Electronic Supplementary Information

Iodide-promoted transformations of imidazopyridines into sulfur-bridged imidazopyridines or 1,2,4-thiadiazoles

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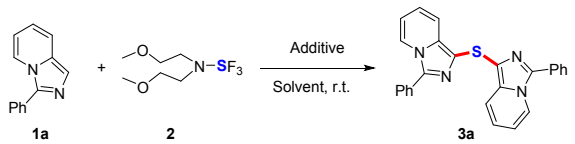
Contents:

1. General Information

Unless otherwise indicated, all commercial reagents and solvents were used without additional purification. Ammonium-^{15}N chloride (abundance: ≥98 atom % ^{15}N, chemical purity ≥ 98%) was purchased from Aladdin Reagents.^{1}H NMR spectra were recorded with Bruker AVANCE 600M or AVANCE 400M NMR spectrometer. Chemical shifts (in ppm) were referenced to tetramethylsilane in $CDCl_3$ as an internal standard. ^{13}C NMR spectra were obtained by the same NMR spectrometer and were calibrated with $CDCl_3$. HRMS were recorded on an AB SCIEX Triple TOF 6600 LC/MS (ESI) or Aglient 7250& JEOL-JMS-T100LP AccuTOF (EI).

2. Experimental Procedure

2.1 Table S1 Optimization of reaction conditions [a]



Entry	Additive	Solvent	Yield (%)[b]
1	NH_4I	EtOAc	71
2	**NaI**	**EtOAc**	**95**
3	TBAI	EtOAc	78
4	I_2	EtOAc	trace
5	NIS	EtOAc	9
6	I_2O_5	EtOAc	trace
7	$NaIO_4$	EtOAc	trace
8	$PhI(OAc)_2$	EtOAc	trace
9[c]	NaI	EtOAc	38
10	-	EtOAc	0
11	NaI	DCE	68
12	NaI	THF	trace
13	NaI	CH_3CN	86
14	NaI	DMSO	67
15	NaI	EtOH	45
16	NaI	PhMe	51
17[d]	NaI	EtOAc	87
18[e]	NaI	EtOAc	83

[a] Reaction conditions: **1a** (0.3 mmol), **2** (0.55 mmol), additive (0.3 mmol), solvent (2 mL), room temperature, 15 minutes. [b] Isolated yield. [c] NaI (0.06 mmol) was used. [d] 0.5 mmol of **2** was used. [e] 0.6 mmol of **2** was used.

2.2 Synthesis of sulfur-bridged imidazopyridines 3

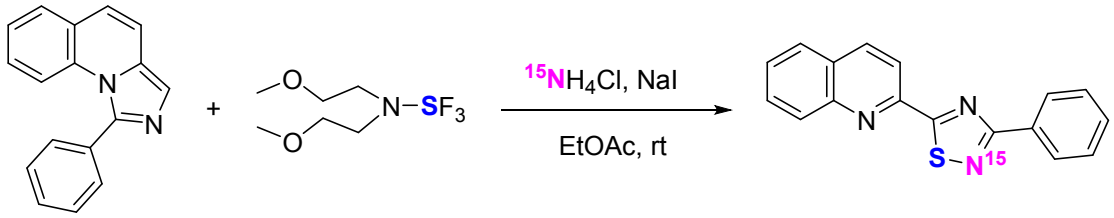
Imidazopyridines (0.3 mmol), NaI (0.3 mmol) and EtOAc (2 mL) were successively added to a 10 mL sealed tube, followed by addition of BAST (0.55 mmol). The mixture was stirred at room temperature for 15 minutes. The reaction was quenched with 20% aqueous sodium carbonate and extracted with CH_2Cl_2. The combined organic layers were dried over Na_2SO_4, filtered, and evaporated under vacuum. The residue was purified by column chromatography on silica gel to afford the desired sulfur-bridged imidazopyridines **3**.

2.3 Synthesis of 1,2,4-thiadiazoles 4

Method A: Imidazo[1,5-a]pyridines (0.3 mmol), NH_4I (0.3 mmol), and EtOAc (2 mL) were successively added to a 10 mL sealed tube, followed by addition of BAST (0.6 mmol). The mixture was stirred at room temperature for 12 h. The reaction was quenched with 20% aqueous sodium carbonate and extracted with CH_2Cl_2. The combined organic layers were dried over Na_2SO_4, filtered, and evaporated under vacuum. The residue was purified by column chromatography on silica gel to afford the 1,2,4-thiadiazoles **4**.

Method B: Imidazo[1,5-a]pyridines (0.3 mmol), NaI (0.3 mmol), NH_4Cl (0.6 mmol), and EtOAc (2 mL) were successively added to a 10 mL sealed tube, followed by addition of BAST (0.6 mmol). The mixture was stirred at room temperature for 12 h. The reaction was quenched with 20% aqueous sodium carbonate and extracted with CH_2Cl_2. The combined organic layers were dried over Na_2SO_4, filtered, and evaporated under vacuum. The residue was purified by column chromatography on silica gel to afford the 1,2,4-thiadiazoles **4**.

3. ^{15}N Labeling Experiment

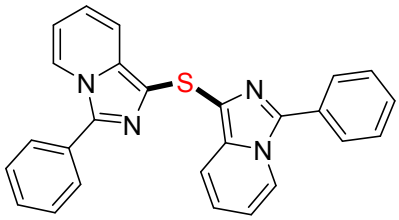


4l

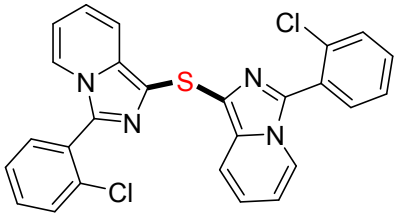
1-phenylimidazo[1,5-a]quinoline (**1t**, 0.3 mmol), NaI (0.3 mmol), $^{15}NH_4Cl$ (0.6 mmol), and EtOAc (2 mL) were successively added to a 10 mL sealed tube, followed by addition of BAST (0.6 mmol). The mixture was stirred at room temperature for 12 h. The reaction was quenched with 20% aqueous sodium carbonate and extracted with CH_2Cl_2. The combined organic layers were dried over Na_2SO_4, filtered, and evaporated under vacuum. The residue was purified by column chromatography on silica gel to afford the ^{15}N-labeled product **4l** in 78% yield.

4l: ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.44-8.38 (m, 3H), 8.33 (d, J = 8.5 Hz, 1H), 8.16 (d, J = 8.5 Hz, 1H), 7.87 (d, J = 8.1 Hz, 1H), 7.78 (t, J = 7.7 Hz, 1H), 7.62 (t, J = 7.6 Hz, 1H), 7.55-7.48 (m, 3H).; ^{13}C NMR (100 MHz, $CDCl_3$) δ 189.4, 173.9 (d, J = 3 Hz), 149.1, 147.9, 137.6, 133.0 (d, J = 8 Hz), 130.4, 130.4, 129.7, 129.3, 128.8, 128.3 (d, J = 2 Hz), 128.1, 127.8, 118.1; HRMS (ESI): calcd for $C_{17}H_{12}{}^{15}NN_2S$ $[M+H]^+$ 291.0717, found 291.0719.

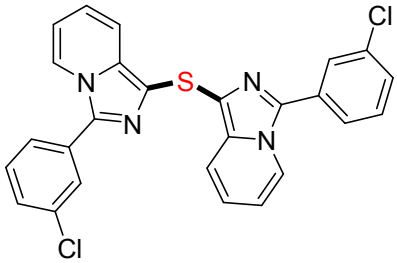
4. Characterization Data of Products



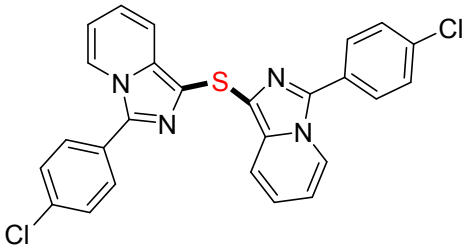
bis(3-phenylimidazo[1,5-a]pyridin-1-yl)sulfane (**3a**): mp: 205-208 °C; ^{1}H NMR (600 MHz, $CDCl_3$) δ 8.17 (dd, J = 7.2, 1.2 Hz, 2H), 8.08 (d, J = 9.2 Hz, 2H), 7.76-7.73 (m, 4H), 7.50-7.46 (m, 4H), 7.41 (t, J = 7.4 Hz, 2H), 6.85 (dd, J = 9.2, 6.4 Hz, 2H), 6.59 (td, J = 6.9, 6.3, 1.2 Hz, 2H); ^{13}C NMR (150 MHz, $CDCl_3$) δ 137.7, 133.7, 129.4, 129.0, 128.9, 128.2, 122.6, 121.6, 120.5, 119.5, 114.0; HRMS (ESI): calcd for $C_{26}H_{19}N_4S$ $[M+H]^+$ 419.1330, found 419.1325.



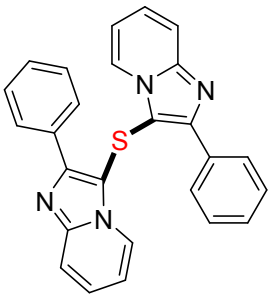
bis(3-(2-chlorophenyl)imidazo[1,5-a]pyridin-1-yl)sulfane (**3b**): mp: 170-172 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.96 (dd, J = 9.2, 1.2 Hz, 1H), 7.56 – 7.52 (m, 2H), 7.48 (dd, J = 7.8, 1.5 Hz, 1H), 7.41-7.33 (m, 2H), 6.82 (dd, J = 9.2, 6.3 Hz, 1H), 6.58-6.54 (m, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 135.3, 134.2, 133.4, 133.2, 130.9, 129.9, 128.9, 127.2, 122.7, 122.5, 120.4, 119.0, 113.3; HRMS (ESI): calcd for $C_{26}H_{17}Cl_2N_4S$ $[M+H]^+$ 487.0551, found 487.0546.



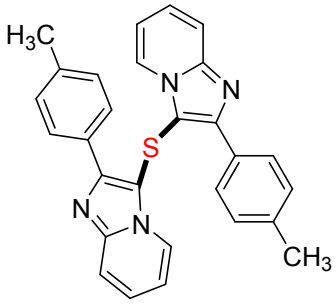
bis(3-(3-chlorophenyl)imidazo[1,5-a]pyridin-1-yl)sulfane (**3c**): mp: 163-165 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.12 (d, J = 7.2 Hz, 2H), 7.99 (dt, J = 9.2, 1.3 Hz, 2H), 7.72 (t, J = 1.8 Hz, 2H), 7.58 (dt, J = 7.5, 1.7 Hz, 2H), 7.38 – 7.30 (m, 4H), 6.83 (dd, J = 9.2, 6.4 Hz, 2H), 6.60 – 6.56 (m, 2H).; ^{13}C NMR (100 MHz, $CDCl_3$) δ 136.4, 134.9, 133.9, 131.5, 130.2, 128.8, 128.1, 125.9, 123.4, 121.4, 120.7, 119.3, 114.2; HRMS (ESI): calcd for $C_{26}H_{17}Cl_2N_4S$ $[M+H]^+$ 487.0551, found 487.0546.



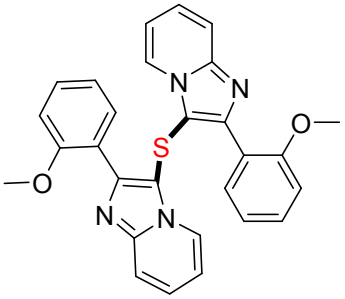
bis(3-(4-chlorophenyl)imidazo[1,5-a]pyridin-1-yl)sulfane (**3d**): mp: 189-192 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.13 – 8.06 (m, 4H), 7.70 – 7.67 (m, 4H), 7.46 – 7.43 (m, 4H), 6.88 (ddd, J = 9.2, 6.4, 0.9 Hz, 2H), 6.65-6.61 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 136.8, 134.8, 133.9, 129.4, 129.3, 128.3, 123.3, 121.5, 120.6, 119.5, 114.2; HRMS (ESI): calcd for $C_{26}H_{17}Cl_2N_4S$ $[M+H]^+$ 487.0551, found 487.0546.



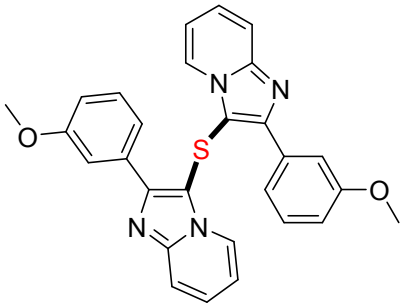
bis(2-phenylimidazo[1,2-a]pyridin-3-yl)sulfane (**3e**): pale yellow solid, mp: 249-252 °C; ^{1}H NMR (600 MHz, $CDCl_3$) δ 8.10-8.09 (m, 4H), 7.62-7.59 (m, 4H), 7.56-7.53 (m, 4H), 7.49 (d, J = 8.9 Hz, 2H), 7.09-7.06 (m, 2H), 6.33 (td, J = 6.9, 1.1 Hz, 2H); ^{13}C NMR (150 MHz, $CDCl_3$) δ 150.8, 146.5, 133.7, 129.5, 128.9, 128.6, 126.4, 125.3, 117.3, 112.6, 107.5; HRMS (ESI): calcd for $C_{26}H_{19}N_4S$ $[M+H]^+$ 419.1330, found 419.1325.



bis(2-(p-tolyl)imidazo[1,2-a]pyridin-3-yl)sulfane (**3f**): pale yellow solid, mp: 262-263 °C; ^{1}H NMR (600 MHz, $CDCl_3$) δ 8.03-8.02 (m, 4H), 7.60 (dt, J = 6.9, 1.2 Hz, 2H), 7.53 (d, J = 8.9 Hz, 2H), 7.42 (d, J = 7.9 Hz, 4H), 7.12 (ddd, J = 8.5, 6.8, 1.3 Hz, 2H), 6.38 (td, J = 6.9, 1.2 Hz, 2H), 2.52 (s, 6H); ^{13}C NMR (150 MHz, $CDCl_3$) δ 150.5, 146.2, 139.0, 130.4, 129.4, 129.3, 126.6, 125.4, 117.2, 112.8, 107.0, 21.5; HRMS (ESI): calcd for $C_{28}H_{23}N_4S$ $[M+H]^+$ 447.1643, found 447.1657.

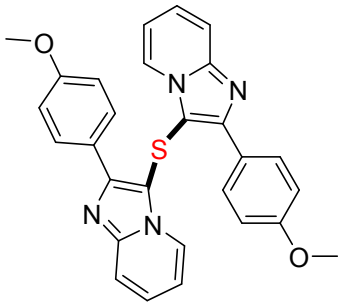


bis(2-(2-methoxyphenyl)imidazo[1,2-a]pyridin-3-yl)sulfane (**3g**): pale yellow solid, mp: 196-197 °C; ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.55-7.52 (m, 4H), 7.51-7.48 (m, 4H), 7.17 (td, J = 7.4, 1.0 Hz, 2H), 7.13 (dd, J = 8.7, 1.0 Hz, 2H), 7.09 (ddd, J = 8.9, 6.8, 1.4 Hz, 2H), 6.42 (td, J = 6.8, 1.2 Hz, 2H), 3.84 (s, 6H); ^{13}C NMR (150 MHz, $CDCl_3$) δ 157.5, 148.5, 146.4, 132.6, 130.5, 125.6, 124.9, 123.1, 120.6, 117.4, 112.2, 111.2, 109.8, 55.6; HRMS (ESI): calcd for $C_{28}H_{23}N_4O_2S$ $[M+H]^+$ 479.1536, found 479.1541.

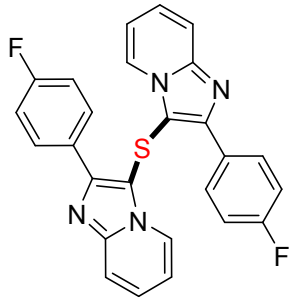


bis(2-(3-methoxyphenyl)imidazo[1,2-a]pyridin-3-yl)sulfane (**3h**): pale yellow solid, mp: 153-155 °C; ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.73 (dt, J = 7.6, 1.2 Hz, 2H), 7.65-7.64 (m, 4H), 7.60 (d, J = 9.0 Hz, 2H), 7.54 (t, J = 7.9 Hz, 2H), 7.18 (ddd, J = 8.5, 6.9, 1.2 Hz, 2H), 7.13 (ddd, J = 8.3, 2.6, 0.9 Hz, 2H), 6.45 (td, J = 6.9, 1.1 Hz, 2H), 3.95 (s, 6H); ^{13}C NMR (150 MHz, $CDCl_3$) δ 159.9, 149.9, 145.9, 134.1, 129.8,

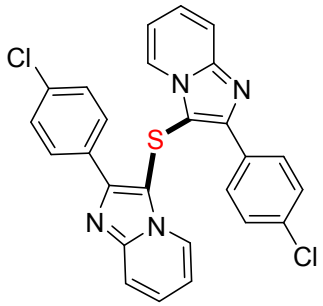
127.3, 125.5, 122.0, 117.1, 115.7, 114.3, 113.3, 107.5, 55.6; HRMS (ESI): calcd for $C_{28}H_{23}N_4O_2S$ $[M+H]^+$ 479.1536,



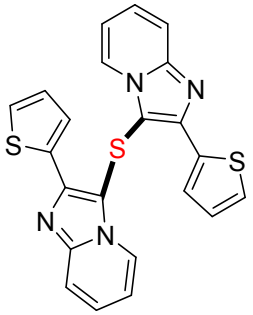
bis(2-(4-methoxyphenyl)imidazo[1,2-a]pyridin-3-yl)sulfane (**3i**): pale yellow solid, mp: 221-223 °C; ^{1}H NMR (600 MHz, $CDCl_3$) δ 8.11-8.08 (m, 4H), 7.63 (dt, J = 6.9, 1.2 Hz, 2H), 7.59 (d, J = 8.9 Hz, 2H), 7.18-7.14 (m, 6H), 6.46 (td, J = 6.9, 1.1 Hz, 2H), 3.95 (s, 6H); ^{13}C NMR (150 MHz, $CDCl_3$) δ 160.5, 149.6, 145.8, 130.9, 127.2, 125.4, 125.1, 116.9, 114.2, 113.2, 106.5, 55.5; HRMS (ESI): calcd for $C_{28}H_{23}N_4O_2S$ $[M+H]^+$ 479.1542, found 479.1538.



bis(2-(4-fluorophenyl)imidazo[1,2-a]pyridin-3-yl)sulfane (**3j**): pale yellow solid, mp: 249-252 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.14 – 8.09 (m, 4H), 7.57 (ddt, J = 16.8, 9.0, 1.1 Hz, 4H), 7.35 – 7.29 (m, 4H), 7.17 (ddd, J = 8.9, 6.8, 1.3 Hz, 2H), 6.46 (td, J = 6.8, 1.2 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 163.5 ($^1J_{CF}$ = 248 Hz), 149.8, 146.4, 131.4 ($^3J_{CF}$ = 8 Hz), 129.7 ($^4J_{CF}$ = 2 Hz), 127.0, 125.2, 117.5, 115.9 ($^2J_{CF}$ = 21 Hz), 113.1, 107.1; HRMS (ESI): calcd for $C_{26}H_{17}F_2N_4S$ $[M+H]^+$ 455.1141, found 455.1165.

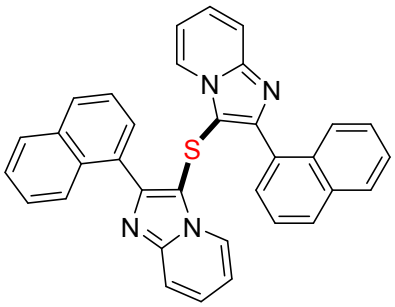


bis(2-(4-chlorophenyl)imidazo[1,2-a]pyridin-3-yl)sulfane (**3k**): pale yellow solid, mp: 295-297 °C; ^{1}H NMR (600 MHz, $CDCl_3$) δ 8.10-8.08 (m, 4H), 7.62-7.59 (m, 8H), 7.21 (td, J = 7.0, 3.4 Hz, 2H), 6.52-6.50 (m, 2H); ^{13}C NMR (150 MHz, $CDCl_3$) δ 149.0, 146.1, 135.4, 131.4, 130.7, 129.0, 127.4, 125.1, 117.4, 113.5, 107.1; HRMS (ESI): calcd for $C_{26}H_{17}Cl_2N_4S$ $[M+H]^+$ 487.0545, found 487.0548.

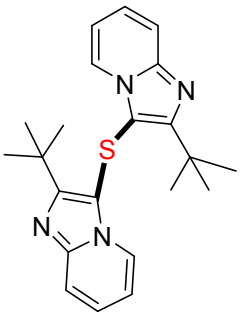


bis(2-(thiophen-2-yl)imidazo[1,2-a]pyridin-3-yl)sulfane (**3l**): pale yellow solid, mp: 218-219 °C; ^{1}H NMR (600 MHz, $CDCl_3$) δ 8.17 (dd, J = 3.6, 1.0 Hz, 2H), 7.94 (dt, J = 6.9, 1.1 Hz, 2H), 7.56-7.52 (m,

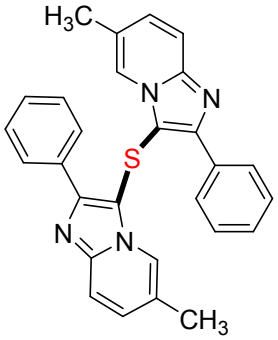
4H), 7.29 (dd, J = 5.1, 3.6 Hz, 2H), 7.14 (ddd, J = 8.6, 6.9, 1.2 Hz, 2H), 6.55 (td, J = 6.8, 1.1 Hz, 2H); ^{13}C NMR (150 MHz, $CDCl_3$) δ 146.8, 145.2, 136.5, 127.9, 127.4, 126.7, 125.2, 117.3, 113.2, 104.5; HRMS (ESI): calcd for $C_{22}H_{15}N_4S_3$ $[M+H]^+$ 431.0459, found 431.0453.



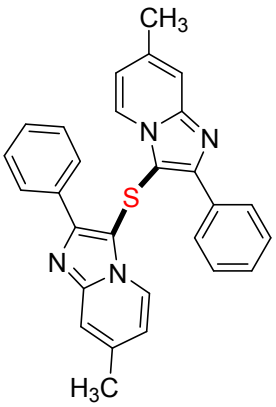
bis(2-(naphthalen-1-yl)imidazo[1,2-a]pyridin-3-yl)sulfane (3m): pale yellow solid, mp: 258-260 °C; ^{1}H NMR (600 MHz, $CDCl_3$) δ 8.68-8.63 (m, 2H), 8.30 (dd, J = 8.4, 1.7 Hz, 2H), 8.10 (d, J = 8.5 Hz, 2H), 8.06-8.04 (m, 2H), 8.00-7.98 (m, 2H), 7.63-7.56 (m, 8H), 7.09 (ddd, J = 8.7, 6.9, 1.3 Hz, 2H), 6.18 (td, J = 6.9, 1.1 Hz, 2H); ^{13}C NMR (150 MHz, $CDCl_3$) δ 150.2, 146.3, 133.5, 133.3, 130.5, 129.0, 128.7, 128.4, 127.9, 126.9, 126.9, 126.8, 126.6, 125.3, 117.2, 113.0, 107.3; HRMS (ESI): calcd for $C_{34}H_{23}N_4S$ $[M+H]^+$ 519.1643, found 519.1638.



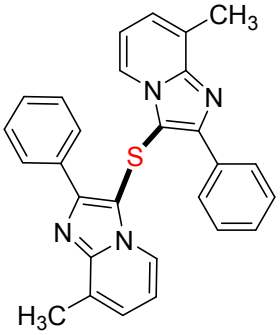
bis(2-(tert-butyl)imidazo[1,2-a]pyridin-3-yl)sulfane (3n): pale yellow oil; ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.83 (dt, J = 6.9, 1.2 Hz, 2H), 7.54 (dt, J = 8.9, 1.2 Hz, 2H), 7.06 (ddd, J = 8.8, 6.8, 1.3 Hz, 2H), 6.54 (td, J = 6.9, 1.2 Hz, 2H), 1.64 (s, 18H); ^{13}C NMR (150 MHz, $CDCl_3$) δ 158.0, 145.0, 124.6, 123.6, 117.3, 112.5, 102.6, 34.1, 30.7; HRMS (ESI): calcd for $C_{22}H_{27}N_4S$ $[M+H]^+$ 379.1956, found 379.1951.



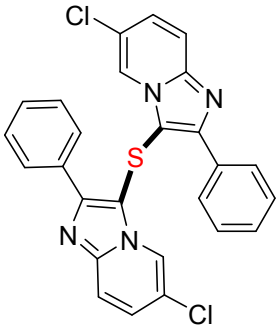
bis(6-methyl-2-phenylimidazo[1,2-a]pyridin-3-yl)sulfane (3o): pale yellow solid, mp: 286-287 °C; ^{1}H NMR (600 MHz, $CDCl_3$) δ 8.19-8.17 (m, 4H), 7.65 (t, J = 7.7 Hz, 4H), 7.58-7.55 (m, 2H), 7.46-7.42 (m, 4H), 6.99-6.98 (m, 2H), 1.75 (s, 6H); ^{13}C NMR (150 MHz, $CDCl_3$) δ 149.9, 145.2, 133.6, 129.9, 129.4, 128.9, 128.8, 123.7, 122.9, 116.4, 106.8, 17.8; HRMS (ESI): calcd for $C_{28}H_{23}N_4S$ $[M+H]^+$ 447.1638, found 447.1640.



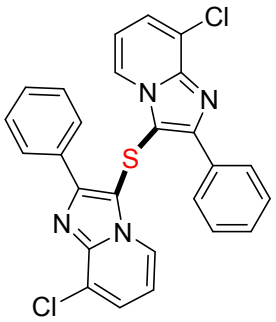
bis(7-methyl-2-phenylimidazo[1,2-a]pyridin-3-yl)sulfane (3p): pale yellow solid, mp: 195-197 °C; ^{1}H NMR (600 MHz, $CDCl_3$) δ 8.11-8.09 (m, 4H), 7.62-7.59 (m, 4H), 7.55-7.53 (m, 2H), 7.41 (dd, J = 7.0, 0.9 Hz, 2H), 7.25-7.24 (m, 2H), 6.17 (dd, J = 7.1, 1.7 Hz, 2H), 2.26 (s, 6H); ^{13}C NMR (150 MHz, $CDCl_3$) δ 150.6, 146.9, 137.6, 133.9, 129.5, 128.7, 128.5, 124.5, 115.9, 115.1, 106.7, 21.2; HRMS (ESI): calcd for $C_{28}H_{23}N_4S$ $[M+H]^+$ 447.1638, found 447.1641.



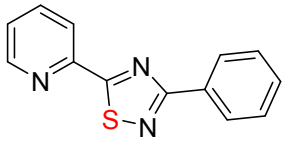
bis(8-methyl-2-phenylimidazo[1,2-a]pyridin-3-yl)sulfane (**3q**): pale yellow solid, mp: 217-219 °C; ^{1}H NMR (600 MHz, $CDCl_3$) δ 8.09-8.07 (m, 4H), 7.62-7.59 (m, 4H), 7.56-7.53 (m, 2H), 7.42 (d, J = 6.8 Hz, 2H), 6.90 (d, J = 6.9 Hz, 2H), 6.28 (t, J = 6.9 Hz, 2H), 2.54 (s, 6H); ^{13}C NMR (150 MHz, $CDCl_3$) δ 150.2, 146.5, 133.8, 129.8, 128.8, 128.5, 127.3, 125.4, 123.2, 112.6, 108.0, 16.7; HRMS (ESI): calcd for $C_{28}H_{23}N_4S$ $[M+H]^+$ 447.1638, found 447.1642.



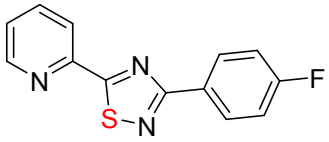
bis(6-chloro-2-phenylimidazo[1,2-a]pyridin-3-yl)sulfane (3r): pale yellow solid, mp: 264-267 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.09 – 8.06 (m, 4H), 7.67 – 7.62 (m, 4H), 7.60 – 7.55 (m, 4H), 7.46 (dd, J = 9.4, 0.9 Hz, 2H), 7.08 (dd, J = 9.4, 2.0 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 151.8, 144.9, 132.8, 129.5, 129.3, 129.0, 128.1, 123.7, 121.3, 117.7, 107.7; HRMS (ESI): calcd for $C_{26}H_{17}Cl_2N_4S$ $[M+H]^+$ 487.0551, found 487.0546.



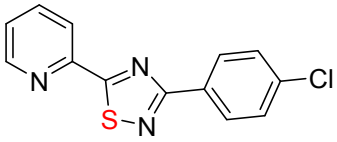
bis(8-chloro-2-phenylimidazo[1,2-a]pyridin-3-yl)sulfane (3s): pale yellow solid, mp: 272-273 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.09 (d, J = 7.1 Hz, 4H), 7.63-7.55 (m, 6H), 7.43 (d, J = 6.6 Hz, 2H), 7.17 (d, J = 7.1 Hz, 2H), 6.28 (t, J = 6.9 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 151.5, 143.9, 133.1, 129.8, 129.3, 128.7, 125.5, 124.0, 123.2, 112.3, 109.2; HRMS (ESI): calcd for $C_{26}H_{17}Cl_2N_4S$ $[M+H]^+$ 487.0551, found 487.0546.



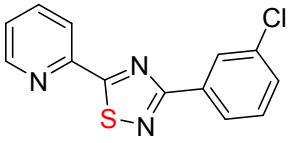
3-phenyl-5-(pyridin-2-yl)-1,2,4-thiadiazole (4a): white solid, mp: 126-128°C; ^{1}H NMR (600 MHz, $CDCl_3$) δ 8.68 (d, J = 4.7 Hz, 1H), 8.40-8.38 (m, 2H), 8.31 (d, J = 7.8 Hz, 1H), 7.90 (td, J = 7.7, 1.6 Hz, 1H), 7.53-7.47 (m, 3H), 7.44 (ddd, J = 7.6, 4.7, 1.2 Hz, 1H); ^{13}C NMR (150 MHz, $CDCl_3$) δ 189.0, 173.9, 149.9, 149.3, 137.5, 132.9, 130.3, 128.7, 128.2, 126.2, 120.6; HRMS (ESI): calcd for $C_{13}H_{10}N_3S$ $[M+H]^+$ 240.0595, found 240.0590.



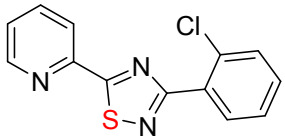
3-(4-fluorophenyl)-5-(pyridin-2-yl)-1,2,4-thiadiazole (4b): white solid, mp: 165-167 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.67 (d, J = 4.8 Hz, 1H), 8.37 (dd, J = 8.5, 5.5 Hz, 2H), 8.26 (d, J = 7.9 Hz, 1H), 7.88 (t, J = 7.8 Hz, 1H), 7.43 (dd, J = 7.6, 4.9 Hz, 1H), 7.18 (t, J = 8.5 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 189.3, 172.9, 164.2 ($^1J_{CF}$ = 248 Hz), 150.0, 149.2, 137.5, 130.3 ($^3J_{CF}$ = 9 Hz), 129.3 ($^4J_{CF}$ = 3 Hz), 126.2, 120.5, 115.7 ($^2J_{CF}$ = 22 Hz); HRMS (EI): calcd for $C_{13}H_8FN_3S$ $[M]^+$ 257.0419, found 257.0417.



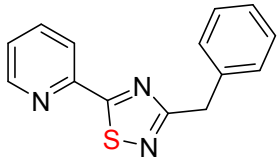
3-(4-chlorophenyl)-5-(pyridin-2-yl)-1,2,4-thiadiazole (4c): white solid, mp: 175-178 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.67 (d, J = 4.6 Hz, 1H), 8.29 (dd, J = 20.4, 7.9 Hz, 3H), 7.89 (t, J = 7.7 Hz, 1H), 7.45 (dd, J = 14.1, 6.9 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 189.4, 172.8, 150.0, 149.2, 137.5, 136.4, 131.4, 129.5, 129.0, 126.3, 120.5; HRMS (EI): calcd for $C_{13}H_8ClN_3S$ $[M]^+$ 273.0124, found 273.0122.



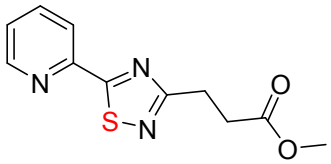
3-(3-chlorophenyl)-5-(pyridin-2-yl)-1,2,4-thiadiazole (4d): white solid, mp: 161-163 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.69-8.67 (m, 1H), 8.38 (d, J = 2.0 Hz, 1H), 8.29-8.23 (m, 2H), 7.90 (tt, J = 7.8, 1.9 Hz, 1H), 7.44 (td, J = 5.8, 2.8 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 189.5, 172.5, 150.0, 149.2, 137.5, 134.7, 134.5, 130.3, 130.0, 128.4, 126.3, 126.2, 120.6; HRMS (EI): calcd for $C_{13}H_8ClN_3S$ $[M]^+$ 273.0127, found 273.0122.



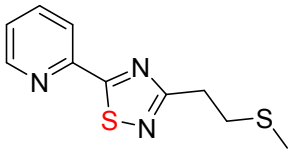
3-(2-chlorophenyl)-5-(pyridin-2-yl)-1,2,4-thiadiazole (4e): white solid, mp: 104-105 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.68 (d, J = 4.8 Hz, 1H), 8.25 (d, J = 7.9 Hz, 1H), 8.02 (dd, J = 6.7, 3.0 Hz, 1H), 7.87 (t, J = 7.7 Hz, 1H), 7.55-7.53 (m, 1H), 7.45-7.38 (m, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 188.8, 172.0, 150.0, 149.2, 137.5, 133.2, 132.2, 132.1, 130.9, 130.8, 126.8, 126.3, 120.6. HRMS (EI): calcd for $C_{13}H_8ClN_3S$ $[M]^+$ 273.0120, found 273.0122.



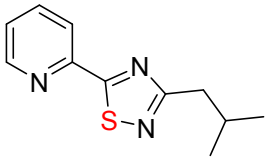
3-benzyl-5-(pyridin-2-yl)-1,2,4-thiadiazole (4f): pale yellow solid, mp: 83-85 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.63 (d, J = 4.8 Hz, 1H), 8.15-8.13 (m, 1H), 7.82 (tt, J = 7.8, 1.5 Hz, 1H), 7.41-7.31 (m, 5H), 7.26-7.22 (m, 1H), 4.39 (s, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 189.3, 176.2, 149.9, 149.2, 137.4, 137.2, 129.1, 128.6, 126.8, 126.1, 120.5, 39.5; HRMS (EI): calcd for $C_{14}H_{11}N_3S$ $[M]^+$ 253.0671, found 253.0668.



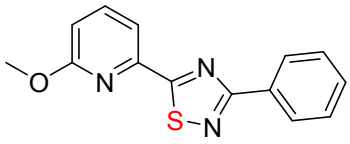
methyl 3-(5-(pyridin-2-yl)-1,2,4-thiadiazol-3-yl)propanoate (4g): colorless oil; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.62 (d, J = 4.8 Hz, 1H), 8.11 (d, J = 7.8 Hz, 1H), 7.84 (td, J = 7.8, 1.9 Hz, 1H), 7.41-7.37 (m, 1H), 3.69 (s, 3H), 3.35 (t, J = 7.4 Hz, 2H), 2.94 (t, J = 7.4 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 189.1, 176.0, 173.0, 150.0, 149.2, 137.4, 126.1, 120.4, 51.8, 31.8, 28.3; HRMS (EI): calcd for $C_{11}H_{11}N_3O_2S$ $[M]^+$ 249.0565, found 249.0566.



3-(2-(methylthio)ethyl)-5-(pyridin-2-yl)-1,2,4-thiadiazole (4h): colorless oil; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.64 (d, J = 4.7 Hz, 1H), 8.13 (dt, J = 7.9, 1.2 Hz, 1H), 7.84 (td, J = 7.7, 1.9 Hz, 1H), 7.42-7.38 (m, 1H), 3.35-3.30 (m, 2H), 3.05 (t, J = 7.5 Hz, 2H), 2.15 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 189.1, 176.0, 150.0, 149.2, 137.4, 126.1, 120.4, 33.2, 32.2, 15.5; HRMS (EI): calcd for $C_{10}H_{11}N_3S_2$ $[M]^+$ 237.0388, found 237.0389.

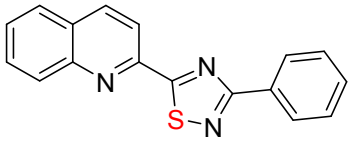


3-isobutyl-5-(pyridin-2-yl)-1,2,4-thiadiazole (4i): colorless oil; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.64 (d, J = 4.8 Hz, 1H), 8.15 (d, J = 7.9 Hz, 1H), 7.84 (td, J = 7.8, 1.9 Hz, 1H), 7.41-7.38 (m, 1H), 2.91 (d, J = 7.2 Hz, 2H), 2.36-2.26 (m, 1H), 0.99 (d, J = 6.8 Hz, 6H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 188.6, 177.7, 149.9, 149.4, 137.4, 126.0, 120.4, 42.1, 28.3, 22.5; HRMS (EI): calcd for $C_{11}H_{13}N_3S$ $[M]^+$ 219.0821, found 219.0825.



5-(6-methoxypyridin-2-yl)-3-phenyl-1,2,4-thiadiazole (4j): pale yellow solid, mp: 103-105 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.40-8.37 (m, 2H), 7.88 (d, J = 7.2 Hz, 1H), 7.74 (t, J = 7.8 Hz, 1H), 7.53-

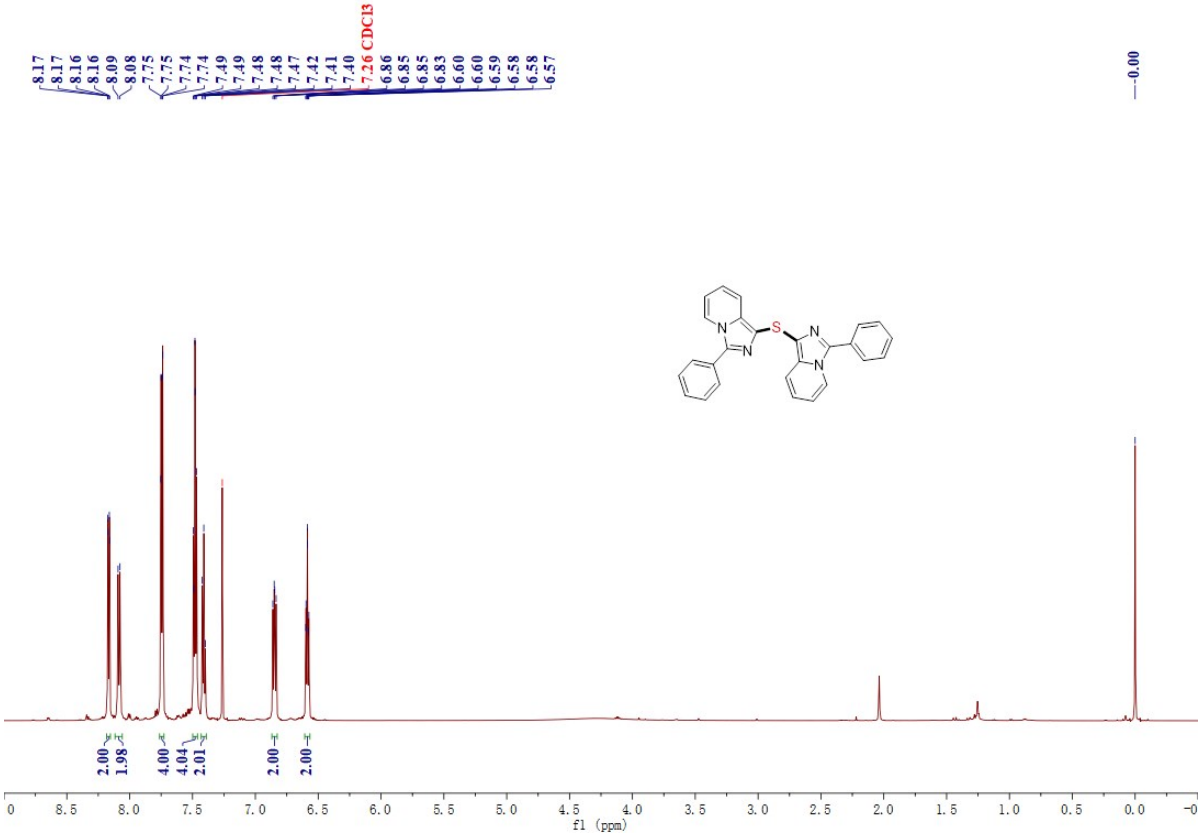
7.47 (m, 3H), 6.89 (d, J = 8.3 Hz, 1H), 4.02 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 189.2, 173.9, 163.8, 146.4, 139.7, 133.0, 130.3, 128.7, 128.2, 114.4, 113.3, 53.7; HRMS (EI): calcd for $C_{14}H_{10}N_3OS$ $[M]^+$ 268.0539, found 268.0539.

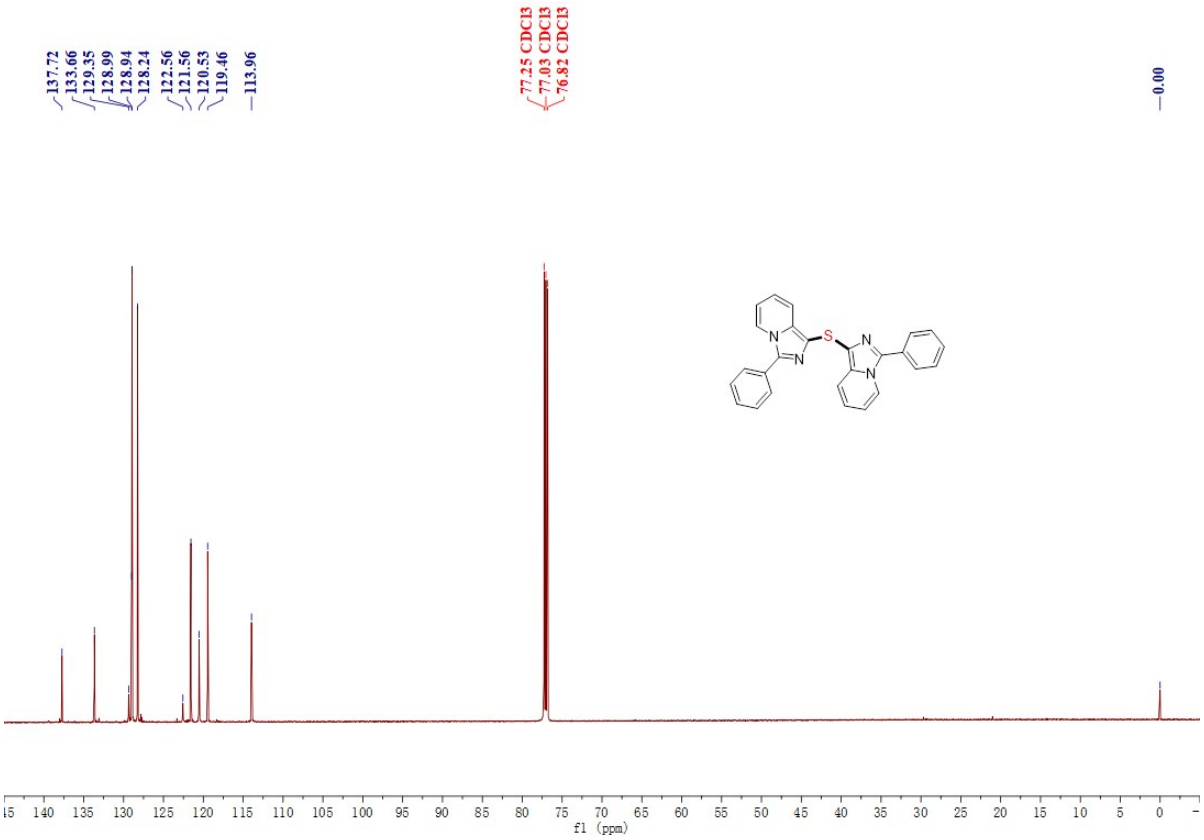


3-phenyl-5-(quinolin-2-yl)-1,2,4-thiadiazole (4k): white solid, mp: 171-173 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.44-8.37 (m, 3H), 8.32 (d, J = 8.5 Hz, 1H), 8.15 (d, J = 8.5 Hz, 1H), 7.86 (d, J = 8.2 Hz, 1H), 7.78 (ddd, J = 8.6, 6.8, 1.7 Hz, 1H), 7.63-7.59 (m, 1H), 7.55-7.48 (m, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 189.4, 173.9, 149.1, 147.9, 137.6, 133.0, 130.4, 130.4, 129.7, 129.3, 128.7, 128.3, 128.0, 127.8, 118.1; HRMS (EI): calcd for $C_{17}H_{11}N_3S$ $[M]^+$ 289.0665, found 289.0668.

5. ^{1}H NMR and ^{13}C NMR Spectra

3a





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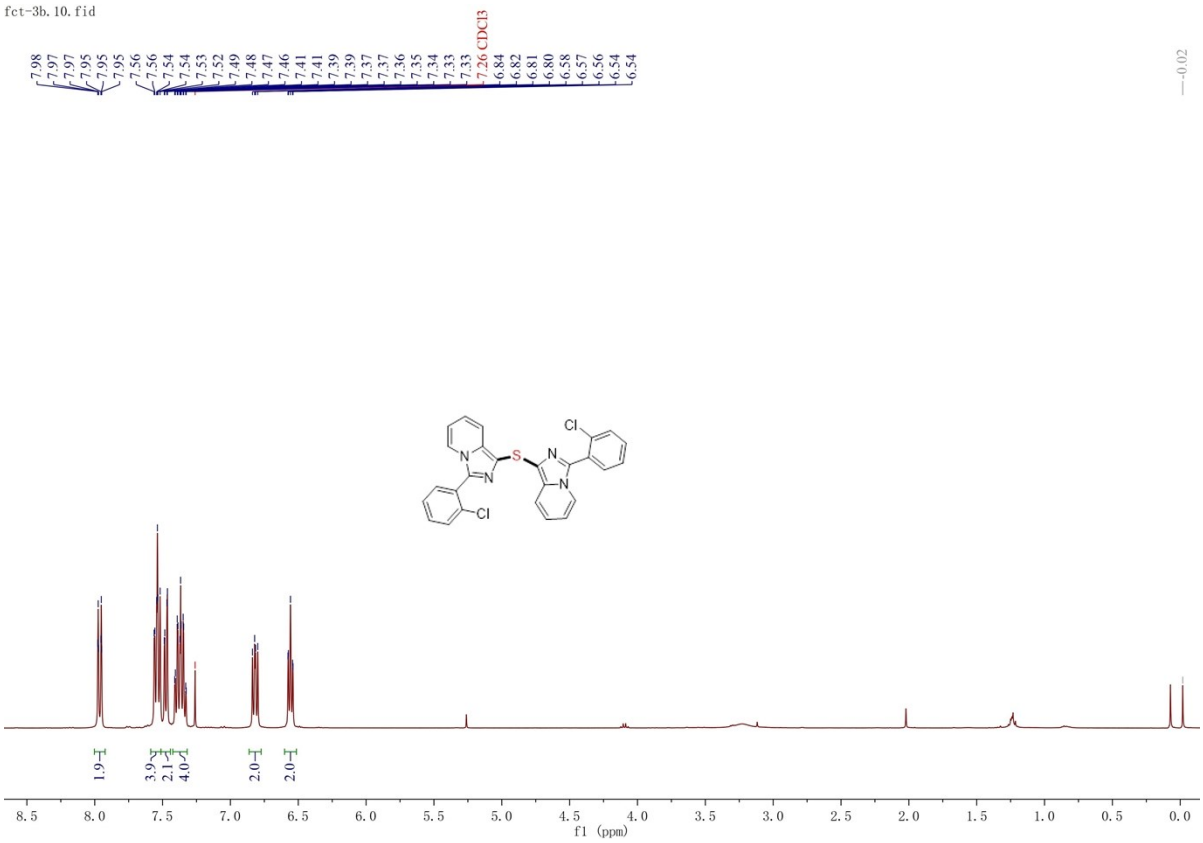
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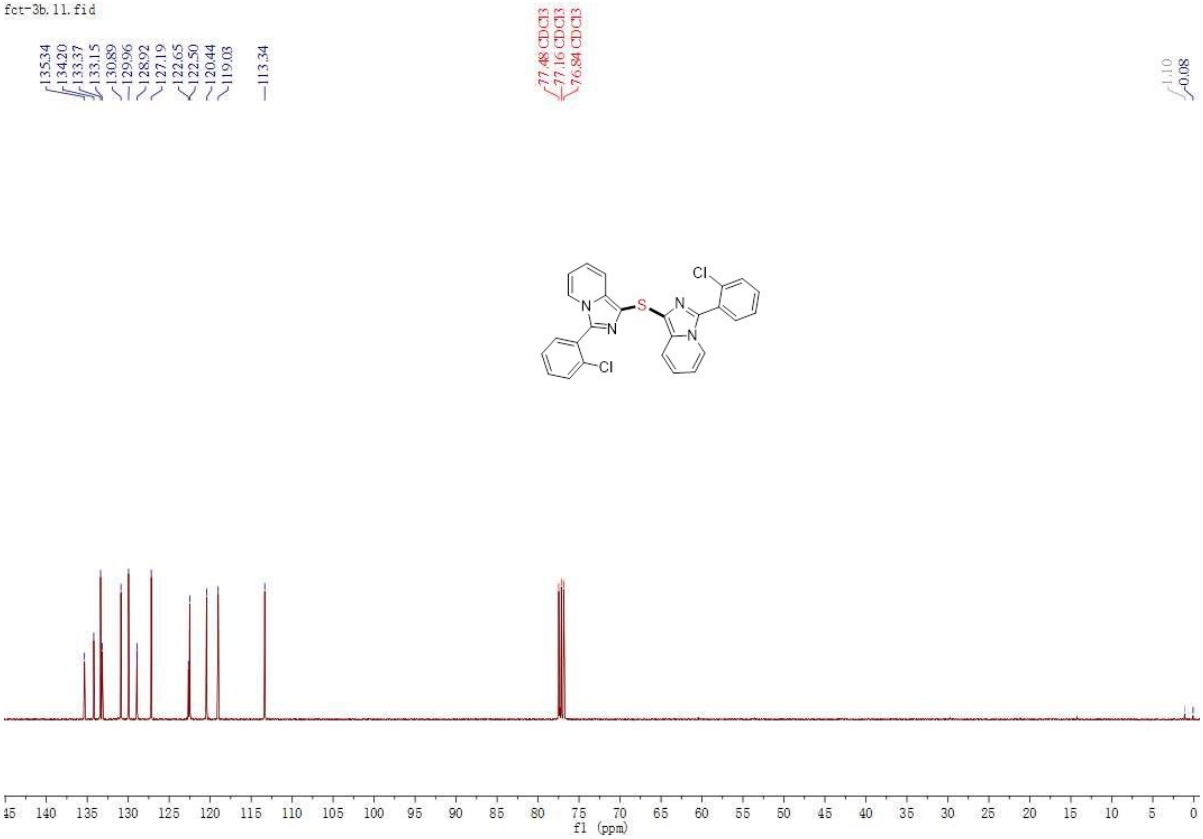
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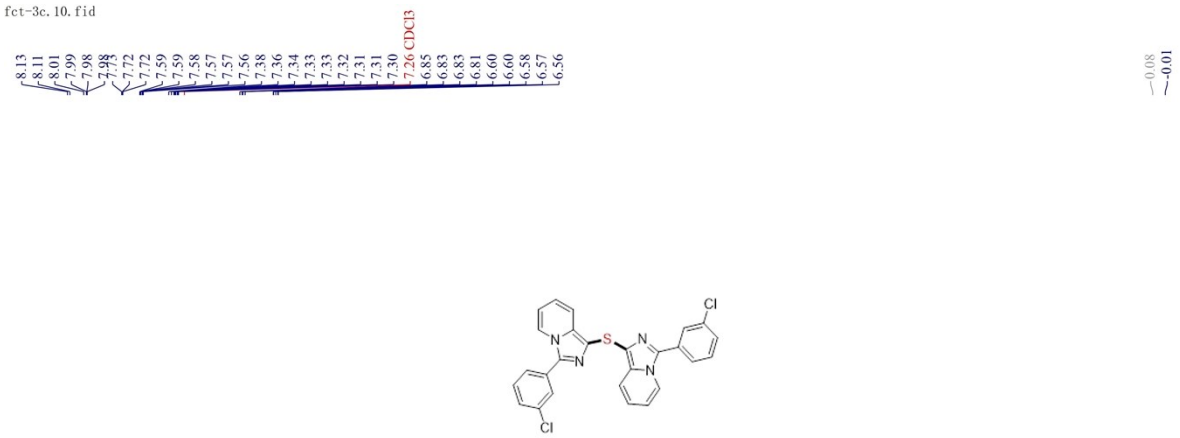
3c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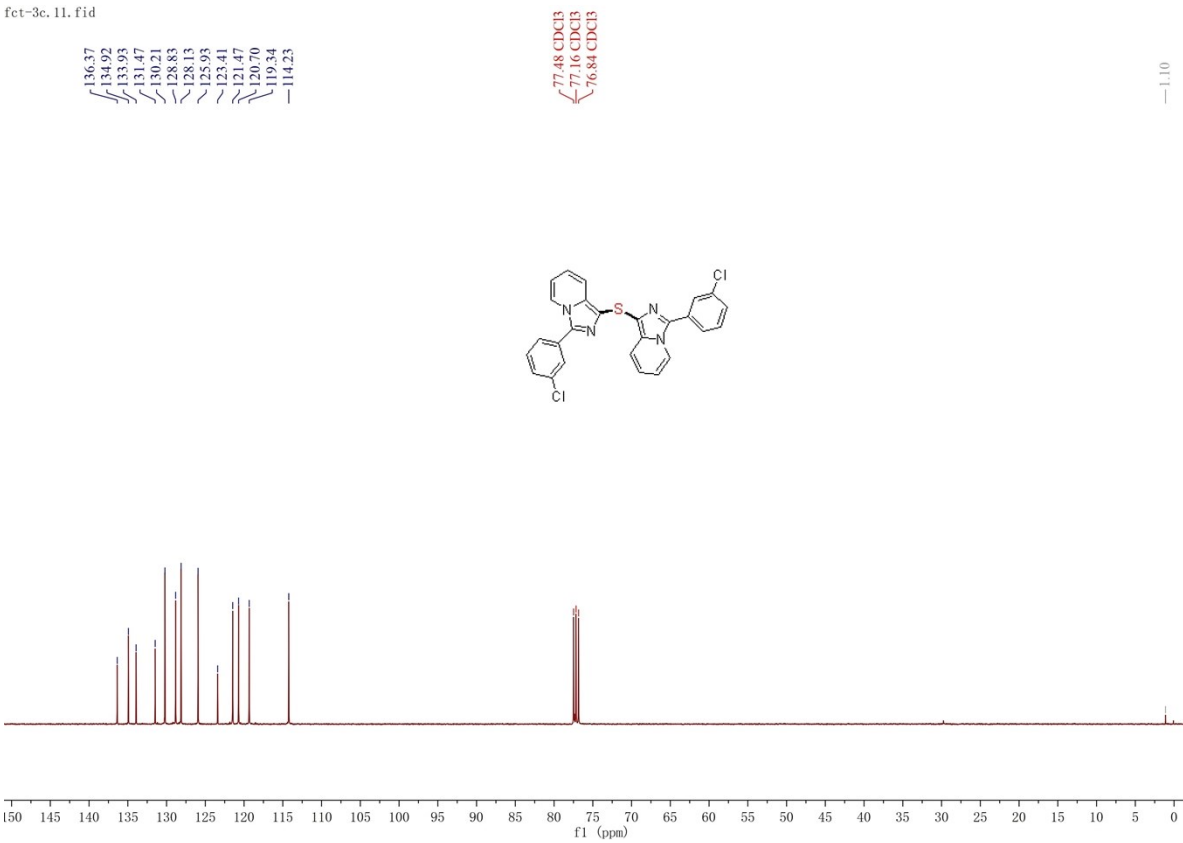
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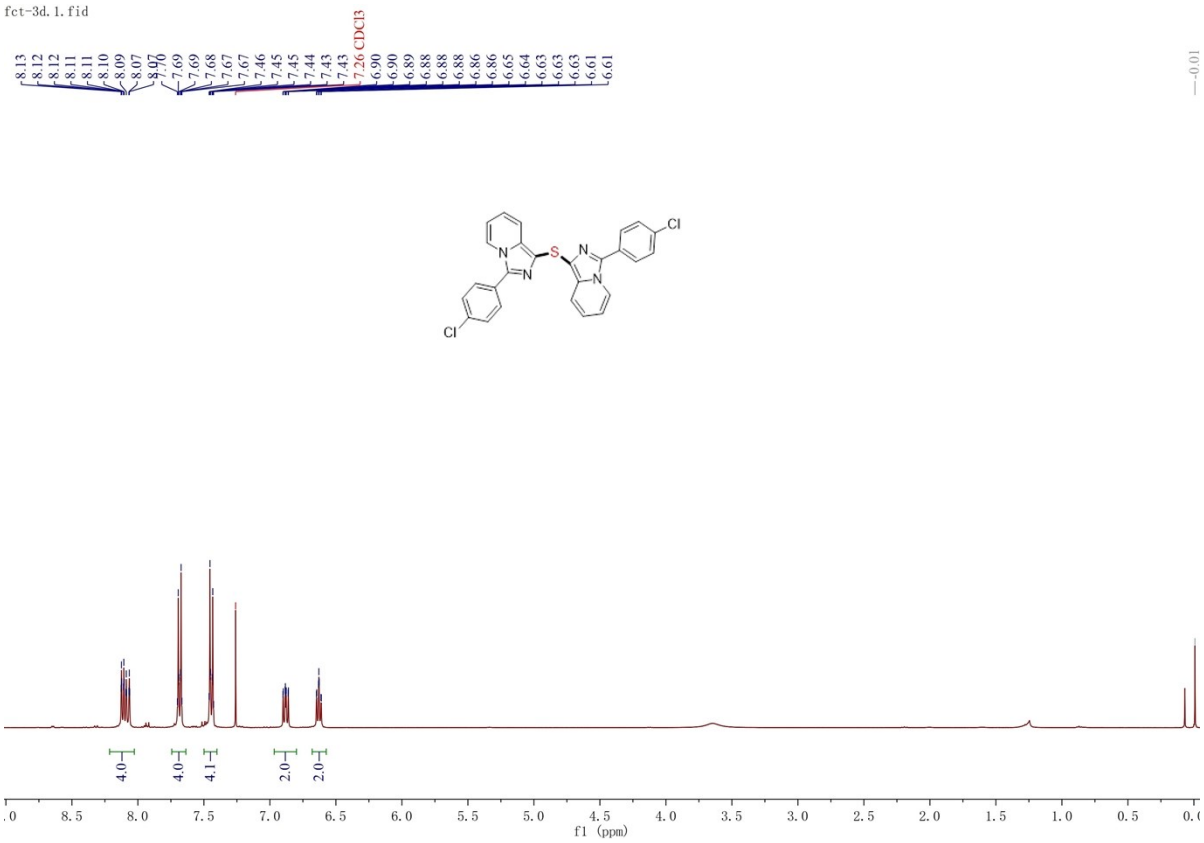
3d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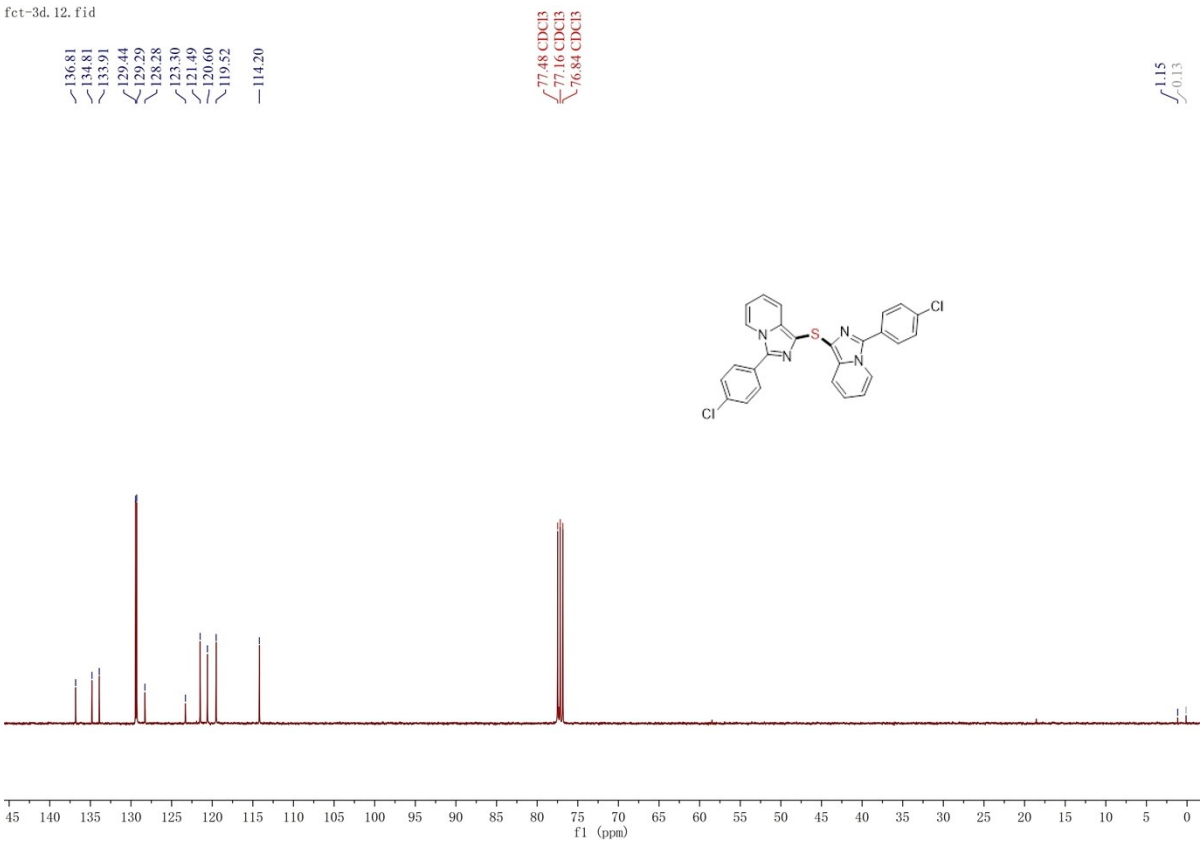
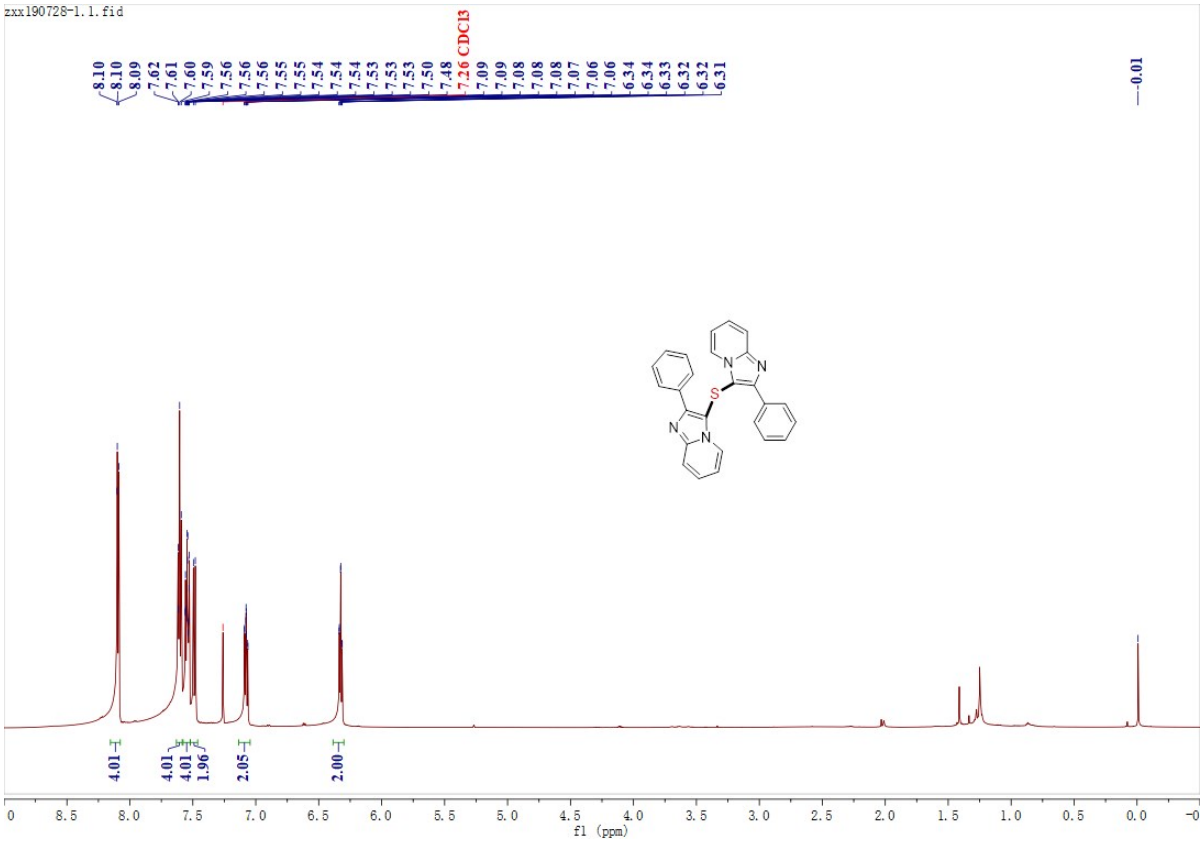
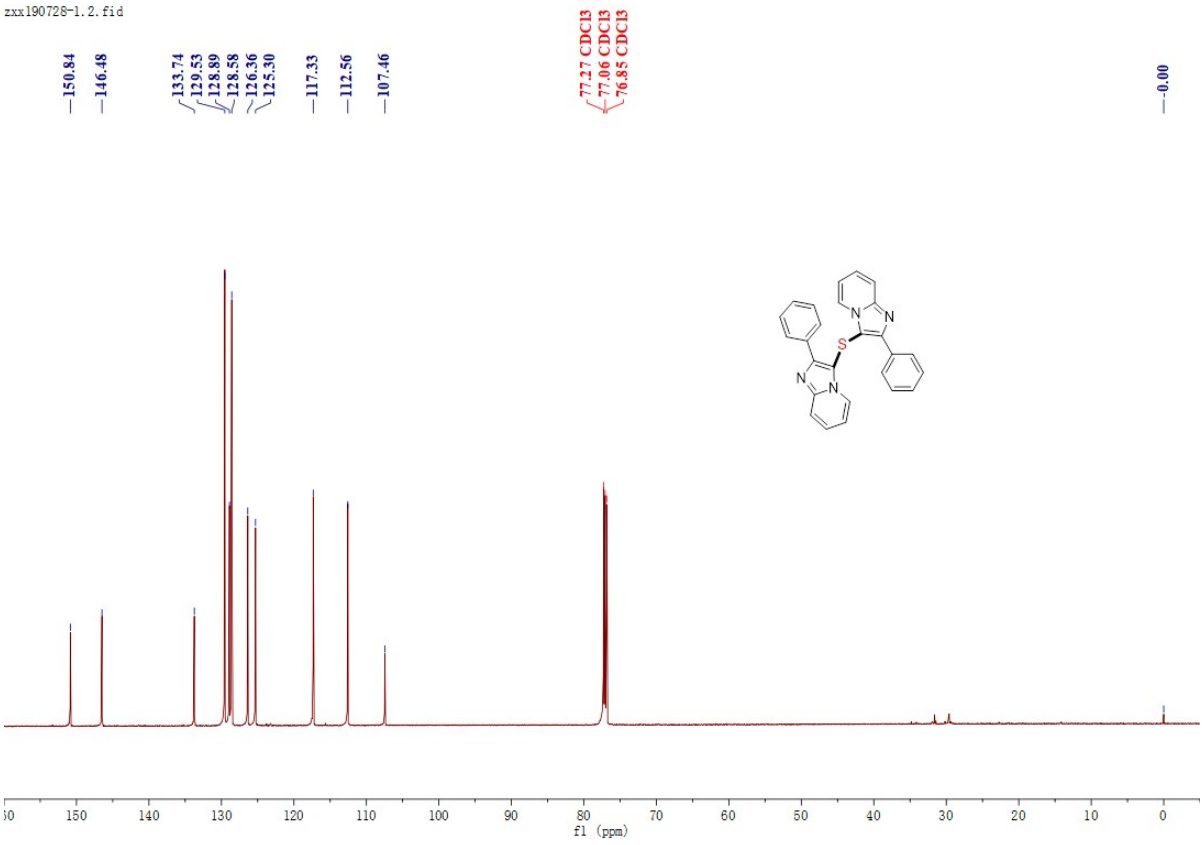
6.63
6.63
6.63
6.61
6.61
0.01
Cl
S
N
N
N
Cl
4.0
4.0
4.1
2.0
2.0
f1 (ppm)

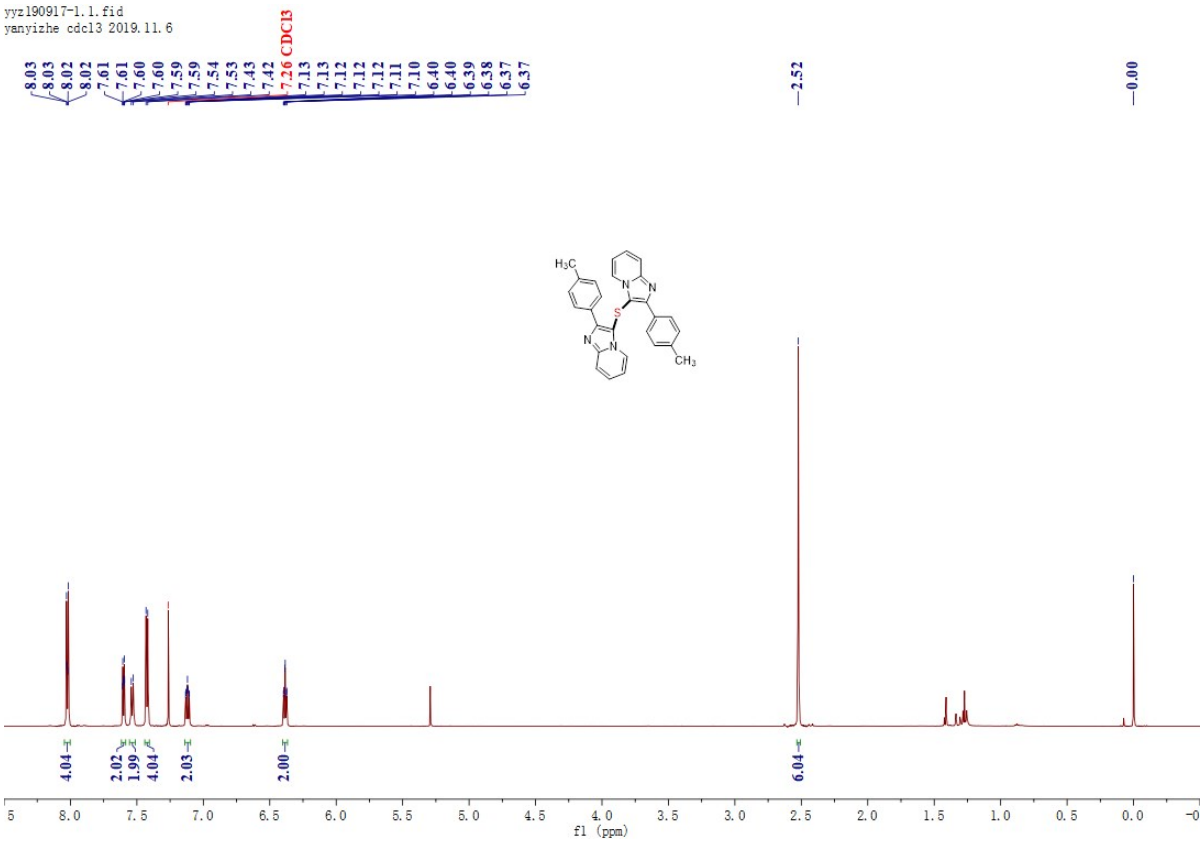

fct-3d.12.fid
136.81
134.81
133.91
129.44
129.29
128.28
123.30
121.49
120.60
119.52
114.20
77.48 CDCl3
77.16 CDCl3
76.84 CDCl3
1.15
0.13
Cl
S
N
N
N
Cl
f1 (ppm)

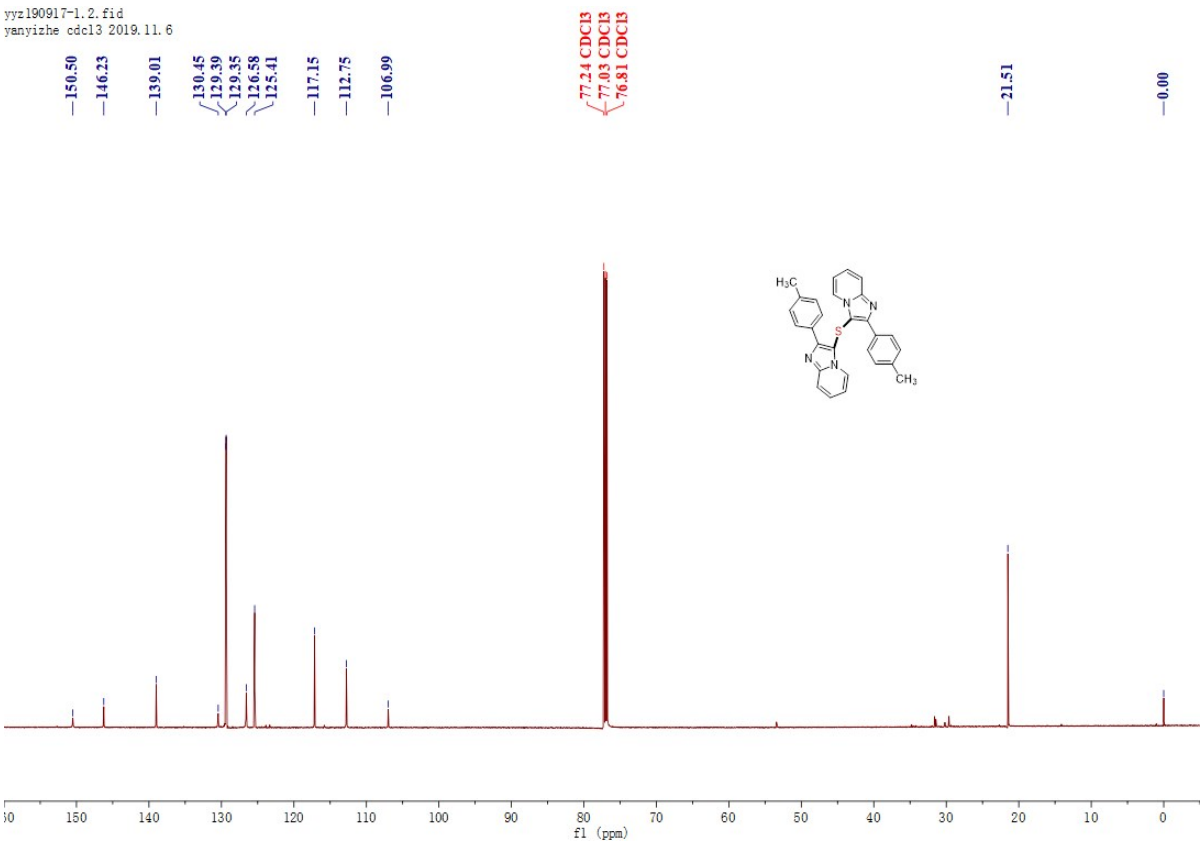
3e


zxx190728-1.1.fid
8.10
8.10
8.09
7.62
7.61
7.60
7.59
7.56
7.56
7.56
7.55
7.55
7.54
7.54
7.54
7.53
7.53
7.53
7.50
7.48
7.26 CDCl3
7.09
7.09
7.08
7.08
7.08
7.07
7.06
7.06
6.34
6.34
6.33
6.32
6.32
6.31
0.01
4.01
4.01
4.01
1.96
2.05
2.00
f1 (ppm)


zxx190728-1.2.fid
150.84
146.48
133.74
129.53
128.89
128.58
126.36
125.30
117.33
112.56
107.46
77.27 CDCl3
77.06 CDCl3
76.85 CDCl3
0.00
f1 (ppm)

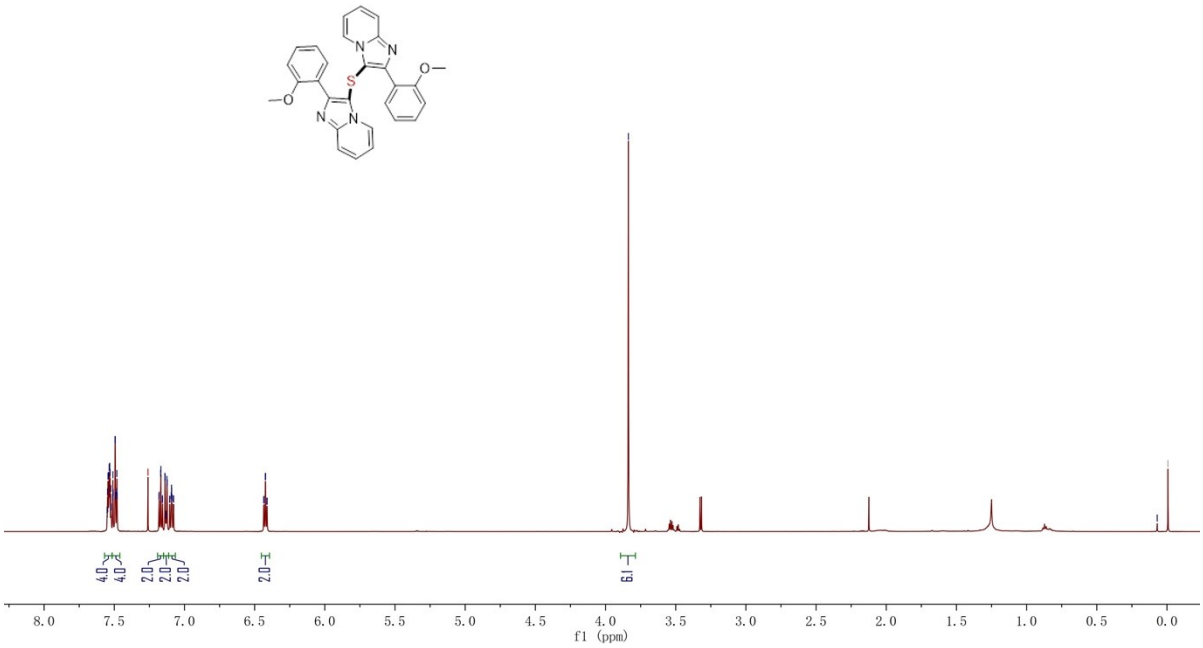
3f


yyz190917-1.1.fid
yanyizhe cdcl3 2019.11.6
8.03
8.03
8.02
8.02
7.61
7.61
7.60
7.60
7.59
7.59
7.54
7.53
7.43
7.42
7.26 CDCl3
7.13
7.13
7.12
7.12
7.12
7.11
7.10
6.40
6.40
6.39
6.38
6.37
6.37
2.52
0.00
4.04
2.02
1.99
4.04
2.03
2.00
6.04
f1 (ppm)

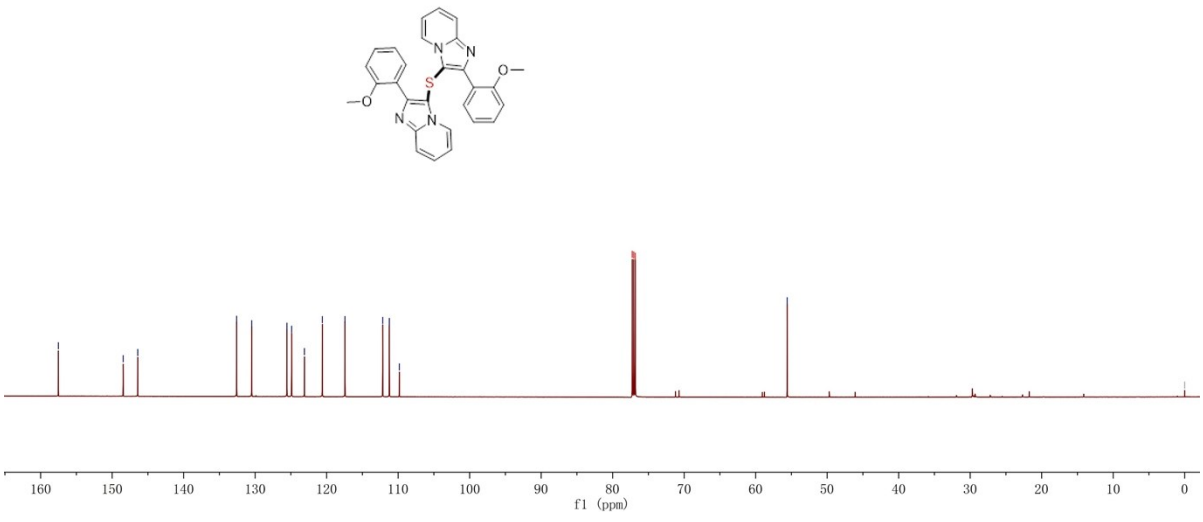

yyz190917-1.2.fid
yanyizhe cdcl3 2019.11.6
150.50
146.23
139.01
130.45
129.39
129.35
126.58
125.41
117.15
112.75
106.99
77.24 CDCl3
77.03 CDCl3
76.81 CDCl3
21.51
0.00
f1 (ppm)

3g

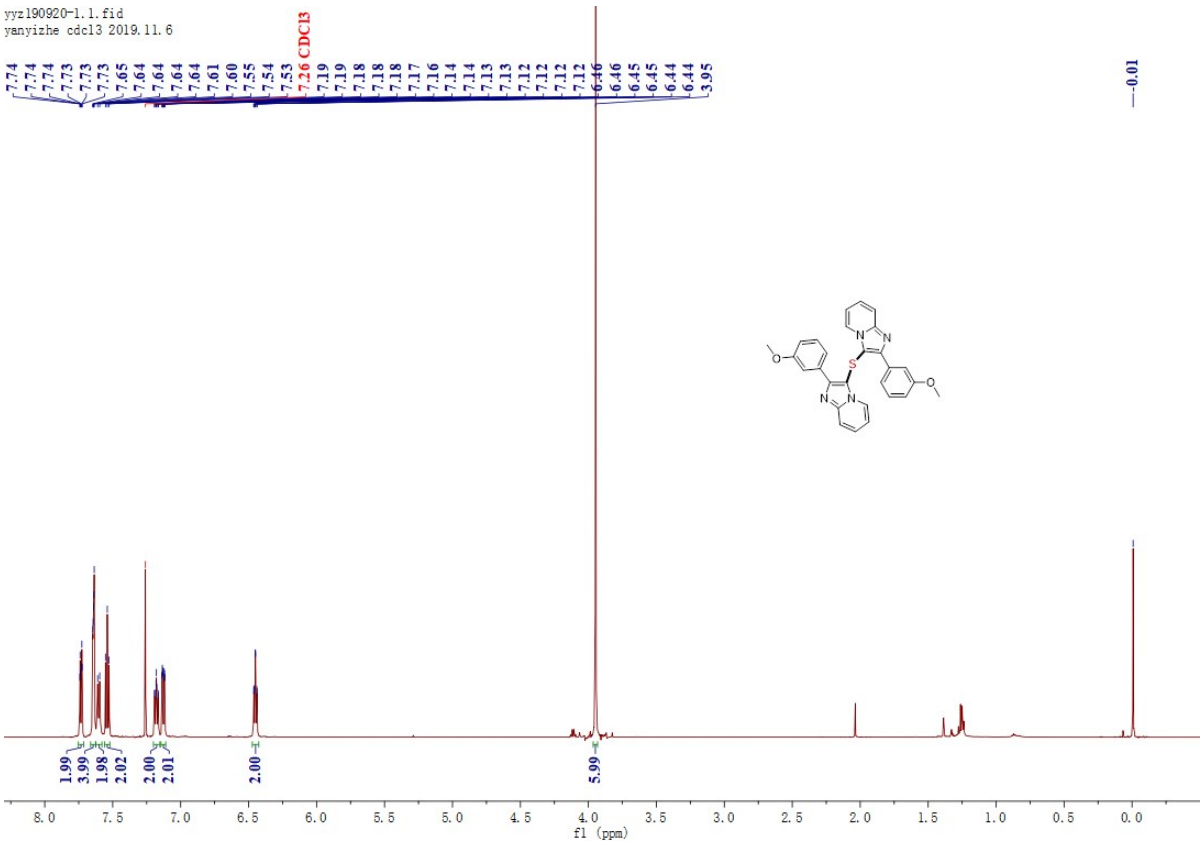
yyz191011-1.1.fid
yanyizhe 2019.11.26

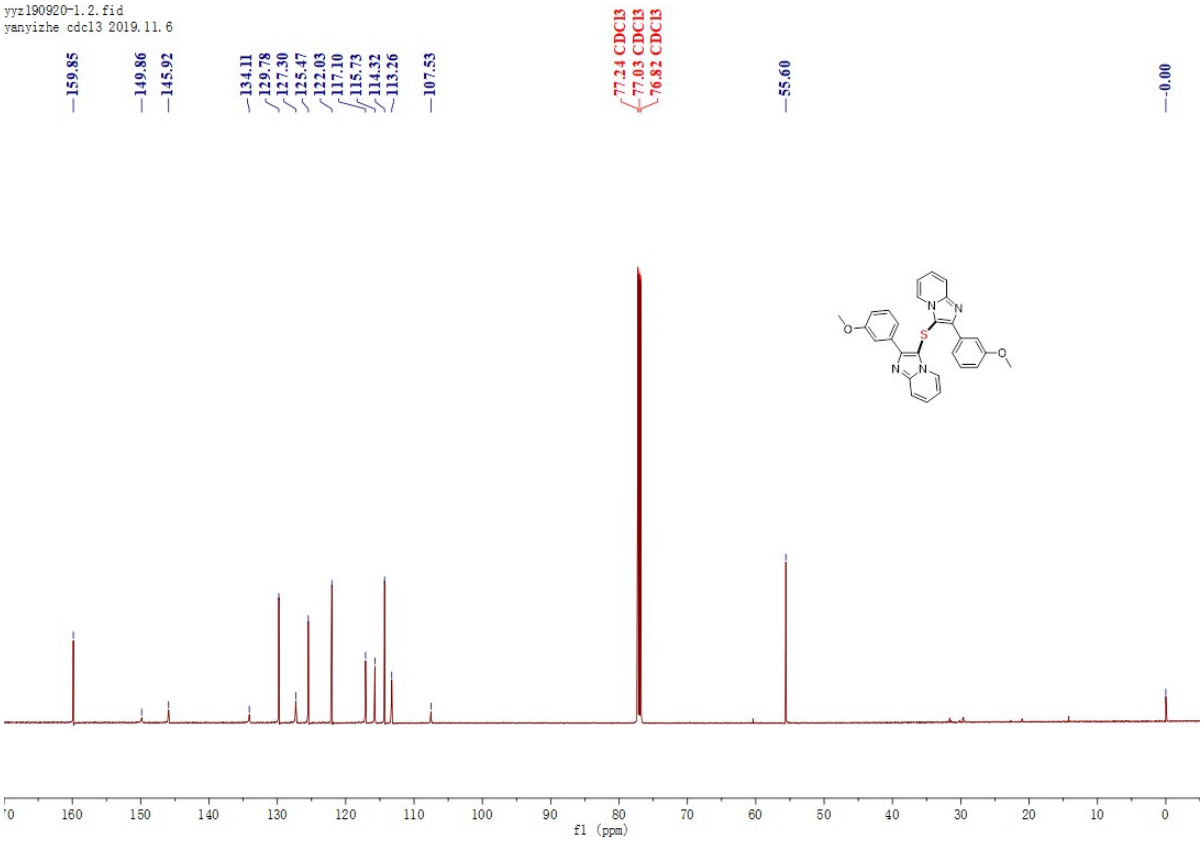


yyz191011-1.2.fid
yanyizhe 2019.11.26

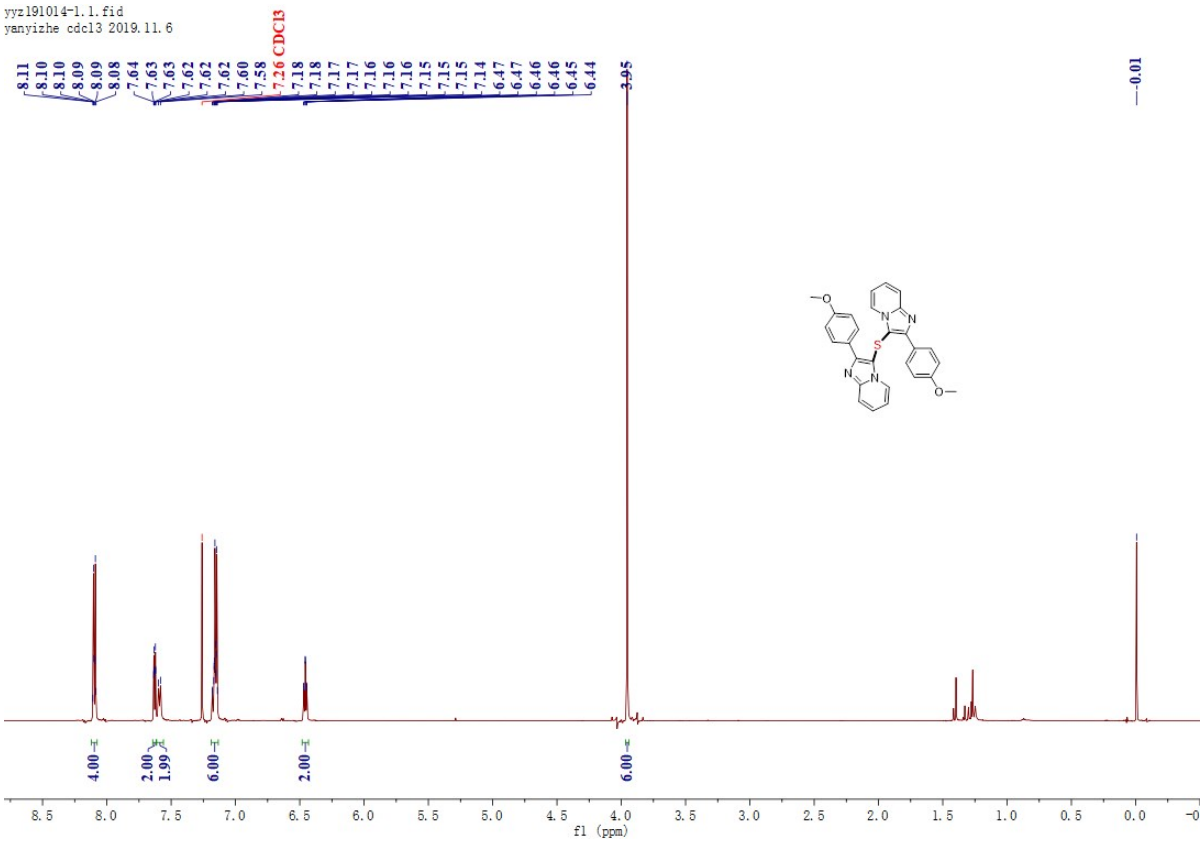


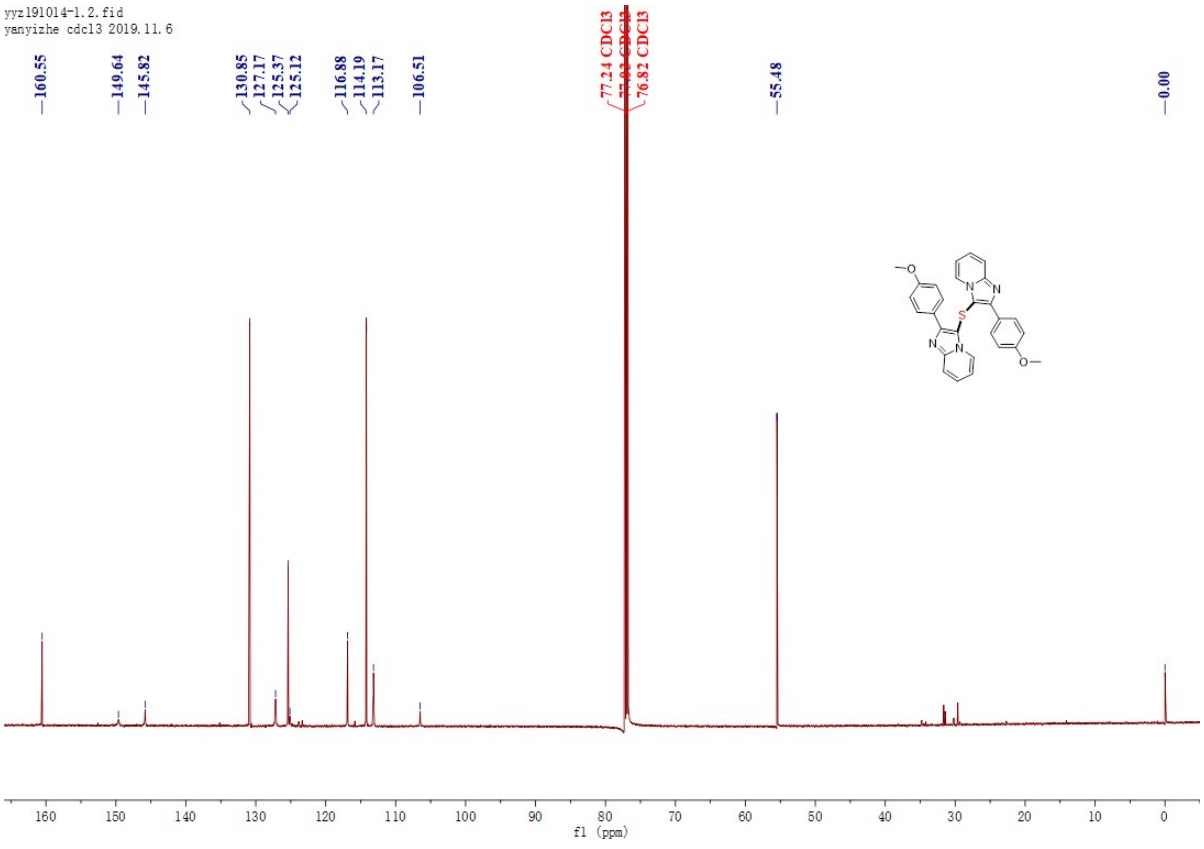
3h


yyz190920-1.1.fid
yanyizhe cdcl3 2019.11.6
7.74
7.74
7.74
7.73
7.73
7.73
7.65
7.64
7.64
7.64
7.61
7.60
7.55
7.54
7.53
7.26 CDCl3
7.19
7.18
7.18
7.18
7.17
7.16
7.14
7.14
7.13
7.13
7.12
7.12
7.12
7.12
6.46
6.46
6.45
6.45
6.44
6.44
3.95
0.01
1.99
3.99
1.98
2.02
2.00
2.01
2.00
5.99
f1 (ppm)

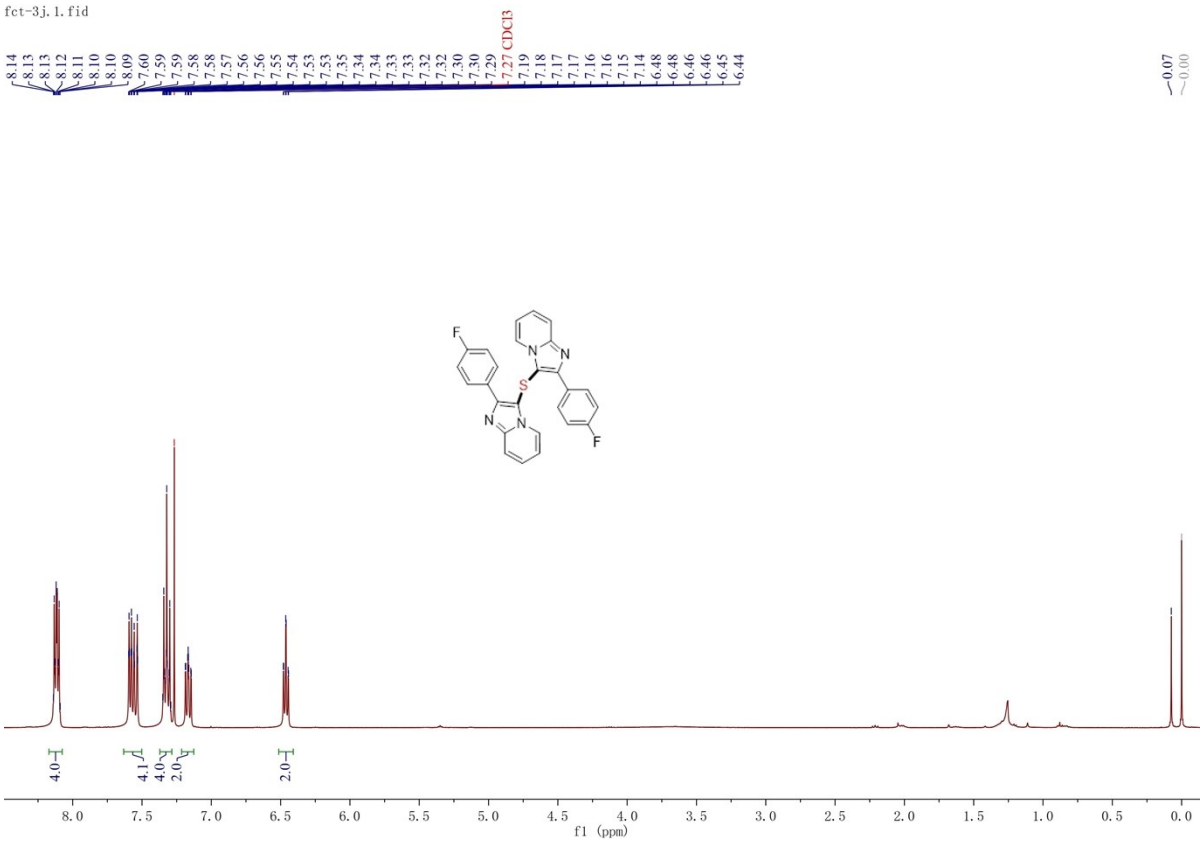

yyz190920-1.2.fid
yanyizhe cdcl3 2019.11.6
159.85
149.86
145.92
134.11
129.78
127.30
125.47
122.03
117.10
115.73
114.32
113.26
107.53
77.24 CDCl3
77.03 CDCl3
76.82 CDCl3
55.60
0.00
f1 (ppm)

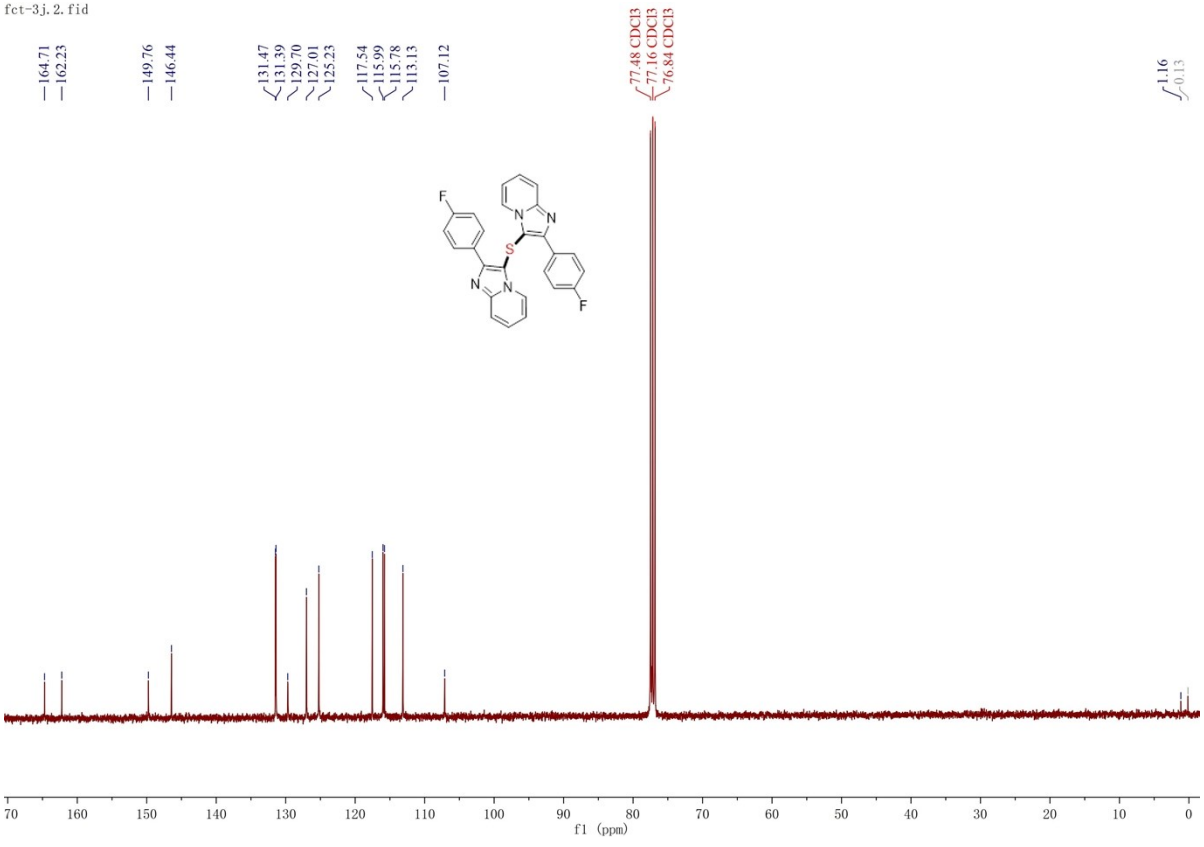
3i


yyz191014-1.1.fid
yanyizhe cdcl3 2019.11.6
8.11
8.10
8.10
8.09
8.09
8.08
7.64
7.63
7.63
7.62
7.62
7.62
7.60
7.58
7.26 CDCl3
7.18
7.18
7.17
7.17
7.16
7.16
7.16
7.15
7.15
7.15
7.14
6.47
6.47
6.46
6.46
6.45
6.44
3.95
-0.01
4.00
2.00
1.99
6.00
2.00
6.00
f1 (ppm)

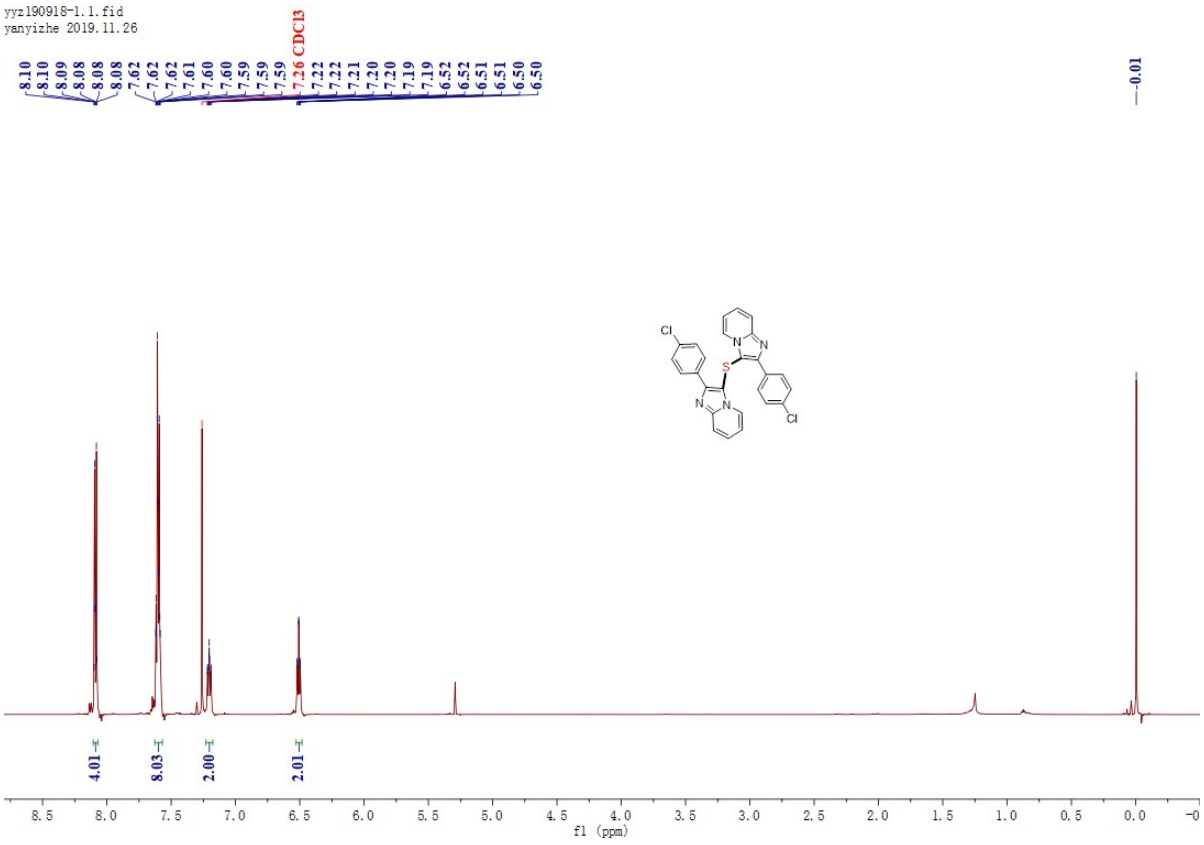

yyz191014-1.2.fid
yanyizhe cdcl3 2019.11.6
160.55
149.64
145.82
130.85
127.17
125.37
125.12
116.88
114.19
113.17
106.51
77.24 CDCl3
77.03 CDCl3
76.82 CDCl3
55.48
0.00
f1 (ppm)

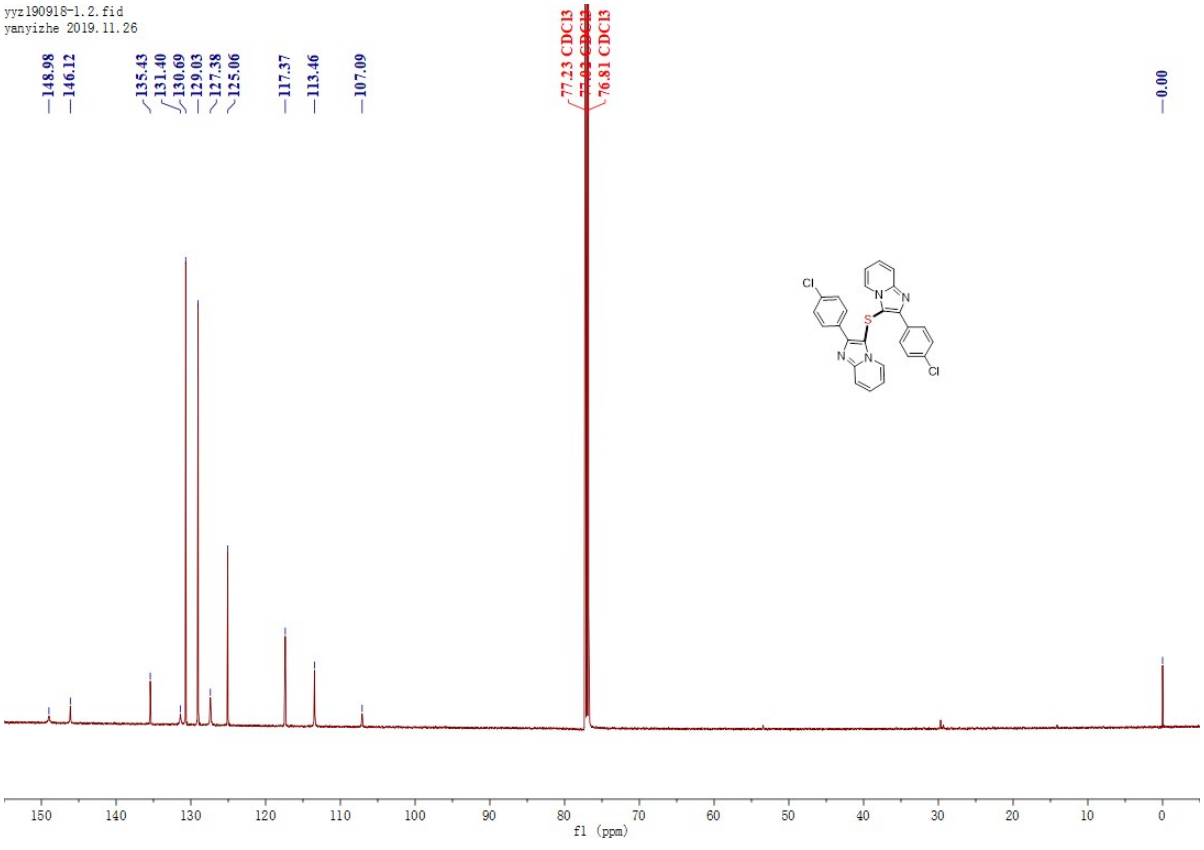
3j


fct-3j.1.fid
8.14
8.13
8.13
8.12
8.11
8.10
8.10
8.09
7.60
7.59
7.59
7.58
7.58
7.57
7.56
7.56
7.55
7.54
7.53
7.53
7.35
7.34
7.34
7.33
7.33
7.32
7.32
7.30
7.30
7.29
7.27 CDCl3
7.19
7.18
7.17
7.17
7.16
7.16
7.15
7.14
6.48
6.48
6.46
6.46
6.45
6.44
0.07
0.00
F
N
N
S
N
N
F
4.0
4.1
4.0
2.0
2.0
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

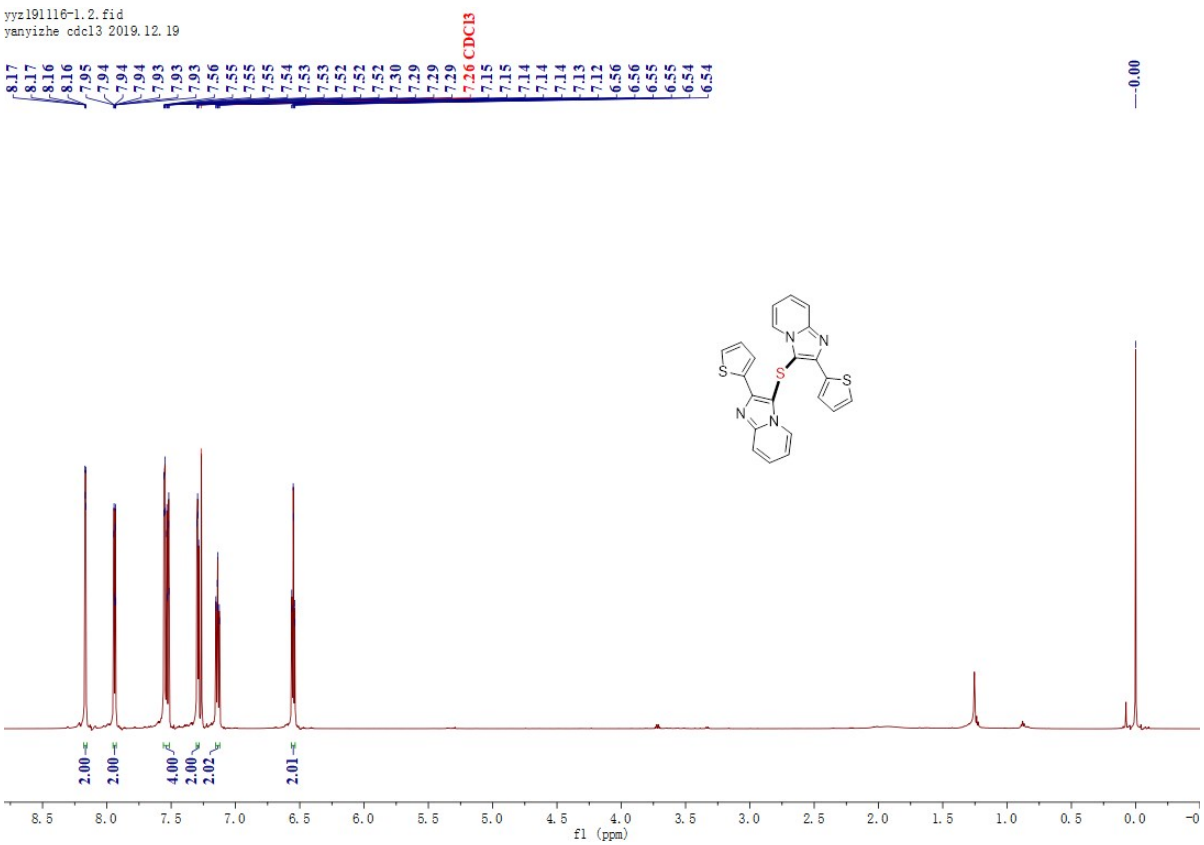

fct-3j.2.fid
164.71
162.23
149.76
146.44
131.47
131.39
129.70
127.01
125.23
117.54
115.99
115.78
113.13
107.12
77.48 CDCl3
77.16 CDCl3
76.84 CDCl3
1.16
0.13
F
N
N
S
N
N
F
70
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

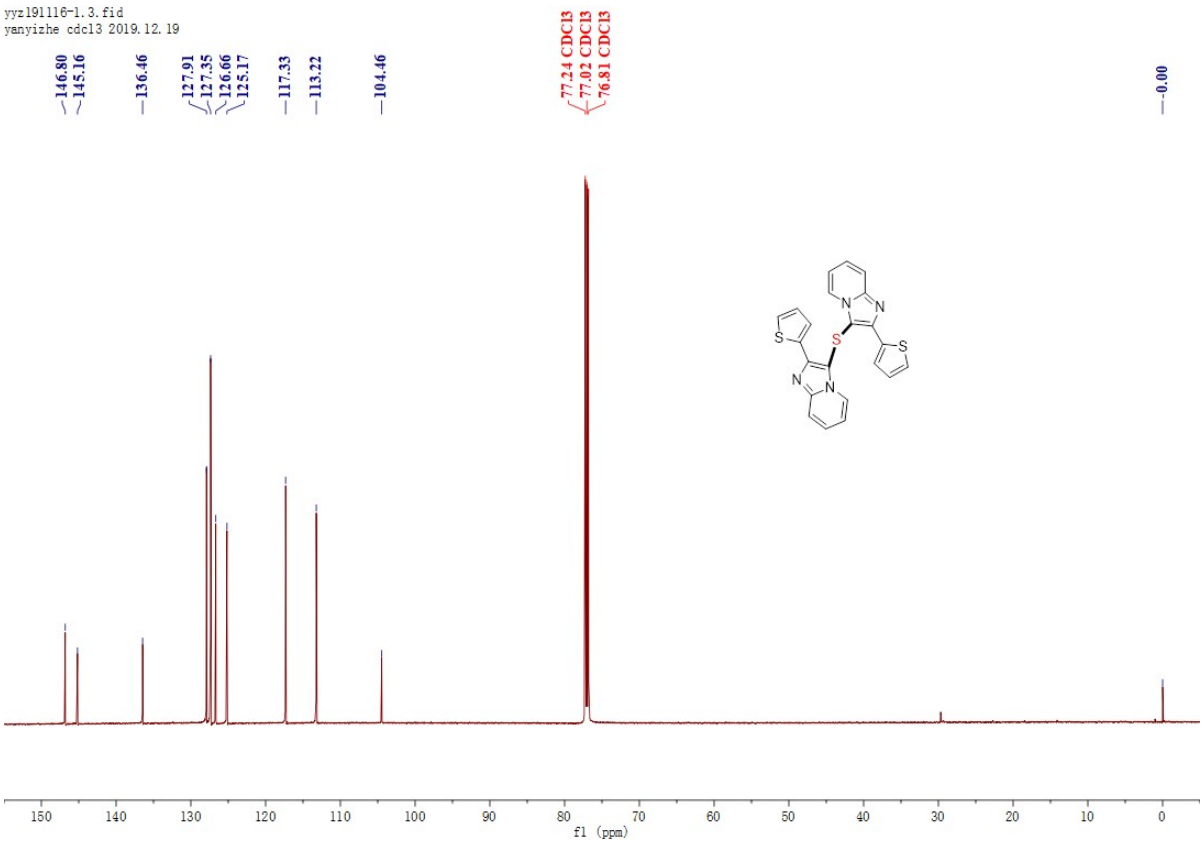
3k


yyz190918-1.1.fid
yanyizhe 2019.11.26
8.10
8.10
8.09
8.08
8.08
8.08
7.62
7.62
7.62
7.61
7.60
7.60
7.59
7.59
7.59
7.26 CDCl3
7.22
7.22
7.21
7.20
7.20
7.19
7.19
6.52
6.52
6.51
6.51
6.50
6.50
-0.01
Cl
N
N
S
N
N
Cl
4.01
8.03
2.00
2.01
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0
f1 (ppm)

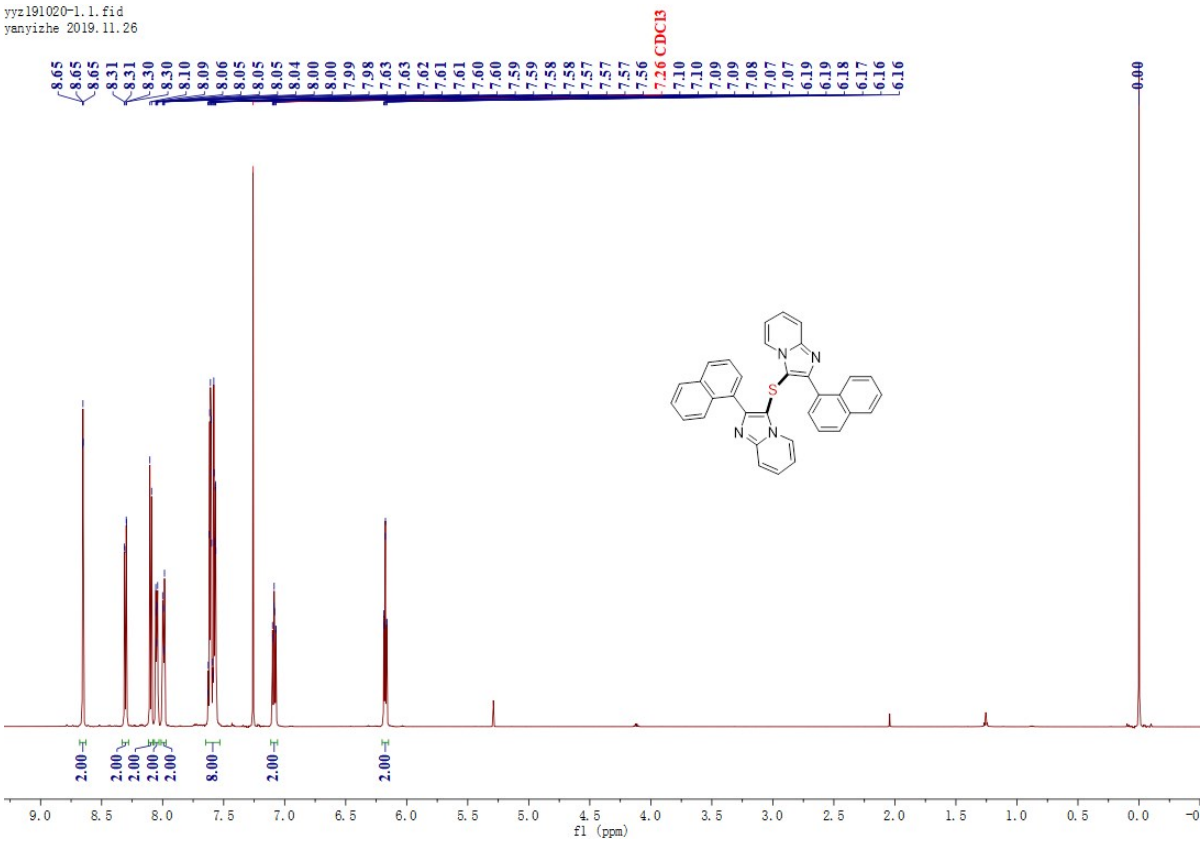

yyz190918-1.2.fid
yanyizhe 2019.11.26
148.98
146.12
135.43
131.40
130.69
129.03
127.38
125.06
117.37
113.46
107.09
77.23 CDCl3
77.03 CDCl3
76.81 CDCl3
-0.00
Cl
N
N
S
N
N
Cl
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

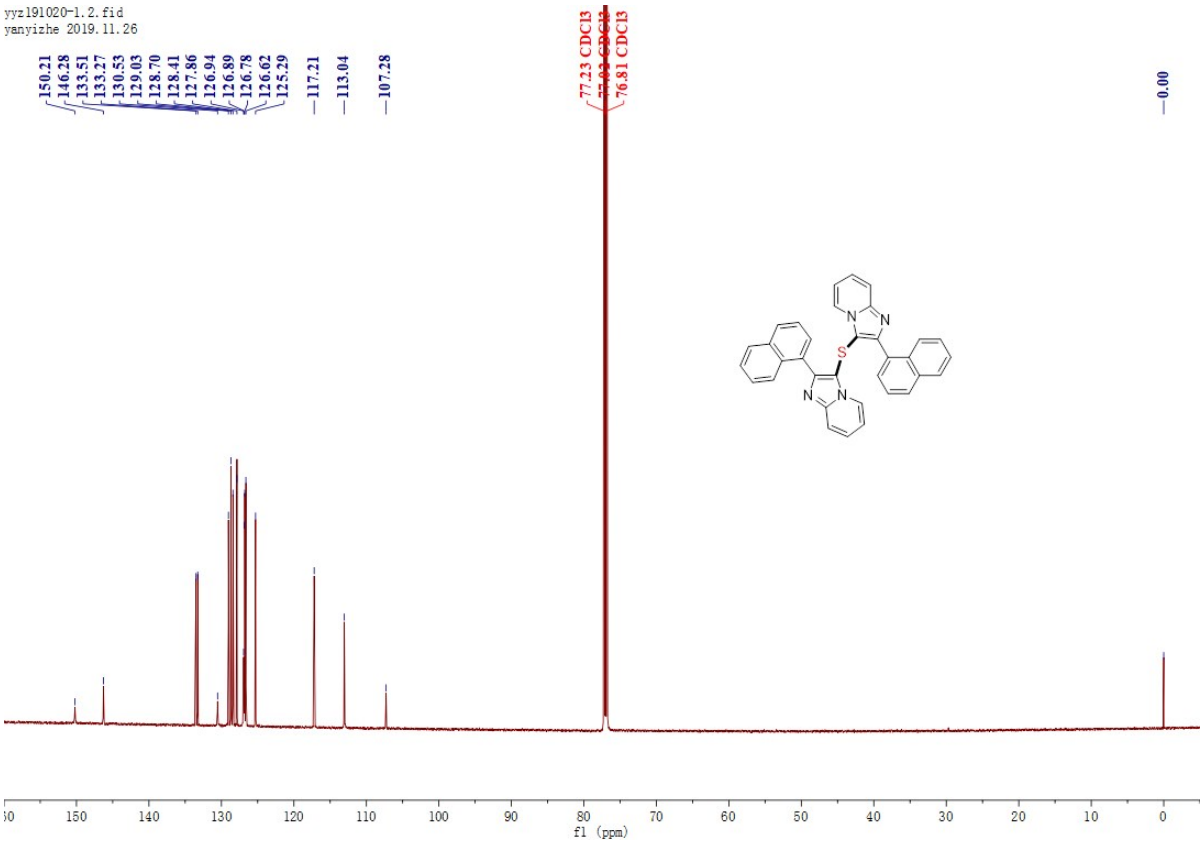
3l


yyz191116-1.2.fid
yanyizhe cdcl3 2019.12.19
7.26 CDCl3
2.00
2.00
4.00
2.00
2.02
2.01
f1 (ppm)

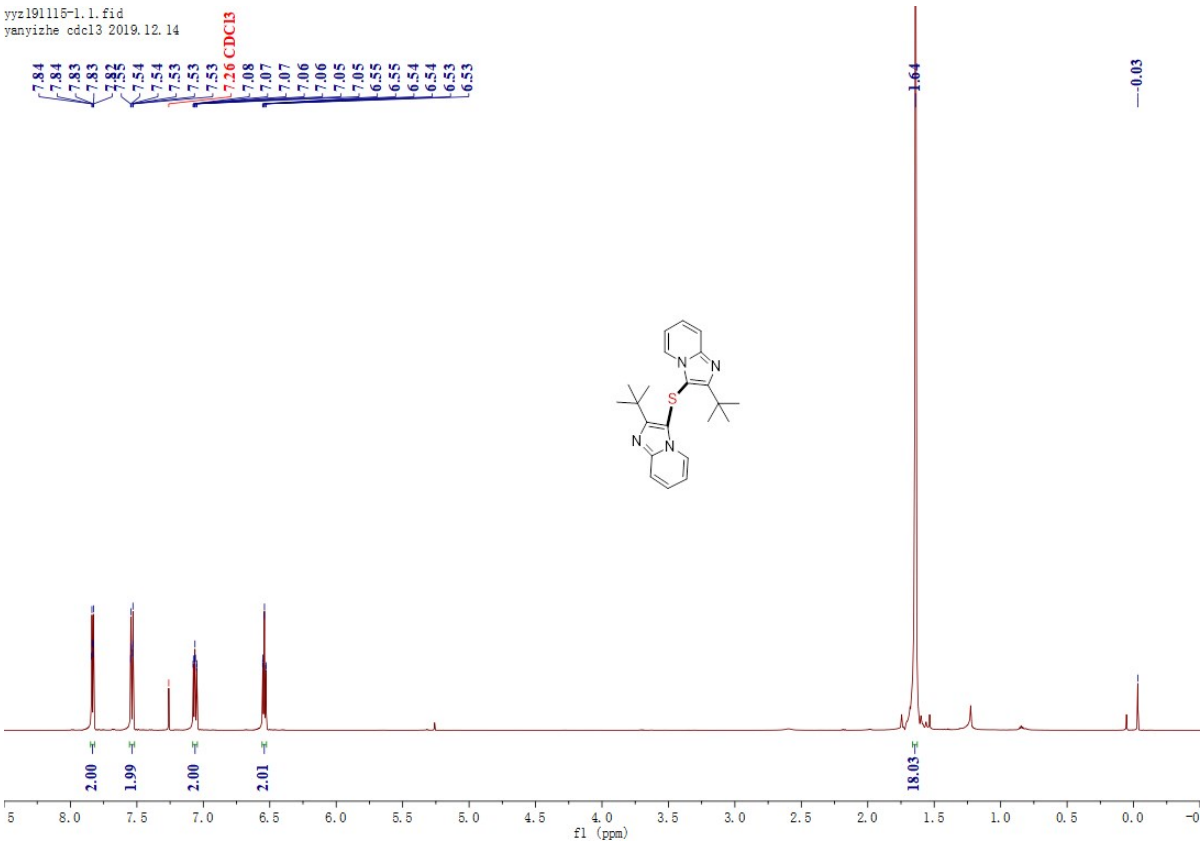

yyz191116-1.3.fid
yanyizhe cdcl3 2019.12.19
146.80
145.16
136.46
127.91
127.35
126.66
125.17
117.33
113.22
104.46
77.24 CDCl3
77.02 CDCl3
76.81 CDCl3
f1 (ppm)

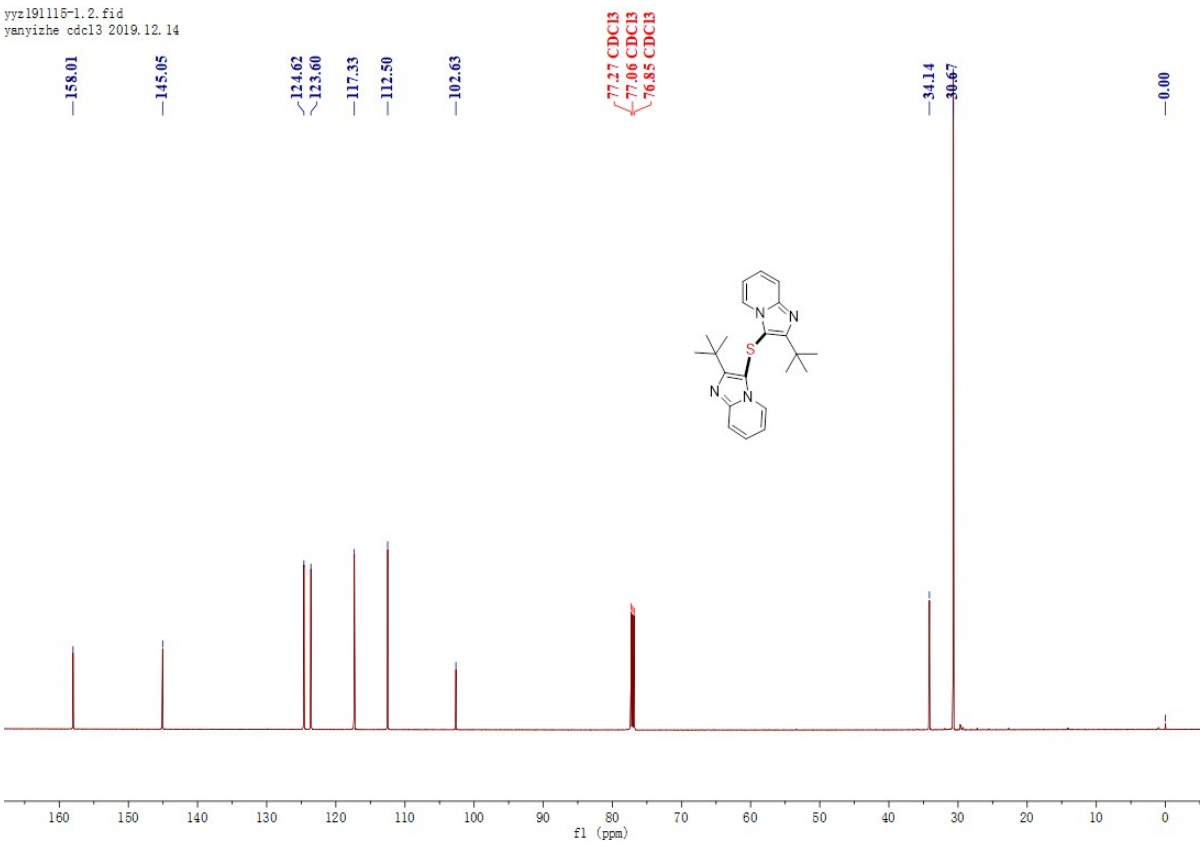
3m


yyz191020-1.1.fid
yanyizhe 2019.11.26
7.26 CDCl3
f1 (ppm)

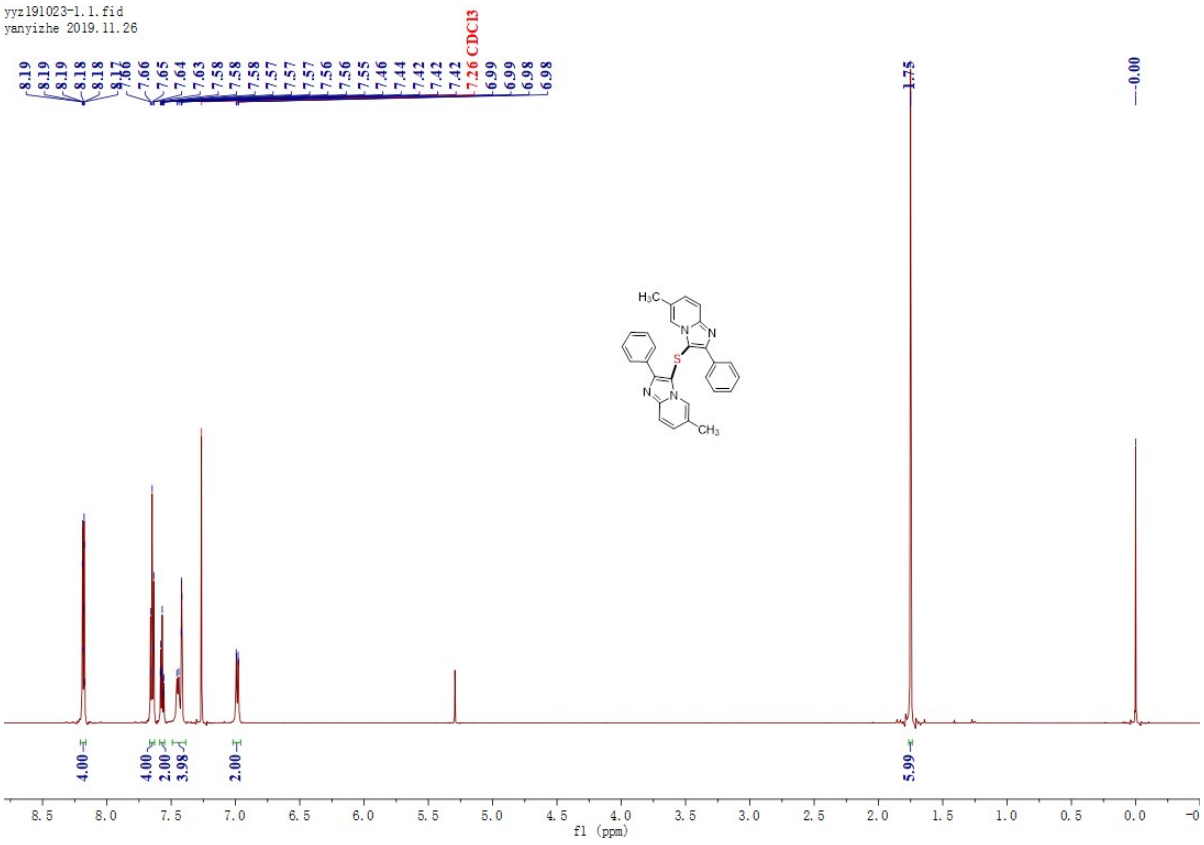

yyz191020-1.2.fid
yanyizhe 2019.11.26
77.23 CDCl3
76.81 CDCl3
f1 (ppm)

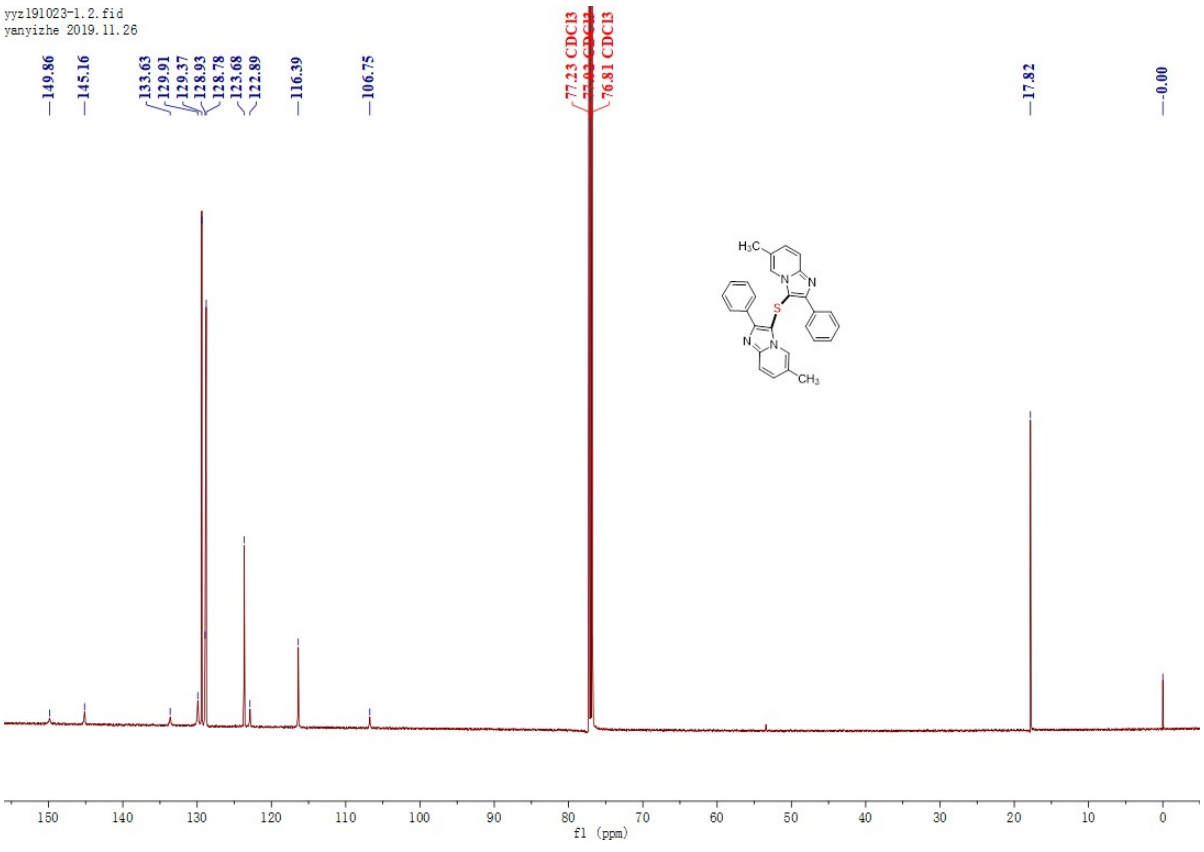
3n


yyz191115-1.1.fid
yanyizhe cdcl3 2019.12.14
7.84
7.84
7.83
7.83
7.82
7.55
7.54
7.54
7.53
7.53
7.53
7.26 CDCl3
7.08
7.07
7.07
7.06
7.06
7.05
7.05
6.55
6.54
6.54
6.53
6.53
1.64
0.03
2.00
1.99
2.00
2.01
18.03
f1 (ppm)

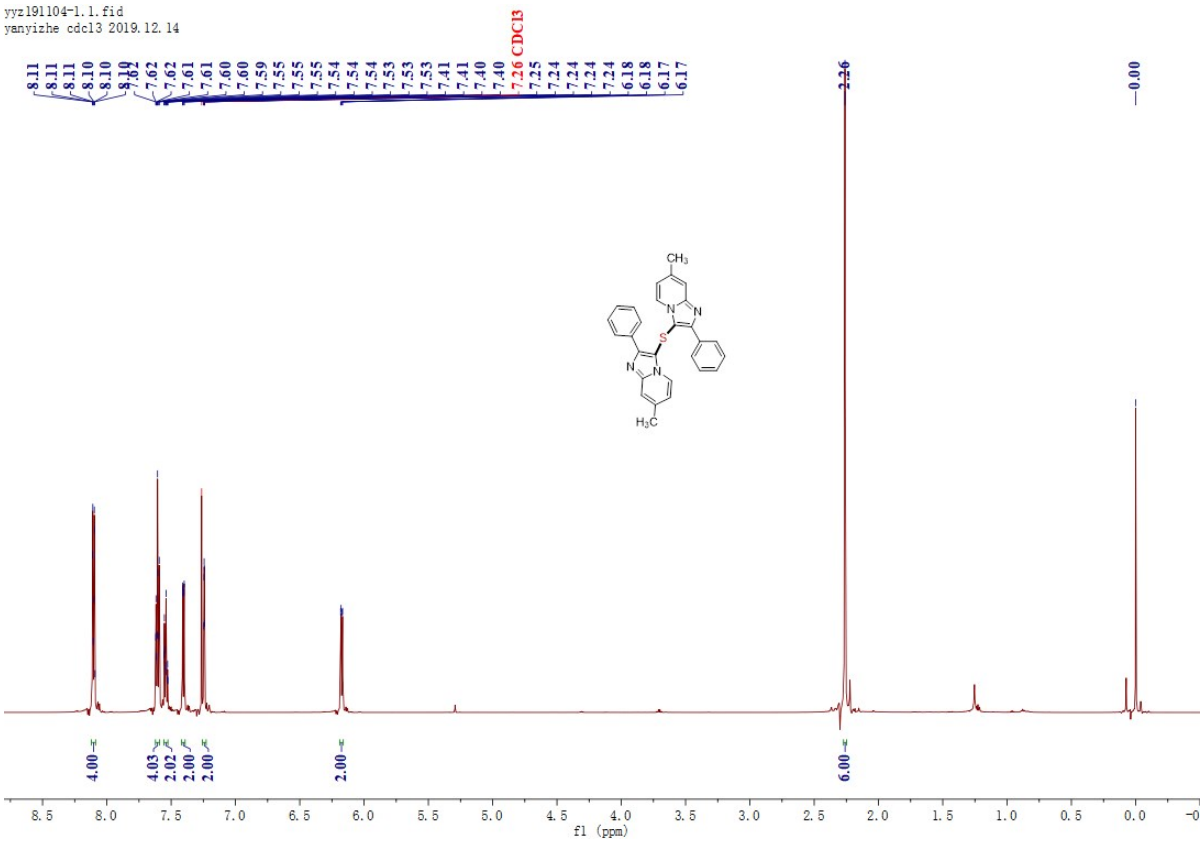

yyz191115-1.2.fid
yanyizhe cdcl3 2019.12.14
158.01
145.05
124.62
123.60
117.33
112.50
102.63
77.27 CDCl3
77.06 CDCl3
76.85 CDCl3
34.14
30.67
0.00
f1 (ppm)

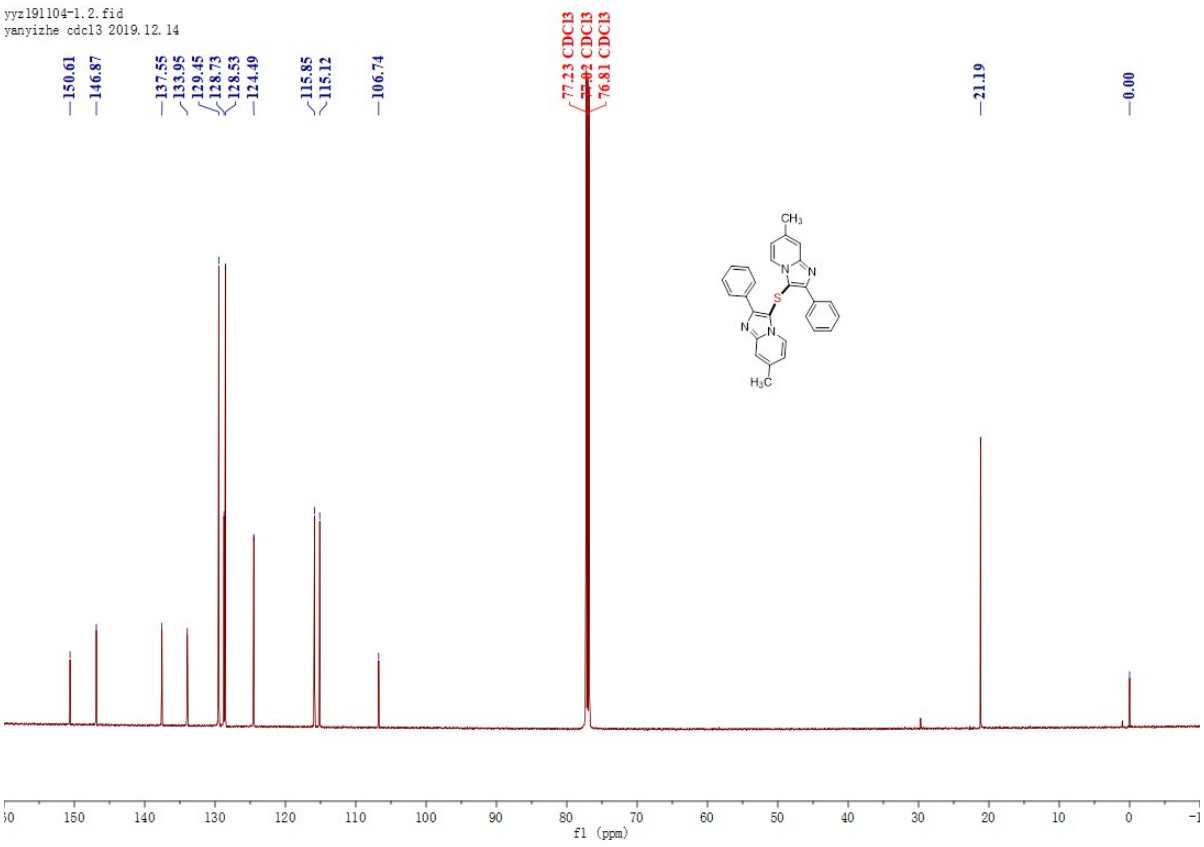
3o


yyz191023-1.1.fid
yanyizhe 2019.11.26
8.19
8.19
8.18
8.18
7.66
7.65
7.64
7.63
7.58
7.58
7.57
7.57
7.56
7.55
7.46
7.44
7.42
7.42
7.26 CDCl3
6.99
6.99
6.98
6.98
1.75
0.00
4.00
4.00
2.00
3.98
2.00
5.99
f1 (ppm)

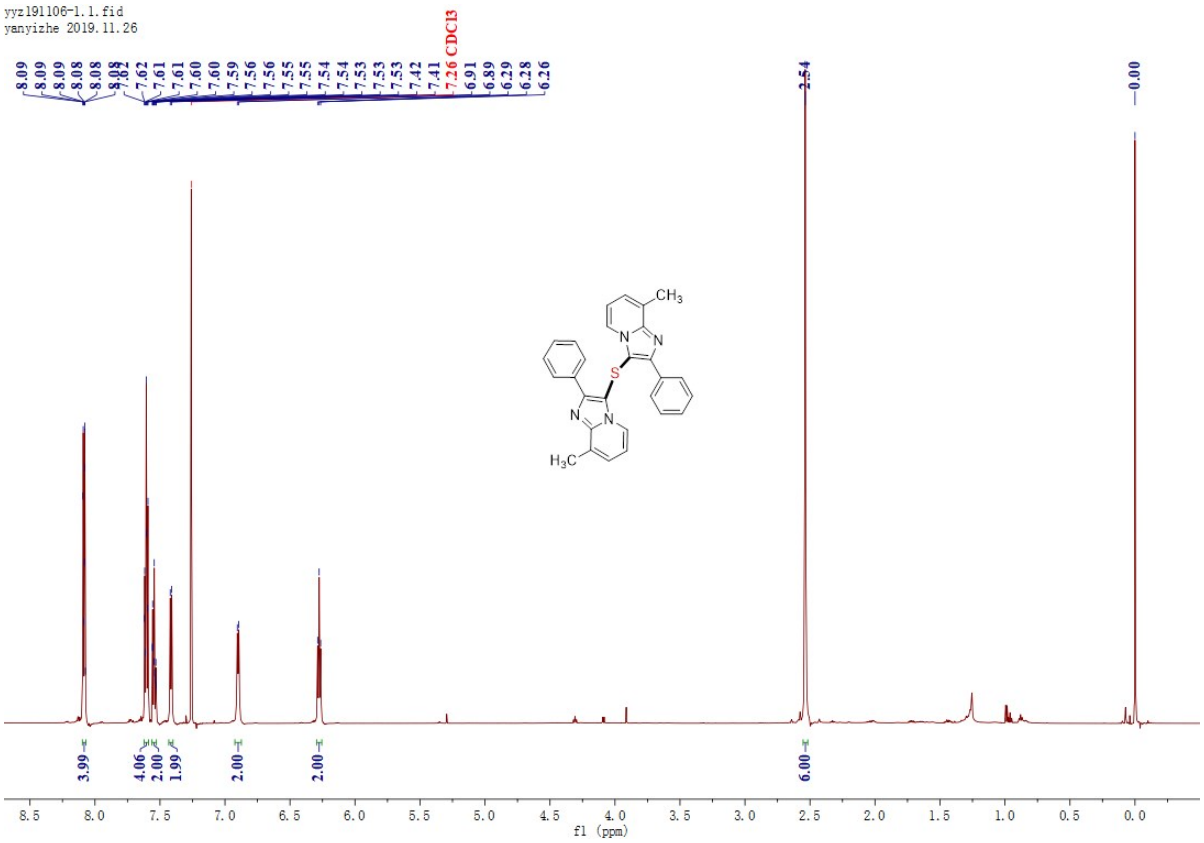

yyz191023-1.2.fid
yanyizhe 2019.11.26
149.86
145.16
133.63
129.91
129.37
128.93
128.78
123.68
122.89
116.39
106.75
77.23 CDCl3
76.81 CDCl3
17.82
0.00
f1 (ppm)

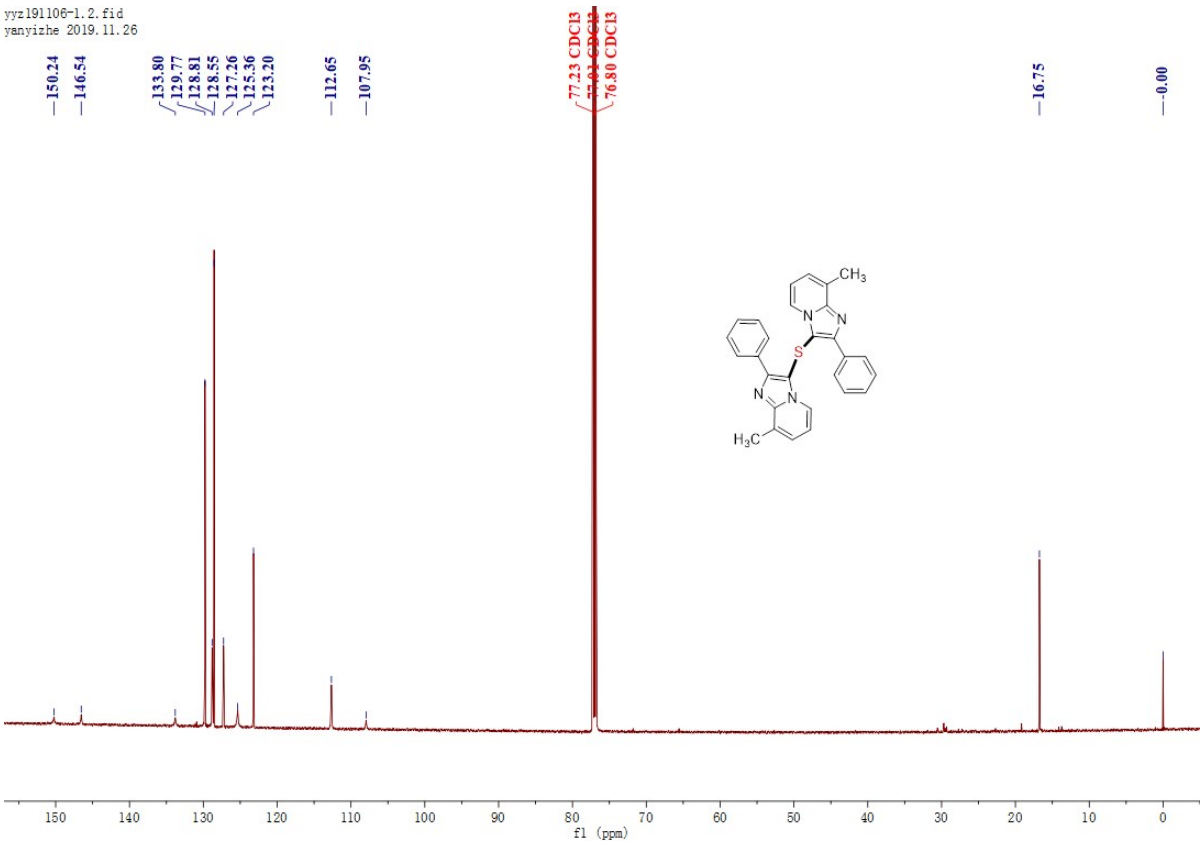
3p


yyz191104-1.1.fid
yanyizhe cdcl3 2019.12.14
8.11
8.11
8.11
8.10
8.10
8.10
7.62
7.62
7.61
7.61
7.60
7.60
7.59
7.55
7.55
7.55
7.54
7.54
7.54
7.53
7.53
7.53
7.41
7.41
7.40
7.40
7.26 CDCl3
7.25
7.24
7.24
7.24
7.24
6.18
6.18
6.17
6.17
2.26
0.00
4.00
4.03
2.02
2.00
2.00
2.00
6.00
f1 (ppm)


yyz191104-1.2.fid
yanyizhe cdcl3 2019.12.14
150.61
146.87
137.55
133.95
129.45
128.73
128.53
124.49
115.85
115.12
106.74
77.23 CDCl3
77.02 CDCl3
76.81 CDCl3
21.19
0.00
f1 (ppm)

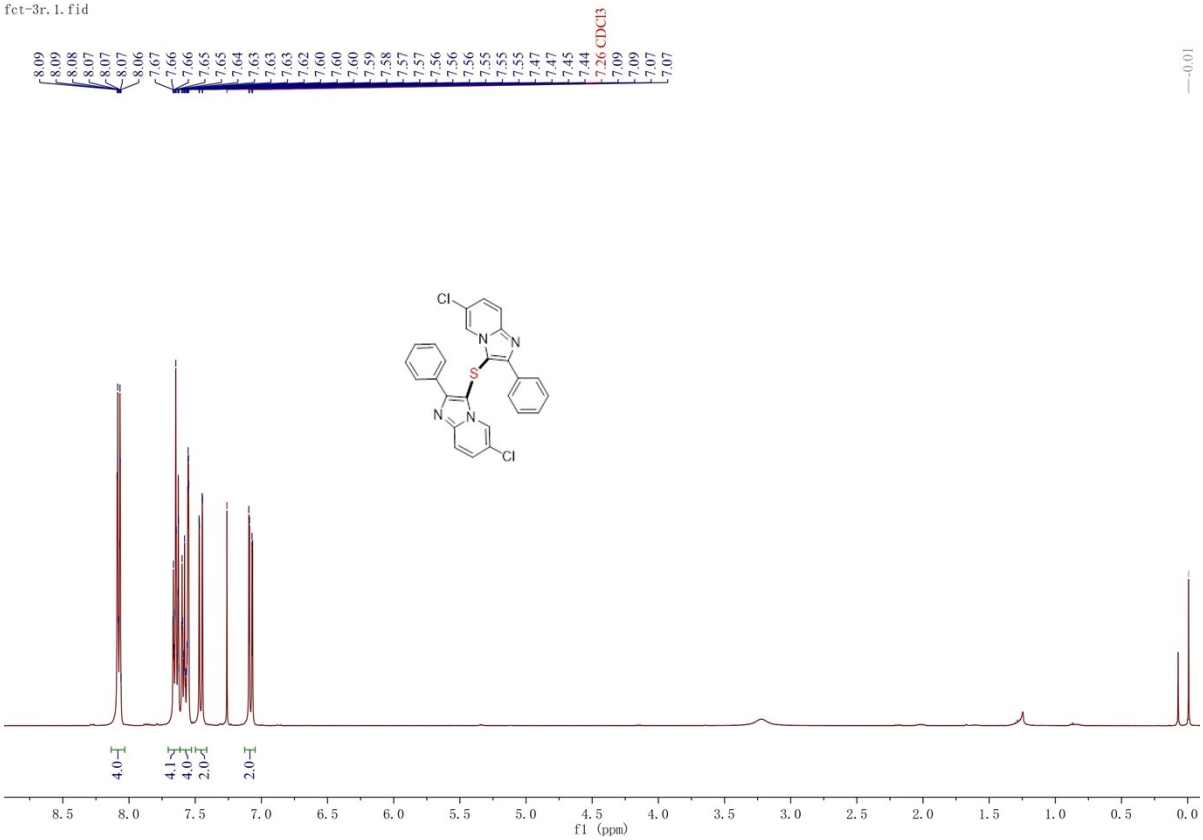
3q


yyz191106-1.1.fid
yanyizhe 2019.11.26
7.26 CDCl3
2.54
0.00
3.99
4.06
2.00
1.99
2.00
2.00
6.00
f1 (ppm)

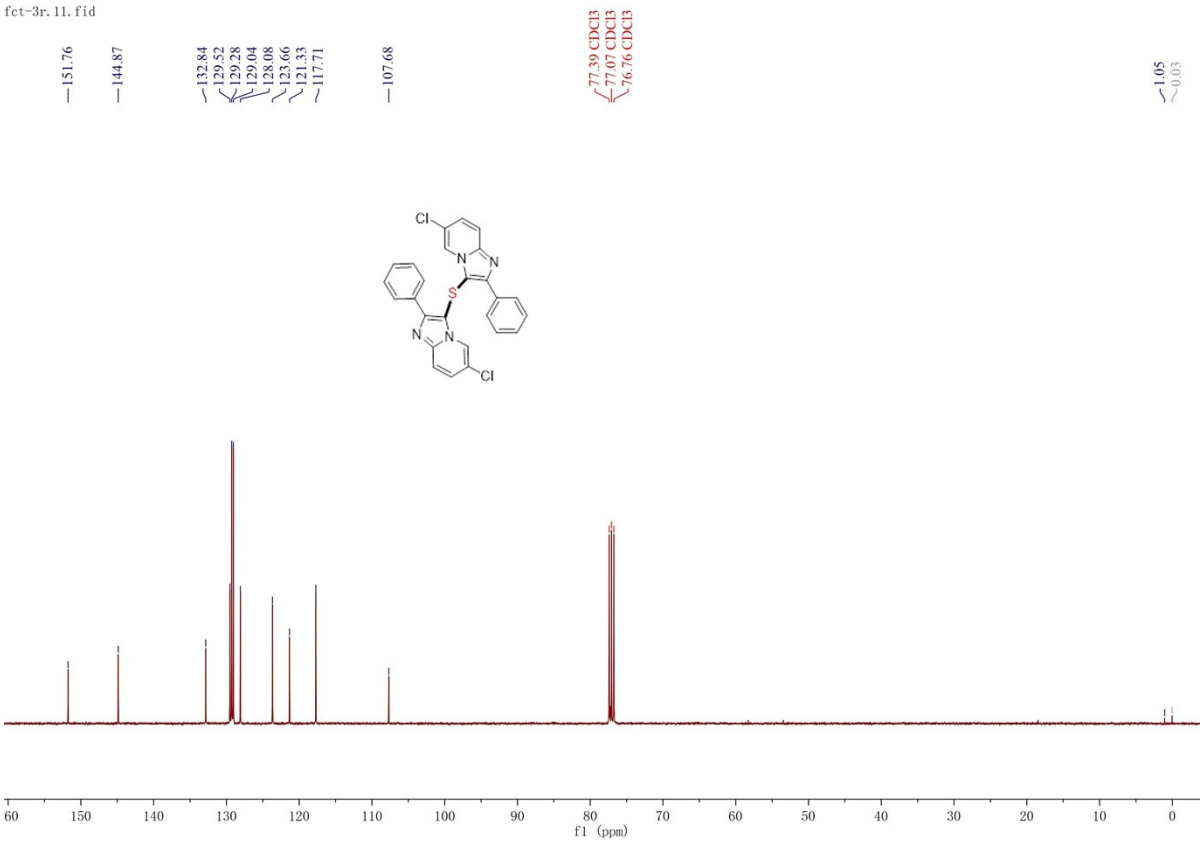

yyz191106-1.2.fid
yanyizhe 2019.11.26
150.24
146.54
133.80
129.77
128.81
128.55
127.26
125.36
123.20
112.65
107.95
77.23 CDCl3
76.80 CDCl3
16.75
0.00
f1 (ppm)

3r

fct-3r.1.fid

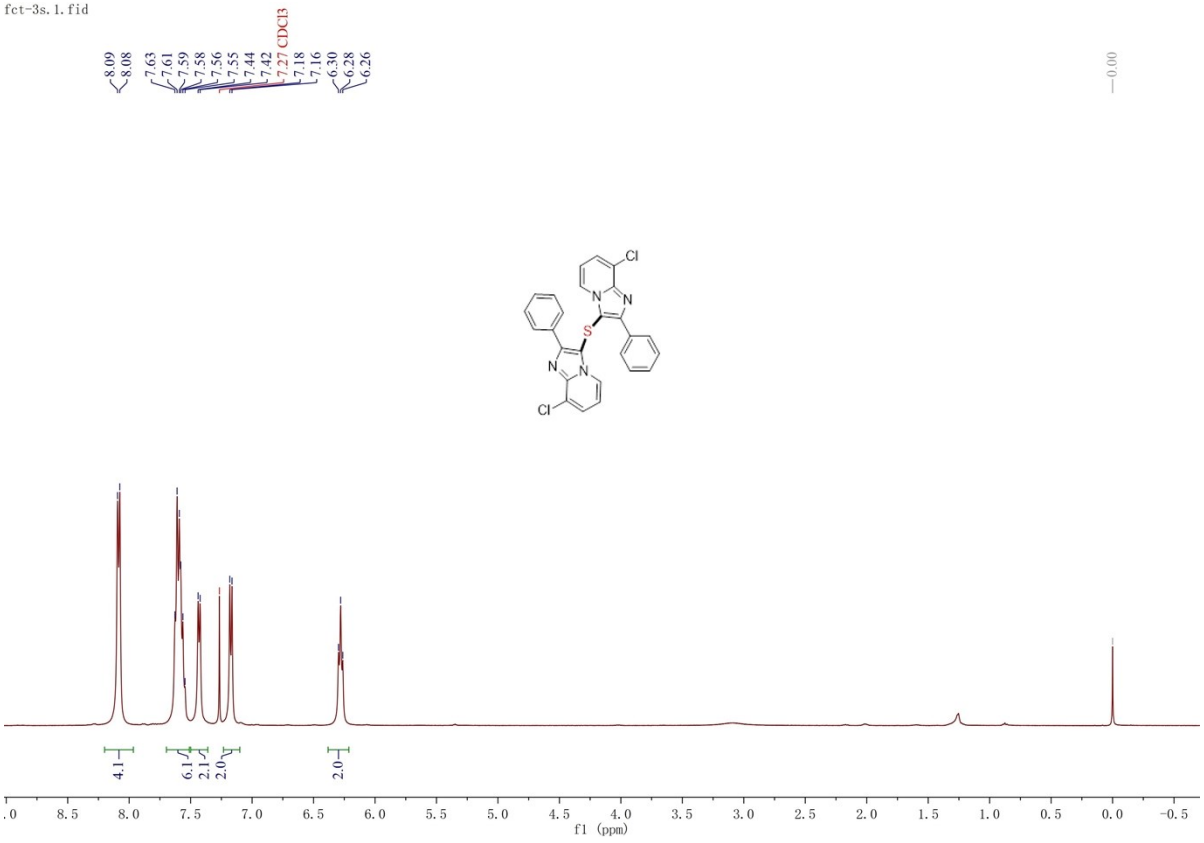


fct-3r.11.fid

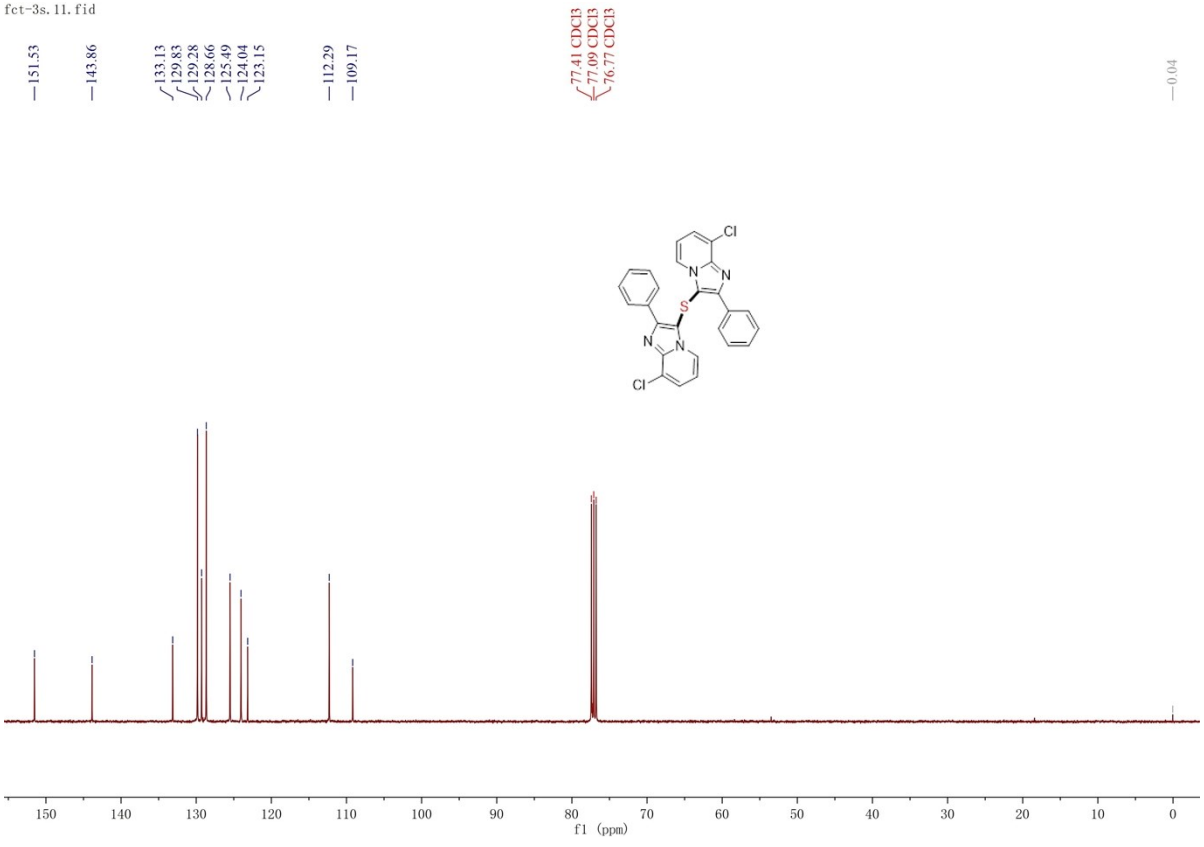


3s

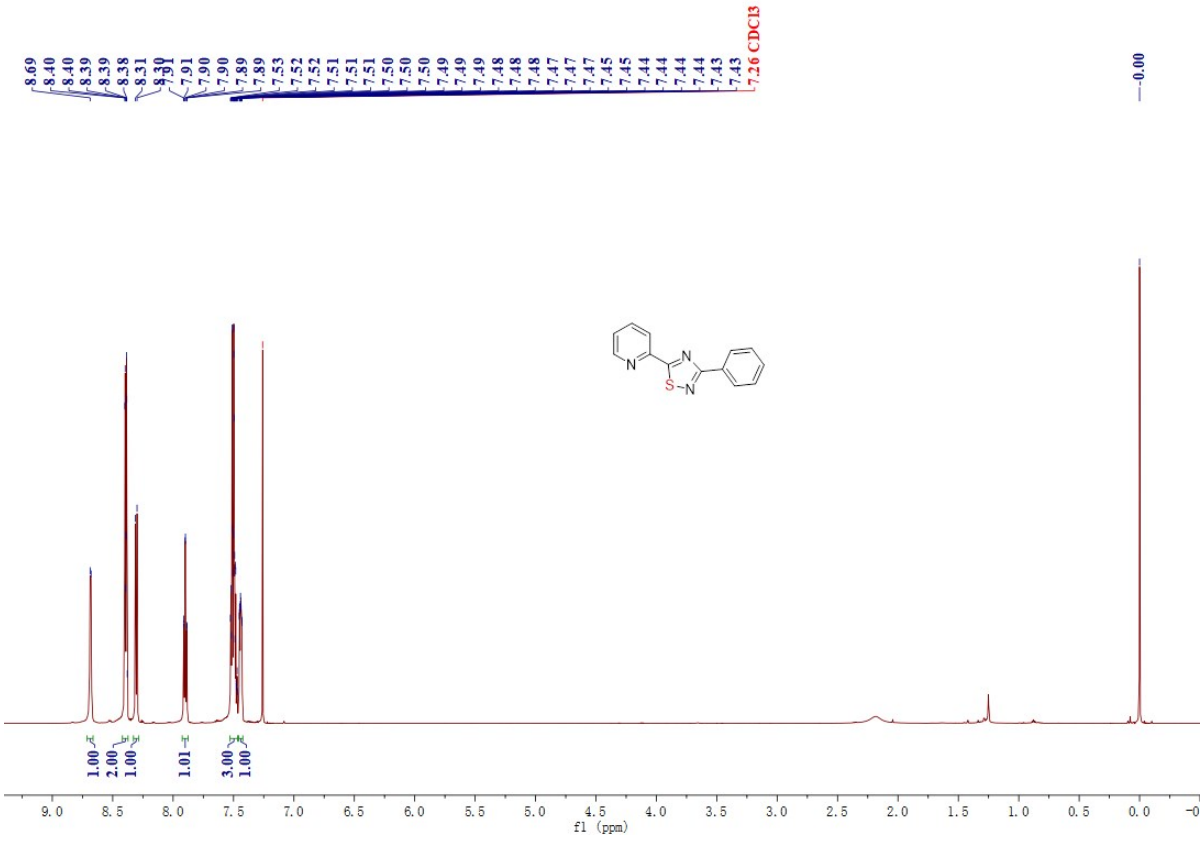
fct-3s.1.fid

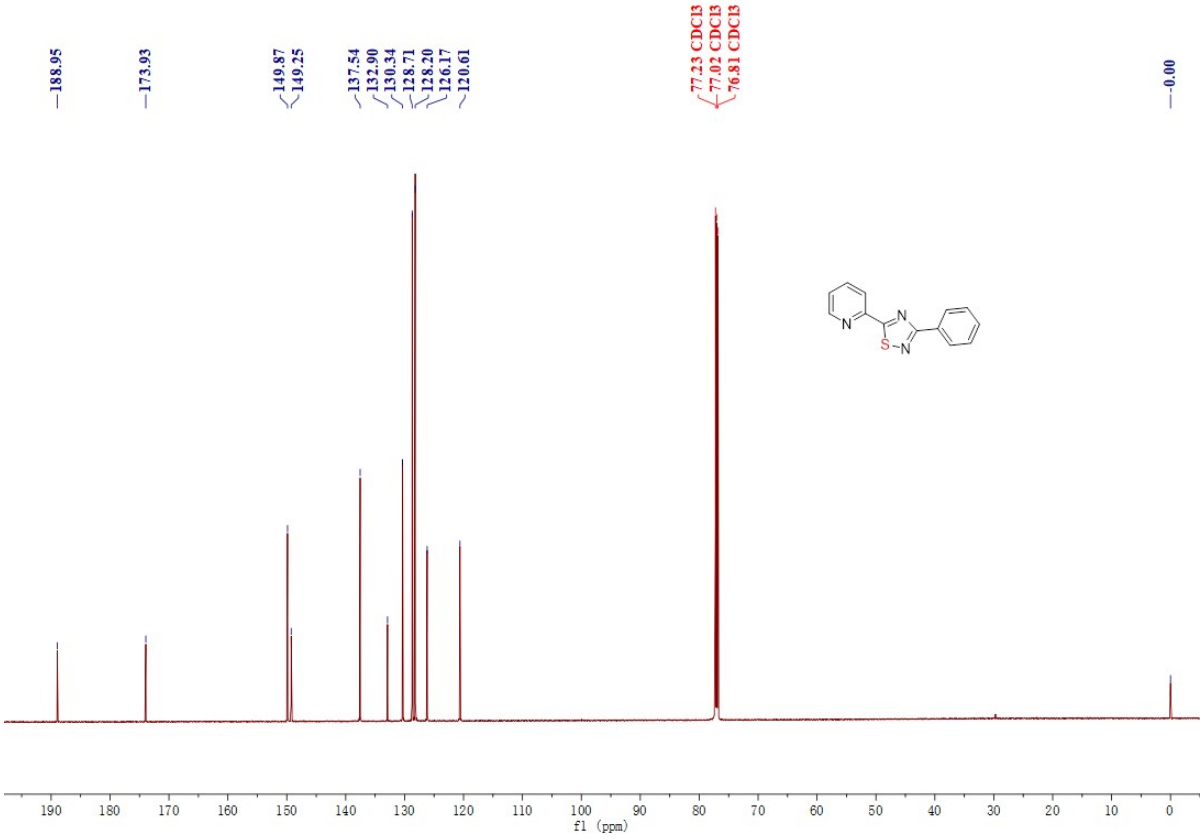


fct-3s.11.fid

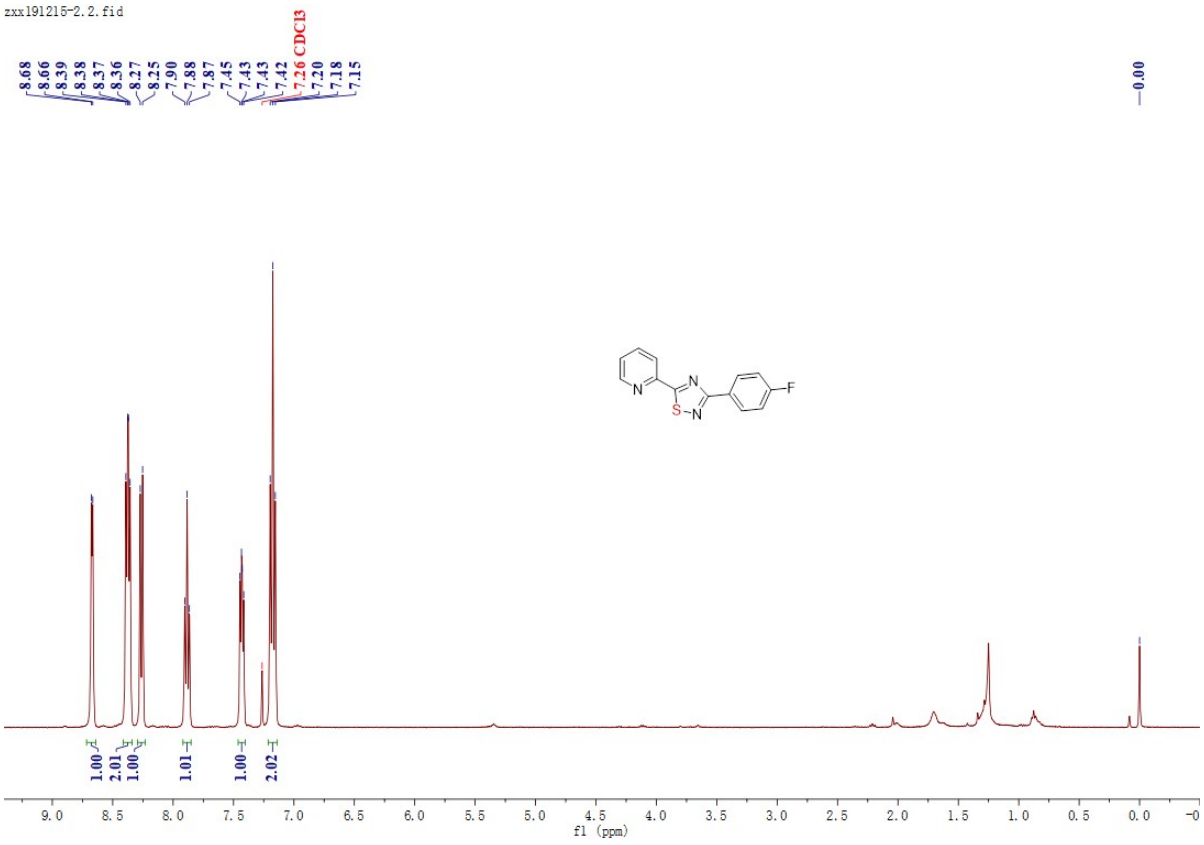


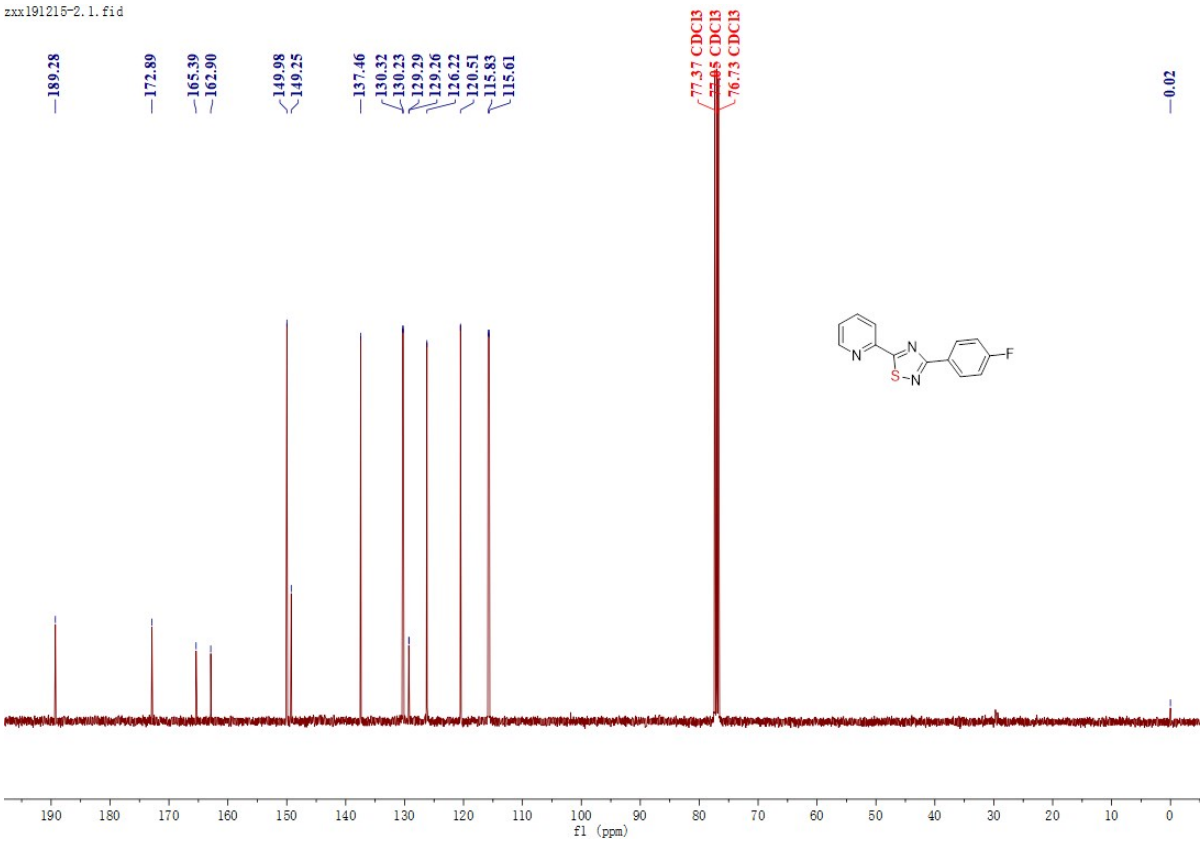
4a


8.69
8.40
8.40
8.39
8.39
8.38
8.31
7.91
7.90
7.90
7.89
7.89
7.53
7.52
7.51
7.51
7.51
7.50
7.50
7.50
7.49
7.49
7.49
7.48
7.48
7.48
7.47
7.47
7.47
7.45
7.44
7.44
7.44
7.44
7.43
7.26 CDCl3
0.00
1.00
2.00
1.00
1.01
3.00
1.00
f1 (ppm)


188.95
173.93
149.87
149.25
137.54
132.90
130.34
128.71
128.20
126.17
120.61
77.23 CDCl3
77.02 CDCl3
76.81 CDCl3
0.00
f1 (ppm)

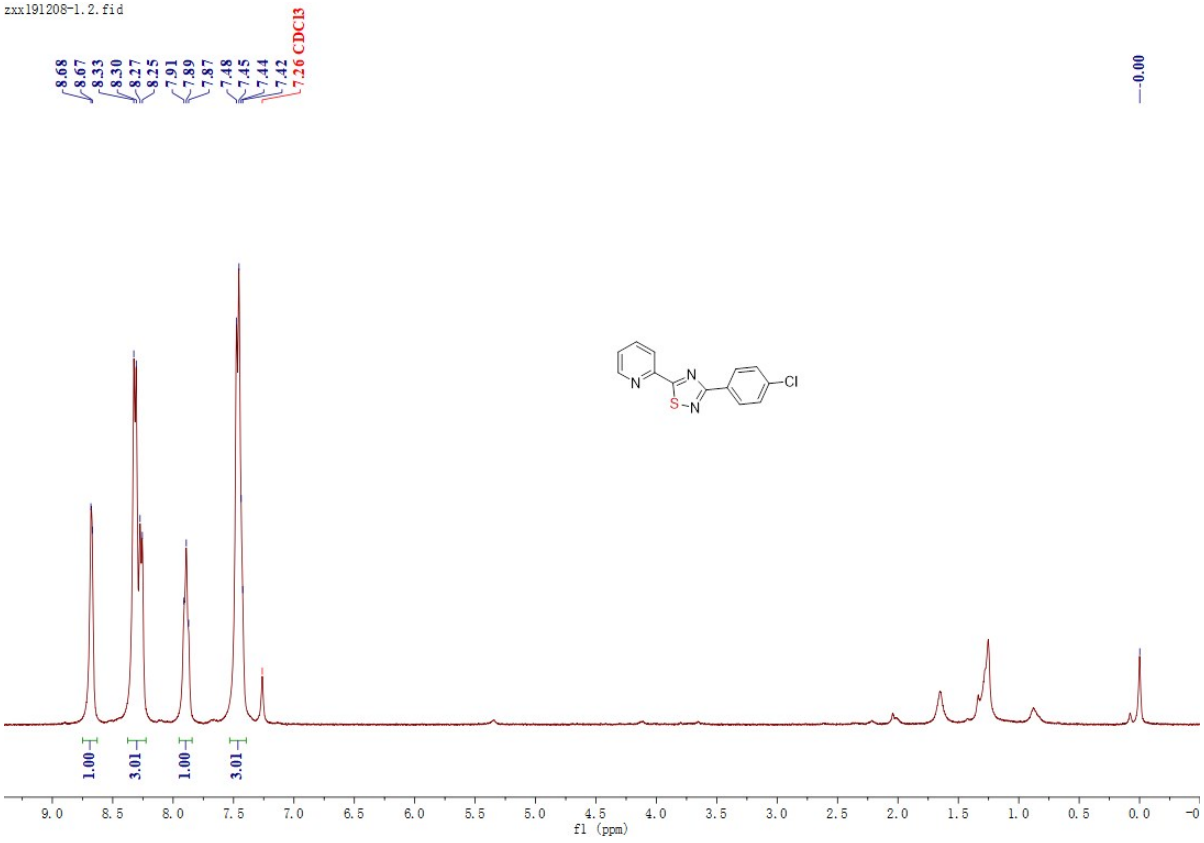
4b


zxx191215-2.2.fid
8.68
8.66
8.39
8.38
8.37
8.36
8.27
8.25
7.90
7.88
7.87
7.45
7.43
7.43
7.42
7.26 CDCl3
7.20
7.18
7.15
0.00
1.00
2.01
1.00
1.01
1.00
2.02
F
f1 (ppm)

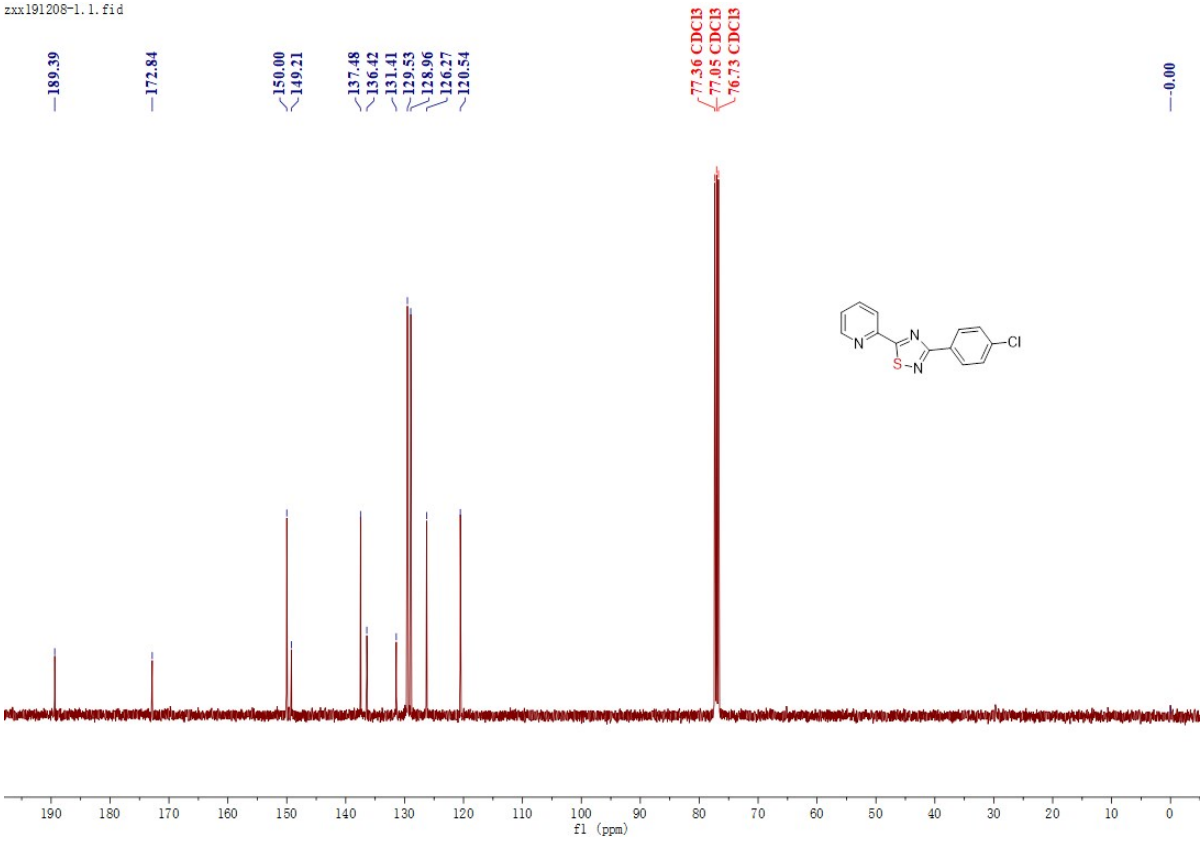

zxx191215-2.1.fid
189.28
172.89
165.39
162.90
149.98
149.25
137.46
130.32
130.23
129.29
129.26
126.22
120.51
115.83
115.61
77.37 CDCl3
77.05 CDCl3
76.73 CDCl3
0.02
F
f1 (ppm)

4c

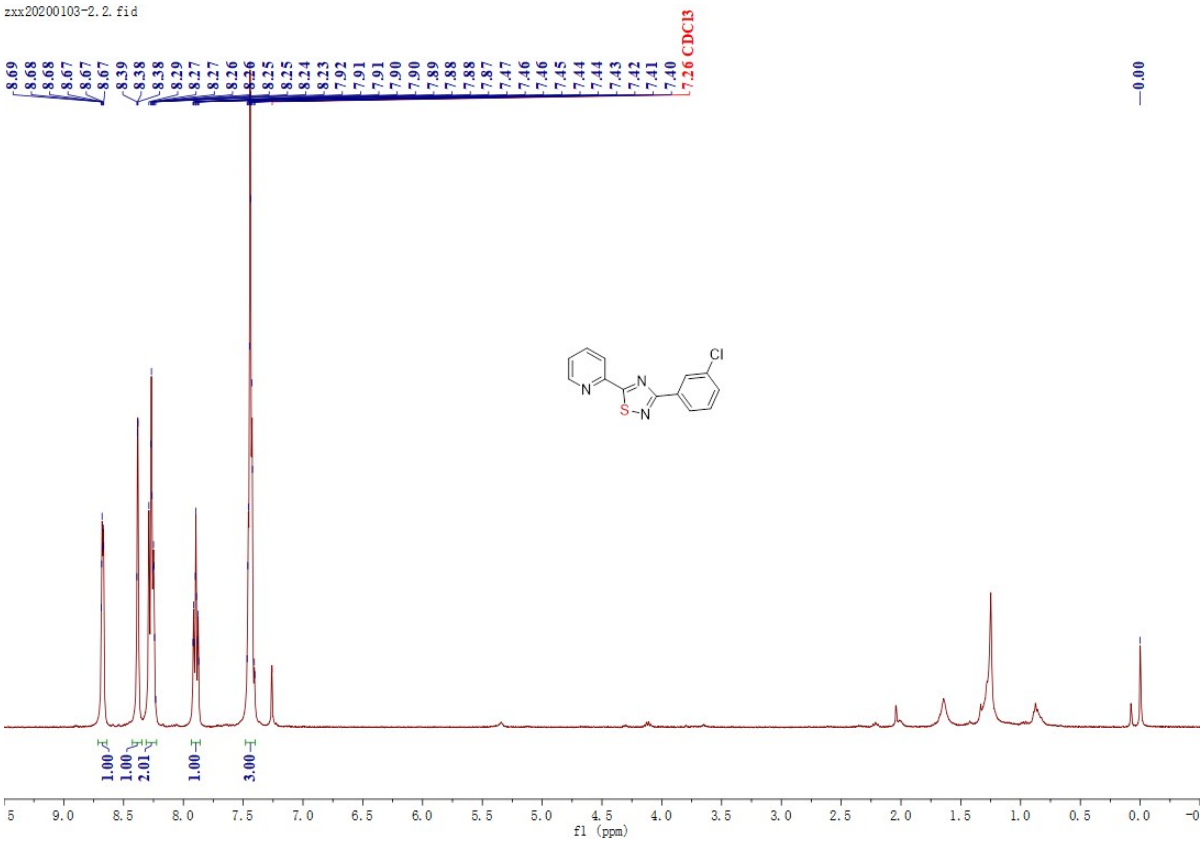
zxx191208-1.2.fid

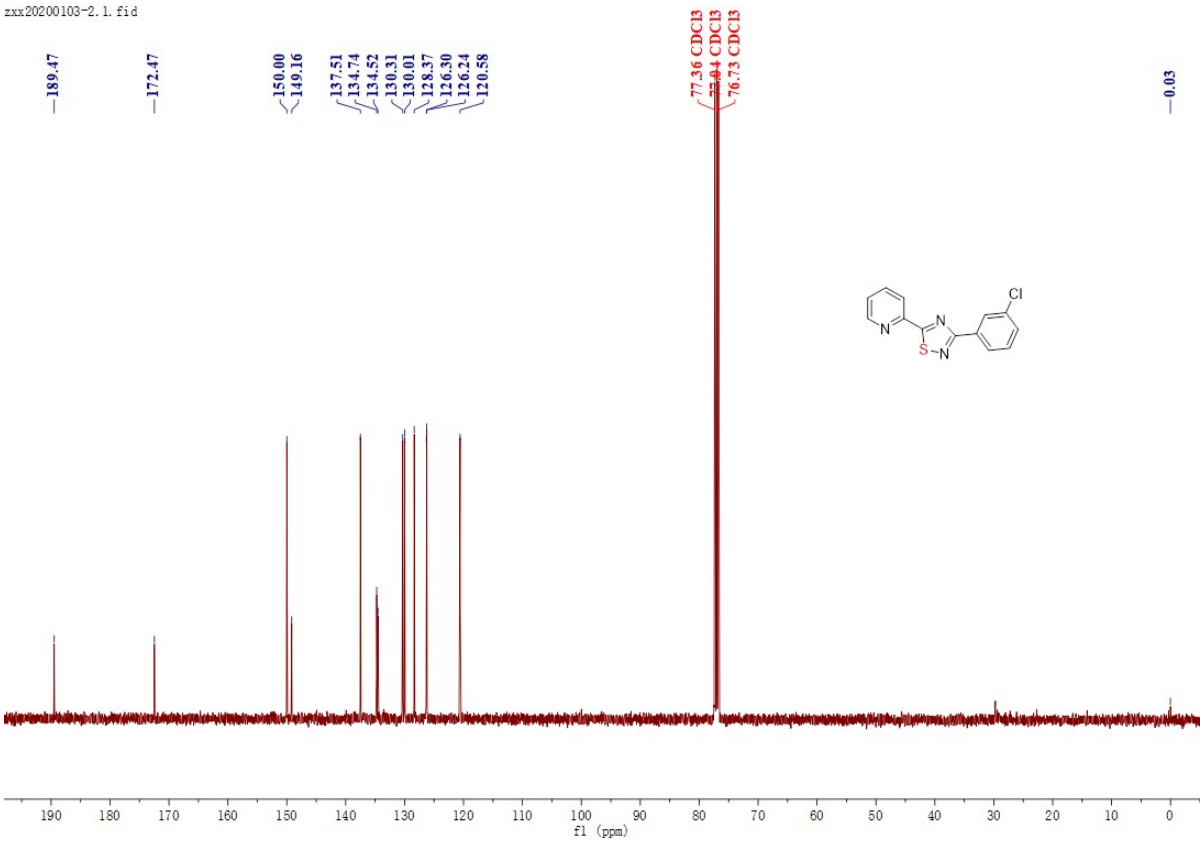


zxx191208-1.1.fid



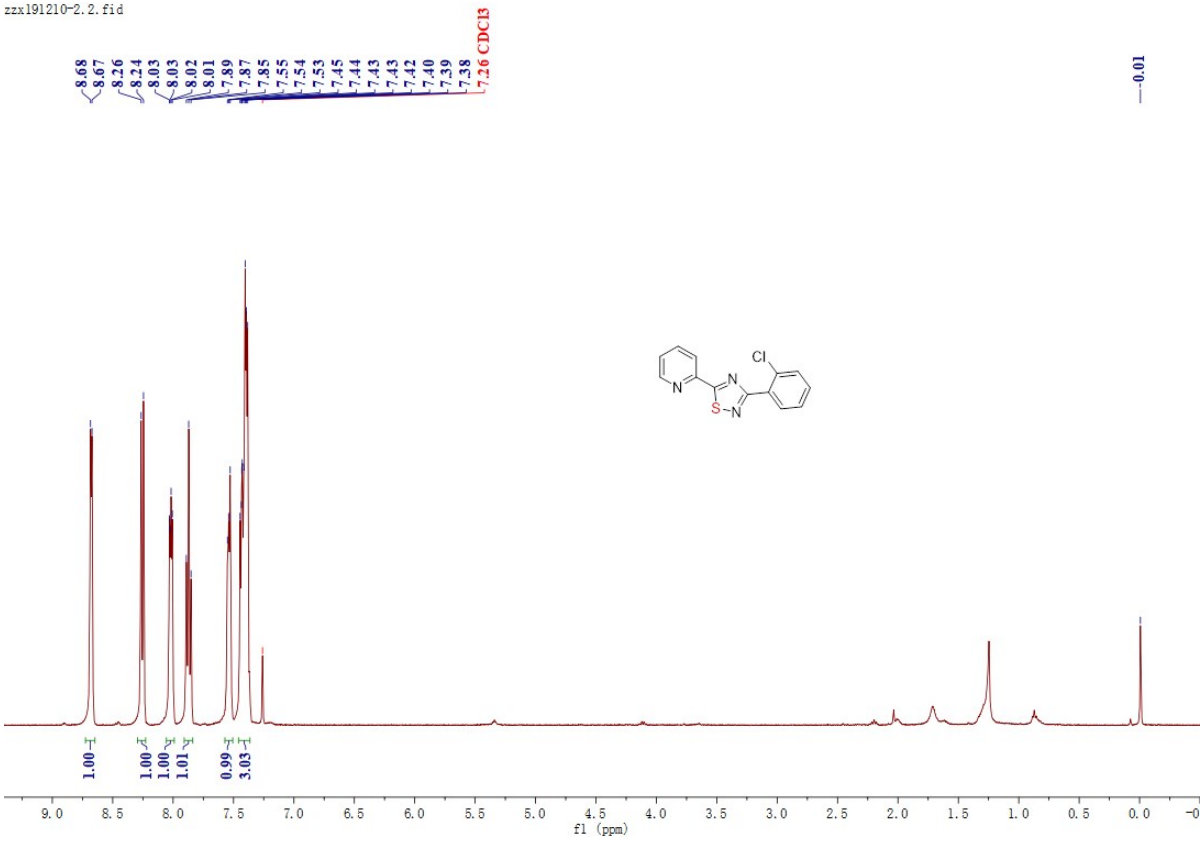
4d


zxx20200103-2.2.fid
8.69
8.68
8.68
8.67
8.67
8.67
8.39
8.38
8.38
8.29
8.27
8.27
8.26
8.26
8.25
8.25
8.24
8.23
7.92
7.91
7.91
7.90
7.90
7.89
7.88
7.88
7.87
7.47
7.46
7.46
7.45
7.44
7.44
7.43
7.42
7.41
7.40
7.26 CDCl3
0.00
1.00
1.00
2.01
1.00
3.00
Cl
N
S
N
f1 (ppm)

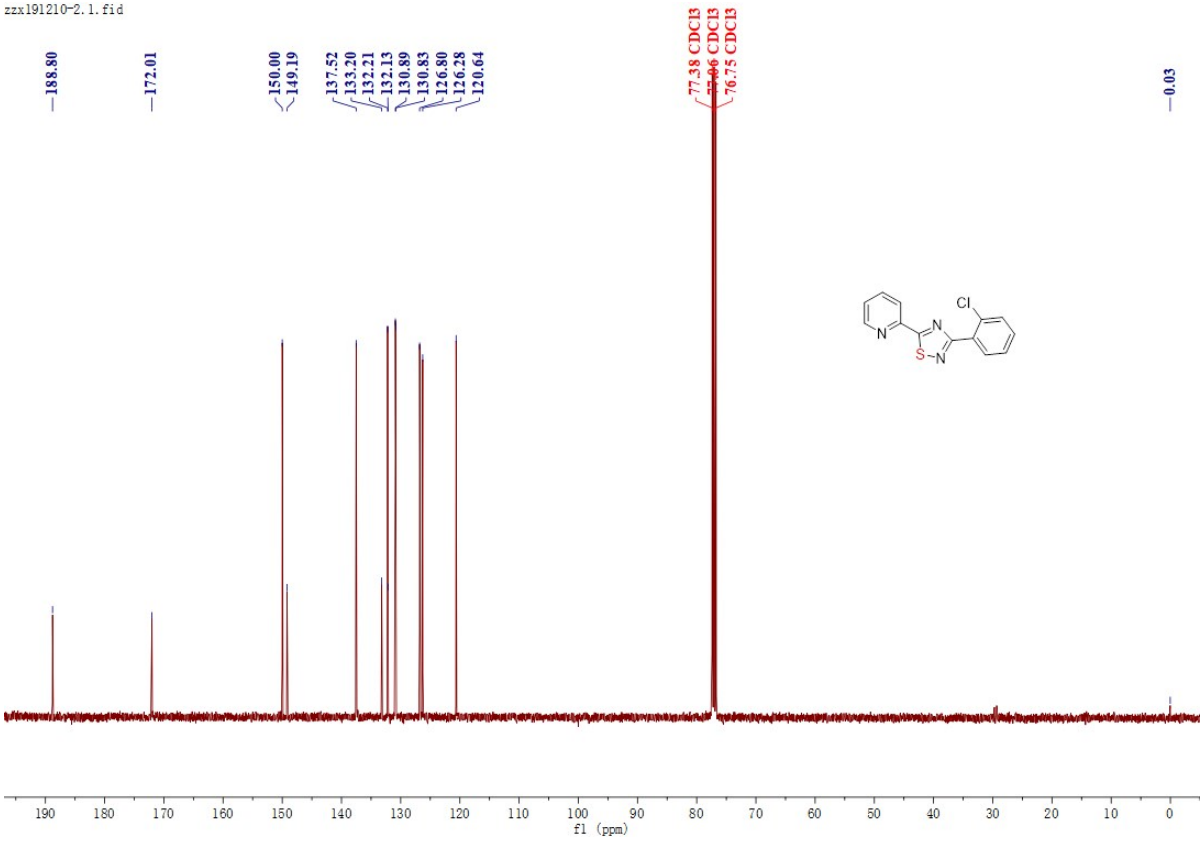

zxx20200103-2.1.fid
189.47
172.47
150.00
149.16
137.51
134.74
134.52
130.31
130.01
128.37
126.30
126.24
120.58
77.36 CDCl3
77.04 CDCl3
76.73 CDCl3
0.03
Cl
N
S
N
f1 (ppm)

4e

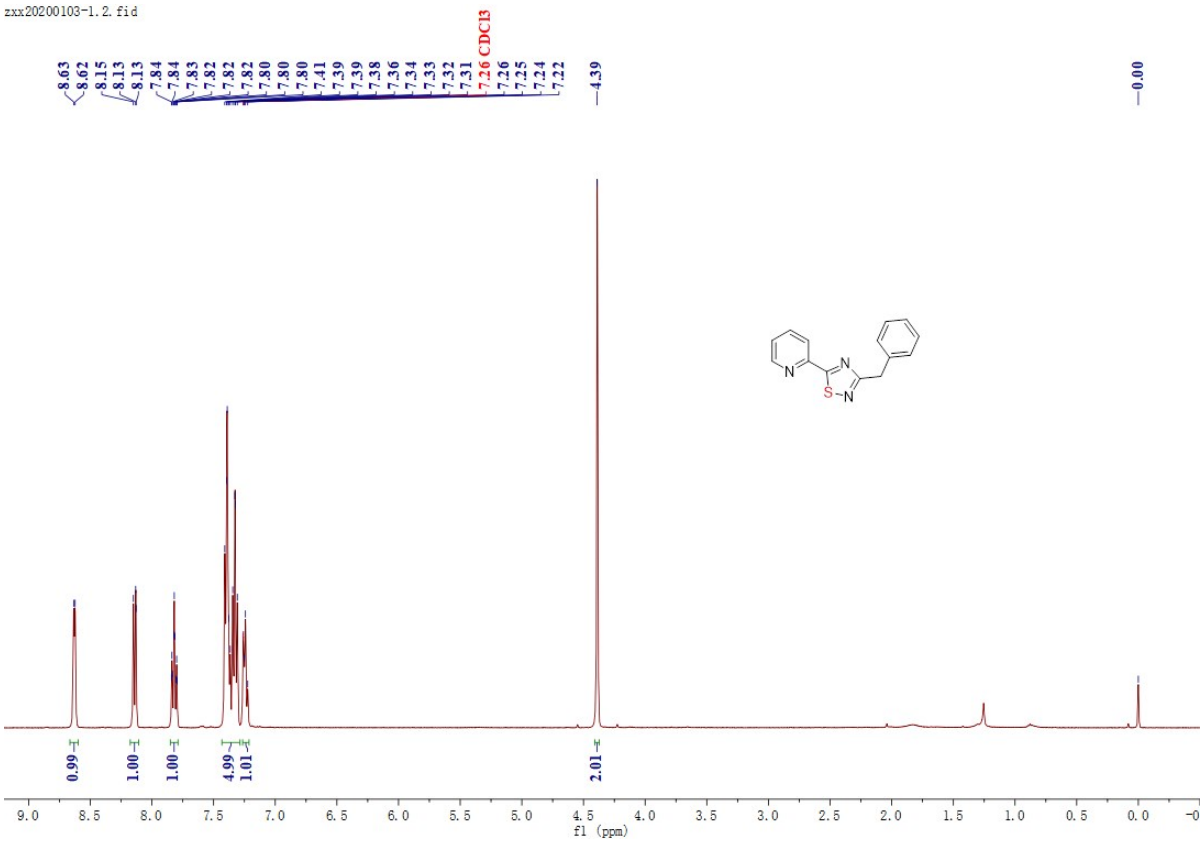
zzx191210-2.2.fid

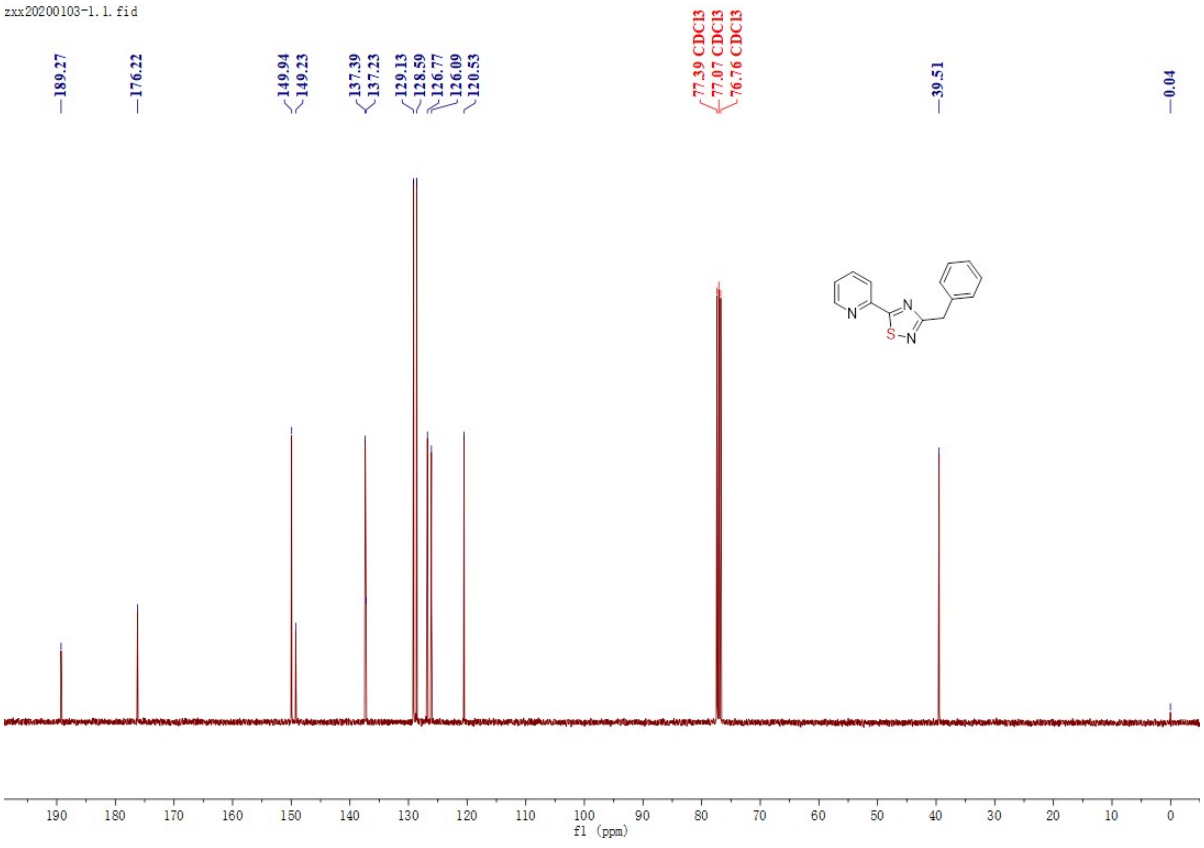


zzx191210-2.1.fid



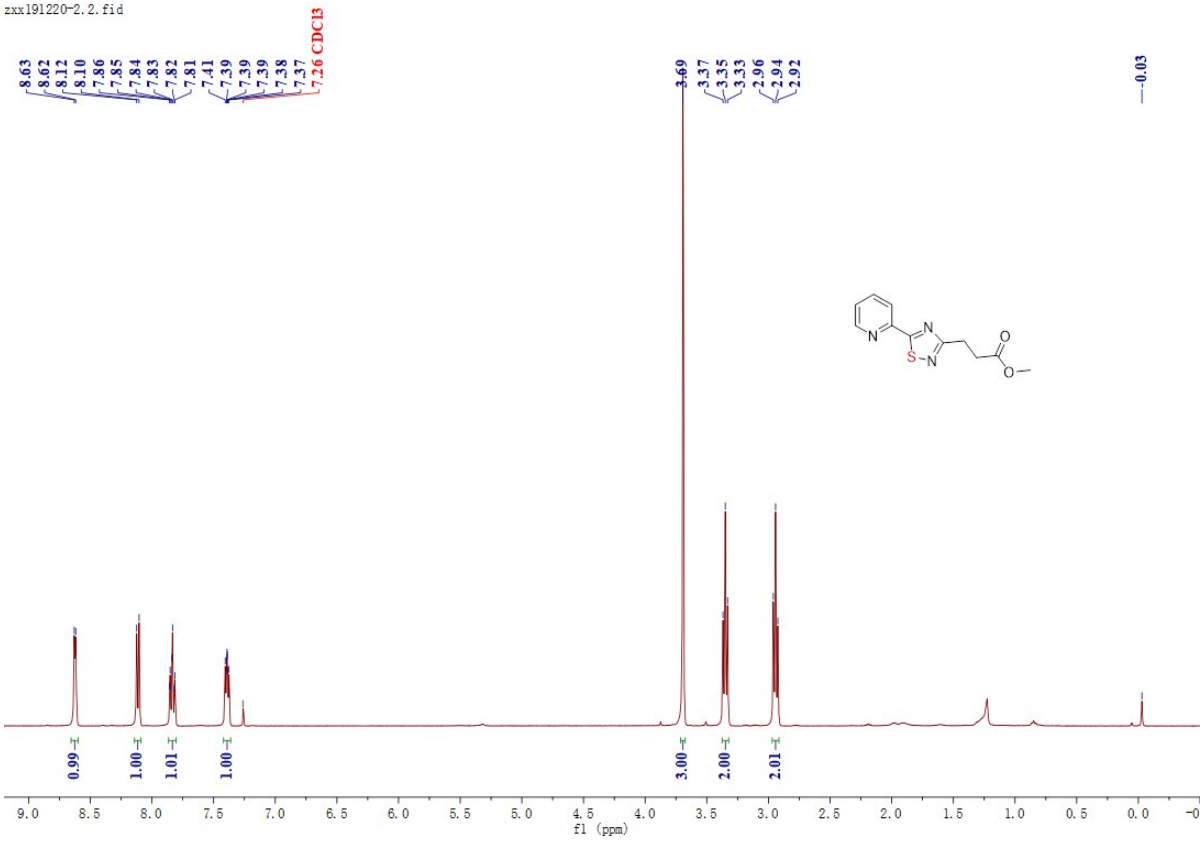
4f


zxx20200103-1.2.fid
8.63
8.62
8.15
8.13
8.13
7.84
7.84
7.83
7.82
7.82
7.82
7.80
7.80
7.80
7.41
7.39
7.39
7.38
7.36
7.34
7.33
7.32
7.31
7.26 CDCl3
7.26
7.25
7.24
7.22
4.39
0.00
0.99
1.00
1.00
4.99
1.01
2.01
f1 (ppm)

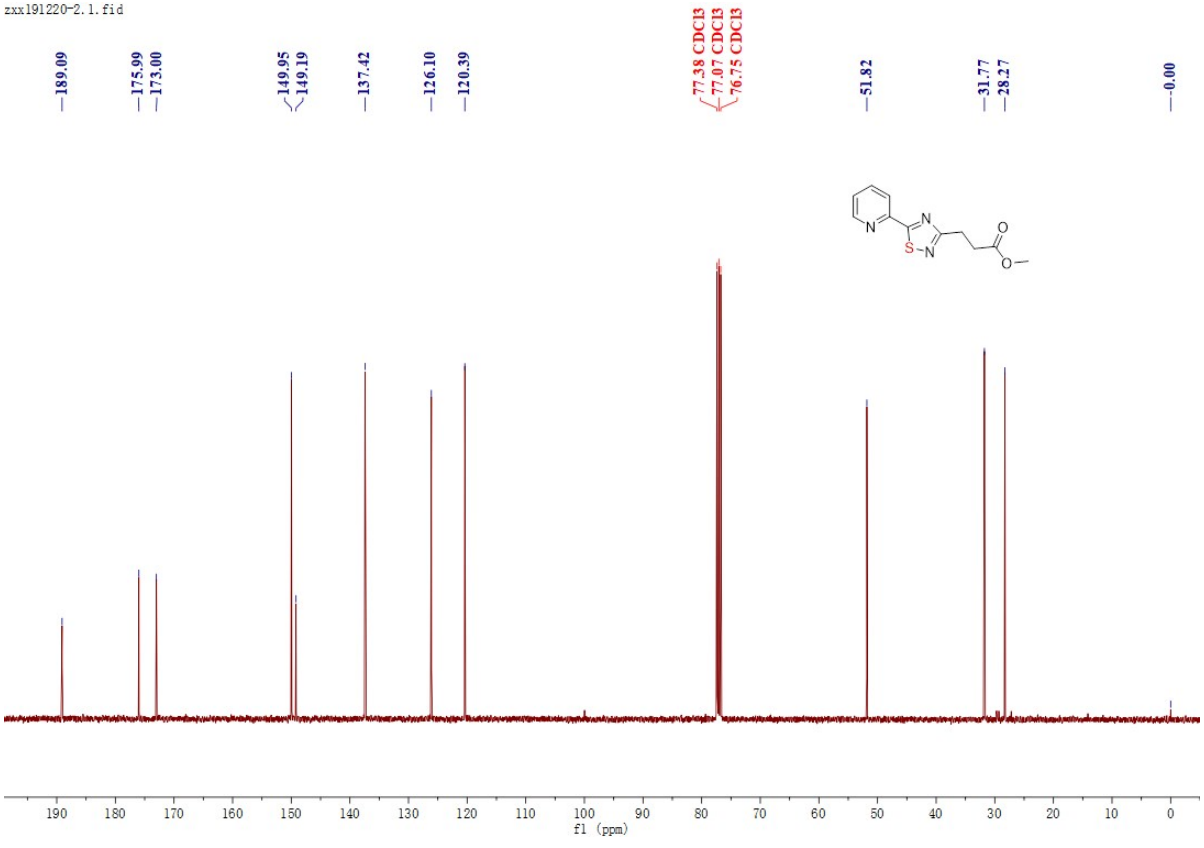

zxx20200103-1.1.fid
189.27
176.22
149.94
149.23
137.39
137.23
129.13
128.59
126.77
126.09
120.53
77.39 CDCl3
77.07 CDCl3
76.76 CDCl3
39.51
0.04
f1 (ppm)

4g

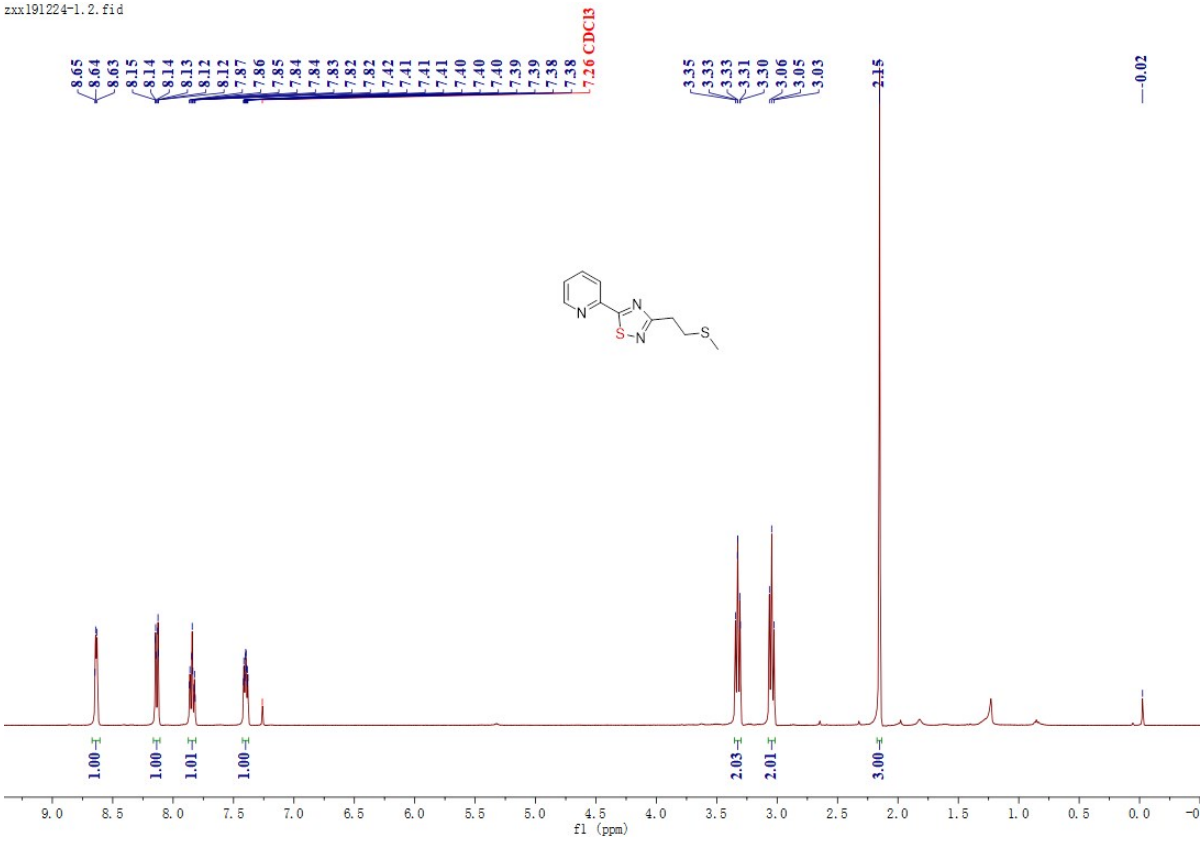
zxx191220-2.2.fid

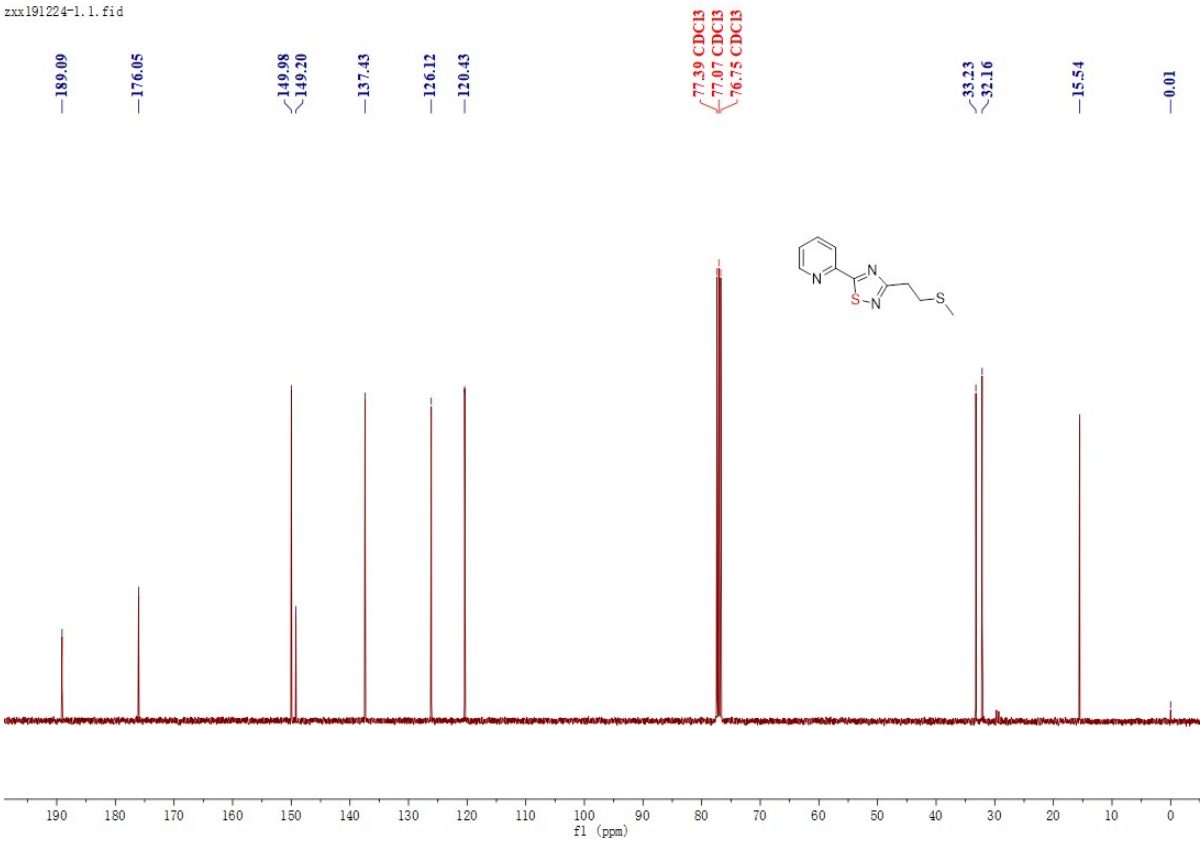


zxx191220-2.1.fid



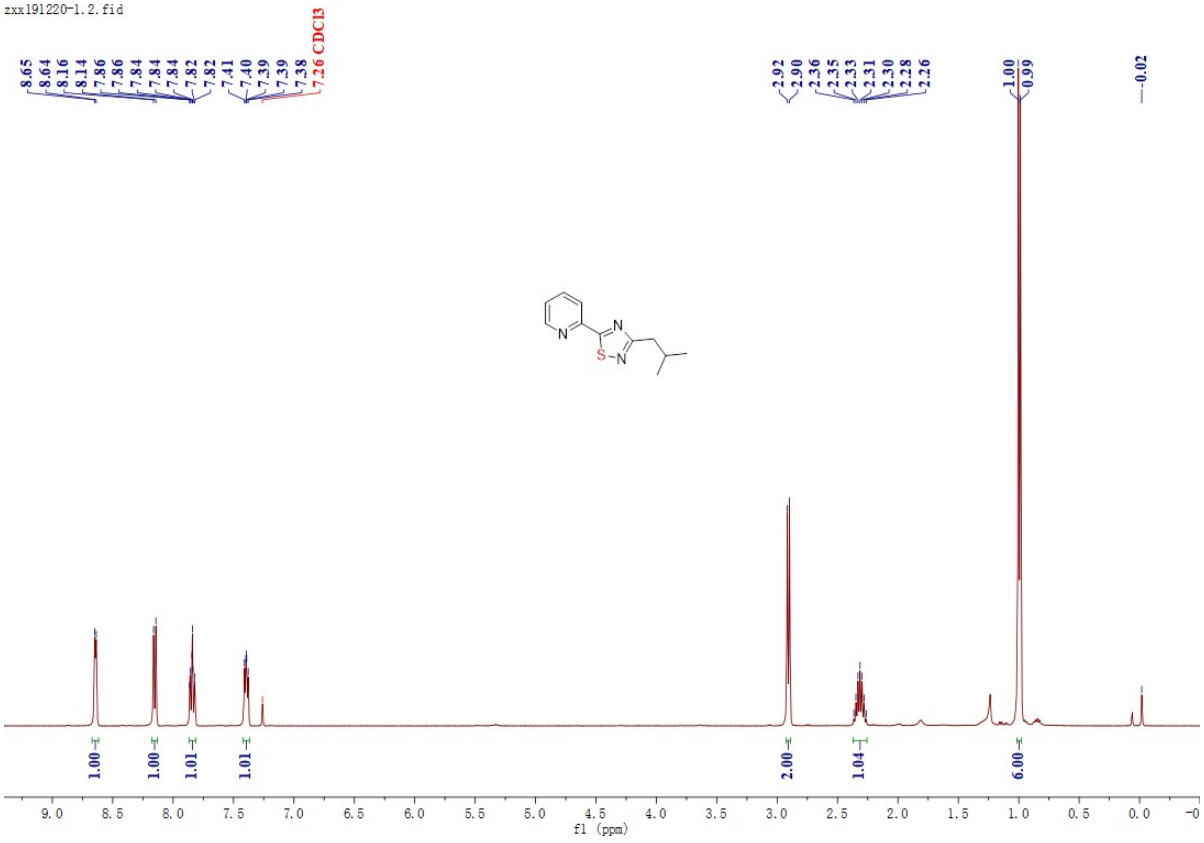
4h


zxx191224-1.2.fid
8.65
8.64
8.63
8.15
8.14
8.14
8.13
8.12
8.12
7.87
7.86
7.85
7.84
7.84
7.83
7.82
7.42
7.41
7.41
7.41
7.40
7.40
7.40
7.39
7.39
7.38
7.38
7.26 CDCl3
3.35
3.33
3.33
3.31
3.30
3.06
3.05
3.03
2.15
0.02
1.00
1.00
1.01
1.00
2.03
2.01
3.00
f1 (ppm)

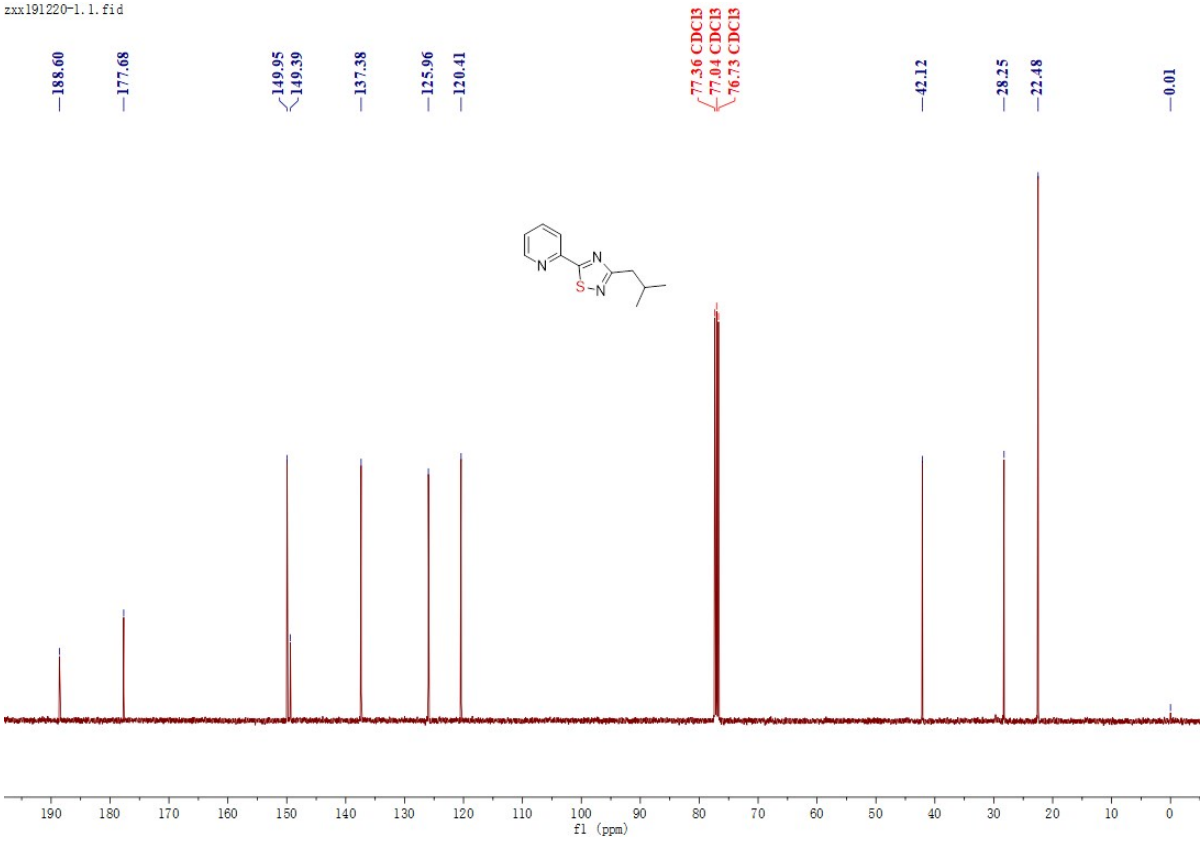

zxx191224-1.1.fid
189.09
176.05
149.98
149.20
137.43
126.12
120.43
77.39 CDCl3
77.07 CDCl3
76.75 CDCl3
33.23
32.16
15.54
0.01
f1 (ppm)

4i

zxx191220-1.2.fid

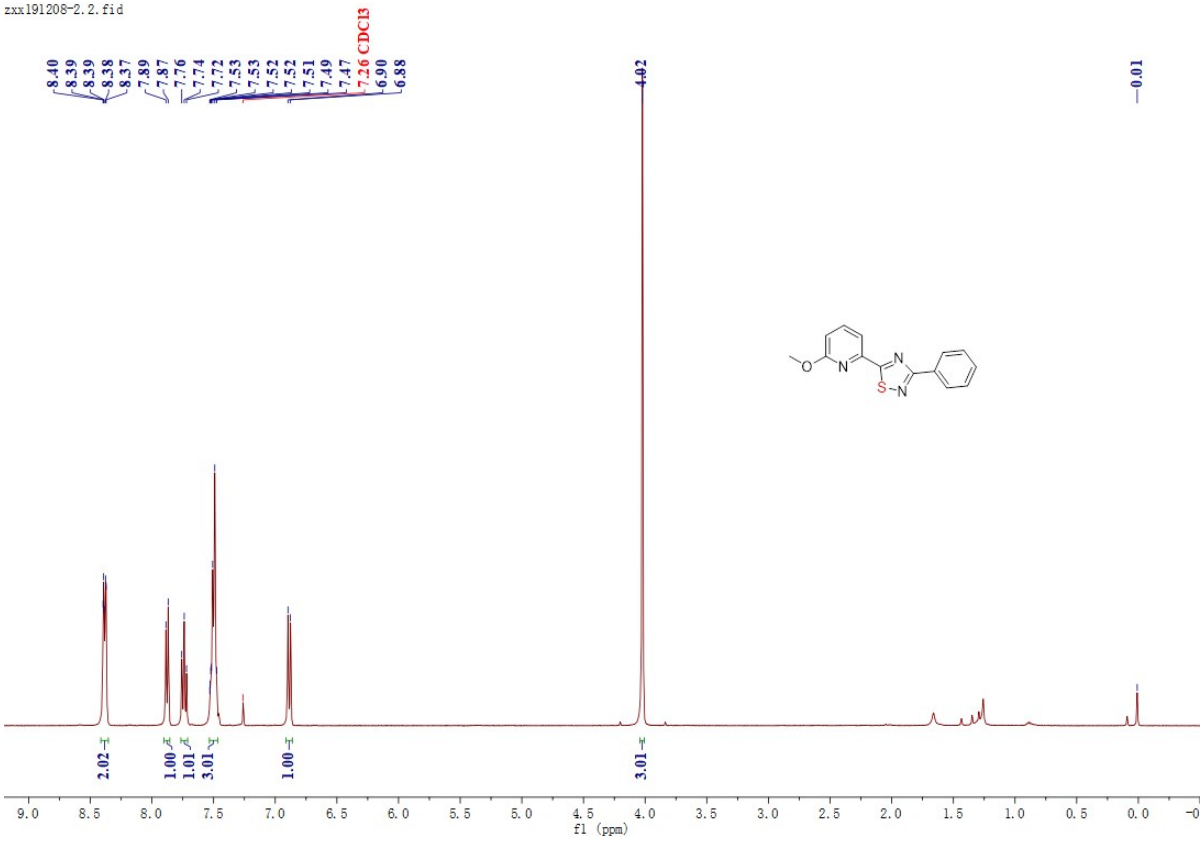


zxx191220-1.1.fid

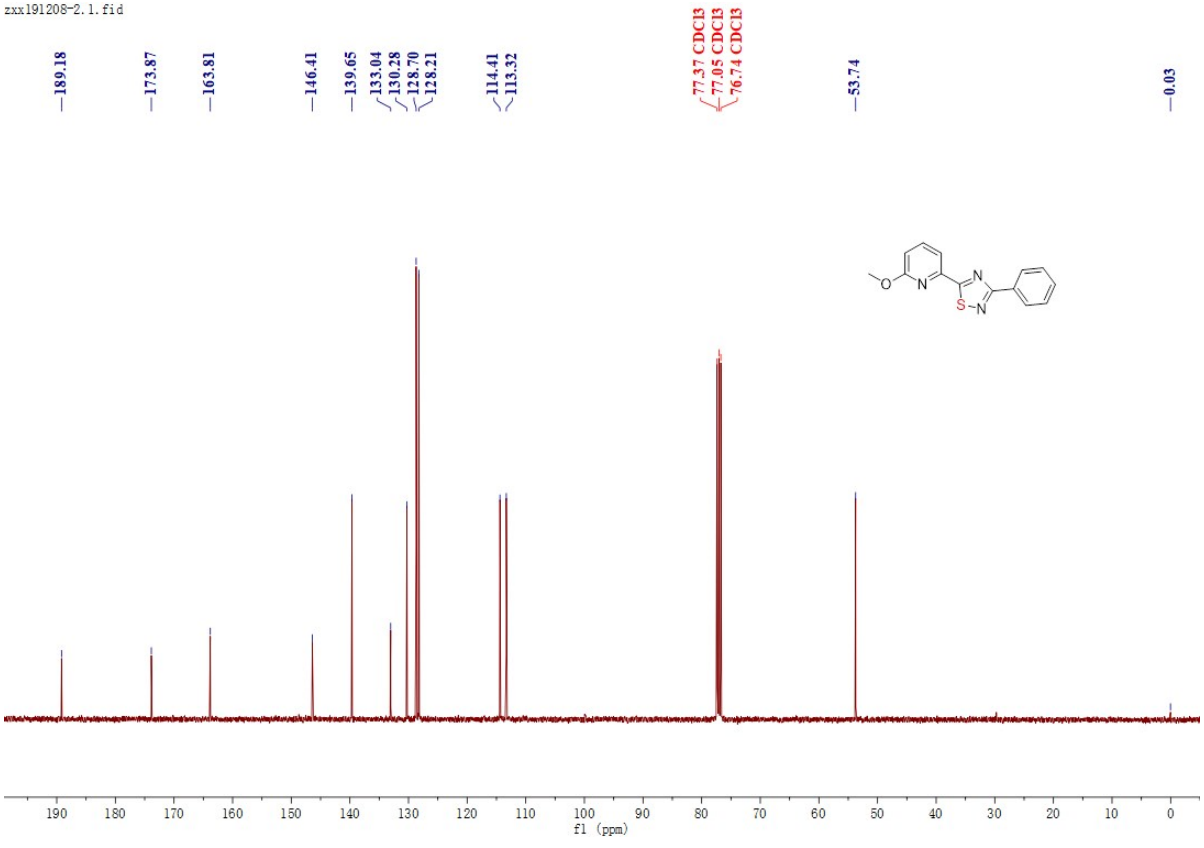


4j

zxx191208-2.2.fid

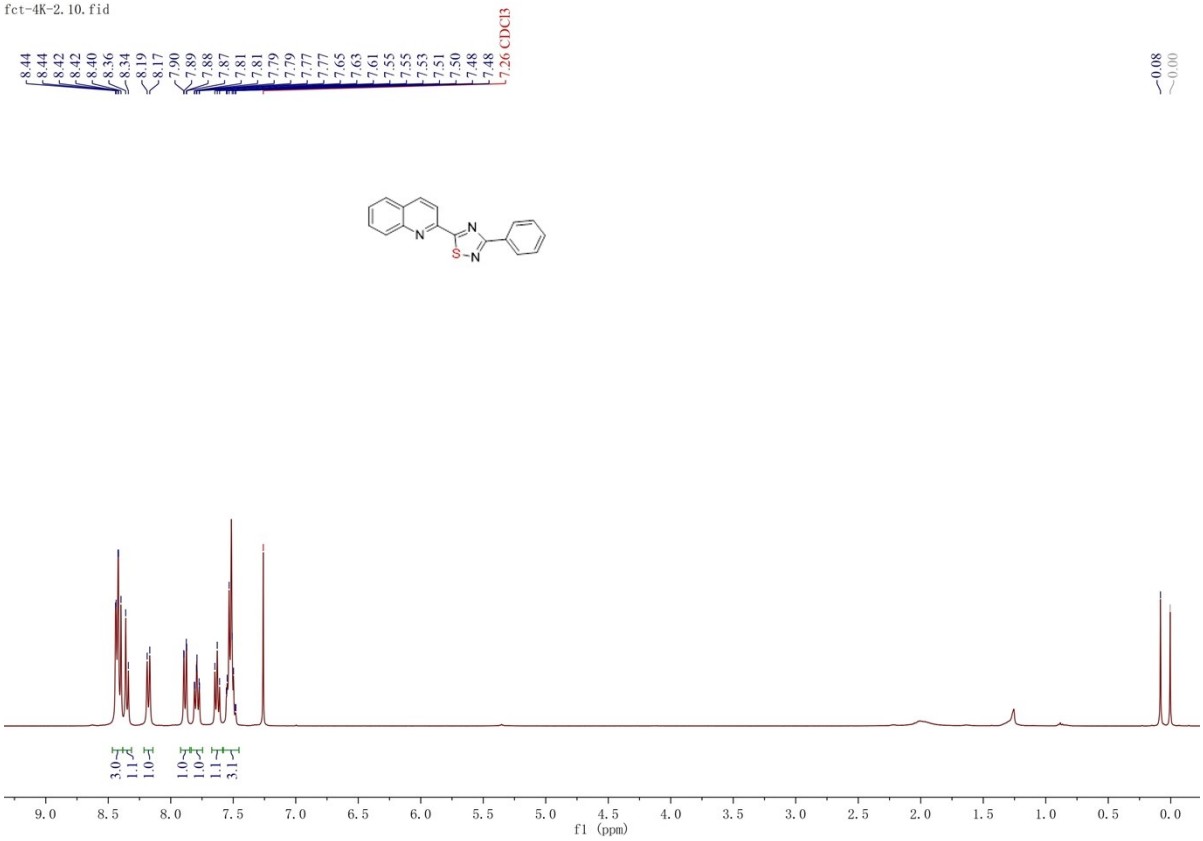


zxx191208-2.1.fid

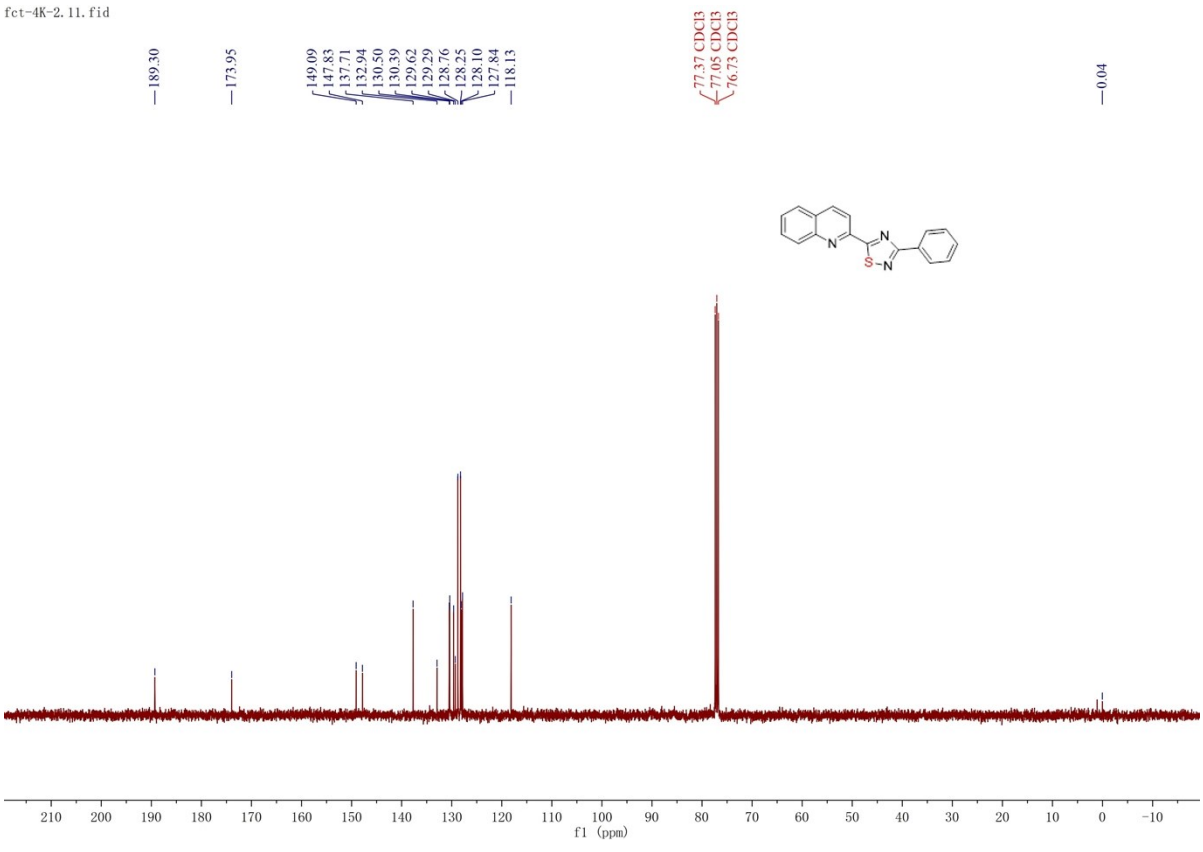


4k

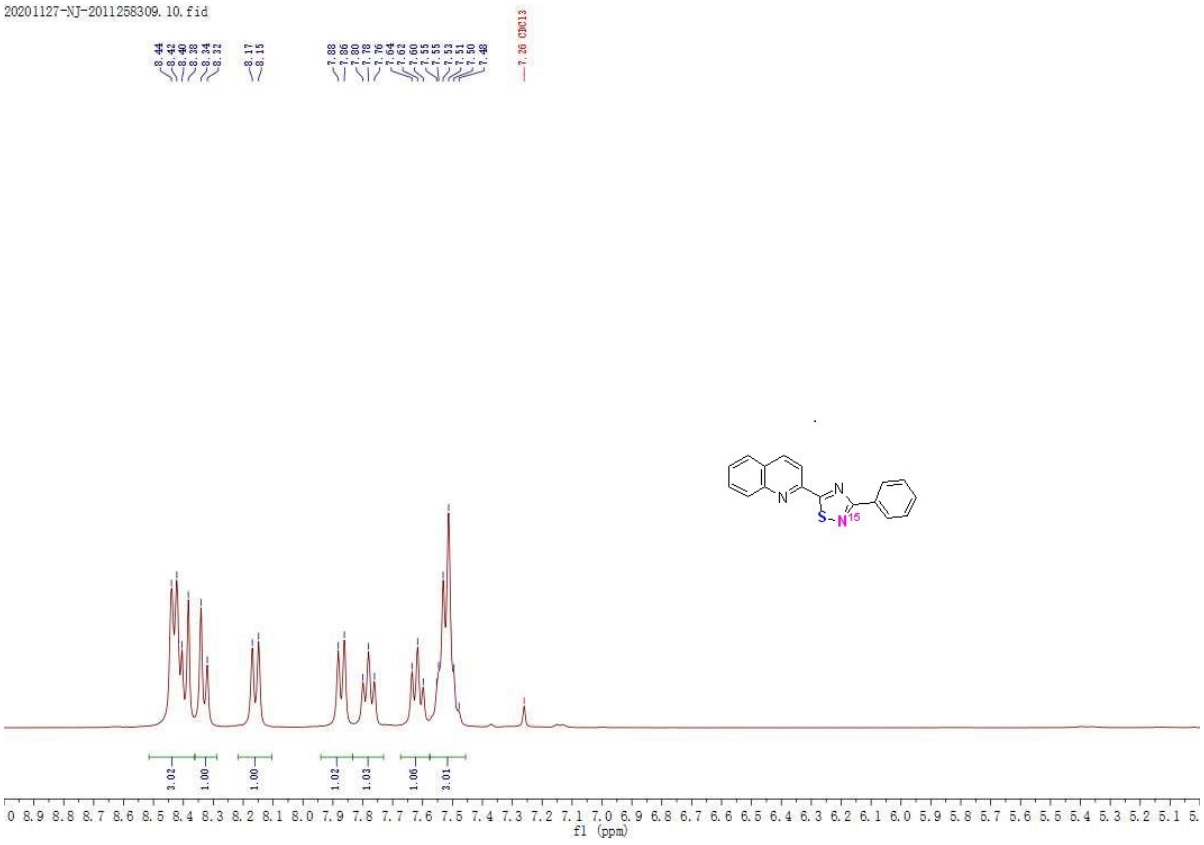
fct-4K-2.10.fid



fct-4K-2.11.fid



4l


20201127-NJ-2011258309.10.fid
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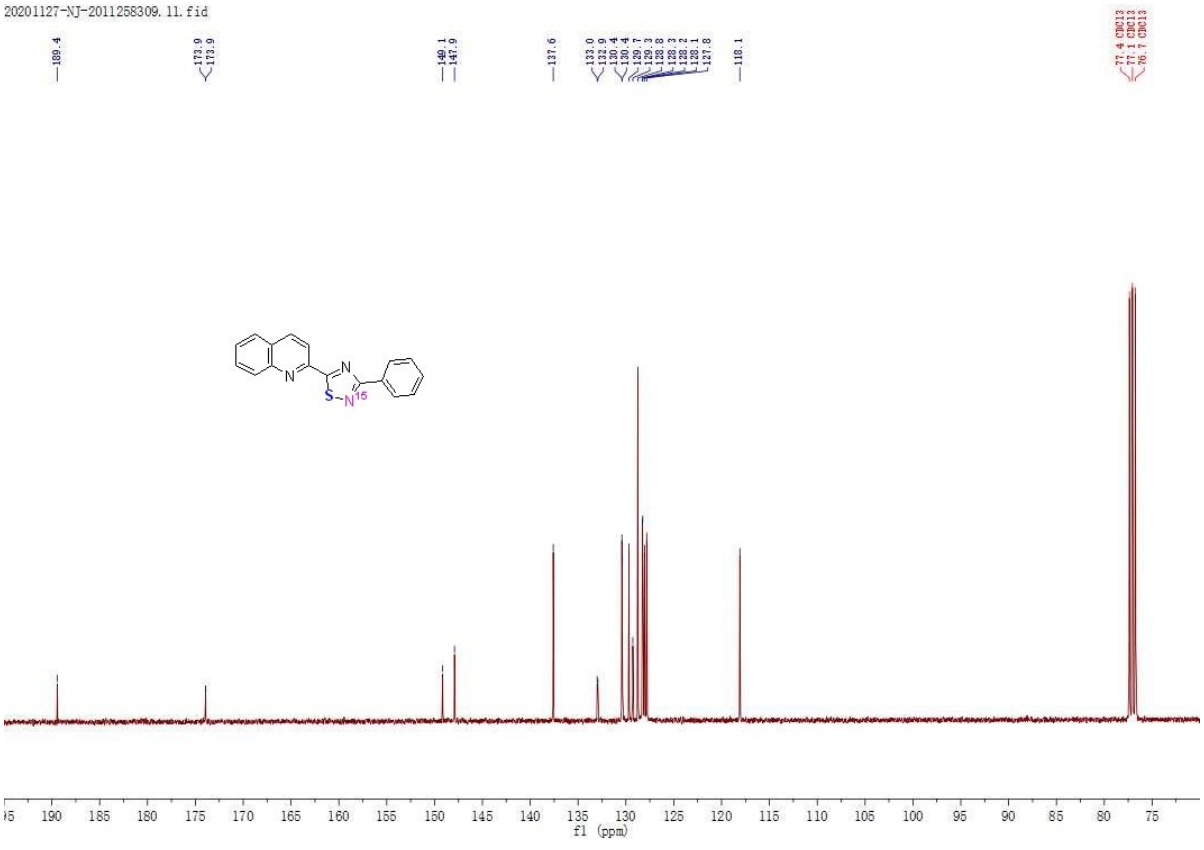
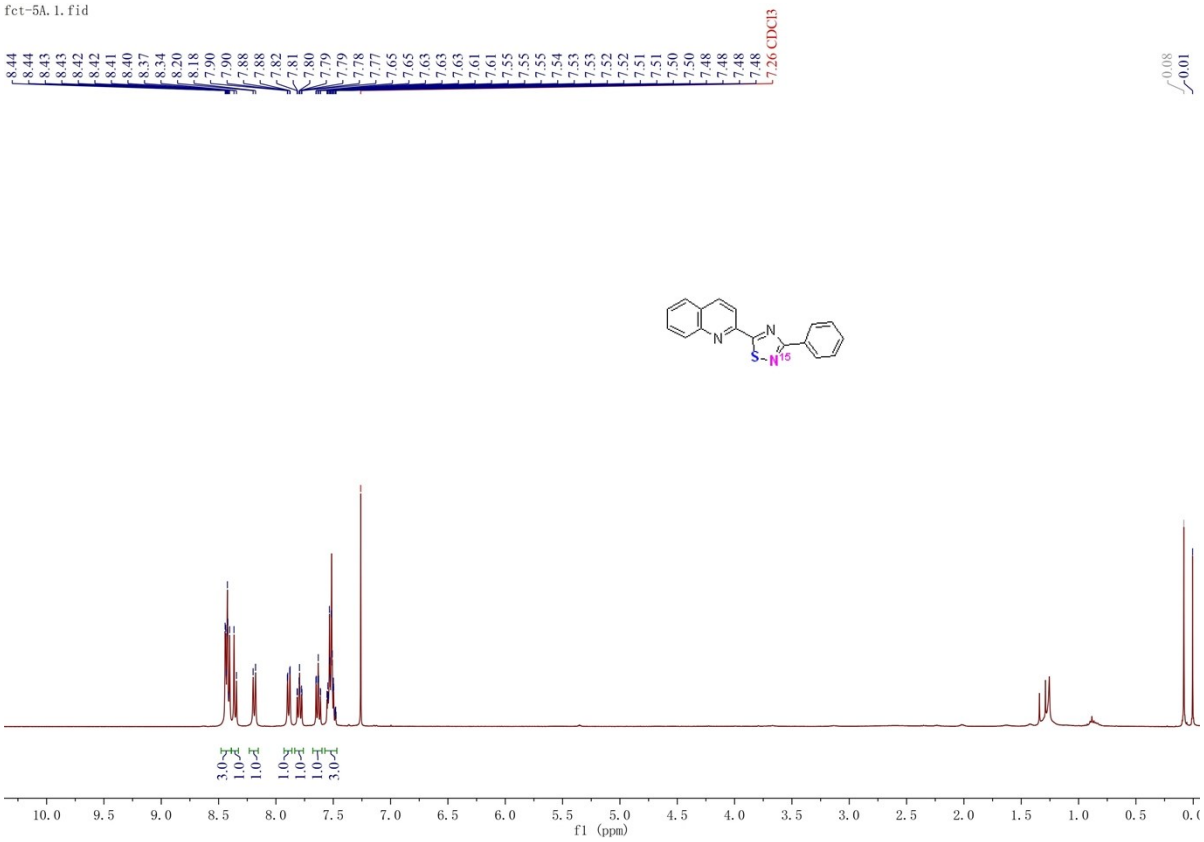
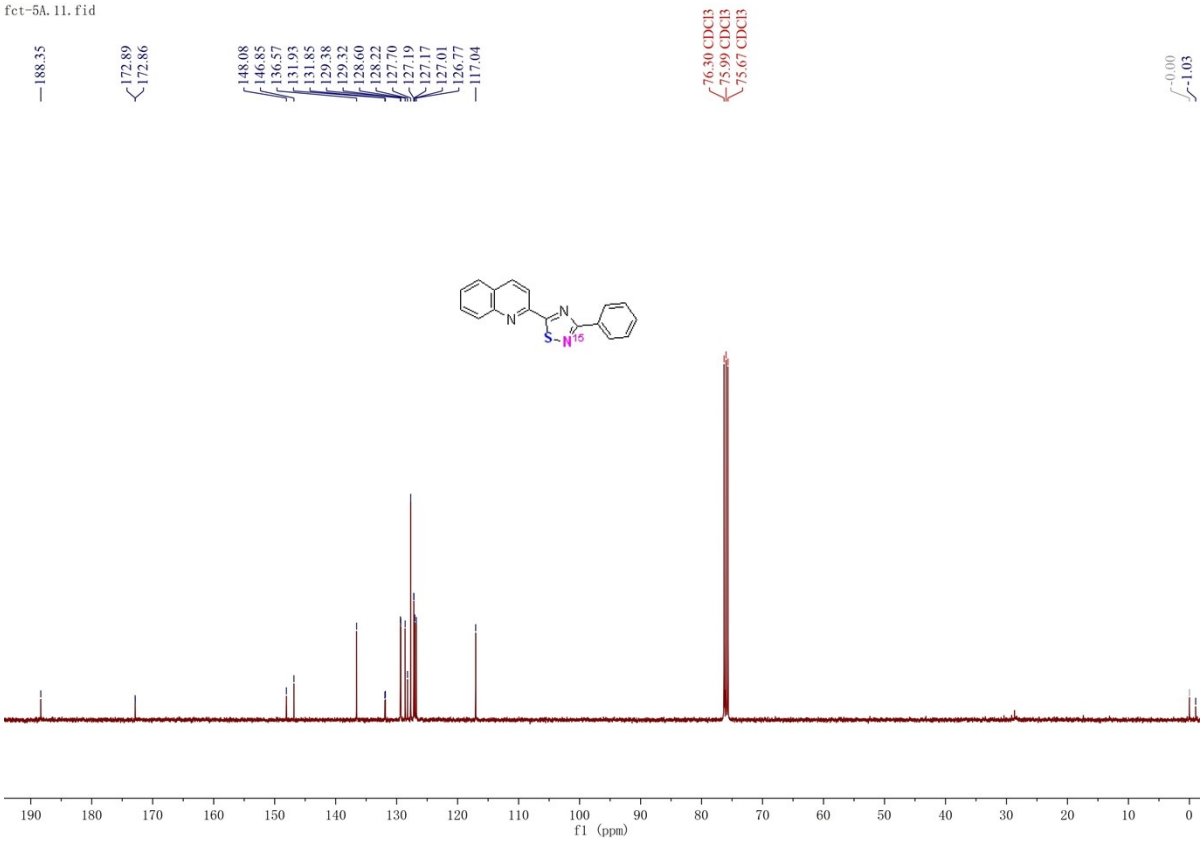
8.43
8.40
8.38
8.34
8.32
8.17
8.15
7.88
7.86
7.80
7.78
7.76
7.64
7.62
7.60
7.55
7.53
7.51
7.50
7.48
7.26 CDCl3
3.02
1.00
1.00
1.02
1.03
1.06
3.01
f1 (ppm)


20201127-NJ-2011258309.11.fid
189.4
173.9
173.9
149.1
147.9
137.6
133.0
132.9
130.4
130.4
129.7
129.3
128.8
128.3
128.2
128.1
127.8
118.1
77.4 CDCl3
77.1 CDCl3
76.7 CDCl3
f1 (ppm)


fct-5A.1.fid
8.44
8.44
8.43
8.43
8.42
8.42
8.41
8.40
8.37
8.34
8.20
8.18
7.90
7.90
7.88
7.88
7.82
7.81
7.80
7.79
7.79
7.78
7.77
7.65
7.65
7.63
7.63
7.63
7.61
7.61
7.55
7.55
7.55
7.54
7.53
7.53
7.52
7.51
7.51
7.50
7.50
7.48
7.48
7.48
7.48
7.26 CDCl3
0.08
0.01
N
S
N15
3.0
1.0
1.0
1.0
1.0
1.0
1.0
3.0
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
f1 (ppm)


fct-5A.11.fid
188.35
172.89
172.86
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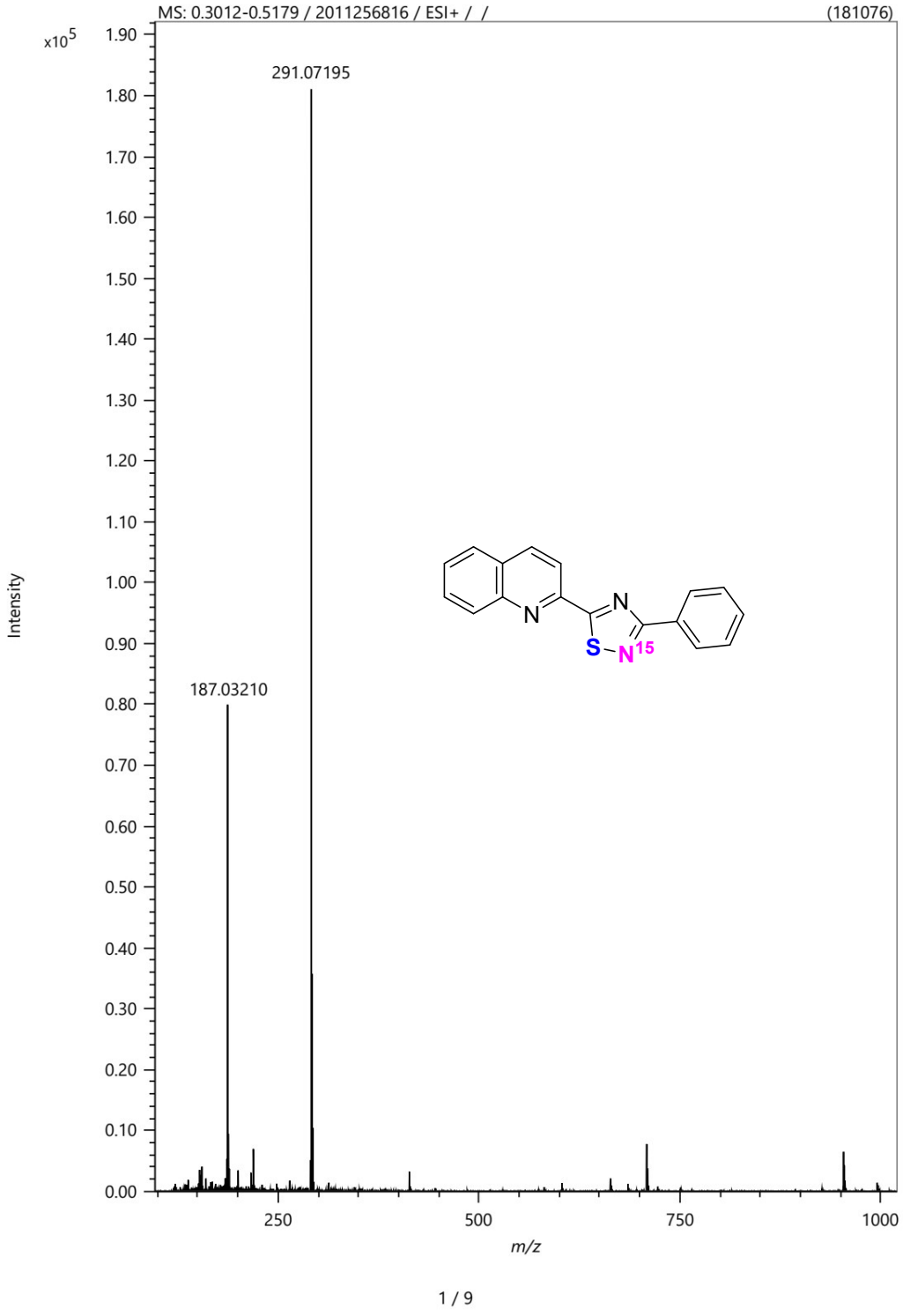
146.85
136.57
131.93
131.85
129.38
129.32
128.60
128.22
127.70
127.19
127.17
127.01
126.77
117.04
76.30 CDCl3
75.99 CDCl3
75.67 CDCl3
-0.00
-1.03
N
S
N15
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

6. HRMS data of ^{15}N-labeled 1,2,4-thiadiazole 4l



7. checkCIF/PLATON of 4a (CCDC 2077857)

checkCIF/PLATON report

You have not supplied any structure factors. As a result the full set of tests cannot be run.

THIS REPORT IS FOR GUIDANCE ONLY. IF USED AS PART OF A REVIEW PROCEDURE FOR PUBLICATION, IT SHOULD NOT REPLACE THE EXPERTISE OF AN EXPERIENCED CRYSTALLOGRAPHIC REFEREE.

No syntax errors found. CIF dictionary Interpreting this report

Datablock: 1

```
Bond precision:  C-C = 0.0034 A          Wavelength=0.71073

Cell:           a=12.8409(15)    b=15.0587(15)       c=12.4189(14)
                alpha=90         beta=106.043(4)     gamma=90
Temperature:    296 K

                Calculated                Reported
Volume          2307.9(4)                 2307.9(4)
Space group     P 21/c                    P 1 21/c 1
Hall group      -P 2ybc                   -P 2ybc
Moiety formula  C13 H9 N3 S               C13 H9 N3 S
Sum formula     C13 H9 N3 S               C13 H9 N3 S
Mr              239.29                    239.29
Dx,g cm-3       1.377                     1.377
Z               8                         8
Mu (mm-1)       0.259                     0.259
F000            992.0                     992.0
F000'           993.27
h,k,lmax        16,18,15                  16,18,15
Nref            4724                      4708
Tmin,Tmax                                 0.699,0.745
Tmin'

Correction method= # Reported T Limits: Tmin=0.699 Tmax=0.745
AbsCorr = MULTI-SCAN

Data completeness= 0.997          Theta(max)= 26.379

R(reflections)= 0.0450( 3173)     wR2(reflections)= 0.1420( 4708)

S = 1.086                 Npar= 307
```

The following ALERTS were generated. Each ALERT has the format
test-name_ALERT_alert-type_alert-level.
Click on the hyperlinks for more details of the test.

Alert level C

PLAT053_ALERT_1_C Minimum Crystal Dimension Missing (or Error) ... Please Check
PLAT054_ALERT_1_C Medium Crystal Dimension Missing (or Error) ... Please Check
PLAT055_ALERT_1_C Maximum Crystal Dimension Missing (or Error) ... Please Check

Alert level G

PLAT003_ALERT_2_G Number of Uiso or Uij Restrained non-H Atoms ... 5 Report
PLAT177_ALERT_4_G The CIF-Embedded .res File Contains DELU Records 3 Report
PLAT860_ALERT_3_G Number of Least-Squares Restraints ..... 3 Note

0 **ALERT level A** = Most likely a serious problem - resolve or explain
0 **ALERT level B** = A potentially serious problem, consider carefully
3 **ALERT level C** = Check. Ensure it is not caused by an omission or oversight
3 **ALERT level G** = General information/check it is not something unexpected

3 ALERT type 1 CIF construction/syntax error, inconsistent or missing data
1 ALERT type 2 Indicator that the structure model may be wrong or deficient
1 ALERT type 3 Indicator that the structure quality may be low
1 ALERT type 4 Improvement, methodology, query or suggestion
0 ALERT type 5 Informative message, check

It is advisable to attempt to resolve as many as possible of the alerts in all categories. Often the minor alerts point to easily fixed oversights, errors and omissions in your CIF or refinement strategy, so attention to these fine details can be worthwhile. In order to resolve some of the more serious problems it may be necessary to carry out additional measurements or structure refinements. However, the purpose of your study may justify the reported deviations and the more serious of these should normally be commented upon in the discussion or experimental section of a paper or in the "special_details" fields of the CIF. checkCIF was carefully designed to identify outliers and unusual parameters, but every test has its limitations and alerts that are not important in a particular case may appear. Conversely, the absence of alerts does not guarantee there are no aspects of the results needing attention. It is up to the individual to critically assess their own results and, if necessary, seek expert advice.

Publication of your CIF in IUCr journals

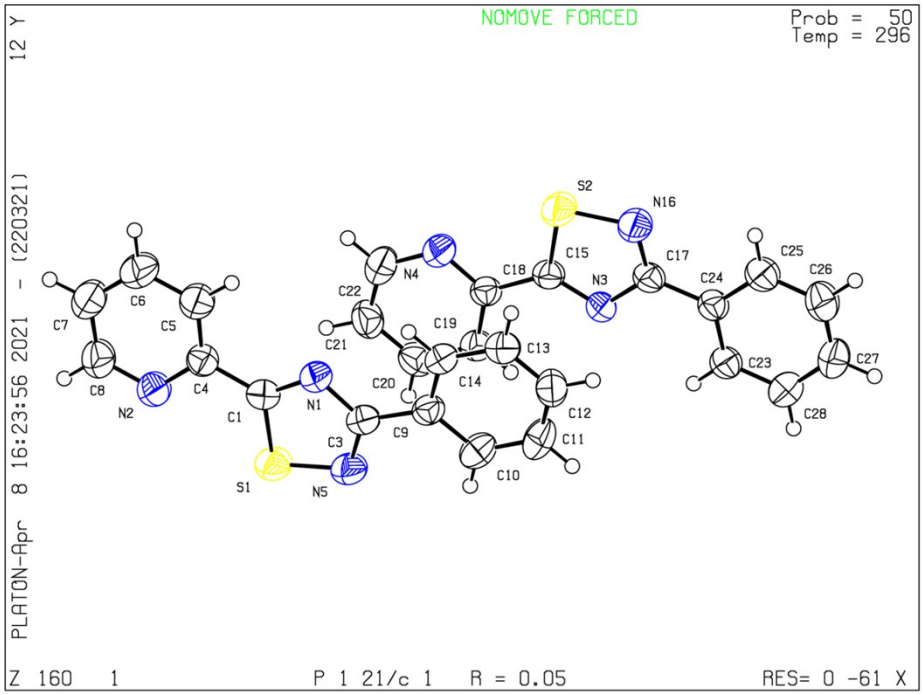
A basic structural check has been run on your CIF. These basic checks will be run on all CIFs submitted for publication in IUCr journals (*Acta Crystallographica*, *Journal of Applied Crystallography*, *Journal of Synchrotron Radiation*); however, if you intend to submit to *Acta Crystallographica Section C* or *E* or *IUCrData*, you should make sure that full publication checks are run on the final version of your CIF prior to submission.

Publication of your CIF in other journals

Please refer to the *Notes for Authors* of the relevant journal for any special instructions relating to CIF submission.

PLATON version of 22/03/2021; check.def file version of 19/03/2021

Datablock 1 - ellipsoid plot



8. ORTEP diagram of 4a (CCDC 2077857)