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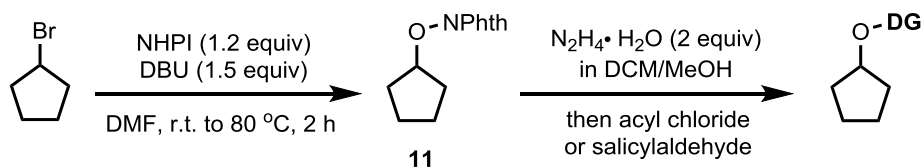
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General Information

Solvents were obtained from Sigma-Aldrich, Alfa-Aesar and Acros and used directly without further purification. Analytical thin layer chromatography was performed on 0.25 mm silica gel 60-F254. Visualization was carried out with UV light (254 nm) and Vogel's permanganate. ^1H NMR spectra were recorded on Bruker AMX-400 (400 MHz) or Bruker DRX-600 (600 MHz) spectrometers. Data for ^1H NMR spectra are reported as follows: chemical shift, multiplicity (br = broad, s = singlet, bs = broad singlet, d = doublet, t = triplet, q = quartet, m = multiplet), coupling constants, and integration. Chemical shifts are reported in parts per million (ppm) referenced to the appropriate solvent signals, for example 7.26 ppm for chloroform-*d*. Coupling constants, J , are reported in Hertz (Hz). ^{13}C NMR spectra were recorded on a Bruker DRX-600 spectrometer (151 MHz). Chemical shifts are reported in parts per million (ppm) referenced to the appropriate solvent signals, for example 77.00 ppm for chloroform-*d*. High-resolution mass spectra (HRMS) were recorded on an Agilent Mass spectrometer using ESI-TOF (electrospray ionization-time of flight). Optical rotations were recorded on a Rudolph Research Analytical Autopol III Automatic Polarimeter.

Experimental Procedures

Preparation of DG2- to DG10-tethered cyclopentanol



Synthesis of intermediate **11**:

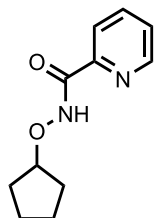
To a 50-mL round-bottom flask were added cyclopentyl bromide (3.0 g, 20 mmol) and *N*-hydroxy phthalimide (4.0 g, 24 mmol), the mixture was dissolved with DMF (20 mL). To the resulting solution was added 1,8-Diazabicyclo(5.4.0)undec-7-ene (DBU, 4.5 mL, 30 mmol) slowly, after which the resulting mixture was stirred at 80 °C for 2 hours. After cooling to room temperature, the reaction solution was poured into a HCl ice-water (10 mL conc. HCl in 200 g ice, pH ~1) while vigorously stirring, during which a pale yellow solid precipitated. The solid was collected by filtration and washed with sat. aq. NaHCO₃ and then with water, dried under air to give product **11** (4.1 g, yield: 87%).

General procedure for installation of DG2 and DG3:

To a 50-mL round-bottom flask were added **11** (231 mg, 1.0 mmol), CH₂Cl₂ (20 mL) and MeOH (2.0 mL); then, hydrazine monohydrate (0.1 mL, 2.0 mmol) was added. The resulting mixture was stirred at room temperature for 1 h, during which a significant amount of solid precipitated. Anhydrous sodium sulfate (~5 g) was then added, the mixture was filtered through a Büchner funnel, and the filtrate was used directly for the next step.

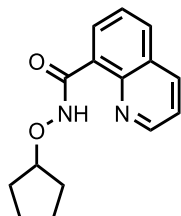
In another 25 mL-round-bottom flask was placed pyridine-2-carboxylic acid (250 mg, 2.0 mmol) or quinoline-8-carboxylic acid (350 mg, 2.0 mmol), dissolved with CH₂Cl₂ (10 mL), then DMF (2 drops) was added. To this solution was added oxalyl chloride (0.35 mL, 4.0 mmol) dropwise. After addition, the mixture was stirred for 2 hours. The solvent was evaporated under reduced pressure and the residue was dissolved with CH₂Cl₂ (5 mL). This solution was added to the solution prepared in the

first step, followed by triethyl amine (1.4 mL). The resulting mixture was stirred at room temperature for 2 hours. The solution was washed with water and the organic phase was dried with anhydrous sodium sulfate. The product was purified by flash column chromatography.



N-(cyclopentyloxy)picolinamide (DG2-CP)

The product is obtained as a white solid, 62% yield. ^1H NMR (600 MHz, Chloroform-*d*) δ 10.26 (s, 1H), 8.51 (s, 1H), 8.18 (d, $J = 7.8$ Hz, 1H), 7.86 (t, $J = 8.4$ Hz, 1H), 7.45 (d, $J = 4.7$ Hz, 1H), 4.67 (s, 1H), 1.96 (s, 2H), 1.89 – 1.68 (m, 4H), 1.59 (s, 2H); ^{13}C NMR (151 MHz, Chloroform-*d*) δ 161.39, 148.83, 147.67, 136.98, 126.09, 121.91, 87.78, 30.77, 23.23; HRMS (ESI-TOF) m/z Calcd. for $\text{C}_{11}\text{H}_{15}\text{N}_2\text{O}_2^+$ ($\text{M}+\text{H}$) $^+$: 207.1128; found: 207.1131.

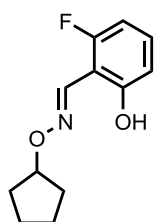


N-(cyclopentyloxy)quinoline-8-carboxamide (DG3-CP)

The product is obtained as a white solid, 54% yield. ^1H NMR (600 MHz, Chloroform-*d*) δ 9.00 – 8.88 (m, 1H), 8.88 – 8.78 (m, 1H), 8.36 – 8.21 (m, 1H), 8.05 – 7.90 (m, 1H), 7.75 – 7.60 (m, 1H), 7.51 (ddd, $J = 16.3, 8.4, 4.0$ Hz, 1H), 4.78 (ddt, $J = 8.6, 5.7, 2.8$ Hz, 1H), 2.19 – 1.99 (m, 2H), 1.98 – 1.73 (m, 4H), 1.62 (dtd, $J = 17.1, 9.6, 8.8, 5.3$ Hz, 2H); ^{13}C NMR (151 MHz, Chloroform-*d*) δ 164.12, 149.44, 144.74, 137.88, 133.70, 131.99, 128.42, 126.59, 121.06, 87.49, 31.39, 23.84; HRMS (ESI-TOF) m/z Calcd. for $\text{C}_{15}\text{H}_{17}\text{N}_2\text{O}_2^+$ ($\text{M}+\text{H}$) $^+$: 257.1285; found: 257.1289.

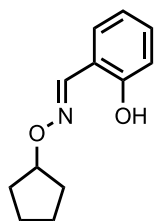
General procedure for installation of DG4–DG8 and DG10:

To a 50 mL-round-bottom flask were added **11** (346 mg, 1.5 mmol), CH₂Cl₂ (20 mL) and MeOH (2.0 mL); then hydrazine monohydrate (0.1 mL, 2.0 mmol) was added. The resulting mixture was stirred at room temperature for 1 h, during which a significant amount of solid precipitated. Anhydrous sodium sulfate (~5 g) was then added, and the resulting mixture was filtered. To the filtrate were added HOAc (0.15 mL) and salicylaldehyde (1.0 mmol), and the mixture was stirred at room temperature for 1 h. The volatiles were evaporated under reduced pressure and the residue was purified by flash column chromatography to give the desired product.



(E)-2-fluoro-6-hydroxybenzaldehyde O-cyclopentyl oxime (DG4-CP)

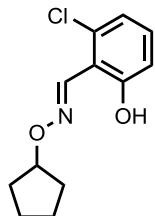
The product is obtained as a colorless oil, 88% yield. ¹H NMR (600 MHz, Chloroform-*d*) δ 10.35 (s, 1H), 8.45 (s, 1H), 7.27 – 7.18 (m, 1H), 6.82 – 6.75 (m, 1H), 6.61 (ddd, *J* = 10.2, 8.2, 1.0 Hz, 1H), 4.81 (dt, *J* = 5.7, 2.9 Hz, 1H), 1.89 (m, 4H), 1.83 – 1.73 (m, 2H), 1.64 (m, 2H); ¹³C NMR (151 MHz, Chloroform-*d*) δ 161.94, 160.28, 158.66, 158.63, 144.77, 144.72, 131.42, 131.35, 112.41, 112.39, 106.14, 106.05, 105.80, 105.66, 86.38, 31.78, 23.70; HRMS (ESI-TOF) *m/z* Calcd. for C₁₂H₁₅FNO₂⁺ (M+H)⁺: 224.1081; found: 224.1087.



(E)-2-hydroxybenzaldehyde O-cyclopentyl oxime (DG5-CP)

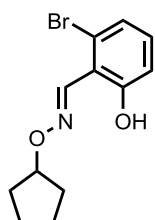
The product is obtained as a colorless oil, 91% yield. ¹H NMR (600 MHz, Chloroform-*d*) δ 10.11 (s, 1H), 8.16 (s, 1H), 7.29 (ddd, *J* = 8.5, 7.3, 1.7 Hz, 1H), 7.17 (dd, *J* = 7.7, 1.7 Hz, 1H), 7.02 (dd, *J* = 8.2, 1.0 Hz, 1H), 6.92 (td, *J* = 7.5, 1.1 Hz, 1H), 4.81 (tt, *J* = 5.8, 2.9 Hz, 1H), 1.90 (m, 4H), 1.83 – 1.72 (m, 2H), 1.64 (m, 2H); ¹³C

NMR (151 MHz, Chloroform-*d*) δ 157.40, 151.05, 130.85, 130.48, 119.44, 116.64, 116.59, 86.05, 31.76, 23.71; HRMS (ESI-TOF) m/z Calcd. for $C_{12}H_{16}NO_2^+$ (M+H) $^+$: 206.1176; found: 206.1181.



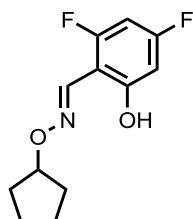
(E)-2-chloro-6-hydroxybenzaldehyde O-cyclopentyl oxime (DG6-CP)

The product is obtained as a colorless oil, 84% yield. 1H NMR (600 MHz, Chloroform-*d*) δ 10.71 (s, 1H), 8.70 (s, 1H), 7.17 (t, $J = 8.1$ Hz, 1H), 6.98 – 6.85 (m, 2H), 4.82 (dt, $J = 5.8, 2.9$ Hz, 1H), 2.01 – 1.83 (m, 4H), 1.78 (d, $J = 4.6$ Hz, 2H), 1.73 – 1.58 (m, 2H); ^{13}C NMR (151 MHz, Chloroform-*d*) δ 158.54, 148.10, 133.66, 130.70, 120.14, 115.23, 113.97, 86.07, 31.39, 23.31; HRMS (ESI-TOF) m/z Calcd. for $C_{12}H_{15}ClNO_2^+$ (M+H) $^+$: 240.0786; found: 240.0792.



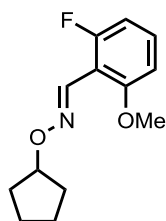
(E)-2-bromo-6-hydroxybenzaldehyde O-cyclopentyl oxime (DG7-CP)

The product is obtained as a colorless oil, 78% yield. 1H NMR (600 MHz, Chloroform-*d*) δ 10.78 (s, 1H), 8.69 (s, 1H), 7.17 – 7.05 (m, 2H), 6.95 (dd, $J = 7.9, 1.3$ Hz, 1H), 4.82 (tt, $J = 5.8, 2.9$ Hz, 1H), 2.01 – 1.85 (m, 4H), 1.85 – 1.73 (m, 2H), 1.70 – 1.59 (m, 2H); ^{13}C NMR (151 MHz, Chloroform-*d*) δ 158.51, 150.64, 131.00, 123.73, 123.58, 115.97, 115.24, 86.11, 31.40, 23.33; HRMS (ESI-TOF) m/z Calcd. for $C_{12}H_{15}BrNO_2^+$ (M+H) $^+$: 284.0281; found: 284.0283.



(*E*)-2,4-difluoro-6-hydroxybenzaldehyde *O*-cyclopentyl oxime (DG8-CP)

The product is obtained as a colorless oil, 86% yield. ¹H NMR (600 MHz, Chloroform-*d*) δ 10.66 (s, 1H), 8.36 (s, 1H), 6.56 – 6.48 (m, 1H), 6.44 – 6.36 (m, 1H), 4.79 (dt, *J* = 5.6, 2.6 Hz, 1H), 1.88 (m, 4H), 1.76 (td, *J* = 7.6, 7.1, 4.0 Hz, 2H), 1.69 – 1.59 (m, 2H); ¹³C NMR (151 MHz, Chloroform-*d*) δ 164.44, 164.33, 162.78, 162.67, 161.83, 161.72, 160.16, 160.05, 159.46, 159.42, 159.36, 159.32, 143.72, 143.67, 102.48, 102.42, 102.39, 99.83, 99.81, 99.67, 99.64, 94.98, 94.81, 94.63, 86.02, 31.34, 23.26; HRMS (ESI-TOF) *m/z* Calcd. for C₁₂H₁₄F₂NO₂⁺ (*M*+*H*)⁺: 242.0987; found: 242.0992.

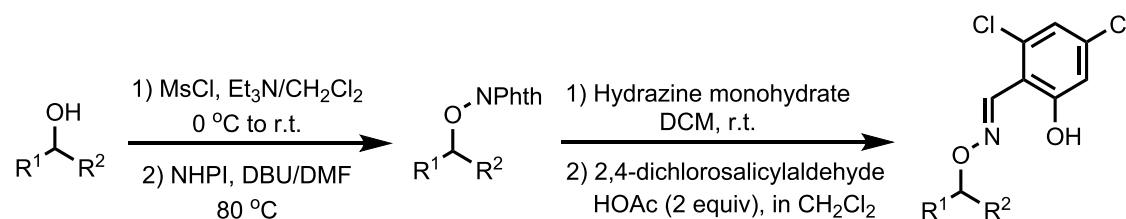


(*E*)-2-fluoro-6-methoxybenzaldehyde *O*-cyclopentyl oxime (DG10-CP)

The product is obtained as a colorless oil, 85% yield. ¹H NMR (600 MHz, Chloroform-*d*) δ 8.31 (s, 1H), 7.26 (td, *J* = 8.4, 6.3 Hz, 1H), 6.78 – 6.72 (m, 1H), 6.70 (d, *J* = 8.4 Hz, 1H), 4.86 (m, 1H), 3.87 (s, 3H), 1.95 – 1.83 (m, 4H), 1.80 – 1.71 (m, 2H), 1.66 – 1.56 (m, 2H); ¹³C NMR (151 MHz, Chloroform-*d*) δ 161.42, 159.73, 158.50, 158.45, 141.03, 141.01, 130.05, 129.97, 109.49, 109.40, 108.29, 108.14, 106.05, 106.03, 84.91, 55.67, 31.64, 23.37; HRMS (ESI-TOF) *m/z* Calcd. for C₁₃H₁₇FNO₂⁺ (*M*+*H*)⁺: 238.1238; found: 238.1239.

Preparation of DG9-tethered alcohol substrates

General procedure A:

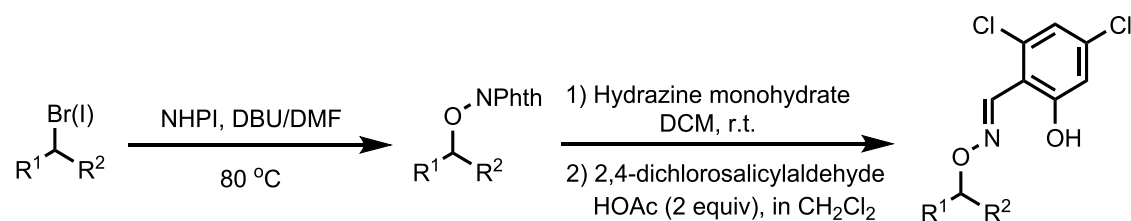


Alcohol (5.0 mmol) and Et₃N (10 mmol) were dissolved in anhydrous

dichloromethane (30 mL), and cooled with an ice-bath. To the pre-cooled solution was added MsCl (7.5 mmol) dropwise and the resulting mixture was stirred at room temperature for 1 h before poured into 1 M HCl solution (50 mL). The organic phase was collected, and the aqueous phase was extracted with CH₂Cl₂ (2×30 mL). The CH₂Cl₂ solution was dried with anhydrous sodium sulfate and concentrated under reduced pressure. The residue was dissolved in DMF (10 mL), then *N*-hydroxyphthalimide (5.0 mmol) and DBU (10 mmol) were added. The resulting dark brown solution was stirred at 80 °C for 4 h before being poured into aq. HCl (1 M, 80 mL). For solid products, the precipitate was collected and washed with sat. aq. NaHCO₃, dried under air. For the oil product the aqueous solution was extracted with 50 mL CH₂Cl₂ (×2). The combined CH₂Cl₂ solution was washed with brine and dried with anhydrous sodium sulfate. The solvent was evaporated under reduced pressure and the residue was used in the next step without further purification.

The crude intermediate was dissolved in CH₂Cl₂ (40 mL), then hydrazine monohydrate (10 mmol) and MeOH (5 mL) were added sequentially. The reaction was stirred at room temperature for 1 h. The white solid formed during the reaction was filtered, and to the filtrate were added AcOH (20 mmol) and 2,4-dichlorosalicylaldehyde (3.0 mmol). The resulting mixture was stirred at room temperature for 1 h, then washed with sat. aq. NaHCO₃ and extracted with 40 mL CH₂Cl₂, the organic phase was dried with anhydrous Na₂SO₄, evaporated and purified by flash column chromatography to give the desired product.

General procedure B:

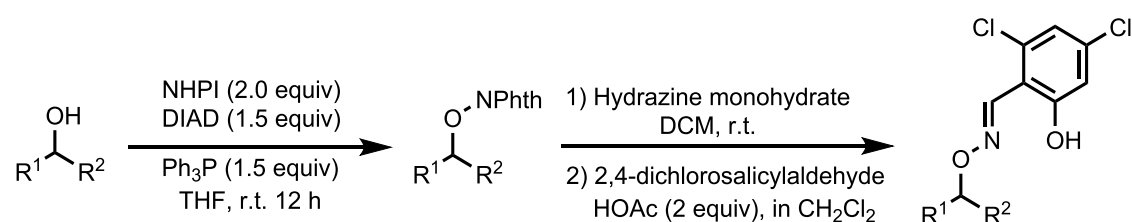


Alkyl bromide or alkyl iodide (7.0 mmol) was dissolved in DMF (10 mL), then *N*-hydroxyphthalimide (NHPI, 5.0 mmol) and 1,8-Diazabicyclo(5.4.0)undec-7-ene

(DBU, 10.0 mmol) were added. The resulting dark brown solution was stirred at 80 °C until the deep color disappeared (typically 1 h). The reaction mixture was cooled to room temperature and poured into aq. HCL (1 M, 80 mL). For solid products, the precipitate was collected and washed with sat. aq. NaHCO₃ and water, dried under air. For oil products, the aqueous solution was extracted with CH₂Cl₂ (50 mL × 2); organic layers were combined, washed with brine (× 3) and dried with anhydrous sodium sulfate. The solvent was evaporated under reduced pressure and the residue was used in the next step without further purification.

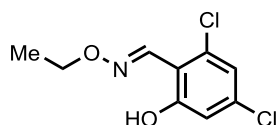
The resulting intermediate was dissolved in CH₂Cl₂ (40 mL), then hydrazine monohydrate (10 mmol) and MeOH (5 mL) were added sequentially. The reaction was stirred at room temperature for 1 h. The white solid formed during the reaction was filtered, and to the filtrate were added AcOH (20 mmol) and 2,4-dichlorosalicylaldehyde (3.0 mmol). The resulting mixture was stirred at room temperature for 1 h, then washed with sat. aq. NaHCO₃ and extracted with 40 mL CH₂Cl₂. The organic phase was dried with anhydrous Na₂SO₄, concentrated under reduced pressure, and purified by flash column chromatography to give the desired product.

General procedure C:



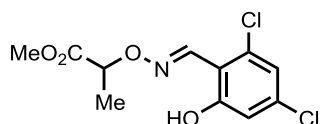
Alcohol (5.0 mmol), NHPI (10.0 mmol) and Ph₃P (7.5 mmol) were dissolved in anhydrous THF (30 mL), and cooled with an ice-bath. To the pre-cooled solution was added DIAD (7.5 mmol) dropwise and the resulting mixture was stirred at room temperature for 12 h. The solvent was evaporated, and the residue was purified by flash column chromatography.

The intermediate was dissolved in CH₂Cl₂ (40 mL), then hydrazine monohydrate (10 mmol) and MeOH (5 mL) were added sequentially. The reaction was stirred at room temperature for 1 h. The white solid formed during the reaction was filtered, and to the filtrate were added AcOH (20 mmol) and 2,4-dichlorosalicylaldehyde (3.0 mmol). The resulting mixture was stirred at room temperature for 1 h, then washed with sat. aq. NaHCO₃ and extracted with CH₂Cl₂ (40 mL). The organic phase was dried with anhydrous Na₂SO₄, evaporated and purified by flash column chromatography to give the desired product.



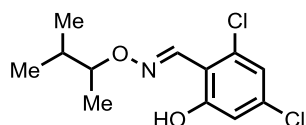
(E)-2,4-dichloro-6-hydroxybenzaldehyde O-ethyl oxime (1a)

Following General procedure B, the product is obtained as a colorless oil, 78% yield. ¹H NMR (600 MHz, Chloroform-*d*) δ 10.75 (s, 1H), 8.64 (s, 1H), 6.96 (d, *J* = 2.0 Hz, 1H), 6.91 (dd, *J* = 2.0, 0.6 Hz, 1H), 4.26 (q, *J* = 7.1 Hz, 2H), 1.35 (t, *J* = 7.1 Hz, 3H); ¹³C NMR (151 MHz, Chloroform-*d*) δ 158.77, 147.59, 136.06, 134.20, 120.41, 115.70, 112.62, 70.42, 13.79; HRMS (ESI-TOF) *m/z* Calcd. for C₉H₁₀Cl₂NO₂⁺ (M+H)⁺: 234.0089; found: 234.0085.



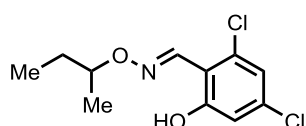
Methyl (E)-2-(((2,4-dichloro-6-hydroxybenzylidene)amino)oxy)propanoate (1b)

Following General procedure A, the product is obtained as a colorless oil, 68% yield. ¹H NMR (600 MHz, Chloroform-*d*) δ 10.23 (s, 1H), 8.73 (s, 1H), 6.97 (d, *J* = 1.9 Hz, 1H), 6.90 (dd, *J* = 2.1, 0.6 Hz, 1H), 4.82 (q, *J* = 7.0 Hz, 1H), 3.79 (s, 3H), 1.57 (d, *J* = 7.0 Hz, 3H); ¹³C NMR (151 MHz, Chloroform-*d*) δ 171.40, 158.74, 149.17, 136.73, 134.67, 120.60, 115.81, 112.22, 77.91, 51.88, 16.27; HRMS (ESI-TOF) *m/z* Calcd. for C₁₁H₁₂Cl₂NO₄⁺ (M+H)⁺: 292.0143; found: 292.0145.



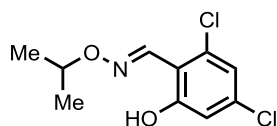
(E)-2,4-dichloro-6-hydroxybenzaldehyde O-(3-methylbutan-2-yl) oxime (1c)

Following General procedure A, the product is obtained as a colorless oil, 54% yield. ^1H NMR (600 MHz, Chloroform-*d*) δ 10.87 (s, 1H), 8.62 (s, 1H), 6.95 (d, $J = 2.0$ Hz, 1H), 6.90 (dd, $J = 2.0, 0.6$ Hz, 1H), 4.11 (m, 1H), 1.96 (pd, $J = 6.8, 5.8$ Hz, 1H), 1.26 (d, $J = 6.5$ Hz, 3H), 0.96 (dd, $J = 19.4, 6.8$ Hz, 6H); ^{13}C NMR (151 MHz, Chloroform-*d*) δ 159.19, 147.44, 136.25, 134.48, 120.78, 116.07, 113.33, 85.61, 32.12, 18.47, 17.46, 15.78; HRMS (ESI-TOF) m/z Calcd. for $\text{C}_{12}\text{H}_{16}\text{Cl}_2\text{NO}_2^+$ (M+H) $^+$: 276.0558; found: 276.0562.



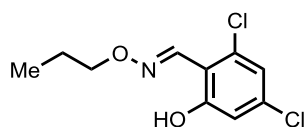
(E)-2,4-dichloro-6-hydroxybenzaldehyde O-(sec-butyl) oxime (1d)

Following General procedure B, the product is obtained as a colorless oil, 82% yield. ^1H NMR (600 MHz, Chloroform-*d*) δ 10.85 (s, 1H), 8.62 (s, 1H), 6.94 (d, $J = 2.1$ Hz, 1H), 6.90 (d, $J = 2.0$ Hz, 1H), 4.25 (h, $J = 6.3$ Hz, 1H), 1.79 – 1.70 (m, 1H), 1.65 – 1.57 (m, 1H), 1.31 (d, $J = 6.3$ Hz, 3H), 0.97 (t, $J = 7.5$ Hz, 3H); ^{13}C NMR (151 MHz, Chloroform-*d*) δ 159.19, 147.57, 136.27, 134.50, 120.76, 116.07, 113.29, 82.13, 28.19, 18.92, 9.57; HRMS (ESI-TOF) m/z Calcd. for $\text{C}_{11}\text{H}_{14}\text{Cl}_2\text{NO}_2^+$ (M+H) $^+$: 262.0396; found: 262.0391.



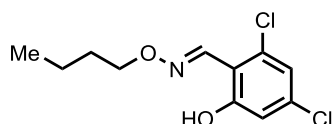
(E)-2,4-dichloro-6-hydroxybenzaldehyde O-isopropyl oxime (1e)

Following General procedure B, the product is obtained as a colorless oil, 84% yield. ^1H NMR (600 MHz, Chloroform-*d*) δ 10.84 (s, 1H), 8.62 (s, 1H), 6.95 (d, $J = 2.0$ Hz, 1H), 6.91 (dd, $J = 2.1, 0.6$ Hz, 1H), 4.47 (hept, $J = 6.3$ Hz, 1H), 1.33 (d, $J = 6.3$ Hz, 6H); ^{13}C NMR (151 MHz, Chloroform-*d*) δ 159.22, 147.69, 136.35, 134.58, 120.81, 116.10, 113.27, 77.13, 21.31; HRMS (ESI-TOF) m/z Calcd. for $\text{C}_{10}\text{H}_{12}\text{Cl}_2\text{NO}_2^+$ (M+H) $^+$: 248.0245; found: 248.0246.



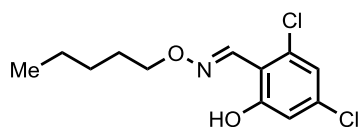
(E)-2,4-dichloro-6-hydroxybenzaldehyde O-propyl oxime (1f)

Following General procedure B, the product is obtained as a colorless oil, 92% yield. ^1H NMR (600 MHz, Chloroform-*d*) δ 10.72 (s, 1H), 8.61 (s, 1H), 6.92 (d, $J = 2.0$ Hz, 1H), 6.88 (dd, $J = 2.0, 0.6$ Hz, 1H), 4.15 (t, $J = 6.7$ Hz, 2H), 1.75 (h, $J = 7.4$ Hz, 2H), 1.00 (t, $J = 7.4$ Hz, 3H); ^{13}C NMR (151 MHz, Chloroform-*d*) δ 159.16, 147.82, 136.39, 134.53, 120.74, 116.09, 113.01, 76.82, 22.07, 10.21; HRMS (ESI-TOF) m/z Calcd. for $\text{C}_{10}\text{H}_{12}\text{Cl}_2\text{NO}_2^+$ ($\text{M}+\text{H}$) $^+$: 248.0245; found: 248.0240.



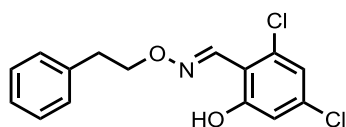
(E)-2,4-dichloro-6-hydroxybenzaldehyde O-butyl oxime (1g)

Following General procedure B, the product is obtained as a colorless oil, 85% yield. ^1H NMR (600 MHz, Chloroform-*d*) δ 10.76 (s, 1H), 8.63 (s, 1H), 6.96 (d, $J = 2.0$ Hz, 1H), 6.91 (dd, $J = 2.0, 0.6$ Hz, 1H), 4.20 (t, $J = 6.7$ Hz, 2H), 1.71 (ddt, $J = 9.0, 7.8, 6.6$ Hz, 2H), 1.48 – 1.41 (m, 2H), 0.97 (t, $J = 7.4$ Hz, 3H); ^{13}C NMR (151 MHz, Chloroform-*d*) δ 159.22, 147.90, 136.47, 134.62, 120.85, 116.15, 113.10, 75.16, 30.81, 19.01, 13.81; HRMS (ESI-TOF) m/z Calcd. for $\text{C}_{11}\text{H}_{14}\text{Cl}_2\text{NO}_2^+$ ($\text{M}+\text{H}$) $^+$: 262.0402; found: 262.0401.



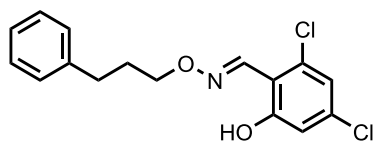
(E)-2,4-dichloro-6-hydroxybenzaldehyde O-pentyl oxime (1h)

Following General procedure B, the product is obtained as a colorless oil, 75% yield. ^1H NMR (600 MHz, Chloroform-*d*) δ 10.75 (s, 1H), 8.62 (s, 1H), 6.95 (d, $J = 2.0$ Hz, 1H), 6.90 (dd, $J = 2.0, 0.5$ Hz, 1H), 4.19 (t, $J = 6.7$ Hz, 2H), 1.78 – 1.68 (m, 2H), 1.43 – 1.33 (m, 4H), 0.97 – 0.89 (m, 3H); ^{13}C NMR (151 MHz, Chloroform-*d*) δ 159.20, 147.85, 136.44, 134.59, 120.81, 116.13, 113.08, 75.42, 28.45, 27.92, 22.42, 13.95; HRMS (ESI-TOF) m/z Calcd. for $\text{C}_{12}\text{H}_{16}\text{Cl}_2\text{NO}_2^+$ ($\text{M}+\text{H}$) $^+$: 276.0558; found: 276.0553.



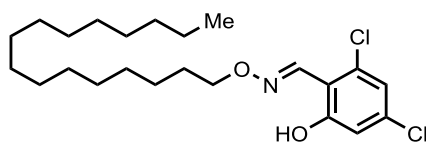
(E)-2,4-dichloro-6-hydroxybenzaldehyde O-phenethyl oxime (1i)

Following General procedure A, the product is obtained as a colorless oil, 66% yield. ^1H NMR (600 MHz, Chloroform-*d*) δ 10.67 (s, 1H), 8.68 (s, 1H), 7.38 – 7.34 (m, 2H), 7.31 – 7.26 (m, 3H), 6.99 (d, $J = 2.0$ Hz, 1H), 6.95 (dd, $J = 2.0, 0.6$ Hz, 1H), 4.46 (t, $J = 7.0$ Hz, 2H), 3.09 (t, $J = 7.0$ Hz, 2H); ^{13}C NMR (151 MHz, Chloroform-*d*) δ 159.20, 148.40, 137.62, 136.64, 134.70, 128.87, 128.56, 126.58, 120.88, 116.17, 112.93, 75.71, 35.31; HRMS (ESI-TOF) m/z Calcd. for $\text{C}_{15}\text{H}_{14}\text{Cl}_2\text{NO}_2^+$ ($\text{M}+\text{H}$) $^+$: 310.0402; found: 310.0401.



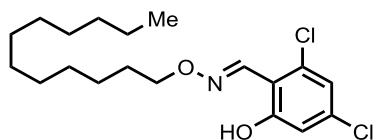
(E)-2,4-dichloro-6-hydroxybenzaldehyde O-(3-phenylpropyl) oxime (1j)

Following General procedure A, the product is obtained as a colorless oil, 74% yield. ^1H NMR (600 MHz, Chloroform-*d*) δ 10.69 (s, 1H), 8.65 (d, $J = 0.6$ Hz, 1H), 7.33 – 7.27 (m, 2H), 7.24 – 7.16 (m, 3H), 6.97 (d, $J = 2.1$ Hz, 1H), 6.92 (dd, $J = 2.0, 0.6$ Hz, 1H), 4.21 (t, $J = 6.5$ Hz, 2H), 2.75 (dd, $J = 8.4, 6.9$ Hz, 2H), 2.11 – 2.03 (m, 2H); ^{13}C NMR (151 MHz, Chloroform-*d*) δ 159.22, 148.17, 141.17, 136.58, 134.68, 128.46, 128.43, 126.04, 120.89, 116.18, 113.03, 74.42, 31.93, 30.30; HRMS (ESI-TOF) m/z Calcd. for $\text{C}_{16}\text{H}_{16}\text{Cl}_2\text{NO}_2^+$ ($\text{M}+\text{H}$) $^+$: 324.0558; found: 324.0555.



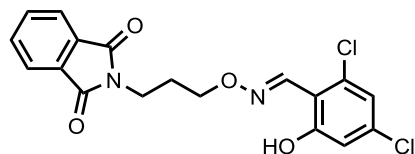
(E)-2,4-dichloro-6-hydroxybenzaldehyde O-hexadecyl oxime (1k)

Following General procedure A, the product is obtained as a white solid, 72% yield. ^1H NMR (600 MHz, Chloroform-*d*) δ 10.76 (s, 1H), 8.63 (s, 1H), 6.96 (d, $J = 2.0$ Hz, 1H), 6.91 (d, $J = 2.0$ Hz, 1H), 4.19 (t, $J = 6.7$ Hz, 2H), 1.72 (dt, $J = 14.7, 6.8$ Hz, 2H), 1.44 – 1.23 (m, 26H), 0.88 (t, $J = 7.0$ Hz, 3H); ^{13}C NMR (151 MHz, Chloroform-*d*) δ 159.22, 147.89, 136.46, 134.62, 120.84, 116.15, 113.10, 75.47, 31.92, 29.70, 29.69, 29.69, 29.67, 29.66, 29.64, 29.56, 29.51, 29.36, 29.35, 28.76, 25.77, 22.69, 14.12; HRMS (ESI-TOF) m/z Calcd. for $\text{C}_{23}\text{H}_{38}\text{Cl}_2\text{NO}_2^+$ ($\text{M}+\text{H}$) $^+$: 430.2280; found: 430.2274.



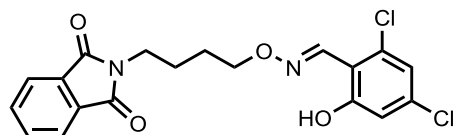
(E)-2,4-dichloro-6-hydroxybenzaldehyde O-dodecyl oxime (1l)

Following General procedure A, the product is obtained as a white solid, 74% yield. ^1H NMR (600 MHz, Chloroform-*d*) δ 10.76 (s, 1H), 8.63 (s, 1H), 6.96 (d, $J = 2.0$ Hz, 1H), 6.91 (d, $J = 2.0$ Hz, 1H), 4.19 (t, $J = 6.7$ Hz, 2H), 1.72 (dt, $J = 14.8, 6.8$ Hz, 2H), 1.42 – 1.23 (m, 18H), 0.88 (t, $J = 7.0$ Hz, 3H); ^{13}C NMR (151 MHz, Chloroform-*d*) δ 159.22, 147.89, 136.46, 134.61, 120.84, 116.15, 113.10, 75.47, 31.91, 29.64, 29.63, 29.56, 29.52, 29.35, 28.76, 25.77, 22.69, 14.11; HRMS (ESI-TOF) m/z Calcd. for $\text{C}_{19}\text{H}_{30}\text{Cl}_2\text{NO}_2^+$ ($\text{M}+\text{H}$) $^+$: 374.1654; found: 374.1647.



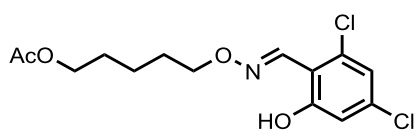
(E)-2,4-dichloro-6-hydroxybenzaldehyde O-(3-(1,3-dioxoisindolin-2-yl)propyl) oxime (1m)

Following General procedure A from N-Boc protected 1, 3-amino alcohol, then remove the Boc in TFA/ CH_2Cl_2 and re-protect the amine with phthalic anhydride. The product is obtained as a pale yellow solid, 42% yield. ^1H NMR (600 MHz, Chloroform-*d*) δ 10.52 (s, 1H), 8.48 (d, $J = 0.6$ Hz, 1H), 7.84 (dd, $J = 5.4, 3.1$ Hz, 2H), 7.77 – 7.67 (m, 2H), 6.93 (d, $J = 2.0$ Hz, 1H), 6.89 (dd, $J = 2.0, 0.6$ Hz, 1H), 4.28 (t, $J = 6.1$ Hz, 2H), 3.87 (t, $J = 6.8$ Hz, 2H), 2.14 (m, 2H); ^{13}C NMR (151 MHz, Chloroform-*d*) δ 168.29, 159.18, 148.50, 136.69, 134.71, 133.99, 132.05, 123.26, 120.86, 116.19, 112.82, 72.90, 35.18, 27.65; HRMS (ESI-TOF) m/z Calcd. for $\text{C}_{18}\text{H}_{15}\text{Cl}_2\text{N}_2\text{O}_4^+$ ($\text{M}+\text{H}$) $^+$: 393.0409; found: 393.0406.



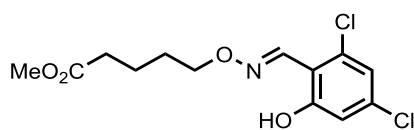
(E)-2,4-dichloro-6-hydroxybenzaldehyde O-(4-(1,3-dioxoisindolin-2-yl)butyl) oxime (1n)

Following General procedure A from N-Boc protected 1, 4-amino alcohol, then remove the Boc in TFA/CH₂Cl₂ and re-protect the amine with phthalic anhydride. The product is obtained as a pale yellow solid, 48% yield. ¹H NMR (600 MHz, Chloroform-*d*) δ 10.61 (s, 1H), 8.60 (s, 1H), 7.83 (dd, *J* = 5.4, 3.0 Hz, 2H), 7.70 (dd, *J* = 5.4, 3.0 Hz, 2H), 6.95 (d, *J* = 2.0 Hz, 1H), 6.90 – 6.86 (m, 1H), 4.23 (t, *J* = 6.2 Hz, 2H), 3.75 (t, *J* = 6.9 Hz, 2H), 1.85 – 1.76 (m, 4H); ¹³C NMR (151 MHz, Chloroform-*d*) δ 168.40, 159.18, 148.26, 136.56, 134.69, 133.92, 132.03, 123.22, 120.85, 116.16, 112.95, 74.48, 37.52, 28.40, 26.04, 24.97; HRMS (ESI-TOF) *m/z* Calcd. for C₁₉H₁₇Cl₂N₂O₄⁺ (M+H)⁺: 407.0565; found: 407.0558.



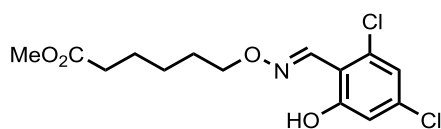
(*E*)-5-(((2,4-dichloro-6-hydroxybenzylidene)amino)oxy)pentyl acetate (1o)

Following General procedure A, the product is obtained as a colorless oil, 62% yield. ¹H NMR (600 MHz, Chloroform-*d*) δ 10.69 (s, 1H), 8.62 (s, 1H), 6.95 (d, *J* = 2.0 Hz, 1H), 6.90 (dd, *J* = 2.1, 0.6 Hz, 1H), 4.20 (t, *J* = 6.6 Hz, 2H), 4.08 (t, *J* = 6.6 Hz, 2H), 2.04 (s, 3H), 1.80 – 1.73 (m, 2H), 1.73 – 1.65 (m, 2H), 1.52 – 1.45 (m, 2H); ¹³C NMR (151 MHz, Chloroform-*d*) δ 171.12, 159.18, 148.08, 136.55, 134.63, 120.85, 116.14, 112.98, 75.02, 64.21, 28.38, 28.33, 22.34, 20.95; HRMS (ESI-TOF) *m/z* Calcd. for C₁₄H₁₈Cl₂NO₄⁺ (M+H)⁺: 334.0613; found: 334.0613.



Methyl (*E*)-5-(((2,4-dichloro-6-hydroxybenzylidene)amino)oxy)pentanoate (1p)

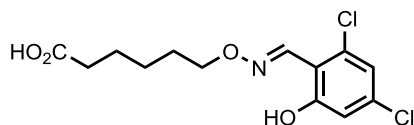
Following General procedure A, the product is obtained as a colorless oil, 72% yield. ¹H NMR (600 MHz, Chloroform-*d*) δ 10.66 (s, 1H), 8.63 (s, 1H), 6.96 (d, *J* = 2.1 Hz, 1H), 6.91 (dd, *J* = 2.1, 0.6 Hz, 1H), 4.26 – 4.16 (m, 2H), 3.68 (s, 3H), 2.44 – 2.33 (m, 2H), 1.84 – 1.69 (m, 4H); ¹³C NMR (151 MHz, Chloroform-*d*) δ 173.68, 159.20, 148.21, 136.60, 134.68, 120.88, 116.17, 112.97, 74.74, 51.57, 33.59, 28.13, 21.32; HRMS (ESI-TOF) *m/z* Calcd. for C₁₃H₁₆Cl₂NO₄⁺ (M+H)⁺: 320.0456; found: 320.0460.



Methyl (*E*)-6-(((2,4-dichloro-6-hydroxybenzylidene)amino)oxy)hexanoate (1q)

Following General procedure A, the product is obtained as a colorless oil, 70% yield.

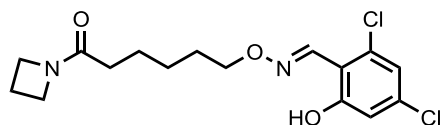
^1H NMR (600 MHz, Chloroform-*d*) δ 10.67 (s, 1H), 8.60 (s, 1H), 6.92 (d, $J = 2.0$ Hz, 1H), 6.88 (dd, $J = 2.0, 0.6$ Hz, 1H), 4.17 (t, $J = 6.6$ Hz, 2H), 3.65 (s, 3H), 2.32 (t, $J = 7.5$ Hz, 2H), 1.76 – 1.64 (m, 4H), 1.47 – 1.39 (m, 2H); ^{13}C NMR (151 MHz, Chloroform-*d*) δ 173.83, 159.14, 147.96, 136.45, 134.56, 120.76, 116.09, 112.94, 74.97, 51.44, 33.81, 28.37, 25.32, 24.57; HRMS (ESI-TOF) m/z Calcd. for $\text{C}_{14}\text{H}_{18}\text{Cl}_2\text{NO}_4^+$ ($\text{M}+\text{H}$) $^+$: 334.0613; found: 334.0607.



(*E*)-6-(((2,4-dichloro-6-hydroxybenzylidene)amino)oxy)hexanoic acid (1r)

Following General procedure A, the product is obtained as a white solid, 61% yield.

^1H NMR (600 MHz, Chloroform-*d*) δ 10.69 (s, 1H), 8.63 (s, 1H), 6.96 (d, $J = 2.0$ Hz, 1H), 6.91 (dd, $J = 2.0, 0.6$ Hz, 1H), 4.20 (t, $J = 6.5$ Hz, 2H), 2.39 (t, $J = 7.4$ Hz, 2H), 1.81 – 1.67 (m, 4H), 1.48 (tdd, $J = 10.6, 8.4, 4.3$ Hz, 2H); ^{13}C NMR (151 MHz, Chloroform-*d*) δ 179.29, 159.20, 148.11, 136.56, 134.66, 120.87, 116.16, 113.01, 74.97, 33.77, 28.40, 25.30, 24.34; HRMS (ESI-TOF) m/z Calcd. for $\text{C}_{13}\text{H}_{16}\text{Cl}_2\text{NO}_4^+$ ($\text{M}+\text{H}$) $^+$: 320.0456; found: 320.0459.

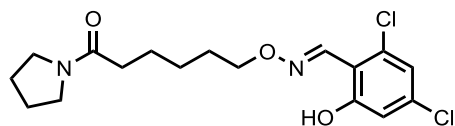


(*E*)-2,4-dichloro-6-hydroxybenzaldehyde O-(6-(azetidin-1-yl)-6-oxohexyl) oxime (1s)

Following General procedure A, the product is obtained as a pale-yellow oil, 45% yield.

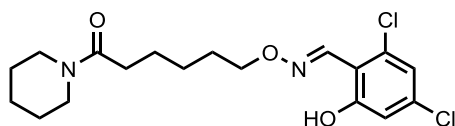
^1H NMR (600 MHz, Chloroform-*d*) δ 10.67 (s, 1H), 8.58 (s, 1H), 6.91 (d, $J = 2.0$ Hz, 1H), 6.89 – 6.84 (m, 1H), 4.16 (t, $J = 6.6$ Hz, 2H), 4.09 (t, $J = 7.5$ Hz, 2H), 3.98 (t, $J = 7.6$ Hz, 2H), 2.22 (m, 2H), 2.08 – 2.01 (m, 2H), 1.71 (dt, $J = 14.7, 6.6$ Hz, 2H), 1.66 – 1.62 (m, 2H), 1.45 – 1.37 (m, 2H); ^{13}C NMR (151 MHz, Chloroform-*d*) δ

172.85, 159.08, 147.85, 136.37, 134.51, 120.72, 116.02, 112.93, 75.02, 50.01, 47.68, 30.80, 28.47, 25.52, 24.42, 14.92; HRMS (ESI-TOF) m/z Calcd. for $C_{16}H_{21}Cl_2N_2O_3^+$ (M+H)⁺: 359.0929; found: 359.0937.



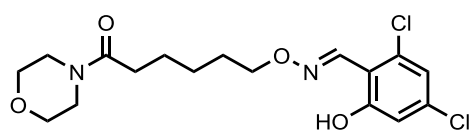
(E)-2,4-dichloro-6-hydroxybenzaldehyde O-(6-oxo-6-(pyrrolidin-1-yl)hexyl) oxime (1t)

Following General procedure A, the product is obtained as a pale-yellow oil, 48% yield. ¹H NMR (600 MHz, Chloroform-*d*) δ 10.69 (s, 1H), 8.59 (s, 1H), 6.92 (d, J = 2.0 Hz, 1H), 6.87 (d, J = 2.0 Hz, 1H), 4.17 (t, J = 6.6 Hz, 2H), 3.44 (t, J = 6.9 Hz, 2H), 3.38 (t, J = 6.8 Hz, 2H), 2.26 (t, J = 7.5 Hz, 2H), 1.92 (m, 2H), 1.82 (m, 2H), 1.71 (ddt, J = 30.3, 15.3, 7.1 Hz, 4H), 1.44 (m, 2H); ¹³C NMR (151 MHz, Chloroform-*d*) δ 171.51, 159.10, 147.85, 136.38, 134.52, 120.73, 116.04, 112.96, 75.09, 46.59, 45.60, 34.44, 28.54, 26.00, 25.61, 24.49, 24.30; HRMS (ESI-TOF) m/z Calcd. for $C_{17}H_{23}Cl_2N_2O_3^+$ (M+H)⁺: 373.1080; found: 373.1084.



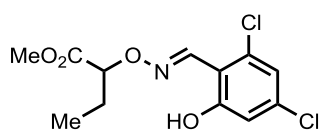
(E)-2,4-dichloro-6-hydroxybenzaldehyde O-(6-oxo-6-(piperidin-1-yl)hexyl) oxime (1u)

Following General procedure A, the product is obtained as a pale-yellow oil, 42% yield. ¹H NMR (600 MHz, Chloroform-*d*) δ 10.65 (s, 1H), 8.55 (s, 1H), 6.88 (d, J = 2.0 Hz, 1H), 6.83 (d, J = 2.0 Hz, 1H), 4.14 (t, J = 6.6 Hz, 2H), 3.49 (dd, J = 5.6, 2.3 Hz, 2H), 3.34 (d, J = 5.5 Hz, 2H), 2.29 (dd, J = 8.3, 7.1 Hz, 2H), 1.64 – 1.57 (m, 6H), 1.52 – 1.46 (m, 6H); ¹³C NMR (151 MHz, Chloroform-*d*) δ 170.97, 158.96, 147.66, 136.21, 134.36, 120.54, 115.88, 112.80, 74.92, 72.82, 46.49, 42.48, 32.89, 28.74, 28.38, 26.33, 25.72, 25.46, 25.35, 25.10, 24.87, 24.31, 21.60; HRMS (ESI-TOF) m/z Calcd. for $C_{18}H_{25}Cl_2N_2O_3^+$ (M+H)⁺: 387.1237; found: 387.1240.



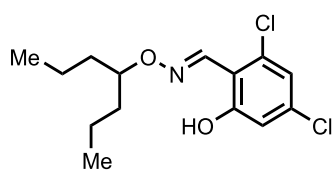
(E)-2,4-dichloro-6-hydroxybenzaldehyde O-(6-morpholino-6-oxohexyl) oxime (1v)

Following General procedure A, the product is obtained as a pale-yellow oil, 51% yield. ^1H NMR (600 MHz, Chloroform-*d*) δ 10.80 – 10.59 (m, 1H), 8.75 – 8.50 (m, 1H), 7.02 – 6.83 (m, 2H), 4.20 (dt, $J = 16.1, 6.8$ Hz, 2H), 3.65 (tt, $J = 15.9, 7.9$ Hz, 8H), 2.34 – 2.31 (m, 2H), 1.88 – 1.84 (m, 2H), 1.69 (dq, $J = 16.1, 7.6$ Hz, 4H); ^{13}C NMR (151 MHz, Chloroform-*d*) δ 171.35, 159.11, 147.92, 136.43, 134.54, 120.81, 116.07, 112.94, 75.03, 66.86, 41.83, 32.77, 28.56, 25.61, 24.77, 21.81; HRMS (ESI-TOF) m/z Calcd. for $\text{C}_{17}\text{H}_{23}\text{Cl}_2\text{N}_2\text{O}_4^+$ ($\text{M}+\text{H}$) $^+$: 389.1029; found: 389.1035.



Methyl (E)-2-(((2,4-dichloro-6-hydroxybenzylidene)amino)oxy)butanoate (1w)

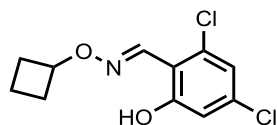
Following General procedure A, the product is obtained as a colorless oil, 68% yield. ^1H NMR (600 MHz, Chloroform-*d*) δ 10.23 (s, 1H), 8.74 (s, 1H), 6.95 (d, $J = 2.0$ Hz, 1H), 6.88 (dd, $J = 2.0, 0.6$ Hz, 1H), 4.66 (dd, $J = 7.4, 5.4$ Hz, 1H), 3.78 (s, 3H), 2.02 – 1.89 (m, 2H), 1.05 (t, $J = 7.5$ Hz, 3H); ^{13}C NMR (151 MHz, Chloroform-*d*) δ 170.93, 158.73, 149.11, 136.66, 134.61, 120.56, 115.78, 112.23, 83.04, 51.72, 24.02, 9.05; HRMS (ESI-TOF) m/z Calcd. for $\text{C}_{12}\text{H}_{14}\text{Cl}_2\text{NO}_4^+$ ($\text{M}+\text{H}$) $^+$: 306.0294; found: 306.0296.



(E)-2,4-dichloro-6-hydroxybenzaldehyde O-heptan-4-yl oxime (1x)

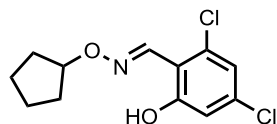
Following General procedure A, the product is obtained as a colorless oil, 65% yield. ^1H NMR (600 MHz, Chloroform-*d*) δ 10.88 (s, 1H), 8.62 (s, 1H), 6.95 (d, $J = 2.0$ Hz, 1H), 6.90 (dd, $J = 2.0, 0.6$ Hz, 1H), 4.19 (tt, $J = 7.4, 5.1$ Hz, 1H), 1.67 (dddd, $J = 14.0, 10.1, 7.4, 5.3$ Hz, 2H), 1.57 (dddd, $J = 14.0, 9.9, 6.0, 5.1$ Hz, 2H), 1.51 – 1.34 (m, 4H), 0.94 (t, $J = 7.4$ Hz, 6H); ^{13}C NMR (151 MHz, Chloroform-*d*) δ 159.19, 147.33,

136.23, 134.46, 120.78, 116.06, 113.39, 84.64, 35.80, 18.58, 14.10; HRMS (ESI-TOF) m/z Calcd. for $C_{14}H_{20}Cl_2NO_2^+$ ($M+H$) $^+$: 304.0866; found: 304.0863.



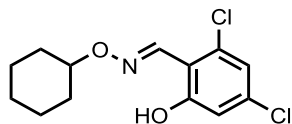
(E)-2,4-dichloro-6-hydroxybenzaldehyde O-cyclobutyl oxime (1y)

Following General procedure B, the product is obtained as a colorless oil, 55% yield. 1H NMR (600 MHz, Chloroform-*d*) δ 10.74 (s, 1H), 8.64 (s, 1H), 6.95 (d, $J = 2.0$ Hz, 1H), 6.90 (dd, $J = 2.0, 0.5$ Hz, 1H), 4.85 – 4.69 (m, 1H), 2.39 – 2.30 (m, 2H), 2.22 – 2.11 (m, 2H), 1.89 – 1.79 (m, 1H), 1.71 – 1.60 (m, 1H); ^{13}C NMR (151 MHz, Chloroform-*d*) δ 159.24, 148.35, 136.54, 134.67, 120.82, 116.14, 113.09, 77.61, 29.38, 12.78; HRMS (ESI-TOF) m/z Calcd. for $C_{11}H_{12}Cl_2NO_2^+$ ($M+H$) $^+$: 260.0240; found: 260.0237.



(E)-2,4-dichloro-6-hydroxybenzaldehyde O-cyclopentyl oxime (1z)

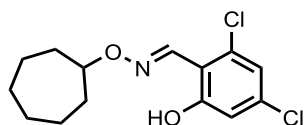
Following General procedure B, the product is obtained as a colorless oil, 85% yield. 1H NMR (600 MHz, Chloroform-*d*) δ 10.87 (s, 1H), 8.59 (s, 1H), 6.94 (d, $J = 2.1$ Hz, 1H), 6.90 (dd, $J = 2.1, 0.6$ Hz, 1H), 4.79 (tt, $J = 5.7, 3.1$ Hz, 1H), 1.86 (tq, $J = 12.0, 6.9, 6.3$ Hz, 4H), 1.74 (qt, $J = 8.7, 4.7$ Hz, 2H), 1.67 – 1.58 (m, 2H); ^{13}C NMR (151 MHz, Chloroform-*d*) δ 159.24, 147.86, 136.33, 134.54, 120.77, 116.10, 113.25, 86.75, 31.82, 23.72; HRMS (ESI-TOF) m/z Calcd. for $C_{12}H_{14}Cl_2NO_2^+$ ($M+H$) $^+$: 274.0396; found: 274.0398.



(E)-2,4-dichloro-6-hydroxybenzaldehyde O-cyclohexyl oxime (1aa)

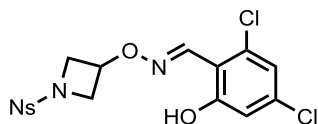
Following General procedure B, the product is obtained as a colorless oil, 74% yield. 1H NMR (600 MHz, Chloroform-*d*) δ 10.87 (s, 1H), 8.63 (s, 1H), 6.94 (d, $J = 2.0$ Hz, 1H), 6.91 – 6.88 (m, 1H), 4.17 (ddd, $J = 13.2, 9.3, 3.9$ Hz, 1H), 2.07 – 1.95 (m, 2H), 1.79 (dq, $J = 13.1, 4.2$ Hz, 2H), 1.58 (dt, $J = 12.5, 3.9$ Hz, 1H), 1.55 – 1.45 (m, 2H),

1.36 (tdd, $J = 13.6, 10.5, 3.3$ Hz, 2H), 1.32 – 1.24 (m, 1H); ^{13}C NMR (151 MHz, Chloroform-*d*) δ 159.19, 147.61, 136.25, 134.50, 120.74, 116.06, 113.28, 82.25, 31.30, 25.47, 23.61; HRMS (ESI-TOF) m/z Calcd. for $\text{C}_{13}\text{H}_{16}\text{Cl}_2\text{NO}_2^+$ ($\text{M}+\text{H}$) $^+$: 288.0553; found: 288.0557.



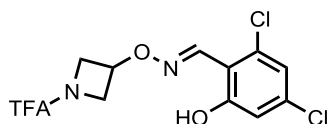
(E)-2,4-dichloro-6-hydroxybenzaldehyde O-cycloheptyl oxime (1ab)

Following General procedure A, the product is obtained as a colorless oil, 54% yield. ^1H NMR (600 MHz, Chloroform-*d*) δ 10.86 (s, 1H), 8.59 (s, 1H), 6.92 (d, $J = 2.1$ Hz, 1H), 6.88 (d, $J = 2.0$ Hz, 1H), 4.35 (tt, $J = 8.3, 4.6$ Hz, 1H), 2.04 (dddd, $J = 13.3, 7.6, 4.5, 2.8$ Hz, 2H), 1.81 – 1.74 (m, 2H), 1.70 (ddq, $J = 11.7, 7.0, 3.0$ Hz, 2H), 1.58 (tq, $J = 8.6, 5.6, 4.5$ Hz, 4H), 1.46 (dtt, $J = 12.3, 6.0, 3.8$ Hz, 2H); ^{13}C NMR (151 MHz, Chloroform-*d*) δ 159.16, 147.46, 136.19, 134.43, 120.68, 116.01, 113.26, 85.16, 33.07, 28.52, 22.76; HRMS (ESI-TOF) m/z Calcd. for $\text{C}_{14}\text{H}_{18}\text{Cl}_2\text{NO}_2^+$ ($\text{M}+\text{H}$) $^+$: 302.0709; found: 302.0711.



(E)-2,4-dichloro-6-hydroxybenzaldehyde O-(1-((2-nitrophenyl)sulfonyl)azetidine-3-yl) oxime (1ad)

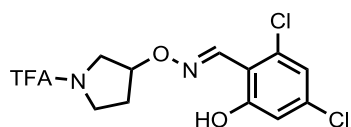
Following General procedure C, first with the Boc protection of azetidine, then changed the Boc to Ns, the product is obtained as pale yellow solid, 34% yield. ^1H NMR (600 MHz, Chloroform-*d*) δ 10.04 (s, 1H), 8.71 (s, 1H), 8.06 (dd, $J = 6.9, 2.7$ Hz, 1H), 7.77 – 7.70 (m, 3H), 7.00 – 6.94 (m, 1H), 6.89 (d, $J = 2.0$ Hz, 1H), 5.04 (tt, $J = 6.5, 4.4$ Hz, 1H), 4.41 (dd, $J = 9.7, 6.6$ Hz, 2H), 4.27 (dd, $J = 9.7, 4.3$ Hz, 2H); ^{13}C NMR (151 MHz, Chloroform-*d*) δ 159.17, 150.90, 148.17, 137.60, 135.17, 133.88, 132.14, 132.11, 130.66, 124.46, 121.19, 116.36, 112.31, 70.26, 57.71; HRMS (ESI-TOF) m/z Calcd. for $\text{C}_{16}\text{H}_{14}\text{Cl}_2\text{N}_3\text{O}_6\text{S}^+$ ($\text{M}+\text{H}$) $^+$: 445.9975; found: 445.9978.



(E)-2,4-dichloro-6-hydroxybenzaldehyde

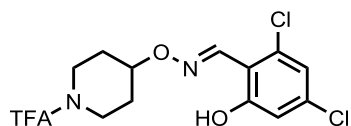
O-(1-(2,2,2-trifluoroacetyl)azetidin-3-yl) oxime (1ae)

Following General procedure C, first with the Boc protection of azetidine, then changed the Boc to TFA, the product is obtained as pale yellow solid, 42% yield. ¹H NMR (600 MHz, Chloroform-*d*) δ 10.00 (s, 1H), 8.78 (s, 1H), 7.05 – 6.98 (m, 1H), 6.97 – 6.89 (m, 1H), 5.17 (ddd, *J* = 10.5, 6.5, 3.9 Hz, 1H), 4.71 (dd, *J* = 10.1, 7.5 Hz, 1H), 4.48 (dd, *J* = 11.7, 5.6 Hz, 2H), 4.30 – 4.21 (m, 1H); ¹³C NMR (151 MHz, Chloroform-*d*) δ 159.20, 156.46 (q, *J* = 37.5 Hz), 151.37, 137.91, 135.31, 121.36, 116.44, 115.92 (q, *J* = 288.4 Hz), 112.19, 71.76, 58.22 (q, *J* = 2.11 Hz), 55.29; HRMS (ESI-TOF) *m/z* Calcd. for C₁₂H₁₀Cl₂F₃N₂O₃⁺ (M+H)⁺: 357.0021; found: 357.0026.



(E)-2,4-dichloro-6-hydroxybenzaldehyde O-(1-(2,2,2-trifluoroacetyl)pyrrolidin-3-yl) oxime (1af)

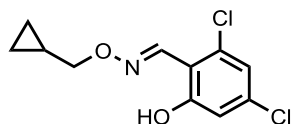
Following General procedure C, first with the Boc protection of pyrrolidine, then changed the Boc to TFA, the product is obtained as pale yellow solid, 48% yield. The NMR corresponding to the major rotamer. ¹H NMR (600 MHz, Chloroform-*d*) δ 10.28 (s, 1H), 8.70 (d, *J* = 4.4 Hz, 1H), 7.01 (dt, *J* = 3.6, 1.6 Hz, 1H), 6.95 (d, *J* = 1.9 Hz, 1H), 5.11 – 4.96 (m, 1H), 4.09 – 3.67 (m, 4H), 2.50 – 2.35 (m, 1H), 2.31 – 2.13 (m, 1H); ¹³C NMR (151 MHz, Chloroform-*d*) δ 159.16, 155.86 (q, *J* = 37.5 Hz), 150.14, 137.39, 135.06, 121.18, 116.31, 116.15 (q, *J* = 287.4 Hz), 112.50, 80.32, 52.30, 45.48, 28.53; HRMS (ESI-TOF) *m/z* Calcd. for C₁₃H₁₂Cl₂F₃N₂O₃⁺ (M+H)⁺: 371.0172; found: 371.0175.



(E)-2,4-dichloro-6-hydroxybenzaldehyde O-(1-(2,2,2-trifluoroacetyl)piperidin-4-yl) oxime (1ag)

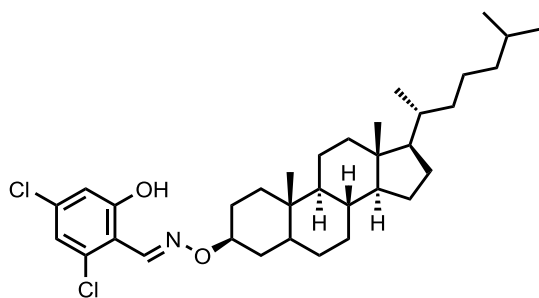
Following General procedure C, first with the Boc protection of pyrrolidine, then changed the Boc to TFA, the product is obtained as pale yellow solid, 44% yield. ¹H

NMR (600 MHz, Chloroform-*d*) δ 10.47 (s, 1H), 8.71 (s, 1H), 6.99 (d, $J = 2.0$ Hz, 1H), 6.93 (dd, $J = 2.0, 0.5$ Hz, 1H), 4.52 (tt, $J = 6.7, 3.5$ Hz, 1H), 3.86 – 3.53 (m, 4H), 2.11 – 2.00 (m, 2H), 2.00 – 1.86 (m, 2H); ^{13}C NMR (151 MHz, Chloroform-*d*) δ 159.18, 155.51 (q, $J = 35.8$ Hz), 149.20, 137.11, 134.90, 121.12, 116.48 (q, $J = 288.4$ Hz), 116.27, 112.76, 77.36, 42.21 (q, $J = 3.8$ Hz), 40.09, 30.53, 29.56; HRMS (ESI-TOF) m/z Calcd. for $\text{C}_{14}\text{H}_{14}\text{Cl}_2\text{F}_3\text{N}_2\text{O}_3^+$ ($\text{M}+\text{H}$) $^+$: 385.0328; found: 385.0332.



(*E*)-2,4-dichloro-6-hydroxybenzaldehyde O-cyclopropylmethyl oxime (1ah)

Following General procedure A, the product is obtained as a colorless oil, 58% yield. ^1H NMR (600 MHz, Chloroform-*d*) δ 10.76 (s, 1H), 8.66 (d, $J = 0.6$ Hz, 1H), 6.95 (d, $J = 2.1$ Hz, 1H), 6.90 (dd, $J = 2.1, 0.6$ Hz, 1H), 4.01 (d, $J = 7.2$ Hz, 2H), 1.26 – 1.14 (m, 1H), 0.70 – 0.59 (m, 2H), 0.39 – 0.29 (m, 2H); ^{13}C NMR (151 MHz, Chloroform-*d*) δ 159.22, 148.00, 136.47, 134.63, 120.81, 116.12, 113.08, 79.96, 9.95, 3.17; HRMS (ESI-TOF) m/z Calcd. for $\text{C}_{11}\text{H}_{12}\text{Cl}_2\text{NO}_2^+$ ($\text{M}+\text{H}$) $^+$: 260.0240; found: 260.0241.



(*E*)-2,4-dichloro-6-hydroxybenzaldehyde O-((3*S*,8*R*,9*S*,10*S*,13*R*,14*S*,17*R*)-10,13-dimethyl-17-((*R*)-6-methylheptan-2-yl)hexadecahydro-1*H*-cyclopenta[*a*]phenanthren-3-yl) oxime (4)

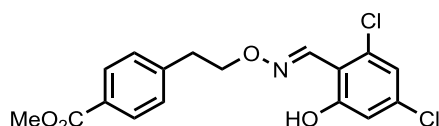
Following General procedure C, the product is obtained as a white solid, 48% yield. $[\alpha]_D^{20} = +24.7$ ($c=1$, CHCl_3); ^1H NMR (600 MHz, Chloroform-*d*) δ 10.96 (s, 1H), 8.69 (s, 1H), 6.98 (t, $J = 2.2$ Hz, 1H), 6.93 (d, $J = 2.0$ Hz, 1H), 4.43 (q, $J = 2.9$ Hz, 1H), 2.04 – 1.93 (m, 2H), 1.83 (dtd, $J = 13.3, 9.3, 5.6$ Hz, 1H), 1.77 – 1.65 (m, 3H), 1.64 – 1.44 (m, 8H), 1.43 – 1.33 (m, 4H), 1.31 – 1.19 (m, 5H), 1.19 – 1.08 (m, 5H),

1.07 – 0.97 (m, 3H), 0.95 – 0.86 (m, 9H), 0.84 (s, 3H), 0.68 (s, 3H); ^{13}C NMR (151 MHz, Chloroform-*d*) δ 158.80, 147.03, 135.78, 134.05, 120.33, 115.64, 112.94, 79.17, 56.07, 55.83, 53.70, 42.13, 39.56, 39.10, 39.06, 35.72, 35.36, 35.33, 35.02, 32.05, 31.95, 31.47, 28.03, 27.79, 27.55, 25.06, 23.72, 23.41, 22.37, 22.10, 20.34, 18.21, 11.62, 10.96; HRMS (ESI-TOF) m/z Calcd. for $\text{C}_{34}\text{H}_{52}\text{Cl}_2\text{NO}_2^+$ (M+H) $^+$: 576.3370; found: 576.3373.

C(sp³)-H arylation of aliphatic alcohols

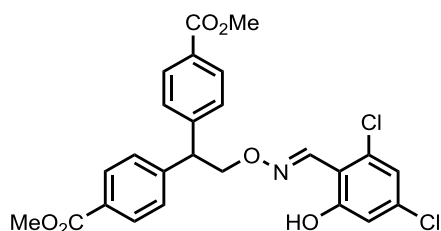
General procedure D:

To a 8-mL vial were added **DG9**-tethered alcohol (0.1 mmol), methyl 4-iodobenzoate (78 mg, 0.3 mmol), palladium acetate (2.3 mg, 0.01 mmol), 3-nitro-5-trifluoromethyl-2-pyridone (8.1 mg, 0.04 mmol), and silver trifluoroacetate (33 mg, 0.15 mmol). The mixture was dissolved with 1,1,1,3,3,3-hexafluoro-2-propanol (HFIP, 1.0 mL). The vial was sealed and stirred at 100 °C for 12 hours unless otherwise stated. The reaction was cooled to room temperature, diluted with 5 mL of EtOAc, and filtered through a pad of Celite. The filtrate was concentrated and purified by preparative thin-layer chromatography.



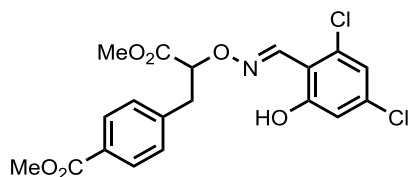
Methyl (*E*)-4-(2-(((2,4-dichloro-6-hydroxybenzylidene)amino)oxy)ethyl)benzoate (**2a**)

Following General procedure D, the product is obtained as a colorless oil, 30% yield. ^1H NMR (600 MHz, CDCl_3) δ 10.55 (br s, 1H), 8.63 (s, 1H), 7.99 (d, $J = 8.4$ Hz, 2H), 7.31 (d, $J = 8.4$ Hz, 2H), 6.96 (d, $J = 2.4$ Hz, 1H), 6.91 (d, $J = 2.4$ Hz, 1H), 4.44 (t, $J = 6.6$ Hz, 2H), 3.91 (s, 3H), 3.10 (t, $J = 6.6$ Hz, 2H); ^{13}C NMR (151 MHz, CDCl_3) δ 166.9, 159.2, 148.7, 143.2, 136.8, 134.8, 129.9, 128.9, 128.6, 121.0, 116.2, 112.9, 75.1, 52.1, 35.3; HRMS (ESI-TOF) m/z Calcd. for $\text{C}_{17}\text{H}_{16}\text{Cl}_2\text{NO}_4^+$ (M+H) $^+$: 368.0456; found: 368.0451.



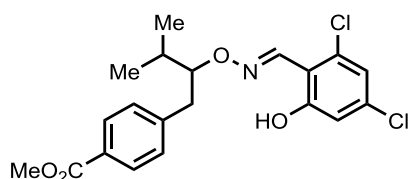
Dimethyl 4,4'-(2-(((2,4-dichloro-6-hydroxybenzylidene)amino)oxy)ethane-1,1-diyl)(*E*)-dibenzoate (2a')

Following General procedure D, the product is obtained as a colorless oil, 45% yield. ^1H NMR (600 MHz, CDCl_3) δ 10.44 (br s, 1H), 8.57 (s, 1H), 8.03 (d, $J = 8.4$ Hz, 4H), 7.35 (d, $J = 8.4$ Hz, 4H), 6.96 (d, $J = 1.8$ Hz, 1H), 6.92 (d, $J = 1.8$ Hz, 1H), 4.79 (d, $J = 7.8$ Hz, 2H), 4.60 (t, $J = 7.8$ Hz, 1H), 3.92 (s, 6H); ^{13}C NMR (151 MHz, CDCl_3) δ 166.7, 159.2, 149.2, 145.3, 137.0, 134.9, 130.1, 129.1, 128.3, 121.0, 116.2, 112.7, 77.0, 52.2, 50.1; HRMS (ESI-TOF) m/z Calcd. for $\text{C}_{25}\text{H}_{22}\text{Cl}_2\text{NO}_6^+$ ($\text{M}+\text{H}$) $^+$: 502.0819; found: 502.0822.



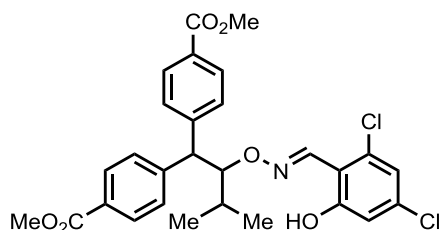
Methyl (*E*)-4-(2-(((2,4-dichloro-6-hydroxybenzylidene)amino)oxy)-3-methoxy-3-oxopropyl)benzoate (2b)

Following General procedure D, the product is obtained as a colorless oil, 65% yield. ^1H NMR (600 MHz, Chloroform-*d*) δ 10.09 (s, 1H), 8.73 (d, $J = 0.6$ Hz, 1H), 8.06 – 7.99 (m, 2H), 7.39 – 7.33 (m, 2H), 6.99 (d, $J = 2.0$ Hz, 1H), 6.91 (dd, $J = 2.0, 0.6$ Hz, 1H), 4.98 (dd, $J = 8.3, 5.0$ Hz, 1H), 3.93 (s, 3H), 3.78 (s, 3H), 3.34 – 3.25 (m, 2H); ^{13}C NMR (151 MHz, Chloroform-*d*) δ 170.50, 166.80, 159.16, 150.10, 140.95, 137.39, 135.18, 129.89, 129.33, 129.11, 121.12, 116.28, 112.46, 82.56, 52.43, 52.10, 37.22; HRMS (ESI-TOF) m/z Calcd. for $\text{C}_{19}\text{H}_{18}\text{Cl}_2\text{NO}_6^+$ ($\text{M}+\text{H}$) $^+$: 426.0511; found: 426.0513.



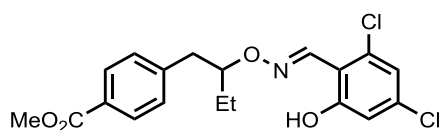
Methyl (E)-4-(2-(((2,4-dichloro-6-hydroxybenzylidene)amino)oxy)-3-methylbutyl) benzoate (2c)

Following General procedure D, the product is obtained as a colorless oil, 52% yield. ^1H NMR (600 MHz, Chloroform-*d*) δ 10.53 (s, 1H), 8.60 (d, $J = 0.6$ Hz, 1H), 7.98 (d, $J = 8.3$ Hz, 2H), 7.32 (d, $J = 8.3$ Hz, 2H), 6.96 (d, $J = 2.0$ Hz, 1H), 6.89 (dd, $J = 2.0, 0.5$ Hz, 1H), 4.26 (dt, $J = 7.6, 5.3$ Hz, 1H), 3.92 (s, 3H), 3.05 – 2.96 (m, 2H), 2.05 (pd, $J = 6.9, 5.2$ Hz, 1H), 1.06 (t, $J = 7.1$ Hz, 6H); ^{13}C NMR (151 MHz, Chloroform-*d*) δ 166.53, 158.66, 147.37, 143.52, 136.04, 134.11, 129.27, 128.86, 127.89, 120.40, 115.64, 112.61, 89.72, 51.56, 36.34, 30.55, 17.87, 17.42; HRMS (ESI-TOF) m/z Calcd. for $\text{C}_{20}\text{H}_{22}\text{Cl}_2\text{NO}_4^+$ ($\text{M}+\text{H}$) $^+$: 410.0926; found: 410.0922.



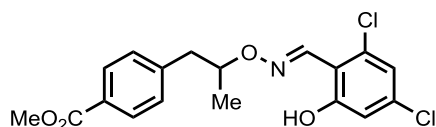
Dimethyl 4,4'-(2-(((2,4-dichloro-6-hydroxybenzylidene)amino)oxy)-3-methylbutane-1,1-diyl)(E)-dibenzoate (2c')

Following General procedure D, the product is obtained as a pale-yellow oil, 20% yield. ^1H NMR (600 MHz, Chloroform-*d*) δ 10.19 (s, 1H), 8.39 (s, 1H), 7.95 (dd, $J = 12.9, 8.4$ Hz, 4H), 7.45 (d, $J = 8.3$ Hz, 2H), 7.36 (d, $J = 8.3$ Hz, 2H), 6.90 (d, $J = 2.0$ Hz, 1H), 6.83 (dd, $J = 2.0, 0.5$ Hz, 1H), 4.82 (dd, $J = 9.1, 4.3$ Hz, 1H), 4.39 (d, $J = 9.0$ Hz, 1H), 3.88 (s, 3H), 3.86 (s, 3H), 1.87 (pd, $J = 6.8, 4.2$ Hz, 1H), 1.02 (d, $J = 6.9$ Hz, 3H), 0.98 (d, $J = 6.8$ Hz, 3H); ^{13}C NMR (151 MHz, Chloroform-*d*) δ 166.73, 166.62, 158.96, 147.29, 146.16, 145.73, 136.54, 134.52, 130.18, 129.88, 128.92, 128.77, 128.34, 120.83, 116.07, 112.77, 91.70, 54.03, 52.11, 52.04, 30.44, 20.47, 16.31; HRMS (ESI-TOF) m/z Calcd. for $\text{C}_{28}\text{H}_{28}\text{Cl}_2\text{NO}_6^+$ ($\text{M}+\text{H}$) $^+$: 544.1294; found: 544.1293.



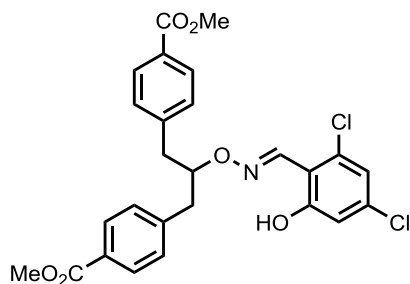
Methyl (*E*)-4-(2-(((2,4-dichloro-6-hydroxybenzylidene)amino)oxy)butyl)benzoate (2d)

Following General procedure D, the product is obtained as a pale-yellow oil, 68% yield. ¹H NMR (600 MHz, Chloroform-*d*) δ 10.62 (s, 1H), 8.62 (s, 1H), 7.97 (d, *J* = 8.3 Hz, 2H), 7.28 (d, *J* = 8.3 Hz, 2H), 6.96 (d, *J* = 2.0 Hz, 1H), 6.90 (d, *J* = 2.0 Hz, 1H), 4.41 – 4.32 (m, 1H), 3.90 (s, 3H), 3.06 (dd, *J* = 14.1, 7.0 Hz, 1H), 2.97 (dd, *J* = 14.1, 5.7 Hz, 1H), 1.74 – 1.66 (m, 2H), 1.00 (t, *J* = 7.4 Hz, 3H); ¹³C NMR (151 MHz, Chloroform-*d*) δ 166.97, 159.17, 148.20, 143.27, 136.60, 134.65, 129.73, 129.42, 128.45, 120.91, 116.13, 113.09, 52.03, 39.65, 26.11, 9.65; HRMS (ESI-TOF) *m/z* Calcd. for C₁₉H₂₀Cl₂NO₄⁺ (M+H)⁺: 396.0764; found: 396.0763.



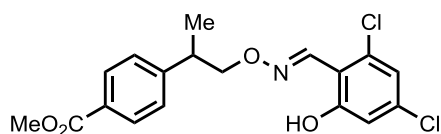
Methyl (*E*)-4-(2-(((2,4-dichloro-6-hydroxybenzylidene)amino)oxy)propyl)benzoate (2e)

Following General procedure D, the product is obtained as a colorless oil, 63% yield. ¹H NMR (600 MHz, Chloroform-*d*) δ 10.64 (s, 1H), 8.62 (s, 1H), 7.98 (d, *J* = 8.3 Hz, 2H), 7.29 (d, *J* = 8.4 Hz, 2H), 6.96 (d, *J* = 2.0 Hz, 1H), 6.90 (dd, *J* = 2.0, 0.5 Hz, 1H), 4.57 (h, *J* = 6.3 Hz, 1H), 3.90 (s, 3H), 3.10 (dd, *J* = 13.9, 6.6 Hz, 1H), 2.92 (dd, *J* = 13.9, 6.2 Hz, 1H), 1.32 (d, *J* = 6.3 Hz, 3H); ¹³C NMR (151 MHz, Chloroform-*d*) δ 166.94, 159.18, 148.30, 142.95, 136.63, 134.68, 129.73, 129.45, 128.53, 120.91, 116.14, 113.04, 81.05, 52.03, 41.75, 19.15; HRMS (ESI-TOF) *m/z* Calcd. for C₁₈H₁₈Cl₂NO₄⁺ (M+H)⁺: 382.0607; found: 382.0609.



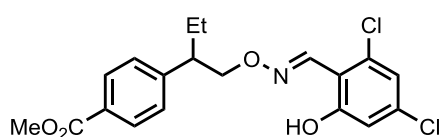
Dimethyl 4,4'-(2-(((2,4-dichloro-6-hydroxybenzylidene)amino)oxy)propane-1,3-diyl)(*E*)-dibenzoate (2e')

Following General procedure D, the product is obtained as a colorless oil, 21% yield. ^1H NMR (600 MHz, Chloroform-*d*) δ 10.38 (s, 1H), 8.62 (s, 1H), 8.00 (d, $J = 8.4$ Hz, 4H), 7.29 (t, $J = 4.2$ Hz, 4H), 6.98 (d, $J = 2.0$ Hz, 1H), 6.91 (dd, $J = 2.0, 0.5$ Hz, 1H), 4.73 (tt, $J = 7.3, 5.4$ Hz, 1H), 3.93 (s, 6H), 3.12 – 2.99 (m, 4H); ^{13}C NMR (151 MHz, Chloroform-*d*) δ 166.88, 159.13, 148.87, 142.62, 136.92, 134.79, 129.83, 129.42, 128.70, 121.01, 116.18, 112.81, 85.27, 52.07, 39.67; HRMS (ESI-TOF) m/z Calcd. for $\text{C}_{26}\text{H}_{24}\text{Cl}_2\text{NO}_6^+$ ($\text{M}+\text{H}$) $^+$: 516.0975; found: 516.0983.



Methyl (E)-4-(1-(((2,4-dichloro-6-hydroxybenzylidene)amino)oxy)propan-2-yl)benzoate (2f)

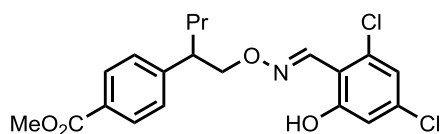
Following General procedure D, the product is obtained as a colorless oil, 85% yield. ^1H NMR (600 MHz, CDCl_3) δ 10.54 (br s, 1H), 8.58 (s, 1H), 8.00 (d, $J = 8.4$ Hz, 2H), 7.32 (d, $J = 8.4$ Hz, 2H), 6.94 (d, $J = 1.8$ Hz, 1H), 6.89 (d, $J = 1.8$ Hz, 1H), 4.34-4.25 (m, 2H), 3.91 (s, 3H), 3.31-3.25 (m, 1H), 1.36 (d, $J = 7.2$ Hz, 3H); ^{13}C NMR (151 MHz, CDCl_3) δ 166.9, 159.2, 148.5, 148.4, 136.8, 134.8, 129.9, 128.7, 127.4, 120.9, 116.2, 112.9, 79.9, 52.1, 39.4, 17.9; HRMS (ESI-TOF) m/z Calcd. for $\text{C}_{18}\text{H}_{18}\text{Cl}_2\text{NO}_4^+$ ($\text{M}+\text{H}$) $^+$: 382.0607; found: 382.0609.



Methyl (E)-4-(1-(((2,4-dichloro-6-hydroxybenzylidene)amino)oxy)butan-2-yl)benzoate (2g)

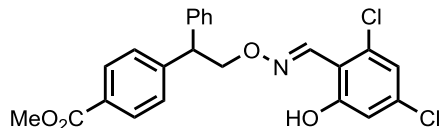
Following General procedure D, the product is obtained as a colorless oil, 82% yield. ^1H NMR (600 MHz, CDCl_3) δ 10.54 (br s, 1H), 8.55 (s, 1H), 8.00 (d, $J = 8.4$ Hz, 2H), 7.28 (d, $J = 8.4$ Hz, 2H), 6.94 (d, $J = 1.8$ Hz, 1H), 6.89 (d, $J = 1.8$ Hz, 1H), 4.38-4.32 (m, 2H), 3.91 (s, 3H), 3.04-2.99 (m, 1H), 1.91-1.84 (m, 1H), 1.70-1.62 (m, 1H), 0.85 (t, $J = 7.2$ Hz, 3H); ^{13}C NMR (151 MHz, CDCl_3) δ 167.0, 159.2, 148.4, 147.2, 136.7,

134.7, 129.9, 128.8, 128.0, 120.9, 116.2, 112.9, 78.9, 52.1, 47.1, 25.3, 11.8; HRMS (ESI-TOF) m/z Calcd. for $C_{19}H_{20}Cl_2NO_4^+$ ($M+H$) $^+$: 396.0764; found: 396.0763.



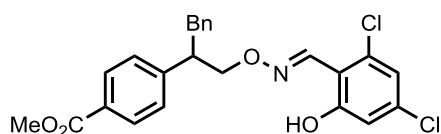
Methyl (E)-4-(1-(((2,4-dichloro-6-hydroxybenzylidene)amino)oxy)pentan-2-yl)benzoate (2h)

Following General procedure D, the product is obtained as a colorless oil, 72% yield. 1H NMR (600 MHz, $CDCl_3$) δ 10.54 (br s, 1H), 8.55 (s, 1H), 8.00 (d, $J = 8.4$ Hz, 2H), 7.29 (d, $J = 8.4$ Hz, 2H), 6.94 (d, $J = 1.8$ Hz, 1H), 6.89 (d, $J = 1.8$ Hz, 1H), 4.36-4.31 (m, 2H), 3.90 (s, 3H), 3.13-3.10 (m, 1H), 1.78-1.75 (m, 1H), 1.67-1.60 (m, 1H), 1.25-1.21 (m, 2H), 0.88 (t, $J = 7.2$ Hz, 3H); ^{13}C NMR (151 MHz, $CDCl_3$) δ 167.0, 159.2, 148.4, 147.5, 136.7, 134.7, 129.9, 128.7, 128.0, 120.9, 116.2, 112.9, 79.1, 52.1, 45.2, 34.4, 20.3, 14.0; HRMS (ESI-TOF) m/z Calcd. for $C_{20}H_{22}Cl_2NO_4^+$ ($M+H$) $^+$: 410.0926; found: 410.0927.



Methyl (E)-4-(2-(((2,4-dichloro-6-hydroxybenzylidene)amino)oxy)-1-phenylethyl)benzoate (2i)

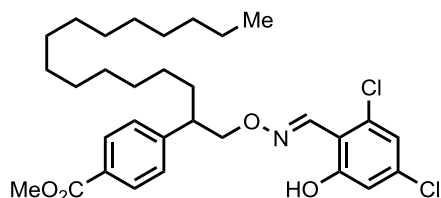
Following General procedure D, the product is obtained as a colorless oil, 61% yield. 1H NMR (600 MHz, $CDCl_3$) δ 10.50 (br s, 1H), 8.56 (s, 1H), 8.00 (d, $J = 8.4$ Hz, 2H), 7.36 (d, $J = 8.4$ Hz, 2H), 7.34-7.33 (m, 2H), 7.28-7.24 (m, 3H), 6.94 (d, $J = 1.8$ Hz, 1H), 6.91 (d, $J = 1.8$ Hz, 1H), 4.79-4.74 (m, 2H), 4.53 (t, $J = 7.2$ Hz, 1H), 3.90 (s, 3H); ^{13}C NMR (151 MHz, $CDCl_3$) δ 166.8, 159.2, 148.9, 146.3, 140.2, 136.9, 134.9, 130.0, 128.9, 128.4, 128.2, 127.2, 121.0, 116.2, 112.8, 77.4, 52.1, 50.1; HRMS (ESI-TOF) m/z Calcd. for $C_{23}H_{20}Cl_2NO_4^+$ ($M+H$) $^+$: 444.0764; found: 444.0761.



Methyl (*E*)-4-(1-(((2,4-dichloro-6-hydroxybenzylidene)amino)oxy)-3-phenylpropan-2-yl)benzoate (2j)

Following General procedure D, the product is obtained as a colorless oil, 54% yield.

^1H NMR (600 MHz, CDCl_3) δ 10.48 (br s, 1H), 8.58 (s, 1H), 8.00 (d, $J = 8.4$ Hz, 2H), 7.28 (d, $J = 8.4$ Hz, 2H), 7.26-7.23 (m, 2H), 7.20-7.18 (m, 1H), 7.08-7.06 (m, 2H), 6.97 (d, $J = 1.8$ Hz, 1H), 6.92 (d, $J = 1.8$ Hz, 1H), 4.43 (d, $J = 6.6$ Hz, 2H), 3.93 (s, 3H), 3.47-3.43 (m, 1H), 3.17-3.13 (m, 1H), 3.01-2.98 (m, 1H); ^{13}C NMR (151 MHz, CDCl_3) δ 166.9, 159.2, 148.6, 146.7, 138.7, 136.8, 134.8, 129.9, 129.0, 128.9, 128.4, 128.1, 126.4, 121.0, 116.2, 112.8, 77.8, 52.1, 47.1, 39.0; HRMS (ESI-TOF) m/z Calcd. for $\text{C}_{24}\text{H}_{22}\text{Cl}_2\text{NO}_4^+$ ($\text{M}+\text{H}$) $^+$: 458.0920; found: 458.0919.

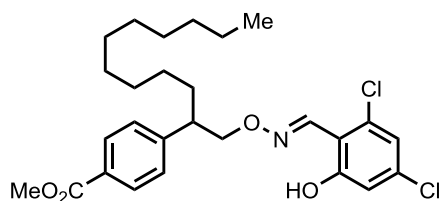


Methyl

(*E*)-4-(1-(((2,4-dichloro-6-hydroxybenzylidene)amino)oxy)hexadecan-2-yl)benzoate (2k)

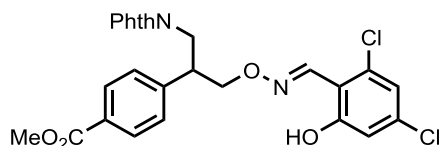
Following General procedure D, the product is obtained as a colorless oil, 71% yield.

^1H NMR (600 MHz, CDCl_3) δ 10.54 (br s, 1H), 8.55 (s, 1H), 8.00 (d, $J = 8.4$ Hz, 2H), 7.28 (d, $J = 8.4$ Hz, 2H), 6.94 (d, $J = 1.8$ Hz, 1H), 6.89 (d, $J = 1.8$ Hz, 1H), 4.36-4.32 (m, 2H), 3.90 (s, 3H), 3.12-3.07 (m, 1H), 1.82-1.77 (m, 1H), 1.67-1.60 (m, 1H), 1.30-1.16 (m, 24H), 0.88 (t, $J = 7.2$ Hz, 3H); ^{13}C NMR (151 MHz, CDCl_3) δ 166.96, 159.20, 148.42, 147.53, 136.72, 134.73, 129.90, 128.72, 127.95, 120.92, 116.19, 112.86, 79.15, 52.04, 45.45, 32.27, 31.94, 29.70, 29.69, 29.67, 29.66, 29.62, 29.57, 29.55, 29.43, 29.38, 27.15, 22.71, 14.14; HRMS (ESI-TOF) m/z Calcd. for $\text{C}_{31}\text{H}_{44}\text{Cl}_2\text{NO}_4^+$ ($\text{M}+\text{H}$) $^+$: 564.2647; found: 564.2657.



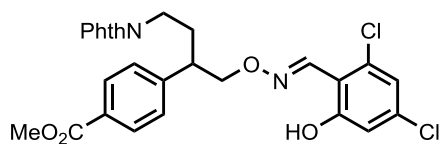
Methyl (E)-4-(1-(((2,4-dichloro-6-hydroxybenzylidene)amino)oxy)dodecan-2-yl)benzoate (2l)

Following General procedure D, the product is obtained as a colorless oil, 76% yield. ¹H NMR (600 MHz, CDCl₃) δ 10.54 (br s, 1H), 8.55 (s, 1H), 8.00 (d, *J* = 8.4 Hz, 2H), 7.28 (d, *J* = 8.4 Hz, 2H), 6.93 (d, *J* = 2.4 Hz, 1H), 6.89 (d, *J* = 2.4 Hz, 1H), 4.36-4.30 (m, 2H), 3.90 (s, 3H), 3.12-3.07 (m, 1H), 1.83-1.77 (m, 1H), 1.67-1.61 (m, 1H), 1.29-1.17 (m, 16H), 0.87 (t, *J* = 7.2 Hz, 3H); ¹³C NMR (151 MHz, CDCl₃) δ 166.95, 159.19, 148.42, 147.53, 136.72, 134.73, 129.90, 128.72, 127.95, 120.91, 116.19, 112.86, 79.15, 52.04, 45.45, 32.26, 31.90, 29.57, 29.56, 29.55, 29.42, 29.31, 27.14, 22.69, 14.12; HRMS (ESI-TOF) *m/z* Calcd. for C₂₇H₃₆Cl₂NO₄⁺ (M+H)⁺: 508.2021; found: 508.2023.



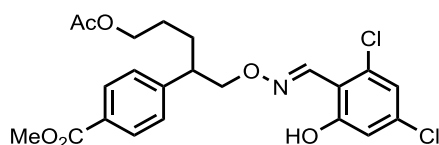
Methyl (E)-4-(1-(((2,4-dichloro-6-hydroxybenzylidene)amino)oxy)-3-(1,3-dioxoisindolin-2-yl)propan-2-yl)benzoate (2m)

Following General procedure D, the product is obtained as a pale-yellow oil, 58% yield. ¹H NMR (600 MHz, CDCl₃) δ 10.31 (br s, 1H), 8.40 (s, 1H), 7.99 (d, *J* = 8.4 Hz, 2H), 7.80-7.78 (m, 2H), 7.69-7.67 (m, 2H), 7.39 (d, *J* = 8.4 Hz, 2H), 6.92 (d, *J* = 2.4 Hz, 1H), 6.88 (d, *J* = 2.4 Hz, 1H), 4.50-4.45 (m, 2H), 4.13-4.03 (m, 2H), 3.89 (s, 3H), 3.84-3.80 (m, 1H); ¹³C NMR (151 MHz, CDCl₃) δ 168.1, 166.7, 159.2, 148.9, 143.4, 136.9, 134.8, 134.1, 131.7, 130.1, 129.6, 128.2, 123.4, 120.9, 116.3, 112.6, 77.0, 52.1, 43.5, 40.5; HRMS (ESI-TOF) *m/z* Calcd. for C₂₆H₂₁Cl₂N₂O₆⁺ (M+H)⁺: 527.0771; found: 527.0768.



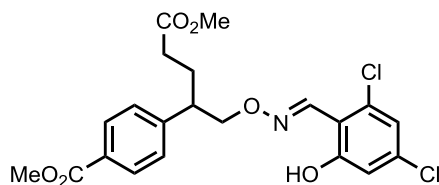
Methyl (E)-4-(1-(((2,4-dichloro-6-hydroxybenzylidene)amino)oxy)-4-(1,3-dioxoisindolin-2-yl)butan-2-yl)benzoate (2n)

Following General procedure D, the product is obtained as a pale-yellow oil, 62% yield. ^1H NMR (600 MHz, CDCl_3) δ 10.38 (s, 1H), 8.54 (s, 1H), 7.91 (d, $J = 8.4$ Hz, 2H), 7.72 (dd, $J = 5.4, 3.0$ Hz, 2H), 7.62 (dd, $J = 5.4, 3.0$ Hz, 2H), 7.32 (d, $J = 8.4$ Hz, 2H), 6.93 (d, $J = 2.4$ Hz, 1H), 6.85 (d, $J = 2.4$ Hz, 1H), 4.29 (d, $J = 6.6$ Hz, 2H), 3.87 (s, 3H), 3.67 (t, $J = 6.6$ Hz, 2H), 3.20-3.16 (m, 1H), 2.27-2.17 (m, 2H); ^{13}C NMR (151 MHz, CDCl_3) δ 168.2, 166.7, 159.1, 148.8, 145.6, 136.8, 134.8, 133.8, 131.8, 130.0, 128.9, 128.0, 123.1, 120.9, 116.2, 112.7, 78.6, 52.0, 43.4, 36.2, 30.2; HRMS (ESI-TOF) m/z Calcd. for $\text{C}_{27}\text{H}_{23}\text{Cl}_2\text{N}_2\text{O}_6^+$ ($\text{M}+\text{H}$) $^+$: 541.0933; found: 541.0938.



Methyl (E)-4-(5-acetoxy-1-(((2,4-dichloro-6-hydroxybenzylidene)amino)oxy)pentan-2-yl)benzoate (2o)

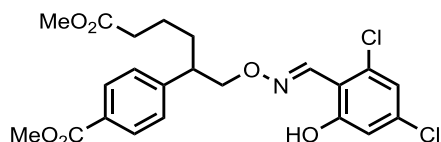
Following General procedure D, the product is obtained as a colorless oil, 69% yield. ^1H NMR (600 MHz, CDCl_3) δ 10.48 (s, 1H), 8.56 (s, 1H), 8.01 (d, $J = 8.4$ Hz, 2H), 7.29 (d, $J = 8.4$ Hz, 2H), 6.94 (d, $J = 2.4$ Hz, 1H), 6.89 (d, $J = 2.4$ Hz, 1H), 4.34 (d, $J = 6.6$ Hz, 2H), 4.02 (t, $J = 6.6$ Hz, 2H), 3.90 (s, 3H), 3.14-3.09 (m, 1H), 2.02 (s, 3H), 1.94-1.88 (m, 1H), 1.72-1.68 (m, 1H), 1.59-1.50 (m, 2H); ^{13}C NMR (151 MHz, CDCl_3) δ 171.1, 166.8, 159.2, 148.6, 146.6, 136.8, 134.8, 130.1, 129.0, 127.9, 121.0, 116.2, 112.8, 78.9, 64.0, 52.1, 45.1, 28.6, 26.3, 20.9; HRMS (ESI-TOF) m/z Calcd. for $\text{C}_{22}\text{H}_{24}\text{Cl}_2\text{NO}_6^+$ ($\text{M}+\text{H}$) $^+$: 468.0981; found: 468.0984.



Methyl (E)-4-(1-(((2,4-dichloro-6-hydroxybenzylidene)amino)oxy)-5-methoxy-5-oxopentan-2-yl)benzoate (2p)

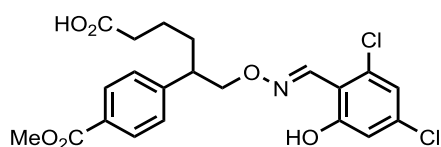
Following General procedure D, the product is obtained as a colorless oil, 70% yield. ^1H NMR (600 MHz, CDCl_3) δ 10.45 (s, 1H), 8.56 (s, 1H), 8.00 (d, $J = 8.4$ Hz, 2H), 7.28 (d, $J = 8.4$ Hz, 2H), 6.93 (d, $J = 2.4$ Hz, 1H), 6.88 (d, $J = 2.4$ Hz, 1H), 4.34 (d, J

= 7.2 Hz, 2H), 3.90 (s, 3H), 3.61 (s, 3H), 3.19-3.12 (m, 1H), 2.25-2.18 (m, 3H), 2.01-1.92 (m, 1H); ¹³C NMR (151 MHz, CDCl₃) δ 173.3, 166.8, 159.2, 148.7, 145.9, 136.8, 134.8, 130.1, 129.2, 128.0, 121.0, 116.2, 112.8, 78.7, 52.1, 51.6, 44.7, 31.6, 27.3; HRMS (ESI-TOF) m/z Calcd. for C₂₁H₂₂Cl₂NO₆⁺ (M+H)⁺: 454.0824; found: 454.0823.



Methyl (E)-4-(1-(((2,4-dichloro-6-hydroxybenzylidene)amino)oxy)-6-methoxy-6-oxohexan-2-yl)benzoate (2q)

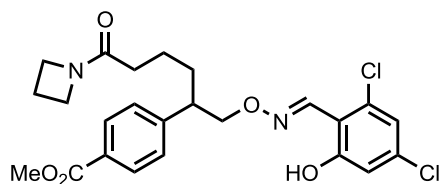
Following General procedure D, the product is obtained as a colorless oil, 73% yield. ¹H NMR (600 MHz, CDCl₃) δ 10.47 (s, 1H), 8.54 (s, 1H), 8.00 (d, *J* = 8.4 Hz, 2H), 7.28 (d, *J* = 8.4 Hz, 2H), 6.93 (d, *J* = 2.4 Hz, 1H), 6.88 (d, *J* = 2.4 Hz, 1H), 4.32 (d, *J* = 7.2 Hz, 2H), 3.90 (s, 3H), 3.63 (s, 3H), 3.12-3.09 (m, 1H), 2.30-2.27 (m, 2H), 1.88-1.82 (m, 1H), 1.73-1.66 (m, 1H), 1.59-1.48 (m, 2H); ¹³C NMR (151 MHz, CDCl₃) δ 173.6, 166.9, 159.2, 148.6, 146.7, 136.8, 134.8, 130.0, 129.0, 127.9, 120.9, 116.2, 112.8, 78.9, 52.1, 51.5, 45.3, 33.8, 31.6, 22.6; HRMS (ESI-TOF) m/z Calcd. for C₂₂H₂₄Cl₂NO₆⁺ (M+H)⁺: 468.0981; found: 468.0985.



(E)-6-(((2,4-dichloro-6-hydroxybenzylidene)amino)oxy)-5-(4-(methoxycarbonyl)phenyl)hexanoic acid (2r)

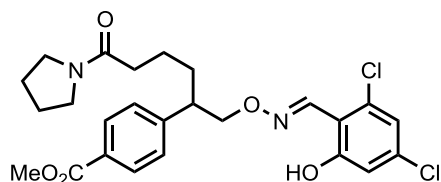
Following General procedure D, the product is obtained as a colorless oil, 71% yield. ¹H NMR (600 MHz, CDCl₃) δ 10.48 (s, 1H), 8.56 (s, 1H), 8.00 (d, *J* = 8.4 Hz, 2H), 7.29 (d, *J* = 8.4 Hz, 2H), 6.94 (d, *J* = 2.4 Hz, 1H), 6.89 (d, *J* = 2.4 Hz, 1H), 4.33 (d, *J* = 7.2 Hz, 2H), 3.91 (s, 3H), 3.12-3.10 (m, 1H), 2.37-2.28 (m, 2H), 1.91-1.86 (m, 1H), 1.75-1.68 (m, 1H), 1.60-1.49 (m, 2H); ¹³C NMR (151 MHz, CDCl₃) δ 178.9, 166.9, 159.2, 148.6, 146.6, 136.8, 134.8, 130.1, 129.0, 127.9, 121.0, 116.2, 112.8, 78.9, 52.1,

45.3, 33.7, 31.5, 22.3; HRMS (ESI-TOF) m/z Calcd. for $C_{21}H_{21}Cl_2NO_6^+$ (M+H)⁺: 454.0819; found: 454.0816.



Methyl (E)-4-(6-(azetidin-1-yl)-1-(((2,4-dichloro-6-hydroxybenzylidene)amino)oxy)-6-oxohexan-2-yl)benzoate (2s)

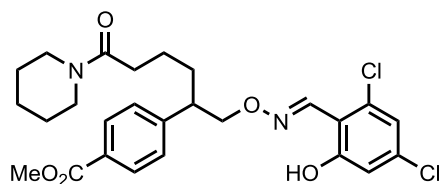
Following General procedure D, the product is obtained as a pale-yellow oil, 58% yield. ¹H NMR (600 MHz, CDCl₃) δ 10.49 (br s, 1H), 8.55 (s, 1H), 8.00 (d, $J = 8.4$ Hz, 2H), 7.29 (d, $J = 8.4$ Hz, 2H), 6.94 (d, $J = 2.4$ Hz, 1H), 6.89 (d, $J = 2.4$ Hz, 1H), 4.35-4.30 (m, 2H), 4.01 (t, $J = 7.8$ Hz, 4H), 3.90 (s, 3H), 3.13-3.08 (m, 1H), 2.25-2.20 (m, 2H), 2.03 (td, $J = 7.2, 2.0$ Hz, 2H), 1.89-1.83 (m, 1H), 1.73-1.66 (m, 1H), 1.58-1.46 (m, 2H); ¹³C NMR (151 MHz, CDCl₃) δ 172.1, 166.5, 158.7, 148.1, 146.4, 136.3, 134.3, 129.5, 128.4, 127.5, 120.5, 115.7, 112.4, 78.5, 51.6, 48.7 (broad), 44.9, 31.4, 30.3, 22.0, 14.6; HRMS (ESI-TOF) m/z Calcd. for $C_{24}H_{27}Cl_2N_2O_5^+$ (M+H)⁺: 493.1292; found: 493.1295.



Methyl (E)-4-(1-(((2,4-dichloro-6-hydroxybenzylidene)amino)oxy)-6-oxo-6-(pyrrolidin-1-yl)hexan-2-yl)benzoate (2t)

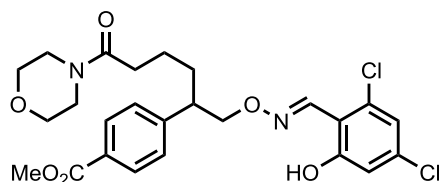
Following General procedure D, the product is obtained as a pale-yellow oil, 48% yield. ¹H NMR (600 MHz, CDCl₃) δ 10.50 (br s, 1H), 8.55 (s, 1H), 7.99 (d, $J = 8.4$ Hz, 2H), 7.30 (d, $J = 8.4$ Hz, 2H), 6.94 (d, $J = 2.4$ Hz, 1H), 6.89 (d, $J = 2.4$ Hz, 1H), 4.37-4.31 (m, 2H), 3.90 (s, 3H), 3.42-3.32 (br 4H), 3.14-3.11 (m, 1H), 2.26-2.20 (m, 2H), 1.92-1.83 (m, 5H), 1.76-1.70 (m, 1H), 1.65-1.51 (m, 2H); ¹³C NMR (151 MHz, CDCl₃) δ 170.7, 166.5, 158.7, 148.1, 146.6, 136.3, 134.3, 129.5, 128.4, 127.5, 120.5, 115.7, 112.4, 78.6, 51.6, 46.1 (broad), 45.4 (broad), 45.0, 33.9, 31.4, 25.6 (broad),

23.9 (broad), 22.1; HRMS (ESI-TOF) m/z Calcd. for $C_{25}H_{29}Cl_2N_2O_5^+$ (M+H)⁺: 507.1454; found: 507.1457.



Methyl (E)-4-(1-(((2,4-dichloro-6-hydroxybenzylidene)amino)oxy)-6-oxo-6-(piperidin-1-yl)hexan-2-yl)benzoate (2u)

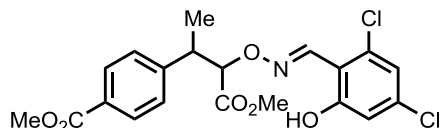
Following General procedure D, the product is obtained as a pale-yellow oil, 54% yield. ¹H NMR (600 MHz, Chloroform-*d*) δ 10.52 (s, 1H), 8.57 (s, 1H), 8.02 (d, J = 8.3 Hz, 2H), 7.32 (d, J = 8.3 Hz, 2H), 6.97 (d, J = 2.0 Hz, 1H), 6.92 (d, J = 2.0 Hz, 1H), 4.41 – 4.30 (m, 2H), 3.93 (s, 3H), 3.65 – 3.20 (m, 4H), 3.15 (dq, J = 11.9, 6.8 Hz, 1H), 2.41 – 2.25 (m, 2H), 1.95 – 1.89 (m, 1H), 1.77 – 1.71 (m, 1H), 1.65 – 1.47 (m, 8H); ¹³C NMR (151 MHz, Chloroform-*d*) δ 170.72, 166.88, 159.16, 148.50, 146.96, 136.73, 134.73, 129.95, 128.84, 127.97, 120.91, 116.17, 112.81, 78.97, 52.04, 45.41, 32.98, 31.86, 25.94 (broad), 24.46, 22.98; HRMS (ESI-TOF) m/z Calcd. for $C_{26}H_{31}Cl_2N_2O_5^+$ (M+H)⁺: 521.1610; found: 521.1613.



Methyl (E)-4-(1-(((2,4-dichloro-6-hydroxybenzylidene)amino)oxy)-6-morpholino-6-oxohexan-2-yl)benzoate (2v)

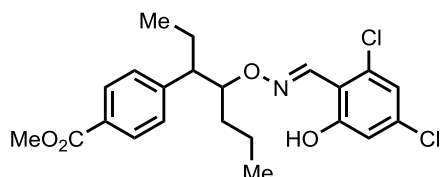
Following General procedure D, the product is obtained as a pale-yellow oil, 62% yield. ¹H NMR (600 MHz, Chloroform-*d*) δ 10.49 (s, 1H), 8.55 (s, 1H), 8.00 (d, J = 8.3 Hz, 2H), 7.30 (d, J = 8.4 Hz, 2H), 6.95 (d, J = 2.0 Hz, 1H), 6.89 (dd, J = 2.0, 0.6 Hz, 1H), 4.38 – 4.29 (m, 2H), 3.90 (s, 3H), 3.61 (d, J = 14.9 Hz, 6H), 3.45 – 3.26 (m, 2H), 3.13 (dtd, J = 9.7, 6.9, 4.9 Hz, 1H), 2.32 – 2.22 (m, 2H), 1.89 (ddt, J = 13.4, 10.7, 5.4 Hz, 1H), 1.76 – 1.69 (m, 1H), 1.62 – 1.49 (m, 2H); ¹³C NMR (151 MHz, Chloroform-*d*) δ 170.54, 166.40, 158.71, 148.11, 146.38, 136.35, 134.30, 129.54,

128.48, 127.52, 120.51, 115.74, 112.34, 78.49, 66.43, 66.09, 51.63, 45.42, 44.97, 41.42, 32.31, 31.36, 22.27; HRMS (ESI-TOF) m/z Calcd. for $C_{25}H_{29}Cl_2N_2O_6^+$ (M+H)⁺: 523.1403; found: 523.1404.



Methyl (E)-4-(3-(((2,4-dichloro-6-hydroxybenzylidene)amino)oxy)-4-methoxy-4-oxobutan-2-yl)benzoate (2w)

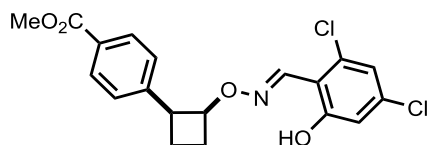
Following General procedure D, the product is obtained as a pale-yellow oil, 74% yield (dr = 4:1). ¹H NMR (600 MHz, Chloroform-*d*) δ 10.10 (s, 1H), 8.65 (s, 1H), 8.02 (d, J = 8.4 Hz, 2H), 7.38 (d, J = 8.2 Hz, 2H), 6.96 (d, J = 2.0 Hz, 1H), 6.90 (d, J = 1.6 Hz, 1H), 4.87 (d, J = 7.5 Hz, 1H), 3.92 (s, 3H), 3.76 (s, 2H), 3.64 (s, 1H), 3.48 (m, 1H), 1.43 (d, J = 7.2 Hz, 3H); ¹³C NMR (151 MHz, Chloroform-*d*) δ 170.43, 166.81, 159.12, 149.96, 146.34, 137.30, 135.11, 129.81, 129.08, 127.86, 121.06, 116.24, 112.40, 86.53, 52.23, 52.05, 41.39, 17.56; HRMS (ESI-TOF) m/z Calcd. for $C_{20}H_{20}Cl_2NO_6^+$ (M+H)⁺: 440.0662; found: 440.0655.



Methyl (E)-4-(4-(((2,4-dichloro-6-hydroxybenzylidene)amino)oxy)heptan-3-yl)benzoate (2x)

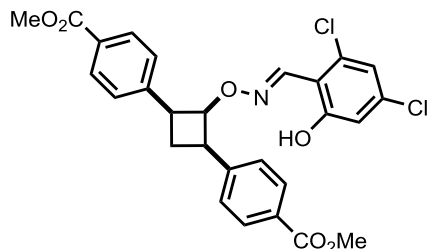
Following General procedure D, the product is obtained as a pale-yellow oil, 63% yield (dr = 5:1). ¹H NMR (600 MHz, Chloroform-*d*) δ 10.73 (s, 1H), 8.58 (s, 1H), 8.00 (d, J = 8.2 Hz, 2H), 7.33 (d, J = 8.3 Hz, 2H), 7.00 – 6.97 (m, 1H), 6.95 – 6.91 (m, 1H), 4.42 (dt, J = 9.2, 4.4 Hz, 1H), 3.92 (s, 3H), 2.88 (dt, J = 10.2, 4.9 Hz, 1H), 2.02 – 1.71 (m, 2H), 1.60 – 1.36 (m, 4H), 0.90 (t, J = 7.1 Hz, 3H), 0.82 (t, J = 7.4 Hz, 3H); ¹³C NMR (151 MHz, Chloroform-*d*) δ 167.02, 159.11, 147.49, 146.54, 136.44, 134.53, 129.50, 129.04, 128.48, 120.85, 116.07, 113.12, 87.85, 51.98, 51.59, 33.80,

24.49, 18.93, 13.95, 12.13; HRMS (ESI-TOF) m/z Calcd. for $C_{22}H_{26}Cl_2NO_4^+$ (M+H)⁺: 438.1239; found: 438.1241.



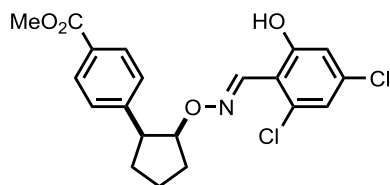
Methyl (E)-4-(2-(((2,4-dichloro-6-hydroxybenzylidene)amino)oxy)cyclobutyl)benzoate (2y)

Following General procedure D, the product is obtained as a colorless oil, 18% yield. ¹H NMR (600 MHz, Chloroform-*d*) δ 10.39 (s, 1H), 8.32 (d, $J = 0.5$ Hz, 1H), 7.95 (d, $J = 8.4$ Hz, 2H), 7.36 (d, $J = 8.1$ Hz, 2H), 6.89 (d, $J = 2.0$ Hz, 1H), 6.84 (d, $J = 2.0$ Hz, 1H), 5.14 – 5.07 (m, 1H), 3.97 (q, $J = 7.5$ Hz, 1H), 3.88 (s, 3H), 2.57 – 2.42 (m, 2H), 2.37 – 2.26 (m, 2H); ¹³C NMR (151 MHz, Chloroform-*d*) δ 167.01, 159.12, 149.09, 144.26, 136.70, 134.70, 129.39, 128.38, 128.30, 120.81, 116.10, 112.72, 80.58, 51.98, 45.56, 26.51, 21.63; HRMS (ESI-TOF) m/z Calcd. for $C_{19}H_{18}Cl_2NO_4^+$ (M+H)⁺: 394.0607; found: 394.0607.



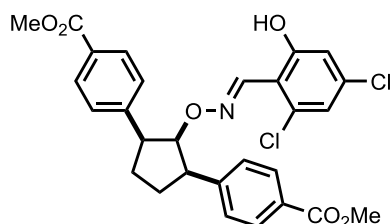
Dimethyl 4,4'-(2-(((2,4-dichloro-6-hydroxybenzylidene)amino)oxy)cyclobutane-1,3-diyl)(E)-dibenzoate (2y')

Following General procedure D, the product is obtained as a pale-yellow solid, 58% yield. ¹H NMR (600 MHz, Chloroform-*d*) δ 9.79 (s, 1H), 8.08 (s, 1H), 7.96 (d, $J = 8.3$ Hz, 4H), 7.38 (d, $J = 8.2$ Hz, 4H), 6.80 (d, $J = 2.0$ Hz, 1H), 6.72 (d, $J = 2.0$ Hz, 1H), 5.39 (td, $J = 5.6, 4.1$ Hz, 1H), 3.99 (ddd, $J = 11.0, 7.8, 5.9$ Hz, 2H), 3.88 (s, 6H), 3.17 (q, $J = 11.0$ Hz, 1H), 2.75 (dtd, $J = 11.6, 7.9, 3.9$ Hz, 1H); ¹³C NMR (151 MHz, Chloroform-*d*) δ 166.87, 158.87, 149.26, 143.11, 136.64, 134.49, 129.45, 128.50, 128.29, 120.64, 115.98, 112.32, 86.25, 52.00, 41.75, 29.42; HRMS (ESI-TOF) m/z Calcd. for $C_{27}H_{24}Cl_2NO_6^+$ (M+H)⁺: 528.0981; found: 528.0988.



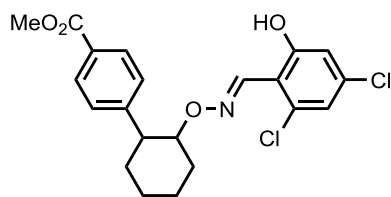
Methyl (E)-4-(2-(((2,4-dichloro-6-hydroxybenzylidene)amino)oxy)cyclopentyl)benzoate (2z)

Following General procedure D, the product is obtained as a colorless oil, 66% yield, dr > 20:1. ¹H NMR (600 MHz, Chloroform-*d*) δ 10.39 (s, 1H), 8.35 (s, 1H), 7.94 (d, *J* = 8.4 Hz, 2H), 7.38 (d, *J* = 8.3 Hz, 2H), 6.89 (d, *J* = 2.0 Hz, 1H), 6.82 (d, *J* = 2.0 Hz, 1H), 4.90 (td, *J* = 5.0, 2.2 Hz, 1H), 3.88 (s, 3H), 3.25 (ddd, *J* = 12.1, 7.8, 4.9 Hz, 1H), 2.25 – 2.06 (m, 4H), 2.01 (ddt, *J* = 17.8, 11.8, 6.0 Hz, 1H), 1.79 (ddd, *J* = 13.9, 11.9, 6.5 Hz, 1H); ¹³C NMR (151 MHz, Chloroform-*d*) δ 167.01, 159.07, 148.05, 145.06, 136.40, 134.48, 129.34, 128.78, 128.29, 120.73, 116.03, 112.92, 88.14, 51.96, 50.35, 31.72, 29.01, 22.22; HRMS (ESI-TOF) *m/z* Calcd. for C₂₀H₂₀Cl₂NO₄⁺ (M+H)⁺: 408.0764; found: 408.0758.



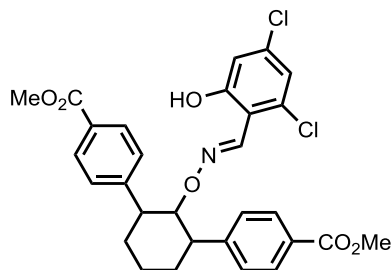
Dimethyl 4,4'-(2-(((2,4-dichloro-6-hydroxybenzylidene)amino)oxy)cyclopentane-1,3-diyl)(E)-dibenzoate (2z')

Following General procedure D, the product is obtained as a pale-yellow oil, 16% yield. ¹H NMR (600 MHz, Chloroform-*d*) δ 9.52 (s, 1H), 8.13 (s, 1H), 7.95 (d, *J* = 8.3 Hz, 4H), 7.42 (d, *J* = 8.3 Hz, 4H), 6.83 (d, *J* = 2.0 Hz, 1H), 6.68 (d, *J* = 2.0 Hz, 1H), 4.99 (t, *J* = 4.0 Hz, 1H), 3.88 (s, 6H), 3.64 (dq, *J* = 6.9, 4.0 Hz, 2H), 2.55 – 2.44 (m, 2H), 2.39 (ddt, *J* = 13.8, 8.1, 4.3 Hz, 2H); ¹³C NMR (151 MHz, Chloroform-*d*) δ 166.87, 158.74, 147.53, 144.84, 136.32, 134.28, 129.44, 128.76, 128.49, 120.54, 115.86, 112.43, 91.29, 51.96, 50.57, 28.23; HRMS (ESI-TOF) *m/z* Calcd. for C₂₈H₂₆Cl₂NO₆⁺ (M+H)⁺: 542.1132; found: 542.1128.



Methyl (E)-4-(2-(((2,4-dichloro-6-hydroxybenzylidene)amino)oxy)cyclohexyl)benzoate (2aa)

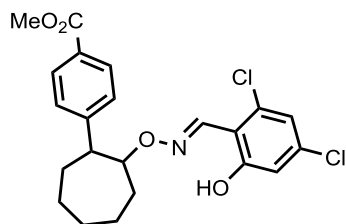
Following General procedure D, the product is obtained as a pale-yellow oil, 31% yield, dr = 1.5:1. ^1H NMR (600 MHz, Chloroform-*d*) δ 10.43 (s, 1H), 8.56 (s, 1H), 7.96 (d, $J = 8.4$ Hz, 2H), 7.37 (d, $J = 8.4$ Hz, 2H), 6.91 (d, $J = 2.0$ Hz, 1H), 6.87 – 6.80 (m, 1H), 4.65 – 4.28 (m, 1H), 3.88 (s, 3H), 2.95 – 2.78 (m, 1H), 2.38 – 2.21 (m, 1H), 2.16 – 1.92 (m, 2H), 1.87 – 1.76 (m, 1H), 1.71 – 1.41 (m, 4H); ^{13}C NMR (151 MHz, Chloroform-*d*) δ 167.02, 166.95, 159.09, 159.05, 148.76, 148.73, 147.87, 147.77, 136.34, 134.49, 134.42, 129.79, 129.47, 128.28, 128.00, 127.57, 120.74, 120.72, 116.03, 115.99, 113.01, 112.99, 85.95, 82.86, 51.94, 49.35, 46.85, 34.08, 32.13, 30.43, 25.88, 25.83, 25.66, 24.68, 19.57; HRMS (ESI-TOF) m/z Calcd. for $\text{C}_{21}\text{H}_{22}\text{Cl}_2\text{NO}_4^+$ ($\text{M}+\text{H}$) $^+$: 422.0920; found: 422.0924.



Dimethyl 4,4'-(2-(((2,4-dichloro-6-hydroxybenzylidene)amino)oxy)cyclohexane-1,3-diyl)(E)-dibenzoate (2aa')

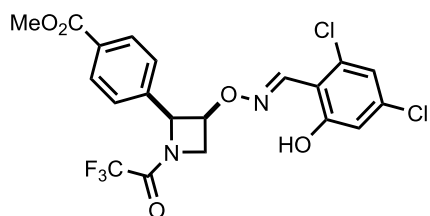
Following General procedure D, the product is obtained as a pale-yellow oil, 62% yield, dr = 2:1. ^1H NMR (600 MHz, Chloroform-*d*) δ 8.99 (s, 1H), 8.34 (s, 1H), 7.94 (d, $J = 8.4$ Hz, 4H), 7.37 (d, $J = 8.3$ Hz, 4H), 6.83 (d, $J = 2.0$ Hz, 1H), 6.59 (d, $J = 2.0$ Hz, 1H), 4.69 (s, 1H), 3.88 (s, 6H), 3.12 (d, $J = 12.8$ Hz, 2H), 2.32 – 2.13 (m, 3H), 1.99 – 1.86 (m, 2H), 1.68 (dtd, $J = 13.2, 9.5, 3.5$ Hz, 1H); ^{13}C NMR (151 MHz, Chloroform-*d*) δ 166.88, 158.70, 147.87, 146.75, 136.22, 134.20, 129.57, 128.50,

127.97, 120.43, 115.82, 112.34, 88.02, 51.93, 48.31, 25.70, 25.14; HRMS (ESI-TOF) m/z Calcd. for $C_{29}H_{28}Cl_2NO_6^+$ (M+H) $^+$: 556.1288; found: 556.1289.



Methyl (E)-4-(2-(((2,4-dichloro-6-hydroxybenzylidene)amino)oxy)cycloheptyl)benzoate (2ab)

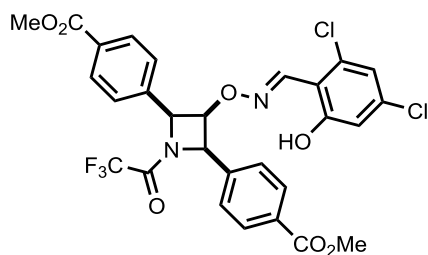
Following General procedure D, the product is obtained as a colorless oil, 76% yield, dr = 2:1. 1H NMR (600 MHz, Chloroform-*d*) δ 10.49 (s, 1H), 8.60 (s, 1H), 7.97 (d, J = 8.5 Hz, 2H), 7.37 (d, J = 8.2 Hz, 2H), 6.95 (d, J = 2.0 Hz, 1H), 6.86 (dd, J = 2.0, 0.5 Hz, 1H), 4.63 (dt, J = 5.9, 2.9 Hz, 1H), 3.90 (s, 3H), 3.08 (dt, J = 11.2, 2.8 Hz, 1H), 2.28 – 2.08 (m, 2H), 1.95 – 1.61 (m, 8H); ^{13}C NMR (151 MHz, Chloroform-*d*) δ 167.00, 159.11, 150.79, 147.83, 137.68, 134.47, 130.99, 129.54, 127.93, 120.75, 116.06, 113.02, 86.63, 49.47, 32.67, 29.19, 27.52, 27.35, 22.65; HRMS (ESI-TOF) m/z Calcd. for $C_{22}H_{24}Cl_2NO_4^+$ (M+H) $^+$: 436.1077; found: 436.1076.



Methyl (E)-4-(3-(((2,4-dichloro-6-hydroxybenzylidene)amino)oxy)-1-(2,2,2-trifluoroacetyl)azetidin-2-yl)benzoate (2ae)

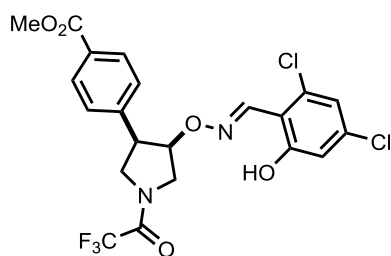
Following General procedure D, the product is obtained as a pale-yellow oil, 39% yield. 1H NMR (600 MHz, Chloroform-*d*) δ 9.77 (s, 1H), 8.21 (s, 1H), 8.02 (d, J = 8.4 Hz, 2H), 7.38 (d, J = 8.1 Hz, 2H), 6.93 (t, J = 2.4 Hz, 1H), 6.88 (dd, J = 3.8, 2.0 Hz, 1H), 5.82 (d, J = 6.8 Hz, 1H), 5.40 (tt, J = 7.1, 3.9 Hz, 1H), 4.88 (dd, J = 11.0, 7.3 Hz, 1H), 4.59 (dd, J = 11.2, 4.2 Hz, 1H), 3.91 (s, 3H); ^{13}C NMR (151 MHz, Chloroform-*d*) δ 158.98, 151.29, 137.86, 136.91, 135.14, 129.73, 127.38, 127.20,

121.20, 116.32, 73.91, 73.66, 69.45, 56.47, 53.96, 52.16; HRMS (ESI-TOF) m/z Calcd. for $C_{20}H_{16}Cl_2F_3N_2O_5^+$ (M+H) $^+$: 491.0388; found: 491.0388.



Dimethyl 4,4'-(3-(((2,4-dichloro-6-hydroxybenzylidene)amino)oxy)-1-(2,2,2-trifluoroacetyl)azetidine-2,4-diyl)(E)-dibenzoate (2ae')

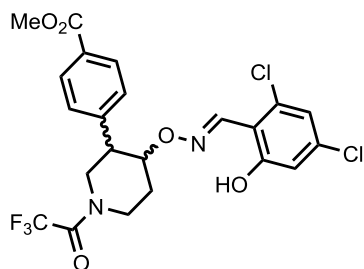
Following General procedure D, the product is obtained as a pale-yellow oil, 6% yield. ¹H NMR (600 MHz, Chloroform-*d*) δ 9.65 (s, 1H), 8.12 (d, *J* = 3.3 Hz, 4H), 7.85 (s, 1H), 7.50 (s, 4H), 6.87 (d, *J* = 2.0 Hz, 1H), 6.84 (dd, *J* = 2.0, 0.5 Hz, 1H), 6.09 (s, 2H), 5.61 (t, *J* = 7.2 Hz, 1H), 3.93 (s, 6H); ¹³C NMR (151 MHz, Chloroform-*d*) δ 158.89, 151.45, 137.87, 135.09, 130.28, 126.98, 121.16, 116.27, 111.63, 76.60, 52.23; HRMS (ESI-TOF) m/z Calcd. for $C_{28}H_{22}Cl_2F_3N_2O_7^+$ (M+H) $^+$: 625.0756; found: 625.0766.



Methyl (E)-4-(4-(((2,4-dichloro-6-hydroxybenzylidene)amino)oxy)-1-(2,2,2-trifluoroacetyl)pyrrolidin-3-yl)benzoate (2af)

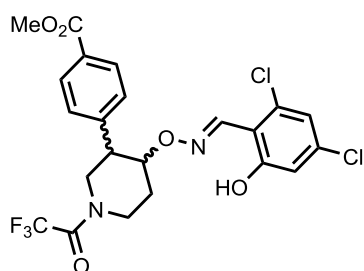
Following General procedure D, the product is obtained as a pale-yellow oil, 42% yield (rotamers). ¹H NMR (400 MHz, Chloroform-*d*) δ 9.90 (s, 1H), 8.54 (s, 1H), 8.06 (d, *J* = 8.3 Hz, 2H), 7.42 (d, *J* = 8.6 Hz, 2H), 6.97 (dd, *J* = 3.1, 2.0 Hz, 1H), 6.89 (ddd, *J* = 2.6, 2.0, 0.5 Hz, 1H), 5.14 (dt, *J* = 14.3, 3.9 Hz, 1H), 4.34 – 3.96 (m, 4H), 3.94 (s, 3H), 3.84 – 3.70 (m, 1H); ¹³C NMR (151 MHz, Chloroform-*d*) δ 166.52, 159.08, 150.48, 150.31, 139.23, 138.90, 137.60, 137.51, 135.14, 135.01, 129.95, 128.48,

121.17, 116.31, 112.21, 84.32, 82.02, 53.09, 52.22, 49.38, 48.37, 45.80; HRMS (ESI-TOF) m/z Calcd. for $C_{21}H_{18}Cl_2F_3N_2O_5^+$ ($M+H$) $^+$: 505.0545; found: 505.0547.



Methyl (E)-4-(4-(((2,4-dichloro-6-hydroxybenzylidene)amino)oxy)-1-(2,2,2-trifluoroacetyl)piperidin-3-yl)benzoate (2ag)

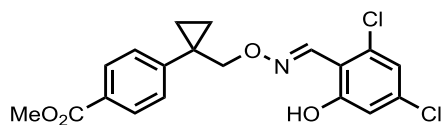
Following General procedure D, the product is obtained as a pale-yellow oil, 41% yield (major diastereomer). 1H NMR (600 MHz, Chloroform-*d*) δ 10.05 (s, 1H), 8.66 (s, 1H), 8.04 (t, $J = 8.0$ Hz, 2H), 7.38 (d, $J = 8.3$ Hz, 2H), 6.98 (t, $J = 2.0$ Hz, 1H), 6.87 (t, $J = 2.3$ Hz, 1H), 4.78 (dd, $J = 5.4, 2.6$ Hz, 1H), 4.71 – 4.55 (m, 1H), 4.03 (m, 1H), 3.93 (s, 3H), 3.65 – 3.15 (m, 3H), 2.42 – 2.33 (m, 1H), 2.06 – 1.97 (m, 1H); ^{13}C NMR (151 MHz, Chloroform-*d*) δ 166.65, 159.06, 155.53, 149.35, 143.41, 143.10, 137.25, 134.83, 129.98, 129.87, 127.96, 121.10, 116.25, 112.41, 79.80, 52.13, 46.37, 45.18, 42.53, 40.48, 30.37, 29.29; HRMS (ESI-TOF) m/z Calcd. for $C_{22}H_{20}Cl_2F_3N_2O_5^+$ ($M+H$) $^+$: 519.0696; found: 519.0698.



Methyl (E)-4-(4-(((2,4-dichloro-6-hydroxybenzylidene)amino)oxy)-1-(2,2,2-trifluoroacetyl)piperidin-3-yl)benzoate (2ag')

Following General procedure D, the product is obtained as a pale-yellow oil, 20% yield (minor diastereomer, rotamers). 1H NMR (600 MHz, Chloroform-*d*) δ 10.14 (d, $J = 11.0$ Hz, 1H), 8.49 (d, $J = 13.8$ Hz, 1H), 8.06 (t, $J = 8.1$ Hz, 2H), 7.37 (t, $J = 8.2$ Hz, 2H), 6.95 (dd, $J = 3.7, 2.0$ Hz, 1H), 6.89 (t, $J = 2.0$ Hz, 1H), 4.81 – 4.62 (m, 2H), 4.16 (m, 1H), 3.93 (s, 3H), 3.45 – 3.31 (m, 1H), 3.07 (dd, $J = 24.7, 10.1$ Hz, 2H), 2.50

– 2.42 (m, 1H), 1.90 – 1.80 (m, 1H); ^{13}C NMR (151 MHz, Chloroform-*d*) δ 166.59, 159.03, 155.53, 149.34, 143.01, 142.74, 137.11, 130.36, 130.22, 129.67, 127.75, 127.68, 121.08, 82.77, 52.15, 48.51, 47.41, 47.01, 42.13, 31.16, 30.51; HRMS (ESI-TOF) m/z Calcd. for $\text{C}_{22}\text{H}_{20}\text{Cl}_2\text{F}_3\text{N}_2\text{O}_5^+$ (M+H) $^+$: 519.0696; found: 519.0699 .



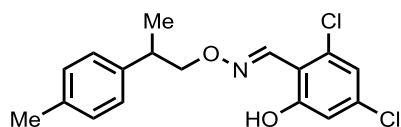
Methyl (E)-4-(1-(((2,4-dichloro-6-hydroxybenzylidene)amino)oxy)methyl)cyclopropylbenzoate (2ah)

Following General procedure D, the product is obtained as a colorless oil, 32% yield. ^1H NMR (600 MHz, Chloroform-*d*) δ 10.54 (s, 1H), 8.60 (s, 1H), 7.97 (d, $J = 8.7$ Hz, 2H), 7.39 (d, $J = 8.7$ Hz, 2H), 6.95 (d, $J = 2.0$ Hz, 1H), 6.89 (dd, $J = 2.0, 0.6$ Hz, 1H), 4.32 (s, 2H), 3.90 (s, 3H), 0.91 – 0.82 (m, 4H); ^{13}C NMR (151 MHz, Chloroform-*d*) δ 166.91, 159.21, 148.49, 147.79, 136.73, 134.76, 129.69, 128.38, 128.29, 120.93, 116.17, 112.89, 82.15, 52.04, 29.70, 24.96, 12.83; HRMS (ESI-TOF) m/z Calcd. for $\text{C}_{19}\text{H}_{18}\text{Cl}_2\text{NO}_4^+$ (M+H) $^+$: 394.0607; found: 394.0602.

C(sp³)-H arylation of DG9-tethered 1-propanol

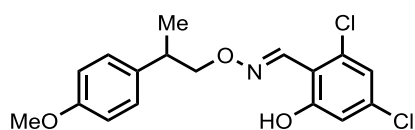
General procedure E:

To a 8-mL vial were added **DG9**-tethered 1-propanol (0.1 mmol), aryl iodide (0.3 mmol), palladium acetate (2.3 mg, 0.01 mmol), 3-nitro-5-trifluoromethyl-2-pyridone (8.1 mg, 0.04 mmol) and silver trifluoroacetate (33 mg, 0.15 mmol). the mixture was dissolved with 1,1,1,3,3,3-hexafluoro-2-propanol (HFIP, 1.0 mL). The vial was sealed and stirred at 100 °C for 12 hours unless otherwise stated. The reaction was cooled to room temperature and diluted with 5 mL of EtOAc and filtered through a pad of Celite. The filtrate was concentrated and purified by preparative thin-layer chromatography.



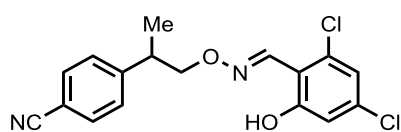
(E)-2,4-dichloro-6-hydroxybenzaldehyde O-(2-(p-tolyl)propyl) oxime (3a)

Following General procedure E, the product is obtained as a colorless oil, 88% yield. ^1H NMR (600 MHz, Chloroform-*d*) δ 10.65 (s, 1H), 8.62 (s, 1H), 7.15 (s, 4H), 6.96 (d, $J = 2.0$ Hz, 1H), 6.91 (d, $J = 2.0$ Hz, 1H), 4.31 (dd, $J = 10.5, 6.9$ Hz, 1H), 4.22 (dd, $J = 10.5, 7.5$ Hz, 1H), 3.19 (m, 1H), 2.34 (s, 3H), 1.34 (d, $J = 7.0$ Hz, 3H); ^{13}C NMR (151 MHz, Chloroform-*d*) δ 159.21, 148.23, 139.96, 136.57, 136.30, 134.67, 129.27, 127.15, 120.87, 116.15, 112.99, 80.61, 38.82, 21.02, 18.12; HRMS (ESI-TOF) m/z Calcd. for $\text{C}_{17}\text{H}_{18}\text{Cl}_2\text{NO}_2^+$ ($\text{M}+\text{H}$) $^+$: 338.0709; found: 338.0714.



(E)-2,4-dichloro-6-hydroxybenzaldehyde O-(2-(4-methoxyphenyl)propyl) oxime (3b)

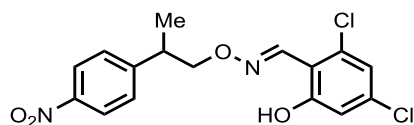
Following General procedure E, the product is obtained as a colorless oil, 66% yield. ^1H NMR (600 MHz, Chloroform-*d*) δ 10.64 (s, 1H), 8.61 (s, 1H), 7.17 (d, $J = 8.5$ Hz, 2H), 6.96 (d, $J = 2.0$ Hz, 1H), 6.91 (d, $J = 2.4$ Hz, 1H), 6.88 (d, $J = 8.7$ Hz, 2H), 4.29 (dd, $J = 10.5, 6.9$ Hz, 1H), 4.20 (dd, $J = 10.5, 7.5$ Hz, 1H), 3.80 (s, 3H), 3.17 (m, 1H), 1.33 (d, $J = 7.0$ Hz, 3H); ^{13}C NMR (151 MHz, Chloroform-*d*) δ 159.20, 158.36, 148.22, 136.58, 135.02, 134.67, 128.20, 120.87, 116.15, 113.98, 112.98, 80.70, 55.25, 38.40, 18.16; HRMS (ESI-TOF) m/z Calcd. for $\text{C}_{17}\text{H}_{18}\text{Cl}_2\text{NO}_3^+$ ($\text{M}+\text{H}$) $^+$: 354.0658; found: 354.0659.



(E)-4-(1-(((2,4-dichloro-6-hydroxybenzylidene)amino)oxy)propan-2-yl)benzonitrile (3c)

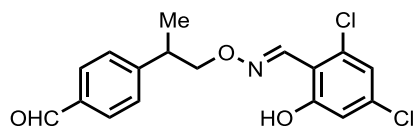
Following General procedure E, the product is obtained as a colorless oil, 68% yield. ^1H NMR (600 MHz, Chloroform-*d*) δ 10.43 (s, 1H), 8.58 (s, 1H), 7.63 (d, $J = 8.4$ Hz, 2H), 7.36 (d, $J = 8.1$ Hz, 2H), 6.96 (d, $J = 2.0$ Hz, 1H), 6.93 – 6.88 (m, 1H), 4.35 – 4.24 (m, 2H), 3.29 (m, 1H), 1.36 (d, $J = 7.1$ Hz, 3H); ^{13}C NMR (151 MHz, Chloroform-*d*) δ 159.14, 148.70, 148.67, 136.92, 134.79, 132.43, 128.19, 121.01,

118.81, 116.22, 112.70, 110.71, 79.55, 39.51, 17.72; HRMS (ESI-TOF) m/z Calcd. for $C_{17}H_{15}Cl_2N_2O_2^+$ ($M+H$) $^+$: 349.0505; found: 349.0513.



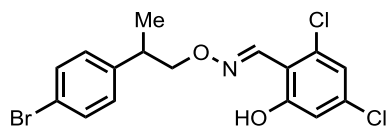
(E)-2,4-dichloro-6-hydroxybenzaldehyde O-(2-(4-nitrophenyl)propyl) oxime (3d)

Following General procedure E, the product is obtained as a pale-yellow oil, 57% yield. 1H NMR (600 MHz, Chloroform-*d*) δ 10.43 (s, 1H), 8.58 (s, 1H), 8.20 (d, J = 8.8 Hz, 2H), 7.42 (d, J = 8.6 Hz, 2H), 6.96 (d, J = 2.0 Hz, 1H), 6.93 – 6.88 (m, 1H), 4.38 – 4.27 (m, 2H), 3.35 (m, 1H), 1.39 (d, J = 7.1 Hz, 3H); ^{13}C NMR (151 MHz, Chloroform-*d*) δ 159.15, 150.81, 148.78, 146.91, 136.97, 134.82, 128.24, 123.87, 121.04, 116.23, 112.69, 79.51, 39.36, 17.82; HRMS (ESI-TOF) m/z Calcd. for $C_{16}H_{15}Cl_2N_2O_4^+$ ($M+H$) $^+$: 369.0403; found: 369.0402.



(E)-4-(1-(((2,4-dichloro-6-hydroxybenzylidene)amino)oxy)propan-2-yl)benzaldehyde (3e)

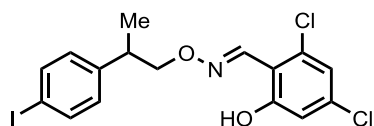
Following General procedure E, the product is obtained as a colorless oil, 84% yield. 1H NMR (600 MHz, Chloroform-*d*) δ 10.49 (s, 1H), 9.99 (s, 1H), 8.58 (s, 1H), 7.85 (d, J = 8.2 Hz, 2H), 7.42 (d, J = 8.1 Hz, 2H), 6.95 (d, J = 2.0 Hz, 1H), 6.90 (d, J = 2.0 Hz, 1H), 4.40 – 4.26 (m, 2H), 3.32 (m, 1H), 1.38 (d, J = 7.0 Hz, 3H); ^{13}C NMR (151 MHz, Chloroform-*d*) δ 191.81, 159.15, 150.31, 148.58, 136.82, 135.22, 134.75, 130.10, 128.04, 120.96, 116.19, 112.77, 79.76, 39.58, 17.83; HRMS (ESI-TOF) m/z Calcd. for $C_{17}H_{16}Cl_2NO_3^+$ ($M+H$) $^+$: 352.0502; found: 352.0508.



(E)-2,4-dichloro-6-hydroxybenzaldehyde O-(2-(4-bromophenyl)propyl) oxime (3f)

Following General procedure E, the product is obtained as a colorless oil, 80% yield.

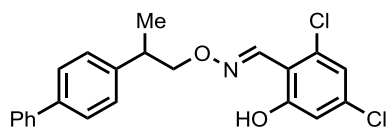
^1H NMR (600 MHz, Chloroform-*d*) δ 10.55 (s, 1H), 8.60 (s, 1H), 7.45 (d, $J = 8.4$ Hz, 2H), 7.13 (d, $J = 8.3$ Hz, 2H), 6.96 (d, $J = 2.0$ Hz, 1H), 6.91 (dd, $J = 2.0, 0.5$ Hz, 1H), 4.32 – 4.18 (m, 2H), 3.19 (m, 1H), 1.33 (d, $J = 7.0$ Hz, 3H); ^{13}C NMR (151 MHz, Chloroform-*d*) δ 159.18, 148.46, 142.03, 136.74, 134.73, 131.66, 129.05, 120.94, 120.51, 116.19, 112.86, 80.07, 38.79, 17.93; HRMS (ESI-TOF) m/z Calcd. for $\text{C}_{16}\text{H}_{15}\text{BrCl}_2\text{NO}_2^+$ ($\text{M}+\text{H}$) $^+$: 401.9658; found: 401.9661.



(*E*)-2,4-dichloro-6-hydroxybenzaldehyde O-(2-(4-iodophenyl)propyl) oxime (3g)

Following General procedure E, the product is obtained as a colorless oil, 72% yield.

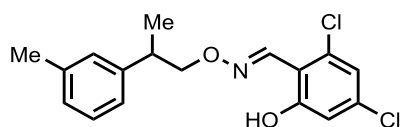
^1H NMR (600 MHz, Chloroform-*d*) δ 10.55 (s, 1H), 8.59 (s, 1H), 7.65 (d, $J = 8.4$ Hz, 2H), 7.00 (d, $J = 8.1$ Hz, 2H), 6.96 (d, $J = 2.0$ Hz, 1H), 6.91 (dd, $J = 2.0, 0.5$ Hz, 1H), 4.33 – 4.18 (m, 2H), 3.17 (m, 1H), 1.32 (d, $J = 7.0$ Hz, 3H); ^{13}C NMR (151 MHz, Chloroform-*d*) δ 159.19, 148.48, 142.74, 137.64, 136.74, 134.74, 129.38, 120.94, 116.19, 112.86, 91.94, 80.04, 38.89, 17.89; HRMS (ESI-TOF) m/z Calcd. for $\text{C}_{16}\text{H}_{15}\text{ICl}_2\text{NO}_2^+$ ($\text{M}+\text{H}$) $^+$: 449.9519; found: 449.9519.



(*E*)-2,4-dichloro-6-hydroxybenzaldehyde O-(2-([1,1'-biphenyl]-4-yl)propyl) oxime (3h)

Following General procedure E, the product is obtained as a colorless oil, 78% yield.

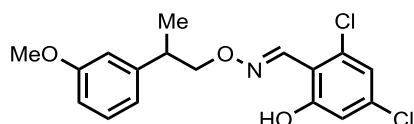
^1H NMR (600 MHz, Chloroform-*d*) δ 10.65 (s, 1H), 8.66 (s, 1H), 7.60 (ddt, $J = 10.4, 8.5, 1.7$ Hz, 4H), 7.51 – 7.44 (m, 2H), 7.41 – 7.33 (m, 3H), 6.99 (d, $J = 2.0$ Hz, 1H), 6.96 – 6.92 (m, 1H), 4.42-4.30 (m, 2H), 3.30 (m, 1H), 1.43 (d, $J = 7.0$ Hz, 3H); ^{13}C NMR (151 MHz, Chloroform-*d*) δ 159.21, 148.34, 142.07, 140.86, 139.74, 136.63, 134.70, 128.71, 127.72, 127.33, 127.15, 127.03, 120.89, 116.17, 112.95, 80.45, 38.97, 18.05; HRMS (ESI-TOF) m/z Calcd. for $\text{C}_{22}\text{H}_{20}\text{Cl}_2\text{NO}_2^+$ ($\text{M}+\text{H}$) $^+$: 400.0866; found: 400.0862.



(E)-2,4-dichloro-6-hydroxybenzaldehyde O-(2-(m-tolyl)propyl) oxime (3i)

Following General procedure E, the product is obtained as a colorless oil, 80% yield.

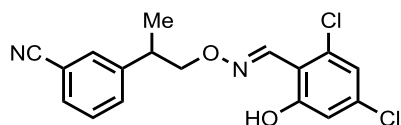
^1H NMR (600 MHz, Chloroform-*d*) δ 10.65 (s, 1H), 8.62 (s, 1H), 7.25 – 7.20 (m, 1H), 7.10 – 7.03 (m, 3H), 6.96 (d, J = 2.0 Hz, 1H), 6.91 (dd, J = 2.0, 0.5 Hz, 1H), 4.36 – 4.19 (m, 2H), 3.18 (m, 1H), 2.36 (s, 3H), 1.35 (d, J = 7.0 Hz, 3H); ^{13}C NMR (151 MHz, Chloroform-*d*) δ 159.21, 148.26, 142.95, 138.17, 136.59, 134.68, 128.47, 128.07, 127.51, 124.29, 120.87, 116.16, 112.99, 80.56, 21.47, 18.12; HRMS (ESI-TOF) m/z Calcd. for $\text{C}_{17}\text{H}_{18}\text{Cl}_2\text{NO}_2^+$ ($\text{M}+\text{H}$) $^+$: 338.0709; found: 338.0711.



(E)-2,4-dichloro-6-hydroxybenzaldehyde O-(2-(3-methoxyphenyl)propyl) oxime (3j)

Following General procedure E, the product is obtained as a colorless oil, 78% yield.

^1H NMR (600 MHz, Chloroform-*d*) δ 10.65 (s, 1H), 8.64 (s, 1H), 7.29 (d, J = 7.7 Hz, 1H), 6.98 (d, J = 2.0 Hz, 1H), 6.93 (d, J = 2.0 Hz, 1H), 6.87 (d, J = 7.6 Hz, 1H), 6.81 (dd, J = 9.2, 1.6 Hz, 2H), 4.35 (dd, J = 10.5, 6.9 Hz, 1H), 4.25 (dd, J = 10.5, 7.4 Hz, 1H), 3.84 (s, 3H), 3.22 (m, 1H), 1.37 (d, J = 7.0 Hz, 3H); ^{13}C NMR (151 MHz, Chloroform-*d*) δ 159.75, 159.21, 148.30, 144.71, 136.61, 134.69, 129.56, 120.88, 119.65, 116.16, 113.43, 112.95, 111.69, 80.41, 55.17, 39.31, 18.06; HRMS (ESI-TOF) m/z Calcd. for $\text{C}_{17}\text{H}_{18}\text{Cl}_2\text{NO}_3^+$ ($\text{M}+\text{H}$) $^+$: 354.0658; found: 354.0662.

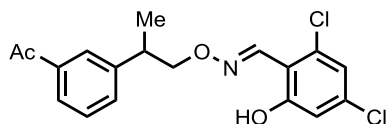


(E)-3-(1-(((2,4-dichloro-6-hydroxybenzylidene)amino)oxy)propan-2-yl)benzonitrile (3k)

Following General procedure E, the product is obtained as a colorless oil, 62% yield.

^1H NMR (600 MHz, Chloroform-*d*) δ 10.45 (s, 1H), 8.59 (s, 1H), 7.58 – 7.52 (m, 2H), 7.52 – 7.47 (m, 1H), 7.44 (t, J = 8.0 Hz, 1H), 6.96 (d, J = 2.1 Hz, 1H), 6.90 (dd, J =

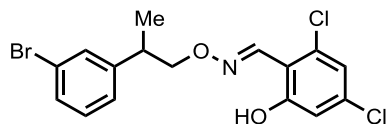
2.1, 0.5 Hz, 1H), 4.34 – 4.23 (m, 2H), 3.26 (m, 1H), 1.37 (d, $J = 7.0$ Hz, 3H); ^{13}C NMR (151 MHz, Chloroform-*d*) δ 159.16, 148.73, 144.59, 136.90, 134.81, 131.98, 131.01, 130.53, 129.38, 121.00, 118.83, 116.21, 112.72, 112.71, 79.65, 39.01, 29.69, 17.76; HRMS (ESI-TOF) m/z Calcd. for $\text{C}_{17}\text{H}_{15}\text{Cl}_2\text{N}_2\text{O}_2^+$ ($\text{M}+\text{H}$) $^+$: 349.0505; found: 349.0507.



(E)-2,4-dichloro-6-hydroxybenzaldehyde O-(2-(3-acetylphenyl)propyl) oxime (3l)

Following General procedure E, the product is obtained as a colorless oil, 81% yield.

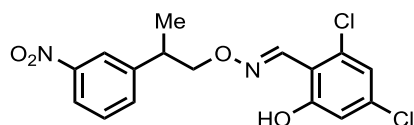
^1H NMR (600 MHz, Chloroform-*d*) δ 10.55 (s, 1H), 8.61 (s, 1H), 7.91 – 7.83 (m, 2H), 7.50 – 7.44 (m, 2H), 6.97 (d, $J = 2.0$ Hz, 1H), 6.92 (d, $J = 2.4$ Hz, 1H), 4.40 – 4.27 (m, 2H), 3.32 (m, 1H), 2.63 (s, 3H), 1.41 (d, $J = 7.0$ Hz, 3H); ^{13}C NMR (151 MHz, Chloroform-*d*) δ 198.09, 159.17, 148.48, 143.72, 136.73, 134.73, 132.19, 128.82, 127.03, 126.94, 120.92, 116.18, 112.83, 80.08, 39.25, 26.68, 17.99; HRMS (ESI-TOF) m/z Calcd. for $\text{C}_{18}\text{H}_{18}\text{Cl}_2\text{NO}_3^+$ ($\text{M}+\text{H}$) $^+$: 366.0658; found: 366.0663.



(E)-2,4-dichloro-6-hydroxybenzaldehyde O-(2-(3-bromophenyl)propyl) oxime (3m)

Following General procedure E, the product is obtained as a colorless oil, 86% yield.

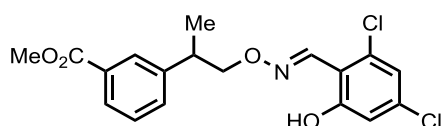
^1H NMR (600 MHz, Chloroform-*d*) δ 10.55 (s, 1H), 8.61 (s, 1H), 7.51 – 7.34 (m, 2H), 7.24 – 7.11 (m, 2H), 6.96 (d, $J = 2.0$ Hz, 1H), 6.91 (d, $J = 2.0$ Hz, 1H), 4.31-4.22 (m, 2H), 3.19 (m, 1H), 1.34 (d, $J = 7.0$ Hz, 3H); ^{13}C NMR (151 MHz, Chloroform-*d*) δ 159.19, 148.53, 145.42, 136.74, 134.76, 130.42, 130.15, 129.88, 126.04, 122.68, 120.93, 116.19, 112.86, 80.02, 39.08, 17.93; HRMS (ESI-TOF) m/z Calcd. for $\text{C}_{16}\text{H}_{15}\text{BrCl}_2\text{NO}_2^+$ ($\text{M}+\text{H}$) $^+$: 401.9658; found: 401.9661.



(E)-2,4-dichloro-6-hydroxybenzaldehyde O-(2-(3-nitrophenyl)propyl) oxime (3n)

Following General procedure E, the product is obtained as a colorless oil, 88% yield.

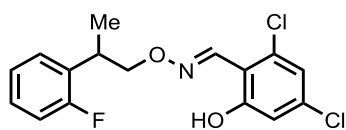
^1H NMR (600 MHz, Chloroform-*d*) δ 10.46 (s, 1H), 8.61 (s, 1H), 8.20 – 8.09 (m, 2H), 7.61 (d, $J = 7.7$ Hz, 1H), 7.53 (t, $J = 7.9$ Hz, 1H), 6.98 (d, $J = 2.0$ Hz, 1H), 6.92 (dd, $J = 2.0, 0.5$ Hz, 1H), 4.41 – 4.28 (m, 2H), 3.38 (m, 1H), 1.43 (d, $J = 7.1$ Hz, 3H); ^{13}C NMR (151 MHz, Chloroform-*d*) δ 159.15, 148.78, 148.49, 145.21, 136.91, 134.81, 133.76, 129.49, 122.25, 121.93, 121.00, 116.21, 112.70, 79.62, 39.11, 17.82; HRMS (ESI-TOF) m/z Calcd. for $\text{C}_{16}\text{H}_{15}\text{Cl}_2\text{N}_2\text{O}_4^+$ ($\text{M}+\text{H}$) $^+$: 369.0403; found: 369.0405.



Methyl (E)-3-(1-(((2,4-dichloro-6-hydroxybenzylidene)amino)oxy)propan-2-yl)benzoate (3o)

Following General procedure E, the product is obtained as a colorless oil, 78% yield.

^1H NMR (600 MHz, Chloroform-*d*) δ 10.57 (s, 1H), 8.62 (s, 1H), 8.00 – 7.89 (m, 2H), 7.50 – 7.38 (m, 2H), 6.97 (d, $J = 2.0$ Hz, 1H), 6.93 (dd, $J = 2.0, 0.6$ Hz, 1H), 4.38-4.28 (m, 2H), 3.94 (s, 3H), 3.31 (m, 1H), 1.40 (d, $J = 7.0$ Hz, 3H); ^{13}C NMR (151 MHz, Chloroform-*d*) δ 167.03, 159.19, 148.47, 143.43, 136.70, 134.72, 132.06, 130.47, 128.64, 128.37, 128.04, 120.90, 116.18, 112.87, 80.10, 52.12, 39.15, 17.98; HRMS (ESI-TOF) m/z Calcd. for $\text{C}_{18}\text{H}_{18}\text{Cl}_2\text{NO}_4^+$ ($\text{M}+\text{H}$) $^+$: 382.0607; found: 382.0608.

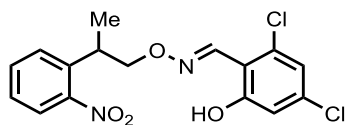


(E)-2,4-dichloro-6-hydroxybenzaldehyde O-(2-(2-fluorophenyl)propyl) oxime (3p)

Following General procedure E, the product is obtained as a colorless oil, 72% yield.

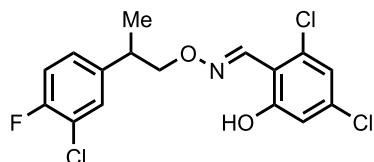
^1H NMR (600 MHz, Chloroform-*d*) δ 10.61 (s, 1H), 8.63 (s, 1H), 7.14 (td, $J = 7.5, 1.3$ Hz, 1H), 7.07 (dd, $J = 10.6, 8.1$ Hz, 1H), 6.98 (d, $J = 2.1$ Hz, 1H), 6.93 (dd, $J = 2.0, 0.5$ Hz, 1H), 4.41 (dd, $J = 10.6, 6.7$ Hz, 1H), 4.29 (dd, $J = 10.6, 7.2$ Hz, 1H), 3.58 (m, 1H), 1.40 (d, $J = 7.0$ Hz, 3H); ^{13}C NMR (151 MHz, Chloroform-*d*) δ 160.93 (d, $J = 244.6$ Hz), 159.22, 148.34, 136.62, 134.70, 129.66 (d, $J = 15.1$ Hz), 128.37 (d, $J = 4.5$

Hz), 128.19 (d, $J = 7.6$ Hz), 124.24 (d, $J = 3.0$ Hz), 120.86, 116.19, 115.57 (d, $J = 22.7$ Hz), 112.94, 79.19 (d, $J = 1.5$ Hz), 32.89 (d, $J = 1.5$ Hz), 16.81 (d, $J = 0.8$ Hz); HRMS (ESI-TOF) m/z Calcd. for $C_{16}H_{15}Cl_2FNO_2^+$ ($M+H$) $^+$: 342.0458; found: 342.0461.



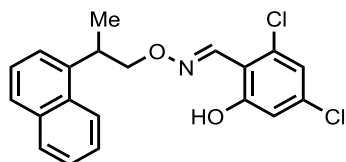
(E)-2,4-dichloro-6-hydroxybenzaldehyde O-(2-(2-nitrophenyl)propyl) oxime (3q)

Following General procedure E, the product is obtained as a pale-yellow oil, 62% yield. 1H NMR (600 MHz, Chloroform- d) δ 10.41 (s, 1H), 8.58 (s, 1H), 7.77 (dd, $J = 8.1, 1.3$ Hz, 1H), 7.62 – 7.56 (m, 1H), 7.50 (dd, $J = 7.9, 1.2$ Hz, 1H), 7.42 – 7.34 (m, 1H), 6.95 (d, $J = 2.0$ Hz, 1H), 6.91 – 6.89 (m, 1H), 4.38 (dd, $J = 10.7, 6.9$ Hz, 1H), 4.28 (dd, $J = 10.7, 6.7$ Hz, 1H), 3.85 (h, $J = 6.9$ Hz, 1H), 1.41 (d, $J = 7.0$ Hz, 3H); ^{13}C NMR (151 MHz, Chloroform- d) δ 159.21, 148.67, 137.02, 136.77, 134.79, 132.69, 128.24, 127.50, 124.31, 120.90, 116.24, 112.76, 79.44, 33.49, 17.53; HRMS (ESI-TOF) m/z Calcd. for $C_{16}H_{15}Cl_2N_2O_4^+$ ($M+H$) $^+$: 369.0403; found: 369.0410.



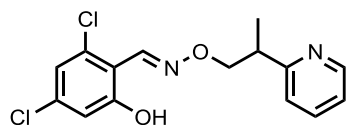
(E)-2,4-dichloro-6-hydroxybenzaldehyde O-(2-(3-chloro-4-fluorophenyl)propyl) oxime (3r)

Following General procedure E, the product is obtained as a colorless oil, 86% yield. 1H NMR (600 MHz, Chloroform- d) δ 10.51 (s, 1H), 8.60 (s, 1H), 7.28 (dd, $J = 7.3, 1.5$ Hz, 1H), 7.14 – 7.05 (m, 2H), 6.96 (d, $J = 2.0$ Hz, 1H), 6.91 (dd, $J = 2.0, 0.5$ Hz, 1H), 4.30 – 4.18 (m, 2H), 3.18 (m, 1H), 1.33 (d, $J = 7.1$ Hz, 3H); ^{13}C NMR (151 MHz, Chloroform- d) δ 159.18, 157.76, 156.12, 148.60, 140.09 (d, $J = 4.5$ Hz), 136.82, 134.78, 129.35, 126.99 (d, $J = 6.8$ Hz), 120.97, 116.59 (d, $J = 20.8$ Hz), 116.21, 112.80, 79.97, 38.50, 18.00; HRMS (ESI-TOF) m/z Calcd. for $C_{16}H_{14}Cl_3FNO_2^+$ ($M+H$) $^+$: 376.0069; found: 376.0073.



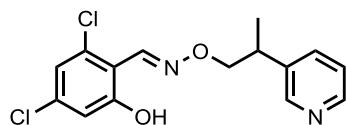
(E)-2,4-dichloro-6-hydroxybenzaldehyde O-(2-(naphthalen-1-yl)propyl) oxime (3s)

Following General procedure E, the product is obtained as a colorless oil, 74% yield. ^1H NMR (600 MHz, Chloroform-*d*) δ 10.66 (s, 1H), 8.61 (s, 1H), 7.87 – 7.78 (m, 3H), 7.69 (s, 1H), 7.51 – 7.42 (m, 2H), 7.40 (dd, $J = 8.4, 1.8$ Hz, 1H), 6.95 (d, $J = 2.0$ Hz, 1H), 6.91 (dd, $J = 2.0, 0.6$ Hz, 1H), 4.44 (dd, $J = 10.6, 7.0$ Hz, 1H), 4.33 (dd, $J = 10.5, 7.3$ Hz, 1H), 3.40 (m, 1H), 1.45 (d, $J = 7.0$ Hz, 3H); ^{13}C NMR (151 MHz, Chloroform-*d*) δ 159.20, 148.37, 140.44, 136.62, 134.70, 133.54, 132.46, 128.24, 127.61, 126.07, 125.77, 125.70, 125.54, 120.89, 116.16, 112.94, 80.38, 39.36, 18.13; HRMS (ESI-TOF) m/z Calcd. for $\text{C}_{20}\text{H}_{18}\text{Cl}_2\text{NO}_2^+$ ($\text{M}+\text{H}$) $^+$: 374.0709; found: 374.0713.



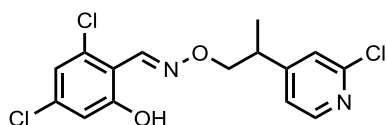
(E)-2,4-dichloro-6-hydroxybenzaldehyde O-(2-(pyridin-2-yl)propyl) oxime (3t)

Following General procedure E, the product is obtained as a pale-yellow oil, 38% yield. ^1H NMR (600 MHz, Chloroform-*d*) δ 10.54 (s, 1H), 8.58 (d, $J = 4.8$ Hz, 1H), 8.56 (s, 1H), 7.62 (td, $J = 7.7, 1.8$ Hz, 1H), 7.18 (d, $J = 7.8$ Hz, 1H), 7.17 – 7.12 (m, 1H), 6.93 (d, $J = 2.0$ Hz, 1H), 6.89 (d, $J = 2.0$ Hz, 1H), 4.56 (dd, $J = 10.6, 7.5$ Hz, 1H), 4.37 (dd, $J = 10.6, 6.5$ Hz, 1H), 3.37 (m, 1H), 1.37 (d, $J = 7.0$ Hz, 3H); ^{13}C NMR (151 MHz, Chloroform-*d*) δ 162.39, 159.18, 149.47, 148.20, 136.56, 136.50, 134.64, 122.49, 121.76, 120.82, 116.15, 112.93, 79.33, 41.32, 17.20; HRMS (ESI-TOF) m/z Calcd. for $\text{C}_{15}\text{H}_{15}\text{Cl}_2\text{N}_2\text{O}_2^+$ ($\text{M}+\text{H}$) $^+$: 325.0505; found: 325.0511.



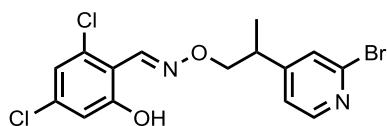
(E)-2,4-dichloro-6-hydroxybenzaldehyde O-(2-(pyridin-3-yl)propyl) oxime (3u)

Following General procedure E, the product is obtained as a pale-yellow oil, 34% yield. ^1H NMR (600 MHz, Chloroform-*d*) δ 10.49 (s, 1H), 8.59 (s, 1H), 8.51 (d, $J = 11.6$ Hz, 2H), 7.57 (dddd, $J = 7.9, 2.2, 1.6, 0.5$ Hz, 1H), 7.30 – 7.26 (m, 1H), 6.95 (d, $J = 2.0$ Hz, 1H), 6.90 (dd, $J = 2.0, 0.5$ Hz, 1H), 4.37 – 4.23 (m, 2H), 3.25 (m, 1H), 1.38 (d, $J = 7.1$ Hz, 3H); ^{13}C NMR (151 MHz, Chloroform-*d*) δ 159.16, 149.33, 148.63, 148.24, 138.36, 136.82, 134.78, 134.56, 123.52, 120.97, 116.19, 112.78, 79.75, 36.94, 17.69; HRMS (ESI-TOF) m/z Calcd. for $\text{C}_{15}\text{H}_{15}\text{Cl}_2\text{N}_2\text{O}_2^+$ ($\text{M}+\text{H}$) $^+$: 325.0505; found: 325.0507.



(*E*)-2,4-dichloro-6-hydroxybenzaldehyde O-(2-(2-chloropyridin-4-yl)propyl) oxime (3v)

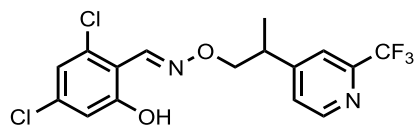
Following General procedure E, the product is obtained as a pale-yellow oil, 70% yield. ^1H NMR (600 MHz, Chloroform-*d*) δ 10.40 (s, 1H), 8.59 (s, 1H), 8.33 (s, 1H), 7.22 (s, 1H), 7.11 (dd, $J = 5.1, 1.4$ Hz, 1H), 6.96 (d, $J = 2.0$ Hz, 1H), 6.91 (d, $J = 2.0$ Hz, 1H), 4.38 – 4.22 (m, 2H), 3.21 (m, 1H), 1.35 (d, $J = 7.1$ Hz, 3H); ^{13}C NMR (151 MHz, Chloroform-*d*) δ 159.15, 155.53, 151.90, 149.80, 148.96, 137.01, 134.86, 123.23, 121.55, 121.04, 116.24, 112.63, 78.86, 38.66, 17.27; HRMS (ESI-TOF) m/z Calcd. for $\text{C}_{15}\text{H}_{14}\text{Cl}_3\text{N}_2\text{O}_2^+$ ($\text{M}+\text{H}$) $^+$: 359.0115; found: 359.0113.



(*E*)-2,4-dichloro-6-hydroxybenzaldehyde O-(2-(2-bromopyridin-4-yl)propyl) oxime (3w)

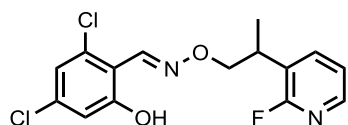
Following General procedure E, the product is obtained as a pale-yellow oil, 58% yield. ^1H NMR (600 MHz, Chloroform-*d*) δ 10.42 (s, 1H), 8.62 (s, 1H), 8.33 (d, $J = 5.4$ Hz, 1H), 7.40 (d, $J = 1.5$ Hz, 1H), 7.16 (dd, $J = 5.1, 1.2$ Hz, 1H), 6.98 (d, $J = 2.0$ Hz, 1H), 6.93 (dd, $J = 2.0, 0.4$ Hz, 1H), 4.38 – 4.25 (m, 2H), 3.22 (dt, $J = 13.9, 7.0$ Hz, 1H), 1.37 (d, $J = 7.1$ Hz, 3H); ^{13}C NMR (151 MHz, Chloroform-*d*) δ 159.15, 155.27, 150.22, 148.98, 142.64, 137.01, 134.87, 127.04, 121.94, 121.04, 116.24,

112.63, 78.84, 38.60, 17.27; HRMS (ESI-TOF) m/z Calcd. for $C_{15}H_{14}BrCl_2N_2O_2^+$ ($M+H$)⁺: 402.9610; found: 402.9610.



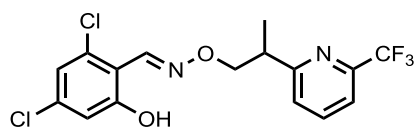
(E)-2,4-dichloro-6-hydroxybenzaldehyde O-(2-(2-(trifluoromethyl)pyridin-4-yl)propyl) oxime (3x)

Following General procedure E, the product is obtained as a pale-yellow oil, 71% yield. ¹H NMR (600 MHz, Chloroform-*d*) δ 10.37 (s, 1H), 8.67 (d, $J = 5.0$ Hz, 1H), 8.59 (s, 1H), 7.57 (s, 1H), 7.38 (d, $J = 5.0$ Hz, 1H), 6.96 (d, $J = 2.0$ Hz, 1H), 6.91 (dd, $J = 2.0, 0.5$ Hz, 1H), 4.33 (d, $J = 6.9$ Hz, 2H), 3.32 (m, 1H), 1.39 (d, $J = 7.1$ Hz, 3H); ¹³C NMR (151 MHz, Chloroform-*d*) δ 159.15, 154.22, 150.19, 149.06, 148.58 (q, $J = 34.4$ Hz), 137.08, 134.89, 125.42, 121.53 (q, $J = 274.8$ Hz), 121.07, 119.51 (q, $J = 2.7$ Hz), 116.25, 112.58, 78.81, 38.93, 17.26; HRMS (ESI-TOF) m/z Calcd. for $C_{16}H_{14}Cl_2F_3N_2O_2^+$ ($M+H$)⁺: 393.0379; found: 393.0387.



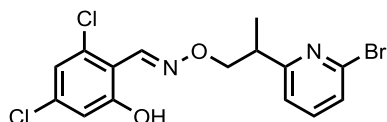
(E)-2,4-dichloro-6-hydroxybenzaldehyde O-(2-(2-fluoropyridin-3-yl)propyl) oxime (3y)

Following General procedure E, the product is obtained as a pale-yellow oil, 56% yield. ¹H NMR (600 MHz, Chloroform-*d*) δ 10.48 (s, 1H), 8.60 (s, 1H), 8.17 – 8.06 (m, 1H), 7.77 – 7.64 (m, 1H), 7.25 – 7.13 (m, 1H), 6.97 (d, $J = 2.0$ Hz, 1H), 6.93 (d, $J = 2.0$ Hz, 1H), 4.37 (ddd, $J = 47.9, 10.7, 6.8$ Hz, 2H), 3.50 (m, 1H), 1.40 (d, $J = 7.1$ Hz, 3H); ¹³C NMR (151 MHz, Chloroform-*d*) δ 161.68 (d, $J = 238.9$ Hz), 159.19, 148.68, 145.72 (d, $J = 1.5$ Hz), 139.10, 136.84, 134.78, 124.74 (d, $J = 29.1$ Hz), 121.64, 120.95, 116.22, 112.75, 78.36, 33.14, 16.43; HRMS (ESI-TOF) m/z Calcd. for $C_{15}H_{14}Cl_2FN_2O_2^+$ ($M+H$)⁺: 343.0411; found: 343.0417.



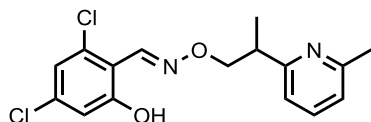
(E)-2,4-dichloro-6-hydroxybenzaldehyde O-(2-(6-(trifluoromethyl)pyridin-2-yl)propyl) oxime (3z)

Following General procedure E, the product is obtained as a pale-yellow oil, 88% yield. ^1H NMR (600 MHz, Chloroform-*d*) δ 10.47 (s, 1H), 8.56 (d, $J = 0.5$ Hz, 1H), 7.79 (td, $J = 7.8, 0.7$ Hz, 1H), 7.54 (dd, $J = 7.8, 0.9$ Hz, 1H), 7.38 (d, $J = 7.8$ Hz, 1H), 6.94 (d, $J = 2.0$ Hz, 1H), 6.89 (dd, $J = 2.1, 0.5$ Hz, 1H), 4.56 (dd, $J = 10.6, 7.6$ Hz, 1H), 4.40 (dd, $J = 10.6, 6.1$ Hz, 1H), 3.51 – 3.40 (m, 1H), 1.40 (d, $J = 7.0$ Hz, 3H); ^{13}C NMR (151 MHz, Chloroform-*d*) δ 163.36, 159.18, 148.40, 147.99 (q, $J = 33.2$ Hz), 137.64, 136.69, 134.69, 125.12, 121.50 (q, $J = 274.8$), 120.87, 118.36 (q, $J = 3.0$ Hz), 116.18, 112.82, 78.96, 41.22, 16.98; HRMS (ESI-TOF) m/z Calcd. for $\text{C}_{16}\text{H}_{14}\text{Cl}_2\text{F}_3\text{N}_2\text{O}_2^+$ ($\text{M}+\text{H}$) $^+$: 393.0379; found: 393.0383.



(E)-2,4-dichloro-6-hydroxybenzaldehyde O-(2-(6-bromopyridin-2-yl)propyl) oxime (3aa)

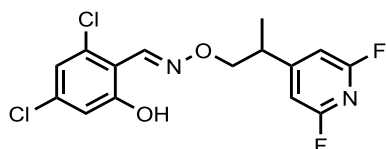
Following General procedure E, the product is obtained as a pale-yellow oil, 66% yield. ^1H NMR (600 MHz, Chloroform-*d*) δ 10.52 (s, 1H), 8.59 (s, 1H), 7.50 (d, $J = 7.7$ Hz, 1H), 7.36 (d, $J = 7.8$ Hz, 1H), 7.16 (d, $J = 7.5$ Hz, 1H), 6.96 (d, $J = 2.0$ Hz, 1H), 6.92 (d, $J = 2.0$ Hz, 1H), 4.55 (dd, $J = 10.6, 7.5$ Hz, 1H), 4.38 (dd, $J = 10.6, 6.2$ Hz, 1H), 3.41 – 3.31 (m, 1H), 1.38 (d, $J = 7.0$ Hz, 3H); ^{13}C NMR (151 MHz, Chloroform-*d*) δ 164.06, 159.18, 148.38, 141.94, 138.72, 136.66, 134.67, 126.16, 121.18, 120.85, 116.18, 112.84, 78.90, 41.12, 17.03; HRMS (ESI-TOF) m/z Calcd. for $\text{C}_{15}\text{H}_{14}\text{BrCl}_2\text{N}_2\text{O}_2^+$ ($\text{M}+\text{H}$) $^+$: 402.9610; found: 402.9611.



(E)-2,4-dichloro-6-hydroxybenzaldehyde O-(2-(6-methylpyridin-2-yl)propyl) oxime (3ab)

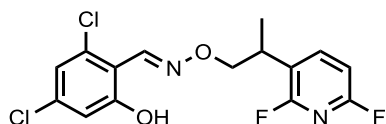
Following General procedure E, the product is obtained as a pale-yellow oil, 70% yield. ^1H NMR (600 MHz, Chloroform-*d*) δ 10.57 (s, 1H), 8.57 (s, 1H), 7.51 (t, $J =$

7.7 Hz, 1H), 6.99 (t, $J = 8.0$ Hz, 2H), 6.94 (d, $J = 2.0$ Hz, 1H), 6.89 (dd, $J = 2.0, 0.5$ Hz, 1H), 4.55 (dd, $J = 10.5, 7.2$ Hz, 1H), 4.36 (dd, $J = 10.5, 6.5$ Hz, 1H), 3.34 (m, 1H), 2.54 (s, 3H), 1.36 (d, $J = 7.0$ Hz, 3H); ^{13}C NMR (151 MHz, Chloroform-*d*) δ 161.70, 159.20, 158.08, 148.10, 136.70, 136.50, 134.62, 121.30, 120.80, 118.94, 116.15, 112.98, 79.49, 41.40, 24.49, 17.27; HRMS (ESI-TOF) m/z Calcd. for $\text{C}_{16}\text{H}_{17}\text{Cl}_2\text{N}_2\text{O}_2^+$ ($\text{M}+\text{H}$) $^+$: 339.0662; found: 339.0665.



(*E*)-2,4-dichloro-6-hydroxybenzaldehyde O-(2-(2,6-difluoropyridin-4-yl)propyl)oxime (3ac)

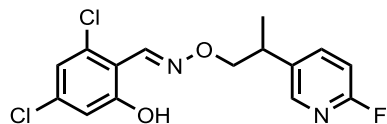
Following General procedure E, the product is obtained as a pale-yellow oil, 62% yield. ^1H NMR (600 MHz, Chloroform-*d*) δ 10.35 (s, 1H), 8.60 (s, 1H), 6.97 (d, $J = 2.0$ Hz, 1H), 6.91 (dd, $J = 2.0, 0.6$ Hz, 1H), 6.72 (s, 2H), 4.31 (d, $J = 6.8$ Hz, 2H), 3.30 (h, $J = 6.9$ Hz, 1H), 1.37 (d, $J = 7.1$ Hz, 3H); ^{13}C NMR (151 MHz, Chloroform-*d*) δ 162.93 (t, $J = 7.5$ Hz), 161.99 (dd, $J = 246.6, 16.0$ Hz), 159.15, 149.14, 137.13, 134.93, 121.10, 116.27, 112.57, 104.85 (dd, $J = 30.1, 12.1$ Hz), 78.62, 38.96 (t, $J = 2.7$ Hz), 17.26; HRMS (ESI-TOF) m/z Calcd. for $\text{C}_{15}\text{H}_{13}\text{Cl}_2\text{F}_2\text{N}_2\text{O}_2^+$ ($\text{M}+\text{H}$) $^+$: 361.0317; found: 361.0319.



(*E*)-2,4-dichloro-6-hydroxybenzaldehyde O-(2-(2,6-difluoropyridin-3-yl)propyl)oxime (3ad)

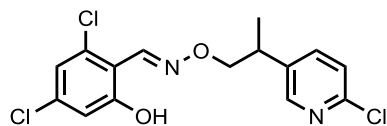
Following General procedure E, the product is obtained as a pale-yellow oil, 60% yield. ^1H NMR (600 MHz, Chloroform-*d*) δ 10.44 (s, 1H), 8.61 (s, 1H), 7.85 – 7.77 (m, 1H), 6.98 (d, $J = 2.0$ Hz, 1H), 6.93 (dd, $J = 2.0, 0.5$ Hz, 1H), 6.84 (dd, $J = 8.1, 2.9$ Hz, 1H), 4.40 – 4.29 (m, 2H), 3.50 (m, 1H), 1.39 (d, $J = 7.1$ Hz, 3H); ^{13}C NMR (151 MHz, Chloroform-*d*) δ 160.42 (dd, $J = 154.8, 14.0$ Hz), 159.17, 158.79 (dd, $J = 155.4, 14.3$ Hz), 148.81, 143.28 (dd, $J = 7.6, 5.7$ Hz), 136.94, 134.82, 121.18 (d, $J = 6.0$ Hz),

121.00, 116.24, 112.68, 106.22 (dd, $J = 34.7, 6.0$ Hz), 78.26, 32.44 (d, $J = 3.0$ Hz), 16.53; HRMS (ESI-TOF) m/z Calcd. for $C_{15}H_{13}Cl_2F_2N_2O_2^+$ ($M+H$) $^+$: 361.0317; found: 361.0318.



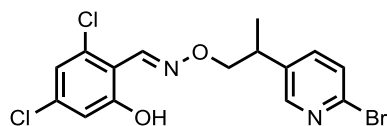
(E)-2,4-dichloro-6-hydroxybenzaldehyde O-(2-(6-fluoropyridin-3-yl)propyl)oxime (3ae)

Following General procedure E, the product is obtained as a pale-yellow oil, 61% yield. 1H NMR (600 MHz, Chloroform- d) δ 10.44 (s, 1H), 8.59 (s, 1H), 8.10 (d, $J = 2.5$ Hz, 1H), 7.68 (td, $J = 8.1, 2.6$ Hz, 1H), 6.96 (d, $J = 2.0$ Hz, 1H), 6.94 – 6.88 (m, 2H), 4.32 – 4.23 (m, 2H), 3.27 (m, 1H), 1.37 (d, $J = 7.1$ Hz, 3H); ^{13}C NMR (151 MHz, Chloroform- d) δ 162.70 (d, $J = 238.6$ Hz), 159.15, 146.64 (d, $J = 15.1$ Hz), 139.71, 136.92, 136.01, 134.82, 121.02, 116.21, 112.70, 109.54, 109.30, 79.64, 36.14, 17.78; HRMS (ESI-TOF) m/z Calcd. for $C_{15}H_{14}Cl_2FN_2O_2^+$ ($M+H$) $^+$: 343.0411; found: 343.0414.



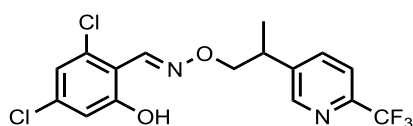
(E)-2,4-dichloro-6-hydroxybenzaldehyde O-(2-(6-chloropyridin-3-yl)propyl)oxime (3af)

Following General procedure E, the product is obtained as a pale-yellow oil, 56% yield. 1H NMR (600 MHz, Chloroform- d) δ 10.43 (s, 1H), 8.59 (s, 1H), 8.28 (d, $J = 2.5$ Hz, 1H), 7.55 (dd, $J = 8.4, 2.7$ Hz, 1H), 7.30 (d, $J = 8.2$ Hz, 1H), 6.96 (d, $J = 2.0$ Hz, 1H), 6.90 (d, $J = 2.0$ Hz, 1H), 4.32 – 4.22 (m, 2H), 3.25 (m, 1H), 1.36 (d, $J = 7.1$ Hz, 3H); ^{13}C NMR (151 MHz, Chloroform- d) δ 159.14, 149.89, 149.07, 148.81, 137.45, 137.35, 136.94, 134.83, 124.19, 121.02, 116.21, 112.68, 79.42, 36.29, 17.61; HRMS (ESI-TOF) m/z Calcd. for $C_{15}H_{14}Cl_3N_2O_2^+$ ($M+H$) $^+$: 359.0115; found: 359.0122.



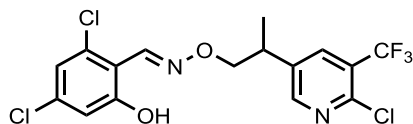
(E)-2,4-dichloro-6-hydroxybenzaldehyde O-(2-(6-bromopyridin-3-yl)propyl) oxime (3ag)

Following General procedure E, the product is obtained as a pale-yellow oil, 57% yield. ¹H NMR (600 MHz, Chloroform-*d*) δ 10.45 (s, 1H), 8.61 (s, 1H), 8.36 – 8.25 (m, 1H), 7.54 – 7.42 (m, 2H), 6.98 (d, *J* = 2.0 Hz, 1H), 6.93 (dd, *J* = 2.0, 0.4 Hz, 1H), 4.30 (qd, *J* = 10.6, 6.9 Hz, 2H), 3.25 (m, 1H), 1.39 (d, *J* = 7.1 Hz, 3H); ¹³C NMR (151 MHz, Chloroform-*d*) δ 159.14, 149.62, 148.83, 140.38, 137.79, 137.27, 136.95, 134.84, 127.99, 121.03, 116.21, 112.67, 79.34, 36.34, 17.55; HRMS (ESI-TOF) *m/z* Calcd. for C₁₅H₁₄BrCl₂N₂O₂⁺ (M+H)⁺: 402.9610; found: 402.9616.



(E)-2,4-dichloro-6-hydroxybenzaldehyde O-(2-(6-(trifluoromethyl)pyridin-3-yl)propyl) oxime (3ah)

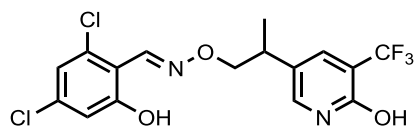
Following General procedure E, the product is obtained as a pale-yellow oil, 62% yield. ¹H NMR (600 MHz, Chloroform-*d*) δ 10.40 (s, 1H), 8.66 (s, 1H), 8.61 (s, 1H), 7.79 (d, *J* = 8.1 Hz, 1H), 7.69 (d, *J* = 8.1 Hz, 1H), 6.98 (d, *J* = 2.0 Hz, 1H), 6.96 – 6.89 (m, 1H), 4.36 (qd, *J* = 10.6, 6.9 Hz, 2H), 3.38 (m, 1H), 1.44 (d, *J* = 7.1 Hz, 3H); ¹³C NMR (151 MHz, Chloroform-*d*) δ 159.14, 149.62, 148.95, 146.79 (q, *J* = 33.2 Hz), 141.87, 137.02, 135.86, 134.87, 121.56 (q, *J* = 274.8 Hz), 121.05, 120.38, 116.22, 112.61, 79.20, 36.90, 17.52; HRMS (ESI-TOF) *m/z* Calcd. for C₁₆H₁₄Cl₂F₃N₂O₂⁺ (M+H)⁺: 393.0379; found: 393.0381.



(E)-2,4-dichloro-6-hydroxybenzaldehyde O-(2-(6-chloro-5-(trifluoromethyl)pyridine-3-yl)propyl) oxime (3ai)

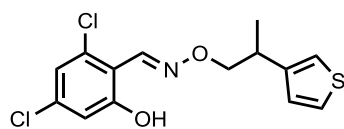
Following General procedure E, the product is obtained as a pale-yellow oil, 78% yield. ¹H NMR (600 MHz, Chloroform-*d*) δ 10.35 (s, 1H), 8.62 (s, 1H), 8.48 (d, *J* = 2.3 Hz, 1H), 7.91 (d, *J* = 2.4 Hz, 1H), 6.99 (d, *J* = 2.0 Hz, 1H), 6.93 (dd, *J* = 2.0, 0.5 Hz, 1H), 4.41 – 4.27 (m, 2H), 3.36 (h, *J* = 7.2 Hz, 1H), 1.43 (d, *J* = 7.1 Hz, 3H); ¹³C

NMR (151 MHz, Chloroform-*d*) δ 159.13, 151.63, 149.16, 147.27, 137.70, 137.13, 135.28 (q, $J = 4.5$ Hz), 134.92, 125.16 (q, $J = 33.2$ Hz), 122.14 (q, $J = 273.3$ Hz), 121.10, 116.24, 112.53, 78.92, 36.25, 17.40; HRMS (ESI-TOF) m/z Calcd. for $C_{16}H_{13}Cl_3F_3N_2O_2^+$ (M+H)⁺: 426.9989; found: 426.9996.



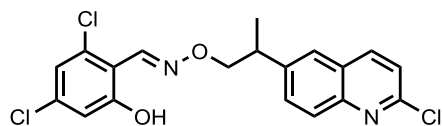
(E)-2,4-dichloro-6-hydroxybenzaldehyde O-(2-(6-hydroxy-5-(trifluoromethyl)pyridin-3-yl)propyl) oxime (3aj)

Following General procedure E, the product is obtained as a pale-yellow oil, 56% yield. ¹H NMR (600 MHz, Chloroform-*d*) δ 10.40 (s, 1H), 8.64 (s, 1H), 7.93 (d, $J = 53.5$ Hz, 1H), 7.83 (d, $J = 2.0$ Hz, 1H), 7.57 (d, $J = 2.2$ Hz, 1H), 6.98 (d, $J = 2.0$ Hz, 1H), 6.95 – 6.89 (m, 1H), 4.29 – 4.18 (m, 2H), 3.10 (m, 1H), 1.34 (d, $J = 7.1$ Hz, 3H); ¹³C NMR (151 MHz, Chloroform-*d*) δ 160.44, 158.71, 148.62, 136.59, 134.48, 128.00, 127.89, 127.83, 127.63, 120.61, 120.25, 115.78, 112.17, 78.59, 35.05, 16.60; HRMS (ESI-TOF) m/z Calcd. for $C_{16}H_{14}Cl_2F_3N_2O_3^+$ (M+H)⁺: 409.0328; found: 409.0331.



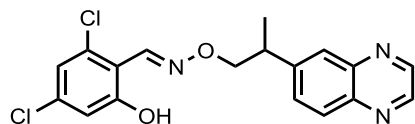
(E)-2,4-dichloro-6-hydroxybenzaldehyde O-(2-(thiophen-3-yl)propyl) oxime (3ak)

Following General procedure E, the product is obtained as a pale-yellow oil, 64% yield. ¹H NMR (600 MHz, Chloroform-*d*) δ 10.60 (s, 1H), 8.64 (s, 1H), 7.30 (dd, $J = 4.9, 2.9$ Hz, 1H), 7.05 (ddd, $J = 3.0, 1.4, 0.7$ Hz, 1H), 7.03 (dd, $J = 5.0, 1.4$ Hz, 1H), 6.96 (d, $J = 2.1$ Hz, 1H), 6.91 (dd, $J = 2.0, 0.6$ Hz, 1H), 4.33 (dd, $J = 10.5, 6.5$ Hz, 1H), 4.22 (dd, $J = 10.5, 7.3$ Hz, 1H), 3.34 (h, $J = 6.9$ Hz, 1H), 1.37 (d, $J = 7.0$ Hz, 3H); ¹³C NMR (151 MHz, Chloroform-*d*) δ 159.19, 148.29, 143.78, 136.65, 134.70, 126.83, 125.70, 120.91, 120.30, 116.18, 112.94, 80.21, 34.75, 17.95; HRMS (ESI-TOF) m/z Calcd. for $C_{14}H_{14}Cl_2NO_2S^+$ (M+H)⁺: 330.0117; found: 330.0115.



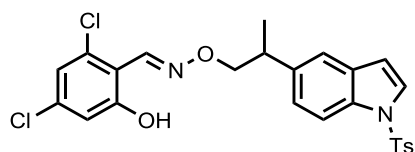
(E)-2,4-dichloro-6-hydroxybenzaldehyde O-(2-(2-chloroquinolin-6-yl)propyl) oxime (3al)

Following General procedure E, the product is obtained as a pale-yellow oil, 33% yield. ^1H NMR (600 MHz, Chloroform-*d*) δ 10.56 (s, 1H), 8.59 (s, 1H), 8.12 (d, $J = 8.7$ Hz, 1H), 7.85 (d, $J = 8.6$ Hz, 1H), 7.67 – 7.59 (m, 2H), 7.08 (d, $J = 8.8$ Hz, 1H), 6.95 (d, $J = 2.0$ Hz, 1H), 6.90 (d, $J = 2.4$ Hz, 1H), 6.89 – 6.84 (m, 1H), 4.44 – 4.30 (m, 2H), 3.40 (m, 1H), 1.44 (d, $J = 7.0$ Hz, 3H); ^{13}C NMR (151 MHz, Chloroform-*d*) δ 158.72, 157.54, 148.08, 143.70, 140.01, 139.93, 136.33, 134.29, 129.62, 127.31, 125.84, 125.07, 120.51, 115.73, 112.39, 111.44, 79.72, 38.68, 17.68; HRMS (ESI-TOF) m/z Calcd. for $\text{C}_{19}\text{H}_{16}\text{Cl}_3\text{N}_2\text{O}_2^+$ ($\text{M}+\text{H}$) $^+$: 409.0272; found: 409.0278.



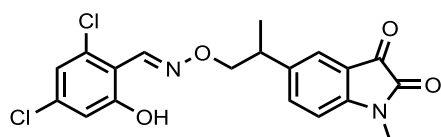
(E)-2,4-dichloro-6-hydroxybenzaldehyde O-(2-(quinoxalin-6-yl)propyl) oxime (3am)

Following General procedure E, the product is obtained as a pale-yellow oil, 35% yield. ^1H NMR (600 MHz, Chloroform-*d*) δ 10.50 (s, 1H), 8.86 – 8.77 (m, 2H), 8.58 (s, 1H), 8.09 (d, $J = 8.6$ Hz, 1H), 7.98 (d, $J = 1.9$ Hz, 1H), 7.70 (dd, $J = 8.7, 2.0$ Hz, 1H), 6.93 (d, $J = 2.0$ Hz, 1H), 6.88 (dd, $J = 2.0, 0.6$ Hz, 1H), 4.47 – 4.35 (m, 2H), 3.48 (m, 1H), 1.48 (d, $J = 7.0$ Hz, 3H); ^{13}C NMR (151 MHz, Chloroform-*d*) δ 158.71, 148.18, 145.19, 144.64, 144.20, 142.65, 141.69, 136.33, 134.31, 129.79, 129.17, 126.77, 120.49, 115.73, 112.34, 79.43, 38.94, 17.52; HRMS (ESI-TOF) m/z Calcd. for $\text{C}_{18}\text{H}_{16}\text{Cl}_2\text{N}_3\text{O}_2^+$ ($\text{M}+\text{H}$) $^+$: 376.0614; found: 376.0621.



(E)-2,4-dichloro-6-hydroxybenzaldehyde O-(2-(1-tosyl-1H-indol-5-yl)propyl) oxime (3an)

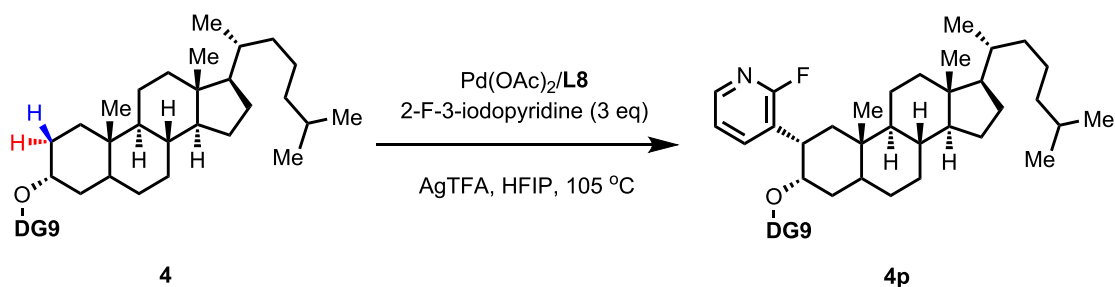
Following General procedure E, the product is obtained as a pale-yellow oil, 82% yield. ^1H NMR (600 MHz, Chloroform-*d*) δ 10.63 (s, 1H), 8.61 (s, 1H), 7.97 (d, $J = 8.6$ Hz, 1H), 7.80 (d, $J = 8.4$ Hz, 2H), 7.57 (d, $J = 3.7$ Hz, 1H), 7.42 (d, $J = 1.6$ Hz, 1H), 7.28 – 7.19 (m, 4H), 6.97 (d, $J = 2.0$ Hz, 1H), 6.90 (dd, $J = 2.0, 0.5$ Hz, 1H), 4.35 (dd, $J = 10.5, 6.8$ Hz, 1H), 4.26 (dd, $J = 10.5, 7.4$ Hz, 1H), 3.30 (m, 1H), 2.36 (s, 3H), 1.39 (d, $J = 7.0$ Hz, 3H); ^{13}C NMR (151 MHz, Chloroform-*d*) δ 159.16, 148.30, 144.87, 138.10, 136.60, 135.37, 134.67, 133.72, 132.98, 130.99, 129.85, 126.82, 126.75, 126.57, 124.09, 120.86, 116.13, 115.31, 113.54, 112.91, 108.79, 107.95, 80.63, 39.08, 21.53, 18.39; HRMS (ESI-TOF) m/z Calcd. for $\text{C}_{25}\text{H}_{23}\text{Cl}_2\text{N}_2\text{O}_4\text{S}^+$ ($\text{M}+\text{H}$) $^+$: 517.0750; found: 517.0754.



(*E*)-2,4-dichloro-6-hydroxybenzaldehyde O-(2-(1-methyl-2,3-dioxindolin-5-yl)propyl) oxime (3ao)

Following General procedure E, the product is obtained as a pale-yellow oil, 74% yield. ^1H NMR (600 MHz, Chloroform-*d*) δ 10.45 (s, 1H), 8.58 (s, 1H), 7.52 (d, $J = 1.9$ Hz, 1H), 7.49 (dd, $J = 8.1, 1.9$ Hz, 1H), 6.95 (d, $J = 2.1$ Hz, 1H), 6.89 (dd, $J = 2.0, 0.5$ Hz, 1H), 6.85 (d, $J = 8.0$ Hz, 1H), 4.29 – 4.22 (m, 2H), 3.24 (m, 4H), 1.34 (d, $J = 7.1$ Hz, 3H); ^{13}C NMR (151 MHz, Chloroform-*d*) δ 183.41, 159.14, 158.29, 150.24, 148.67, 139.17, 137.54, 136.88, 134.78, 123.90, 120.99, 117.70, 116.19, 112.73, 110.02, 79.83, 38.65, 26.25, 17.78; HRMS (ESI-TOF) m/z Calcd. for $\text{C}_{19}\text{H}_{17}\text{Cl}_2\text{N}_2\text{O}_4^+$ ($\text{M}+\text{H}$) $^+$: 407.0560; found: 407.0565.

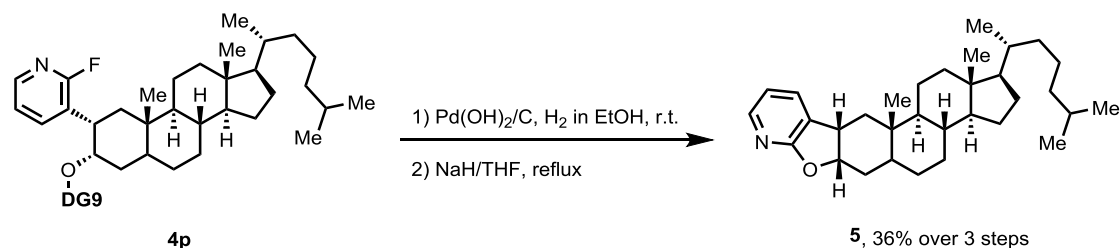
C-H arylation/annulation of dihydrocholesterol



(E)-2,4-dichloro-6-hydroxybenzaldehyde O-((2S,3S,8R,9S,10S,13R,14S,17R)-2-(2-fluoropyridin-3-yl)-10,13-dimethyl-17-((R)-6-methylheptan-2-yl)hexadecahydro-1H-cyclopenta[a]phenanthren-3-yl) oxime (4p)

To a 8-mL vial were added **DG9**-tethered dihydrocholesterol (**4**, 115 mg, 0.2 mmol), 2-fluoro-3-iodopyridine (134 mg, 0.6 mmol), palladium acetate (4.5 mg, 0.02 mmol), 3-nitro-5-trifluoromethyl-2-pyridone (16.2 mg, 0.08 mmol) and silver trifluoroacetate (110 mg, 0.5 mmol). The mixture was dissolved with 1,1,1,3,3,3-hexafluoro-2-propanol (HFIP, 2.0 mL). The vial was sealed and stirred at 105 °C for 12 hours. The reaction was cooled to room temperature and diluted with 5 mL of EtOAc, filtered through a pad of Celite, the filtrate was concentrated and purified by preparative thin-layer chromatography (hexanes : EtOAc = 4:1) to give **4p** as a white solid (68 mg, 51% yield).

$[\alpha]_D^{20} = +57.6$ ($c=0.25$, CHCl_3); $^1\text{H NMR}$ (600 MHz, Chloroform- d) δ 10.36 (s, 1H), 8.53 (s, 1H), 8.07 (d, $J = 4.6$ Hz, 1H), 7.80 (t, $J = 8.6$ Hz, 1H), 7.14 (ddd, $J = 7.5, 4.8, 1.4$ Hz, 1H), 6.94 (d, $J = 2.0$ Hz, 1H), 6.87 (d, $J = 2.0$ Hz, 1H), 4.58 (d, $J = 2.4$ Hz, 1H), 3.40 (dt, $J = 13.5, 3.2$ Hz, 1H), 2.01 (dt, $J = 12.6, 3.2$ Hz, 1H), 1.91 – 1.67 (m, 6H), 1.61 – 1.49 (m, 4H), 1.44 – 1.24 (m, 8H), 1.22 – 1.05 (m, 7H), 1.01 (d, $J = 10.1$ Hz, 5H), 0.95 – 0.84 (m, 10H), 0.69 (s, 3H); $^{13}\text{C NMR}$ (151 MHz, Chloroform- d) δ 161.31 (d, $J = 237$ Hz), 159.14, 147.74, 145.26 (d, $J = 15$ Hz), 140.15 (d, $J = 6$ Hz), 136.50, 134.40, 124.78 (d, $J = 28.7$ Hz), 121.29 (d, $J = 4.5$ Hz), 120.75, 116.17, 112.90, 81.34, 56.51, 56.31, 54.34, 42.61, 39.96, 39.50, 39.03, 37.44, 36.44, 36.16, 35.80, 35.38, 34.70, 33.09, 31.86, 28.23, 28.00, 27.94, 24.17, 23.86, 22.82, 22.55, 20.92, 18.67, 12.28, 12.11; HRMS (ESI-TOF) m/z Calcd. for $\text{C}_{39}\text{H}_{54}\text{Cl}_2\text{FN}_2\text{O}_2^+$ ($\text{M}+\text{H}$) $^+$: 671.3541; found: 671.3538.

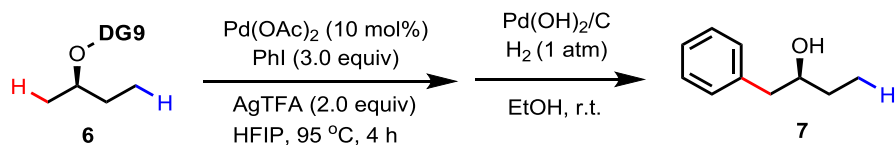


(1R,3aS,3bR,6aS,11bS,12aS,12bS,14aR)-12a,14a-dimethyl-1-((R)-6-methylheptan-2-yl)-2,3,3a,3b,4,5,5a,6,6a,11b,12,12a,12b,13,14,14a-hexadecahydro-1H-cyclopenta[7',8']phenanthro[3',2':4,5]furo[2,3-b]pyridine (5)

Product **4p** was dissolved with EtOH (1.0 mL) in an 8-mL vial. Pd(OH)₂ on activated carbon (20 wt. %, 20 mg) and HOAc (20 μL) were added sequentially. The vial was equipped with a H₂ balloon, evacuated and backfilled with H₂ (× 3), the resulting mixture was stirred at room temperature under H₂ atmosphere for 24 h. The reaction mixture was filtered, the filtrate was concentrated under reduced pressure, and the residue was dissolved with anhydrous THF (2.0 mL), then NaH (60 wt. % in mineral oil, 20 mg, 0.5 mmol) was added. The resulting mixture was stirred at 65 °C for 12 hours. The reaction solution was poured into 20 mL of water, extracted with EtOAc (10 mL × 3), the organic phase was combined and dried with anhydrous sodium sulfate. The solvent was evaporated, and the residue purified by preparative thin-layer chromatography (hexanes : EtOAc = 3:1) to give annulation product **5** (33 mg, white solid, 70% yield over 2 steps).

$[\alpha]_D^{20} = +16.2$ ($c=1$, CHCl₃); ¹H NMR (600 MHz, Chloroform-*d*) δ 8.04 – 7.95 (m, 1H), 7.42 (dd, $J = 7.1, 1.4$ Hz, 1H), 6.78 (dd, $J = 7.1, 5.3$ Hz, 1H), 4.64 (t, $J = 4.8$ Hz, 1H), 3.14 (dt, $J = 12.4, 6.4$ Hz, 1H), 2.08 (dd, $J = 15.7, 3.6$ Hz, 1H), 2.00 – 1.84 (m, 2H), 1.83 – 1.75 (m, 1H), 1.73 – 1.63 (m, 2H), 1.57 – 1.40 (m, 4H), 1.36 – 1.16 (m, 8H), 1.15 – 0.93 (m, 8H), 0.89 – 0.80 (m, 13H), 0.64 (s, 5H); ¹³C NMR (151 MHz, Chloroform-*d*) δ 167.24, 145.57, 132.36, 128.79, 116.71, 81.28, 56.38, 56.12, 53.49, 42.82, 42.39, 39.83, 39.49, 38.95, 37.03, 36.11, 35.76, 35.45, 34.66, 31.63, 30.47, 28.43, 28.17, 27.99, 24.17, 23.78, 22.80, 22.54, 20.75, 18.64, 12.02, 11.02; HRMS (ESI-TOF) m/z Calcd. for C₃₂H₅₀NO⁺ (M+H)⁺: 464.3887; found: 464.3888.

Synthesis of enlitalzone precursor via sequential β- and γ-C-H arylations



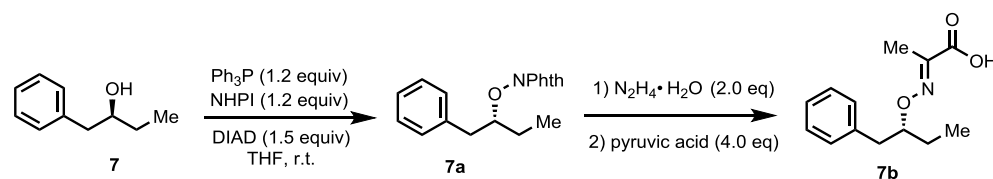
(S)-1-phenylbutan-2-ol (**7**)

To an 8-mL vial were added **6** (261 mg, 1.0 mmol), iodobenzene (600 mg, 3.0 mmol), palladium acetate (23 mg, 0.1 mmol) and silver trifluoroacetate (440 mg, 2.0 mmol). The mixture was dissolved with 1,1,1,3,3,3-hexafluoro-2-propanol (HFIP, 5.0 mL). The vial was sealed and stirred at 95 °C for 4 hours. The reaction was cooled to room temperature, diluted with 10 mL of EtOAc and filtered through a pad of Celite. The filtrate was concentrated, the residue was re-dissolved with 1 mL of EtOAc, then 10 mL of hexane was added. The solution was filtered again through a pad of Celite, the filtrate was concentrated under reduced pressure, and the resulting residue was used directly in the next step.

The crude arylation product was dissolved with EtOH (4 mL) in an 8-mL vial, following which Pd(OH)₂ on activated carbon (20 wt. %, 100 mg) and HOAc (100 μL) were added sequentially. The vial was equipped with a H₂ balloon, evacuated and backfilled with H₂ (× 3), and the resulting mixture was stirred at room temperature under H₂ atmosphere for 24 hours. The reaction mixture was filtered, the filtrate was concentrated under reduced pressure, and the residue was purified by flash column chromatography (hexanes : EtOAc = 5:1) to afford **7** (97 mg, 65% over 2 steps).

Colorless oil, 65% yield from **6**. $[\alpha]_D^{20} = +28.7$ (c=1, CHCl₃); ¹H NMR (600 MHz, Chloroform-*d*) δ 7.41 – 7.32 (m, 2H), 7.31 – 7.23 (m, 3H), 3.78 (ddt, *J* = 8.3, 7.4, 4.6 Hz, 1H), 2.87 (dd, *J* = 13.6, 4.3 Hz, 1H), 2.68 (dd, *J* = 13.6, 8.4 Hz, 1H), 1.66 – 1.51 (m, 3H), 1.03 (t, *J* = 7.5 Hz, 3H); ¹³C NMR (151 MHz, Chloroform-*d*) δ 138.63, 129.38, 128.49, 126.37, 74.00, 43.55, 29.55, 10.02; HRMS (ESI-TOF) *m/z* Calcd. for C₁₀H₁₅O⁺ (M+H)⁺: 151.1117; found: 151.1114.

Directing group installation for γ-C-H arylation:



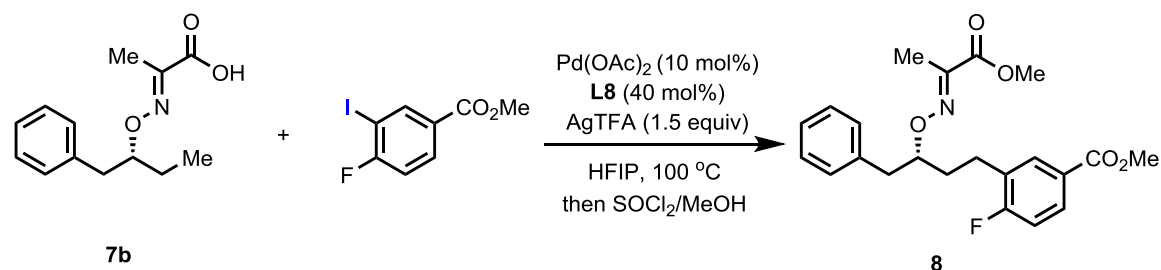
(R,E)-2-(((1-phenylbutan-2-yl)oxy)imino)propanoic acid (**7b**)

Alcohol **7** (75 mg, 0.5 mmol), NHPI (100 mg, 0.6 mmol) and Ph₃P (160 mg, 0.6 mmol) were dissolved in anhydrous THF (3.0 mL) and cooled with an ice-bath. To the pre-cooled solution was added DIAD (180 μL, 0.9 mmol) dropwise and the resulting mixture was stirred at room temperature for 12 h. The solvent was evaporated, and the residue was purified by flash column chromatography (hexanes : EtOAc = 4:1) to give **7a**: 140 mg, 95% yield.

Product **7a** was dissolved in CH₂Cl₂ (5 mL), then hydrazine monohydrate (1.0 mmol) and MeOH (1 mL) were added sequentially. The reaction mixture was stirred at room temperature for 2 h. The white solid formed during the reaction was removed by filtration. To the filtrate was added pyruvic acid (180 mg, 2.0 mmol), and the resulting mixture was stirred for 1 h at room temperature. The solution was extracted with aq. NaOH (4.0 M, 10 mL) and the aqueous phase was washed with CH₂Cl₂ (5 mL, × 2). The aqueous phase was then acidified to pH ~ 2.0 by addition of conc. HCl, then extracted with CH₂Cl₂. The organic phase was dried with anhydrous sodium sulfate and concentrated to give the product **7b** (106 mg, 95% yield).

Colorless oil, 90% yield from **7**. ¹H NMR (600 MHz, Chloroform-*d*) δ 7.34 – 7.29 (m, 2H), 7.27 – 7.23 (m, 1H), 7.22 – 7.16 (m, 2H), 4.46 (tt, *J* = 7.0, 5.6 Hz, 1H), 3.04 – 2.92 (m, 2H), 2.06 (s, 3H), 1.78 – 1.67 (m, 2H), 1.00 (t, *J* = 7.4 Hz, 3H); ¹³C NMR (151 MHz, Chloroform-*d*) δ 163.24, 147.53, 137.77, 129.35, 128.34, 126.45, 88.00, 39.98, 26.18, 10.13, 9.62; HRMS (ESI-TOF) *m/z* Calcd. for C₁₃H₁₈O₃⁺ (*M*+*H*)⁺: 236.1281; found: 236.1276.

γ-C-H arylation of substrate **7b**:

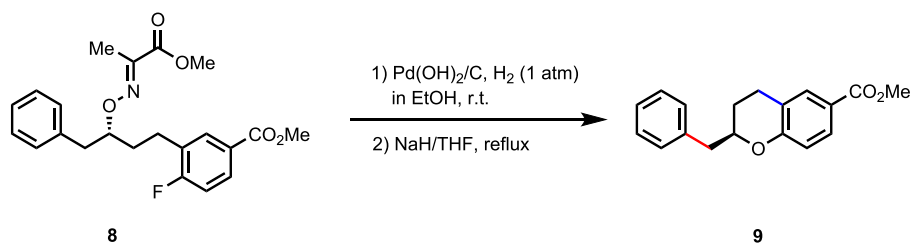


Methyl (R,E)-4-fluoro-3-(3-(((1-methoxy-1-oxopropan-2-ylidene)amino)oxy)-4-phenylbutyl)benzoate (**8**)

To a 8-mL vial were added **7b** (94 mg, 0.4 mmol), methyl 4-fluoro-3-iodobenzoate (168 mg, 0.6 mmol), palladium acetate (9.2 mg, 0.04 mmol), 3-nitro-5-trifluoro-2-pyridone (32 mg, 0.16 mmol) and silver trifluoroacetate (132 mg, 0.6 mmol), and the mixture was dissolved with 1,1,1,3,3,3-hexafluoro-2-propanol (HFIP, 2.0 mL). The vial was sealed and stirred at 100 °C for 4 hours. The reaction was cooled to room temperature, diluted with EtOAc (5 mL), and filtered through a pad of Celite. The filtrate was concentrated, the residue was re-dissolved with methanol (3 mL), then SOCl₂ (0.3 mL) was added dropwise. After stirring for 30 min at room temperature, the solution was concentrated under reduced pressure and the residue was purified by preparative thin-layer chromatography to give the arylation product **8** (78 mg, 49% yield).

Colorless oil, 49% yield from **7b**. ¹H NMR (600 MHz, Chloroform-*d*) δ 7.91 – 7.86 (m, 2H), 7.30 – 7.27 (m, 2H), 7.25 – 7.22 (m, 1H), 7.22 – 7.18 (m, 2H), 7.07 – 7.03 (m, 1H), 4.65 – 4.61 (m, 1H), 3.91 (d, *J* = 2.2 Hz, 3H), 3.88 (s, 3H), 3.10 (dd, *J* = 14.0, 6.1 Hz, 1H), 2.97 (dd, *J* = 14.0, 6.4 Hz, 1H), 2.84 (ddd, *J* = 15.1, 9.5, 6.1 Hz, 1H), 2.80 – 2.71 (m, 1H), 2.06 (s, 3H), 2.01 – 1.94 (m, 2H); ¹³C NMR (151 MHz, Chloroform-*d*) δ 166.25, 164.40, 148.79, 137.47, 132.50, 132.45, 129.66, 129.60, 129.53, 128.88, 128.73, 128.51, 128.27, 126.33, 115.45, 115.29, 84.74, 52.63, 52.11, 40.11, 32.95, 24.85, 11.60; HRMS (ESI-TOF) *m/z* Calcd. for C₂₂H₂₅FNO₅⁺ (M+H)⁺: 402.1711; found: 402.1713.

Synthesis of englitazone precursor **9**:



Methyl (*R*)-2-benzylchromane-6-carboxylate (**9**)

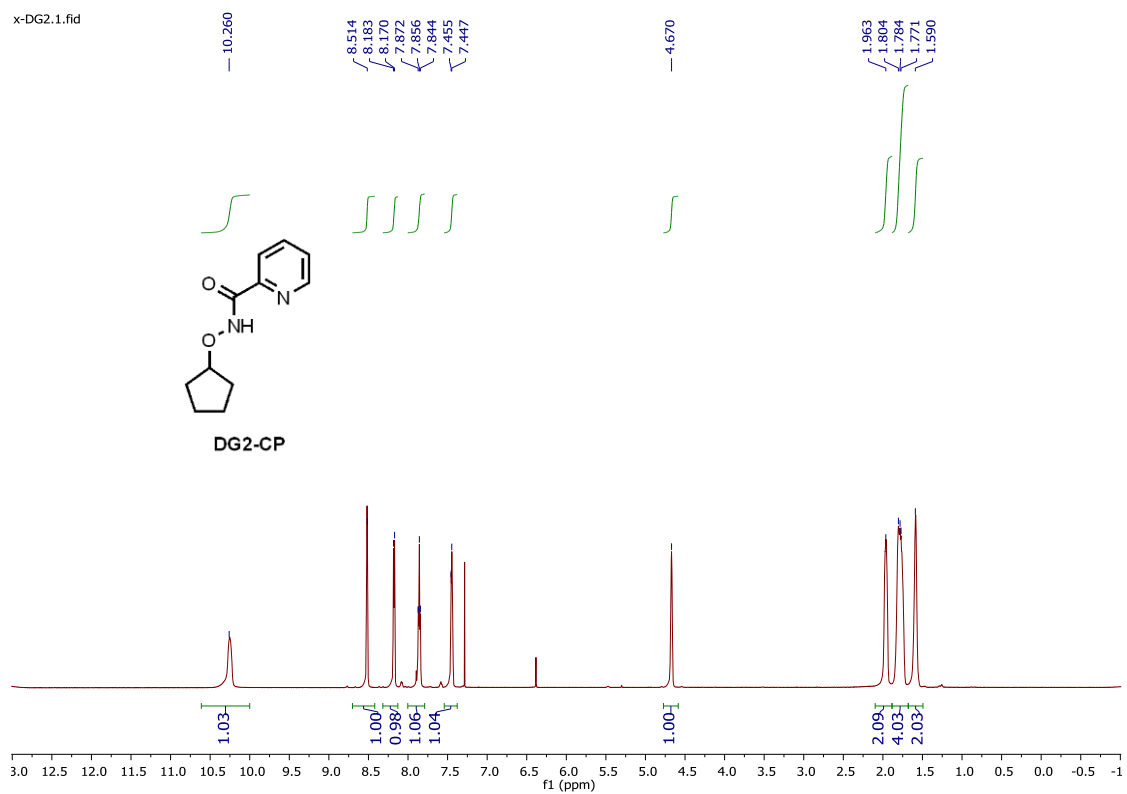
Arylation product **8** (40 mg, 0.1 mmol) was dissolved with EtOH (1.0 mL) in an 8-mL vial, and Pd(OH)₂ on activated carbon (20 wt. %, 20 mg) and HOAc (20 μL) were

added sequentially. The vial was equipped with a H₂ balloon, evacuated and backfilled with H₂ (× 3), and the resulting mixture was stirred at room temperature under H₂ atmosphere for 24 h. The reaction mixture was filtered, the filtrate was concentrated under reduced pressure, and the residue was re-dissolved with anhydrous THF (2.0 mL). Next, NaH (60 wt. % in mineral oil, 20 mg, 0.5 mmol) was added, and the resulting mixture was stirred at 65 °C for 8 h. The reaction solution was poured into water (20 mL) and extracted with EtOAc (10 mL × 3). The organic phase was dried with anhydrous sodium sulfate, concentrated, and the residue was purified by preparative thin-layer chromatography (hexanes : EtOAc = 4:1) to give cyclization product **9** (22 mg, 81% yield over 2 steps).

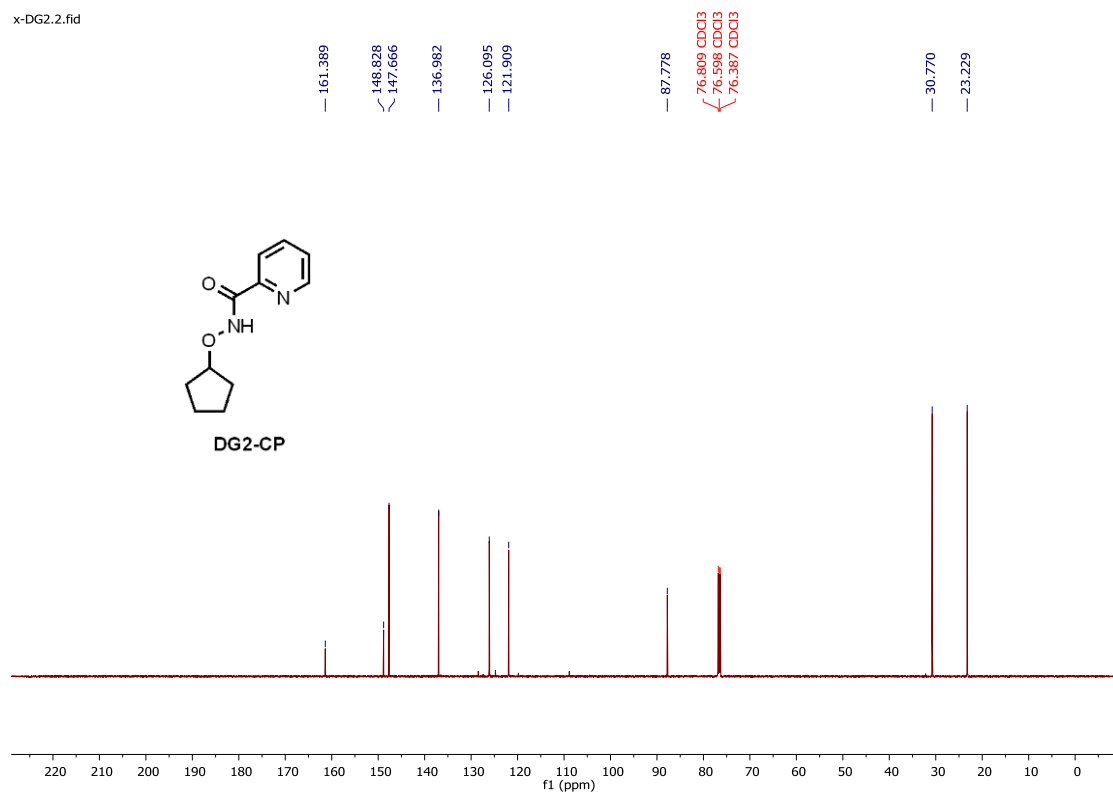
Colorless oil, 81% yield from **8**. $[\alpha]_D^{20} = -94$ ($c=1$, CHCl₃); ¹H NMR (600 MHz, Chloroform-*d*) δ 7.77 (d, $J = 9.5$ Hz, 2H), 7.37 – 7.30 (m, 2H), 7.27 (d, $J = 7.4$ Hz, 2H), 7.26 – 7.22 (m, 1H), 6.84 – 6.79 (m, 1H), 4.30 – 4.25 (m, 1H), 3.86 (s, 3H), 3.14 (dd, $J = 13.7, 6.3$ Hz, 1H), 2.90 (dd, $J = 13.7, 6.8$ Hz, 1H), 2.80 (dd, $J = 10.1, 5.8$ Hz, 2H), 2.05 – 1.99 (m, 1H), 1.72 (m, 1H); ¹³C NMR (151 MHz, Chloroform-*d*) δ 167.07, 158.93, 137.42, 131.59, 129.54, 129.10, 128.42, 126.56, 121.85, 121.70, 116.74, 77.26, 51.79, 41.67, 26.23, 24.44; HRMS (ESI-TOF) m/z Calcd. for C₁₈H₁₉O₃⁺ (M+H)⁺: 283.1329; found: 283.1331.

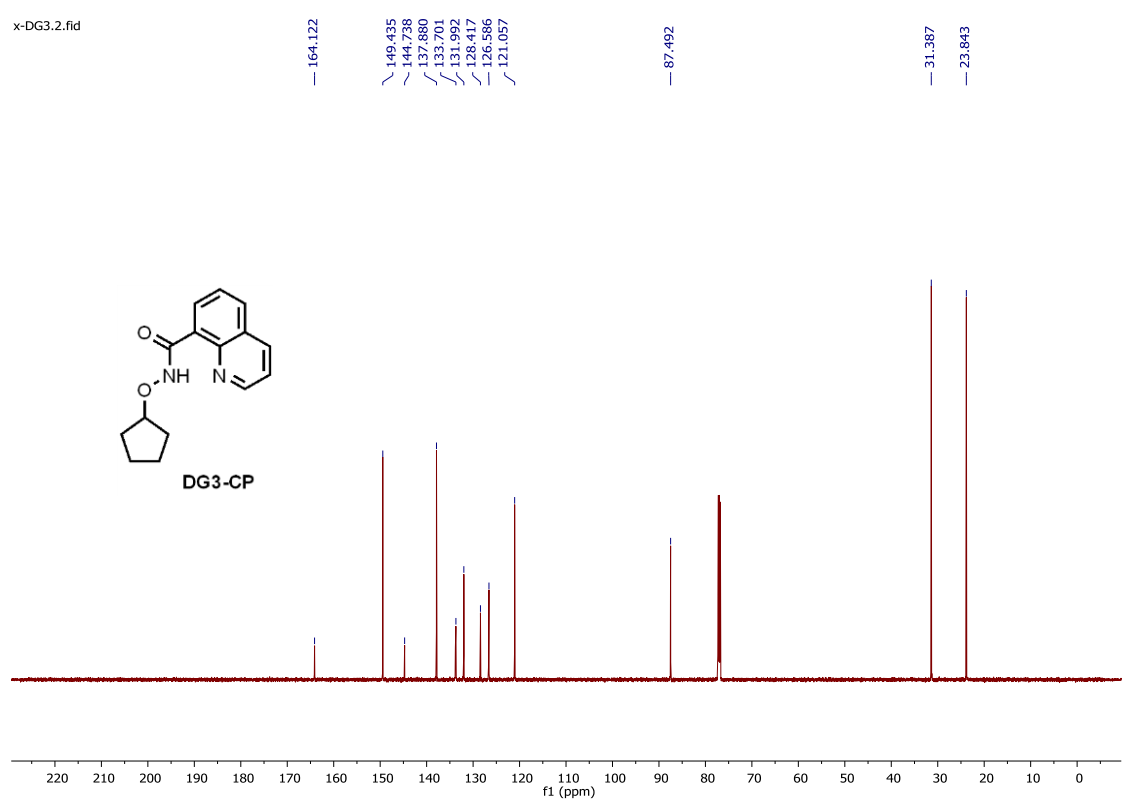
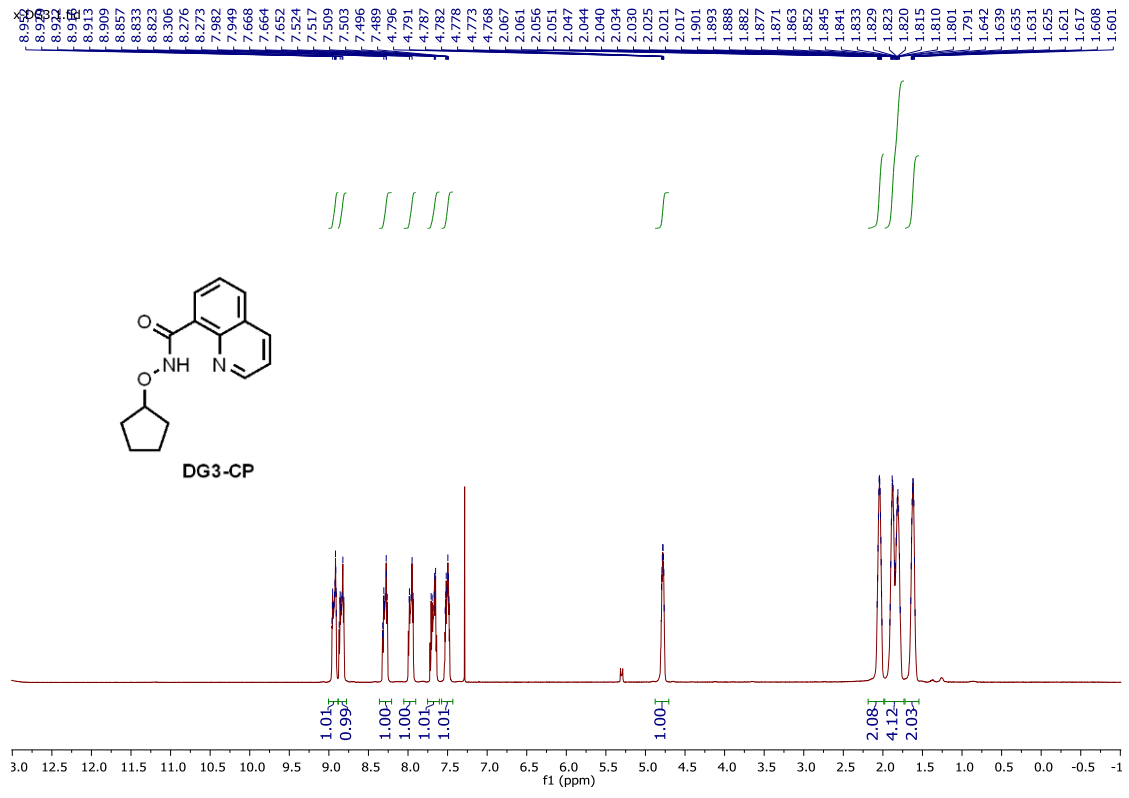
¹H and ¹³C NMR Spectra

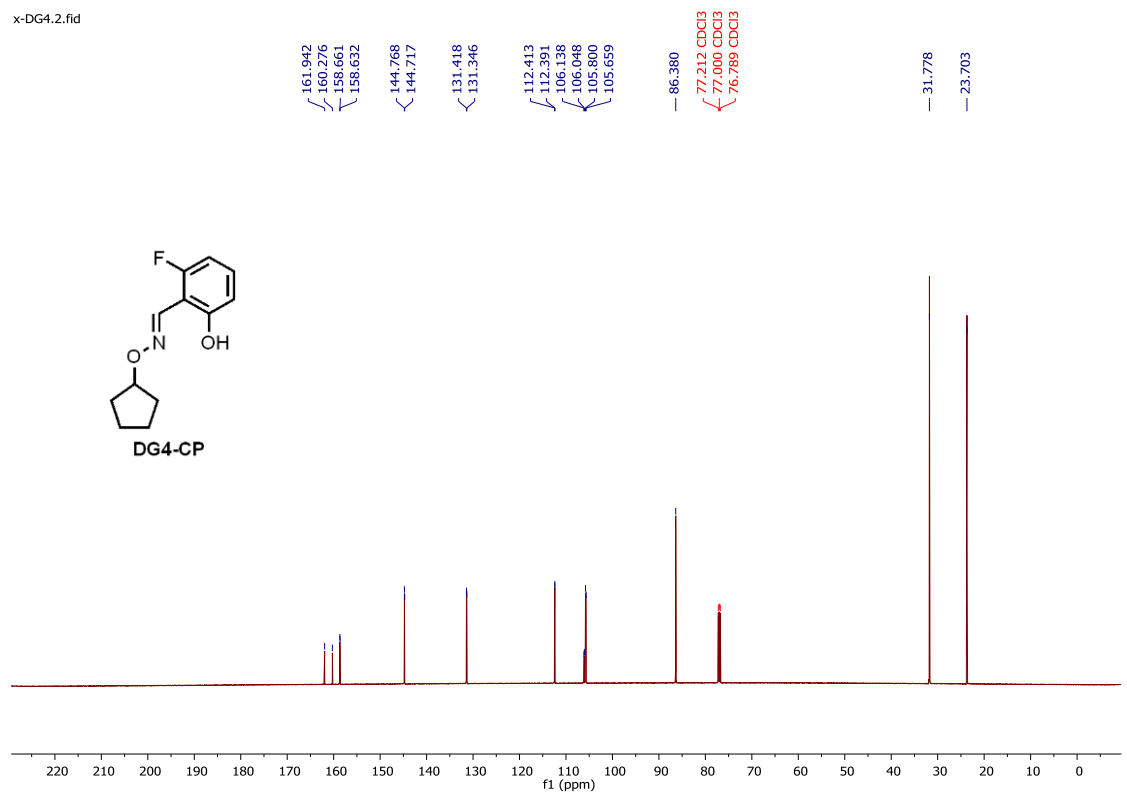
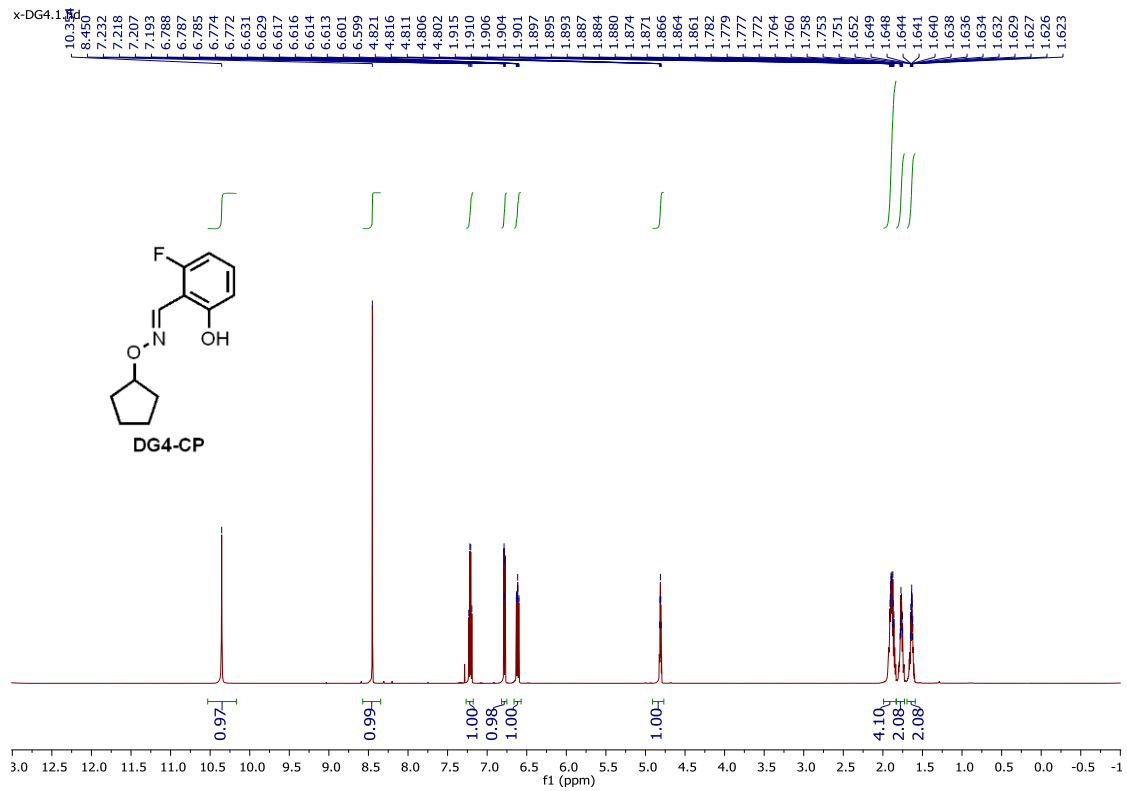
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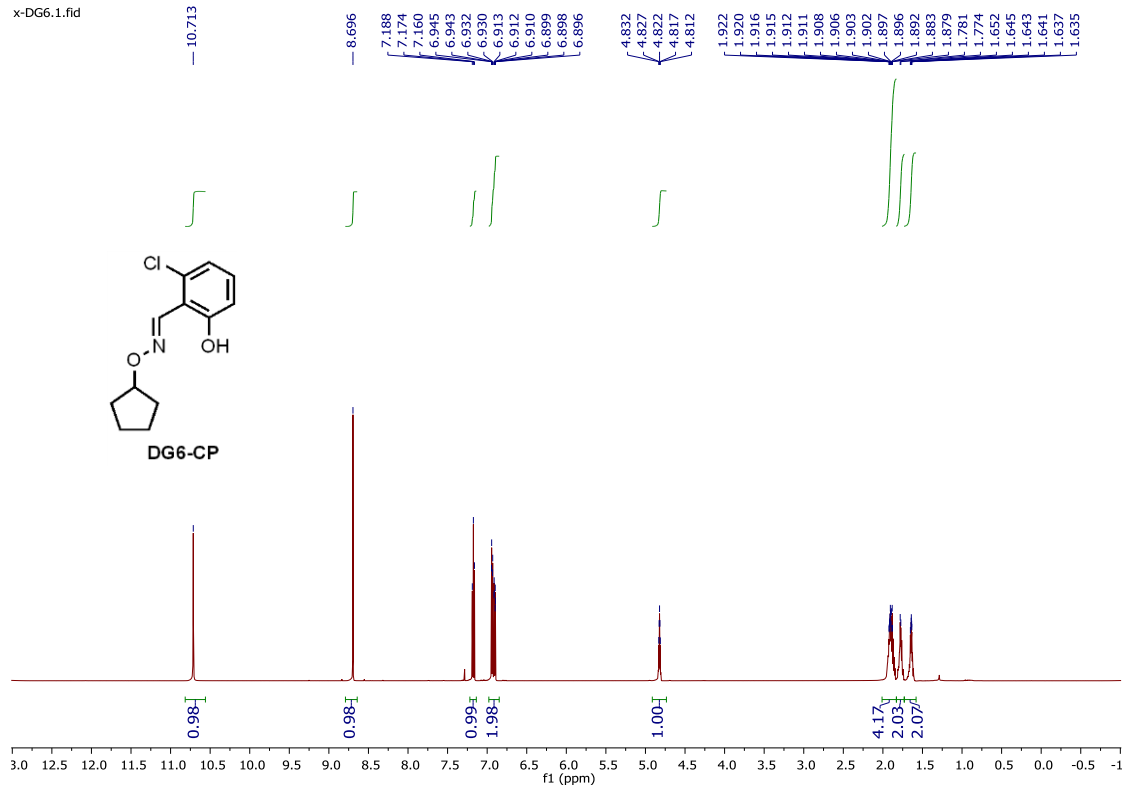
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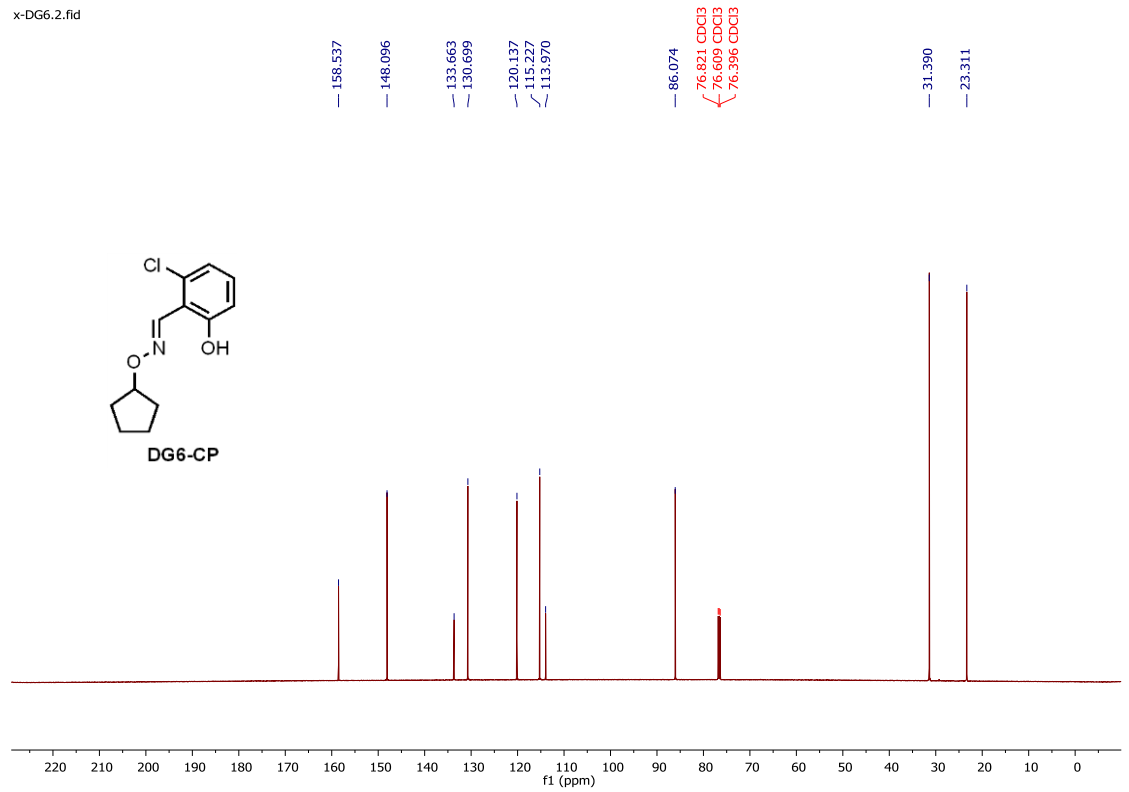


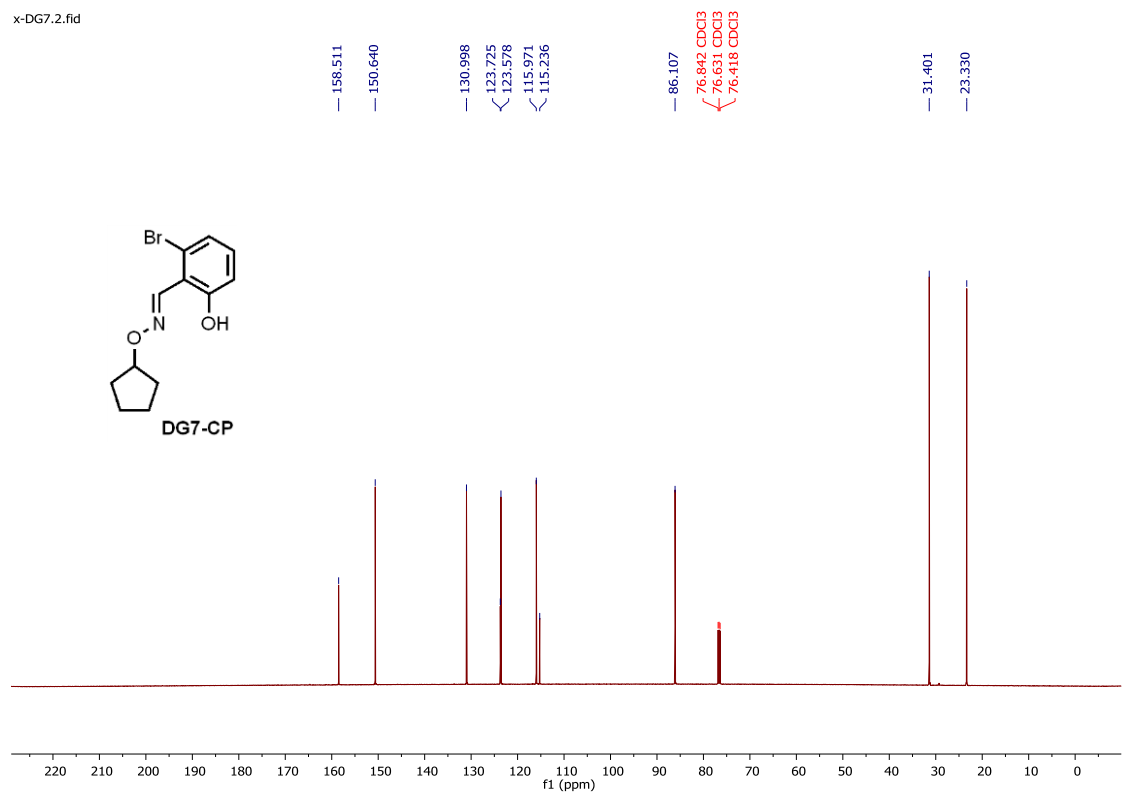
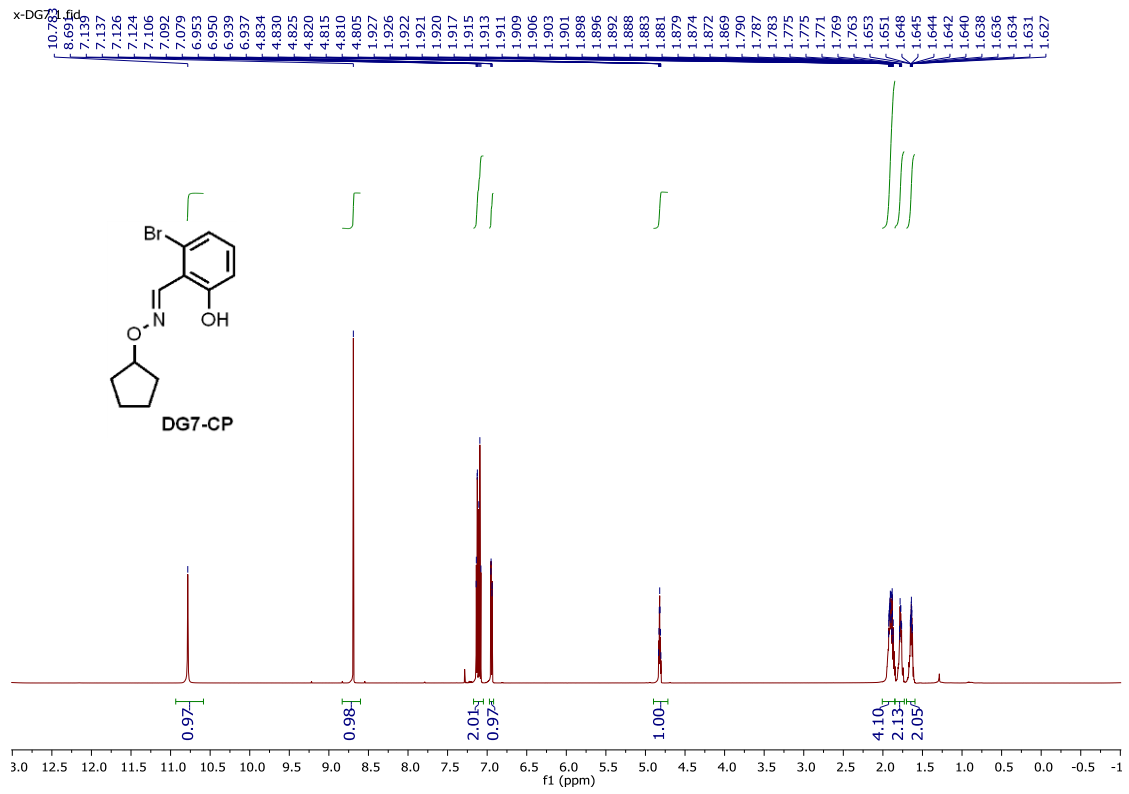


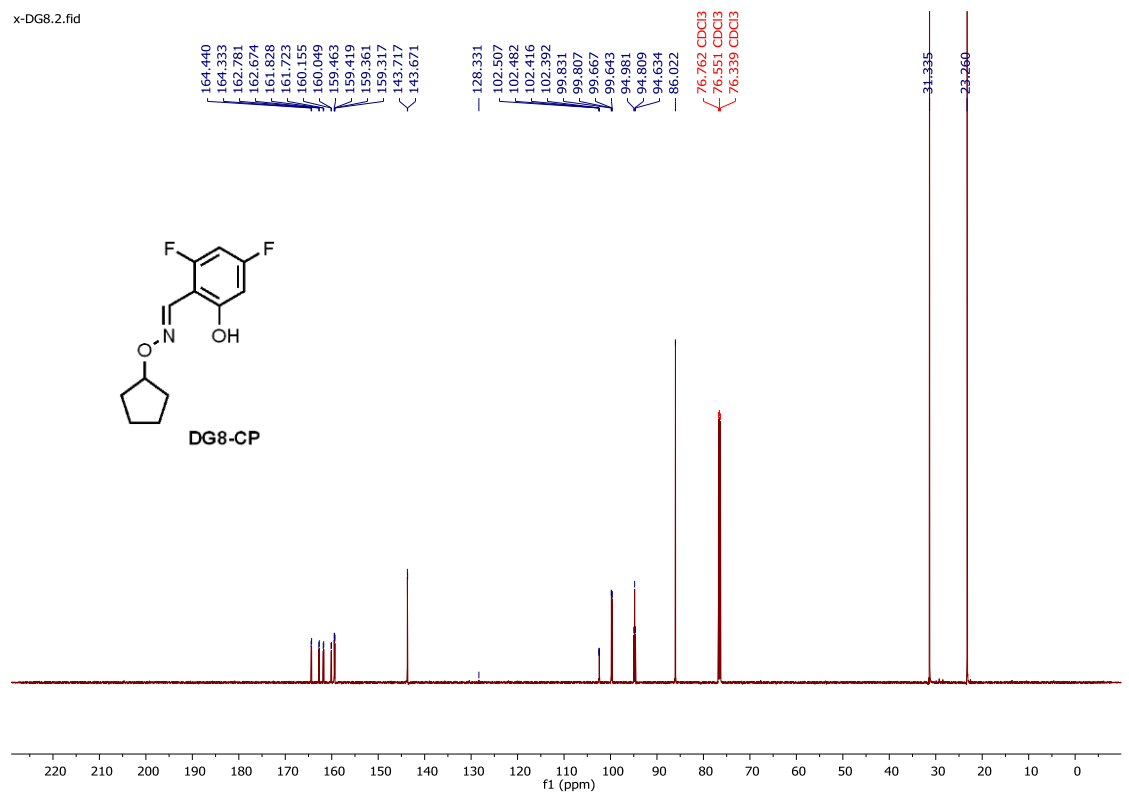
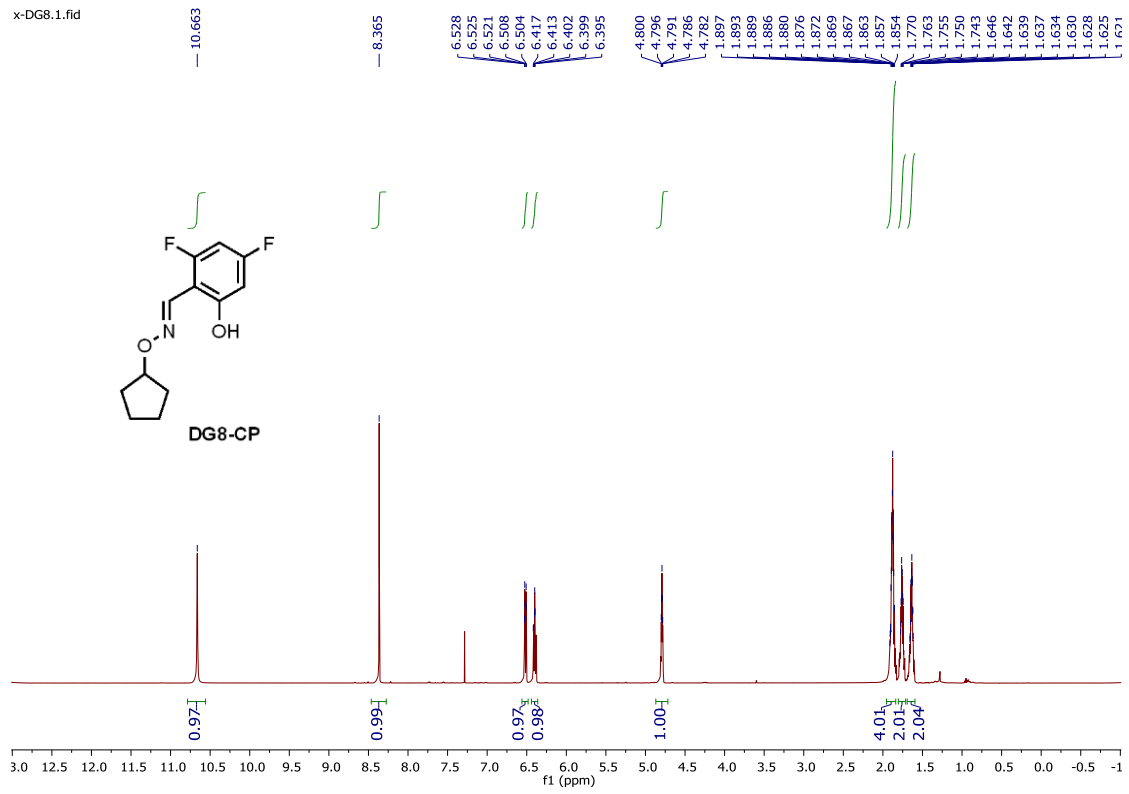
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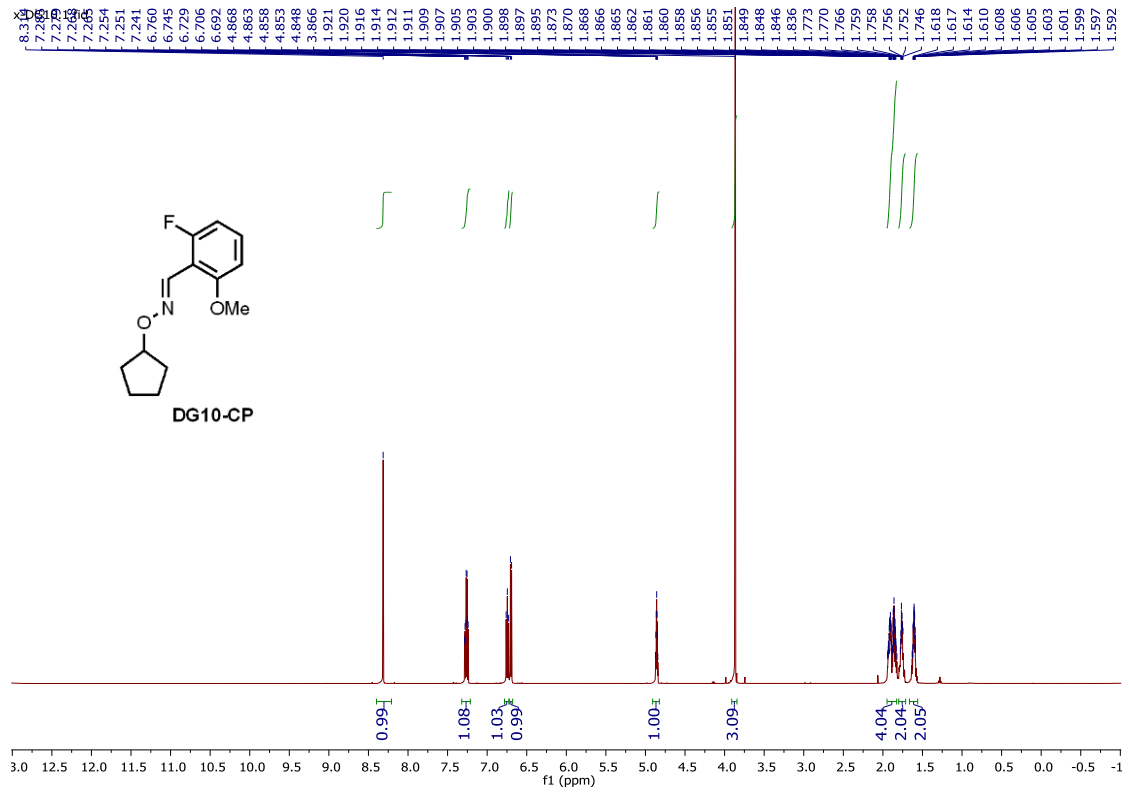


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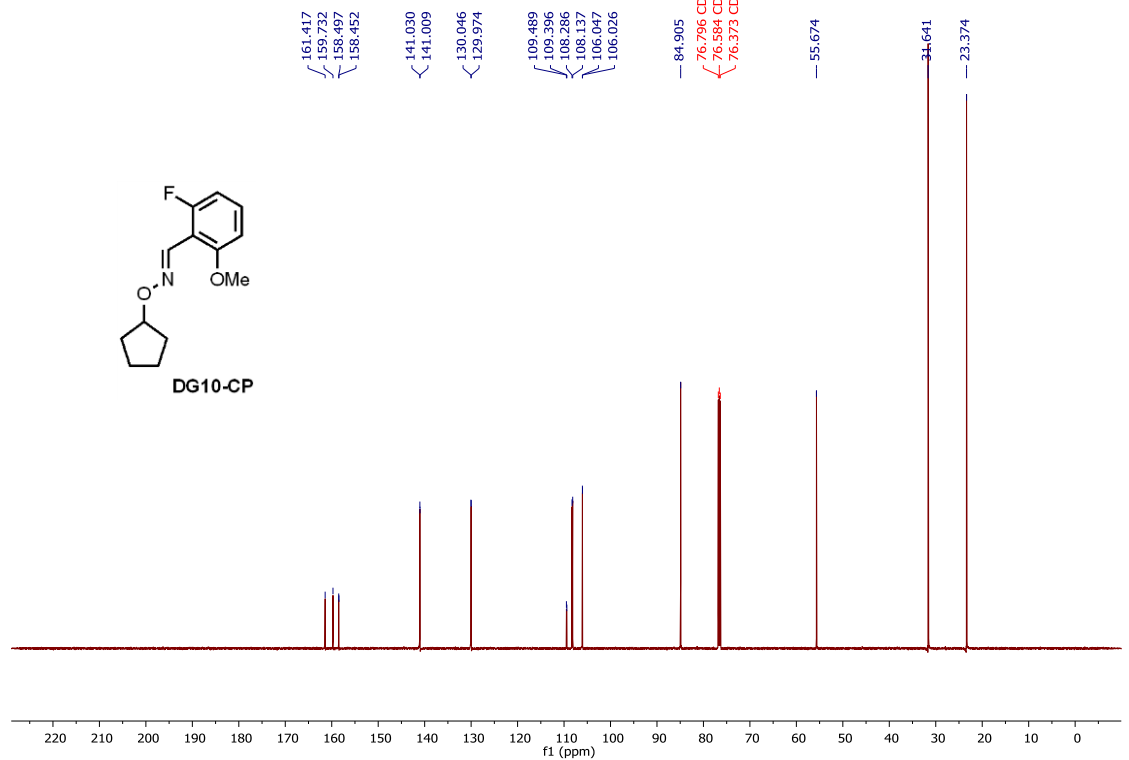




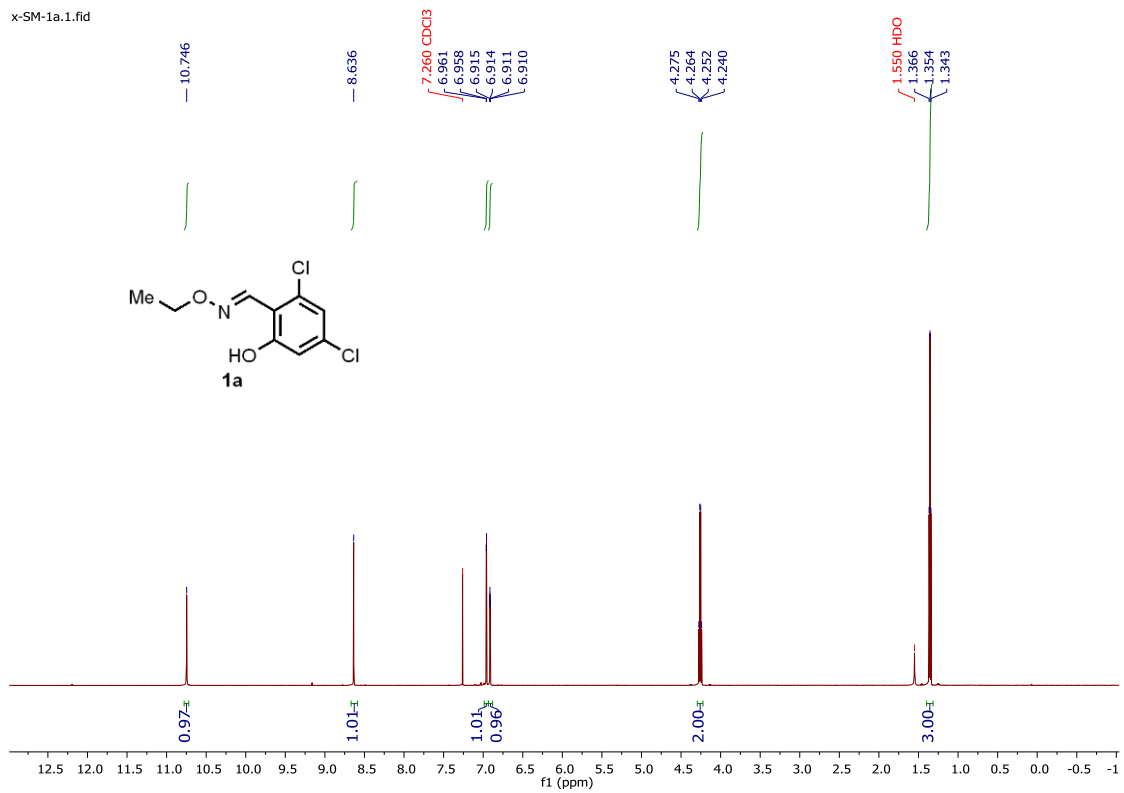




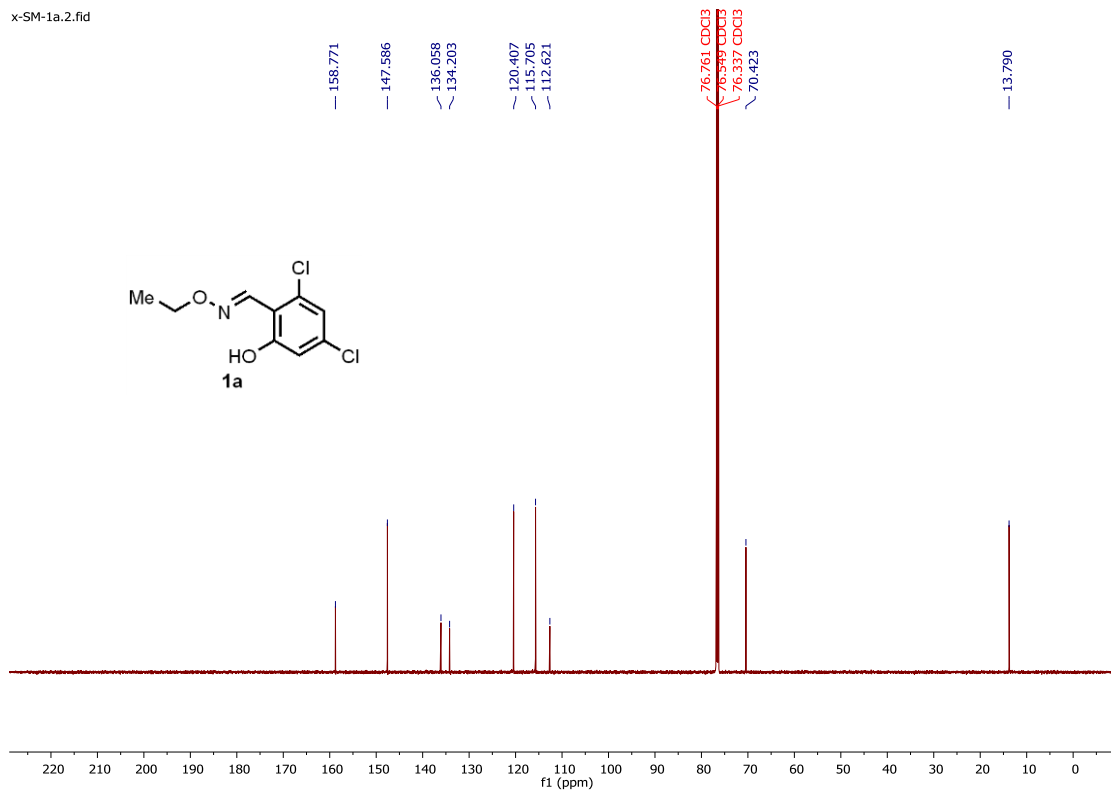
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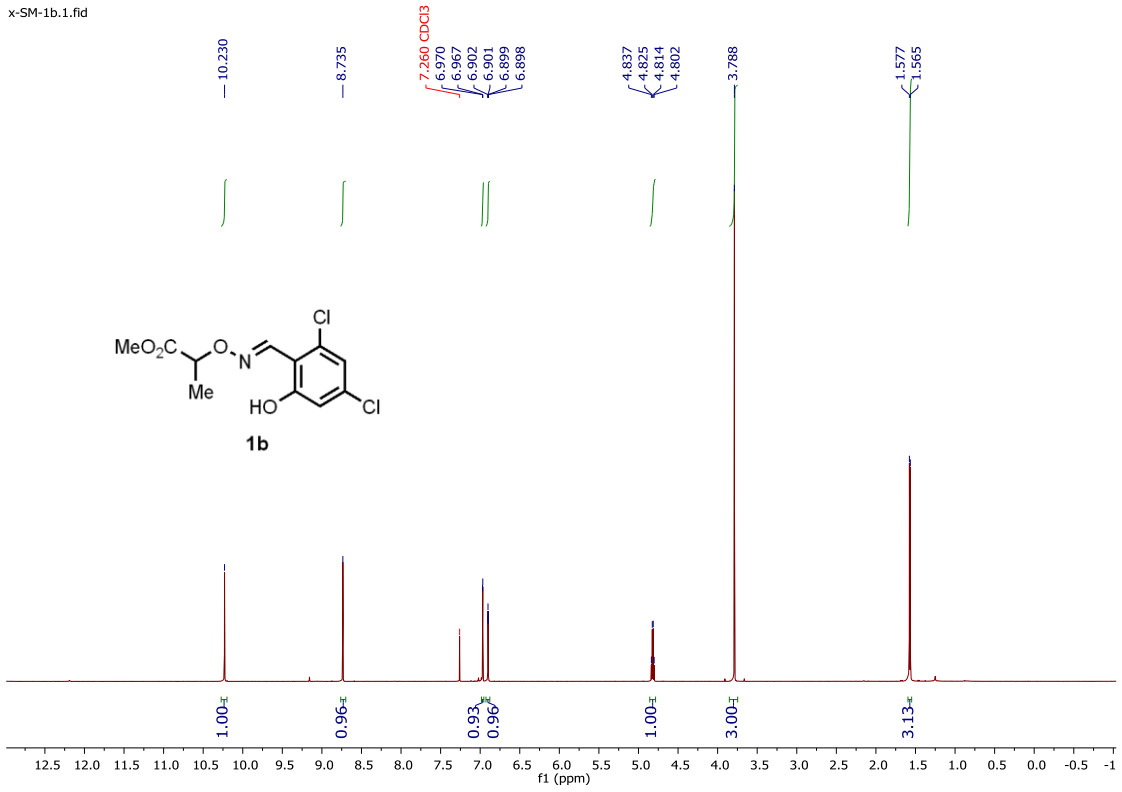
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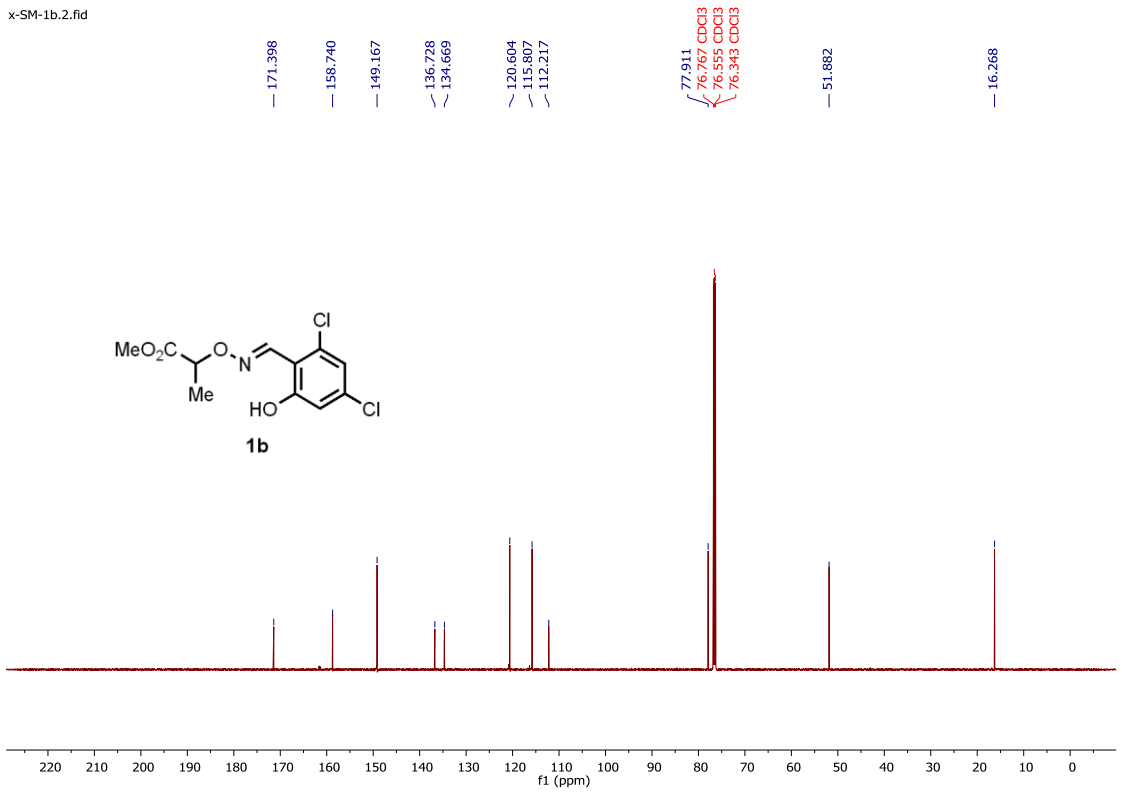
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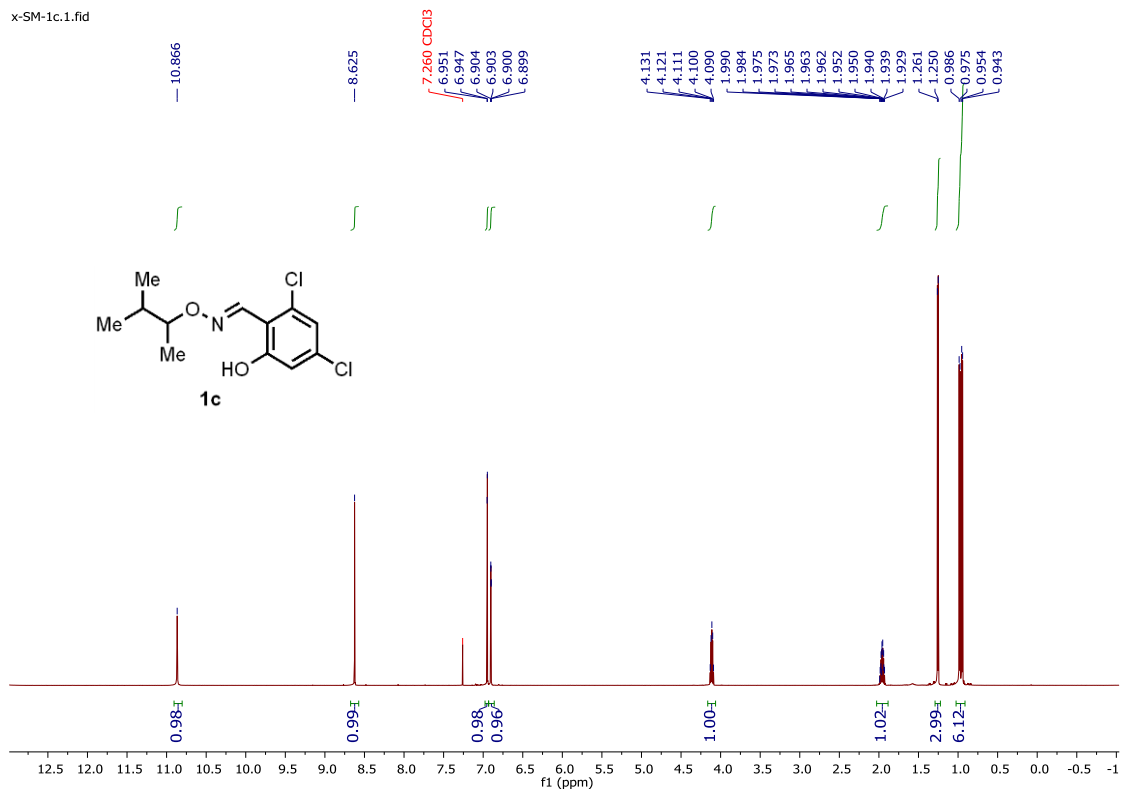
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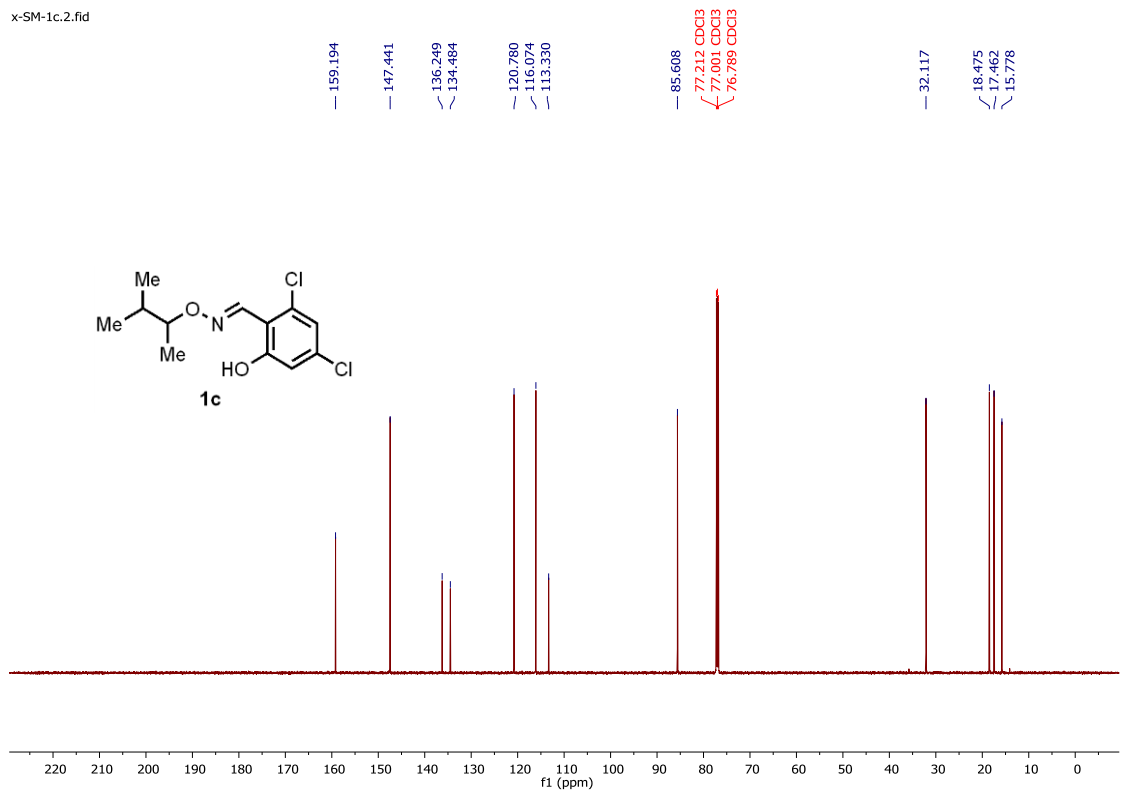
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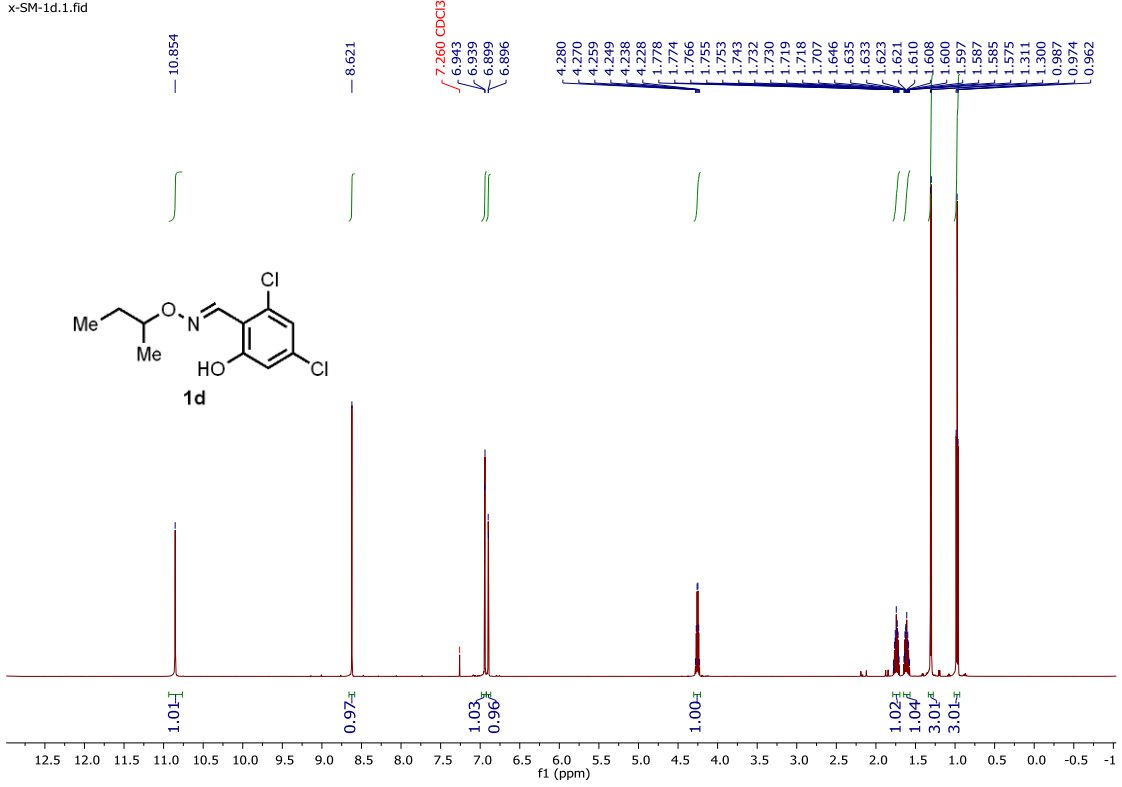
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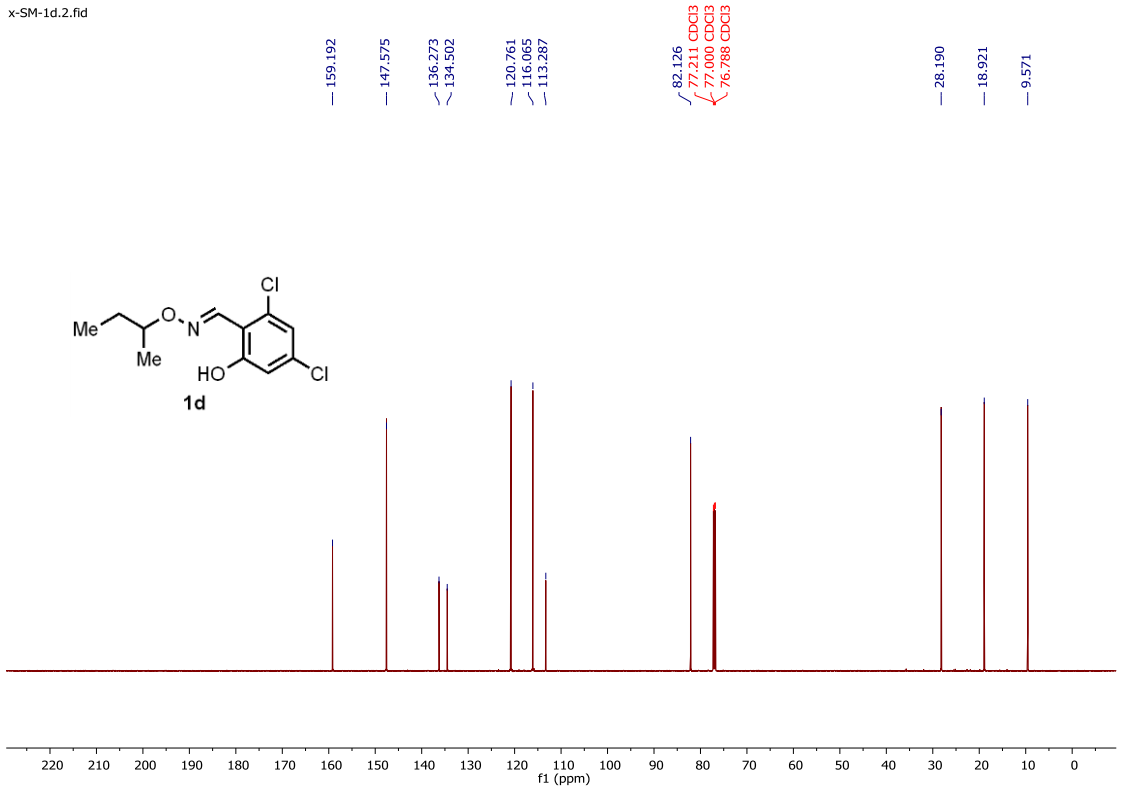
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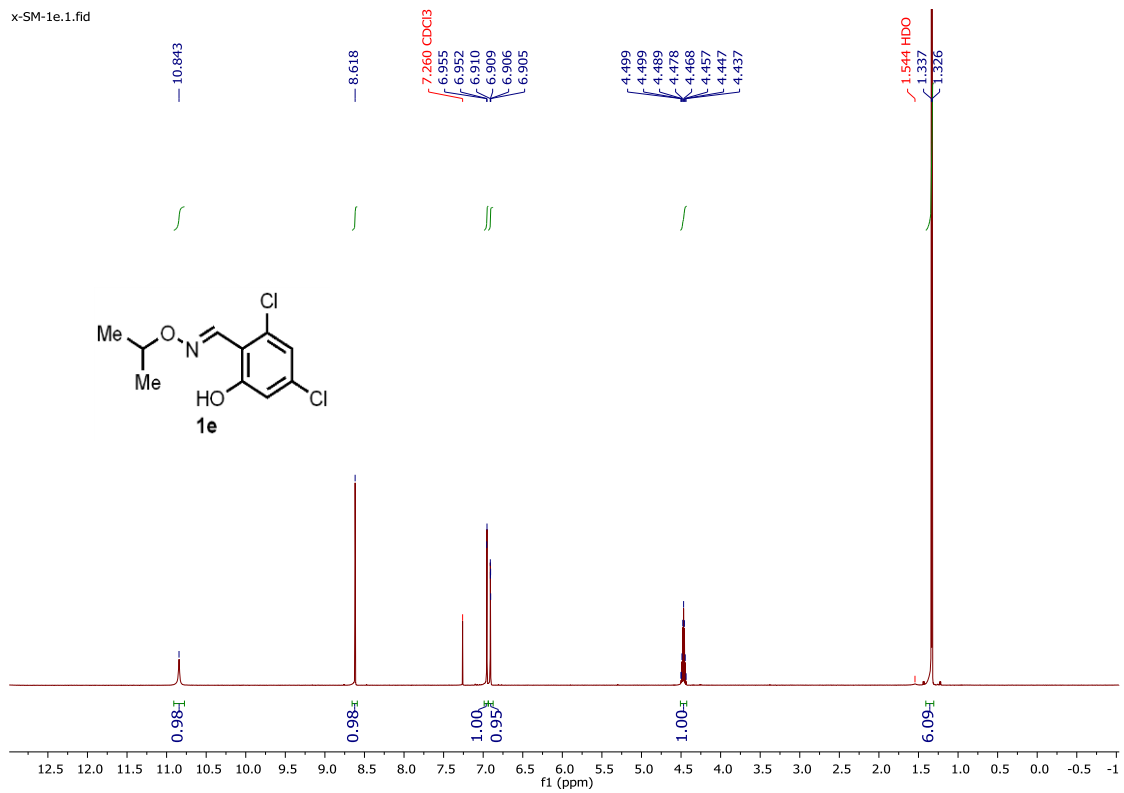
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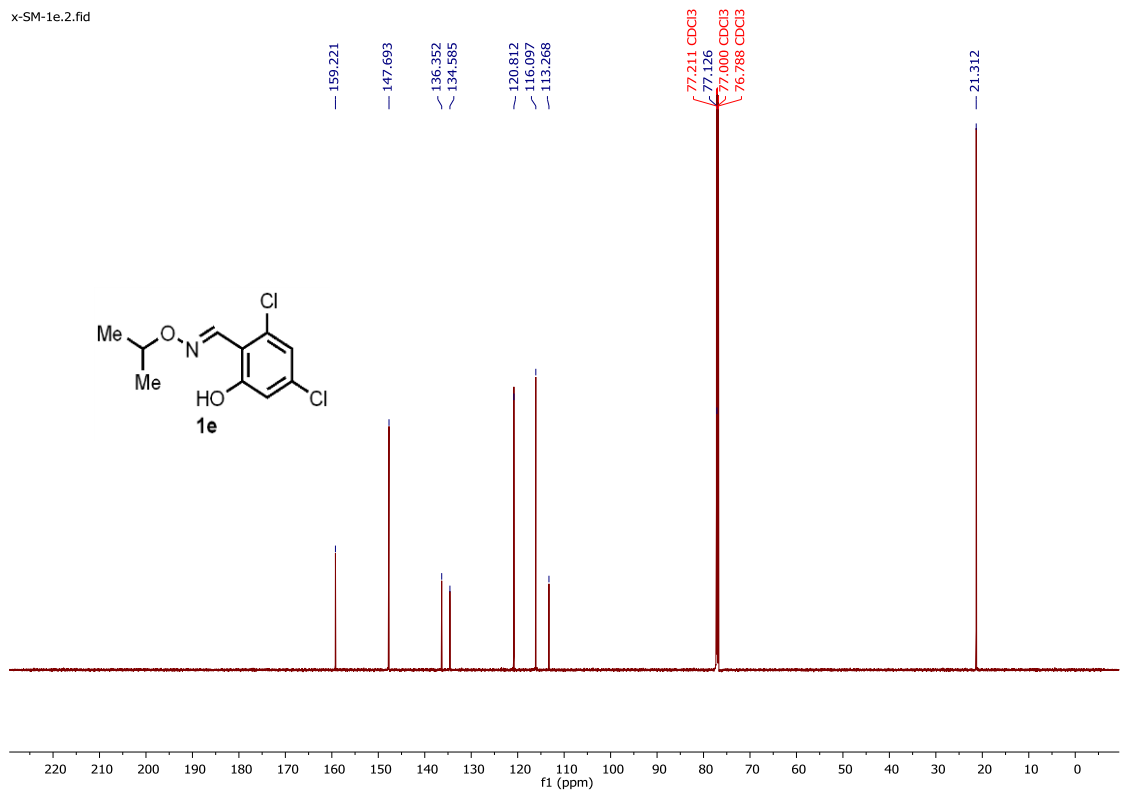
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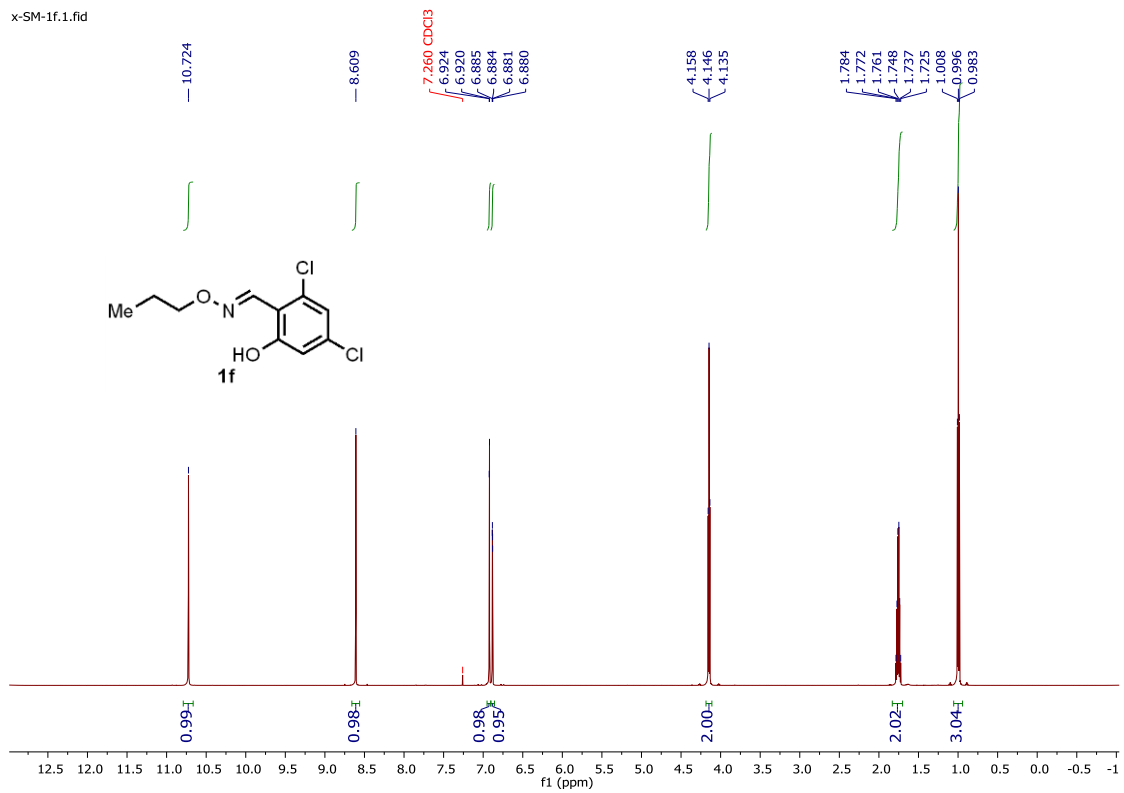
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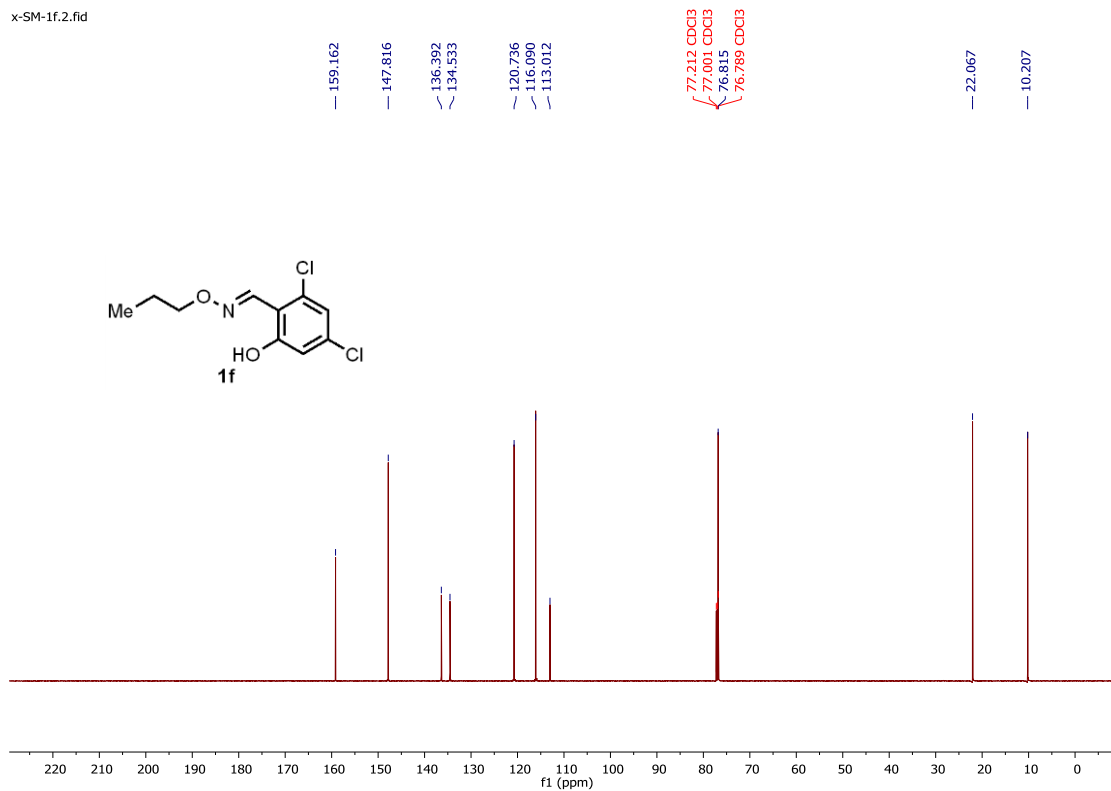
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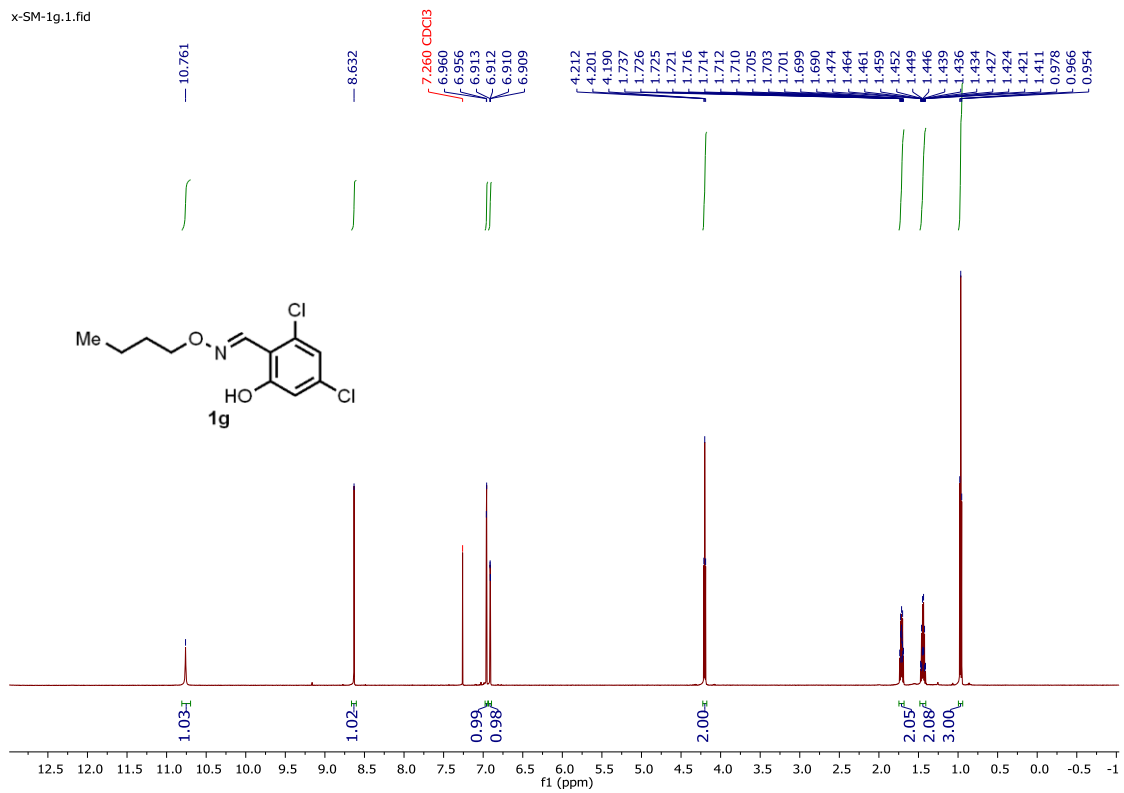
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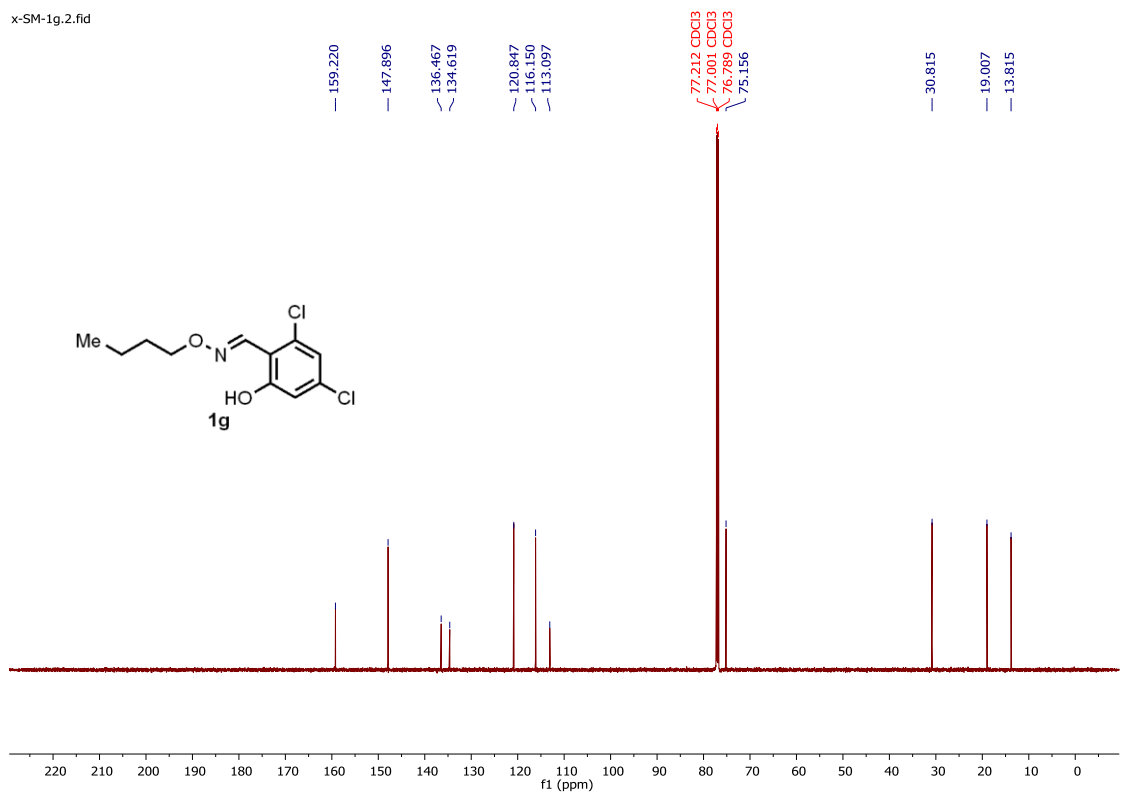
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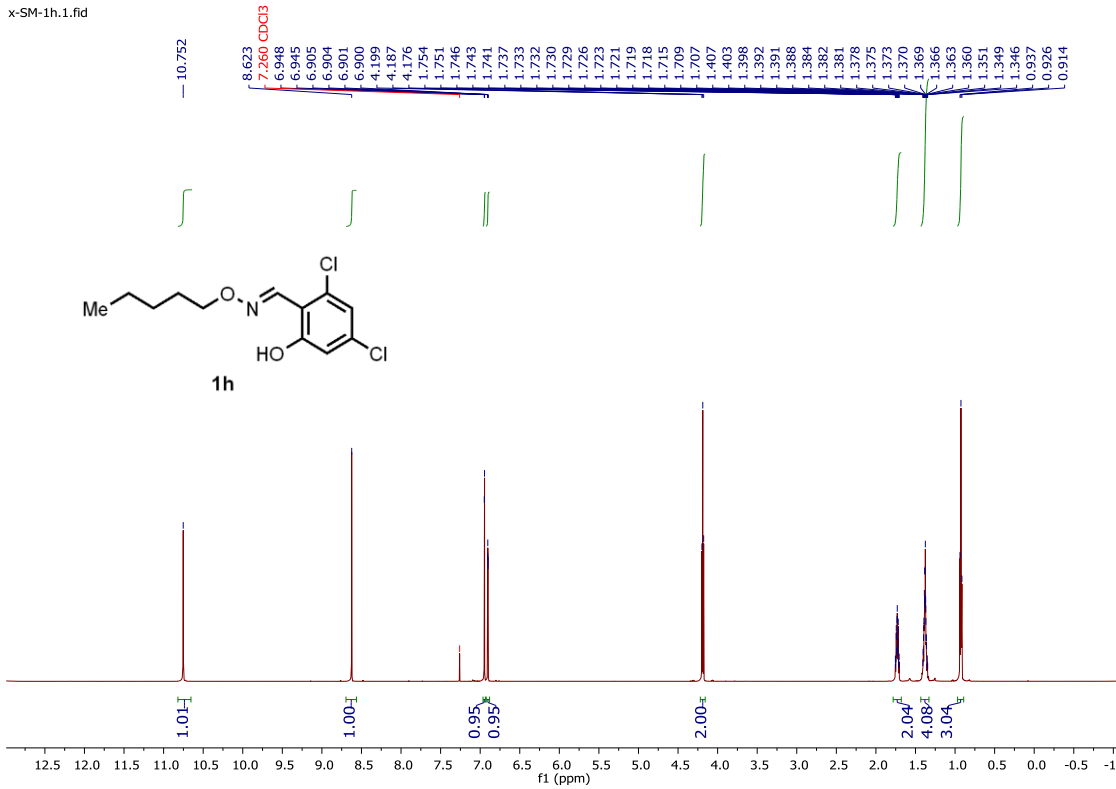
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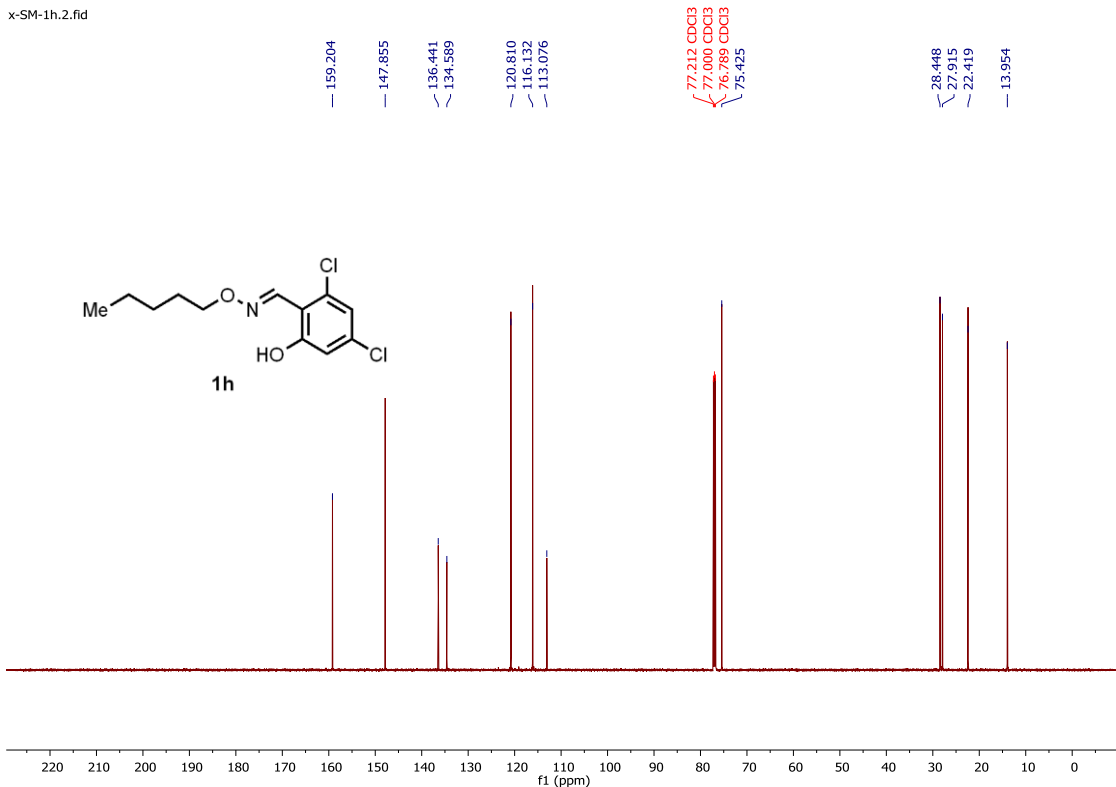
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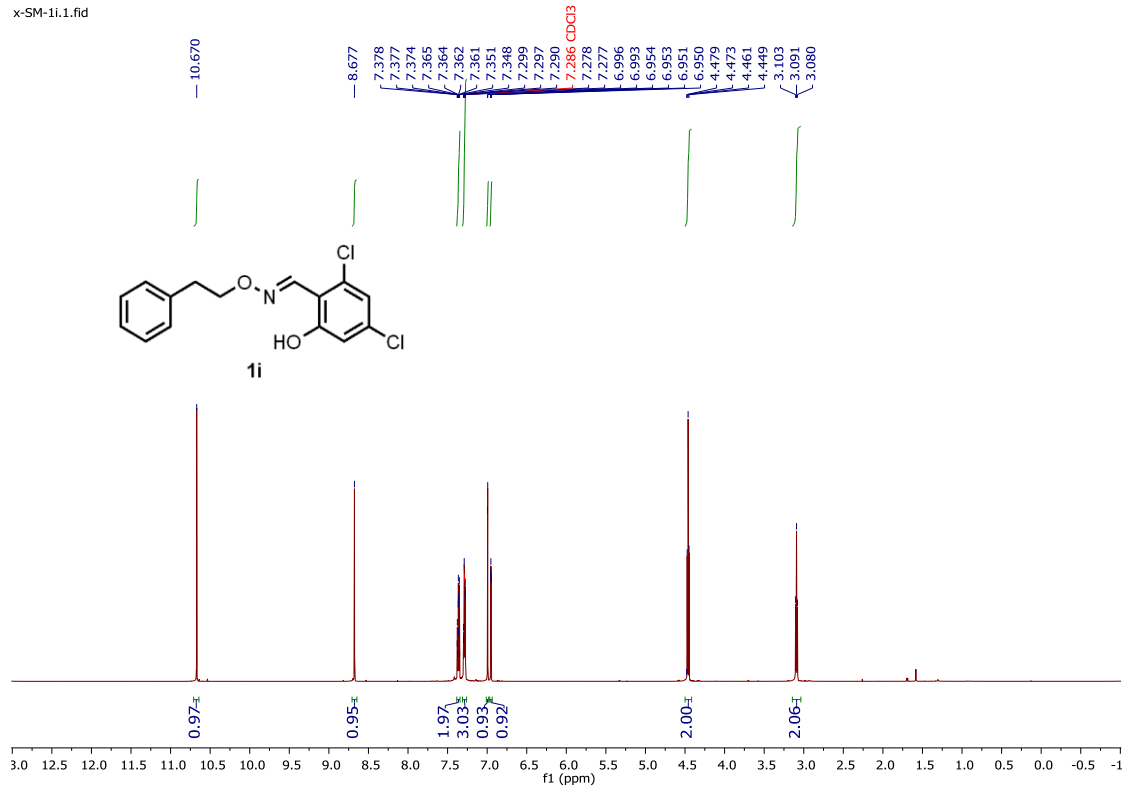
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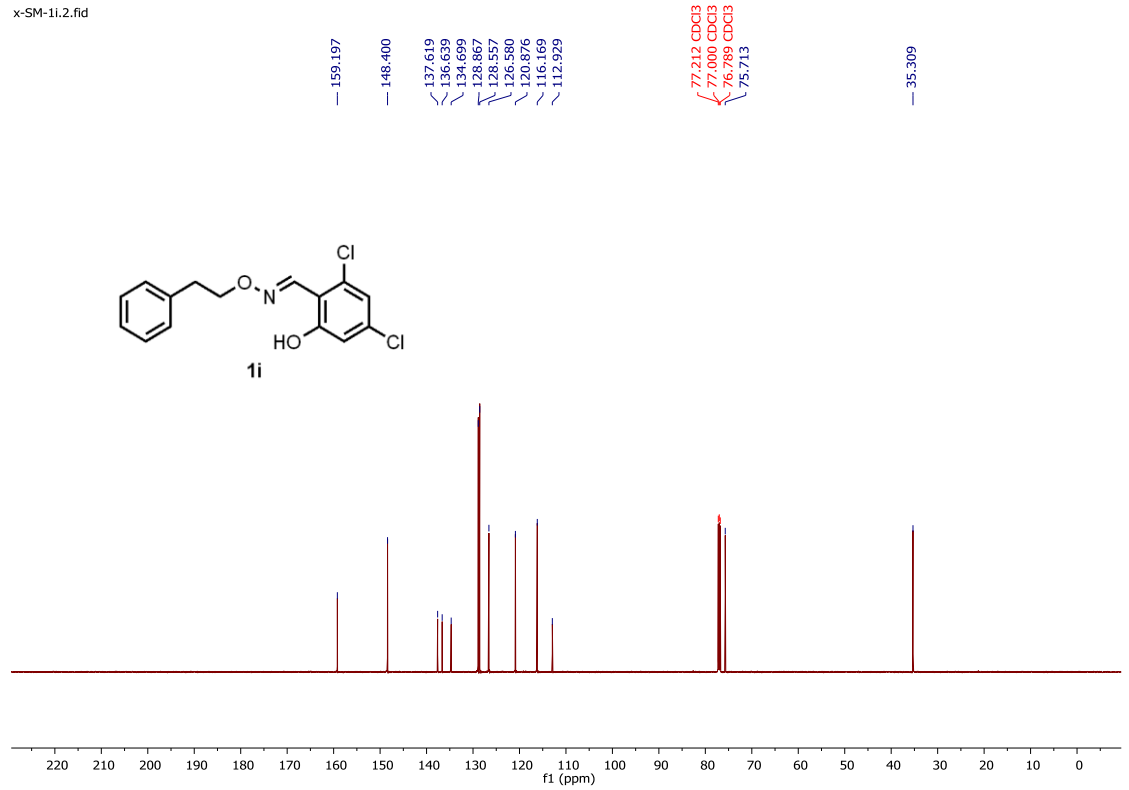
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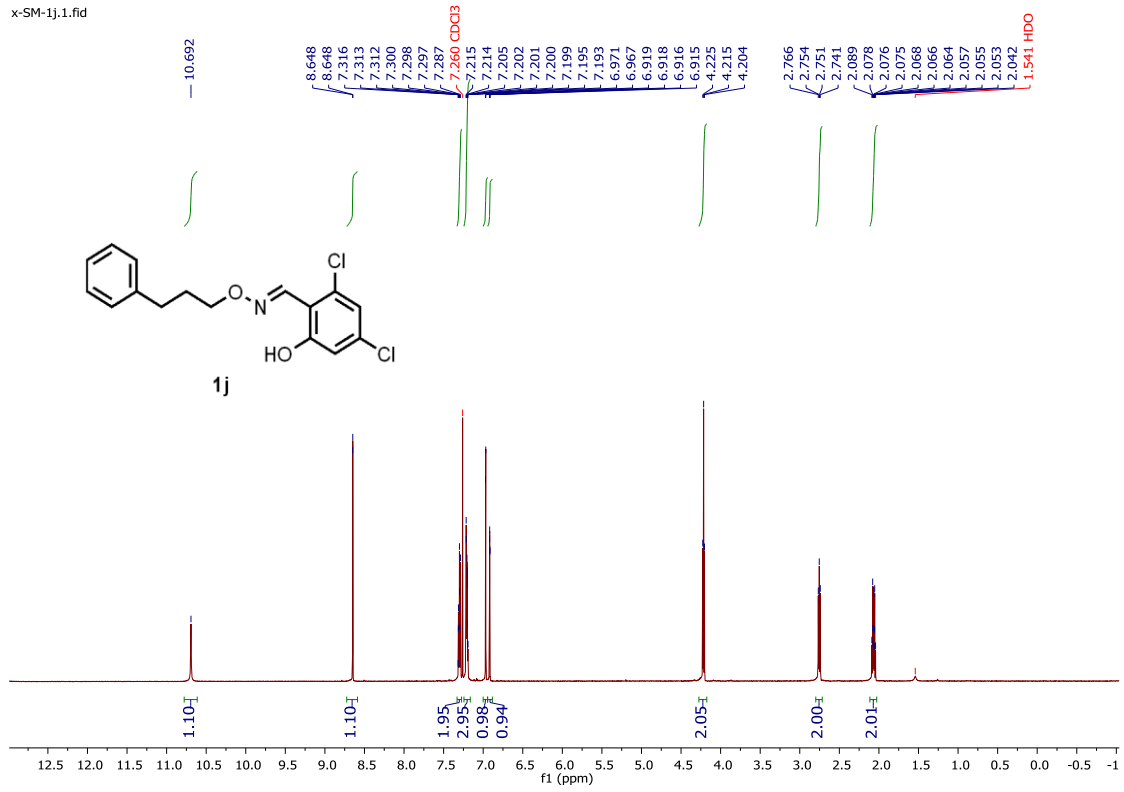
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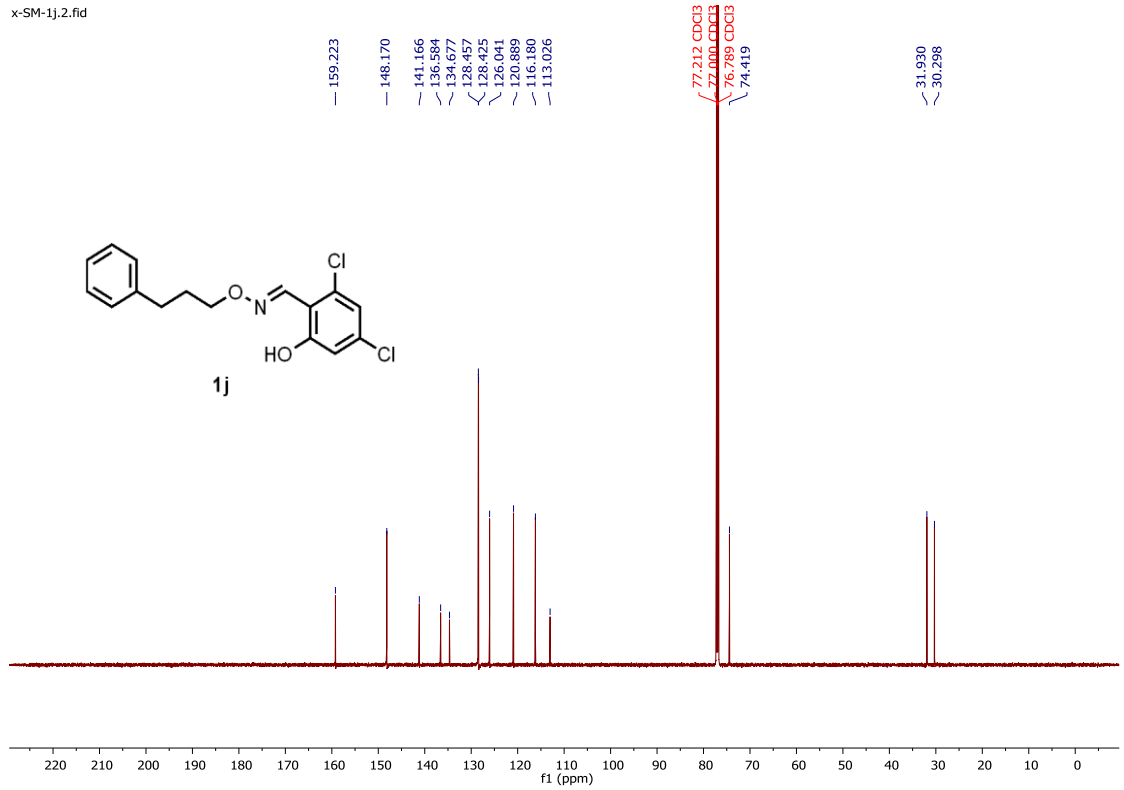
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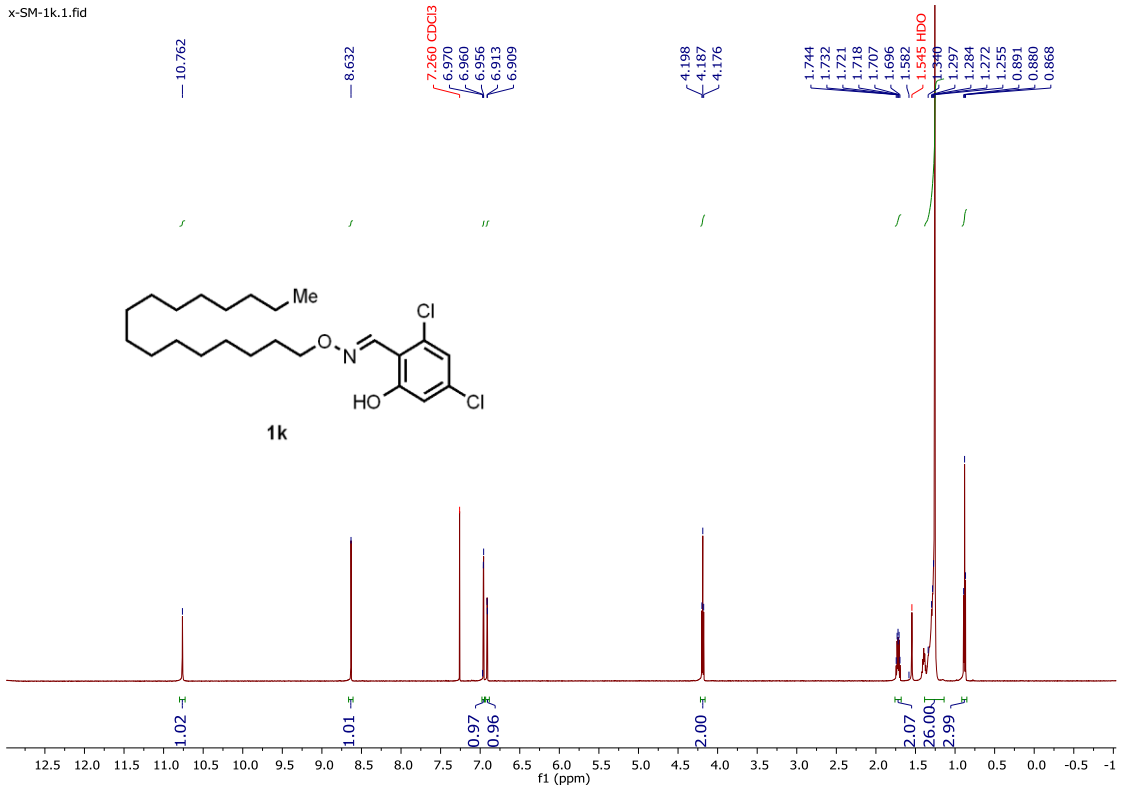
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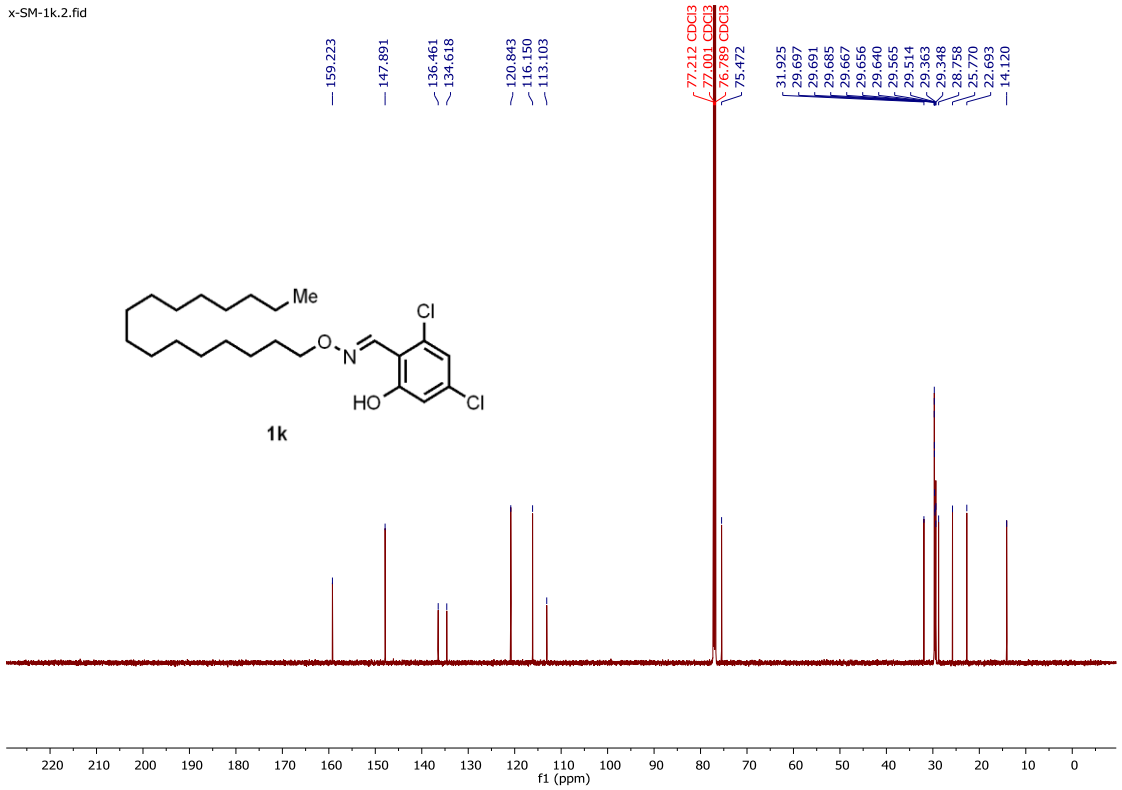
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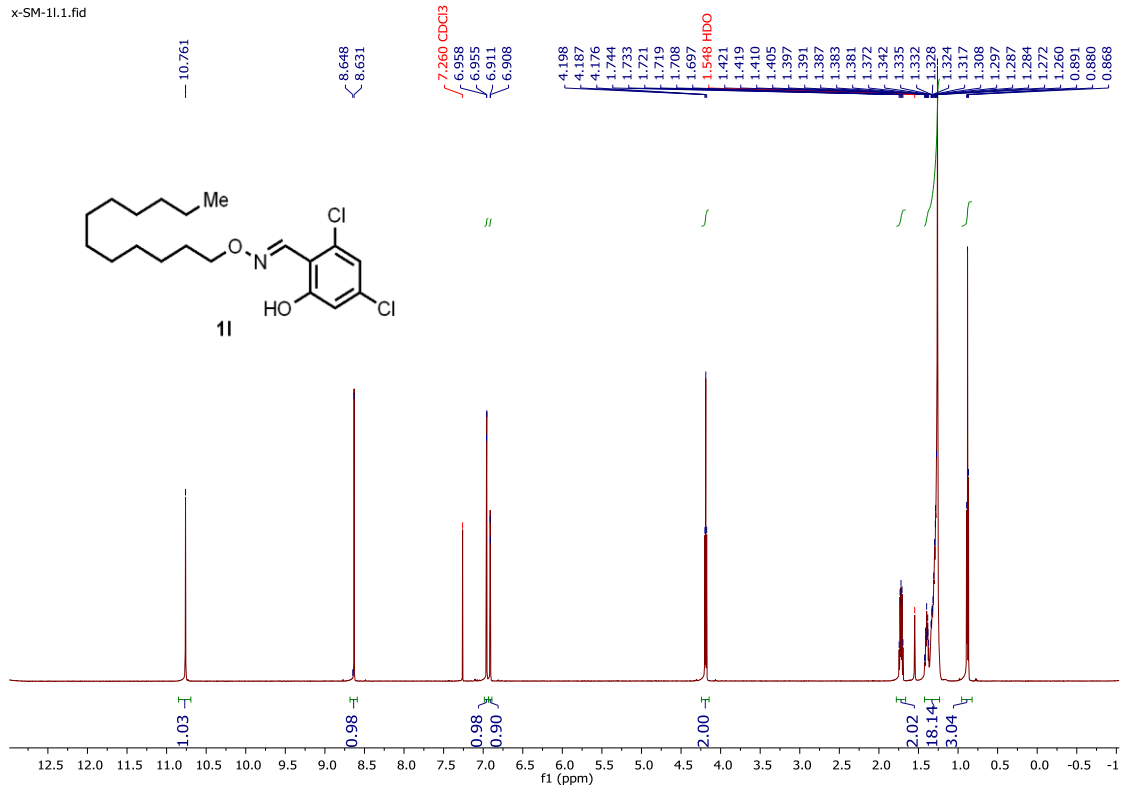
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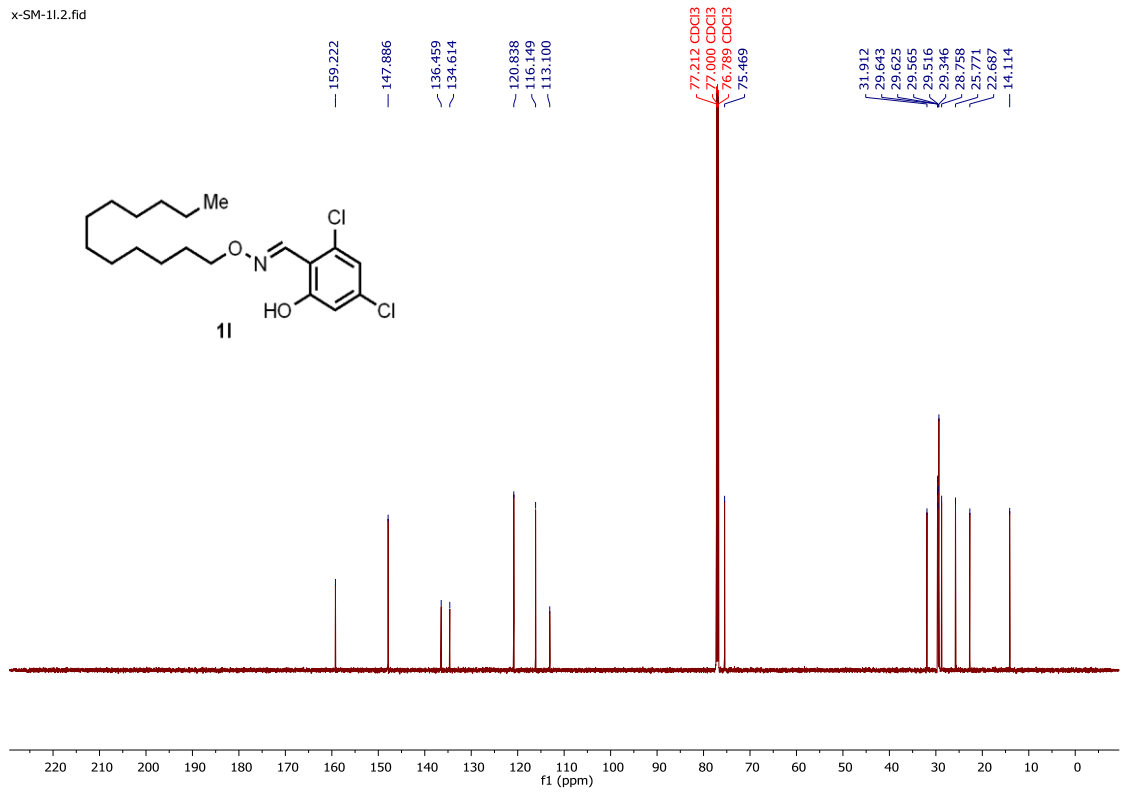
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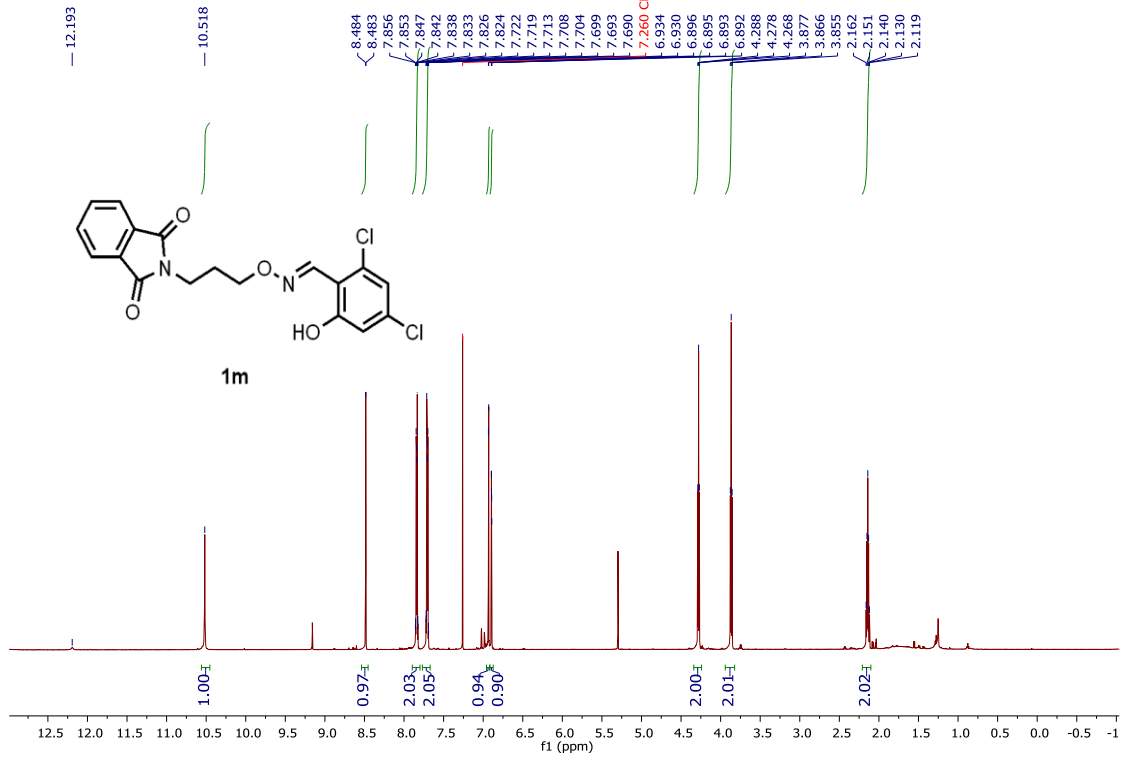
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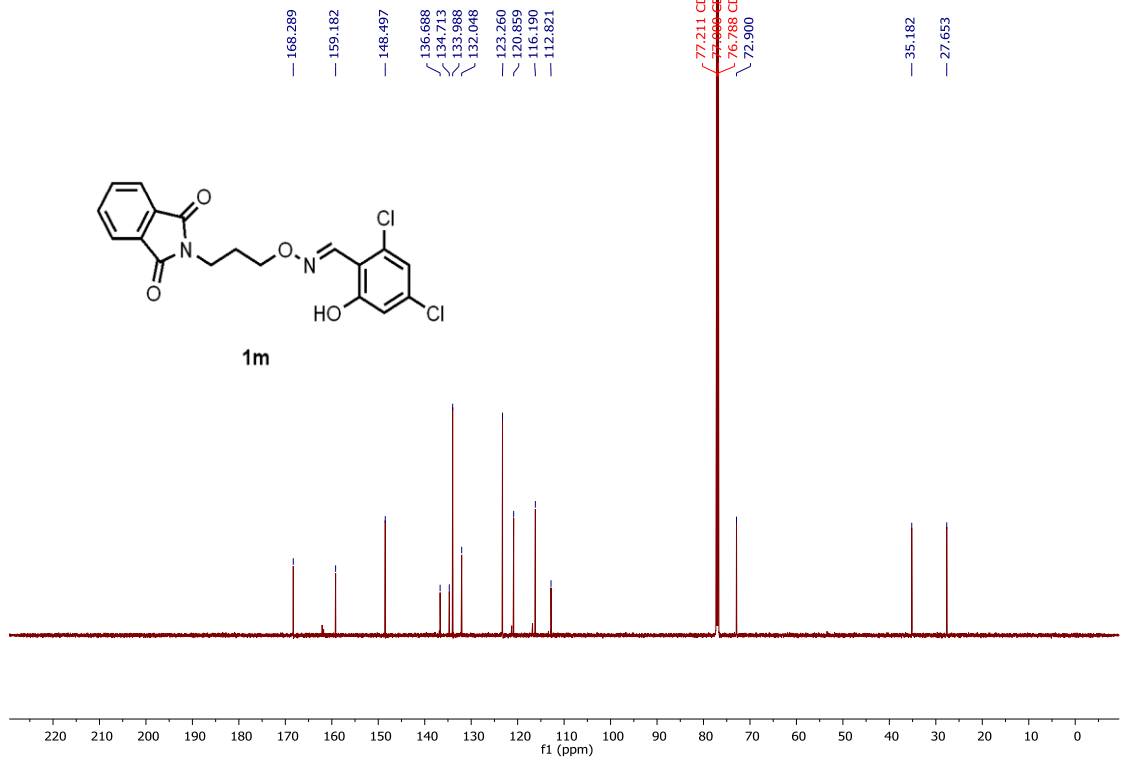
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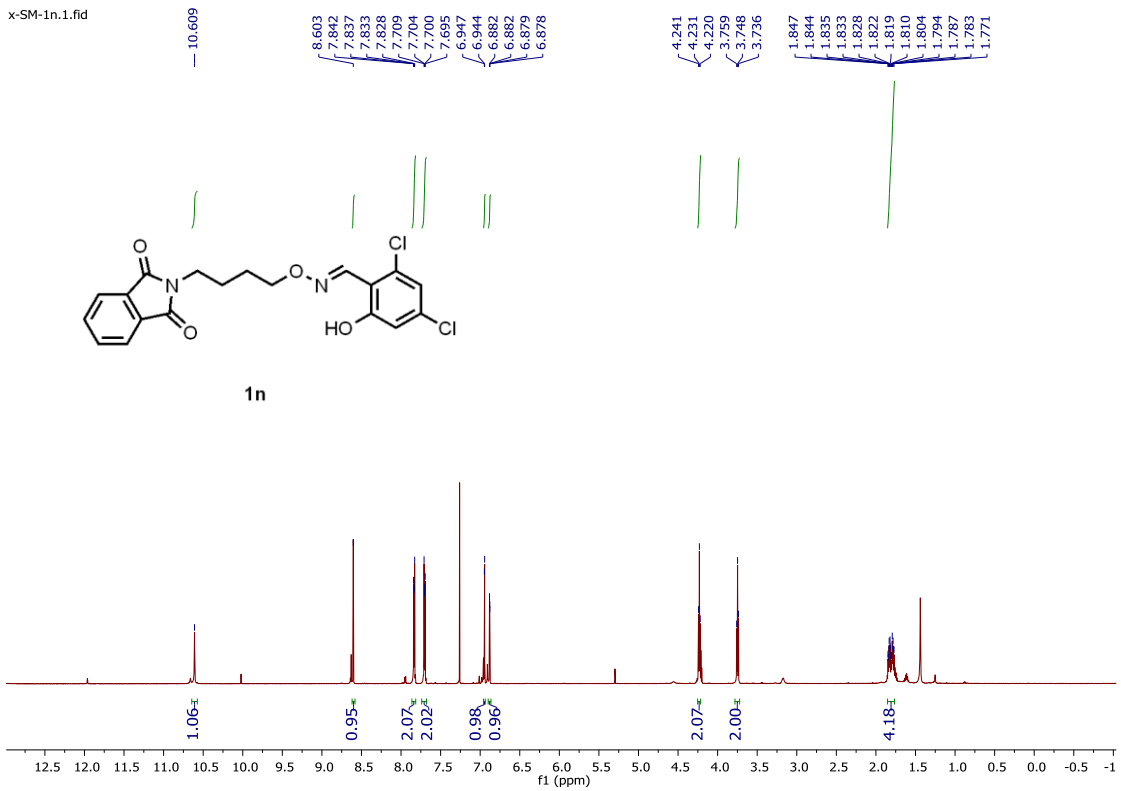
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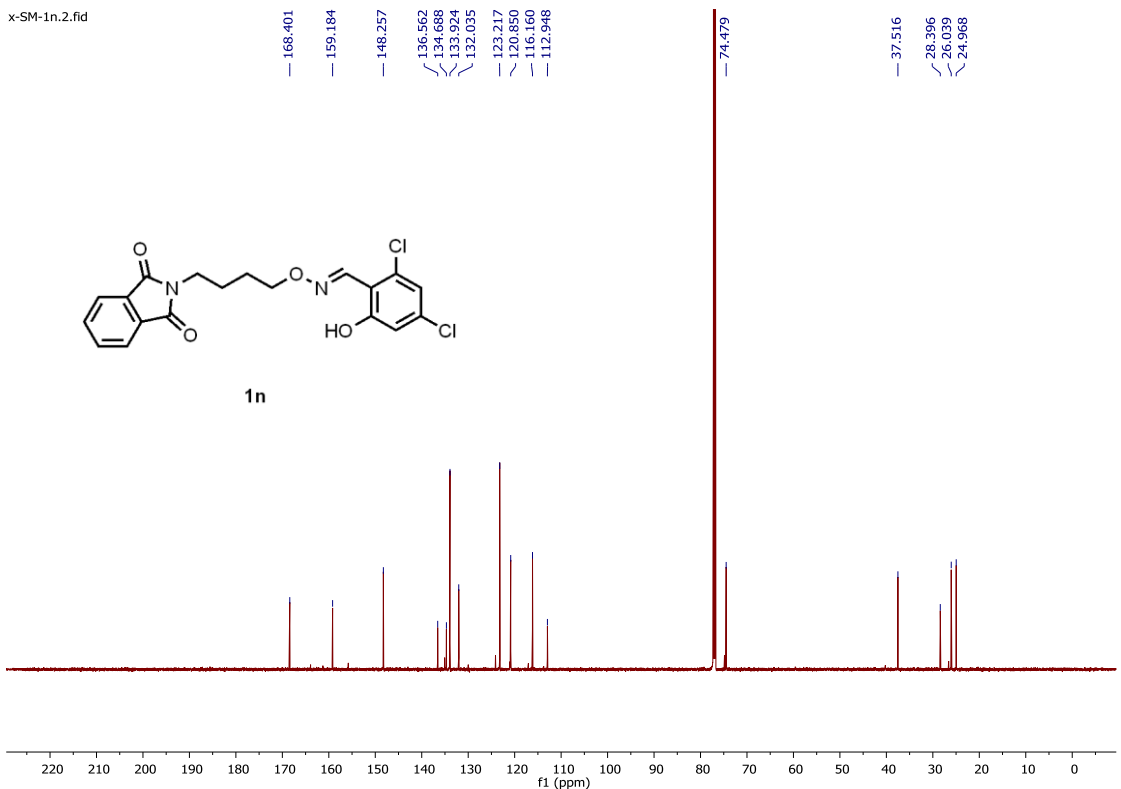
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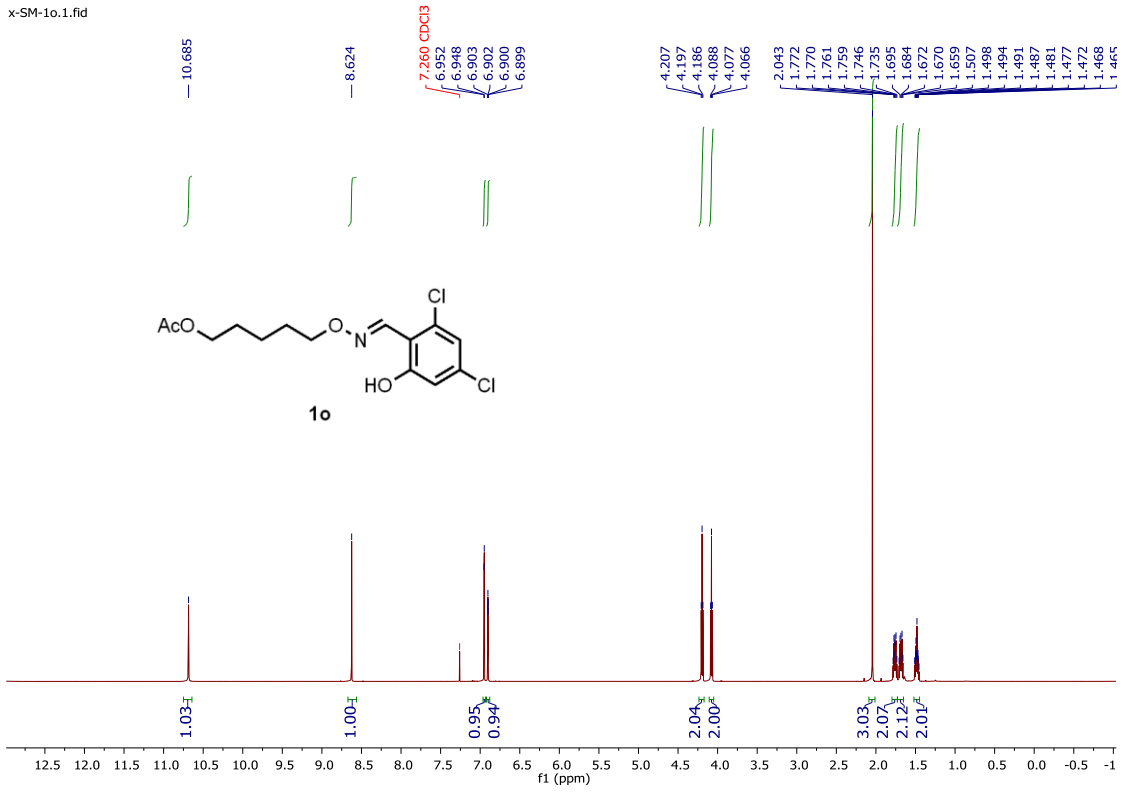
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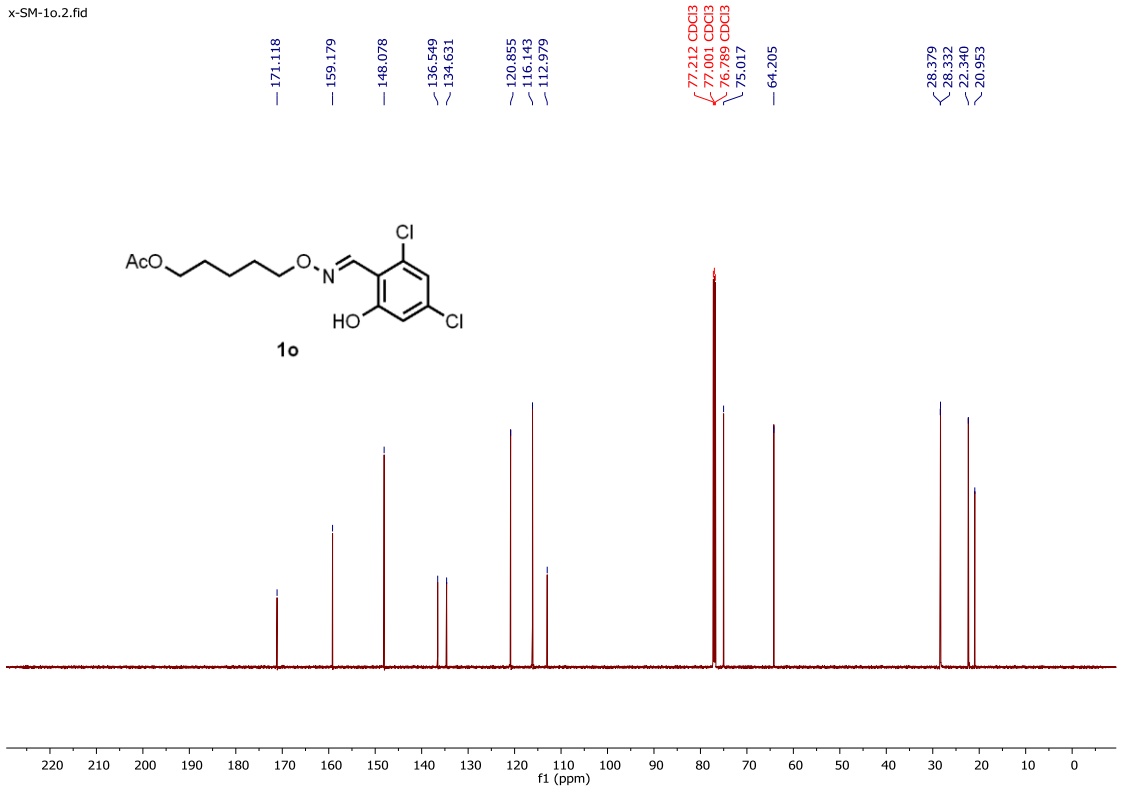
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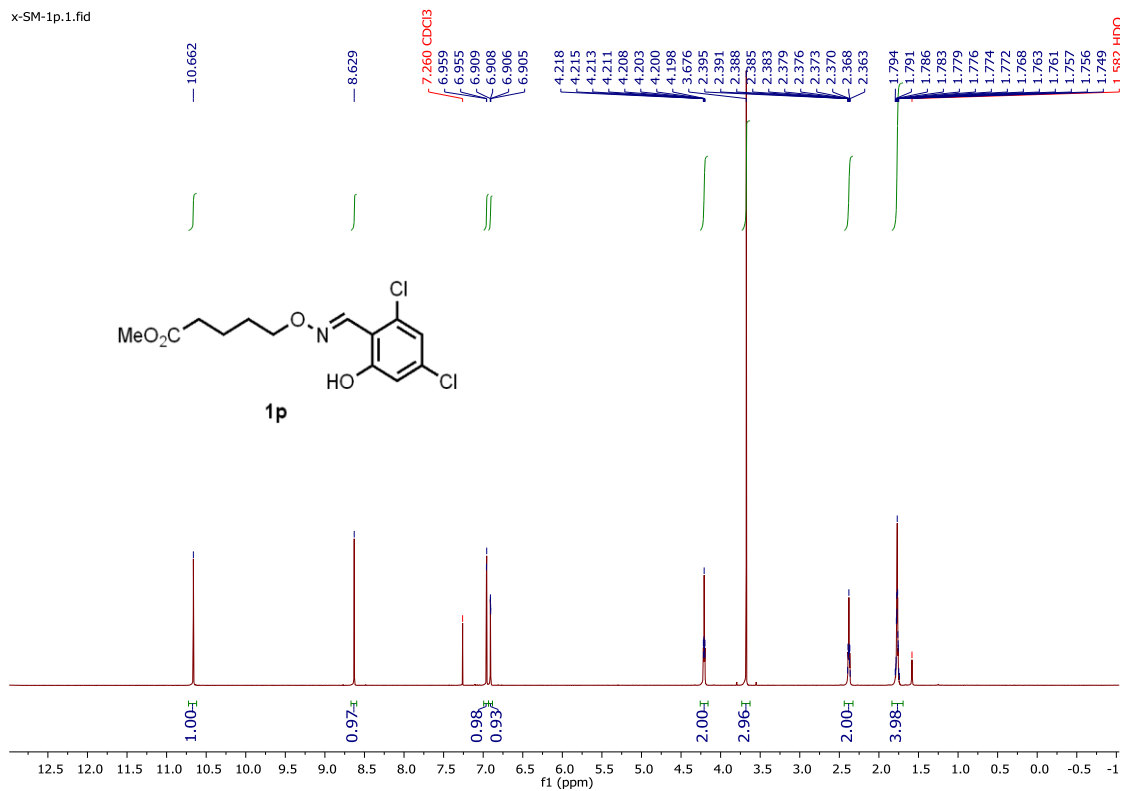
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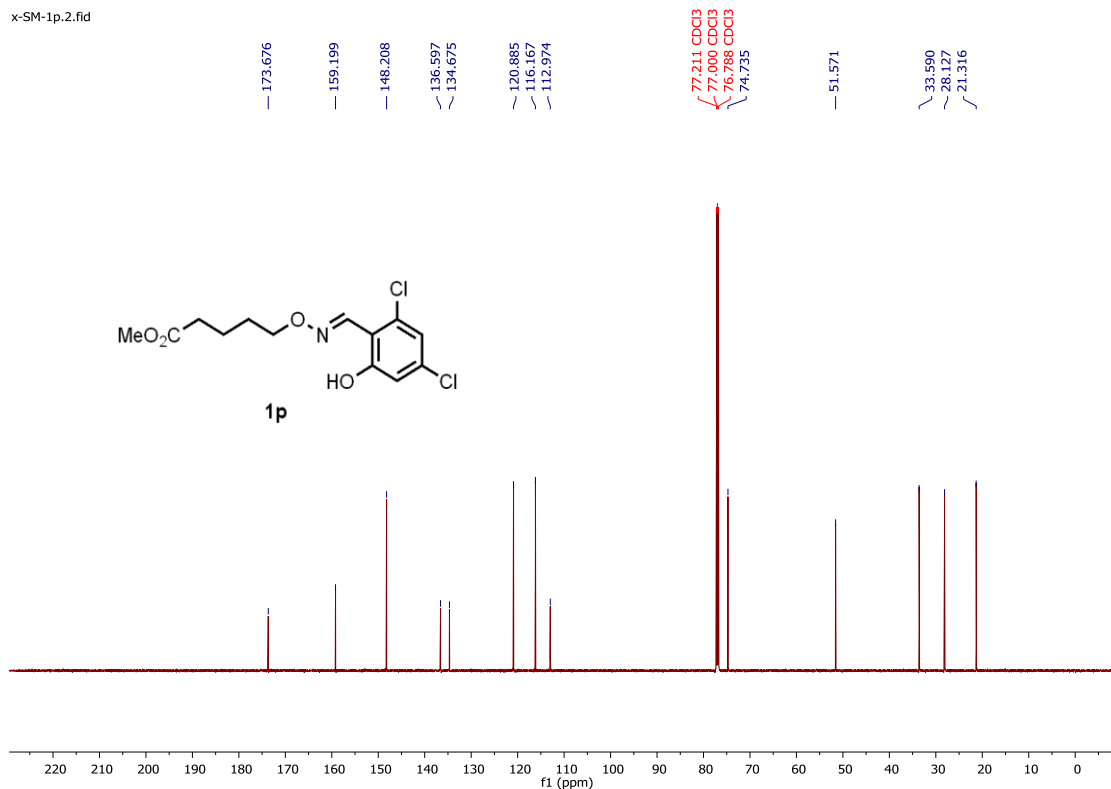
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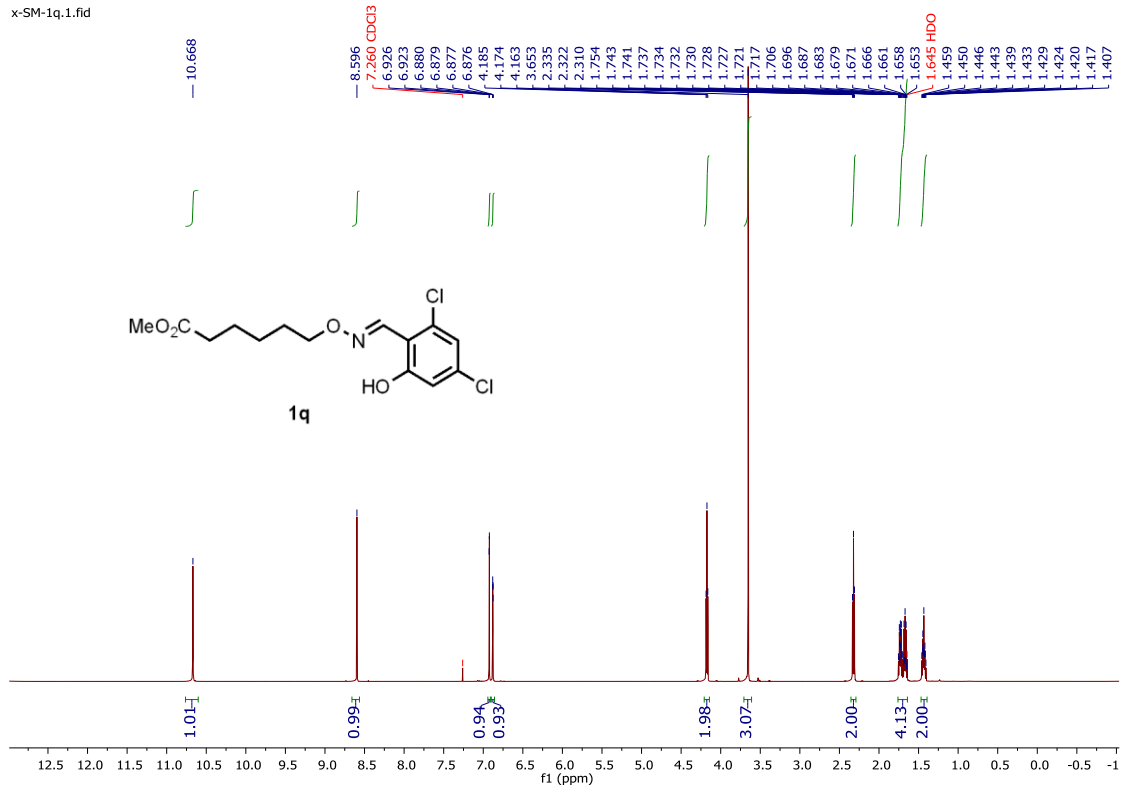
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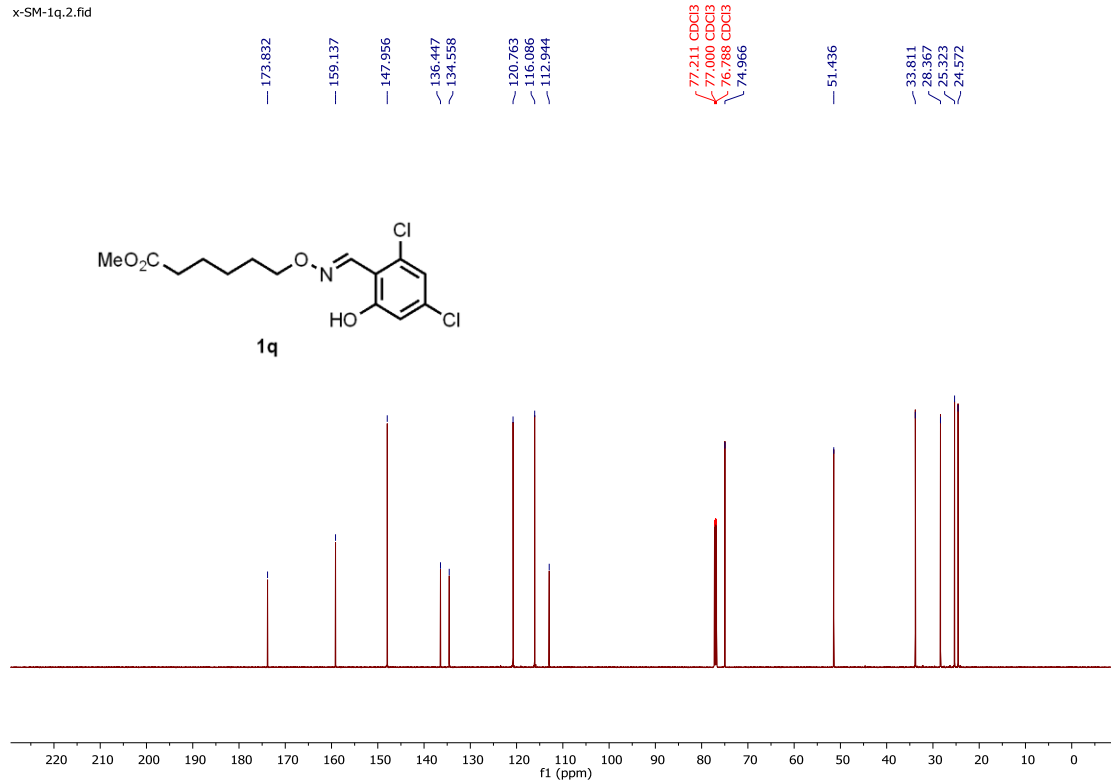
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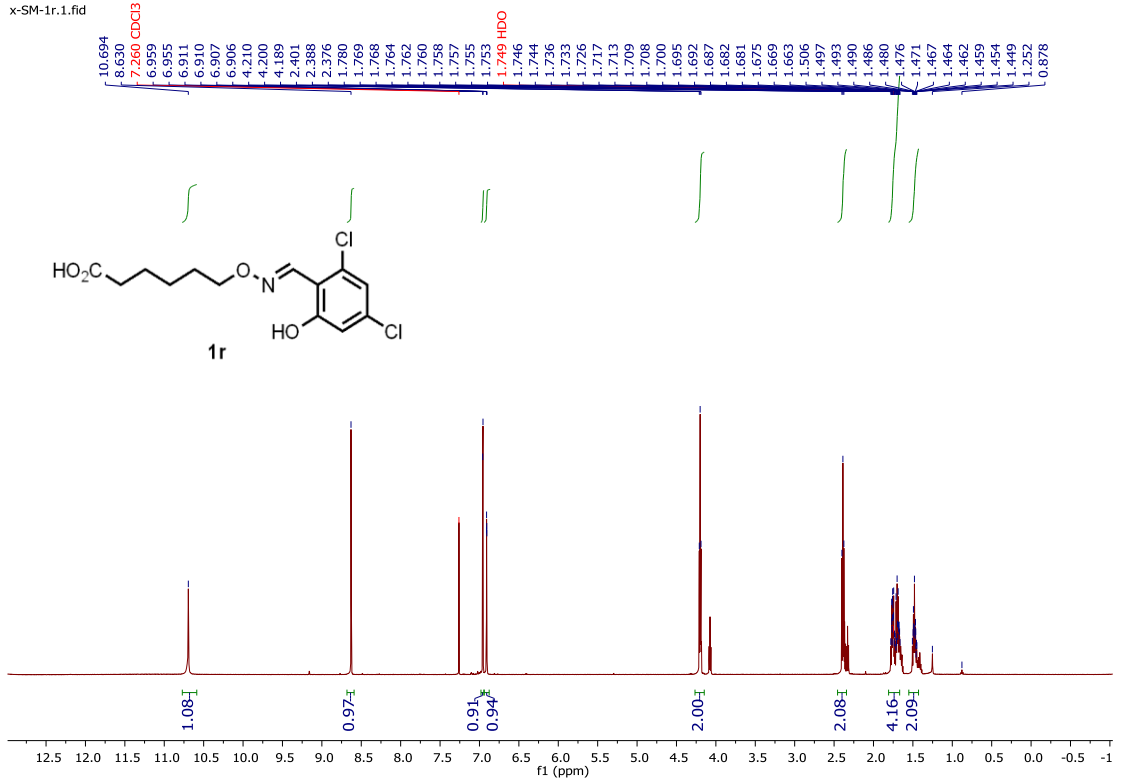
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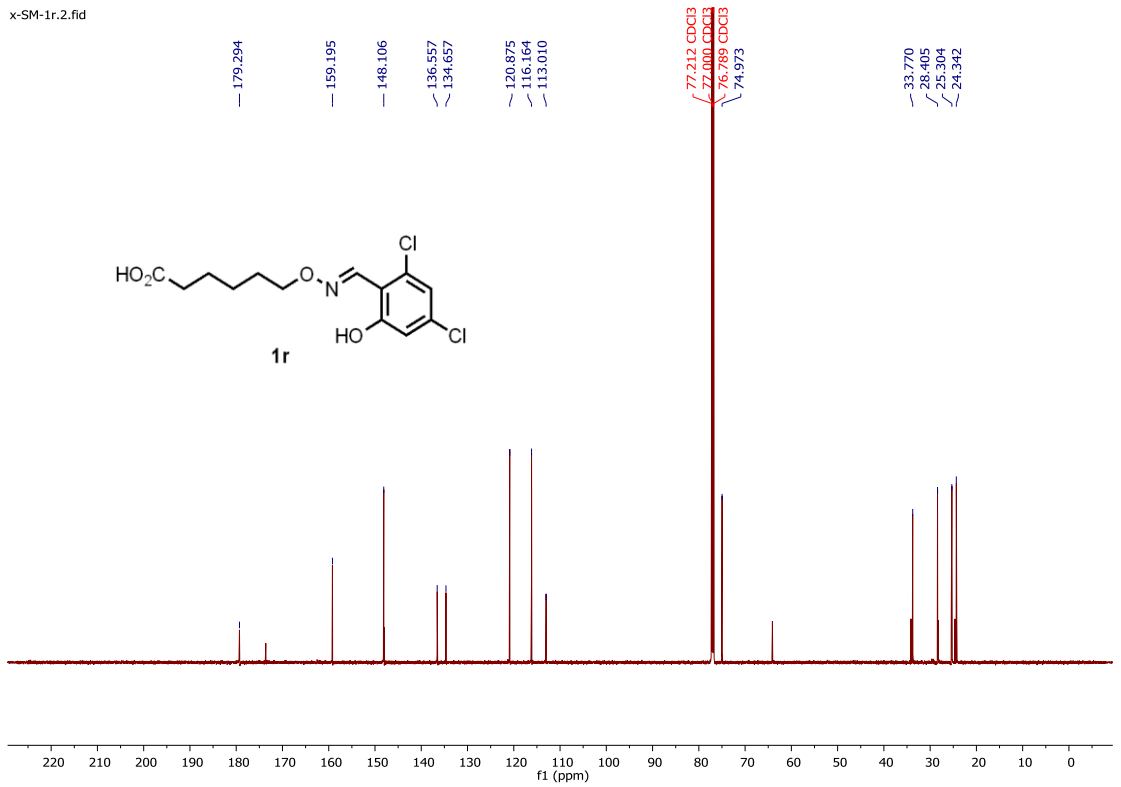
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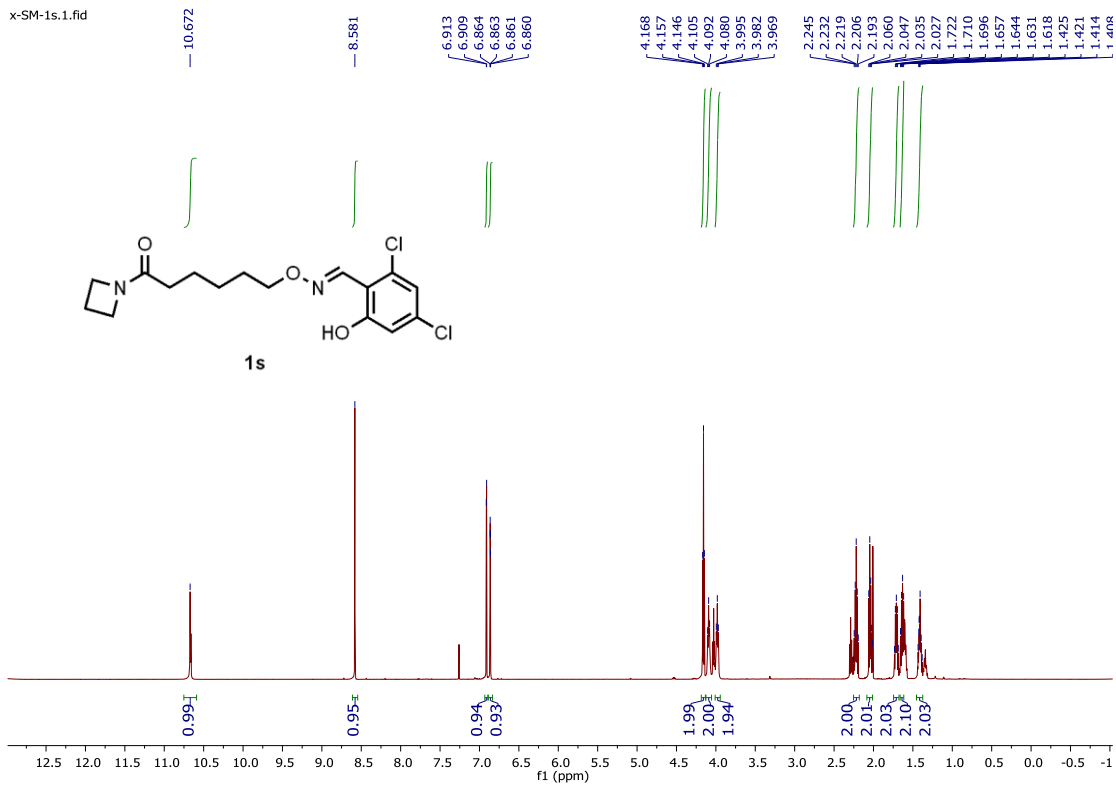
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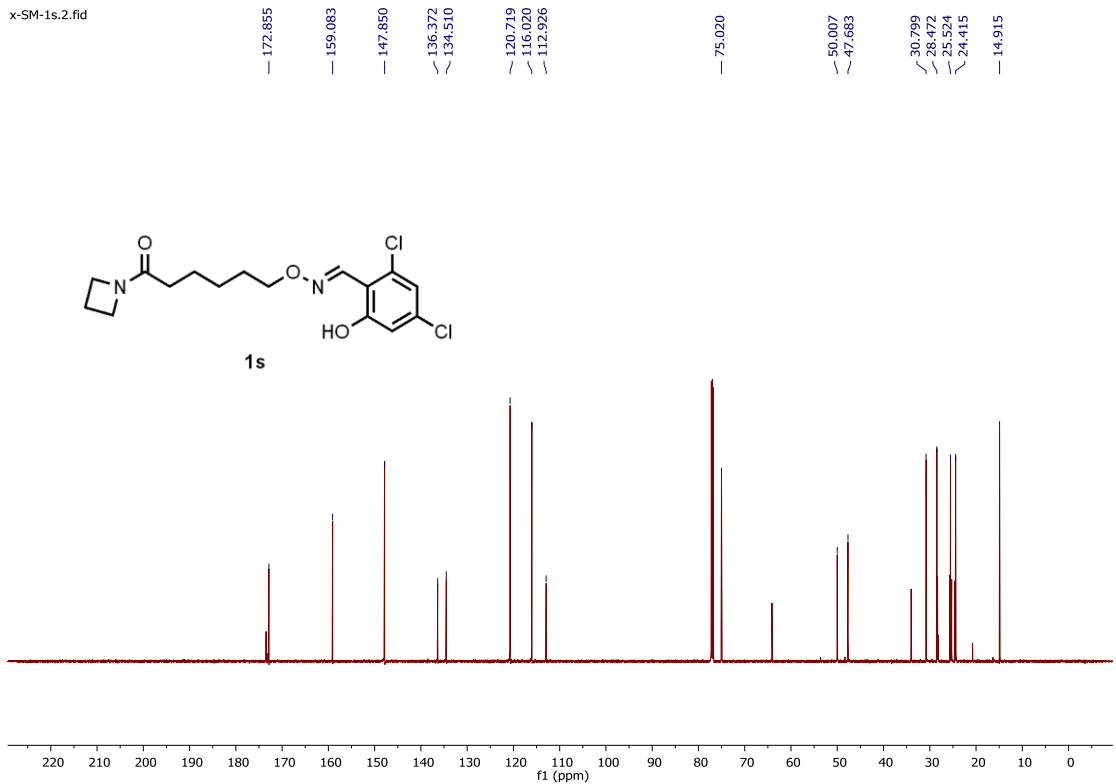
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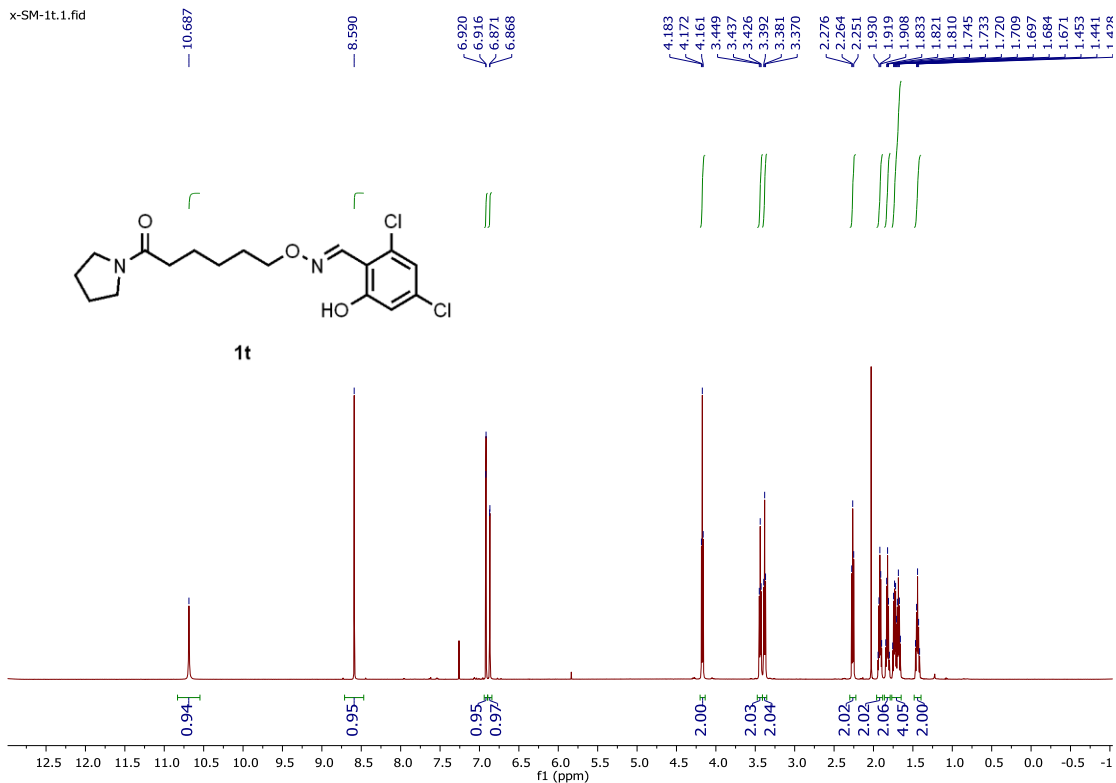
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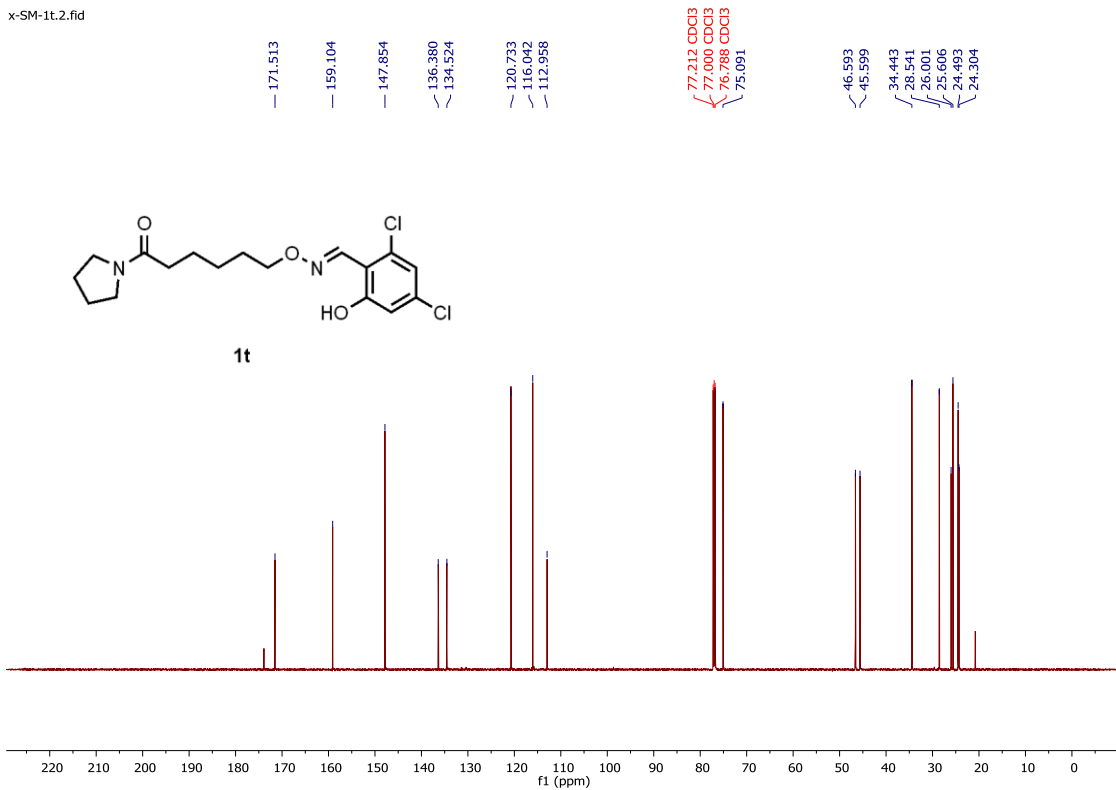
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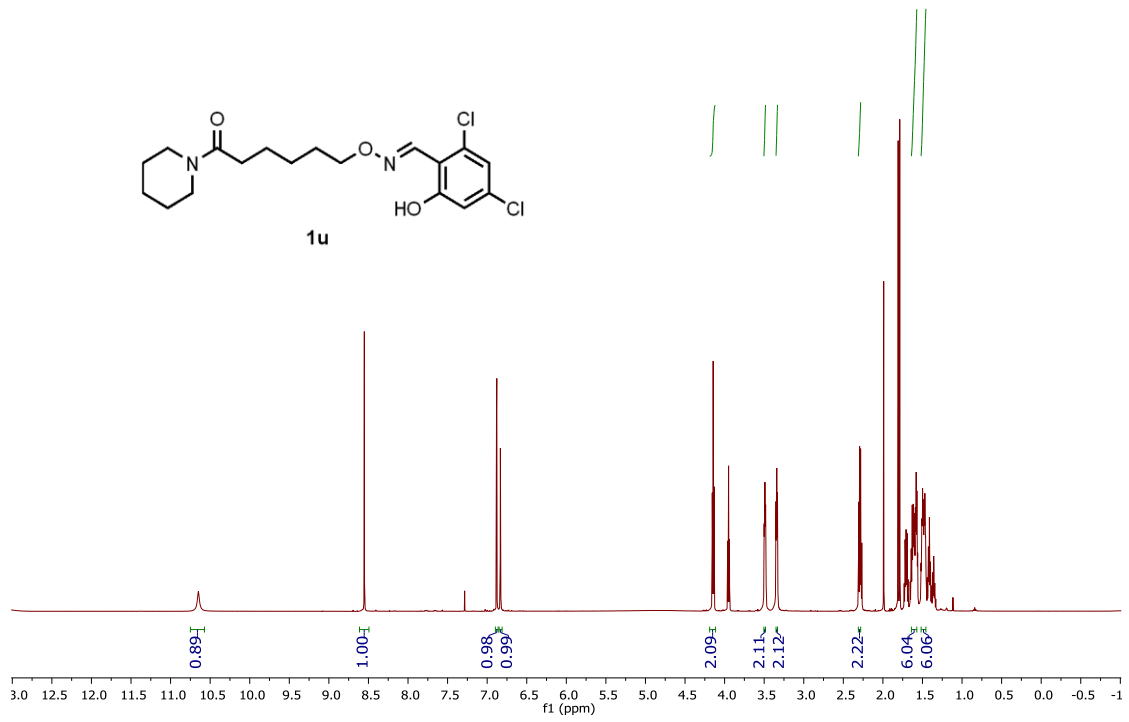
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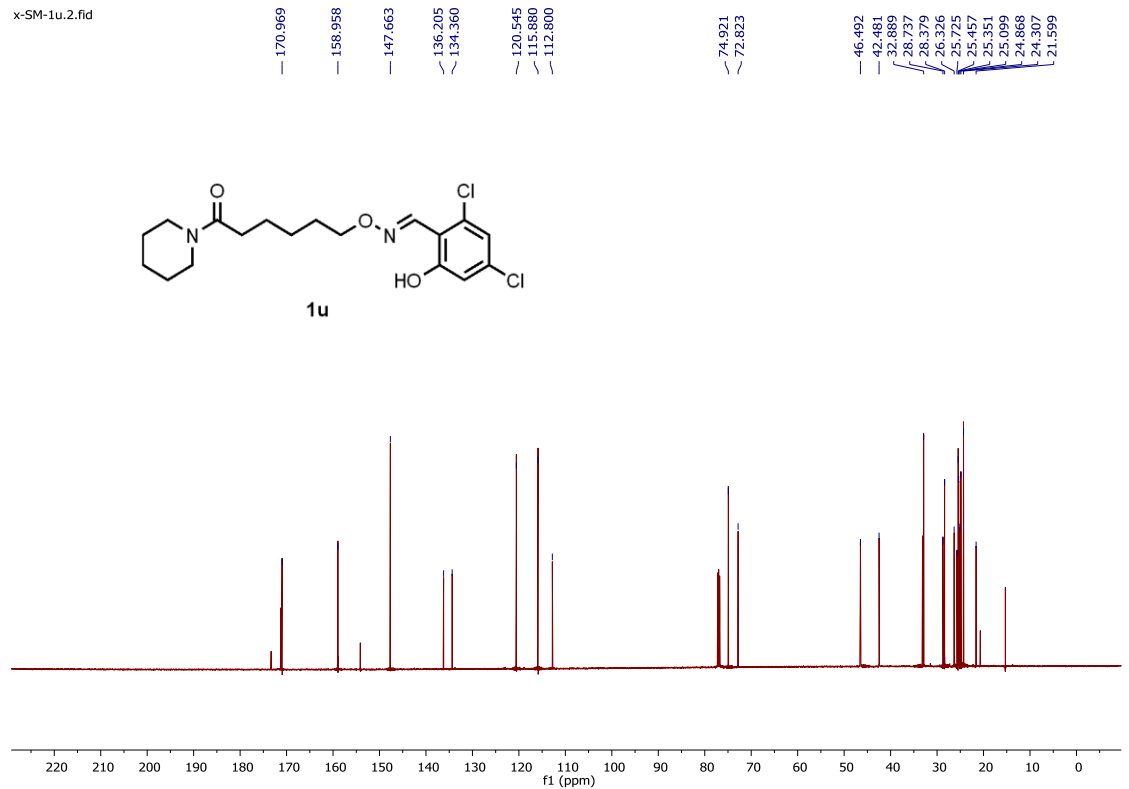
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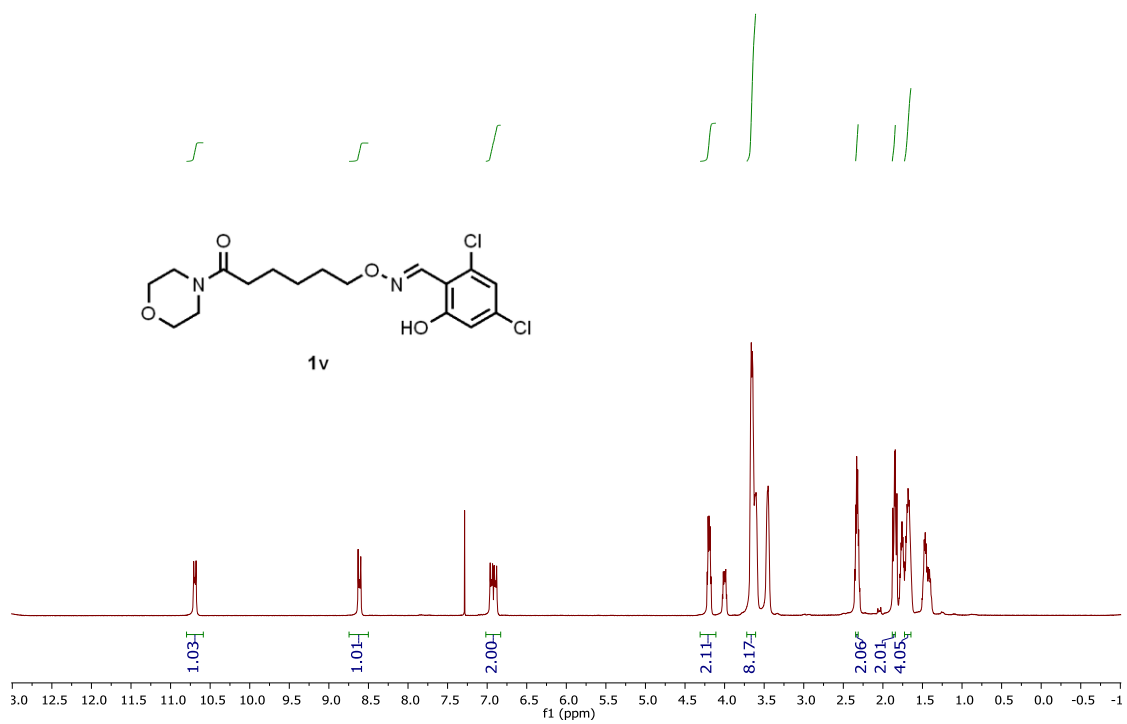
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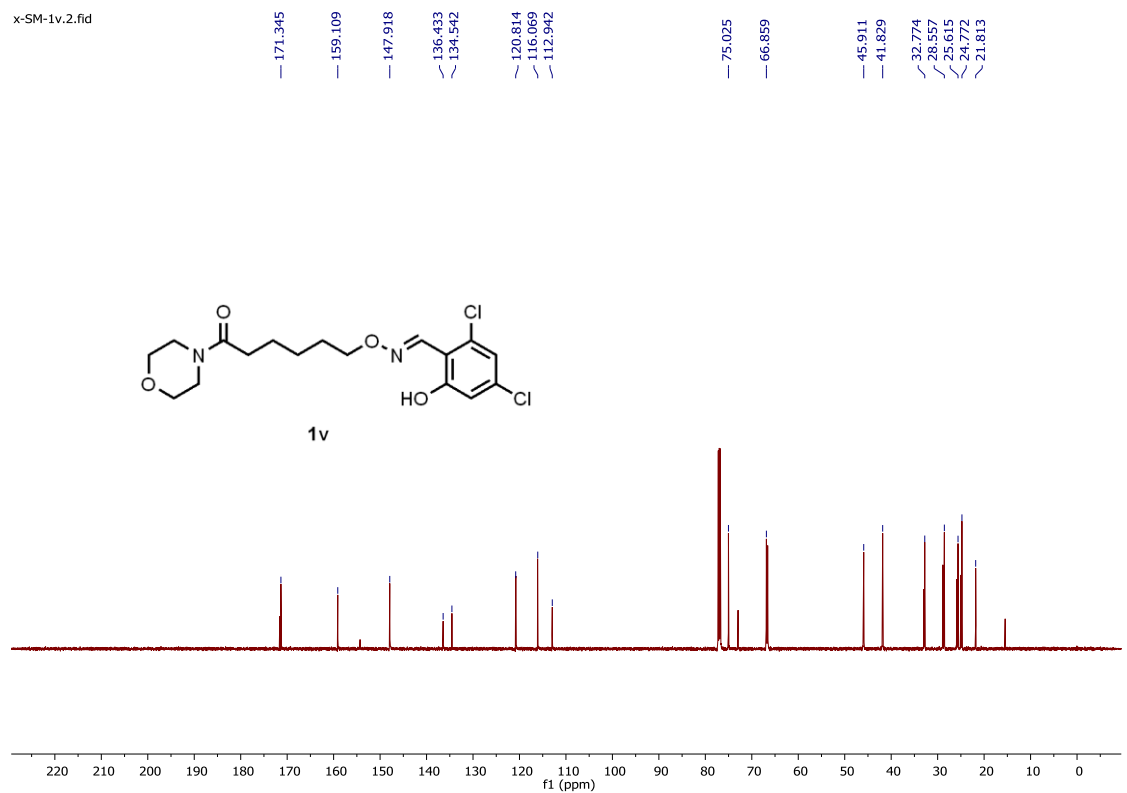
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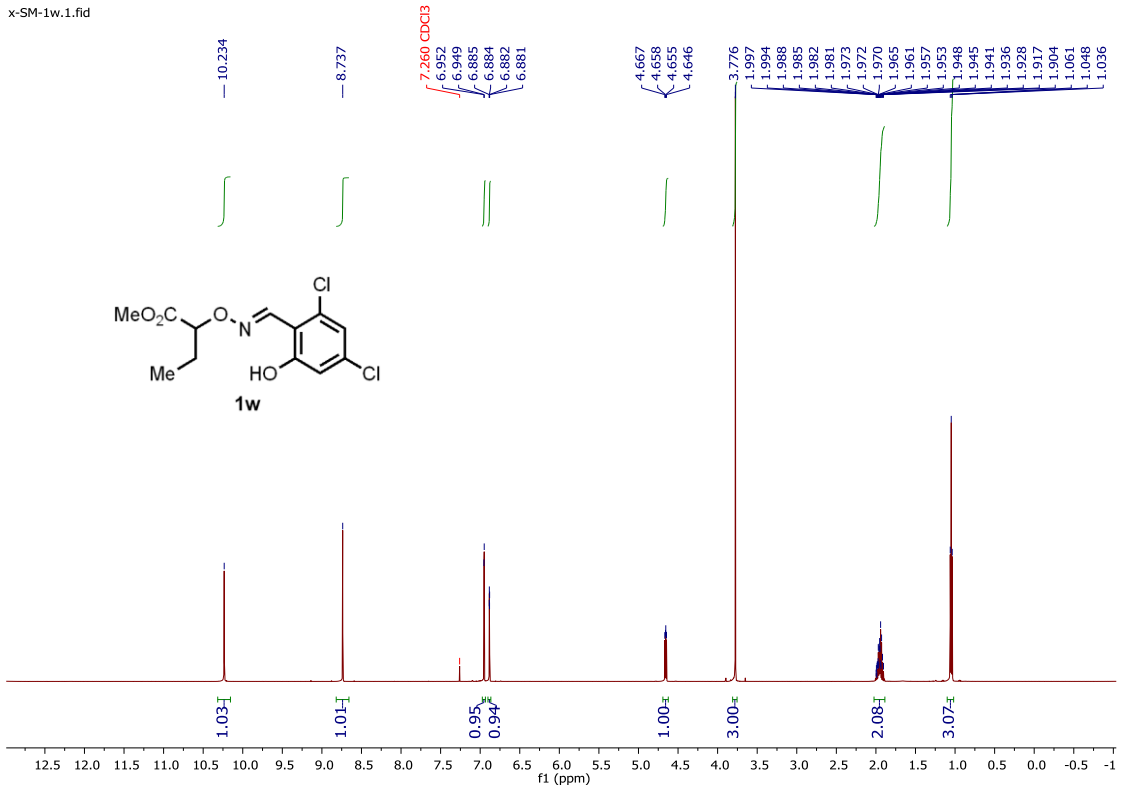
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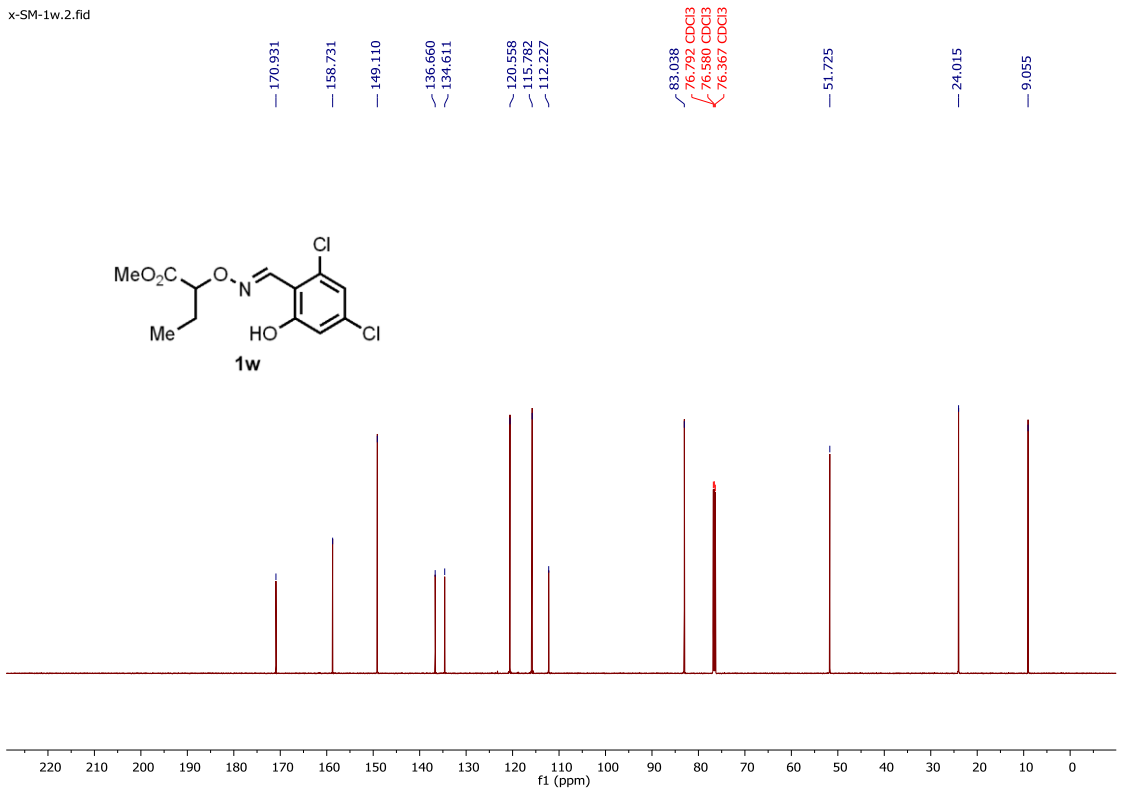
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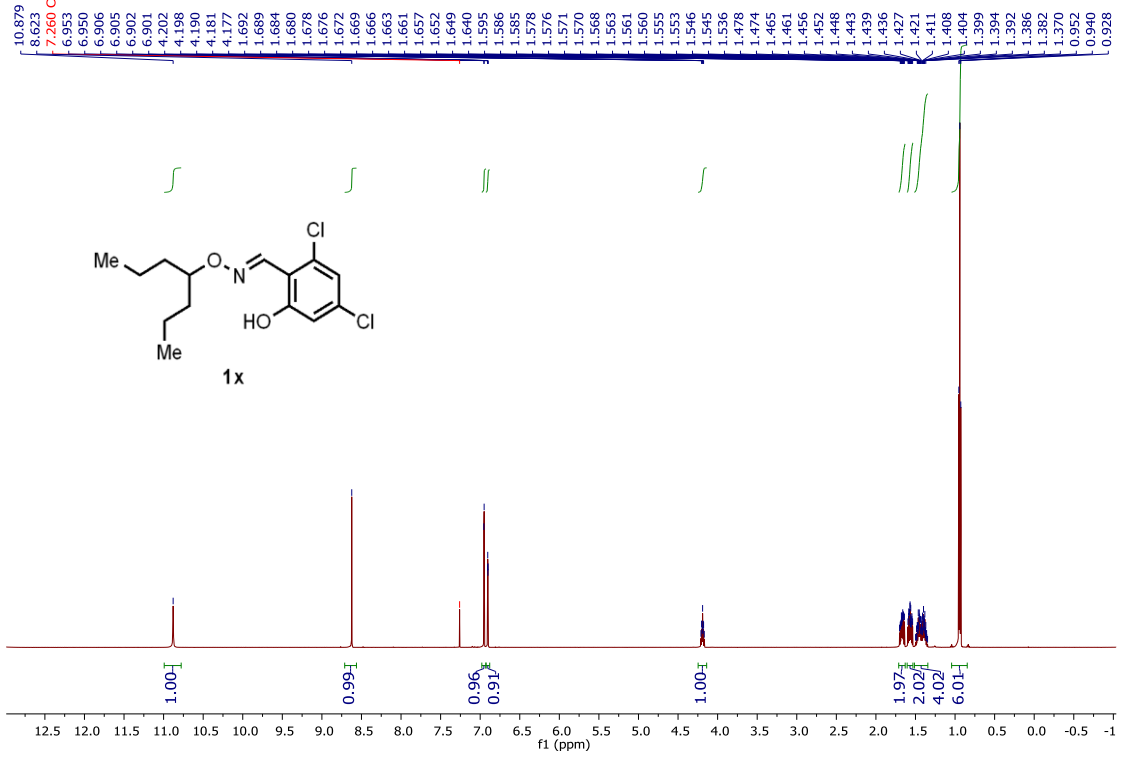
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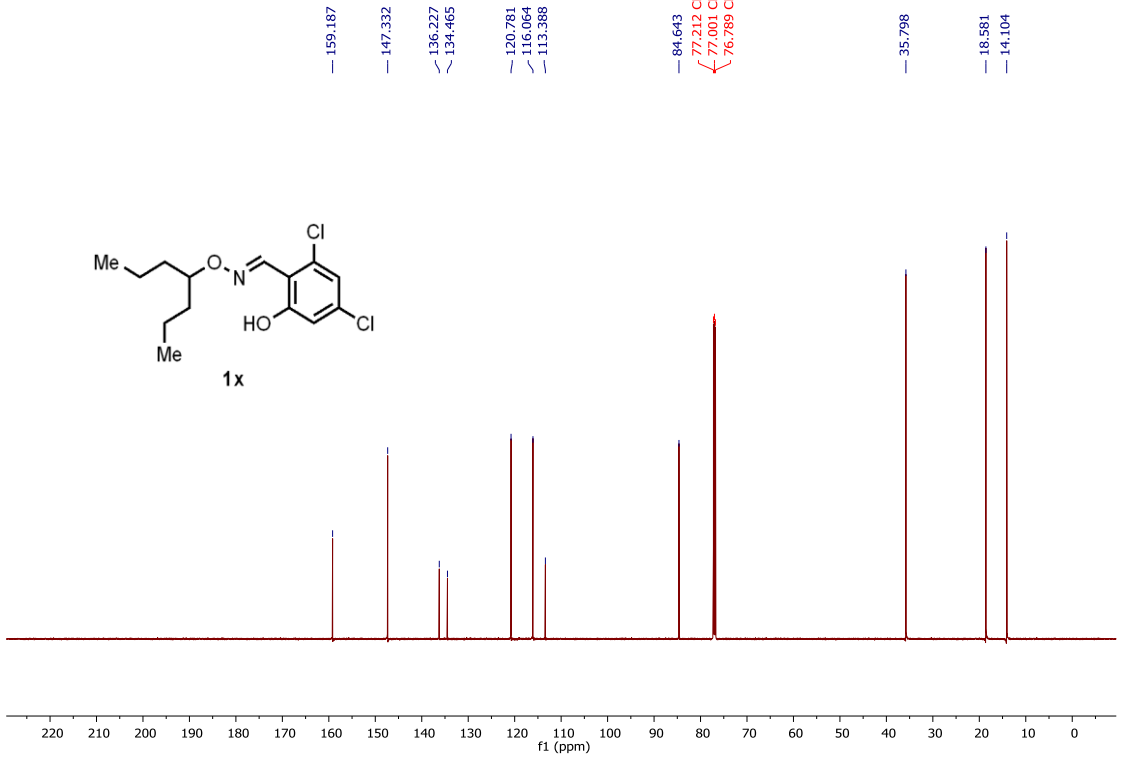
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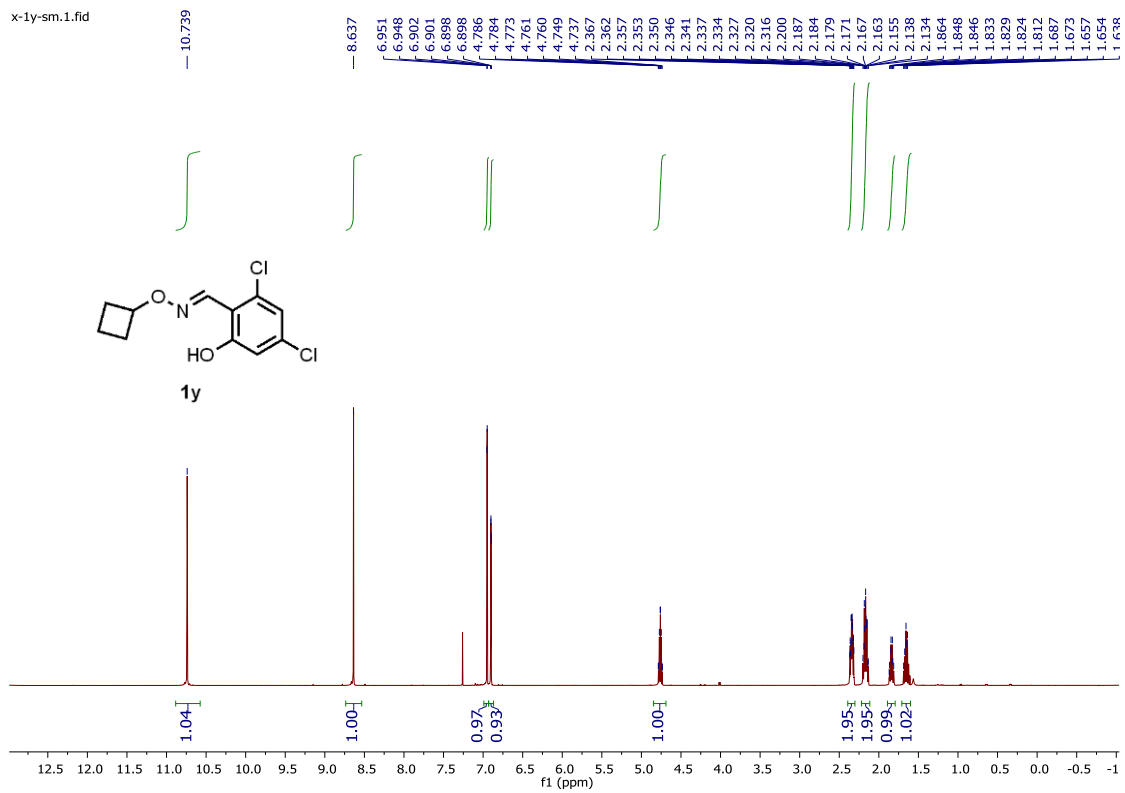
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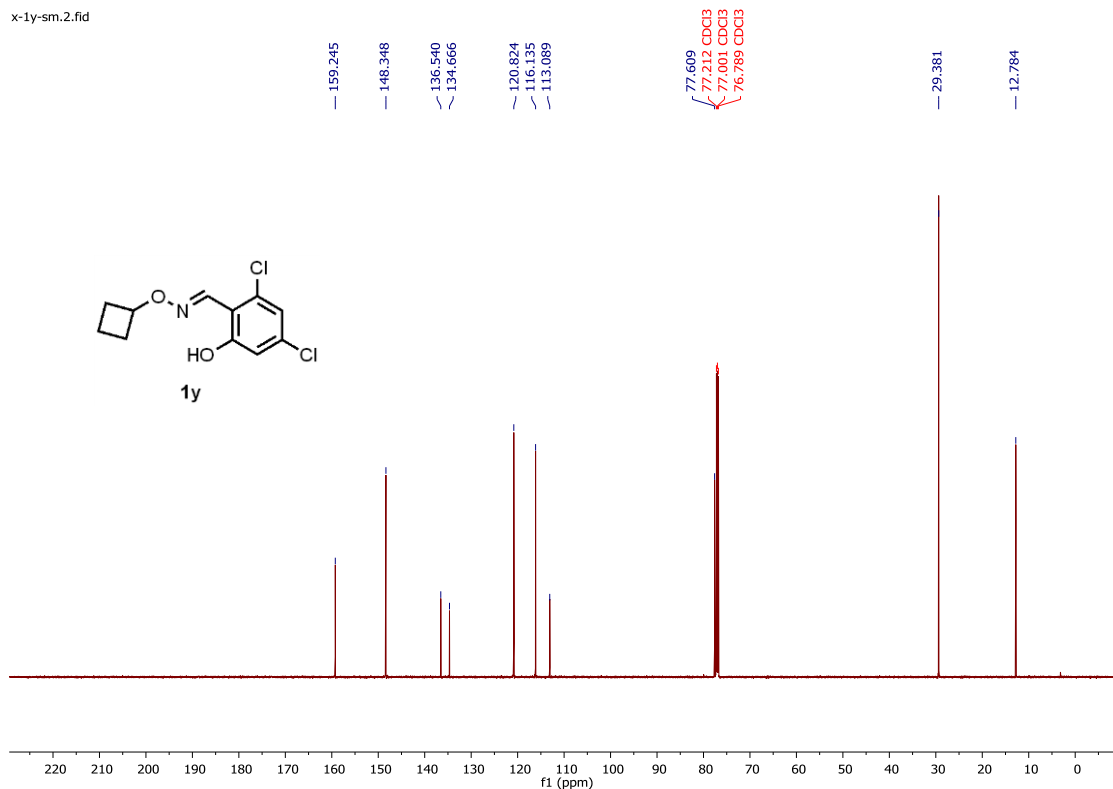
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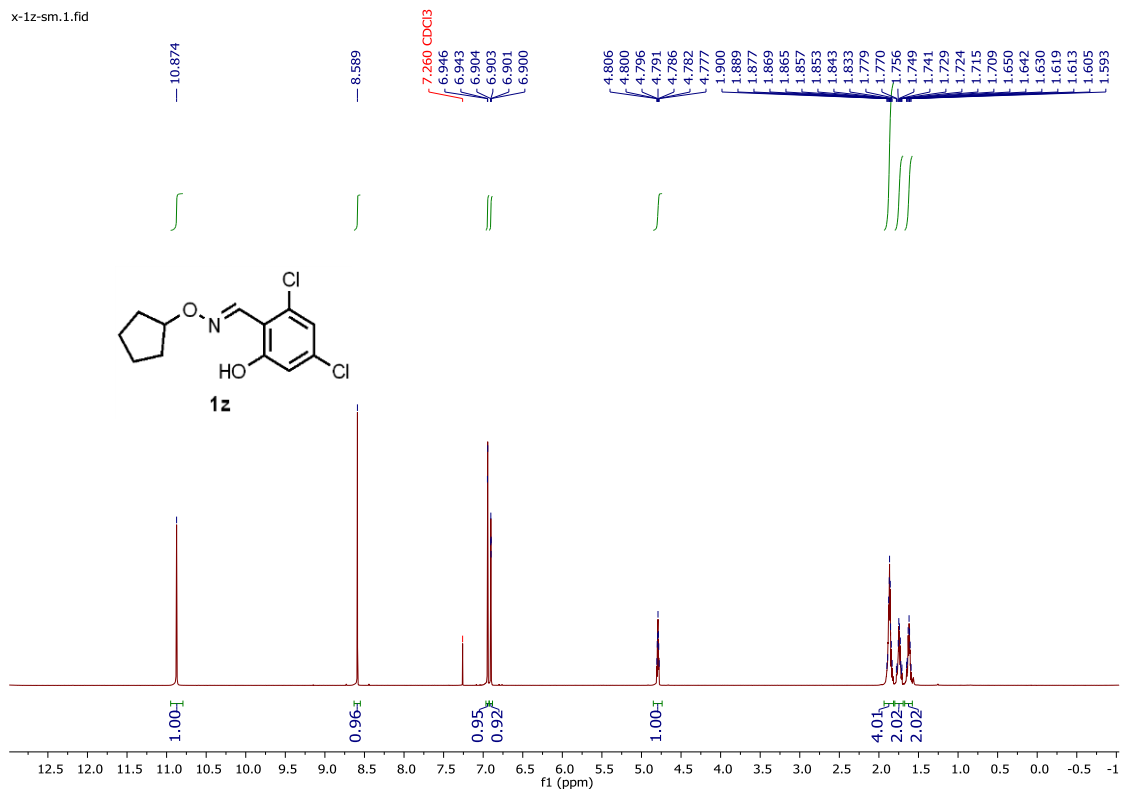
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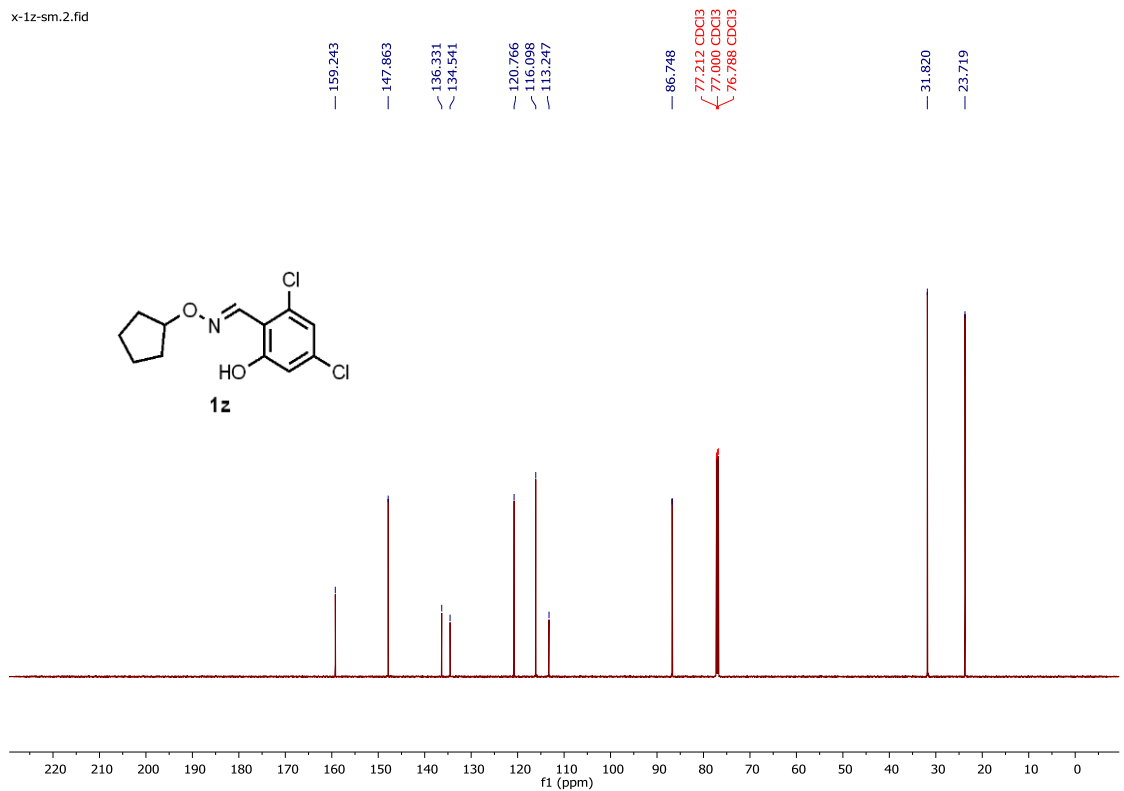
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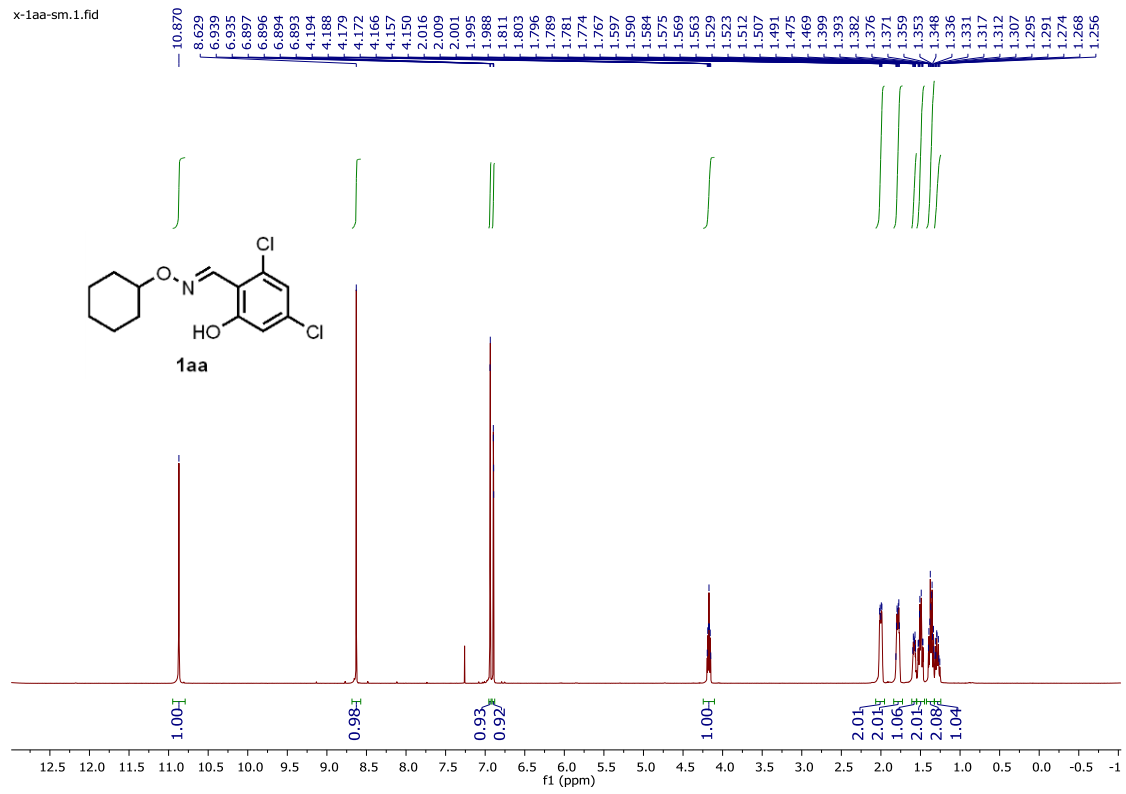
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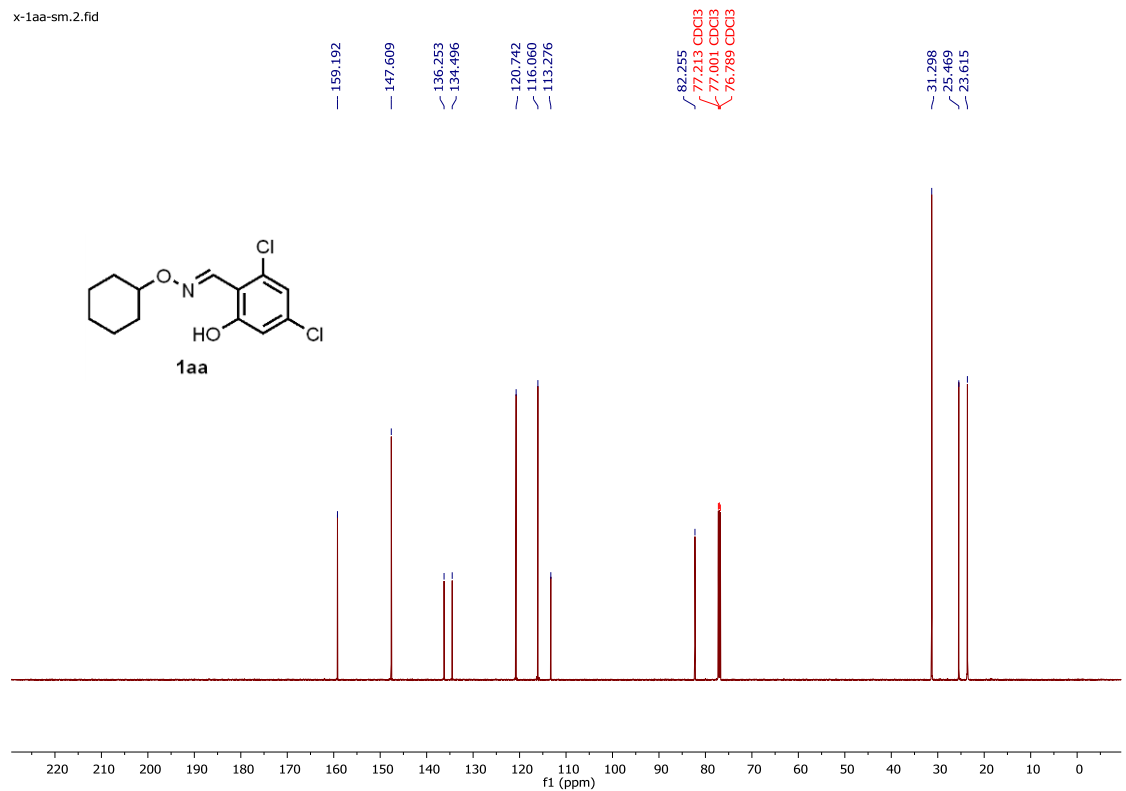
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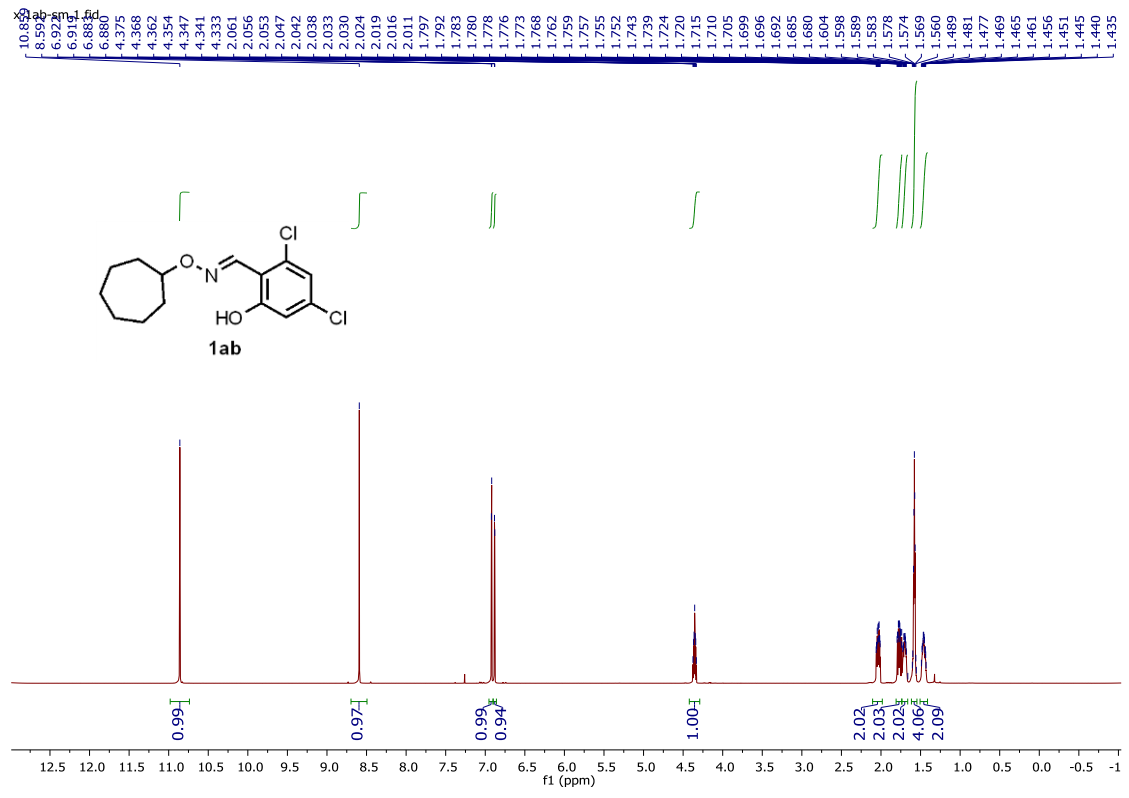


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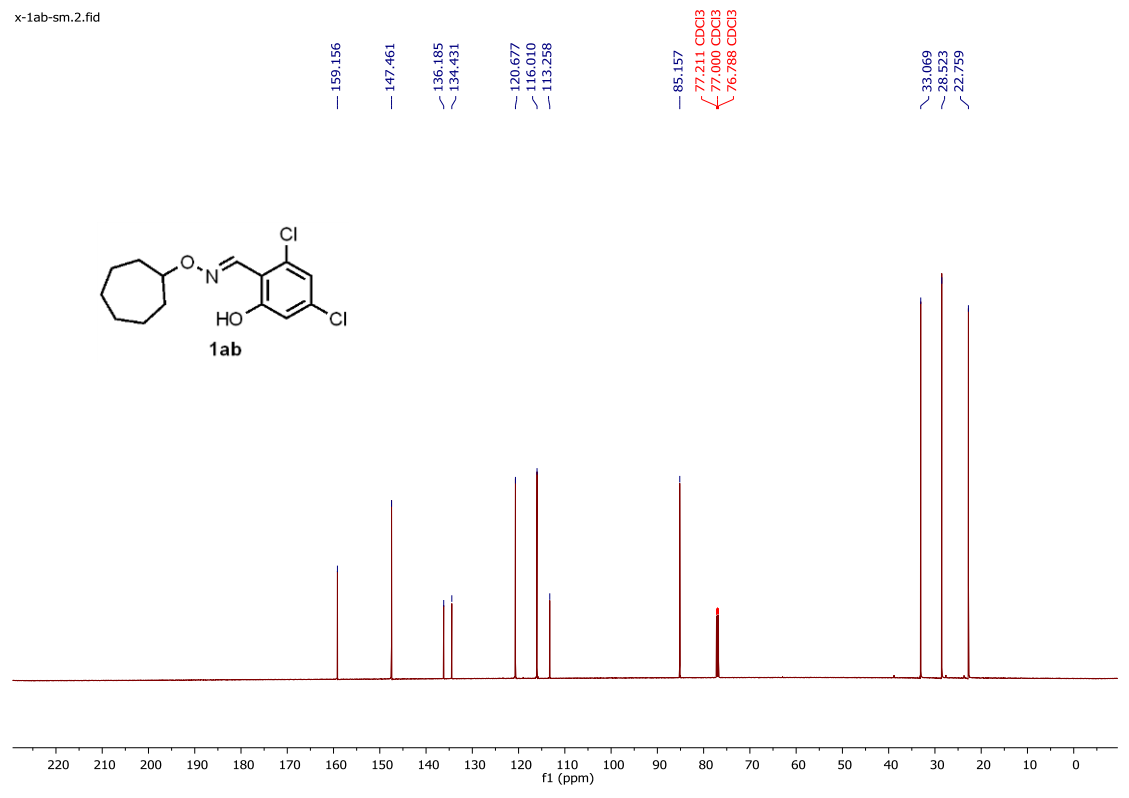


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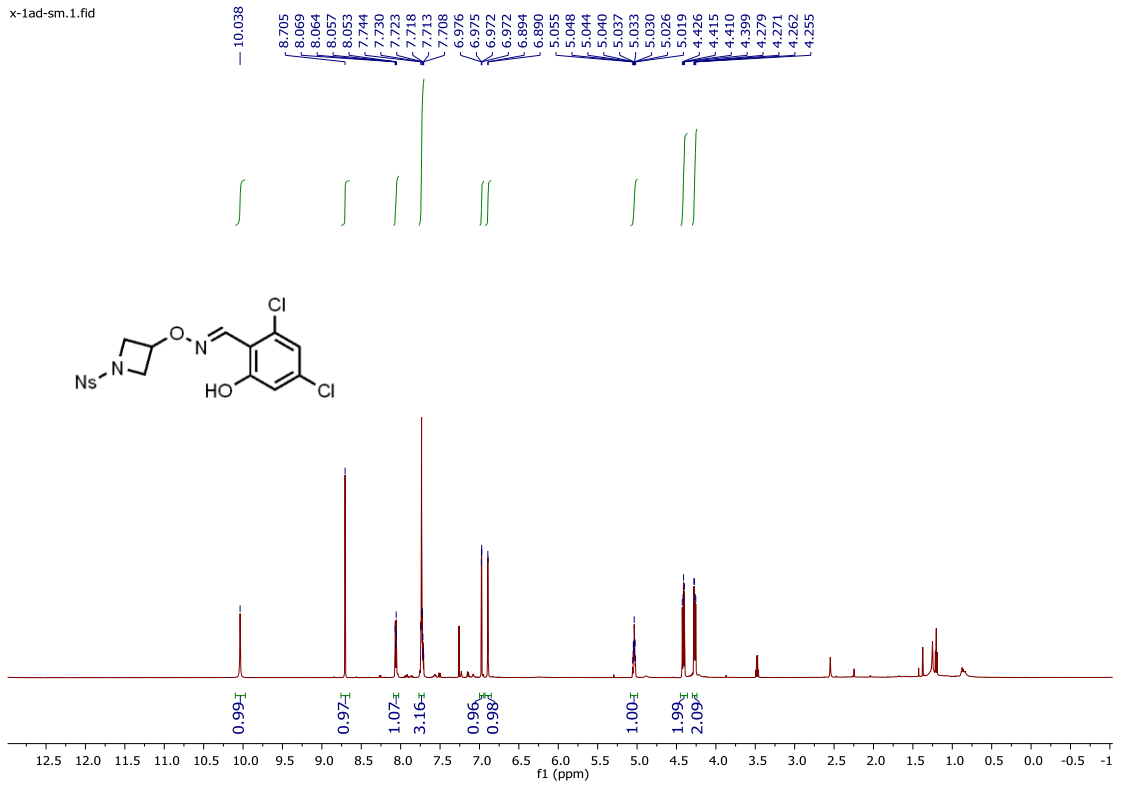




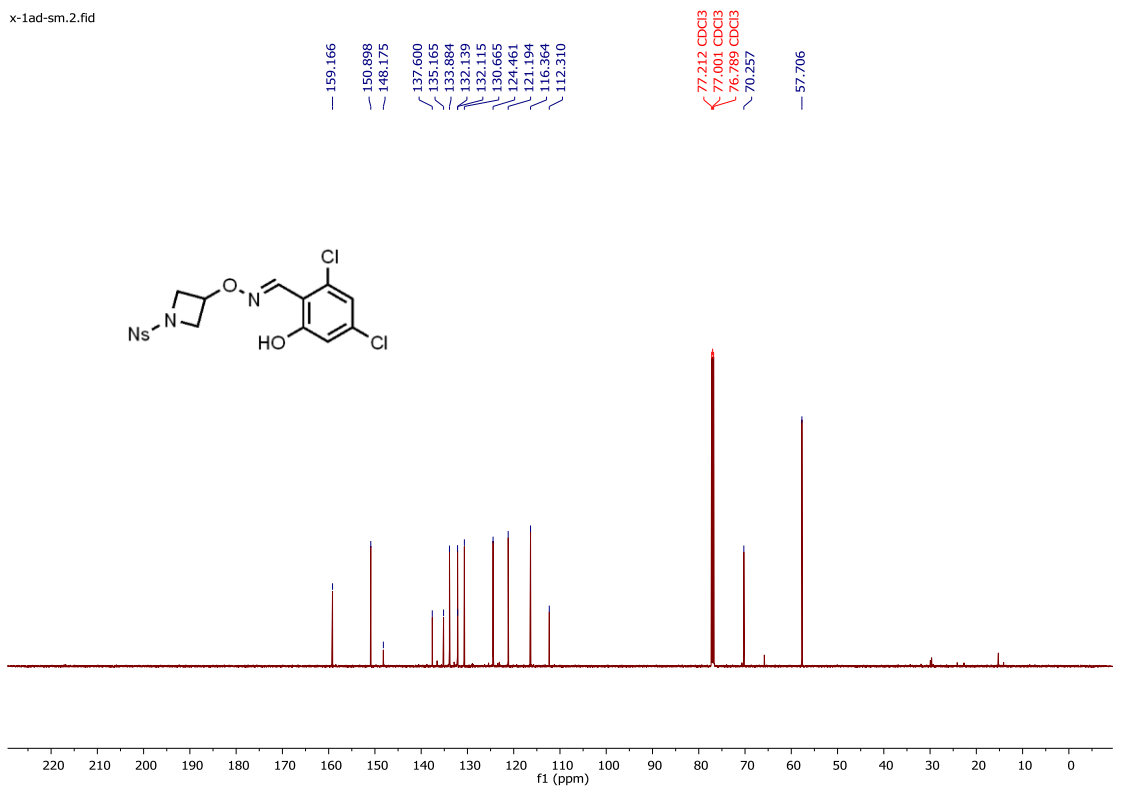
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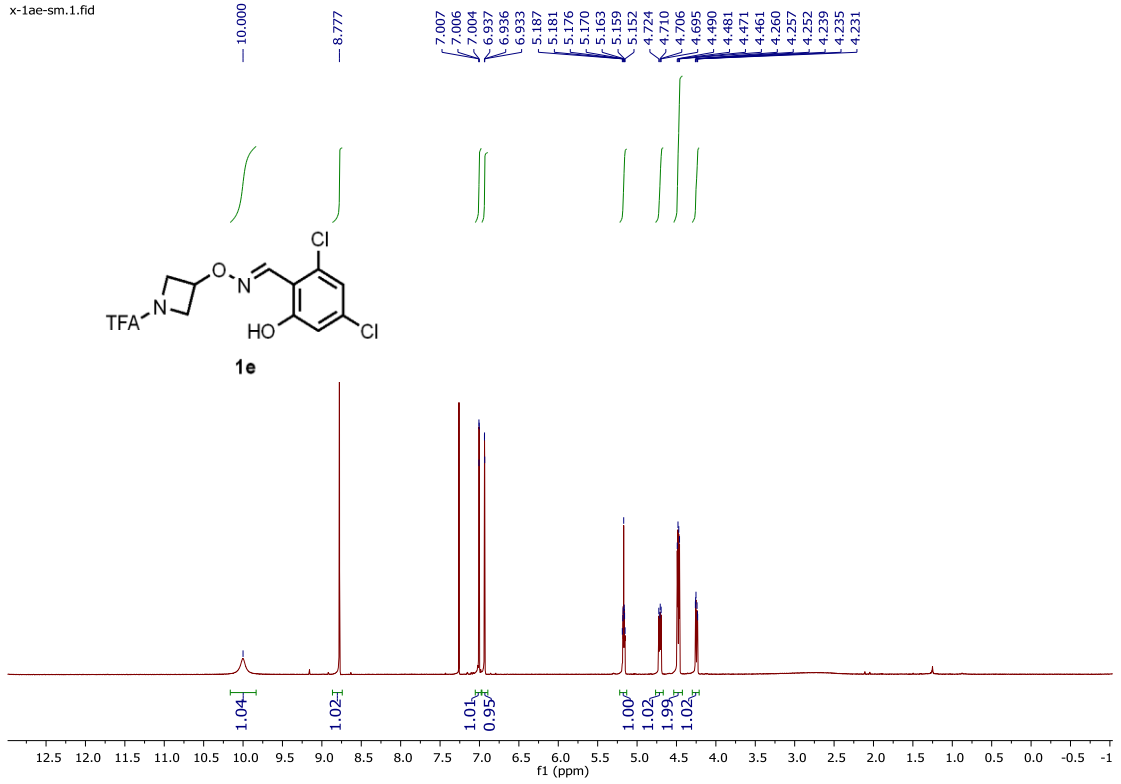
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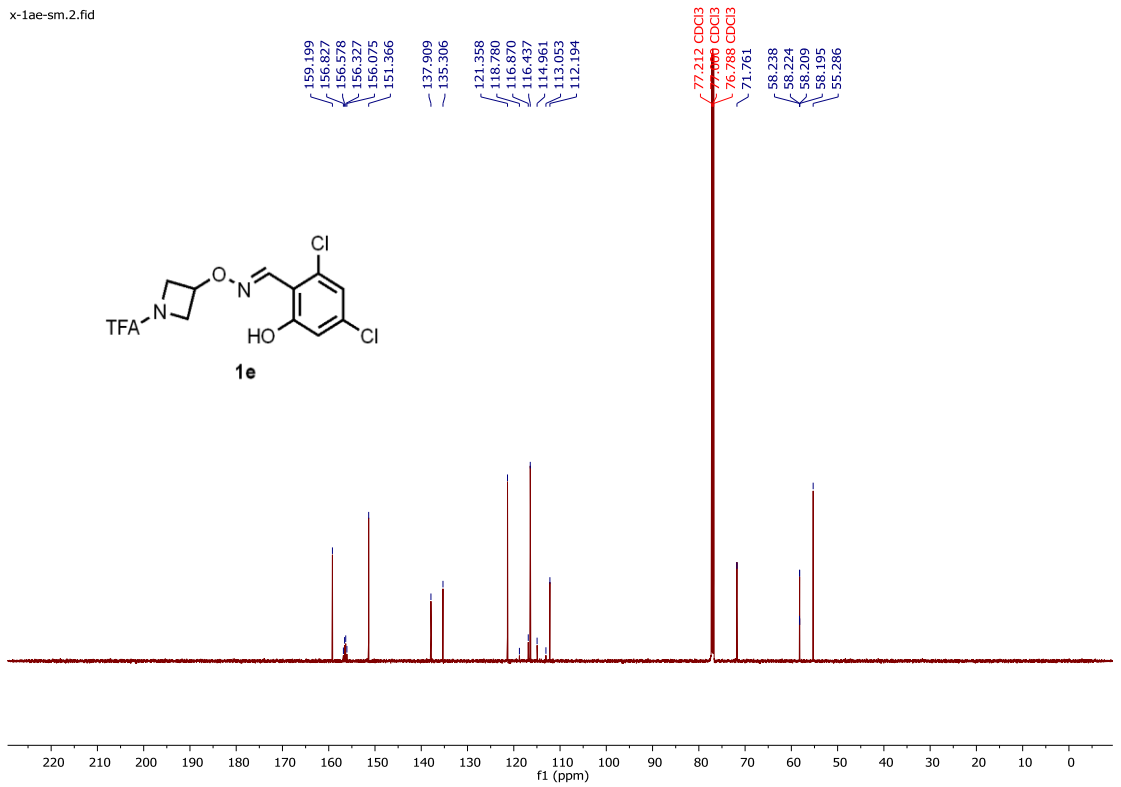
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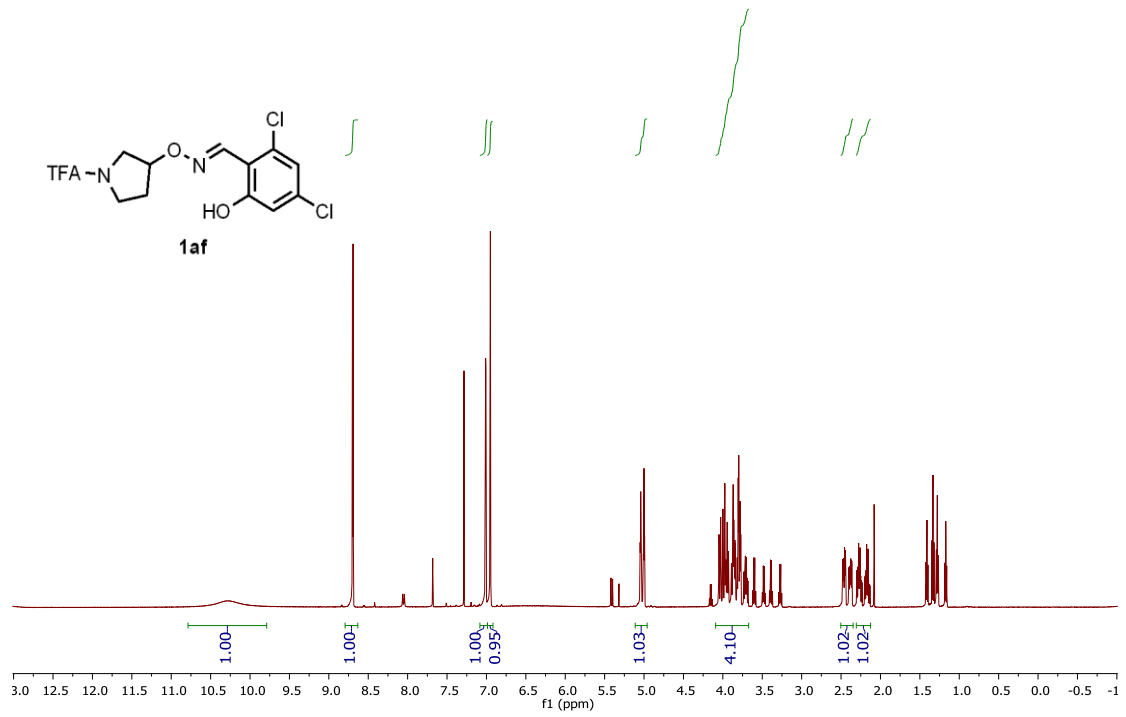
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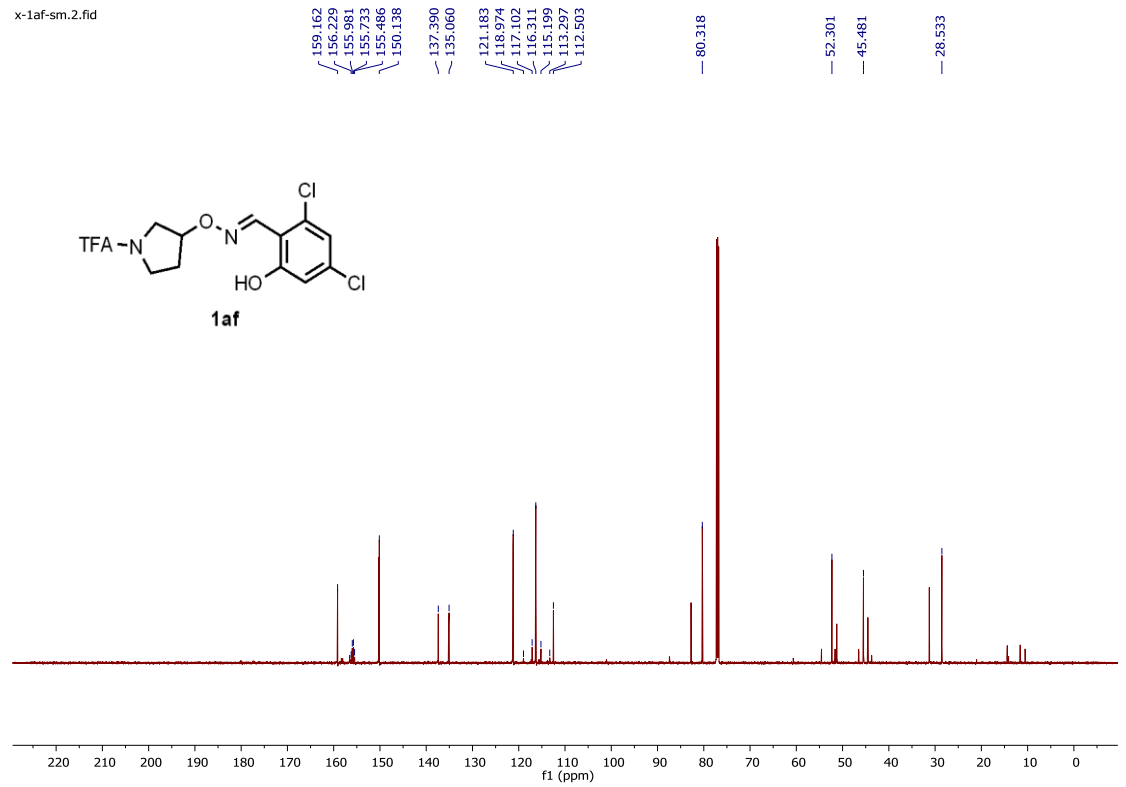
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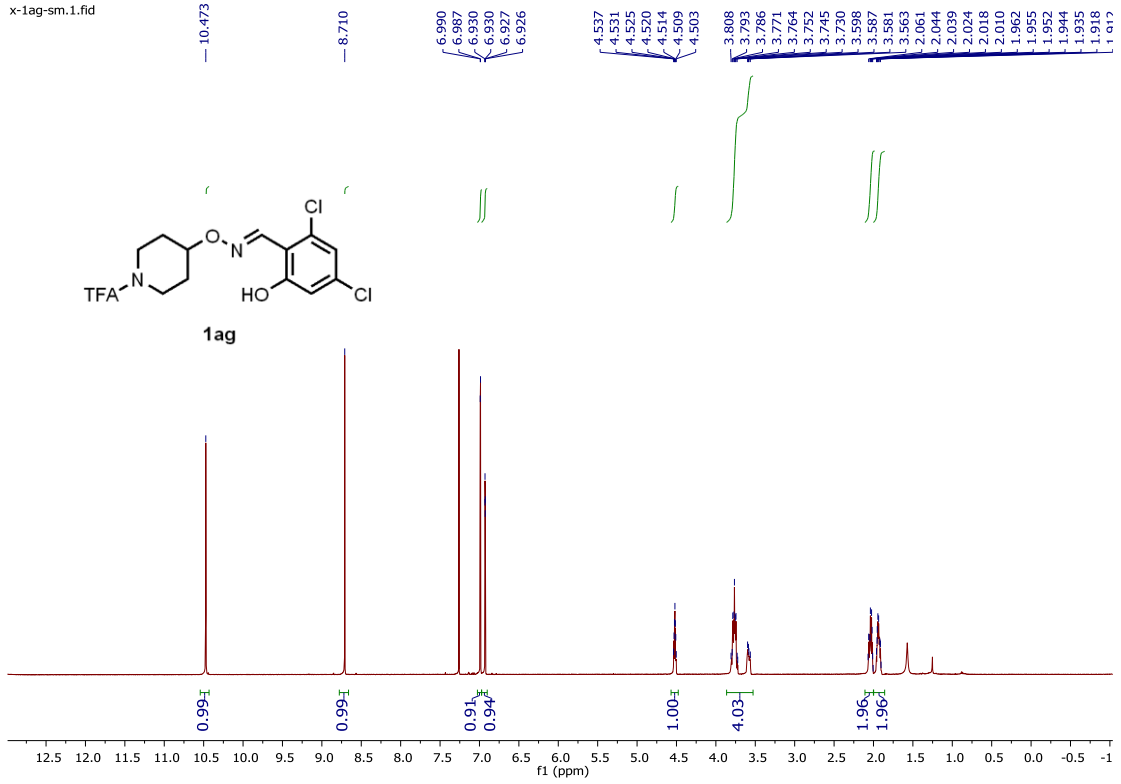
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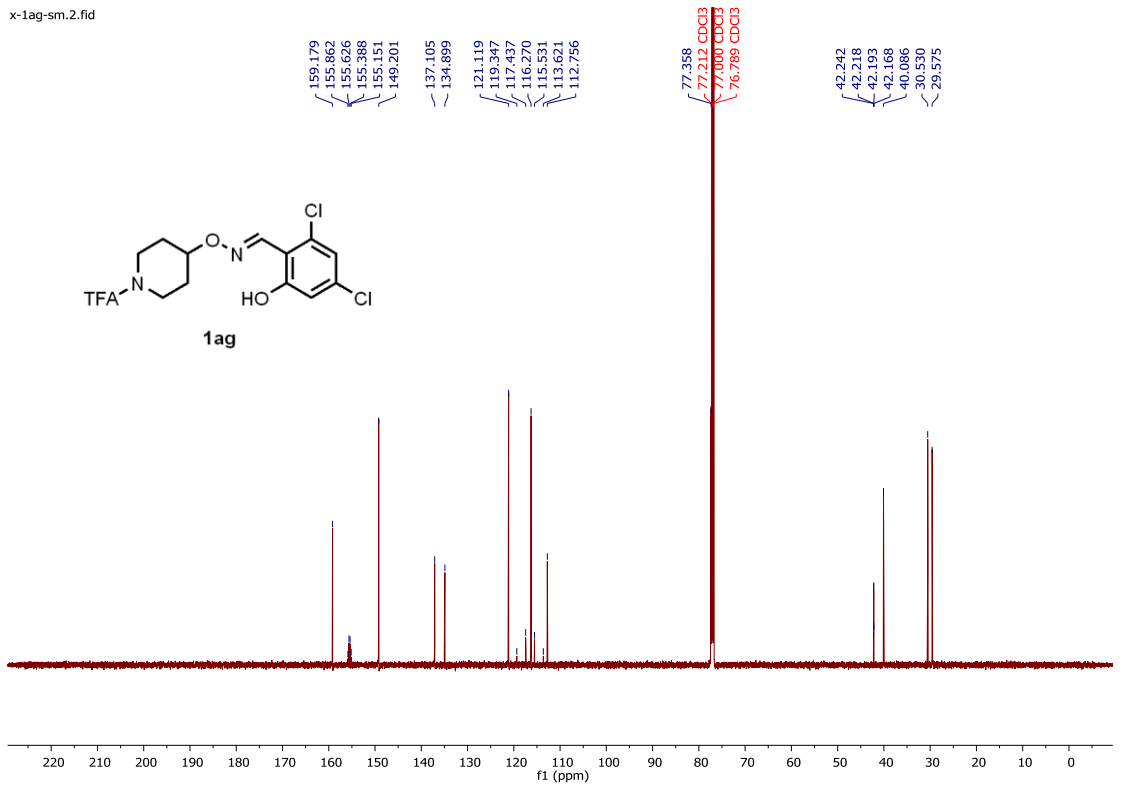
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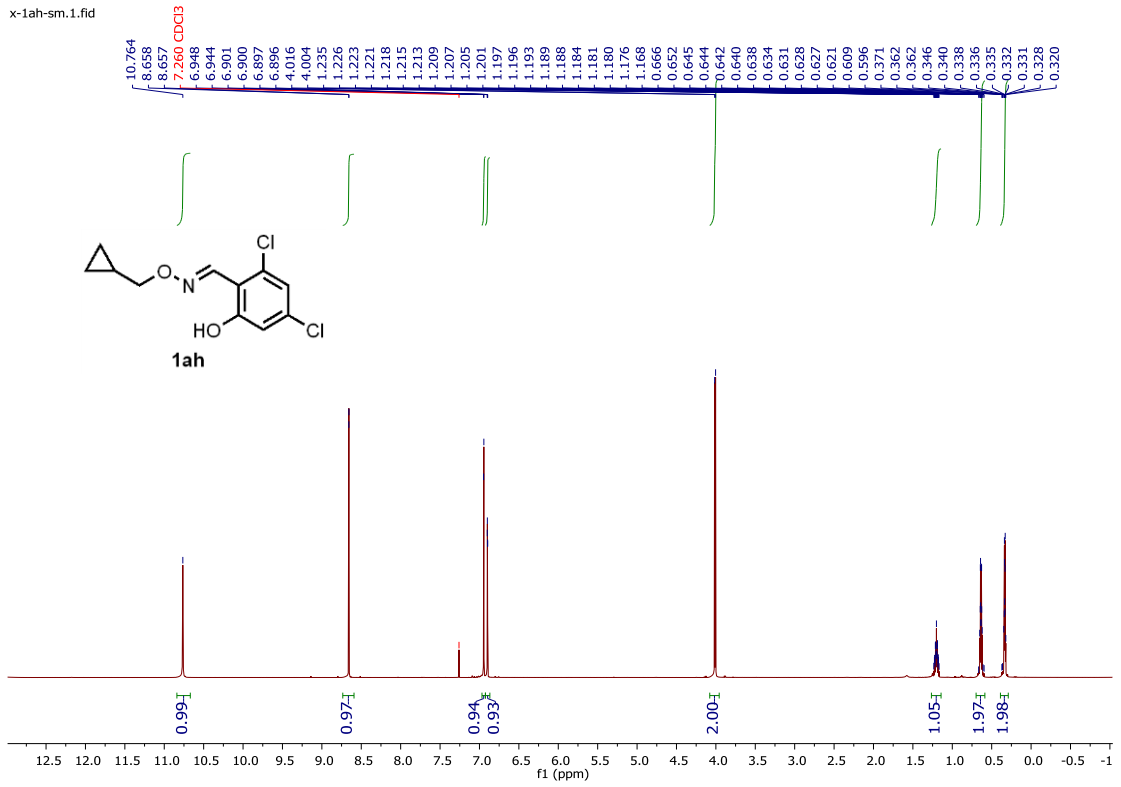
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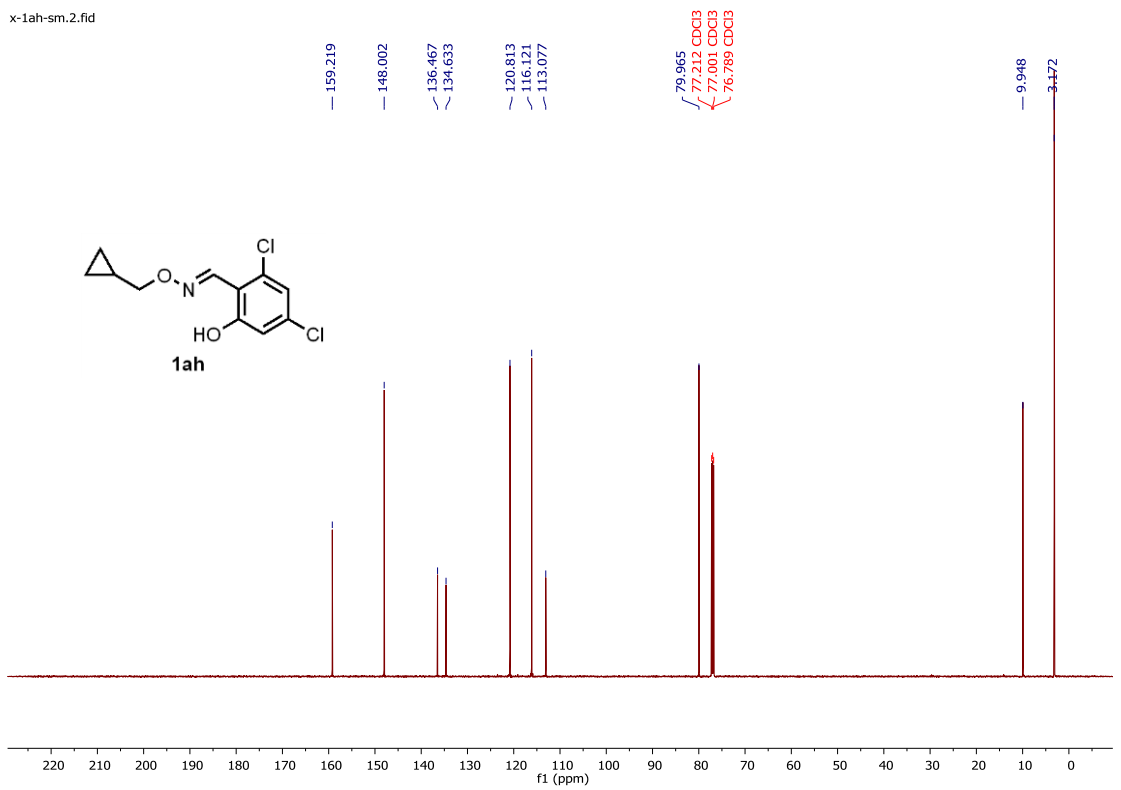
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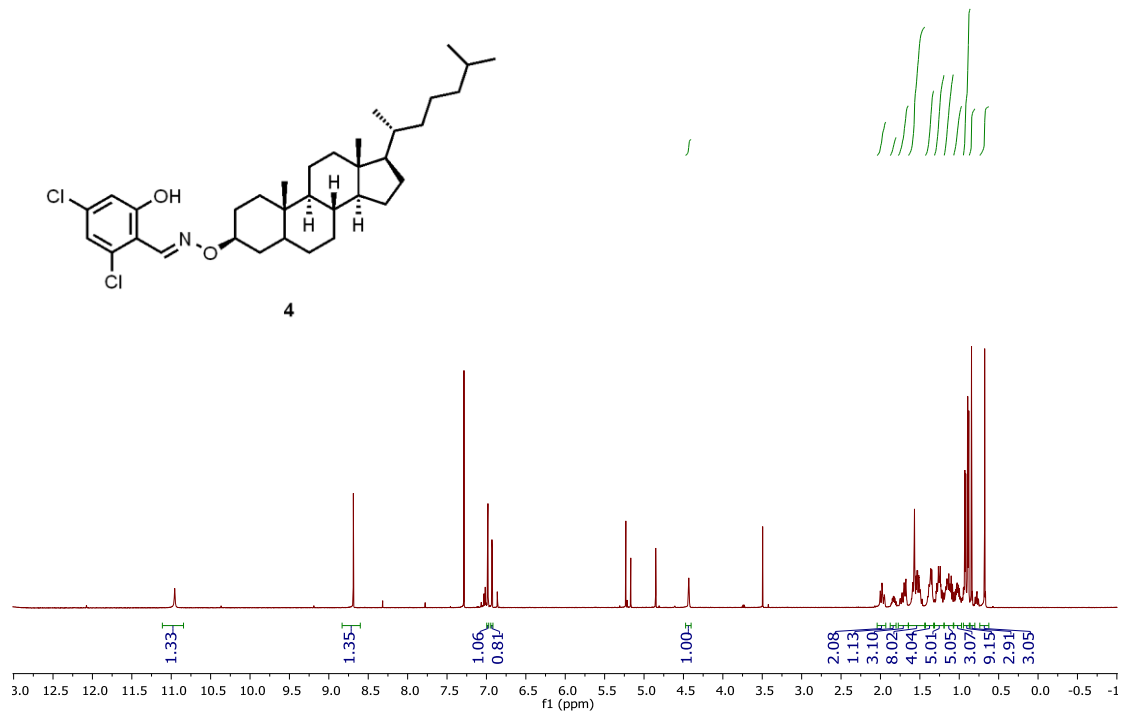
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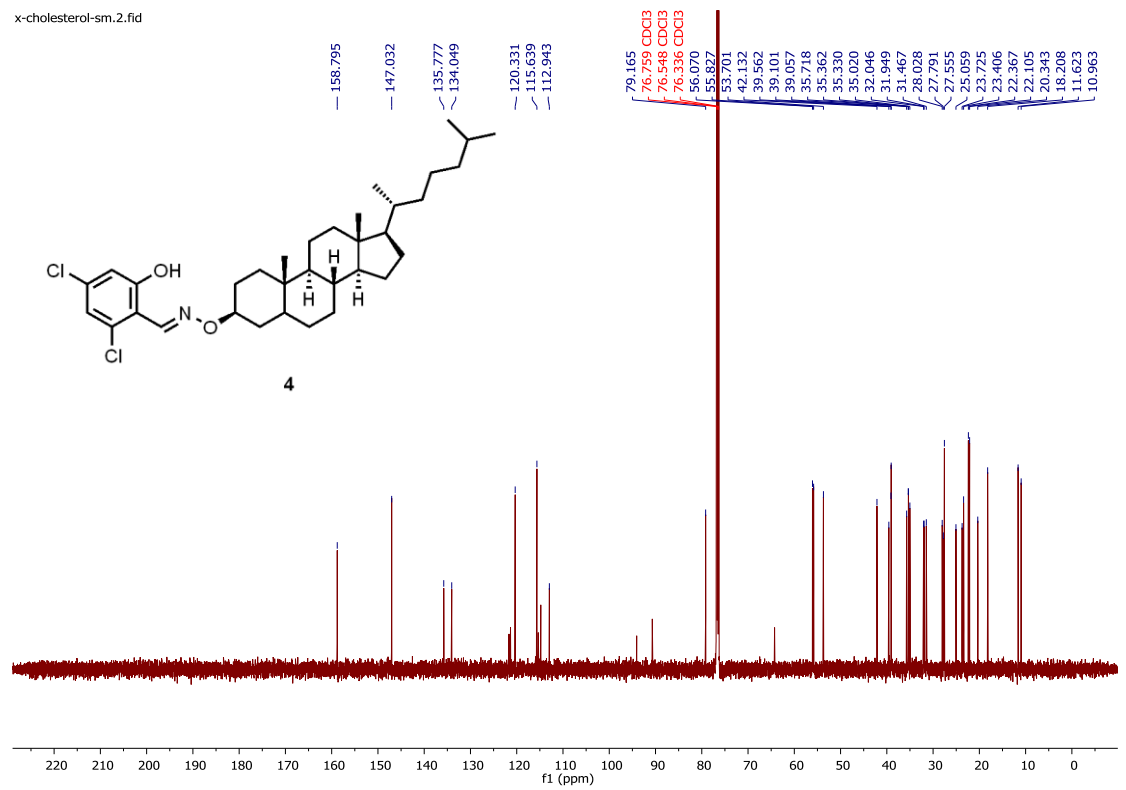
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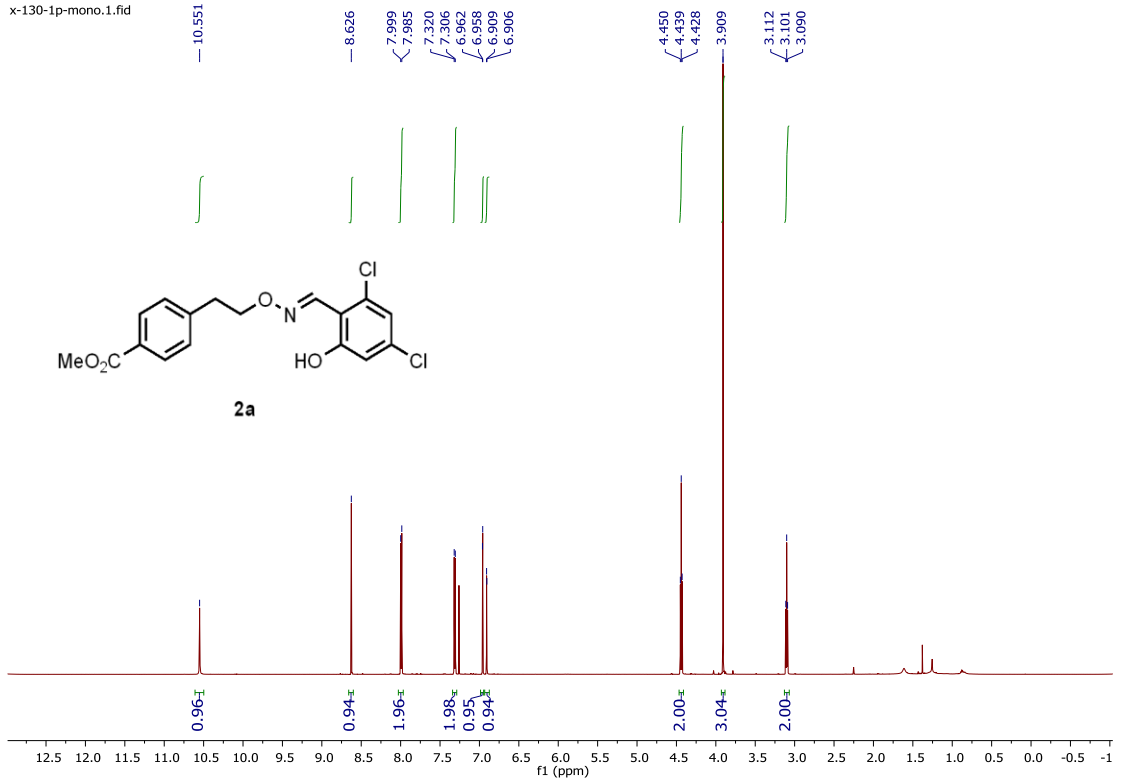
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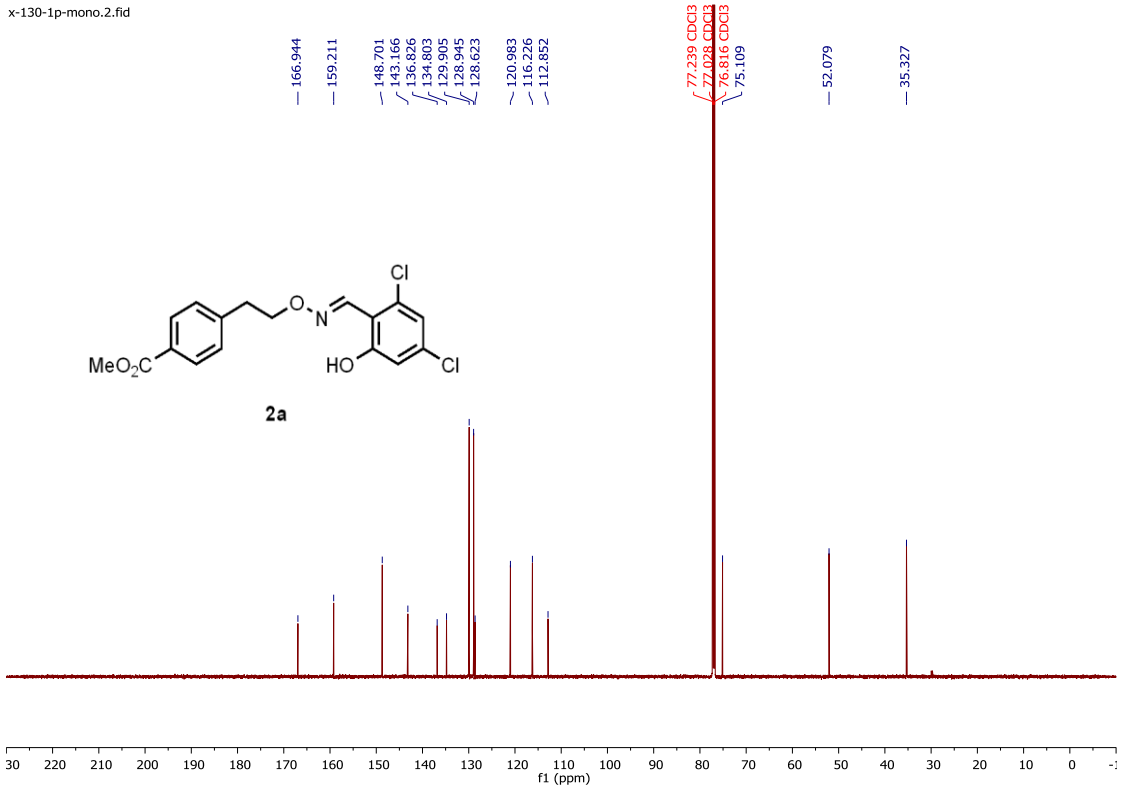
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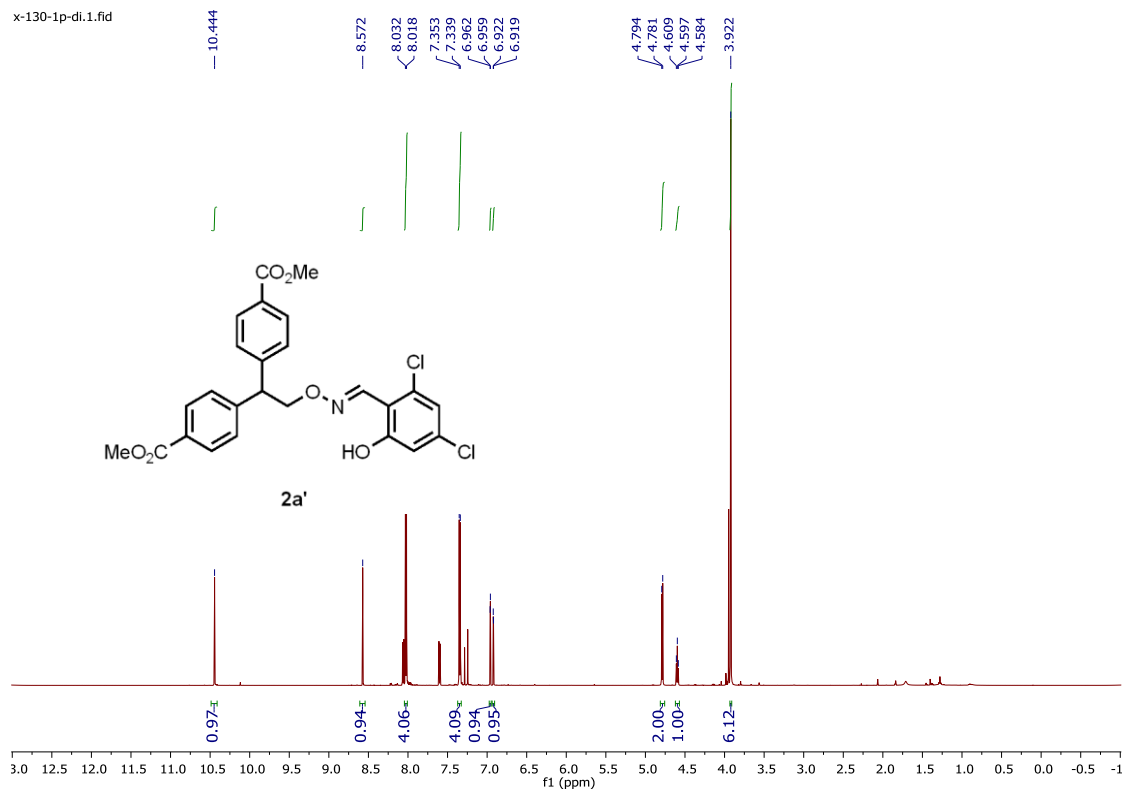
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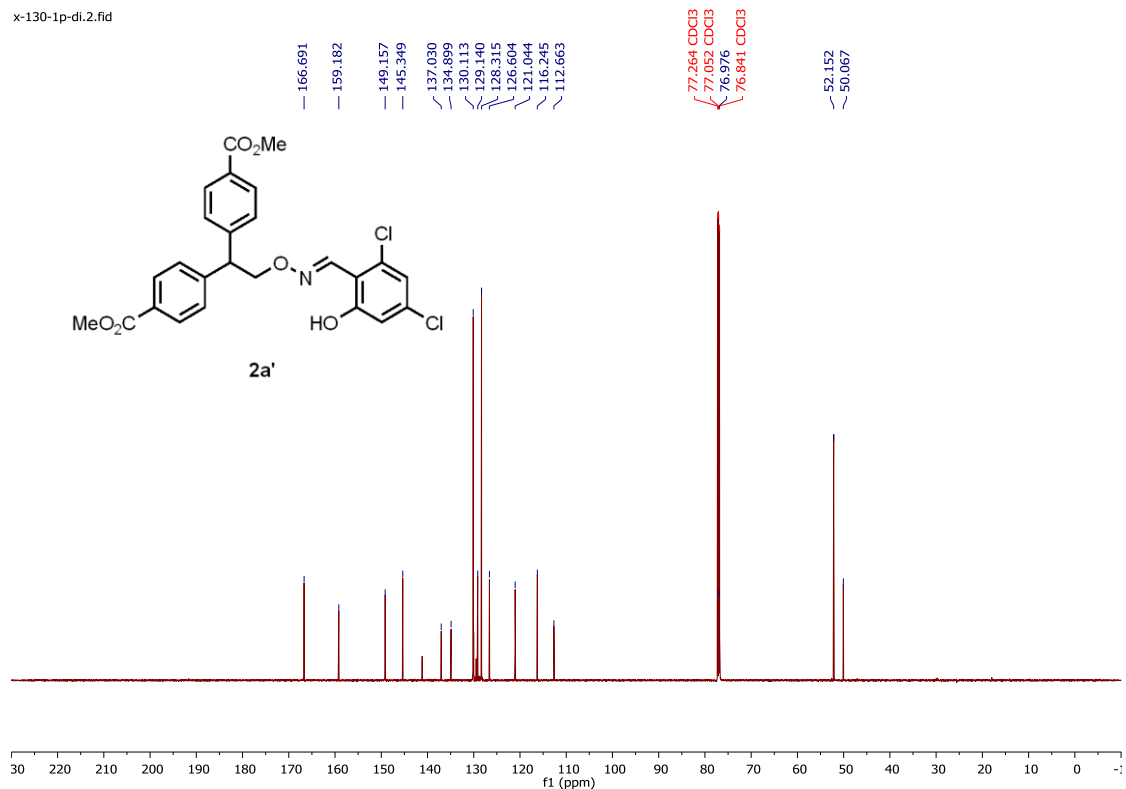
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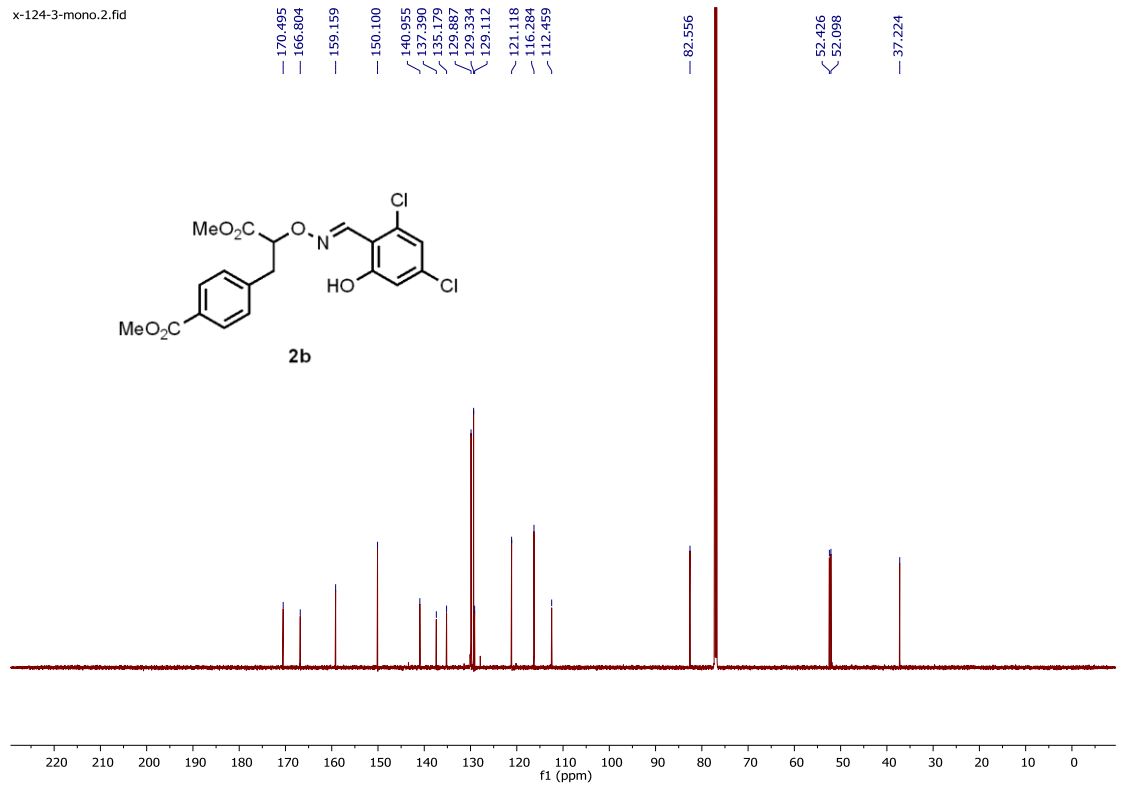
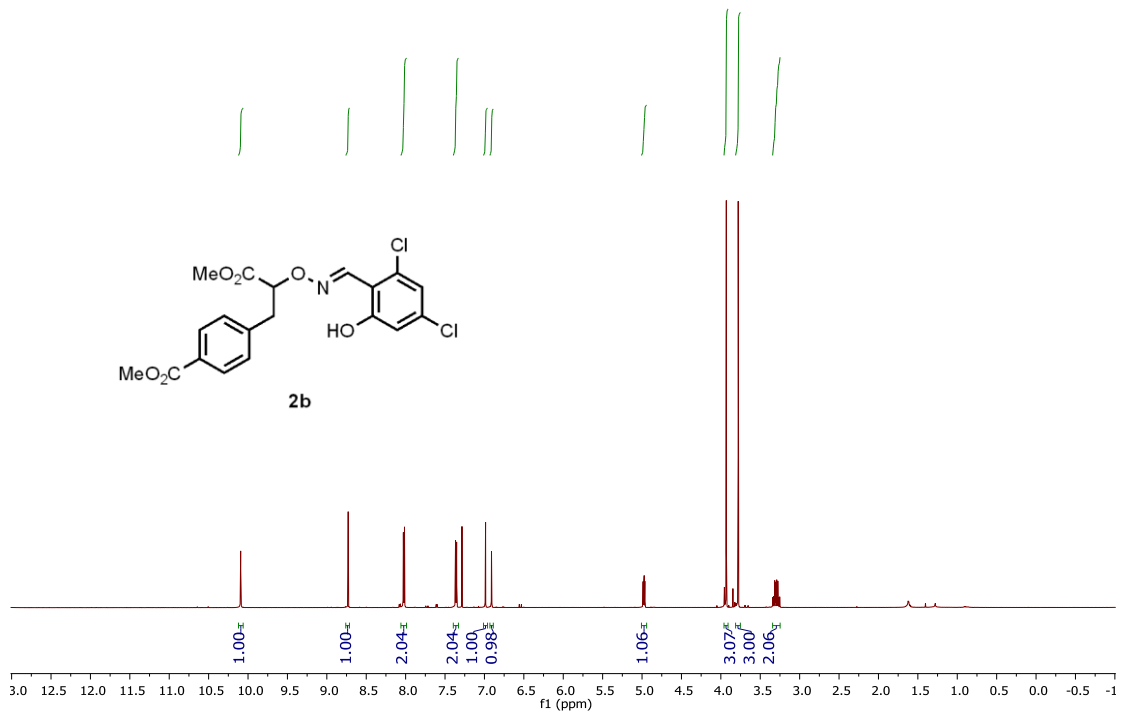


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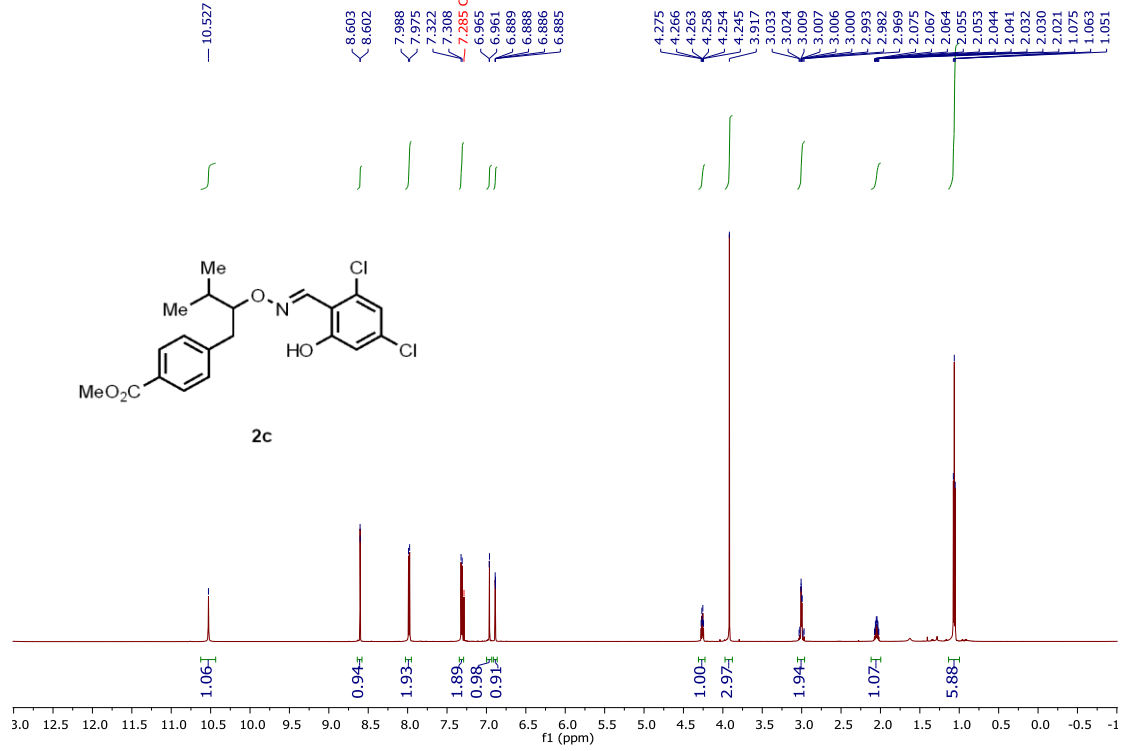


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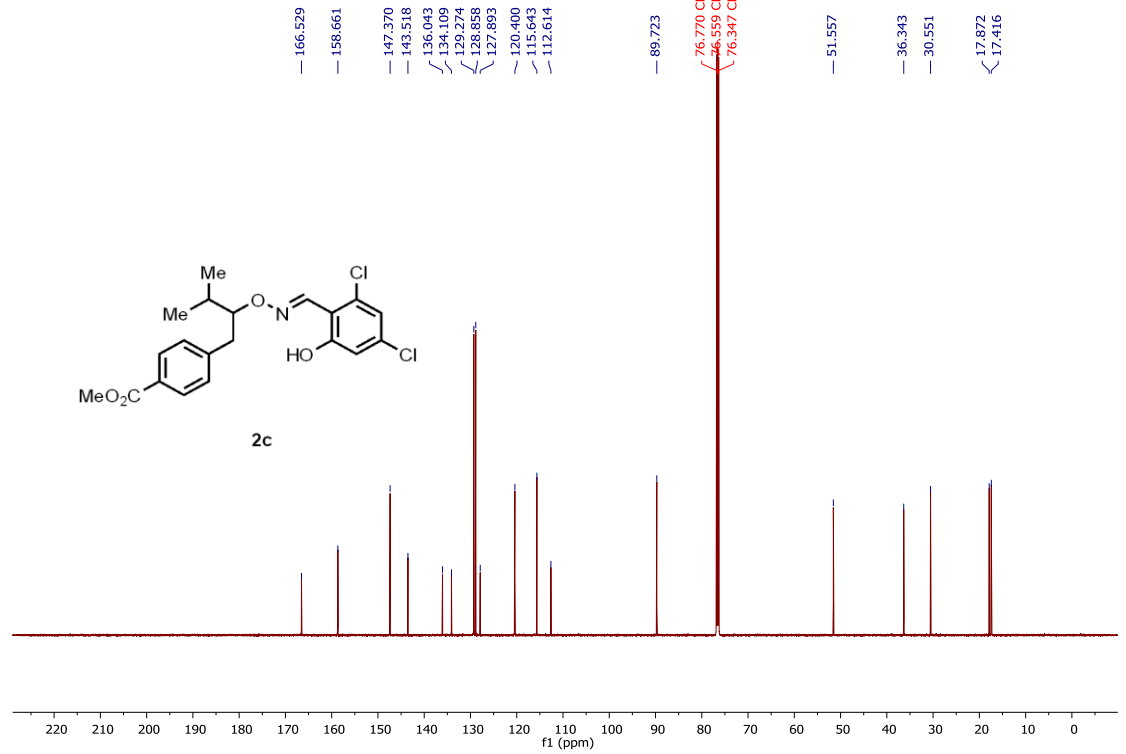




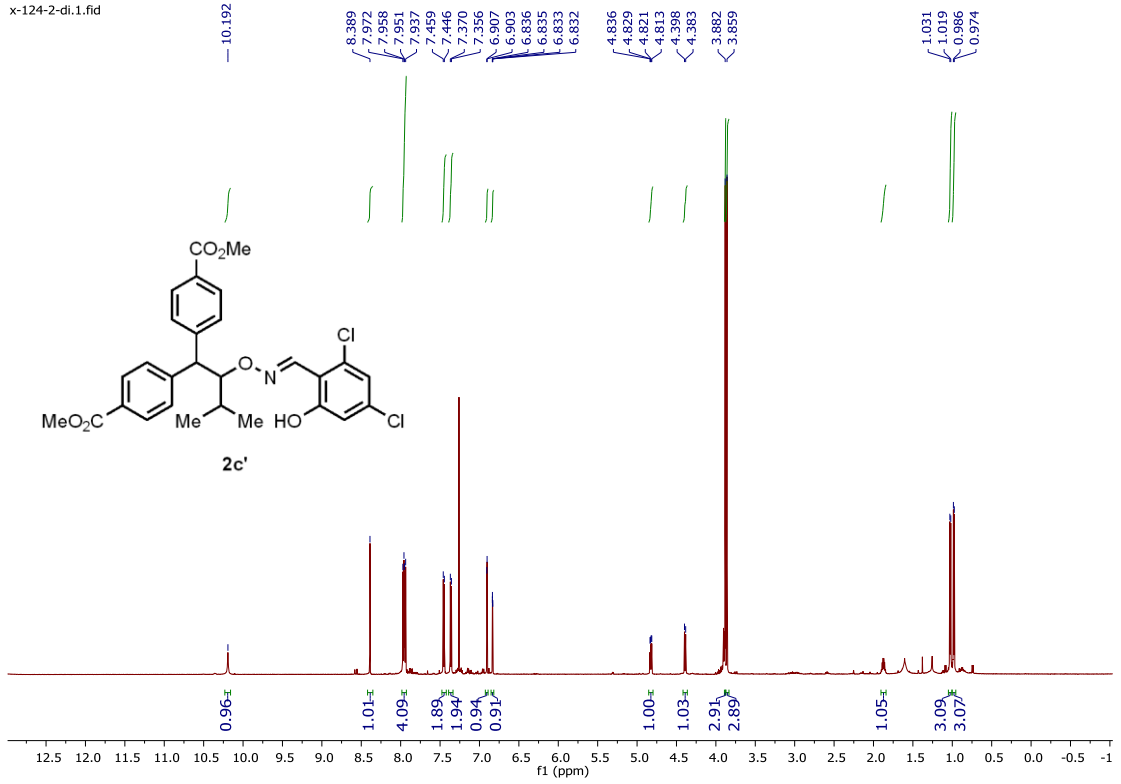
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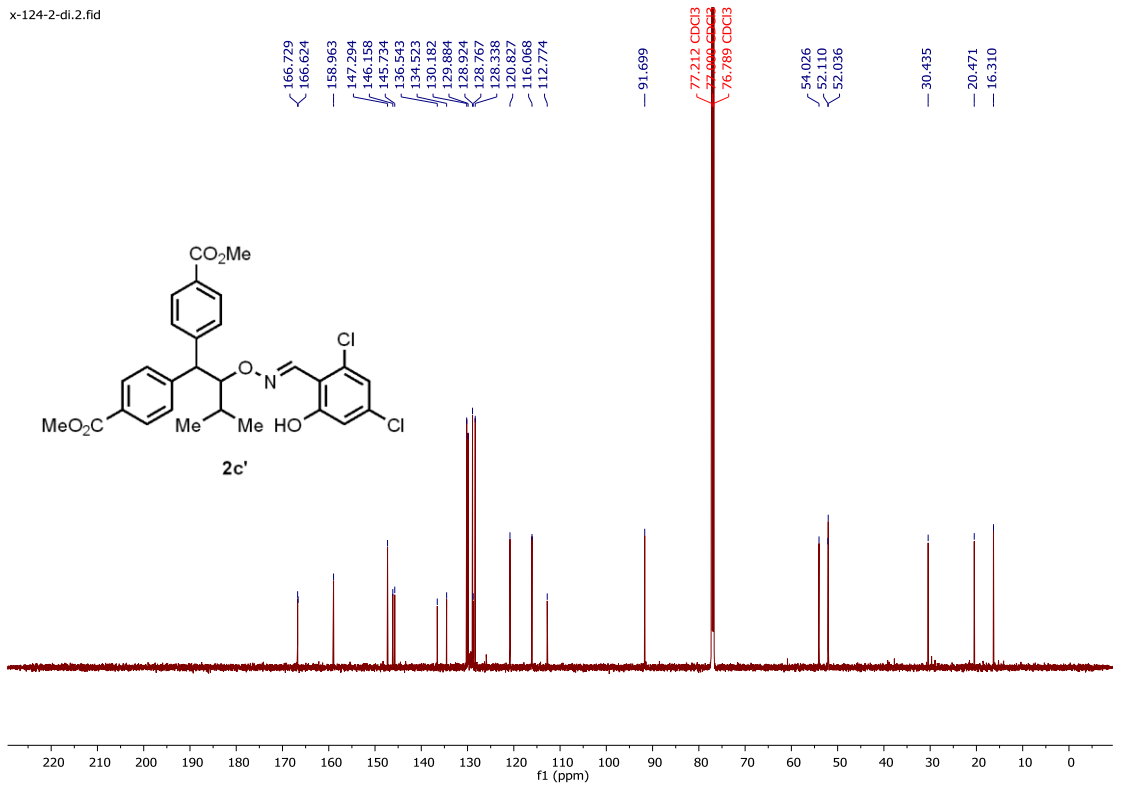
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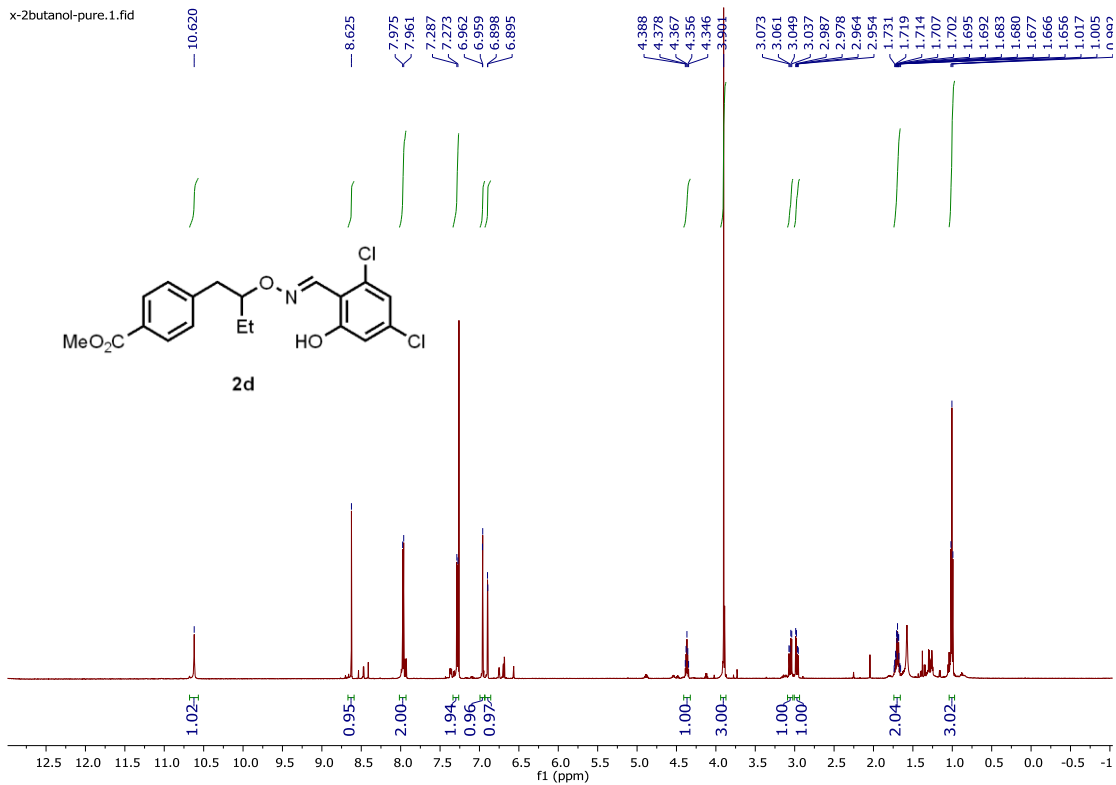
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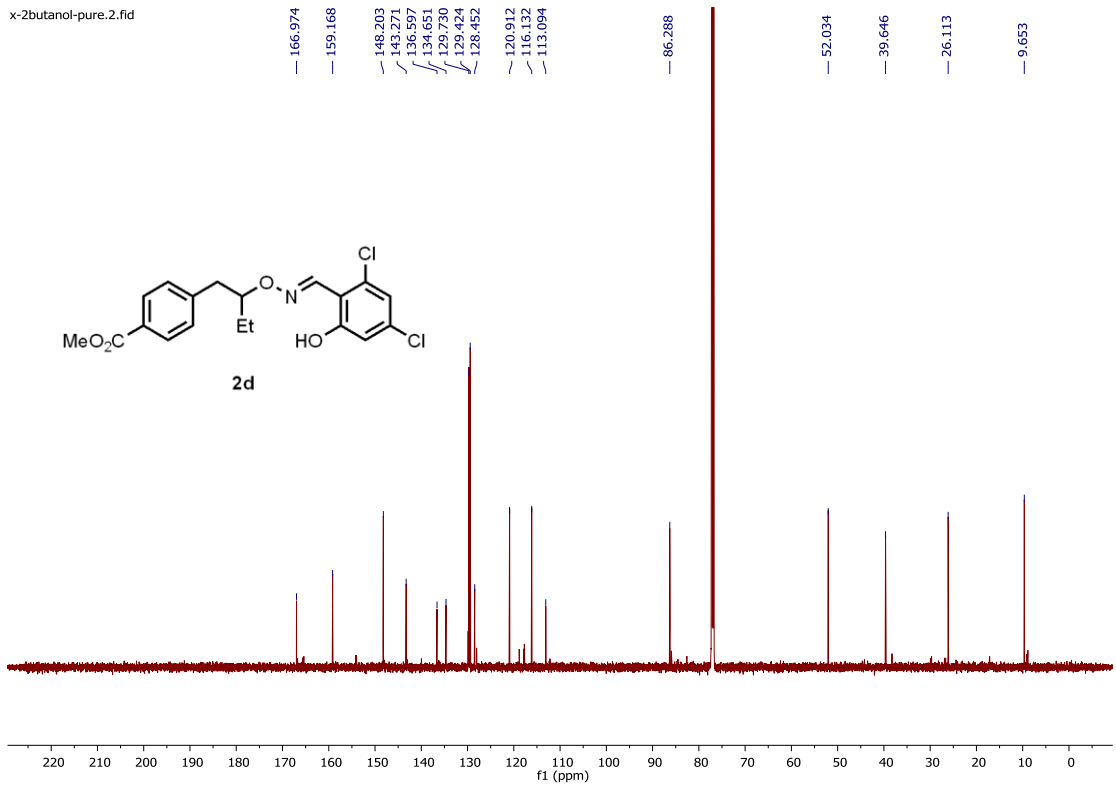
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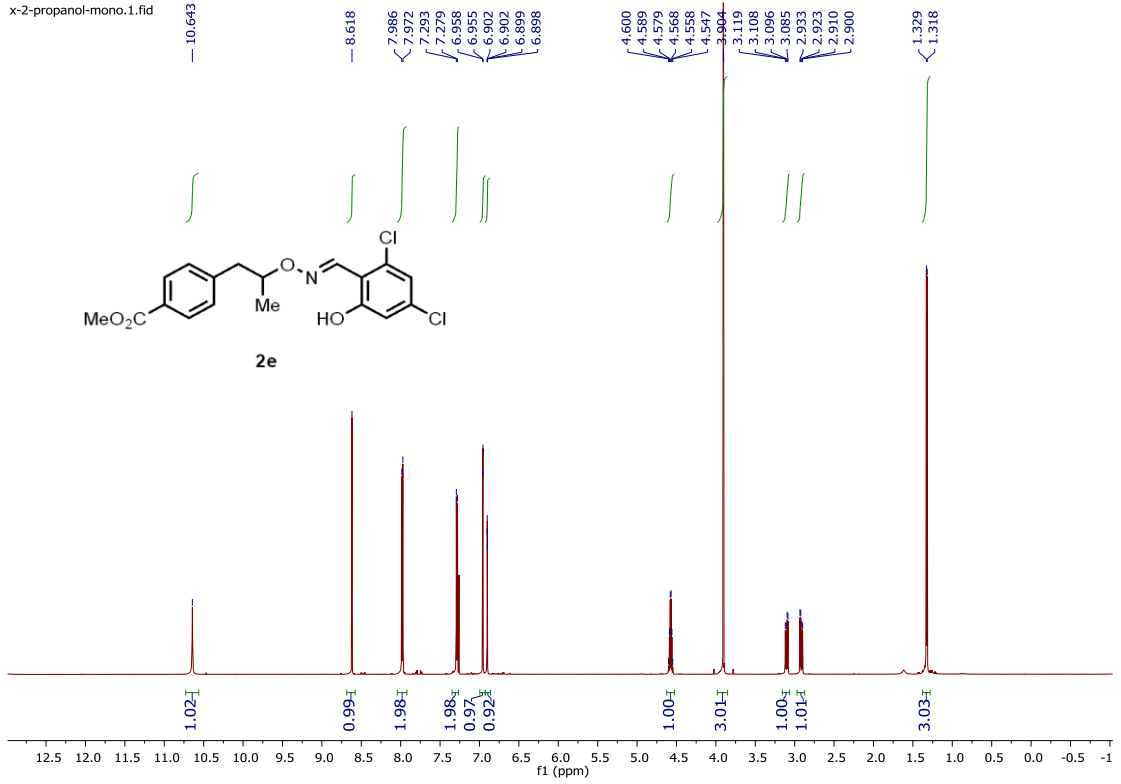
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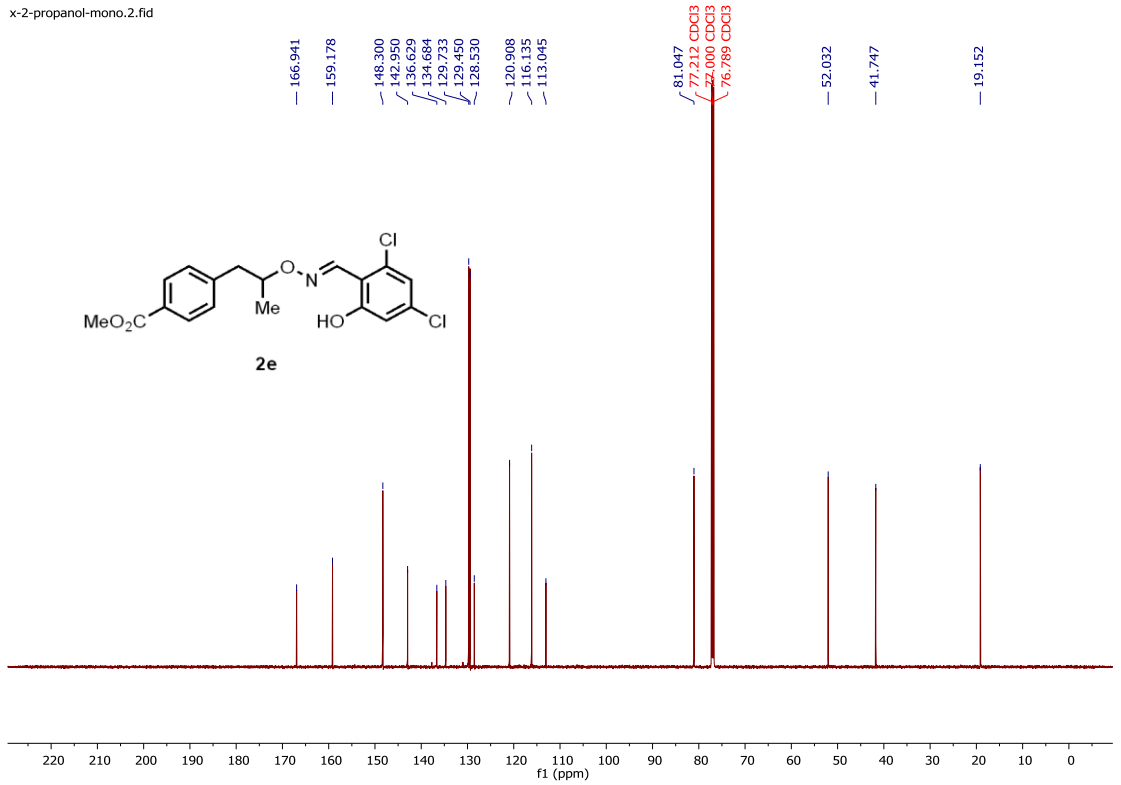
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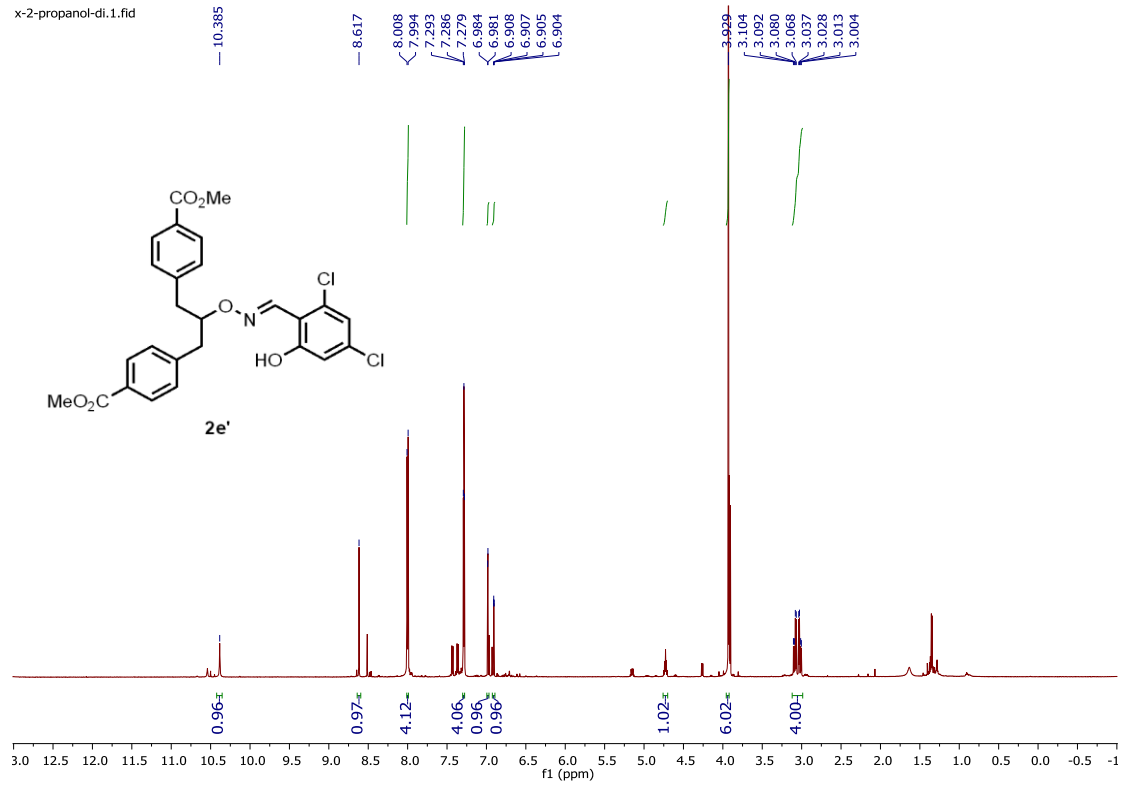
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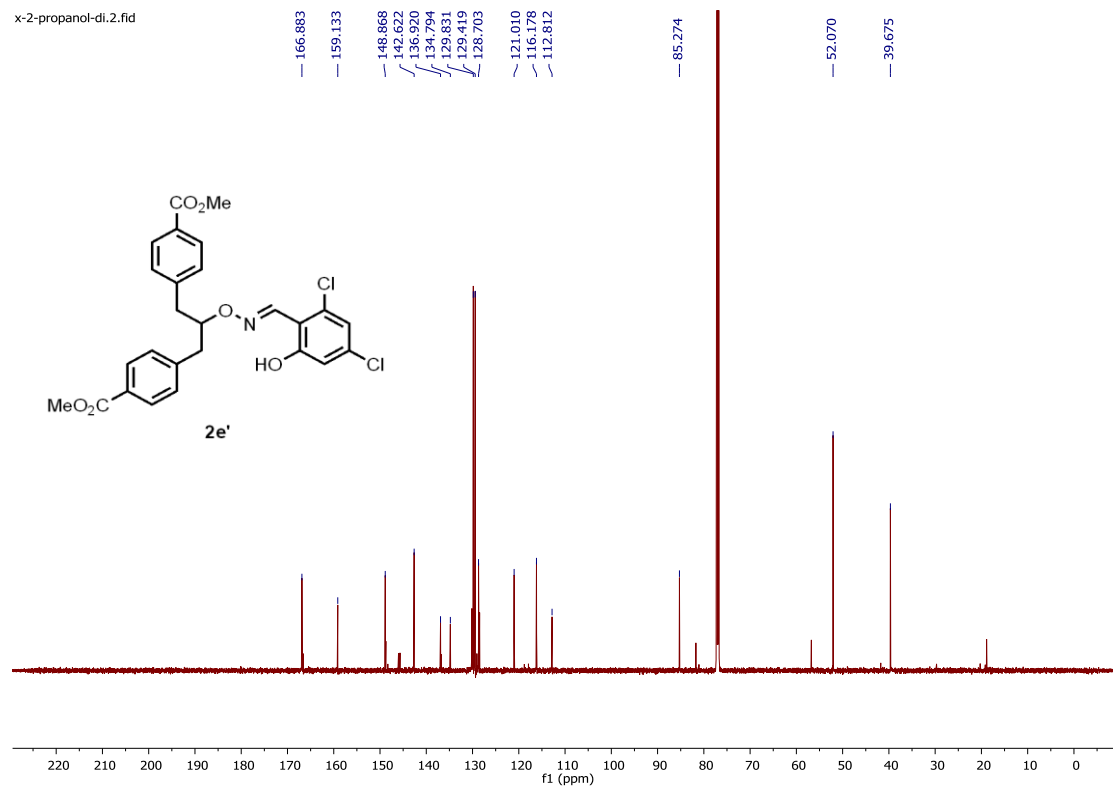
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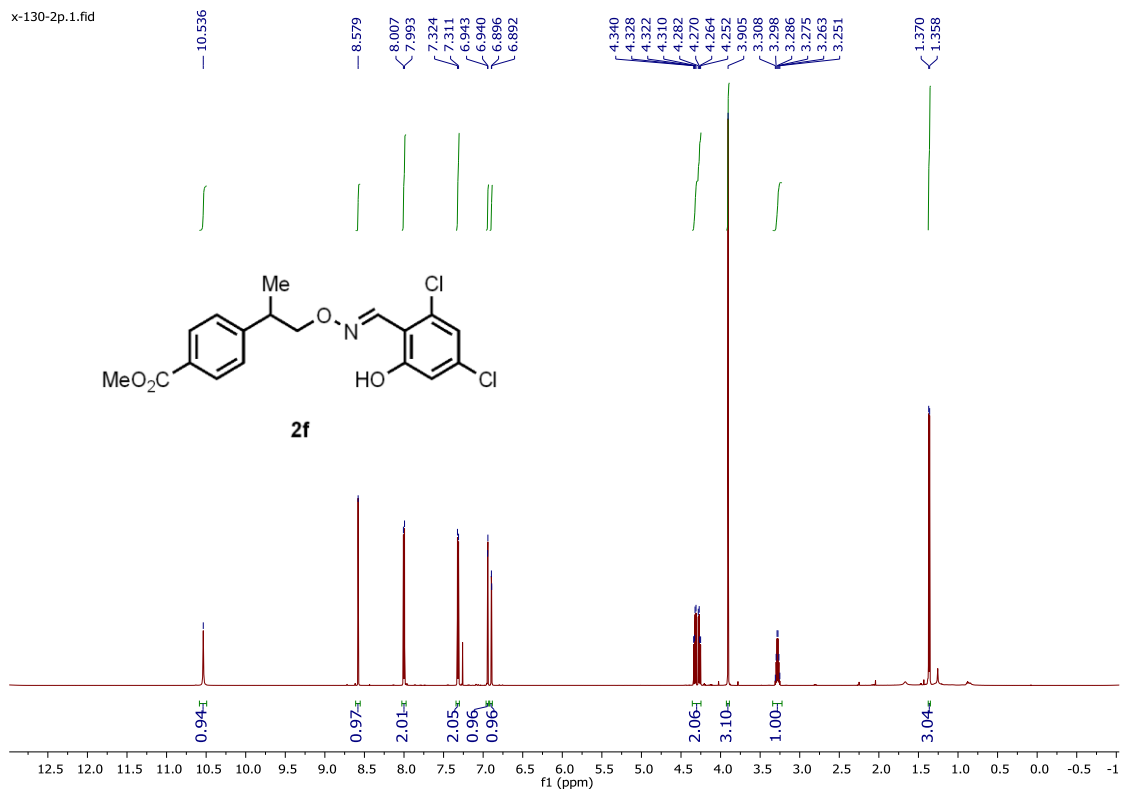
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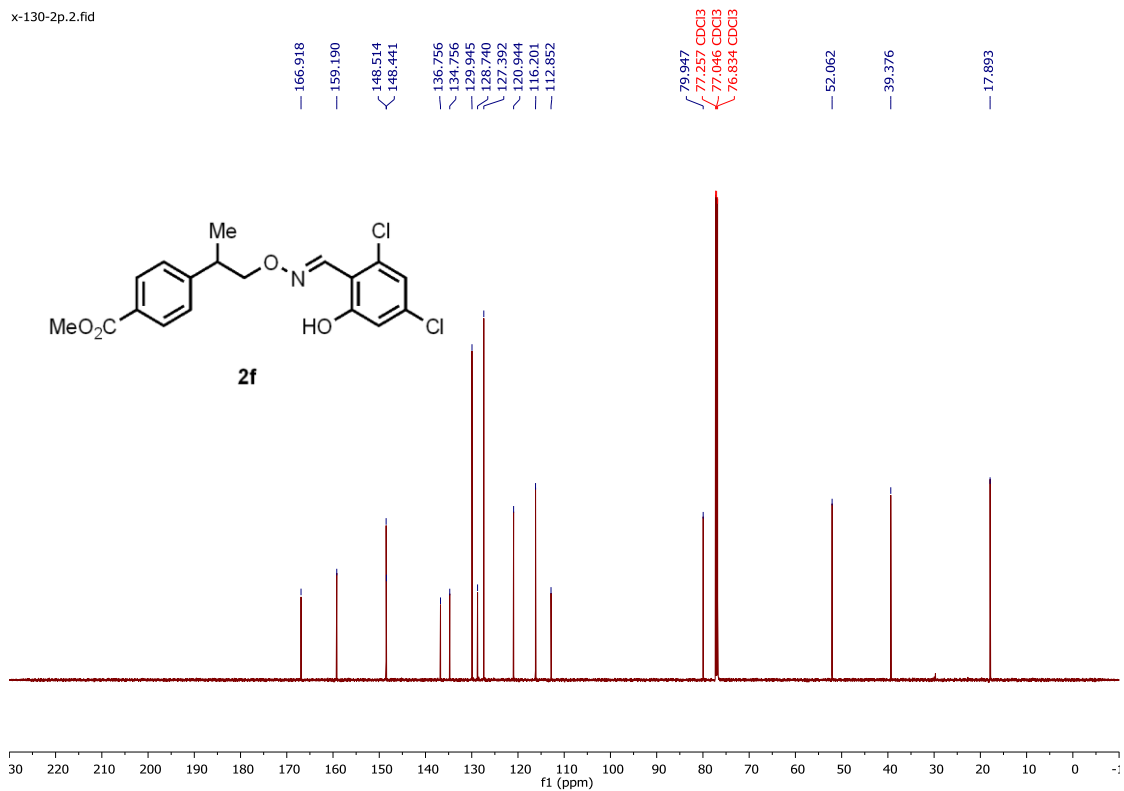
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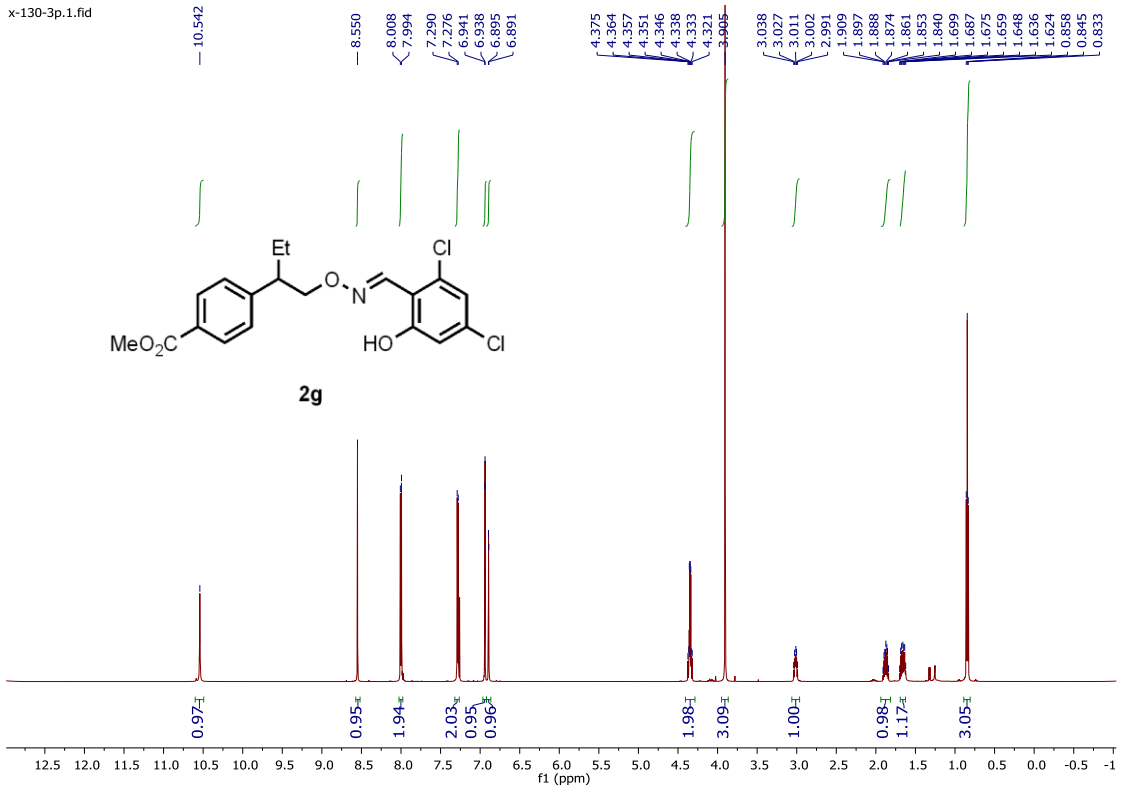
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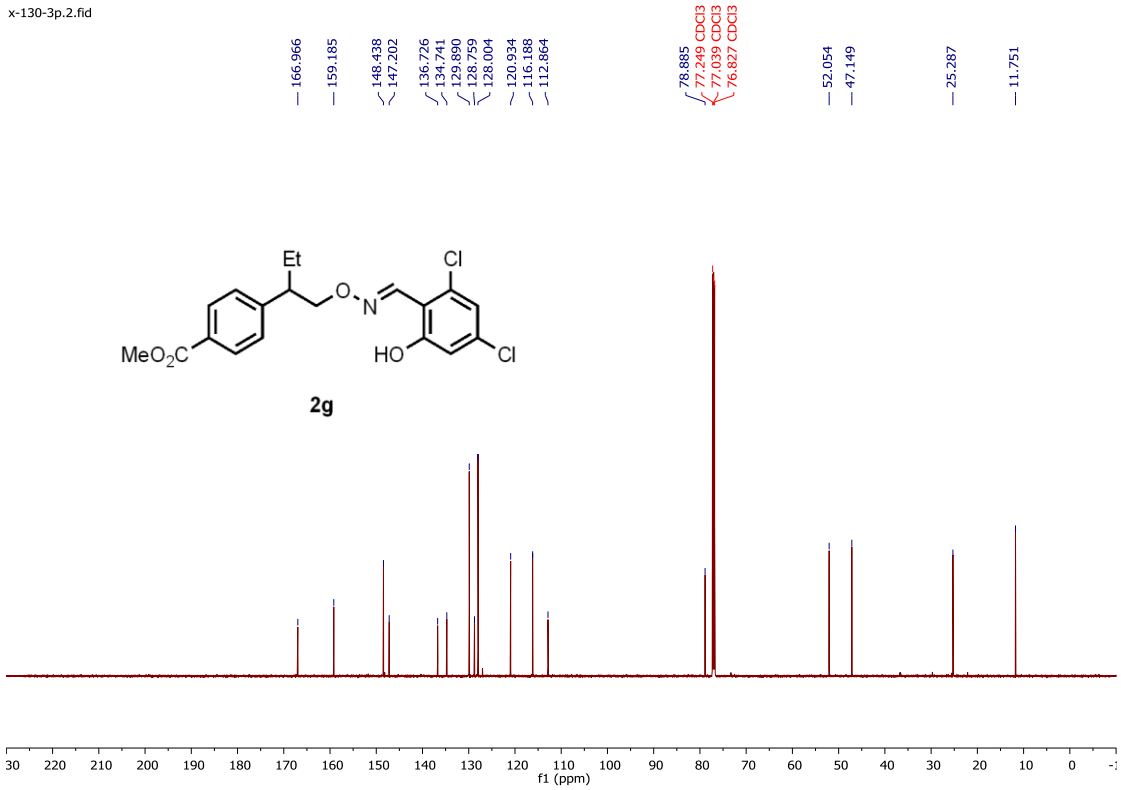
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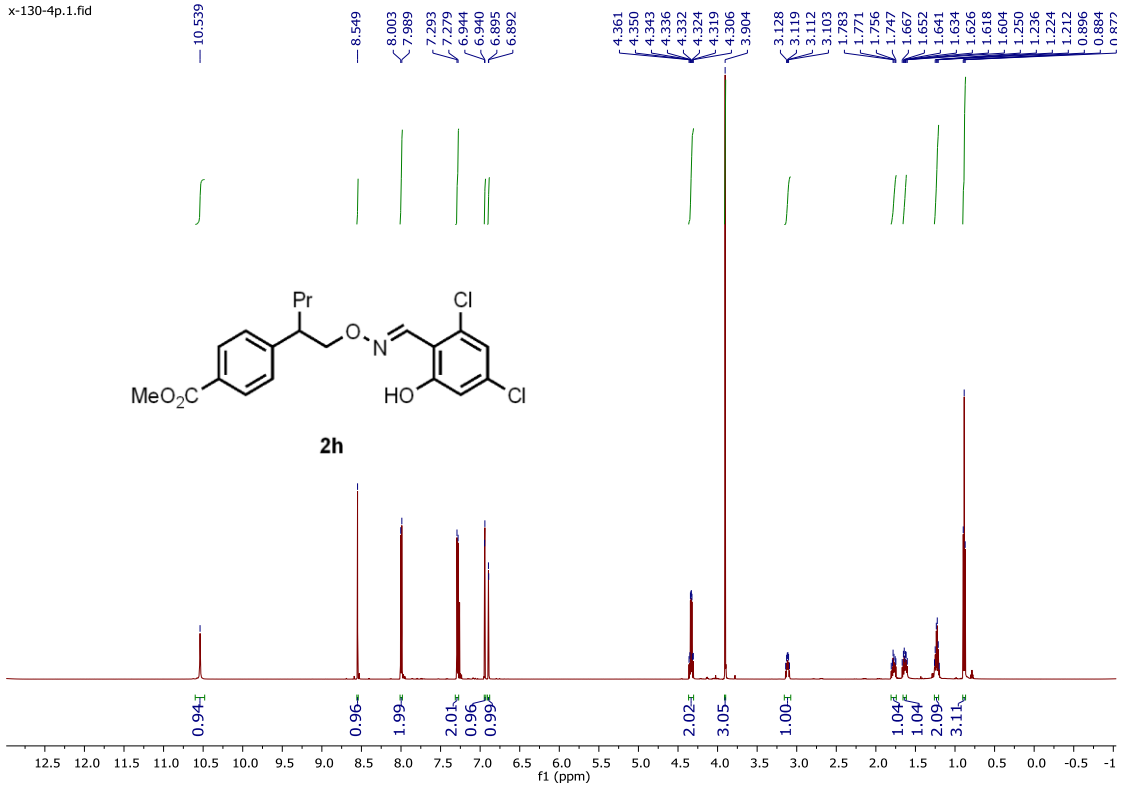
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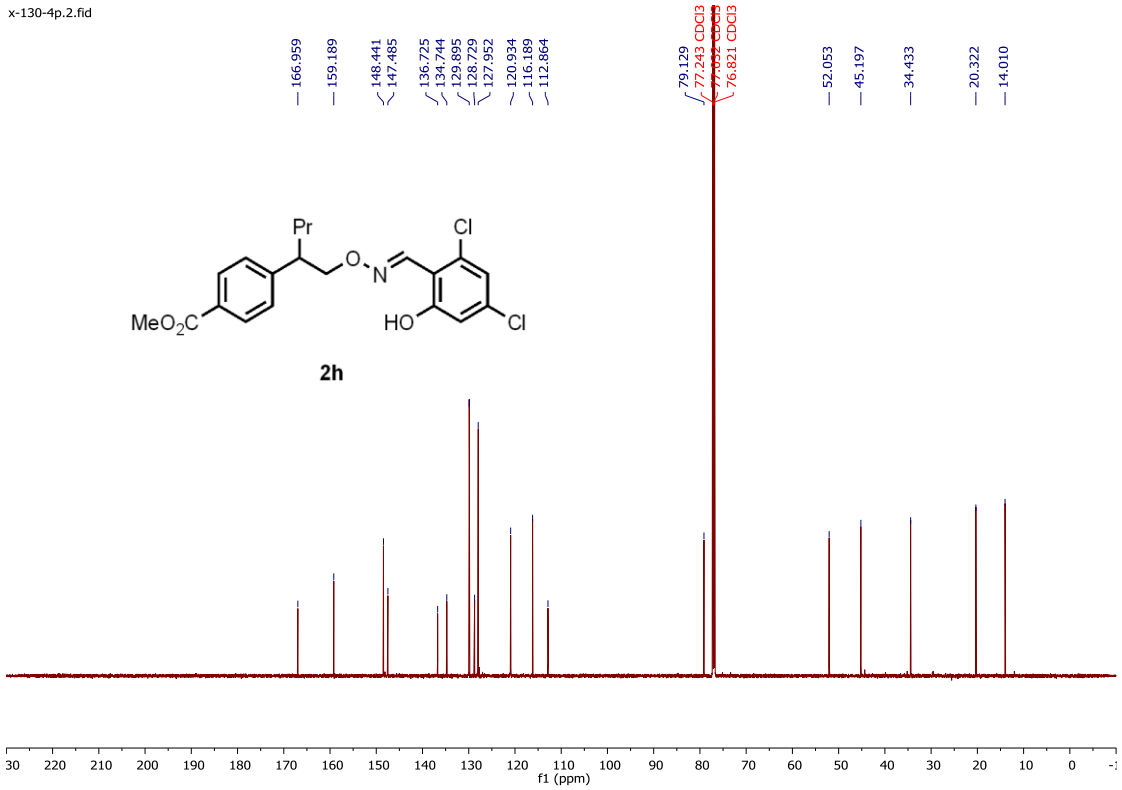
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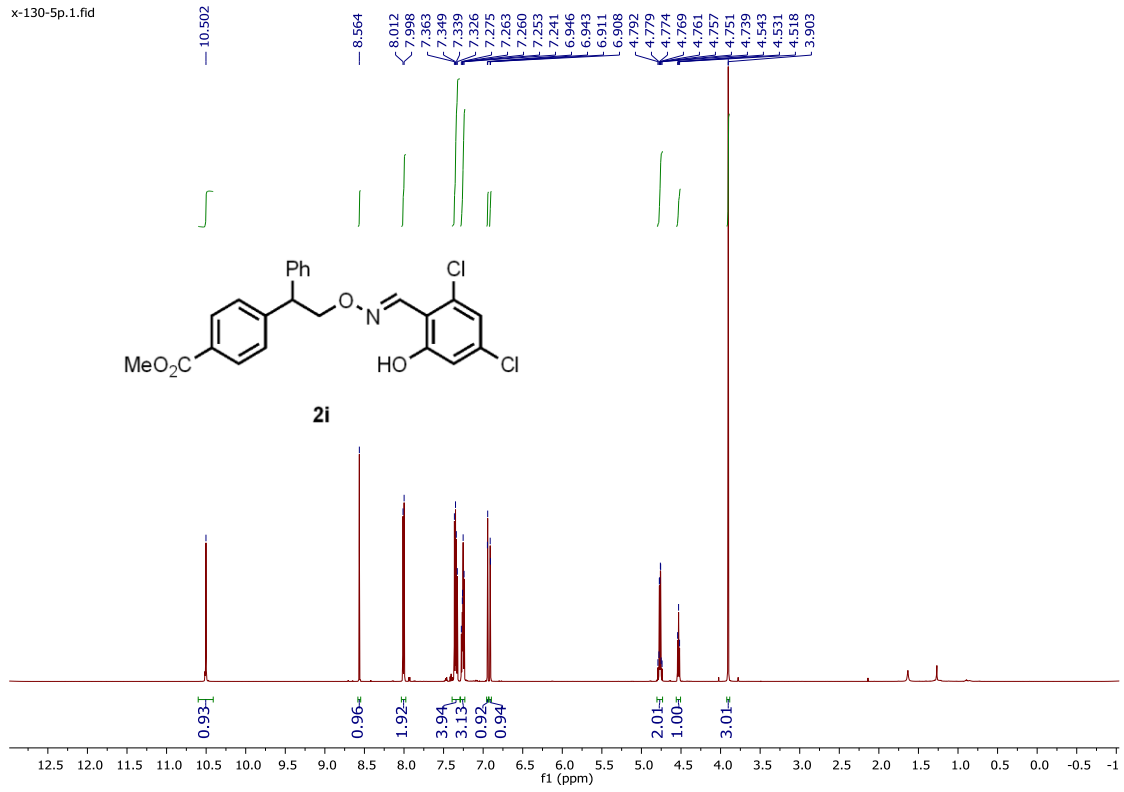
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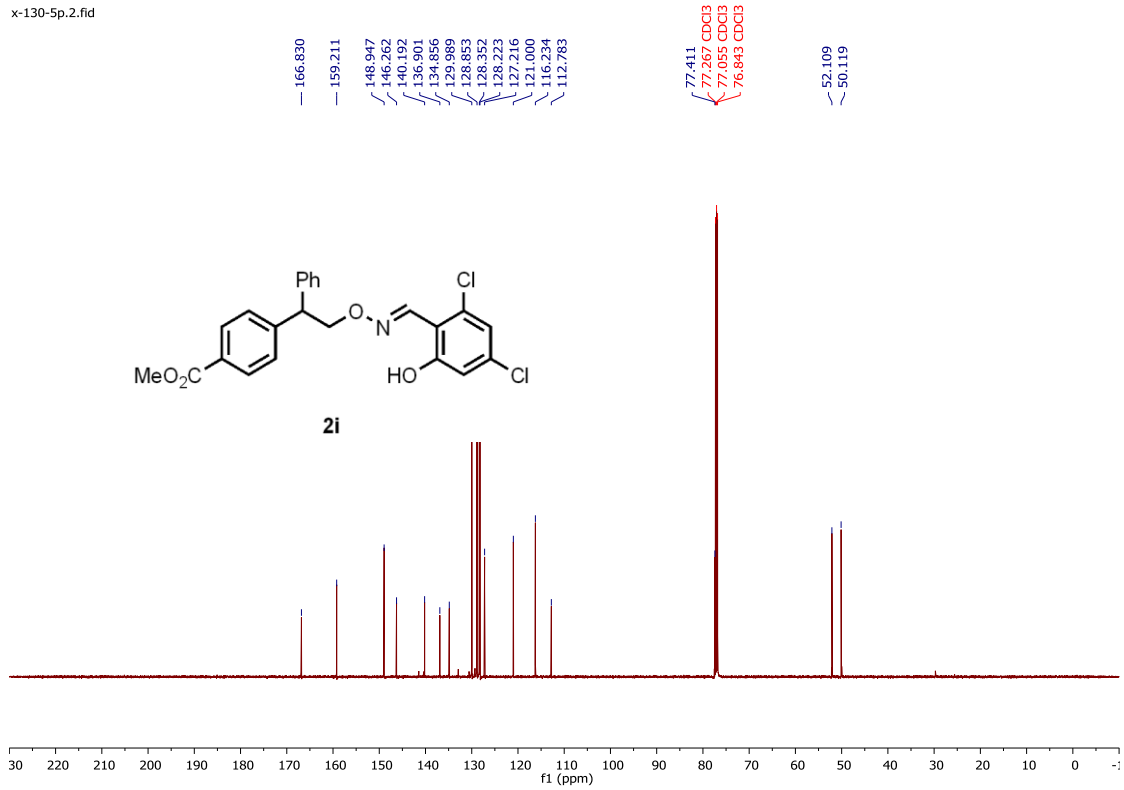
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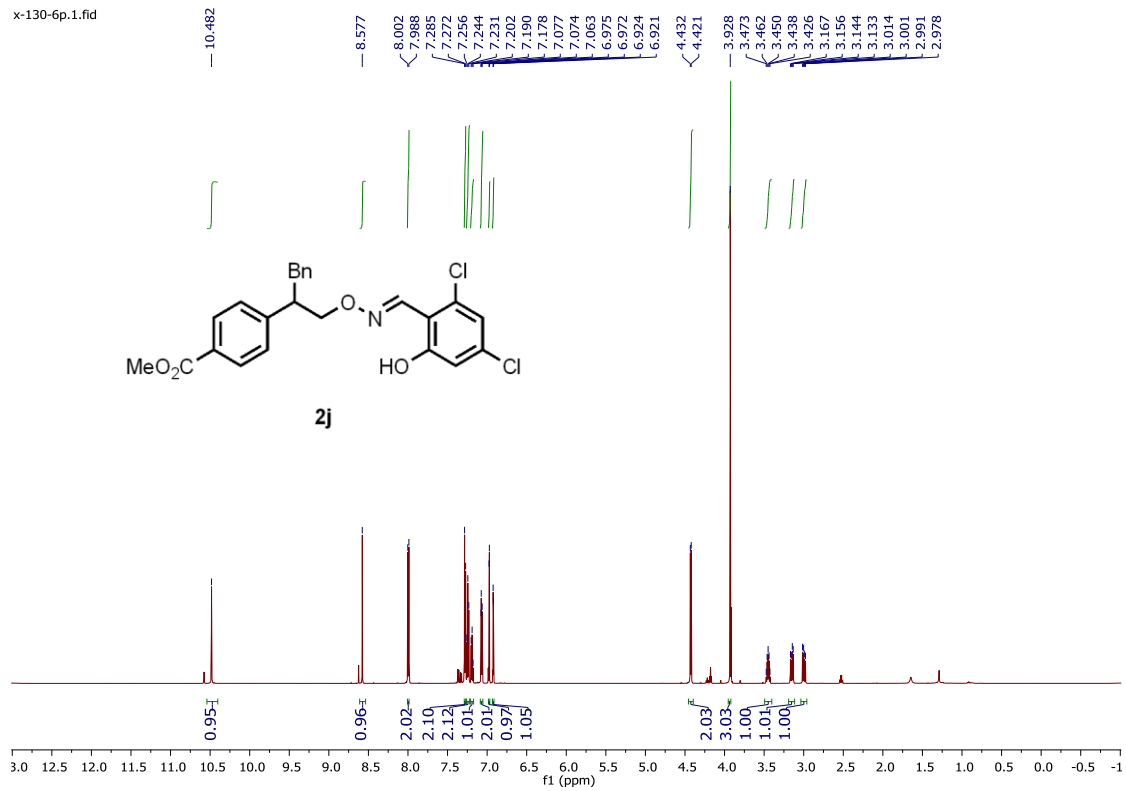
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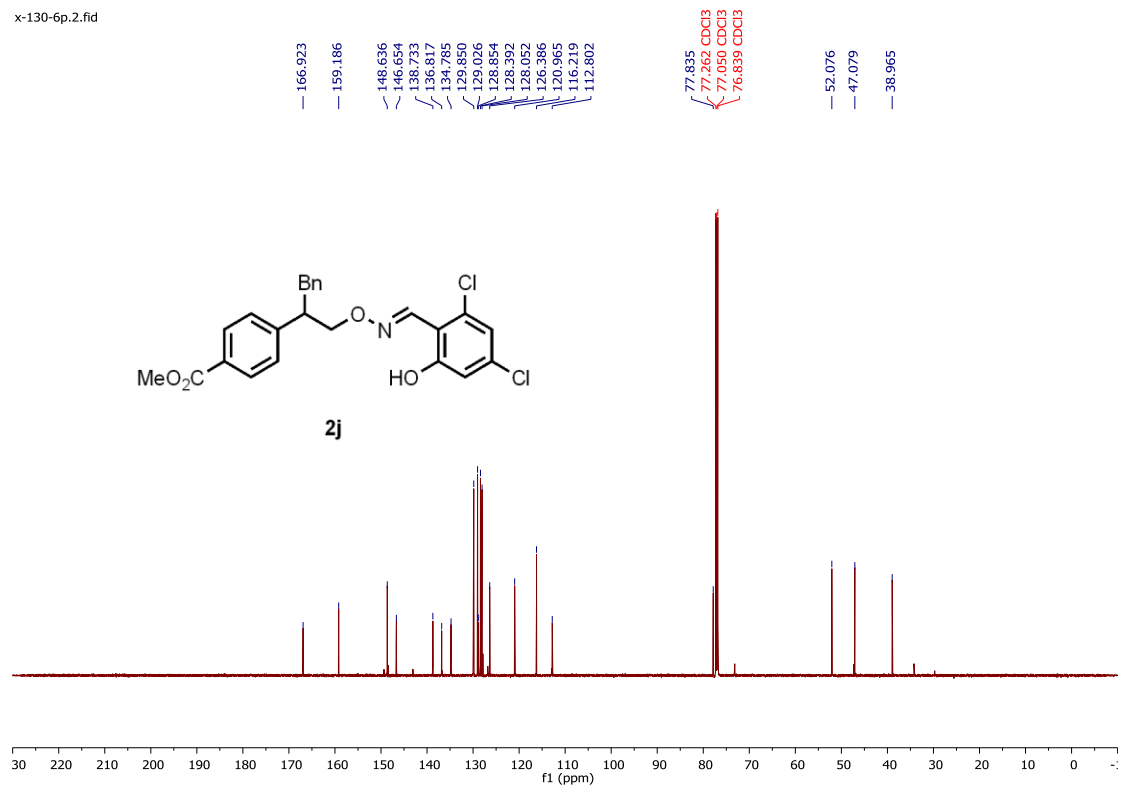
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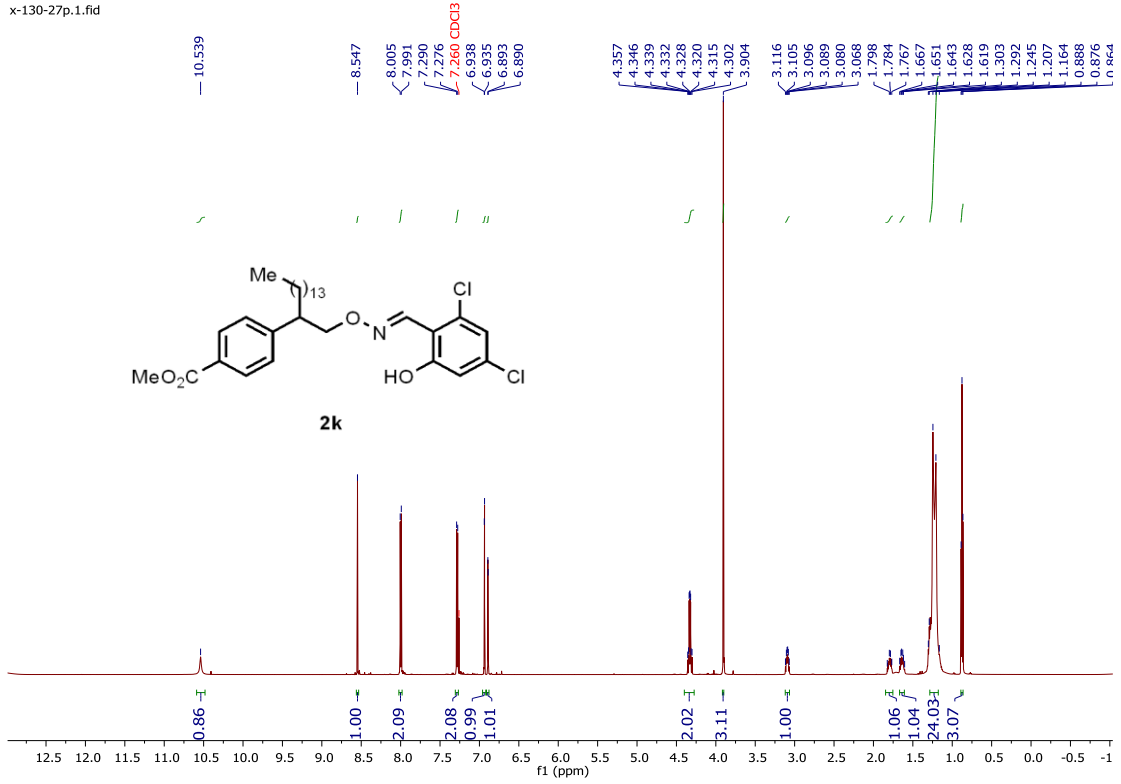
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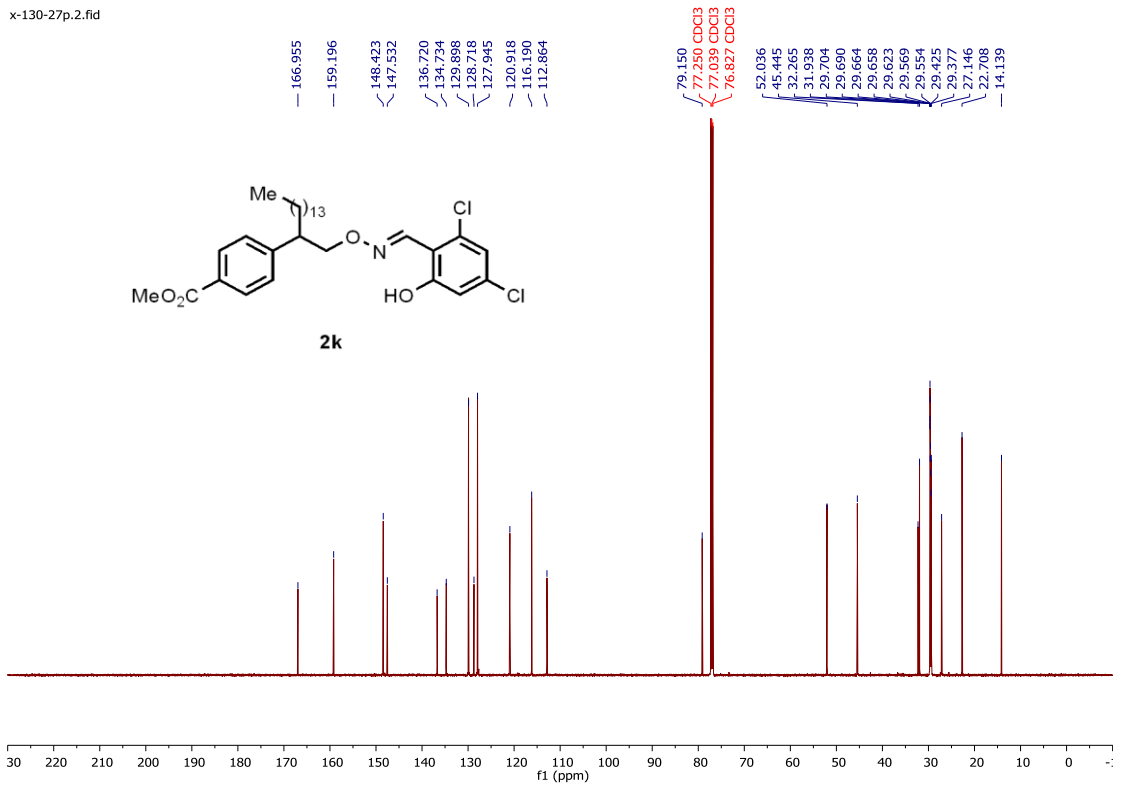
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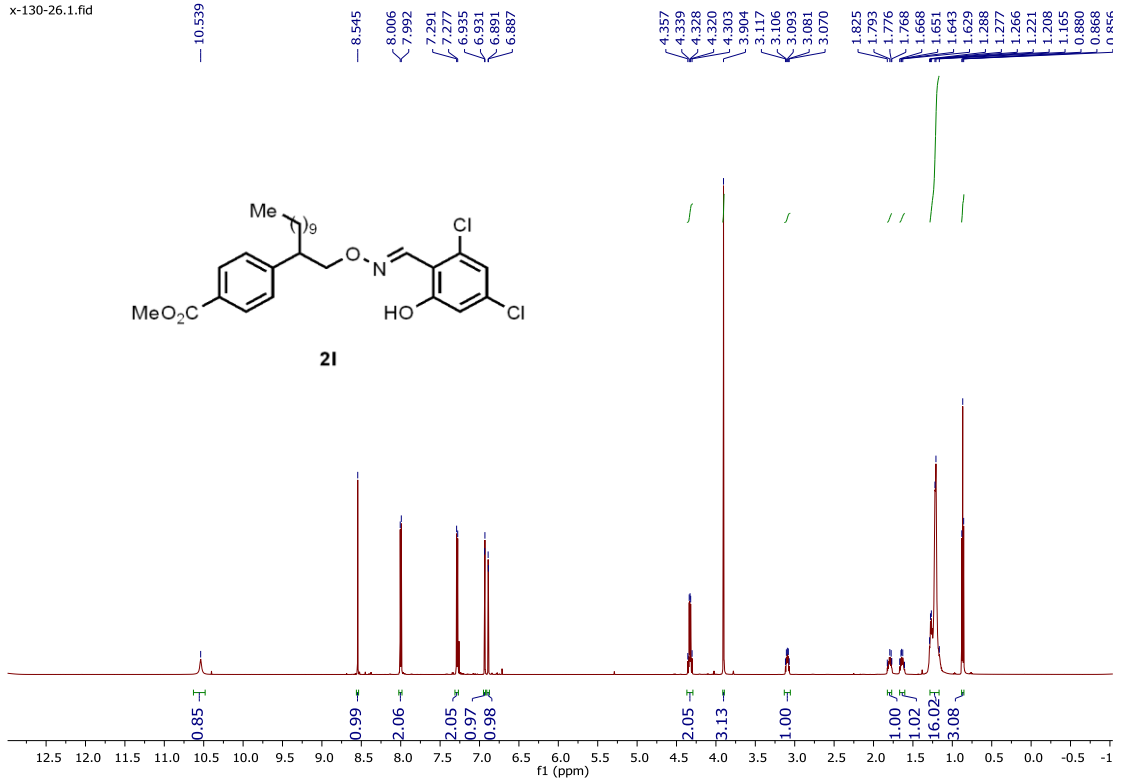
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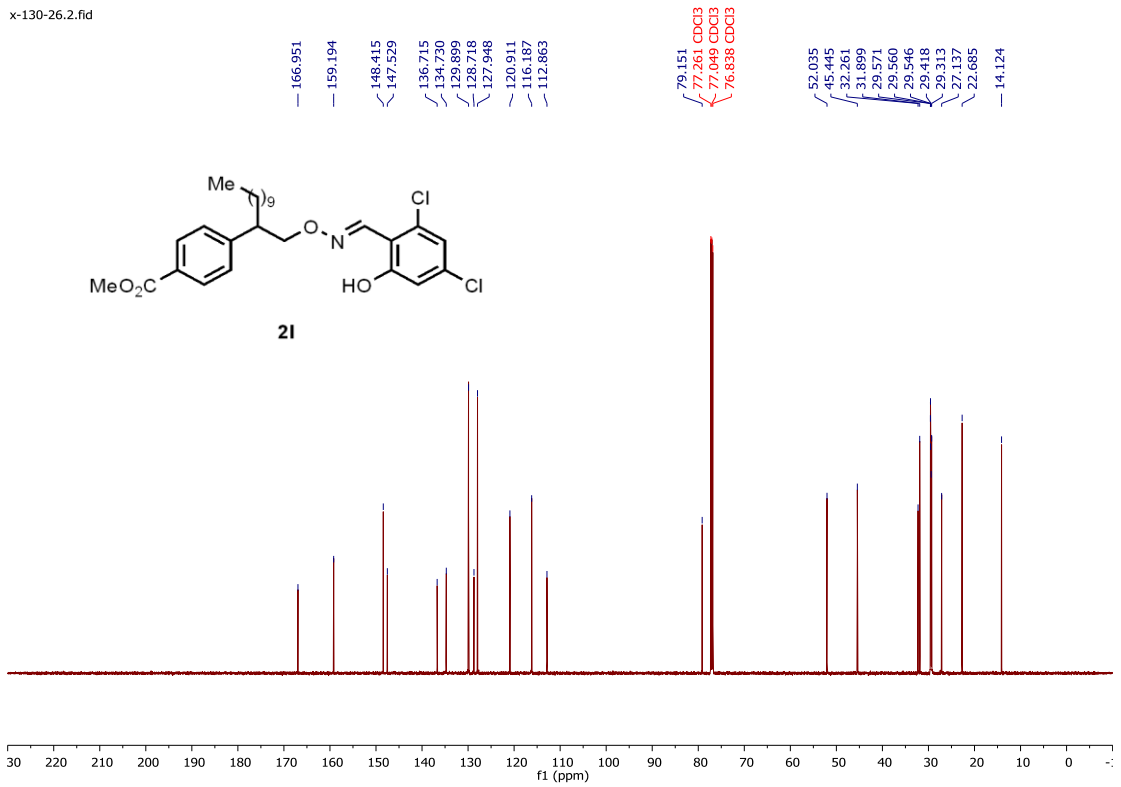
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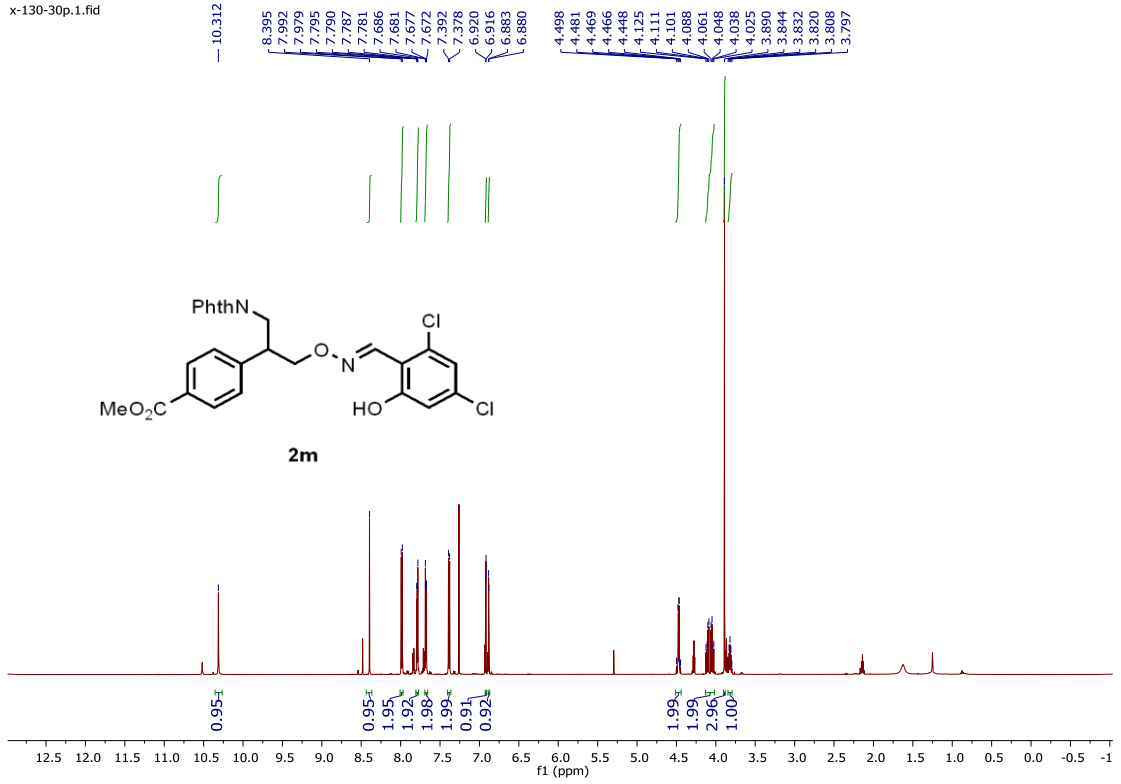
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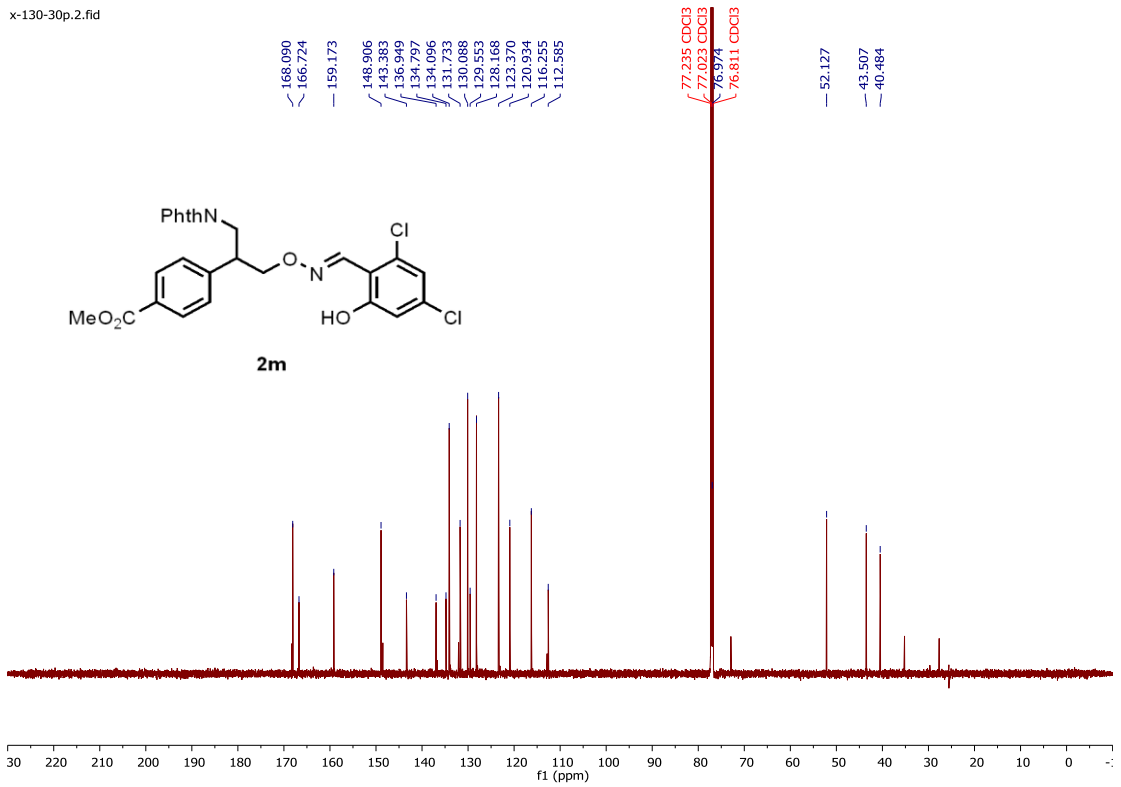
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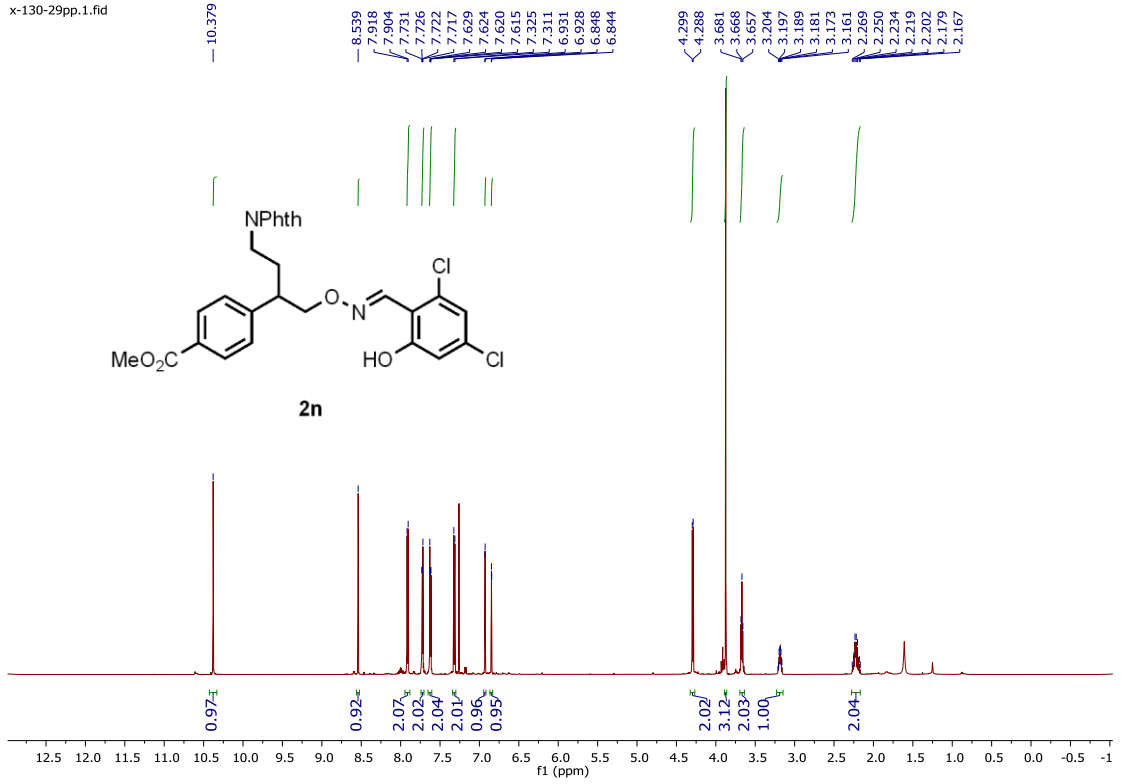
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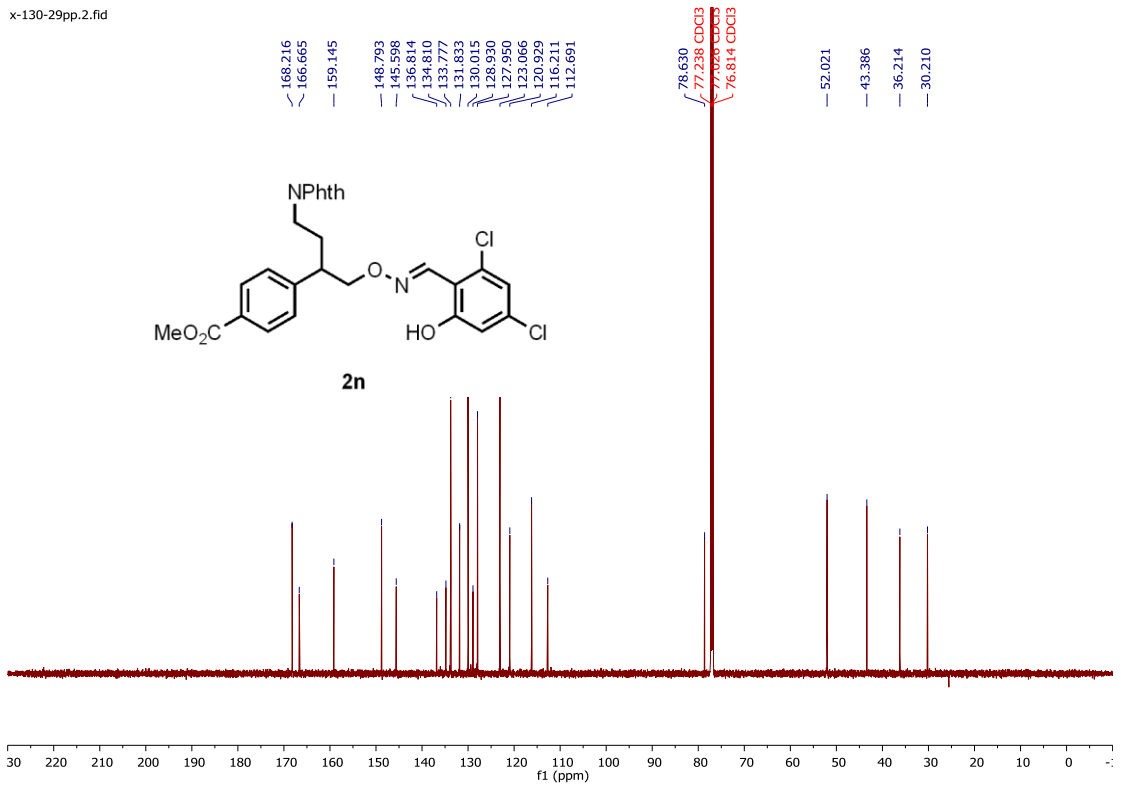
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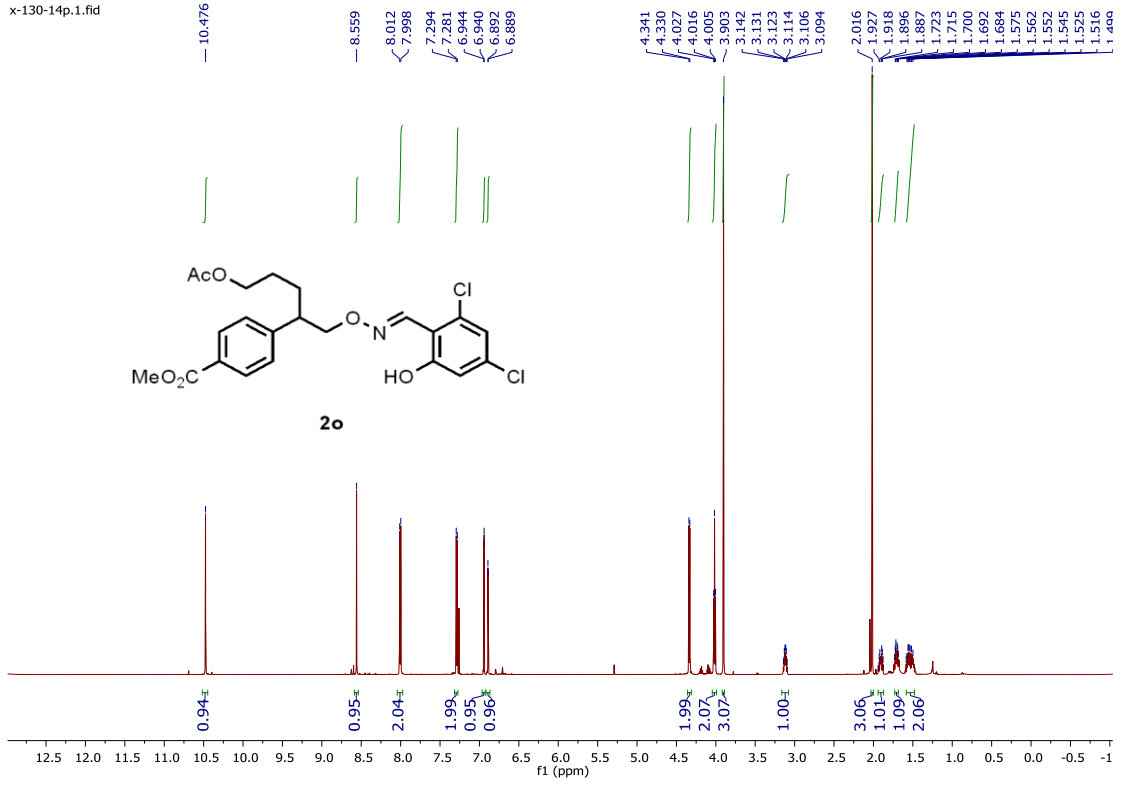
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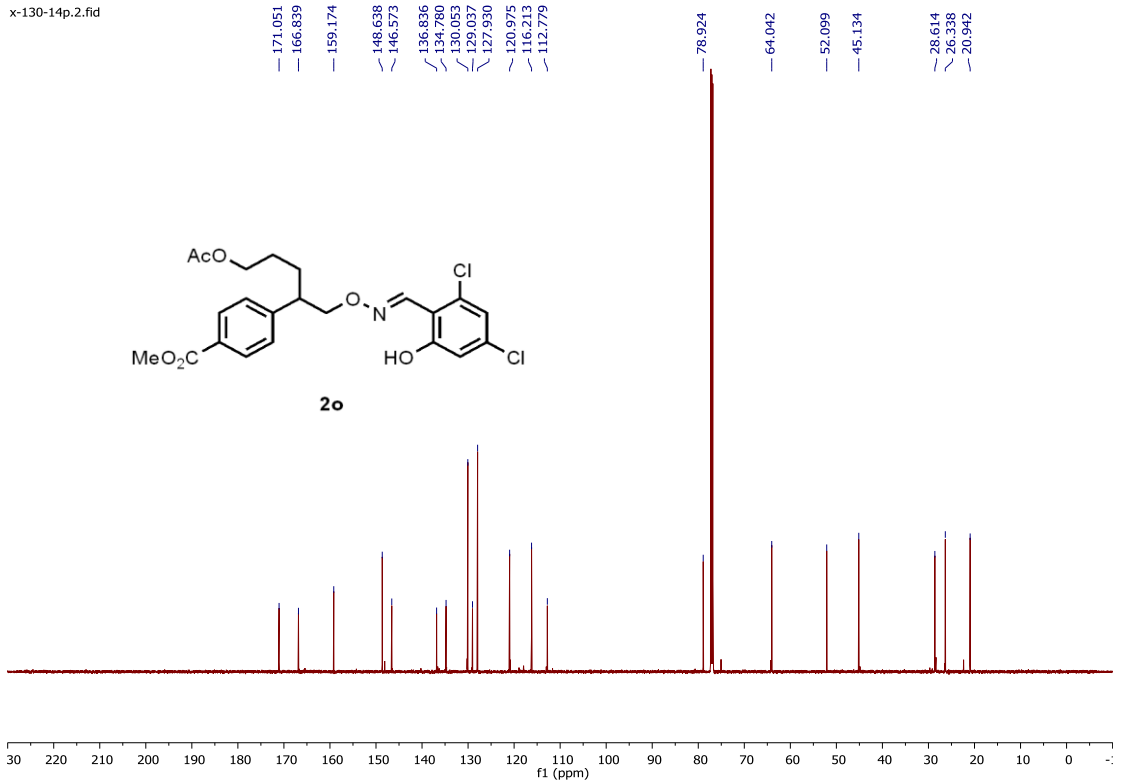
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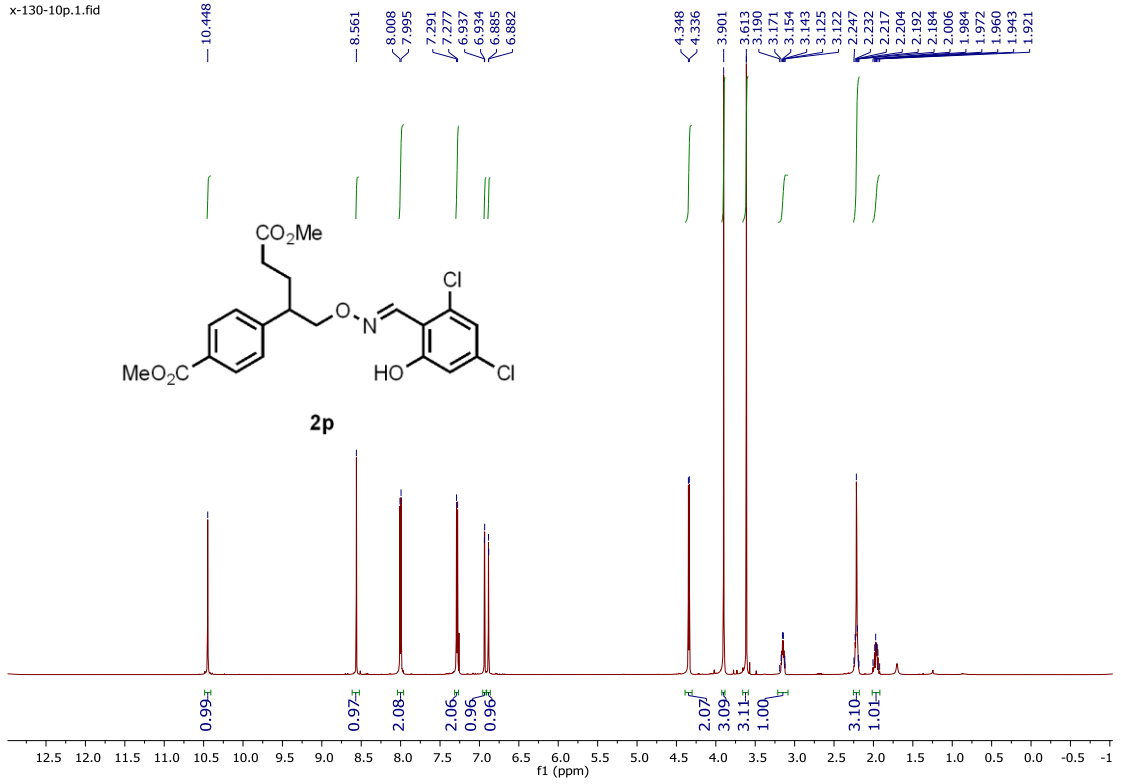
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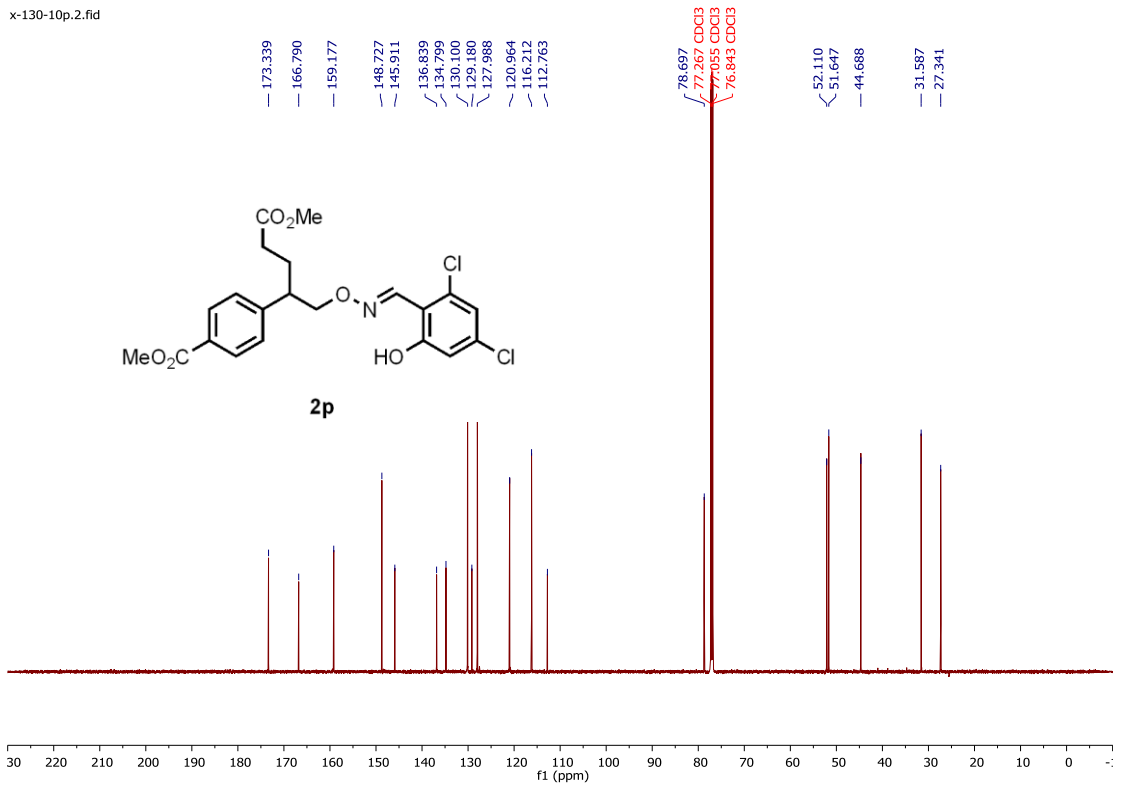
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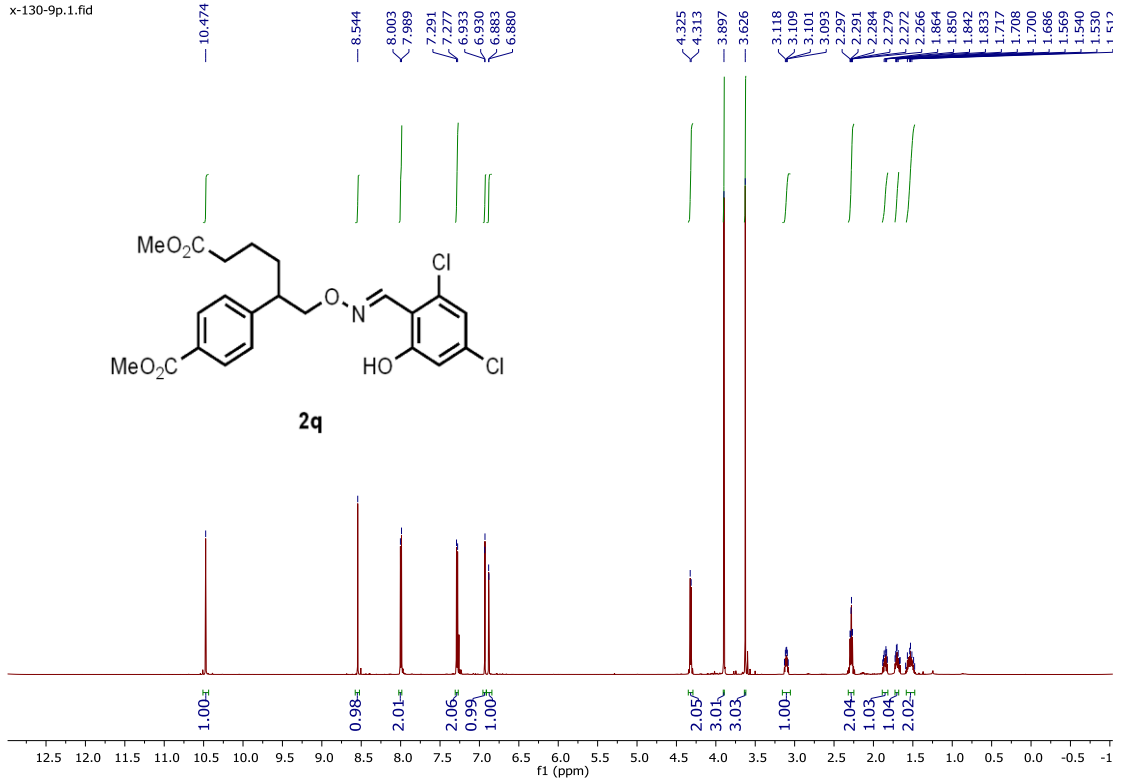
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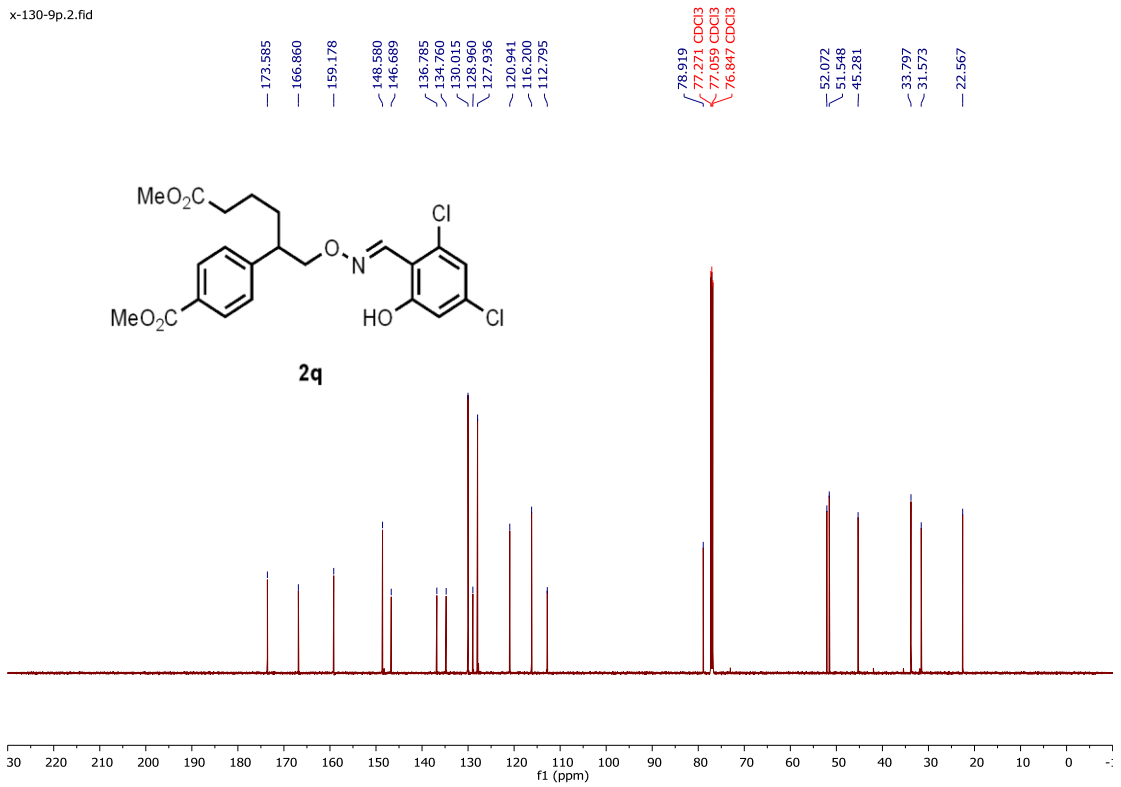
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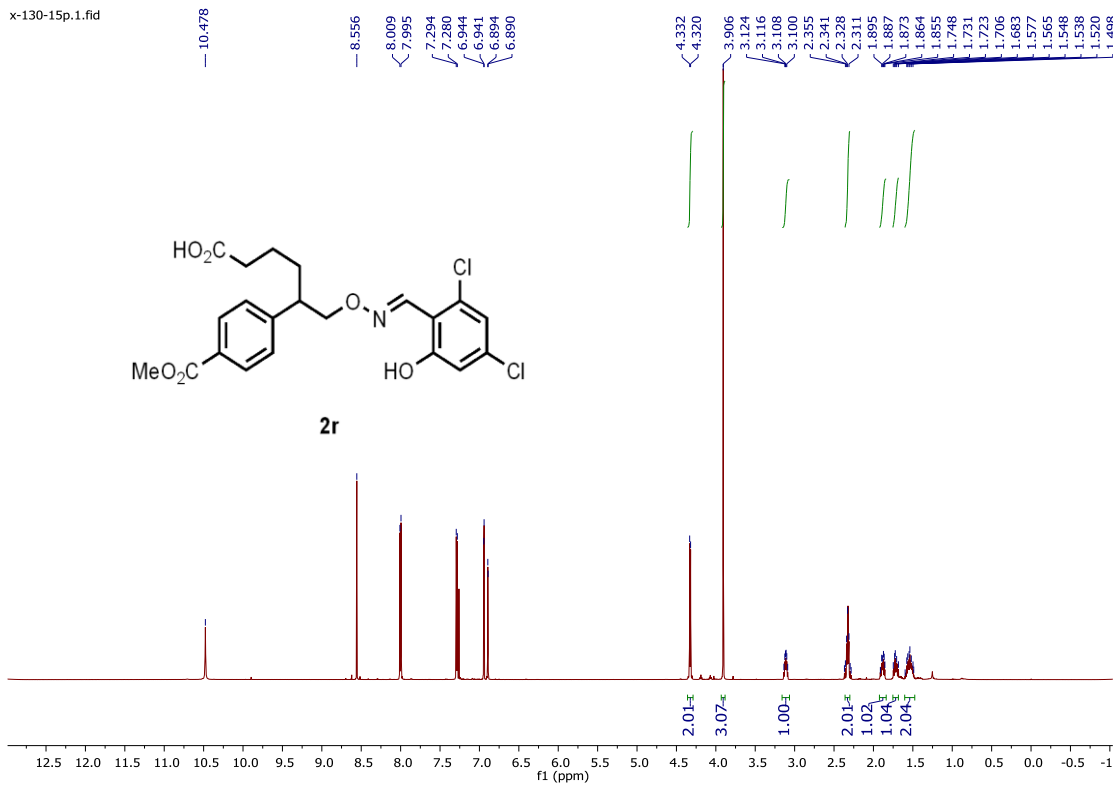
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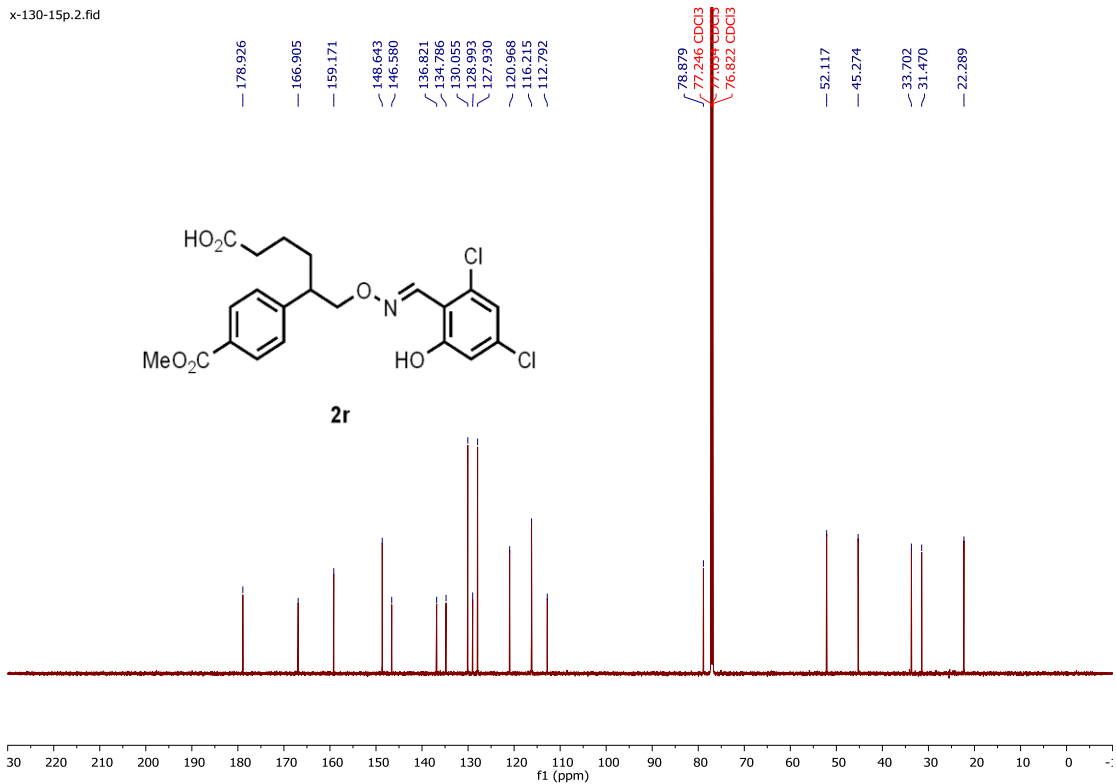
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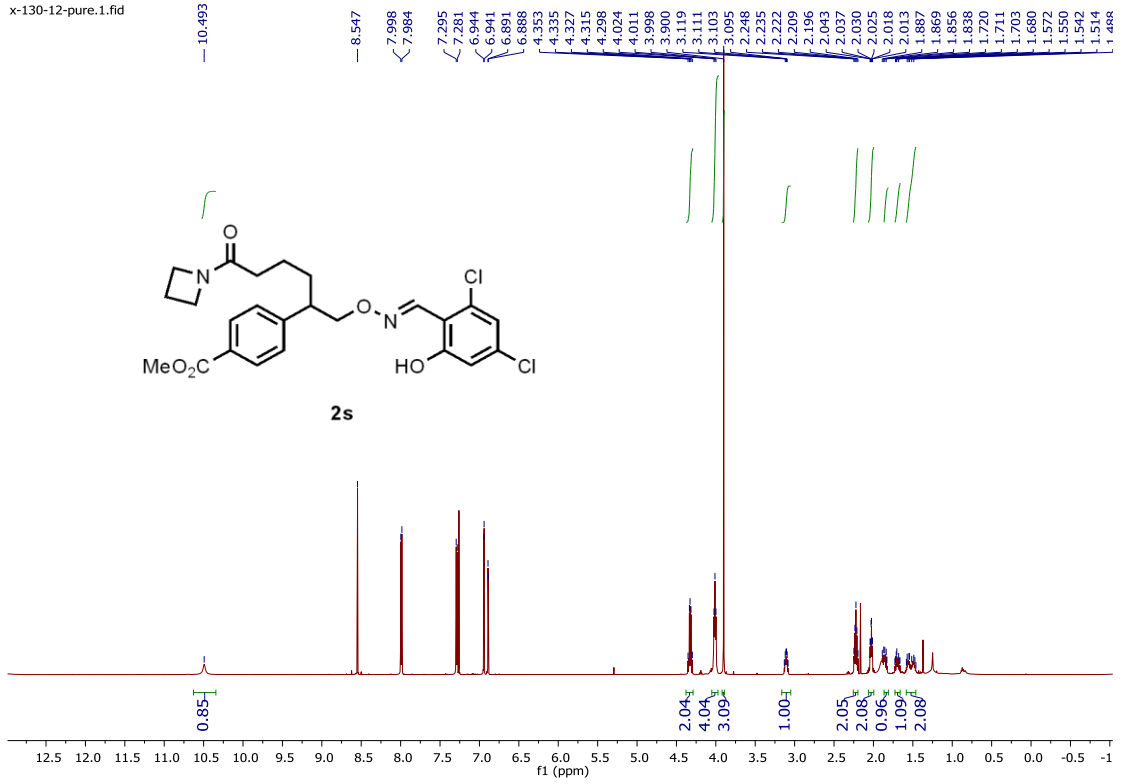
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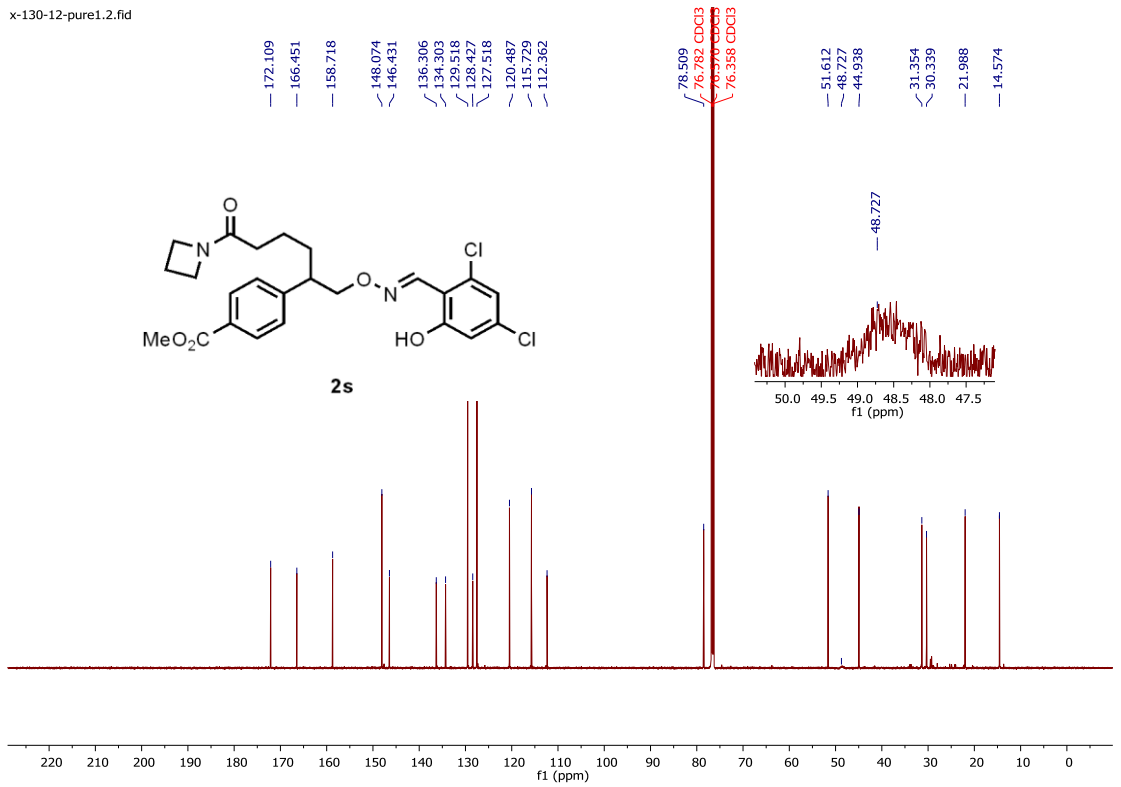
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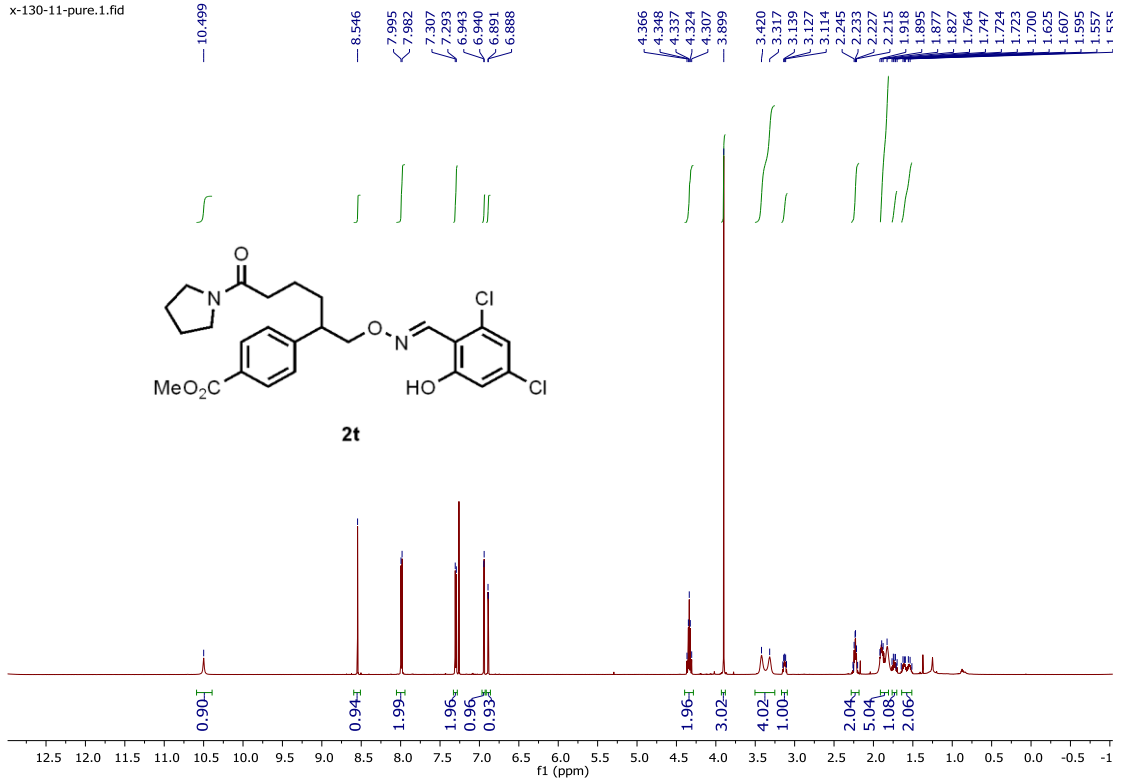
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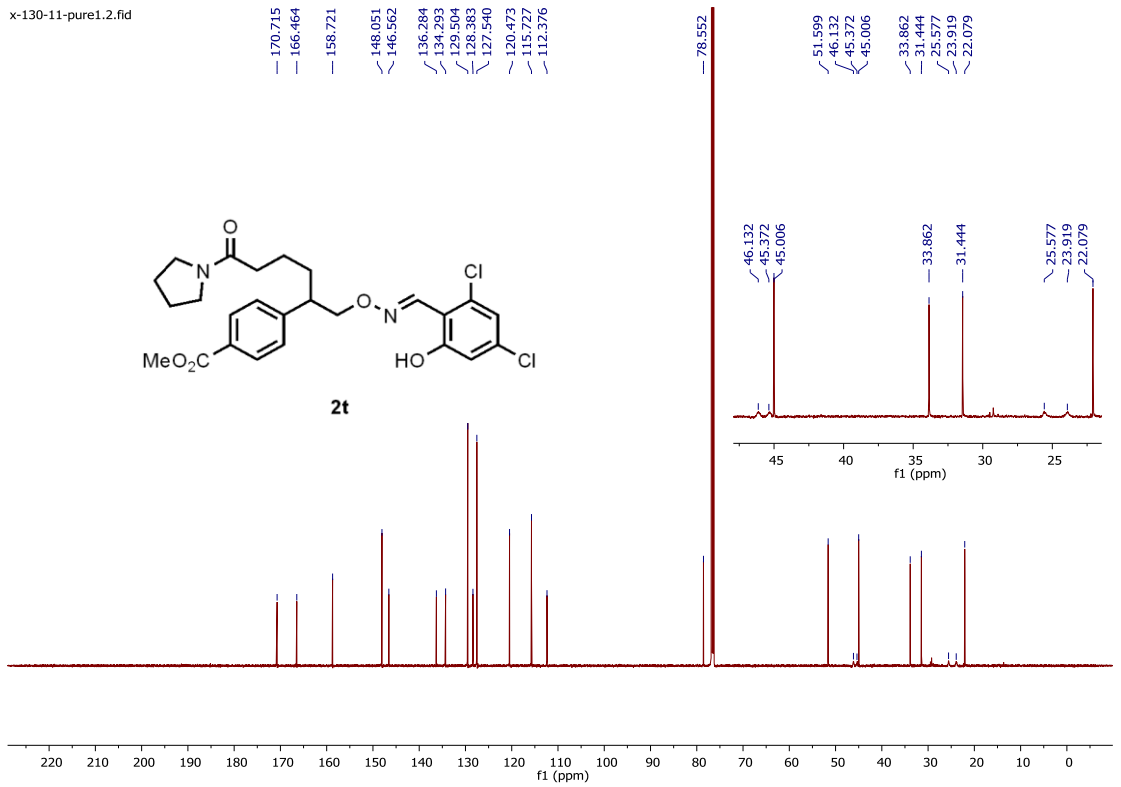
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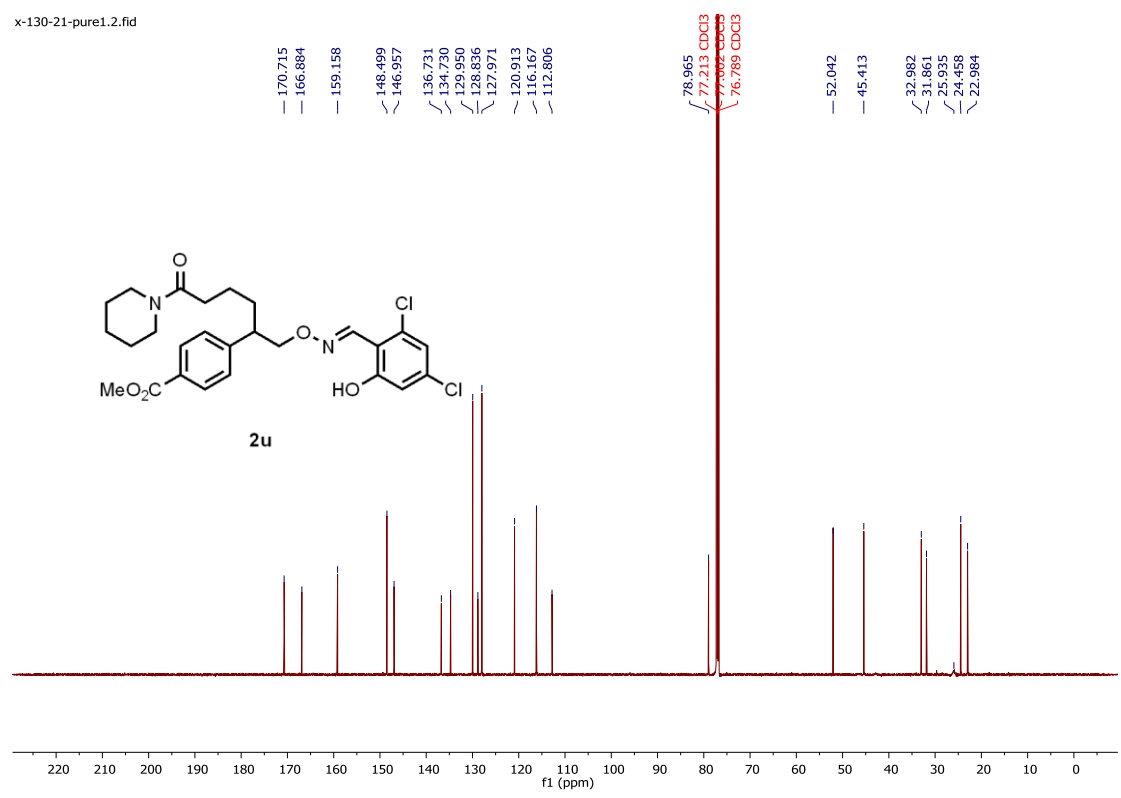
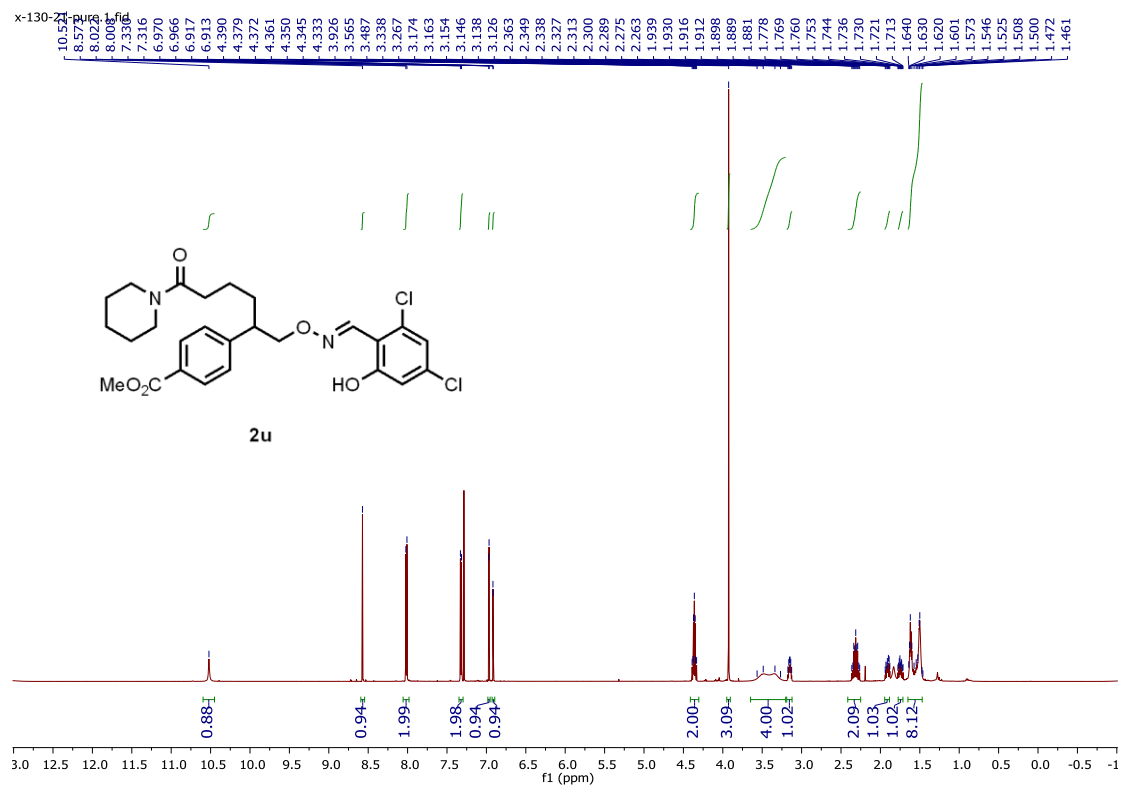


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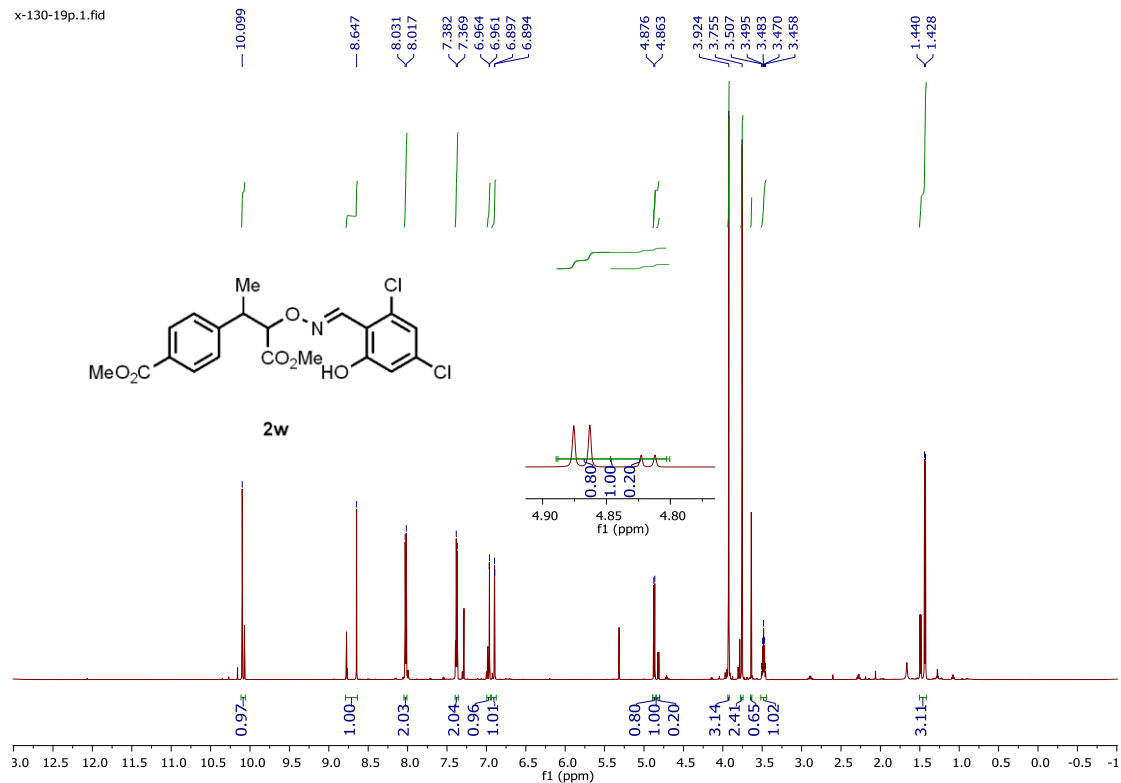


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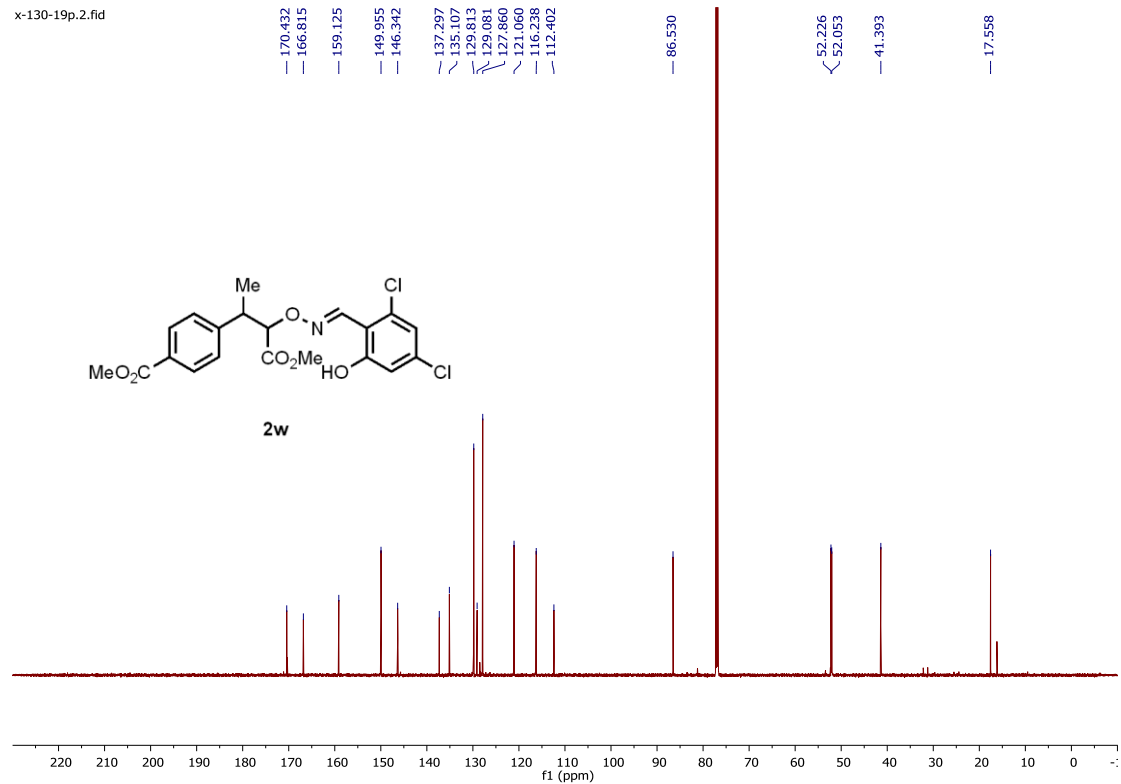




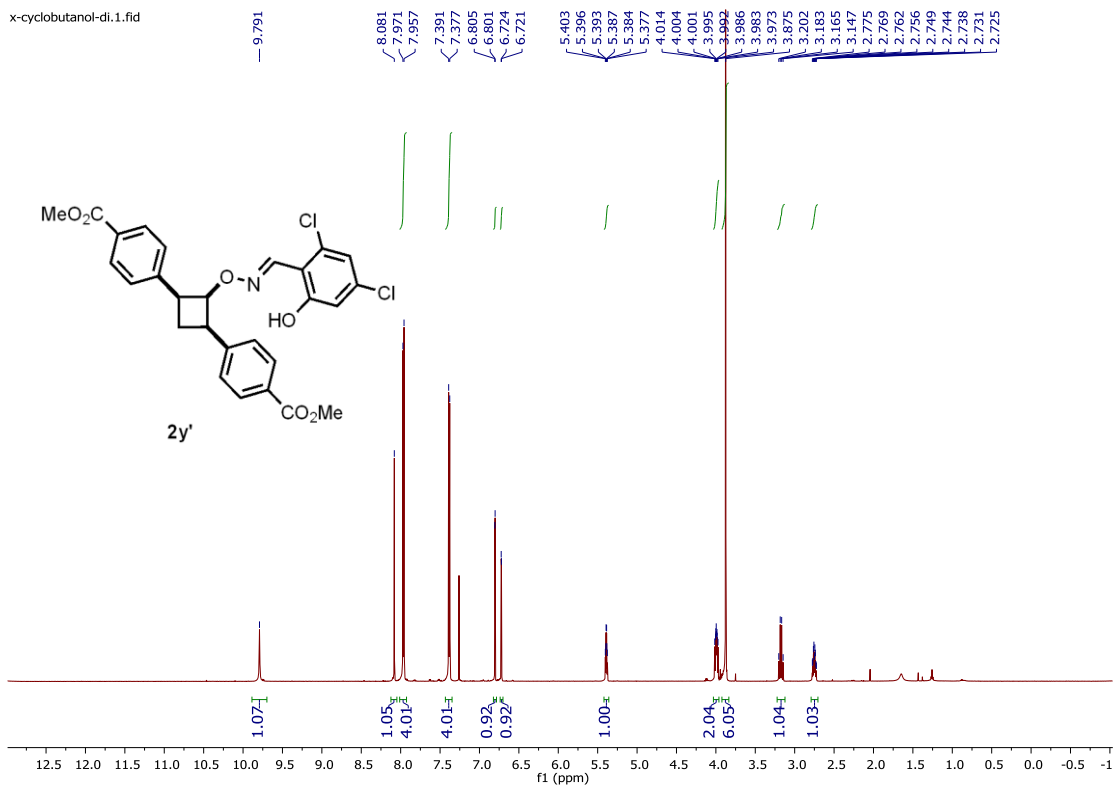
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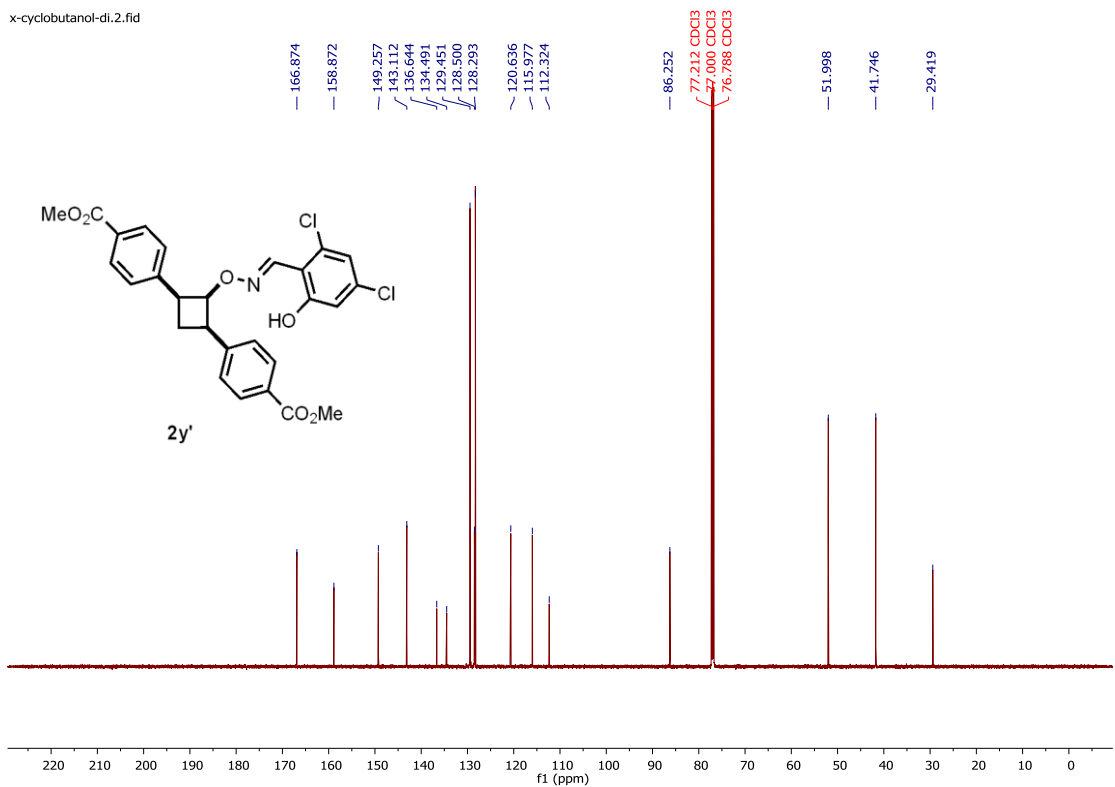
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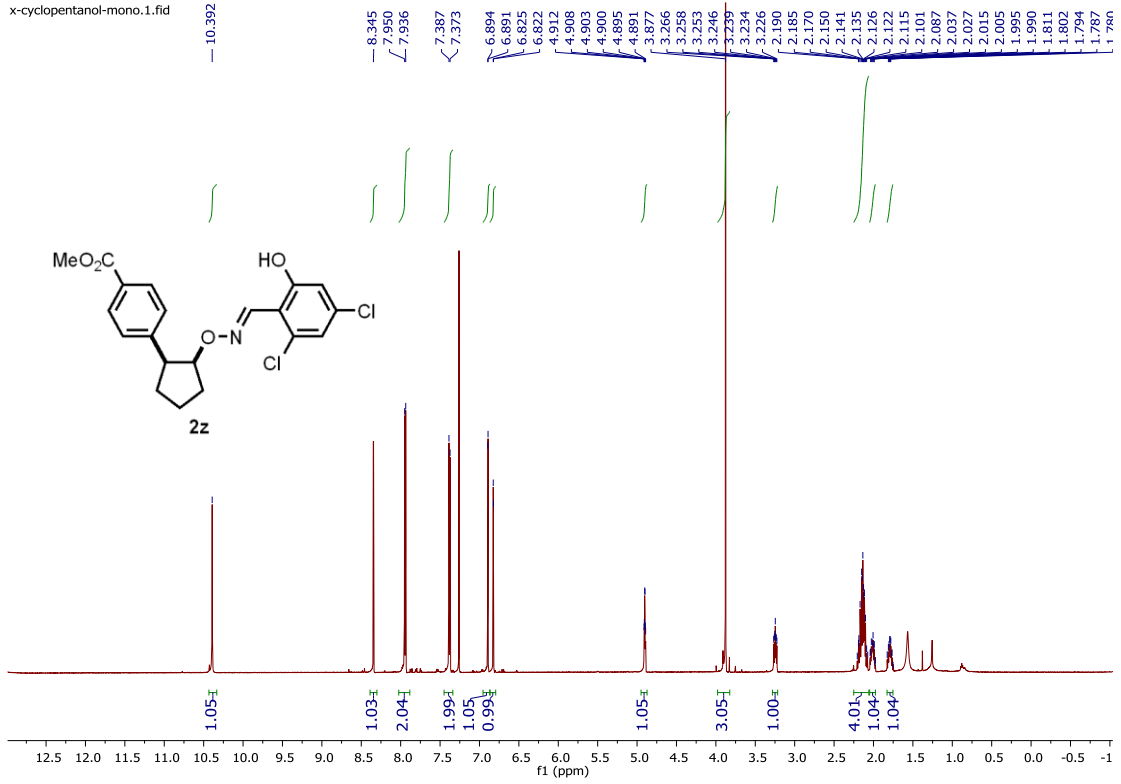
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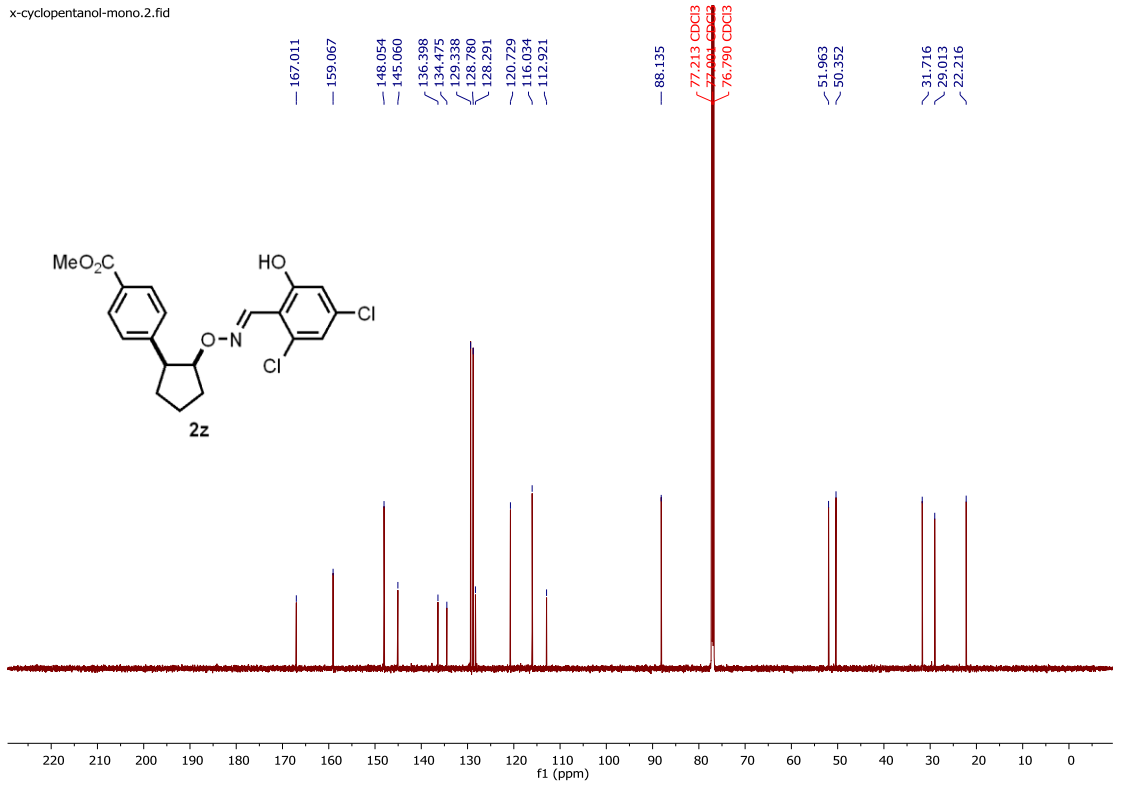
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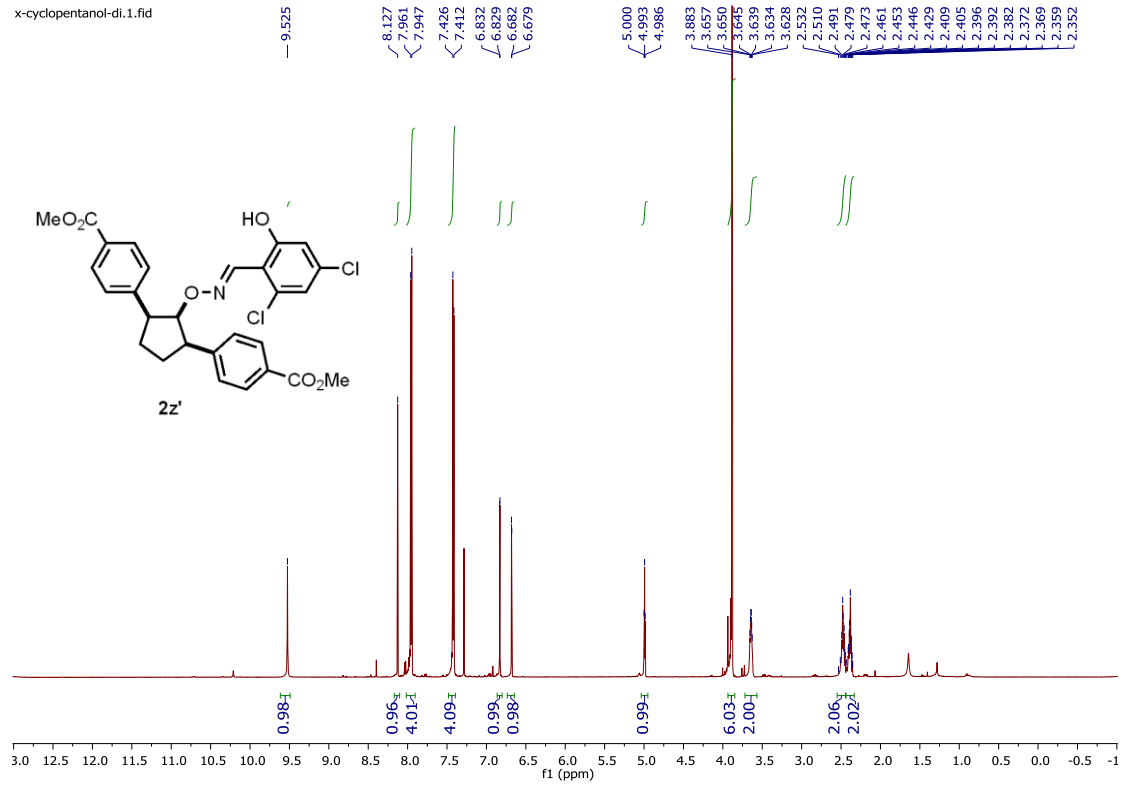
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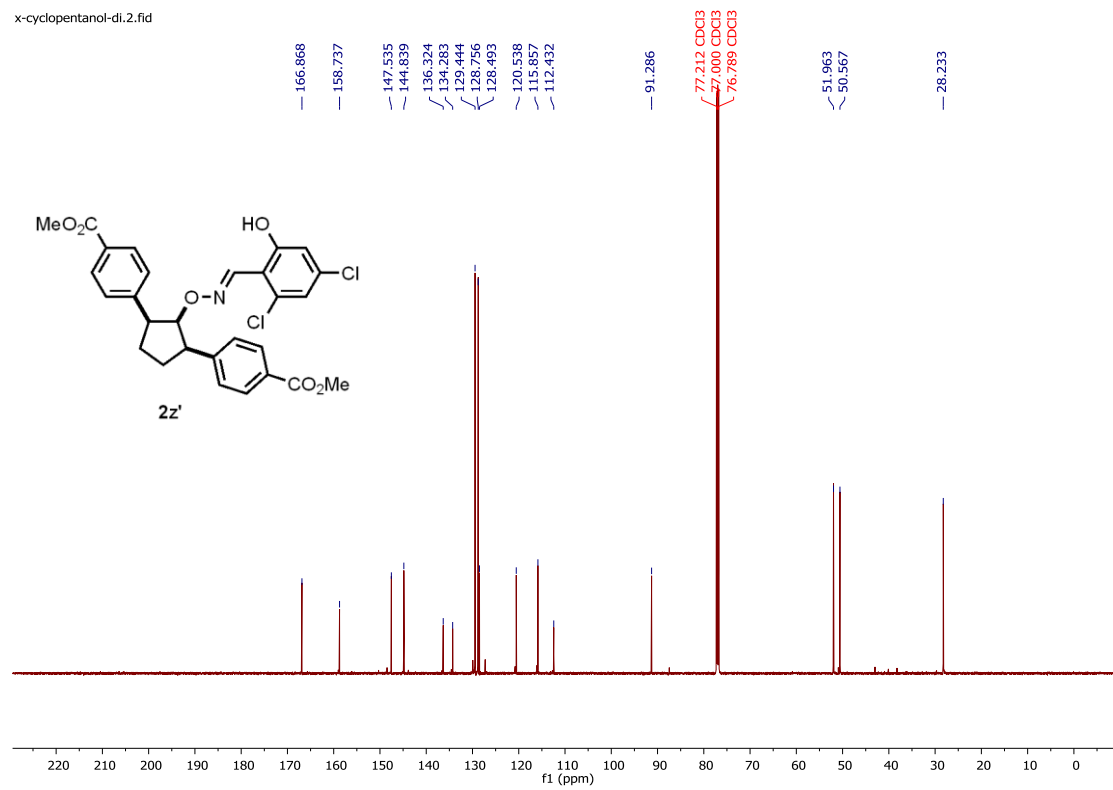
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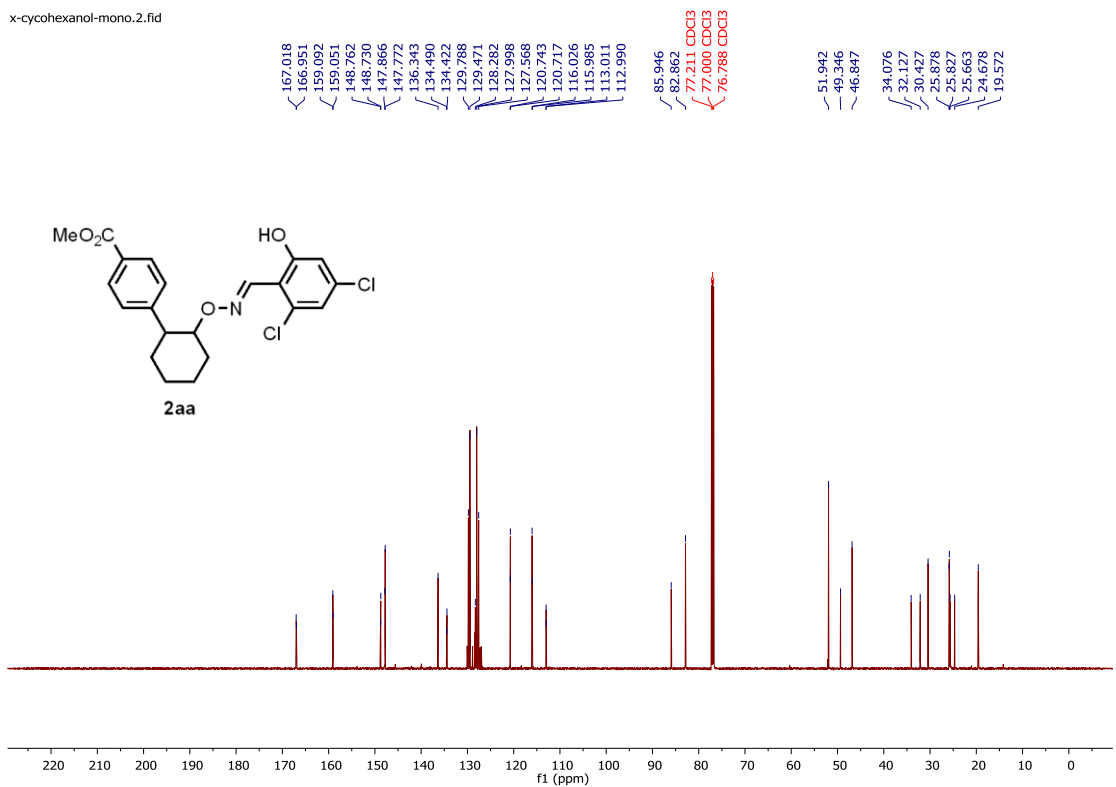
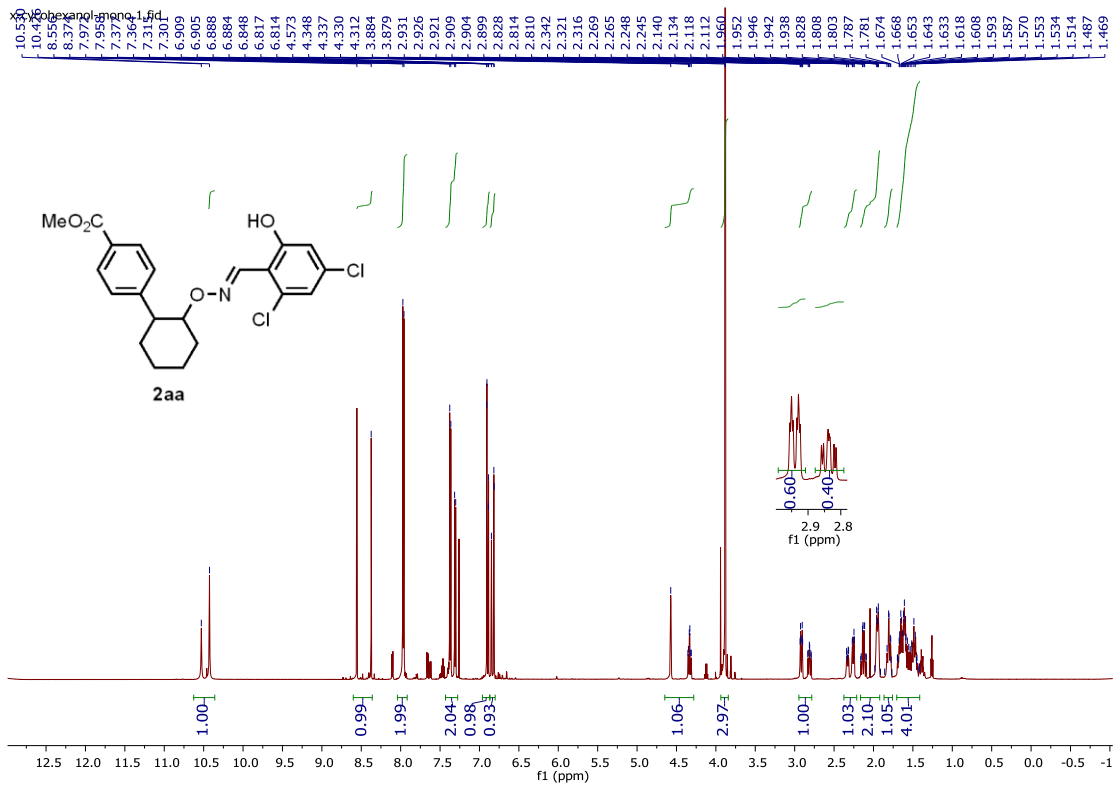


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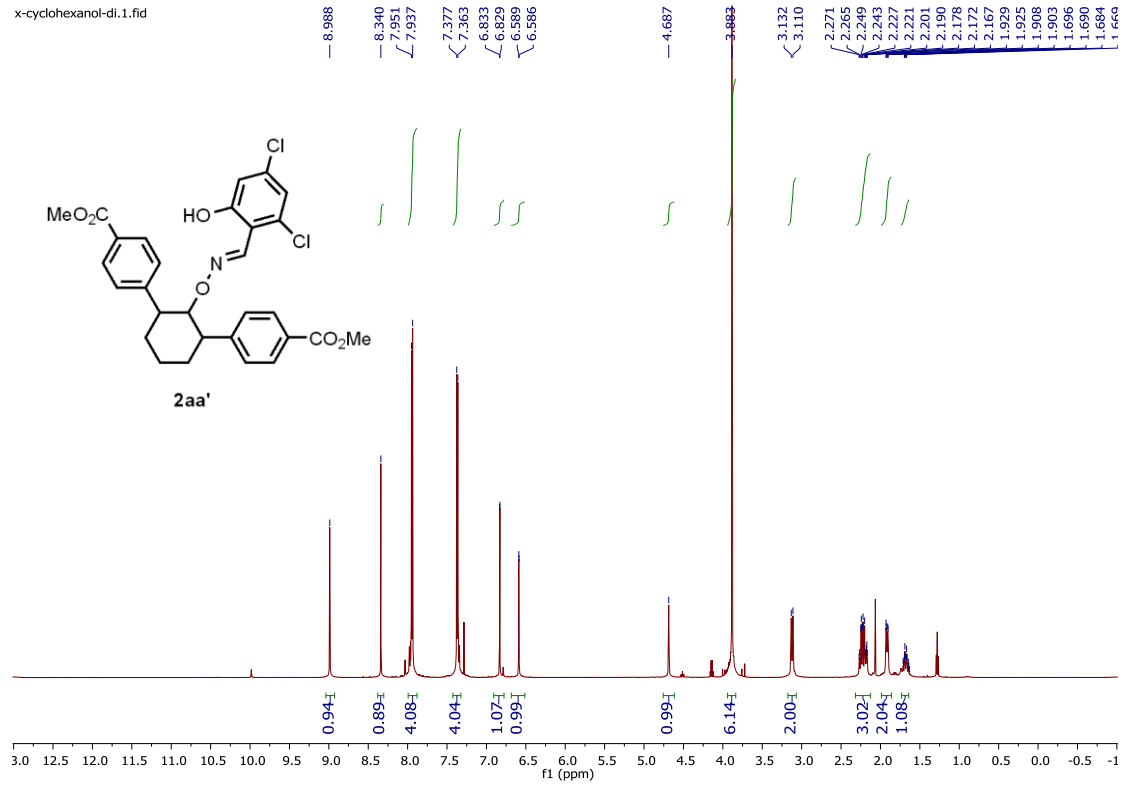


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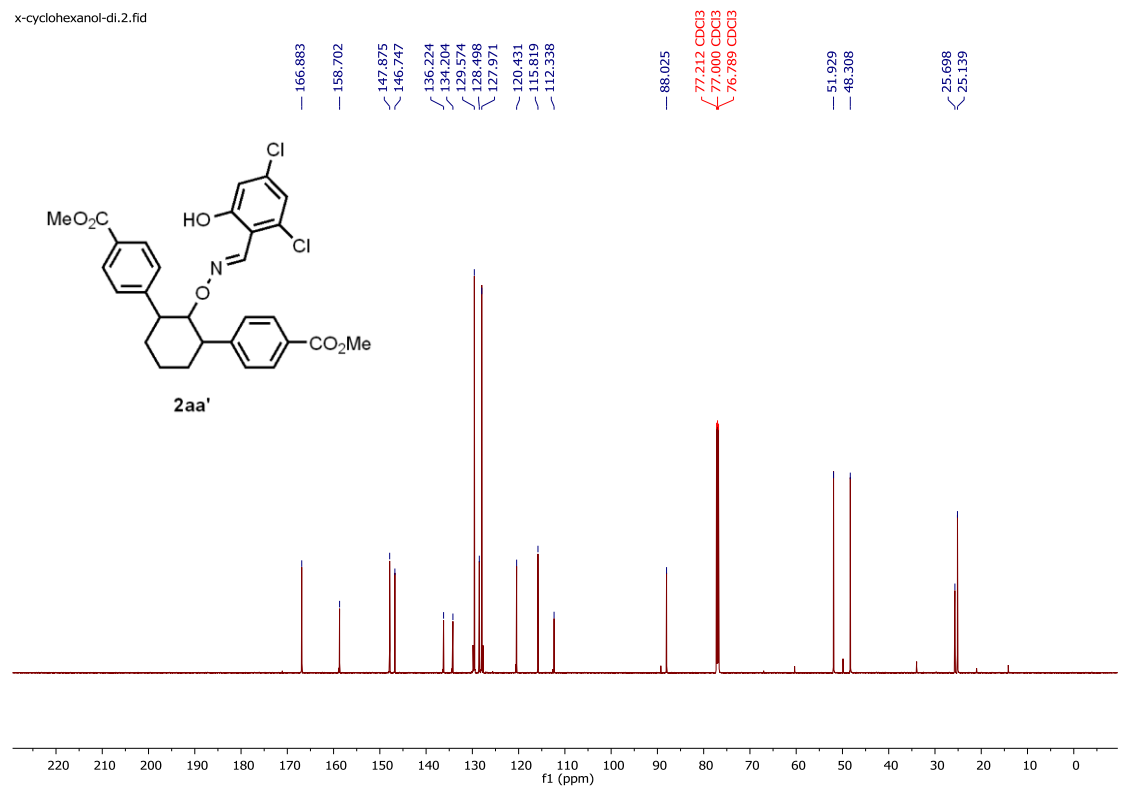




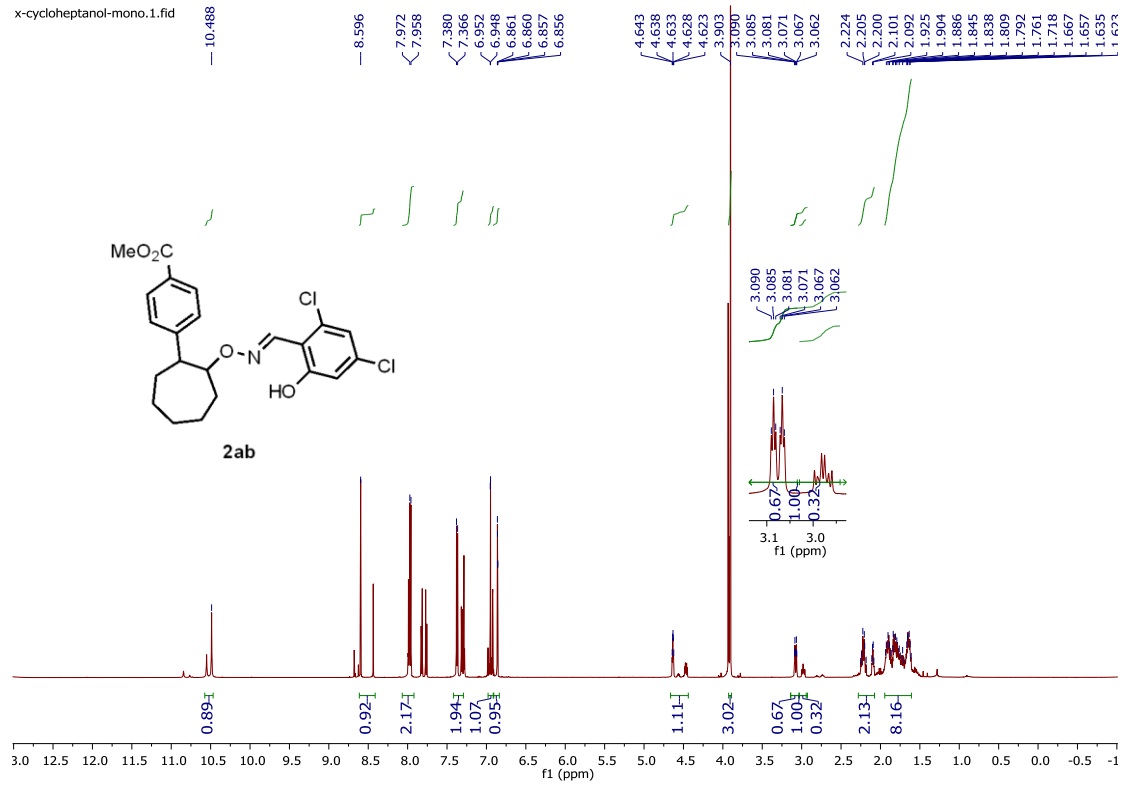
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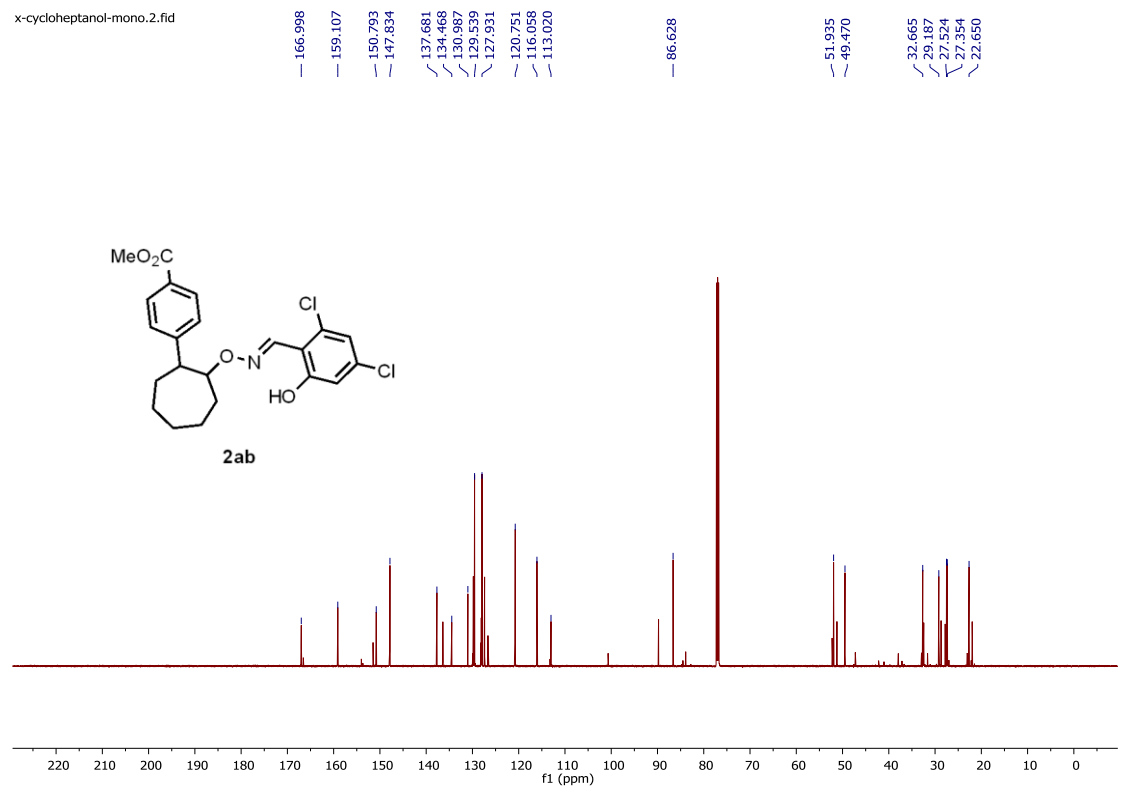
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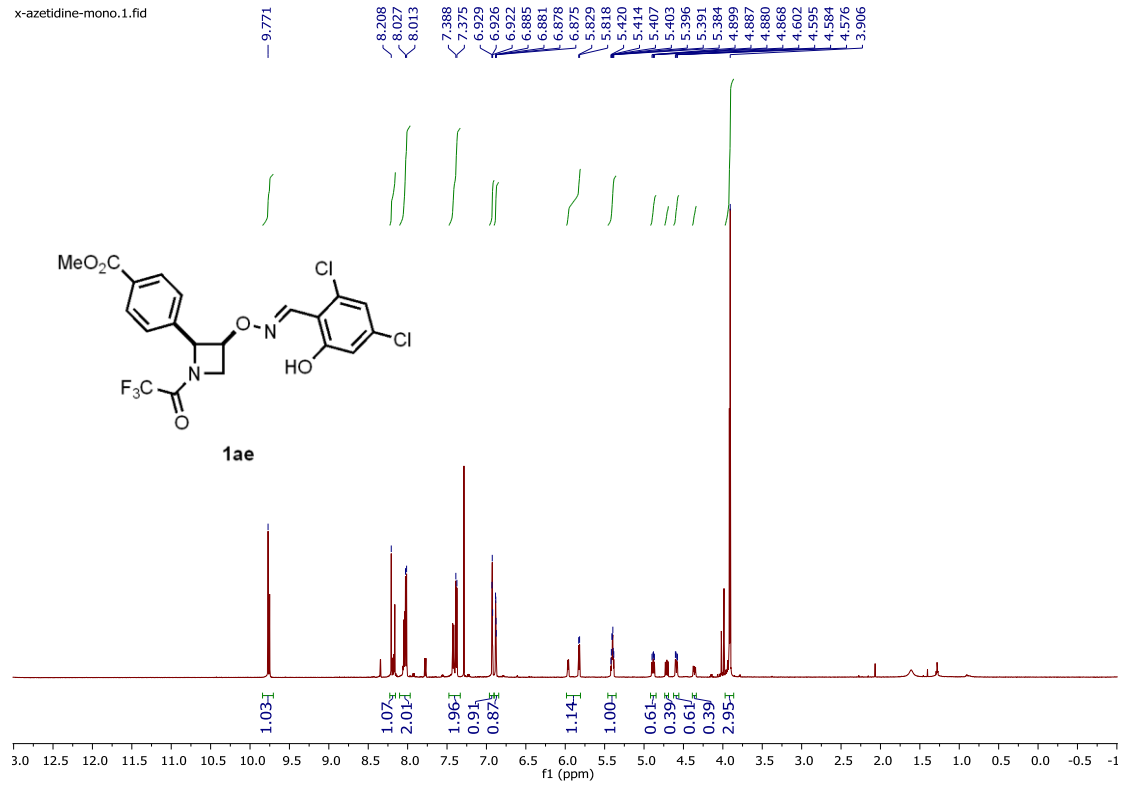
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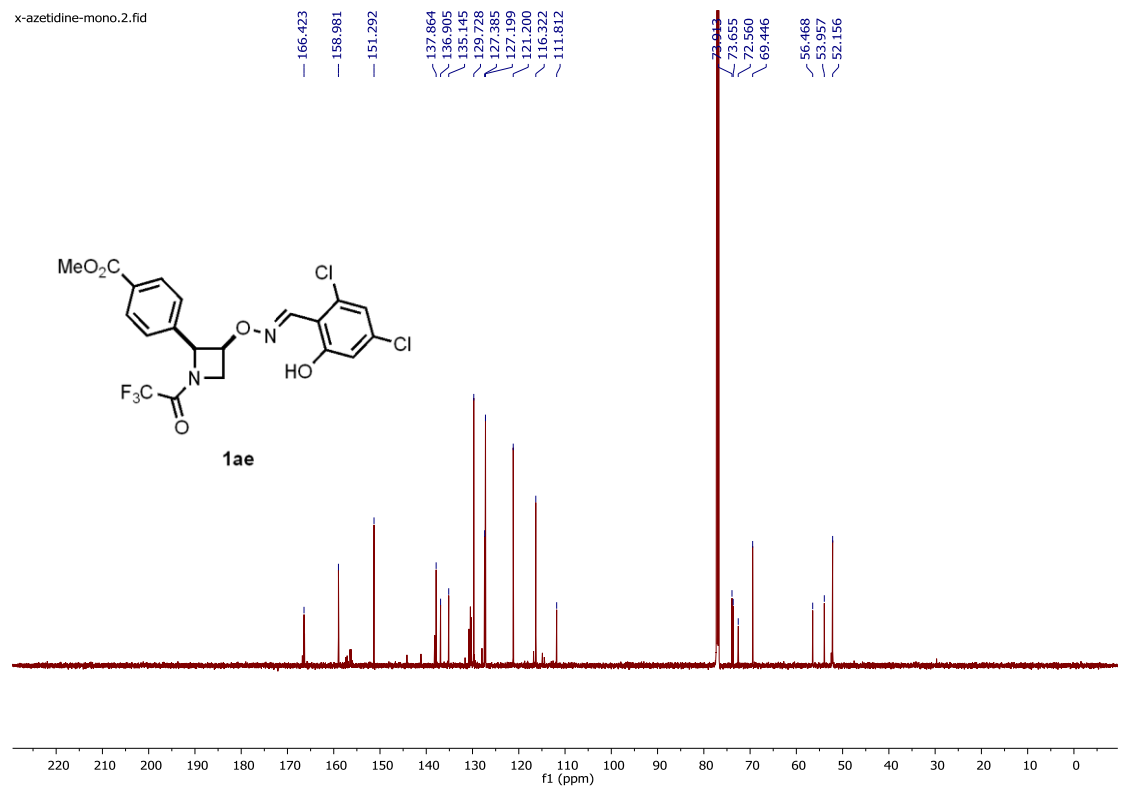
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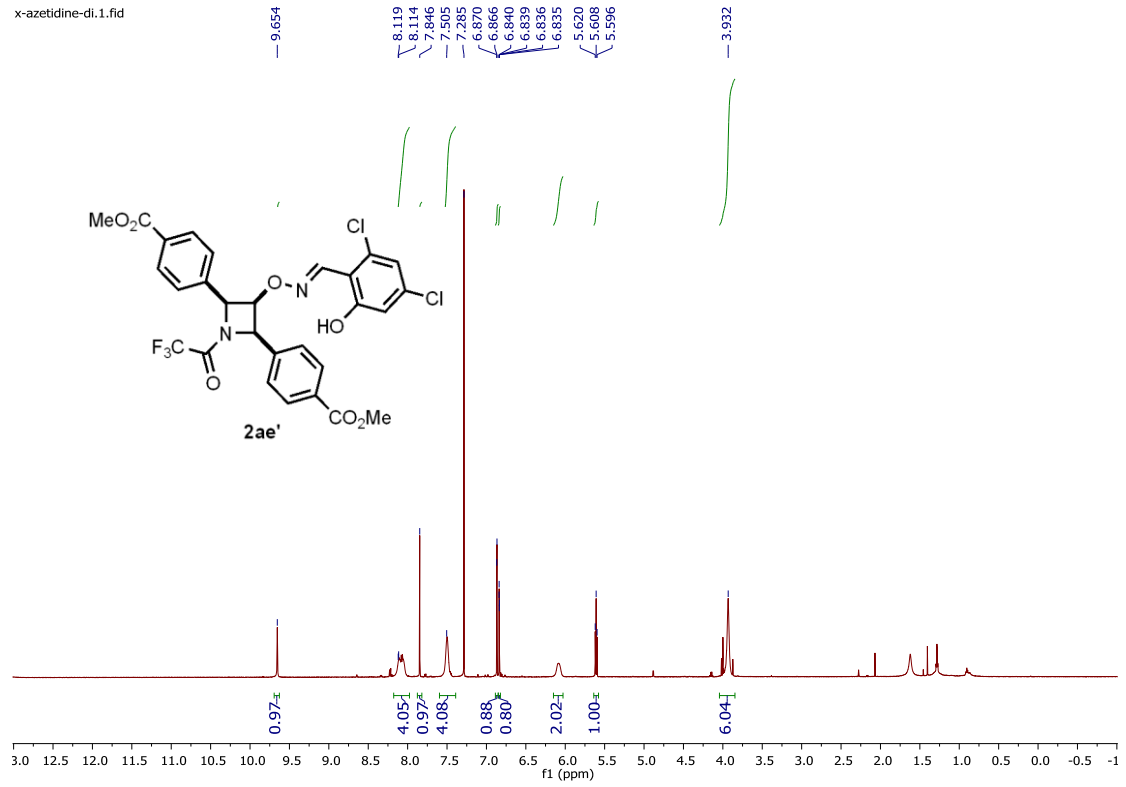
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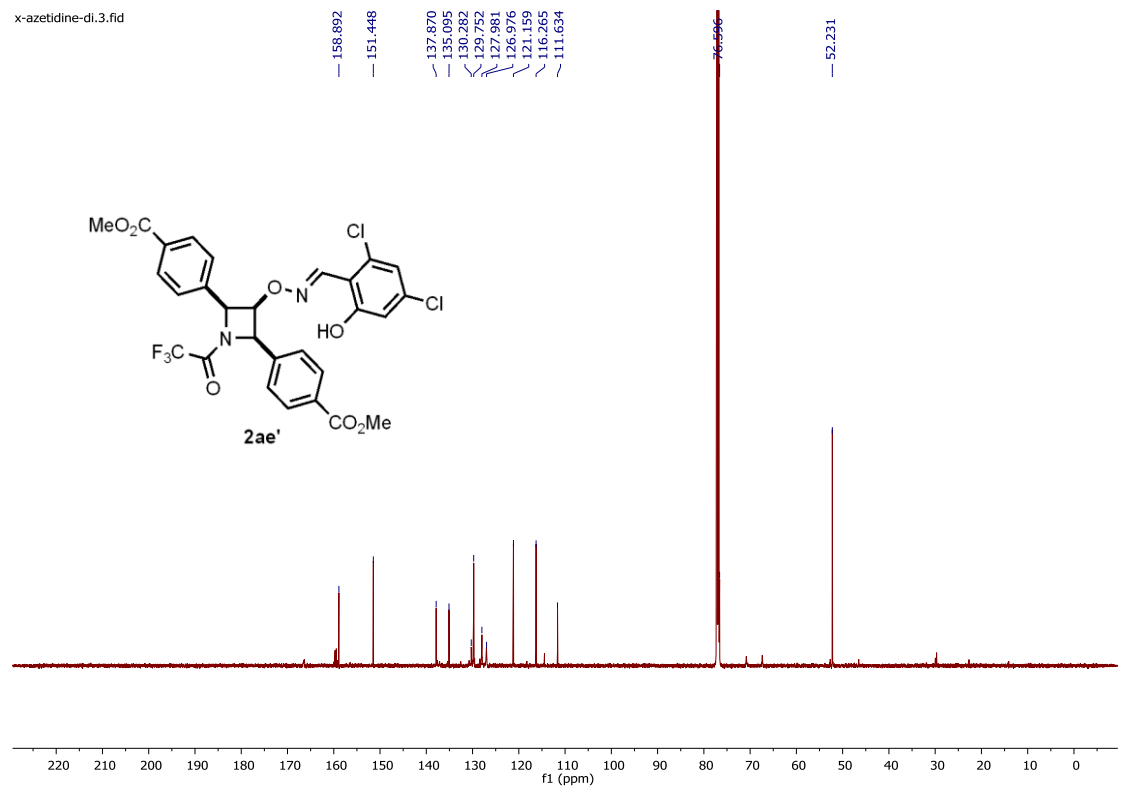
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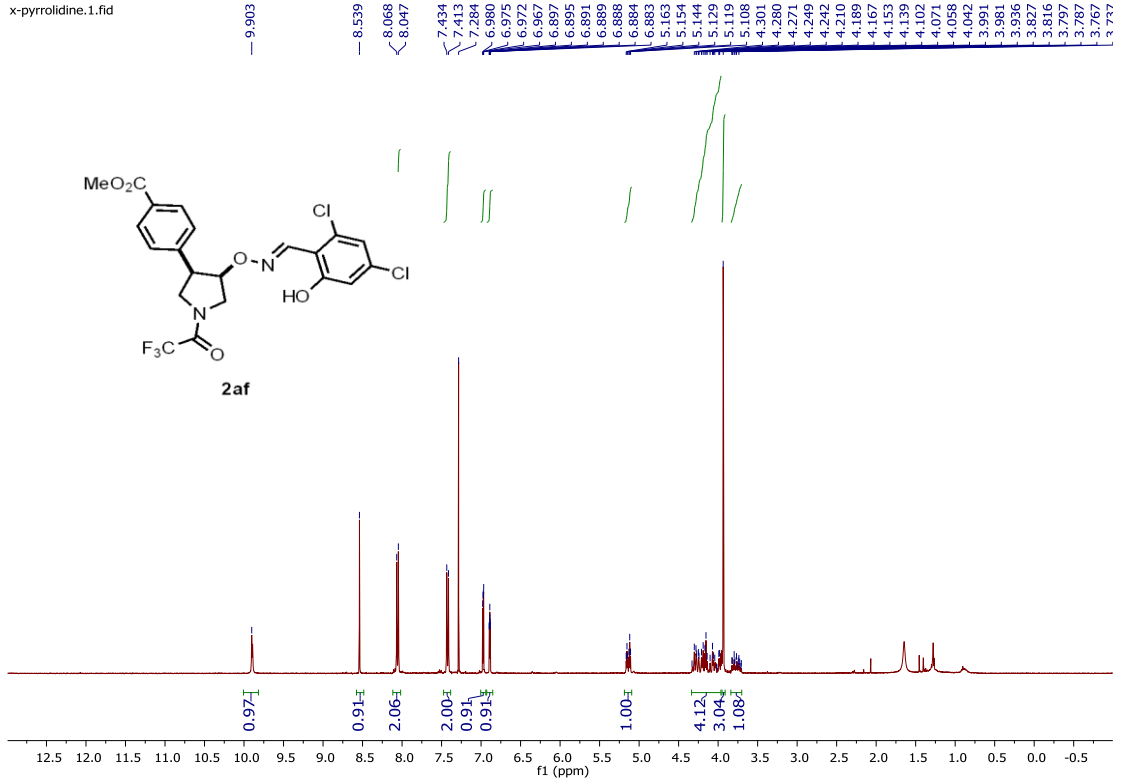
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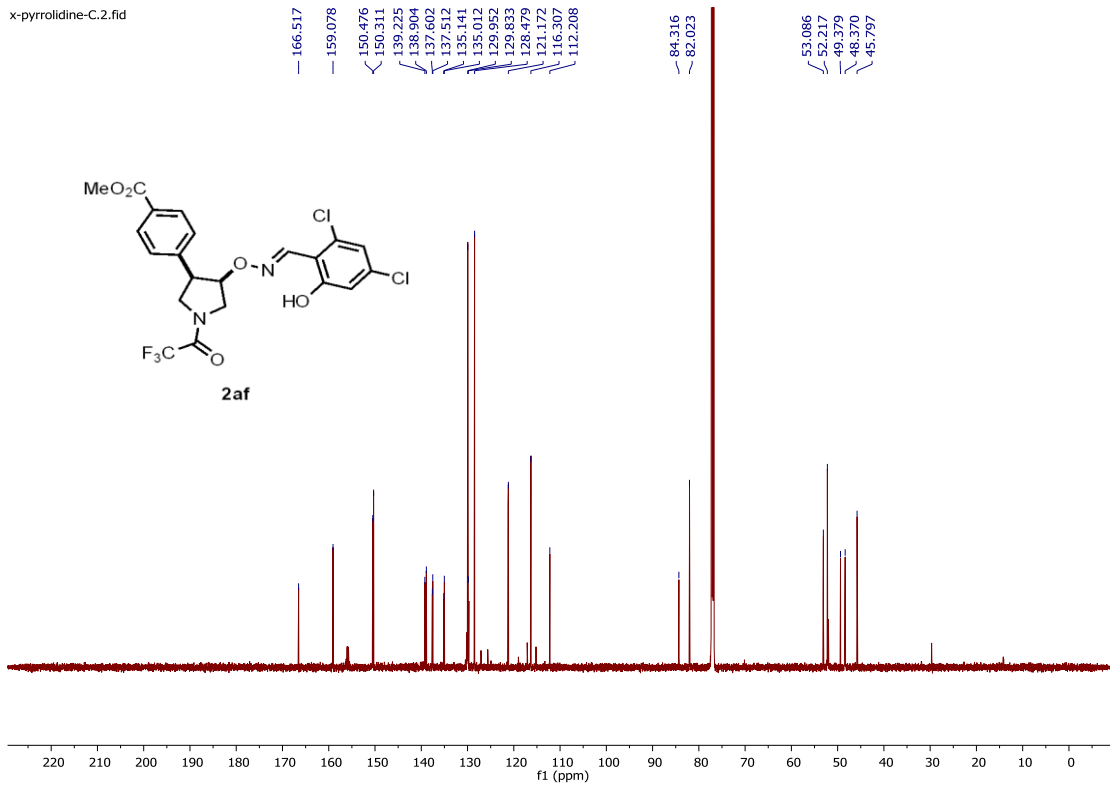
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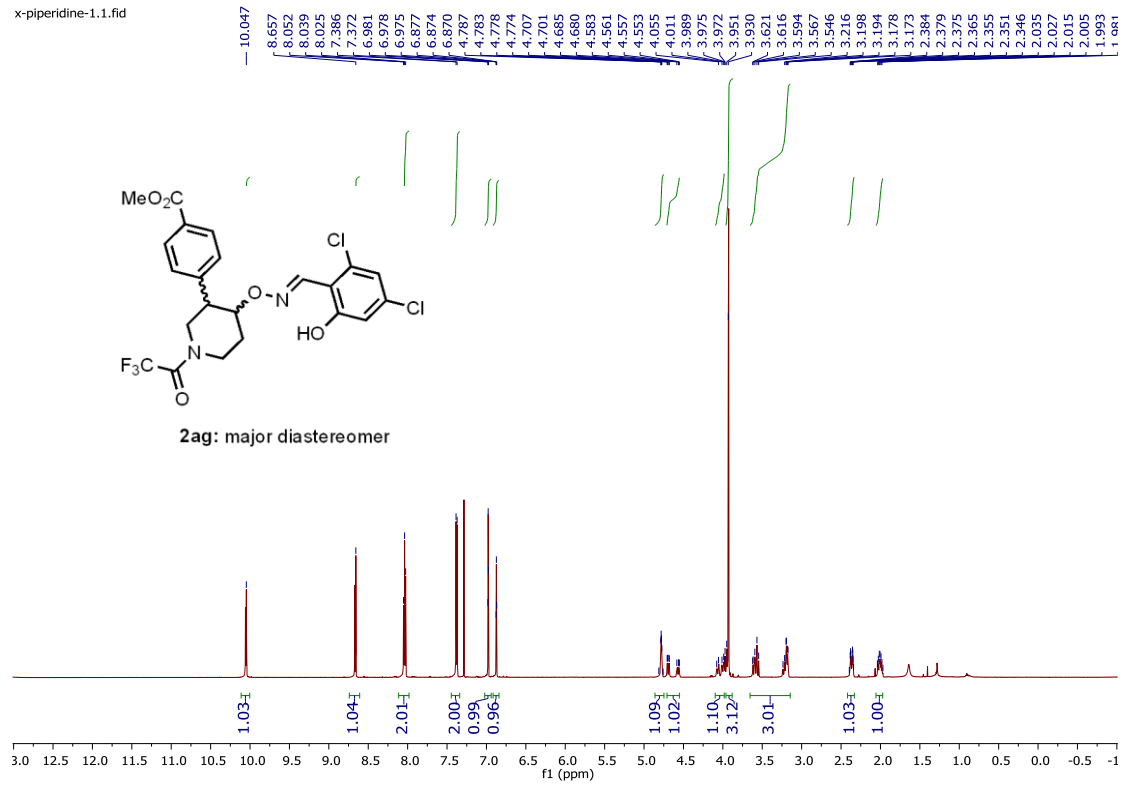
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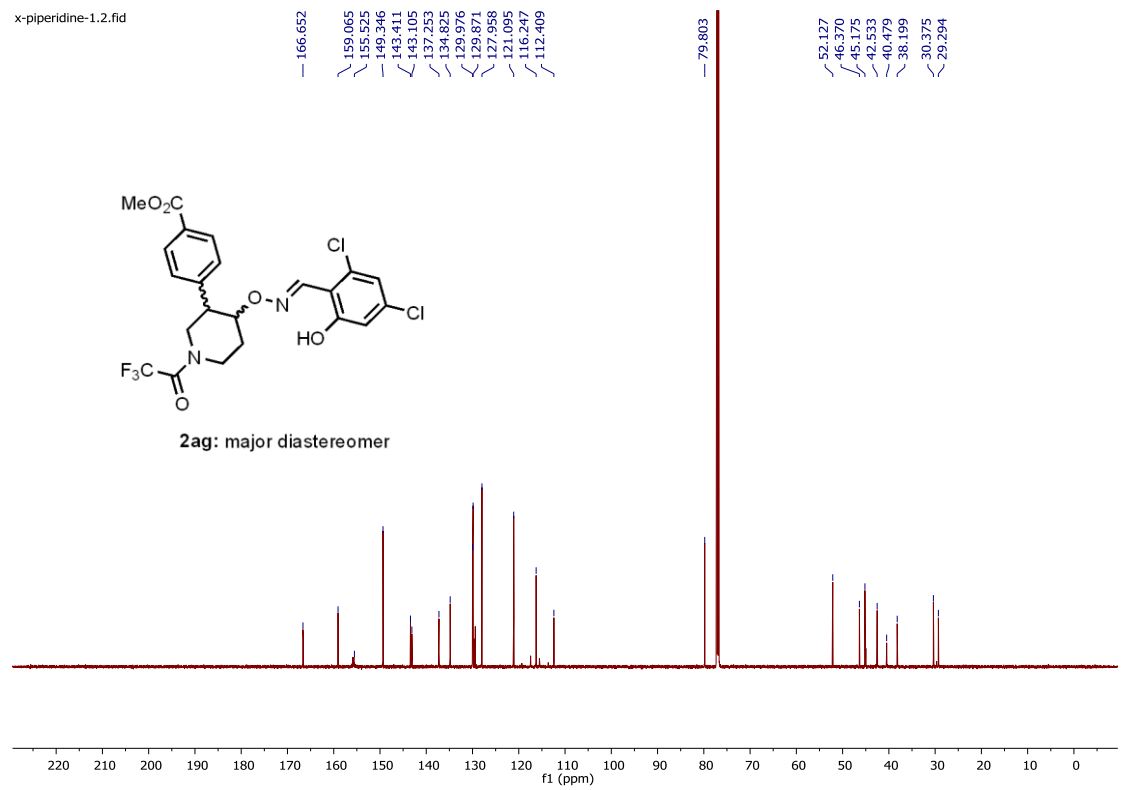
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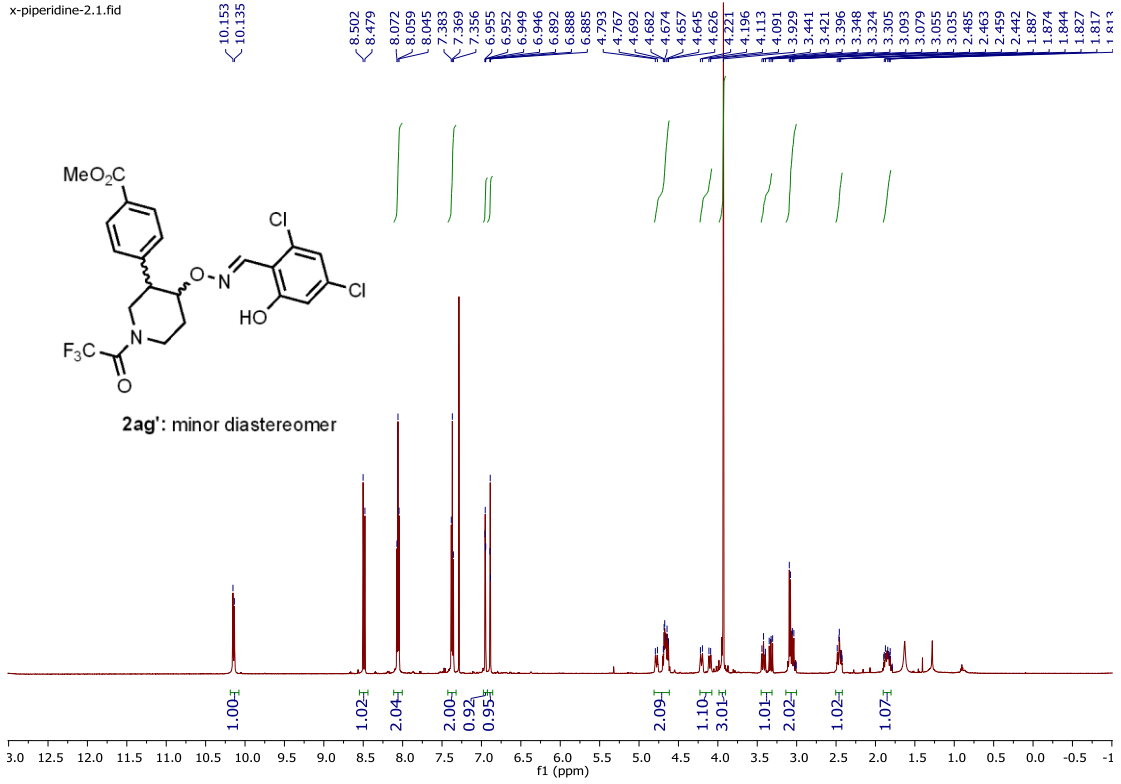
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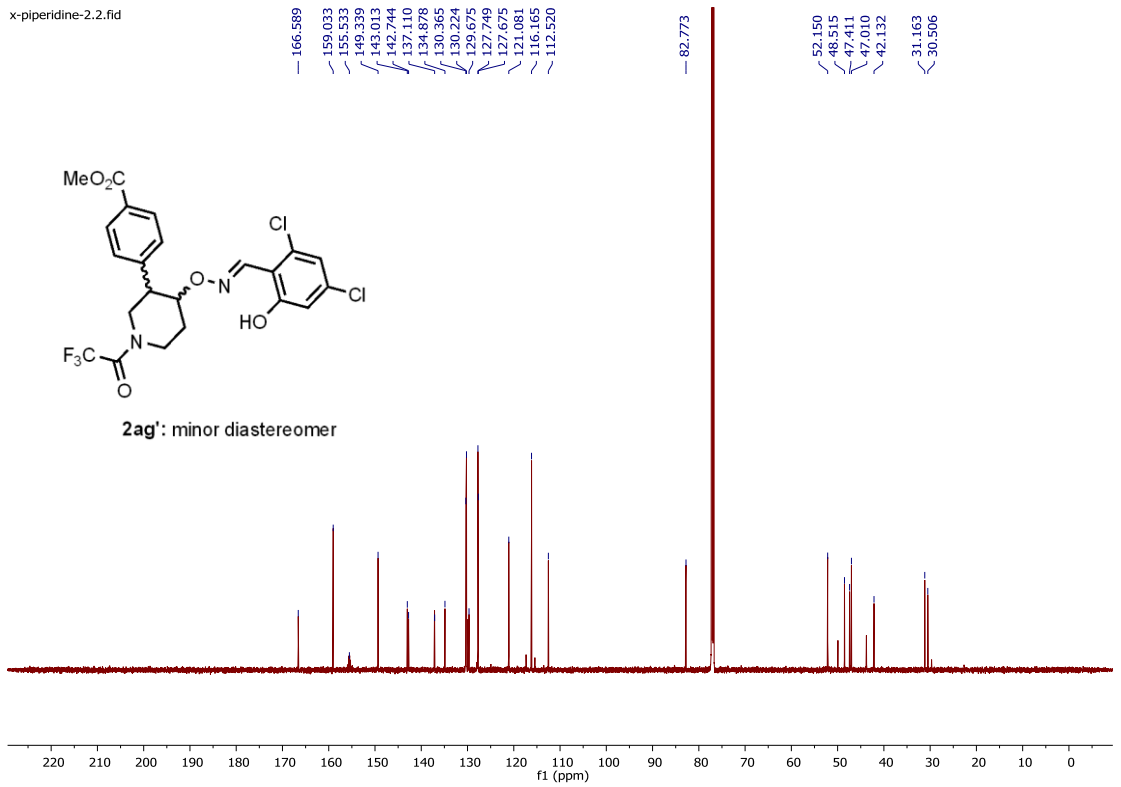
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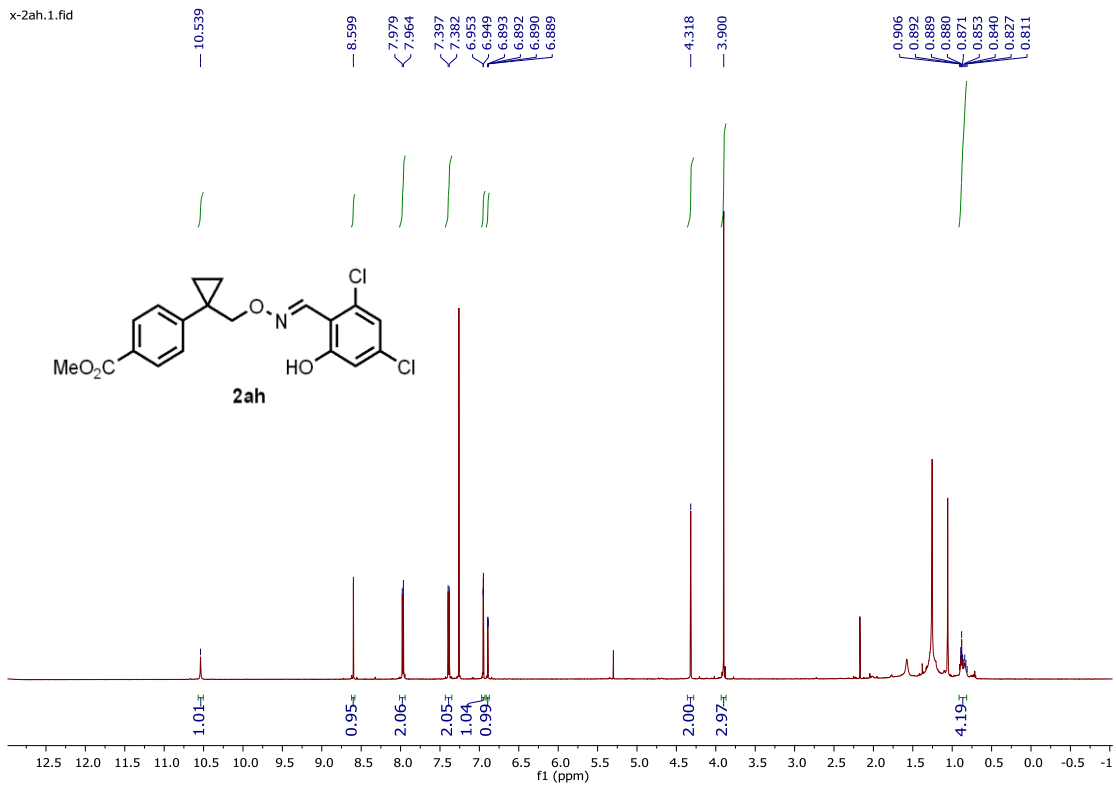
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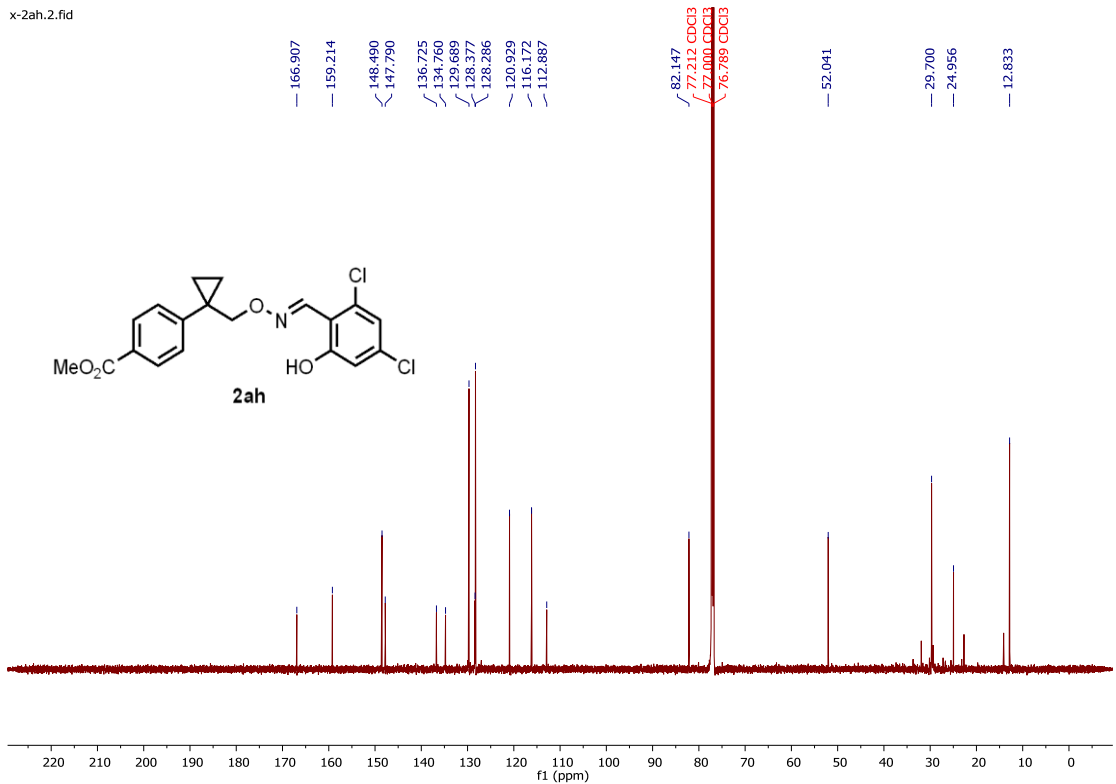
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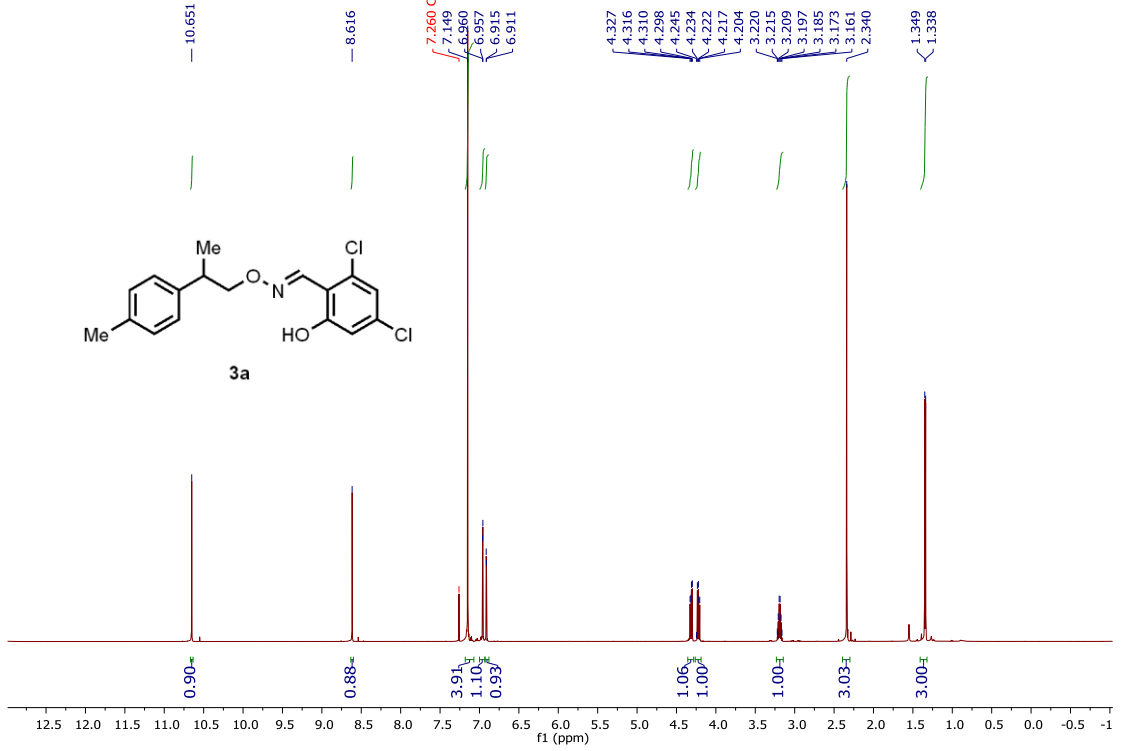
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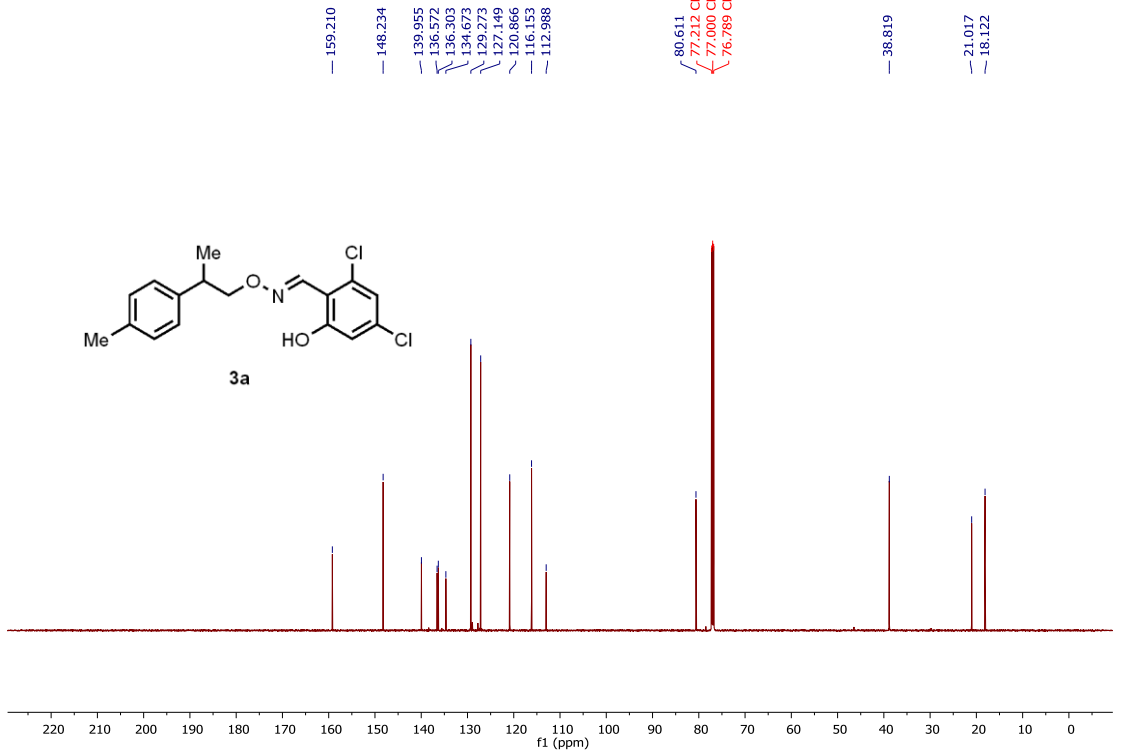
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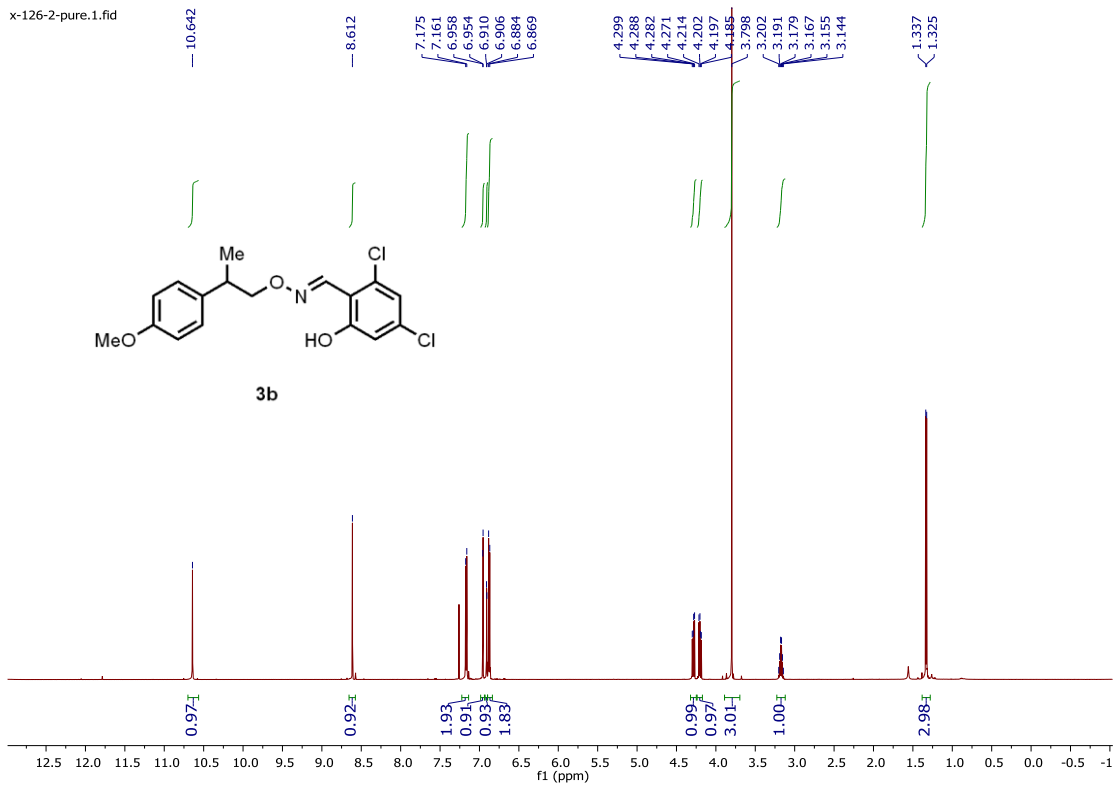
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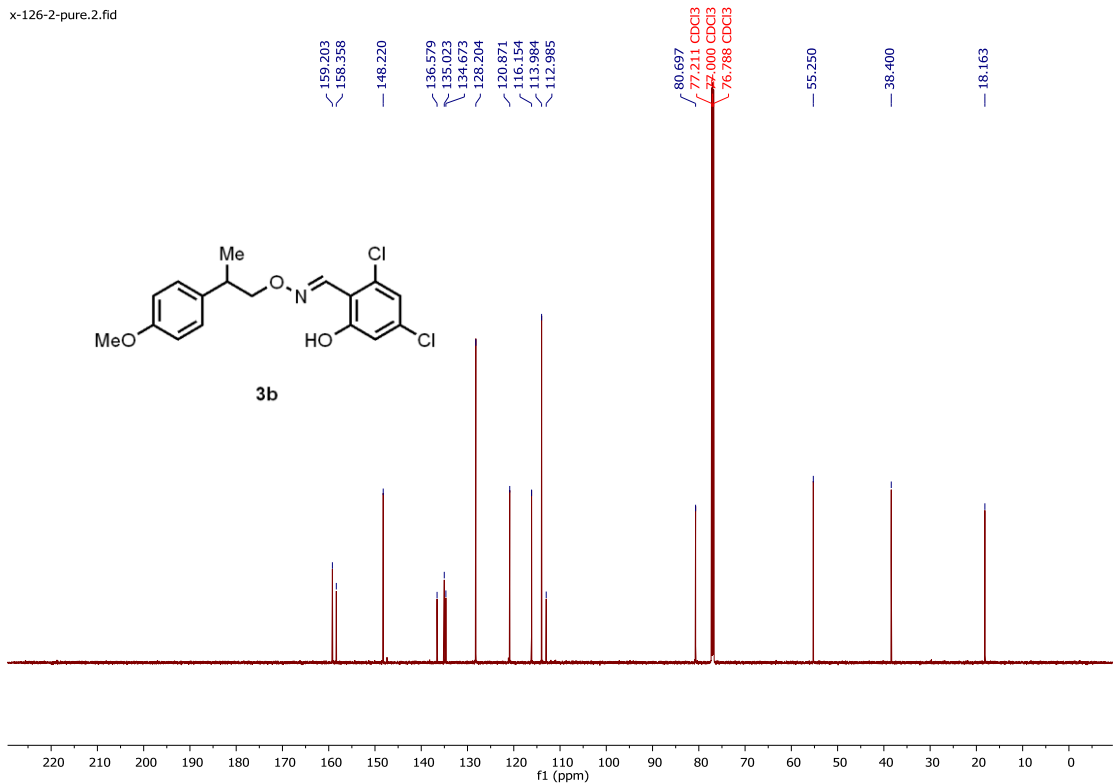
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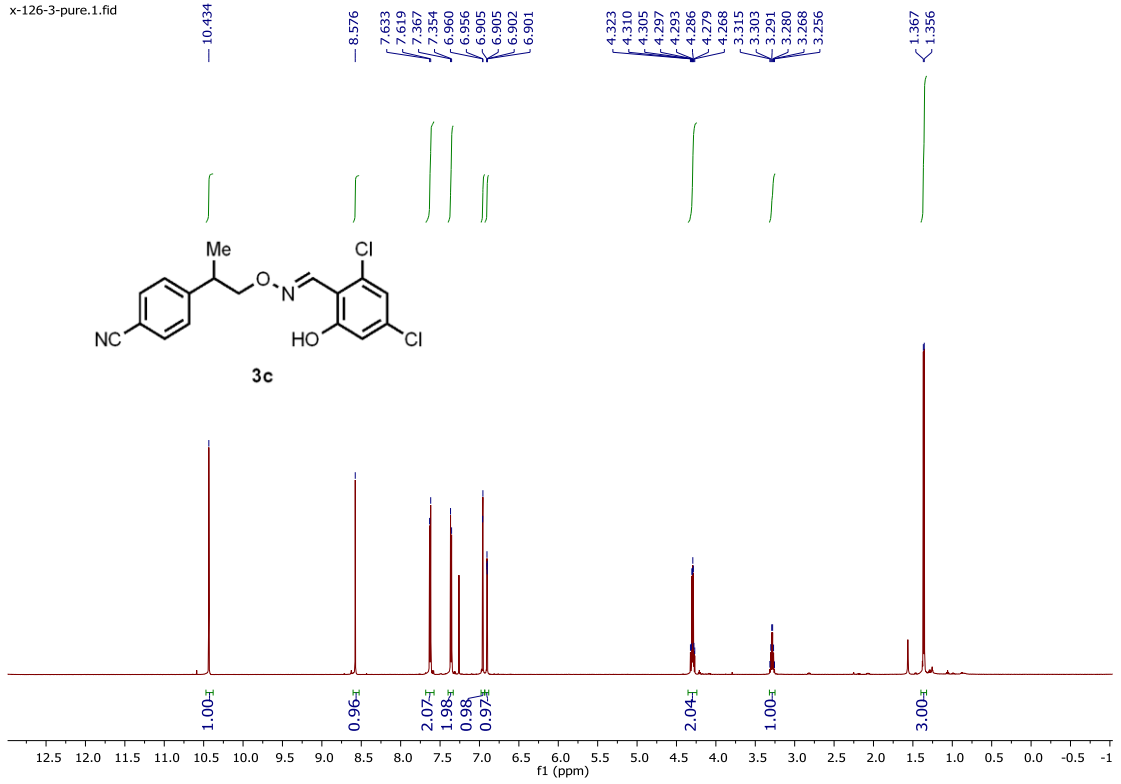
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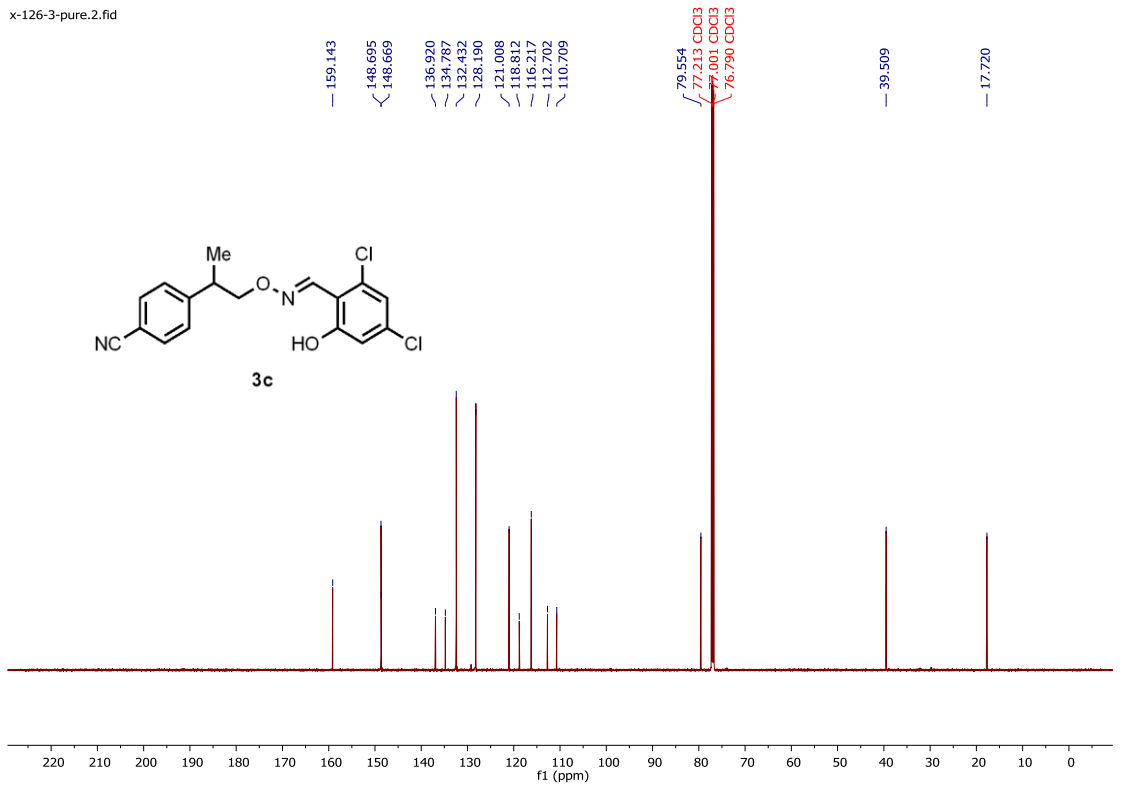
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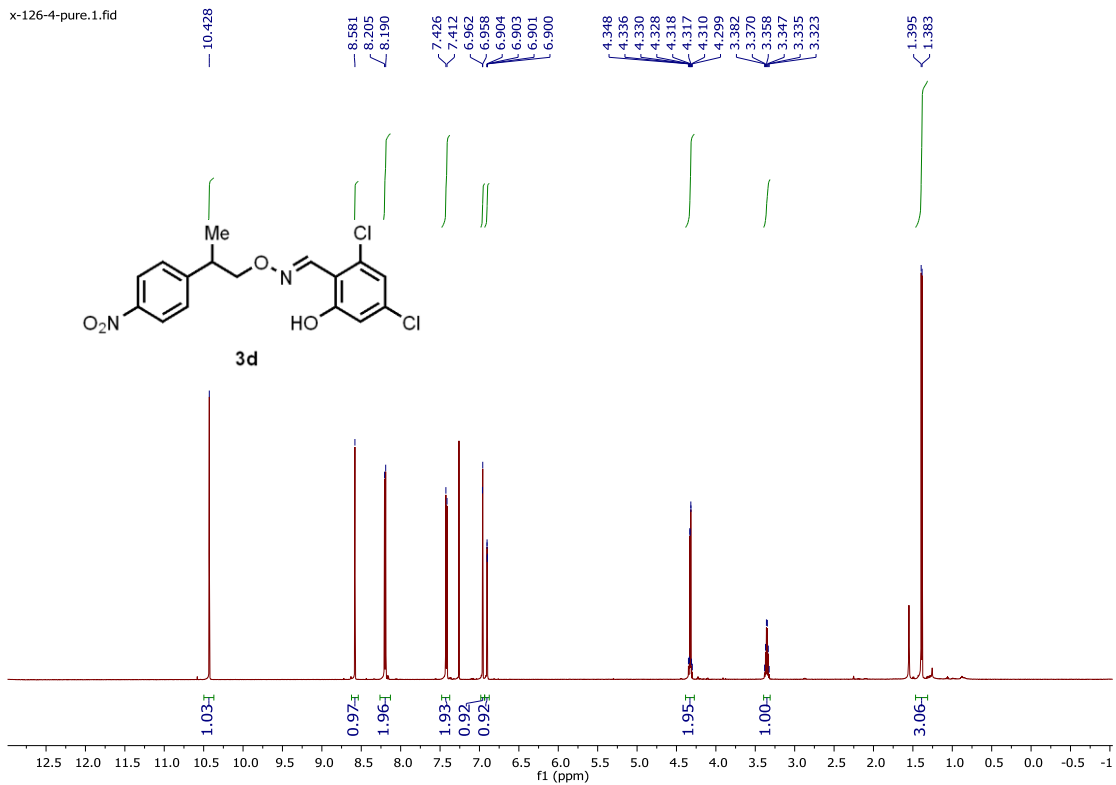
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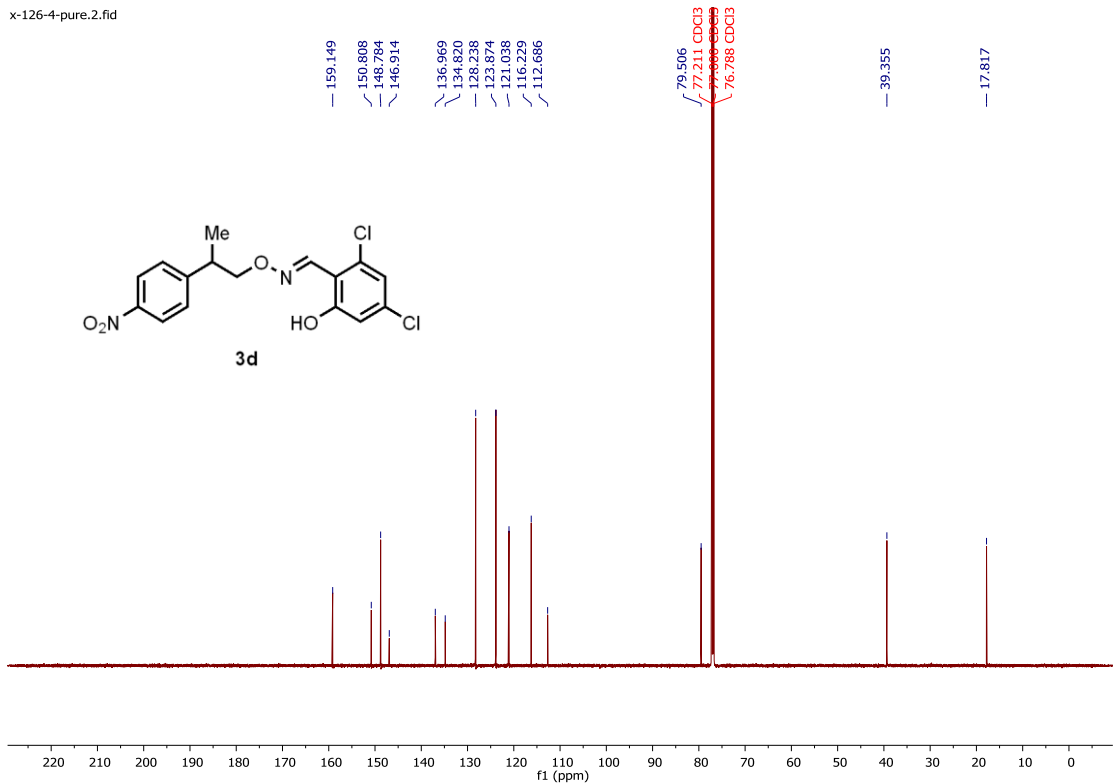
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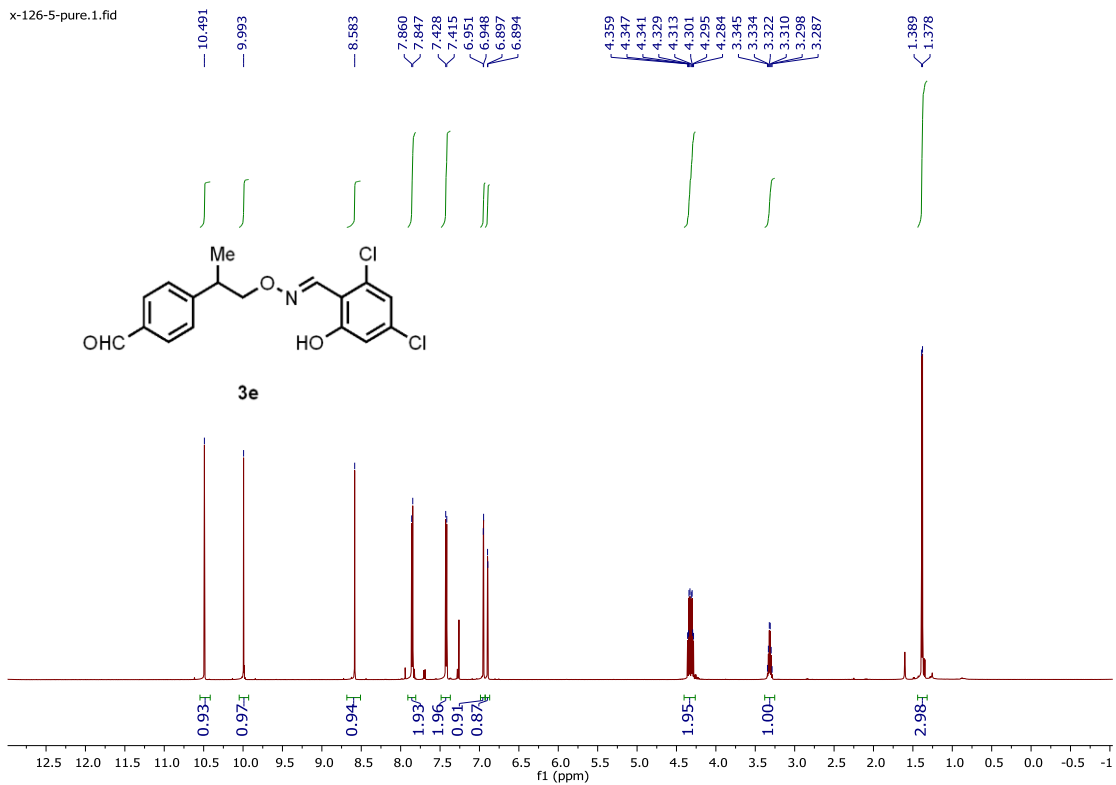
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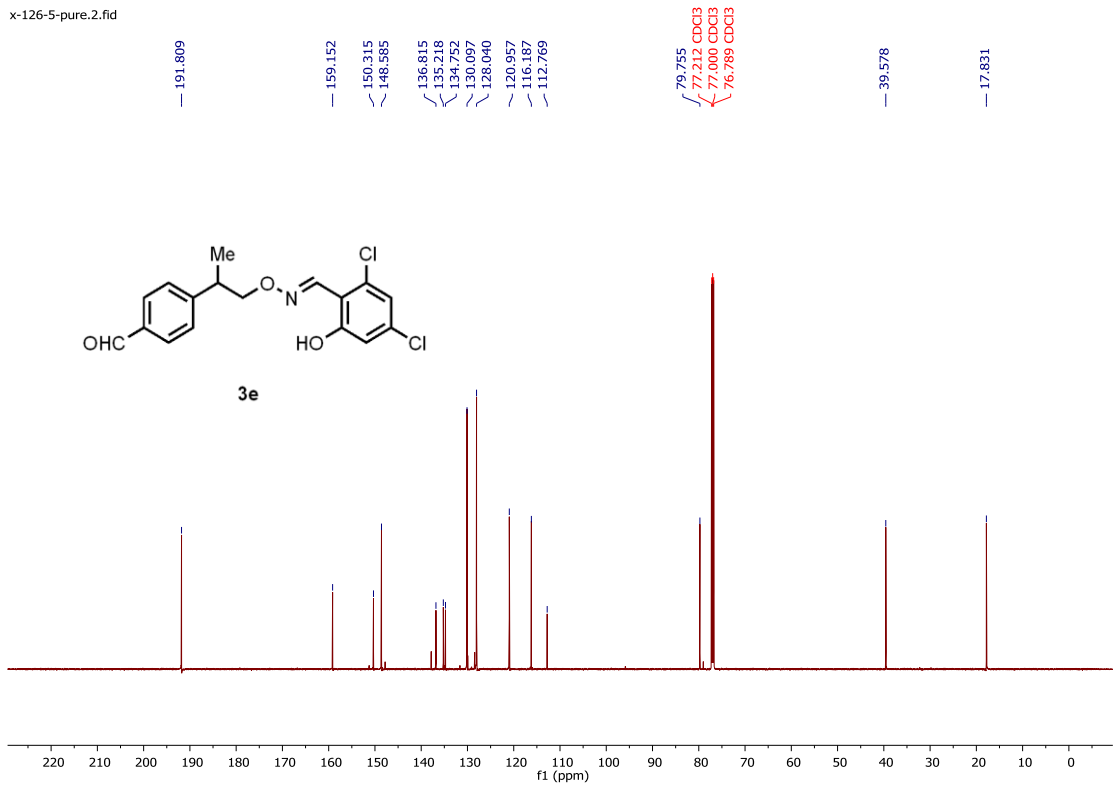
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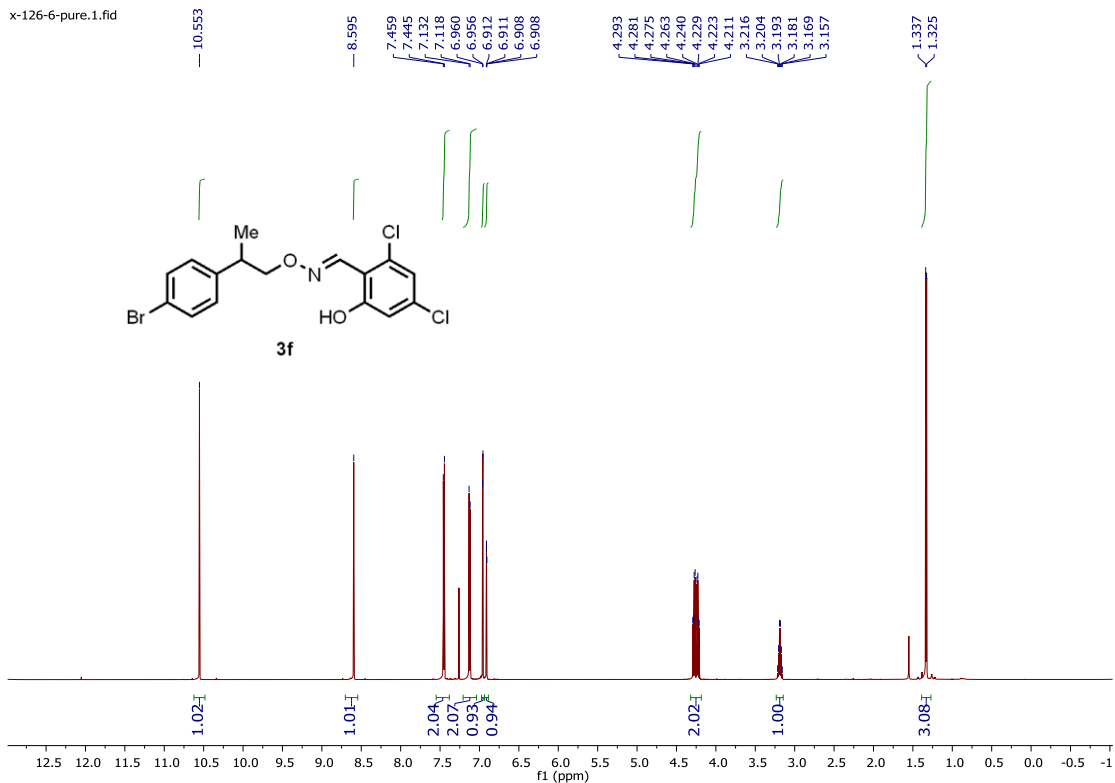
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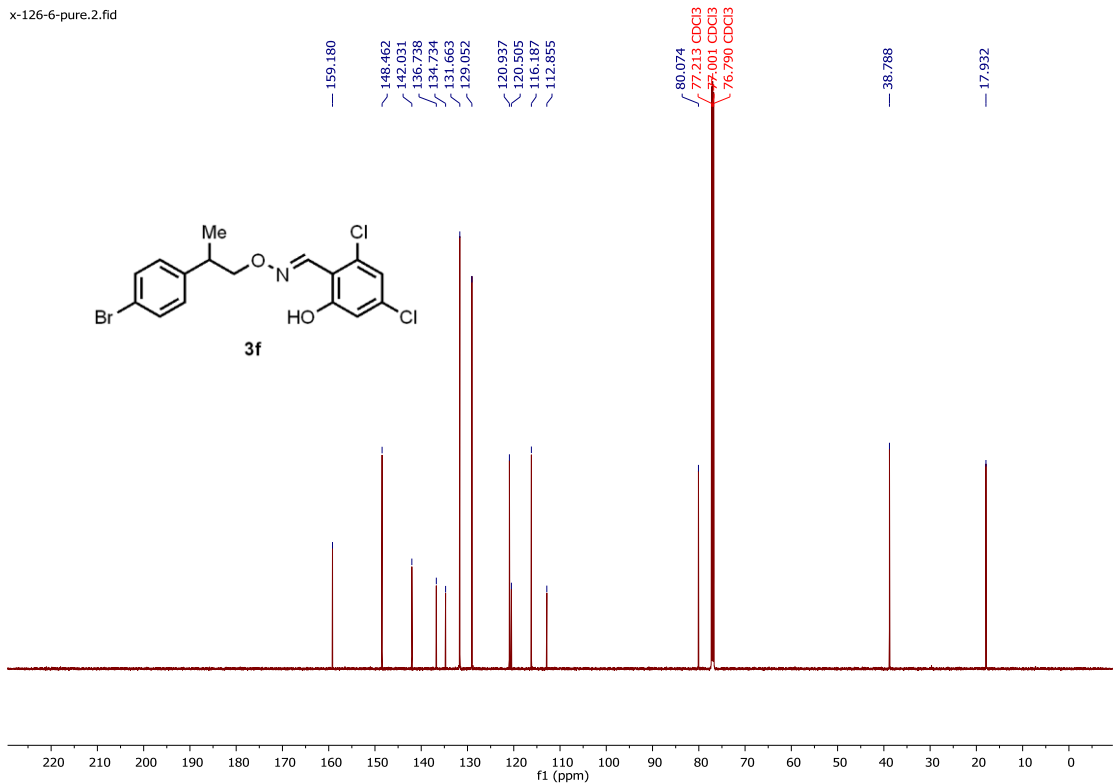
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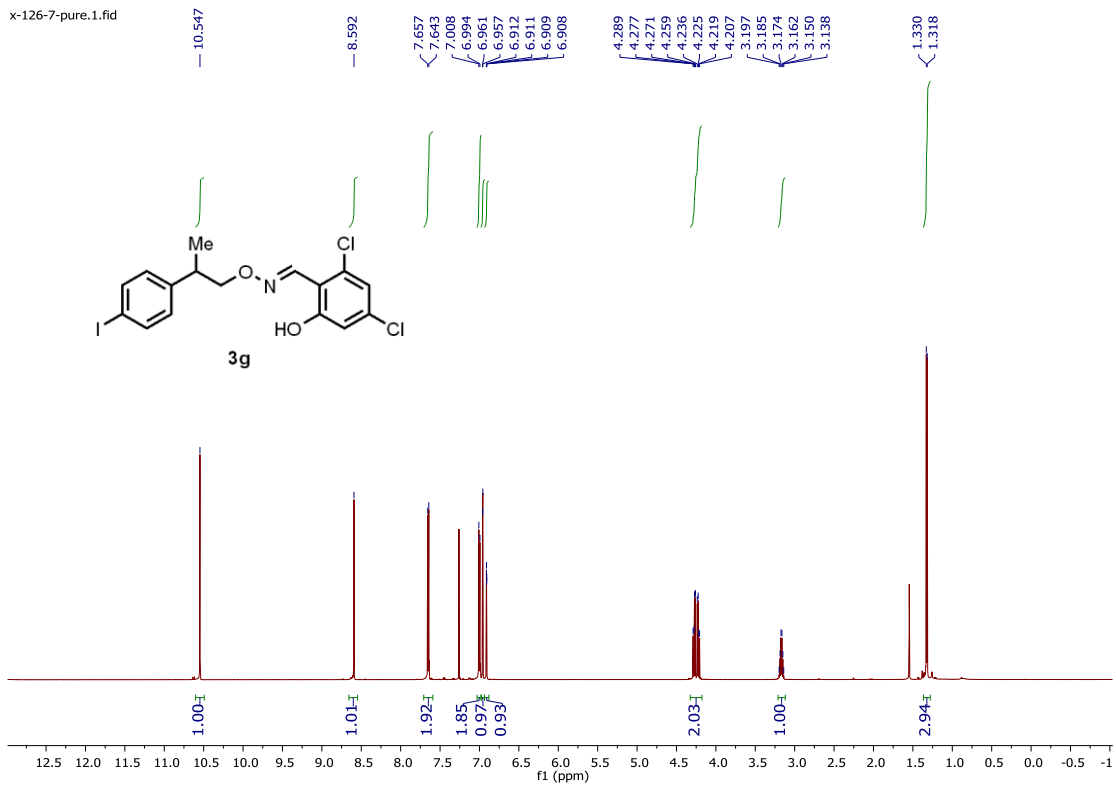
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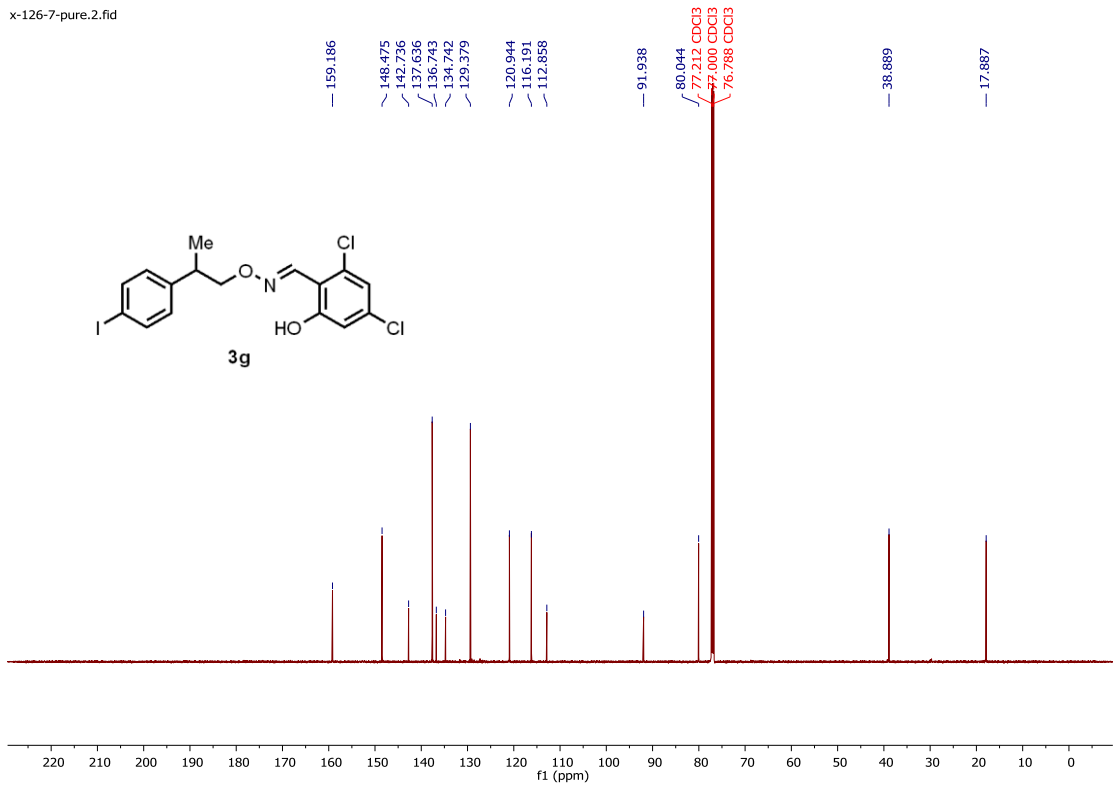
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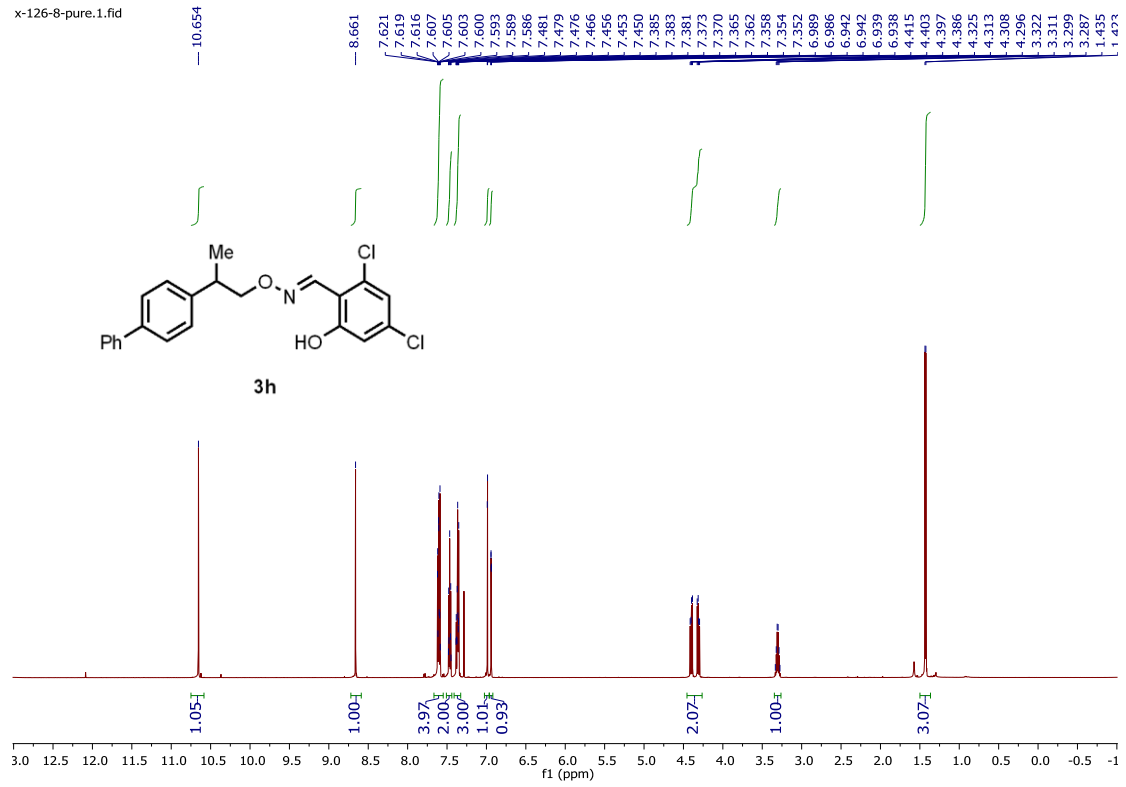
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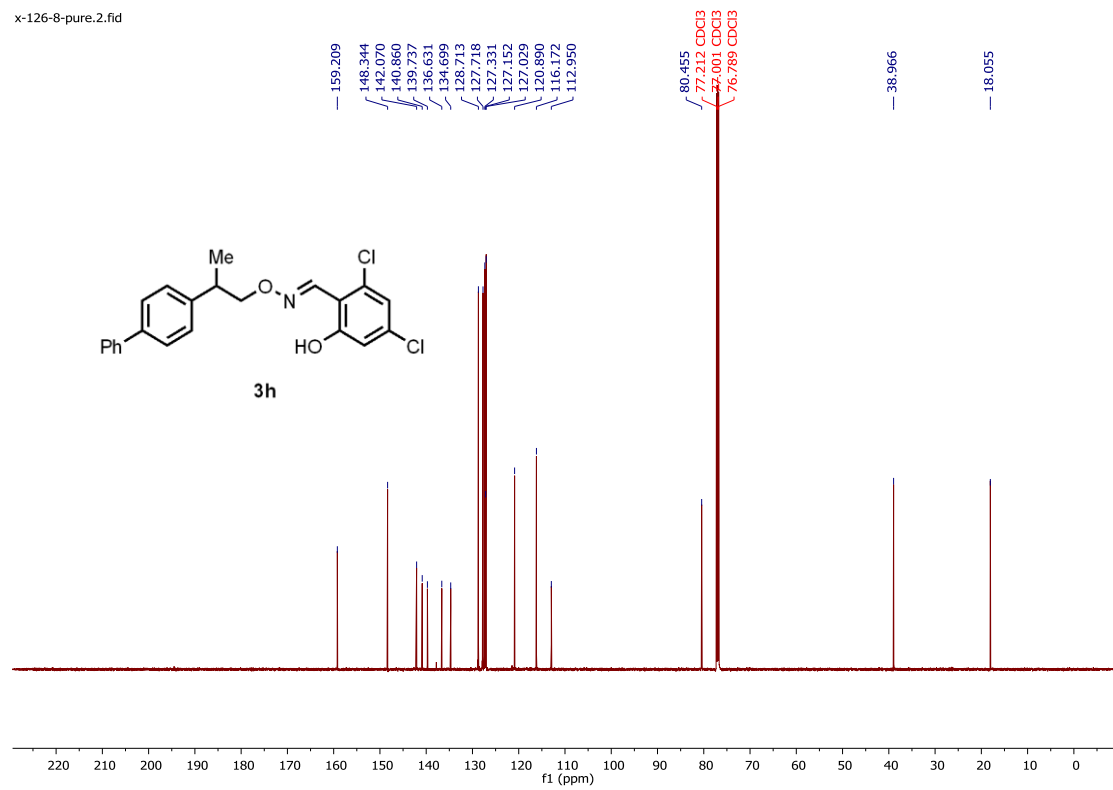
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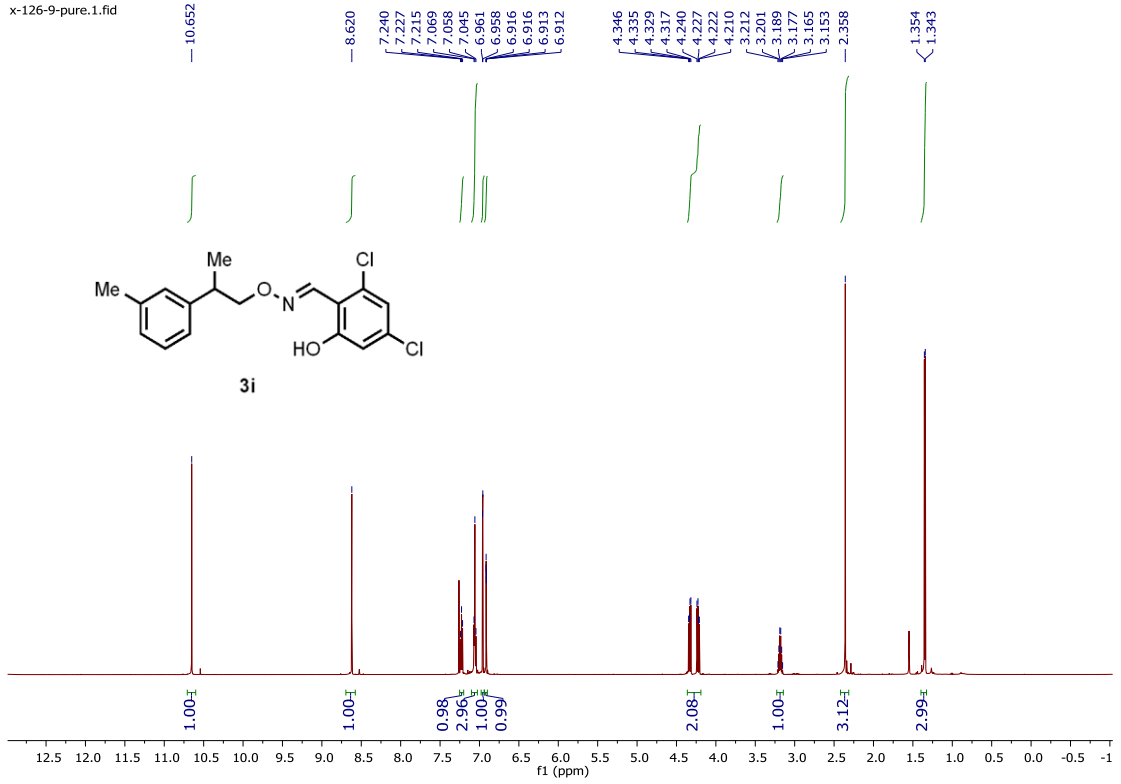
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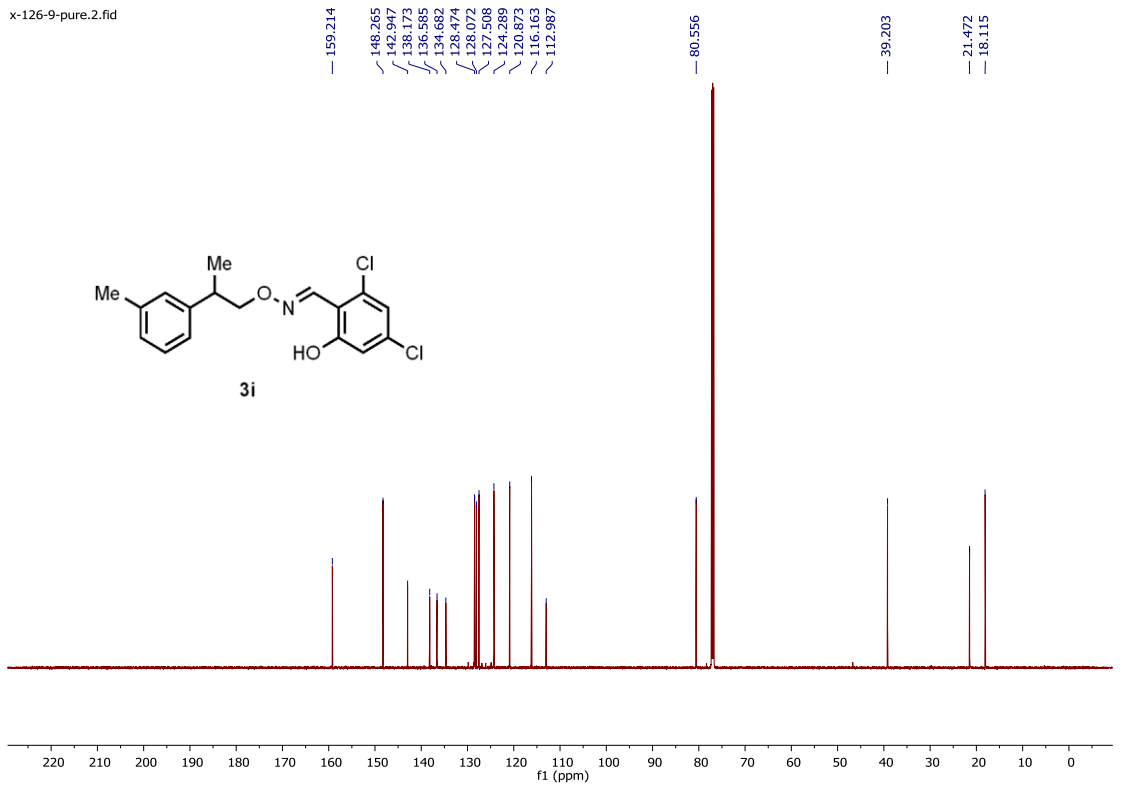
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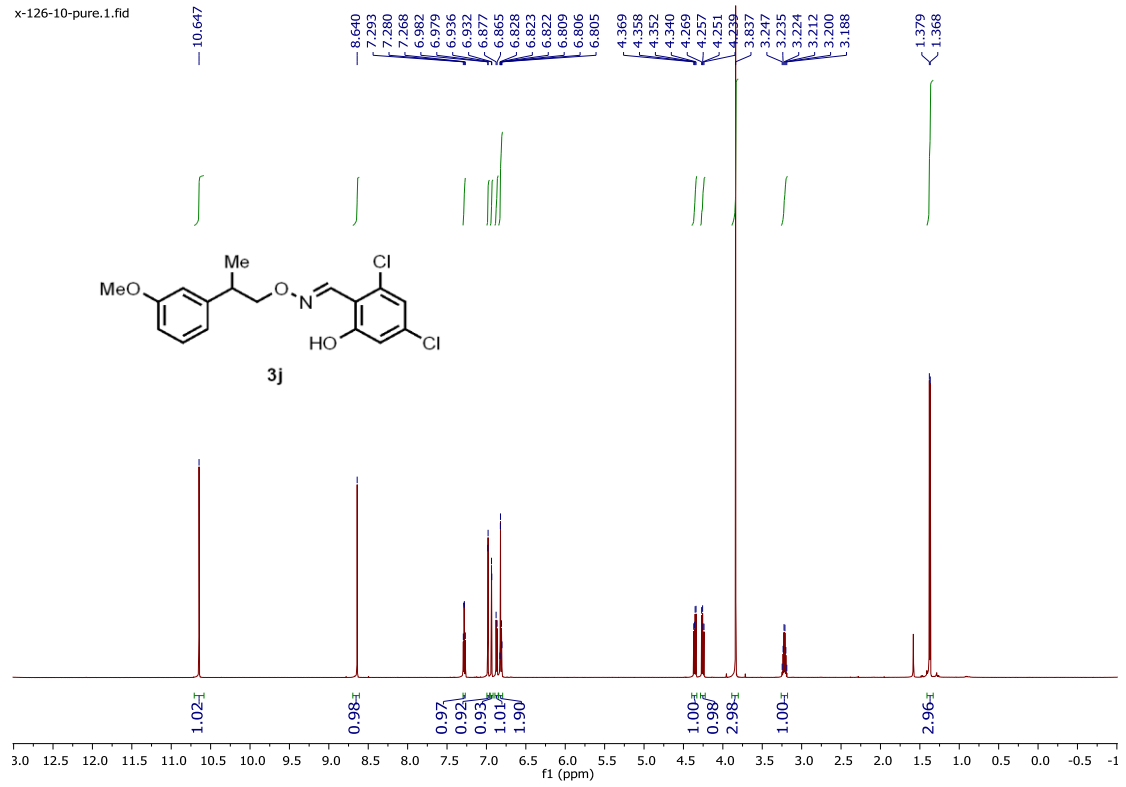
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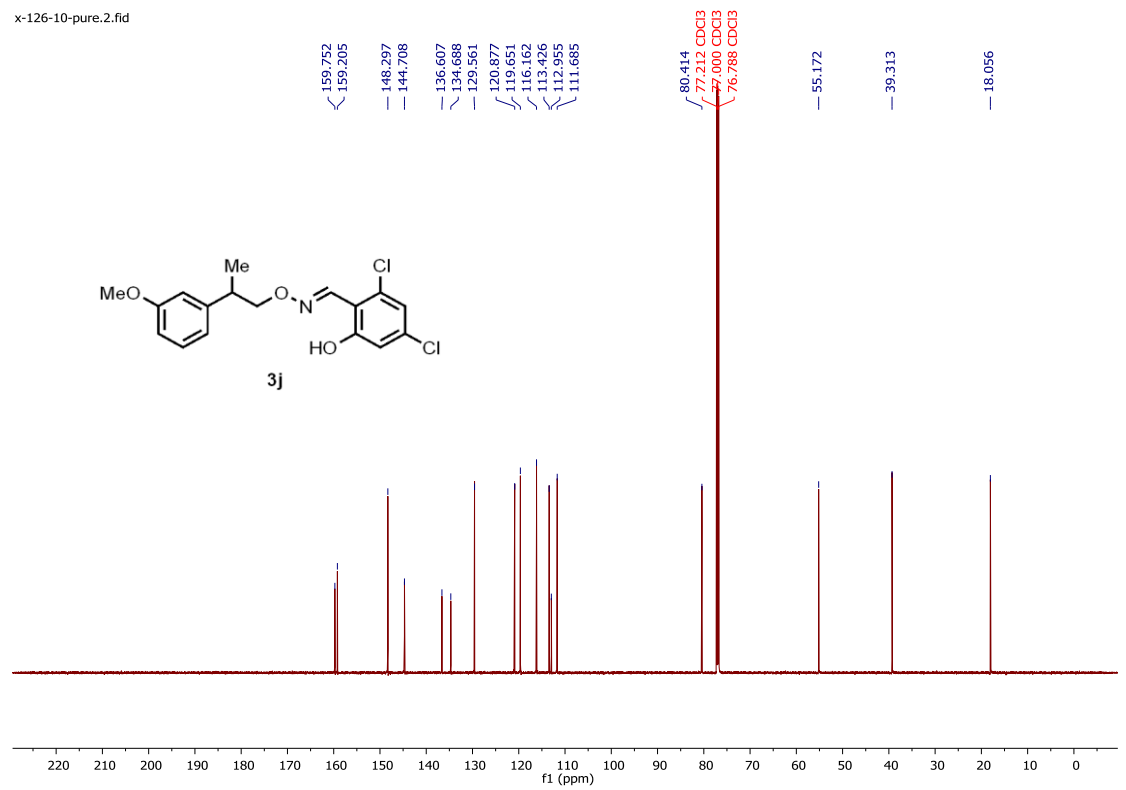
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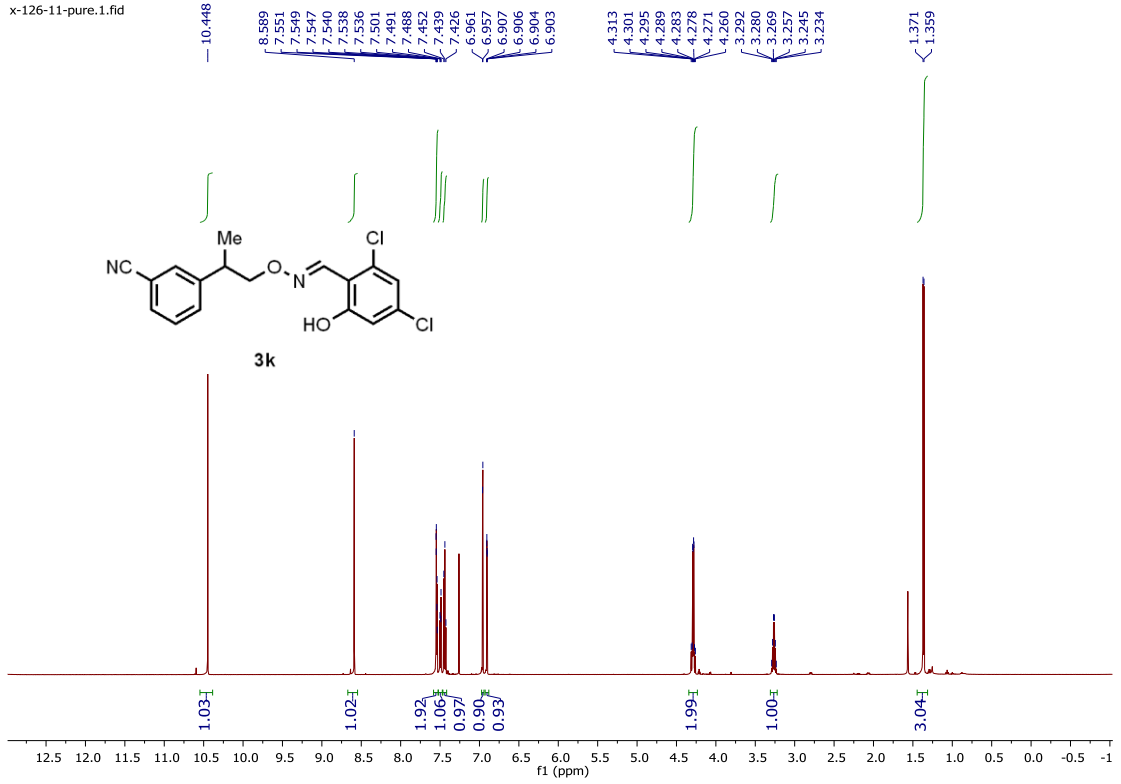
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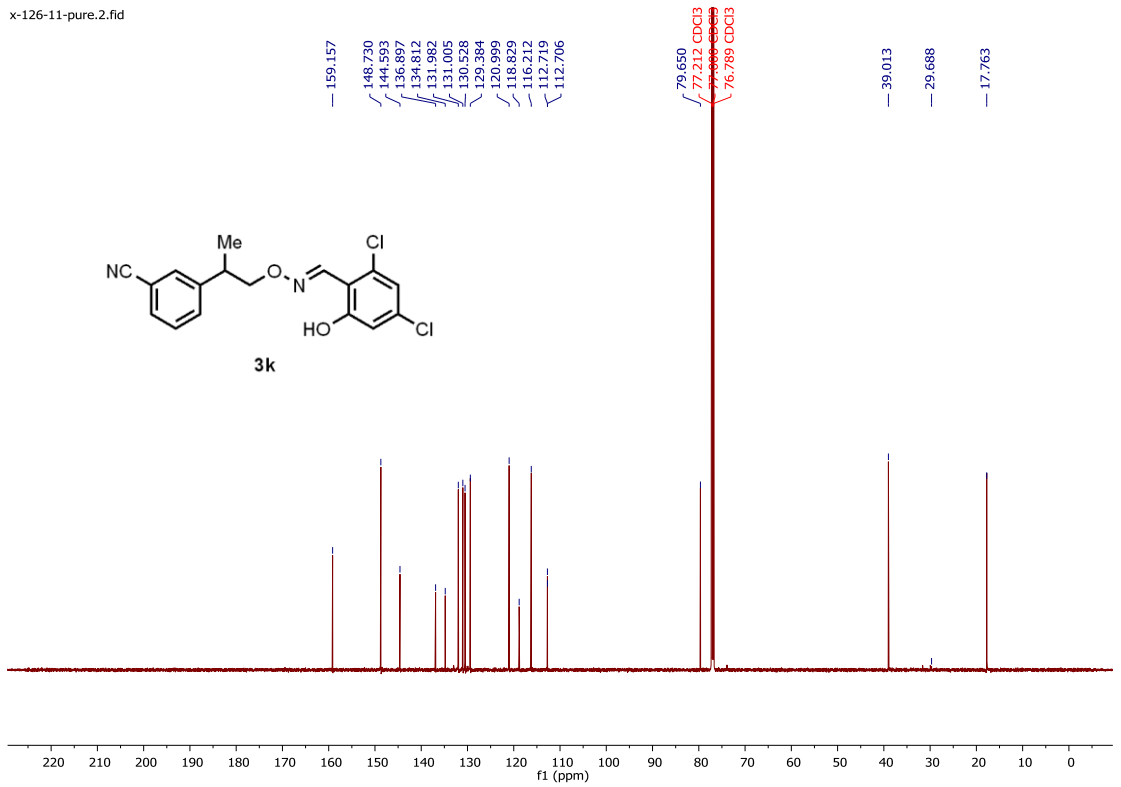
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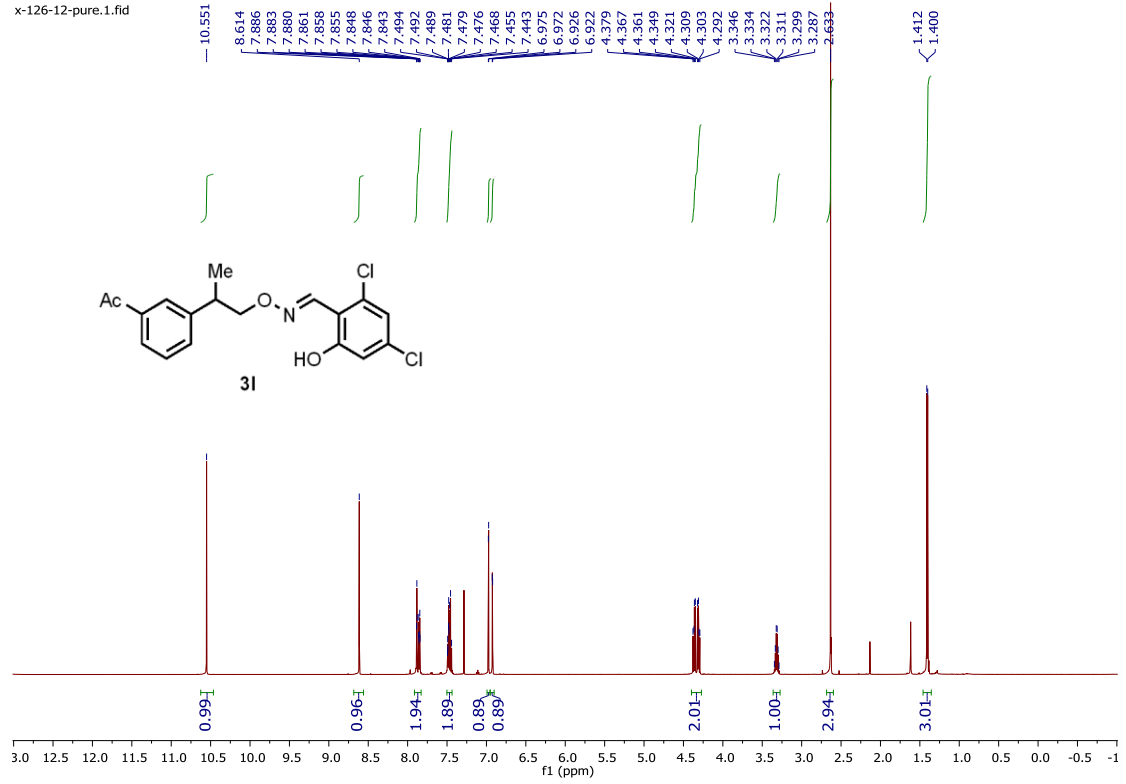
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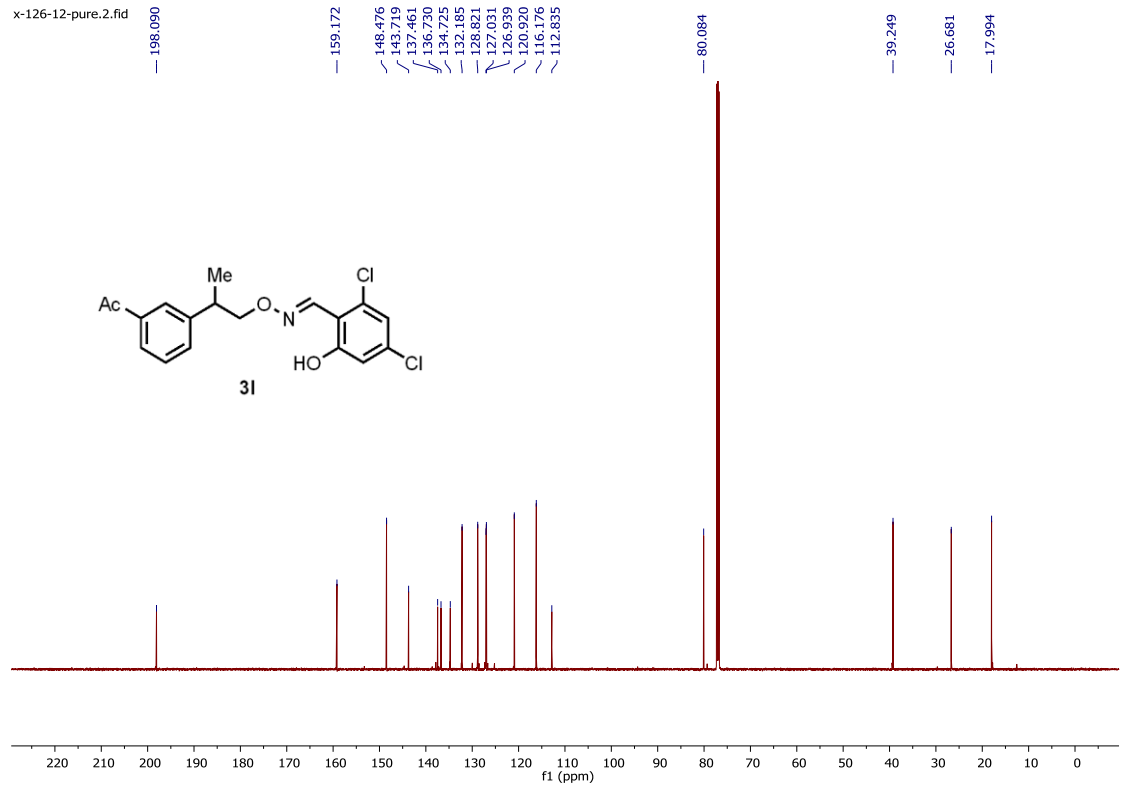
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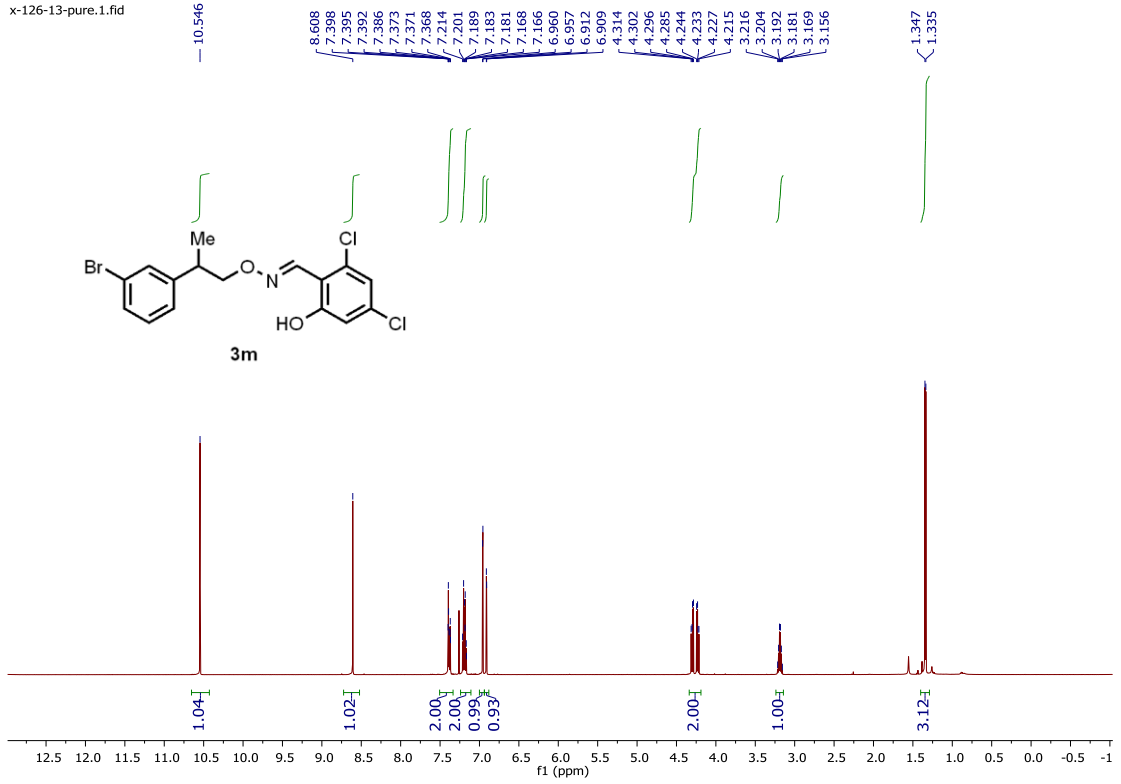
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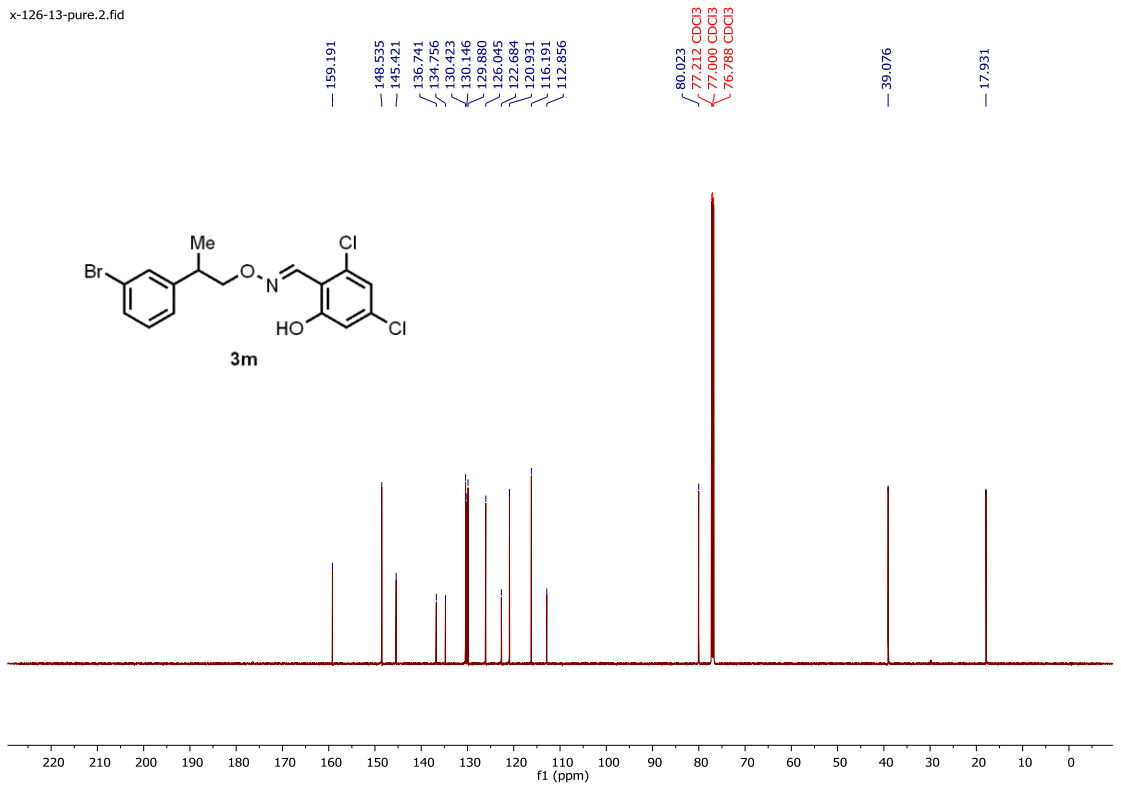
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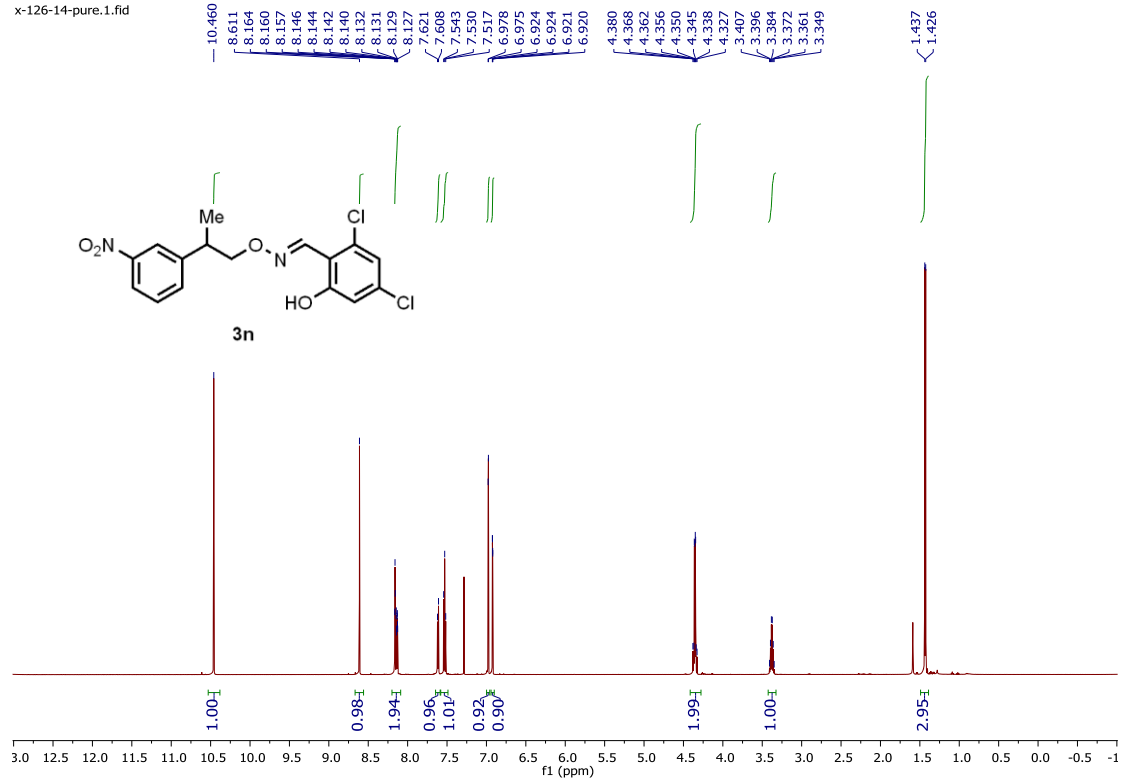
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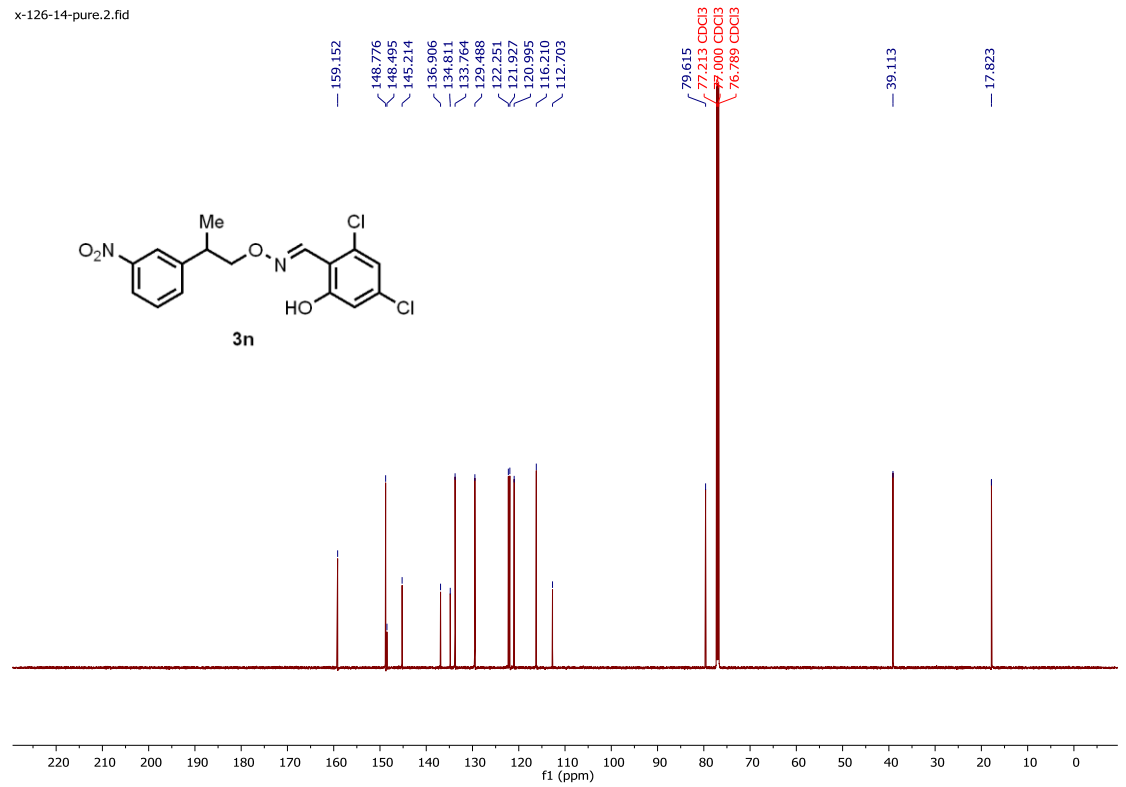
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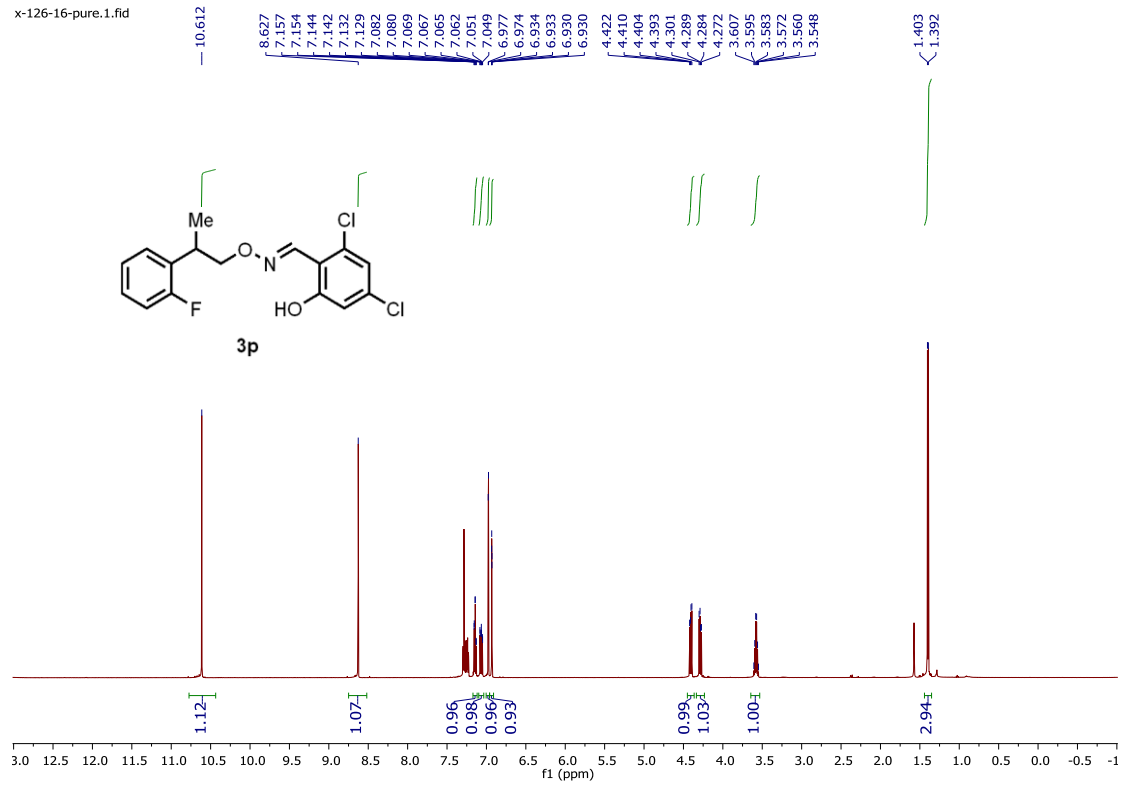
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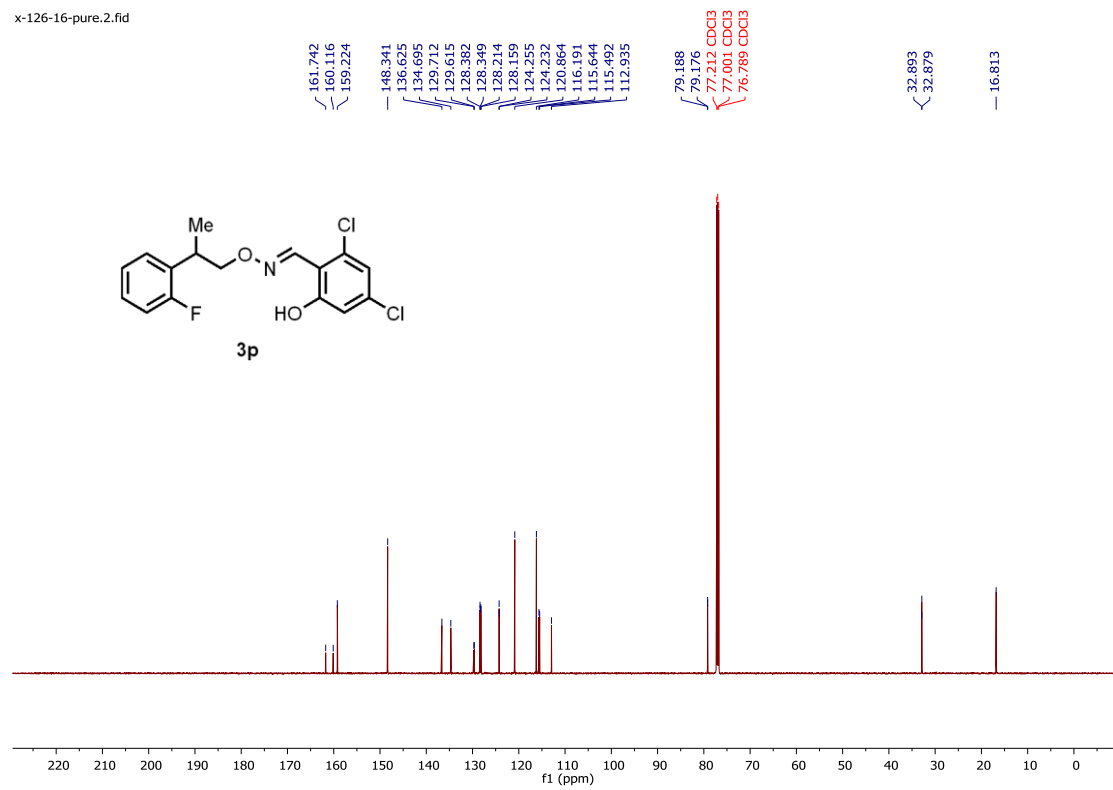
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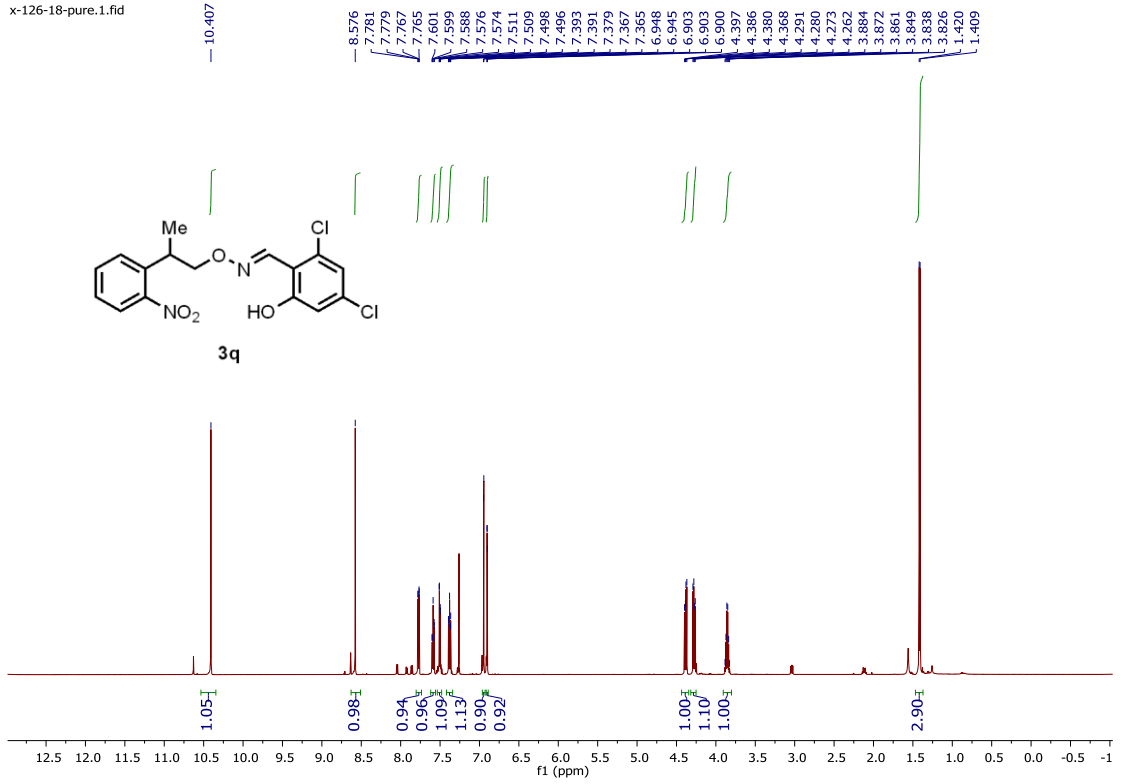
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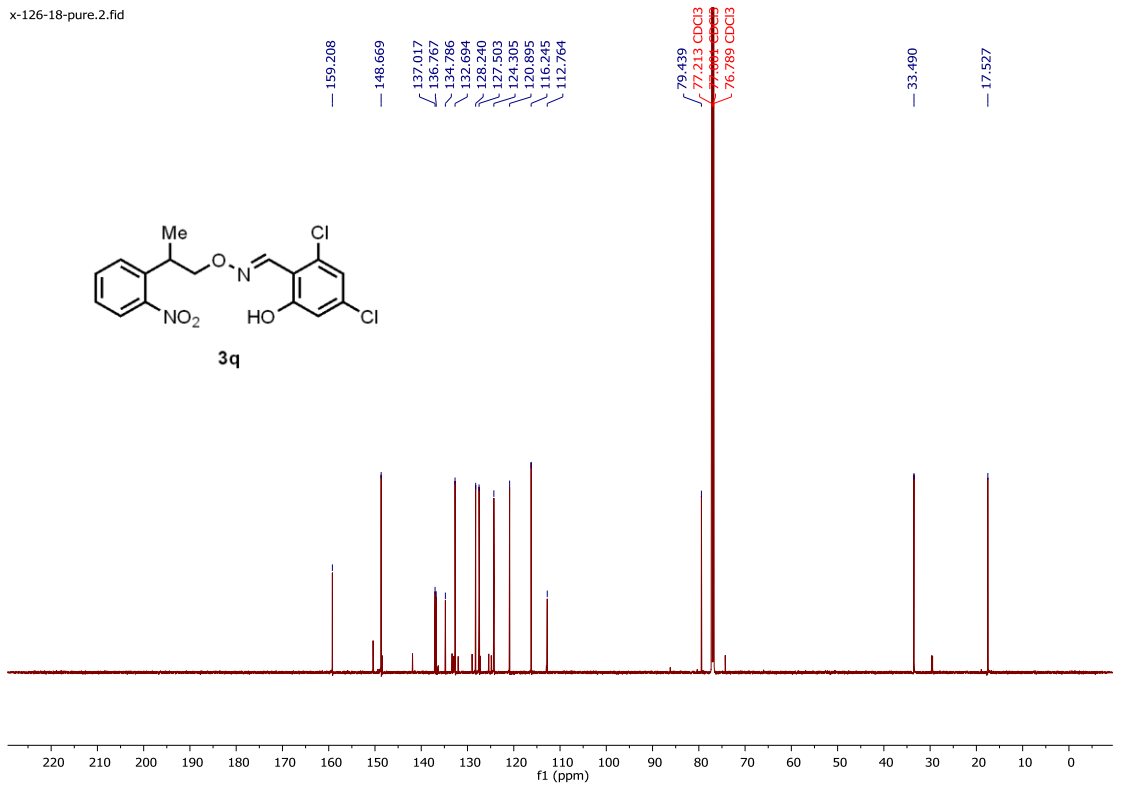
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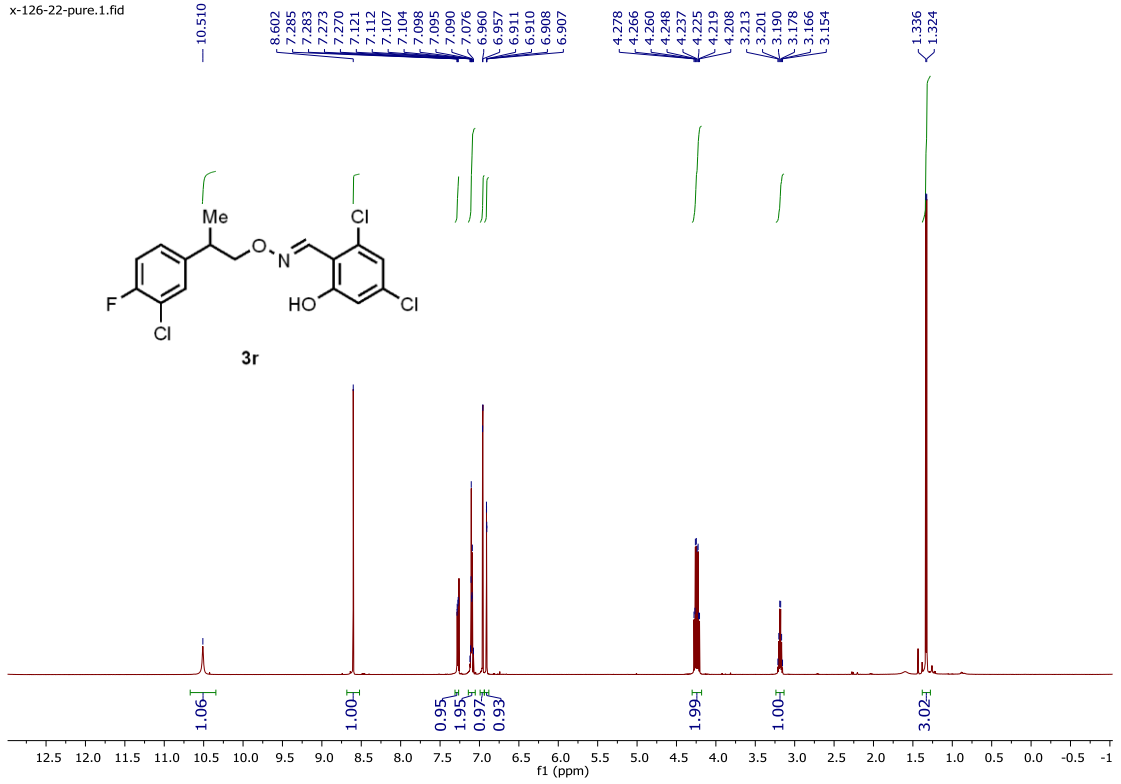
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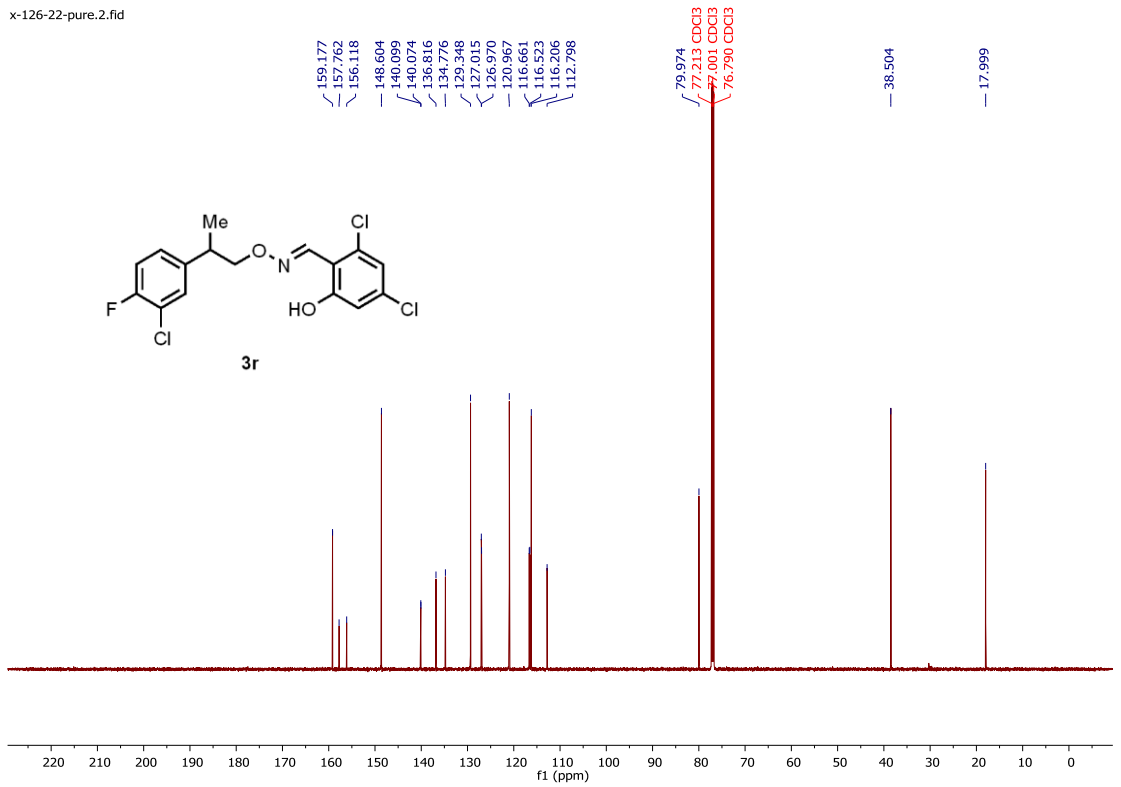
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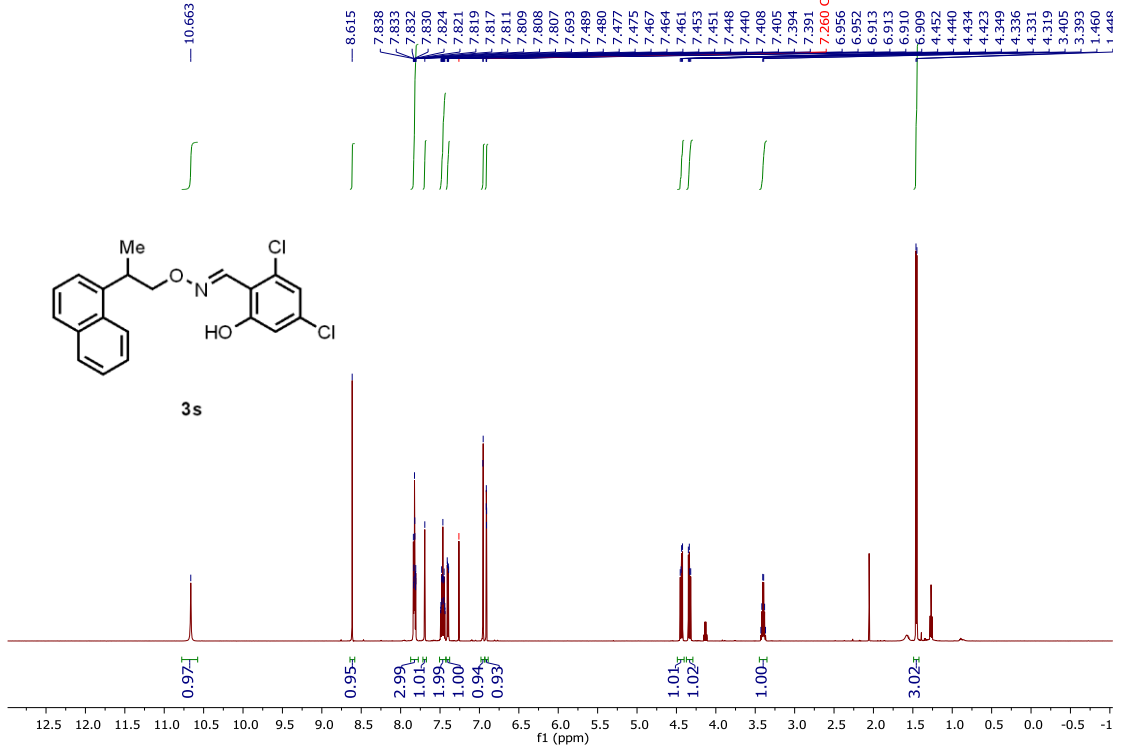
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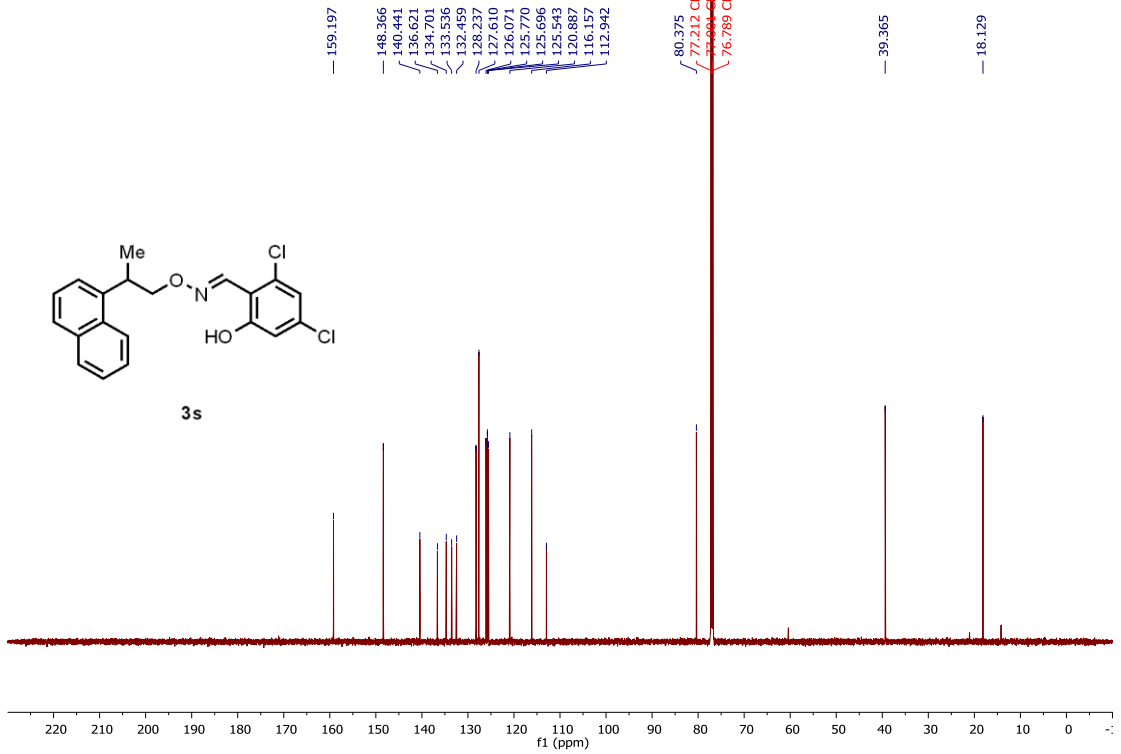
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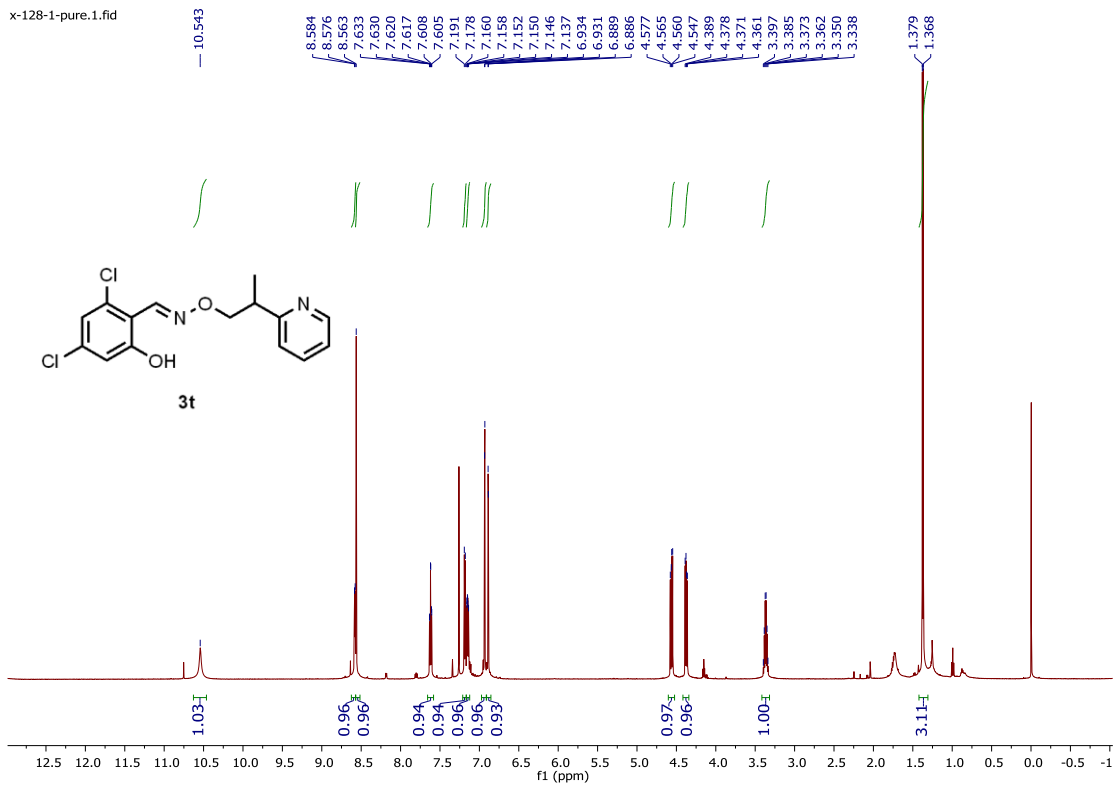
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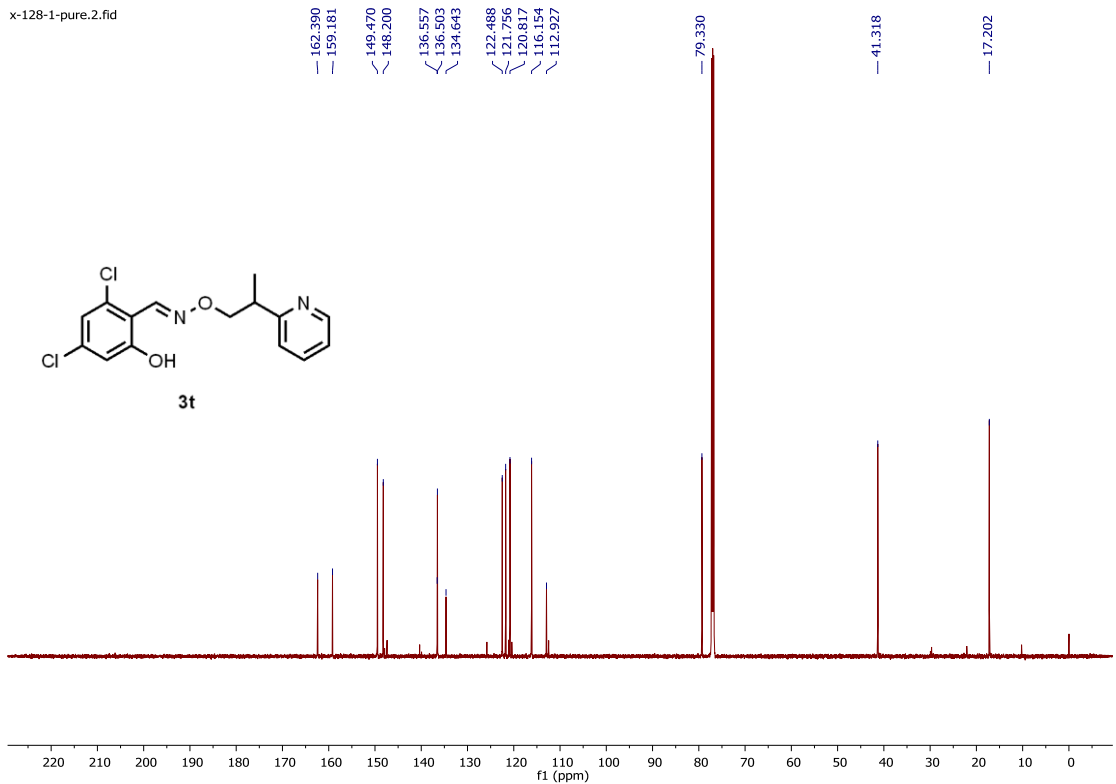
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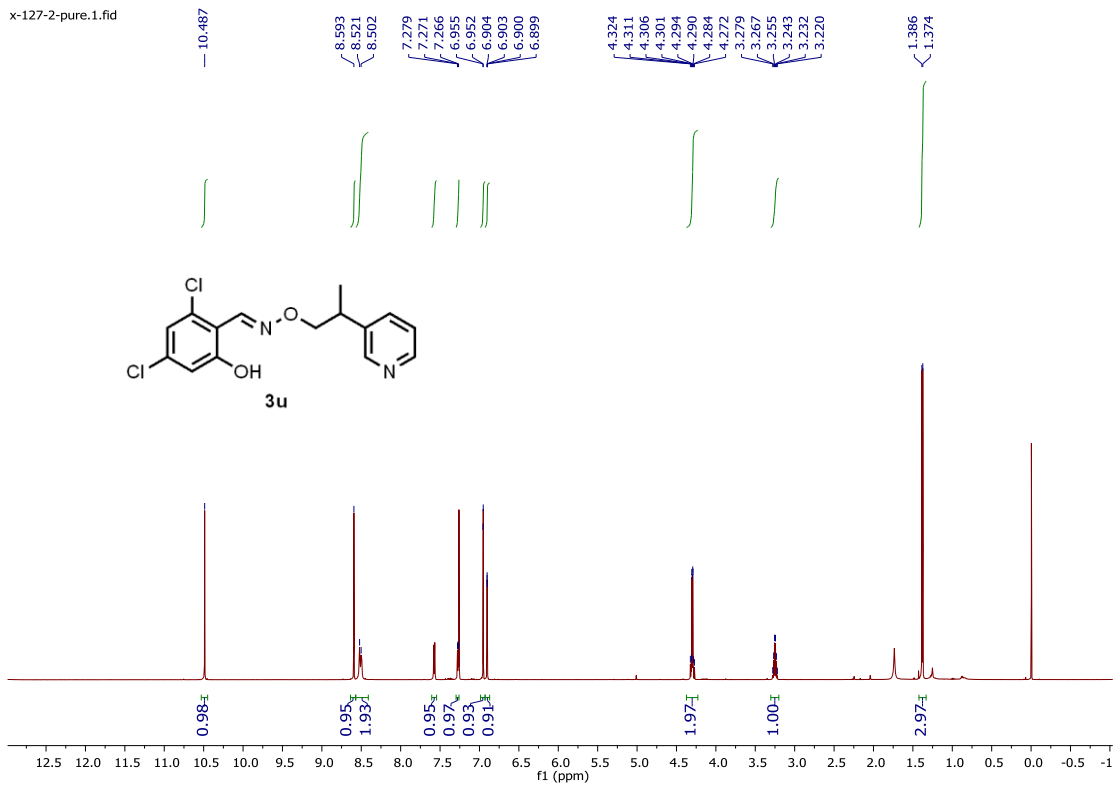
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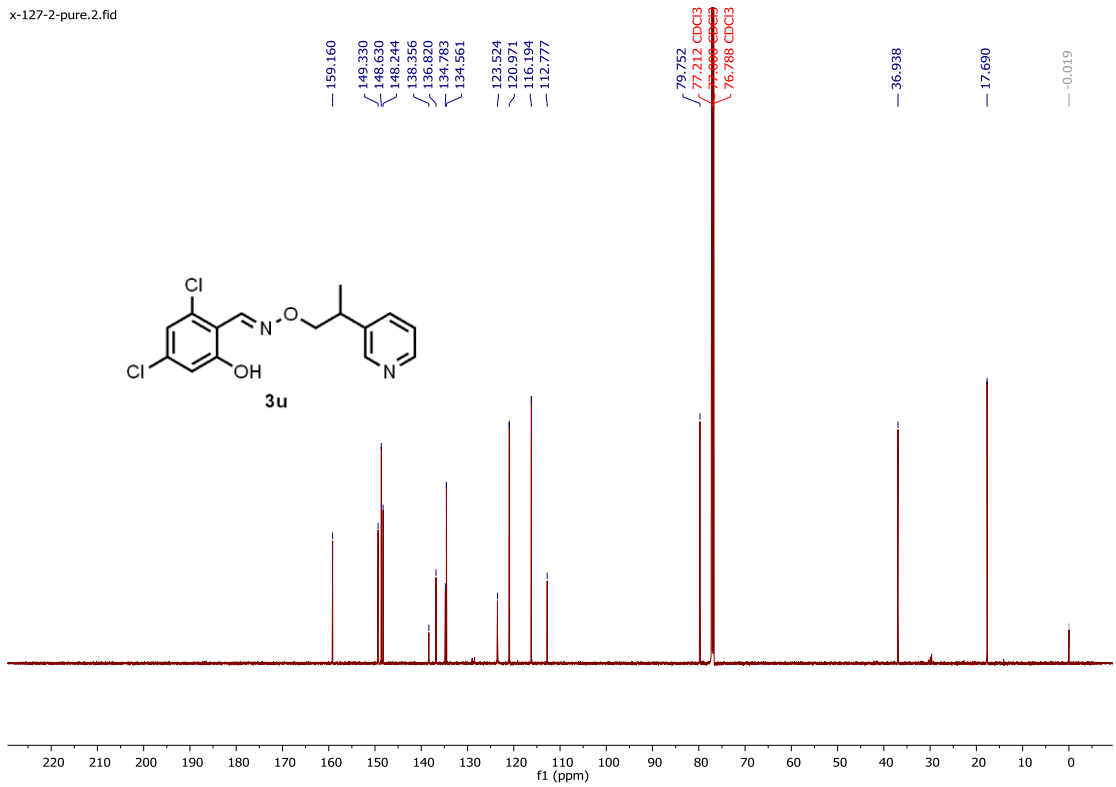
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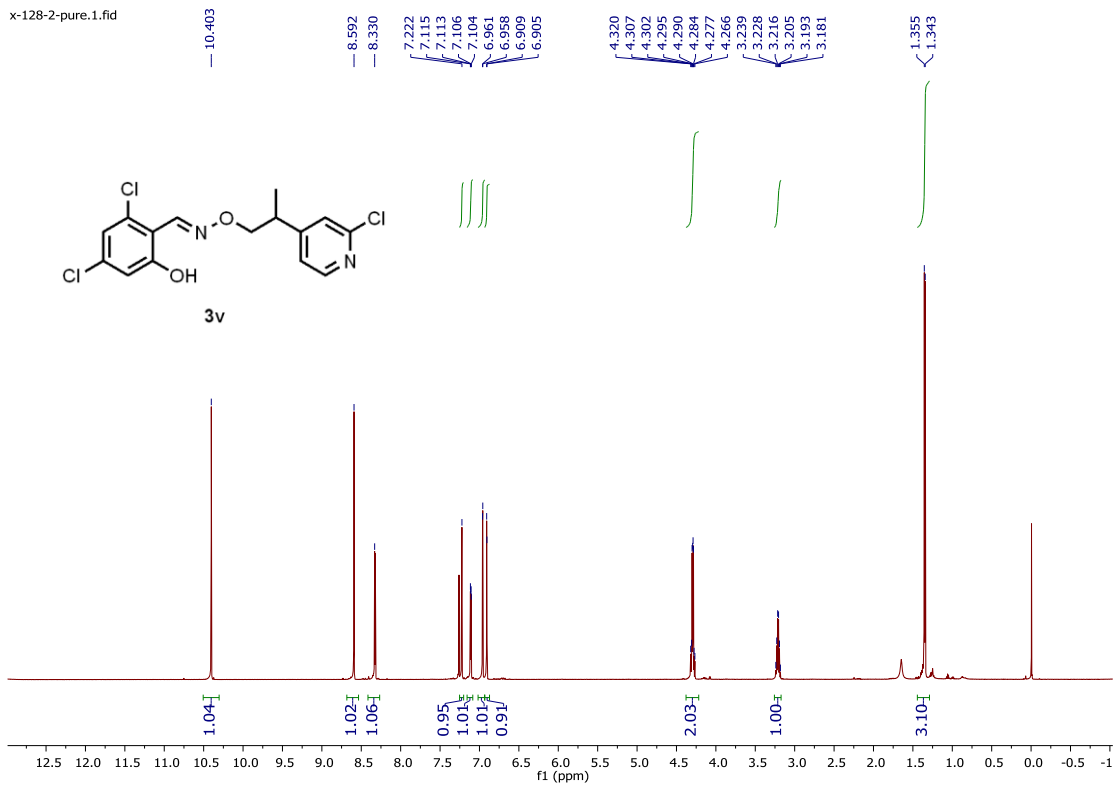
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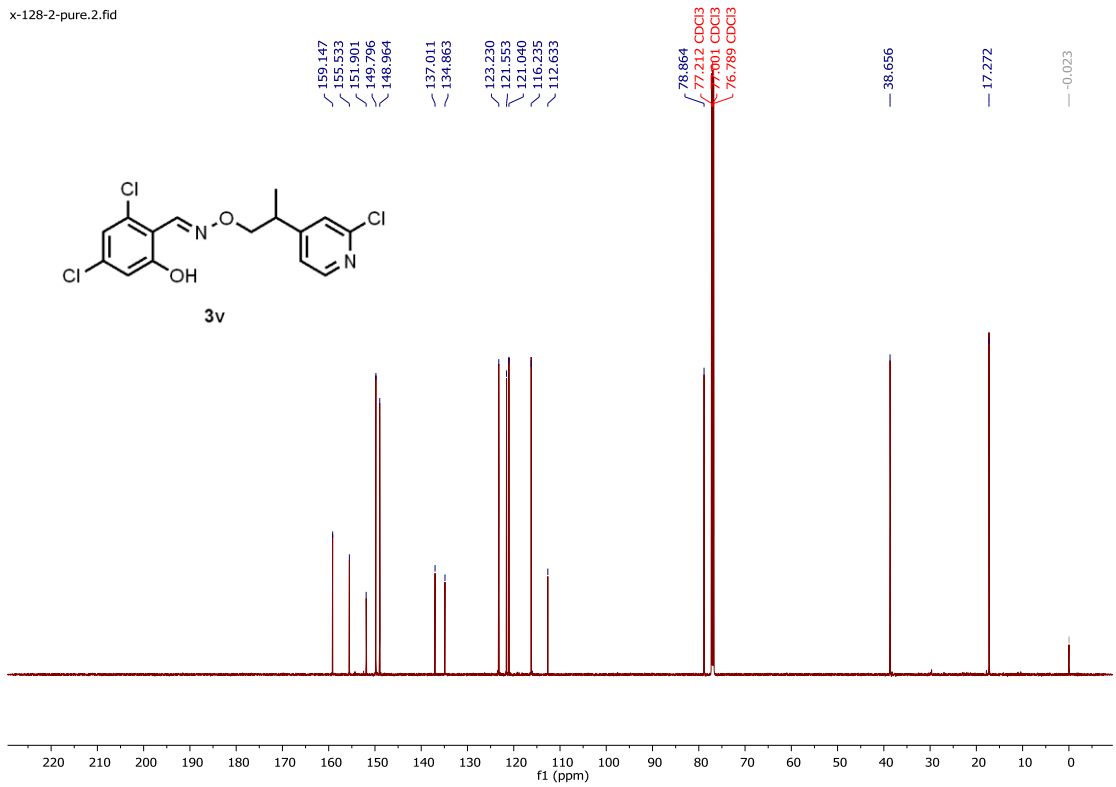
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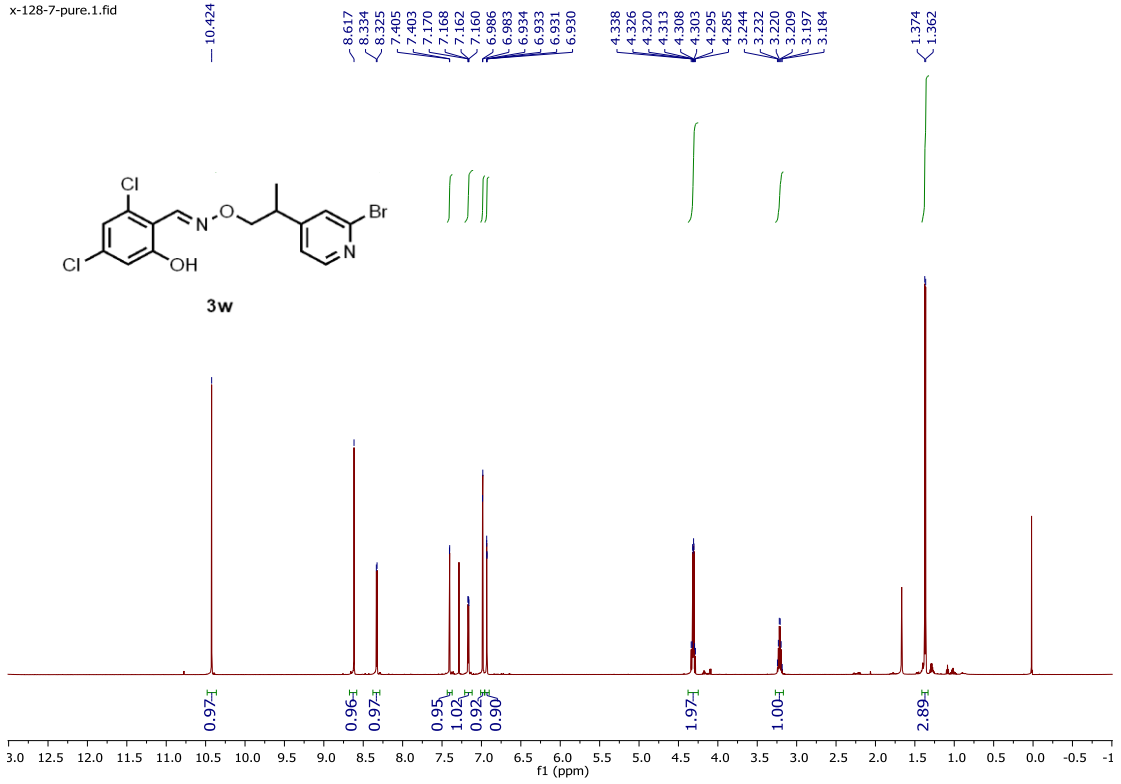
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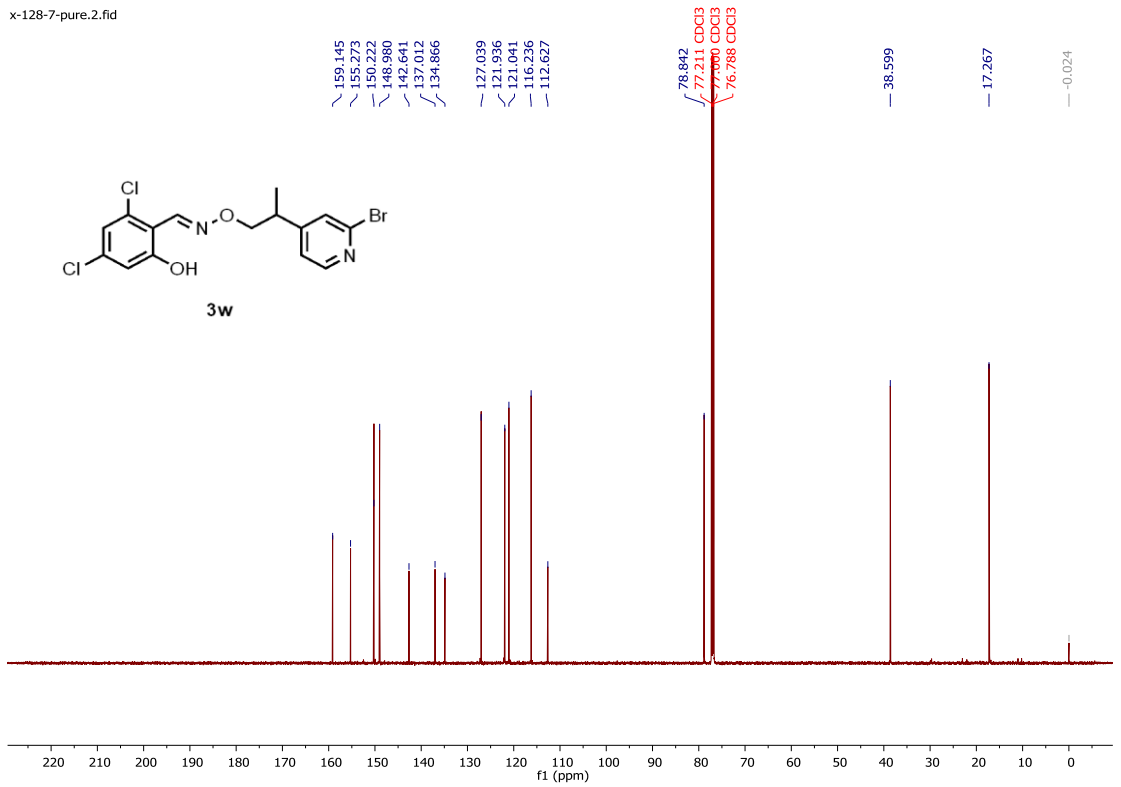
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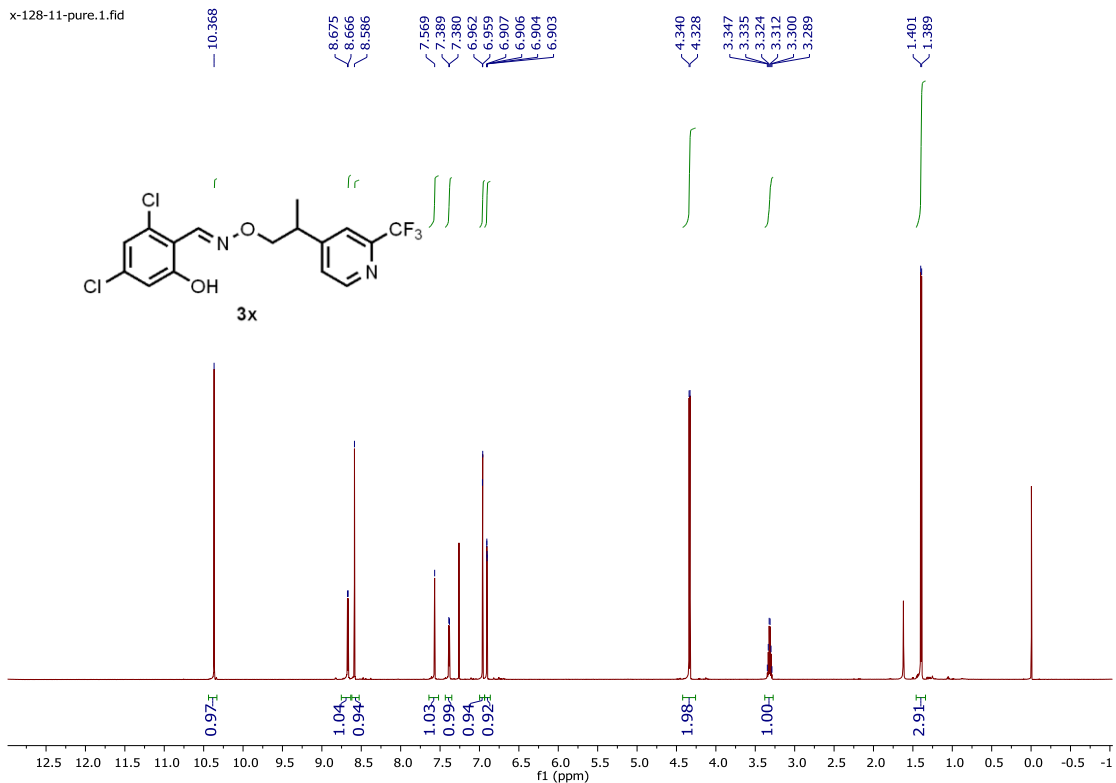
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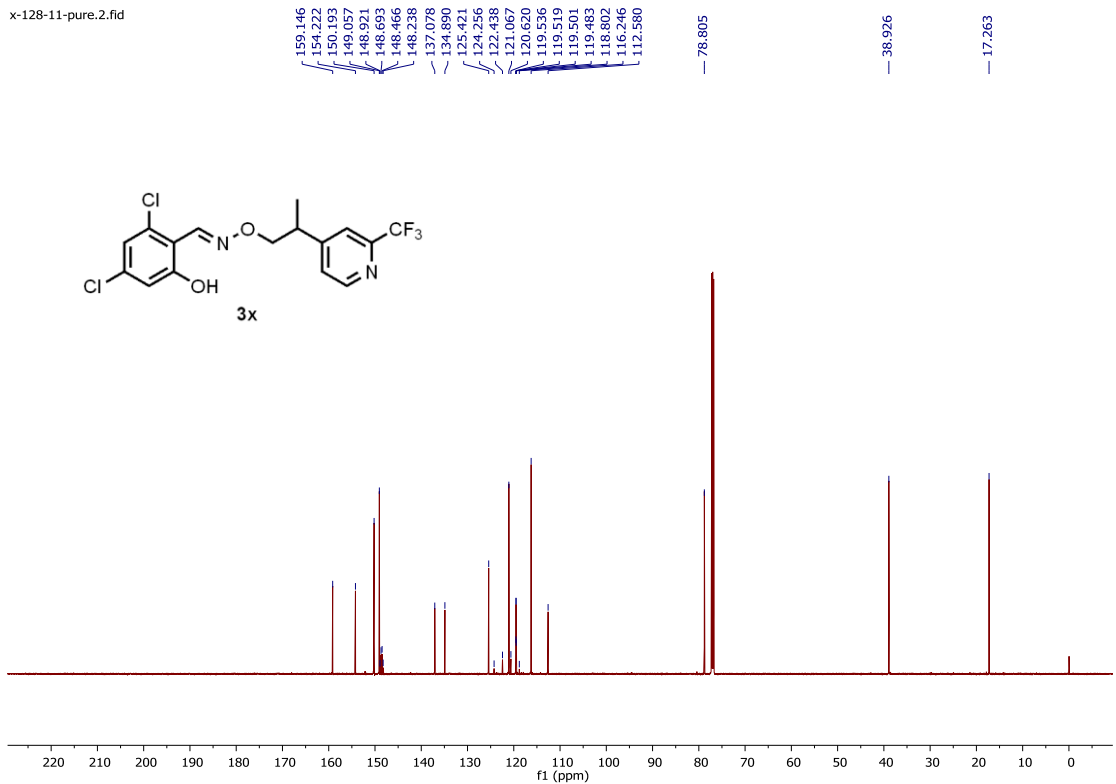
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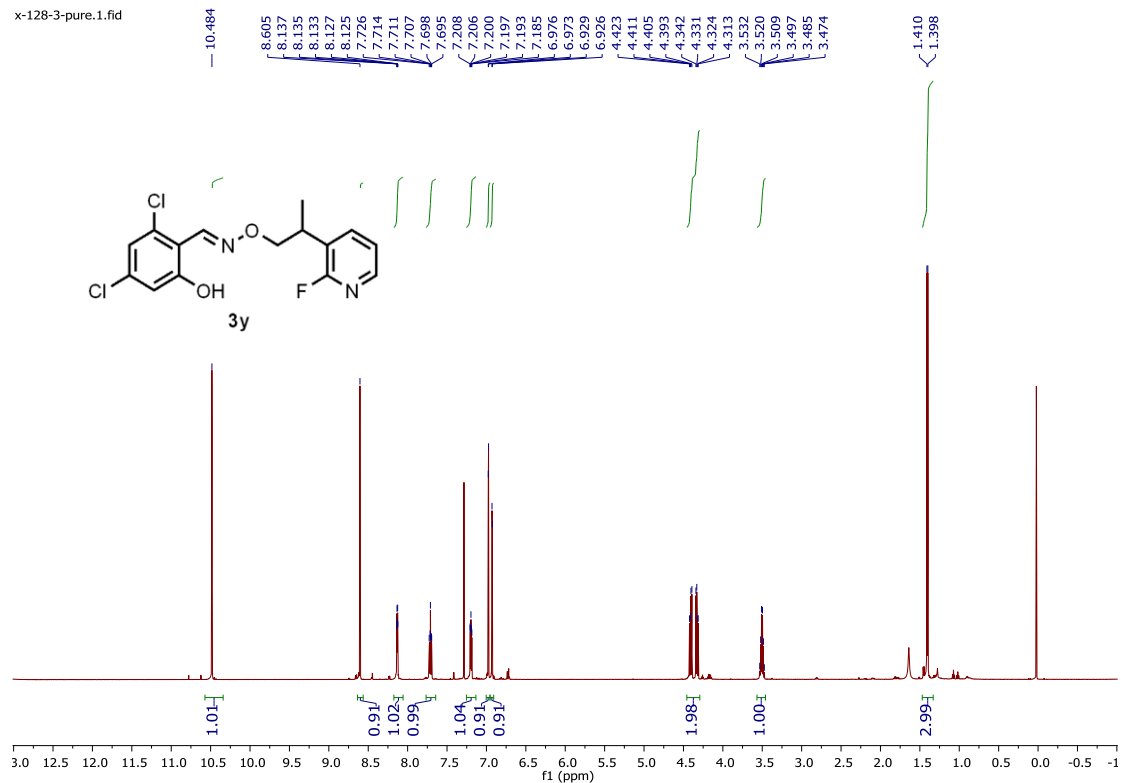
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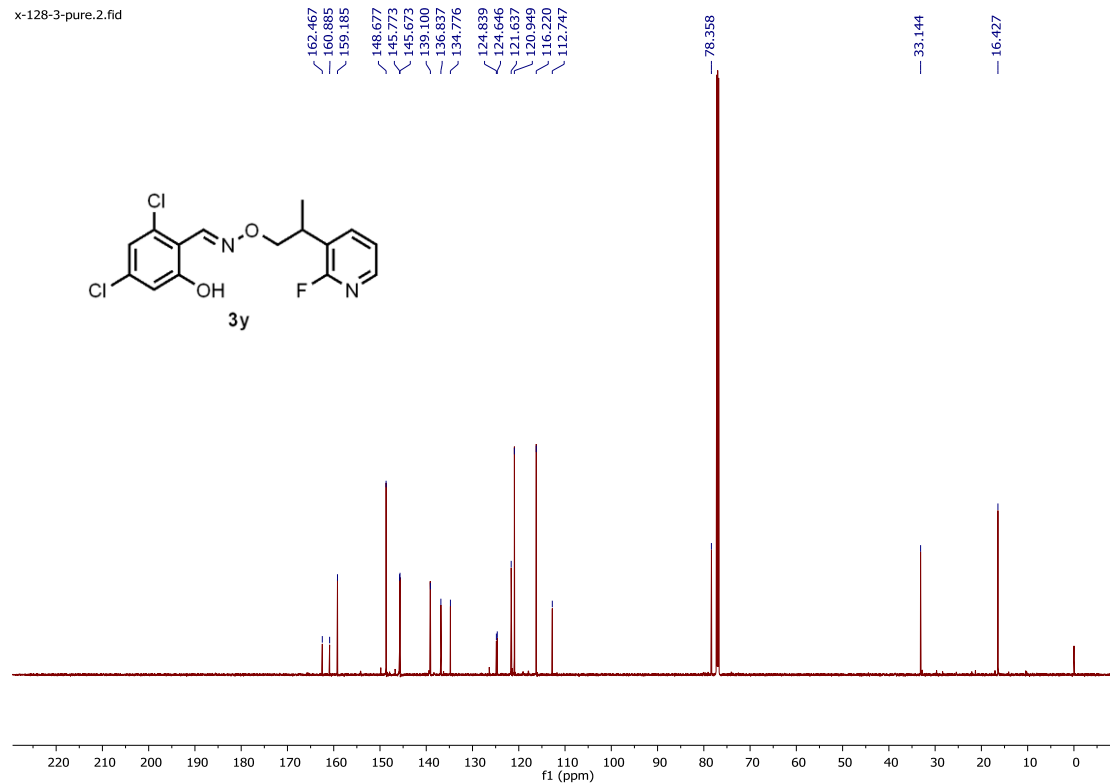
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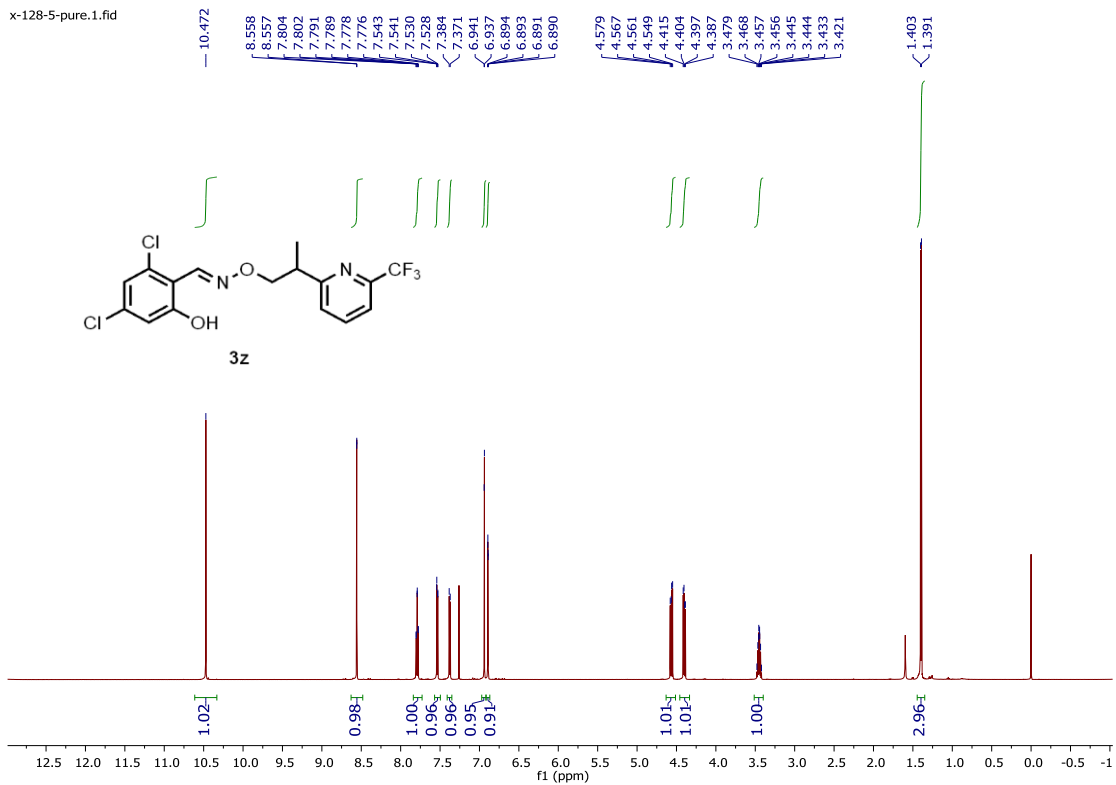
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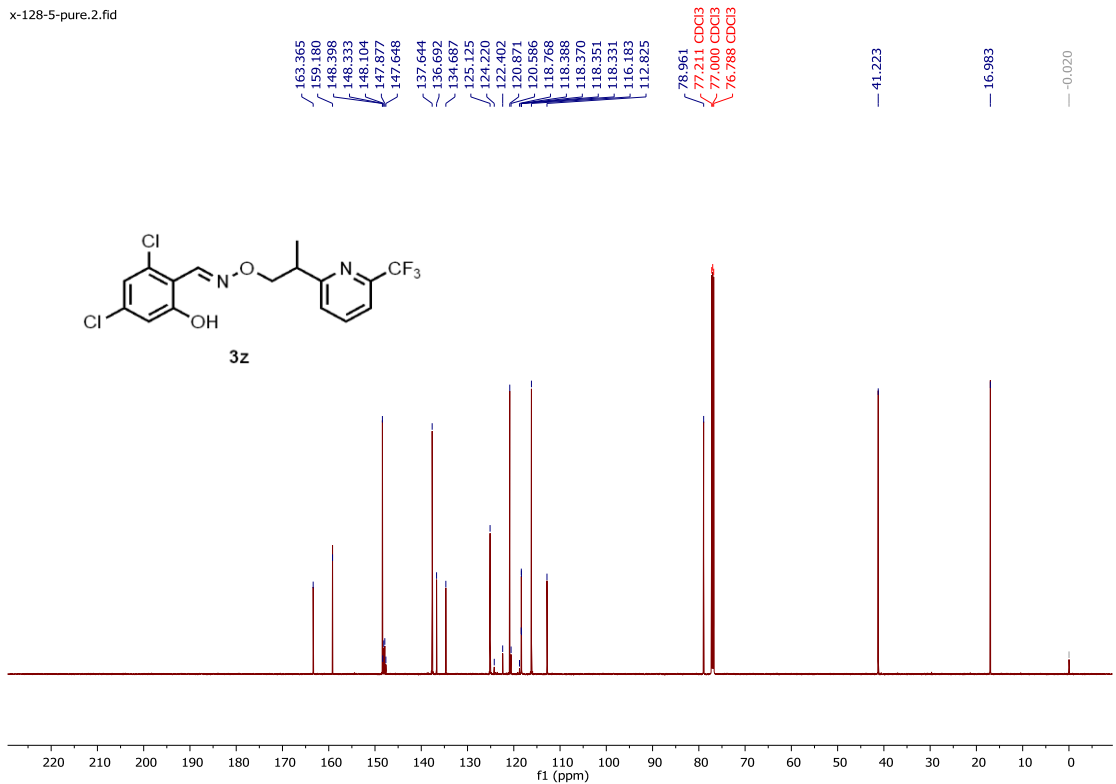
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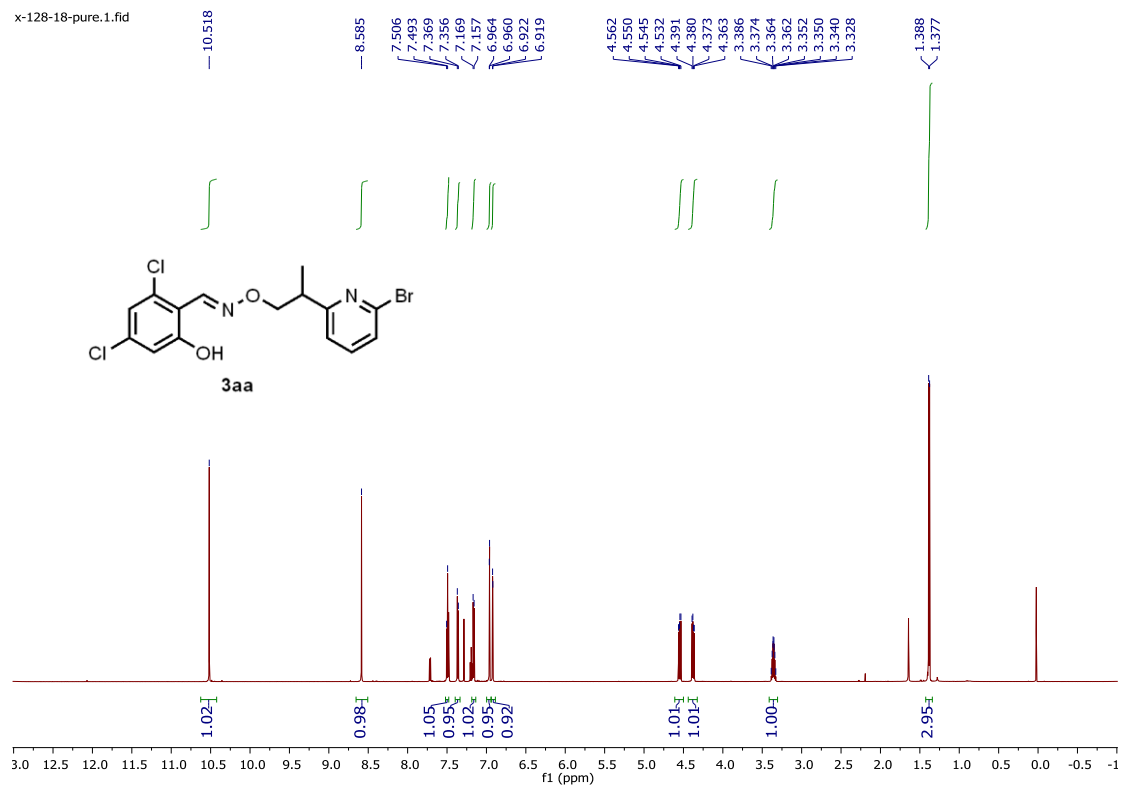
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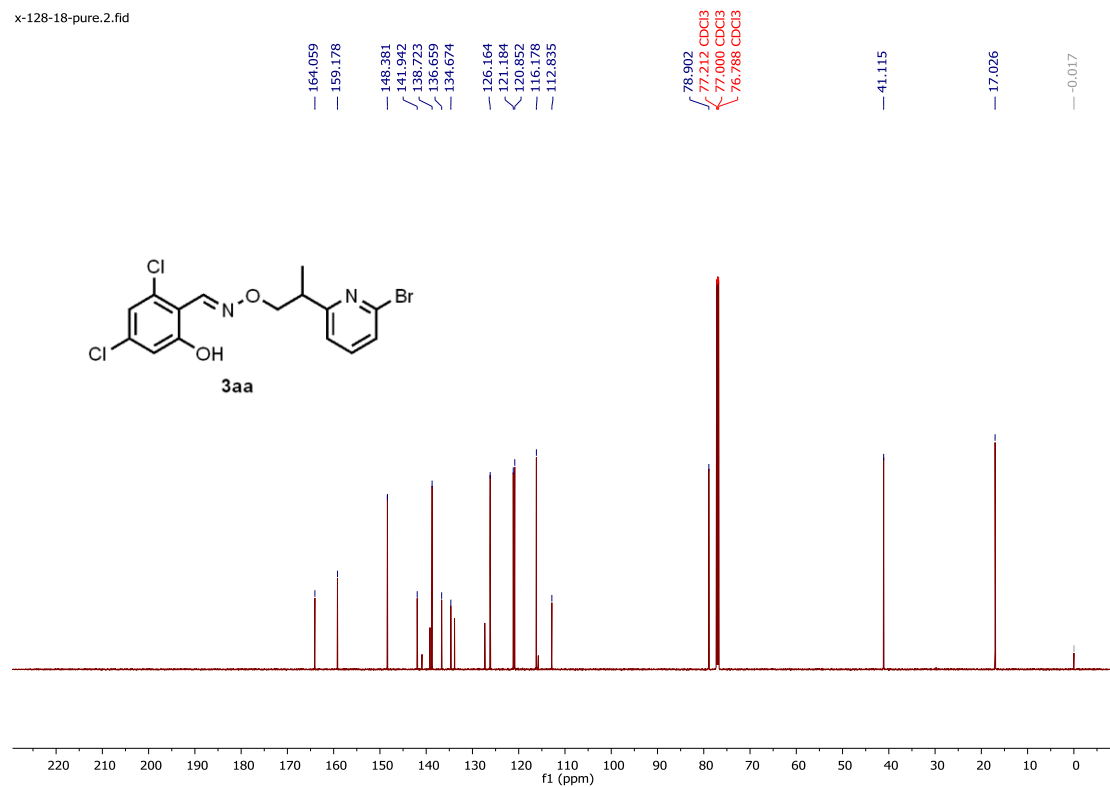
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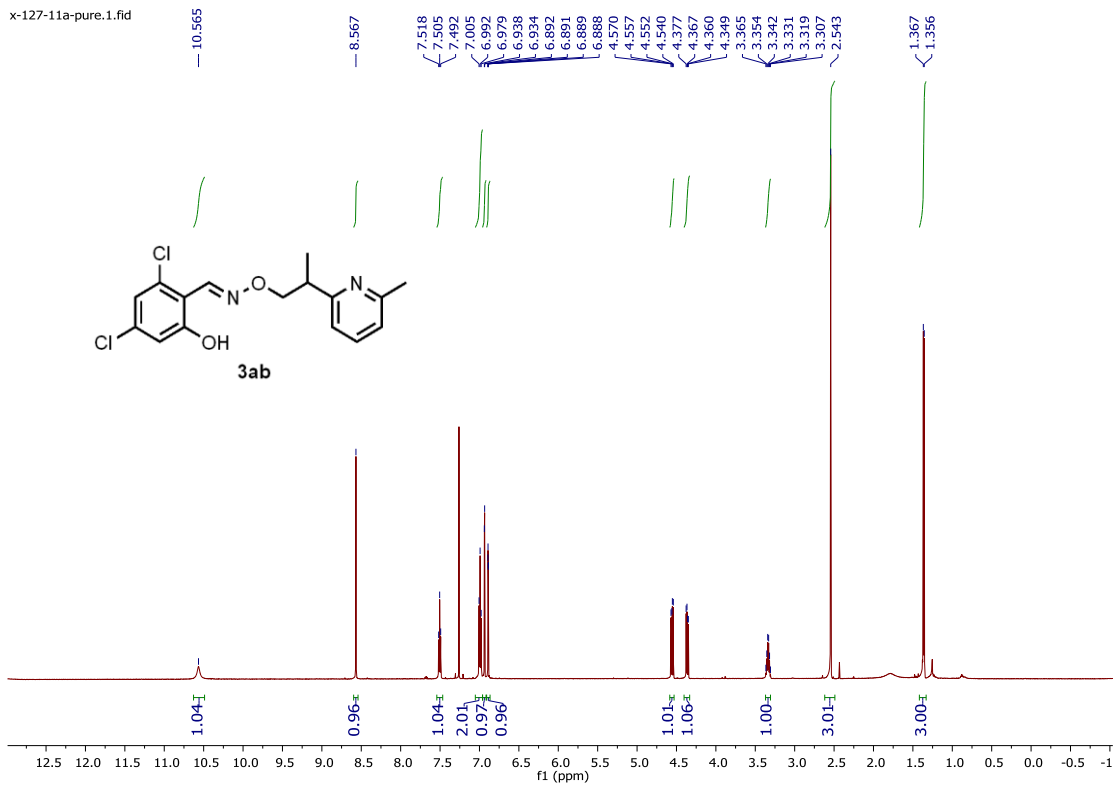
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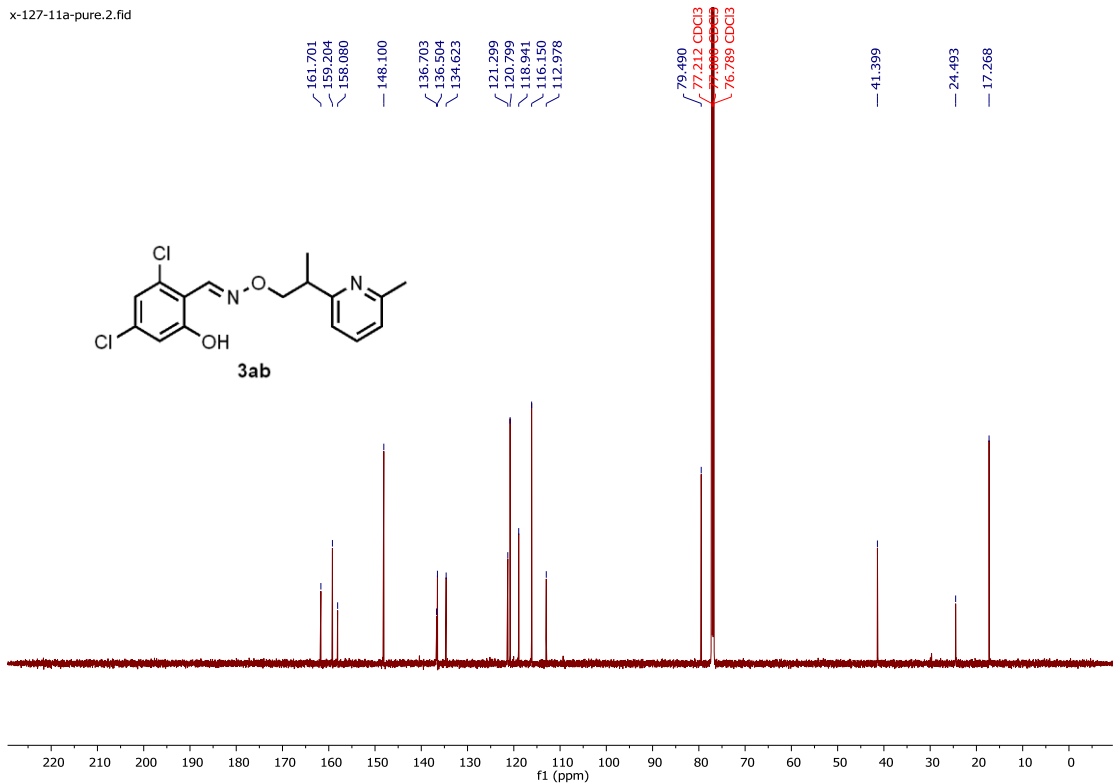
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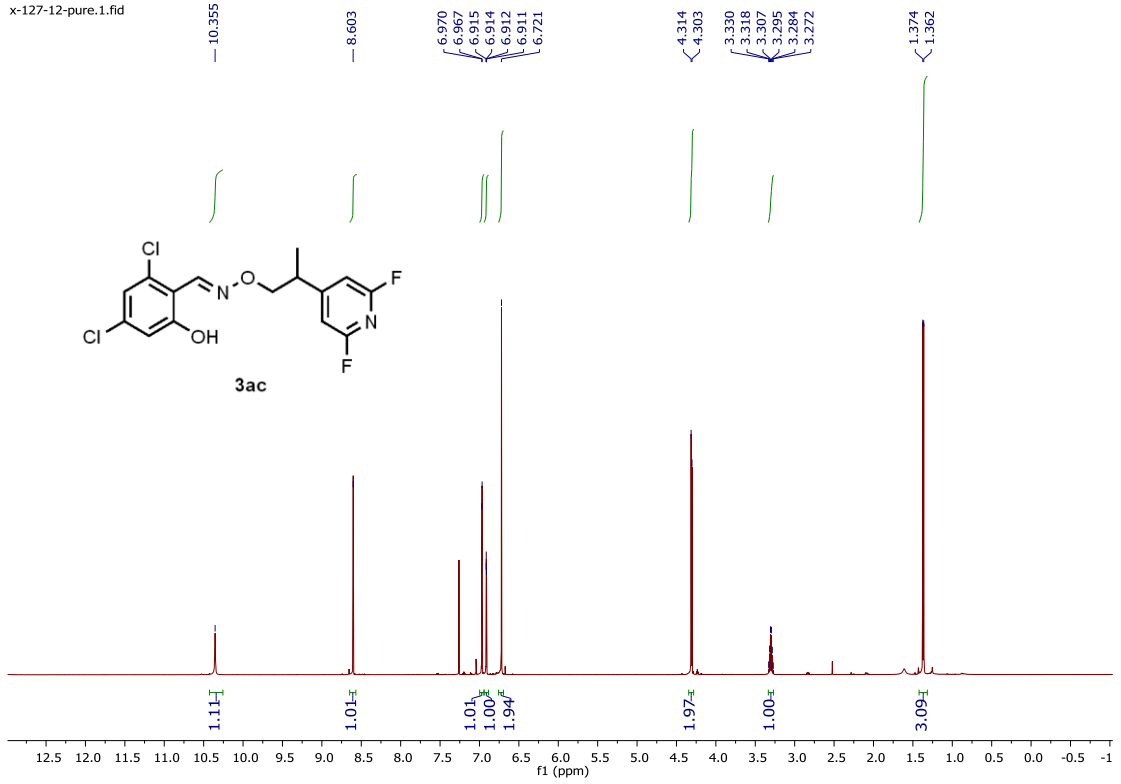
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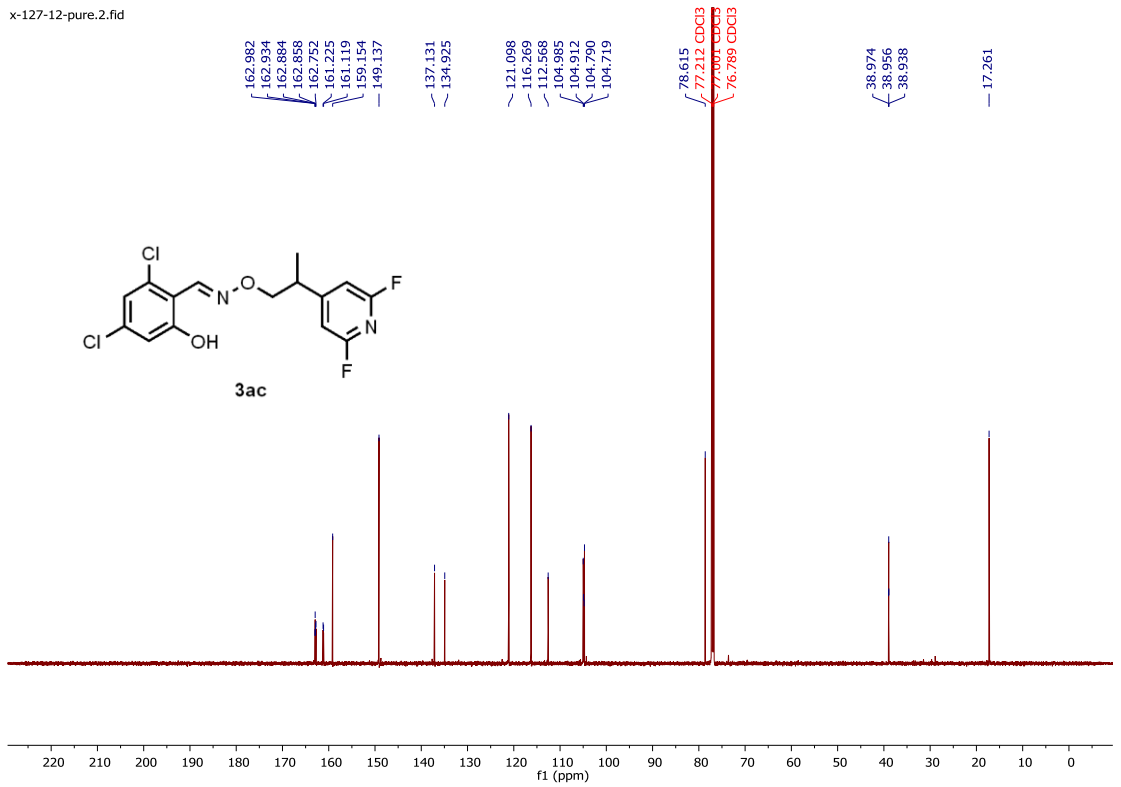
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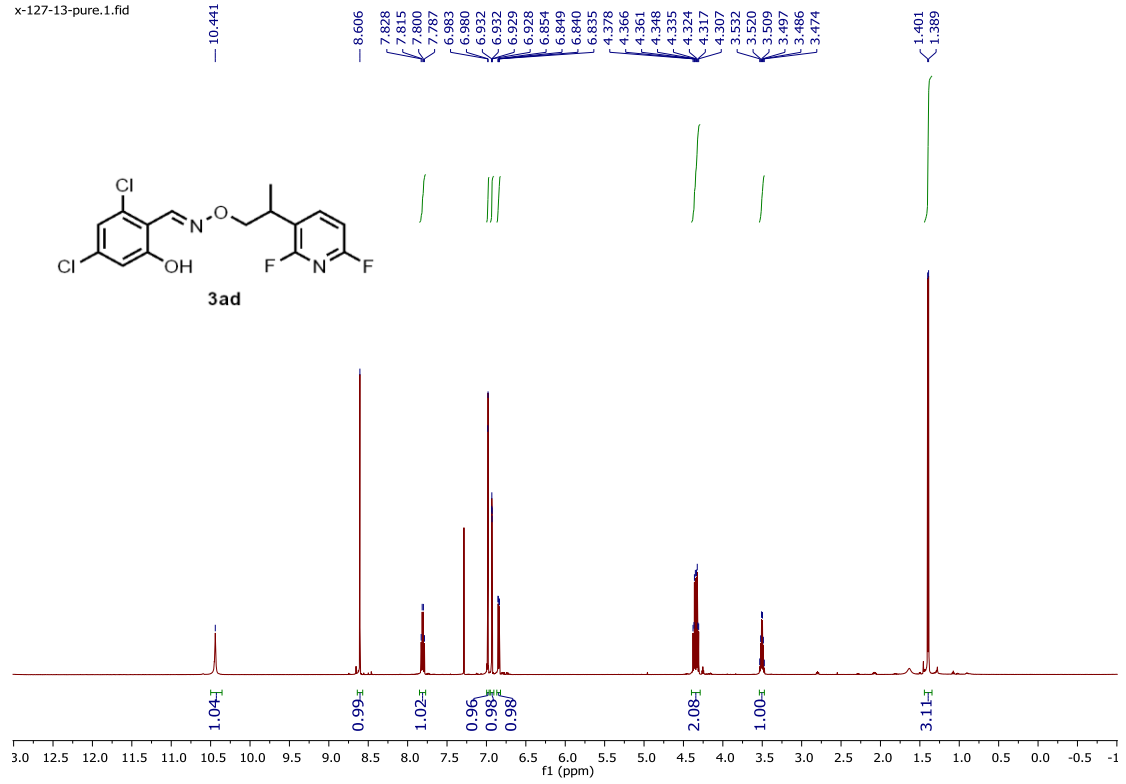
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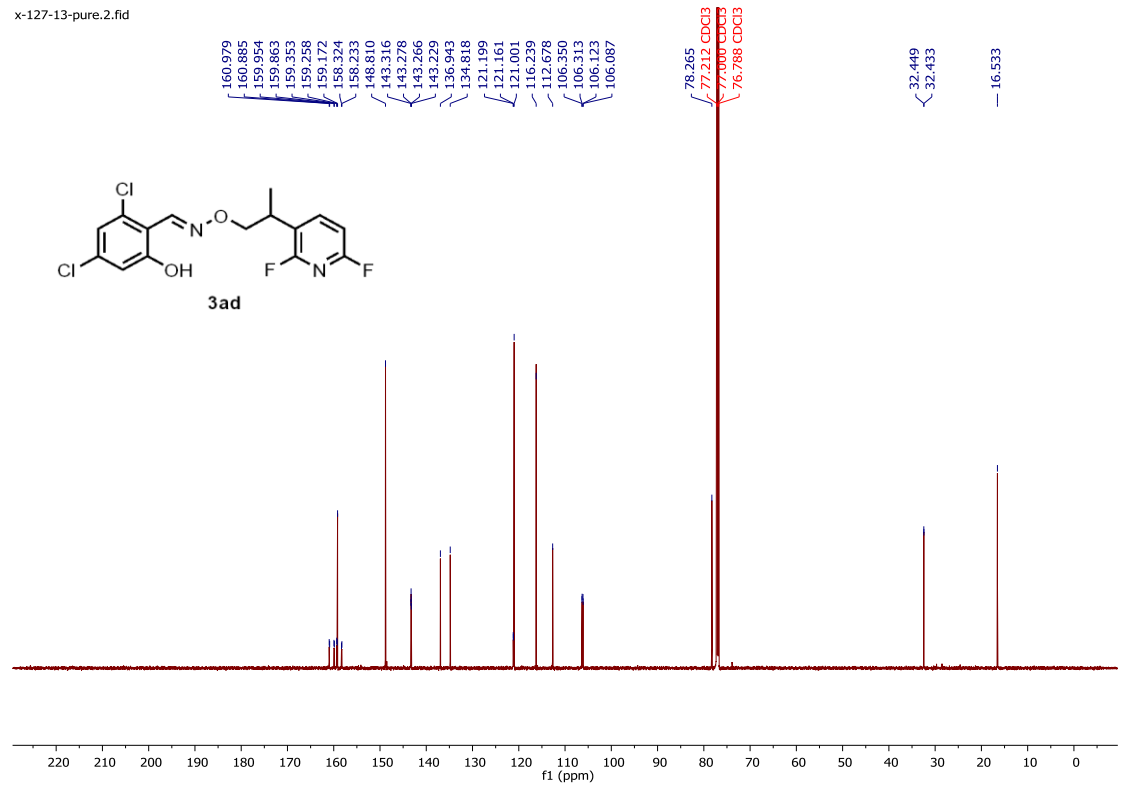
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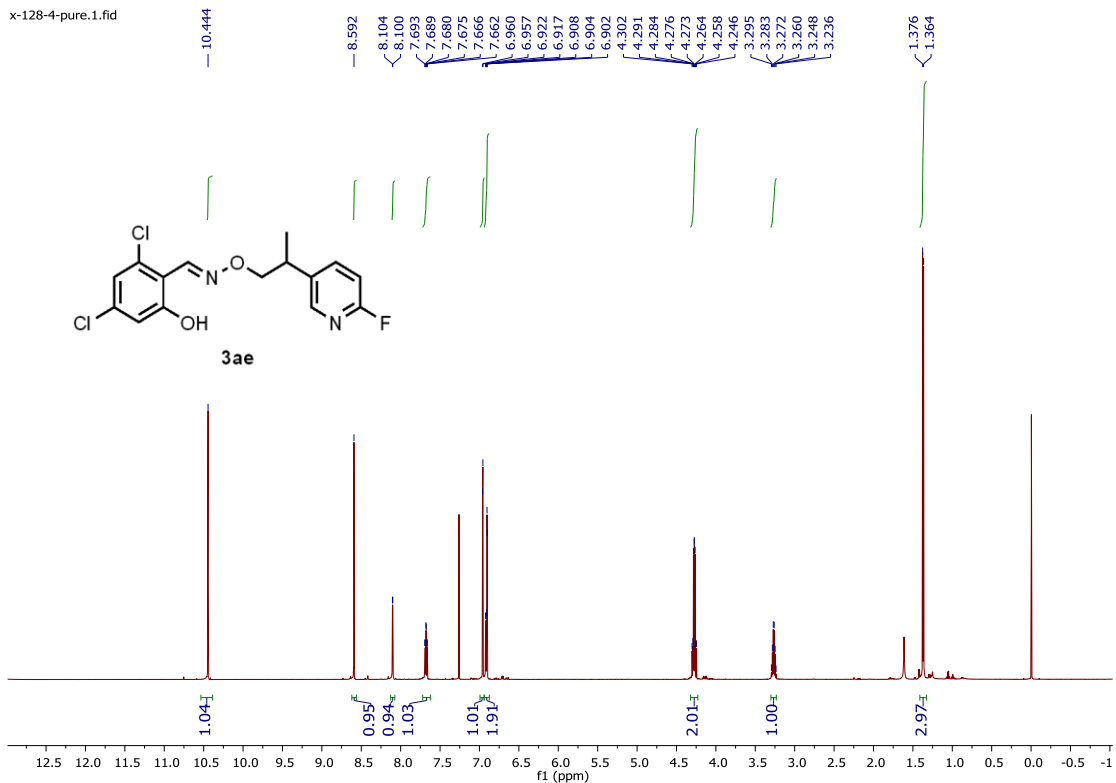
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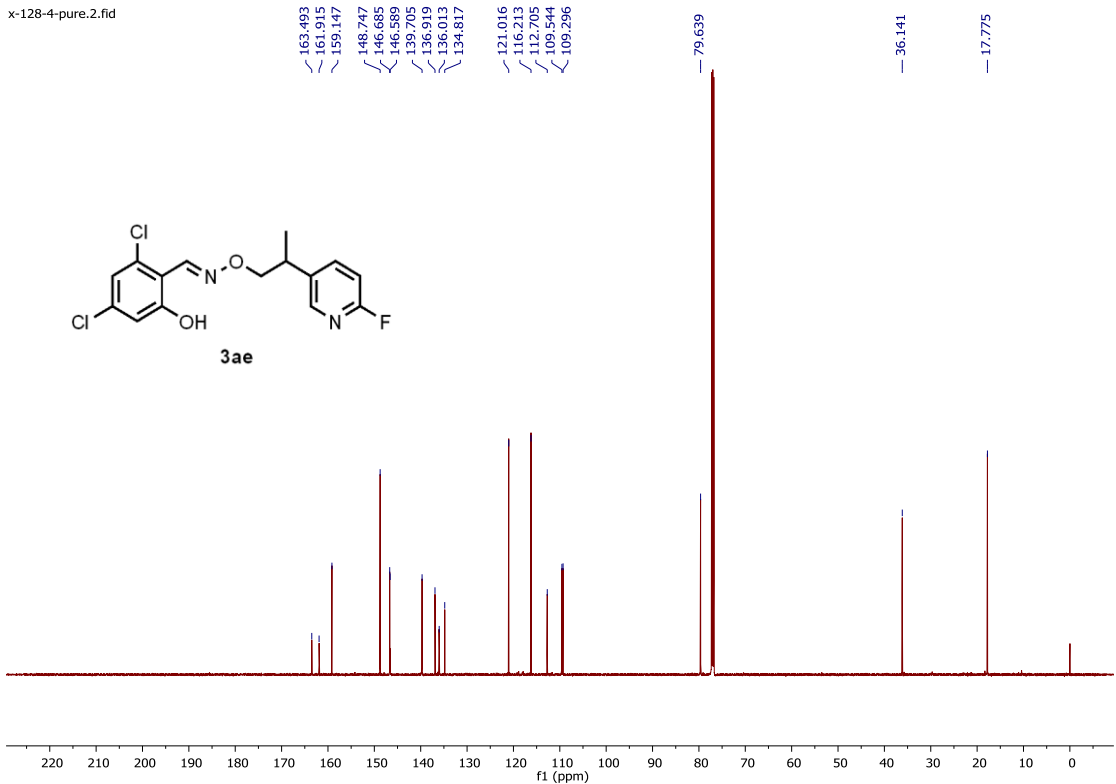
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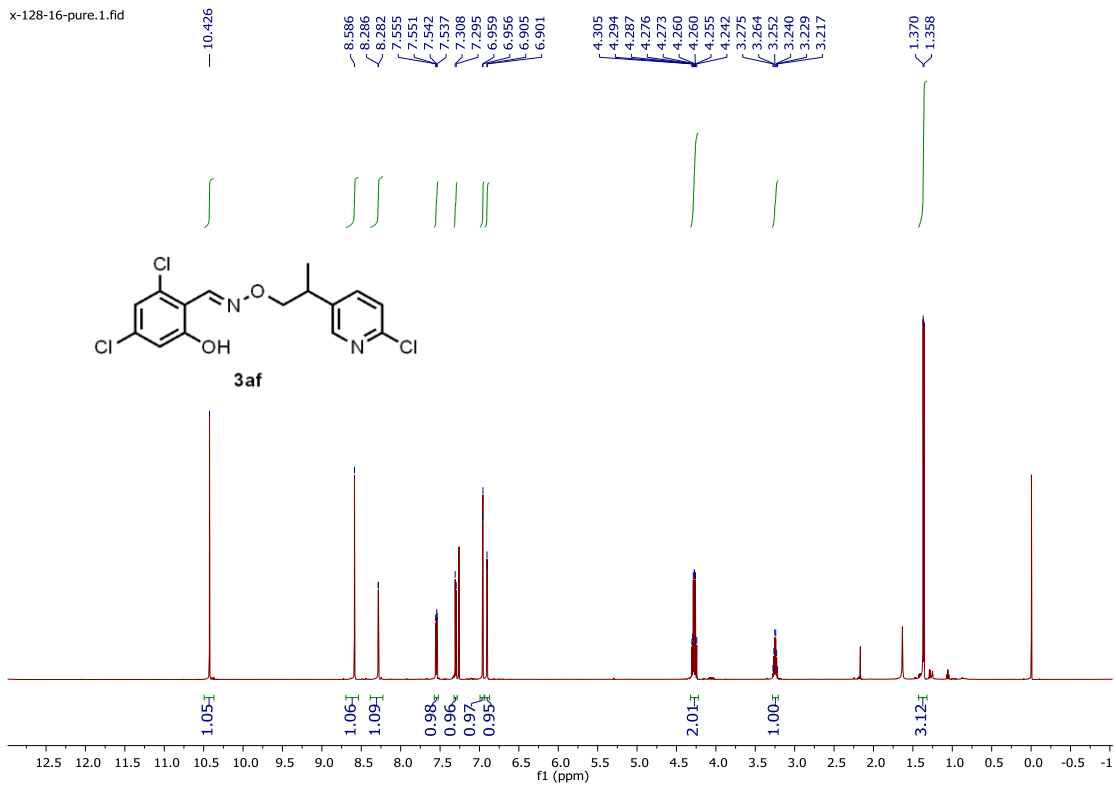
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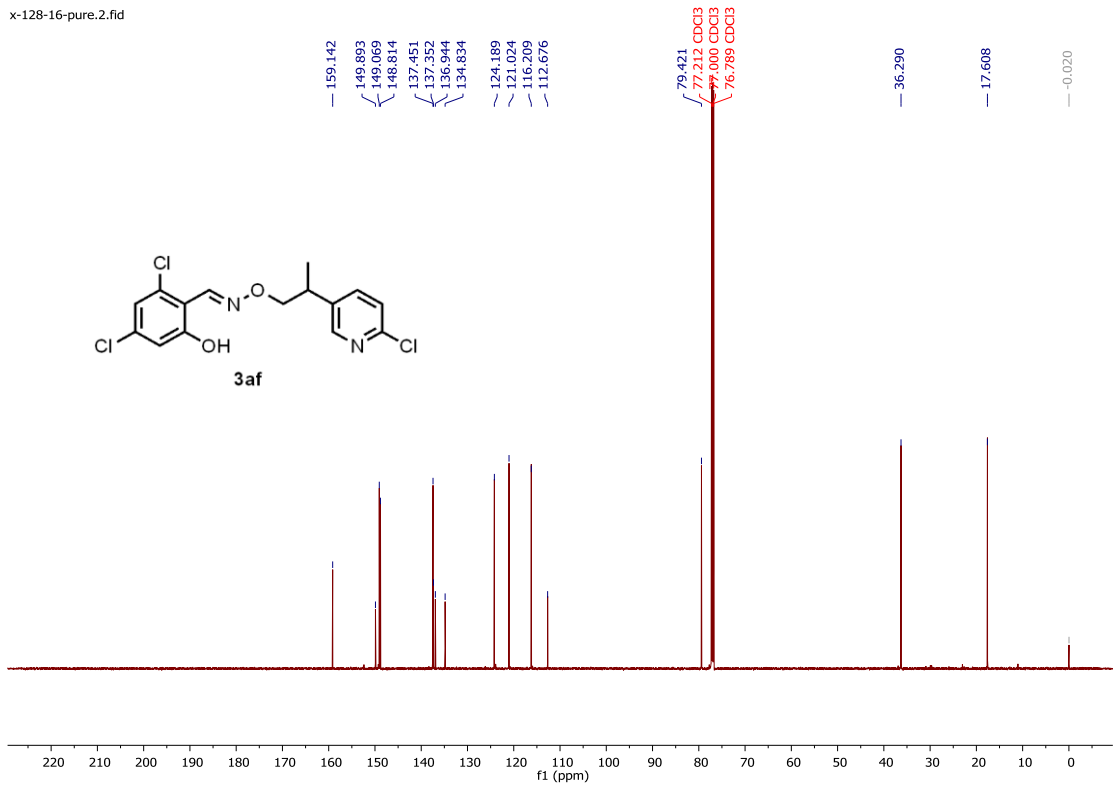
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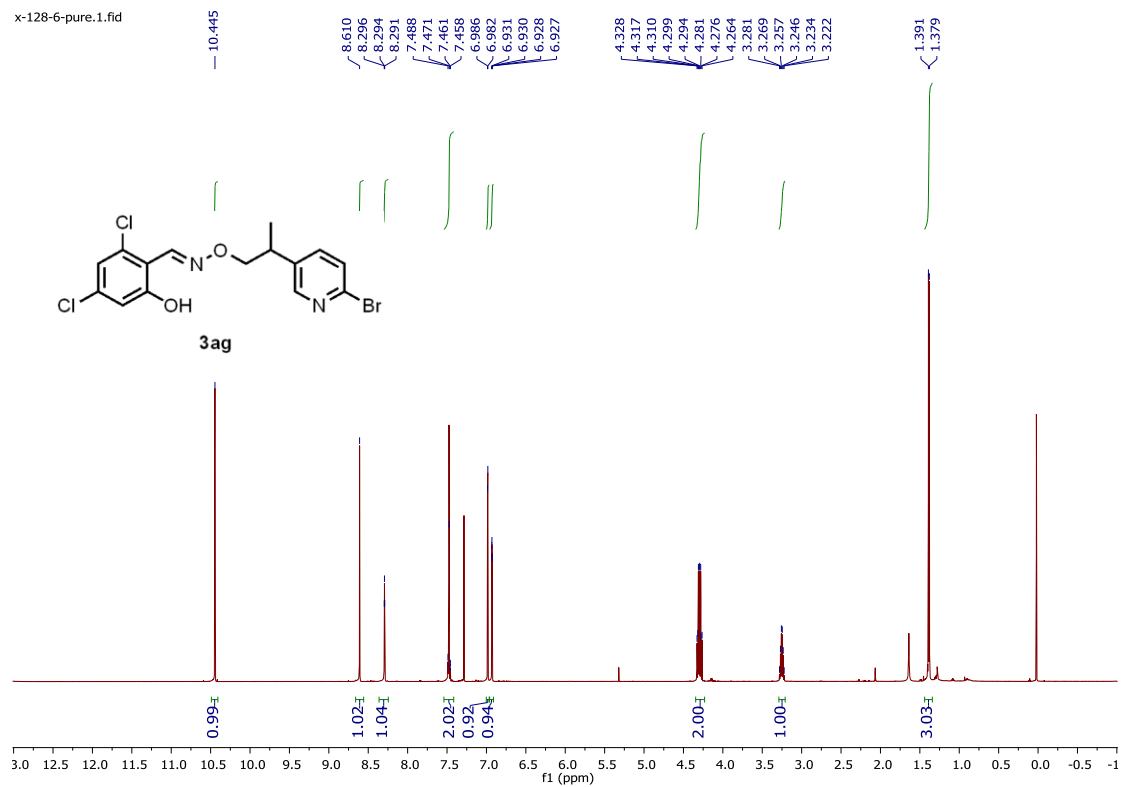
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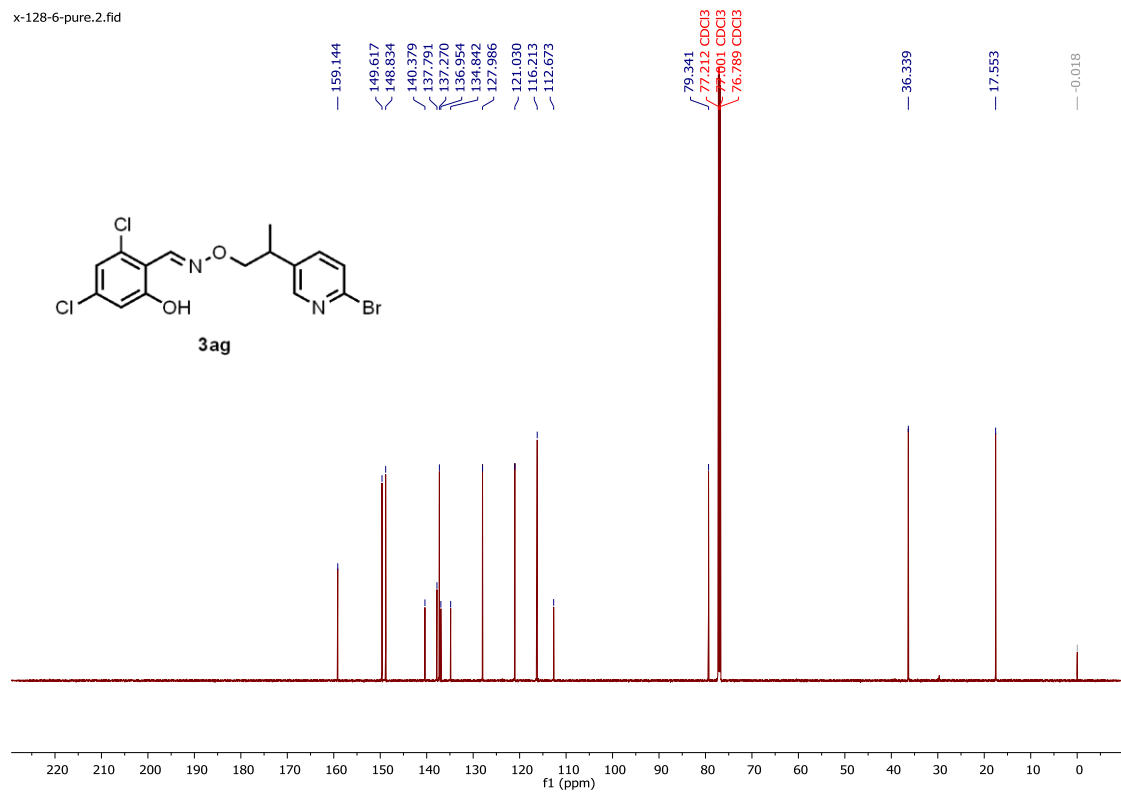
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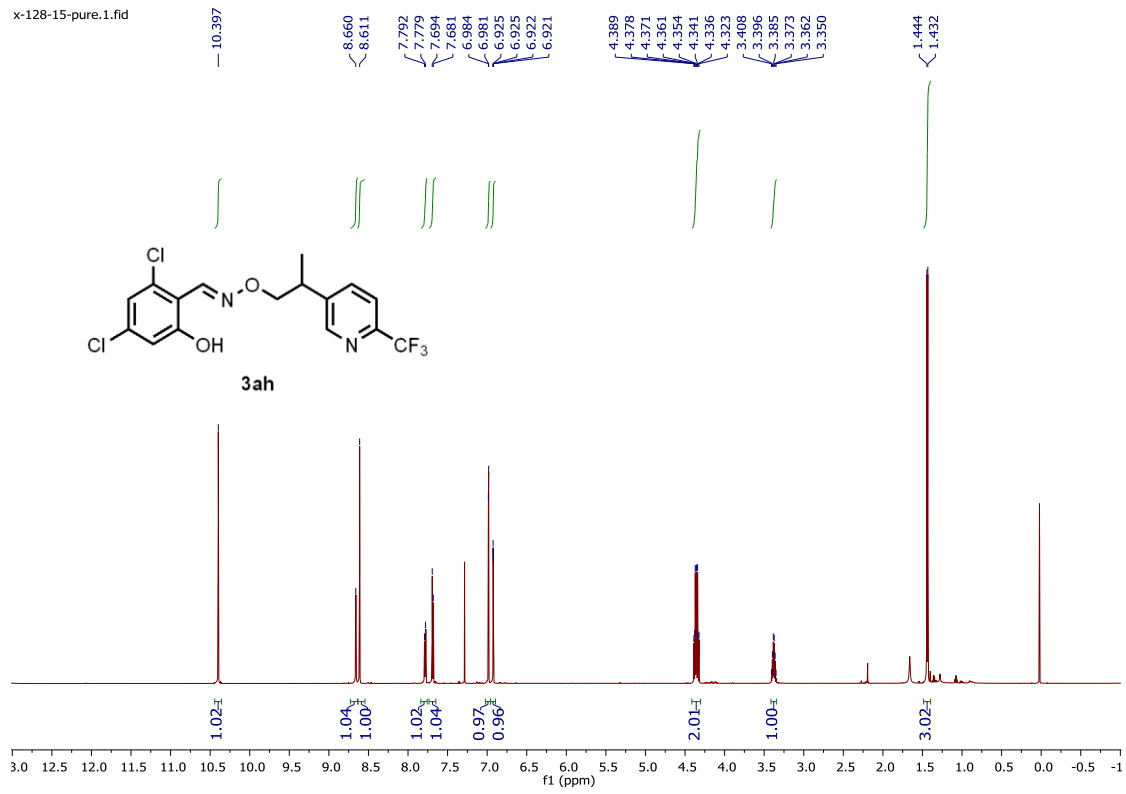
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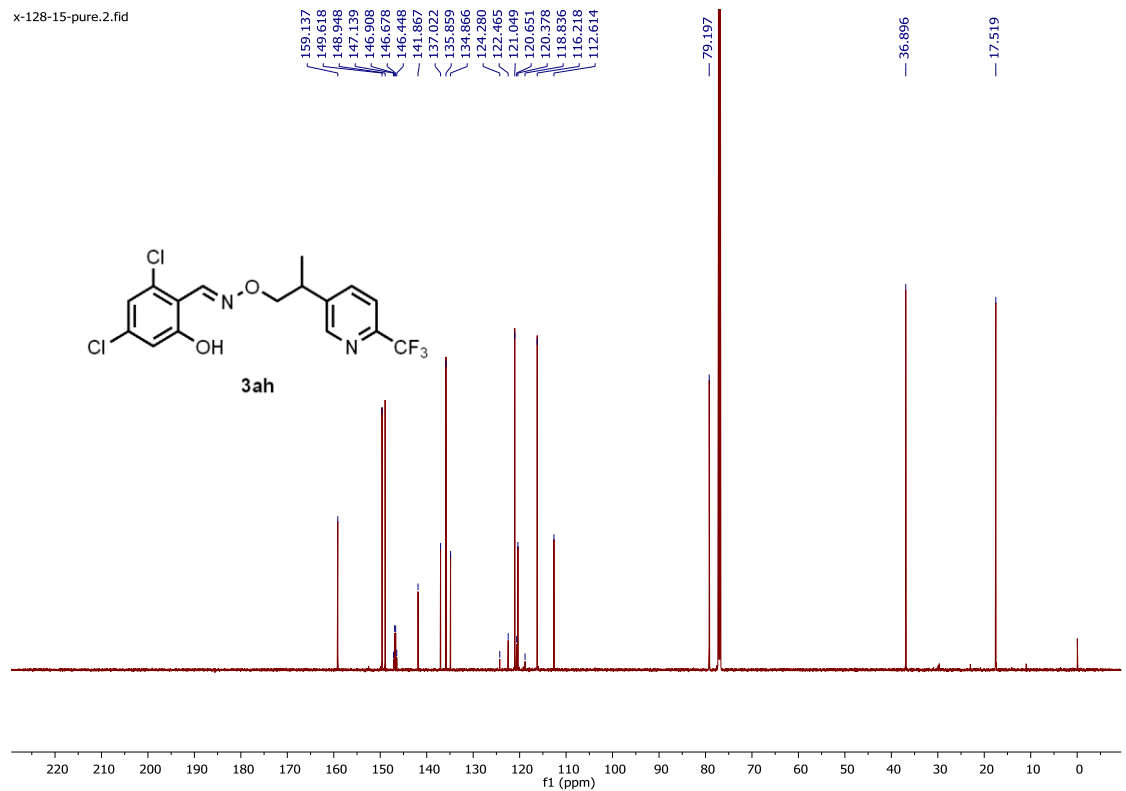
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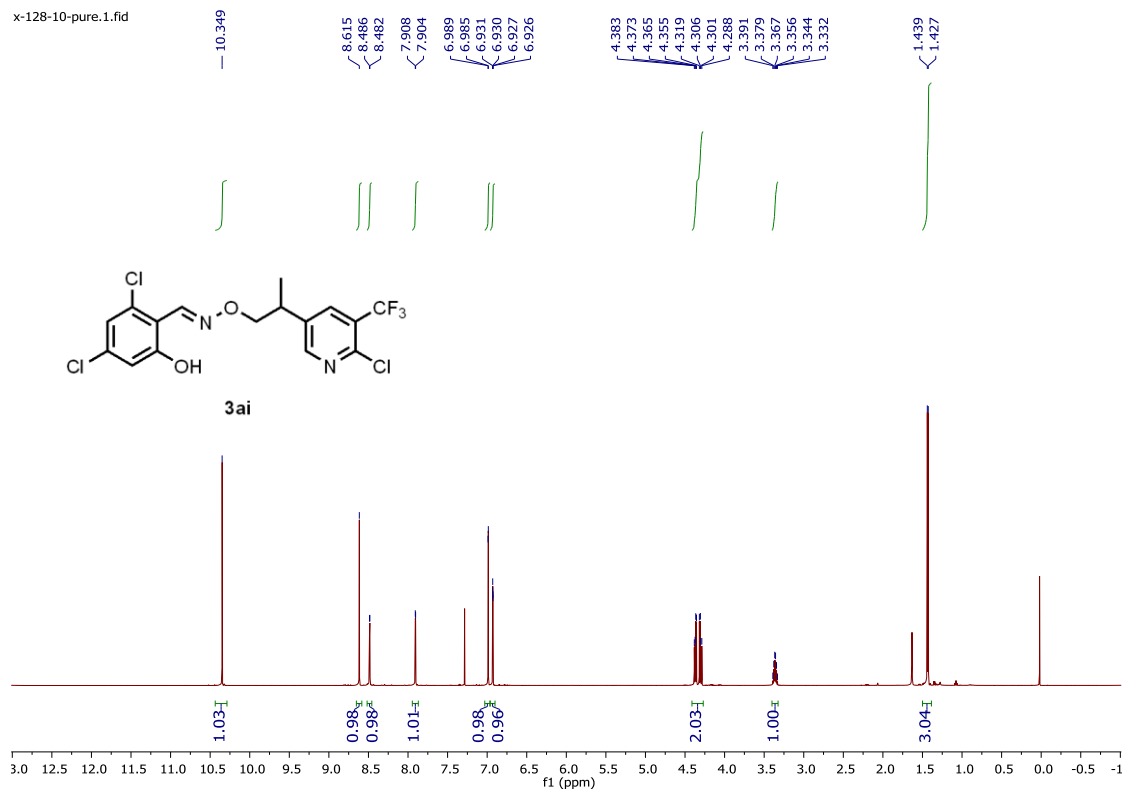
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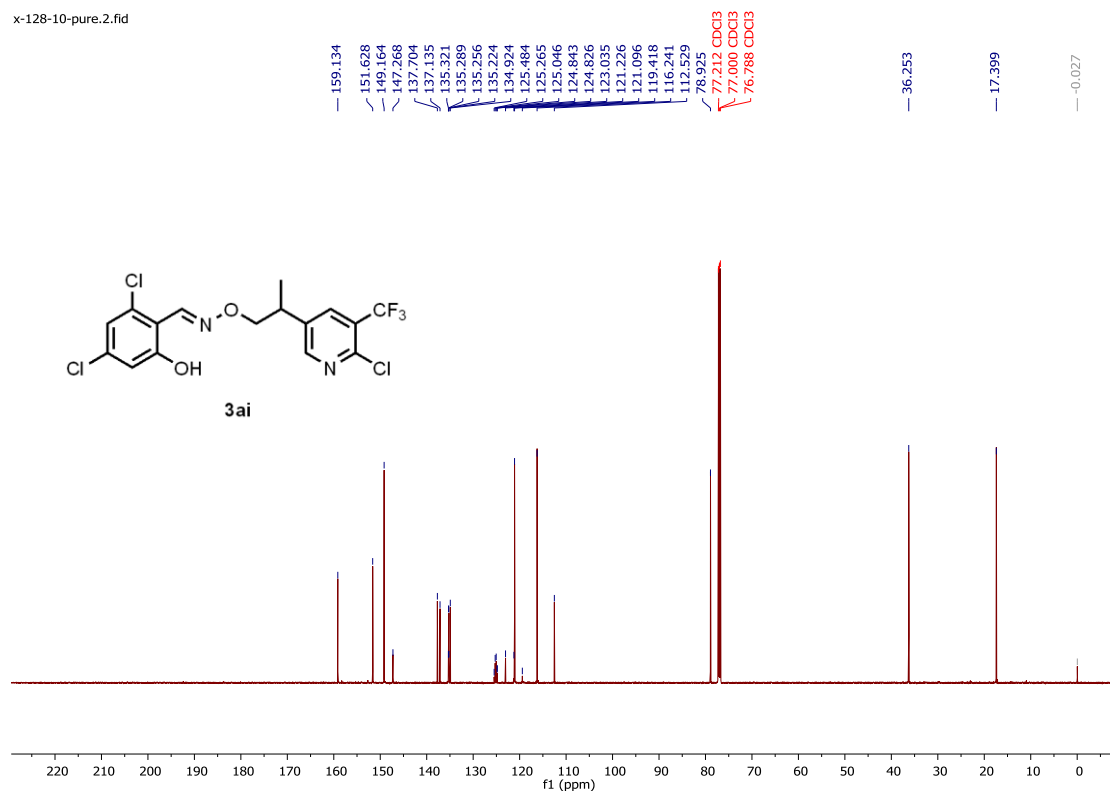
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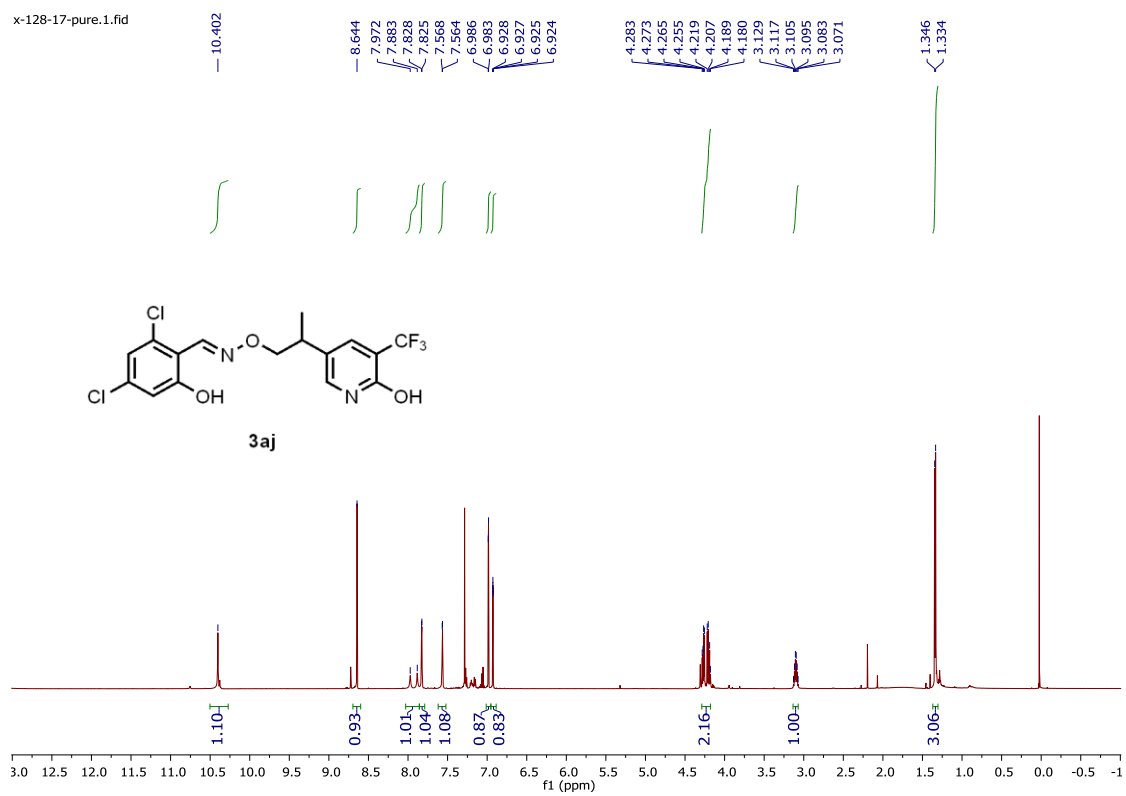
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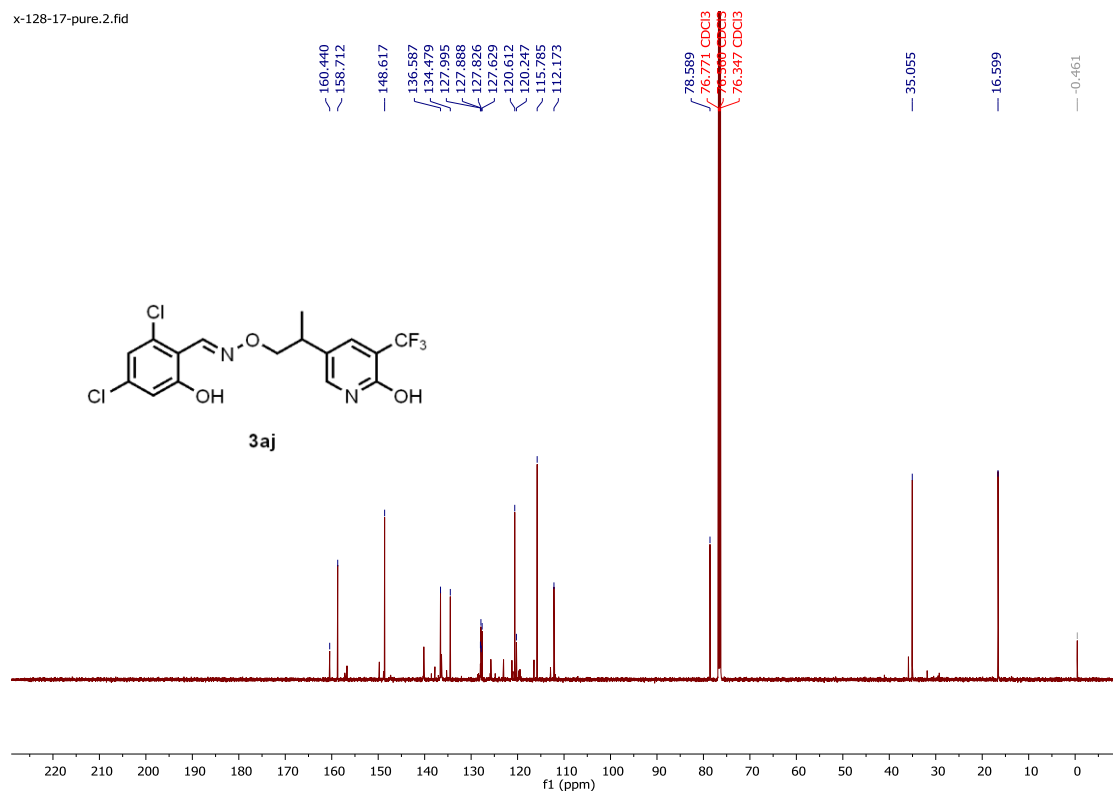
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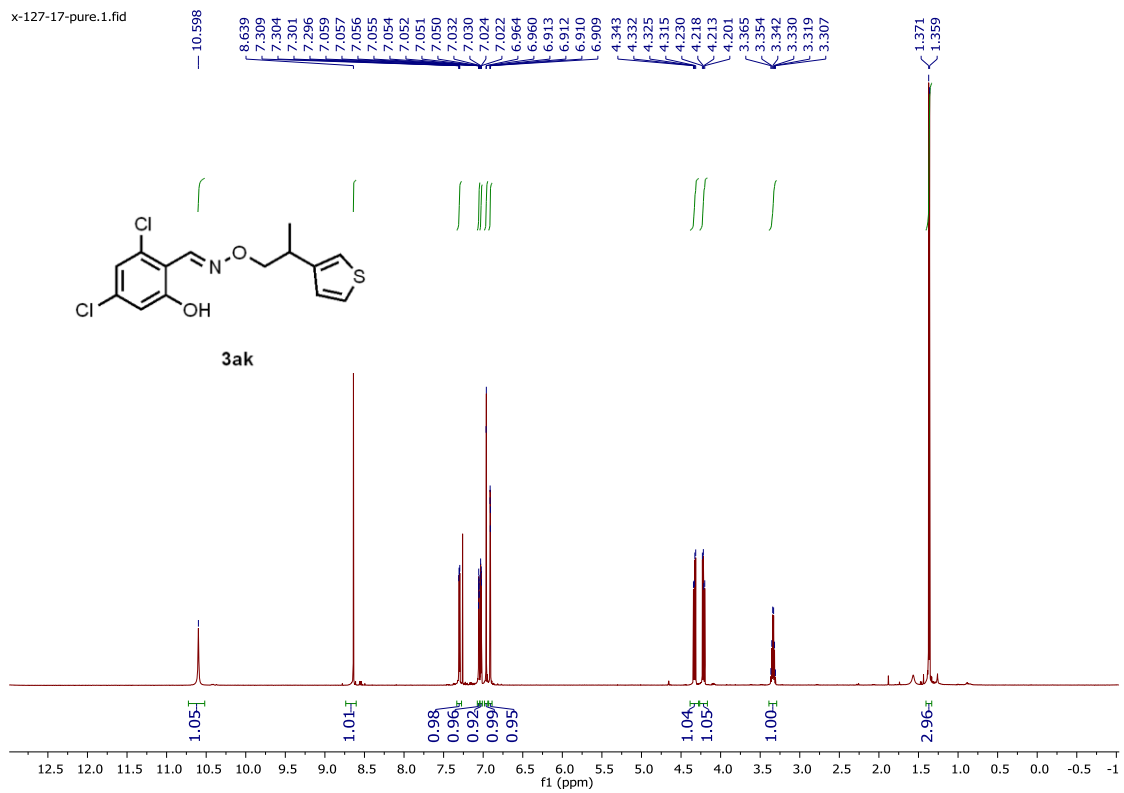
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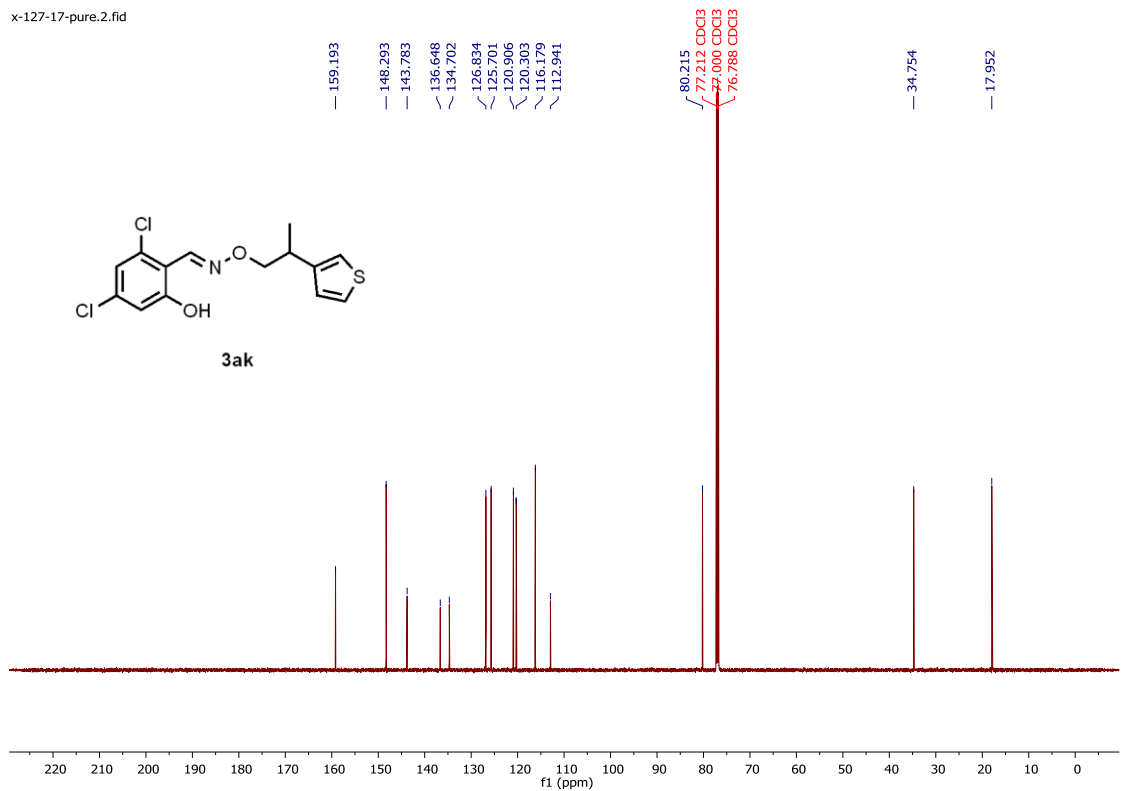
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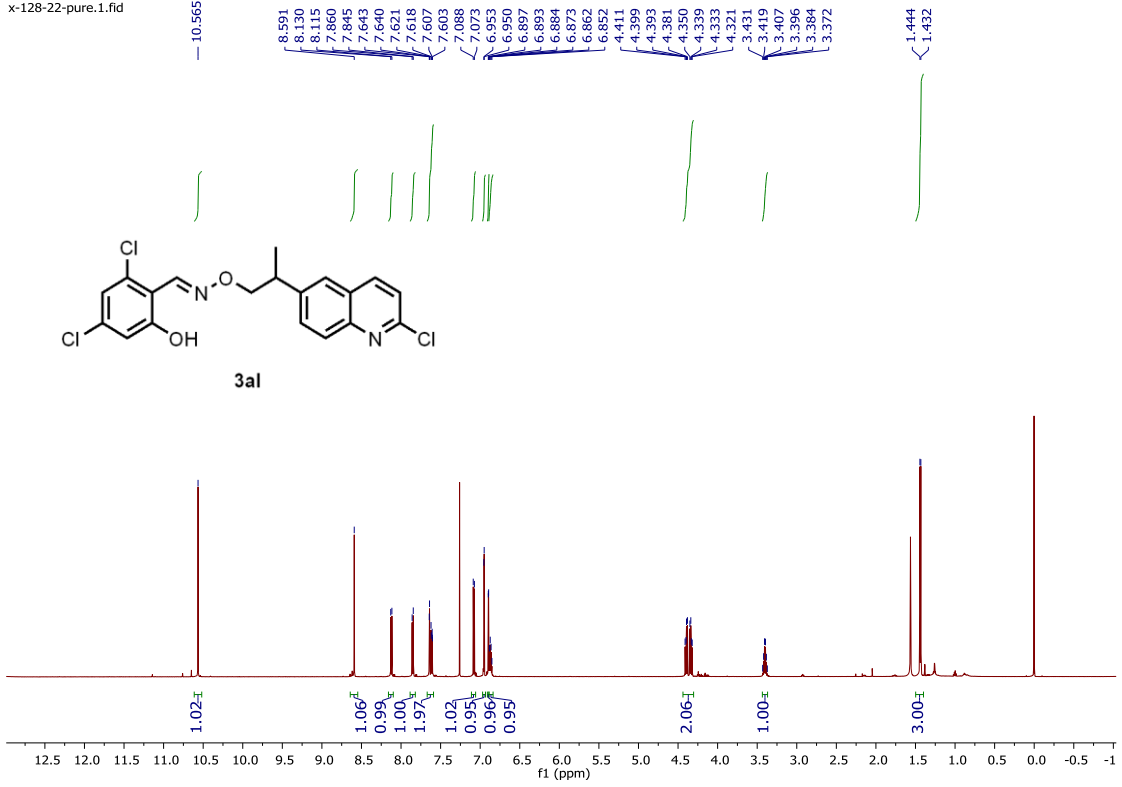
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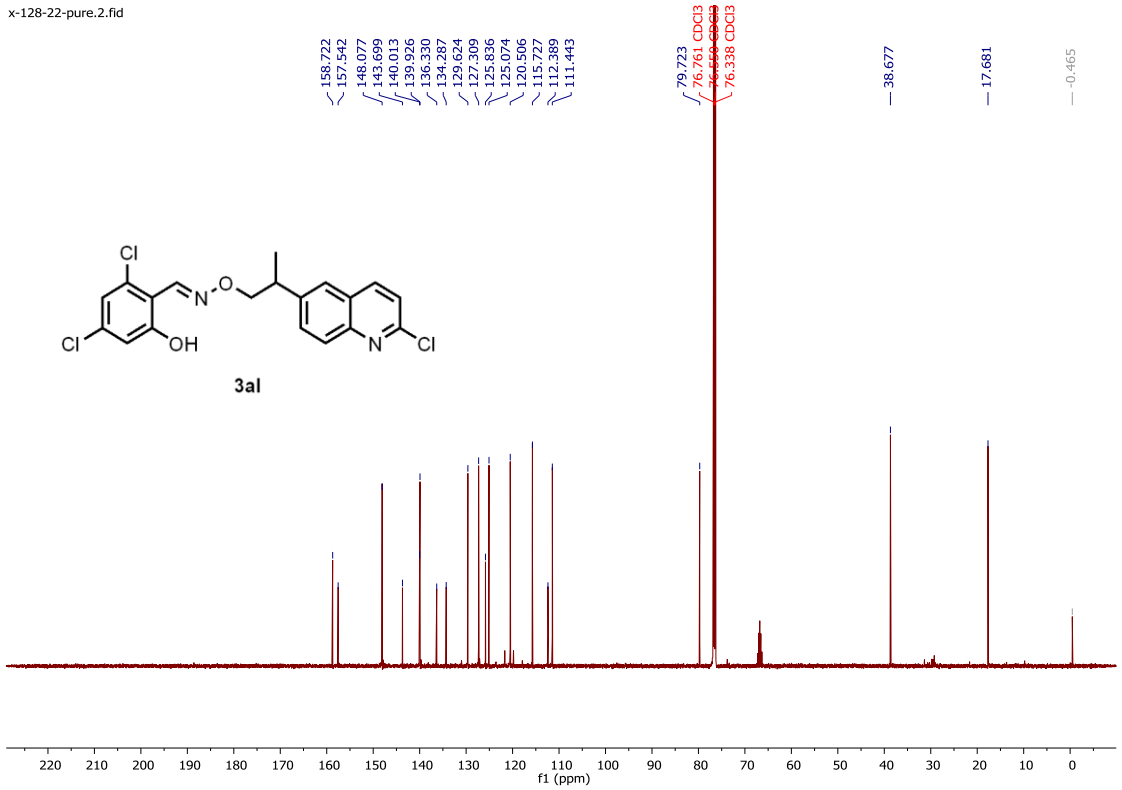
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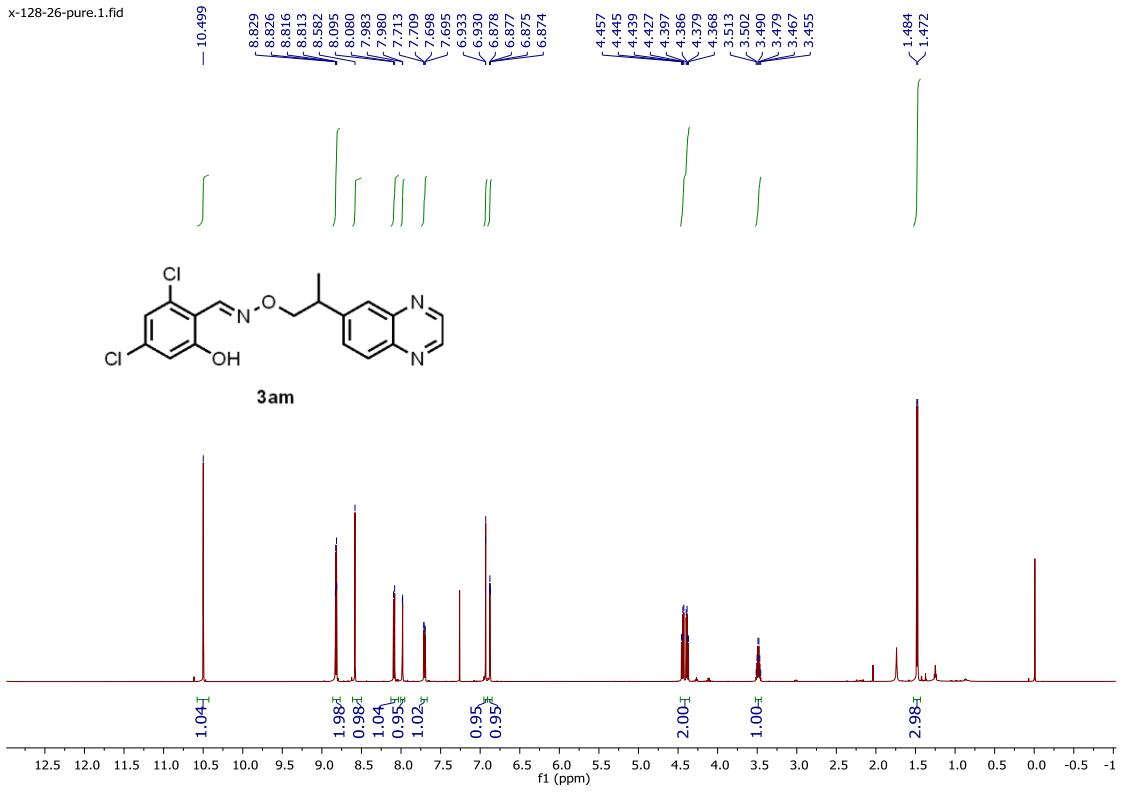
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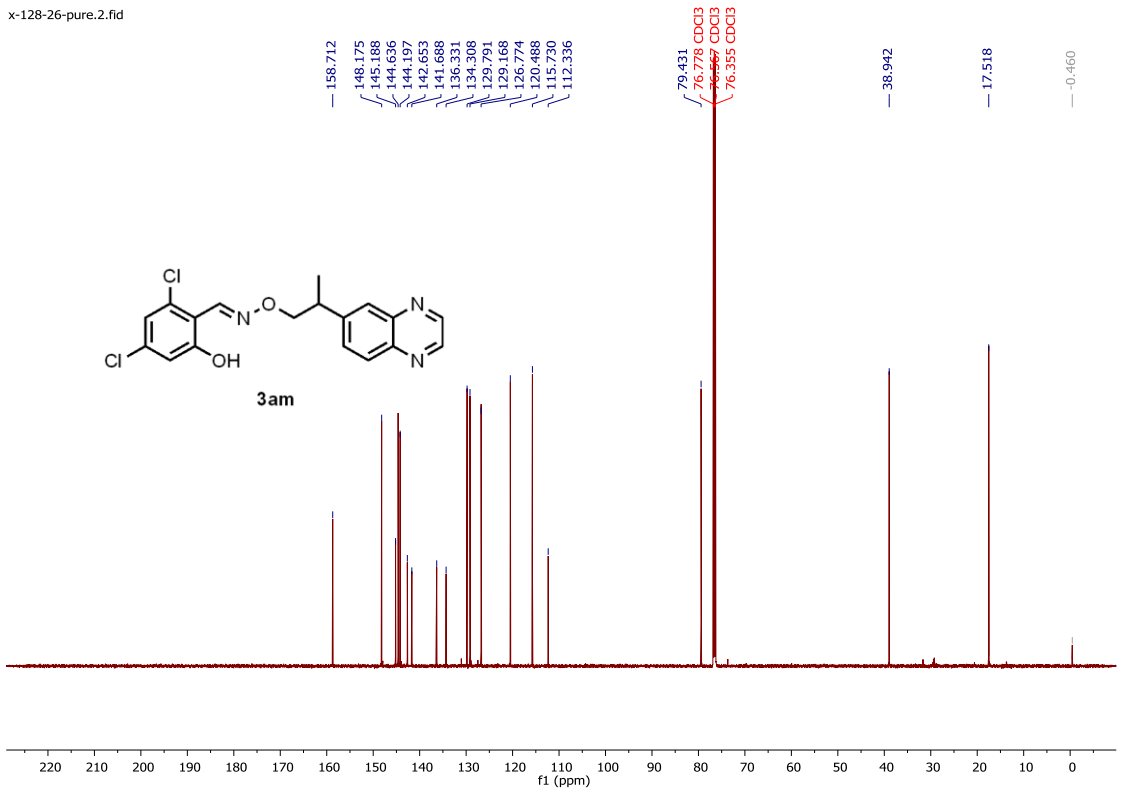
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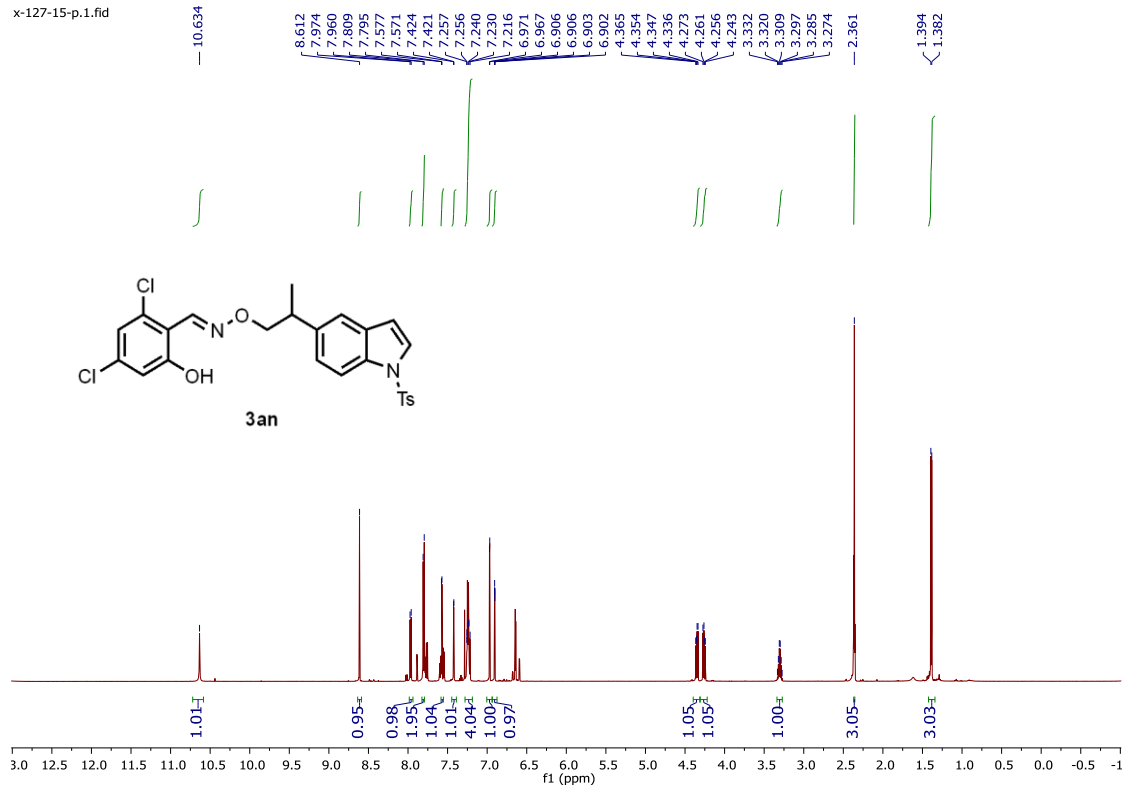
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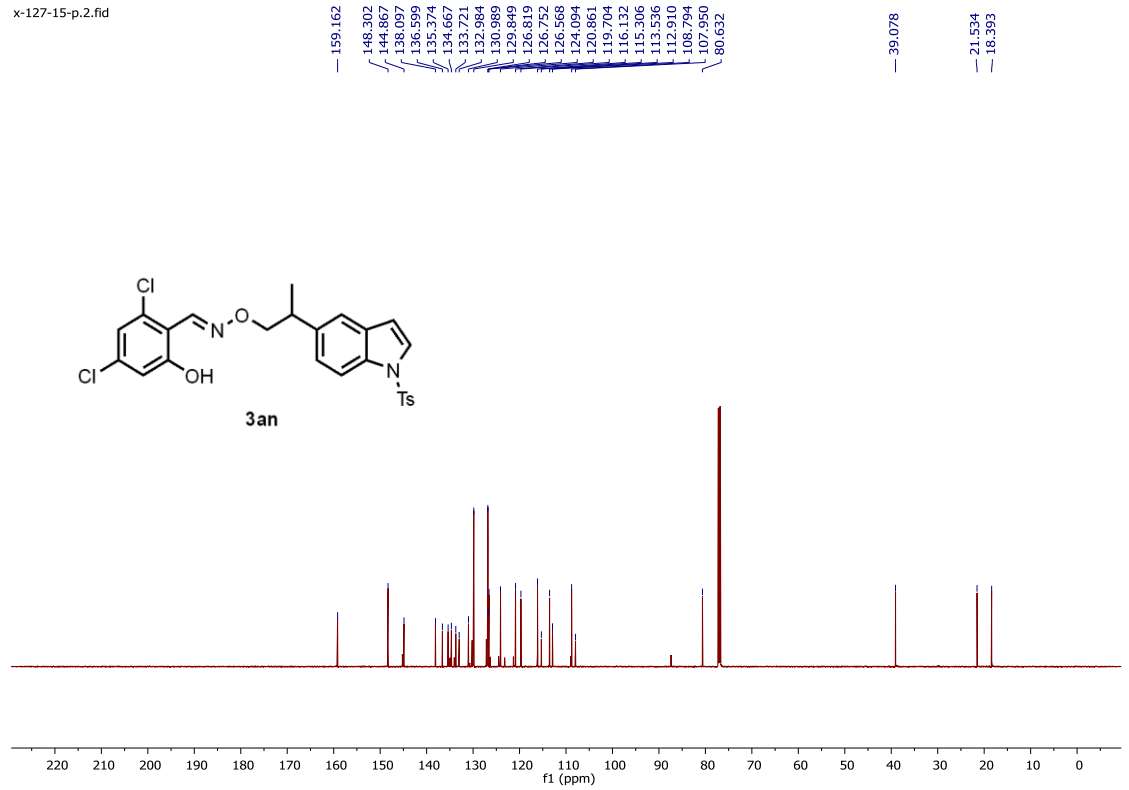
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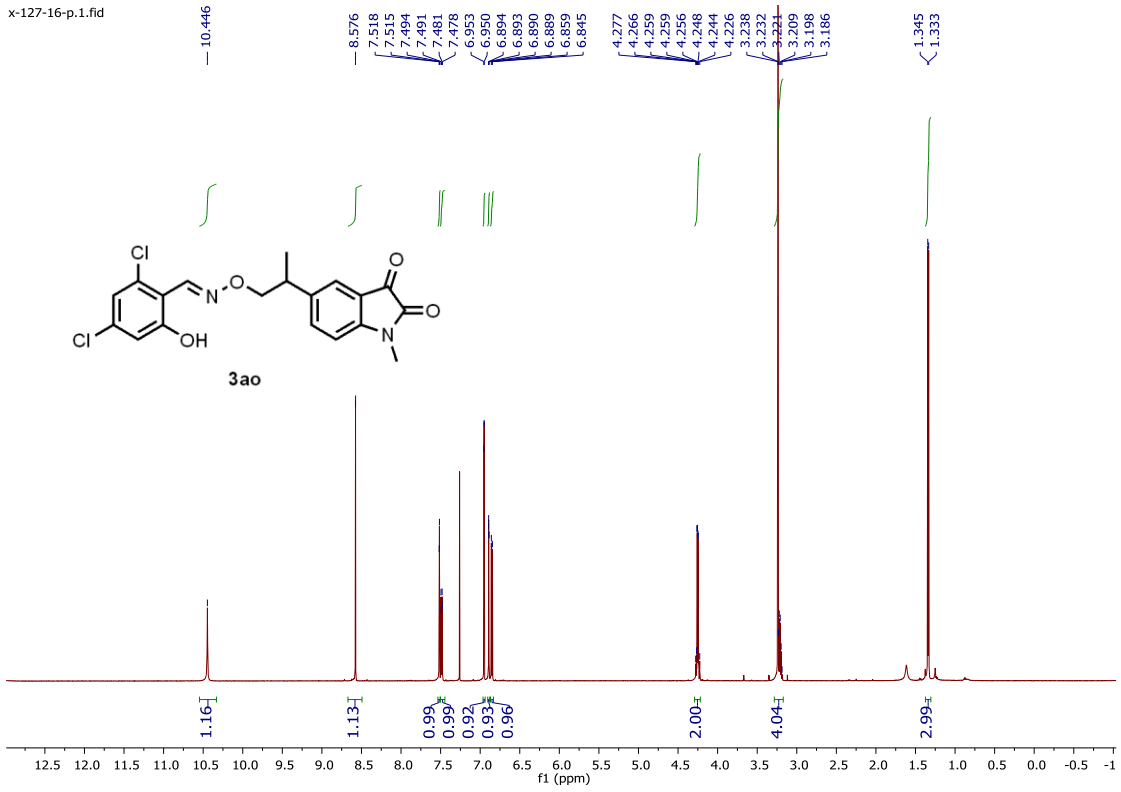
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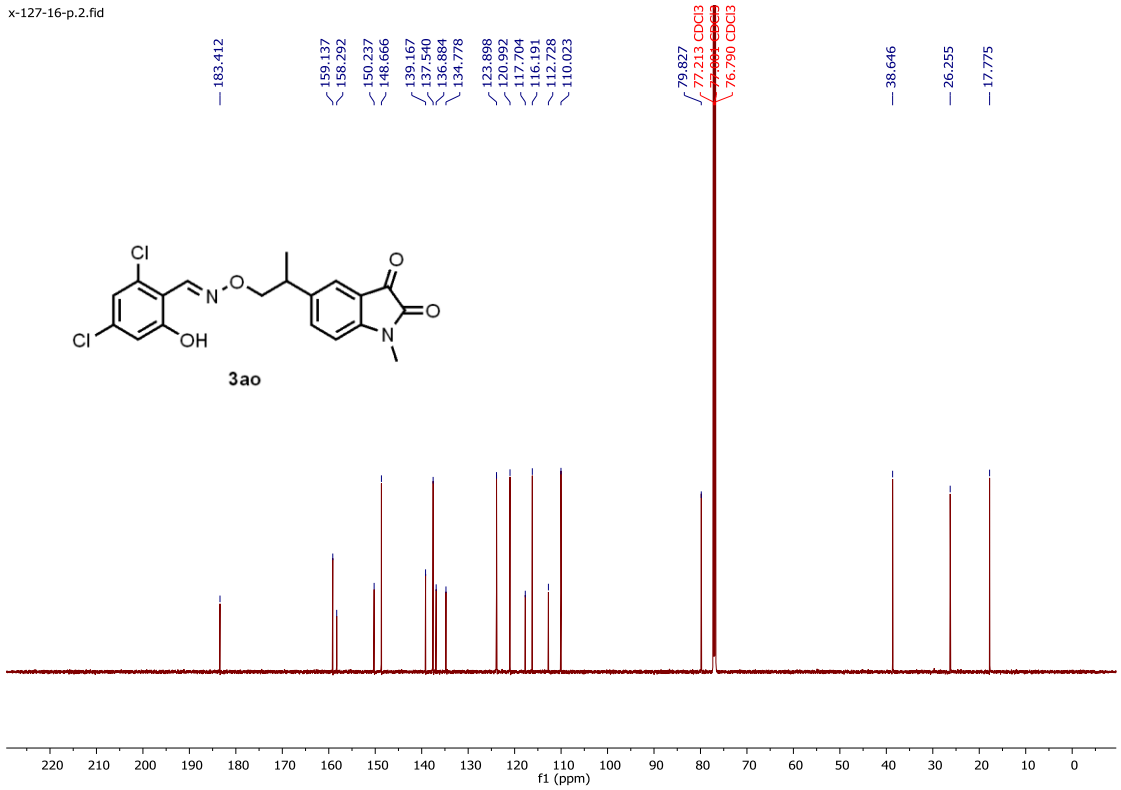
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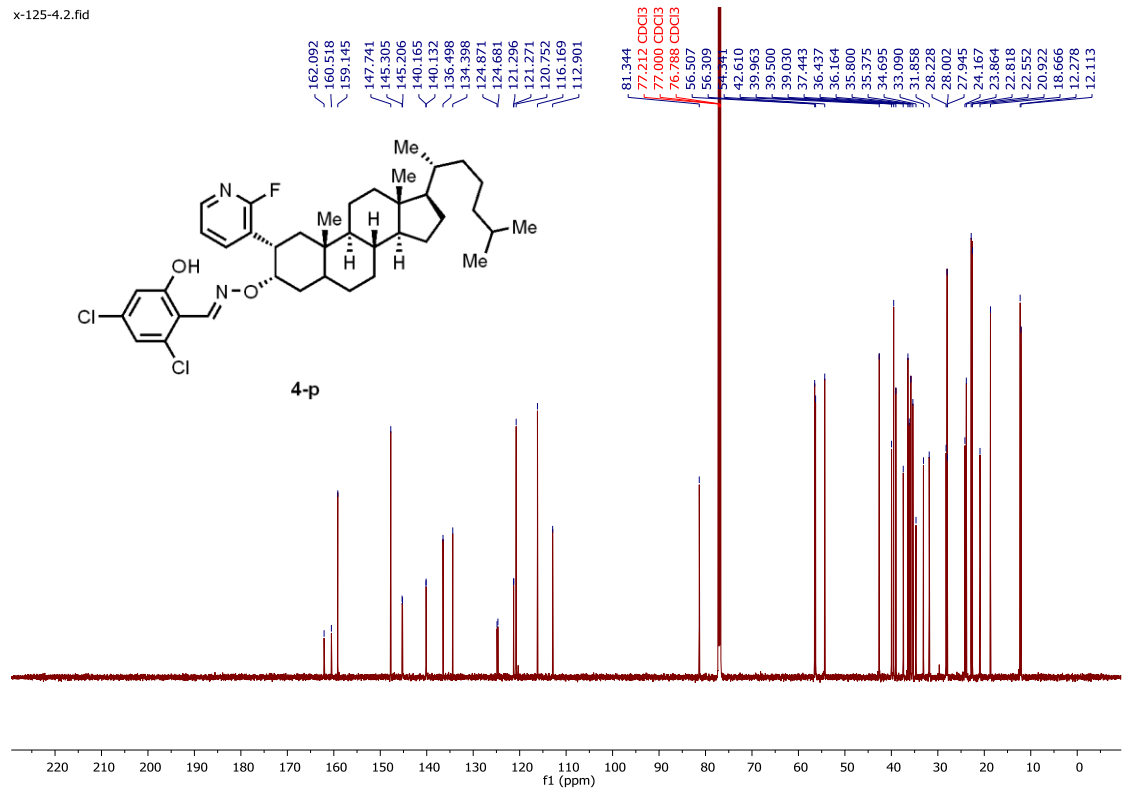
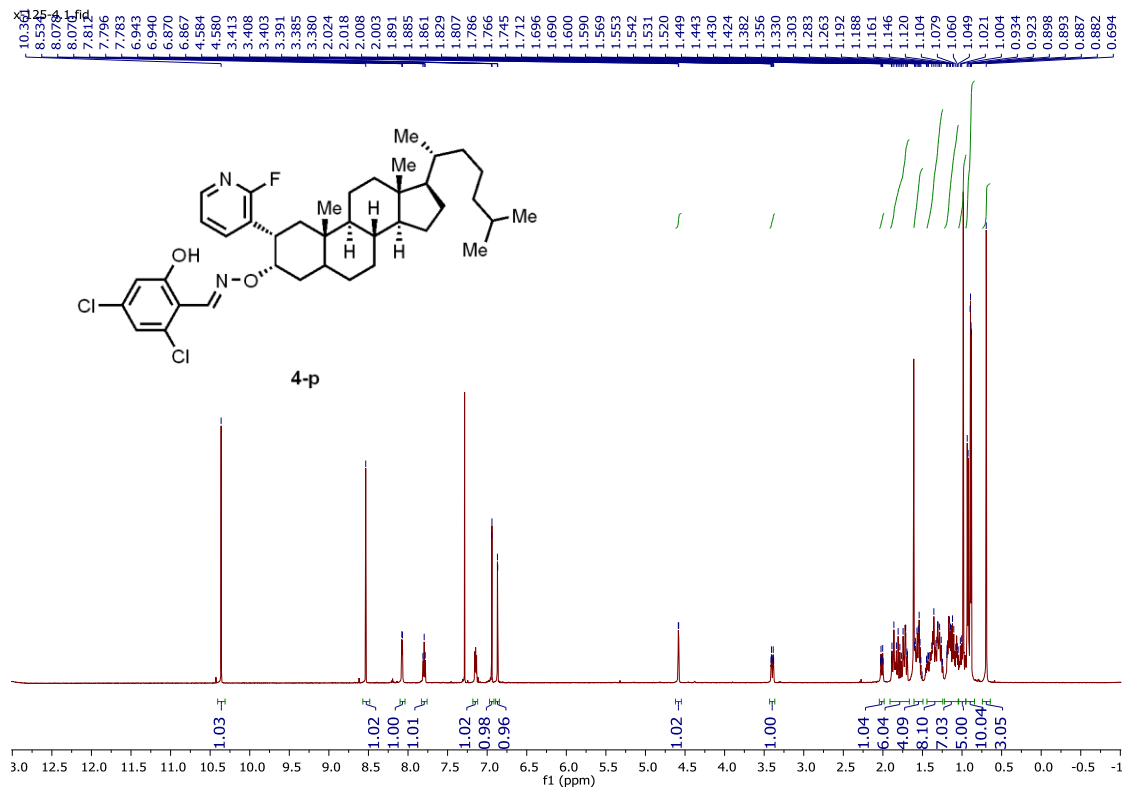


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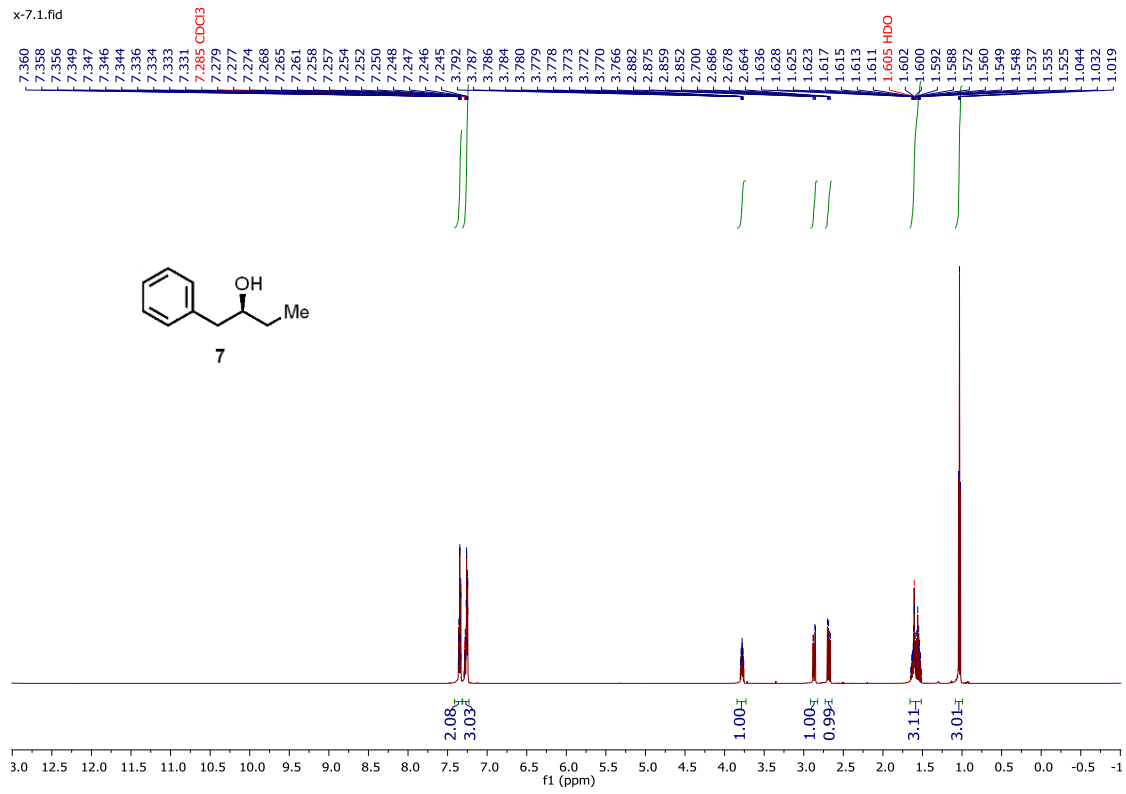


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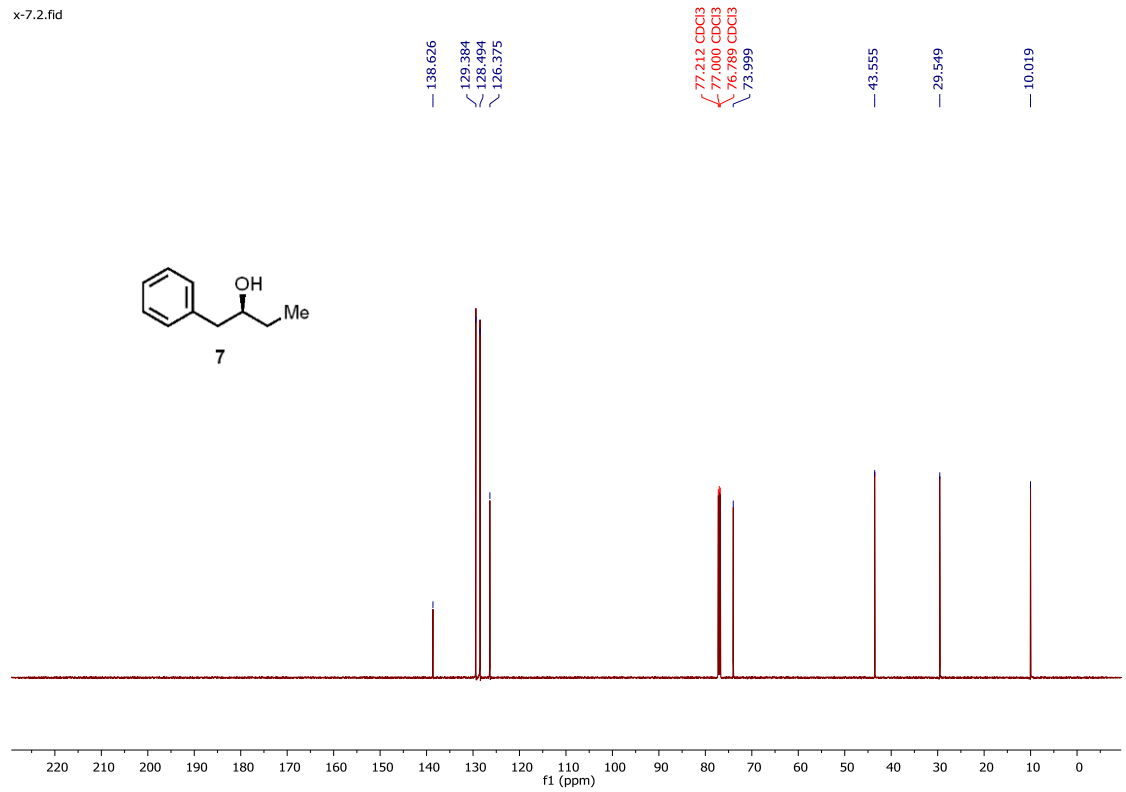




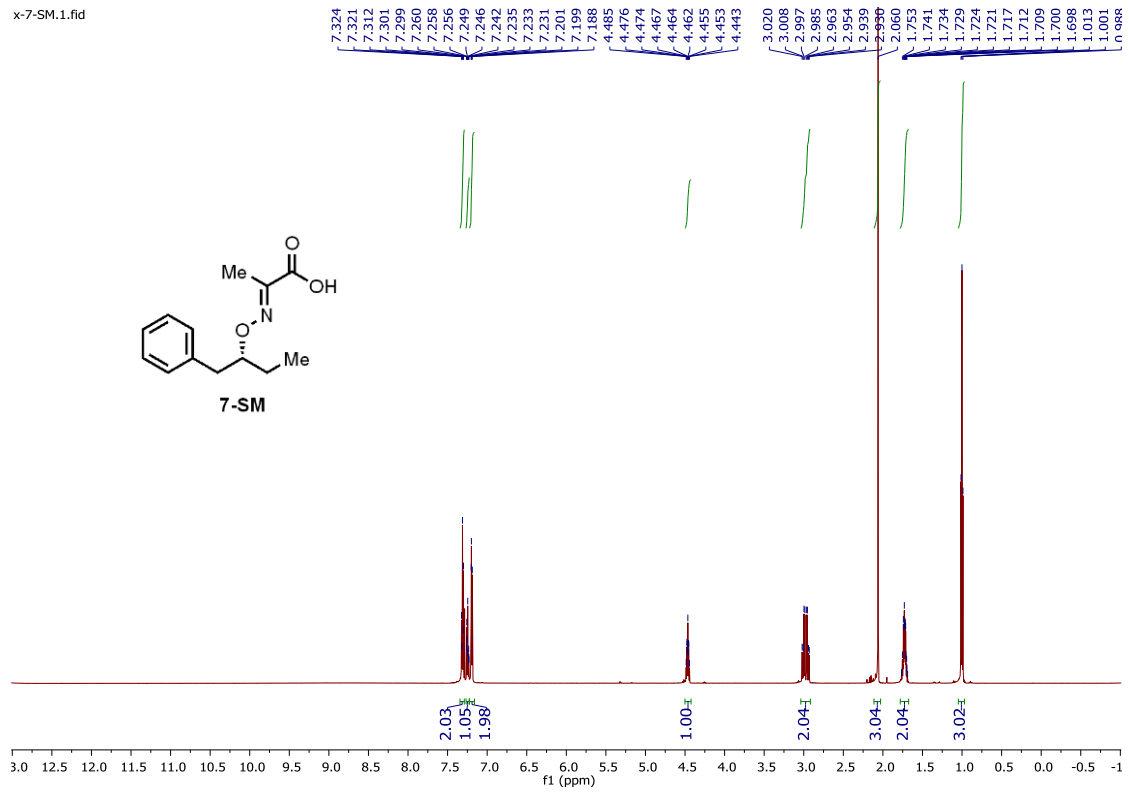
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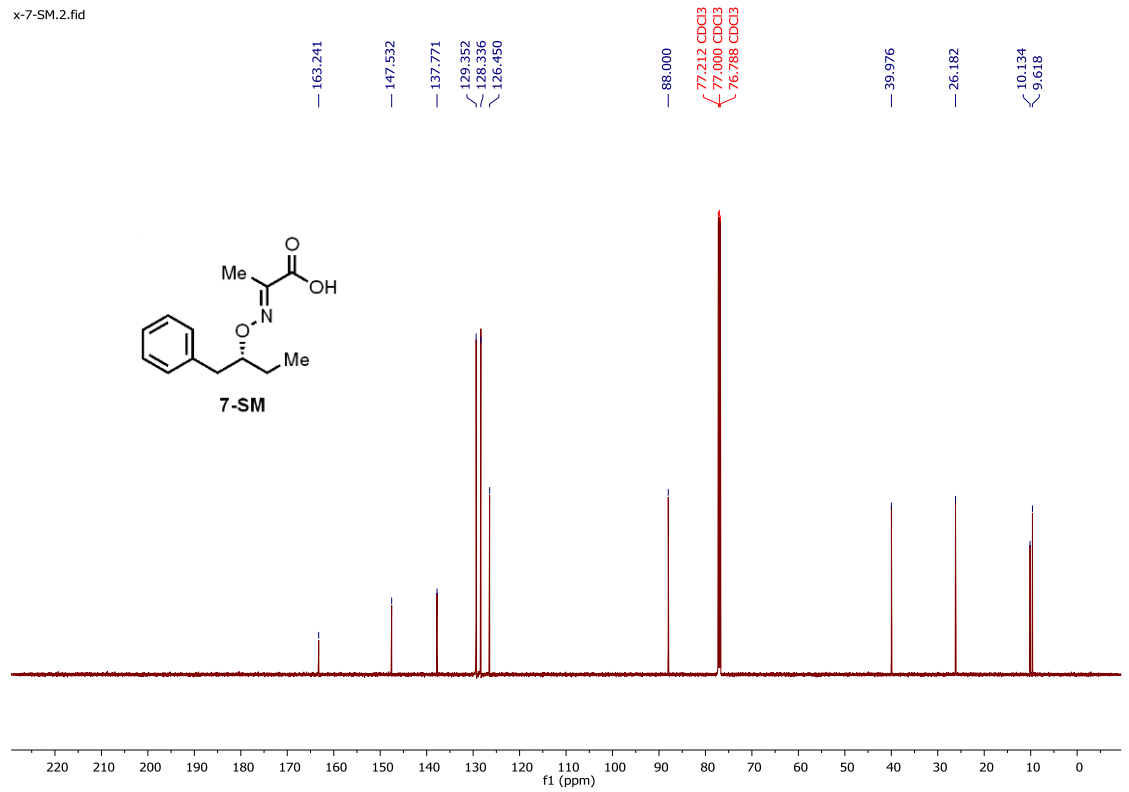
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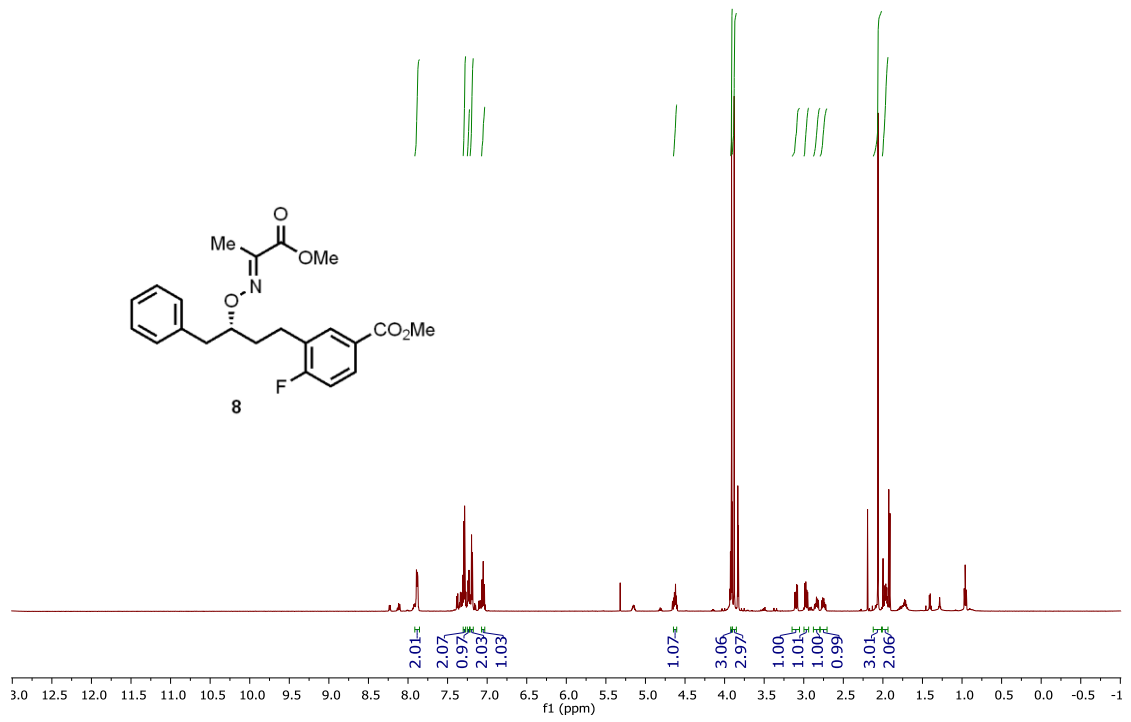
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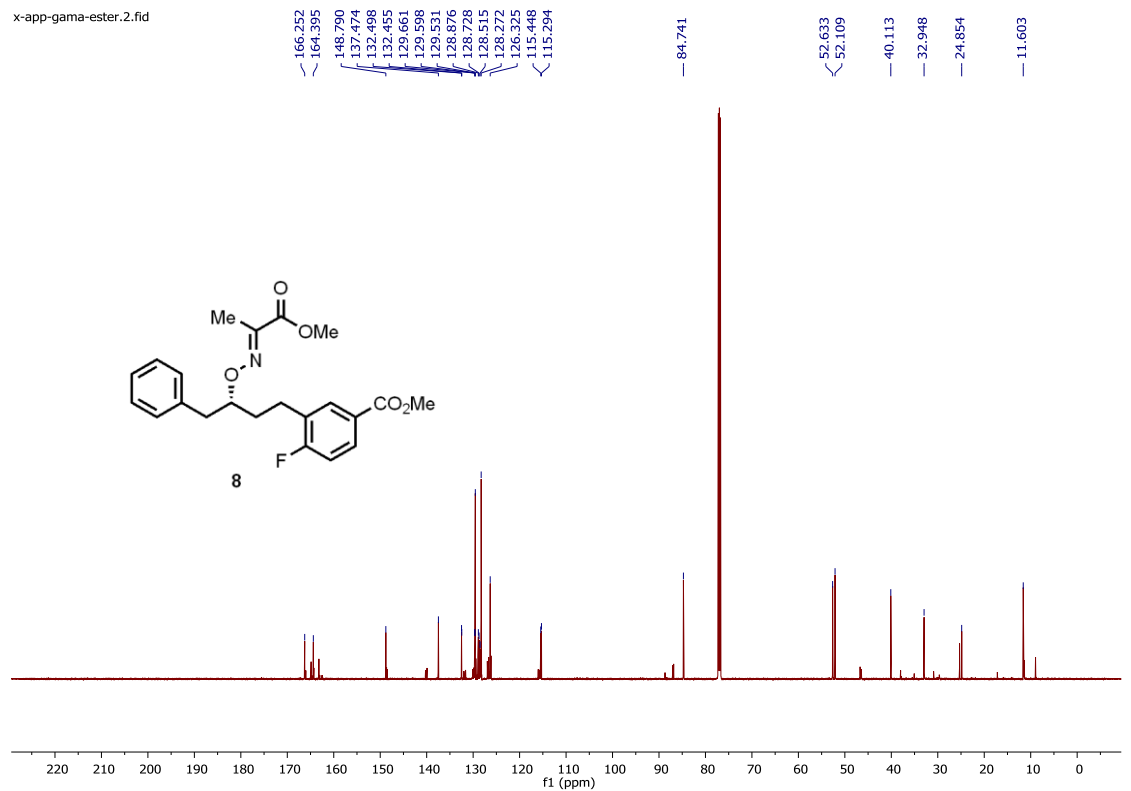
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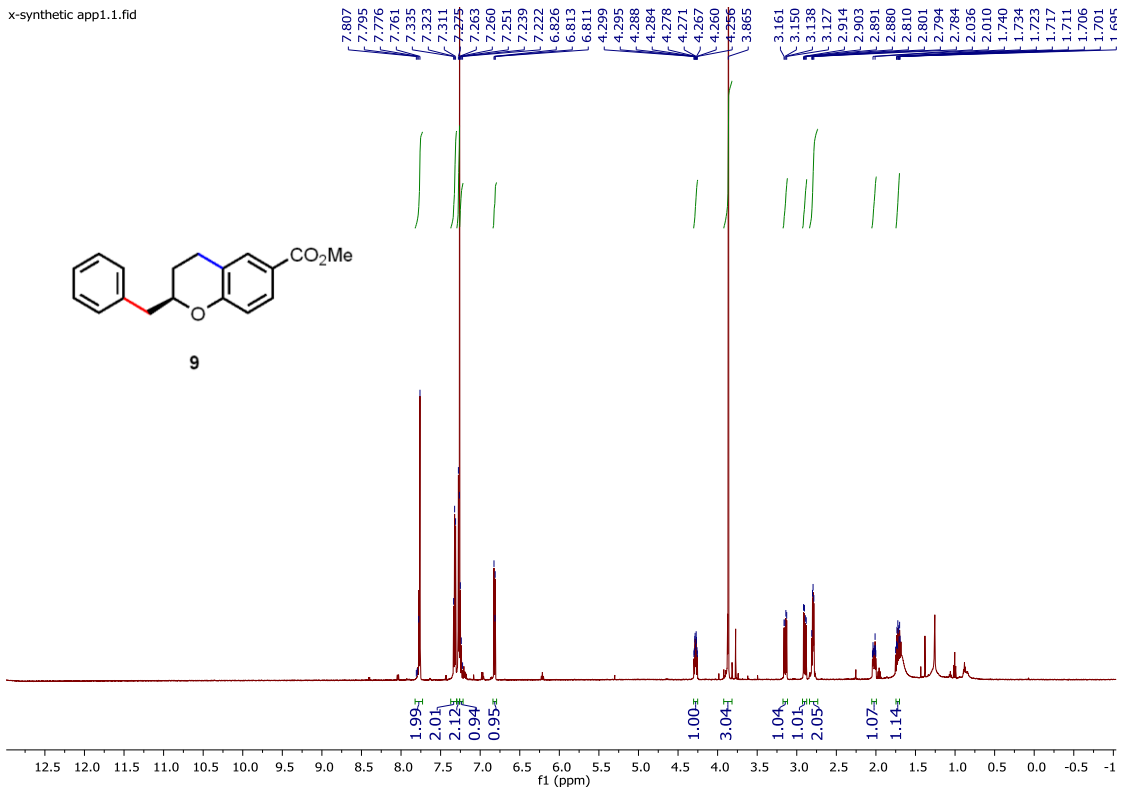
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x-app-gama-ester.2.fid



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x-synthetic app1.2.fid

