
PL1                 3.00 dB
PL1W          12.58925438 W
SFO1          500.1325007 MHz

F2 - Processing parameters
SI                 16384
SF            500.1300000 MHz
WDW                   EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```



BRUKER

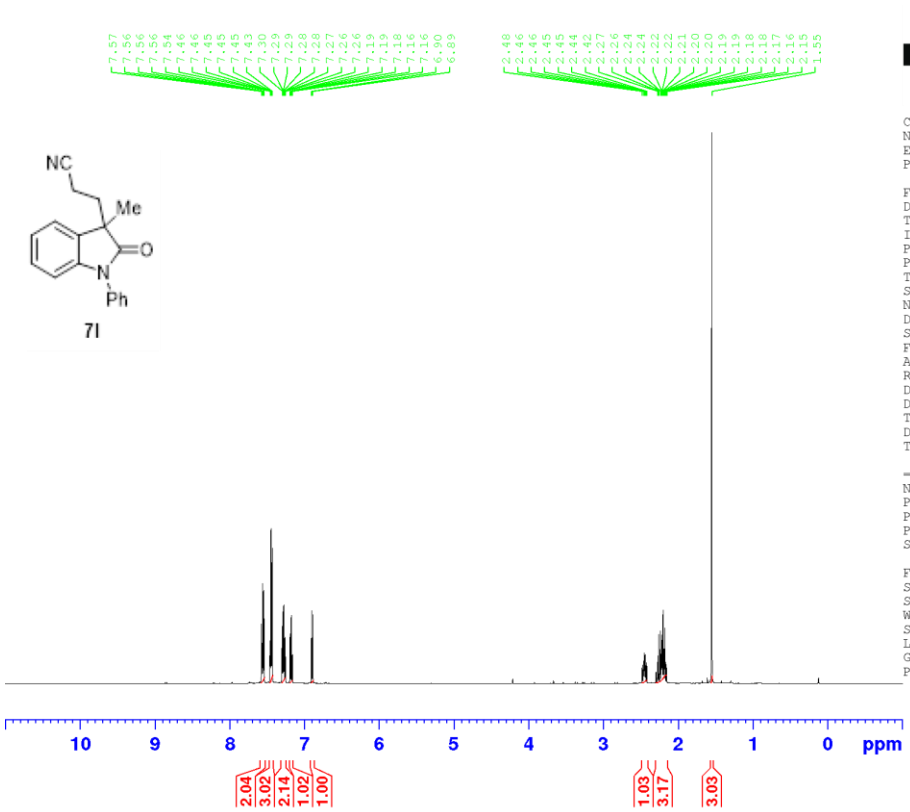
```
Current Data Parameters
NAME          mah-120-13C
EXPNO                 10
PROCNO                 1

F2 - Acquisition Parameters
Date_           20180224
Time               18.49
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG          zgpg30
TD                 65536
SOLVENT            CDCl3
NS                   512
DS                     4
SWH            30303.031 Hz
FIDRES          0.462388 Hz
AQ              1.0813440 sec
RG                 14600
DW                16.500 usec
DE                10.00 usec
TE                297.9 K
D1             1.00000000 sec
D11            0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                 13C
P1                 10.30 usec
PL1          0 dB
PL1W          75.35659027 W
SFO1          125.7703648 MHz

======== CHANNEL f2 ========
CPDPRG[2          waltz16
NUC2                  1H
PCPD2              80.00 usec
PL2                 3.00 dB
PL12               17.54 dB
PL13               17.54 dB
PL2W          12.58925438 W
PL12W          0.44258827 W
PL13W          0.44258827 W
SFO2          500.1320005 MHz

F2 - Processing parameters
SI                 32768
SF            125.7577890 MHz
WDW                   EM
SSB       0
LB                 1.00 Hz
GB        0
PC                 1.40
```

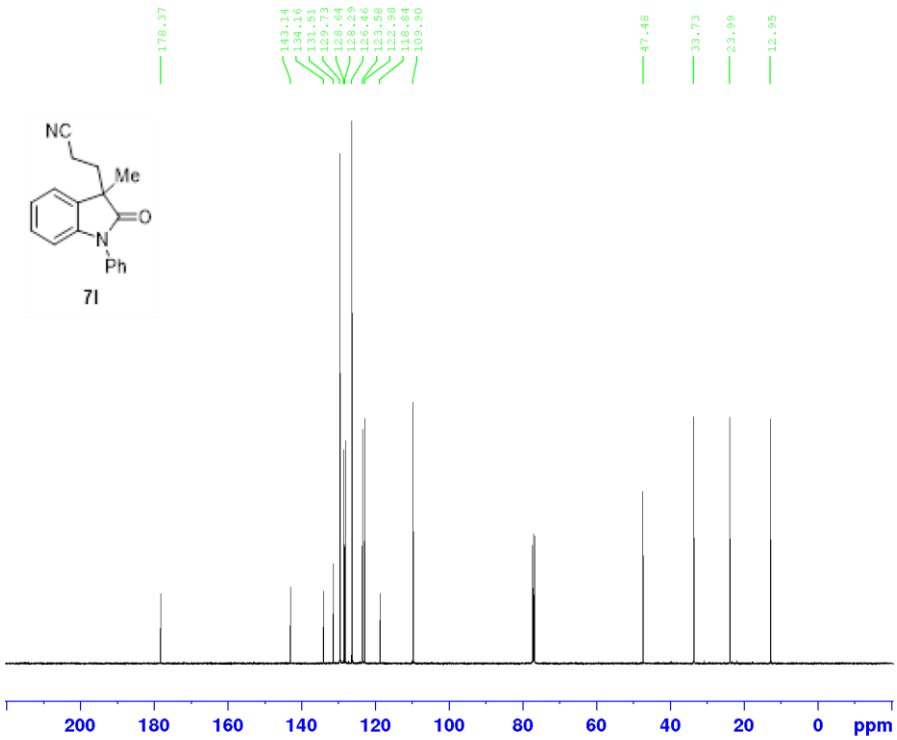


```
Current Data Parameters
NAME                  mah-124-1H
EXPNO                         10
PROCNO                         1

F2 - Acquisition Parameters
Date_                  20180225
Time                      19.05
INSTRUM                   spect
PROBHD     5 mm PABBO BB-
PULPROG                    zg30
TD                        32768
SOLVENT                   CDCl3
NS                          128
DS                            2
SWH                    6009.615 Hz
FIDRES                 0.183399 Hz
AQ                     2.7262976 sec
RG                         50.8
DW                       83.200 usec
DE                       10.00 usec
TE                       295.5 K
D1                     1.00000000 sec
TD0                            1

======== CHANNEL f1 ========
NUC1                          1H
P1                       15.30 usec
PL1                       3.00 dB
PL1W                12.58925438 W
SFO1                500.1325007 MHz

F2 - Processing parameters
SI                         16384
SF                  500.1300000 MHz
WDW                           EM
SSB                0
LB                        0.30 Hz
GB                 0
PC                        1.00
```



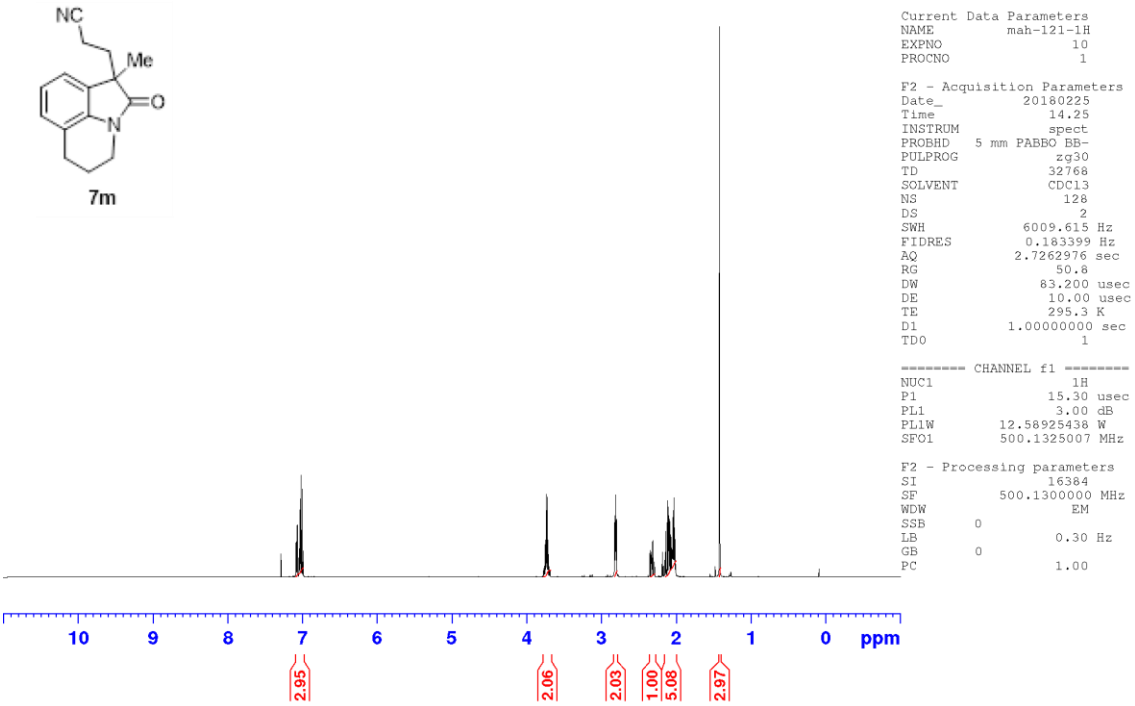
```
Current Data Parameters
NAME                  mah-124-13C
EXPNO                         10
PROCNO                         1

F2 - Acquisition Parameters
Date_                  20180225
Time                      19.25
INSTRUM                   spect
PROBHD     5 mm PABBO BB-
PULPROG                 zgpg30
TD                        65536
SOLVENT                   CDCl3
NS                          512
DS                            4
SWH                   30303.031 Hz
FIDRES                 0.462388 Hz
AQ                     1.0813440 sec
RG                         13000
DW                       16.500 usec
DE                       10.00 usec
TE                       296.6 K
D1                     1.00000000 sec
D11                    0.03000000 sec
TD0                            1

======== CHANNEL f1 ========
NUC1                          13C
P1                       10.30 usec
PL1                 0 dB
PL1W                75.35659027 W
SFO1               125.7703648 MHz

======== CHANNEL f2 ========
CPDPRG[2                waltz16
NUC2                          1H
PCPD2                    80.00 usec
PL2                       3.00 dB
PL12                     17.54 dB
PL13                     17.54 dB
PL2W                12.58925438 W
PL12W                0.44258827 W
PL13W                0.44258827 W
SFO2                500.1320005 MHz

F2 - Processing parameters
SI                         32768
SF                  125.7577890 MHz
WDW                           EM
SSB                0
LB                        1.00 Hz
GB                 0
PC                        1.40
```



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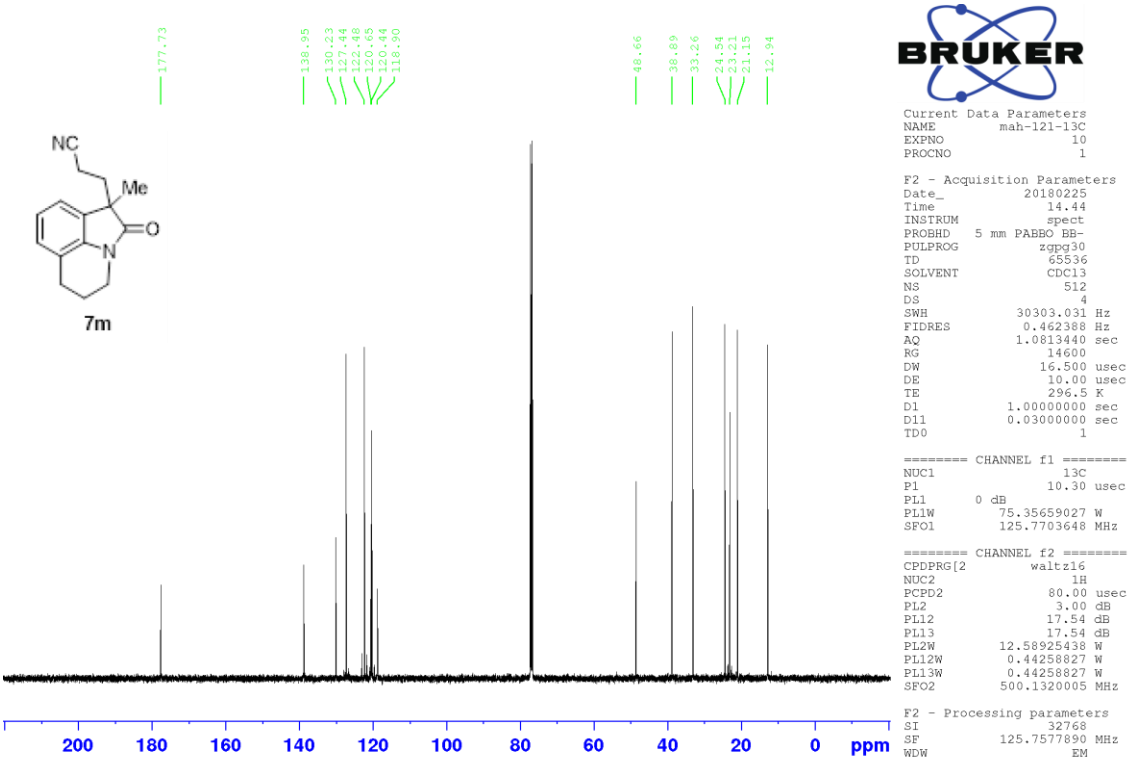
```
Current Data Parameters
NAME                  mah-121-1H
EXPNO                         10
PROCNO                         1

F2 - Acquisition Parameters
Date_                  20180225
Time                      14.25
INSTRUM                   spect
PROBHD    5 mm PABBO BB-
PULPROG                    zg30
TD                        32768
SOLVENT                   CDCl3
NS                          128
DS                            2
SWH                    6009.615 Hz
FIDRES                 0.183399 Hz
AQ                     2.7262976 sec
RG                         50.8
DW                       83.200 usec
DE                       10.00 usec
TE                        295.3 K
D1                     1.00000000 sec
TD0                            1

======== CHANNEL f1 ========
NUC1                          1H
P1                         15.30 usec
PL1                         3.00 dB
PL1W                12.58925438 W
SFO1                500.1325007 MHz

F2 - Processing parameters
SI                         16384
SF                  500.1300000 MHz
WDW                           EM
SSB                0
LB                         0.30 Hz
GB                 0
PC                         1.00
```



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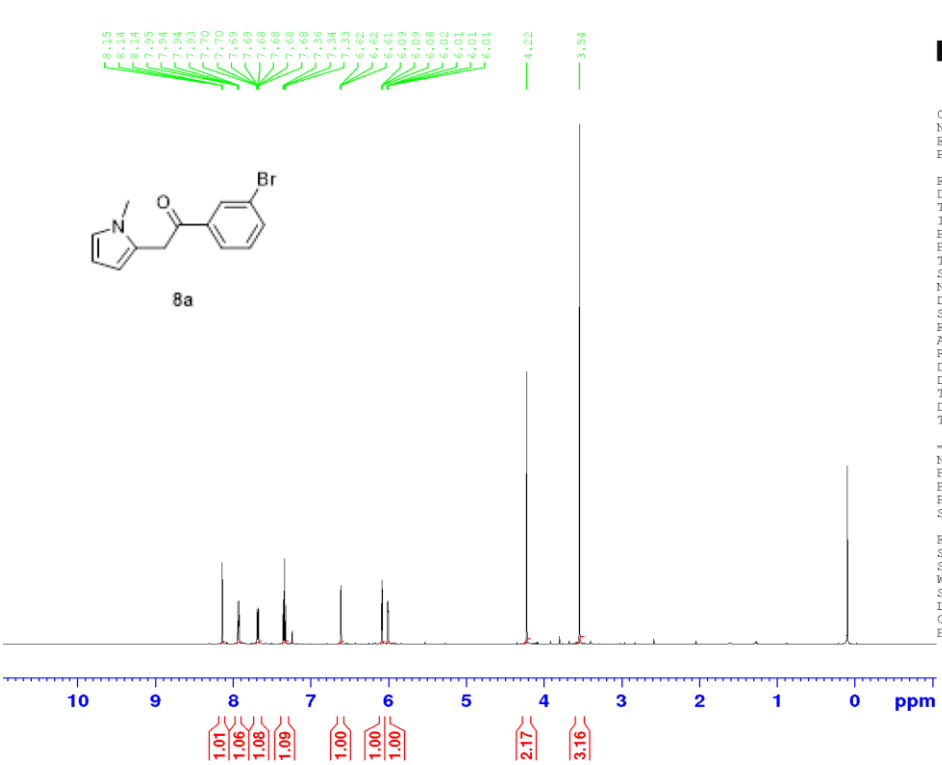
```
Current Data Parameters
NAME                  mah-121-13C
EXPNO                         10
PROCNO                         1

F2 - Acquisition Parameters
Date_                  20180225
Time                      14.44
INSTRUM                   spect
PROBHD    5 mm PABBO BB-
PULPROG                  zgpg30
TD                        65536
SOLVENT                   CDCl3
NS                          512
DS                            4
SWH                  30303.031 Hz
FIDRES                 0.462388 Hz
AQ                     1.0813440 sec
RG                         14600
DW                       16.500 usec
DE                       10.00 usec
TE                        296.5 K
D1                     1.00000000 sec
D11                    0.03000000 sec
TD0                            1

======== CHANNEL f1 ========
NUC1                         13C
P1                         10.30 usec
PL1                 0 dB
PL1W                75.35659027 W
SFO1                125.7703648 MHz

======== CHANNEL f2 ========
CPDPRG[2                waltz16
NUC2                          1H
PCPD2                      80.00 usec
PL2                         3.00 dB
PL12                       17.54 dB
PL13                       17.54 dB
PL2W                12.58925438 W
PL12W                0.44258827 W
PL13W                0.44258827 W
SFO2                500.1320005 MHz

F2 - Processing parameters
SI                         32768
SF                  125.7577890 MHz
WDW                           EM
SSB                0
LB                         1.00 Hz
GB                 0
PC                         1.40
```

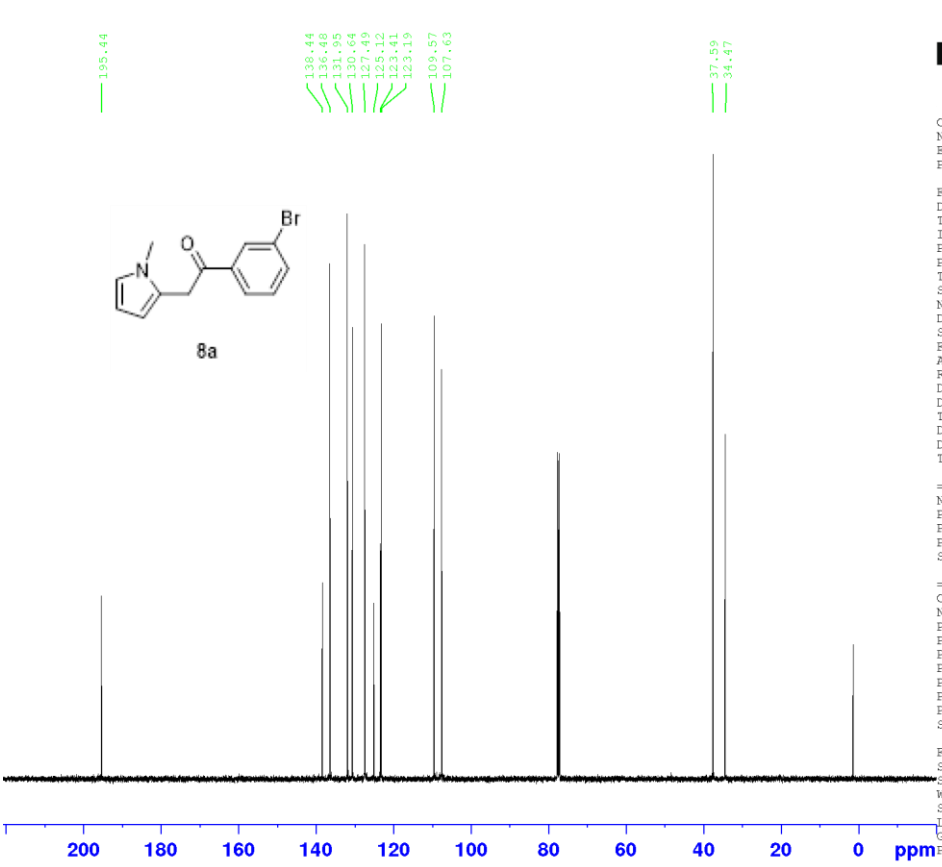



```
Current Data Parameters
NAME        BSCB-387 col
EXPNO                10
PROCNO                1

F2 - Acquisition Parameters
Date_          20180405
Time              16.05
INSTRUM           spect
PROBHD   5 mm PATBO BB-
PULPROG            zg30
TD                32768
SOLVENT           CDCl3
NS                    8
DS                    2
SWH            6009.615 Hz
FIDRES         0.183399 Hz
AQ             2.7262976 sec
RG                 71.8
DW                83.200 usec
DE                10.00 usec
TE                298.0 K
D1             1.00000000 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                  1H
P1                 14.90 usec
PL1                -3.00 dB
PL1W         50.11872482 W
SFO1         500.1325007 MHz

F2 - Processing parameters
SI                16384
SF          500.1300210 MHz
WDW                  EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```



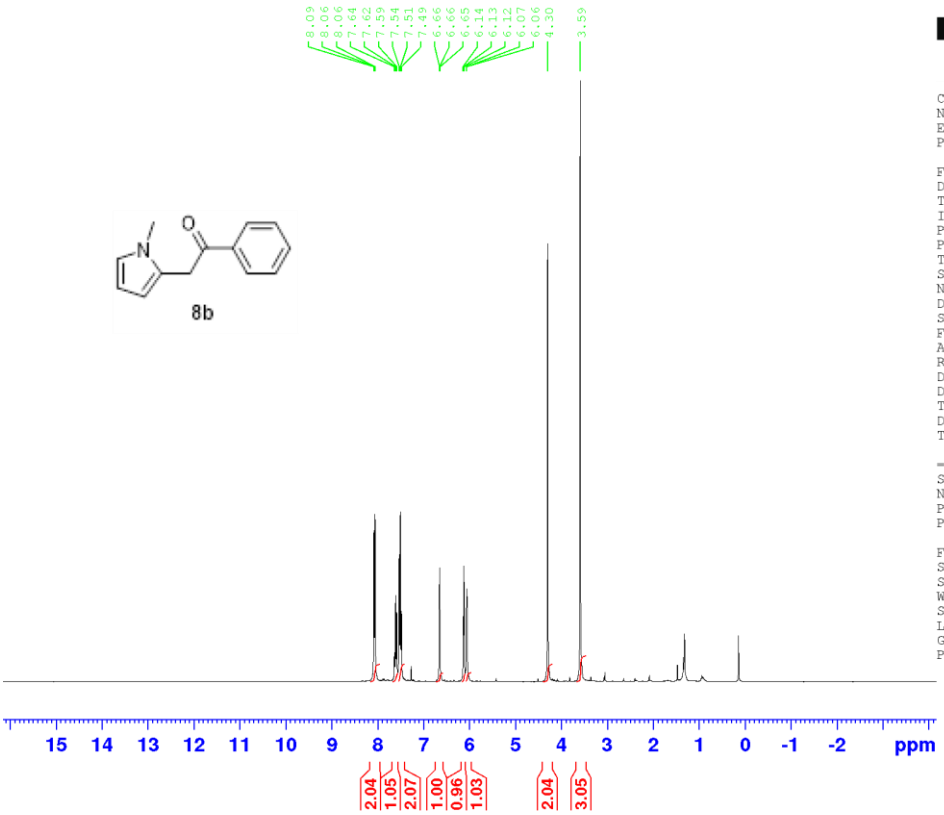

```
Current Data Parameters
NAME        BSCB-387 col
EXPNO                11
PROCNO                1

F2 - Acquisition Parameters
Date_          20180405
Time              17.20
INSTRUM           spect
PROBHD   5 mm PATBO BB-
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                  325
DS                    4
SWH           30303.031 Hz
FIDRES         0.462388 Hz
AQ             1.0813440 sec
RG                11500
DW               16.500 usec
DE                10.00 usec
TE                300.0 K
D1             1.00000000 sec
D11            0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                 13C
P1                 9.75 usec
PL1               -1.00 dB
PL1W         94.86833191 W
SFO1        125.7703648 MHz

======== CHANNEL f2 ========
CPDPRG[2          waltz16
NUC2                  1H
PCPD2              80.00 usec
PL2                -3.00 dB
PL12               11.96 dB
PL13               11.96 dB
PL2W         50.11872482 W
PL12W         1.59955800 W
PL13W         1.59955800 W
SFO2        500.1320005 MHz

F2 - Processing parameters
SI                32768
SF          125.7577441 MHz
WDW                  EM
SSB       0
LB                 1.00 Hz
GB        0
PC                 1.40
```



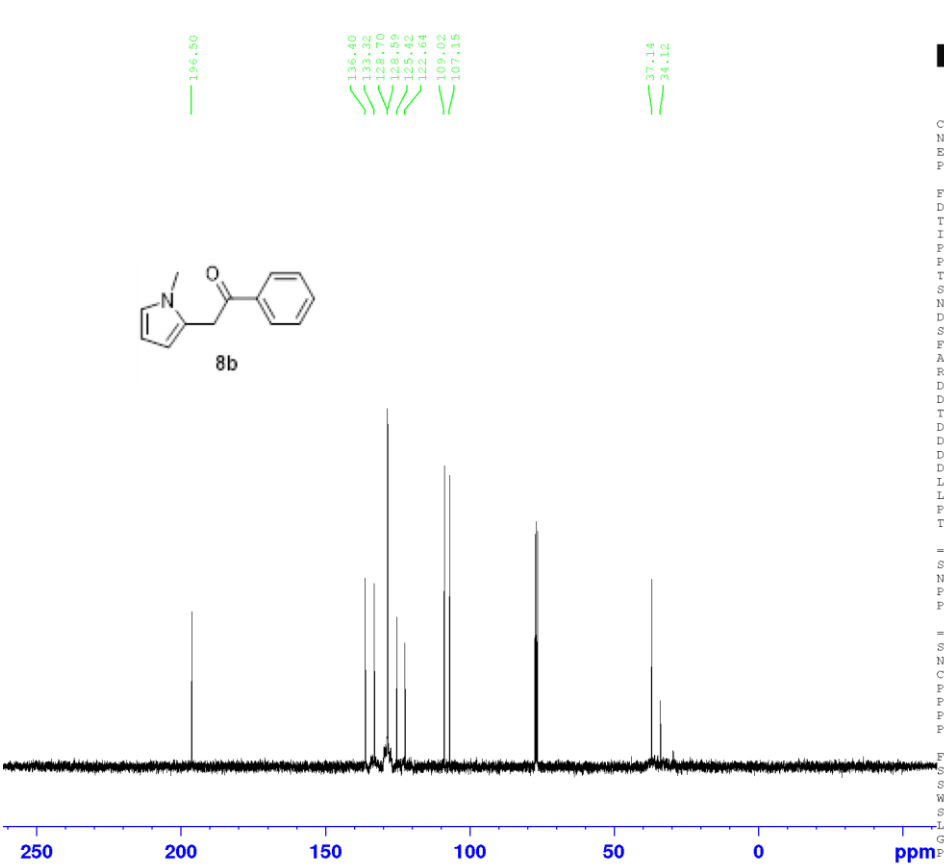
BRUKER

```
Current Data Parameters
NAME        BSCB-318 col
EXPNO                 10
PROCNO                 1

F2 - Acquisition Parameters
Date_           20180221
Time               20.01
INSTRUM       FOURIER300
PROBHD    5 mm DUL 13C-1
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                     8
DS                     2
SWH             6103.516 Hz
FIDRES           0.093132 Hz
AQ               5.3687091 sec
RG                11.0404
DW                81.920 usec
DE                 6.50 usec
TE                298.0 K
D1             1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1          300.1818011 MHz
NUC1                  1H
P1                 11.75 usec
PLW1          20.00000000 W

F2 - Processing parameters
SI                 32768
SF            300.1800000 MHz
WDW                   EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.40
```



BRUKER

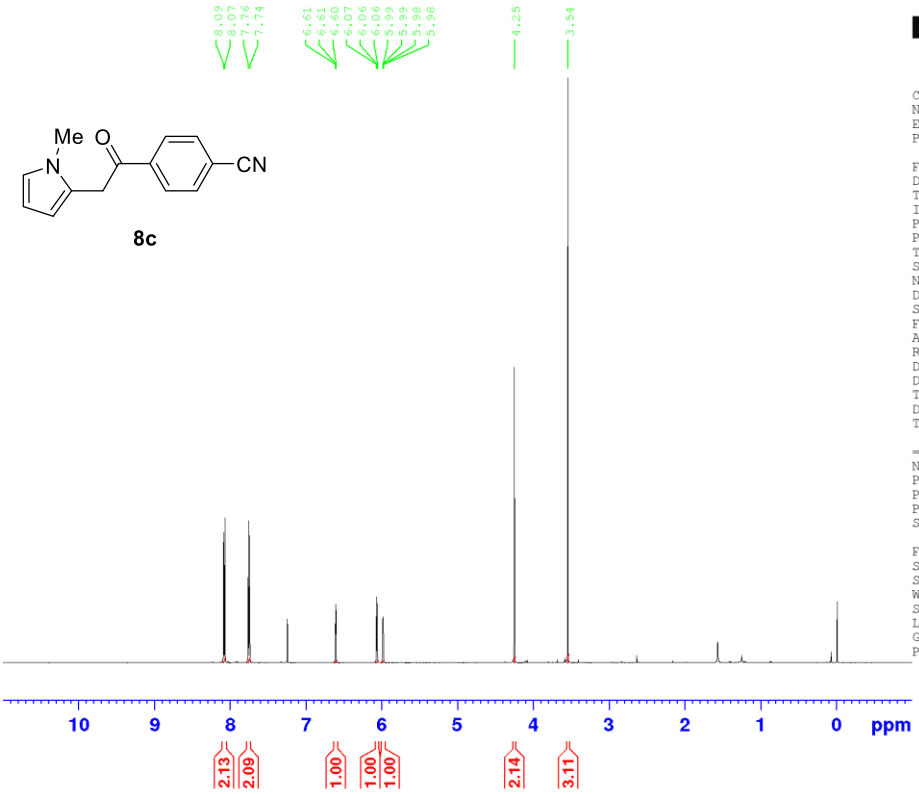
```
Current Data Parameters
NAME        BSCB-318 col
EXPNO                 11
PROCNO                 1

F2 - Acquisition Parameters
Date_           20180222
Time                3.52
INSTRUM       FOURIER300
PROBHD    5 mm DUL 13C-1
PULPROG          zgpg30
TD                 65536
SOLVENT            CDCl3
NS                   512
DS                     4
SWH            24414.063 Hz
FIDRES           0.372529 Hz
AQ               1.3421773 sec
RG                501.187
DW                20.480 usec
DE                 6.50 usec
TE                298.1 K
D1             1.00000000 sec
D11            0.03000000 sec
D31            0.00001050 sec
D40            0.01641260 sec
L4                    45
L5                    32
P32               80.00 usec
TD0                    1

======== CHANNEL f1 ========
SFO1          75.4878688 MHz
NUC1                  13C
P1                10.50 usec
PLW1          50.00000000 W

======== CHANNEL f2 ========
SFO2         300.1812007 MHz
NUC2                  1H
CPDPRG[2          waltz16
PCPD2             80.00 usec
PLW2          20.00000000 W
PLW12          0.43145001 W
PLW13          0.43145001 W

F2 - Processing parameters
SI                 32768
SF            75.4803210 MHz
WDW                   EM
SSB       0
LB                 1.00 Hz
GB        0
PC                 1.40
```



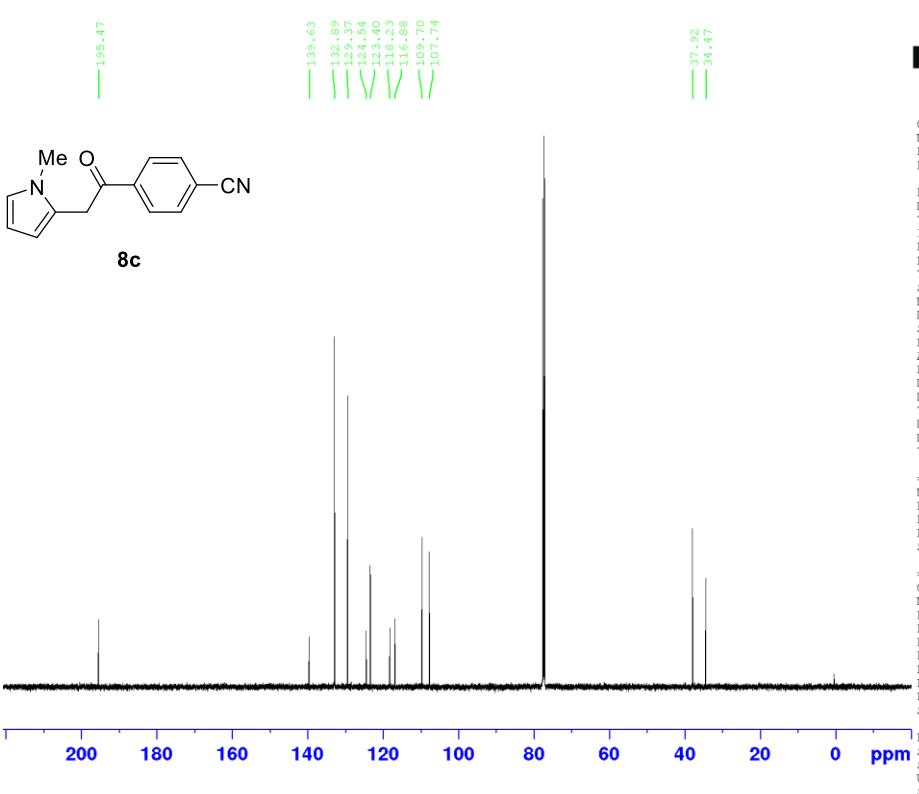
BRUKER

```
Current Data Parameters
NAME         mhbsc-386-1H
EXPNO                  10
PROCNO                  1

F2 - Acquisition Parameters
Date_           20180806
Time              16.26
INSTRUM           spect
PROBHD   5 mm PABBO BB-
PULPROG           zg30
TD               65536
SOLVENT          CDCl3
NS                   8
DS                   2
SWH          10000.000 Hz
FIDRES         0.152588 Hz
AQ           3.2767999 sec
RG                 256
DW              50.000 usec
DE              10.00 usec
TE              298.0 K
D1           1.00000000 sec
TD0                  1

======== CHANNEL f1 ========
NUC1                1H
P1              13.30 usec
PL1              3.00 dB
PL1W       12.58925438 W
SFO1      500.1330008 MHz

F2 - Processing parameters
SI                 32768
SF          500.1300210 MHz
WDW                EM
SSB       0
LB               0.30 Hz
GB        0
PC               1.00
```



BRUKER

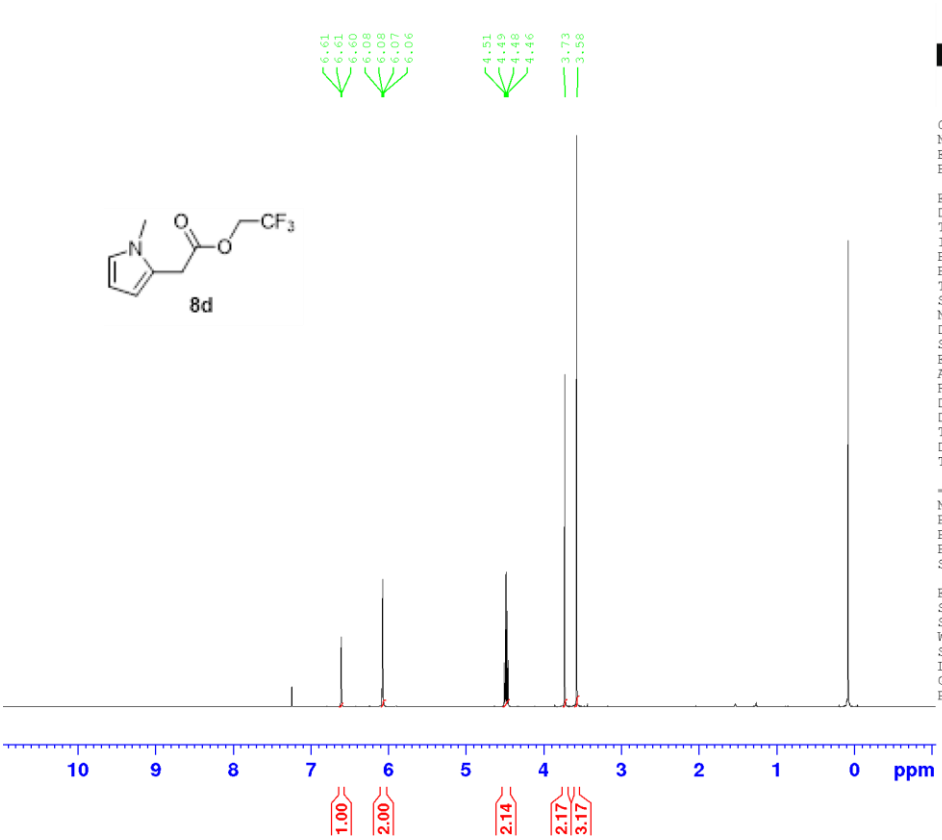
```
Current Data Parameters
NAME       mhbsc-386-13C
EXPNO                 10
PROCNO                 1

F2 - Acquisition Parameters
Date_           20180806
Time              18.03
INSTRUM           spect
PROBHD   5 mm PABBO BB-
PULPROG         zgpg30
TD               65536
SOLVENT          CDCl3
NS                 512
DS                   4
SWH          30303.031 Hz
FIDRES         0.462388 Hz
AQ           1.0813440 sec
RG                13000
DW              16.500 usec
DE              10.00 usec
TE              298.2 K
D1           1.00000000 sec
D11          0.03000000 sec
TD0                  1

======== CHANNEL f1 ========
NUC1               13C
P1               8.60 usec
PL1       0 dB
PL1W       63.80191422 W
SFO1      125.7703648 MHz

======== CHANNEL f2 ========
CPDPRG[2         waltz16
NUC2                1H
PCPD2           80.00 usec
PL2              3.00 dB
PL12            18.58 dB
PL13            18.58 dB
PL2W       12.58925438 W
PL12W       0.34833732 W
PL13W       0.34833732 W
SFO2      500.1320005 MHz

F2 - Processing parameters
SI                 32768
SF          125.7577441 MHz
WDW                EM
SSB       0
LB               1.00 Hz
GB        0
PC               1.40
```

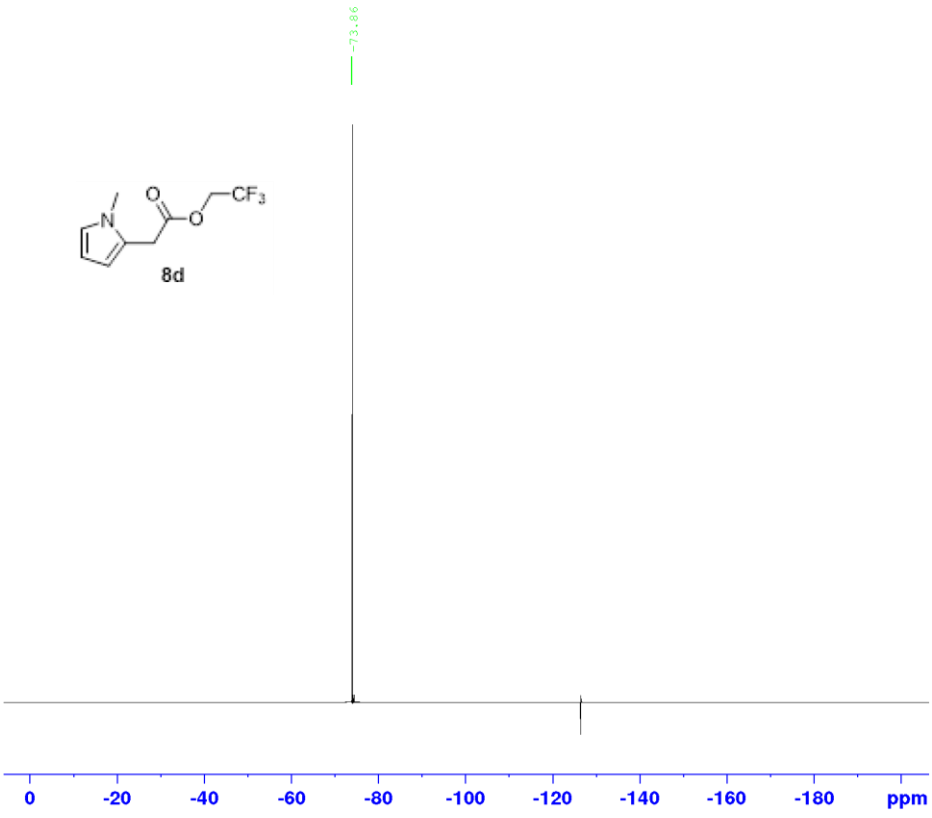



```
Current Data Parameters
NAME        BSCB-388 col
EXPNO                10
PROCNO                1

F2 - Acquisition Parameters
Date_          20180405
Time              16.10
INSTRUM           spect
PROBHD   5 mm PATBO BB-
PULPROG             zg30
TD                 32768
SOLVENT            CDCl3
NS                     4
DS                     2
SWH             6009.615 Hz
FIDRES           0.183399 Hz
AQ              2.7262976 sec
RG                   161
DW                83.200 usec
DE                10.00 usec
TE                297.9 K
D1            1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                 14.90 usec
PL1                -3.00 dB
PL1W         50.11872482 W
SFO1         500.1325007 MHz

F2 - Processing parameters
SI                 16384
SF           500.1300210 MHz
WDW                   EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```



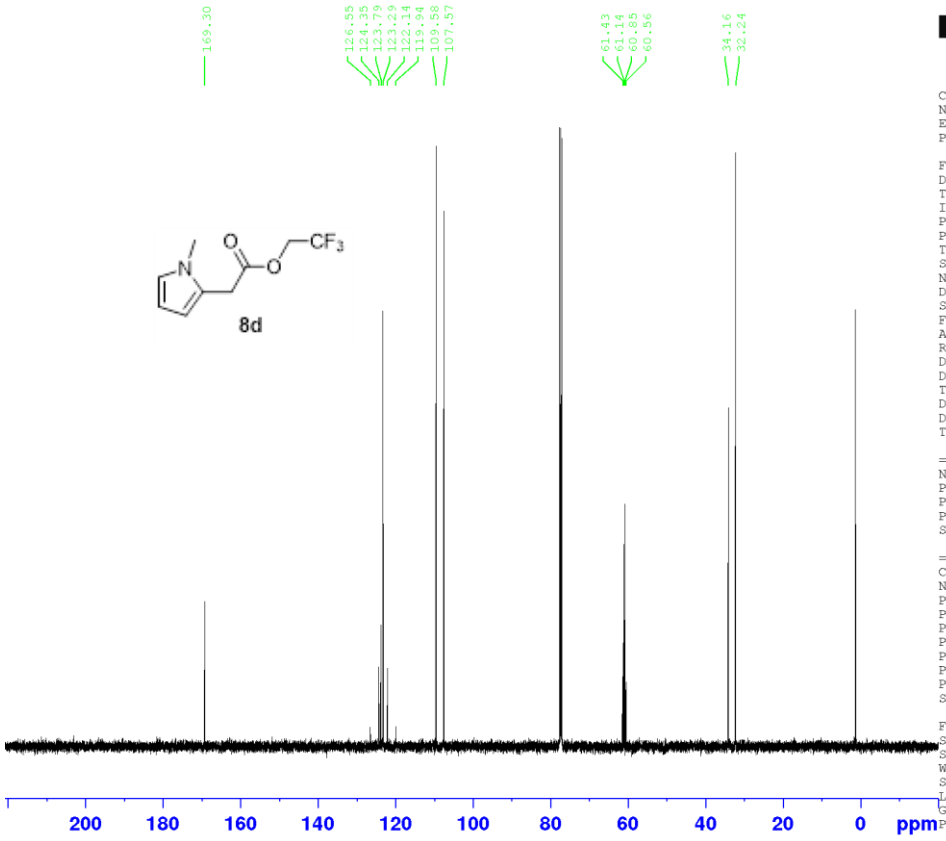

```
Current Data Parameters
NAME        BSCB-388 col
EXPNO                11
PROCNO                1

F2 - Acquisition Parameters
Date_          20180405
Time              16.12
INSTRUM           spect
PROBHD   5 mm PATBO BB-
PULPROG  ICIQ_zgfhigqn.2
TD                131072
SOLVENT            CDCl3
NS                     4
DS                     2
SWH           100000.000 Hz
FIDRES          0.762939 Hz
AQ              0.6553600 sec
RG                   406
DW                5.000 usec
DE                6.50 usec
TE                298.1 K
D1            5.00000000 sec
D11           0.03000000 sec
D12           0.00002000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                 19F
P1                19.50 usec
PL1        0 dB
SFO1         470.5453180 MHz

======== CHANNEL f2 ========
CPDPRG[2          waltz16
NUC2                  1H
PCPD2              80.00 usec
PL2                -3.00 dB
PL12               11.96 dB
PL2W         50.11872482 W
PL12W         1.59955800 W
SFO2         500.1325007 MHz

F2 - Processing parameters
SI                 65536
SF           470.5924245 MHz
WDW                   EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```



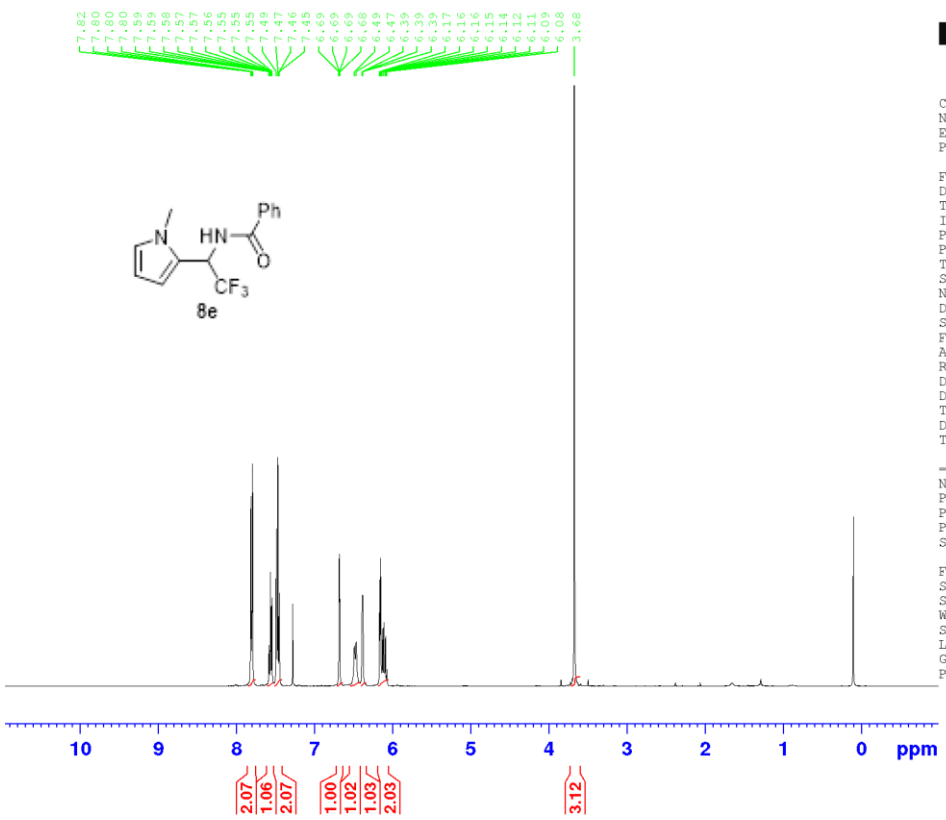

```
Current Data Parameters
NAME        BSCB-388 col
EXPNO                12
PROCNO                1

F2 - Acquisition Parameters
Date_           20180405
Time               16.35
INSTRUM            spect
PROBHD   5 mm PATBO BB-
PULPROG         zgpg30
TD               65536
SOLVENT          CDCl3
NS                 255
DS                   4
SWH          30303.031 Hz
FIDRES        0.462388 Hz
AQ            1.0813440 sec
RG                13000
DW               16.500 usec
DE               10.00 usec
TE               299.6 K
D1            1.00000000 sec
D11           0.03000000 sec
TD0                  1

======== CHANNEL f1 ========
NUC1                13C
P1                9.75 usec
PL1              -1.00 dB
PL1W        94.86833191 W
SFO1       125.7703648 MHz

======== CHANNEL f2 ========
CPDPRG[2        waltz16
NUC2                1H
PCPD2           80.00 usec
PL2              -3.00 dB
PL12             11.96 dB
PL13             11.96 dB
PL2W        50.11872482 W
PL12W        1.59955800 W
PL13W        1.59955800 W
SFO2       500.1320005 MHz

F2 - Processing parameters
SI                 32768
SF         125.7577441 MHz
WDW                   EM
SSB       0
LB                1.00 Hz
GB        0
PC                1.40
```

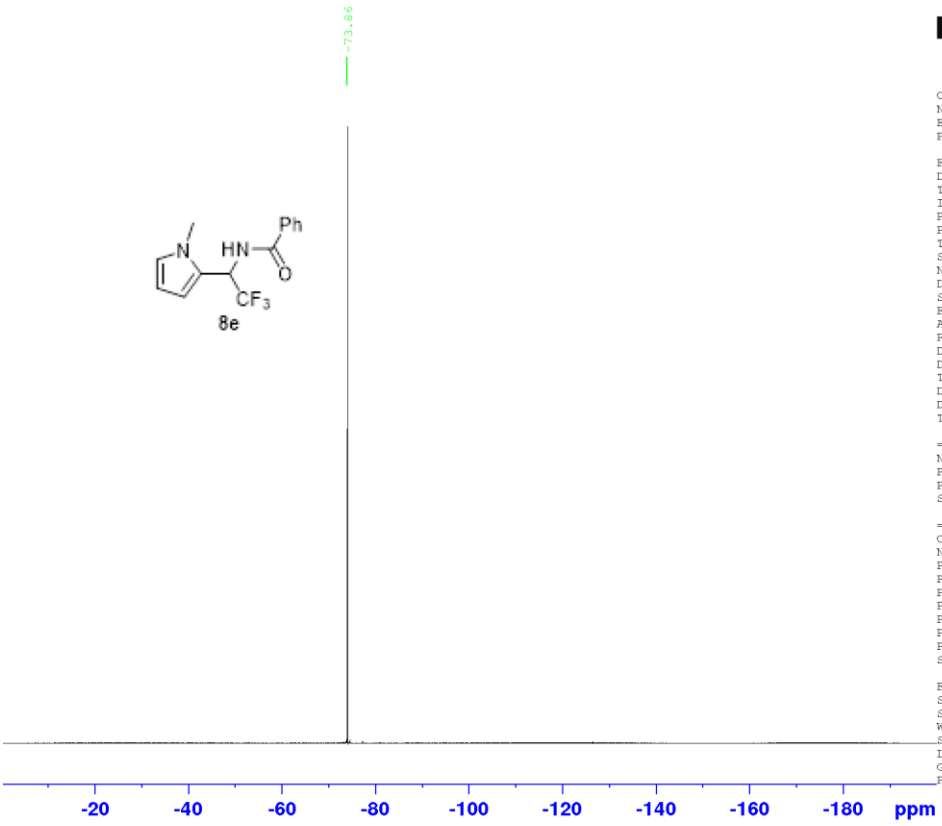



```
Current Data Parameters
NAME        BSCB-324 C+D
EXPNO                10
PROCNO                1

F2 - Acquisition Parameters
Date_           20180223
Time               18.26
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG             zg30
TD                 32768
SOLVENT          CDCl3
NS                     8
DS                     2
SWH           4789.272 Hz
FIDRES        0.146157 Hz
AQ            3.4209793 sec
RG                 161.3
DW               104.400 usec
DE                 6.00 usec
TE                 298.0 K
D1            1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                14.50 usec
PL1               -2.00 dB
PL1W        23.88643074 W
SFO1       400.1320007 MHz

F2 - Processing parameters
SI                 16384
SF         400.1300000 MHz
WDW                   EM
SSB       0
LB                0.30 Hz
GB        0
PC                1.00
```



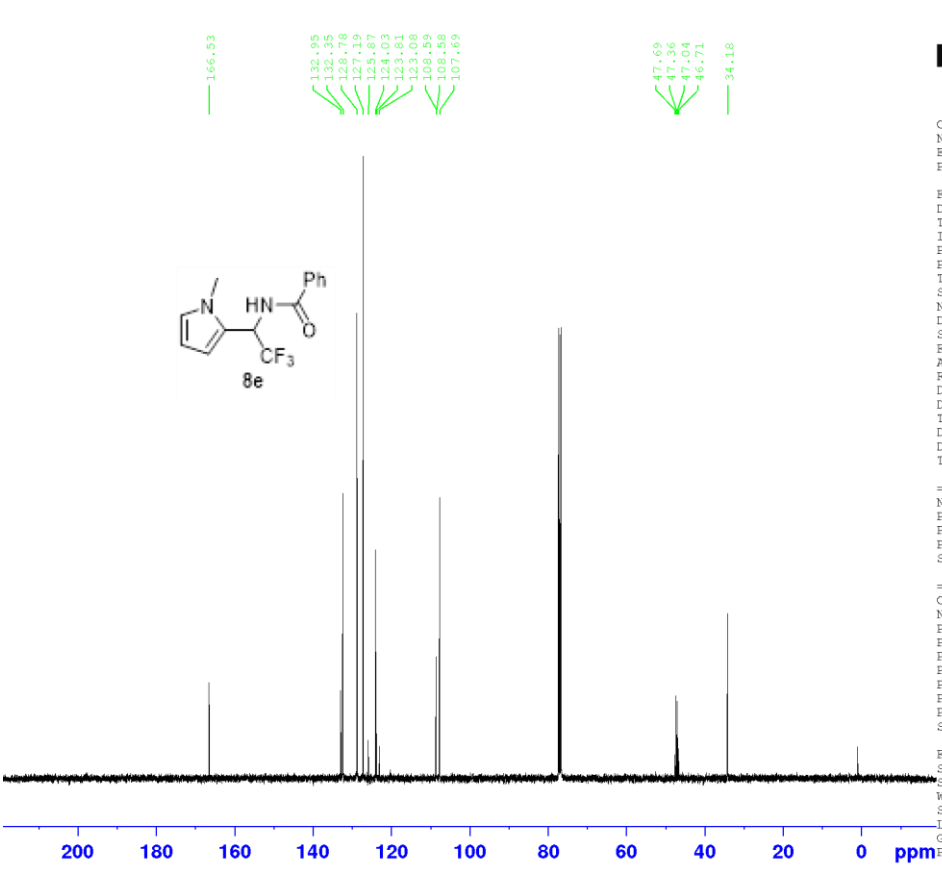

```
Current Data Parameters
NAME         BSCB-324 C+D
EXPNO                 11
PROCNO                 1

F2 - Acquisition Parameters
Date_           20180223
Time               18.28
INSTRUM            spect
PROBHD    5 mm PABBO BB-
PULPROG          zgpg30
TD               131072
SOLVENT            CDCl3
NS                     8
DS                     2
SWH           75187.969 Hz
FIDRES         0.573639 Hz
AQ             0.8716288 sec
RG                  2048
DW                6.650 usec
DE                7.14 usec
TE                298.1 K
D1           5.00000000 sec
D11          0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  19F
P1                 12.00 usec
PL1                -3.00 dB
SFO1         376.4607040 MHz

======== CHANNEL f2 ========
CPDPRG[2          waltz16
NUC2                   1H
PCPD2              80.00 usec
PL2                -3.00 dB
PL12               13.00 dB
PL13               13.00 dB
PL2W         30.07123375 W
PL12W         0.75535524 W
PL13W         0.75535524 W
SFO2         400.1320010 MHz

F2 - Processing parameters
SI                 65536
SF           376.4984036 MHz
WDW                   EM
SSB       0
LB                 1.00 Hz
GB        0
PC                 1.00
```



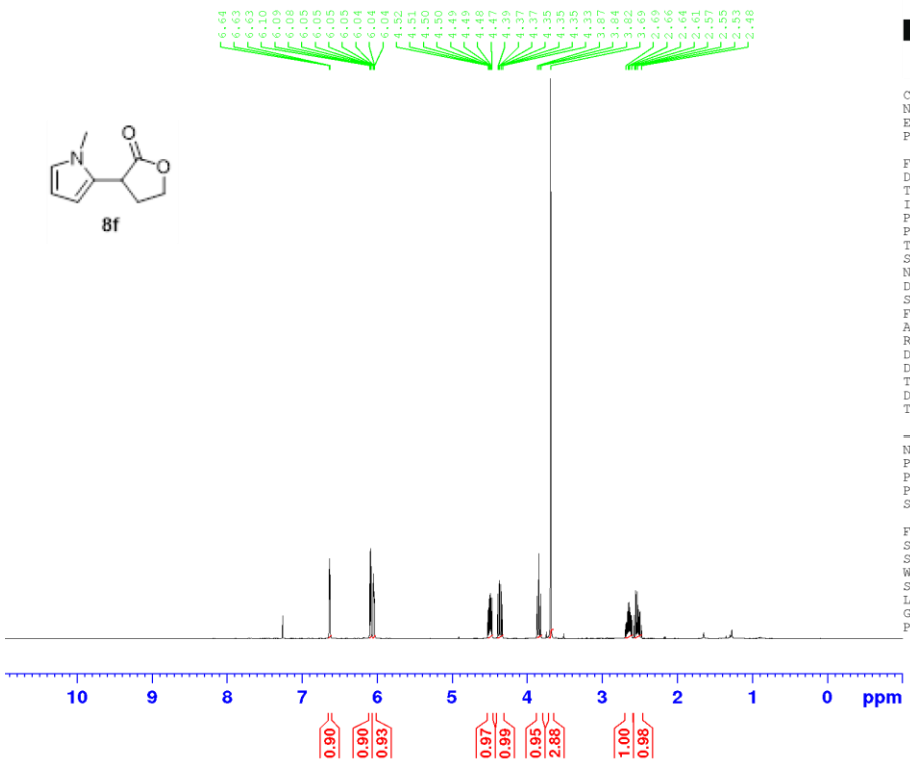

```
Current Data Parameters
NAME         BSCB-324 C+D
EXPNO                 12
PROCNO                 1

F2 - Acquisition Parameters
Date_           20180226
Time               2.23
INSTRUM            spect
PROBHD    5 mm PABBO BB-
PULPROG          zgpg30
TD                65536
SOLVENT            CDCl3
NS                   512
DS                     4
SWH           23980.814 Hz
FIDRES         0.365918 Hz
AQ             1.3664256 sec
RG                 32768
DW                20.850 usec
DE                10.00 usec
TE                298.0 K
D1           1.00000000 sec
D11          0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  13C
P1                 10.00 usec
PL1                -3.00 dB
PL1W         75.17808533 W
SFO1         100.6228298 MHz

======== CHANNEL f2 ========
CPDPRG[2          waltz16
NUC2                   1H
PCPD2              80.00 usec
PL2                -3.00 dB
PL12               13.00 dB
PL13               13.00 dB
PL2W         30.07123375 W
PL12W         0.75535524 W
PL13W         0.75535524 W
SFO2         400.1316005 MHz

F2 - Processing parameters
SI                 32768
SF           100.6127690 MHz
WDW                   EM
SSB       0
LB                 1.00 Hz
GB        0
PC                 1.40
```



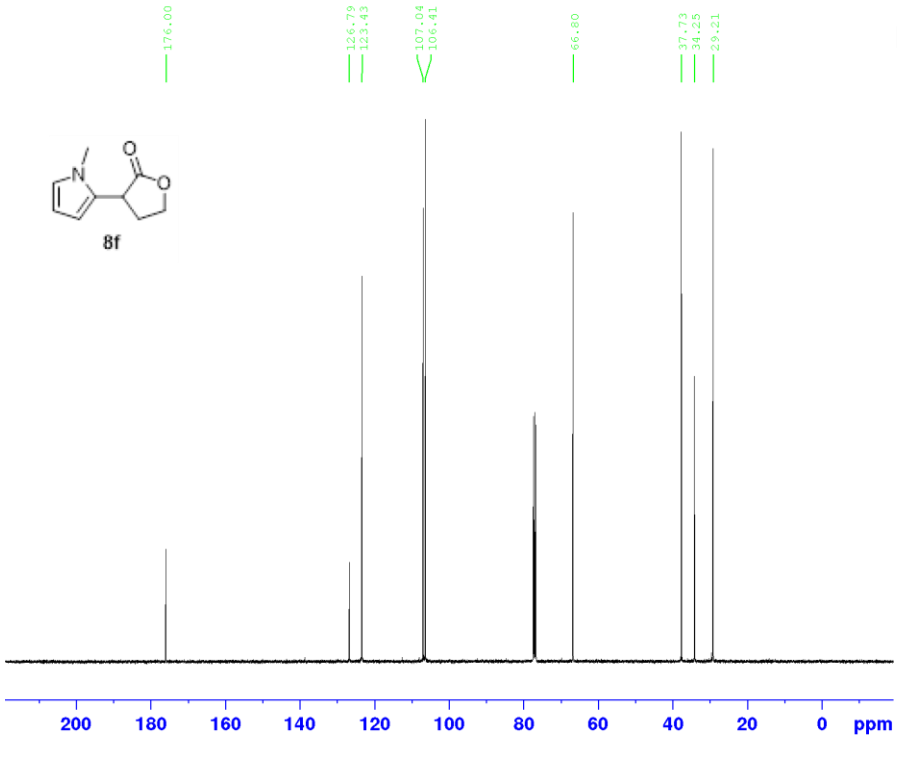
BRUKER

```
Current Data Parameters
NAME          mah-202-1H
EXPNO                 10
PROCNO                 1

F2 - Acquisition Parameters
Date_           20180421
Time                8.20
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG             zg30
TD                 32768
SOLVENT            CDCl3
NS                    32
DS                     2
SWH             4789.272 Hz
FIDRES          0.146157 Hz
AQ              3.4209793 sec
RG                  71.8
DW                104.400 usec
DE                  6.00 usec
TE                298.0 K
D1             1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                 14.50 usec
PL1                -2.00 dB
PL1W         23.88643074 W
SFO1         400.1320007 MHz

F2 - Processing parameters
SI                 16384
SF           400.1300094 MHz
WDW                   EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```



BRUKER

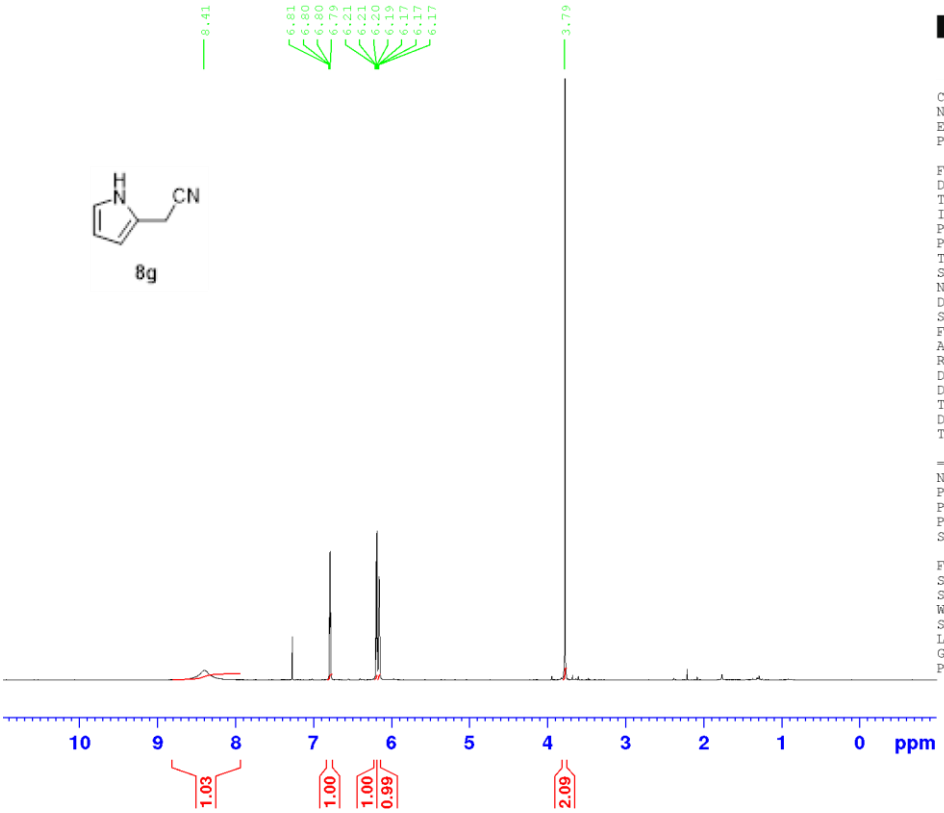
```
Current Data Parameters
NAME          mah-202-13C
EXPNO                 10
PROCNO                 1

F2 - Acquisition Parameters
Date_           20180421
Time               21.28
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG           zgpg30
TD                 65536
SOLVENT            CDCl3
NS                  2048
DS                     4
SWH            23980.814 Hz
FIDRES          0.365918 Hz
AQ              1.3664256 sec
RG                20642.5
DW                 20.850 usec
DE                 10.00 usec
TE                 298.4 K
D1             1.00000000 sec
D11            0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  13C
P1                 10.00 usec
PL1                -3.00 dB
PL1W         75.17808533 W
SFO1         100.6228298 MHz

======== CHANNEL f2 ========
CPDPRG[2          waltz16
NUC2                  1H
PCPD2              80.00 usec
PL2                -3.00 dB
PL12               13.00 dB
PL13               13.00 dB
PL2W         30.07123375 W
PL12W         0.75535524 W
PL13W         0.75535524 W
SFO2         400.1316005 MHz

F2 - Processing parameters
SI                 32768
SF           100.6127690 MHz
WDW                   EM
SSB       0
LB                 1.00 Hz
GB        0
PC                 1.40
```

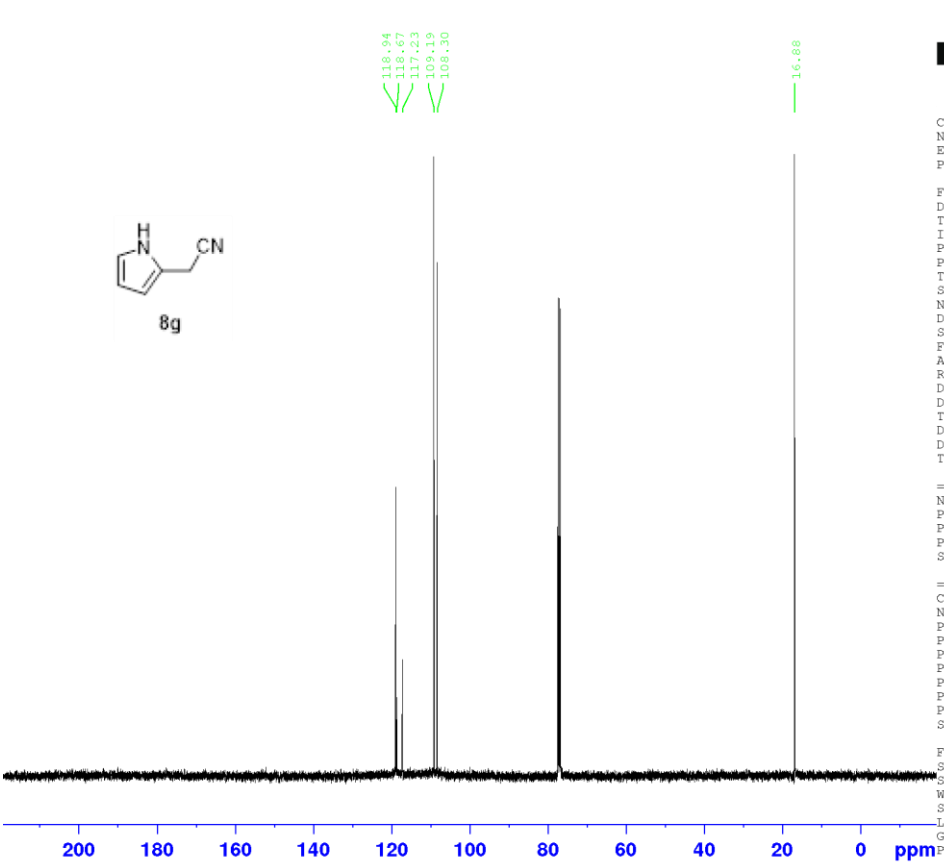



```
Current Data Parameters
NAME        BSCB-404 col
EXPNO                 10
PROCNO                 1

F2 - Acquisition Parameters
Date_           20180418
Time              16.08
INSTRUM           spect
PROBHD   5 mm PABBO BB-
PULPROG            zg30
TD                32768
SOLVENT           CDCl3
NS                    4
DS                    2
SWH            4789.272 Hz
FIDRES        0.146157 Hz
AQ            3.4209793 sec
RG                  128
DW              104.400 usec
DE                6.00 usec
TE                298.1 K
D1          1.00000000 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                  1H
P1                14.50 usec
PL1               -2.00 dB
PL1W        23.88643074 W
SFO1        400.1320007 MHz

F2 - Processing parameters
SI                16384
SF          400.1300000 MHz
WDW                  EM
SSB       0
LB                0.30 Hz
GB        0
PC                1.00
```



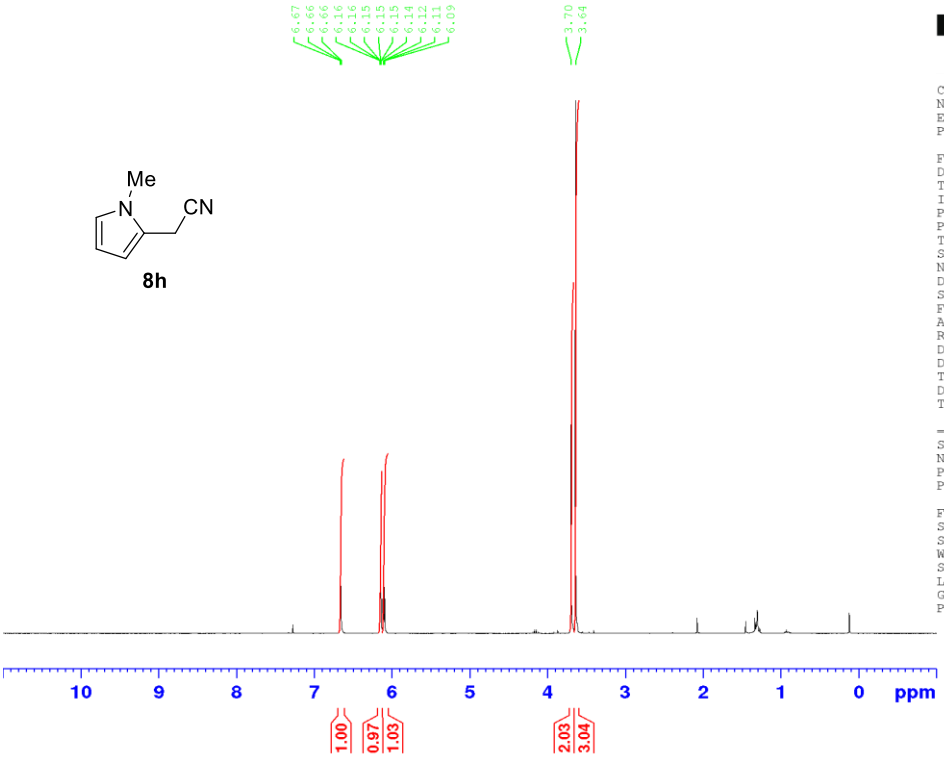

```
Current Data Parameters
NAME        BSCB-404 col
EXPNO                 11
PROCNO                 1

F2 - Acquisition Parameters
Date_           20180419
Time               5.11
INSTRUM           spect
PROBHD   5 mm PABBI 1H/
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                  730
DS                    4
SWH           24038.461 Hz
FIDRES        0.366798 Hz
AQ            1.3631488 sec
RG                23170.5
DW              20.800 usec
DE              10.00 usec
TE                298.0 K
D1          1.00000000 sec
D11         0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                 13C
P1                13.50 usec
PL1               -5.00 dB
PL1W       119.14923859 W
SFO1        100.6303736 MHz

======== CHANNEL f2 ========
CPDPRG[2         waltz16
NUC2                  1H
PCPD2             80.00 usec
PL2               -2.00 dB
PL12              19.16 dB
PL13              19.16 dB
PL2W        23.88643074 W
PL12W        0.18287371 W
PL13W        0.18287371 W
SFO2        400.1616006 MHz

F2 - Processing parameters
SI                32768
SF          100.6203120 MHz
WDW                  EM
SSB       0
LB                1.00 Hz
GB        0
PC                1.40
```

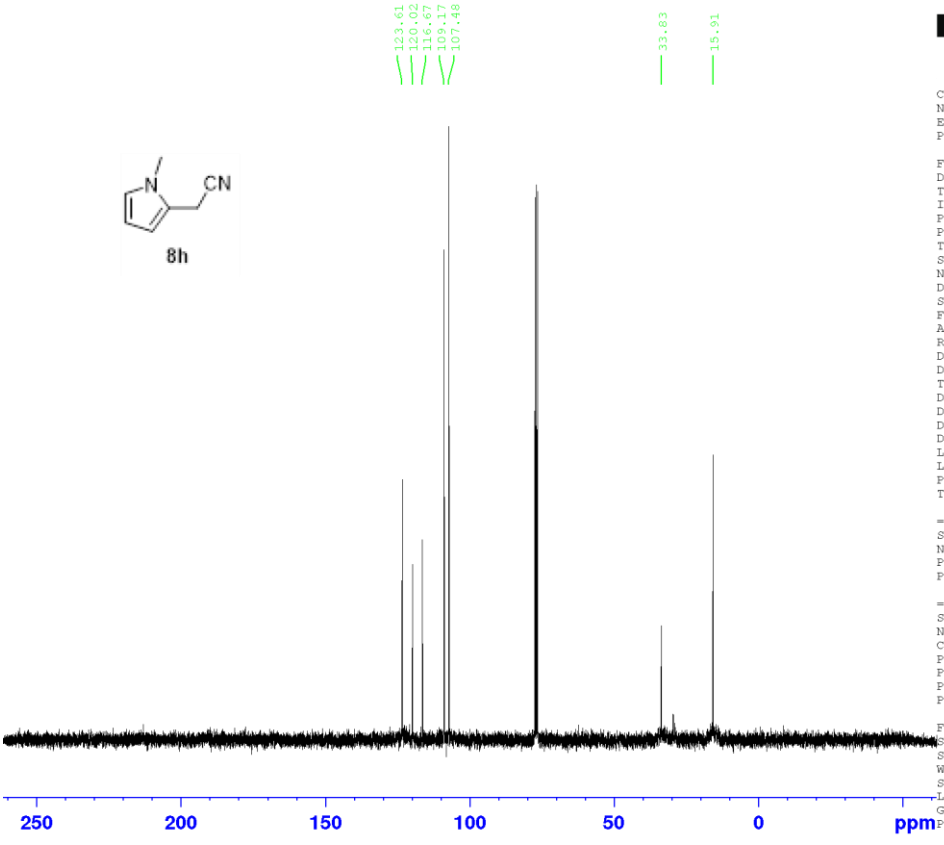



```
Current Data Parameters
NAME        BSCB-319 col
EXPNO                10
PROCNO                1

F2 - Acquisition Parameters
Date_          20180222
Time               4.29
INSTRUM       FOURIER300
PROBHD   5 mm DUL 13C-1
PULPROG            zg30
TD                65536
SOLVENT           CDCl3
NS                    8
DS                    2
SWH            6103.516 Hz
FIDRES         0.093132 Hz
AQ             5.3687091 sec
RG                11.1913
DW                81.920 usec
DE                 6.50 usec
TE                298.0 K
D1             1.00000000 sec
TD0                   1

======== CHANNEL f1 ========
SFO1         300.1818011 MHz
NUC1                   1H
P1                 11.75 usec
PLW1         20.00000000 W

F2 - Processing parameters
SI                32768
SF           300.1800000 MHz
WDW                  EM
SSB       0
LB                0.30 Hz
GB        0
PC                1.40
```



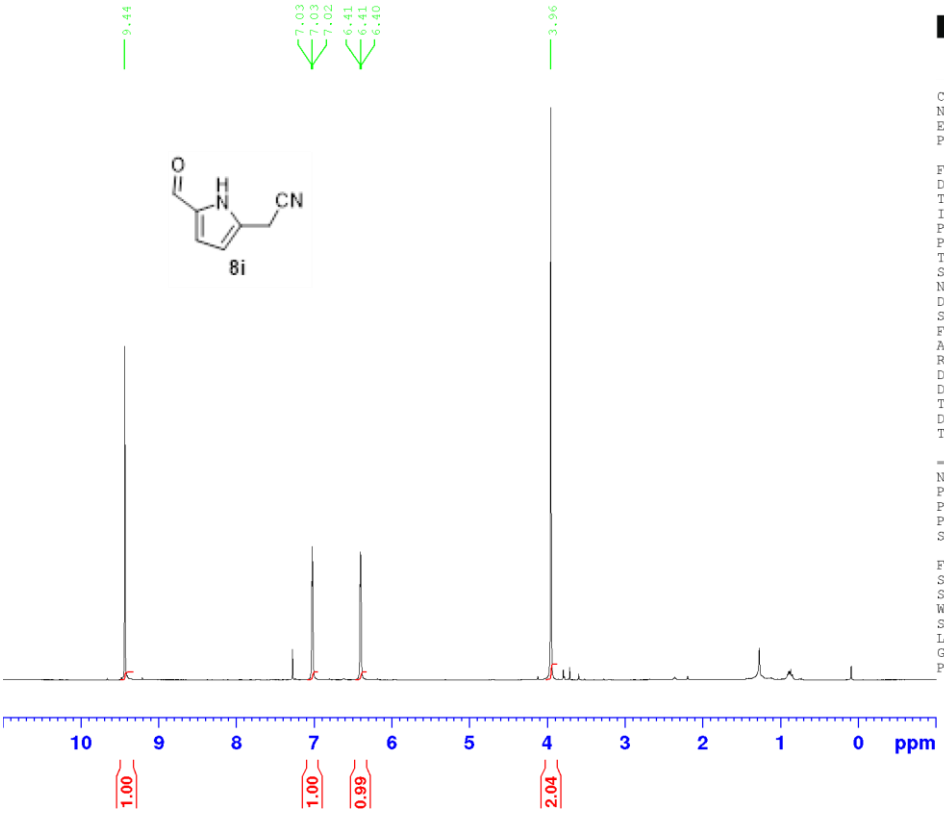

```
Current Data Parameters
NAME        BSCB-319 col
EXPNO                11
PROCNO                1

F2 - Acquisition Parameters
Date_          20180222
Time               4.32
INSTRUM       FOURIER300
PROBHD   5 mm DUL 13C-1
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                  512
DS                    4
SWH           24414.063 Hz
FIDRES         0.372529 Hz
AQ             1.3421773 sec
RG                501.187
DW                20.480 usec
DE                 6.50 usec
TE                298.2 K
D1             1.00000000 sec
D11            0.03000000 sec
D31            0.00001050 sec
D40            0.01641260 sec
L4                    45
L5                    32
P32               80.00 usec
TD0                   1

======== CHANNEL f1 ========
SFO1          75.4878688 MHz
NUC1                  13C
P1                10.50 usec
PLW1         50.00000000 W

======== CHANNEL f2 ========
SFO2         300.1812007 MHz
NUC2                   1H
CPDPRG[2          waltz16
PCPD2             80.00 usec
PLW2         20.00000000 W
PLW12         0.43145001 W
PLW13         0.43145001 W

F2 - Processing parameters
SI                32768
SF           75.4803210 MHz
WDW                  EM
SSB       0
LB             1.00 Hz
GB        0
PC                1.40
```

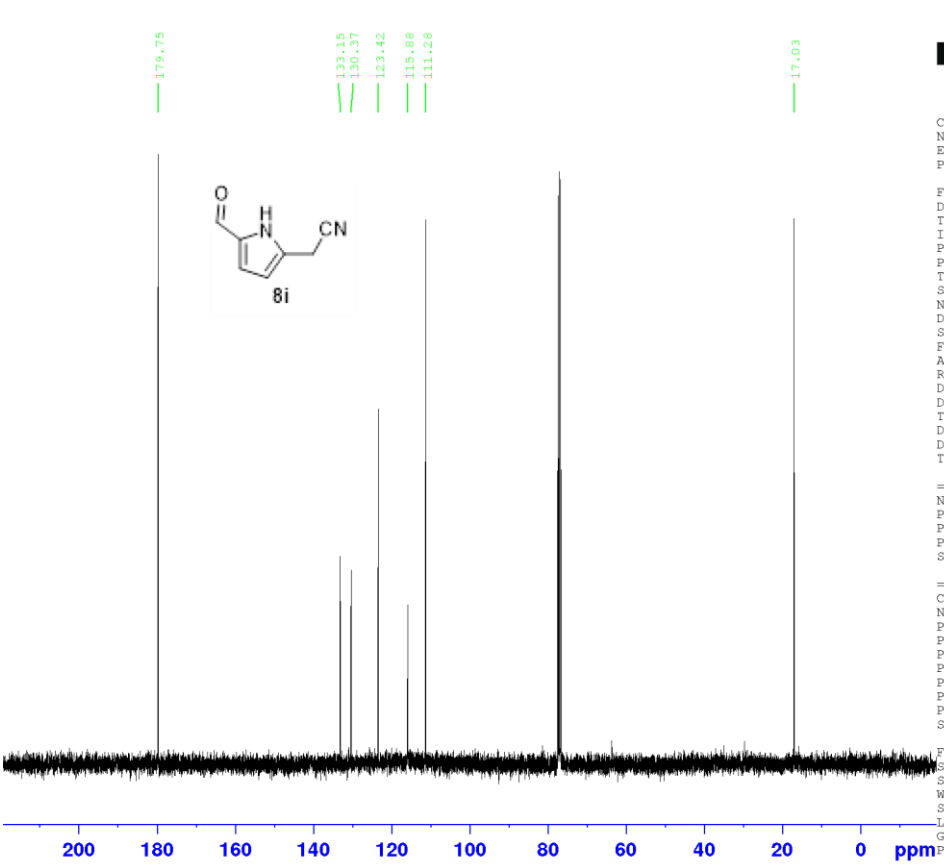



```
Current Data Parameters
NAME      BSCB-344-02 col
EXPNO                 10
PROCNO                 1

F2 - Acquisition Parameters
Date_           20180314
Time              19.38
INSTRUM           spect
PROBHD   5 mm PABBI 1H/
PULPROG           zg30
TD               32768
SOLVENT          CDCl3
NS                   8
DS                   2
SWH           4807.692 Hz
FIDRES         0.146719 Hz
AQ            3.4078720 sec
RG                 114
DW             104.000 usec
DE              10.00 usec
TE              298.0 K
D1           1.00000000 sec
TD0                  1

======== CHANNEL f1 ========
NUC1                1H
P1                6.90 usec
PL1              -2.00 dB
PL1W        23.88643074 W
SFO1        400.1620008 MHz

F2 - Processing parameters
SI                 16384
SF          400.1600000 MHz
WDW                EM
SSB       0
LB              0.30 Hz
GB        0
PC                 1.00
```



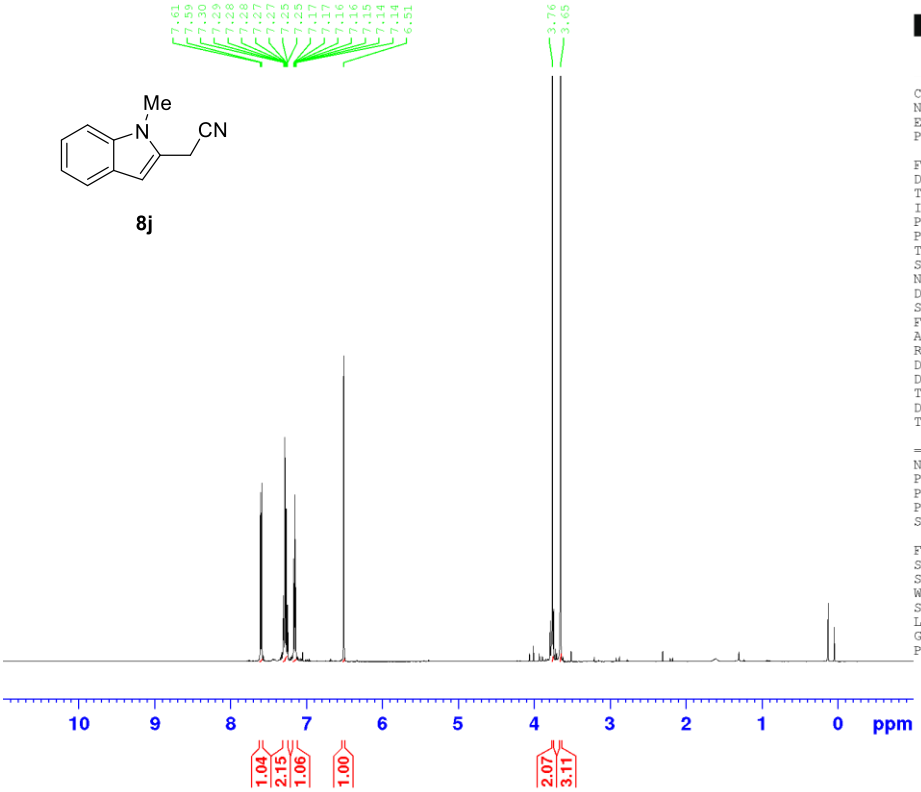

```
Current Data Parameters
NAME      BSCB-344-02 col
EXPNO                 11
PROCNO                 1

F2 - Acquisition Parameters
Date_           20180315
Time               8.14
INSTRUM           spect
PROBHD   5 mm PABBI 1H/
PULPROG          zgpg30
TD               65536
SOLVENT          CDCl3
NS                 203
DS                   4
SWH          24038.461 Hz
FIDRES         0.366798 Hz
AQ            1.3631488 sec
RG                 32768
DW             20.800 usec
DE              10.00 usec
TE              298.0 K
D1           1.00000000 sec
D11          0.03000000 sec
TD0                  1

======== CHANNEL f1 ========
NUC1               13C
P1              13.50 usec
PL1              -5.00 dB
PL1W       119.14923859 W
SFO1        100.6303736 MHz

======== CHANNEL f2 ========
CPDPRG[2         waltz16
NUC2                1H
PCPD2            80.00 usec
PL2              -2.00 dB
PL12             19.16 dB
PL13             19.16 dB
PL2W        23.88643074 W
PL12W        0.18287371 W
PL13W        0.18287371 W
SFO2        400.1616006 MHz

F2 - Processing parameters
SI                 32768
SF          100.6203120 MHz
WDW                EM
SSB       0
LB              1.00 Hz
GB        0
PC                 1.40
```



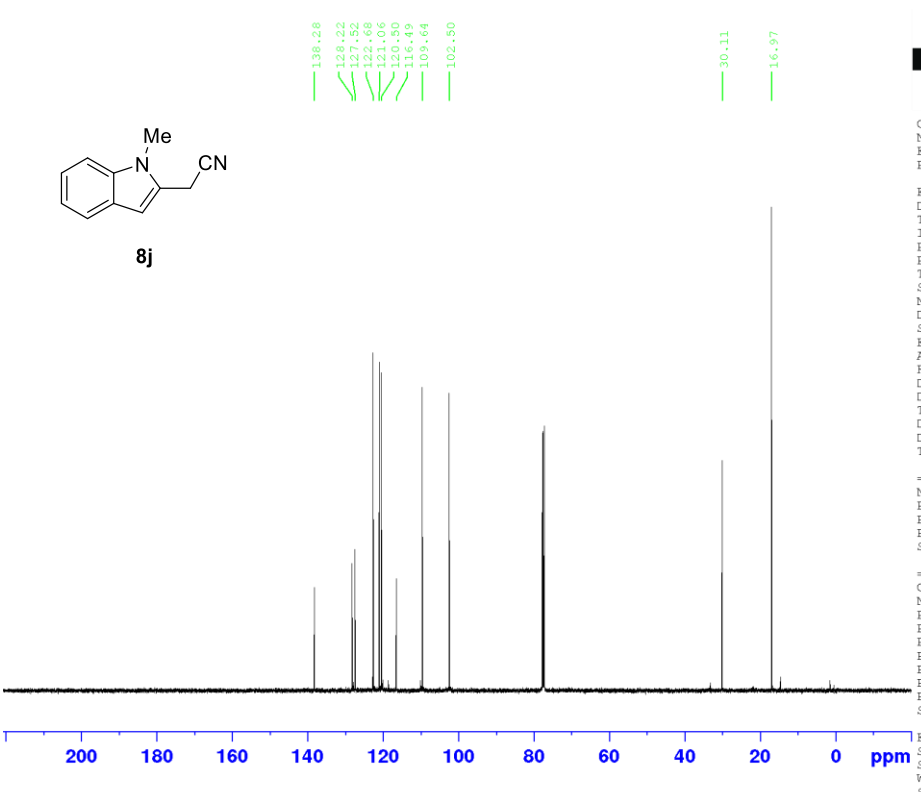
8j

```
Current Data Parameters
NAME          mhbsc-369-1H
EXPNO                   10
PROCNO                   1

F2 - Acquisition Parameters
Date_           20180806
Time              16.33
INSTRUM           spect
PROBHD   5 mm PABBO BB-
PULPROG           zg30
TD              65536
SOLVENT           CDCl3
NS                    8
DS                    2
SWH           10000.000 Hz
FIDRES         0.152588 Hz
AQ            3.2767999 sec
RG                   57
DW              50.000 usec
DE              10.00 usec
TE              298.1 K
D1           1.00000000 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                 1H
P1                13.30 usec
PL1                3.00 dB
PL1W        12.58925438 W
SFO1        500.1330008 MHz

F2 - Processing parameters
SI                32768
SF          500.1300210 MHz
WDW                  EM
SSB       0
LB                0.30 Hz
GB        0
PC                1.00
```



8j

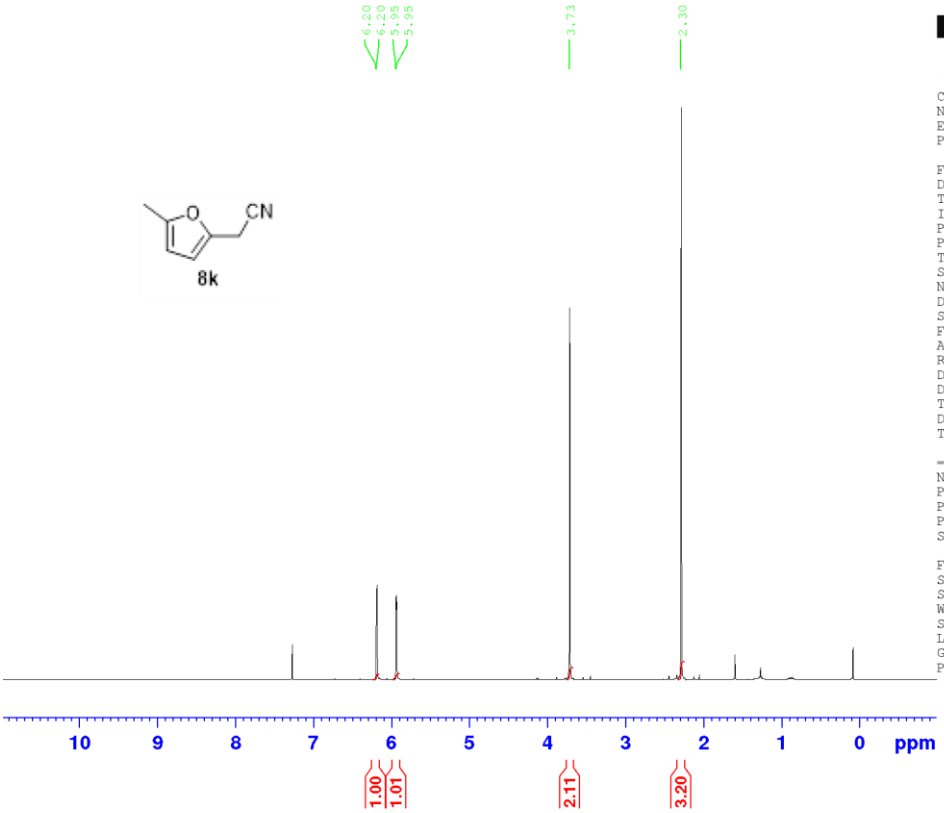
```
Current Data Parameters
NAME         mhbsc-369-13C
EXPNO                   10
PROCNO                   1

F2 - Acquisition Parameters
Date_           20180806
Time              16.45
INSTRUM           spect
PROBHD   5 mm PABBO BB-
PULPROG         zgpg30
TD              65536
SOLVENT           CDCl3
NS                  318
DS                    4
SWH           30303.031 Hz
FIDRES         0.462388 Hz
AQ            1.0813440 sec
RG                13000
DW              16.500 usec
DE              10.00 usec
TE              298.7 K
D1           1.00000000 sec
D11          0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                13C
P1                8.60 usec
PL1       0 dB
PL1W        63.80191422 W
SFO1        125.7703648 MHz

======== CHANNEL f2 ========
CPDPRG[2          waltz16
NUC2                 1H
PCPD2             80.00 usec
PL2                3.00 dB
PL12              18.58 dB
PL13              18.58 dB
PL2W        12.58925438 W
PL12W        0.34833732 W
PL13W        0.34833732 W
SFO2        500.1320005 MHz

F2 - Processing parameters
SI                32768
SF          125.7577441 MHz
WDW                  EM
SSB       0
LB                1.00 Hz
GB        0
PC                1.40
```

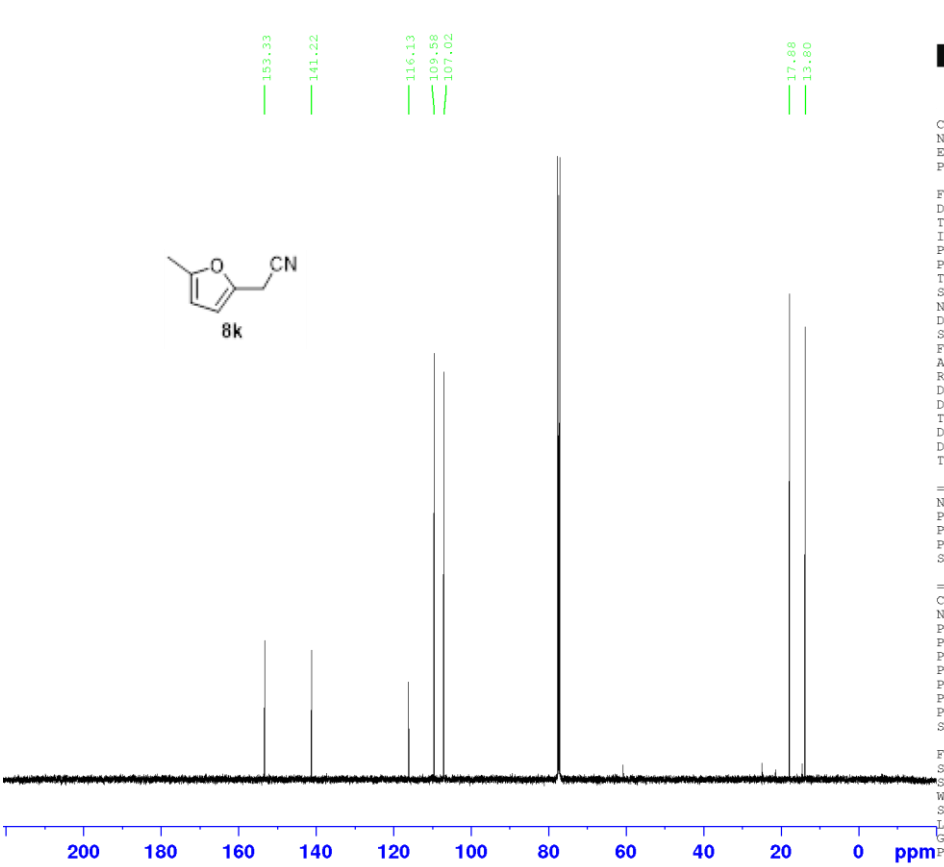



```
Current Data Parameters
NAME        BSCB-343 col
EXPNO                 10
PROCNO                 1

F2 - Acquisition Parameters
Date_           20180319
Time               13.51
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG             zg30
TD                 32768
SOLVENT            CDCl3
NS                     8
DS                     2
SWH             4789.272 Hz
FIDRES          0.146157 Hz
AQ              3.4209793 sec
RG                   181
DW               104.400 usec
DE                 6.00 usec
TE                298.1 K
D1            1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                 14.50 usec
PL1                -2.00 dB
PL1W         23.88643074 W
SFO1         400.1320007 MHz

F2 - Processing parameters
SI                 16384
SF           400.1300000 MHz
WDW                   EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```



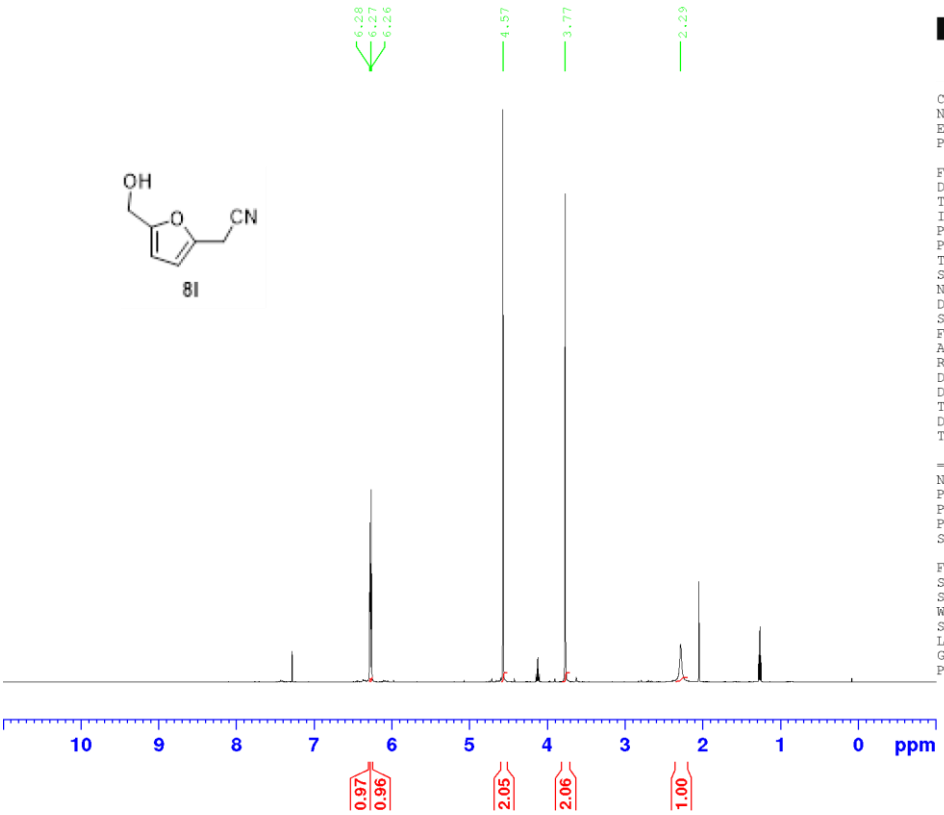

```
Current Data Parameters
NAME      BSCB-343-02 col
EXPNO                 11
PROCNO                 1

F2 - Acquisition Parameters
Date_           20180323
Time               1.08
INSTRUM            spect
PROBHD   5 mm PATBO BB-
PULPROG           zgpg30
TD                 65536
SOLVENT            CDCl3
NS                   512
DS                     4
SWH            30303.031 Hz
FIDRES          0.462388 Hz
AQ              1.0813440 sec
RG                 11500
DW               16.500 usec
DE                 10.00 usec
TE                298.7 K
D1            1.00000000 sec
D11           0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                 13C
P1                 9.75 usec
PL1               -1.00 dB
PL1W         94.86833191 W
SFO1        125.7703648 MHz

======== CHANNEL f2 ========
CPDPRG[2          waltz16
NUC2                  1H
PCPD2              80.00 usec
PL2                -3.00 dB
PL12               11.96 dB
PL13               11.96 dB
PL2W         50.11872482 W
PL12W         1.59955800 W
PL13W         1.59955800 W
SFO2        500.1320005 MHz

F2 - Processing parameters
SI                 32768
SF          125.7577441 MHz
WDW                   EM
SSB       0
LB                 1.00 Hz
GB        0
PC                 1.40
```

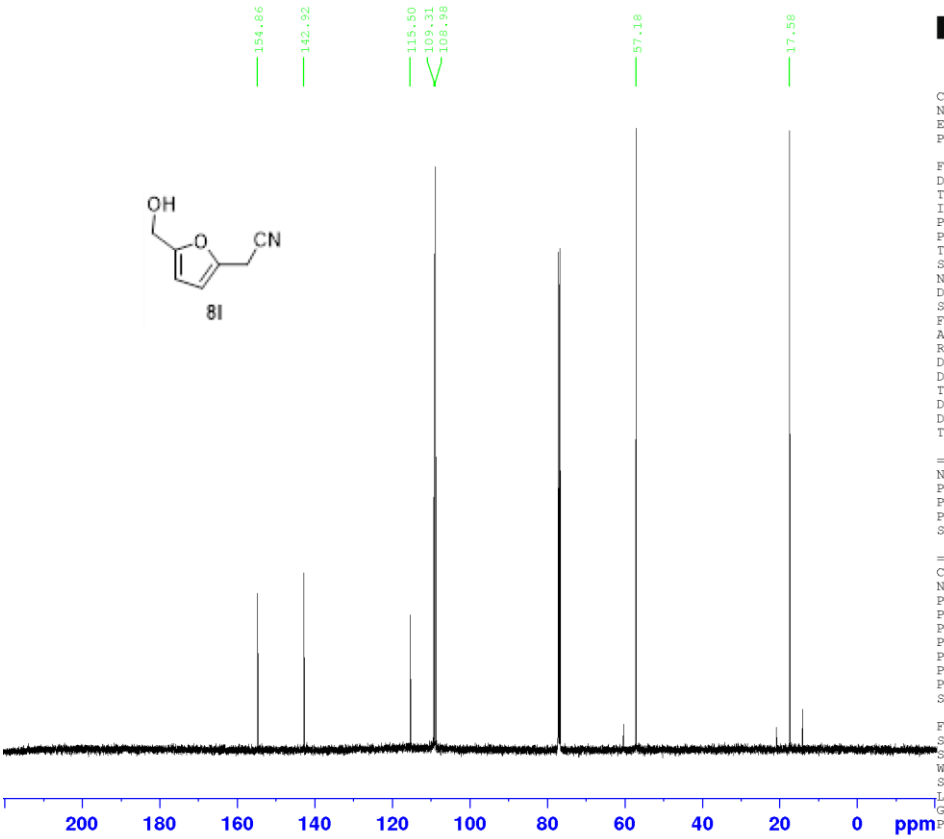



```
Current Data Parameters
NAME      BSCB-332 col C6-D4
EXPNO                 10
PROCNO                 1

F2 - Acquisition Parameters
Date_           20180228
Time              19.05
INSTRUM           spect
PROBHD   5 mm PABBI 1H/
PULPROG           zg30
TD               32768
SOLVENT          CDCl3
NS                   8
DS                   2
SWH           6009.615 Hz
FIDRES         0.183399 Hz
AQ            2.7262976 sec
RG                50.8
DW              83.200 usec
DE              10.00 usec
TE              298.0 K
D1          1.00000000 sec
TD0                  1

======== CHANNEL f1 ========
NUC1                 1H
P1                6.70 usec
PL1               1.00 dB
PL1W        19.95262337 W
SFO1        500.1325007 MHz

F2 - Processing parameters
SI                16384
SF          500.1300000 MHz
WDW                  EM
SSB       0
LB              0.30 Hz
GB        0
PC              1.00
```



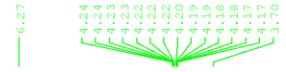

```
Current Data Parameters
NAME      BSCB-332 col C6-D4
EXPNO                 11
PROCNO                 1

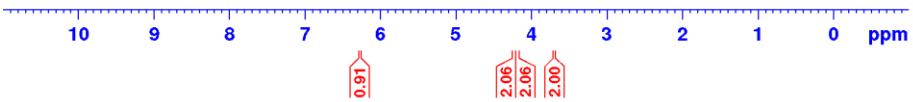
F2 - Acquisition Parameters
Date_           20180301
Time              1.51
INSTRUM           spect
PROBHD   5 mm PABBI 1H/
PULPROG          zgpg30
TD               65536
SOLVENT          CDCl3
NS                 512
DS                   4
SWH          30303.031 Hz
FIDRES         0.462388 Hz
AQ            1.0813440 sec
RG                14600
DW              16.500 usec
DE              10.00 usec
TE              298.0 K
D1          1.00000000 sec
D11         0.03000000 sec
TD0                  1

======== CHANNEL f1 ========
NUC1                13C
P1              18.50 usec
PL1              -0.50 dB
PL1W        84.55149078 W
SFO1        125.7703648 MHz

======== CHANNEL f2 ========
CPDPRG[2         waltz16
NUC2                 1H
PCPD2           80.00 usec
PL2              1.00 dB
PL12            22.54 dB
PL13            22.54 dB
PL2W        19.95262337 W
PL12W        0.13995871 W
PL13W        0.13995871 W
SFO2        500.1320005 MHz

F2 - Processing parameters
SI                32768
SF          125.7577890 MHz
WDW                  EM
SSB       0
LB              1.00 Hz
GB        0
PC              1.40
```





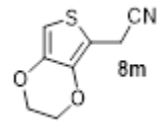
BRUKER

```
Current Data Parameters
NAME        bsbc349-f3.1
EXPNO                 10
PROCNO                 1

F2 - Acquisition Parameters
Date_           20180806
Time               14.32
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG             zg30
TD                 32768
SOLVENT            CDCl3
NS                     8
DS                     2
SWH             4789.272 Hz
FIDRES          0.146157 Hz
AQ              3.4209793 sec
RG                 161.3
DW                104.400 usec
DE                  6.00 usec
TE                298.0 K
D1              1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                 14.50 usec
PL1                -2.00 dB
PL1W          23.88643074 W
SFO1          400.1320007 MHz

F2 - Processing parameters
SI                 16384
SF           400.1300097 MHz
WDW                   no
SSB       0
LB        0 Hz
GB        0
PC                  1.00
```



BRUKER

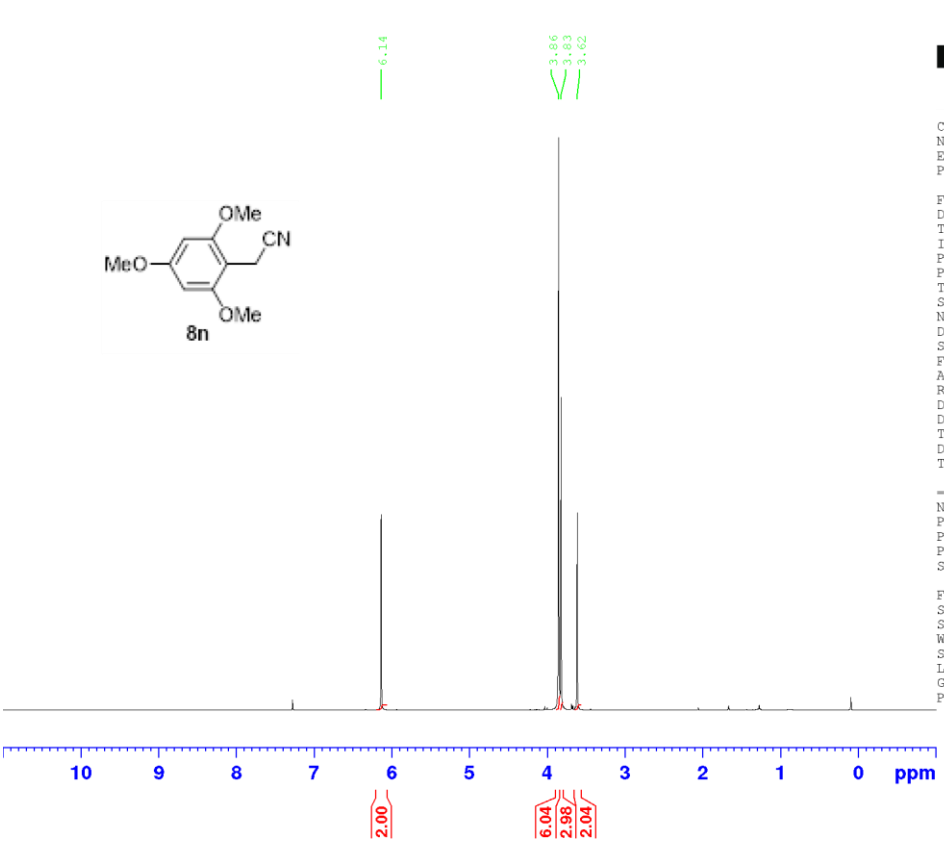
```
Current Data Parameters
NAME      bsbc349-f3.1-13C
EXPNO                   10
PROCNO                   1

F2 - Acquisition Parameters
Date_             20180806
Time                 15.26
INSTRUM              spect
PROBHD   5 mm PABBO BB-
PULPROG            zgpg30
TD                   65536
SOLVENT              CDCl3
NS                     512
DS                       4
SWH              30303.031 Hz
FIDRES            0.462388 Hz
AQ                1.0813440 sec
RG                   13000
DW                  16.500 usec
DE                  10.00 usec
TE                  300.3 K
D1              1.00000000 sec
D11             0.03000000 sec
TD0                      1

======== CHANNEL f1 ========
NUC1                   13C
P1                    8.60 usec
PL1         0 dB
PL1W          63.80191422 W
SFO1         125.7703648 MHz

======== CHANNEL f2 ========
CPDPRG[2          waltz16
NUC2                   1H
PCPD2               80.00 usec
PL2                  3.00 dB
PL12                18.58 dB
PL13                18.58 dB
PL2W          12.58925438 W
PL12W          0.34833732 W
PL13W          0.34833732 W
SFO2         500.1320005 MHz

F2 - Processing parameters
SI                   32768
SF           125.7577753 MHz
WDW                     no
SSB       0
LB        0 Hz
GB        0
PC                    1.40
```

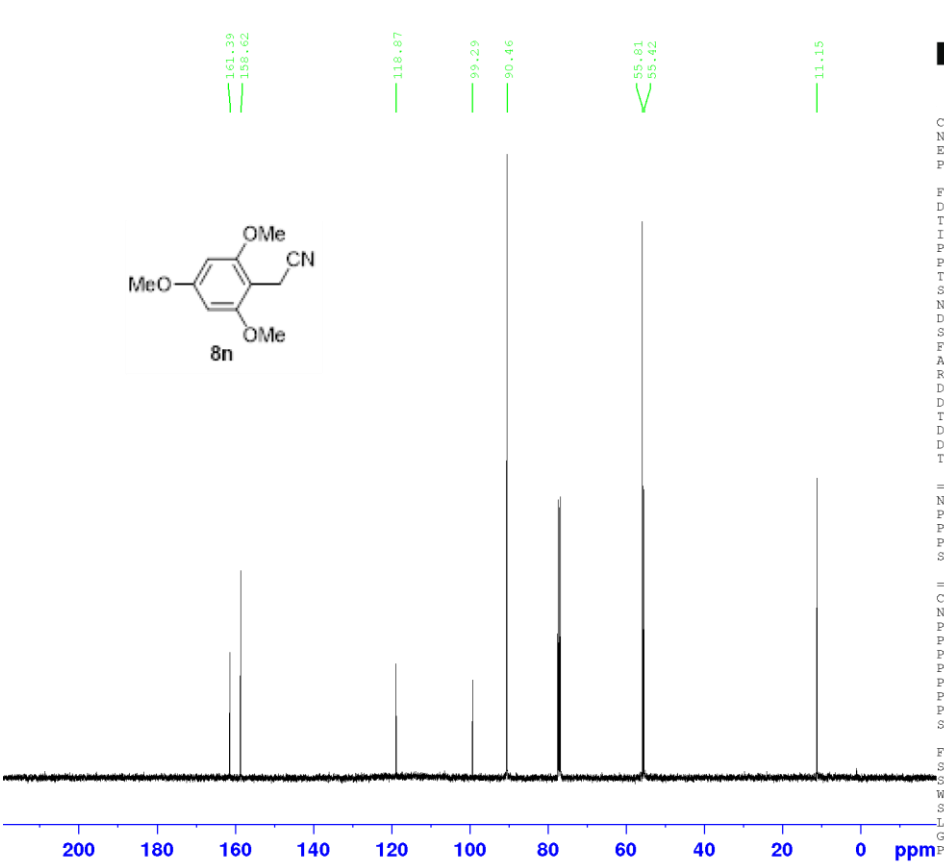



```
Current Data Parameters
NAME        BSCB-356 col
EXPNO                10
PROCNO                1

F2 - Acquisition Parameters
Date_          20180315
Time              19.48
INSTRUM           spect
PROBHD   5 mm PABBI 1H/
PULPROG           zg30
TD               32768
SOLVENT          CDCl3
NS                   8
DS                   2
SWH           4807.692 Hz
FIDRES        0.146719 Hz
AQ            3.4078720 sec
RG                40.3
DW             104.000 usec
DE              10.00 usec
TE              298.0 K
D1          1.00000000 sec
TD0                  1

======== CHANNEL f1 ========
NUC1                1H
P1               6.90 usec
PL1             -2.00 dB
PL1W       23.88643074 W
SFO1       400.1620008 MHz

F2 - Processing parameters
SI                16384
SF         400.1600000 MHz
WDW                  EM
SSB       0
LB              0.30 Hz
GB        0
PC              1.00
```



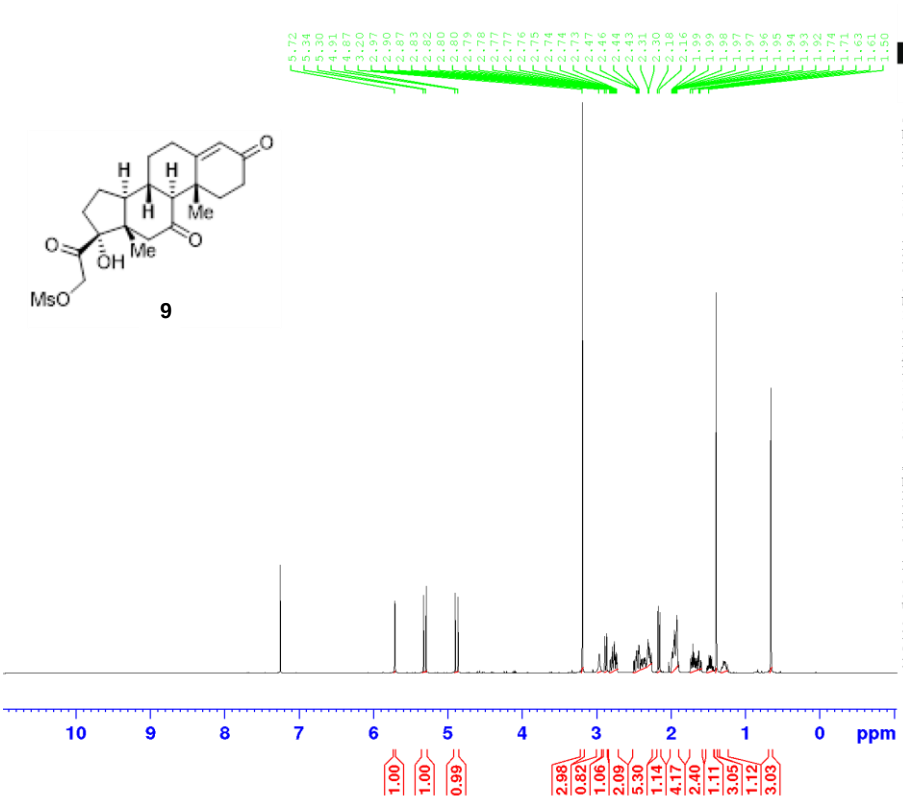

```
Current Data Parameters
NAME        BSCB-356 col
EXPNO                11
PROCNO                1

F2 - Acquisition Parameters
Date_          20180315
Time              20.15
INSTRUM           spect
PROBHD   5 mm PABBI 1H/
PULPROG          zgpg30
TD               65536
SOLVENT          CDCl3
NS                 512
DS                   4
SWH          24038.461 Hz
FIDRES        0.366798 Hz
AQ            1.3631488 sec
RG                26008
DW             20.800 usec
DE              10.00 usec
TE              298.0 K
D1          1.00000000 sec
D11         0.03000000 sec
TD0                  1

======== CHANNEL f1 ========
NUC1               13C
P1              13.50 usec
PL1             -5.00 dB
PL1W      119.14923859 W
SFO1       100.6303736 MHz

======== CHANNEL f2 ========
CPDPRG[2         waltz16
NUC2                1H
PCPD2           80.00 usec
PL2             -2.00 dB
PL12            19.16 dB
PL13            19.16 dB
PL2W       23.88643074 W
PL12W       0.18287371 W
PL13W       0.18287371 W
SFO2       400.1616006 MHz

F2 - Processing parameters
SI                32768
SF         100.6203120 MHz
WDW                  EM
SSB       0
LB              1.00 Hz
GB        0
PC              1.40
```

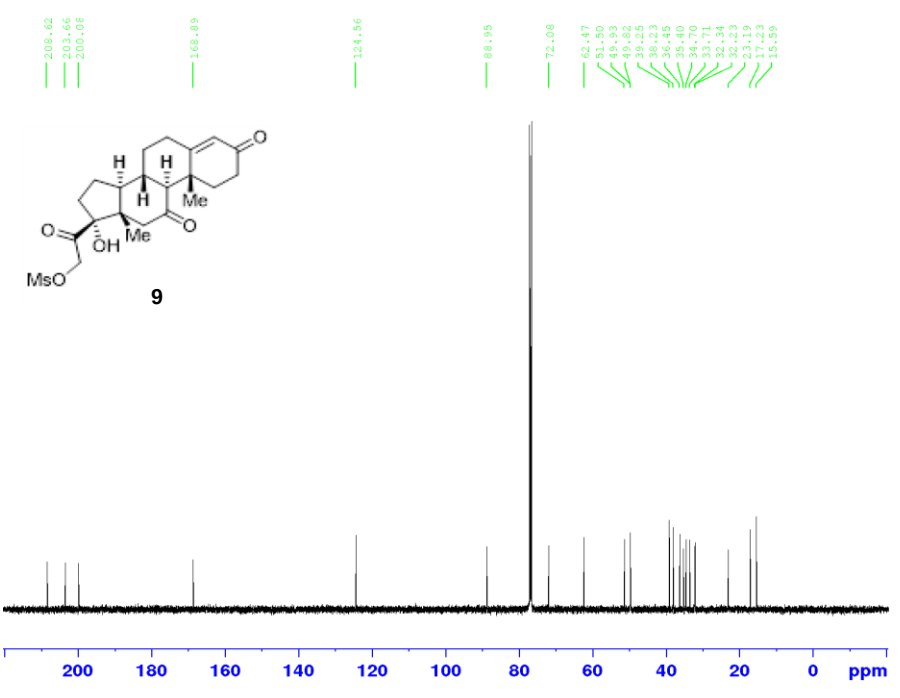



```
Current Data Parameters
NAME          mah-036-1H
EXPNO                 10
PROCNO                 1

F2 - Acquisition Parameters
Date_           20171218
Time               17.26
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG            zg30
TD                32768
SOLVENT           CDCl3
NS                  128
DS                    2
SWH           6009.615 Hz
FIDRES        0.183399 Hz
AQ            2.7262976 sec
RG                  161
DW               83.200 usec
DE               10.00 usec
TE                298.0 K
D1          1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                15.30 usec
PL1                3.00 dB
PL1W        12.58925438 W
SFO1        500.1325007 MHz

F2 - Processing parameters
SI                 16384
SF          500.1300138 MHz
WDW                   EM
SSB       0
LB                0.30 Hz
GB        0
PC                1.00
```



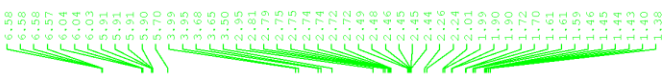

```
Current Data Parameters
NAME          mah-036-13C
EXPNO                 11
PROCNO                 1

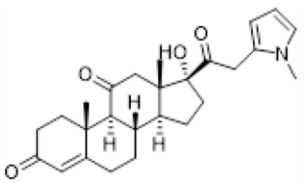
F2 - Acquisition Parameters
Date_           20171218
Time               18.36
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                  512
DS                    4
SWH          30303.031 Hz
FIDRES        0.462388 Hz
AQ            1.0813440 sec
RG                 9200
DW               16.500 usec
DE               10.00 usec
TE                298.1 K
D1          1.00000000 sec
D11         0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                 13C
P1                10.30 usec
PL1          0 dB
PL1W        75.35659027 W
SFO1       125.7703648 MHz

======== CHANNEL f2 ========
CPDPRG[2          waltz16
NUC2                  1H
PCPD2             80.00 usec
PL2                3.00 dB
PL12              17.54 dB
PL13              17.54 dB
PL2W        12.58925438 W
PL12W        0.44258827 W
PL13W        0.44258827 W
SFO2       500.1320005 MHz

F2 - Processing parameters
SI                 32768
SF         125.7577890 MHz
WDW                   EM
SSB       0
LB             1.00 Hz
GB        0
PC                1.40
```





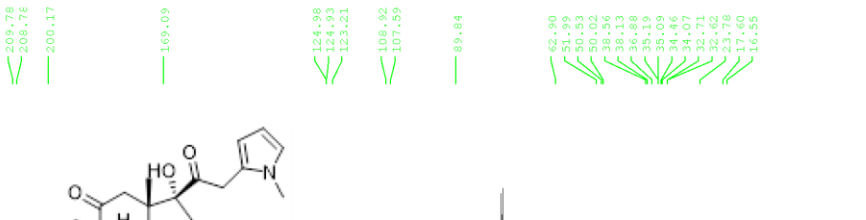
10

```
Current Data Parameters
NAME                  mah-150-1H
EXPNO                         10
PROCNO                         1

F2 - Acquisition Parameters
Date_                   20180313
Time                       23.09
INSTRUM                    spect
PROBHD    5 mm PATBO BB-
PULPROG                    zg30
TD                        32768
SOLVENT                   CDCl3
NS                          128
DS                            2
SWH                    6009.615 Hz
FIDRES                 0.183399 Hz
AQ                     2.7262976 sec
RG                         90.5
DW                       83.200 usec
DE                       10.00 usec
TE                        297.9 K
D1                    1.00000000 sec
TD0                            1

======== CHANNEL f1 ========
NUC1                          1H
P1                         14.90 usec
PL1                        -3.00 dB
PL1W                50.11872482 W
SFO1               500.1325007 MHz

F2 - Processing parameters
SI                         16384
SF                 500.1300210 MHz
WDW                           EM
SSB                0
LB                         0.30 Hz
GB                 0
PC                         1.00
```



10

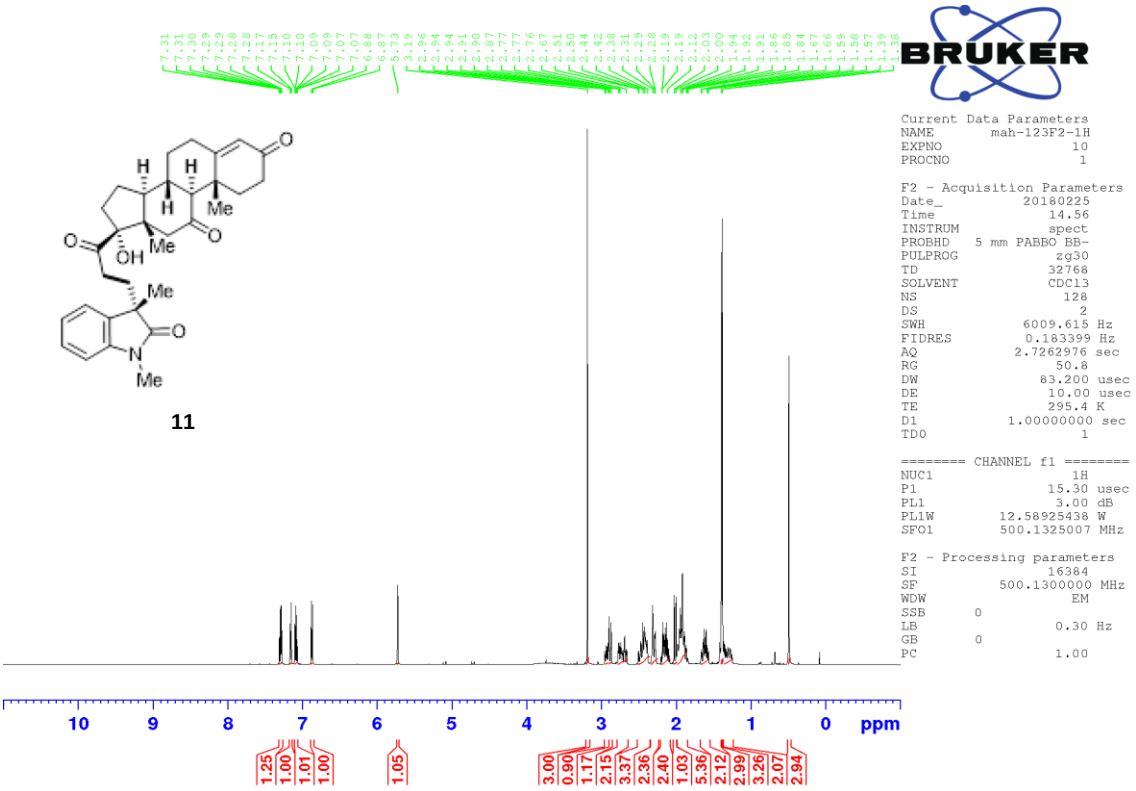
```
Current Data Parameters
NAME                  mah-150-13C
EXPNO                         10
PROCNO                         1

F2 - Acquisition Parameters
Date_                   20180313
Time                       23.28
INSTRUM                    spect
PROBHD    5 mm PATBO BB-
PULPROG                 zgpg30
TD                        65536
SOLVENT                   CDCl3
NS                          512
DS                            4
SWH                   30303.031 Hz
FIDRES                 0.462388 Hz
AQ                     1.0813440 sec
RG                         11500
DW                       16.500 usec
DE                       10.00 usec
TE                        299.6 K
D1                    1.00000000 sec
D11                   0.03000000 sec
TD0                            1

======== CHANNEL f1 ========
NUC1                          13C
P1                          9.75 usec
PL1                        -1.00 dB
PL1W                94.86833191 W
SFO1               125.7703648 MHz

======== CHANNEL f2 ========
CPDPRG[2                waltz16
NUC2                          1H
PCPD2                      80.00 usec
PL2                        -3.00 dB
PL12                       11.96 dB
PL13                       11.96 dB
PL2W                50.11872482 W
PL12W                1.59955800 W
PL13W                1.59955800 W
SFO2               500.1320005 MHz

F2 - Processing parameters
SI                         32768
SF                 125.7577441 MHz
WDW                           EM
SSB                0
LB                         1.00 Hz
GB                 0
PC                         1.40
```



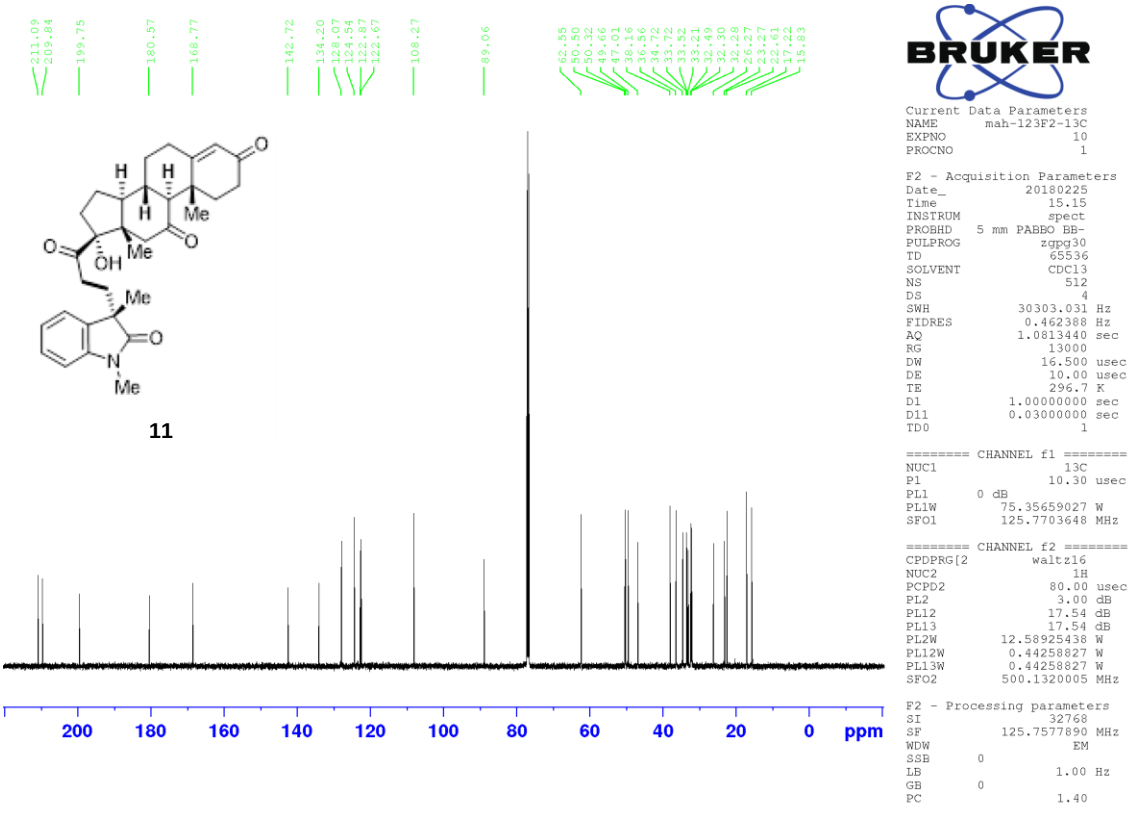
BRUKER

```
Current Data Parameters
NAME        mah-123F2-1H
EXPNO                 10
PROCNO                 1

F2 - Acquisition Parameters
Date_           20180225
Time               14.56
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG             zg30
TD                 32768
SOLVENT            CDCl3
NS                   128
DS                     2
SWH             6009.615 Hz
FIDRES          0.183399 Hz
AQ              2.7262976 sec
RG                  50.8
DW                83.200 usec
DE                10.00 usec
TE                295.4 K
D1            1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                 15.30 usec
PL1                3.00 dB
PL1W         12.58925438 W
SFO1         500.1325007 MHz

F2 - Processing parameters
SI                 16384
SF          500.1300000 MHz
WDW                   EM
SSB       0
LB                0.30 Hz
GB        0
PC                1.00
```



BRUKER

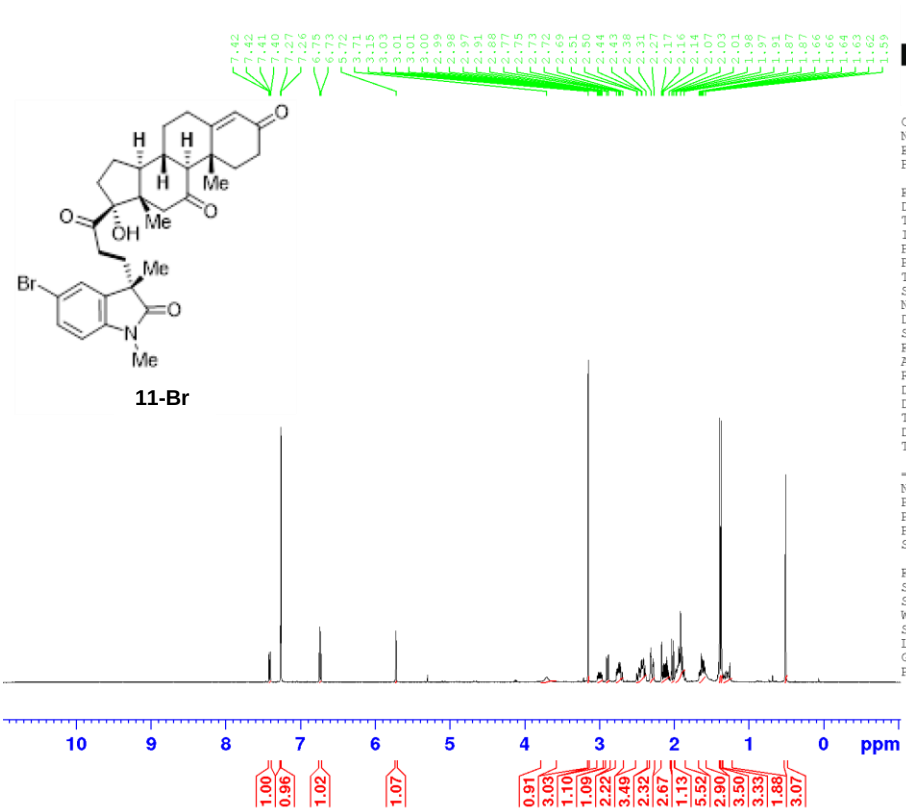
```
Current Data Parameters
NAME       mah-123F2-13C
EXPNO                 10
PROCNO                 1

F2 - Acquisition Parameters
Date_           20180225
Time               15.15
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG          zgpg30
TD                 65536
SOLVENT            CDCl3
NS                   512
DS                     4
SWH            30303.031 Hz
FIDRES          0.462388 Hz
AQ              1.0813440 sec
RG                 13000
DW                16.500 usec
DE                10.00 usec
TE                296.7 K
D1            1.00000000 sec
D11           0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                 13C
P1                10.30 usec
PL1       0 dB
PL1W         75.35659027 W
SFO1         125.7703648 MHz

======== CHANNEL f2 ========
CPDPRG[2          waltz16
NUC2                  1H
PCPD2              80.00 usec
PL2                3.00 dB
PL12               17.54 dB
PL13               17.54 dB
PL2W         12.58925438 W
PL12W         0.44258827 W
PL13W         0.44258827 W
SFO2         500.1320005 MHz

F2 - Processing parameters
SI                 32768
SF          125.7577890 MHz
WDW                   EM
SSB       0
LB                1.00 Hz
GB        0
PC                1.40
```

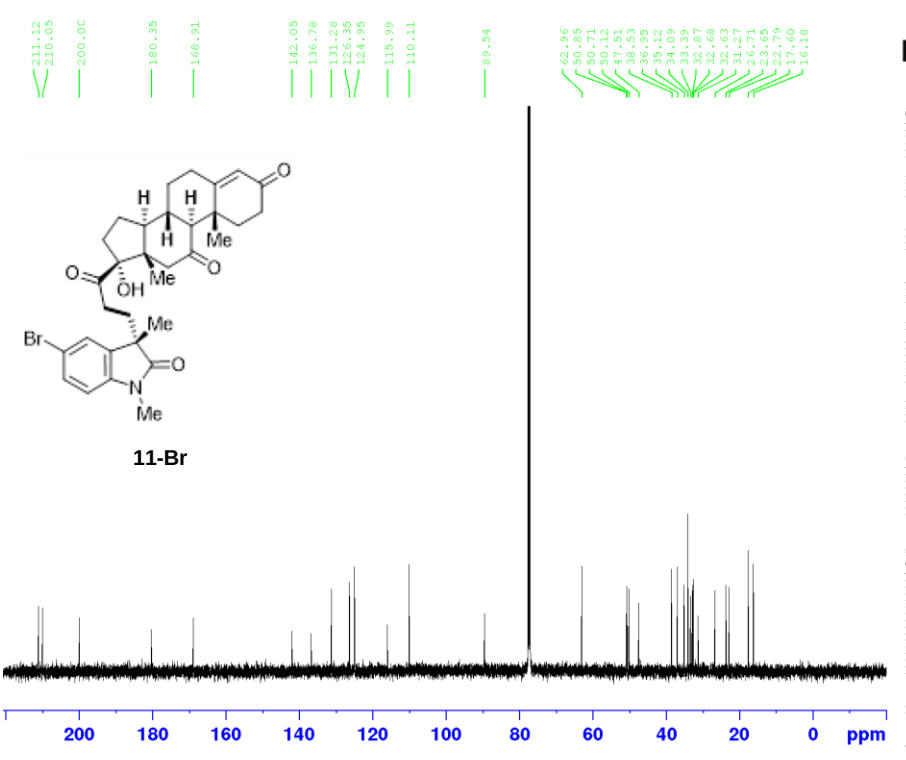



```
Current Data Parameters
NAME                  mah-144-1H
EXPNO                         20
PROCNO                         1

F2 - Acquisition Parameters
Date_                  20180327
Time                      13.08
INSTRUM                   spect
PROBHD     5 mm PATBO BB-
PULPROG                    zg30
TD                        32768
SOLVENT                   CDCl3
NS                            8
DS                            2
SWH                    6009.615 Hz
FIDRES                 0.183399 Hz
AQ                     2.7262976 sec
RG                          575
DW                       83.200 usec
DE                       10.00 usec
TE                       300.0 K
D1                    1.00000000 sec
TD0                           1

======== CHANNEL f1 ========
NUC1                         1H
P1                        14.90 usec
PL1                       -3.00 dB
PL1W                 50.11872482 W
SFO1                500.1325007 MHz

F2 - Processing parameters
SI                        16384
SF                  500.1300133 MHz
WDW                          EM
SSB                 0
LB                        0.30 Hz
GB                  0
PC                        1.00
```



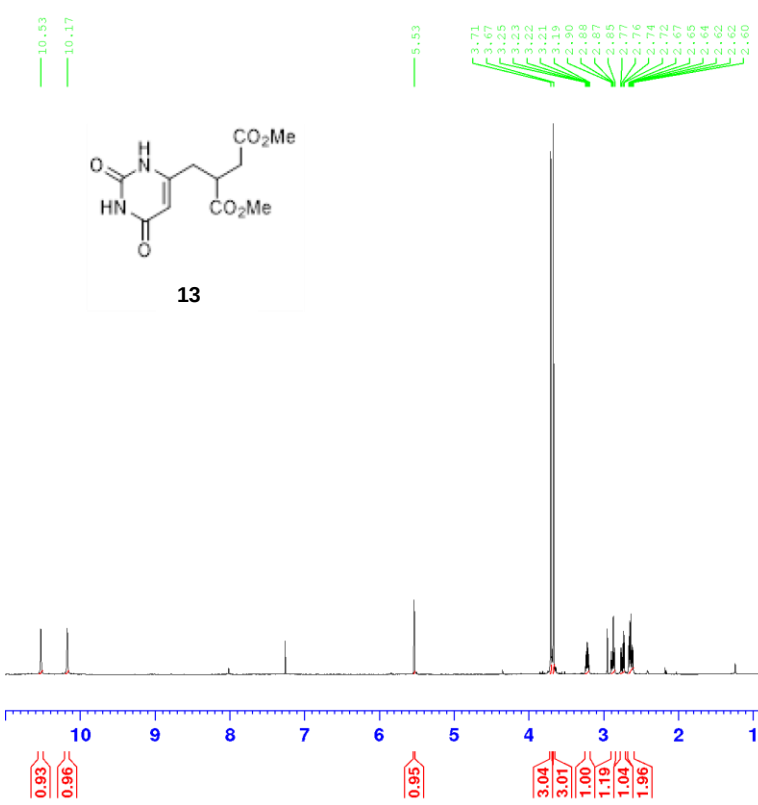

```
Current Data Parameters
NAME              mah-144-13C-2048
EXPNO                         10
PROCNO                         1

F2 - Acquisition Parameters
Date_                  20180328
Time                      0.13
INSTRUM                   spect
PROBHD     5 mm PATBO BB-
PULPROG                 zgpg30
TD                        65536
SOLVENT                   CDCl3
NS                         2048
DS                            4
SWH                30303.031 Hz
FIDRES             0.462388 Hz
AQ                 1.0813440 sec
RG                        11500
DW                       16.500 usec
DE                       10.00 usec
TE                       299.9 K
D1                 1.00000000 sec
D11                0.03000000 sec
TD0                           1

======== CHANNEL f1 ========
NUC1                         13C
P1                         9.75 usec
PL1                       -1.00 dB
PL1W                94.86833191 W
SFO1               125.7703648 MHz

======== CHANNEL f2 ========
CPDPRG[2                waltz16
NUC2                         1H
PCPD2                     80.00 usec
PL2                       -3.00 dB
PL12                      11.96 dB
PL13                      11.96 dB
PL2W                50.11872482 W
PL12W                1.59955800 W
PL13W                1.59955800 W
SFO2               500.1320005 MHz

F2 - Processing parameters
SI                        32768
SF                 125.7577441 MHz
WDW                          EM
SSB                 0
LB                        1.00 Hz
GB                  0
PC                        1.40
```

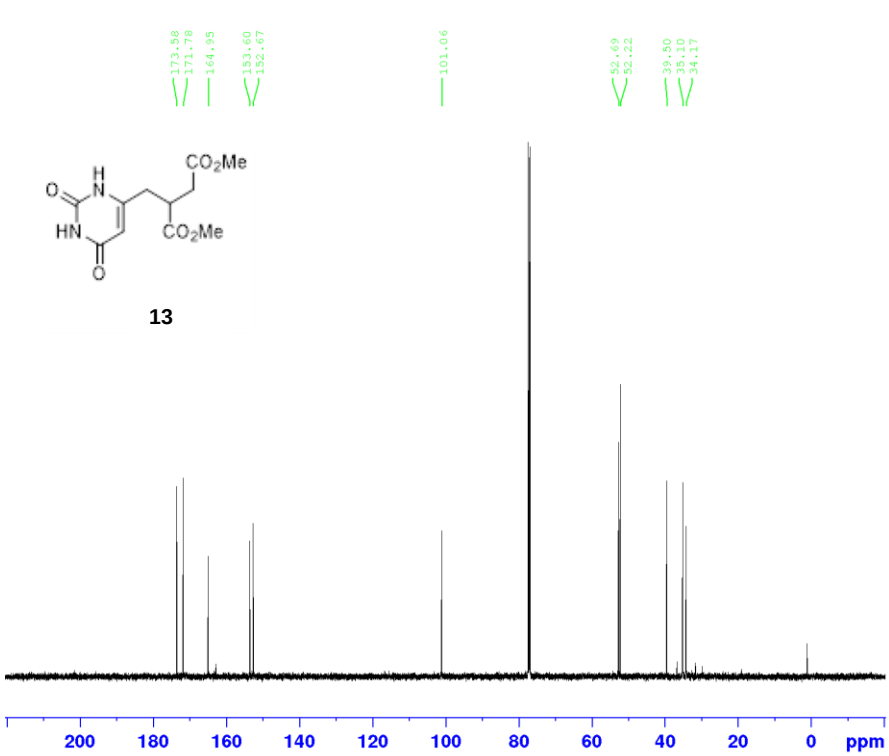



```
Current Data Parameters
NAME      epb239-prod_10
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20180327
Time               6.19
INSTRUM           spect
PROBHD   5 mm PATBO BB-
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                     8
DS                     2
SWH            10000.000 Hz
FIDRES             0.152588 Hz
AQ             3.2767999 sec
RG                  90.5
DW                50.000 usec
DE                10.00 usec
TE                300.0 K
D1             1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                 14.90 usec
PL1                -3.00 dB
PL1W         50.11872482 W
SFO1         500.1330008 MHz

F2 - Processing parameters
SI                 32768
SF           500.1300130 MHz
WDW                   EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```



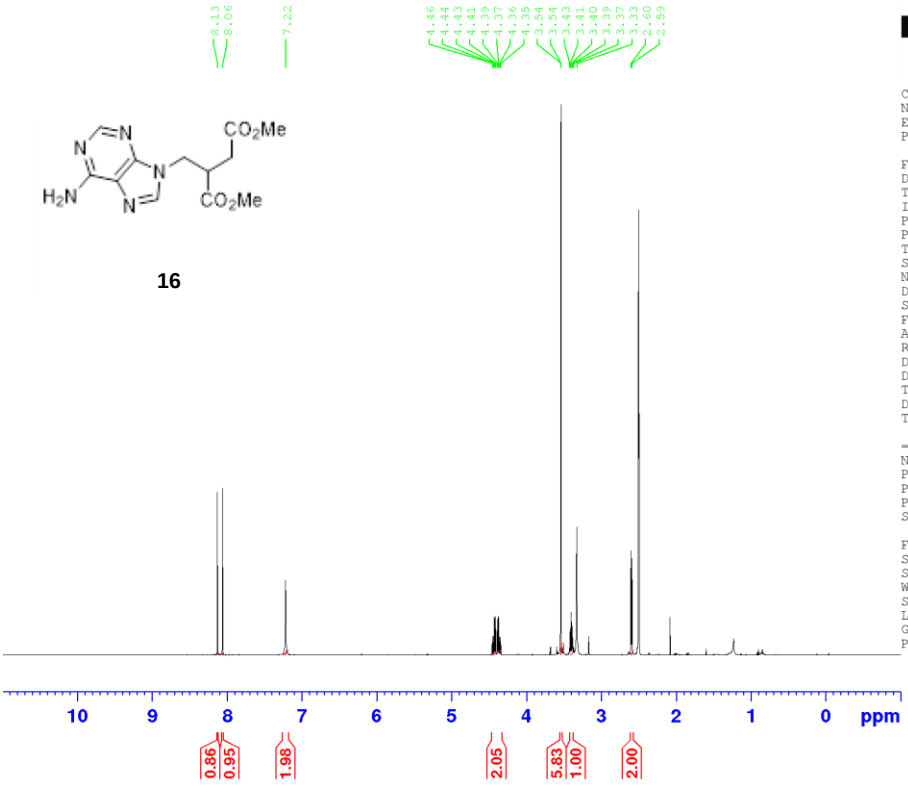

```
Current Data Parameters
NAME      epb239-prod-13C_10
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20180327
Time               6.38
INSTRUM           spect
PROBHD   5 mm PATBO BB-
PULPROG           zgpg30
TD                 65536
SOLVENT            CDCl3
NS                   512
DS                     4
SWH            30303.031 Hz
FIDRES             0.462388 Hz
AQ             1.0813440 sec
RG                 11500
DW                16.500 usec
DE                10.00 usec
TE                300.0 K
D1             1.00000000 sec
D11            0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                 13C
P1                  9.75 usec
PL1                -1.00 dB
PL1W         94.86833191 W
SFO1         125.7703648 MHz

======== CHANNEL f2 ========
CPDPRG[2          waltz16
NUC2                  1H
PCPD2              80.00 usec
PL2                -3.00 dB
PL12               11.96 dB
PL13               11.96 dB
PL2W         50.11872482 W
PL12W         1.59955800 W
PL13W         1.59955800 W
SFO2         500.1320005 MHz

F2 - Processing parameters
SI                 32768
SF           125.7577759 MHz
WDW                   EM
SSB       0
LB                 1.00 Hz
GB        0
PC                 1.40
```



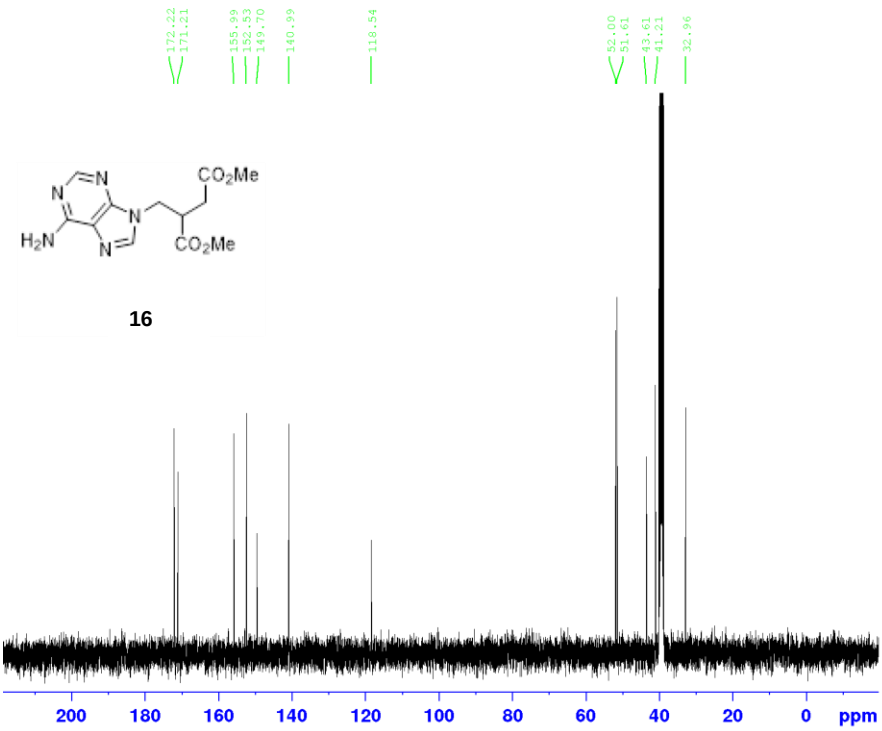
BRUKER

```
Current Data Parameters
NAME      epb303-prod_11
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20180421
Time               5.31
INSTRUM           spect
PROBHD   5 mm PATBO BB-
PULPROG            zg30
TD                65536
SOLVENT            DMSO
NS                  128
DS                    2
SWH           10000.000 Hz
FIDRES            0.152588 Hz
AQ             3.2767999 sec
RG                  322
DW               50.000 usec
DE               10.00 usec
TE                298.0 K
D1            1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                 14.90 usec
PL1                -3.00 dB
PL1W         50.11872482 W
SFO1        500.1330008 MHz

F2 - Processing parameters
SI                 32768
SF          500.1300054 MHz
WDW                   EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```



BRUKER

```
Current Data Parameters
NAME      epb303-prod-13C_10
EXPNO                    1
PROCNO                   1

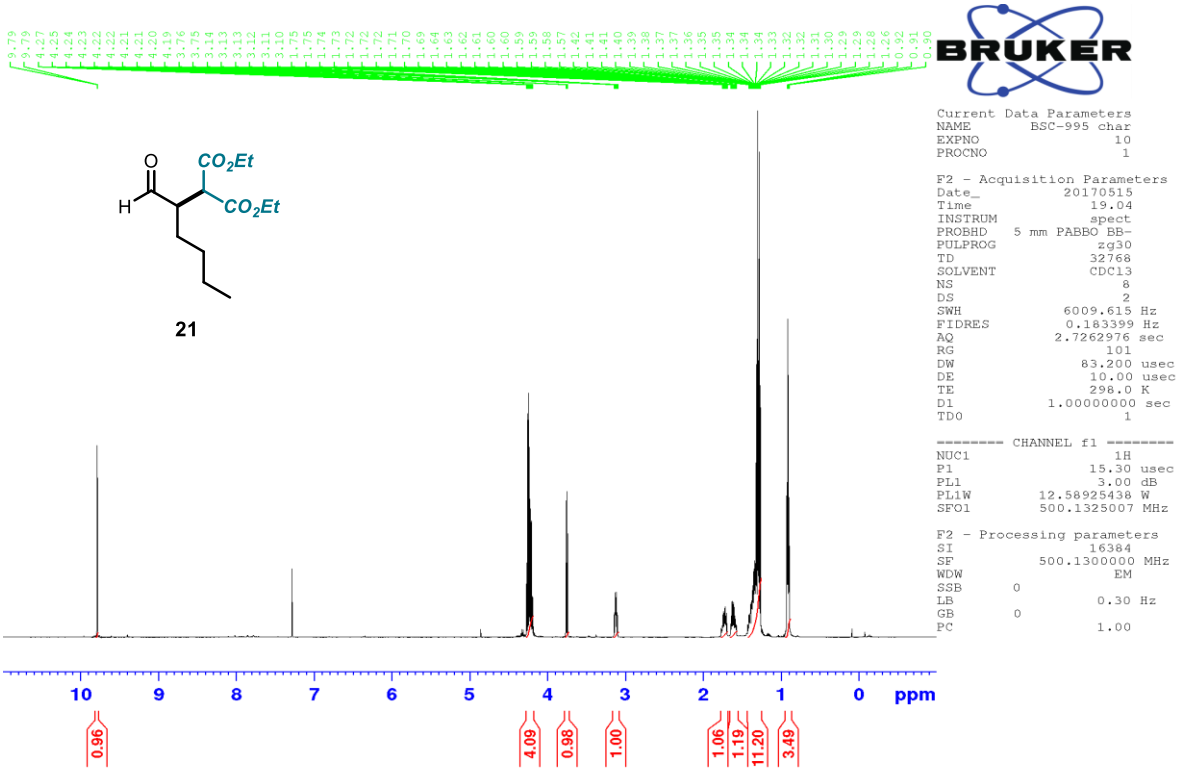
F2 - Acquisition Parameters
Date_           20180423
Time               10.09
INSTRUM           spect
PROBHD   5 mm PABBO BB-
PULPROG          zgpg30
TD                65536
SOLVENT            DMSO
NS                   512
DS                     4
SWH           23980.814 Hz
FIDRES            0.365918 Hz
AQ             1.3664256 sec
RG                 23170.5
DW               20.850 usec
DE               10.00 usec
TE                298.4 K
D1            1.00000000 sec
D11           0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  13C
P1                 10.00 usec
PL1                -3.00 dB
PL1W         75.17808533 W
SFO1        100.6228298 MHz

======== CHANNEL f2 ========
CPDPRG[2          waltz16
NUC2                  1H
PCPD2              80.00 usec
PL2                -3.00 dB
PL12               13.00 dB
PL13               13.00 dB
PL2W         30.07123375 W
PL12W         0.75535524 W
PL13W         0.75535524 W
SFO2        400.1316005 MHz

F2 - Processing parameters
SI                 32768
SF          100.6128143 MHz
WDW                   EM
SSB       0
LB                 1.00 Hz
GB        0
PC                 1.40
```

Asymmetric α-alkylation of aldehydes:

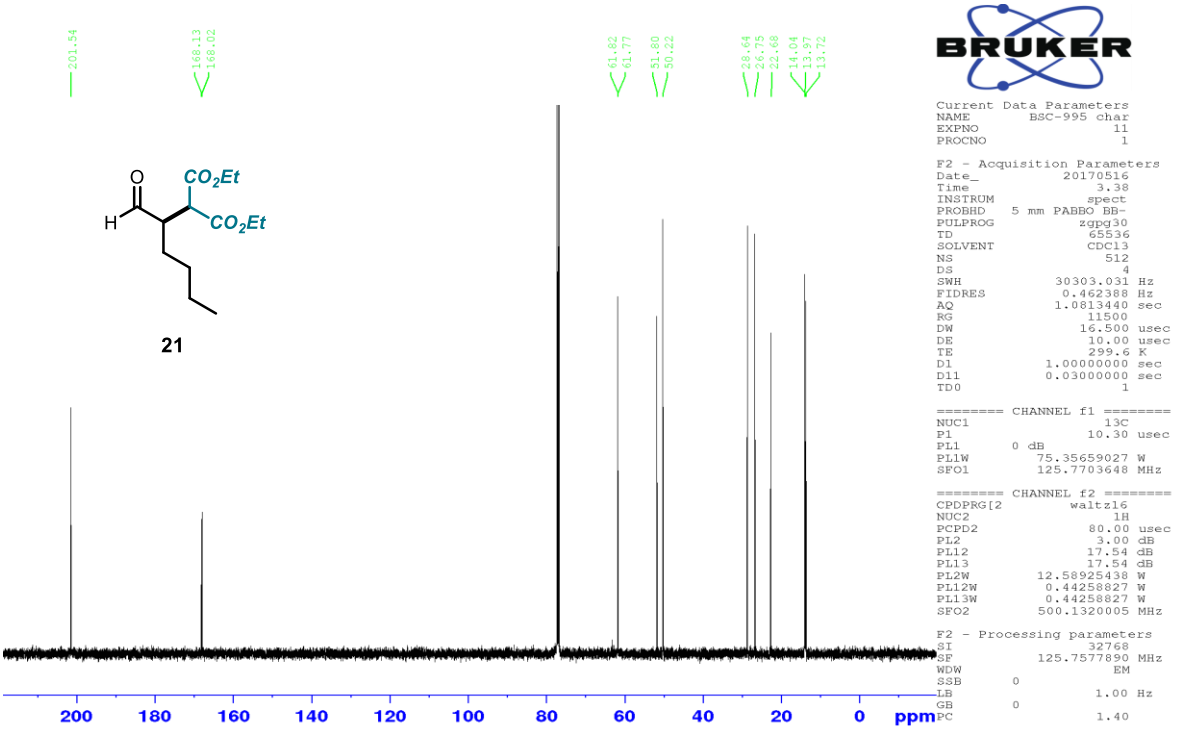



```
Current Data Parameters
NAME        BSC-995 char
EXPNO                10
PROCNO                1

F2 - Acquisition Parameters
Date_            20170515
Time                19.04
INSTRUM             spect
PROBHD    5 mm PABBO BB-
PULPROG             zg30
TD                 32768
SOLVENT            CDCl3
NS                     8
DS                     2
SWH             6009.615 Hz
FIDRES          0.183399 Hz
AQ             2.7262976 sec
RG                   101
DW                83.200 usec
DE                10.00 usec
TE                298.0 K
D1             1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                 15.30 usec
PL1                 3.00 dB
PL1W         12.58925438 W
SFO1        500.1325007 MHz

F2 - Processing parameters
SI                 16384
SF          500.1300000 MHz
WDW                   EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```



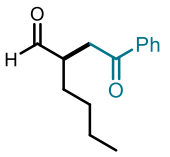

```
Current Data Parameters
NAME        BSC-995 char
EXPNO                11
PROCNO                1

F2 - Acquisition Parameters
Date_            20170516
Time                3.38
INSTRUM             spect
PROBHD    5 mm PABBO BB-
PULPROG           zgpg30
TD                 65536
SOLVENT            CDCl3
NS                   512
DS                     4
SWH            30303.031 Hz
FIDRES          0.462388 Hz
AQ             1.0813440 sec
RG                 11500
DW                16.500 usec
DE                10.00 usec
TE                299.6 K
D1             1.00000000 sec
D11            0.03000000 sec
TD0                    1

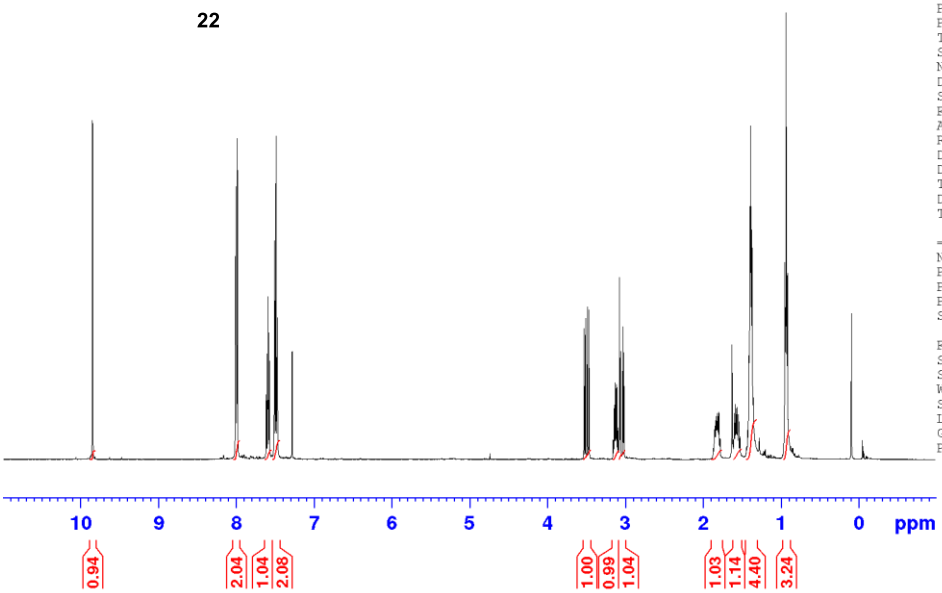
======== CHANNEL f1 ========
NUC1                 13C
P1                 10.30 usec
PL1        0 dB
PL1W         75.35659027 W
SFO1        125.7703648 MHz

======== CHANNEL f2 ========
CPDPRG[2          waltz16
NUC2                  1H
PCPD2              80.00 usec
PL2                 3.00 dB
PL12               17.54 dB
PL13               17.54 dB
PL2W         12.58925438 W
PL12W         0.44258827 W
PL13W         0.44258827 W
SFO2        500.1320005 MHz

F2 - Processing parameters
SI                 32768
SF          125.7577890 MHz
WDW                   EM
SSB       0
LB                 1.00 Hz
GB        0
PC                 1.40
```



22

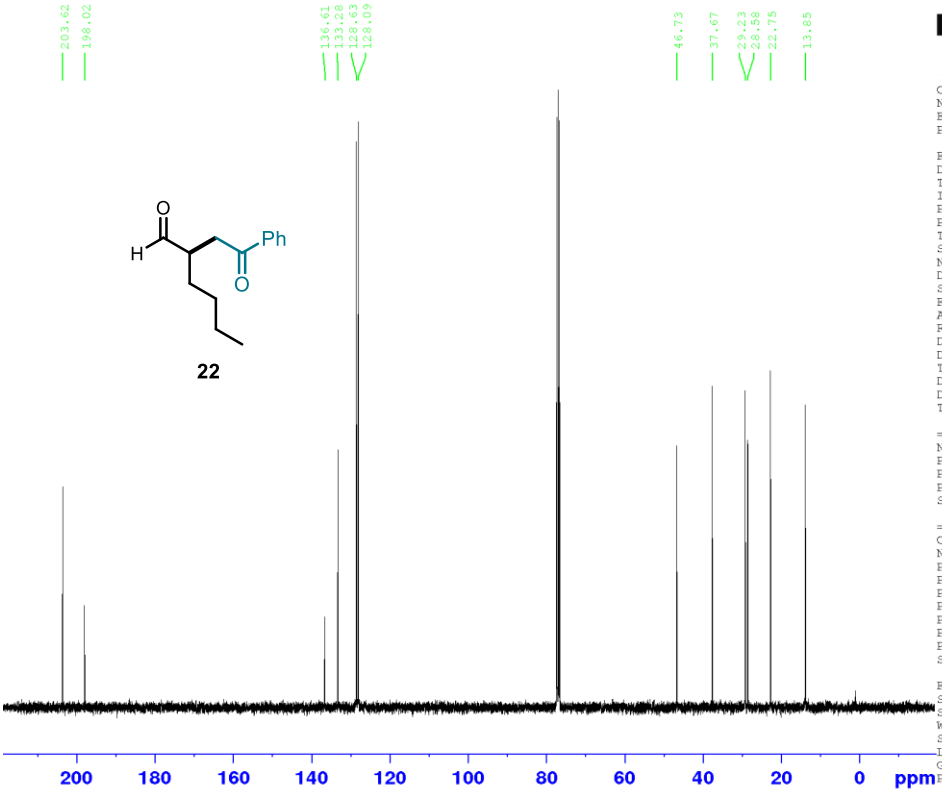



```
Current Data Parameters
NAME      BSCB-025-02 clean
EXPNO                   10
PROCNO                   1

F2 - Acquisition Parameters
Date_           20170601
Time               18.59
INSTRUM            spect
PROBHD    5 mm PABBO BB-
PULPROG             zg30
TD                 32768
SOLVENT            CDCl3
NS                     8
DS                     2
SWH             4789.272 Hz
FIDRES          0.146157 Hz
AQ             3.4209793 sec
RG                   181
DW              104.400 usec
DE                6.00 usec
TE                298.0 K
D1            1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                 14.50 usec
PL1                -2.00 dB
PL1W         23.88643074 W
SFO1         400.1320007 MHz

F2 - Processing parameters
SI                 16384
SF           400.1300000 MHz
WDW                   EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```



22

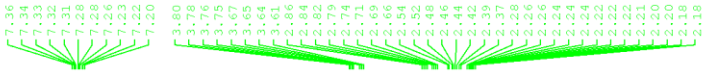
```
Current Data Parameters
NAME      BSCB-025-02 clean
EXPNO                   11
PROCNO                   1

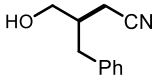
F2 - Acquisition Parameters
Date_           20170602
Time               4.20
INSTRUM            spect
PROBHD    5 mm PABBO BB-
PULPROG           zgpg30
TD                 65536
SOLVENT            CDCl3
NS                   512
DS                     4
SWH            23980.814 Hz
FIDRES          0.365918 Hz
AQ             1.3664256 sec
RG                 26008
DW              20.850 usec
DE                10.00 usec
TE                298.2 K
D1            1.00000000 sec
D11           0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                 13C
P1                 10.00 usec
PL1                -3.00 dB
PL1W         75.17808533 W
SFO1         100.6228298 MHz

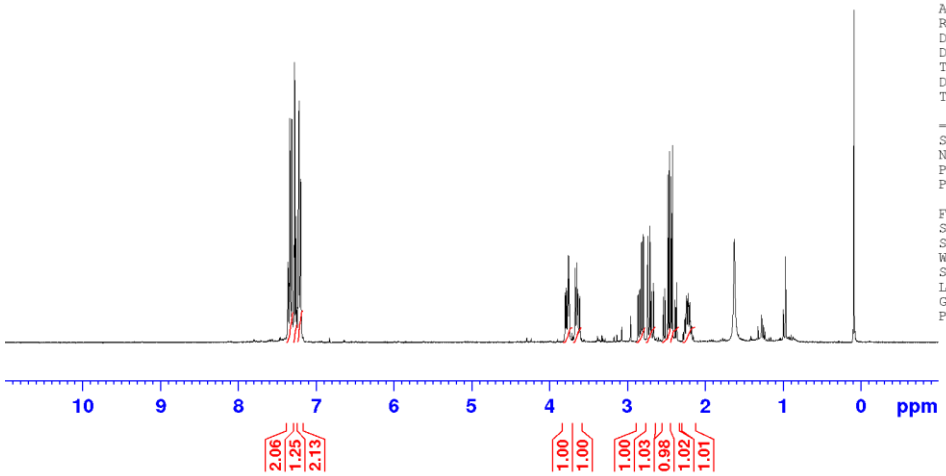
======== CHANNEL f2 ========
CPDPRG[2          waltz16
NUC2                  1H
PCPD2              80.00 usec
PL2                -3.00 dB
PL12               13.00 dB
PL13               13.00 dB
PL2W         30.07123375 W
PL12W         0.75535524 W
PL13W         0.75535524 W
SFO2         400.1316005 MHz

F2 - Processing parameters
SI                 32768
SF           100.6127690 MHz
WDW                   EM
SSB       0
LB                 1.00 Hz
GB        0
PC                 1.40
```





21



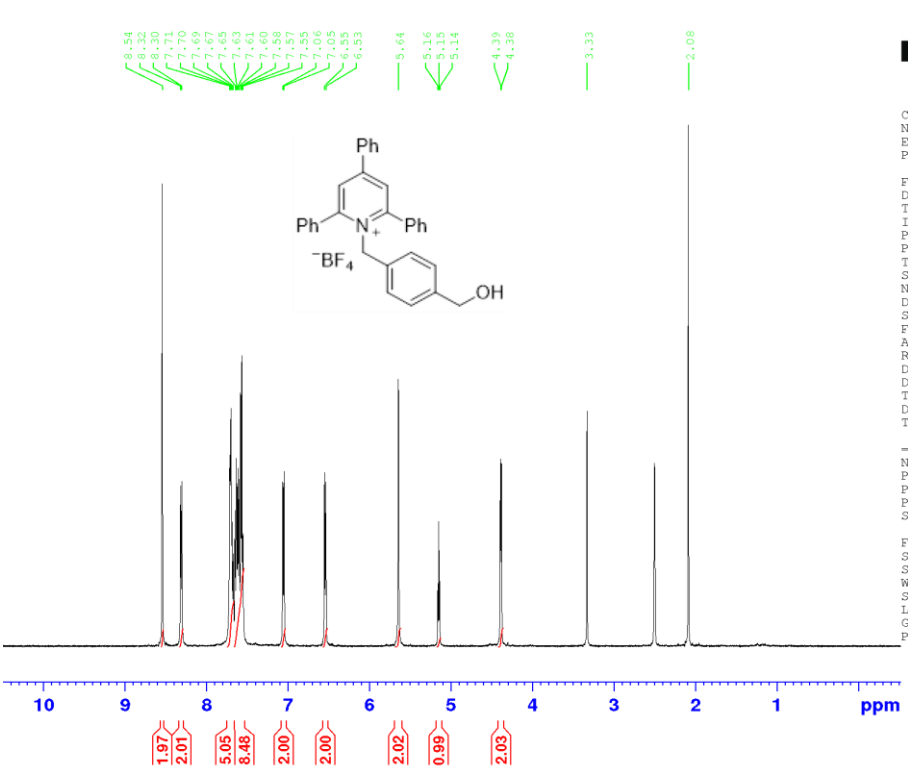

```
Current Data Parameters
NAME         BSC-771 col
EXPNO                 10
PROCNO                 1

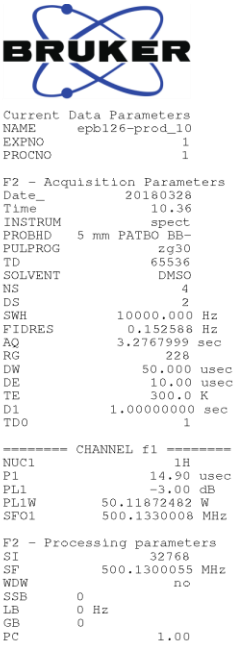
F2 - Acquisition Parameters
Date_           20161201
Time              10.12
INSTRUM       FOURIER300
PROBHD    5 mm DUL 13C-1
PULPROG            zg30
TD                65536
SOLVENT           CDCl3
NS                    8
DS                    2
SWH            6103.516 Hz
FIDRES          0.093132 Hz
AQ             5.3687091 sec
RG               59.9415
DW               81.920 usec
DE                6.50 usec
TE                298.0 K
D1           1.00000000 sec
TD0                   1

======== CHANNEL f1 ========
SFO1         300.1818011 MHz
NUC1                 1H
P1                11.75 usec
PLW1         20.00000000 W

F2 - Processing parameters
SI                32768
SF           300.1800000 MHz
WDW                  EM
SSB       0
LB              0.30 Hz
GB        0
PC              1.40
```

Substrate Synthesis



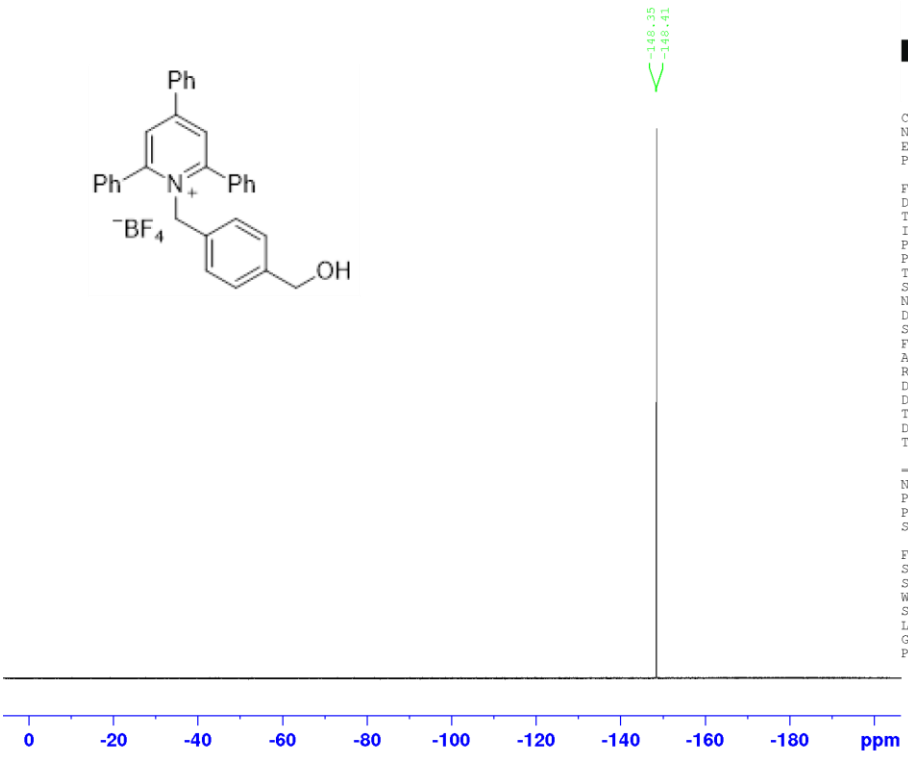


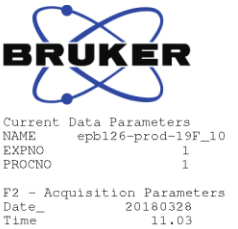
```
Current Data Parameters
NAME      epb126-prod_10
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20180328
Time               10.36
INSTRUM            spect
PROBHD   5 mm PATBO BB-
PULPROG             zg30
TD                 65536
SOLVENT             DMSO
NS                     4
DS                     2
SWH          10000.000 Hz
FIDRES           0.152588 Hz
AQ             3.2767999 sec
RG                   228
DW                50.000 usec
DE                10.00 usec
TE                300.0 K
D1            1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                14.90 usec
PL1               -3.00 dB
PL1W        50.11872482 W
SFO1       500.1330008 MHz

F2 - Processing parameters
SI                 32768
SF          500.1300055 MHz
WDW                   no
SSB       0
LB        0 Hz
GB        0
PC                  1.00
```



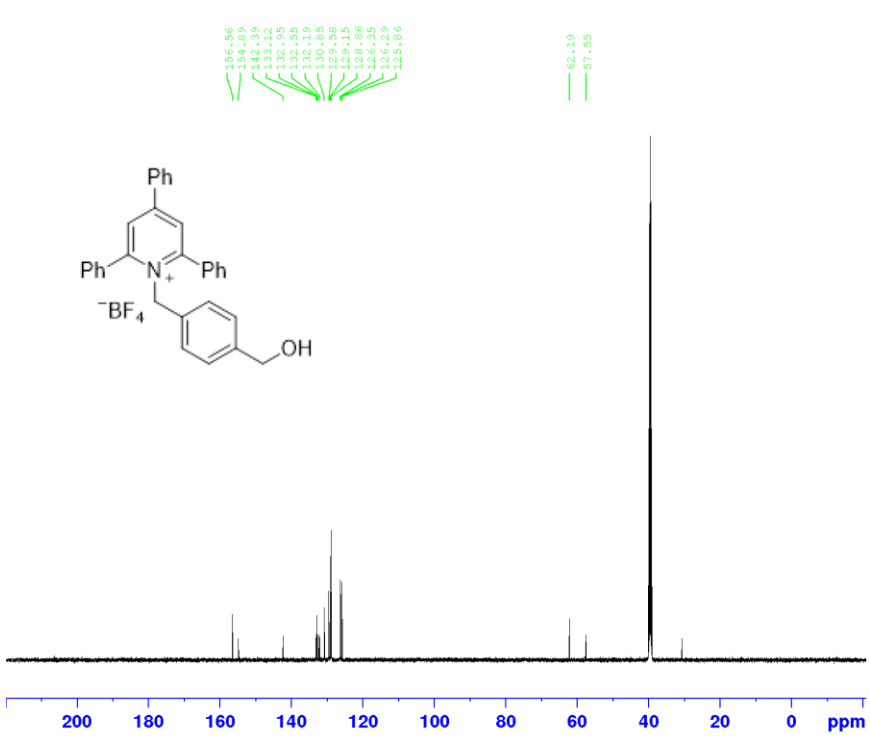


```
Current Data Parameters
NAME      epb126-prod-19F_10
EXPNO                    1
PROCNO                   1

F2 - Acquisition Parameters
Date_           20180328
Time               11.03
INSTRUM            spect
PROBHD   5 mm PATBO BB-
PULPROG             zg30
TD                131072
SOLVENT             DMSO
NS                     8
DS                     2
SWH         100000.000 Hz
FIDRES           0.762939 Hz
AQ             0.6553600 sec
RG                   322
DW                5.000 usec
DE                6.50 usec
TE                298.1 K
D1            5.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                 19F
P1                19.50 usec
PL1       0 dB
SFO1       470.5453180 MHz

F2 - Processing parameters
SI                 65536
SF          470.5924245 MHz
WDW                   no
SSB       0
LB        0 Hz
GB        0
PC                  1.00
```




```
Current Data Parameters
NAME      epb126-prod-13C_10
EXPNO                      1
PROCNO                     1

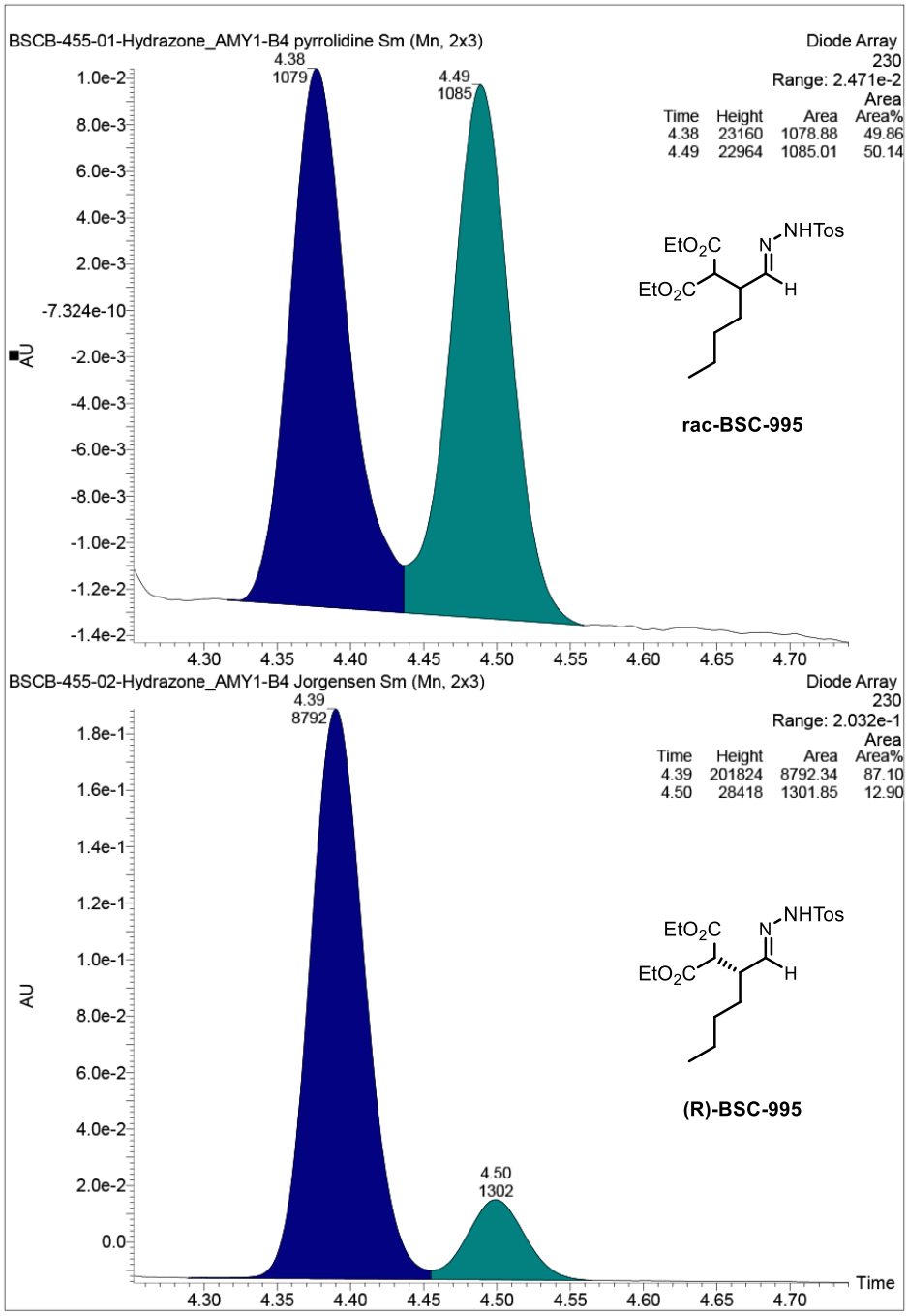
F2 - Acquisition Parameters
Date_              20180328
Time                  10.56
INSTRUM               spect
PROBHD    5 mm PATBO BB-
PULPROG             zgpg30
TD                   65536
SOLVENT                DMSO
NS                       512
DS                         4
SWH              30303.031 Hz
FIDRES            0.462388 Hz
AQ               1.0813440 sec
RG                    6500
DW                  16.500 usec
DE                  10.00 usec
TE                  299.4 K
D1               1.00000000 sec
D11              0.03000000 sec
TD0                        1

======== CHANNEL f1 ========
NUC1                    13C
P1                     9.75 usec
PL1                   -1.00 dB
PL1W           94.86833191 W
SFO1          125.7703648 MHz

======== CHANNEL f2 ========
CPDPRG[2            waltz16
NUC2                    1H
PCPD2                80.00 usec
PL2                  -3.00 dB
PL12                 11.96 dB
PL13                 11.96 dB
PL2W           50.11872482 W
PL12W           1.59955800 W
PL13W           1.59955800 W
SFO2          500.1320005 MHz

F2 - Processing parameters
SI                     32768
SF            125.7578485 MHz
WDW                       EM
SSB         0
LB                    1.00 Hz
GB          0
PC                    1.40
```

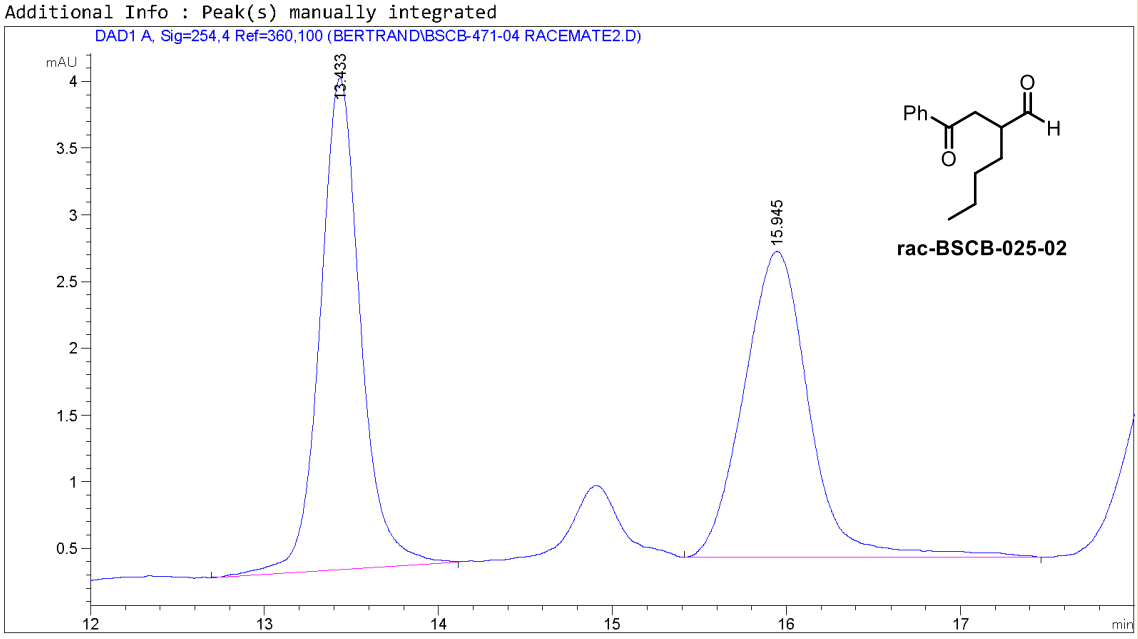
HPLC/UPC^2 traces



Data File C:\CHEM32\1\DATA\BERTRAND\BSCB-471-04 RACEMATE2.D
Sample Name: BSCB-471-04 racemate2

```
=====================================================================
Acq. Operator   : Bertrand
Acq. Instrument : HPLC 1260                      Location : Vial 81
Injection Date  : 7/26/2018 6:05:26 PM
                                                 Inj Volume : 2.000 µl
Acq. Method     : C:\CHEM32\1\METHODS\BSC 98-2.M
Last changed    : 7/26/2018 5:25:13 PM by Bertrand
                  (modified after loading)
Analysis Method : C:\CHEM32\1\METHODS\BSC 98-2.M
Last changed    : 7/27/2018 9:34:26 AM by Bertrand
                  (modified after loading)
Sample Info     : BSCB-471-04 racemate, sample in hexane, column IA, Hex:IPA = 98:2, flow: 0.
                  8 mL/min, 20 deg

Additional Info : Peak(s) manually integrated
```




```
=====================================================================
                        Area Percent Report
=====================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Sample Amount:        :      1.00000  [ng/ul]   (not used in calc.)
Use Multiplier & Dilution Factor with ISTDs

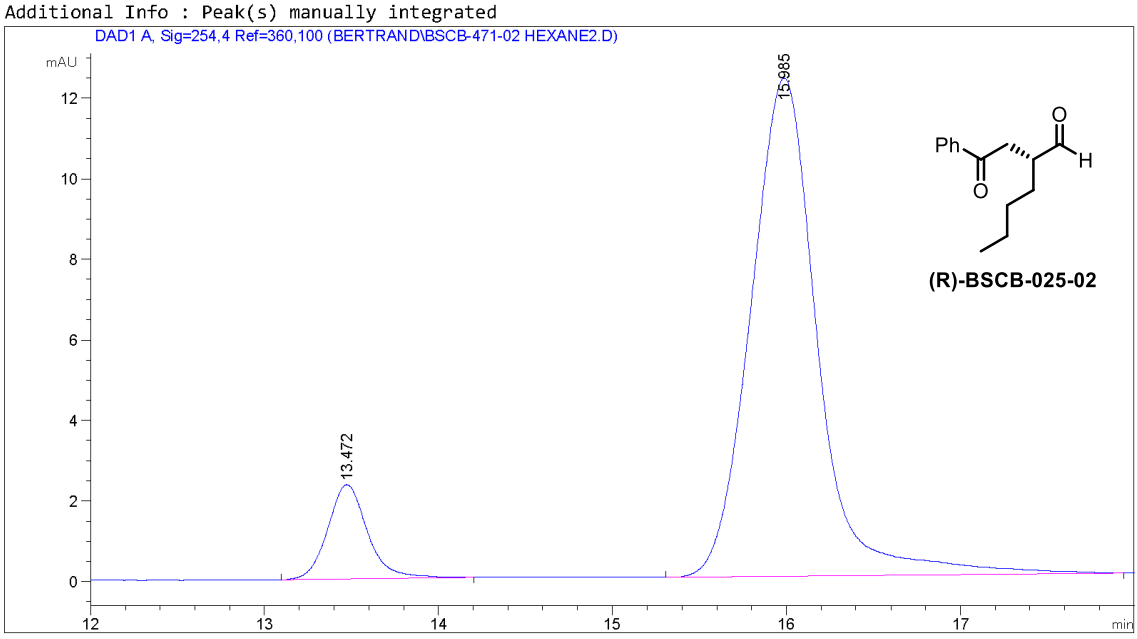

Signal 1: DAD1 A, Sig=254,4 Ref=360,100

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1  13.433 BB    0.2428   59.58977    3.68253  49.9601
   2  15.945 BB    0.3938   59.68485    2.29231  50.0399
```

Data File C:\CHEM32\1\DATA\BERTRAND\BSCB-471-02 HEXANE2.D
Sample Name: BSCB-471-02 hexane2

```
=====================================================================
Acq. Operator   : Bertrand
Acq. Instrument : HPLC 1260                    Location : Vial 71
Injection Date  : 7/26/2018 7:34:46 PM
                                              Inj Volume : 2.000 µl
Acq. Method     : C:\CHEM32\1\METHODS\BSC 98-2.M
Last changed    : 7/26/2018 7:33:47 PM by Bertrand
                  (modified after loading)
Analysis Method : C:\CHEM32\1\METHODS\BSC 98-2.M
Last changed    : 7/27/2018 9:31:19 AM by Bertrand
                  (modified after loading)
Sample Info     : BSCB-471-02 racemate, sample in hexane, column IA, Hex:IPA = 98:2, flow: 0.
                  8 mL/min, 20 deg

Additional Info : Peak(s) manually integrated
```

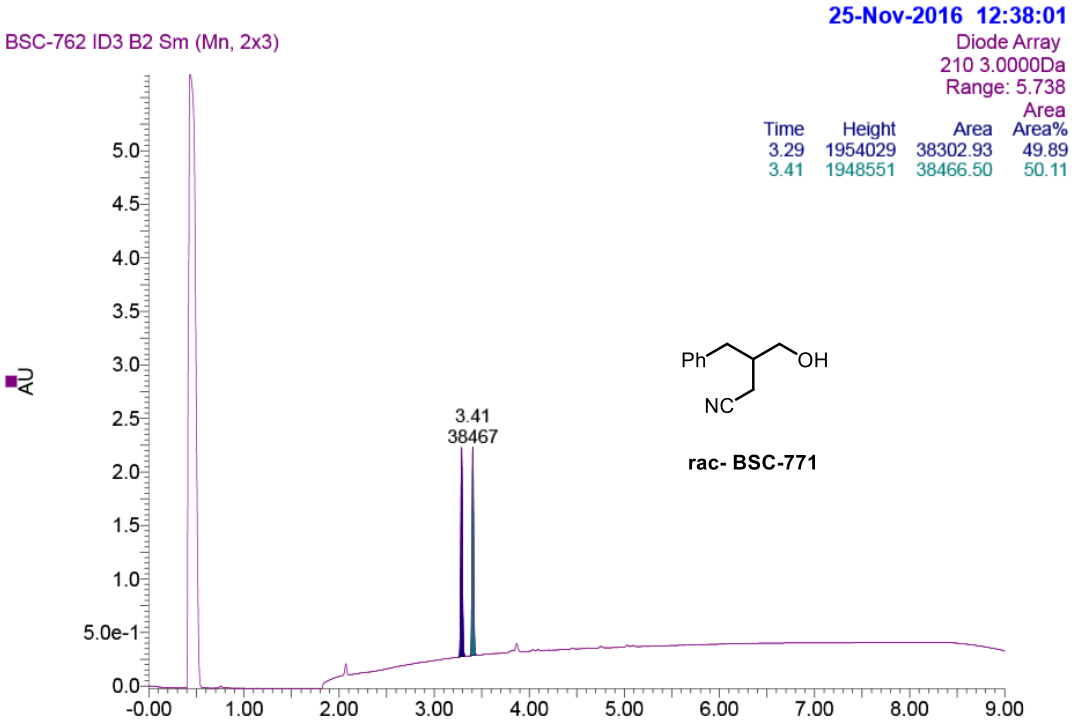


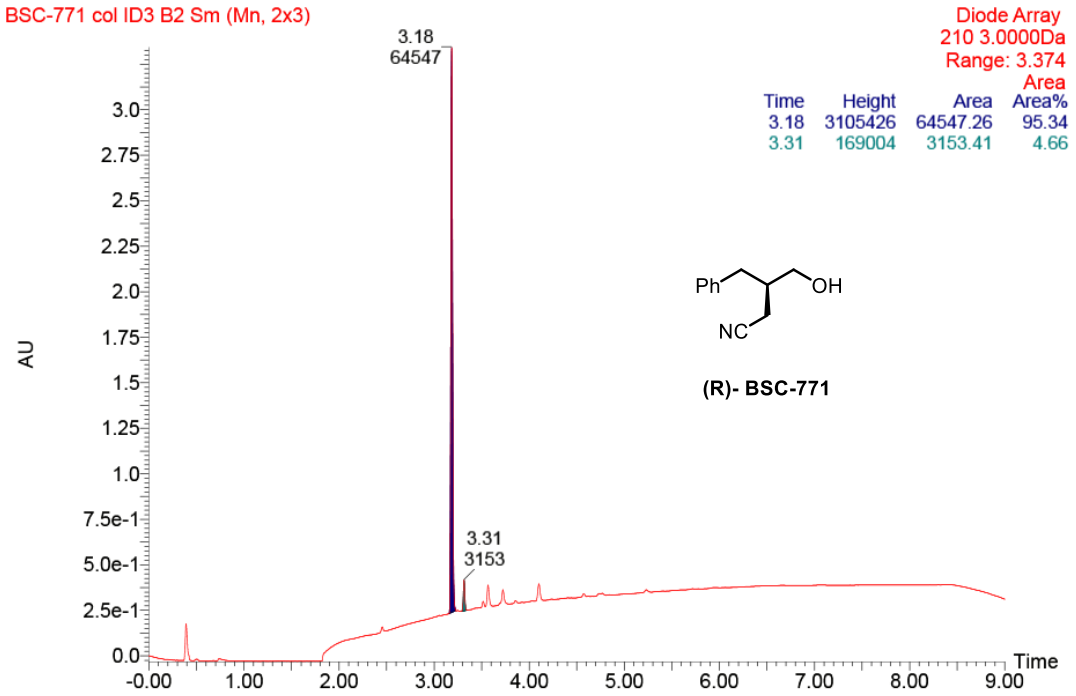

```
=====================================================================
                        Area Percent Report
=====================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Sample Amount:                :      1.00000  [ng/ul]   (not used in calc.)
Use Multiplier & Dilution Factor with ISTDs


Signal 1: DAD1 A, Sig=254,4 Ref=360,100

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1  13.472 BB    0.2350   37.14061    2.34223  10.1276
   2  15.985 BB    0.4029  329.58450   12.36585  89.8724
```


25-Nov-2016 12:38:01
BSC-762 ID3 B2 Sm (Mn, 2x3)
Diode Array
210 3.0000Da
Range: 5.738
Area
Time Height Area Area%
3.29 1954029 38302.93 49.89
3.41 1948551 38466.50 50.11
AU
3.41
38467
Ph
OH
NC
rac- BSC-771


BSC-771 col ID3 B2 Sm (Mn, 2x3)
Diode Array
210 3.0000Da
Range: 3.374
Area
Time Height Area Area%
3.18 3105426 64547.26 95.34
3.31 169004 3153.41 4.66
AU
3.18
64547
3.31
3153
Time
Ph
OH
NC
(R)- BSC-771