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Electrochemical aminotrideuteromethylthiolation of isocyanides with anilines and CD_3SSO_3Na

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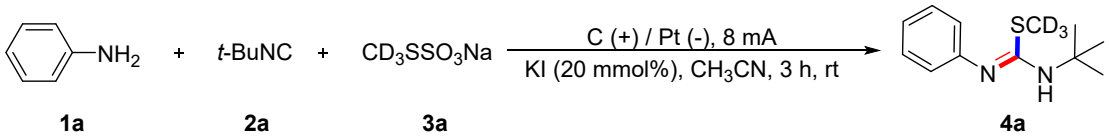
General Information

Anilines and tert-butyl isocyanide were purchased from Energy Chemical Company in China. ^{1}H NMR (500 MHz), ^{13}C NMR (125 MHz) and ^{19}F NMR (470 MHz) spectra were recorded in $CDCl_3$ and DMSO-D6 solutions using a Burker AVANCE 500 spectrometer. High-resolution mass spectra were recorded on an ESI-Q-TOF mass spectrometer. Analysis of crude reaction mixture was done on the Varian 4000 GC/MS and 1200 LC. All reactions were conducted using standard Schlenk techniques. Column chromatography was performed using EM silica gel 60 (300−400 m). Cyclic voltammetry data were measured with a Shanghai Chenhua potentiostat (CHI660E). All the electrochemical synthetic experiments were carried out in Ika stirrer. .

General Experimental Procedures

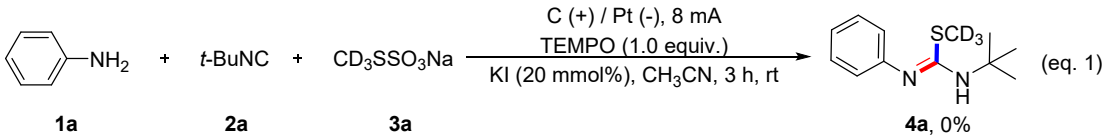
Typical procedure for the preparation of 4a:



1a **2a** **3a** **4a**

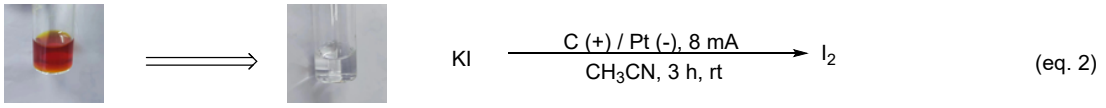
aniline (**1a**, 0.3 mmol), *t*-BuNC (**2a**, 0.3 mmol), CD_3SSO_3Na (**3a**, 0.6 mmol), KI (0.06 mmol) and MeCN (6 mL) were sequentially added to a 15 mL Single neck quartz glass that equipped with a magnetic stirrer bar and sealed with rubber plugs under air atmosphere. A carbon rod (Φ 6 mm) anode and a platinum electrode (10 mm×10 mm×0.3 mm) were used as the cathode in the bottle. About 1.0 cm of the carbon rod and platinum was under the solution. The reaction mixture was stirred and electrolyzed at a constant current of 8 mA under air at room temperature for 3 hours. After completion of the reaction, the solution was concentrated in vacuum. The resulting crude mixture was purified by flash column chromatography to give the desired product **4a**.

Mechanistic Studies



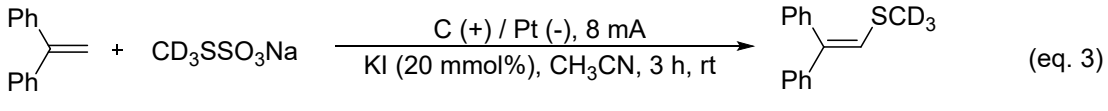
1a **2a** **3a** **4a**, 0% (eq. 1)

aniline (**1a**, 0.3 mmol), *t*-BuNC (**2a**, 0.3 mmol), CD_3SSO_3Na (**3a**, 0.6 mmol), TEMPO (0.3 mmol), KI (0.06 mmol) and MeCN (6 mL) were sequentially added to a 15 mL Single neck quartz glass that equipped with a magnetic stirrer bar and sealed with rubber plugs under air atmosphere. A carbon rod (Φ 6 mm) anode and a platinum electrode (10 mm×10 mm×0.3 mm) were used as the cathode in the bottle. About 1.0 cm of the carbon rod and platinum was under the solution. The reaction mixture was stirred and electrolyzed at a constant current of 8 mA under air at room temperature for 3 hours. After completion of the reaction, and the reaction was filtered through a pad of Celite and diluted with ethyl acetate (10 mL), none of **4a** was detected by GC-MS.



reaction end acetone iodization (eq. 2)

KI (0.06 mmol) and MeCN (6 mL) were sequentially added to a 15 mL Single neck quartz glass that equipped with a magnetic stirrer bar and sealed with rubber plugs under air atmosphere. A carbon rod (Φ 6 mm) anode and a platinum electrode (10 mm×10 mm×0.3 mm) were used as the cathode in the bottle. About 1.0 cm of the carbon rod and platinum was under the solution. The reaction mixture was stirred and electrolyzed at a constant current of 8 mA under air at room temperature for 3 hours. After completion of the reaction, we noticed that the solution turned brown, and then we performed the acetone iodization experiment, and soon the color became colorless. This experimental result confirms that potassium iodide is easily oxidized to iodine.

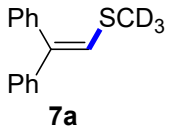


7a, 97% (eq. 3)

1,1-diphenylethylene (**1a**, 0.3 mmol), CD_3SSO_3Na (**3a**, 0.6 mmol), KI (0.06 mmol) and MeCN (6 mL) were sequentially added to a 15 mL Single neck quartz glass that equipped with a magnetic

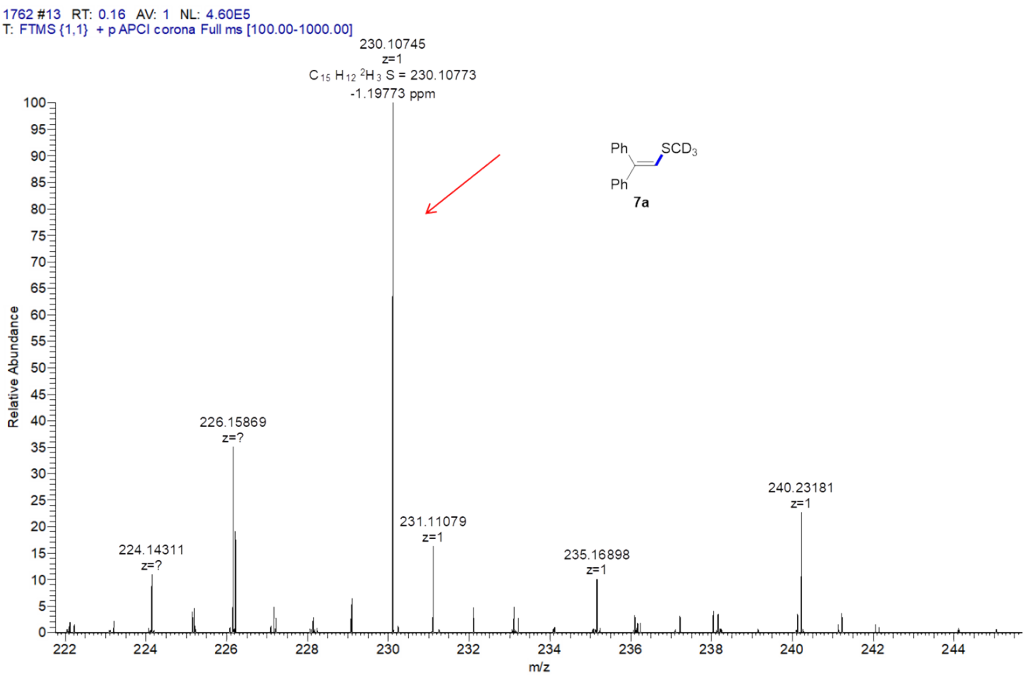
stirrer bar and sealed with rubber plugs under air atmosphere. A carbon rod (Φ 6 mm) anode and a platinum electrode (10 mm×10 mm×0.3 mm) were used as the cathode in the bottle. About 1.0 cm of the carbon rod and platinum was under the solution. The reaction mixture was stirred and electrolyzed at a constant current of 8 mA under air at room temperature for 3 hours. After completion of the reaction, and the reaction was filtered through a pad of Celite and diluted with ethyl acetate (10 mL), **7a** was isolated.

(2,2-diphenylvinyl)(methyl-d3)sulfane



7a

Following the general procedure, using (petroleum ether : EtOAc = 9 : 1) as the eluant afforded a yellow liquid (44.4 mg, 97% yield). **^{1}H NMR** (400 MHz, $CDCl_3$): δ 7.46-7.42 (m, 2H), 7.39-7.25 (m, 8H), 6.60 (s, 1H). **^{13}C NMR** (100 MHz, DMSO-d_6): δ 141.83, 139.58, 138.50, 129.78, 128.41, 128.33, 127.60, 127.06, 126.93. **HRMS** (ESI): calcd for $C_{15}H_{12}D_3S$ $[M + H]^+$ 230.1077, found 230.1074.



Cyclic Voltammetry Studies

The cyclic voltammograms were recorded in an electrolyte of Bu_4NPF_6 (0.1 M) in CH_3CN using a glassy carbon disk working electrode (diameter, 3 mm), a Pt wire auxiliary electrode and a SCE reference electrode. The scan rate is 100 mV/s.

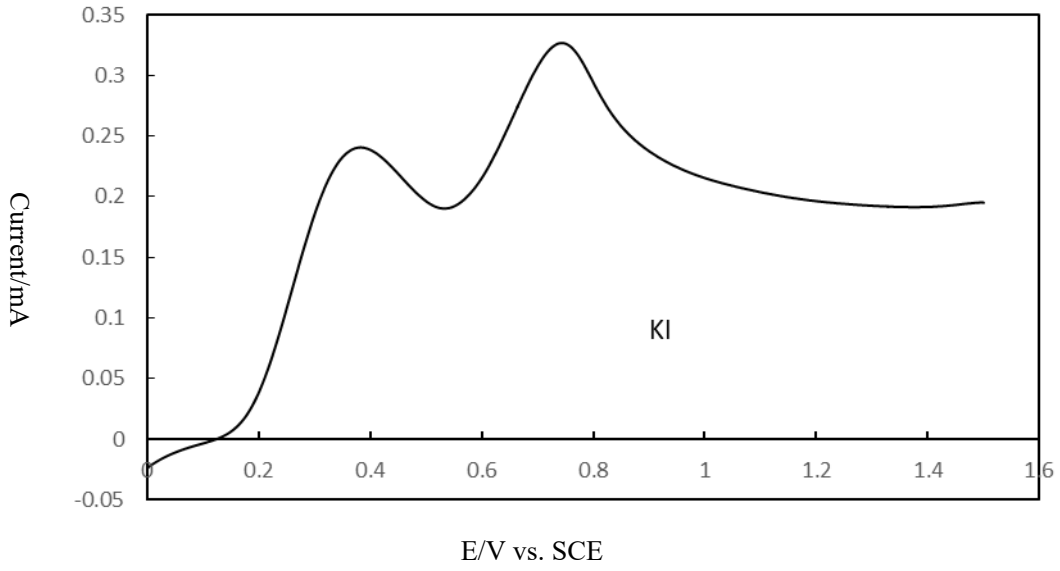


Figure S1:

Figure S1: Cyclic voltammogram of 10 mM KI obtained in CH_3CN containing 0.1 M Bu_4NPF_6 at a 3 mm diameter planar glassy carbon (GC) electrode and at a scan rate of 0.1 V s^{-1} at room temperature. Starting point is 0 v and positive direction of scan.

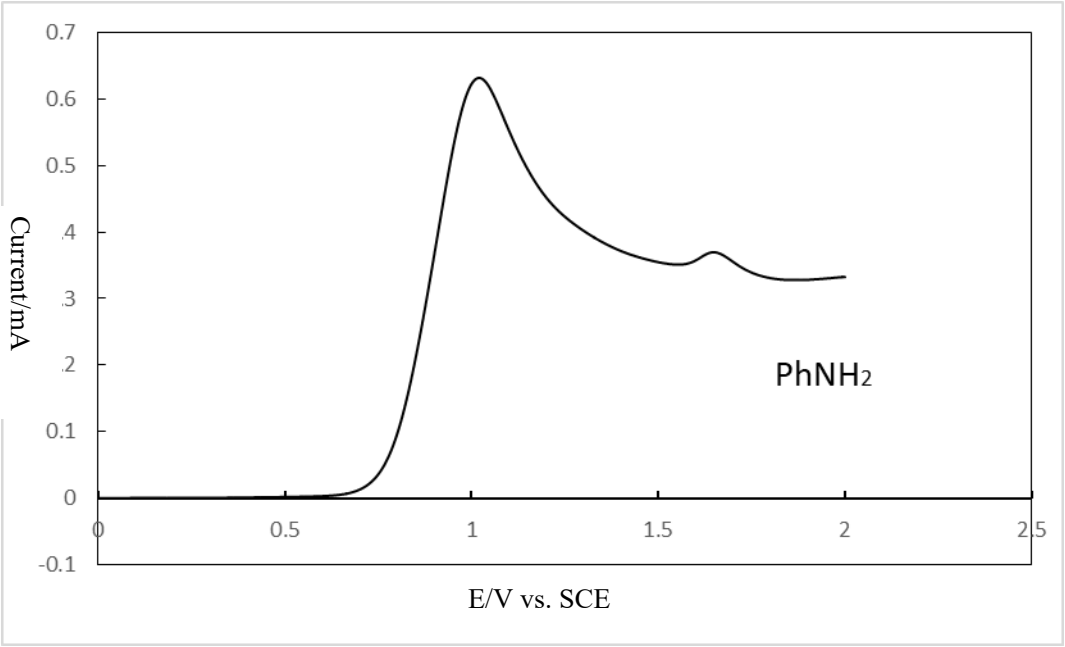


Figure S2;

Figure S2: Cyclic voltammogram of 10 mM $PhNH_2$ obtained in CH_3CN containing 0.1 M Bu_4NPF_6 at a 3 mm diameter planar glassy carbon (GC) electrode and at a scan rate of 0.1 V s^{-1} at room temperature. Starting point is 0 v and positive direction of scan.

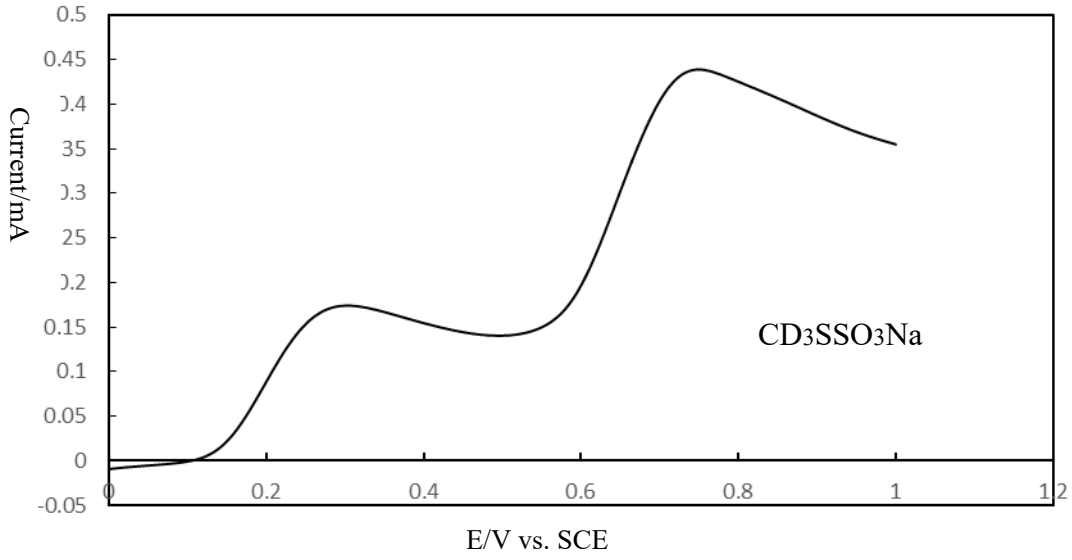
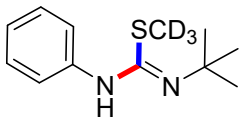


Figure S3

Figure S3: Cyclic voltammogram of 10 mM CD_3SSO_3Na obtained in CH_3CN containing 0.1 M Bu_4NPF_6 at a 3 mm diameter planar glassy carbon (GC) electrode and at a scan rate of 0.1 V s^{-1} at room temperature. Starting point is 0 v and positive direction of scan.

Characterization of Products in Details :

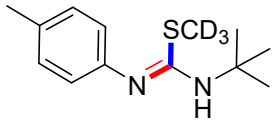
methyl-d3 (Z)-N-(tert-butyl)-N'-phenylcarbamimidothioate



4a

Following the general procedure, using (petroleum ether : EtOAc = 9 : 1) as the eluant afforded a yellow liquid (61.5mg, 91% yield). **^{1}H NMR** (400 MHz, DMSO-d_6) δ 7.23-7.19 (m, 2H), 6.94-6.89 (m, 1H), 6.75-6.73 (m, 2H), 5.74 (s, 1H), 1.41 (s, 9H). **^{13}C NMR** (100 MHz, DMSO-d_6): δ 150.71, 150.20, 128.98, 122.35, 121.94, 52.97, 28.91. **HRMS** (ESI): calcd for $C_{12}H_{16}D_3N_2S$ $[M + H]^+$ 226.1457, found 226.1458.

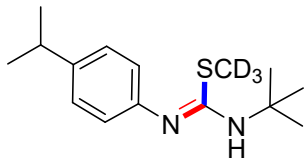
methyl-d3 (Z)-N-(tert-butyl)-N'-(p-tolyl)carbamimidothioate



4b

Following the general procedure, using (petroleum ether : EtOAc = 9 : 1) as the eluant afforded a yellow liquid (66.0 mg, 92% yield). **^{1}H NMR** (400 MHz, Chloroform-*d*) δ 7.30 (s, 1H), 7.10 (d, *J* = 8.0 Hz, 2H), 6.81 (d, *J* = 8.2 Hz, 2H), 2.33 (s, 3H), 1.47 (s, 9H). **^{13}C NMR** (100 MHz, DMSO-d_6): δ 150.09, 148.19, 130.57, 129.49, 122.18, 52.91, 28.92, 20.94. **HRMS** (ESI): calcd for $C_{13}H_{18}D_3N_2S$ $[M + H]^+$ 240.1614, found 240.1623.

methyl-d3 (Z)-N-(tert-butyl)-N'-(4-isopropylphenyl)carbamimidothioate

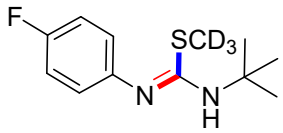


4c

Following the general procedure, using (petroleum ether : EtOAc = 9 : 1) as the eluant afforded a yellow liquid (72.1 mg, 90% yield). **^{1}H NMR** (400 MHz, DMSO-d_6): δ 7.07 (d, *J* = 8.3 Hz, 2H), 6.65 (d, *J* = 8.3 Hz, 2H), 5.65 (s, 1H), 2.82 (p, *J* = 6.9 Hz, 1H), 1.40 (s, 9H), 1.19 (d, *J* = 6.9 Hz,

6H). **^{13}C NMR** (100 MHz, DMSO-d_6): δ 150.00, 148.45, 141.80, 126.74, 122.14, 52.91, 33.32, 28.92, 24.66. **HRMS** (ESI): calcd for $C_{15}H_{22}D_3N_2S$ $[M + H]^+$ 268.1927, found 268.1933.

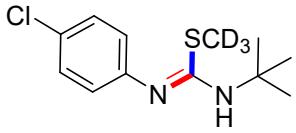
methyl-d3 (Z)-N-(tert-butyl)-N'-(4-fluorophenyl)carbamimidothioate



4d

Following the general procedure, using (petroleum ether : EtOAc = 9 : 1) as the eluant afforded a yellow liquid (62.0 mg, 85% yield). **^{1}H NMR** (400 MHz, DMSO-d_6): δ 7.04-7.00 (m, 2H), 6.74-6.71 (m, 2H), 5.76 (s, 1H), 1.40 (s, 9H). **^{13}C NMR** (100 MHz, DMSO-d_6): δ 158.19 (d, J = 237.1 Hz), 150.94, 147.23, 123.55 (d, J = 7.9 Hz), 115.48 (d, J = 21.9 Hz), 53.02, 28.89. **^{19}F NMR** (375 MHz, DMSO-d_6) δ -123.00 (1F); **HRMS** (ESI): calcd for $C_{12}H_{15}D_3N_2FS$ $[M + H]^+$ 244.1363, found 244.1372.

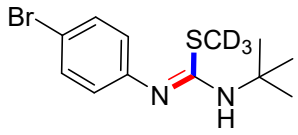
methyl-d3 (Z)-N-(tert-butyl)-N'-(4-chlorophenyl)carbamimidothioate



4e

Following the general procedure, using (petroleum ether : EtOAc = 9 : 1) as the eluant afforded a yellow liquid (68.4 mg, 88% yield). **^{1}H NMR** (400 MHz, DMSO-d_6): δ 7.24 (d, J = 8.7 Hz, 2H), 6.74 (d, J = 8.6 Hz, 2H), 5.89 (s, 1H), 1.39 (s, 9H). **^{13}C NMR** (100 MHz, DMSO-d_6): δ 151.07, 149.70, 128.87, 125.86, 124.08, 53.10, 28.86. **HRMS** (ESI): calcd for $C_{12}H_{15}D_3N_2SCl$ $[M + H]^+$ 260.1068, found 260.1071.

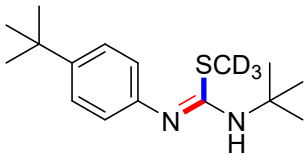
methyl-d3 (Z)-N'-(4-bromophenyl)-N-(tert-butyl)carbamimidothioate



4f

Following the general procedure, using (petroleum ether : EtOAc = 9 : 1) as the eluant afforded a yellow solid (79.1 mg, 87% yield), Mp = 70-71℃. **^{1}H NMR** (400 MHz, DMSO-d_6): δ 7.36 (d, J = 8.6 Hz, 2H), 6.69 (d, J = 8.6 Hz, 2H), 5.90 (s, 1H), 1.39 (s, 9H). **^{13}C NMR** (100 MHz, DMSO-d_6): δ 151.01, 150.10, 131.77, 124.60, 113.84, 53.12, 28.86. **HRMS** (ESI): calcd for $C_{12}H_{15}D_3N_2SBr$ $[M + H]^+$ 304.0562, found 304.0570.

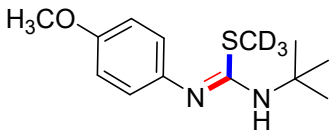
methyl-d3 (Z)-N-(tert-butyl)-N'-(4-(tert-butyl)phenyl)carbamimidothioate



4g

Following the general procedure, using (petroleum ether : EtOAc = 9 : 1) as the eluant afforded a yellow liquid (80.1 mg, 95% yield). **^{1}H NMR** (400 MHz, DMSO-d_6): δ 7.23 (d, J = 8.5 Hz, 2H), 6.67 (d, J = 8.4 Hz, 2H), 5.67 (s, 1H), 1.41 (s, 9H), 1.28 (s, 9H). **^{13}C NMR** (100 MHz, DMSO-d_6): δ 149.97, 148.06, 144.02, 125.59, 121.85, 52.91, 34.34, 31.90, 28.92. **HRMS** (ESI): calcd for $C_{16}H_{24}D_3N_2S$ $[M + H]^+$ 282.2083, found 282.2091.

methyl-d3 (Z)-N-(tert-butyl)-N'-(4-methoxyphenyl)carbamimidothioate

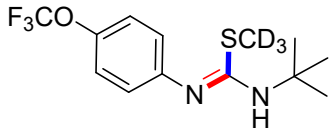


4h

Following the general procedure, using (petroleum ether : EtOAc = 9 : 1) as the eluant afforded a yellow solid (70.4 mg, 92% yield), Mp = 54-55℃. **^{1}H NMR** (400 MHz, DMSO-d_6): δ 6.79 (d, J = 8.8 Hz, 2H), 6.65 (d, J = 8.7 Hz, 2H), 5.59 (s, 1H), 3.71 (s, 3H), 1.39 (s, 9H). **^{13}C NMR** (100 MHz, DMSO-d_6): δ 154.85, 150.30, 144.06, 123.07, 114.33, 55.61, 52.90, 28.94. **HRMS** (ESI): calcd for

$C_{13}H_{18}D_3N_2OS$ $[M + H]^+$ 256.1563, found 256.1567.

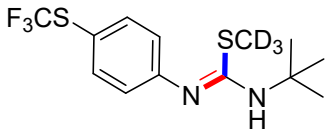
methyl-d3 (Z)-N-(tert-butyl)-N'-(4-(trifluoromethoxy)phenyl)carbamimidothioate



4i

Following the general procedure, using (petroleum ether : EtOAc = 9 : 1) as the eluant afforded a yellow liquid (78.8 mg, 85% yield). **^{1}H NMR** (400 MHz, DMSO-d_6): δ 7.19 (d, J = 8.4 Hz, 2H), 6.81 (d, J = 8.8 Hz, 2H), 5.92 (s, 1H), 1.40 (s, 9H). **^{13}C NMR** (100 MHz, DMSO-d_6): δ 151.23, 150.06, 143.35, 123.58, 121.94, 53.13, 28.84. **^{19}F NMR** (375 MHz, DMSO-d_6) δ -57.05 (3F); **HRMS** (ESI): calcd for $C_{13}H_{15}D_3N_2OSF_3$ $[M + H]^+$ 310.1280, found 310.1271.

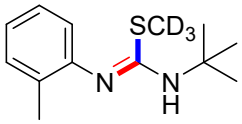
methyl-d3 (Z)-N-(tert-butyl)-N'-(4-((trifluoromethyl)thio)phenyl)carbamimidothioate



4j

Following the general procedure, using (petroleum ether : EtOAc = 9 : 1) as the eluant afforded a yellow solid (79.9 mg, 82% yield), Mp = 55-56℃. **^{1}H NMR** (400 MHz, DMSO-d_6): δ 7.53 (d, J = 8.1 Hz, 2H), 6.88 (d, J = 8.4 Hz, 2H), 6.13 (s, 1H), 1.40 (s, 9H). **^{13}C NMR** (100 MHz, DMSO-d_6): δ 153.80, 151.41, 137.63, 123.81, 114.17, 53.27, 28.82. **^{19}F NMR** (375 MHz, DMSO-d_6) δ -43.18 (3F); **HRMS** (ESI): calcd for $C_{13}H_{15}D_3N_2S_2F_3$ $[M + H]^+$ 326.1052, found 326.1061.

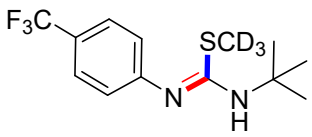
methyl-d3 (Z)-N-(tert-butyl)-N'-(o-tolyl)carbamimidothioate



4k

Following the general procedure, using (petroleum ether : EtOAc = 9 : 1) as the eluant afforded a yellow liquid (60.3 mg, 84% yield). **^{1}H NMR** (400 MHz, DMSO-d_6): δ 7.10 (d, J = 7.4 Hz, 1H), 7.04 (td, J = 7.6, 1.6 Hz, 1H), 6.84 (td, J = 7.4, 1.4 Hz, 1H), 6.64 (dd, J = 7.8, 1.4 Hz, 1H), 5.67 (s, 1H), 2.09 (s, 3H), 1.44 (s, 9H). **^{13}C NMR** (100 MHz, DMSO-d_6): δ 149.73, 149.56, 130.16, 129.55, 126.48, 122.15, 121.61, 52.88, 28.96, 18.57. **HRMS** (ESI): calcd for $C_{13}H_{18}D_3N_2S$ $[M + H]^+$ 240.1614, found 240.1617.

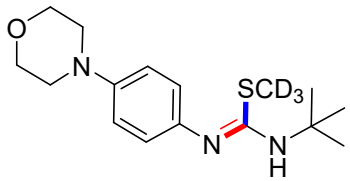
methyl-d3 (Z)-N-(tert-butyl)-N'-(4-(trifluoromethyl)phenyl)carbamimidothioate



4l

Following the general procedure, using (petroleum ether : EtOAc = 9 : 1) as the eluant afforded a yellow liquid (60.6 mg, 69% yield). **^{1}H NMR** (400 MHz, DMSO-d_6): δ 7.54 (d, J = 8.3 Hz, 2H), 6.91 (d, J = 8.2 Hz, 2H), 6.12 (s, 1H), 1.40 (s, 9H). **^{13}C NMR** (100 MHz, DMSO-d_6): δ 154.37, 151.48, 126.17 (q, J = 4.2 Hz), 125.39 (q, J = 269.1 Hz), 122.79, 121.96 (q, J = 32.1 Hz), 53.26, 28.81. **^{19}F NMR** (375 MHz, DMSO-d_6) δ -59.85 (3F); **HRMS** (ESI): calcd for $C_{13}H_{15}D_3N_2SF_3$ $[M + H]^+$ 294.1331, found 294.1335.

methyl-d3 (Z)-N-(tert-butyl)-N'-(4-morpholinophenyl)carbamimidothioate

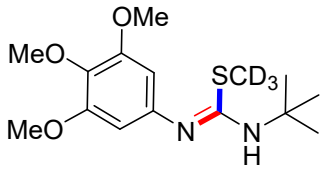


4m

Following the general procedure, using (petroleum ether : EtOAc = 9 : 1) as the eluant afforded a

yellow solid (81.9 mg, 88% yield), Mp = 71-72℃. **^{1}H NMR** (400 MHz, DMSO-d_6): δ 6.82 (d, J = 8.9 Hz, 2H), 6.64 (d, J = 8.7 Hz, 2H), 5.56 (s, 1H), 3.74 (t, J = 4.6 Hz, 4H), 3.01 (t, J = 4.6 Hz, 4H), 1.39 (s, 9H). **^{13}C NMR** (100 MHz, DMSO-d_6): δ 149.97, 146.69, 143.40, 122.76, 116.46, 66.74, 52.88, 50.03, 28.96. **HRMS** (ESI): calcd for $C_{16}H_{23}D_3N_3OS$ $[M + H]^+$ 311.1985, found 311.1990.

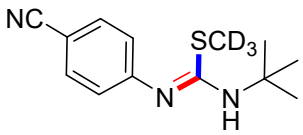
methyl-d3 (Z)-N-(tert-butyl)-N'-(3,4,5-trimethoxyphenyl)carbamimidothioate



4n

Following the general procedure, using (petroleum ether : EtOAc = 9 : 1) as the eluant afforded a yellow solid (85.1 mg, 90% yield), Mp = 67-68℃. **^{1}H NMR** (400 MHz, DMSO-d_6): δ 6.02 (s, 2H), 5.64 (s, 1H), 3.73 (s, 6H), 3.62 (s, 3H), 1.41 (s, 9H). **^{13}C NMR** (100 MHz, DMSO-d_6): δ 153.30, 150.46, 146.86, 133.09, 99.74, 60.58, 56.15, 52.97, 28.95. **HRMS** (ESI): calcd for $C_{15}H_{22}D_3N_2O_3S$ $[M + H]^+$ 316.1774, found 316.1781.

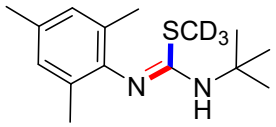
methyl-d3 (Z)-N-(tert-butyl)-N'-(4-cyanophenyl)carbamimidothioate



4o

Following the general procedure, using (petroleum ether : EtOAc = 9 : 1) as the eluant afforded a yellow solid (56.3 mg, 75% yield), Mp = 57-58℃. **^{1}H NMR** (400 MHz, DMSO-d_6): δ 7.63 (d, J = 8.5 Hz, 2H), 6.89 (d, J = 8.5 Hz, 2H), 6.31 (s, 1H), 1.39 (s, 9H). **^{13}C NMR** (100 MHz, DMSO-d_6): δ 155.00, 151.88, 133.38, 123.24, 120.25, 103.43, 53.40, 28.79. **HRMS** (ESI): calcd for $C_{13}H_{15}D_3N_3S$ $[M + H]^+$ 251.1410, found 251.1416.

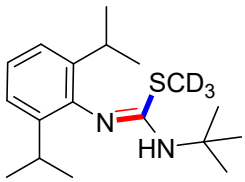
methyl-d3 (Z)-N-(tert-butyl)-N'-mesitylcarbamimidothioate



4p

Following the general procedure, using (petroleum ether : EtOAc = 9 : 1) as the eluant afforded a yellow solid (61.7 mg, 77% yield), Mp = 49-50℃. **^{1}H NMR** (400 MHz, DMSO-d_6): δ 6.74 (s, 2H), 5.46 (s, 1H), 2.18 (s, 3H), 2.01 (s, 6H), 1.47 (s, 9H). **^{13}C NMR** (100 MHz, DMSO-d_6): δ 149.65, 146.01, 130.21, 128.51, 52.77, 28.93, 20.91, 18.51. **HRMS** (ESI): calcd for $C_{15}H_{22}D_3N_2S$ $[M + H]^+$ 268.1927, found 268.1930.

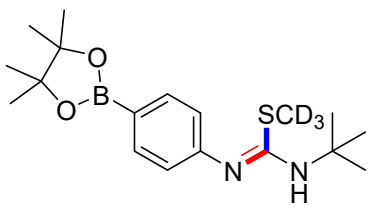
methyl-d3 (Z)-N-(tert-butyl)-N'-(2,6-diisopropylphenyl)carbamimidothioate



4q

Following the general procedure, using (petroleum ether : EtOAc = 9 : 1) as the eluant afforded a yellow liquid (60.3 mg, 65% yield). **^{1}H NMR** (400 MHz, DMSO-d_6): δ 7.00 (d, J = 7.5 Hz, 2H), 6.89 (dd, J = 8.2, 6.9 Hz, 1H), 5.52 (s, 1H), 2.97 (p, J = 6.9 Hz, 2H), 1.46 (s, 9H), 1.19 (d, J = 6.9 Hz, 6H), 1.06 (d, J = 6.9 Hz, 6H). **^{13}C NMR** (100 MHz, DMSO-d_6): δ 150.01, 145.95, 139.07, 122.79, 122.62, 52.74, 28.93, 28.02, 24.32, 23.34. **HRMS** (ESI): calcd for $C_{18}H_{28}D_3N_2S$ $[M + H]^+$ 310.2396, found 310.2402.

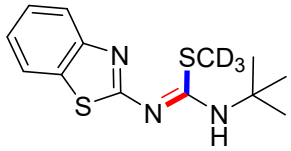
methyl-d3 (Z)-N-(tert-butyl)-N'-(4-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)phenyl)carbamimidothioate



4r

Following the general procedure, using (petroleum ether : EtOAc = 9 : 1) as the eluant afforded a yellow solid (94.8 mg, 90% yield), Mp = 58-59℃. **^{1}H NMR** (400 MHz, DMSO-d_6): δ 7.53 (d, J = 7.8 Hz, 2H), 6.74 (d, J = 7.8 Hz, 2H), 5.89 (s, 1H), 1.39 (s, 9H), 1.29 (s, 12H). **^{13}C NMR** (100 MHz, DMSO-d_6): δ 153.65, 150.39, 135.65, 121.87, 83.72, 53.08, 28.87, 25.22. **HRMS** (ESI): calcd for $C_{18}H_{27}D_3BN_2O_2S$ $[M + H]^+$ 352.2309, found 352.2318.

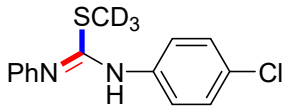
methyl-d3 (Z)-N'-(benzo[d]thiazol-2-yl)-N-(tert-butyl)carbamimidothioate



4s

Following the general procedure, using (petroleum ether : EtOAc = 9 : 1) as the eluant afforded a yellow solid (66.0 mg, 78% yield), Mp = 106-107℃. **^{1}H NMR** (400 MHz, DMSO-d_6): δ 7.83 (d, J = 7.8 Hz, 1H), 7.64 (d, J = 8.0 Hz, 1H), 7.37 (t, J = 7.6 Hz, 1H), 7.24 (t, J = 7.5 Hz, 1H), 1.50 (s, 9H). **^{13}C NMR** (100 MHz, DMSO-d_6): δ 151.43, 132.25, 126.29, 123.73, 121.83, 120.63, 53.81, 29.38. **HRMS** (ESI): calcd for $C_{13}H_{15}D_3N_3S_2$ $[M + H]^+$ 283.1130, found 283.1137.

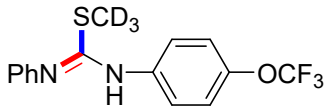
methyl-d3 (Z)-N-(4-chlorophenyl)-N'-phenylcarbamimidothioate



5a

Following the general procedure, using (petroleum ether : EtOAc = 9 : 1) as the eluant afforded a yellow liquid (65.3 mg, 78% yield). **^{1}H NMR** (400 MHz, DMSO-d_6): δ 8.87 (d, J = 28.9 Hz, 1H), 7.76 (dd, J = 33.1, 8.2 Hz, 2H), 7.44-7.36 (m, 4H), 7.10 (t, J = 7.3 Hz, 1H), 6.96 (dd, J = 8.1, 5.4 Hz, 2H). **^{13}C NMR** (100 MHz, DMSO-d_6): δ 149.64, 148.91, 141.13, 140.35, 129.21, 129.07, 128.93, 128.76, 123.83, 123.14, 122.93, 121.98, 121.79, 120.74. **HRMS** (ESI): calcd for $C_{14}H_{11}D_3N_2SCl$ $[M + H]^+$ 280.0755, found 280.0761.

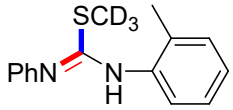
methyl-d3 (Z)-N'-phenyl-N-(4-(trifluoromethoxy)phenyl)carbamimidothioate



5b

Following the general procedure, using (petroleum ether : EtOAc = 9 : 1) as the eluant afforded a yellow liquid (75.0 mg, 76% yield). **^{1}H NMR** (400 MHz, DMSO-d_6): δ 8.84 (d, J = 41.6 Hz, 1H), 7.78 (d, J = 8.5 Hz, 1H), 7.63 (d, J = 8.0 Hz, 1H), 7.29-7.23 (m, 4H), 7.00 (t, J = 7.4 Hz, 1H), 6.94 (d, J = 8.4 Hz, 1H), 6.85 (d, J = 7.8 Hz, 1H). **^{13}C NMR** (100 MHz, DMSO-d_6): δ 151.30, 150.19, 149.61, 149.25, 143.94, 143.35, 141.11, 140.62, 129.18, 128.90, 124.54, 123.39, 123.14, 122.95, 122.06, 121.97, 121.77, 121.46, 120.77, 119.47. **^{19}F NMR** (375 MHz, DMSO-d_6) δ -57.09 (3F); **HRMS** (ESI): calcd for $C_{15}H_{11}D_3N_2OF_3S$ $[M + H]^+$ 330.0967, found 330.0970.

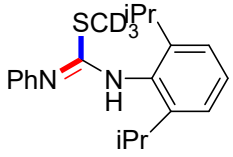
(Z)-1-(l1-methyl)-N'-phenyl-N-(o-tolyl)-l5-sulfanecarboximidamide



5c

Following the general procedure, using (petroleum ether : EtOAc = 9 : 1) as the eluant afforded a yellow solid (55.9 mg, 72% yield), Mp = 47-48℃. **^{1}H NMR** (400 MHz, DMSO-d_6): δ 8.63 (s, 1H), 7.69 (d, J = 8.0 Hz, 2H), 7.29 (t, J = 7.7 Hz, 2H), 7.16 (d, J = 7.4 Hz, 1H), 7.10 (t, J = 7.6 Hz, 1H), 6.99 (t, J = 7.4 Hz, 1H), 6.91 (t, J = 7.5 Hz, 1H), 6.72 (d, J = 7.7 Hz, 1H), 3.38 (s, 3H). **^{13}C NMR** (100 MHz, DMSO-d_6): δ 149.80, 148.70, 141.49, 130.43, 128.93, 126.69, 122.93, 122.76, 121.27, 120.39, 18.54. **HRMS** (ESI): calcd for $C_{15}H_{14}D_3N_2S$ $[M + H]^+$ 260.1301, found 260.1307.

(Z)-N-(2,6-diisopropylphenyl)-1-(l1-methyl)-N'-phenyl-l5-sulfanecarboximidamide

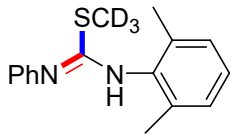


5d

Following the general procedure, using (petroleum ether : EtOAc = 9 : 1) as the eluant afforded a yellow liquid (68.1 mg, 69% yield). **^{1}H NMR** (400 MHz, DMSO-d_6): δ 8.46 (s, 1H), 7.74 (d, J =

8.0 Hz, 2H), 7.31 (t, *J* = 7.7 Hz, 2H), 7.07 (d, *J* = 7.6 Hz, 2H), 6.99 (d, *J* = 8.6 Hz, 2H), 2.99-2.96 (m, 2H), 1.22 (d, *J* = 6.9 Hz, 6H), 1.10 (d, *J* = 6.8 Hz, 6H). **^{13}C NMR** (100 MHz, DMSO-d_6): δ 149.72, 145.09, 141.65, 138.47, 129.02, 123.28, 123.10, 122.73, 120.17, 28.22, 24.11, 23.50. **HRMS** (ESI): calcd for $C_{20}H_{24}D_3N_2S$ $[M + H]^+$ 330.2083, found 330.2093.

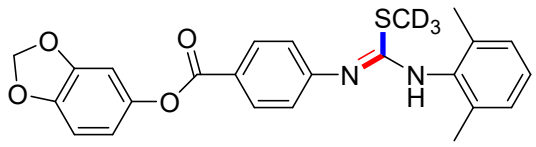
(Z)-N-(2,6-dimethylphenyl)-1-(l1-methyl)-N'-phenyl-l5-sulfanecarboximidamide



5e

Following the general procedure, using (petroleum ether : EtOAc = 9 : 1) as the eluant afforded a yellow solid (55.7 mg, 68% yield), Mp = 40-41℃. **^{1}H NMR** (400 MHz, DMSO-d_6): δ 8.50 (s, 1H), 7.72 (d, *J* = 7.9 Hz, 2H), 7.30 (t, *J* = 7.7 Hz, 2H), 6.99 (d, *J* = 6.8 Hz, 3H), 6.82 (d, *J* = 7.5 Hz, 1H), 2.07 (s, 6H). **^{13}C NMR** (100 MHz, DMSO-d_6): δ 149.67, 147.67, 141.59, 128.95, 128.11, 122.69, 122.51, 120.46, 18.66. **HRMS** (ESI): calcd for $C_{16}H_{16}D_3N_2S$ $[M + H]^+$ 274.1457, found 274.1462.

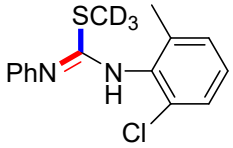
benzo[d][1,3]dioxol-5-yl (Z)-4-((((2,6-dimethylphenyl)amino)((methyl-d3)thio)methylene)amino)benzoate



5f

Following the general procedure, using (petroleum ether : EtOAc = 9 : 1) as the eluant afforded a yellow solid (86.5 mg, 66% yield), Mp = 51-52℃. **^{1}H NMR** (400 MHz, DMSO-d_6): δ 9.09 (s, 1H), 8.02 (d, *J* = 28.9 Hz, 3H), 6.98 (q, *J* = 36.4, 32.3 Hz, 6H), 6.72 (s, 1H), 6.09 (s, 2H), 2.10 (s, 6H). **^{13}C NMR** (100 MHz, DMSO-d_6): δ 164.98, 149.73, 148.17, 147.27, 146.74, 145.59, 145.44, 131.36, 128.22, 127.85, 122.94, 122.11, 119.08, 114.78, 108.44, 104.63, 102.22, 18.65. **HRMS** (ESI): calcd for $C_{24}H_{20}D_3N_2O_4S$ $[M + H]^+$ 438.1567, found 438.1569.

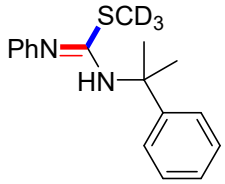
(Z)-N-(2-chloro-6-methylphenyl)-1-(l1-methyl)-N'-phenyl-l5-sulfanecarboximidamide



5g

Following the general procedure, using (petroleum ether : EtOAc = 9 : 1) as the eluant afforded a yellow liquid (62.4 mg, 71% yield). **^{1}H NMR** (400 MHz, DMSO-d_6): δ 8.75 (s, 1H), 7.72 (d, J = 7.9 Hz, 2H), 7.31 (t, J = 7.6 Hz, 2H), 7.26 (d, J = 7.9 Hz, 1H), 7.14 (d, J = 7.5 Hz, 1H), 7.03 (t, J = 7.5 Hz, 1H), 6.91 (t, J = 7.8 Hz, 1H), 2.13 (s, 3H). **^{13}C NMR** (100 MHz, DMSO-d_6): δ 152.03, 146.11, 141.20, 131.26, 129.13, 128.95, 127.31, 125.46, 123.48, 123.14, 120.93, 18.87. **HRMS** (ESI): calcd for $C_{15}H_{13}D_3N_2SCl$ $[M + H]^+$ 294.0911, found 294.0917.

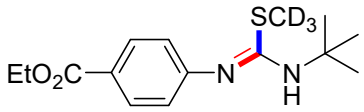
(Z)-1-(l1-methyl)-N'-phenyl-N-(2-phenylpropan-2-yl)-l5-sulfanecarboximidamide



5h

Following the general procedure, using (petroleum ether : EtOAc = 9 : 1) as the eluant afforded a yellow solid (73.2 mg, 85% yield), Mp = 49-50℃. **^{1}H NMR** (400 MHz, DMSO-d_6): δ 7.42 (d, J = 7.9 Hz, 2H), 7.31 (t, J = 7.7 Hz, 2H), 7.17 (t, J = 7.2 Hz, 1H), 7.09 (t, J = 7.7 Hz, 2H), 6.83 (t, J = 7.3 Hz, 1H), 6.39 (d, J = 7.8 Hz, 3H), 1.69 (s, 6H). **^{13}C NMR** (100 MHz, DMSO-d_6): δ 150.48, 149.34, 148.37, 128.78, 128.17, 126.01, 125.51, 121.98, 121.92, 57.55, 30.09. **HRMS** (ESI): calcd for $C_{17}H_{18}D_3N_2S$ $[M + H]^+$ 288.1614, found 288.1622.

ethyl (Z)-4-(((tert-butylamino)((methyl-d3)thio)methylene)amino)benzoate

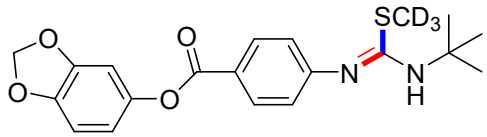


6a

Following the general procedure, using (petroleum ether : EtOAc = 9 : 1) as the eluant afforded a

yellow solid (58.9 mg, 66% yield), Mp = 71-72℃. **^{1}H NMR** (400 MHz, DMSO-d_6): δ 7.83 (d, J = 8.5 Hz, 2H), 6.84 (d, J = 8.5 Hz, 2H), 6.14 (s, 1H), 4.28 (q, J = 7.1 Hz, 2H), 1.40 (s, 9H), 1.32 (t, J = 7.1 Hz, 3H). **^{13}C NMR** (100 MHz, DMSO-d_6): δ 166.27, 155.26, 151.19, 130.54, 123.07, 122.32, 60.64, 53.26, 28.83, 14.78. **HRMS** (ESI): calcd for $C_{15}H_{20}D_3N_2O_2S$ $[M + H]^+$ 298.1669, found 298.1677.

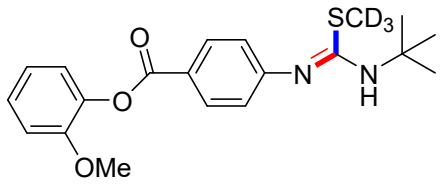
benzo[d][1,3]dioxol-5-yl (Z)-4-(((tert-butylamino)((methyl-d3)thio)methylene)amino)benzoate



6b

Following the general procedure, using (petroleum ether : EtOAc = 9 : 1) as the eluant afforded a yellow solid (80.5 mg, 69% yield), Mp = 103-104℃. **^{1}H NMR** (400 MHz, DMSO-d_6): δ 7.97 (d, J = 8.5 Hz, 2H), 6.97-6.90 (m, 4H), 6.71 (dd, J = 8.4, 2.4 Hz, 1H), 6.25 (s, 1H), 6.09 (s, 2H), 1.41 (s, 9H). **^{13}C NMR** (100 MHz, DMSO-d_6): δ 165.25, 155.97, 151.43, 148.10, 145.60, 145.35, 131.26, 122.49, 121.78, 114.76, 108.39, 104.65, 102.16, 53.30, 28.79. **HRMS** (ESI): calcd for $C_{20}H_{20}D_3N_2O_4S$ $[M + H]^+$ 390.1567, found 390.1568.

2-methoxyphenyl (Z)-4-(((tert-butylamino)((methyl-d3)thio)methylene)amino)benzoate

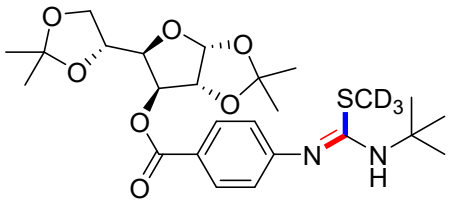


6c

Following the general procedure, using (petroleum ether : EtOAc = 9 : 1) as the eluant afforded a yellow solid (68.6 mg, 61% yield), Mp = 131-132℃. **^{1}H NMR** (400 MHz, DMSO-d_6): δ 7.97 (d, J = 8.2 Hz, 2H), 7.31-6.91 (m, 6H), 6.25 (s, 1H), 3.78 (s, 3H), 1.42 (s, 9H). **^{13}C NMR** (100 MHz, DMSO-d_6): δ 164.49, 155.97, 151.60, 151.43, 131.34, 127.38, 123.61, 122.55, 121.61, 121.08, 113.26, 56.17, 53.31, 28.78. **HRMS** (ESI): calcd for $C_{20}H_{22}D_3N_2O_3S$ $[M + H]^+$ 376.1774, found

376.1783.

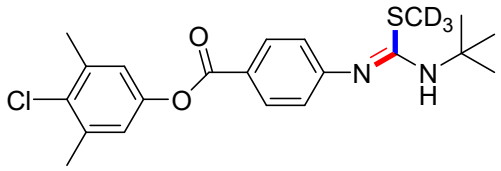
(3aR,5R,6S,6aR)-5-((R)-2,2-dimethyl-1,3-dioxolan-4-yl)-2,2-dimethyltetrahydrofuro[2,3-d][1,3]dioxol-6-yl 4-(((Z)-(tert-butylamino)((methyl-d3)thio)methylene)amino)benzoate



6d

Following the general procedure, using (petroleum ether : EtOAc = 9 : 1) as the eluant afforded a yellow liquid (98.1 mg, 64% yield). **^{1}H NMR** (400 MHz, DMSO-d_6): δ 7.84 (d, J = 8.1 Hz, 2H), 6.87 (d, J = 8.2 Hz, 2H), 6.21 (s, 1H), 5.99 (d, J = 3.7 Hz, 1H), 5.25 (d, J = 3.0 Hz, 1H), 4.68 (d, J = 3.7 Hz, 1H), 4.38 (q, J = 6.1 Hz, 1H), 4.25 (dd, J = 7.3, 3.0 Hz, 1H), 4.07 (q, J = 7.4 Hz, 1H), 3.96 (dd, J = 8.4, 5.2 Hz, 1H), 1.48 (s, 3H), 1.40 (s, 9H), 1.35 (s, 3H), 1.28 (s, 3H), 1.23 (s, 3H). **^{13}C NMR** (100 MHz, DMSO-d_6): δ 165.20, 155.84, 151.35, 130.82, 122.45, 121.99, 111.79, 108.95, 105.20, 83.28, 79.60, 76.34, 72.61, 66.72, 53.27, 28.77, 27.10, 26.92, 26.44, 25.55. **HRMS** (ESI): calcd for $C_{25}H_{34}D_3N_2O_7S$ $[M + H]^+$ 512.2510, found 512.2510.

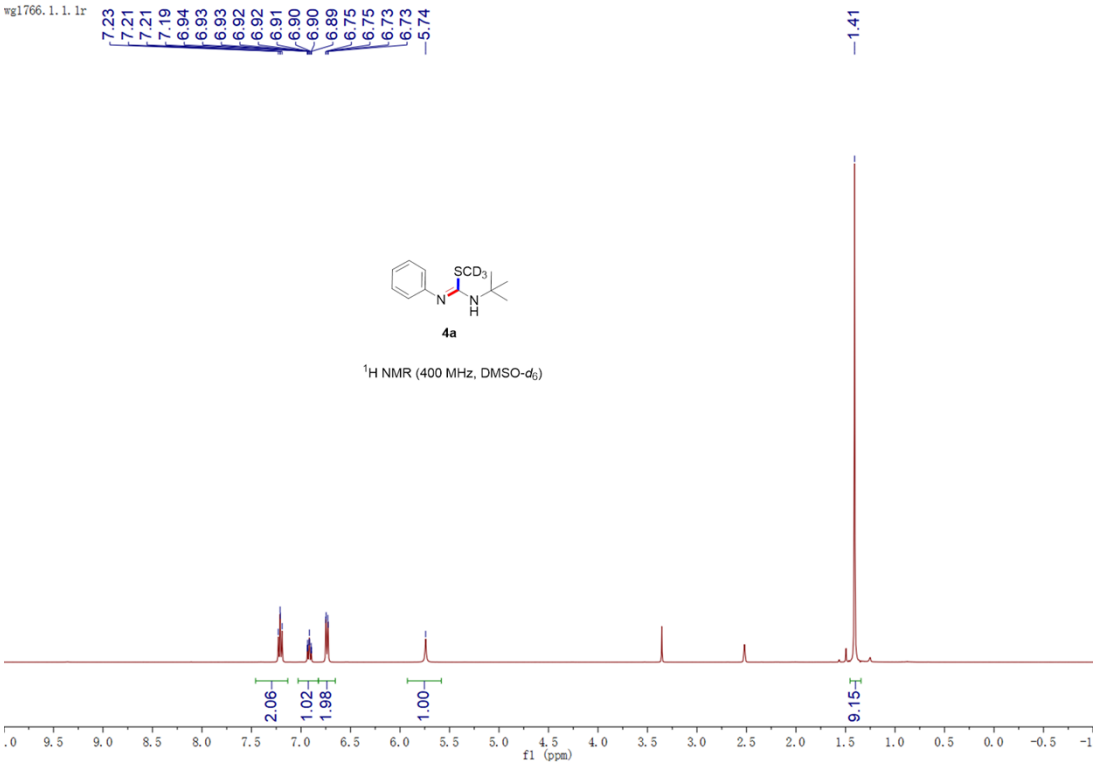
4-chloro-3,5-dimethylphenyl (Z)-4-(((tert-butylamino)((methyl-d3)thio)methylene)amino)benzoate



6e

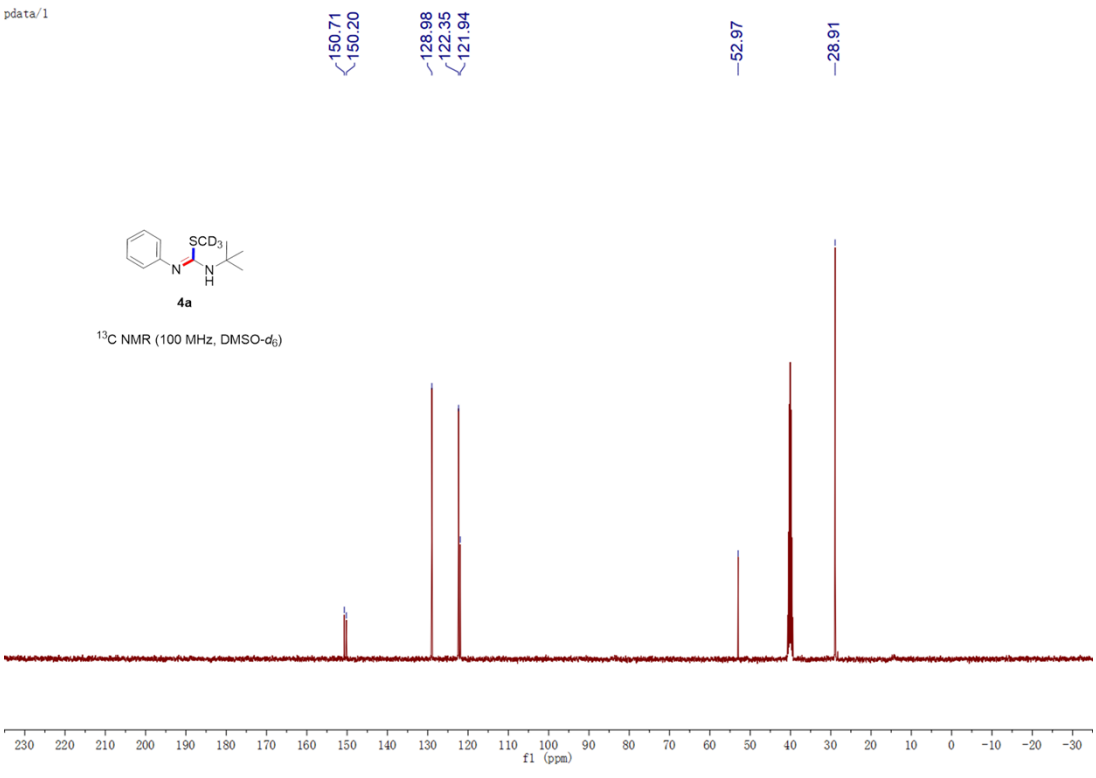
Following the general procedure, using (petroleum ether : EtOAc = 9 : 1) as the eluant afforded a yellow solid (73.3 mg, 60% yield), Mp = 121-122℃. **^{1}H NMR** (400 MHz, DMSO-d_6): δ 7.97 (d, J = 8.5 Hz, 2H), 7.15 (s, 2H), 6.92 (d, J = 8.0 Hz, 2H), 6.27 (s, 1H), 2.37 (s, 6H), 1.41 (s, 9H). **^{13}C NMR** (100 MHz, DMSO-d_6): δ 164.95, 156.08, 151.48, 149.18, 137.37, 131.31, 130.93, 122.57, 121.51, 53.31, 28.77, 20.72. **HRMS** (ESI): calcd for $C_{21}H_{23}D_3N_2O_2SCl$ $[M + H]^+$ 408.1592, found 408.1598.

^{1}H, ^{13}C and ^{19}F NMR spectra of products



4a

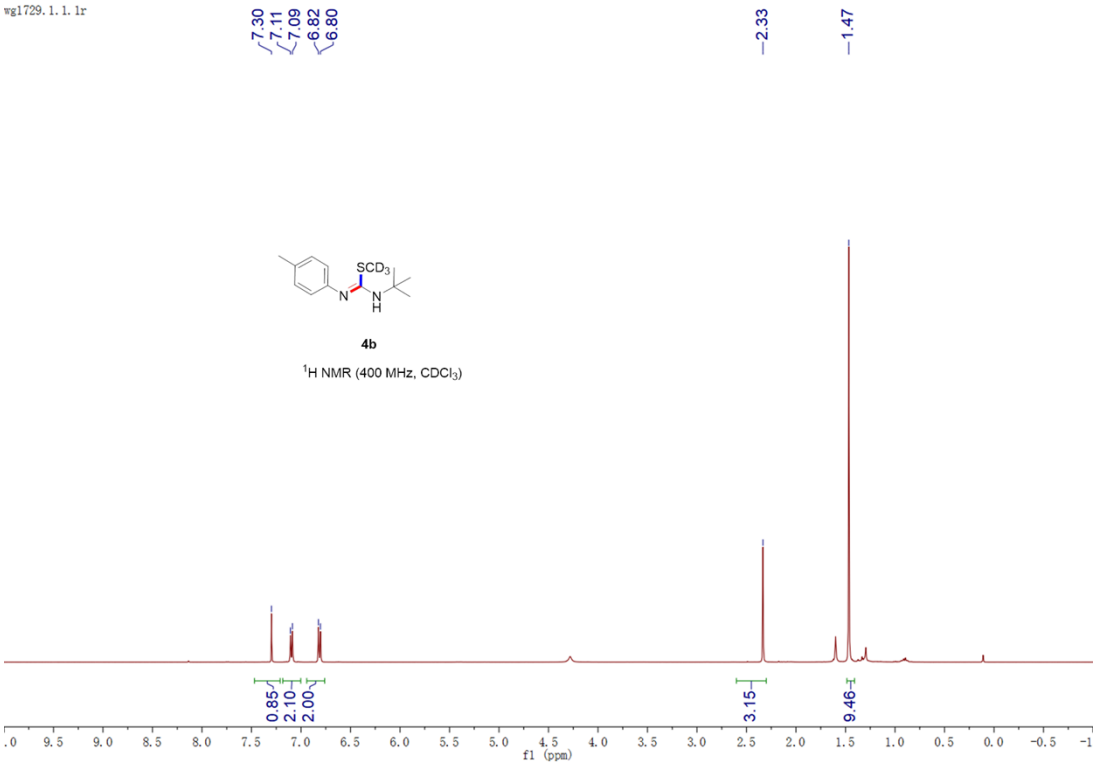
^{1}H NMR (400 MHz, DMSO-d_6)



4a

^{13}C NMR (100 MHz, DMSO-d_6)

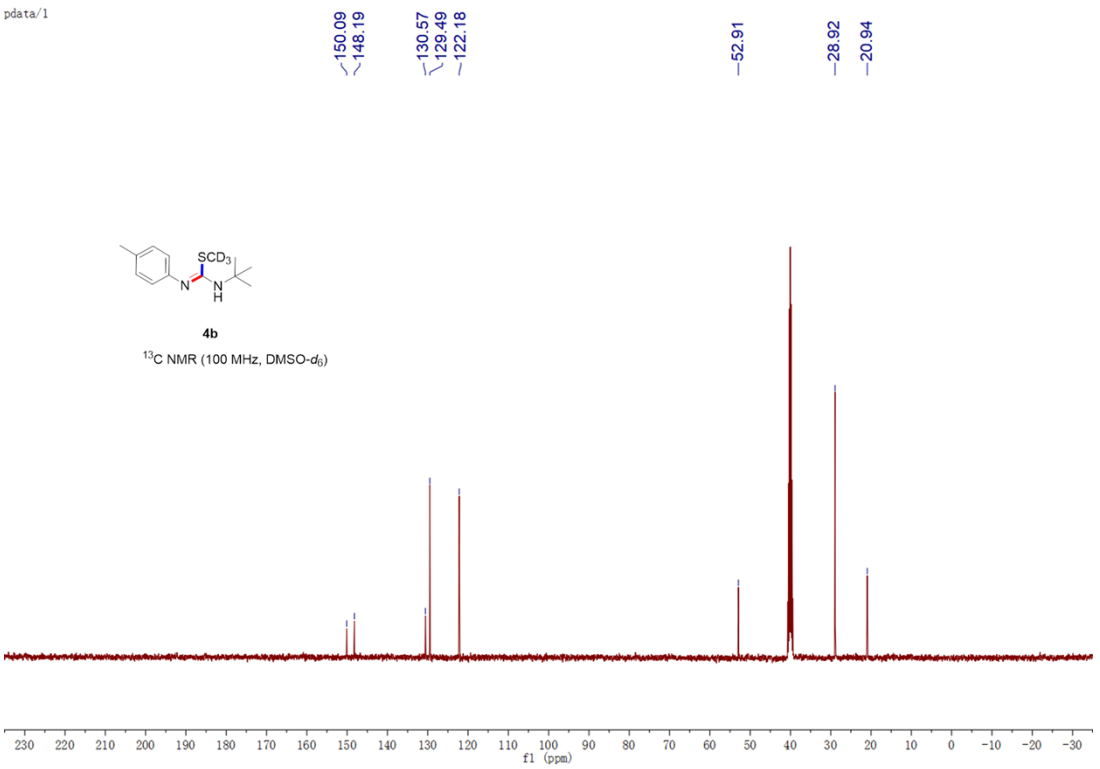
wg1729.1.1.1r



4b

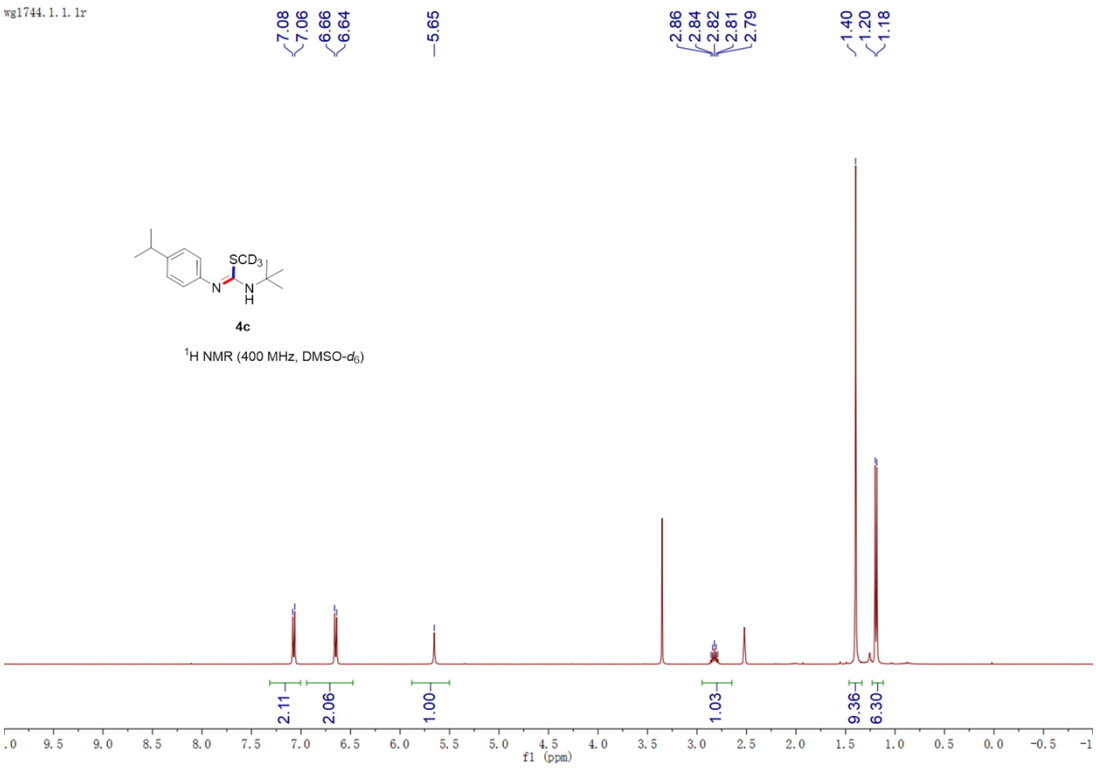
^{1}H NMR (400 MHz, $CDCl_3$)

pdata/1



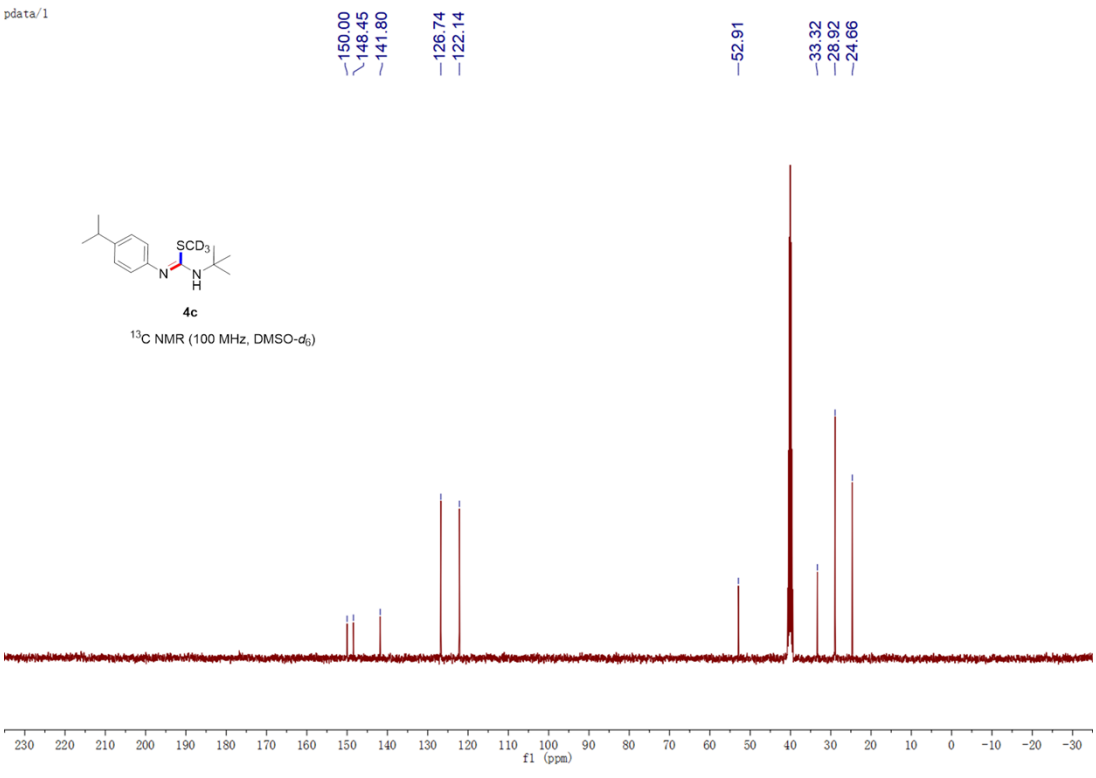
4b

^{13}C NMR (100 MHz, DMSO-d_6)



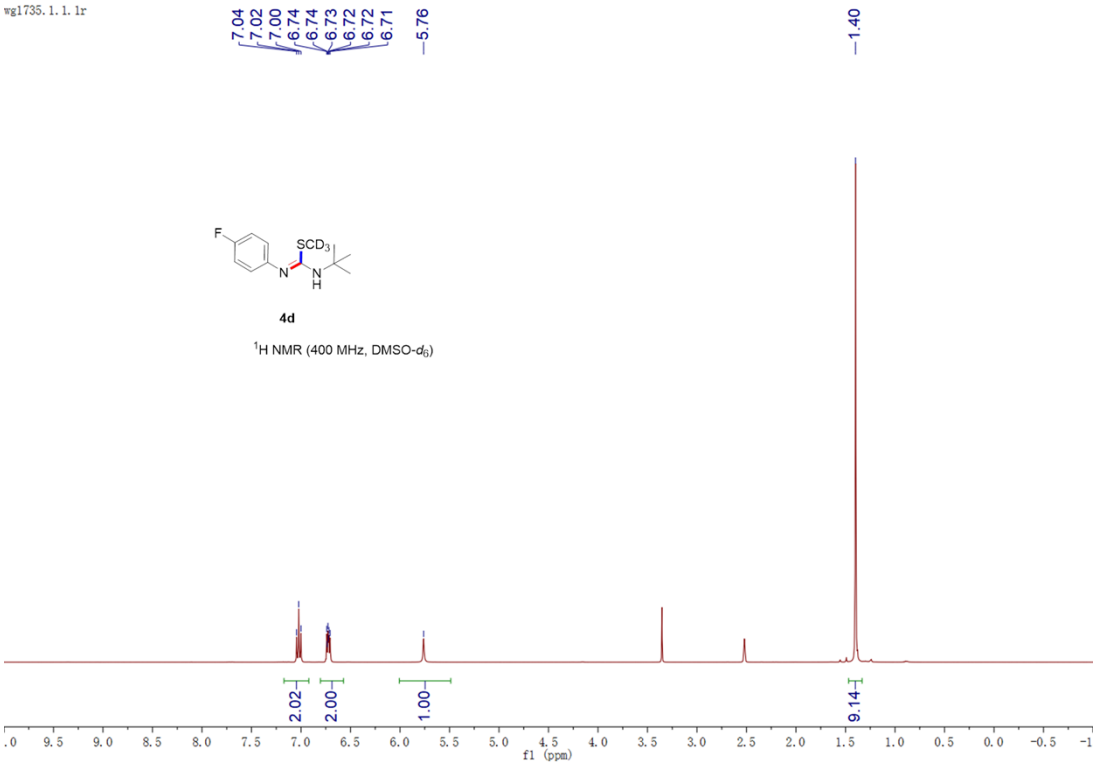
4c

^{1}H NMR (400 MHz, DMSO-d_6)



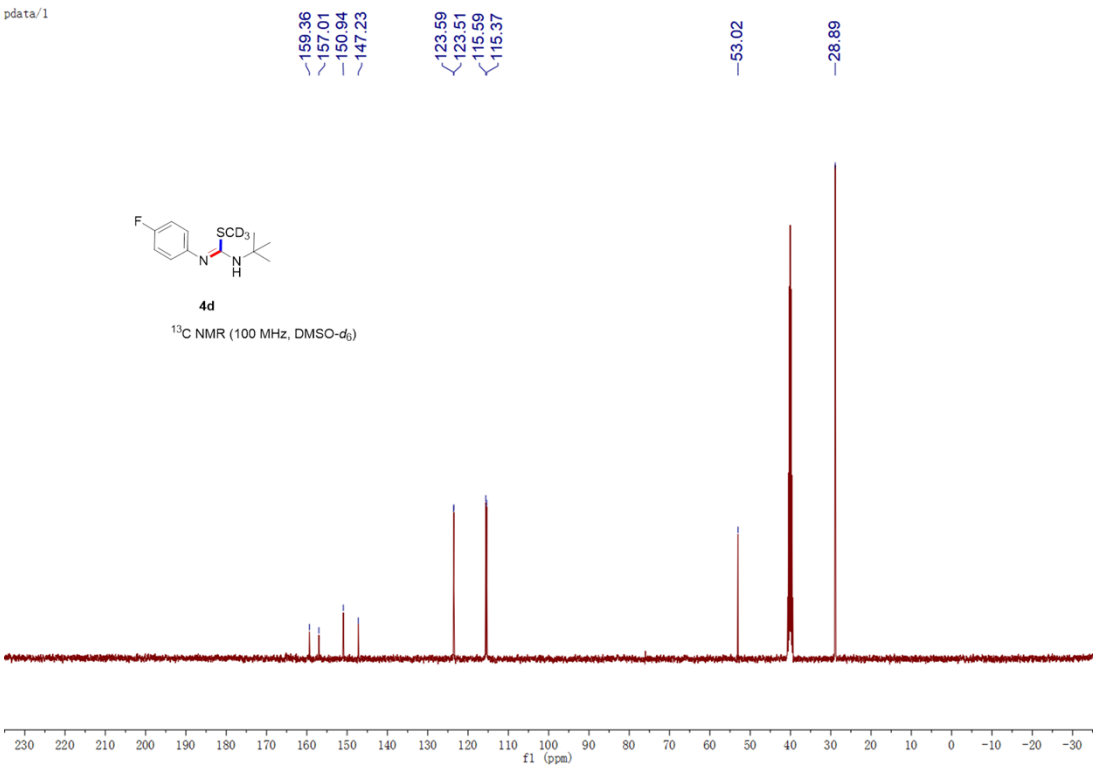
4c

^{13}C NMR (100 MHz, DMSO-d_6)



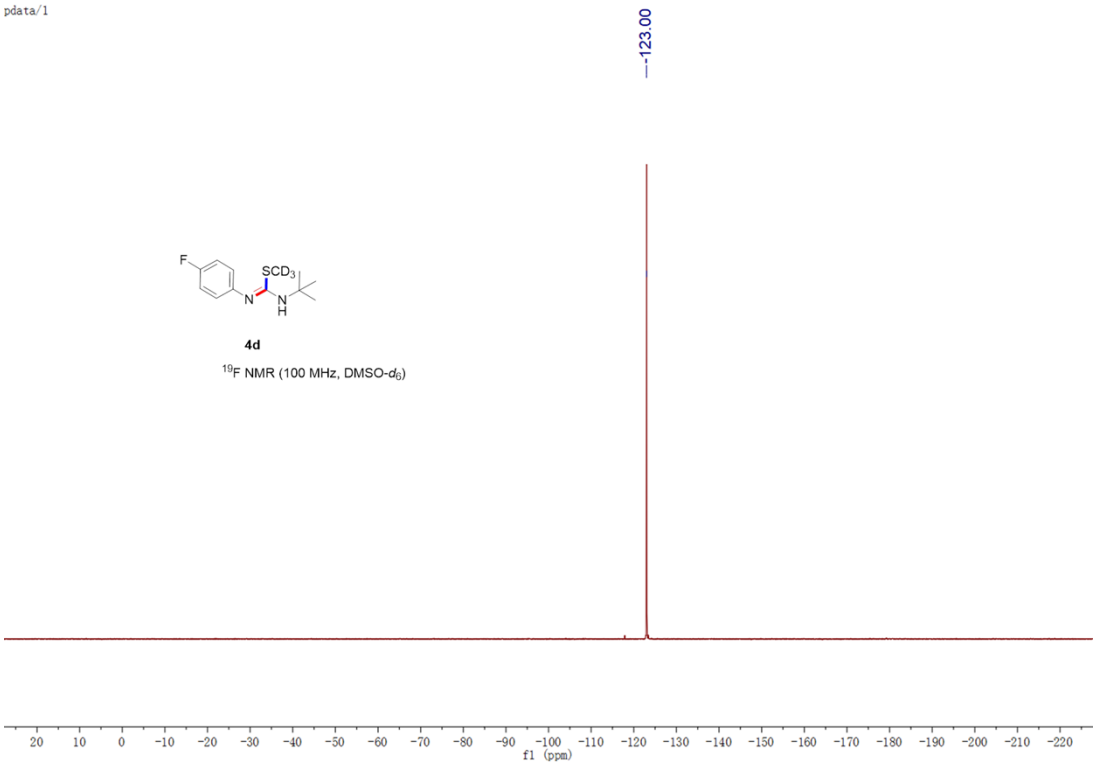
4d

^{1}H NMR (400 MHz, DMSO-d_6)



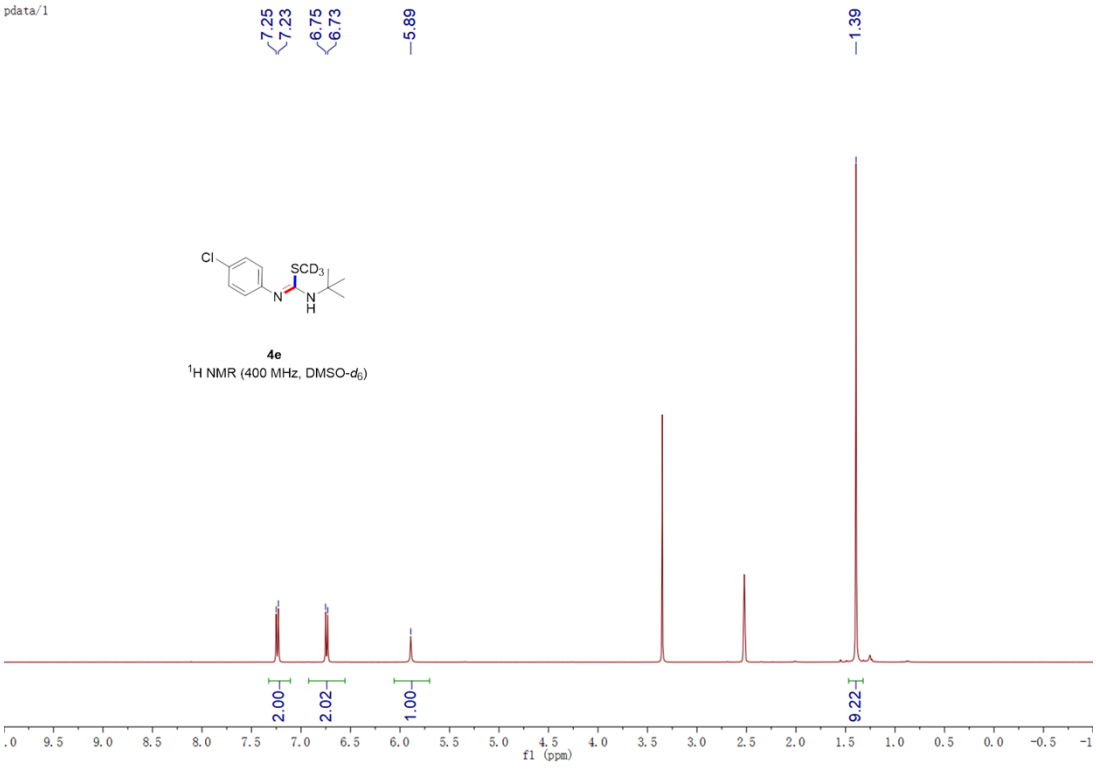
4d

^{13}C NMR (100 MHz, DMSO-d_6)



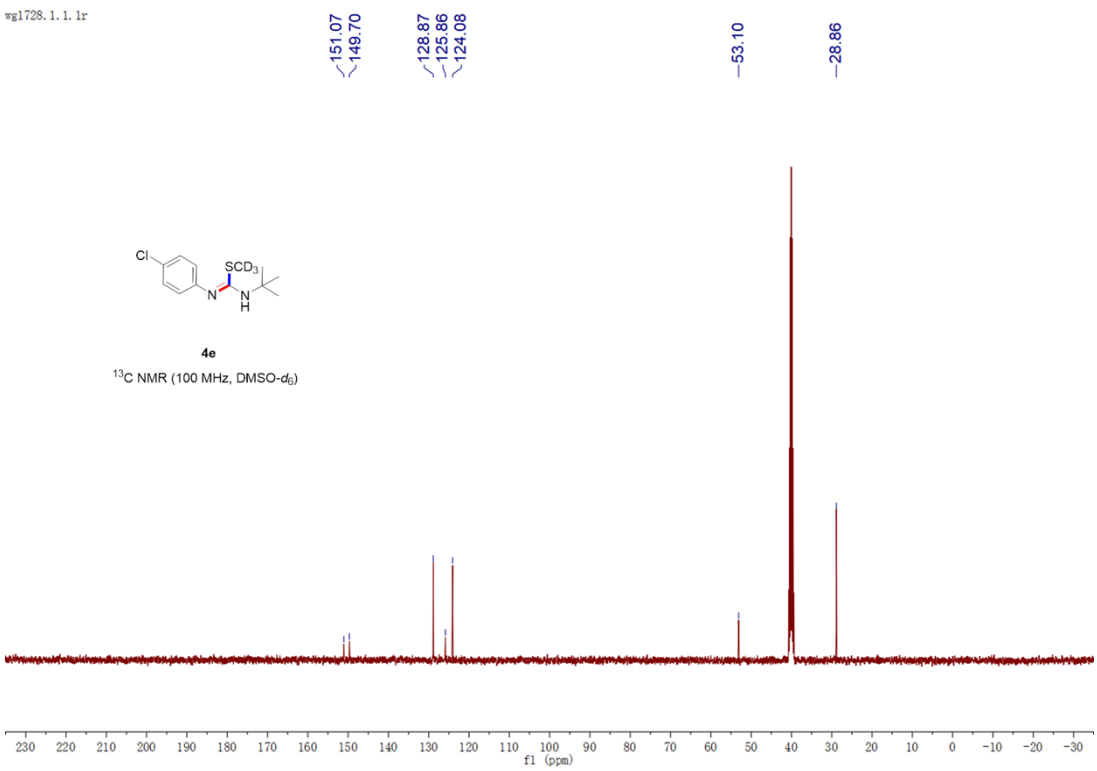
4d

^{19}F NMR (100 MHz, DMSO-d_6)



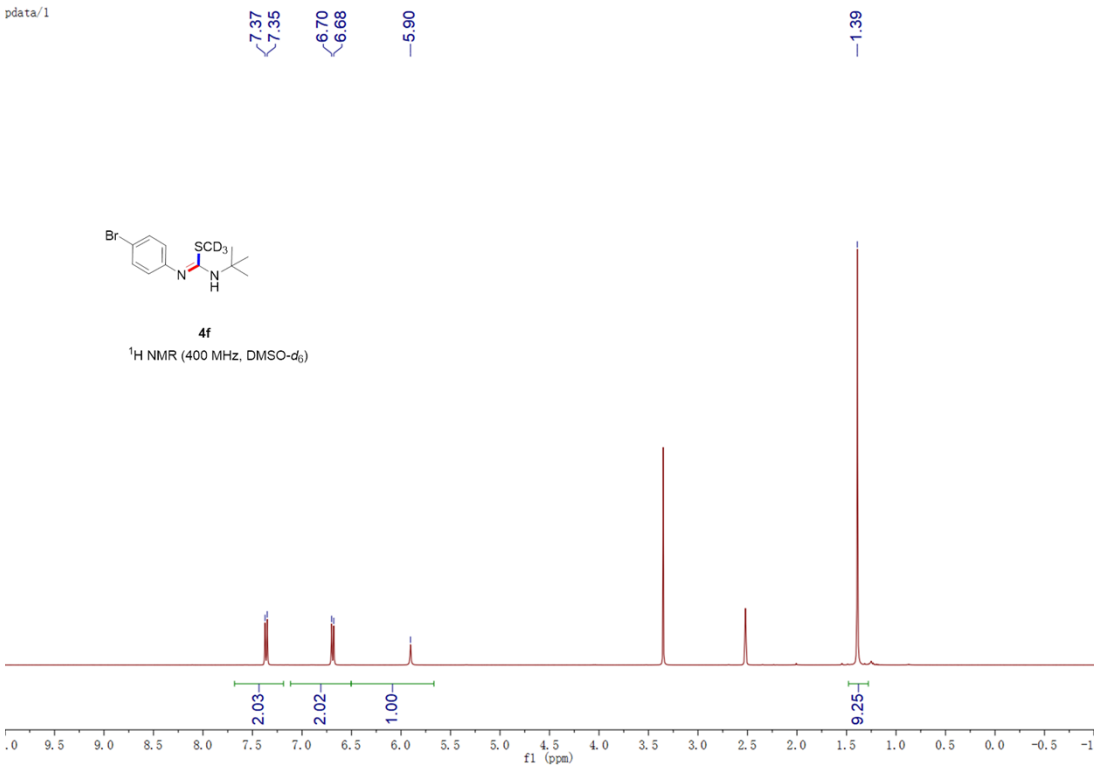
4e

^{1}H NMR (400 MHz, DMSO-d_6)



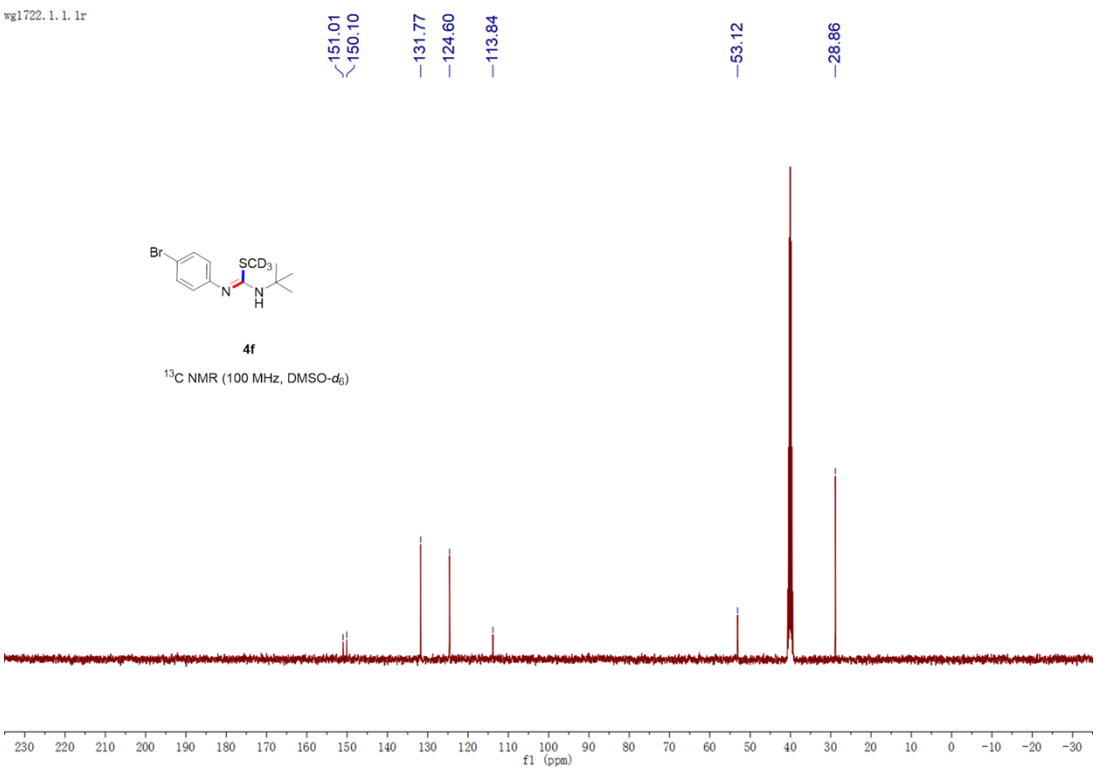
4e

^{13}C NMR (100 MHz, DMSO-d_6)



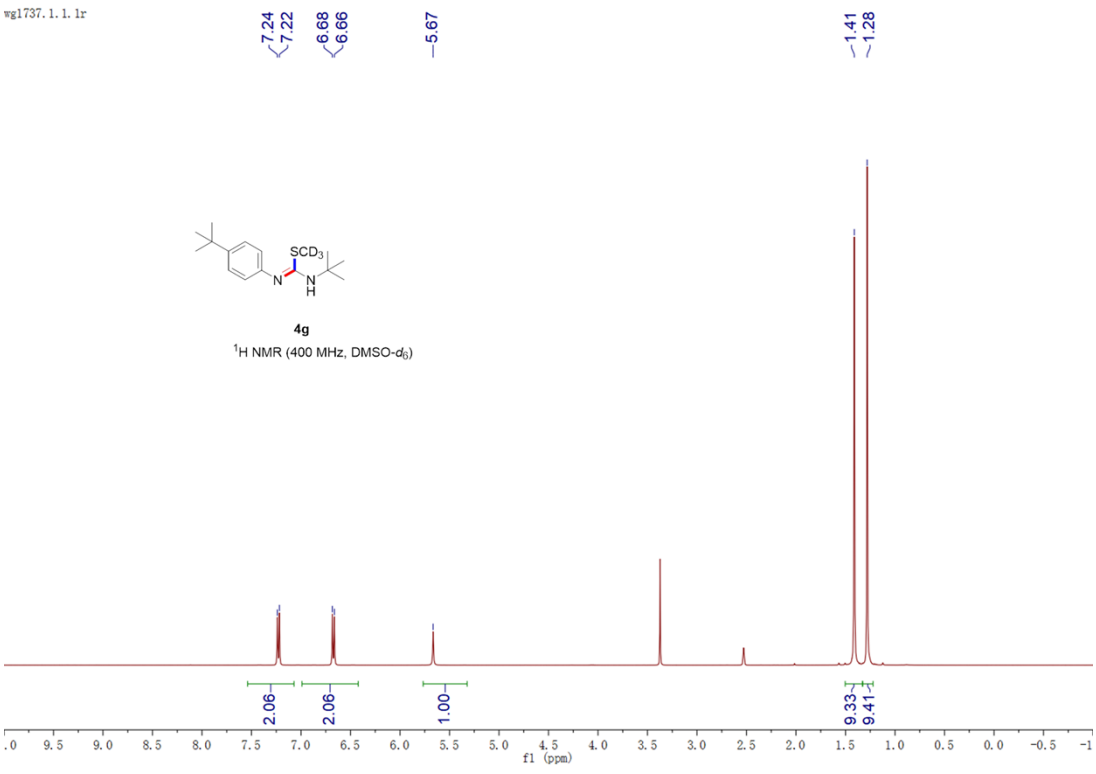
4f

^{1}H NMR (400 MHz, DMSO-d_6)



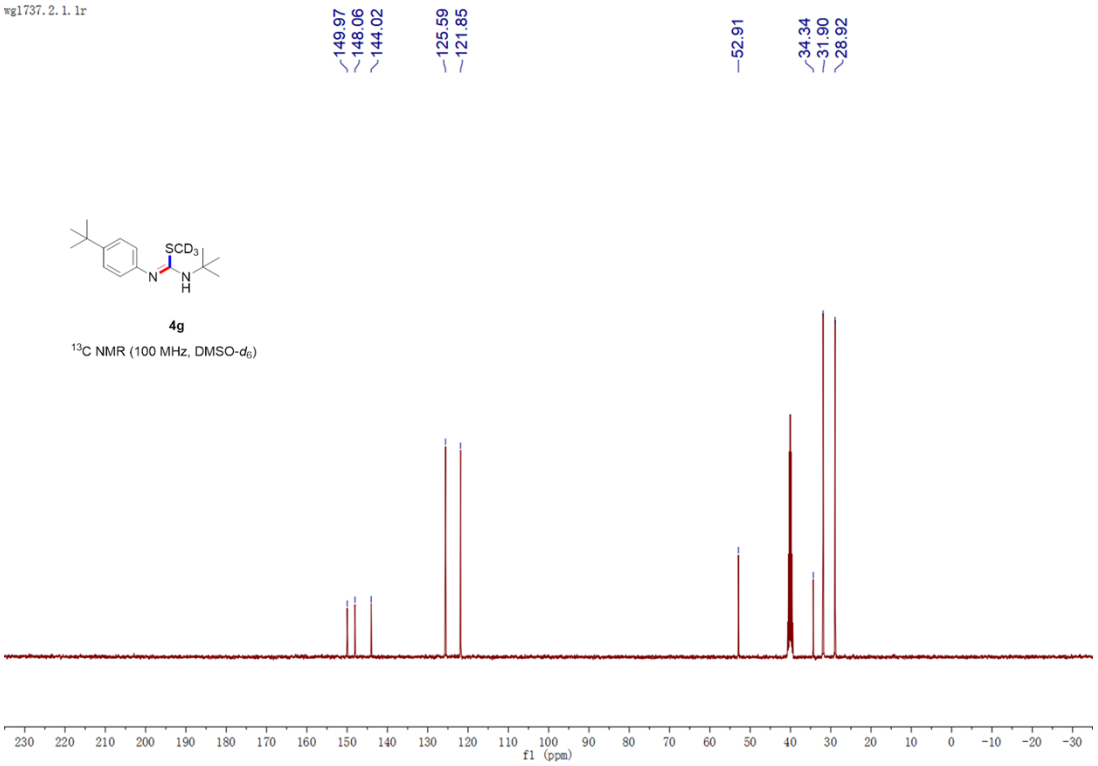
4f

^{13}C NMR (100 MHz, DMSO-d_6)



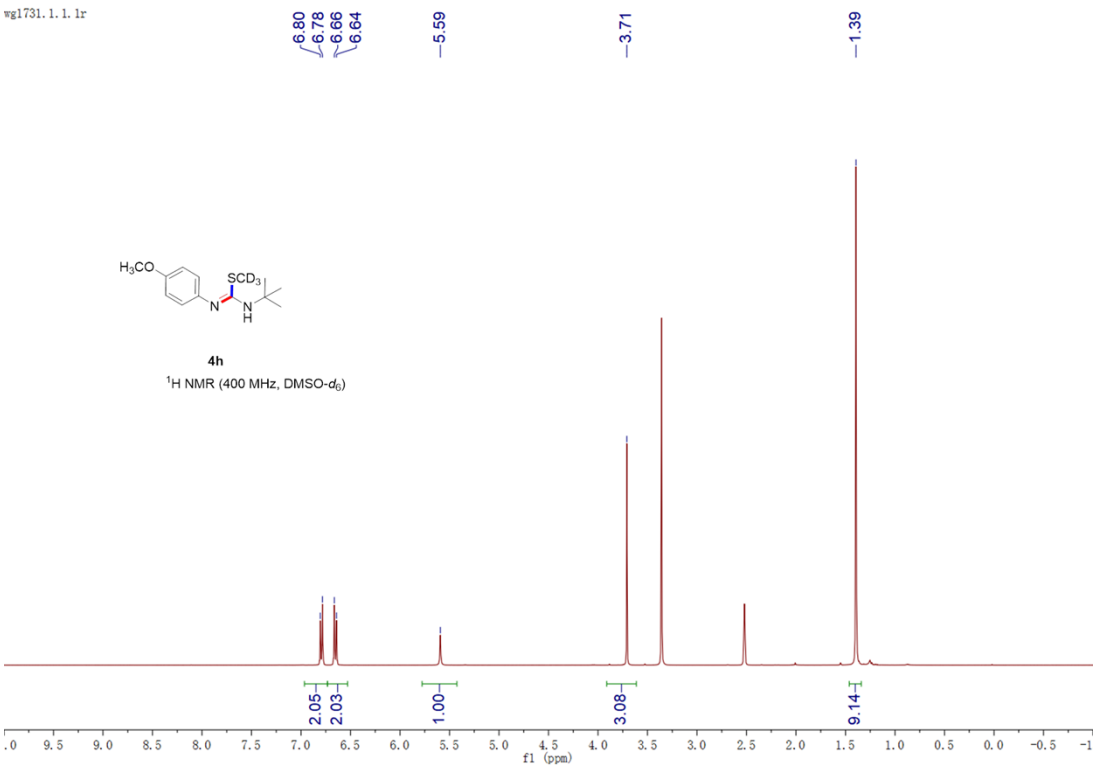
4g

^{1}H NMR (400 MHz, DMSO-d_6)



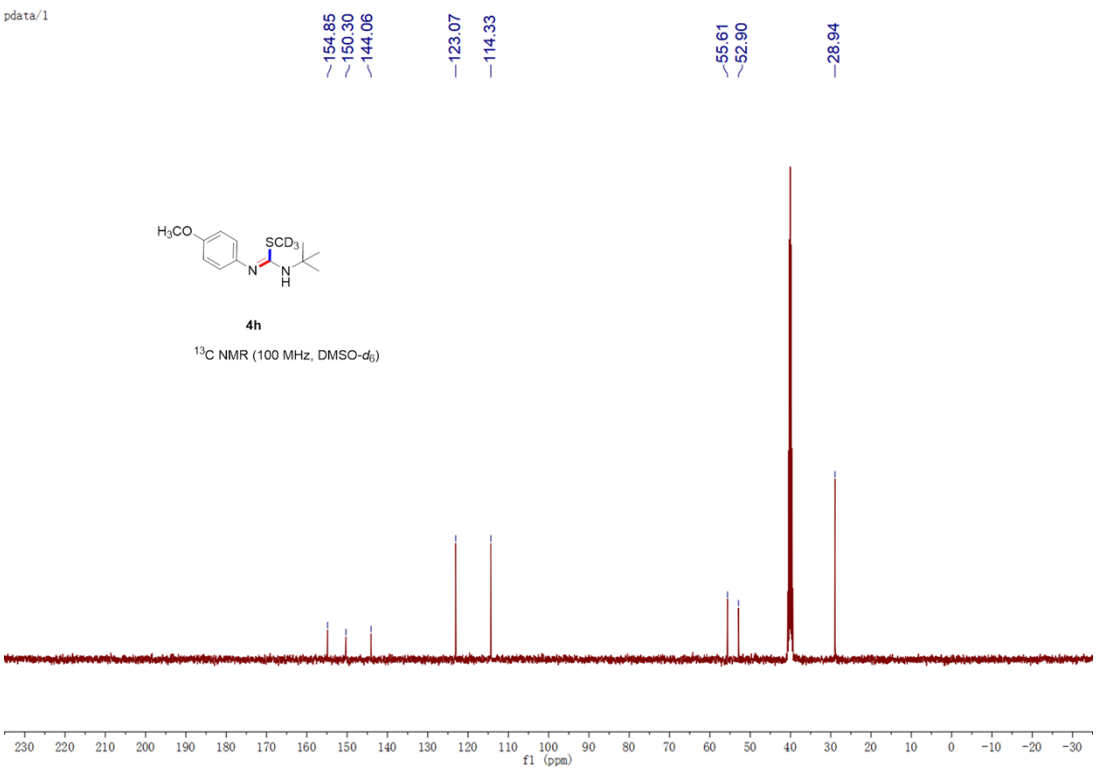
4g

^{13}C NMR (100 MHz, DMSO-d_6)



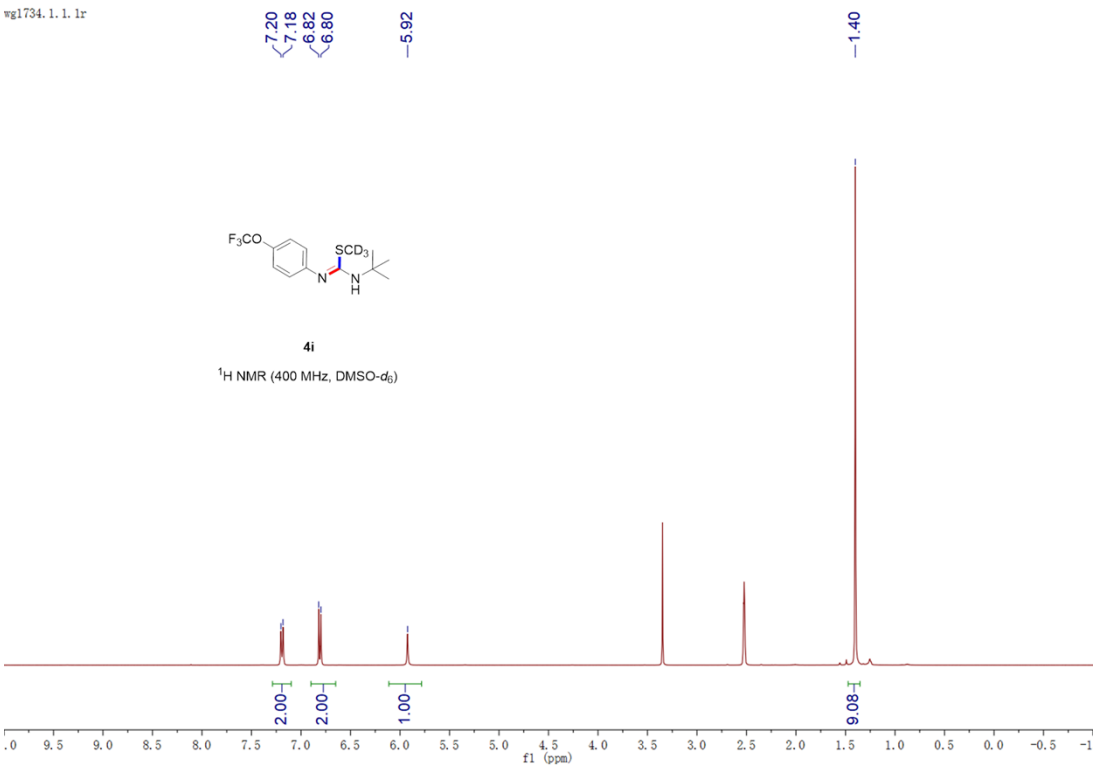
4h

^{1}H NMR (400 MHz, DMSO-d_6)



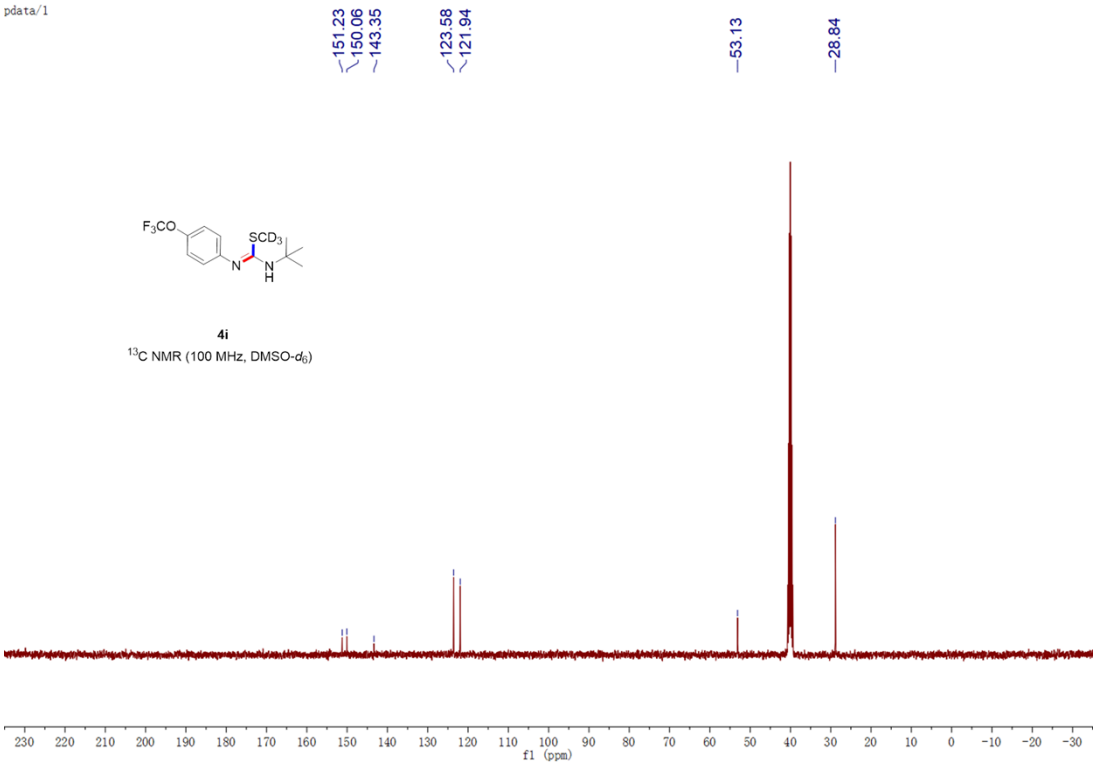
4h

^{13}C NMR (100 MHz, DMSO-d_6)



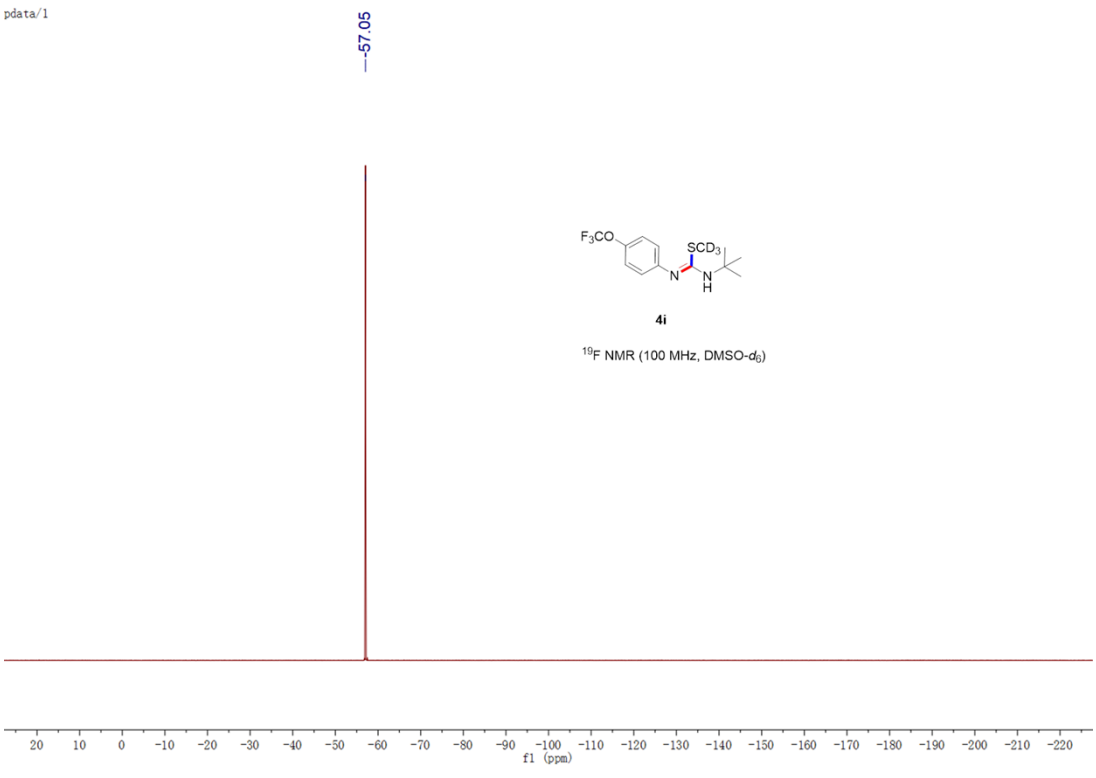
4i

^{1}H NMR (400 MHz, DMSO-d_6)



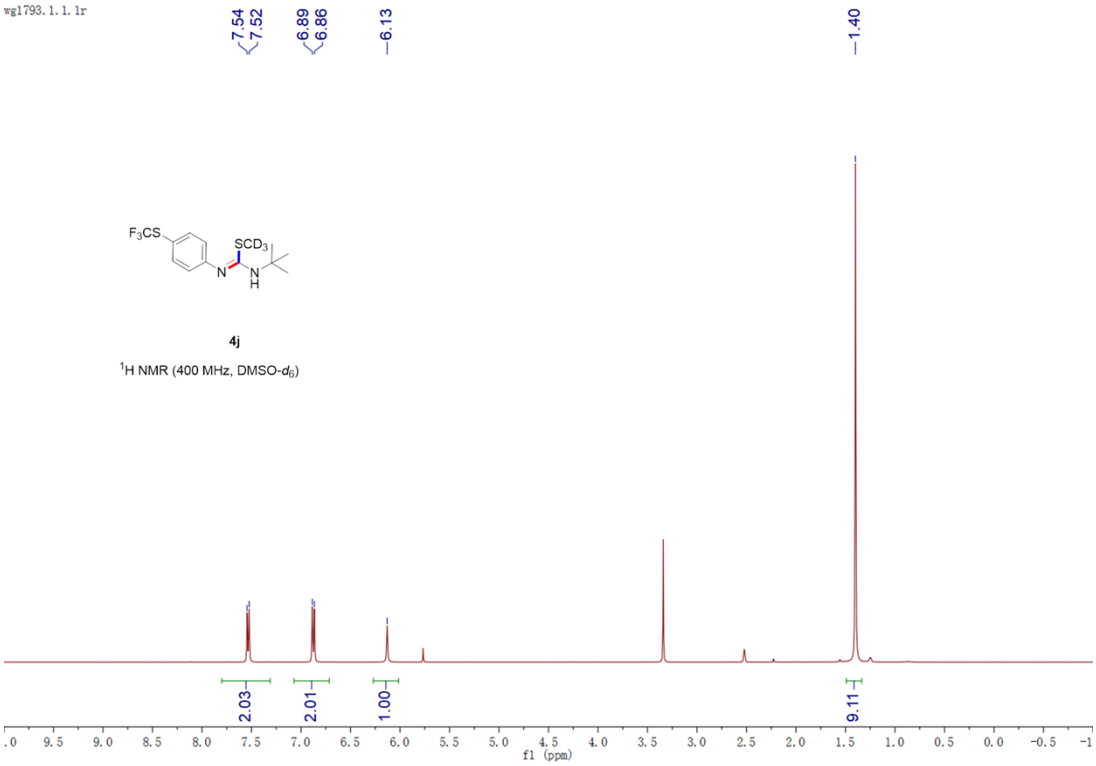
4i

^{13}C NMR (100 MHz, DMSO-d_6)



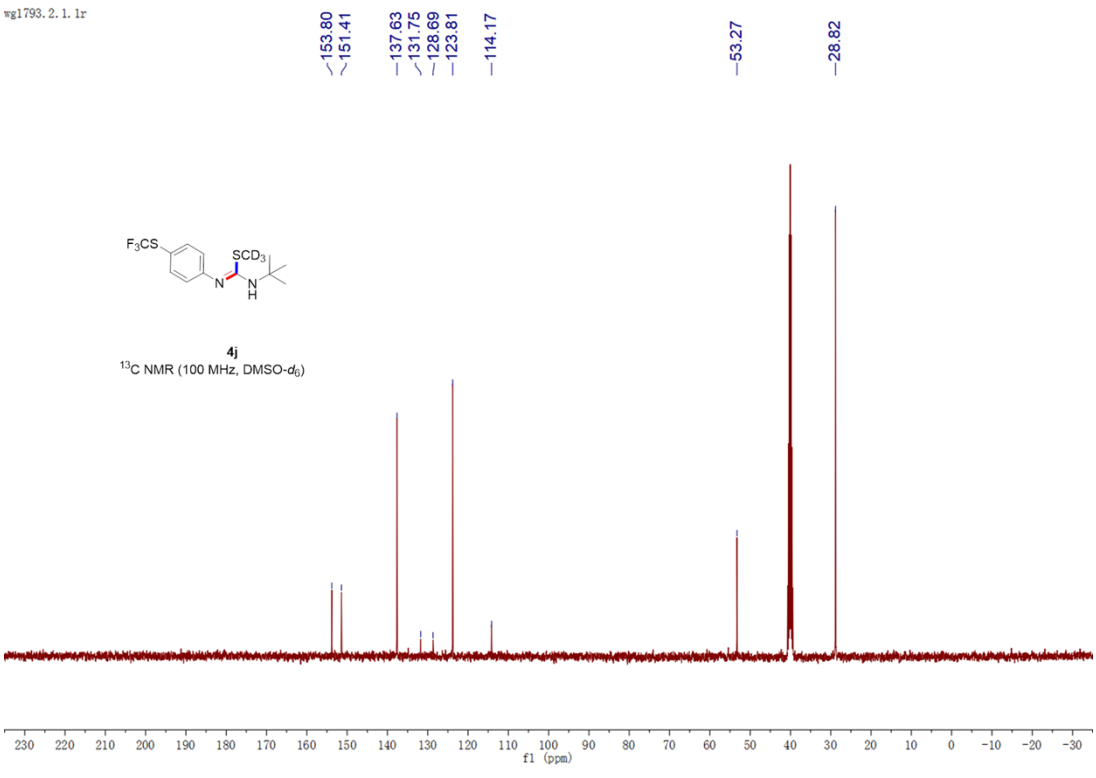
4i

^{19}F NMR (100 MHz, DMSO-d_6)



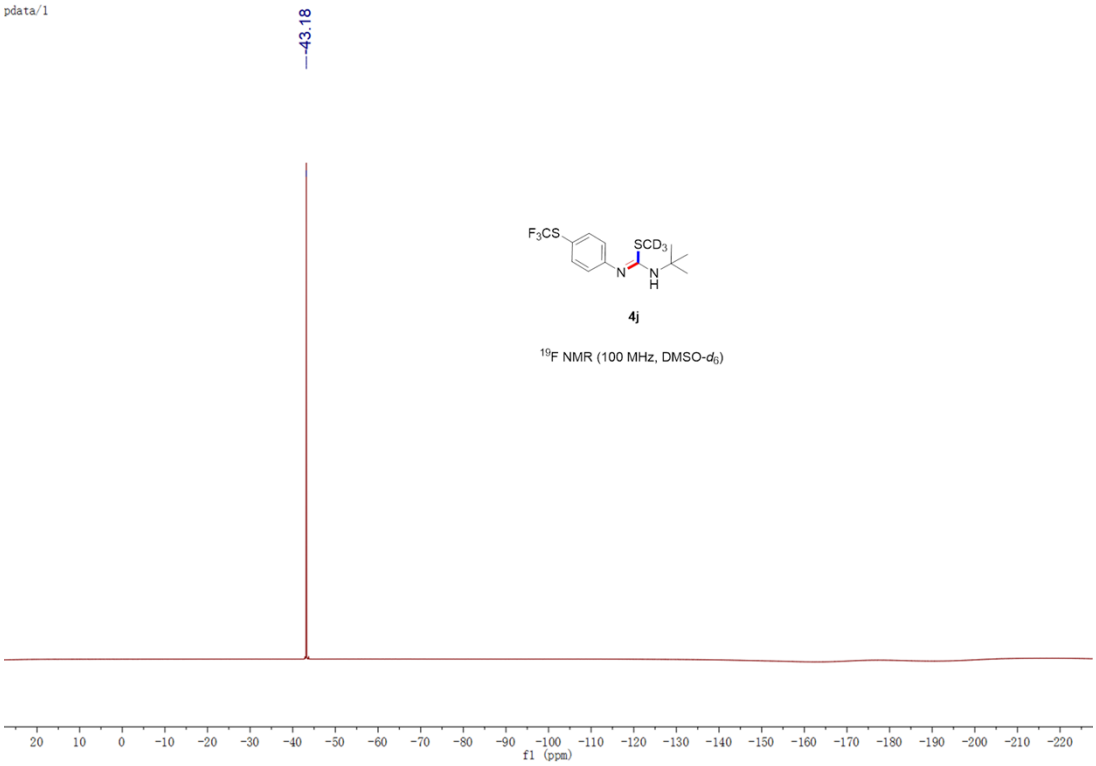
4j

^{1}H NMR (400 MHz, DMSO-d_6)



4j

^{13}C NMR (100 MHz, DMSO-d_6)



4j

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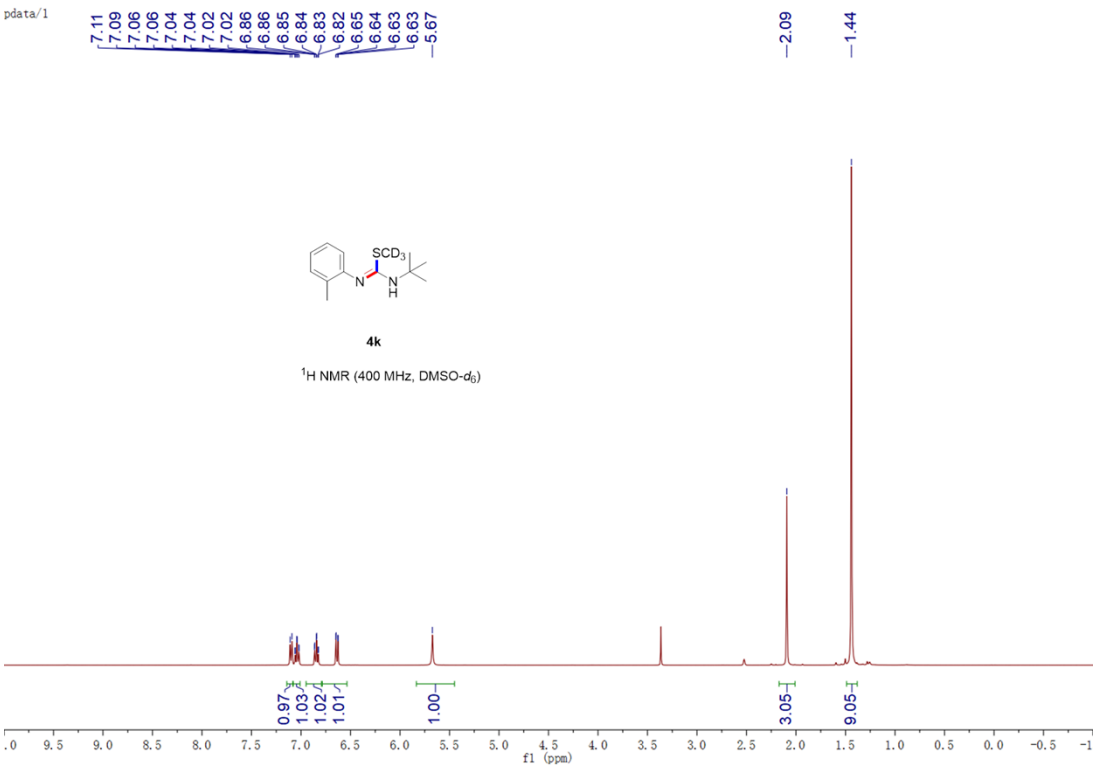
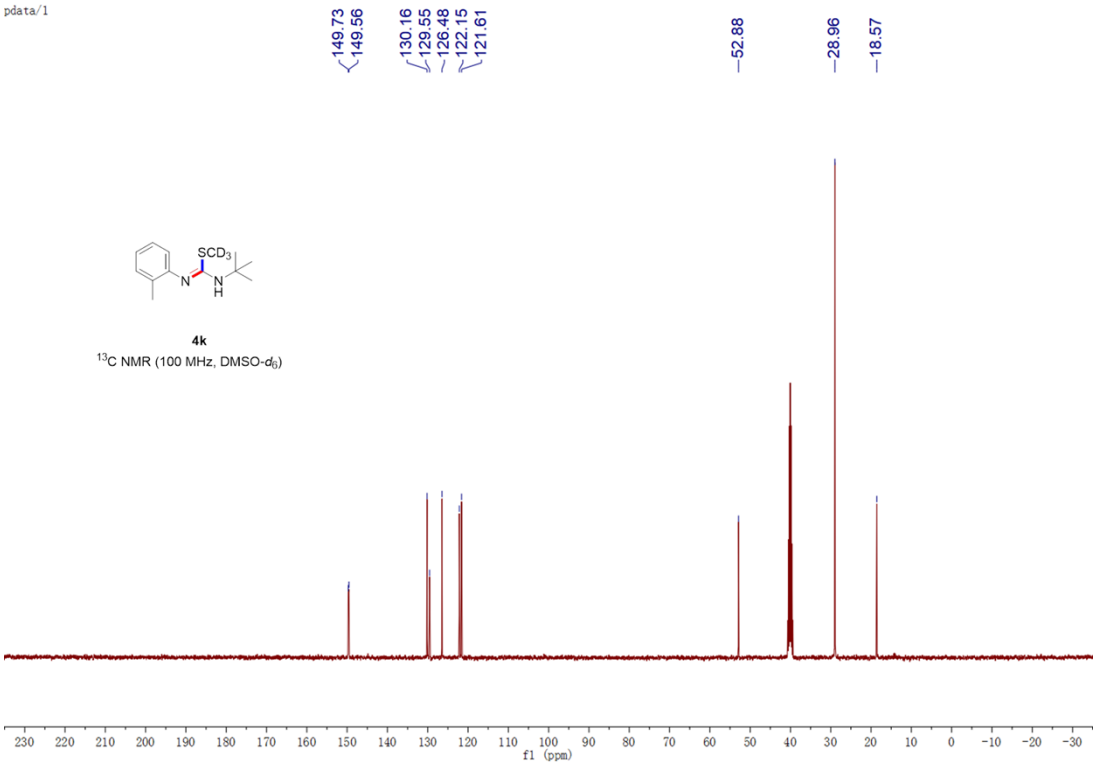


4k

^{1}H NMR (400 MHz, DMSO-d_6)



4k

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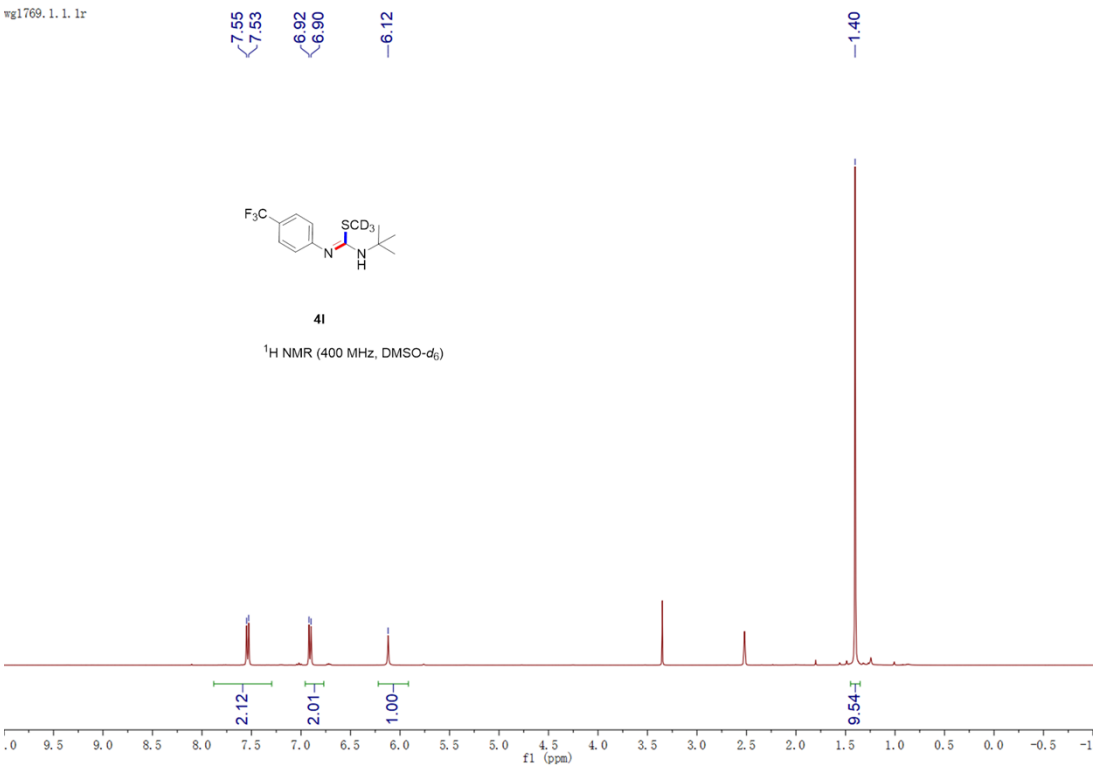
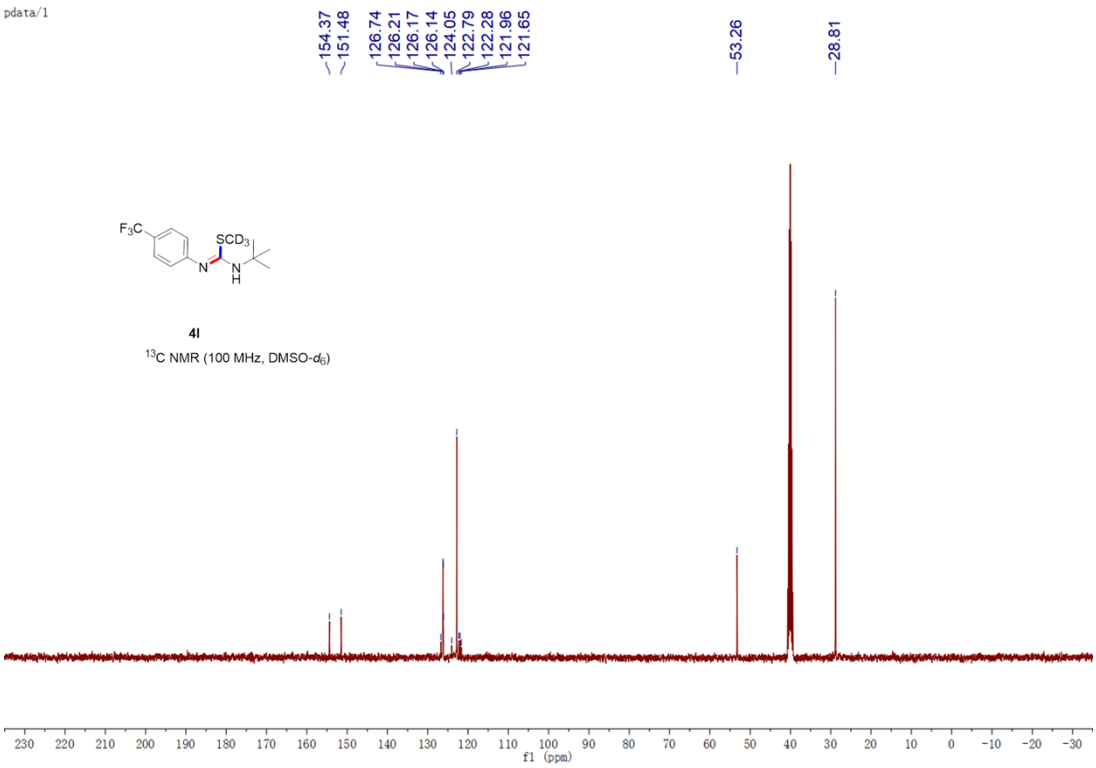


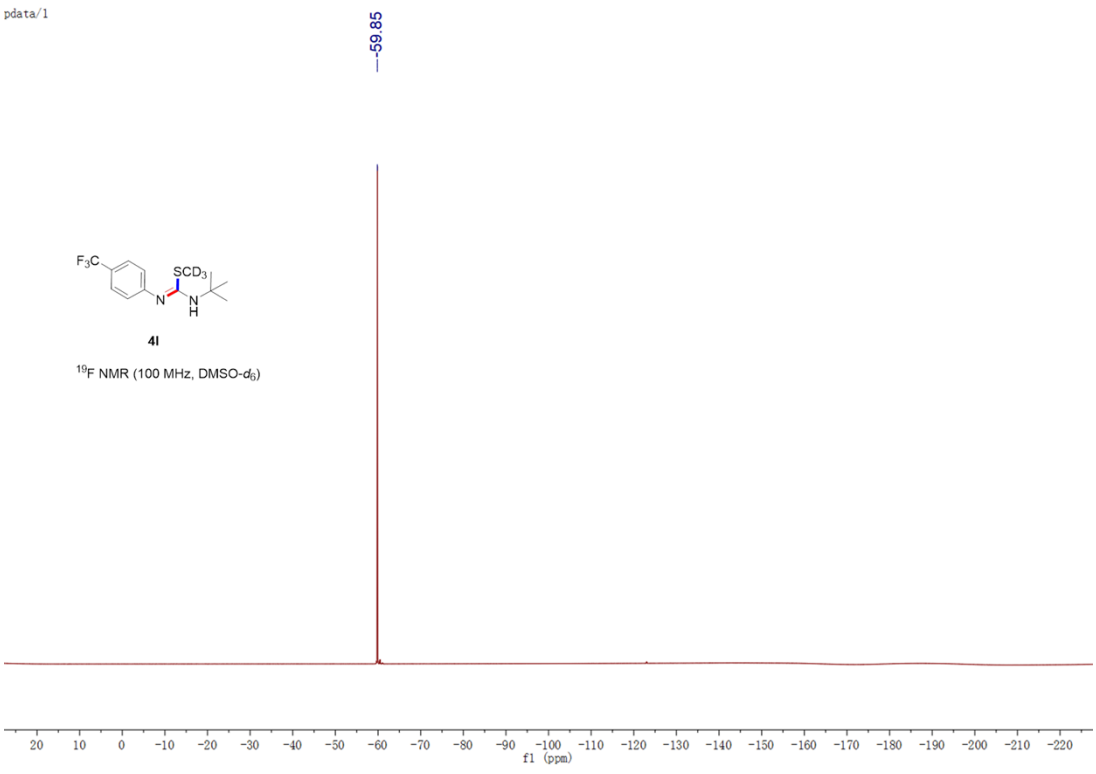
4l

^{1}H NMR (400 MHz, DMSO-d_6)



4l

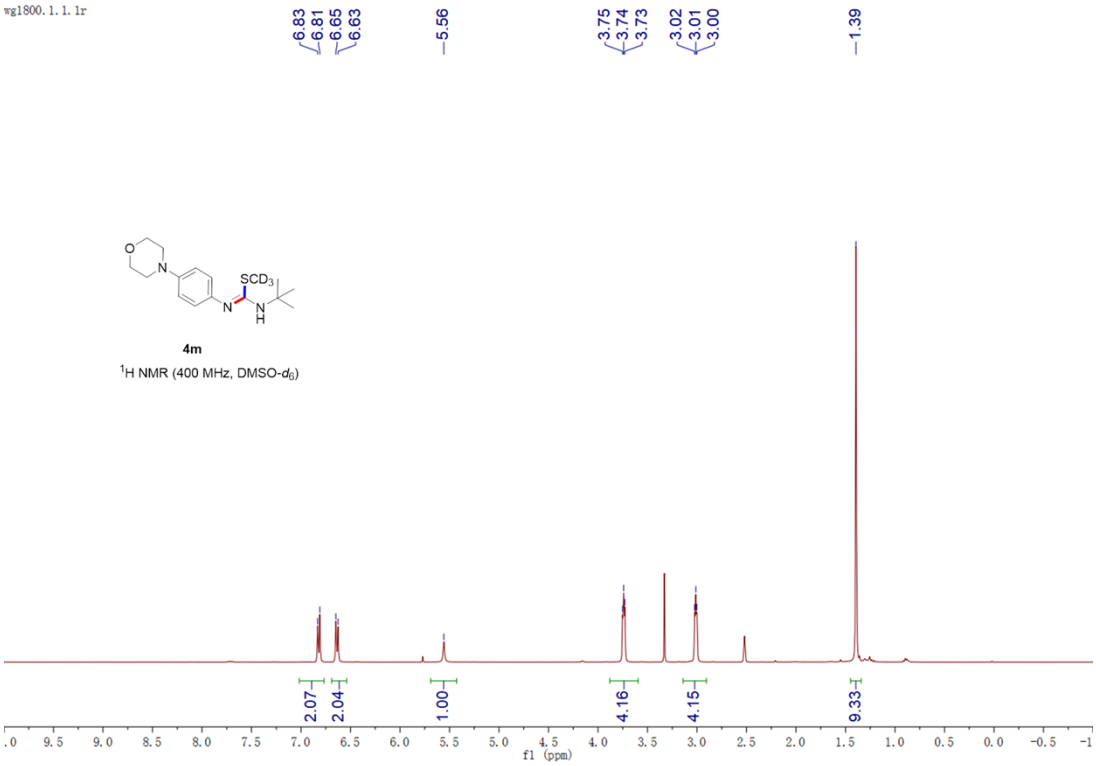
^{13}C NMR (100 MHz, DMSO-d_6)



4l

^{19}F NMR (100 MHz, DMSO-d_6)

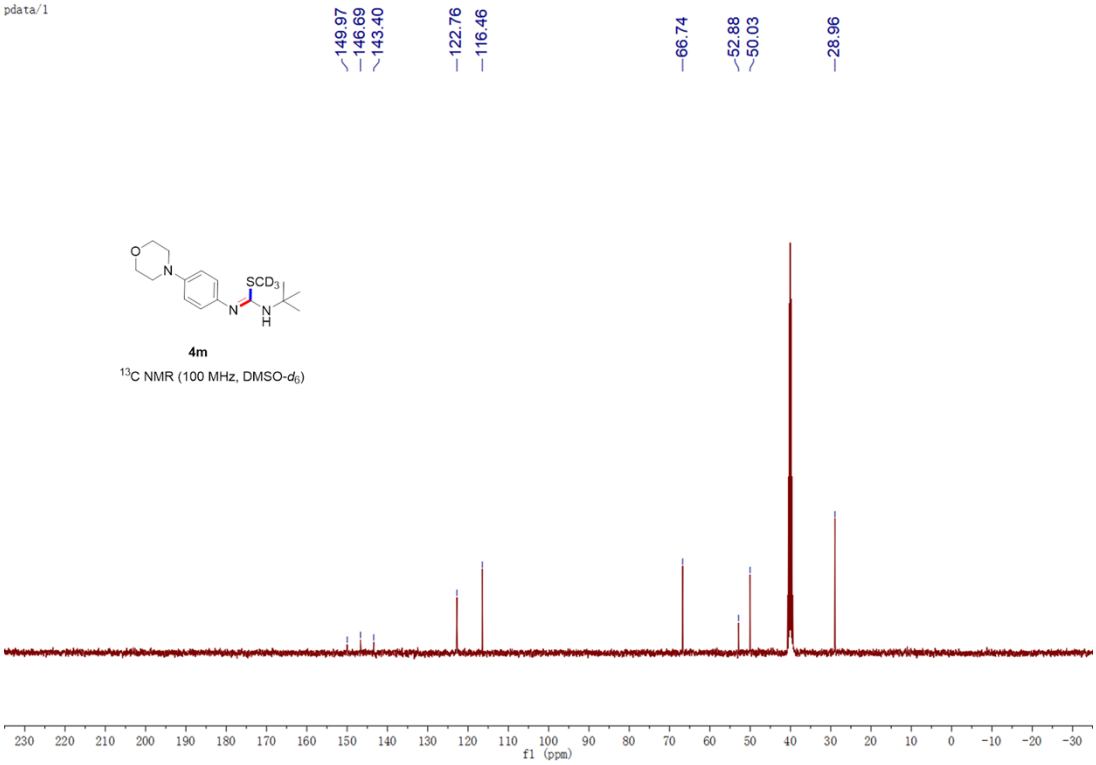
wg1800.1.1.1r



4m

^{1}H NMR (400 MHz, DMSO-d_6)

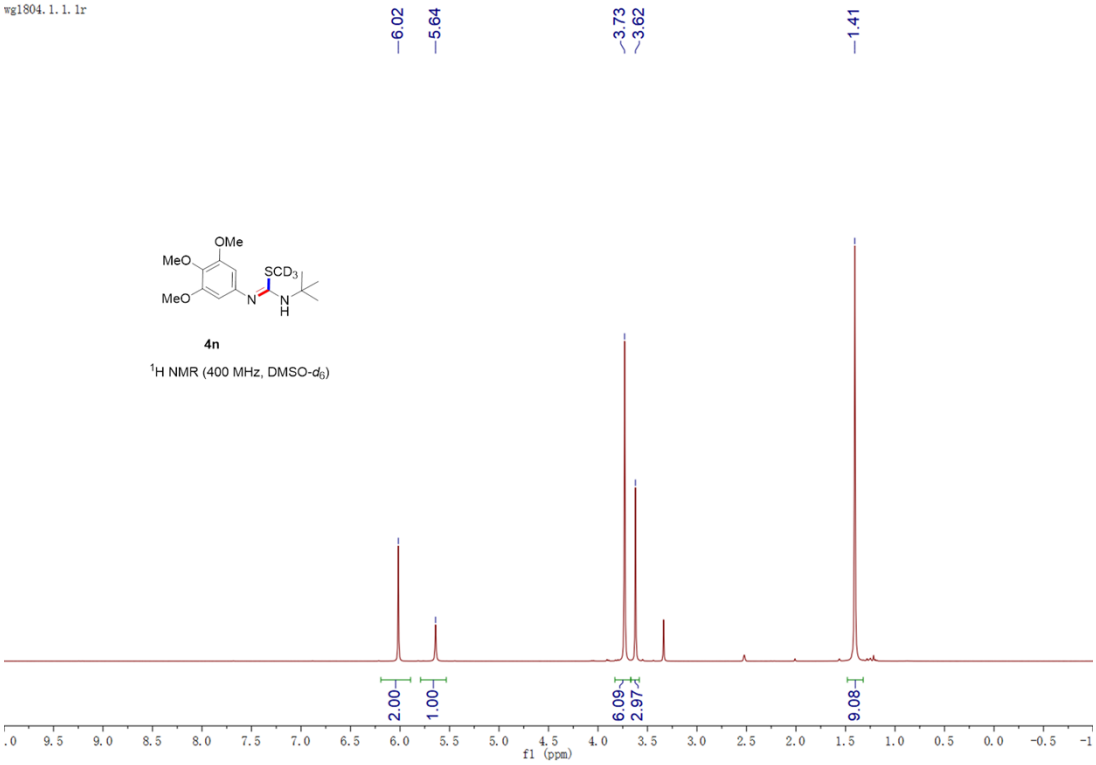
pdata/1



4m

^{13}C NMR (100 MHz, DMSO-d_6)

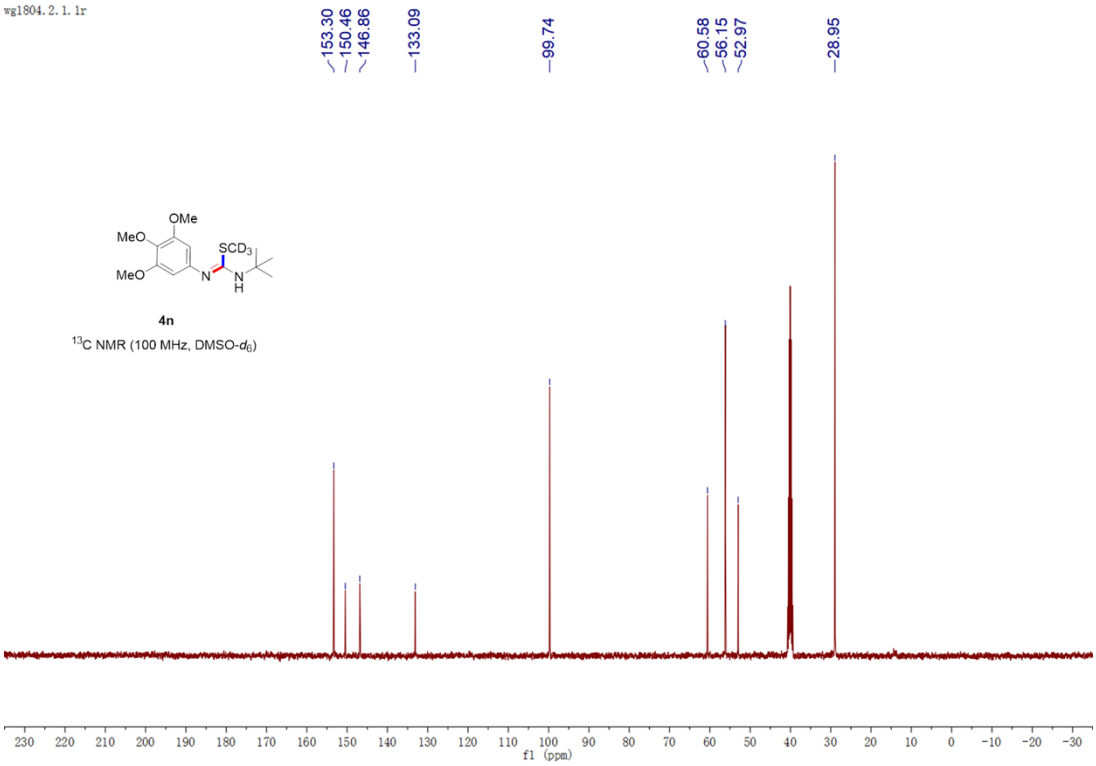
wg1804.1.1.1r



4n

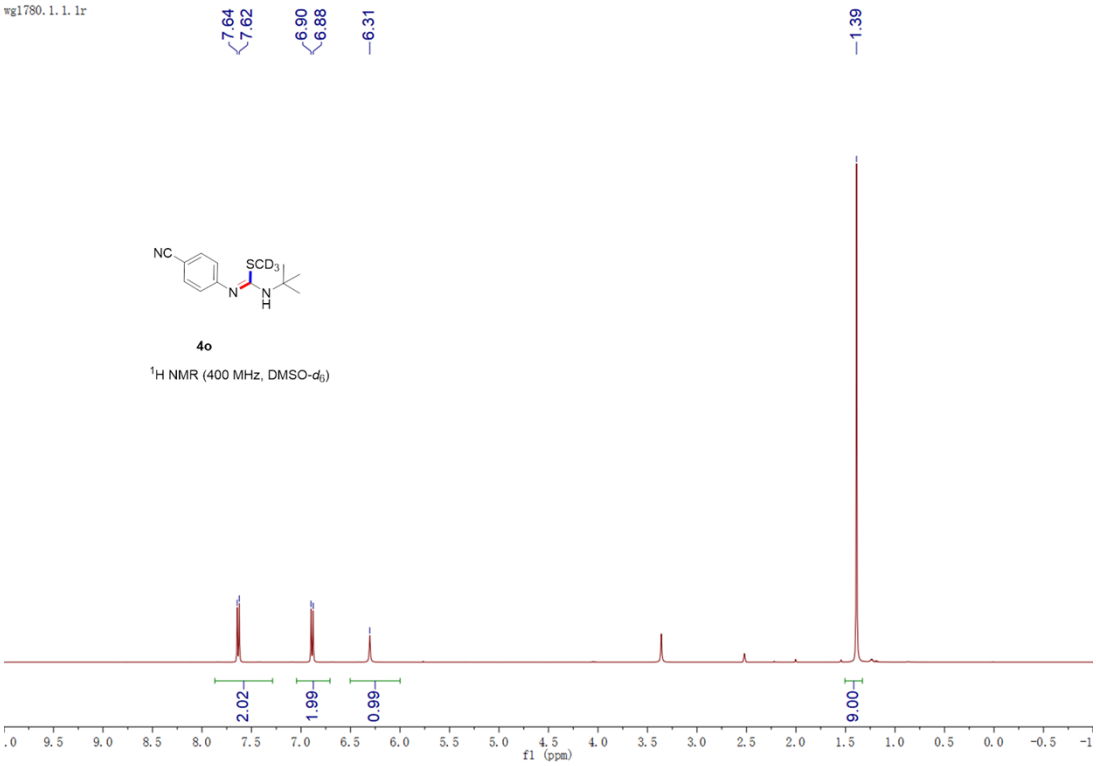
^{1}H NMR (400 MHz, DMSO-d_6)

wg1804.2.1.1r



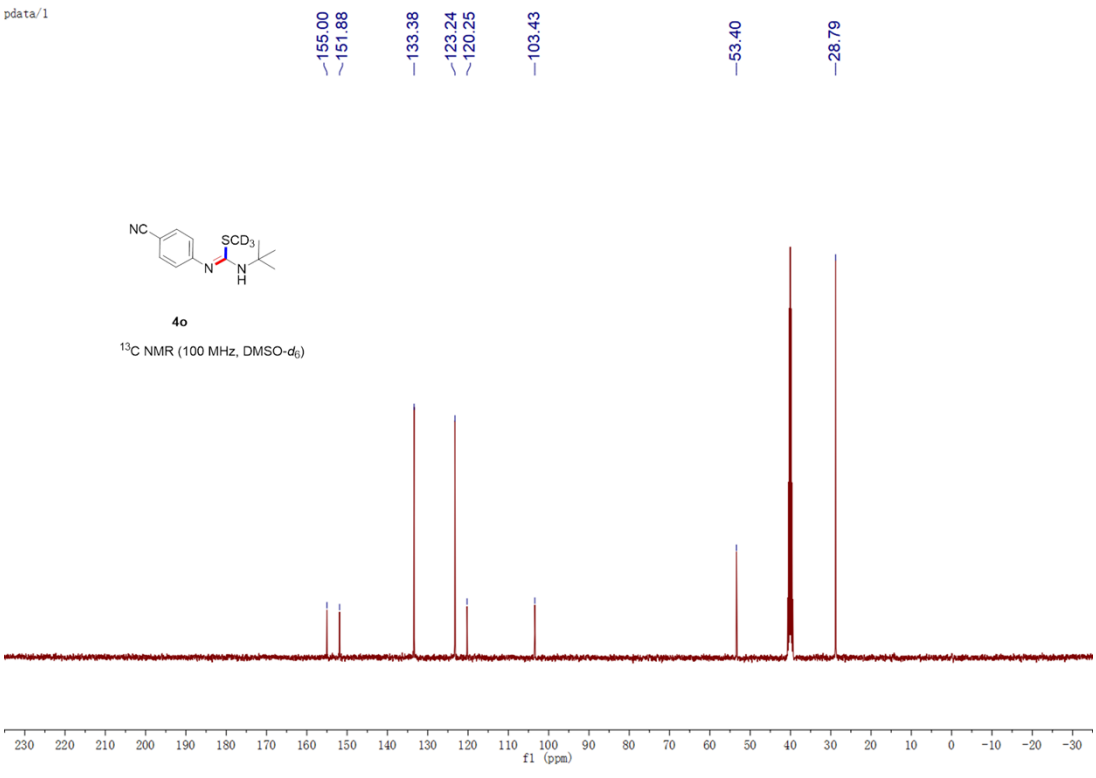
4n

^{13}C NMR (100 MHz, DMSO-d_6)



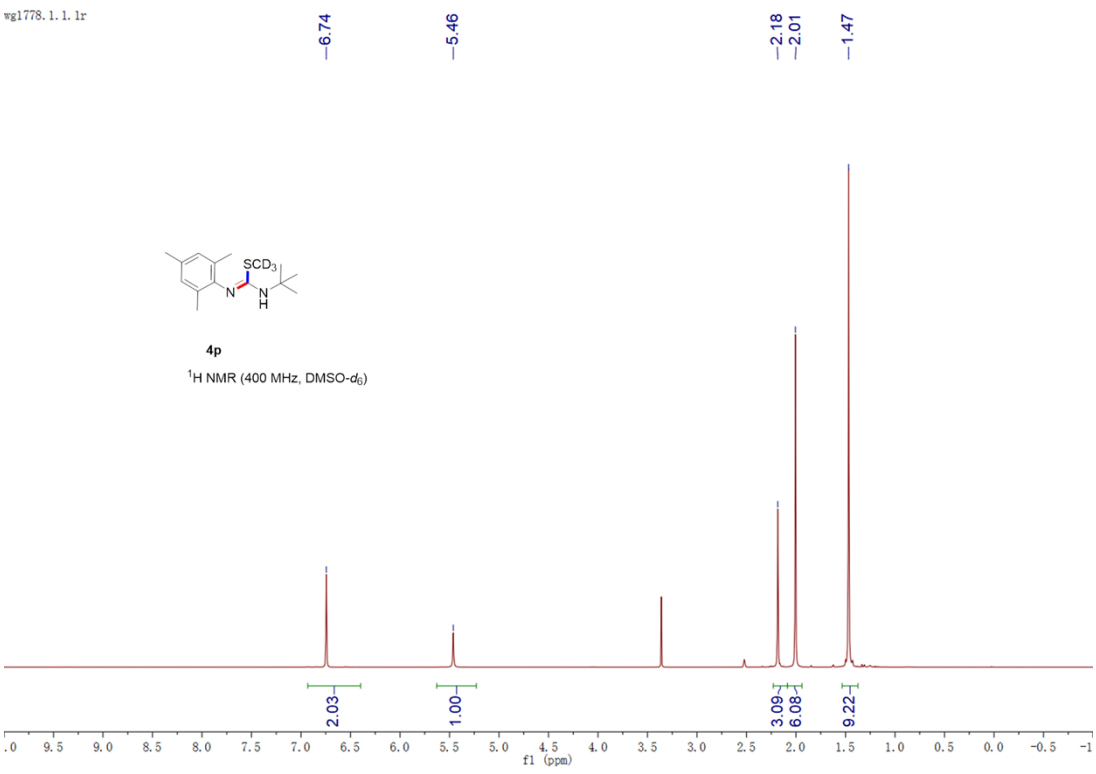
4o

^{1}H NMR (400 MHz, DMSO-d_6)



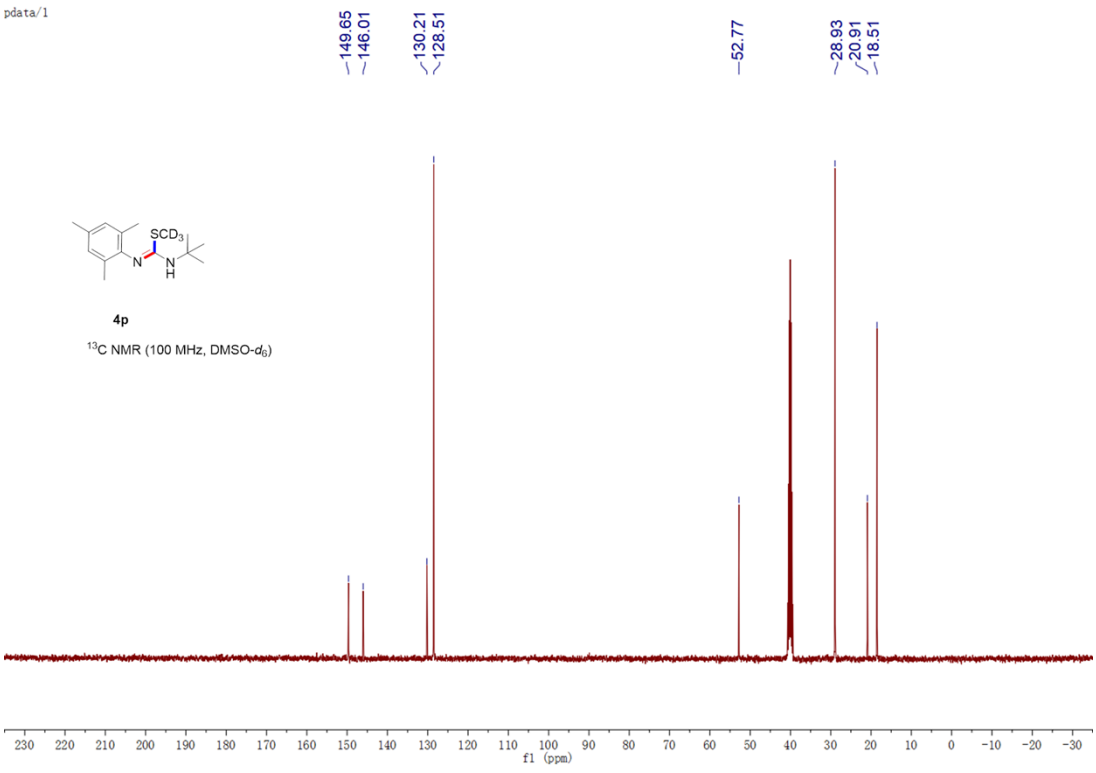
4o

^{13}C NMR (100 MHz, DMSO-d_6)



4p

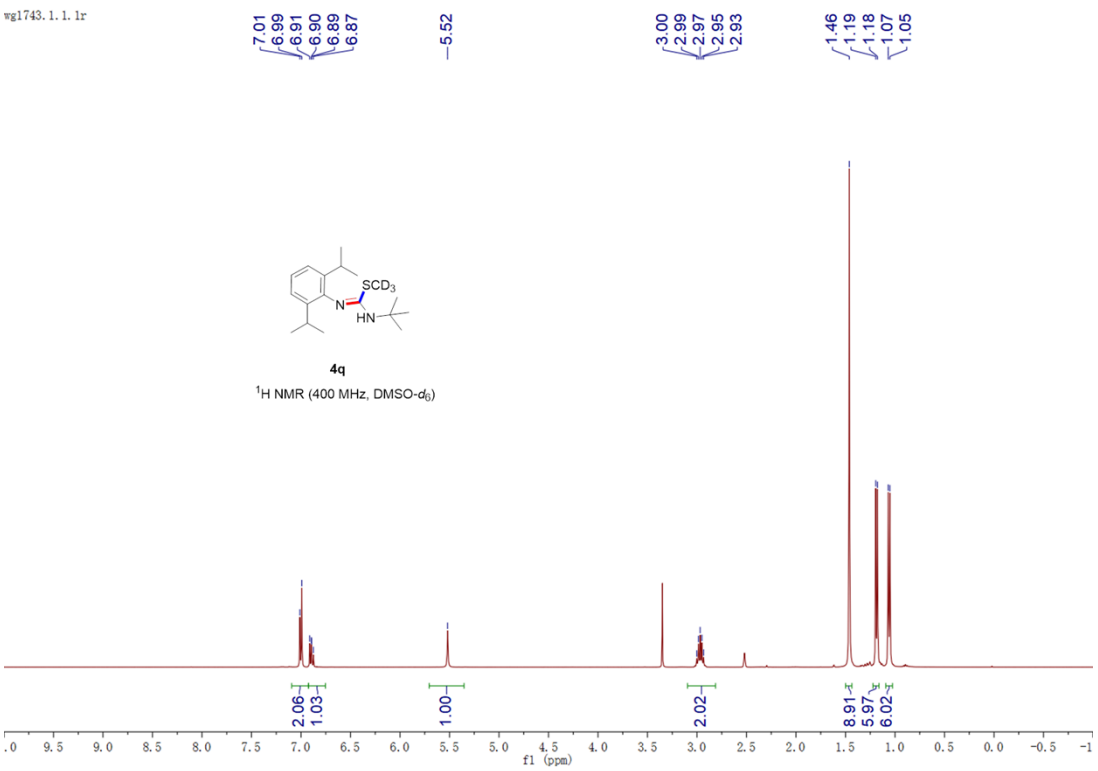
^{1}H NMR (400 MHz, DMSO-d_6)



4p

^{13}C NMR (100 MHz, DMSO-d_6)

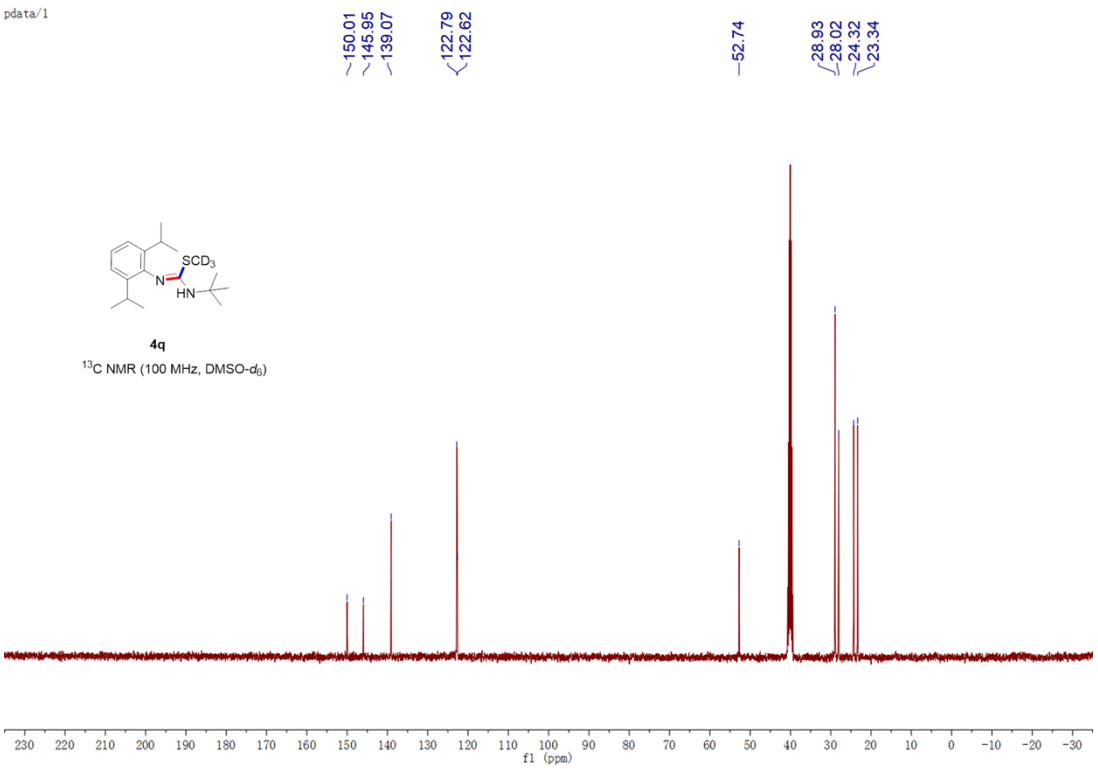
wgl743.1.1.1r



4q

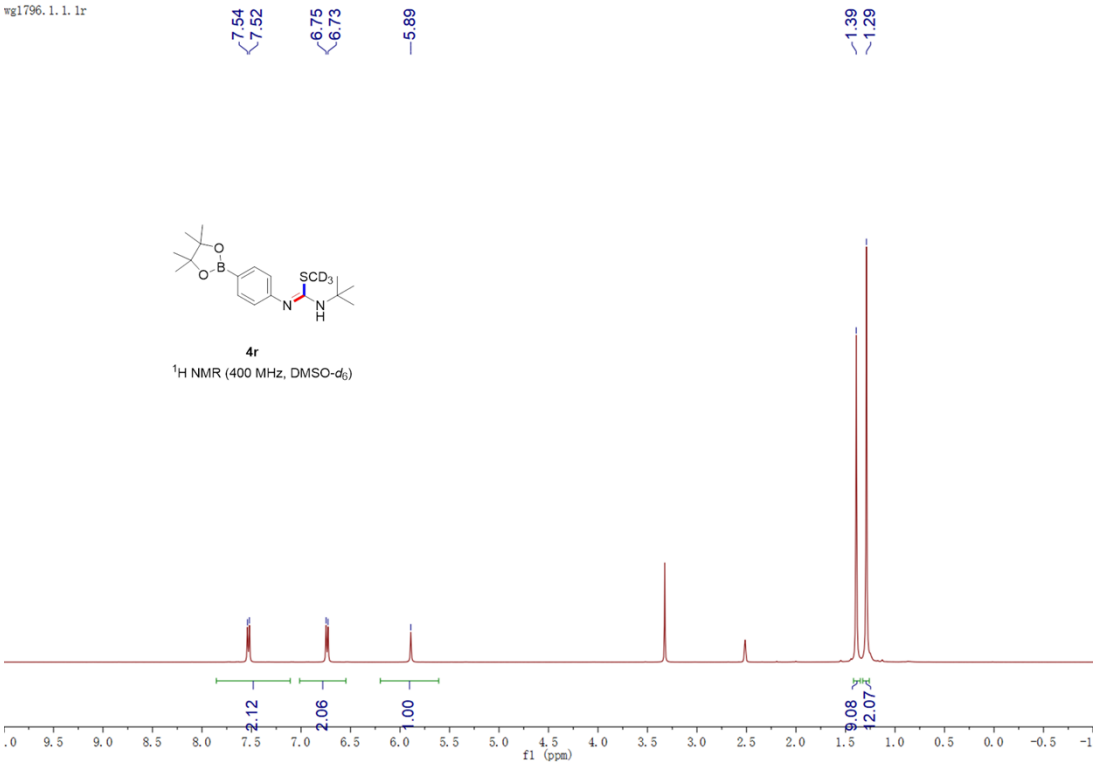
^{1}H NMR (400 MHz, DMSO-d_6)

pdata/1



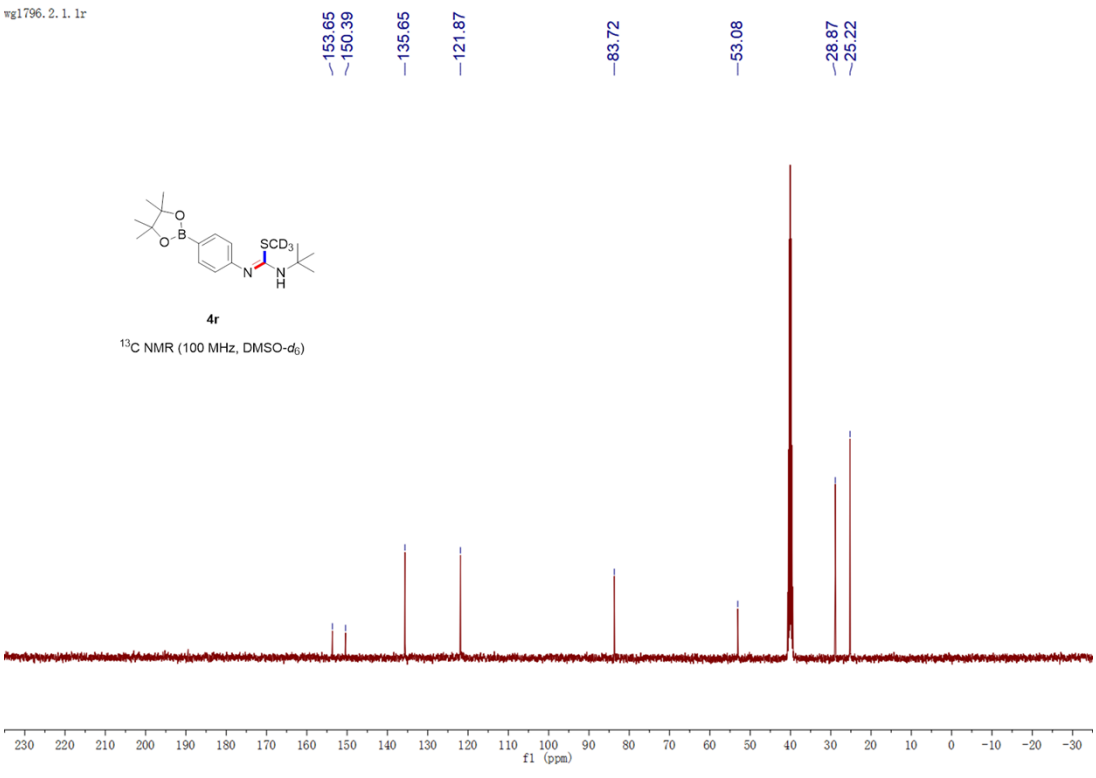
4q

^{13}C NMR (100 MHz, DMSO-d_6)



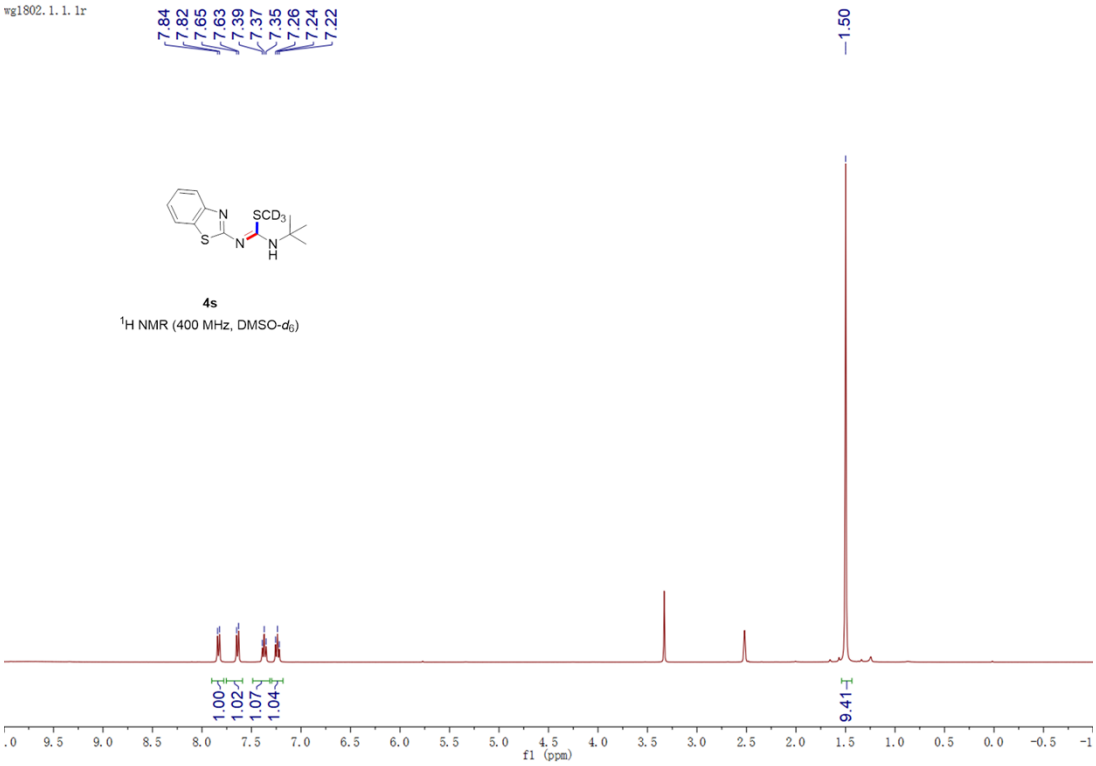
4r

^{1}H NMR (400 MHz, DMSO-d_6)



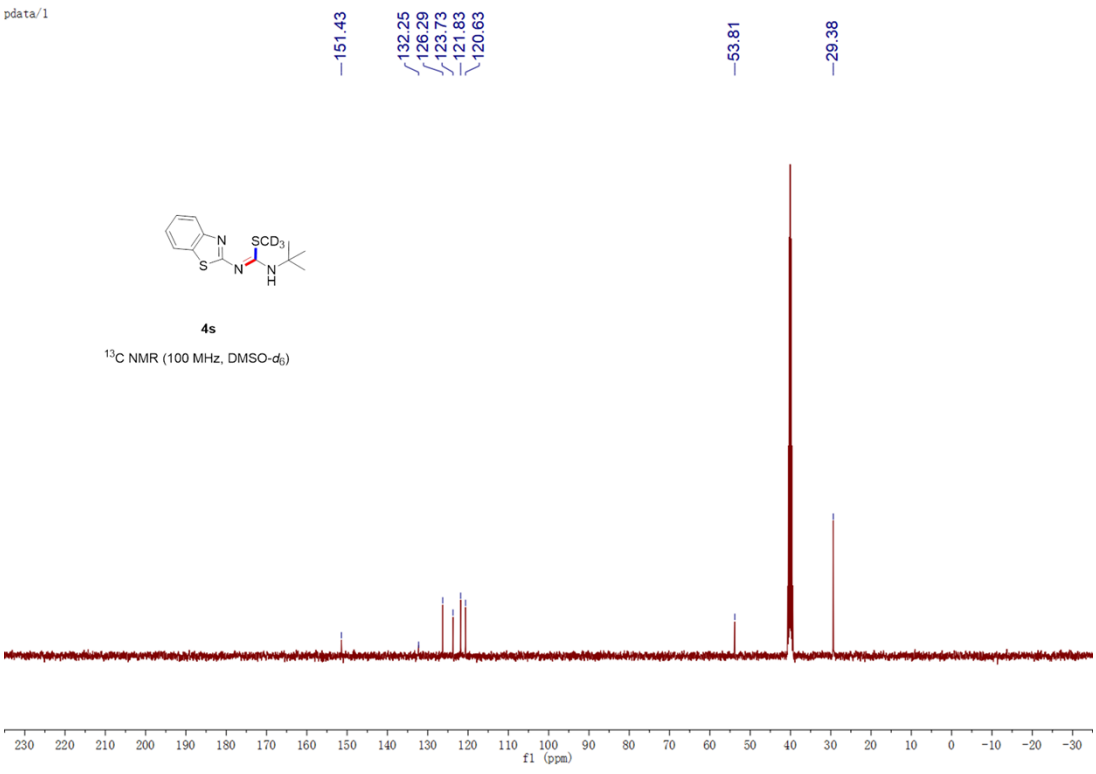
4r

^{13}C NMR (100 MHz, DMSO-d_6)



4s

^{1}H NMR (400 MHz, DMSO-d_6)



4s

^{13}C NMR (100 MHz, DMSO-d_6)

wg1829new.1.1.1r

8.90
8.83
7.81
7.79
7.73
7.71
7.44
7.42
7.41
7.40
7.39
7.38
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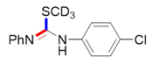
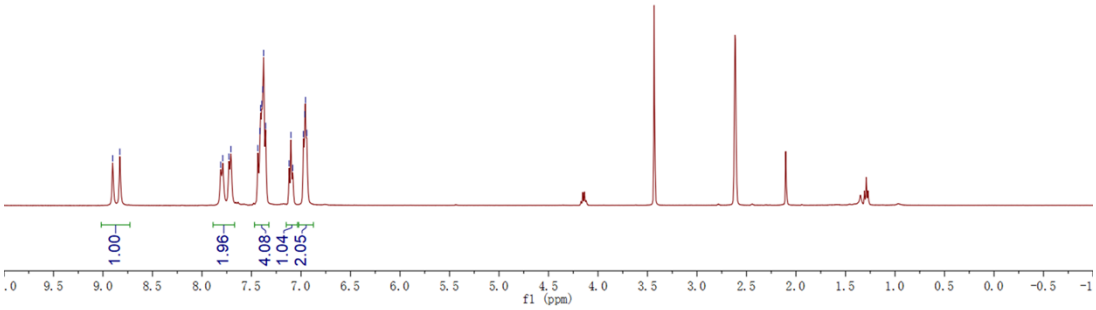
7.10
7.08
6.98
6.96
6.95
6.94



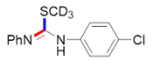
5a

^{1}H NMR (400 MHz, DMSO-d_6)



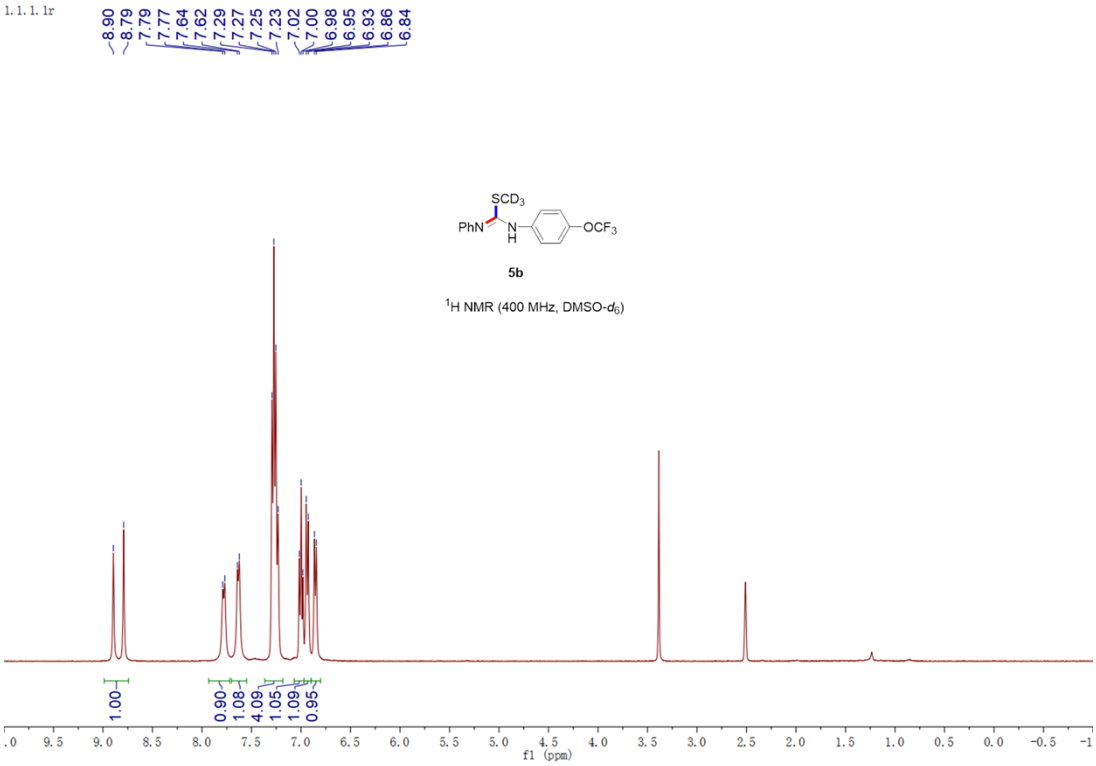
C.1.1.1r

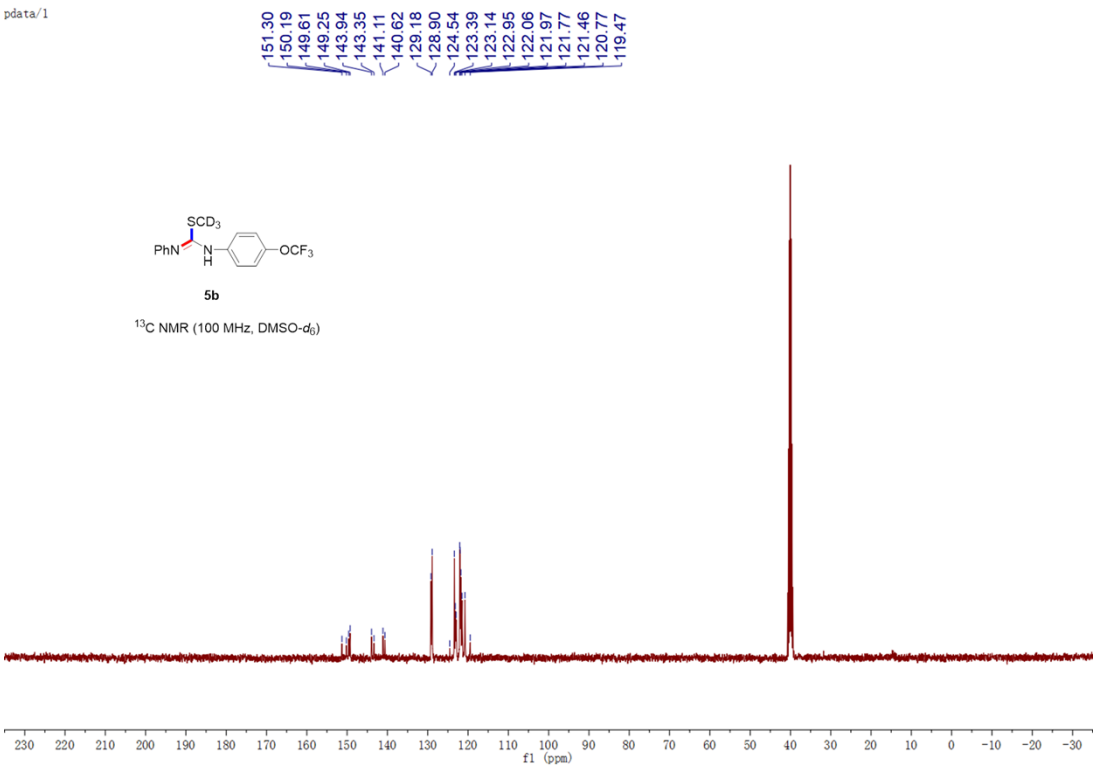
149.64
148.91
141.13
140.35
129.21
129.07
128.93
128.76
123.83
123.14
122.93
121.98
121.79
120.74



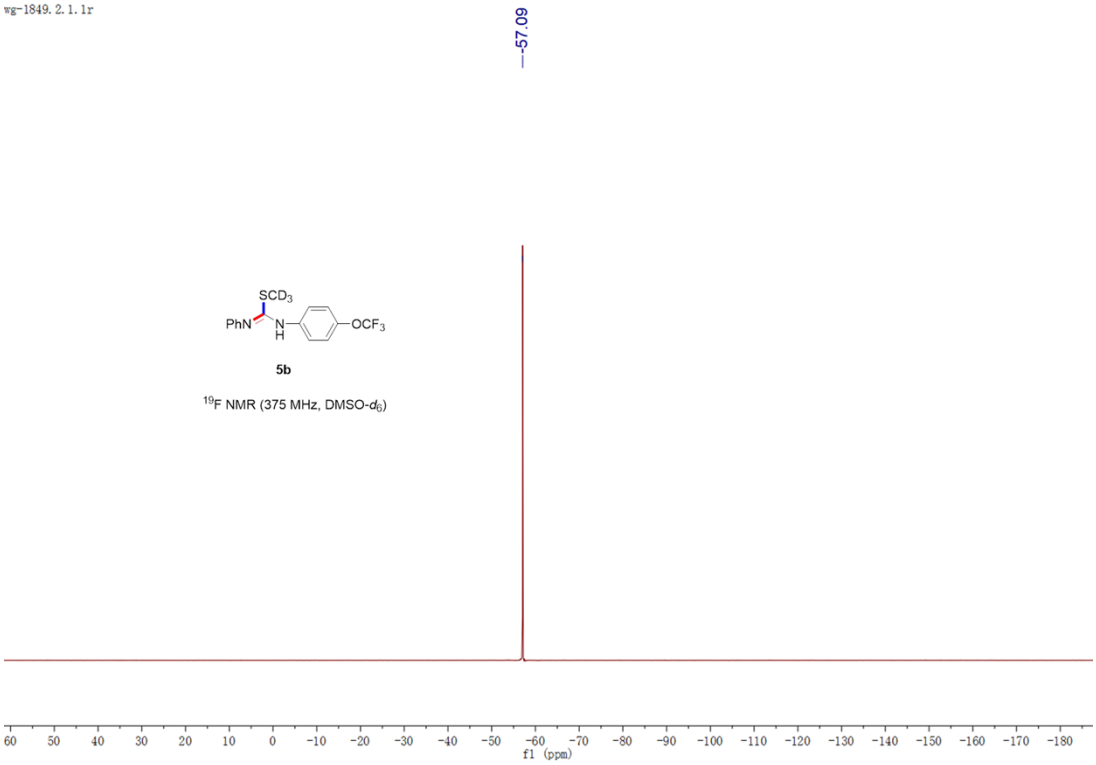
5a

^{13}C NMR (100 MHz, DMSO-d_6)


1.1.1.1r
8.90
8.79
7.79
7.77
7.64
7.62
7.29
7.27
7.25
7.23
7.02
7.00
6.98
6.95
6.93
6.86
6.84
SCD3
PhN
N
H
OCF3
5b
1H NMR (400 MHz, DMSO-d6)
1.00
0.90
1.08
4.09
1.05
1.09
0.95
f1 (ppm)


pdata/1
151.30
150.19
149.61
149.25
143.94
143.35
141.11
140.62
129.18
128.90
124.54
123.39
123.14
122.95
122.06
121.97
121.77
121.46
120.77
119.47
SCD3
PhN
N
H
OCF3
5b
13C NMR (100 MHz, DMSO-d6)
f1 (ppm)

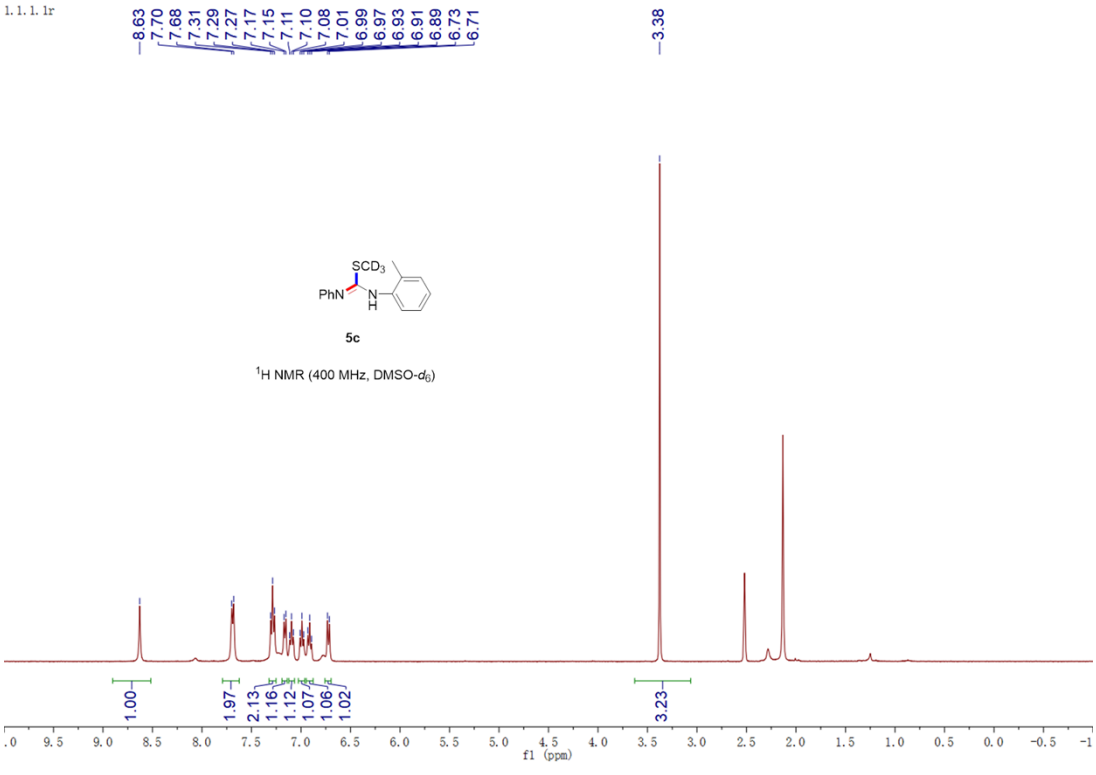
wg-1849.2.1.1r



5b

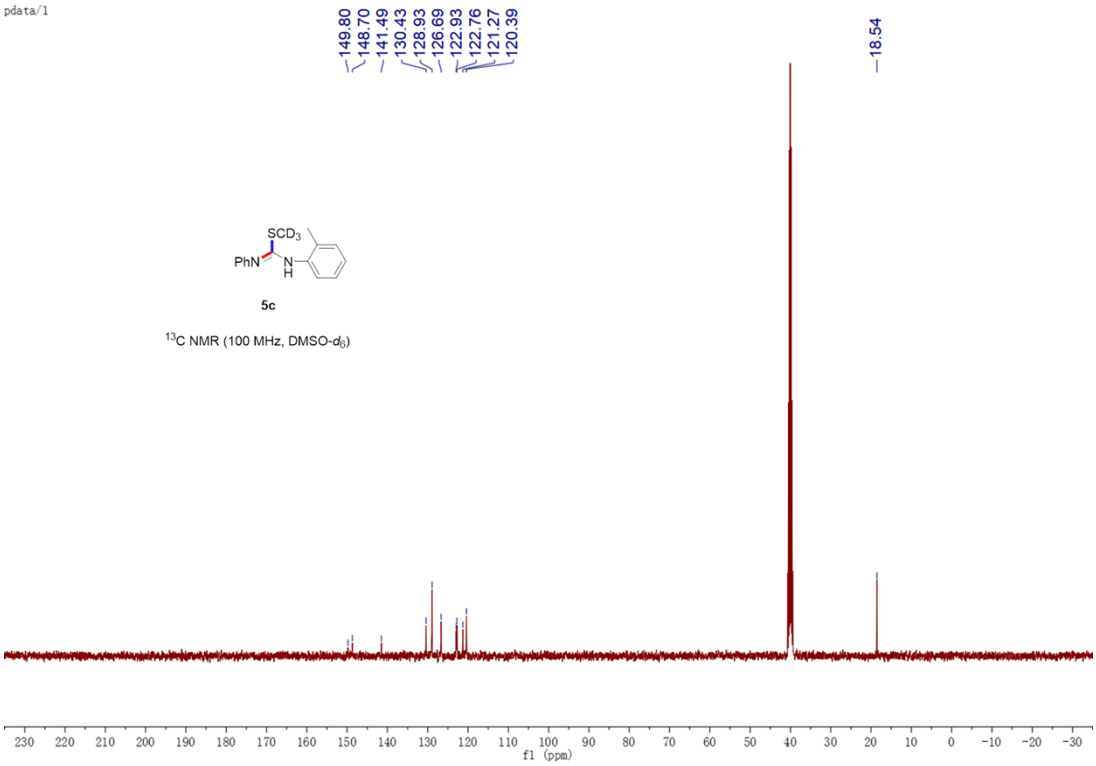
^{19}F NMR (375 MHz, DMSO-d_6)

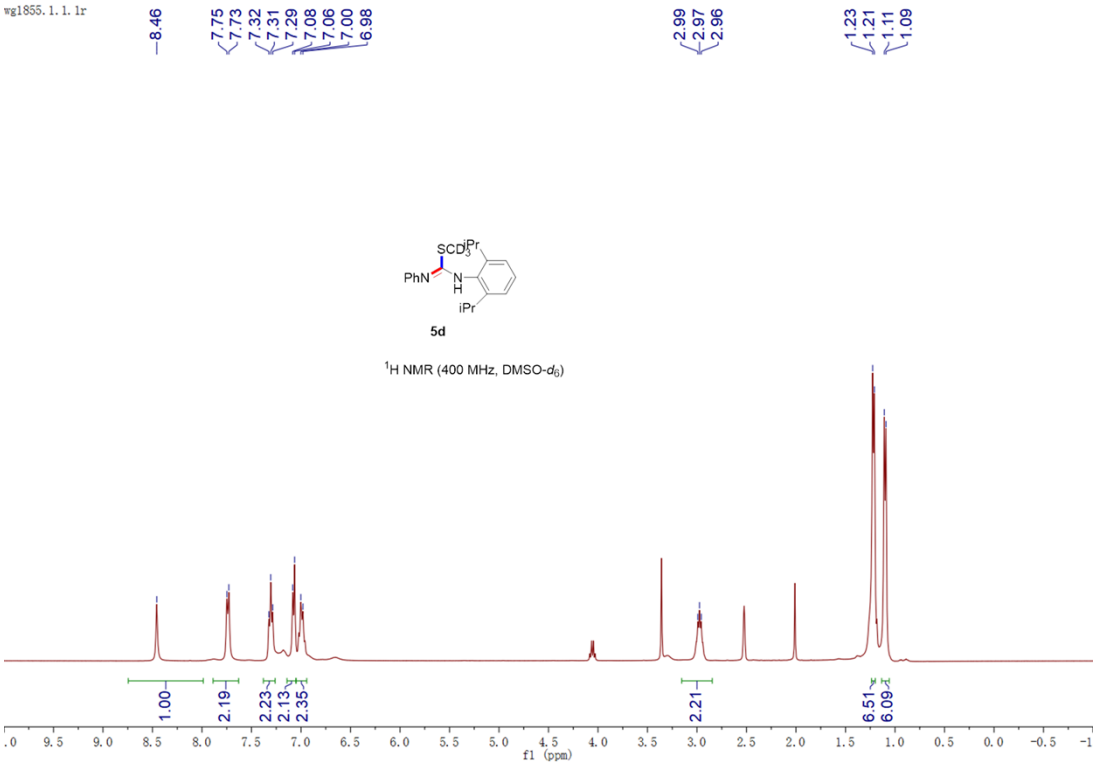
1.1.1.1r



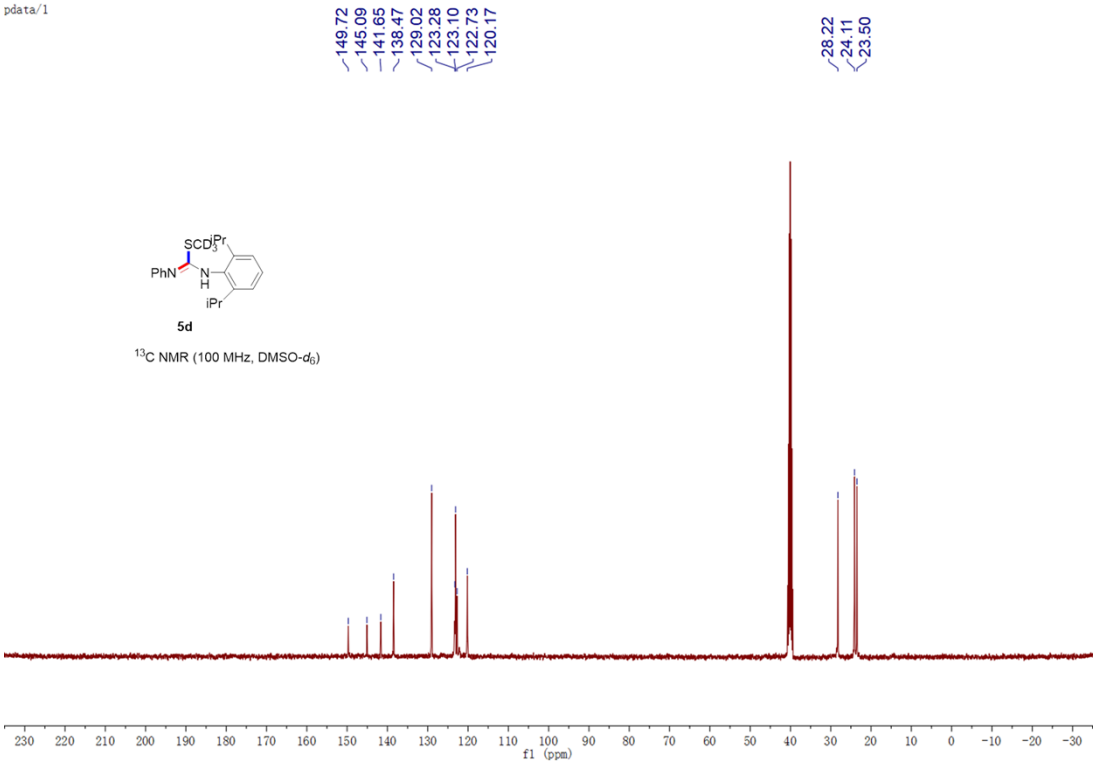
5c

^{1}H NMR (400 MHz, DMSO-d_6)


pdata/1
149.80
148.70
141.49
130.43
128.93
126.69
122.93
122.76
121.27
120.39
18.54
SCD3
PhN
N
H
5c
13C NMR (100 MHz, DMSO-d6)
f1 (ppm)


wg1855.1.1.1r
8.46
7.75
7.73
7.32
7.31
7.29
7.08
7.06
7.00
6.98
2.99
2.97
2.96
1.23
1.21
1.11
1.09
SCD3
iPr
PhN
N
H
iPr
5d
1H NMR (400 MHz, DMSO-d6)
1.00
2.19
2.23
2.13
2.35
2.21
6.51
6.09
f1 (ppm)

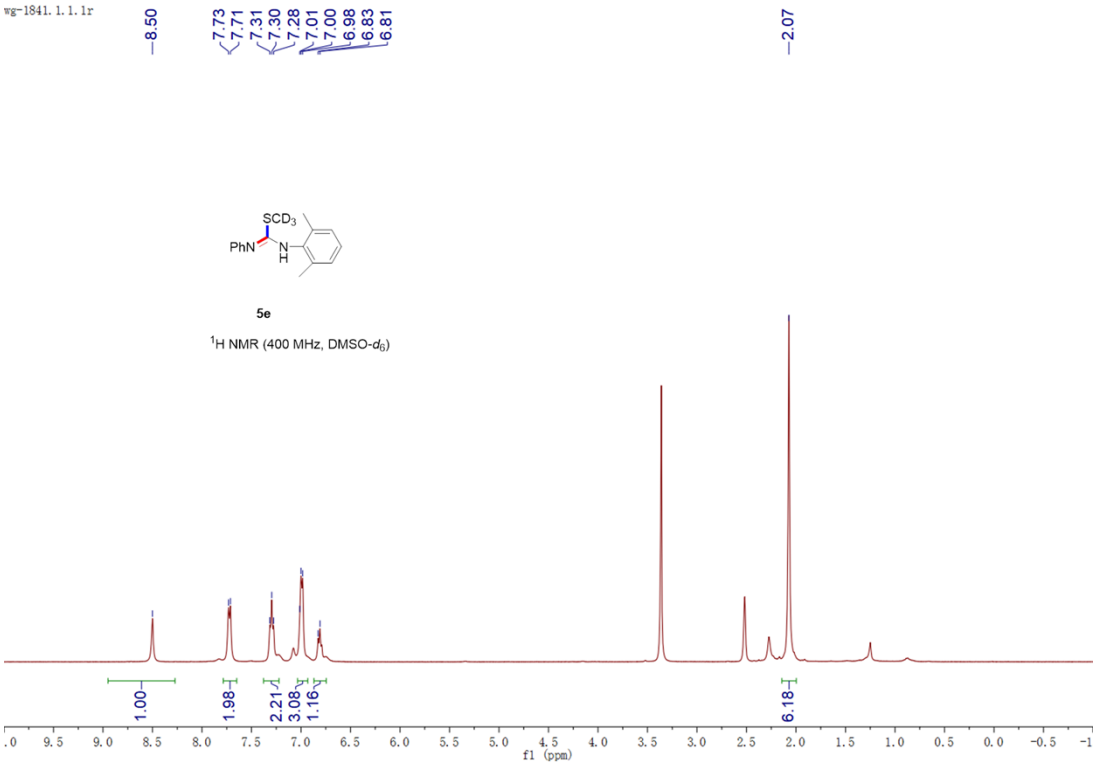
pdata/1



5d

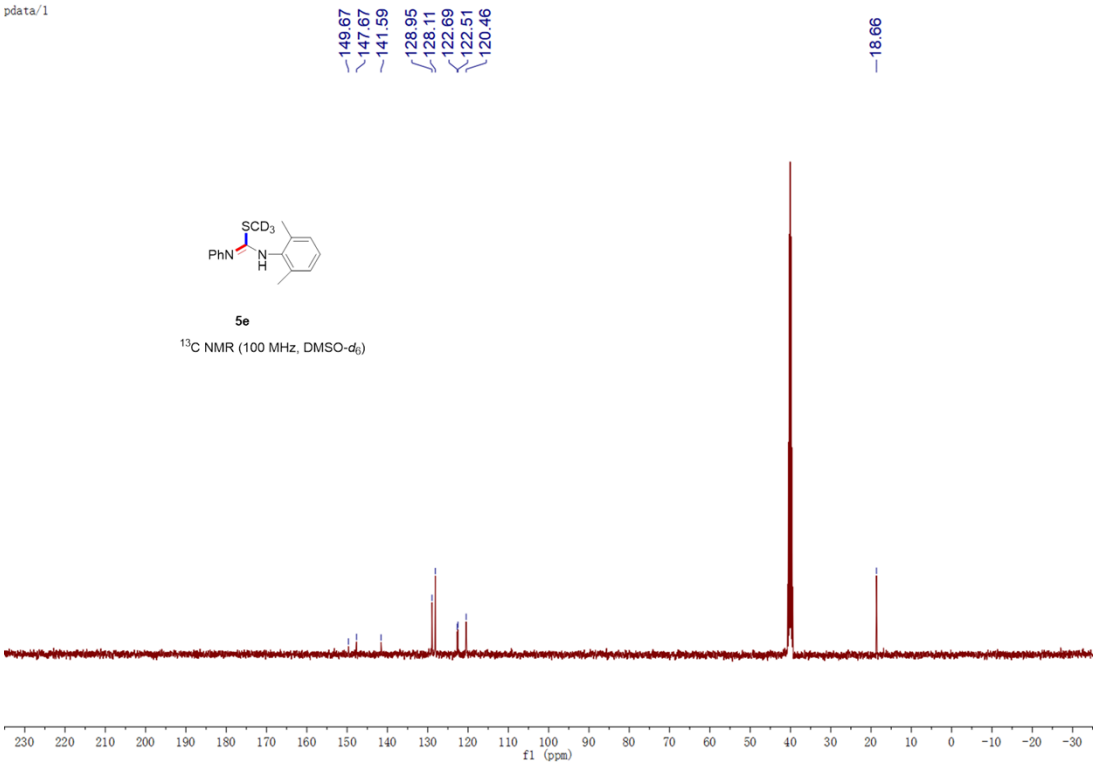
^{13}C NMR (100 MHz, DMSO-d_6)

wg-1841.1.1.1r



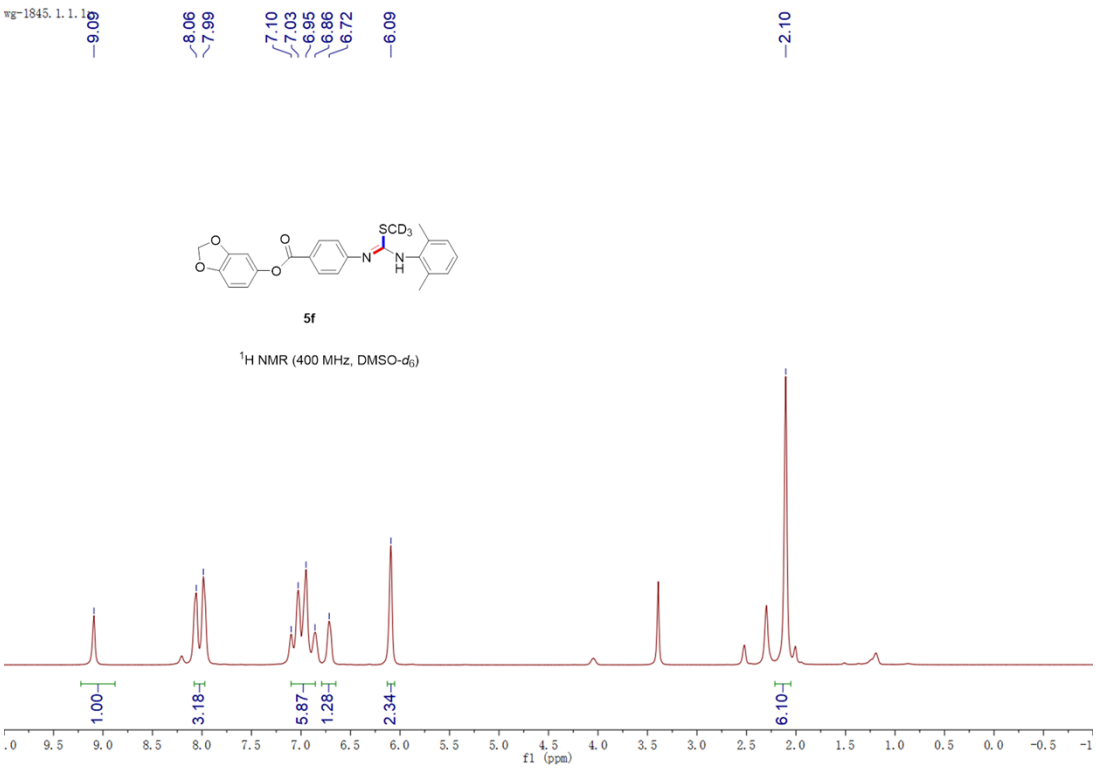
5e

^{1}H NMR (400 MHz, DMSO-d_6)



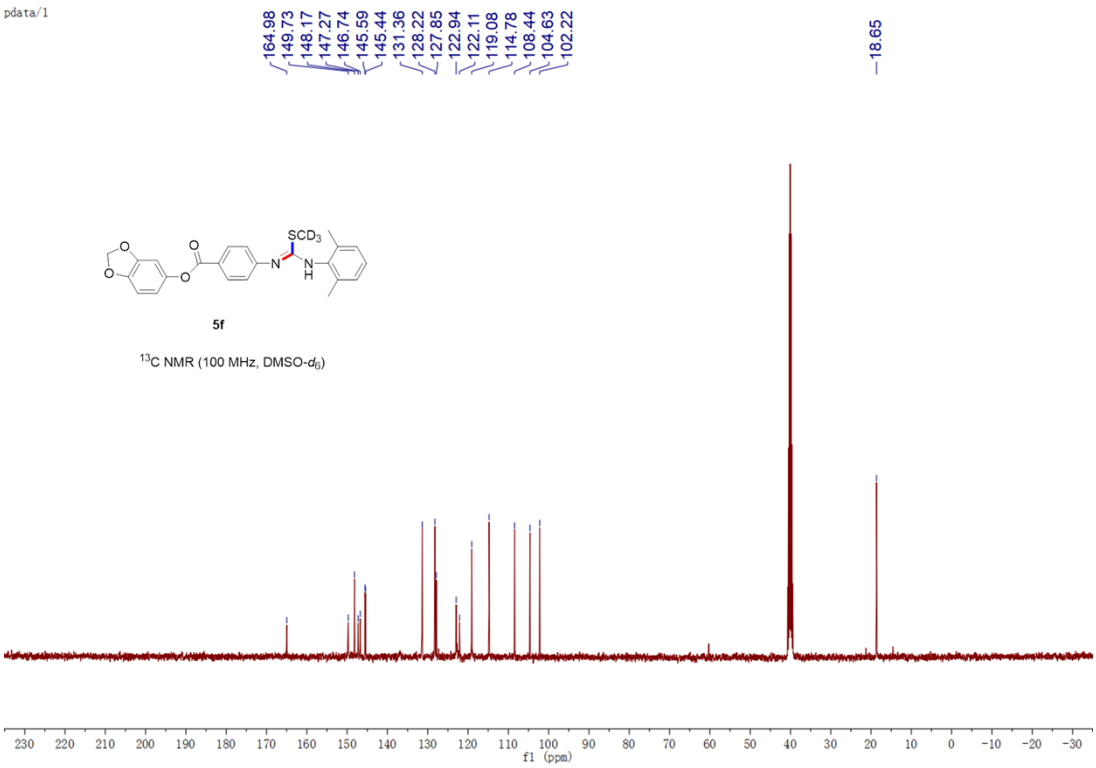
5e

^{13}C NMR (100 MHz, DMSO-d_6)



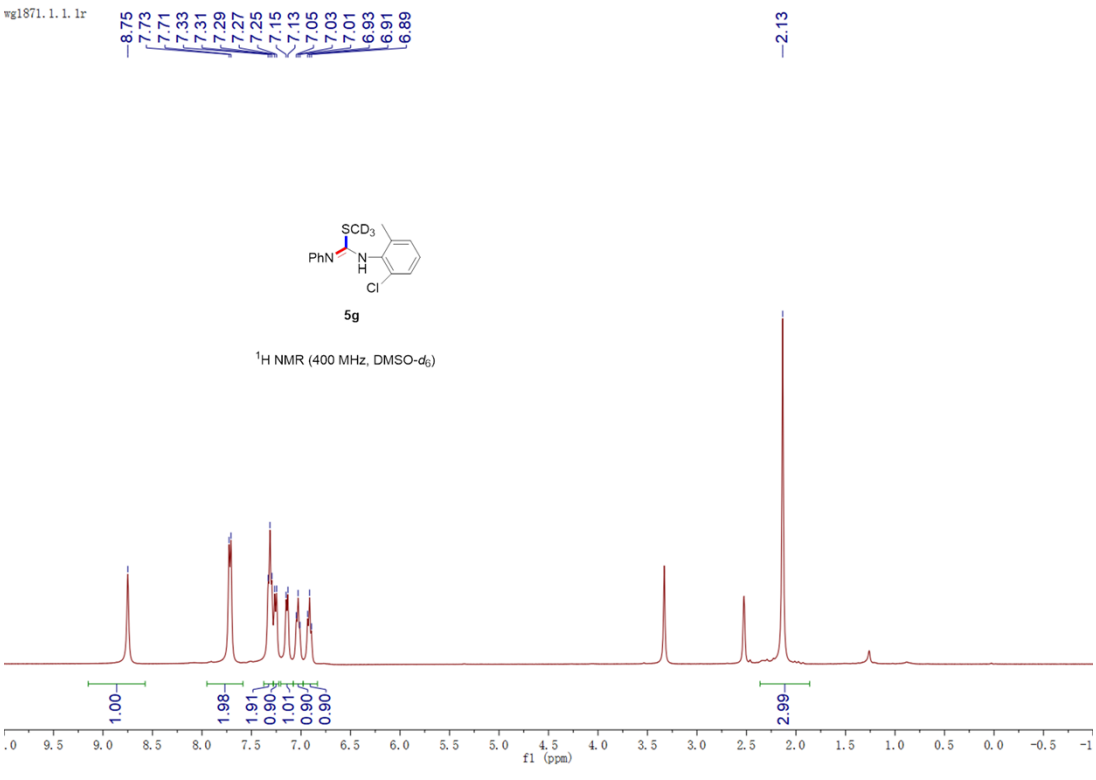
5f

^{1}H NMR (400 MHz, DMSO-d_6)



5f

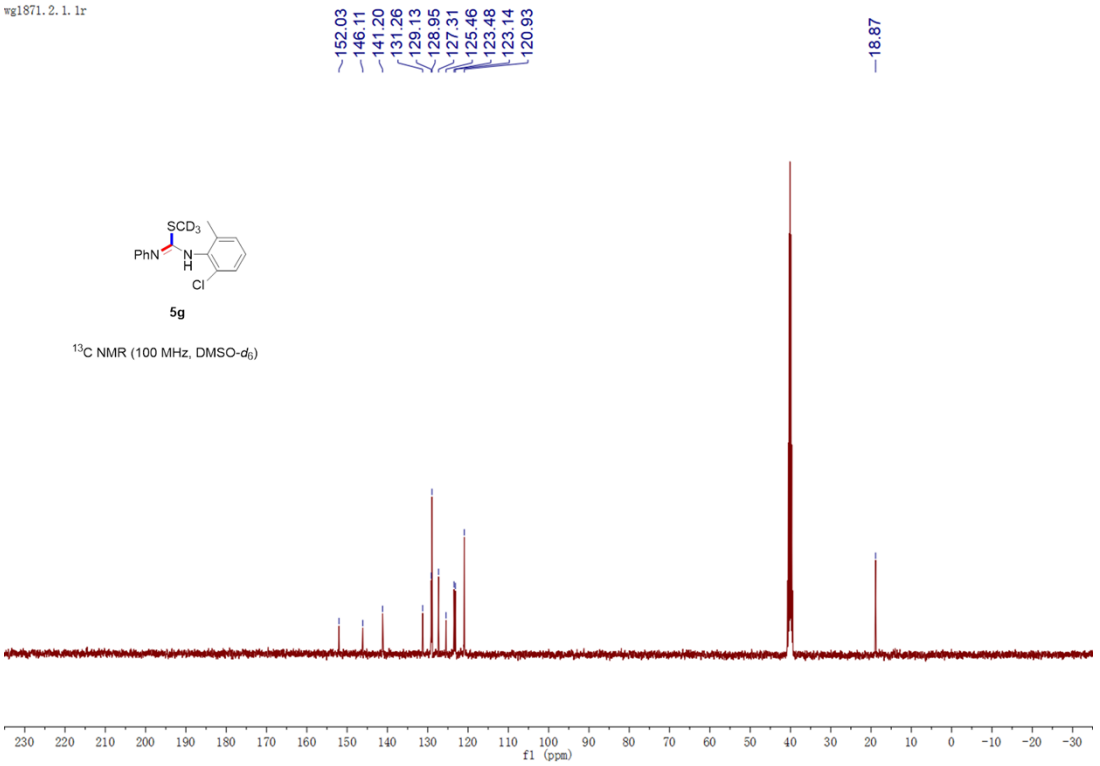
^{13}C NMR (100 MHz, DMSO-d_6)



5g

^{1}H NMR (400 MHz, DMSO-d_6)

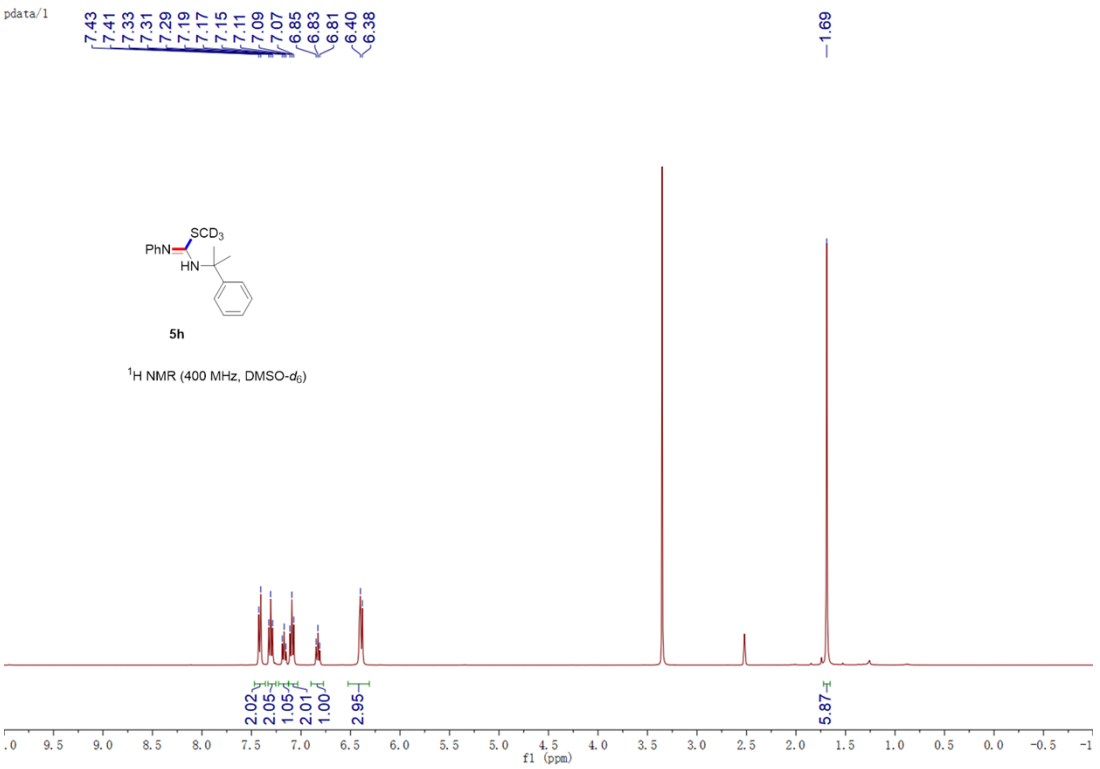
wg1871.2.1.1r



5g

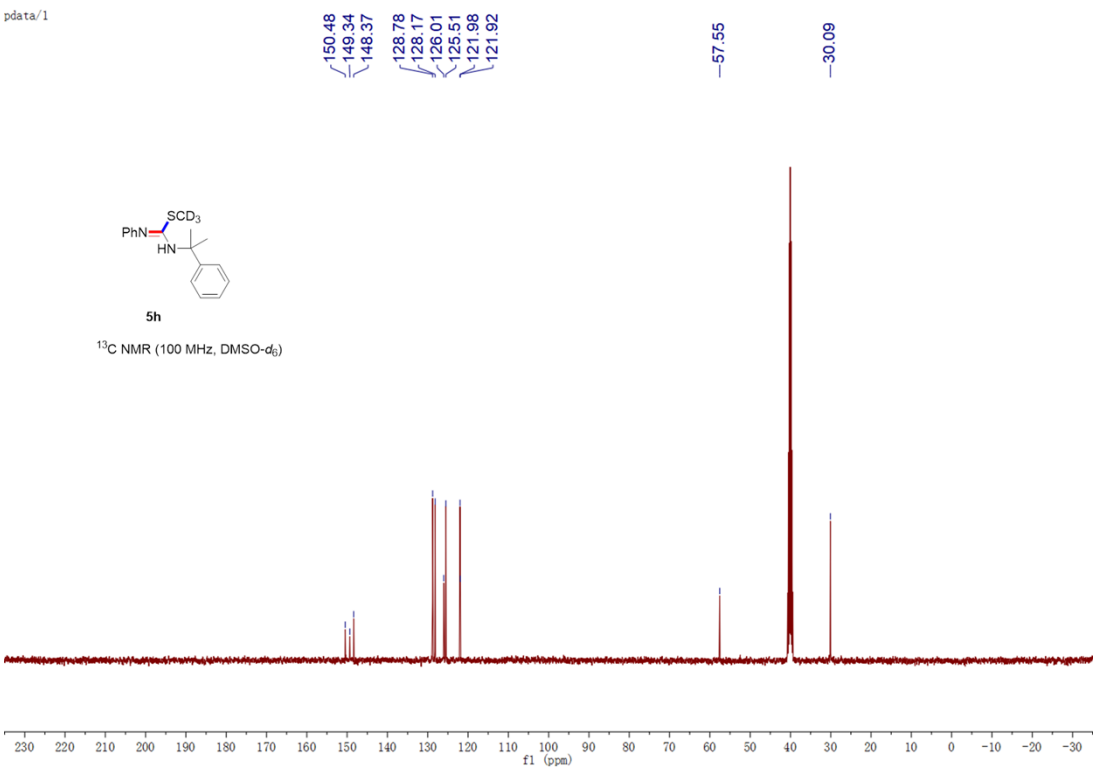
^{13}C NMR (100 MHz, DMSO-d_6)

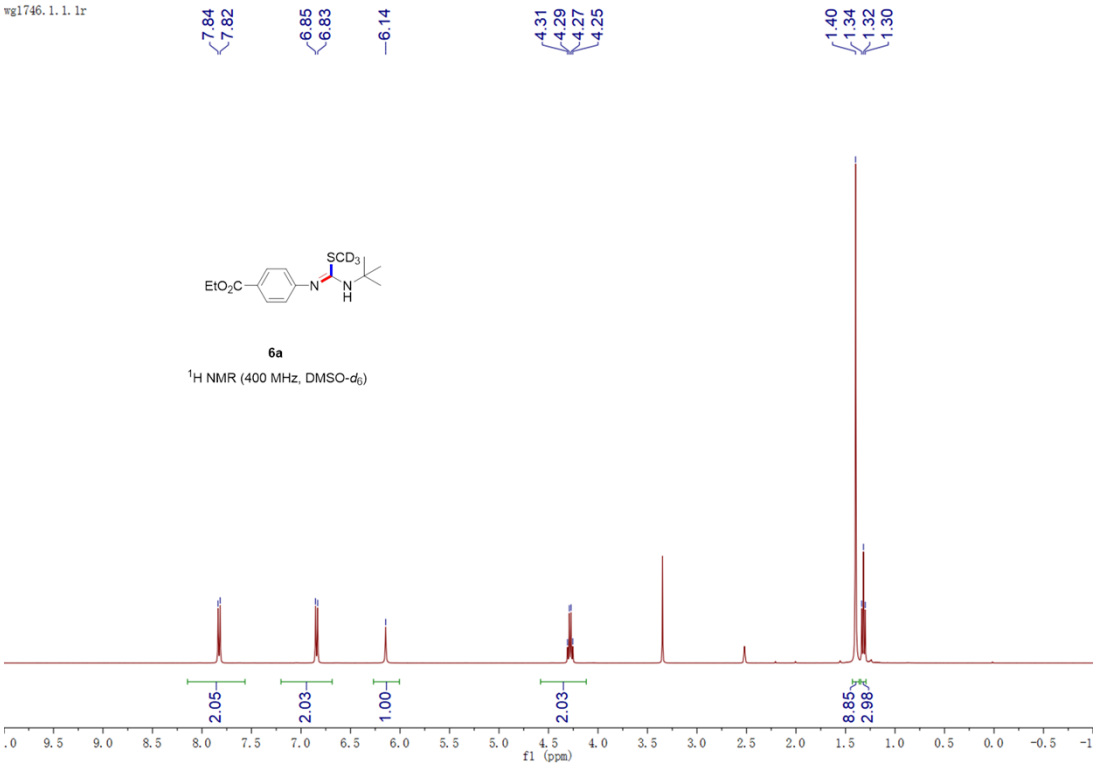
pdata/1

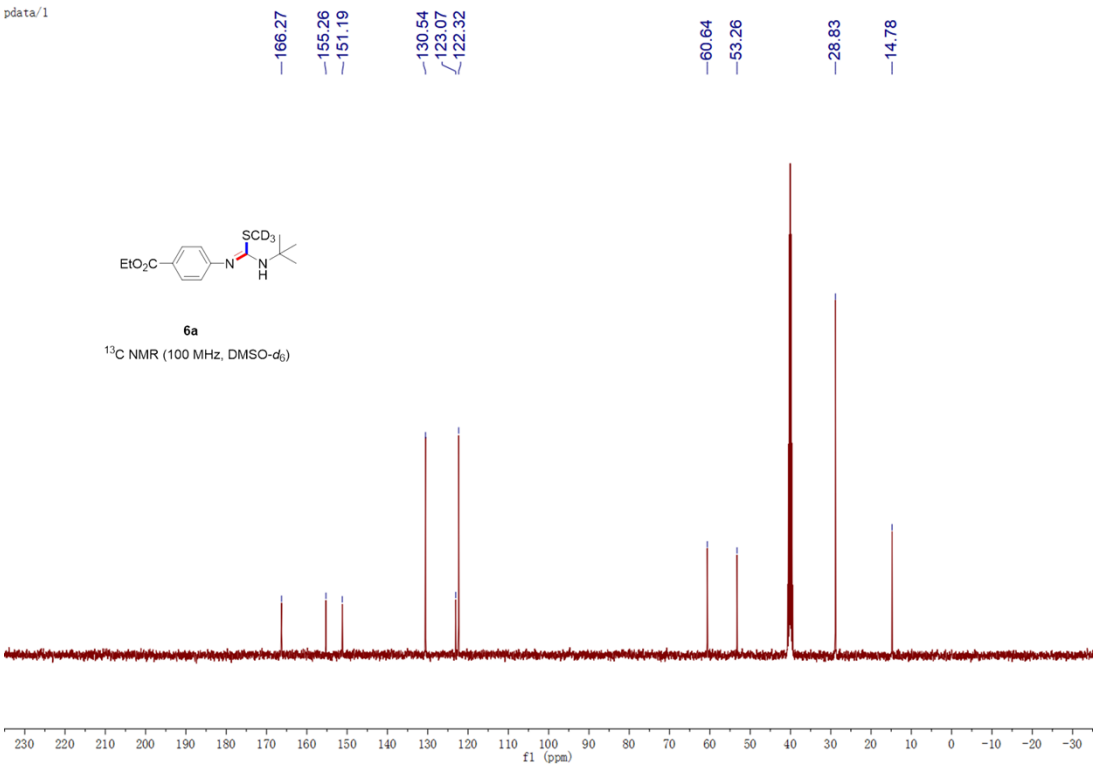


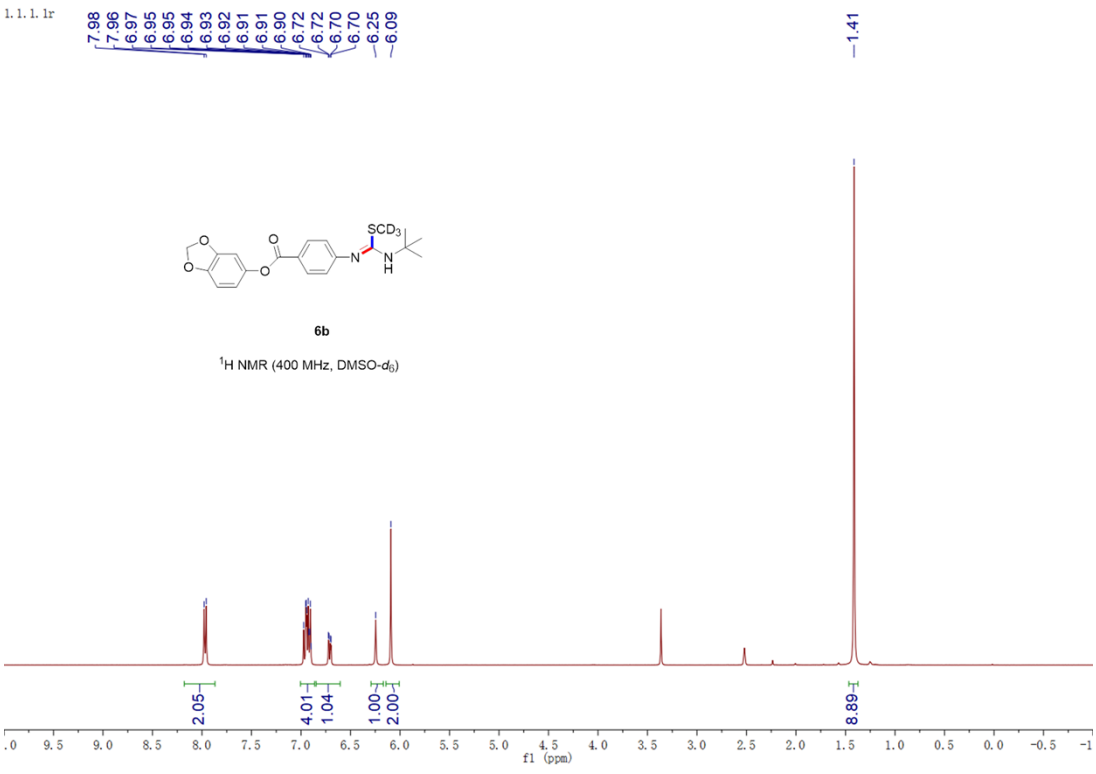
5h

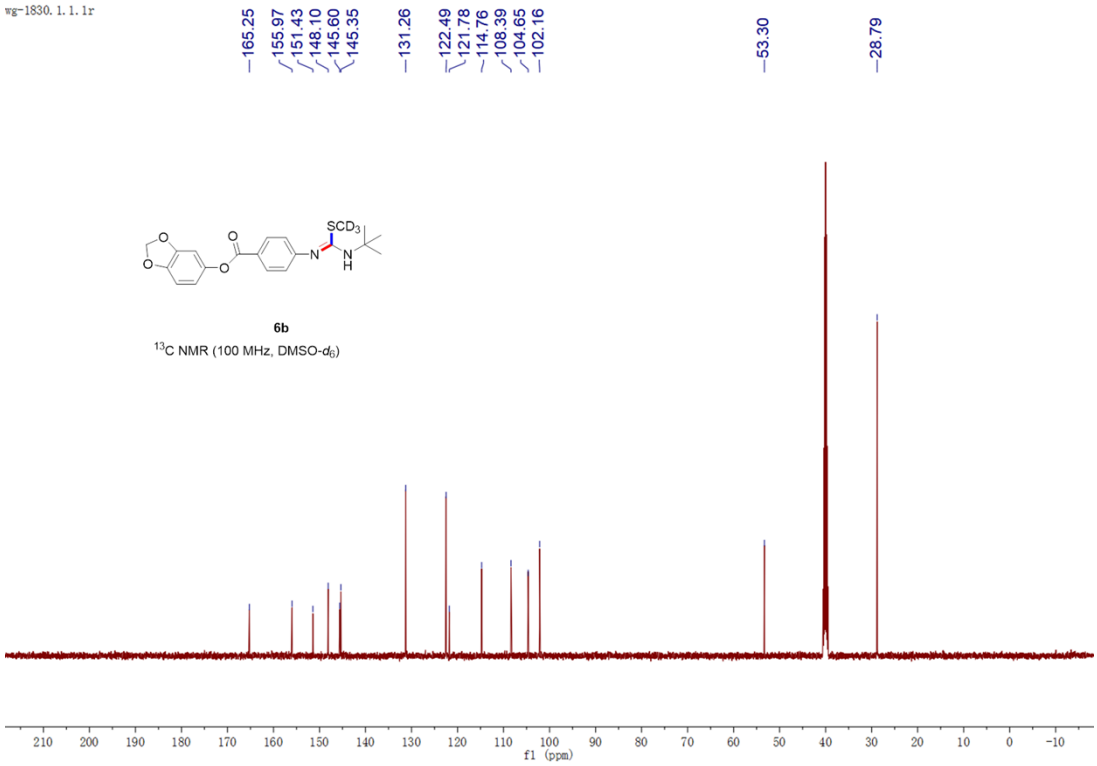
^{1}H NMR (400 MHz, DMSO-d_6)


pdata/1
150.48
149.34
148.37
128.78
128.17
126.01
125.51
121.98
121.92
57.55
30.09
SCD3
PhN
HN
5h
13C NMR (100 MHz, DMSO-d6)
f1 (ppm)


wg1746.1.1.1r
7.84
7.82
6.85
6.83
6.14
4.31
4.29
4.27
4.25
1.40
1.34
1.32
1.30
SCD3
EtO2C
N
N
H
6a
1H NMR (400 MHz, DMSO-d6)
2.05
2.03
1.00
2.03
8.85
2.98
f1 (ppm)

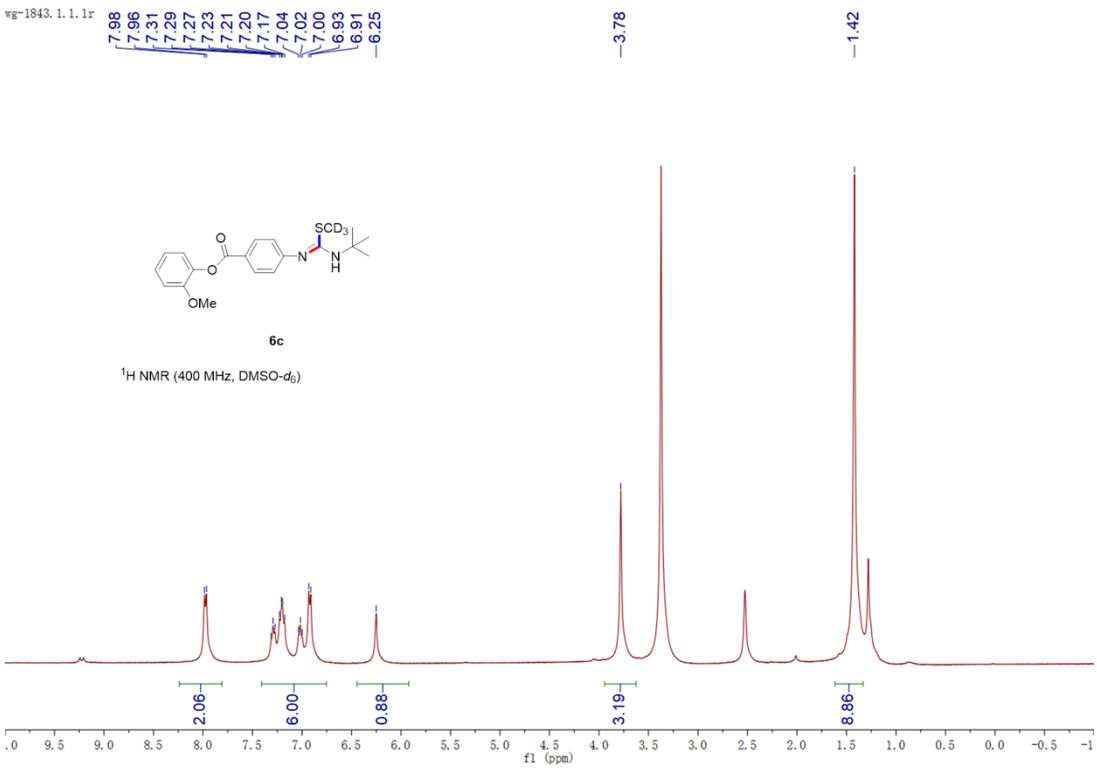

pdata/1
166.27
155.26
151.19
130.54
123.07
122.32
60.64
53.26
28.83
14.78
SCD3
EtO2C
N
N
H
6a
13C NMR (100 MHz, DMSO-d6)
f1 (ppm)


1.1.1.1r
7.98
7.96
6.97
6.95
6.95
6.94
6.93
6.92
6.91
6.91
6.90
6.72
6.72
6.70
6.70
6.25
6.09
1.41
SCD3
O
O
O
O
N
N
H
6b
1H NMR (400 MHz, DMSO-d6)
2.05
4.01
1.04
1.00
2.00
8.89
f1 (ppm)



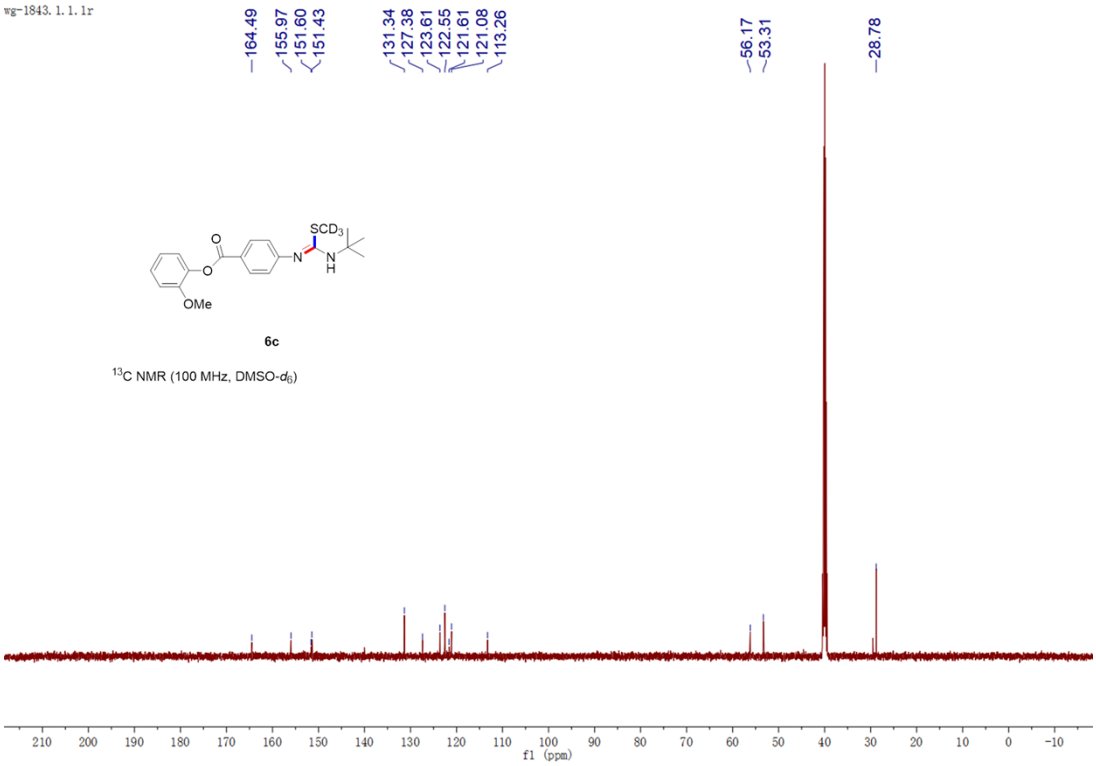
6b

^{13}C NMR (100 MHz, DMSO-d_6)



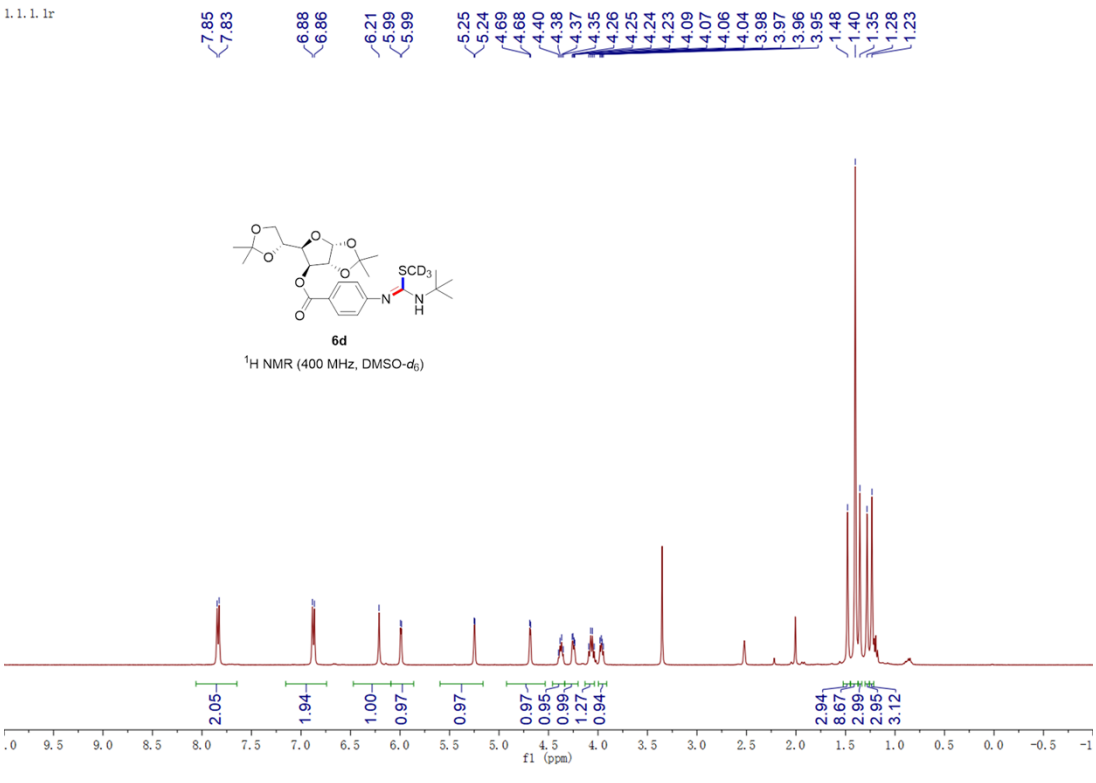
6c

^{1}H NMR (400 MHz, DMSO-d_6)



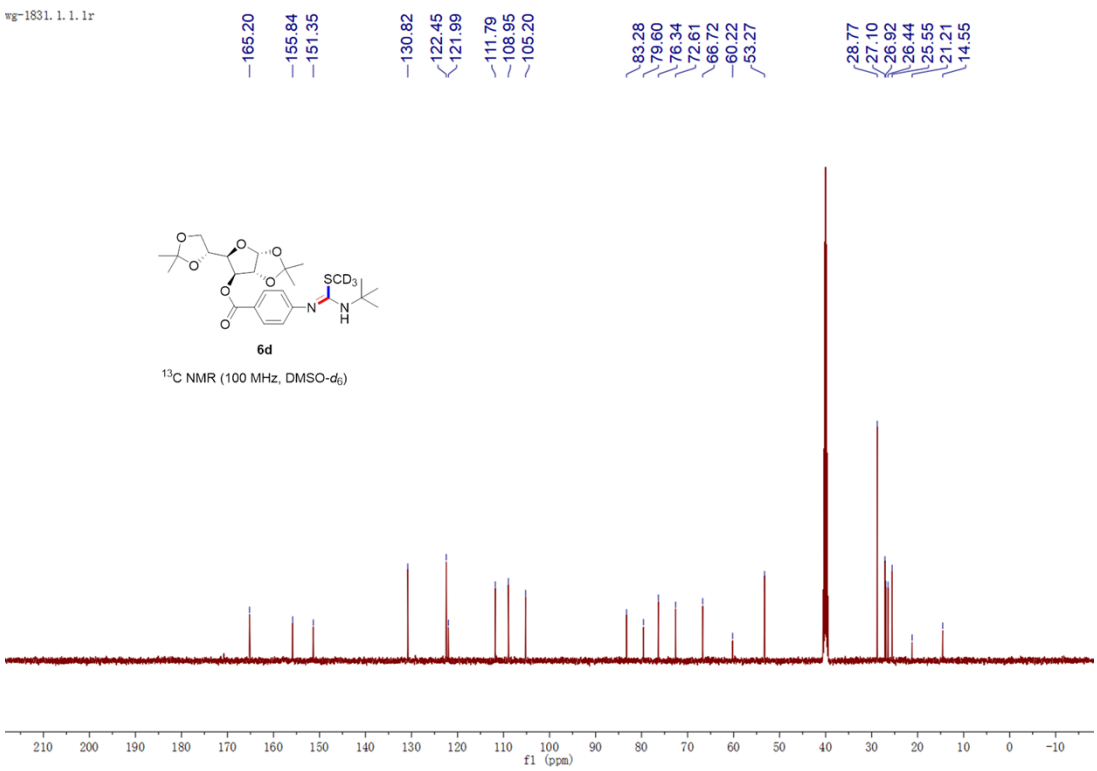
6c

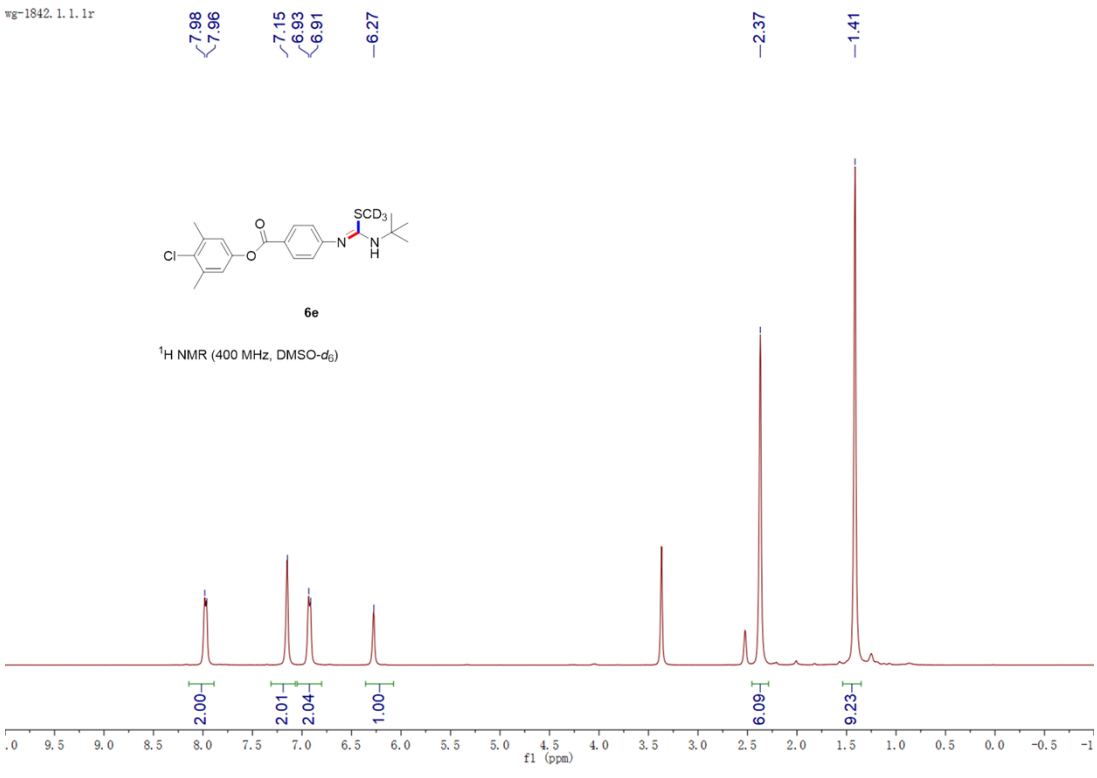
^{13}C NMR (100 MHz, DMSO-d_6)

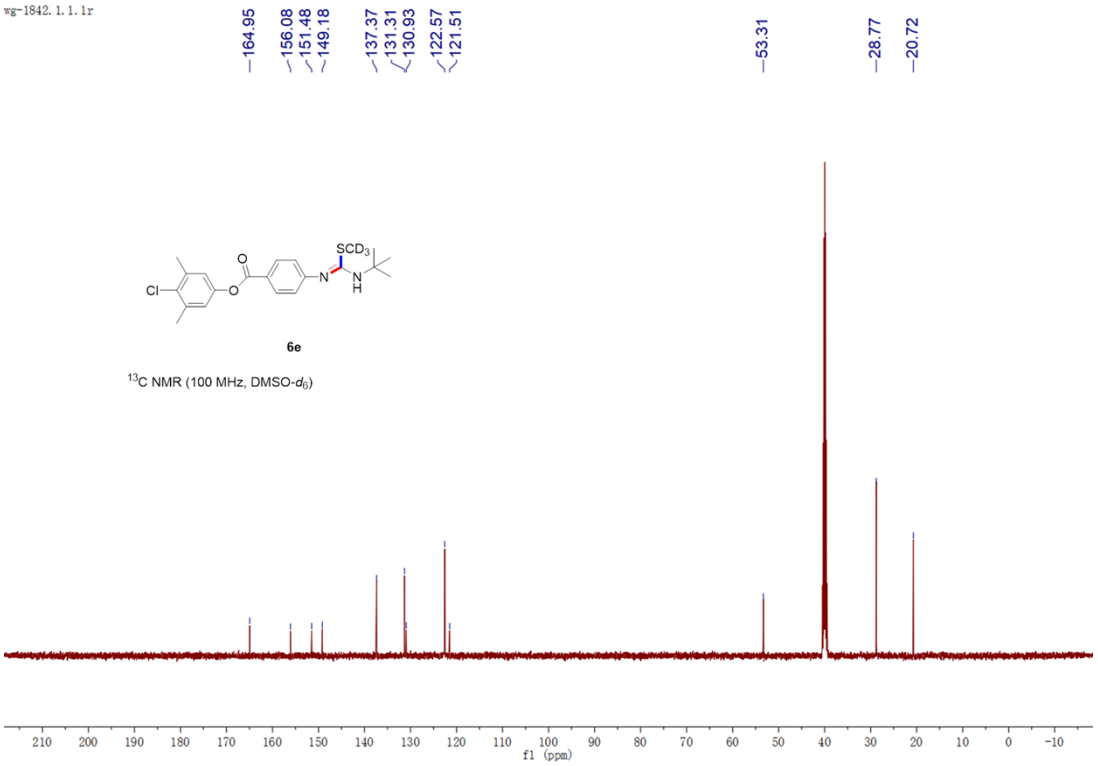


6d

^{1}H NMR (400 MHz, DMSO-d_6)

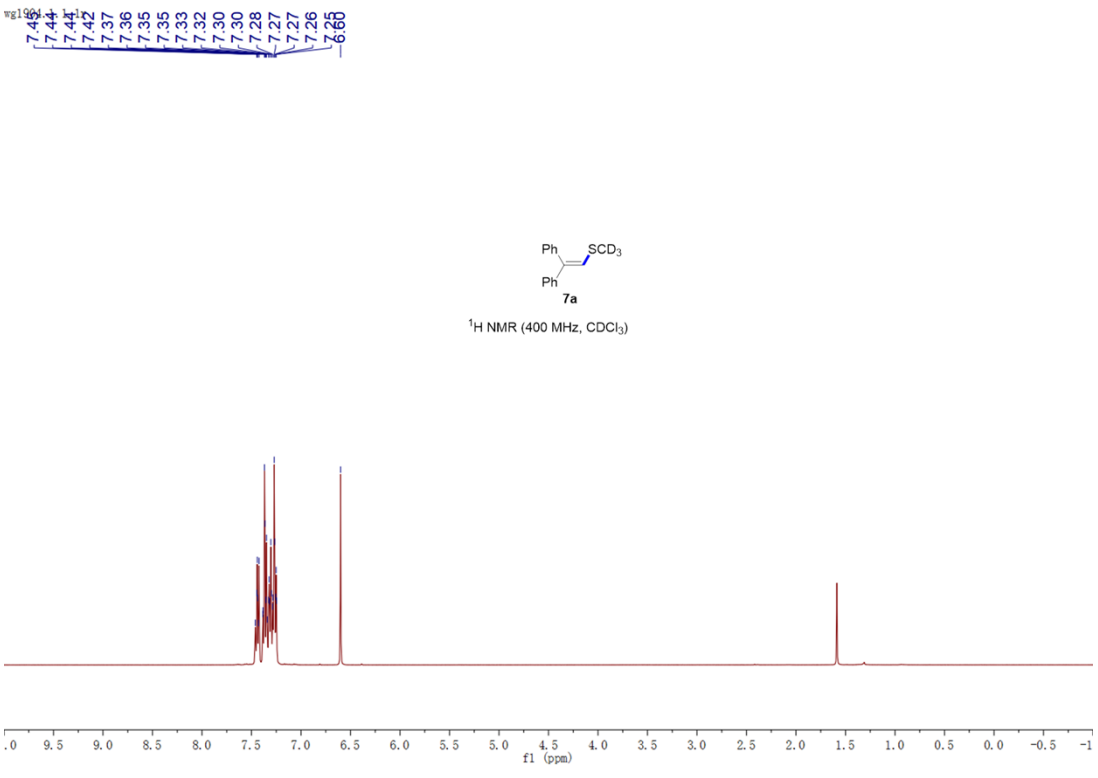

wg-1831.1.1.1r
165.20
155.84
151.35
130.82
122.45
121.99
111.79
108.95
105.20
83.28
79.60
76.34
72.61
66.72
60.22
53.27
28.77
27.10
26.92
26.44
25.55
21.21
14.55
SCD3
N
N
H
O
O
O
O
O
O
O
6d
13C NMR (100 MHz, DMSO-d6)
f1 (ppm)


wg-1842.1.1.1r
7.98
7.96
7.15
6.93
6.91
6.27
2.37
1.41
SCD3
N
N
H
O
O
Cl
6e
1H NMR (400 MHz, DMSO-d6)
2.00
2.01
2.04
1.00
6.09
9.23
f1 (ppm)



6e

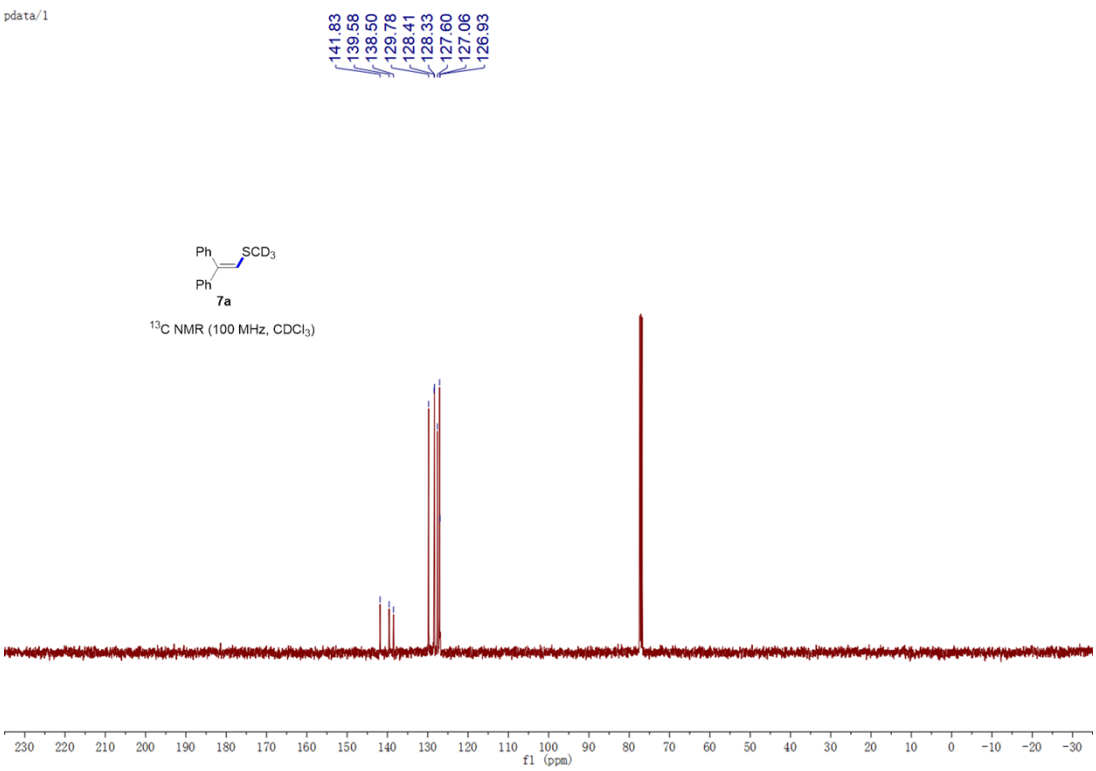
^{13}C NMR (100 MHz, DMSO-d_6)



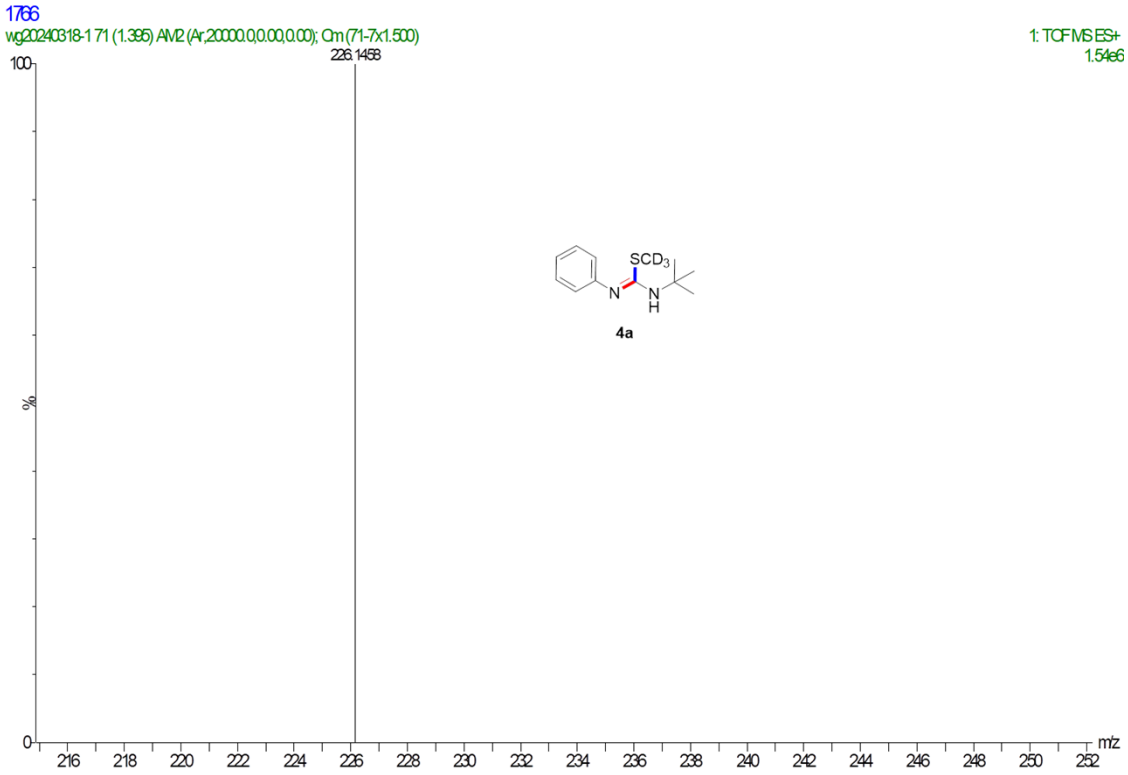
Ph
SCD3
Ph

7a

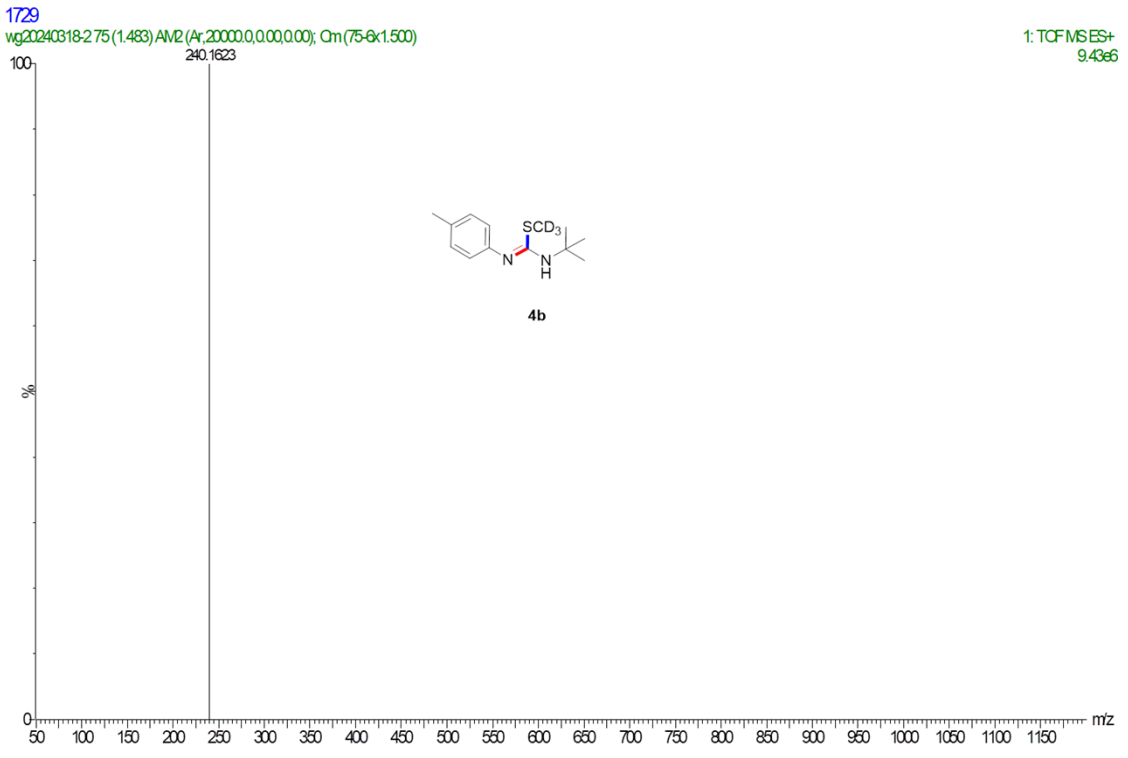
^{1}H NMR (400 MHz, $CDCl_3$)


pdata/1
141.83
139.58
138.50
129.78
128.41
128.33
127.60
127.06
126.93
Ph
SCD3
Ph
7a
13C NMR (100 MHz, CDCl3)
230 220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10 -20 -30
f1 (ppm)

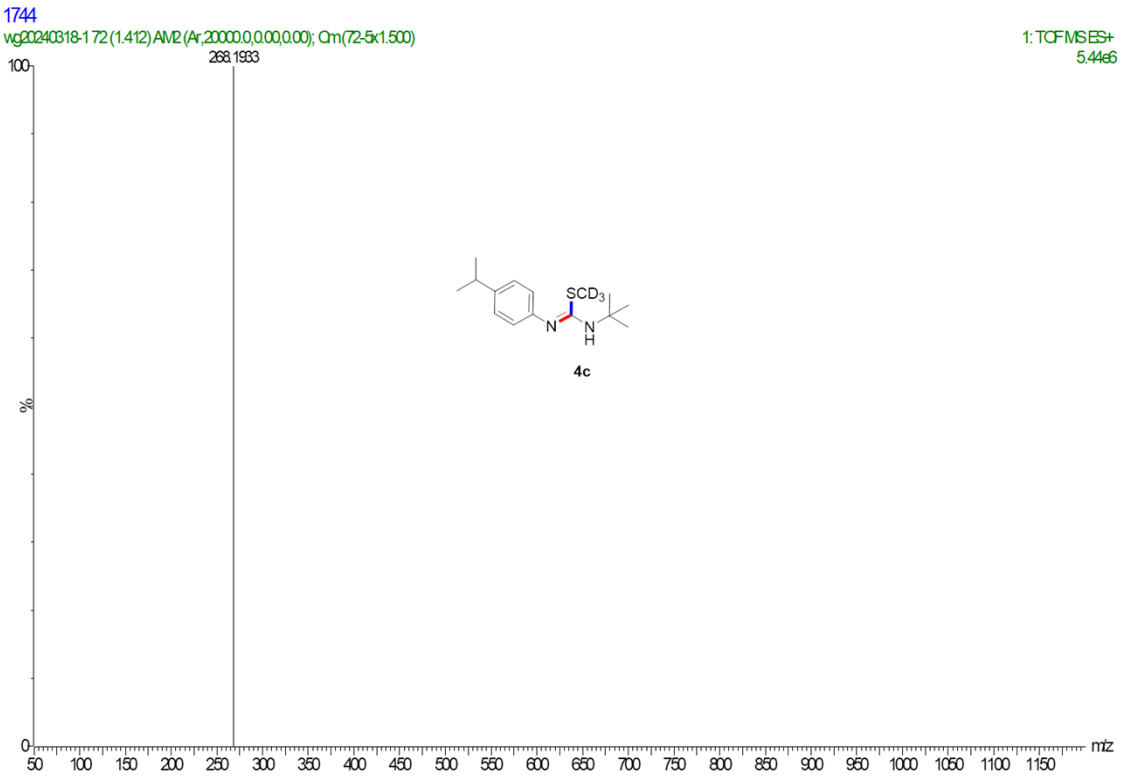
HRMS of Products

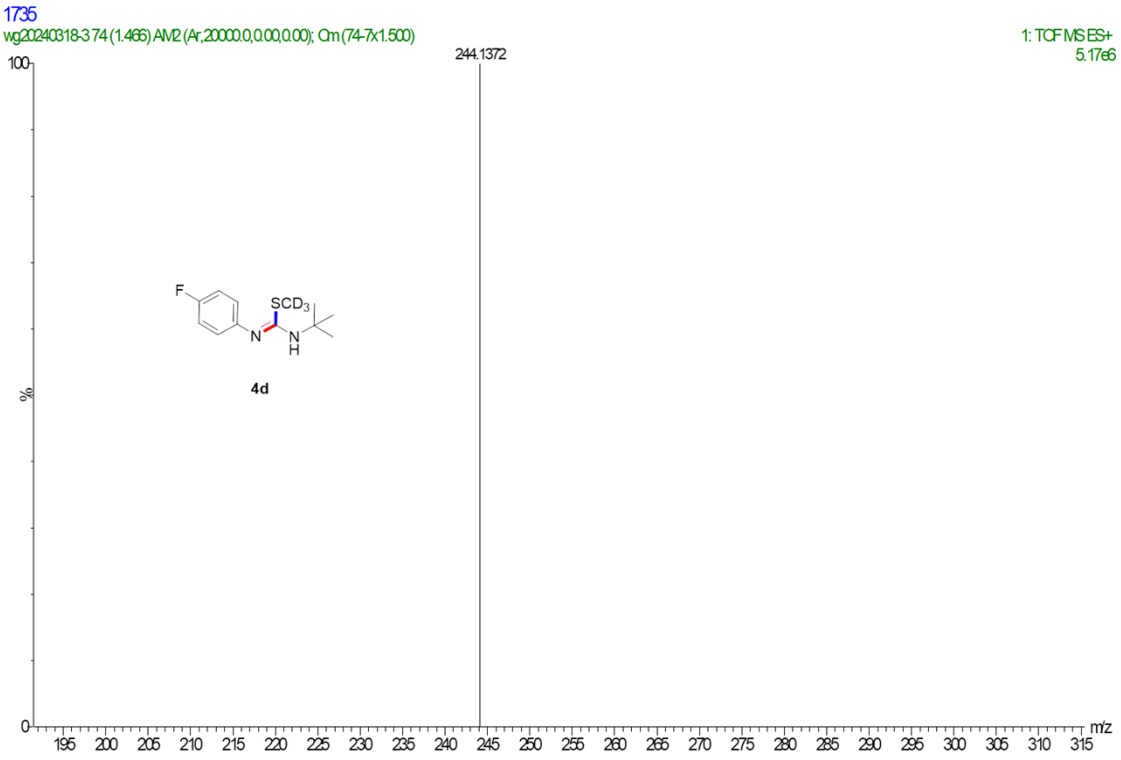


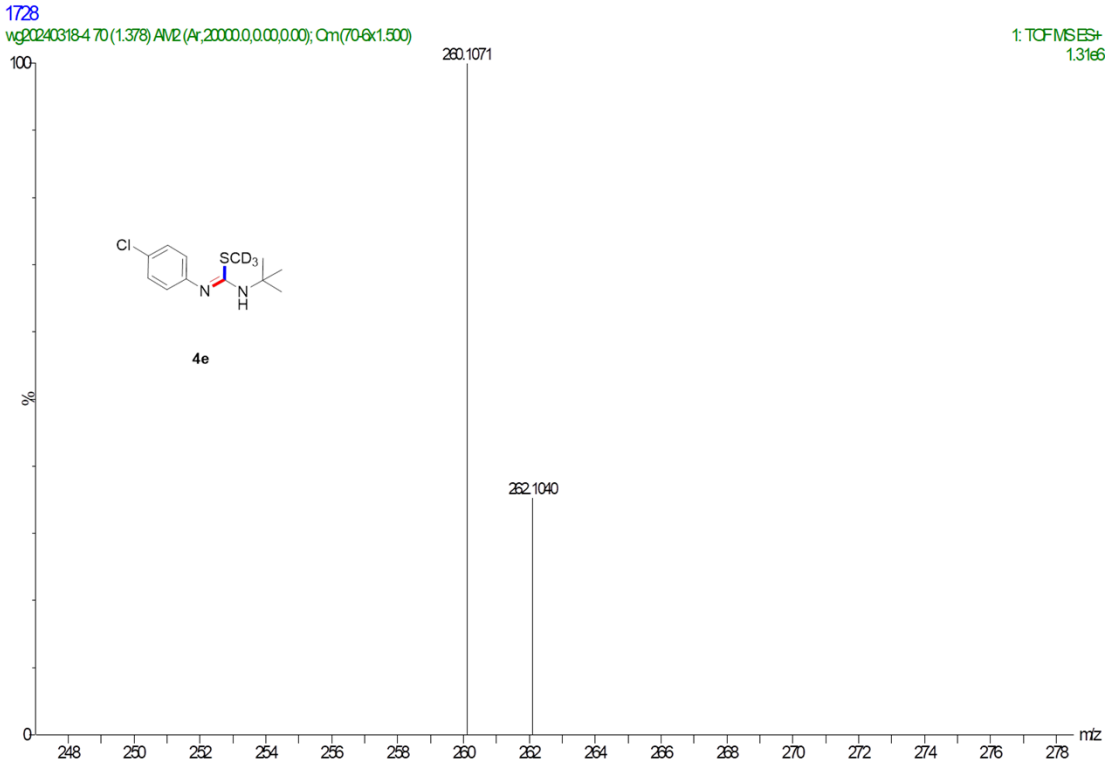
4a

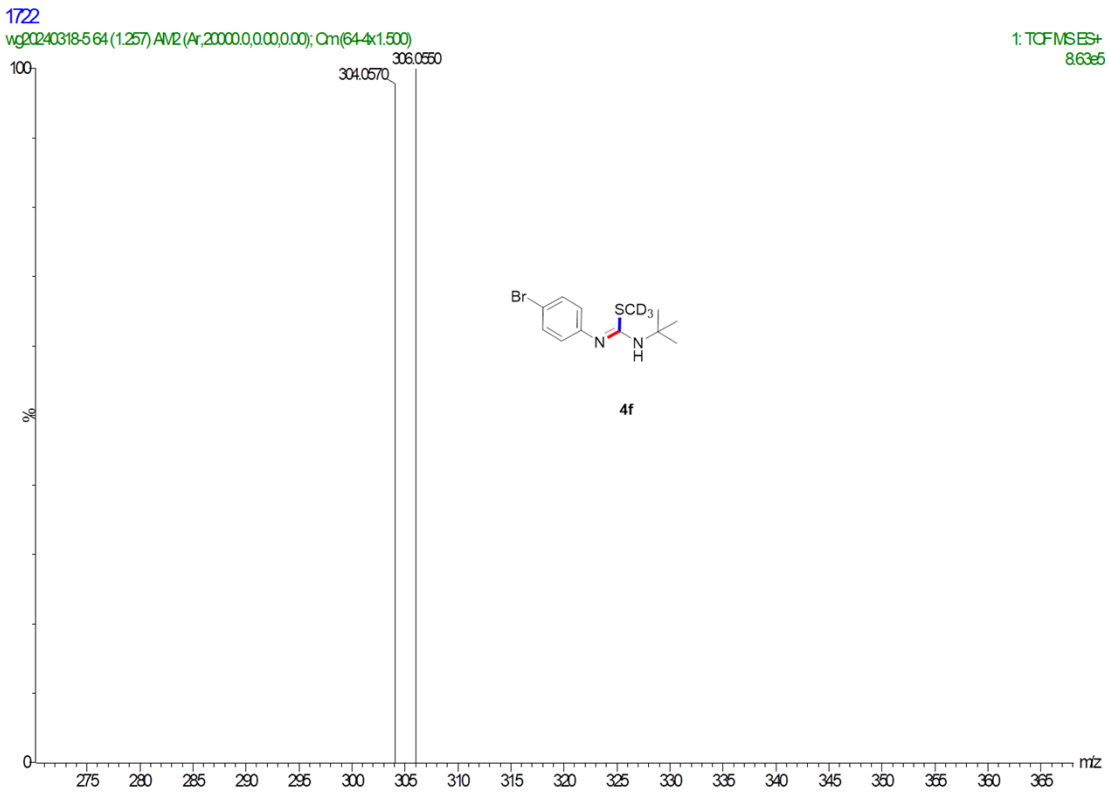


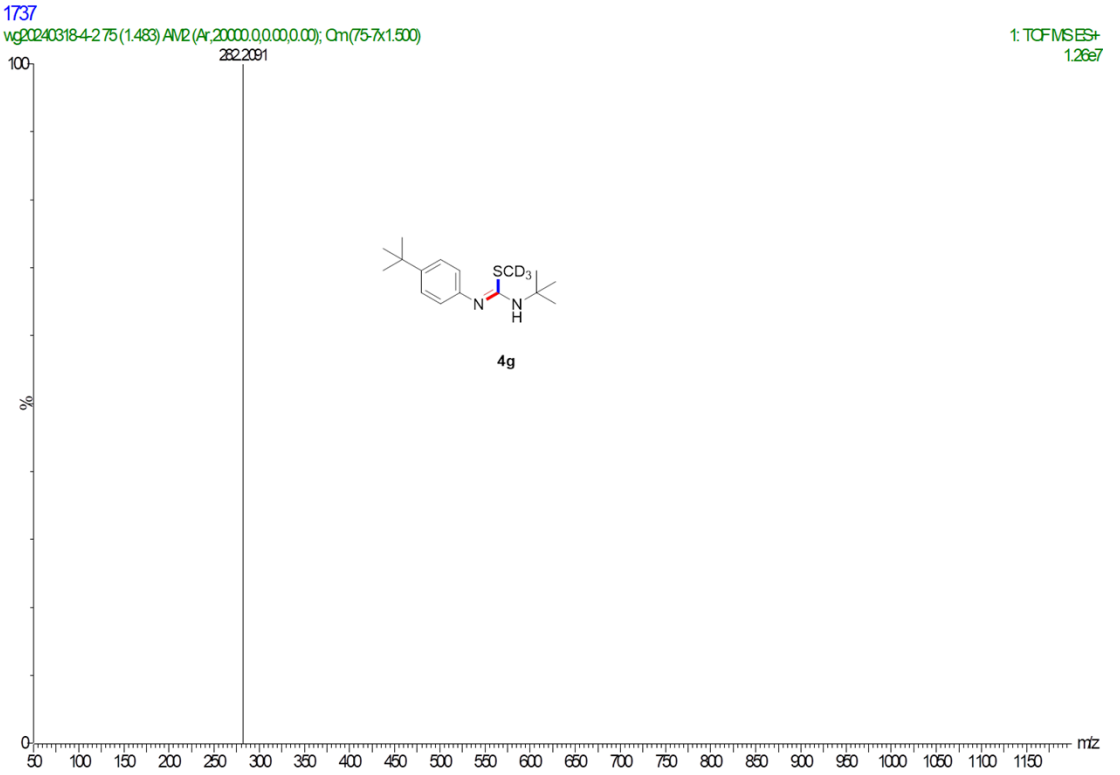
4b


1744
wg20240318-1 72 (1.412) AM2 (Ar,20000.0,0.00,0.00); Cm (72-5x1.500)
1: TOF MS ES+
5.44e6
268.1933
100
%
0
SCD3
N
N
H
4c
m/z
50 100 150 200 250 300 350 400 450 500 550 600 650 700 750 800 850 900 950 1000 1050 1100 1150

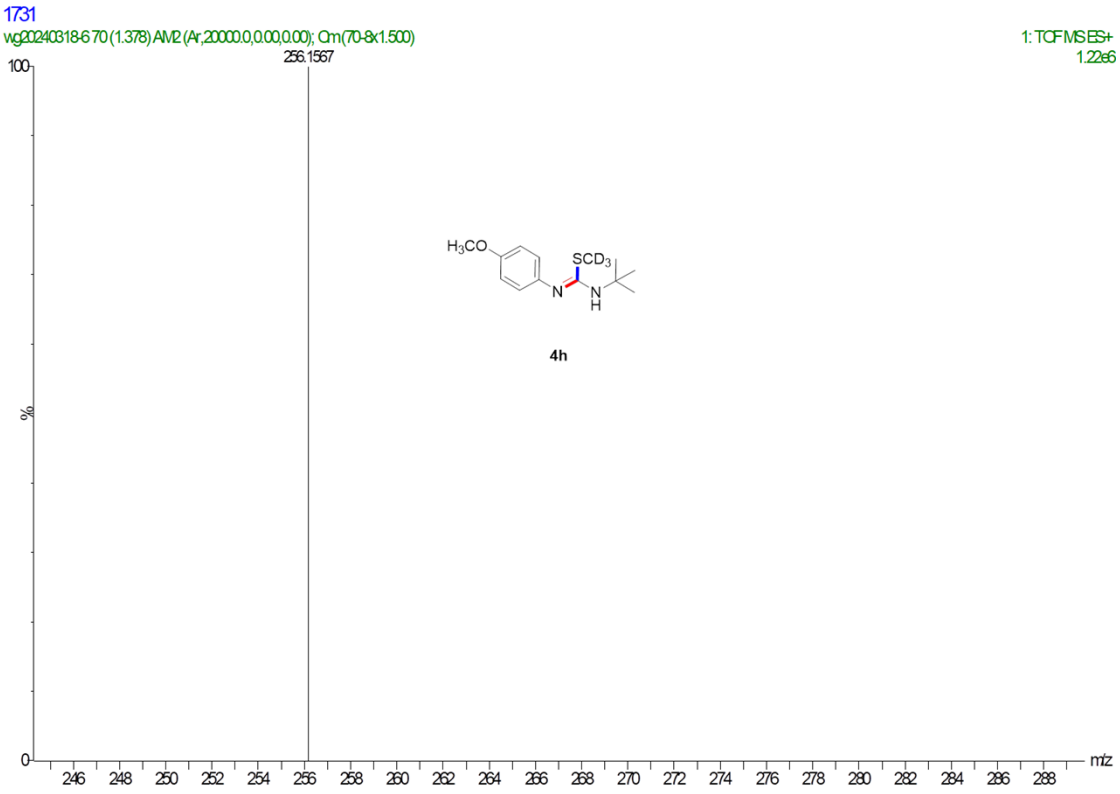

1735
wg20240318-3 74 (1.466) AM2 (Ar,20000.0,0.00,0.00); Cm (74-7x1.500)
1: TOF MS ES+
5.17e6
244.1372
100
%
0
F
SCD3
N
N
H
4d
m/z
195 200 205 210 215 220 225 230 235 240 245 250 255 260 265 270 275 280 285 290 295 300 305 310 315


1728
wg20240318-4 70 (1.378) AM2 (Ar,20000.0,0.00,0.00); Cm(70-6x1.500)
1: TOF MS ES+
1.31e6
260.1071
262.1040
Cl
SCD3
N
N
H
4e
100
%
0
248
250
252
254
256
258
260
262
264
266
268
270
272
274
276
278
m/z

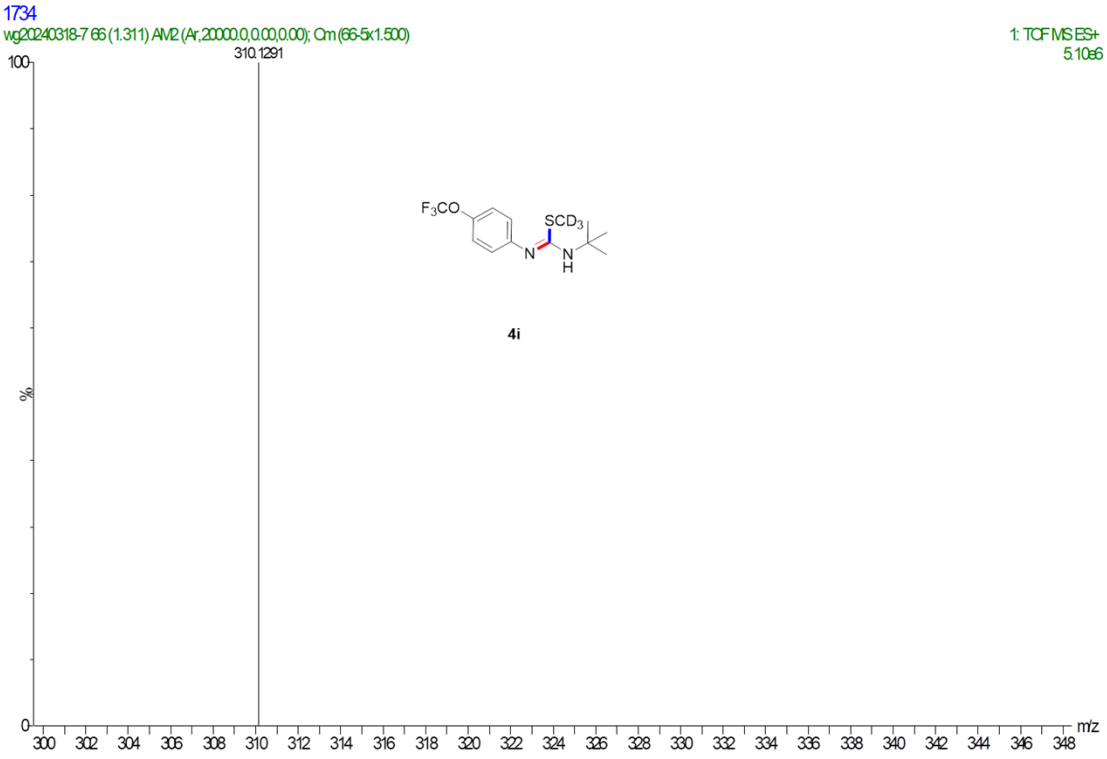

1722
wg20240318-5 64 (1.257) AM2 (Ar,20000.0,0.00,0.00); Cm(64-4x1.500)
1: TOF MS ES+
8.63e5
304.0570
306.0550
Br
SCD3
N
N
H
4f
100
%
0
275
280
285
290
295
300
305
310
315
320
325
330
335
340
345
350
355
360
365
m/z


1737
wg20240318-4-2 75 (1.483) AM2 (Ar,20000.0,0.00,0.00); Cm(75-7x1.500)
1: TOF MS ES+
1.26e7
282.2091
100
%
0
50 100 150 200 250 300 350 400 450 500 550 600 650 700 750 800 850 900 950 1000 1050 1100 1150
m/z
SCD3
N
N
H

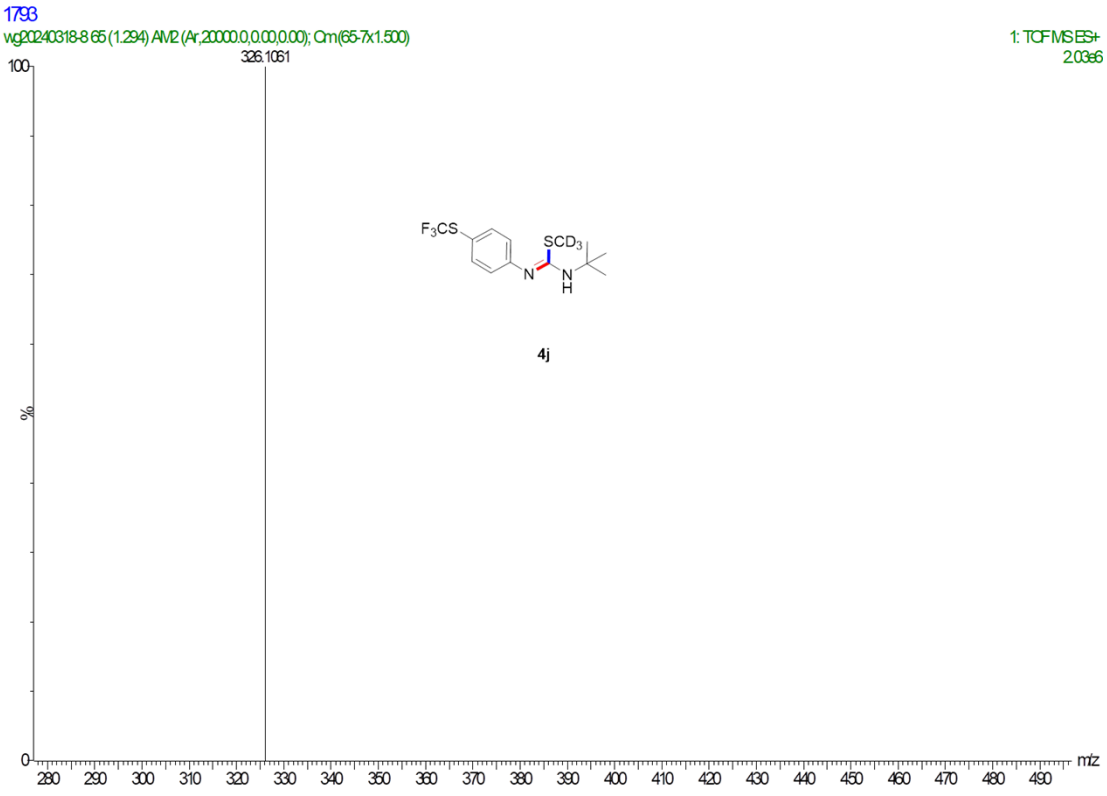
4g


1731
wg20240318-6 70 (1.378) AM2 (Ar,20000.0,0.00,0.00); Cm(70-8x1.500)
1: TOF MS ES+
1.22e6
256.1567
100
%
0
246 248 250 252 254 256 258 260 262 264 266 268 270 272 274 276 278 280 282 284 286 288
m/z
H3CO
SCD3
N
N
H

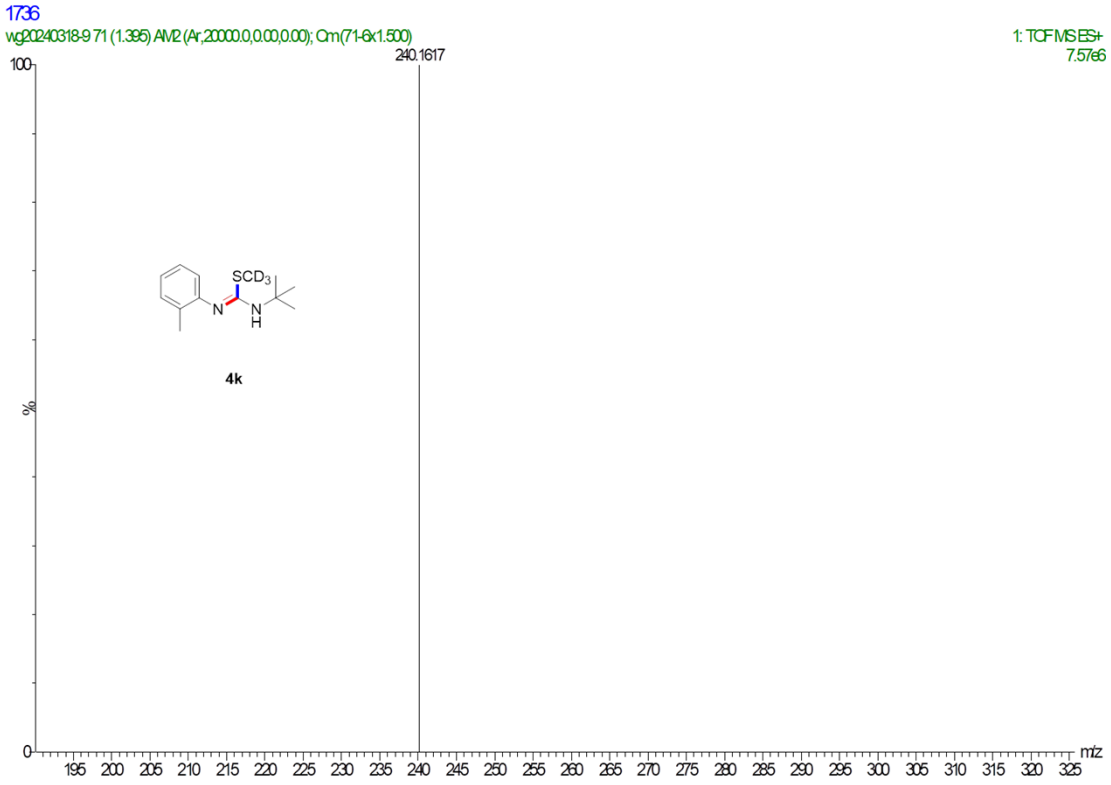
4h

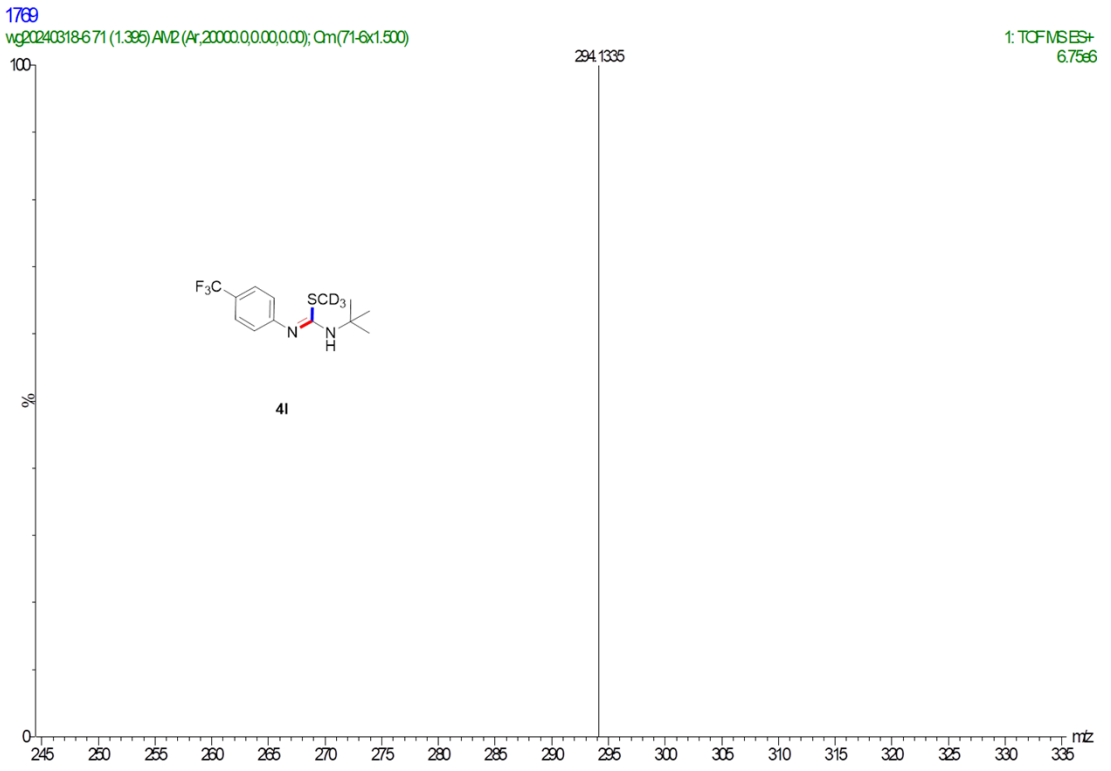

1734
wg20240318-7 66 (1.311) AM2 (Ar,20000.0,0.00,0.00); Cm (66-5x1.500)
1: TOF MS ES+
5.10e6
310.1291
100
%
0
300 302 304 306 308 310 312 314 316 318 320 322 324 326 328 330 332 334 336 338 340 342 344 346 348
m/z
F3CO
SCD3
N
N
H

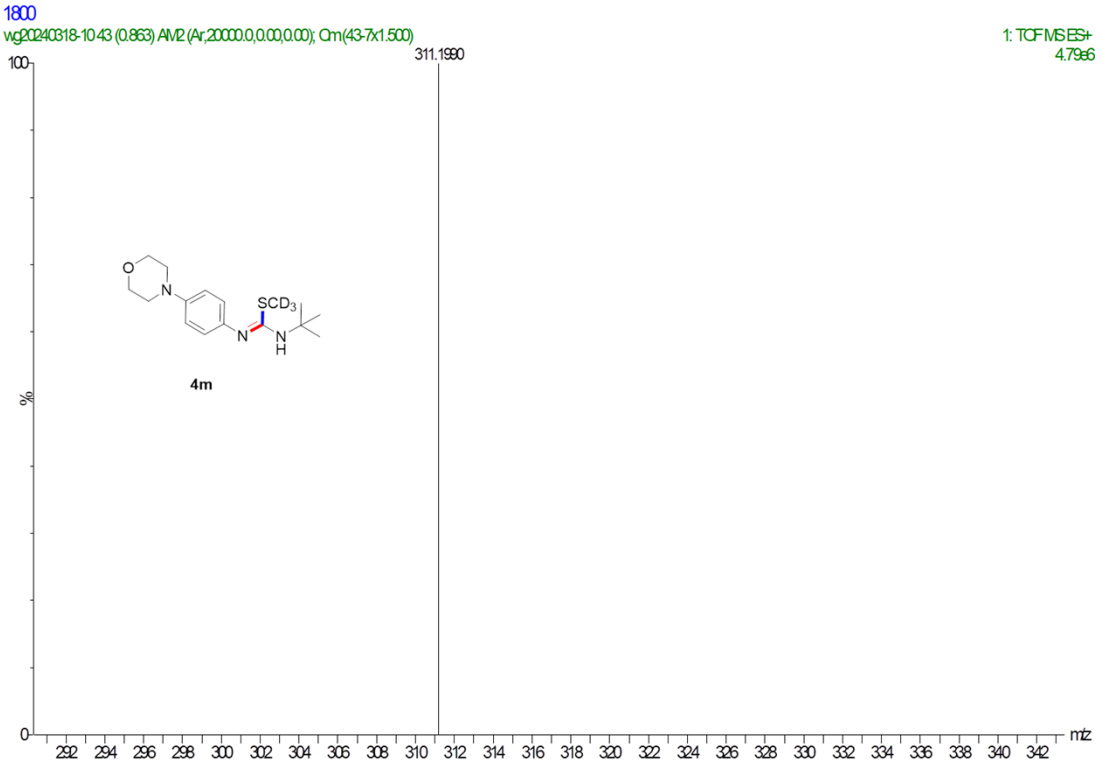
4i

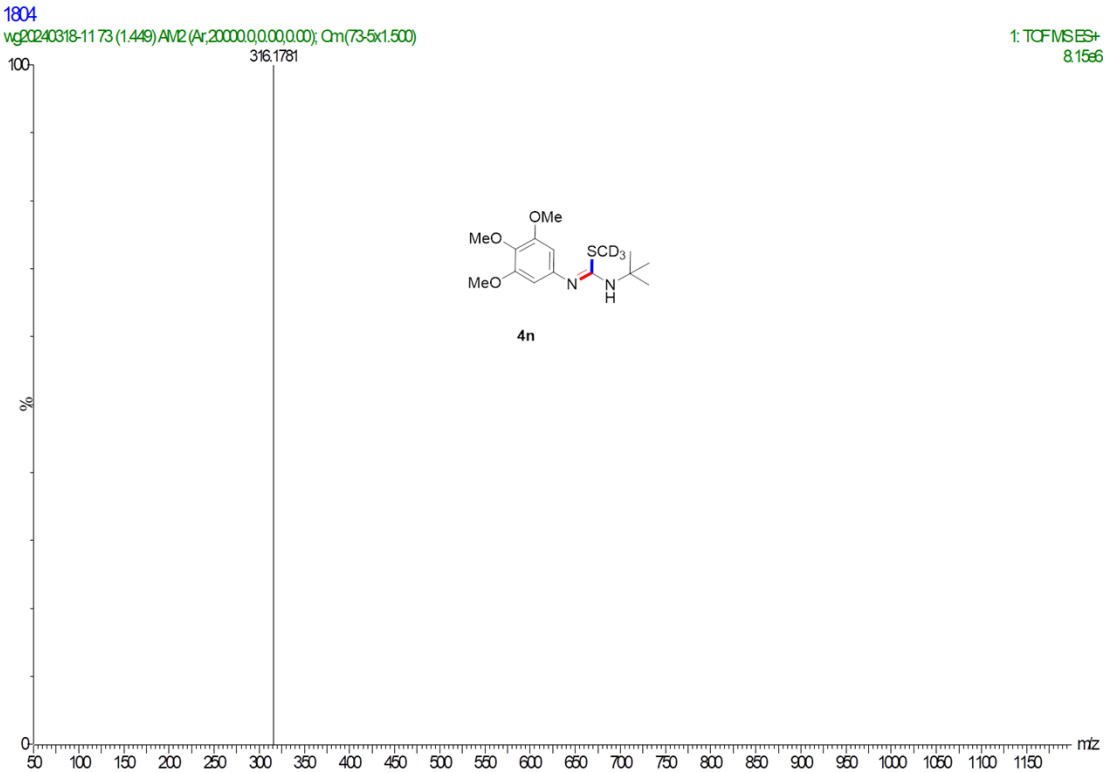

1793
wg20240318-8 65 (1.294) AM2 (Ar,20000.0,0.00,0.00); Cm (65-7x1.500)
1: TOF MS ES+
2.03e6
326.1061
100
%
0
280 290 300 310 320 330 340 350 360 370 380 390 400 410 420 430 440 450 460 470 480 490
m/z
F3CS
SCD3
N
N
H

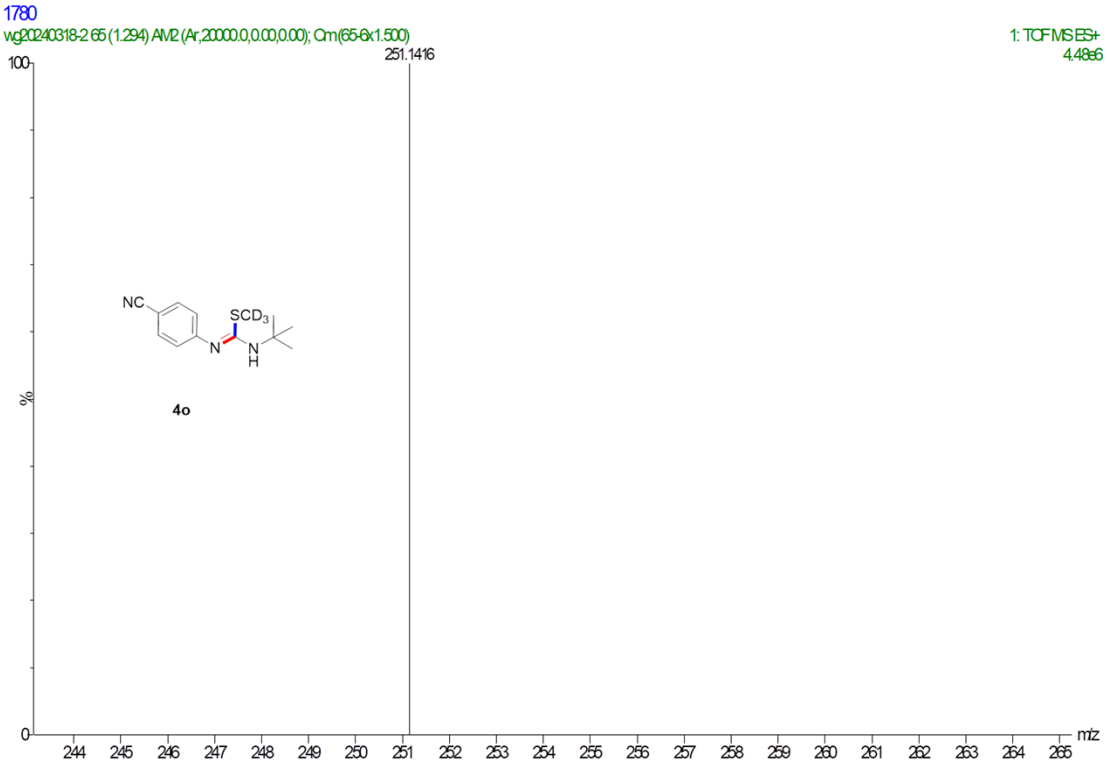
4j

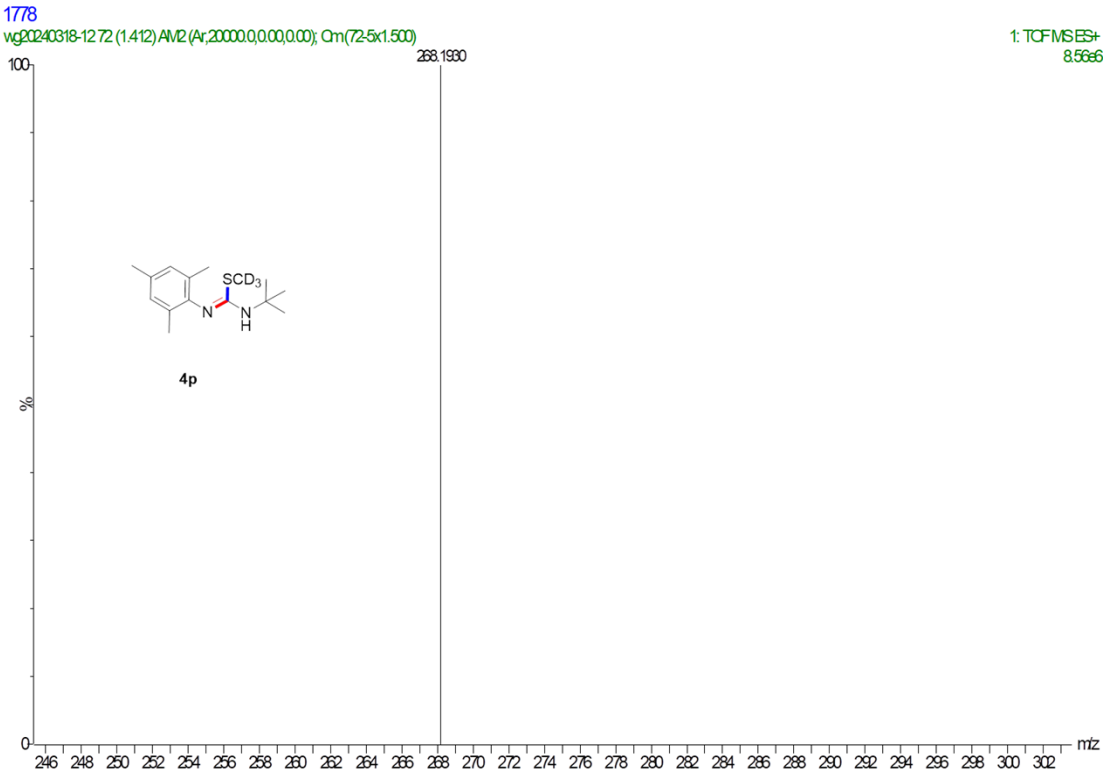

1736
wg20240318-9 71 (1.395) AM2 (Ar,20000.0,0.00,0.00); Cm(71-6x1.500)
1: TOF MS ES+
7.57e6
240.1617
100
%
0
SCD3
N
N
H
4k
195 200 205 210 215 220 225 230 235 240 245 250 255 260 265 270 275 280 285 290 295 300 305 310 315 320 325
m/z

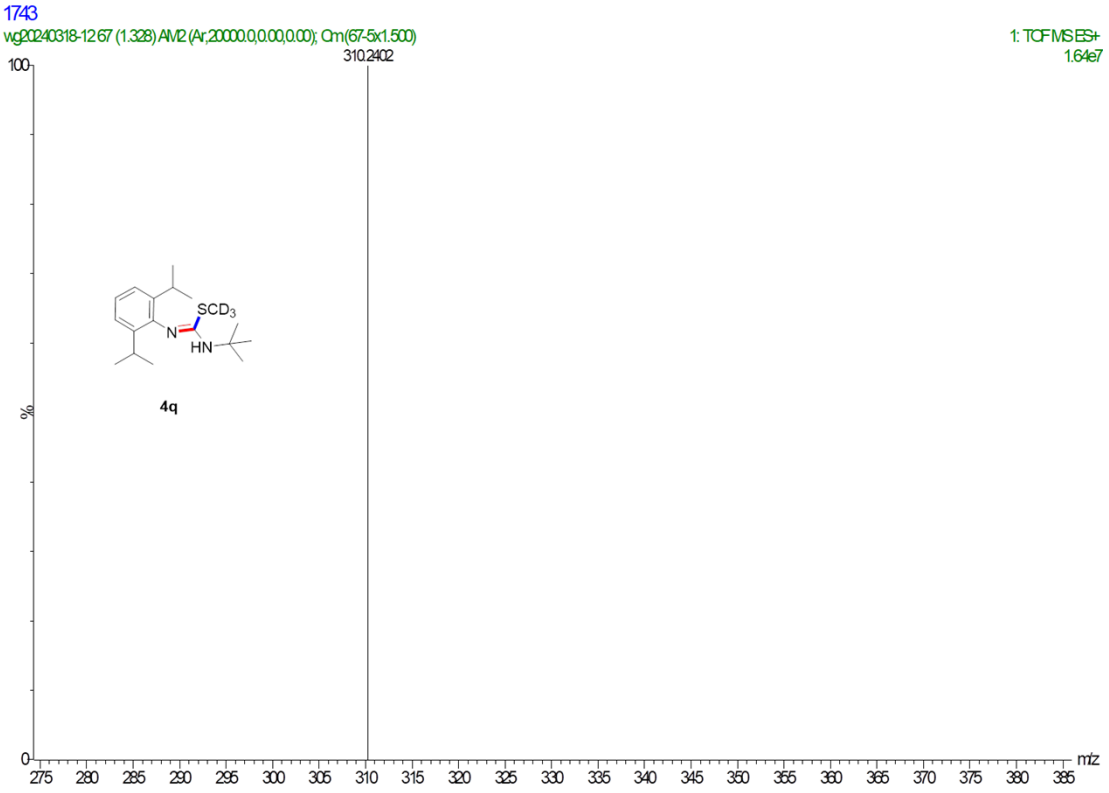

1769
wg20240318-6 71 (1.395) AM2 (Ar,20000.0,0.00,0.00); Cm(71-6x1.500)
1: TOF MS ES+
6.75e6
294.1335
100
%
0
F3C
SCD3
N
N
H
4l
245 250 255 260 265 270 275 280 285 290 295 300 305 310 315 320 325 330 335
m/z

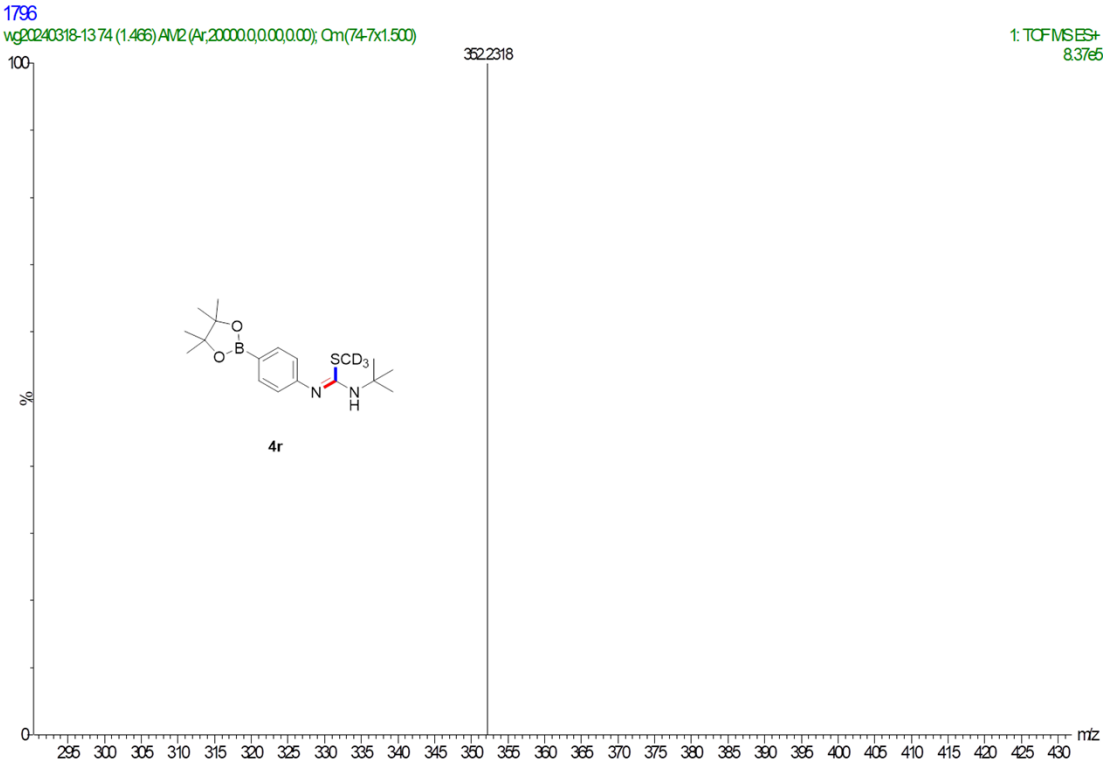

1800
wg20240318-10 43 (0.863) AM2 (Ar,20000.0,0.00,0.00); Cm(43-7x1.500)
1: TOF MS ES+
4.79e6
311.1990
100
%
0
292 294 296 298 300 302 304 306 308 310 312 314 316 318 320 322 324 326 328 330 332 334 336 338 340 342
m/z
O
N
SCD3
N
N
H
4m

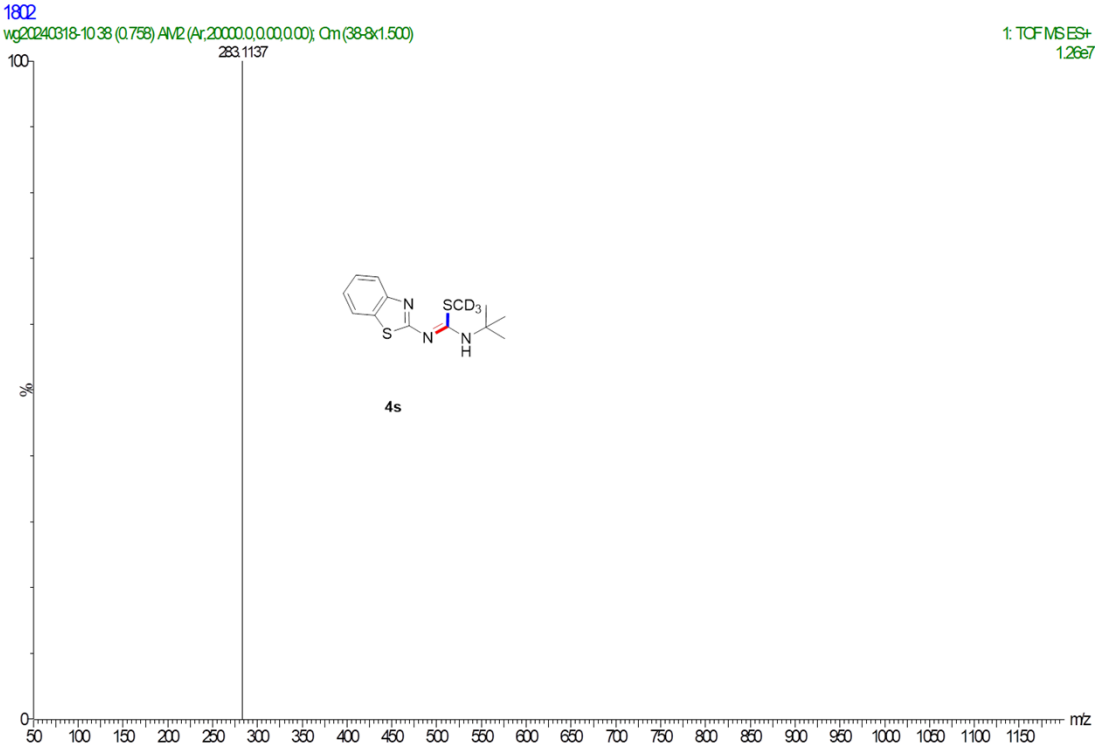

1804
wg20240318-11 73 (1.449) AM2 (Ar,20000.0,0.00,0.00); Cm(73-5x1.500)
1: TOF MS ES+
8.15e6
316.1781
100
%
0
50 100 150 200 250 300 350 400 450 500 550 600 650 700 750 800 850 900 950 1000 1050 1100 1150
m/z
OMe
MeO
MeO
SCD3
N
N
H
4n

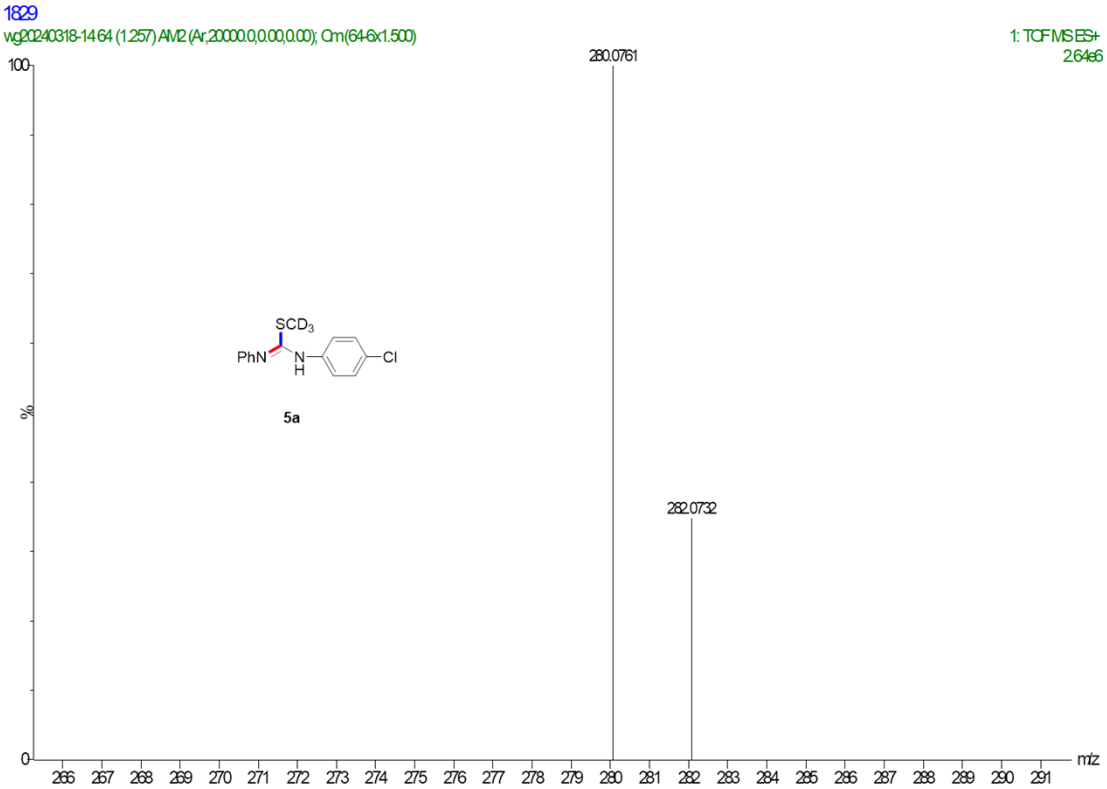

1780
wg20240318-2 65 (1.294) AM2 (Ar,20000.0,0.00,0.00); Cm(65-6x1.500)
1: TOF MS ES+
4.48e6
251.1416
100
%
0
NC
SCD3
N
N
H
4o
244 245 246 247 248 249 250 251 252 253 254 255 256 257 258 259 260 261 262 263 264 265
m/z

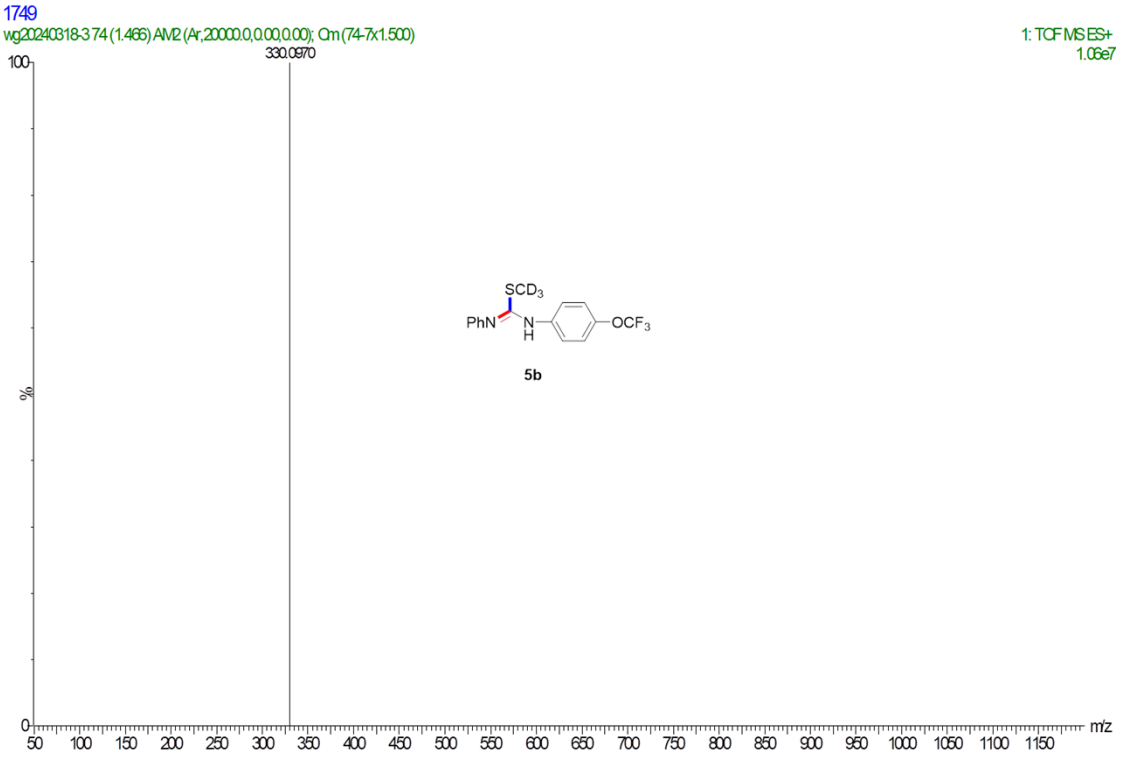

1778
wg20240318-12 72 (1.412) AM2 (Ar,20000.0,0.00,0.00); Cm(72-5x1.500)
1: TOF MS ES+
8.56e6
268.1930
100
%
0
SCD3
N
N
H
4p
246 248 250 252 254 256 258 260 262 264 266 268 270 272 274 276 278 280 282 284 286 288 290 292 294 296 298 300 302
m/z


1743
wg20240318-1267 (1.328) AM2 (Ar,20000.0,0.00,0.00); Cm(67-5x1.500)
1: TOF MS ES+
1.64e7
310.2402
100
%
0
SCD3
N
HN
4q
275 280 285 290 295 300 305 310 315 320 325 330 335 340 345 350 355 360 365 370 375 380 385
m/z

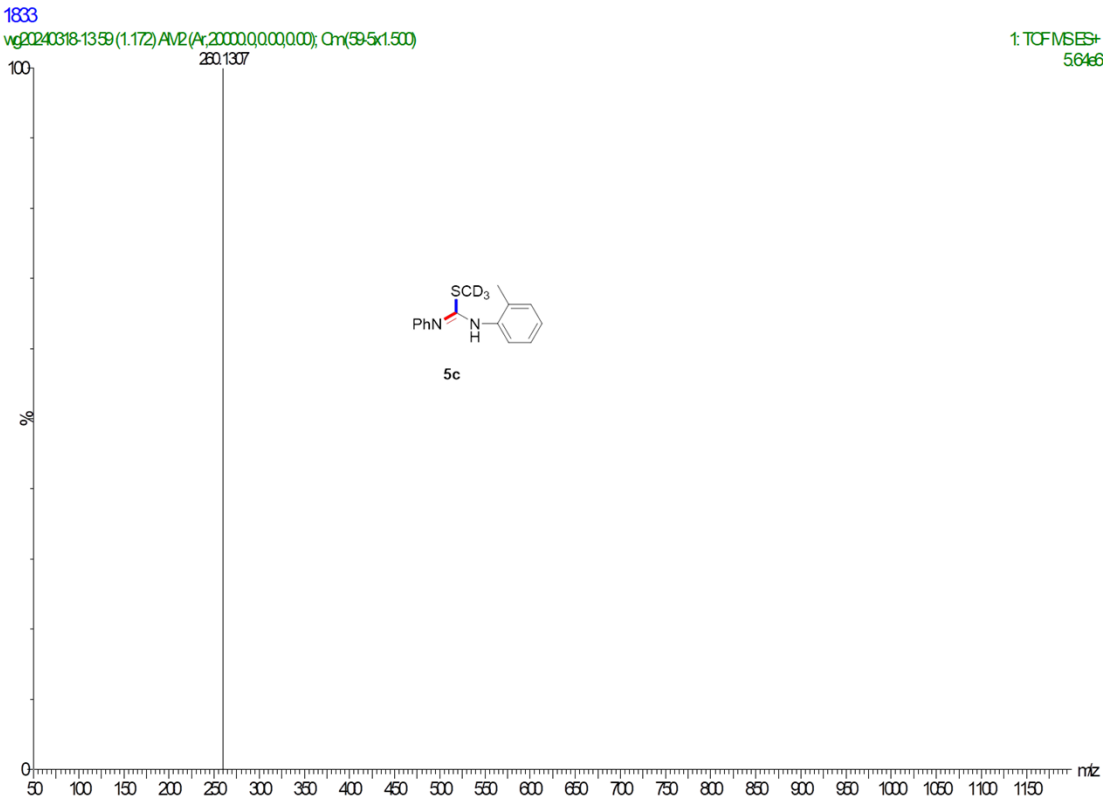

1796
wg20240318-1374 (1.466) AM2 (Ar,20000.0,0.00,0.00); Cm(74-7x1.500)
1: TOF MS ES+
8.37e5
352.2318
100
%
0
O
B
O
SCD3
N
N
H
4r
295 300 305 310 315 320 325 330 335 340 345 350 355 360 365 370 375 380 385 390 395 400 405 410 415 420 425 430
m/z


1802
wg20240318-10 38 (0.758) AM2 (Ar,20000.0,0.00,0.00); Cm (38-8x1.500)
1: TOF MS ES+
1.26e7
283.1137
100
%
0
SCD3
N
S
N
N
H
4s
50 100 150 200 250 300 350 400 450 500 550 600 650 700 750 800 850 900 950 1000 1050 1100 1150
m/z

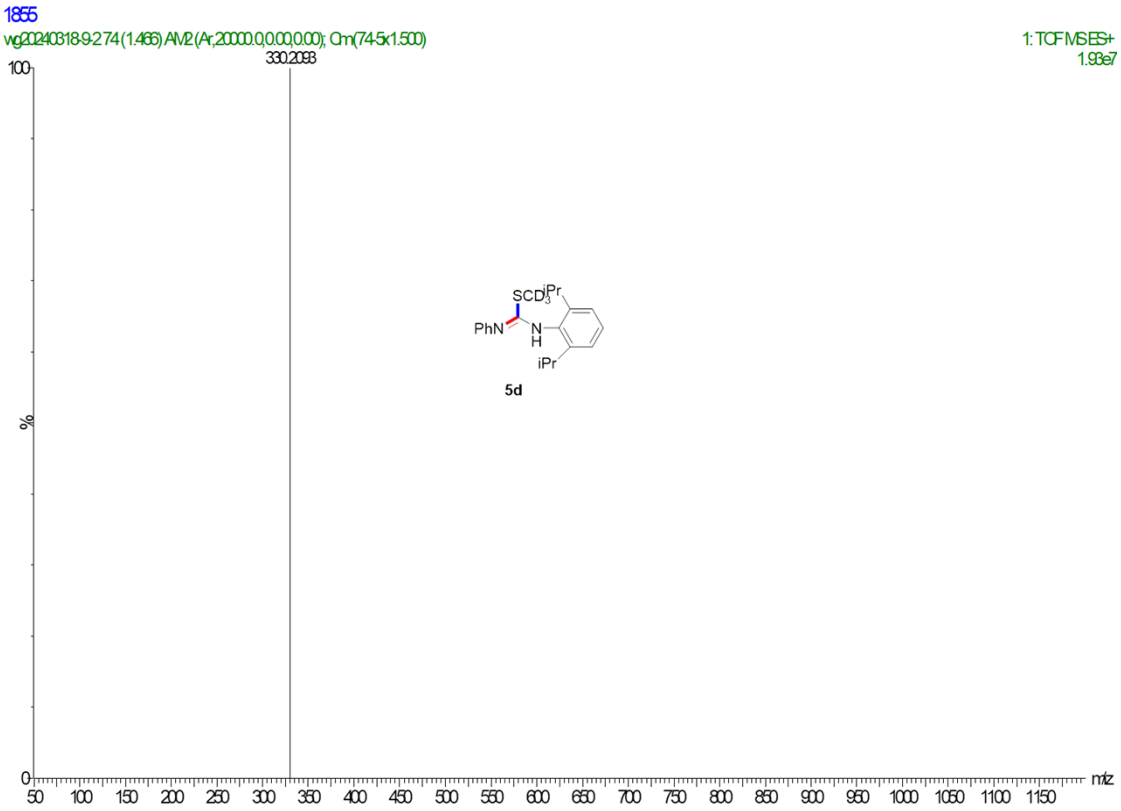

1829
wg20240318-14 64 (1.257) AM2 (Ar,20000.0,0.00,0.00); Cm (64-6x1.500)
1: TOF MS ES+
2.64e6
280.0761
282.0732
100
%
0
SCD3
PhN
N
H
Cl
5a
265 266 267 268 269 270 271 272 273 274 275 276 277 278 279 280 281 282 283 284 285 286 287 288 289 290 291
m/z

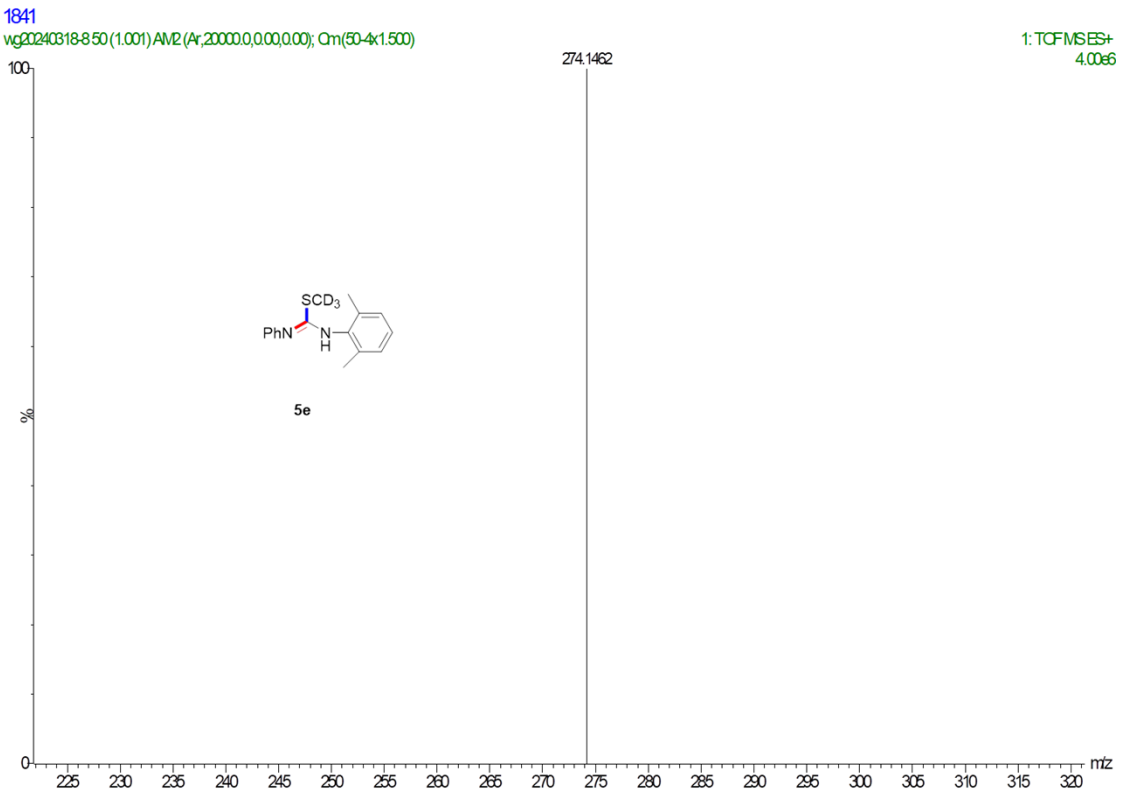

1749
wg20240318-374 (1.466) AM2 (Ar,20000.0,0.00,0.00); Cm (74-7x1.500)
1: TOF MS ES+
1.06e7
330.0970
100
%
0
50 100 150 200 250 300 350 400 450 500 550 600 650 700 750 800 850 900 950 1000 1050 1100 1150
m/z
SCD3
PhN
N
H
OCF3

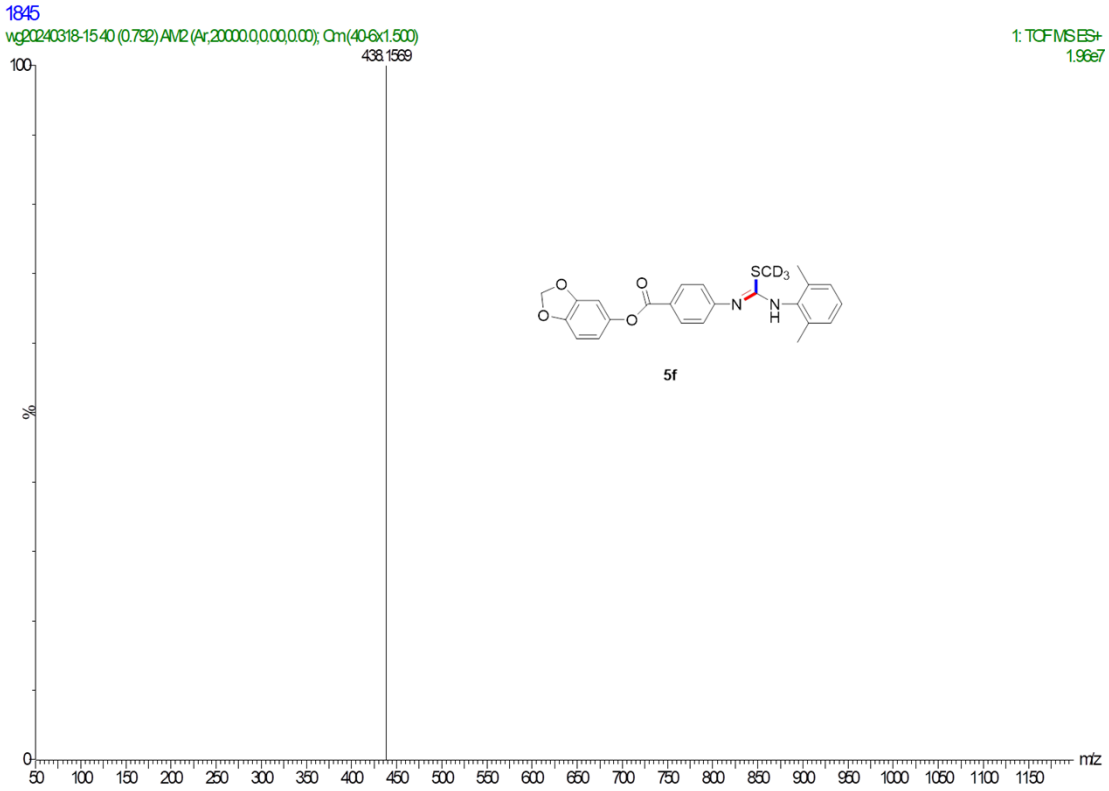
5b


1833
wg20240318-1359 (1.172) AM2 (Ar,20000.0,0.00,0.00); Cm (59-5x1.500)
1: TOF MS ES+
5.64e6
260.1307
100
%
0
50 100 150 200 250 300 350 400 450 500 550 600 650 700 750 800 850 900 950 1000 1050 1100 1150
m/z
SCD3
PhN
N
H

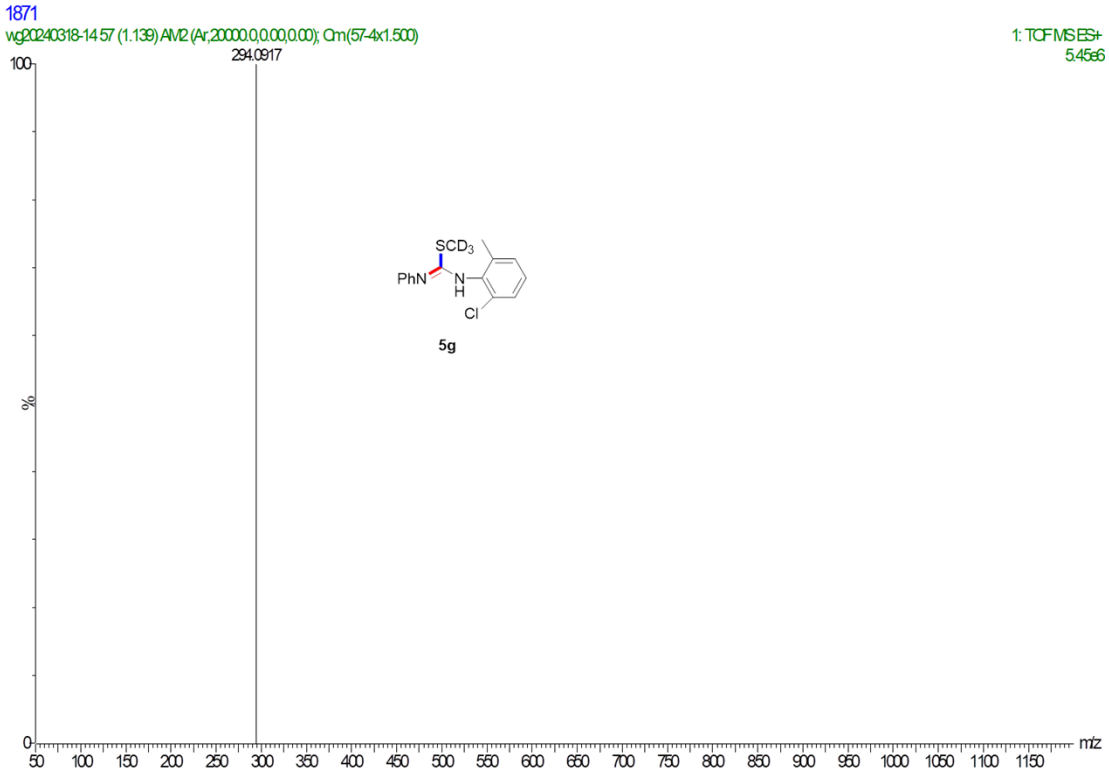
5c


1855
wg20240318-9-2 74 (1.466) AM2 (Ar,20000.0,0.00,0.00); Cm(74-5x1.500)
1: TOF MS ES+
1.93e7
330.2093
100
%
0
50 100 150 200 250 300 350 400 450 500 550 600 650 700 750 800 850 900 950 1000 1050 1100 1150
m/z
SCD3
iPr
PhN
N
H
iPr
5d

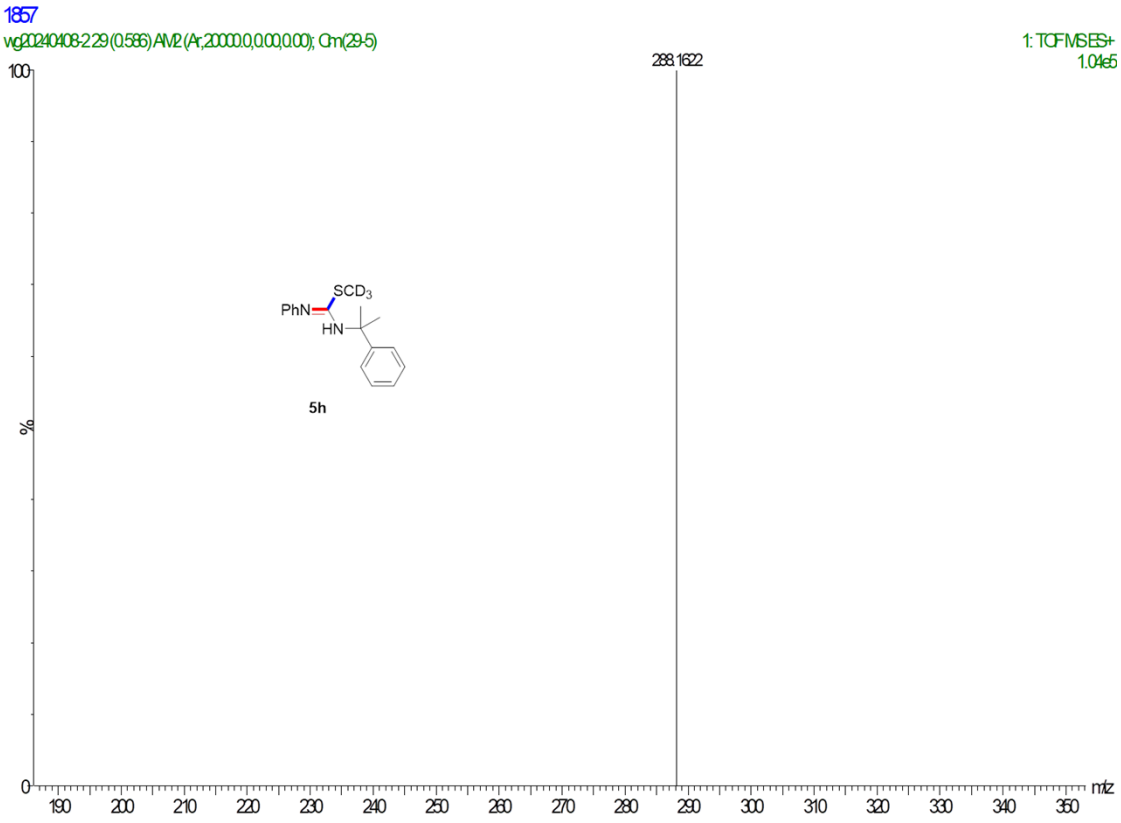

1841
wg20240318-8 50 (1.001) AM2 (Ar,20000.0,0.00,0.00); Cm(50-4x1.500)
1: TOF MS ES+
4.00e6
274.1462
100
%
0
225 230 235 240 245 250 255 260 265 270 275 280 285 290 295 300 305 310 315 320
m/z
SCD3
PhN
N
H
5e

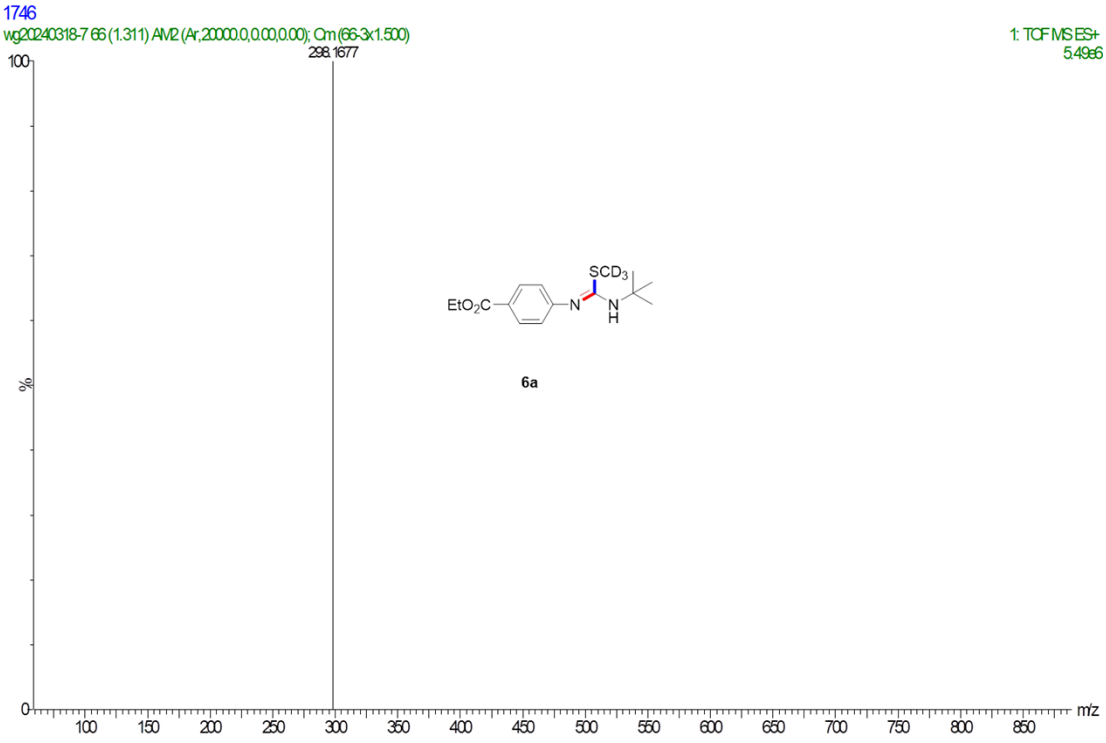

1845
wg20240318-1540 (0.792) AM2 (Ar,20000.0,0.00,0.00); Cm(40-6x1.500)
1: TOF MS ES+
1.96e7
438.1569
100
%
0
m/z
50 100 150 200 250 300 350 400 450 500 550 600 650 700 750 800 850 900 950 1000 1050 1100 1150
SCD3
O
O
O
O
N
N
H

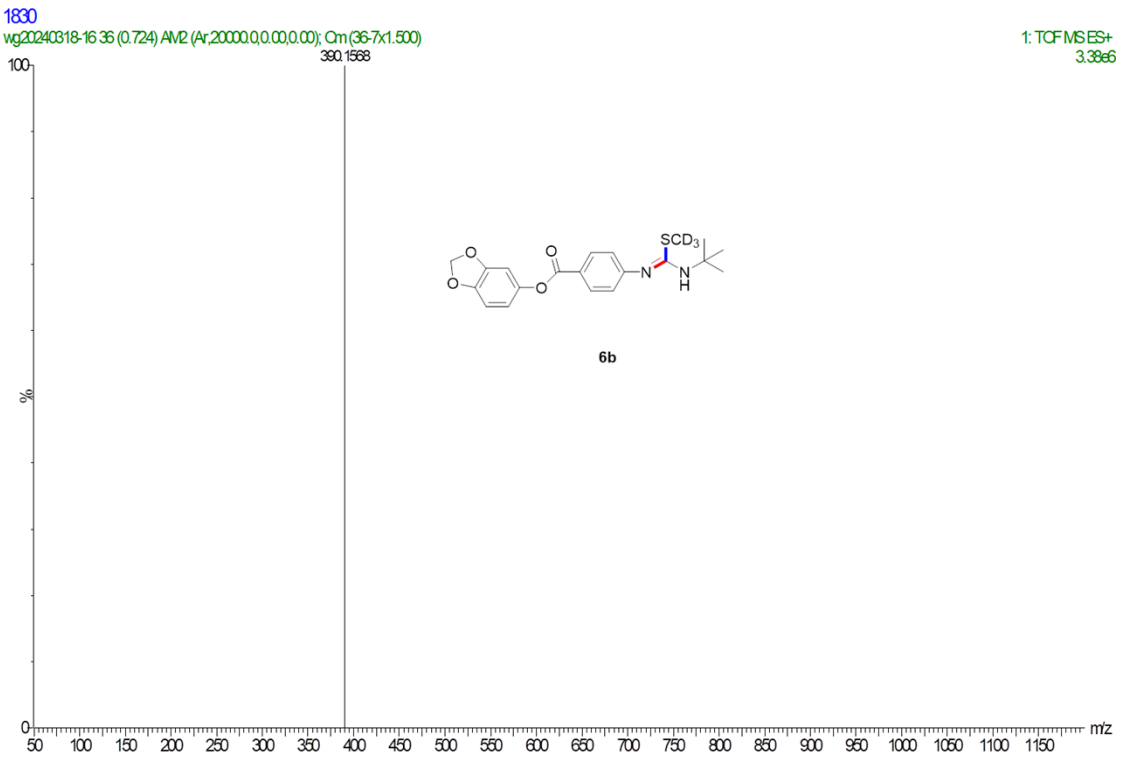
5f


1871
wg20240318-1457 (1.139) AM2 (Ar,20000.0,0.00,0.00); Cm(57-4x1.500)
1: TOF MS ES+
5.45e6
294.0917
100
%
0
m/z
50 100 150 200 250 300 350 400 450 500 550 600 650 700 750 800 850 900 950 1000 1050 1100 1150
SCD3
PhN
N
H
Cl

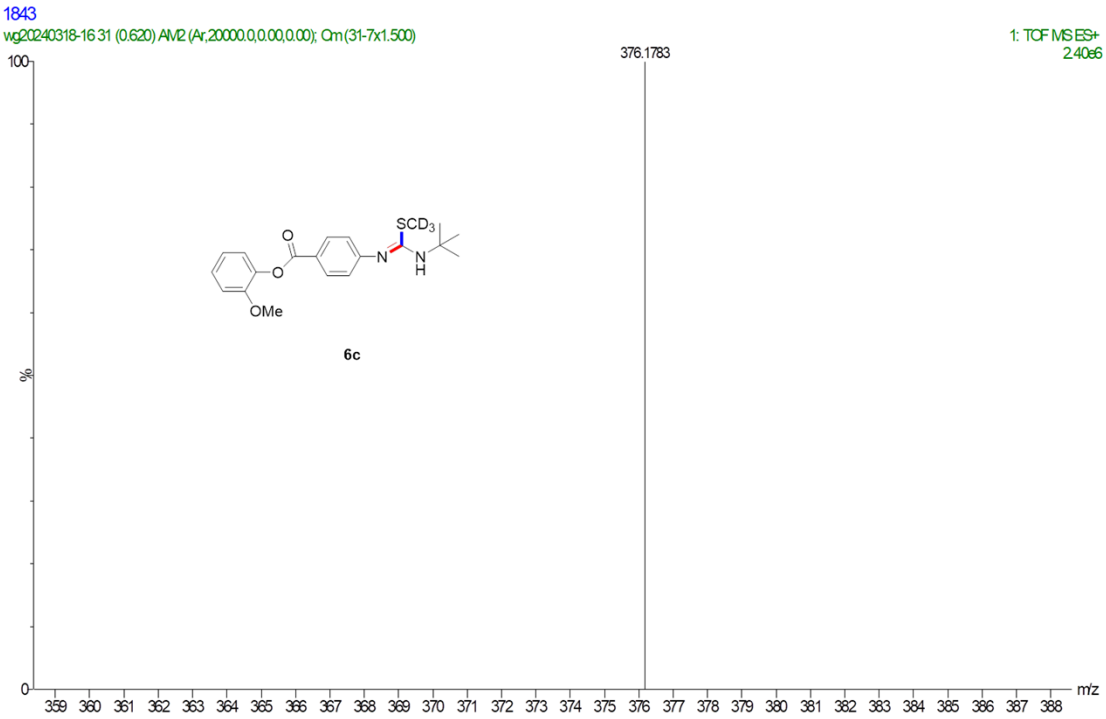
5g


1857
wg20240408-2 29 (0.586) AM2 (Ar,20000.0,0.00,0.00); Cm(29-5)
1: TOF MS ES+
1.04e5
288.1622
100
%
0
PhN
SCD3
HN
5h
190 200 210 220 230 240 250 260 270 280 290 300 310 320 330 340 350
m/z

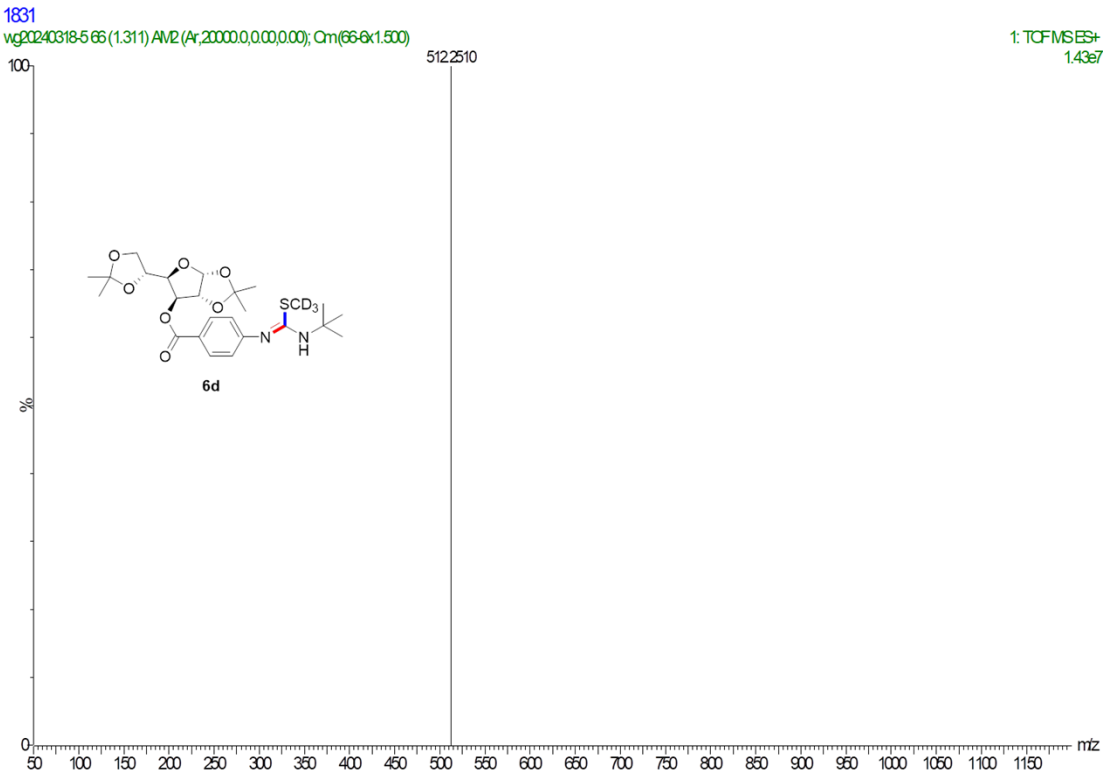

1746
wg20240318-7 66 (1.311) AM2 (Ar,20000.0,0.00,0.00); Cm (66-3x1.500)
1: TOF MS ES+
5.49e6
298.1677
100
%
0
EtO2C
N
SCD3
N
H
6a
100 150 200 250 300 350 400 450 500 550 600 650 700 750 800 850
m/z

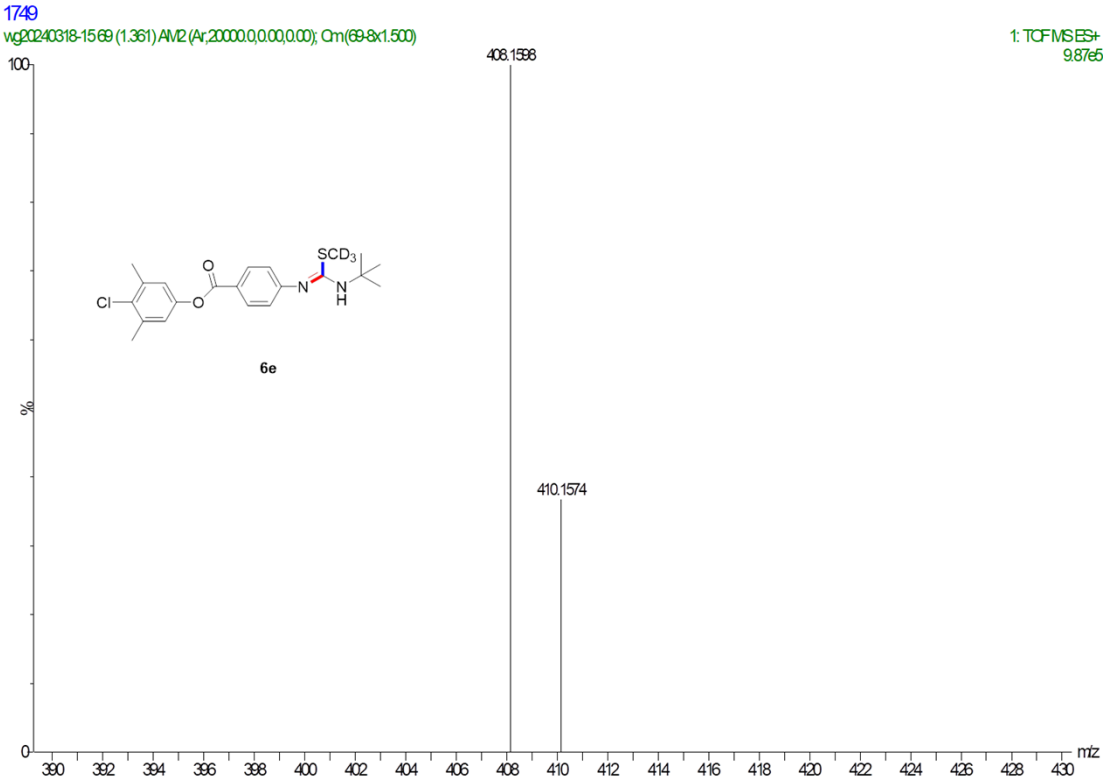

1830
wg20240318-16 36 (0.724) AM2 (Ar,20000.0,0.00,0.00); Cm (36-7x1.500)
1: TOF MS ES+
3.38e6
390.1568
100
%
0
50 100 150 200 250 300 350 400 450 500 550 600 650 700 750 800 850 900 950 1000 1050 1100 1150
m/z
SCD3
O
O
O
O
N
N
H

6b


1843
wg20240318-16 31 (0.620) AM2 (Ar,20000.0,0.00,0.00); Cm (31-7x1.500)
1: TOF MS ES+
2.40e6
376.1783
100
%
0
359 360 361 362 363 364 365 366 367 368 369 370 371 372 373 374 375 376 377 378 379 380 381 382 383 384 385 386 387 388
m/z
SCD3
O
O
N
N
H
OMe

6c


1831
wg20240318-5 66 (1.311) AM2 (Ar,20000.0,0.00,0.00); Cm(66-6x1.500)
1: TOF MS ES+
1.43e7
512.2510
100
%
0
SCD3
N
H
O
6d
50 100 150 200 250 300 350 400 450 500 550 600 650 700 750 800 850 900 950 1000 1050 1100 1150
m/z


1749
wg20240318-15 69 (1.361) AM2 (Ar,20000.0,0.00,0.00); Cm(69-8x1.500)
1: TOF MS ES+
9.87e5
408.1598
410.1574
100
%
0
Cl
O
SCD3
N
H
6e
390 392 394 396 398 400 402 404 406 408 410 412 414 416 418 420 422 424 426 428 430
m/z