Electronic Supplementary Material (ESI) for Organic & Biomolecular Chemistry.

Supporting Information

Efficient, Versatile and Practical Palladium-Catalyzed Highly Regioselective *ortho*-Halogenation of Azoxybenzenes

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Contents:

1. General methods.

All reactions involving air- and moisture-sensitive reagents were carried out under a nitrogen atmosphere. Toluene, DMF, 1, 2-dichloroethane, DMSO, 1, 4- dioxane and CH_3CN were distilled from appropriate drying agents prior to use. All chemicals were purchased from Aldrich and used without further purification. Thin-layer chromatography (TLC) was performed using 60 mesh silica gel plates visualized with short-wavelength UV light (254 nm). Silica gel 60 (230~400 mesh) was used for column chromatography. ^{1}H NMR and ^{13}C NMR spectra were recorded on a Bruker INOVA-400. NMR Spectrums were recorded on a 400 instrument (400 MHz for ^{1}H and 100 MHz for ^{13}C). Chemical shifts (δ) were measured in ppm relative to TMS δ = 0 for ^{1}H, or to chloroform δ = 77.0 for ^{13}C as internal standard. Data are reported as follows: Chemical shift, multiplicity (s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet), Coupling constants, *J*, are reported in hertz. Mass data were measured with Thermo Scientific DSQ II mass spectrometer. Azoxybenzenes were prepared from arylamines, according to the literature[1].

General Catalytic Procedure for *Ortho*-Bromination of Azoxybenzenes with NBS.

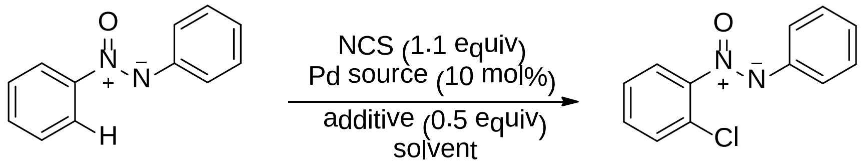
A mixture of azoxybenzene (39.6 mg, 0.2 mmol, 1.0 equiv), $TsOH·H_2O$ (19.0 mg, 0.1 mmol, 0.5 equiv), NBS (39.2 mg, 0.22 mmol, 1.1 equiv) and $Pd(OAc)_2$ (4.5 mg, 0.02 mmol, 10 mol %) in DCE (1 mL) was stirred under air at 40 ^{o}C for 12 h followed by cooling. The volatiles removed under reduced pressure. The contents were subjected to flash chromatography to give the corresponding product (97%) as a pale yellow oil. The purified material was dried under an oil-pump vacuum.

General Catalytic Procedure for *Ortho*-Chlorination/Iodination of Azoxybenzenes with NCS/NIS.

A mixture of azoxybenzene (39.6 mg, 0.2 mmol, 1.0 equiv), NCS/NIS (0.22 mmol, 1.1 equiv) and $Pd(OAc)_2$ (4.5 mg, 0.02 mmol, 10 mol %) in AcOH (1 mL) was stirred under air at 60 ^{o}C or 100 ^{o}C for 12 h as specified in Table 2 and Table 3 followed by cooling. The volatiles removed under reduced pressure. The contents were subjected to flash chromatography to give the corresponding products (96% and 95%) as pale yellow oil. The purified materials were dried under an oil-pump vacuum.

2. Detailed optimization studies for the *ortho*-chlorination/iodination of azoxybenzene with NCS/NIS.

Table S1 Optimization Studies for the *ortho*-chlorination of azoxybenzene with NCS.[a,b]



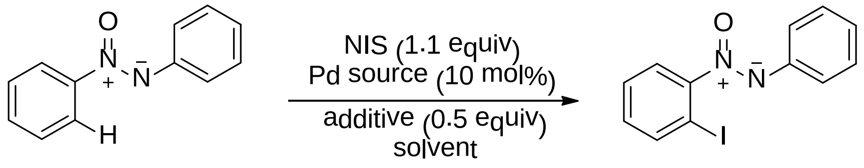
1a **3aa**

Entry	Catalyst	Additive	Solvent	Yield [%][b]
1	$Pd(OAc)_2$		AcOH	96%
2	$Pd(OAc)_2$		DCE	21%
3	$Pd(OAc)_2$		DME	64%
4	$Pd(OAc)_2$		Toluene	40%
5	$Pd(OAc)_2$		MeCN	15%
6	$Pd(OAc)_2$		DMSO	n.r.
7	$Pd(OAc)_2$		DMF	n.r.
8	$Pd(OAc)_2$		Dioxane	trace
9	$Pd(OAc)_2$	AcOH	DCE	55%
10	$Pd(OAc)_2$	$TsOH{\cdot}H_2O$	DCE	44%
11	$Pd(OAc)_2$	TFA	DCE	48%
12	$Pd(OAc)_2$	AcOH	DME	63%
13	$Pd(OAc)_2$	$TsOH{\cdot}H_2O$	DME	69%
14	$Pd(OAc)_2$	TFA	DME	57%
15	$Pd(OAc)_2$	AcOH	Toluene	42%
16	$Pd(OAc)_2$	$TsOH{\cdot}H_2O$	Toluene	35%
17	$Pd(OAc)_2$	TFA	Toluene	32%
18	$Pd(OAc)_2$	AcOH	MeCN	16%
19	$Pd(OAc)_2$	$TsOH{\cdot}H_2O$	MeCN	trace
20	$Pd(OAc)_2$	TFA	MeCN	trace
21	$Pd(OAc)_2$		AcOH	80%[c]
22	$Pd(OAc)_2$		AcOH	52%[d]
23	$Pd(TFA)_2$		AcOH	60%
24	$PdCl_2$		AcOH	59%
25	$PdCl_2(MeCN)_2$		AcOH	30%

[a] All the reactions were carried out in the presence of 0.2 mmol of **1a**, 0.22 mmol of NCS and 0.1 mmol of acid (if any) in 1.0 mL of solvents at 100 °C under air condition. [b] Isolated yields. [c]At 120 °C. [d]At 80 °C.

A mixture of azoxybenzene (39.6 mg, 0.2 mmol, 1.0 equiv), NCS (29.3 mg, 0.22 mmol, 1.1 equiv), Pd catalyst (0.02 mmol, 10 mol %) and acids (if any, 0.1 mmol, 0.5 equiv) in solvent (1 mL) was stirred under air at 100 °C for 12 h followed by cooling. The volatiles removed under reduced pressure. The contents were subjected to flash chromatography to give the corresponding product **3aa** as a pale yellow oil. The purified material was dried under an oil-pump vacuum.

Table S2 Optimization Studies for the *ortho*-iodination of azoxybenzene with NIS.[a,b]



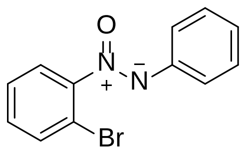
1a **4aa**

Entry	Catalyst	Additive	Solvent	Yield [%][b]
1	$Pd(OAc)_2$		AcOH	95%
2	$Pd(OAc)_2$		DCE	68%
3	$Pd(OAc)_2$		DME	44%
4	$Pd(OAc)_2$		Toluene	18%
5	$Pd(OAc)_2$		MeCN	trace
6	$Pd(OAc)_2$		DMSO	n.r.
7	$Pd(OAc)_2$		DMF	n.r.
8	$Pd(OAc)_2$		Dioxane	44%
9	$Pd(OAc)_2$	AcOH	DCE	68%
10	$Pd(OAc)_2$	$TsOH{\cdot}H_2O$	DCE	31%
11	$Pd(OAc)_2$	TFA	DCE	62%
12	$Pd(OAc)_2$	AcOH	DME	41%
13	$Pd(OAc)_2$	$TsOH{\cdot}H_2O$	DME	55%
14	$Pd(OAc)_2$	TFA	DME	58%
15	$Pd(OAc)_2$	AcOH	Toluene	18%
16	$Pd(OAc)_2$	$TsOH{\cdot}H_2O$	Toluene	trace
17	$Pd(OAc)_2$	TFA	Toluene	15%
18	$Pd(OAc)_2$	AcOH	Dioxane	48%
19	$Pd(OAc)_2$	$TsOH{\cdot}H_2O$	Dioxane	trace
20	$Pd(OAc)_2$	TFA	Dioxane	50%
21	$Pd(OAc)_2$		AcOH	86%[c]
22	$Pd(OAc)_2$		AcOH	44%[d]
23	$Pd(TFA)_2$		AcOH	72%
24	$PdCl_2$		AcOH	62%
25	$PdCl_2(MeCN)_2$		AcOH	60%

[a] All the reactions were carried out in the presence of 0.2 mmol of **1a**, 0.22 mmol of NIS and 0.1 mmol of acid (if any) in 1.0 mL of solvents at 60 °C under air condition. [b] Isolated yields. [c]At 80 °C. [d]At 40 °C.

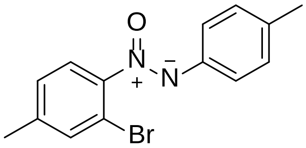
A mixture of azoxybenzene (49.5 mg, 0.2 mmol, 1.0 equiv), NIS (39.2 mg, 0.22 mmol, 1.1 equiv), Pd catalyst (0.02 mmol, 10 mol %) and acids (if any, 0.1 mmol, 0.5 equiv) in solvent (1 mL) was stirred under air at 100 °C for 12 h followed by cooling. The volatiles removed under reduced pressure. The contents were subjected to flash chromatography to give the corresponding product **4aa** as a pale yellow oil. The purified material was dried under an oil-pump vacuum.

3. Characterization of the Products

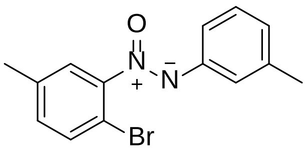


2aa: Pale yellow oil. ^1H NMR (400 MHz, $CDCl_3$) δ: 8.17 (d, *J* = 12.0 Hz, 2 H), 7.71-7.63 (m, 2

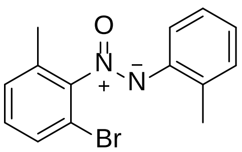
H), 7.52-7.42 (m, 4 H), 7.34 (t, *J* = 8.0 Hz, 1 H). ^{13}C NMR (100 MHz, $CDCl_3$) δ: 149.33, 143.66, 134.03, 130.86, 130.26, 128.77, 128.22, 125.40, 124.81, 115.11. HRMS (ESI) ($[M+Na]^+$) Calcd. for $C_{12}H_9BrN_2ONa^+$: 298.9796, Found $[M+Na]^+$: 298.9744.



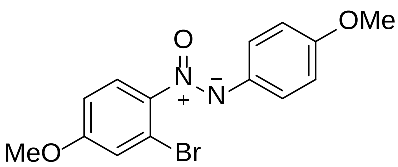
2ba: Pale yellow oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ: 8.10 (d, *J* = 8.0 Hz ,2 H), 7.55-7.43 (m, 2 H), 7.29 (d, *J* = 4 Hz ,2 H), 7.22 (d, *J* = 8.0 Hz ,1 H), 2.41 (d, *J* = 8.0 Hz, 6 H). ^{13}C NMR (100 MHz, $CDCl_3$) δ: 147.10, 141.52, 141.45, 140.86, 134.21, 129.32, 128.73, 125.52, 124.55, 114.75, 21.61, 20.93. HRMS (ESI) ($[M+Na]^+$) Calcd. for $C_{14}H_{13}BrN_2ONa^+$: 327.0103, Found $[M+Na]^+$: 326.9997.



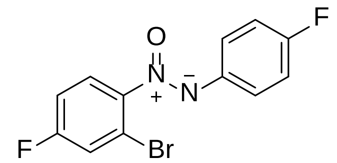
2ca: Deep red oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.95 (d, *J* = 8.0 Hz,2 H), 7.55 (d, *J* = 8.0 Hz, 1 H), 7.46 (d, *J* = 8.0 Hz, 1 H), 7.40-7.36 (m, 1 H), 7.24 (d, *J* = 8.0 Hz, 1 H), 7.15-7.13 (m, 1 H), 2.42 (s, 3 H) , 2.38 (s, 3 H). ^{13}C NMR (100 MHz, $CDCl_3$) δ: 149.11, 143.71, 138.79, 138.57, 133.61, 131.62, 130.98, 128.55, 125.80, 125.29, 122.43, 111.57, 21.43, 20.79. HRMS (ESI) ($[M+H]^+$) Calcd. for $C_{14}H_{14}BrN_2O^+$: 305.0290, Found $[M+H]^+$: 305.0208.



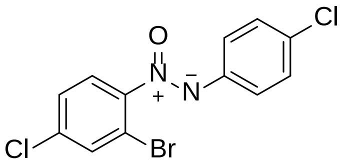
2da: Deep yellow oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ: 8.18-8.15 (m, 1 H), 7.53 (d, *J* = 4.0 Hz, 1 H), 7.34-7.19 (m, 5 H), 2.44 (s, 3 H), 2.42 (s, 3 H). ^{13}C NMR (100 MHz, $CDCl_3$) δ: 148.80, 141.96, 134.90, 132.67, 130.96, 130.87, 130.15, 129.95, 129.12, 125.90, 121.60, 115.26, 18.48, 17.19. HRMS (ESI) ($[M+Na]^+$) Calcd. for $C_{14}H_{13}BrN_2ONa^+$: 327.0103, Found $[M+Na]^+$: 327.0035.



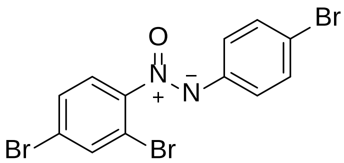
2ea: Pale yellow oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ: 8.28 (d, *J* = 8.0 Hz, 2 H), 7.62 (d, *J* = 12.0 Hz, 1 H), 7.19 (d, *J* = 4.0 Hz, 1 H), 6.98 (d, *J* = 12.0 Hz, 2 H), 6.94-6.92 (m, 1 H), 3.88 (s, 3 H), 3.85 (s, 3 H). ^{13}C NMR (100 MHz, $CDCl_3$) δ: 160.71, 160.30, 142.97, 137.76, 127.82, 125.91, 118.67, 115.97, 113.70, 113.65, 55.92, 55.51.HRMS (ESI) ($[M+Na]^+$) Calcd. for $C_{14}H_{13}BrN_2O_3Na^+$: 359.0002, Found $[M+Na]^+$: 358.9878.



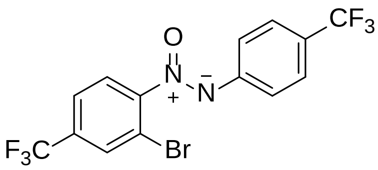
2fa: Pale yellow oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ: 8.25 (dd, *J* = 8.0 Hz, *J* = 12.0 Hz, 2 H), 7.68 (dd, *J* = 4.0 Hz, *J* = 8.0 Hz, 1 H), 7.45 (dd, *J* = 4.0 Hz, *J* = 8.0 Hz, 1 H), 7.17 (t, *J* = 8.0 Hz, 3 H). ^{13}C NMR (100 MHz, $CDCl_3$) δ: 162.92 (d, *J* = 252.0 Hz), 162.16 (d, *J* = 253.0 Hz), 145.82, 140.10(d, *J* = 3.0 Hz), 128.01(d, *J* = 10.0 Hz), 126.36(d, *J* = 9.0 Hz), 121.25(d, *J* = 26.0 Hz), 116.23 (d, *J* = 10.0 Hz), 115.76 (d, *J* = 23.0 Hz), 115.37 (d, *J* = 23.0 Hz). HRMS (ESI) ($[M+Na]^+$) Calcd. for $C_{12}H_7BrF_2N_2ONa^{+:}$ 334.9602, Found $[M+Na]^+$: 334.9737.



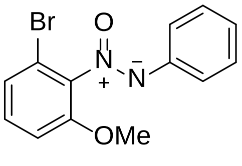
2ga: Pale yellow oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ: 8.14 (d, *J* = 8.0 Hz, 2 H), 7.72 (s, 1 H), 7.62 (d, *J* = 8.0 Hz, 1 H), 7.47-7.42 (m, 3 H). ^{13}C NMR (100 MHz, $CDCl_3$) δ: 147.68, 141.96, 136.40, 135.90, 133.73, 129.02, 128.42, 126.94, 125.79, 115.97. HRMS (ESI) ($[M+Na]^+$) Calcd. for $C_{12}H_7BrCl_2N_2ONa^+$: 366.9017, Found $[M+Na]^+$: 366.9335.



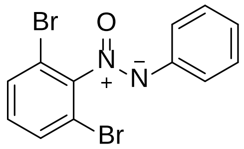
2ha: Pale yellow oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ: 8.06 (d, *J* = 8.0 Hz, 2 H), 7.88 (d, *J* = 4.0 Hz, 1 H), 7.63-7.58 (m, 3 H), 7.54 (d, *J* = 8.0 Hz, 1 H). ^{13}C NMR (100 MHz, $CDCl_3$) δ: 148.09, 142.28, 136.46, 132.02, 131.37, 127.07, 125.98, 124.29, 124.25, 116.13. HRMS (ESI) ($[M+Na]^+$) Calcd. for $C_{12}H_7Br_3N_2ONa^+$: 454.8001, Found $[M+Na]^+$: 454.7833.



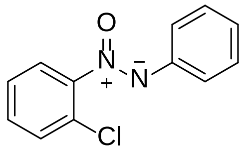
2ia: Pale yellow oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ: 8.22 (d, *J* = 12.0 Hz, 2 H), 8.01 (s, 1 H), 7.79-7.74 (m, 4 H). ^{13}C NMR (100 MHz, $CDCl_3$) δ: 151.06, 145.59, 133.23 (q, $J_{C\text{-}F}$ = 33.0 Hz), 131.62 (q, $J_{C\text{-}F}$ = 33.0 Hz), 131.49 (d, $J_{C\text{-}F}$ = 4.0 Hz), 126.00 (q, $J_{C\text{-}F}$ = 3.0 Hz), 125.57, 125.40, 122.96 (q, $J_{C\text{-}F}$ = 118.0 Hz), 122.95 (q, $J_{C\text{-}F}$ = 118.0 Hz), 115.86. HRMS (ESI) ($[M+Na]^+$) Calcd. for $C_{14}H_7BrF_6N_2ONa^+$: 434.9538, Found $[M+Na]^+$: 434.9381.



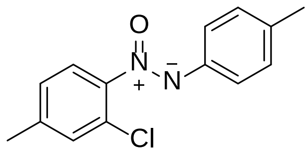
2ka: Pale yellow oil.^{1}HNMR (400 MHz, $CDCl_3$) δ: 8.15 (d, *J* = 8.0 Hz, 2 H), 7.51-7.42 (m, 3 H), 7.26-2.25 (m, 2 H), 7.00 (d, *J* = 8.0 Hz, 1 H). ^{13}C NMR (100 MHz, $CDCl_3$) δ: 152.88, 143.73, 130.59, 130.14, 128.70, 125.48, 125.38, 124.63, 116.46, 111.49, 56.52. HRMS (ESI) ($[M+Na]^+$) Calcd. for $C_{13}H_{11}BrN_2O_2Na^+$: 328.9902, Found $[M+Na]^+$:328.9902.



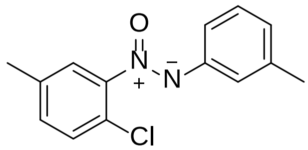
2la: White solid. ^{1}HNMR (400 MHz, $CDCl_3$) δ: 8.15 (d, *J* = 8.0 Hz, 2 H), 7.65 (d, *J* = 8.0 Hz, 2 H), 7.54-7.46 (m, 3 H), 7.20 (t, *J* = 8.0 Hz, 1 H). ^{13}C NMR (100 MHz, $CDCl_3$) δ :143.40, 132.59, 130.92, 130.57, 128.84, 125.42, 116.65. HRMS (ESI) ($[M+Na]^+$) Calcd. for $C_{12}H_8Br_2N_2ONa^+$: 376.8901, Found $[M+Na]^+$: 376.8903.



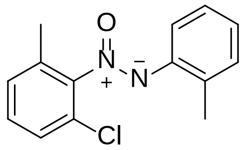
3aa: Deep red oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ: 8.15 (d, *J* = 8.0 Hz, 2 H), 7.68-7.65 (m, 1 H), 7.53-7.47 (m, 3 H), 7.43-7.38 (m, 3 H). ^{13}C NMR (100 MHz, $CDCl_3$) δ: 147.68, 143.70, 130.89, 130.67, 130.22, 128.74, 127.50, 126.74, 125.38, 124.79. HRMS (ESI) ($[M+Na]^+$) Calcd. for $C_{12}H_9ClN_2ONa^+$: 255.0301, Found $[M+Na]^+$: 255.0212.



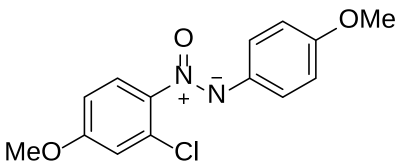
3ba: Pale yellow oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ: 8.10 (d, *J* = 8.0 Hz, 2 H), 7.56 (d, *J* = 8.0 Hz, 1 H), 7.32-7.25 (m, 3 H), 7. 17 (d, *J* = 8.0 Hz, 1 H), 2.41 (d, *J* = 4.0 Hz, 6 H). ^{13}C NMR (100 MHz, $CDCl_3$) δ: 145.54, 141.66, 141.31, 140.79, 131.15, 129.31, 128.04, 126.40, 125.53, 124.63, 21.57, 21.00. HRMS (ESI) ($[M+Na]^+$) Calcd. for $C_{14}H_{13}ClN_2ONa^+$: 283. 0614, Found $[M+Na]^+$: 283.0519.



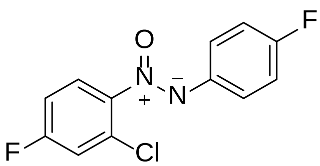
3ca: Deep red oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.97 (d, *J* = 8.0 Hz, 2 H), 7.48 (d, *J* = 4.0 Hz, 1 H), 7.40-7.37 (m, 2 H),7.26-7.21 (m, 2 H), 2.43 (s, 3 H), 2.40 (s, 3 H). ^{13}C NMR (100 MHz, $CDCl_3$) δ: 147.34, 143.65, 138.59, 138.07, 131.41, 131.05, 130.51, 128.56, 125.83, 125.15, 123.47, 122.46, 21.45, 20.78. HRMS (ESI) ($[M+Na]^+$) Calcd. for $C_{14}H_{13}ClN_2ONa^+$: 283.0614, Found $[M+Na]^+$: 283.0512.



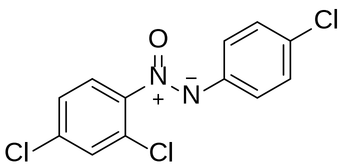
3da: Deep red oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ: 8.12-8.10 (d, *J* = 8.0 Hz, 1 H), 7.36-7.27 (m, 5 H), 7.23 (t, *J* = 8.0 Hz, 1 H), 2.43 (s, 3 H), 2.40 (s, 3 H). ^{13}C NMR (100 MHz, $CDCl_3$) δ: 147.27, 142.10, 134.69, 132.64, 130.86, 129.63, 129.46, 129.03, 127.83, 126.67, 125.94, 121.58, 18.40, 16.96. HRMS (ESI) ($[M+Na]^+$) Calcd. for $C_{14}H_{13}ClN_2ONa^+$: 283.0614, Found $[M+Na]^+$: 283.0611.



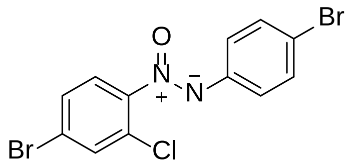
3ea: Pale yellow oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ: 8.29 (s, 1 H), 8.27 (s, 1 H), 7.64 (d, *J* = 12.0 Hz, 1 H), 7.00 (d, *J* = 4.0 Hz, 1 H), 6.99 (d, *J* = 0.0 Hz, 1 H), 6.97 (t, *J* = 4.0 Hz, 1 H), 6.89 (dd, *J* = 4.0 Hz, *J* = 8.0 Hz, 1 H), 3.88 (s, 3 H), 3.85 (s, 3 H). ^{13}C NMR (100 MHz, $CDCl_3$) δ: 160.70, 160.38, 141.31, 137.81, 127.83, 126.00, 115.63, 113.69, 113.04, 55.90, 55.50. HRMS (ESI) ($[M+Na]^+$) Calcd. for $C_{14}H_{13}ClN_2O_3Na^+$: 315.0512, Found $[M+Na]^+$: 315.0404.



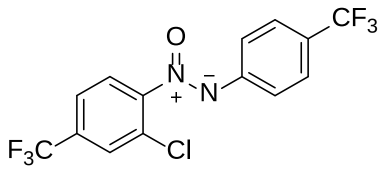
3fa: Pale yellow oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ: 8.27-8.24 (m, 2 H), 7.72-7.69 (dd, *J* = 4.0 Hz, *J* = 8.0 Hz, 1 H), 7.29-7.26 (m, 1 H), 7.20-7.15 (m, 2 H), 7.15-7.10 (m, 1 H). ^{13}C NMR (100 MHz, $CDCl_3$) δ: 162.90 (d, *J* = 252.0Hz), 162.20 (d, *J* = 253.0 Hz), 144.14, 140.10 (d, *J* = 3.0 Hz), 128.40 (d, *J* = 11.0 Hz), 128.03 (d, *J* = 8.0 Hz), 126.44 (d, *J* = 10.0 Hz), 118.26 (d, *J* = 25.0 Hz), 115.75 (d, *J* = 22.0 Hz), 114.80 (d, J = 22.0Hz). HRMS (ESI) ($[M+Na]^+$) Calcd. for $C_{12}H_7ClF_2N_2ONa^+$: 291.0113, Found $[M+Na]^+$: 291.0110.



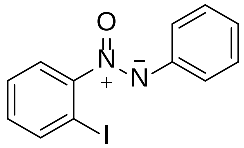
3ga: Pale yellow oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ: 8.14 (d, *J* = 12.0 Hz, 2 H), 7.65 (d, *J* = 8.0 Hz, 1 H), 7.56 (d, *J* = 4.0 Hz, 1 H), 7.46 (d, *J* = 8.0 Hz, 2 H), 7.40 (dd, *J* = 4.0 Hz, *J* = 8.0 Hz, 1 H). ^{13}C NMR (100 MHz, $CDCl_3$) δ: 146.00, 141.97, 136.36, 135.91, 130.80, 129.01, 127.92, 127.82, 126.96, 125.86. HRMS (ESI) ($[M+Na]^+$) Calcd. for $C_{12}H_7Cl_3N_2ONa^+$: 322.9522, Found $[M+Na]^+$: 322.9409.



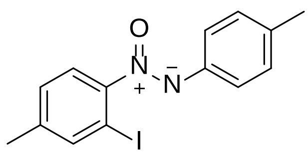
3ha: Pale yellow oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ: 8.07 (s, 1 H), 8.05 (s, 1H), 7.71 (s, 1 H), 7.63-7.62 (m, 1 H), 7.61-7.59 (m, 1 H), 7.57-7.55 (m, 2 H). ^{13}C NMR (100 MHz, $CDCl_3$) δ: ^{13}C NMR (101 MHz, CDCl3) δ: 146.46, 142.33, 133.61, 132.02, 130.76, 127.99, 127.10, 126.03, 124.30, 124.14. HRMS (ESI) ($[M+Na]^+$) Calcd. for $C_{12}H_7Br_2N_2ONa^+$: 410.8511, Found $[M+Na]^+$: 410.8362.



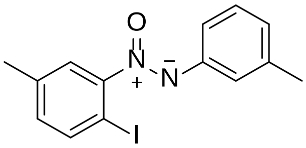
3ia: Pale yellow oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ: 8.22 (d, J = 8.0 Hz, 2 H), 7.84 (d, J = 8.0 Hz, 2 H), 7.77 (d, J = 8.0 Hz, 2 H), 7.72 (dd, J = 4.0 Hz, J = 8.0 Hz, 1 H). ^{13}C NMR (100 MHz, $CDCl_3$) δ: 149.38, 145.61, 133.24 (q, $J_{C\text{-}F}$ = 33.0 Hz), 131.67 (q, $J_{C\text{-}F}$ = 33.0 Hz), 128.47 (q, $J_{C\text{-}F}$ = 4.0 Hz), 127.87, 126.02 (q, $J_{C\text{-}F}$ = 4.0 Hz), 125.61, 125.53, 124.84 (q, $J_{C\text{-}F}$ = 3.0 Hz), 123.02 (q, $J_{C\text{-}F}$ = 105.0 Hz), 123.01 (q, $J_{C\text{-}F}$ = 106.0 Hz). HRMS (ESI) ($[M+Na]^+$) Calcd. for $C_{14}H_7ClF_6N_2ONa^+$: 391.0049, Found $[M+Na]^+$: 390.9896.



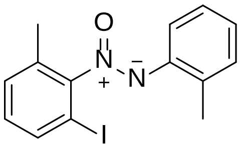
4aa: Pale yellow oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ: 8.17 (t, J = 8.0 Hz, 2 H), 7.94 (d, J = 8.0 Hz, 1 H), 7.65-7.63 (m, 1 H), 7.53-7.42 (m, J = 8.0 Hz, 4 H), 7.21-7.17 (m, 1 H). ^{13}C NMR (100 MHz, $CDCl_3$) δ: 152.60, 143.58, 140.42, 130.99, 130.26, 129.07, 128.77, 125.39, 124.32, 87.97. HRMS (ESI) ($[M+Na]^+$) Calcd. for $C_{12}H_9IN_2ONa^+$: 346.9657, Found $[M+Na]^+$: 346.9650.



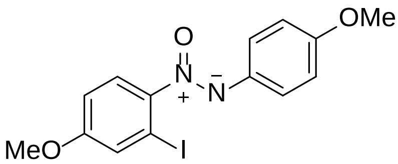
4ba: Pale yellow oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ: 8.11 (d, J = 8.0 Hz, 2 H), 7.77 (s, 1 H), 7.52 (d, J = 8.0 Hz, 1 H), 7.30 (d, J = 8.0 Hz, 2 H), 7.26 (d, J = 4.0 Hz, 1 H), 2.43 (s, 3 H), 2.38 (s, 3 H). ^{13}C NMR (100 MHz, $CDCl_3$) δ: 150.44, 141.45, 140.79, 140.64, 140.09, 129.56, 129.30, 125.50, 124.02, 87.84, 21.60, 20.67. HRMS (ESI) ($[M+Na]^+$) Calcd. for $C_{14}H_{13}IN_2ONa^+$: 374.9970, Found $[M+Na]^+$: 374.9964.



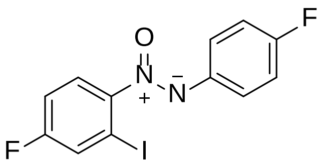
4ca: Pale yellow oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.97 (d, *J* = 4.0 Hz, 2 H), 7.78 (d, *J* = 8.0 Hz, 1 H), 7.44 (d, *J* = 0.0 Hz, 1 H), 7.39 (t, *J* = 8.0 Hz, 1 H), 7.25 (d, *J* = 4.0 Hz, 1 H), 7.00-6.98 (m, 1H), 2.43 (s, 3 H), 2.38 (s, 3 H). ^{13}C NMR (100 MHz, $CDCl_3$) δ: 152.47, 143.58, 139.97, 139.71, 138.55, 131.91, 130.99, 128.53, 125.77, 124.93, 122.43, 83.74, 21.46, 20.82. HRMS (ESI) ($[M+Na]^+$) Calcd. for $C_{14}H_{13}IN_2ONa^+$: 374.9970, Found $[M+Na]^+$: 374.9963.



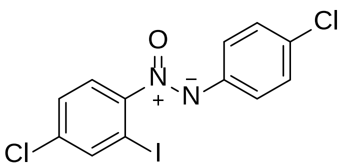
4da: Pale yellow oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ: 8.28 (dd, *J* = 4.0 Hz, *J* = 8.0 Hz, 1 H), 7.72 (d, *J* = 8.0 Hz, 1 H), 7.31-7.23 (m, 4 H), 7.06-7.01 (m, 1 H), 2.43 (d, *J* = 4.0 Hz, 6 H). ^{13}C NMR (100 MHz, CDCl3) δ : 152.23, 141.82, 137.32, 135.18, 132.02, 131.05, 130.84, 130.20, 129.20, 125.81, 121.67, 88.49, 18.60, 17.54. HRMS (ESI) ($[M+Na]^+$) Calcd. for $C_{14}H_{13}IN_2ONa^+$: 374.9970,Found $[M+Na]^+$: 374.9963.



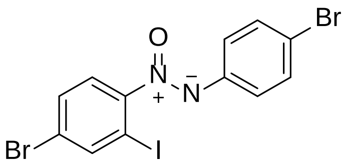
4ea: Pale yellow oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ: 8.30 (t, *J* = 4.0 Hz, 1 H), 8.27 (t, *J* = 4.0 Hz, 1 H), 7.59 (d, *J* = 8.0 Hz, 1 H), 7.43 (d, *J* = 8.0 Hz, 1 H), 7.01-6.99 (m, 1 H), 6.98-6.94 (m, 2 H), 3.86 (s, 3 H), 3.84 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ: 160.67, 160.07, 146.25, 137.67, 127.78, 125.27, 125.09, 114.35, 113.69, 88.60, 55.86, 55.49. HRMS (ESI) ($[M+Na]^+$) Calcd. for $C_{14}H_{13}IN_2O_3Na^+$: 406.9869, Found $[M+Na]^+$: 406.9860.



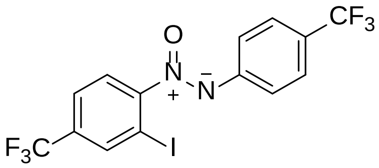
4fa: Pale yellow oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ: 8.27-8.23 (m, 2 H), 7.68-7.63 (m, 2 H), 7.22-7.16 (m, 3 H). ^{13}C NMR (100 MHz, $CDCl_3$) δ: 162.82 (d, *J* = 252.0Hz), 161.79 (d, *J* = 257.0 Hz), 149.00, 139.96 (d, *J* = 3.0 Hz), 127.95 (d, *J* = 8.0 Hz), 127.32 (d, *J* = 25.0 Hz), 125.68 (d, *J* = 10.0 Hz), 116.04 (d, *J* = 23.0 Hz), 115.72 (d, *J* = 23.0 Hz), 88.35 (d, J = 9.0Hz). HRMS (ESI) ($[M+Na]^+$) Calcd. for $C_{12}H_7F_2IN_2ONa^+$: 382.9464, Found $[M+Na]^+$: 382.9326.



4ga: Pale yellow oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ: 8.15 (d, J = 8.0 Hz, 2 H), 7.96 (d, J = 2.0 Hz, 1 H), 7.59 (d, J = 8.0 Hz, 1 H), 7.47 (d, J = 8.0 Hz, 3 H). ^{13}C NMR (100 MHz, $CDCl_3$) δ: 151.01, 141.94, 139.93, 136.33, 135.89, 129.19, 129.03, 126.94, 125.19, 88.46. HRMS (ESI) ([M+Na]$^+$) Calcd. for $C_{12}H_7Cl_2IN_2ONa^+$: 414.8878, Found [M+Na]$^+$: 414.8710.

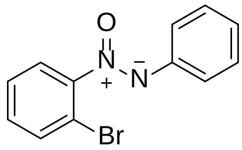


4ha: Pale yellow oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ: 8.11 (s, 1 H), 8.06 (d, J = 8.0 Hz, 2 H), 7.63-7.60 (m, 3 H), 7.52 (d, J = 8.0 Hz, 1 H). ^{13}C NMR (100 MHz, $CDCl_3$) δ: 151.40, 142.56, 142.24, 132.14, 132.02, 127.07, 125.43, 124.38, 124.27, 88.87. HRMS (ESI) ([M+Na]$^+$) Calcd. for $C_{12}H_7Br_2IN_2ONa^+$: 502.7867, Found [M+Na]$^+$: 502.7859.



4ia: Pale yellow oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ: 8.22 (d, J = 8.0 Hz, 3 H), 7.80-7.75 (m, 4 H). ^{13}C NMR (100 MHz, $CDCl_3$) δ: 154.44, 145.55, 137.78 (d, $J_{C\text{-}F}$ = 4.0 Hz), 133.12 (q, $J_{C\text{-}F}$ = 34.0 Hz), 131.62 (q, $J_{C\text{-}F}$ = 32.0 Hz), 126.42 (q, $J_{C\text{-}F}$ = 3.0 Hz), 126.03 (q, $J_{C\text{-}F}$ = 4.0 Hz), 125.58, 124.72, 122.84 (q, $J_{C\text{-}F}$ = 140.0 Hz), 122.83 (q, $J_{C\text{-}F}$ = 142.0 Hz), 88.20. HRMS (ESI) ([M+Na]$^+$) Calcd. for $C_{14}H_7F_6IN_2ONa^+$: 482.9405, Found [M+Na]$^+$: 482.9388.

4. NMR Charts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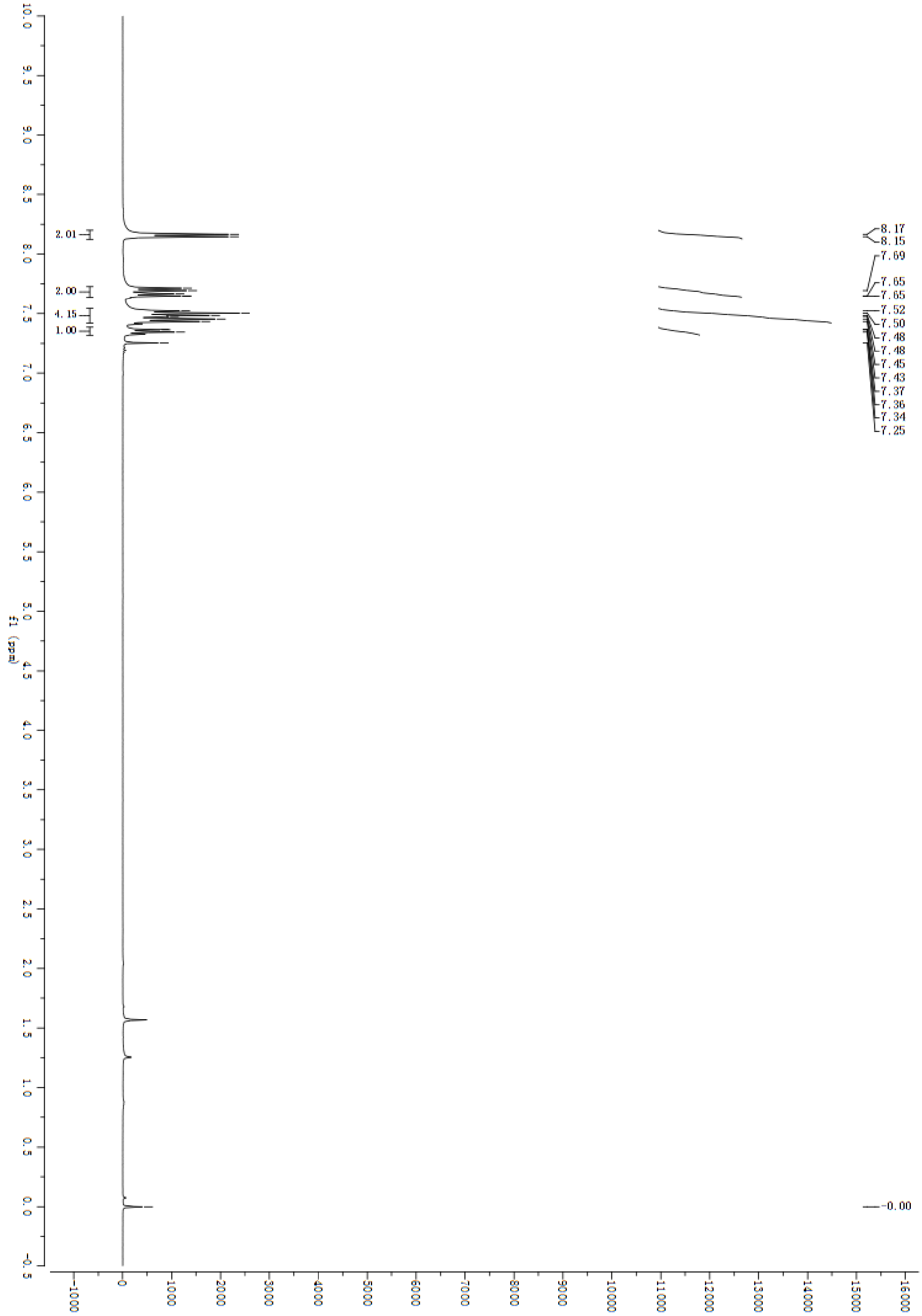
N
+
N
Br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7.48
7.48
7.45
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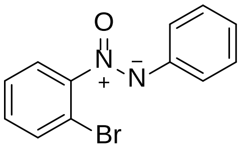
4.15
1.00
f1 (ppm)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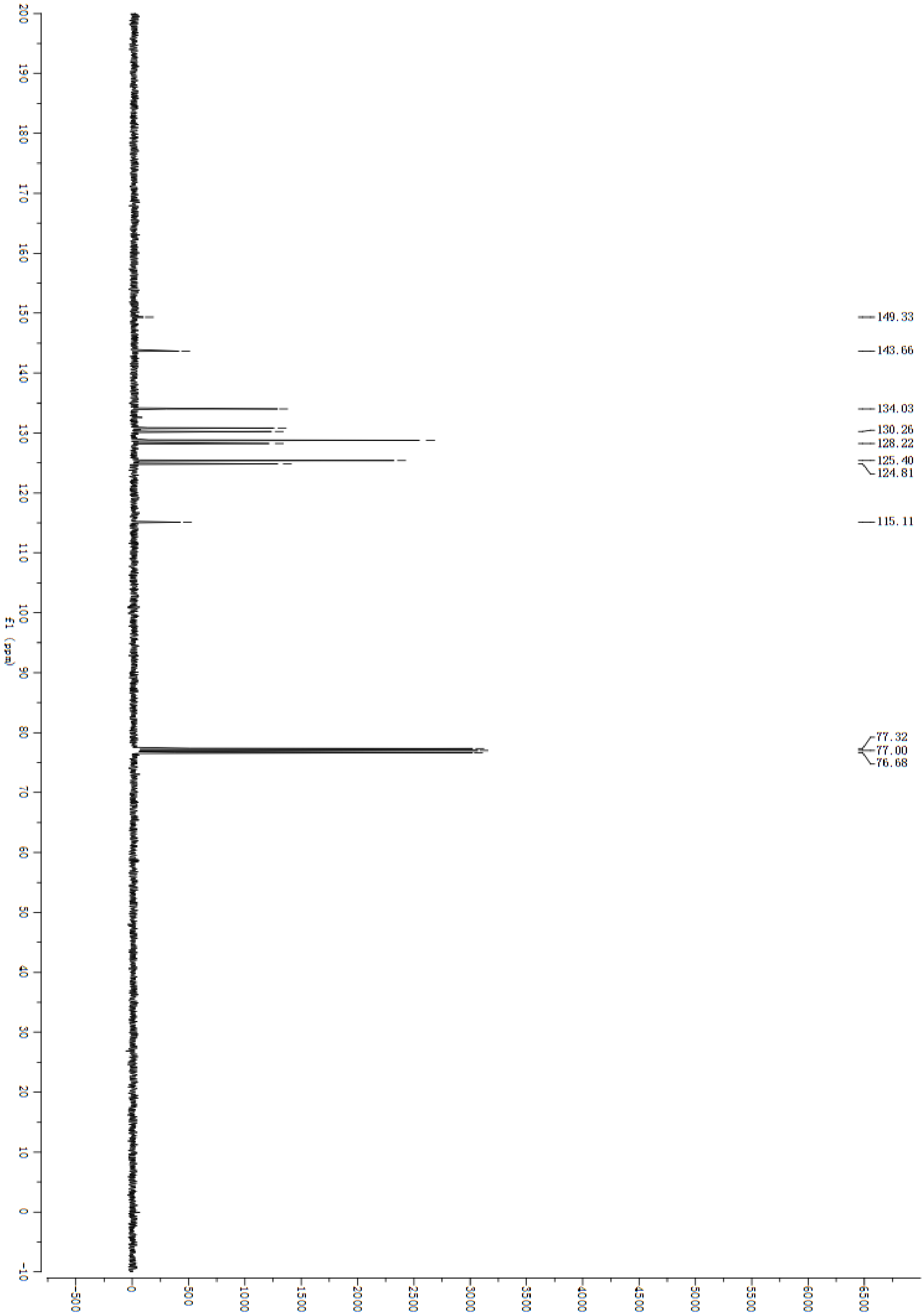
N
+
N
Br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125.40
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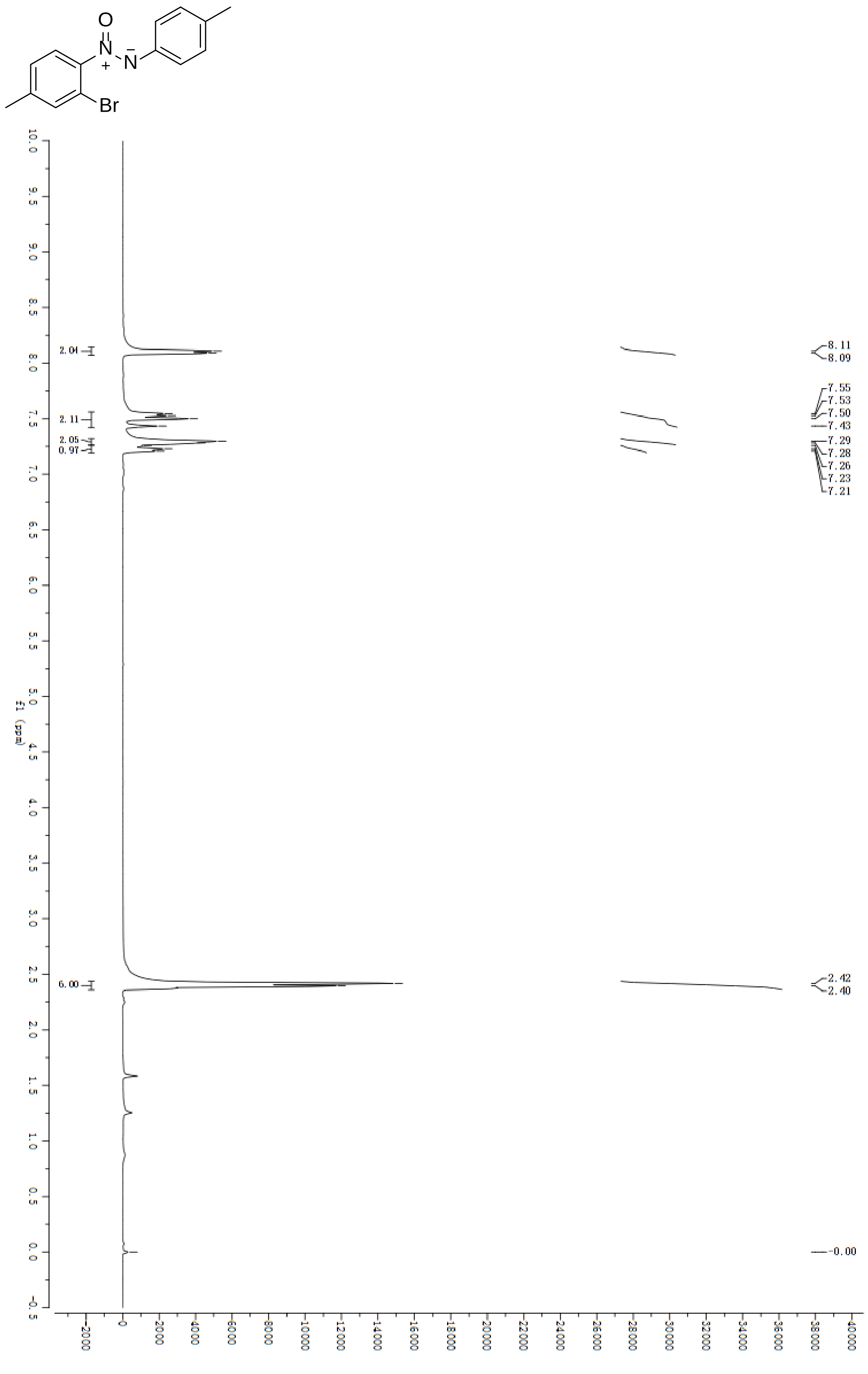
77.00
76.68
f1 (ppm)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+
N
Br
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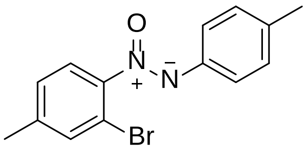
f1 (ppm)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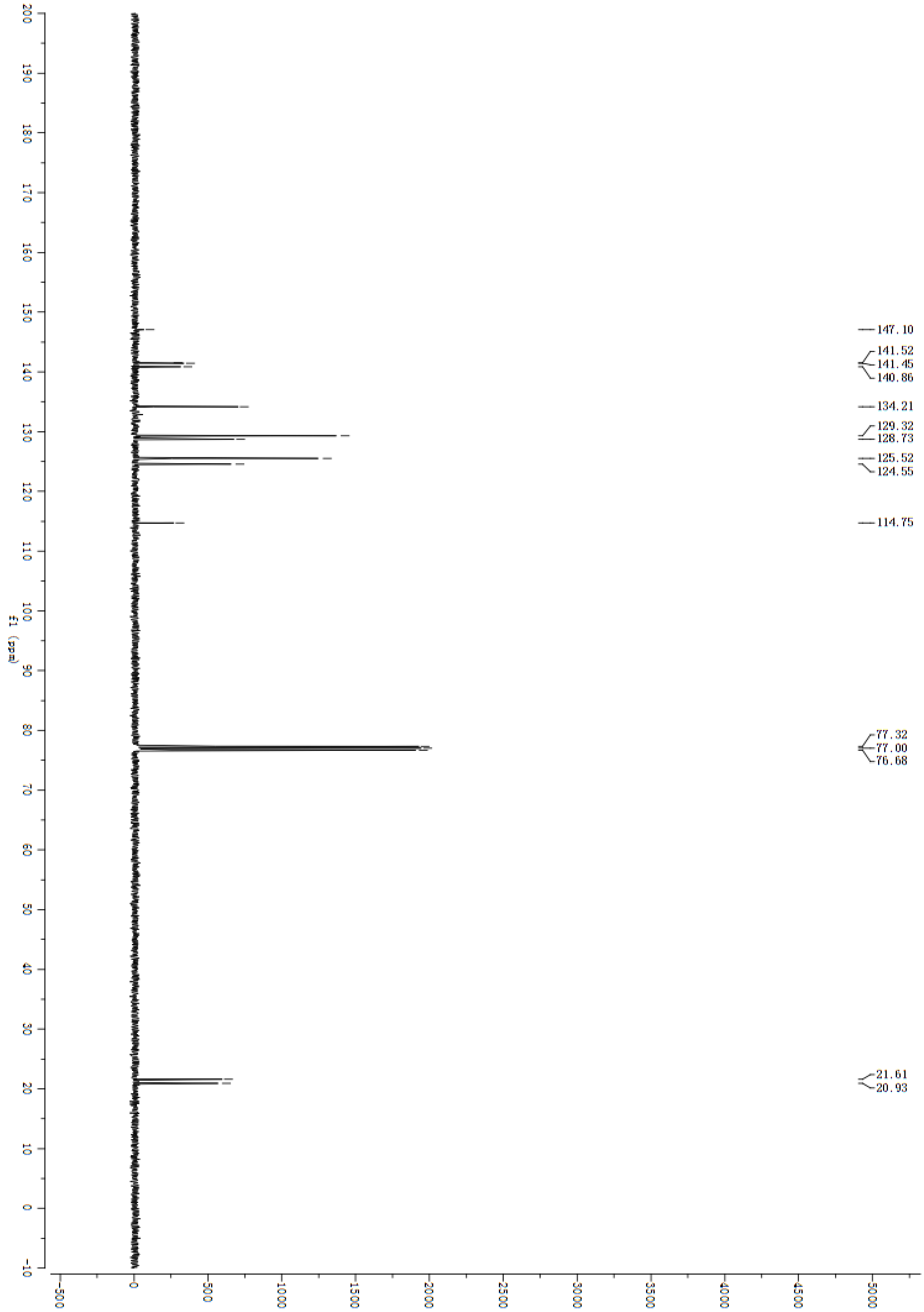
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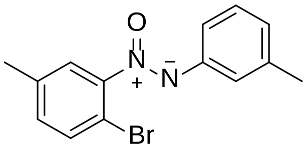
20.93
f1 (ppm)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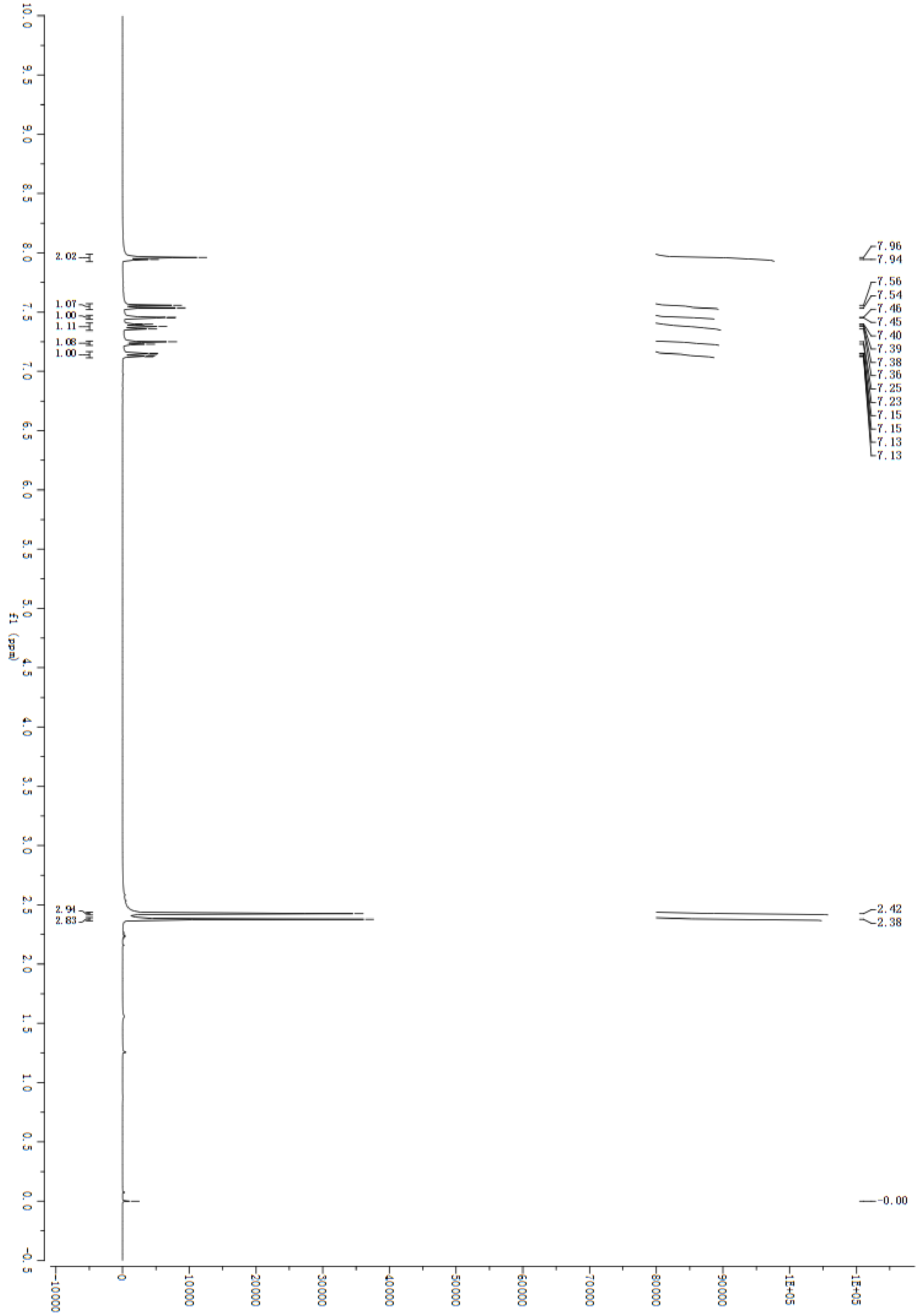
Br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7.13
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1.08
1.00
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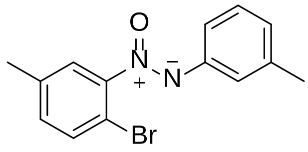
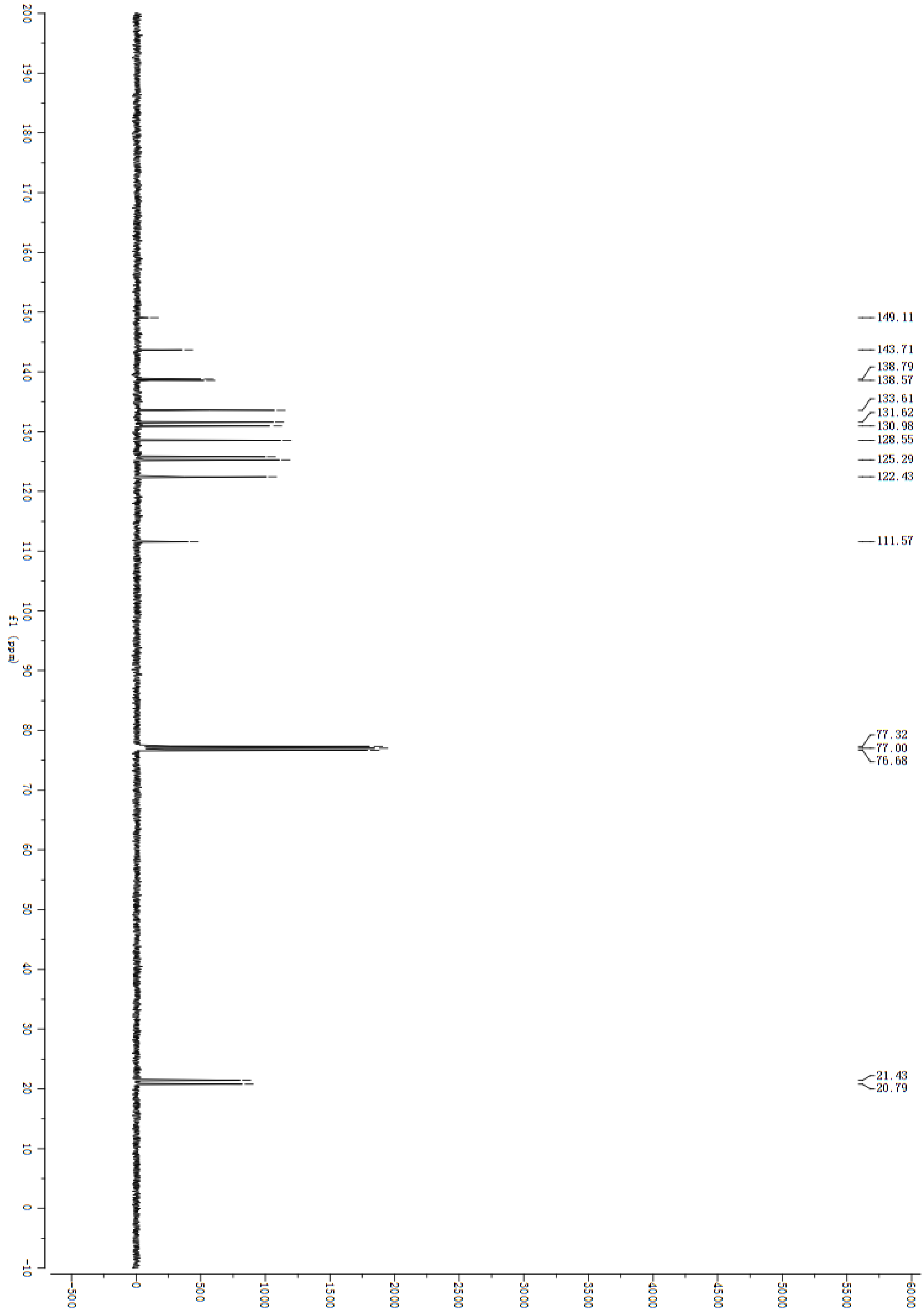
2.83
f1 (ppm)

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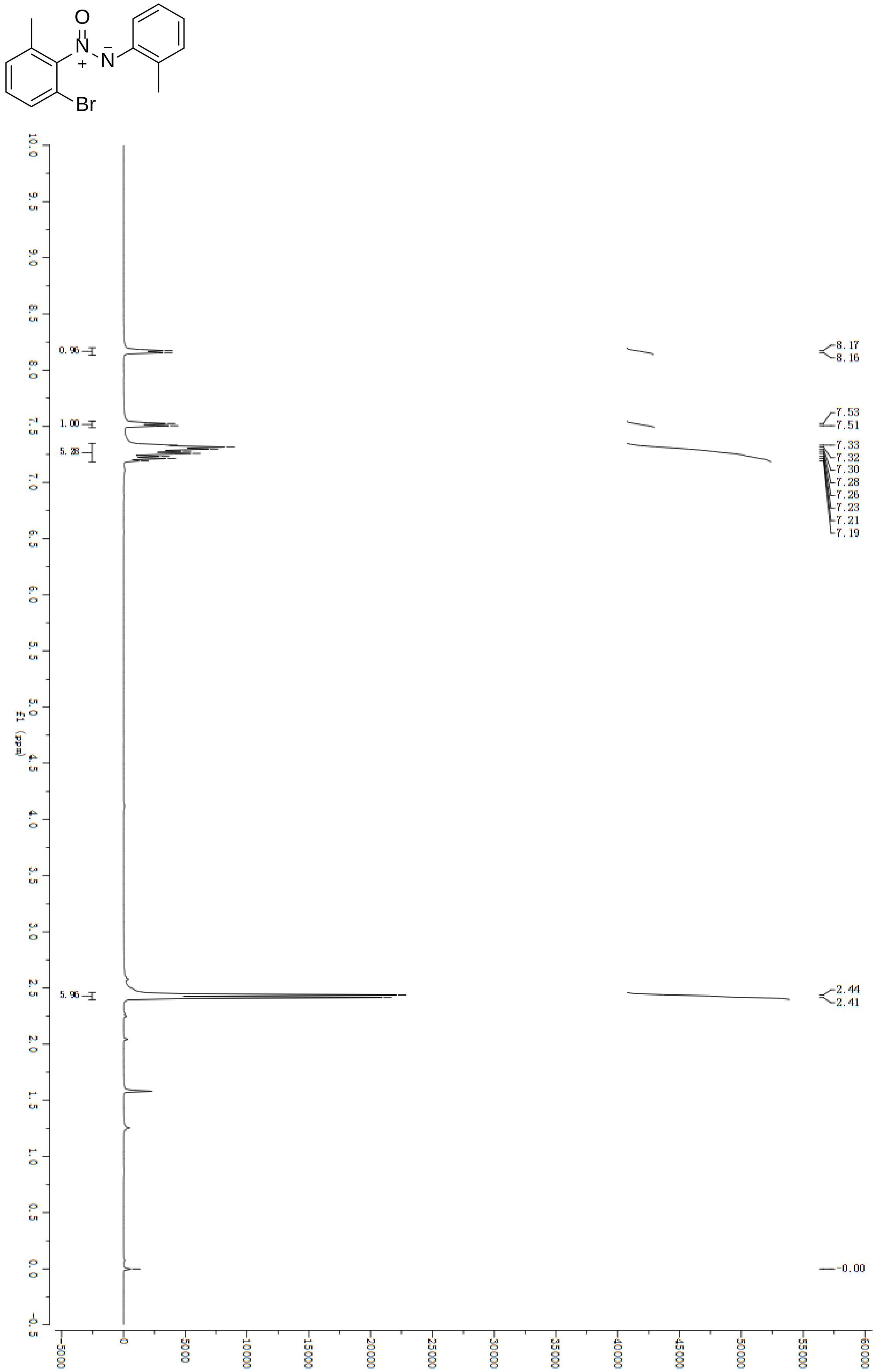
20.79
f1 (ppm)


O
N
+
N
Br
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7.32
7.30
7.28
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7.19
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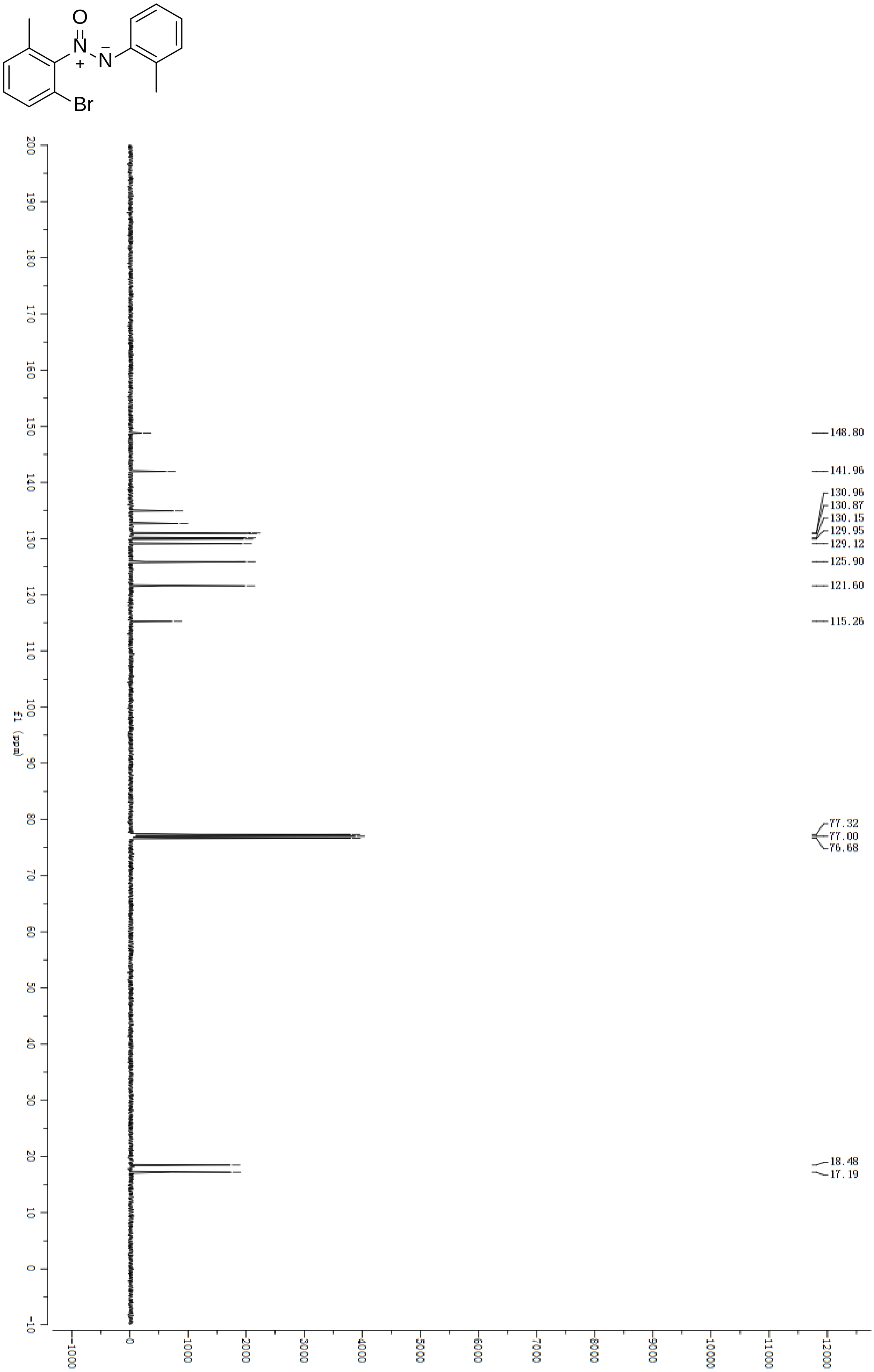
1.00
5.28
5.96
f1 (ppm)



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N
Br
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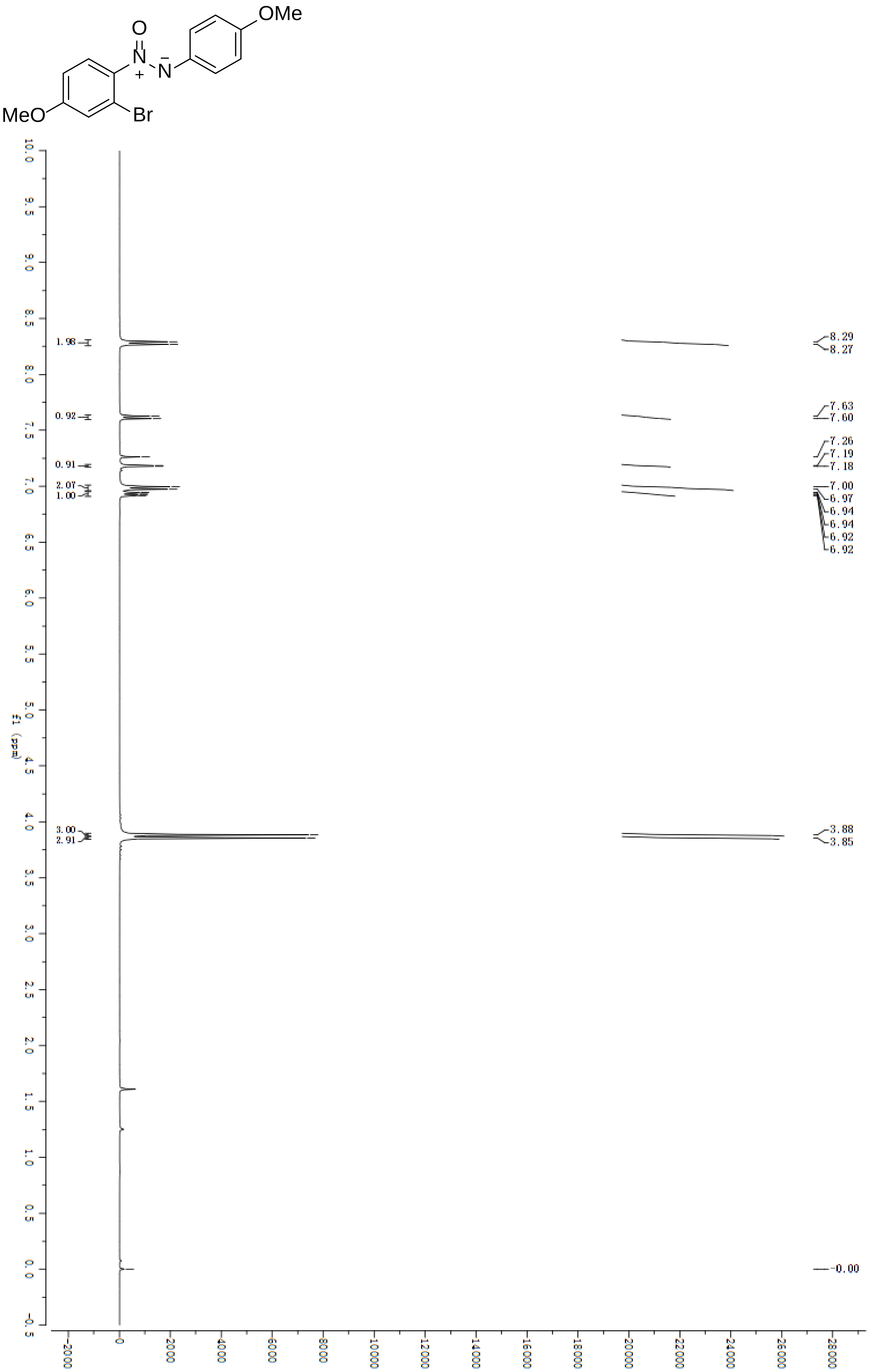
17.19
f1 (ppm)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N
MeO
Br
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7.00
6.97
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6.94
6.92
6.92
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0.91
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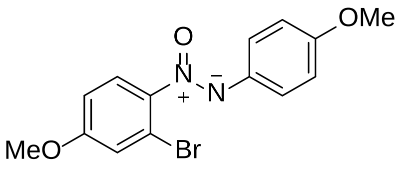
3.00
2.91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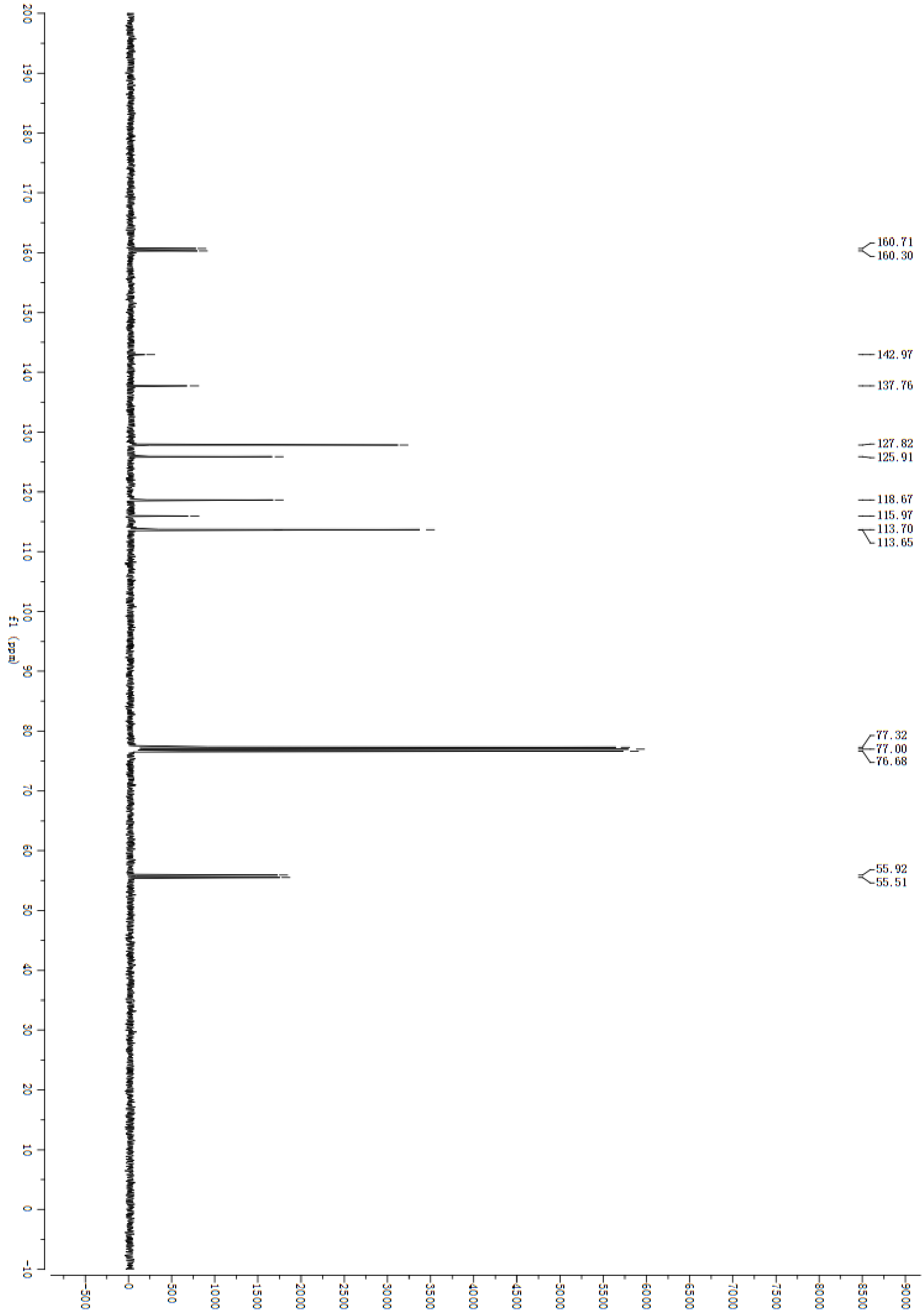
N
N
MeO
Br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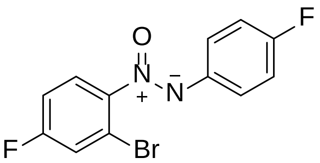
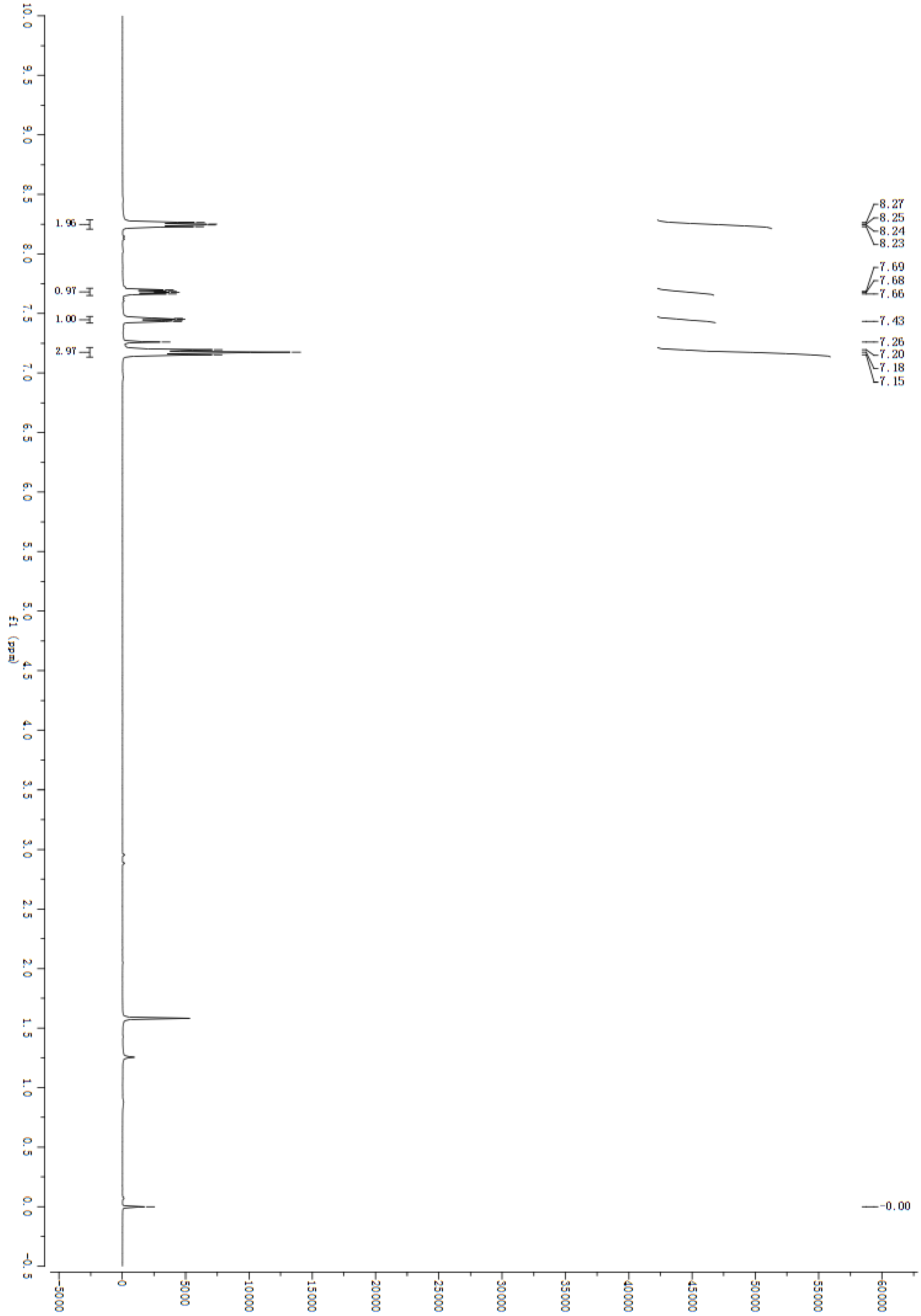
55.51
f1 (ppm)


O
N
N
F
F
Br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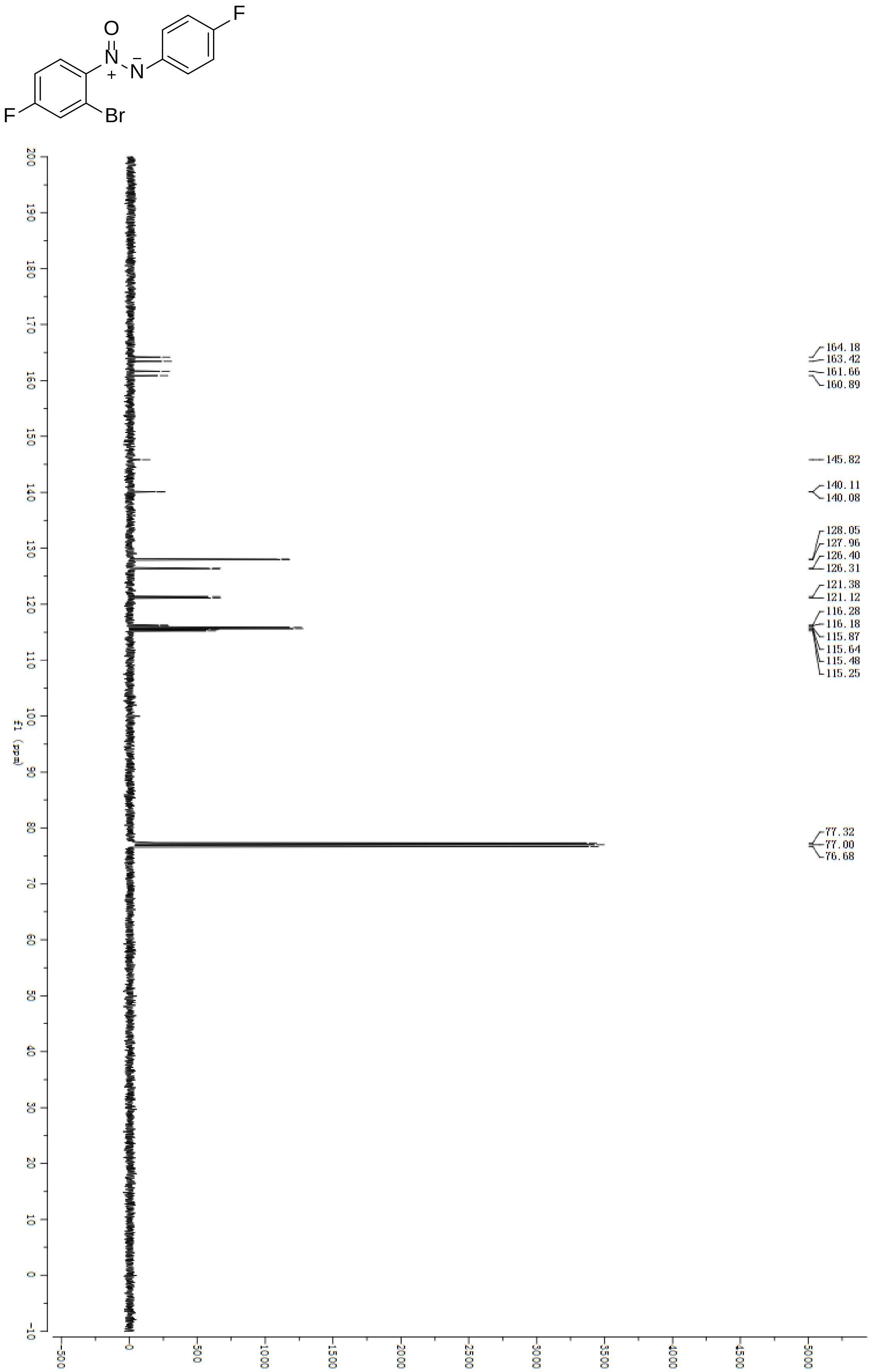
1.96
0.97
1.00
2.97
f1 (ppm)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O
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N
F
Br
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161.66
160.89
145.82
140.11
140.08
128.05
127.96
126.40
126.31
121.38
121.12
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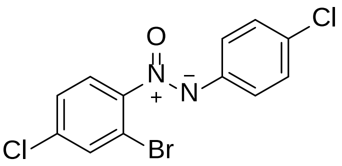
116.18
115.87
115.64
115.48
115.25
77.32
77.00
76.68
f1 (ppm)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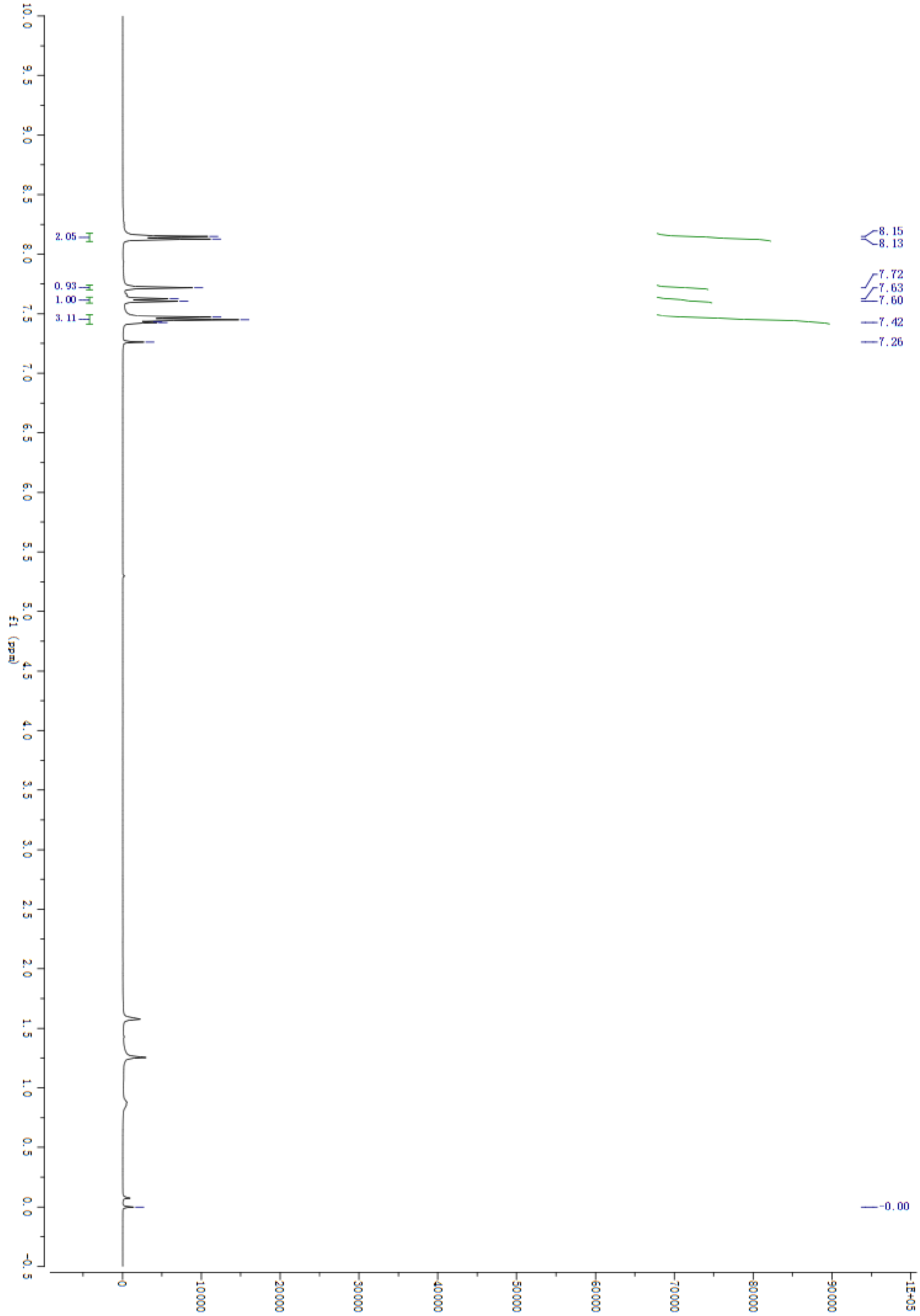
+
N
Cl
Cl
Br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7.42
7.26
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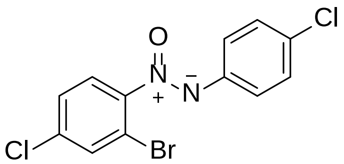
f1 (ppm)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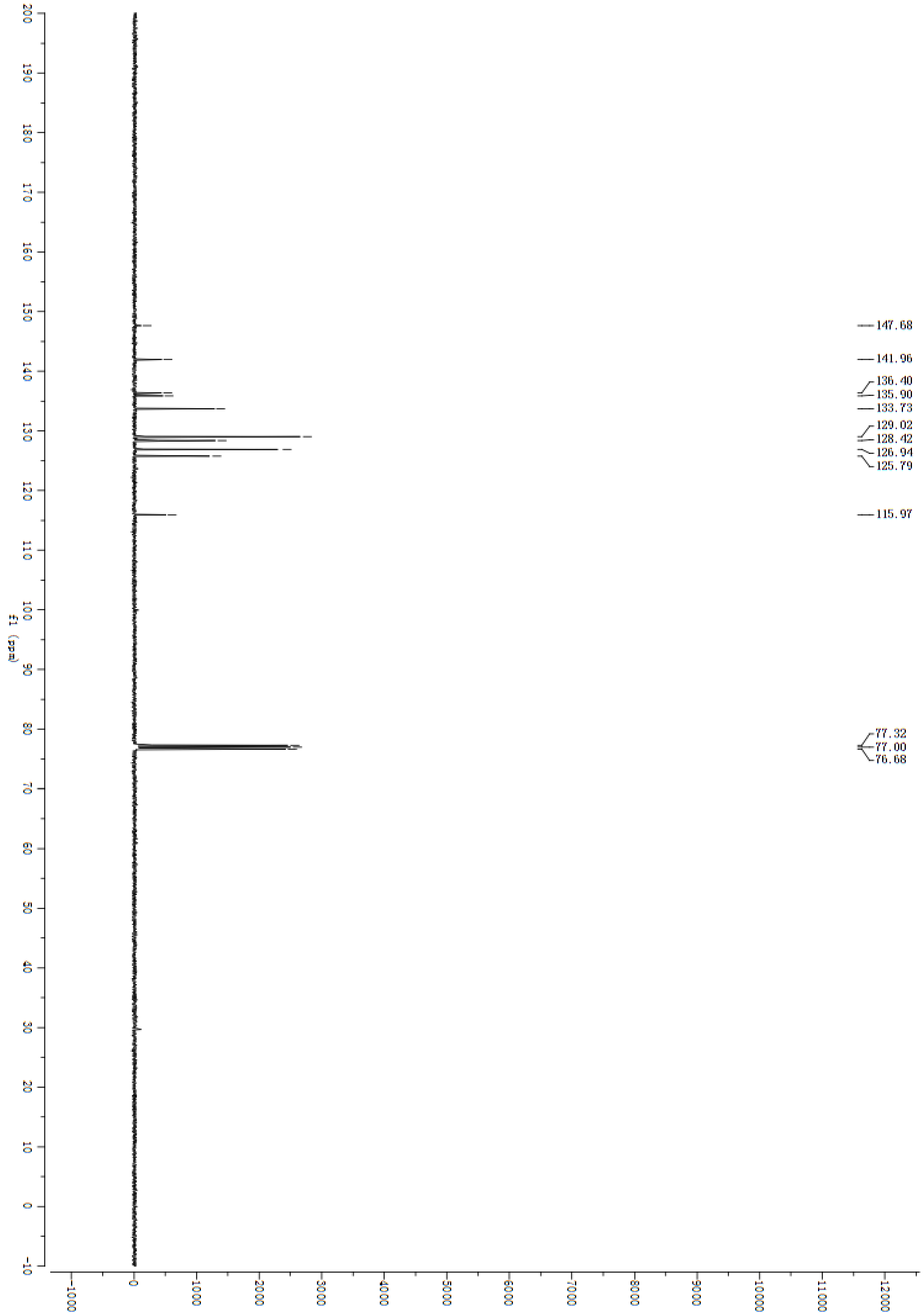
N
+
N
Cl
Cl
Br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126.94
125.79
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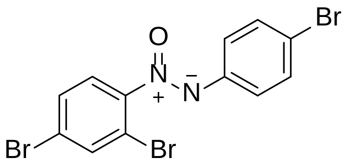
77.32
77.00
76.68
f1 (ppm)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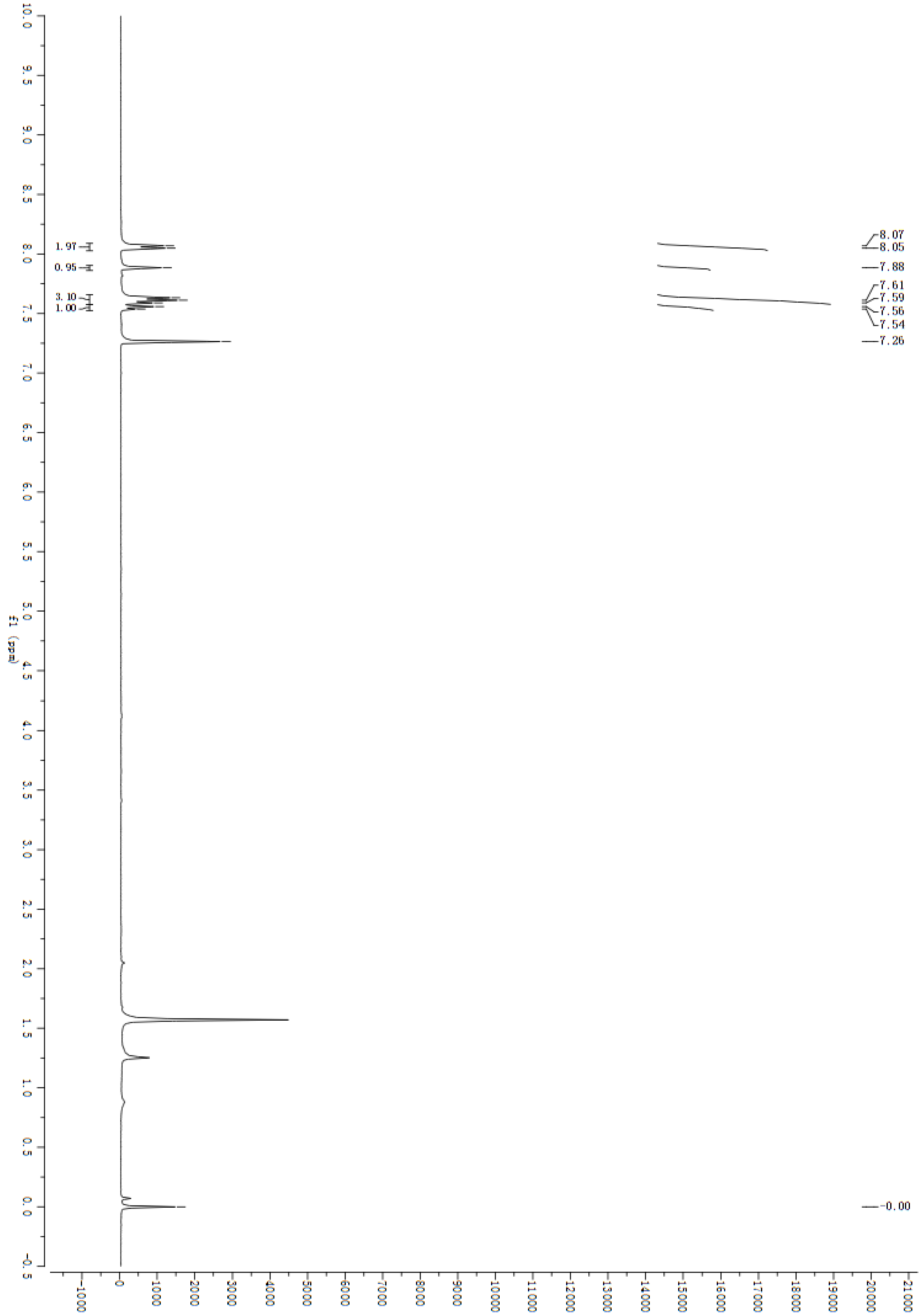
N
N
Br
Br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7.54
7.26
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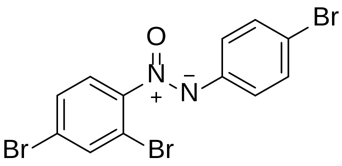
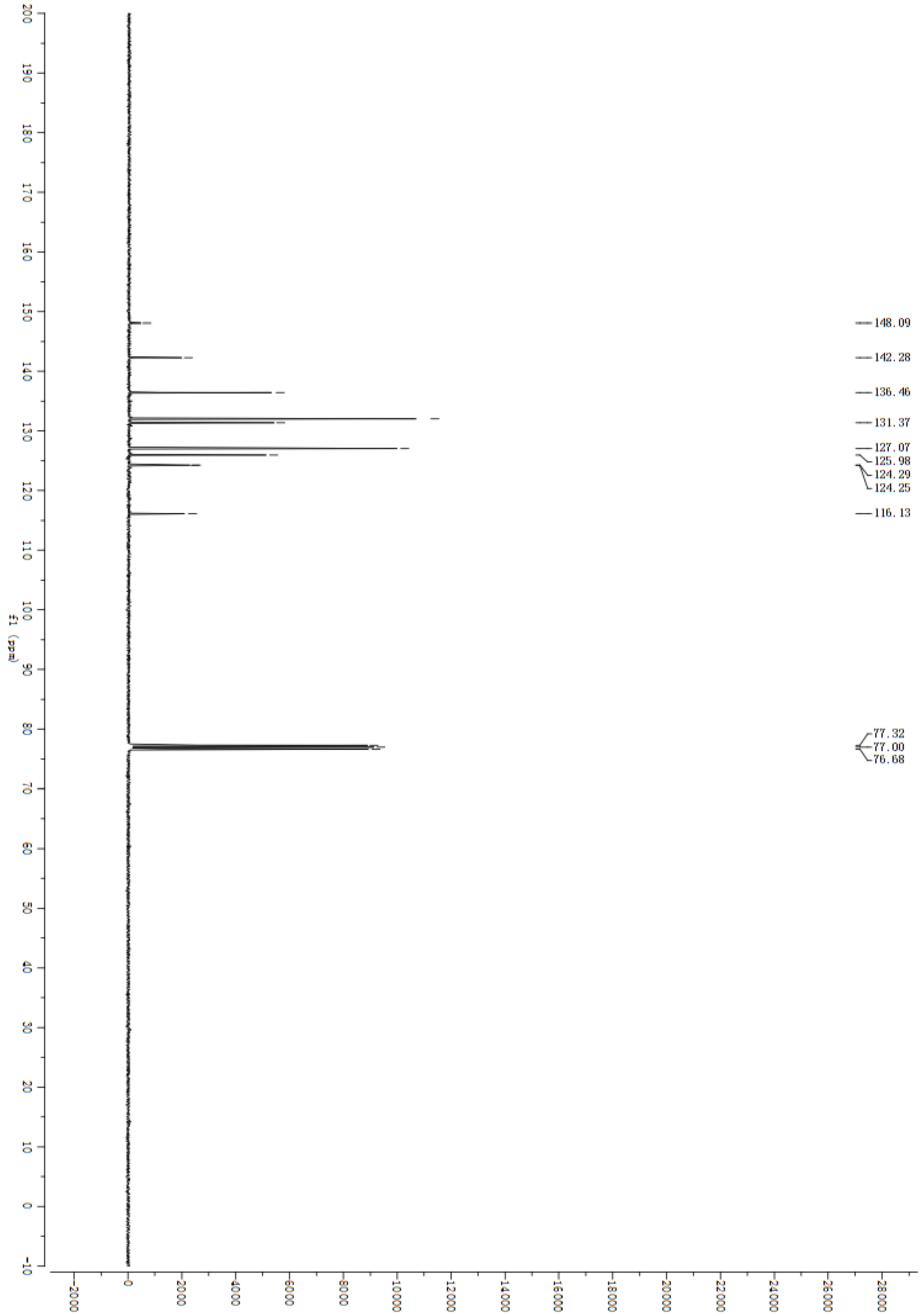
0.95
3.10
1.00
f1 (ppm)

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124.25
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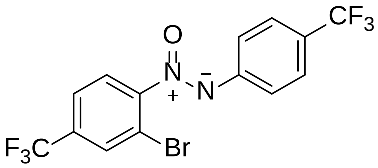
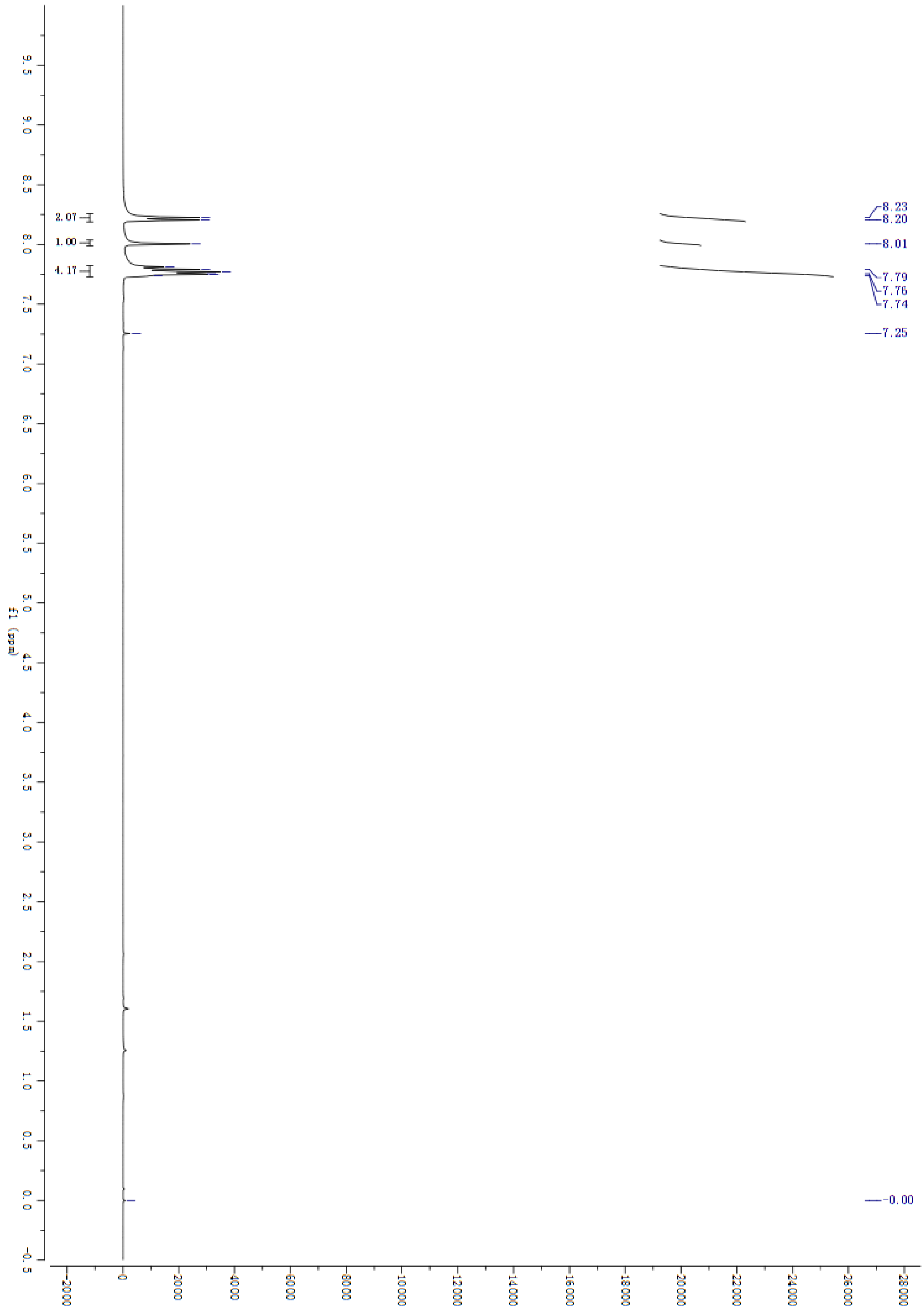
77.00
76.68
f1 (ppm)

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7.74
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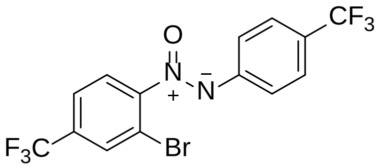
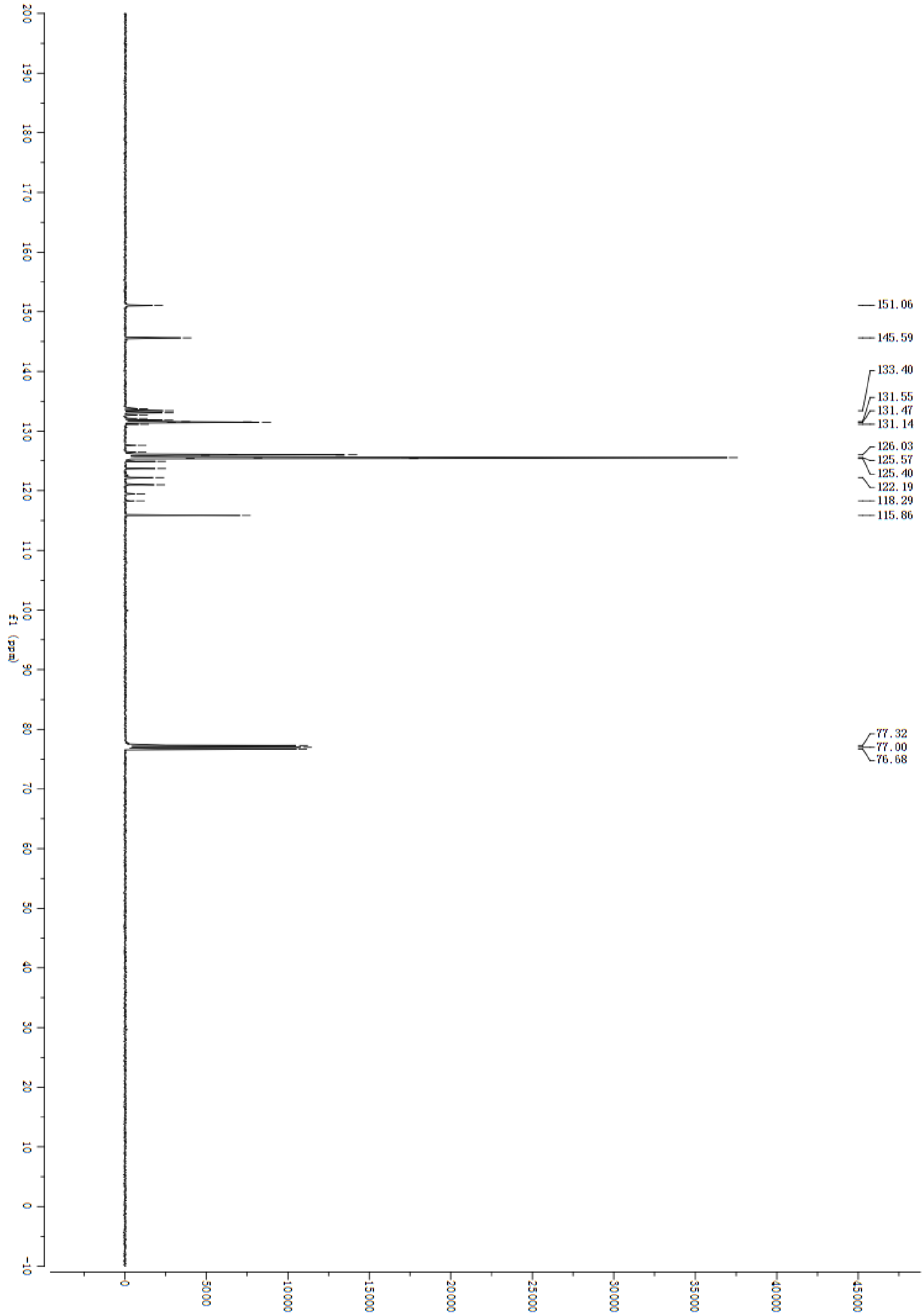
4.17
f1 (ppm)


O
N
+
N
−
CF_3
F_3C
Br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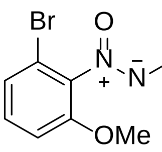
77.32
77.00
76.68
f1 (ppm)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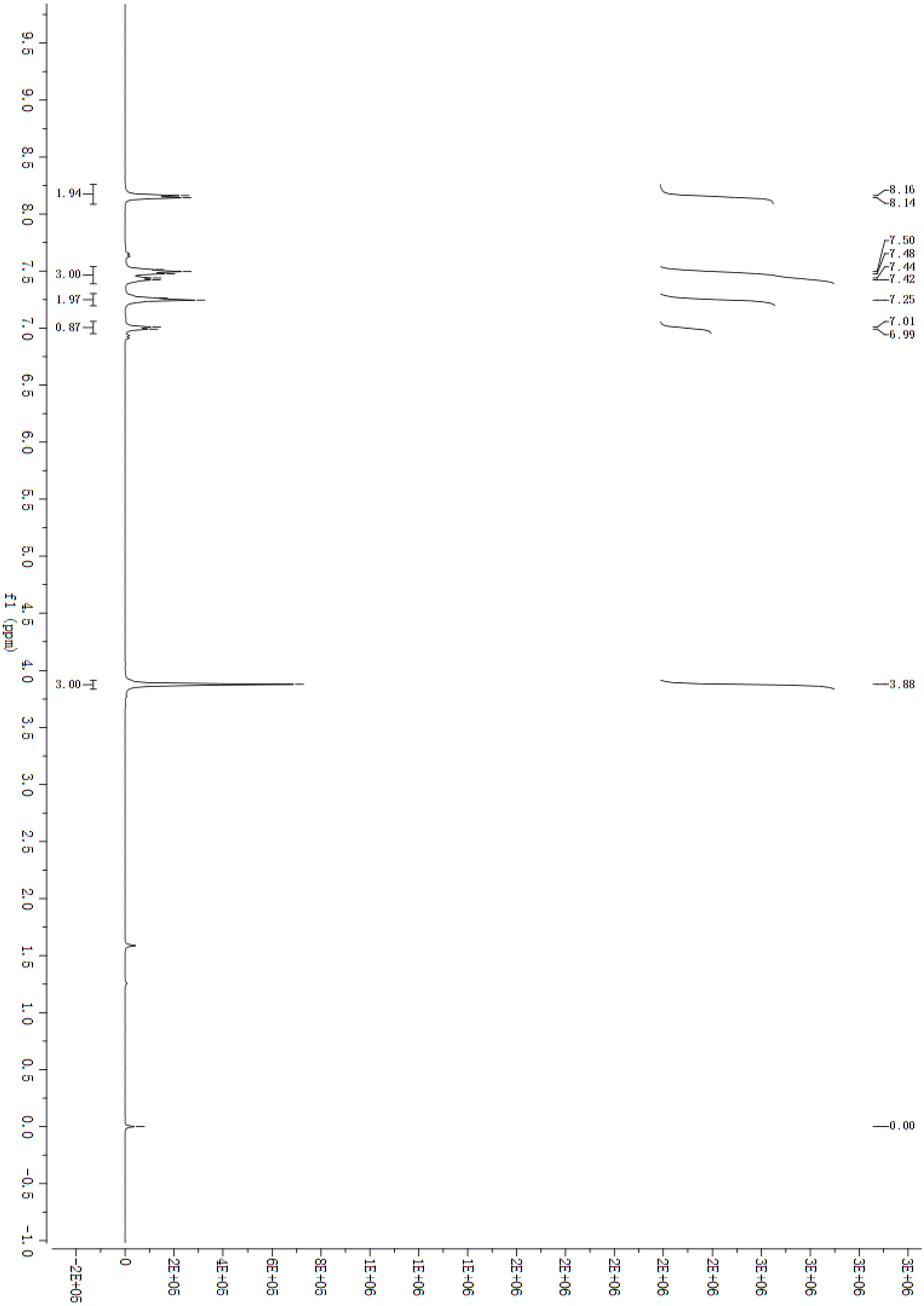
N
+
N
OMe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7.48
7.44
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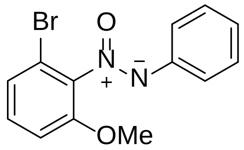
7.25
7.01
6.99
3.88
0.00
1.94
3.00
1.97
0.87
3.00
f1 (ppm)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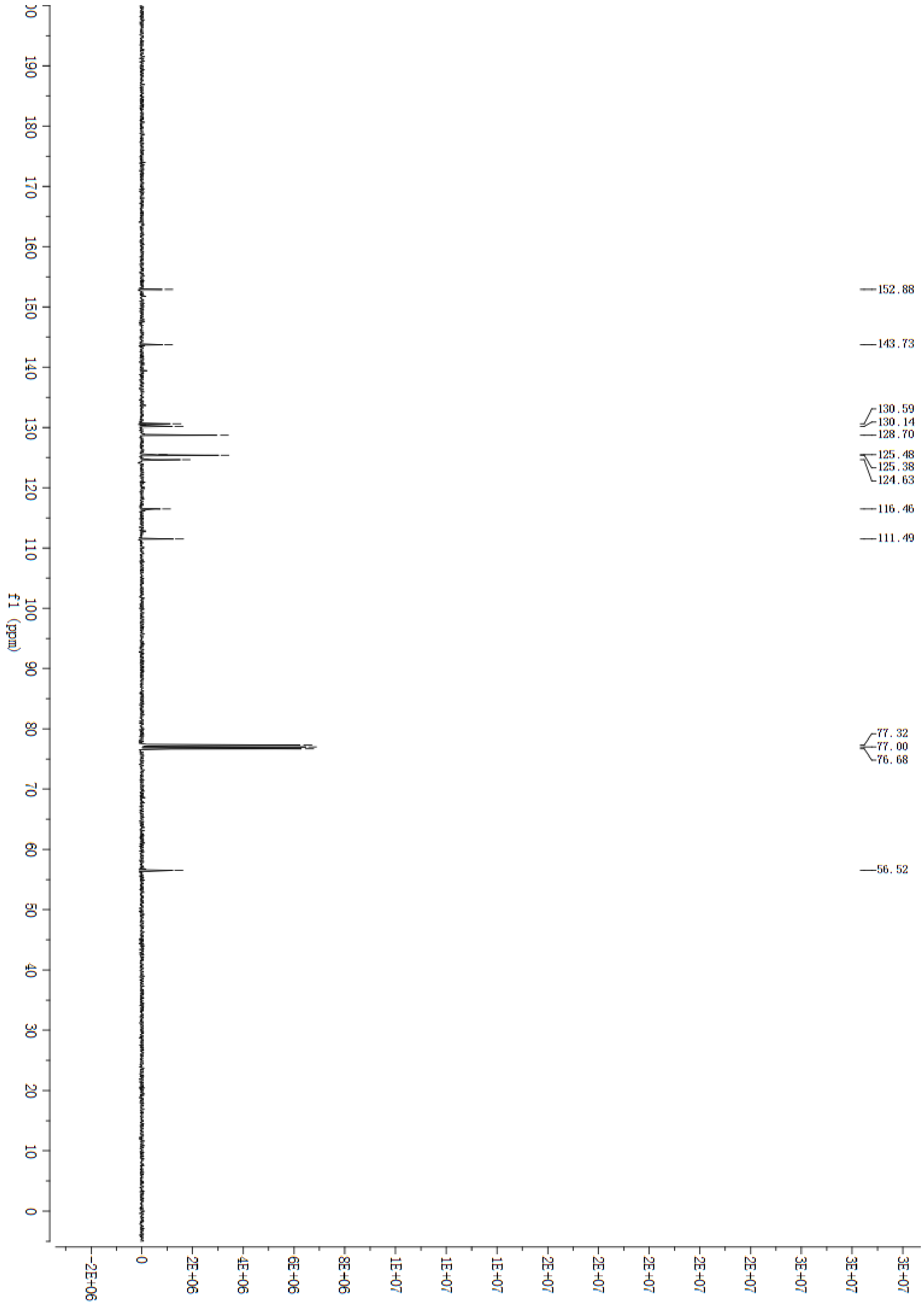
N
OMe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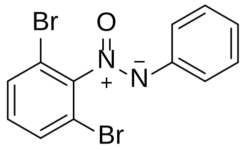
111.49
77.32
77.00
76.68
56.52
f1 (ppm)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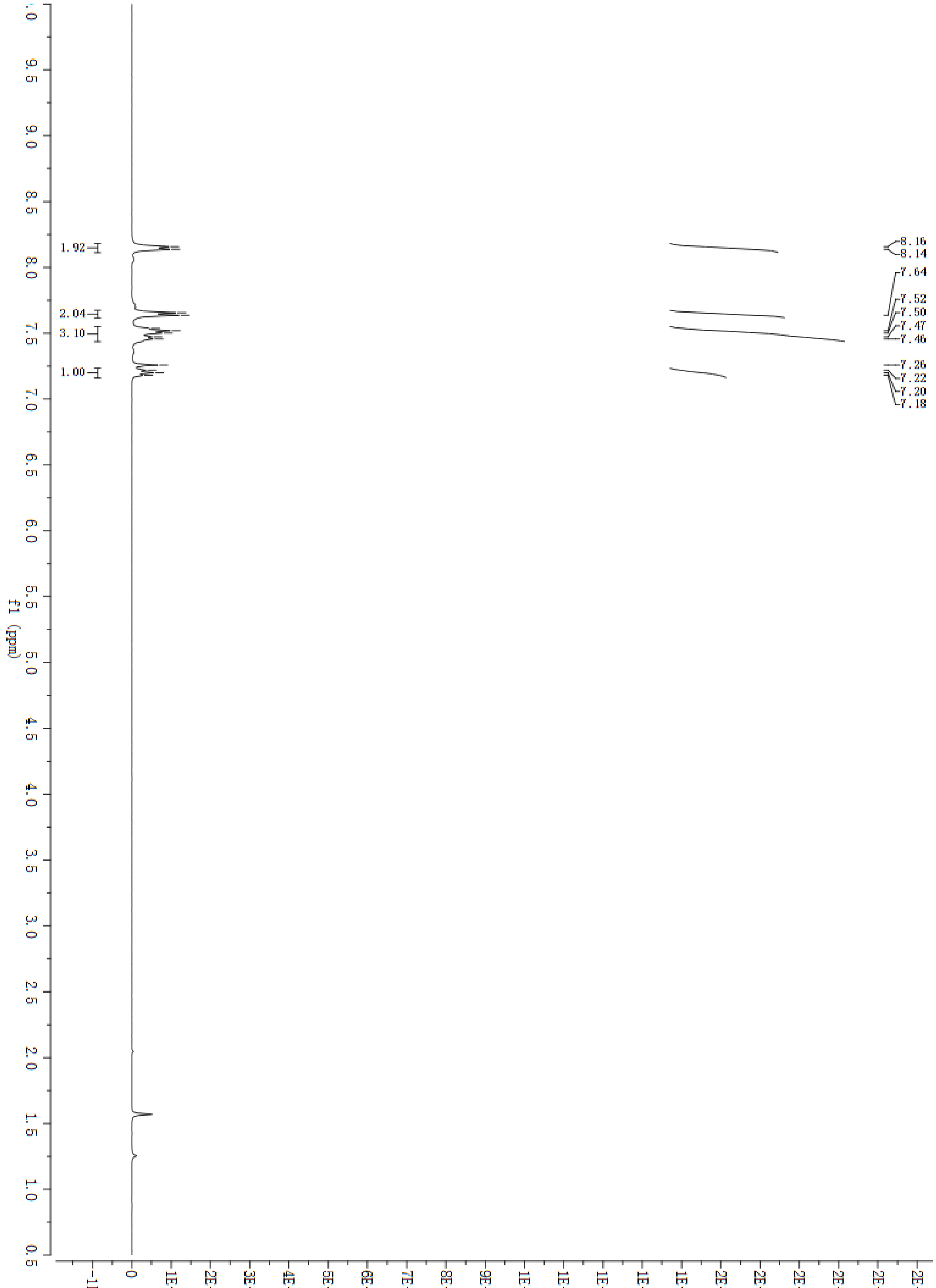
N
Br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7.26
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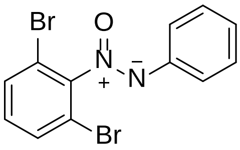
7.20
7.18
1.92
2.04
3.10
1.00
f1 (ppm)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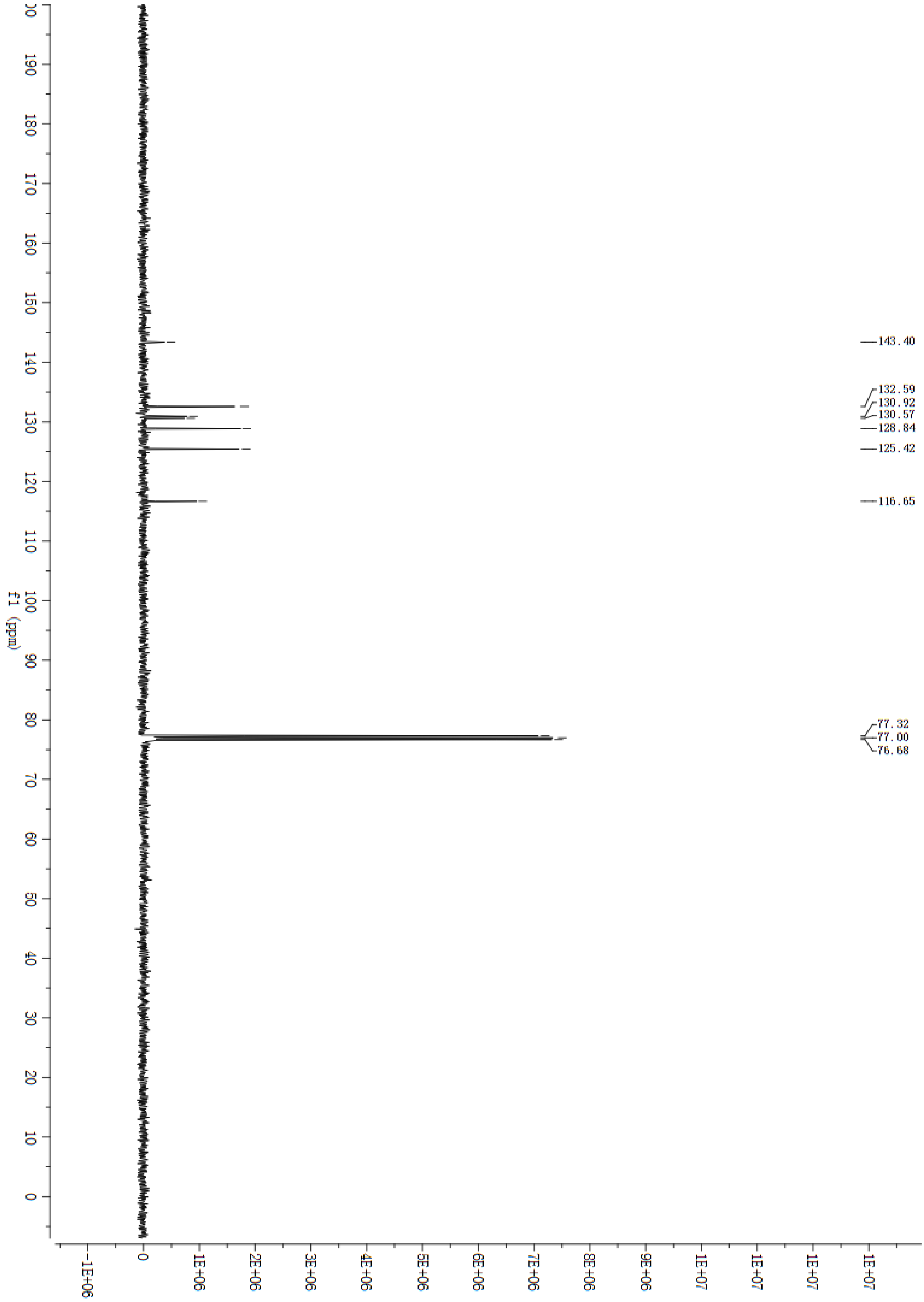
N
+
N
–
Br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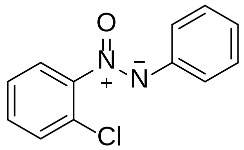
128.84
125.42
116.65
77.32
77.00
76.68
f1 (ppm)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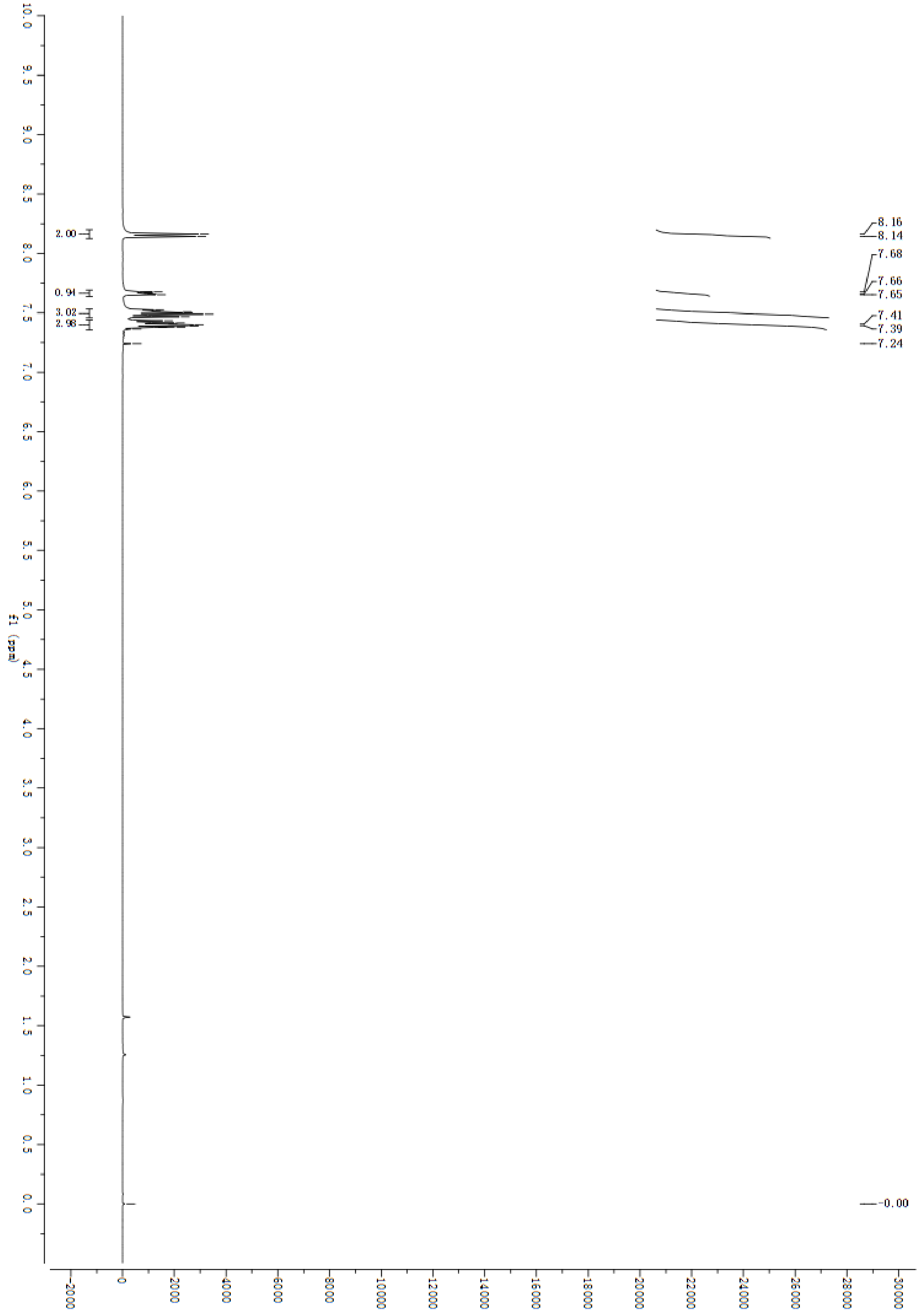
+
N
Cl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7.24
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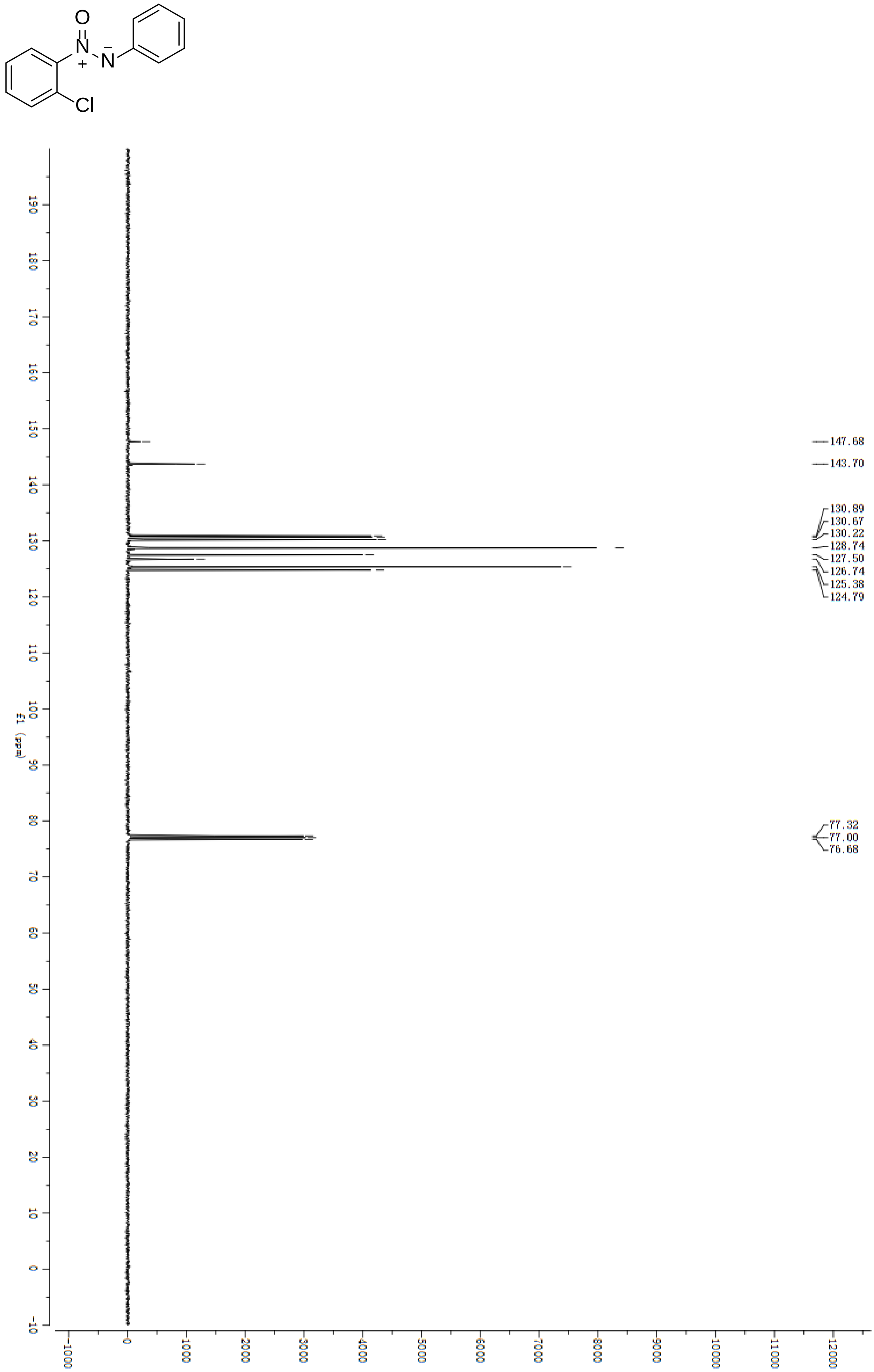
0.94
3.02
2.98
f1 (ppm)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N
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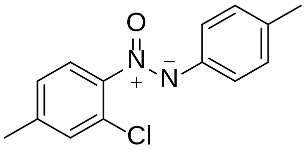
77.32
77.00
76.68
f1 (ppm)



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+
N
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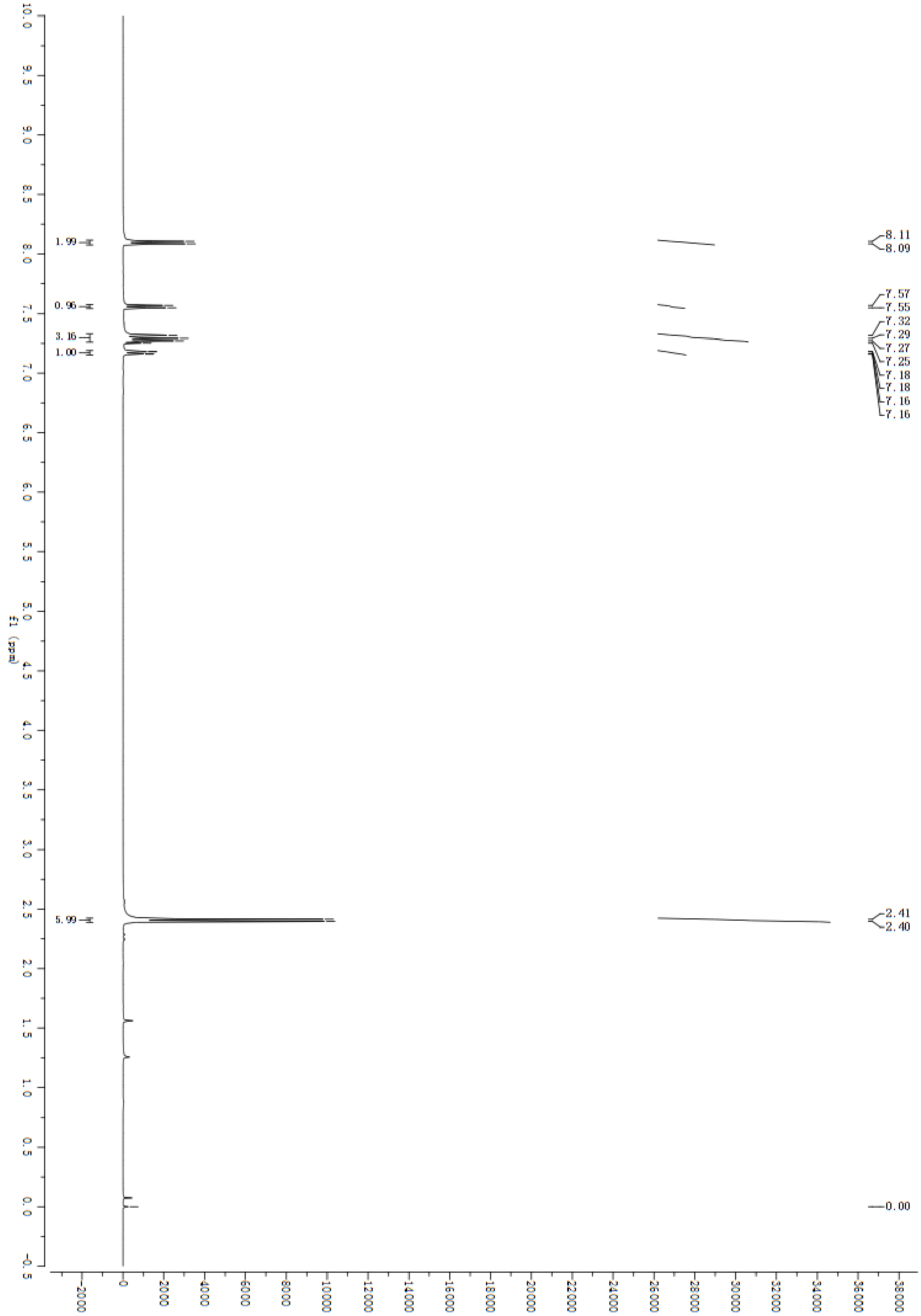

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7.27
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2.41
2.40
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1.99
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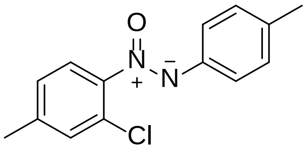
3.16
1.00
5.99
f1 (ppm)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N
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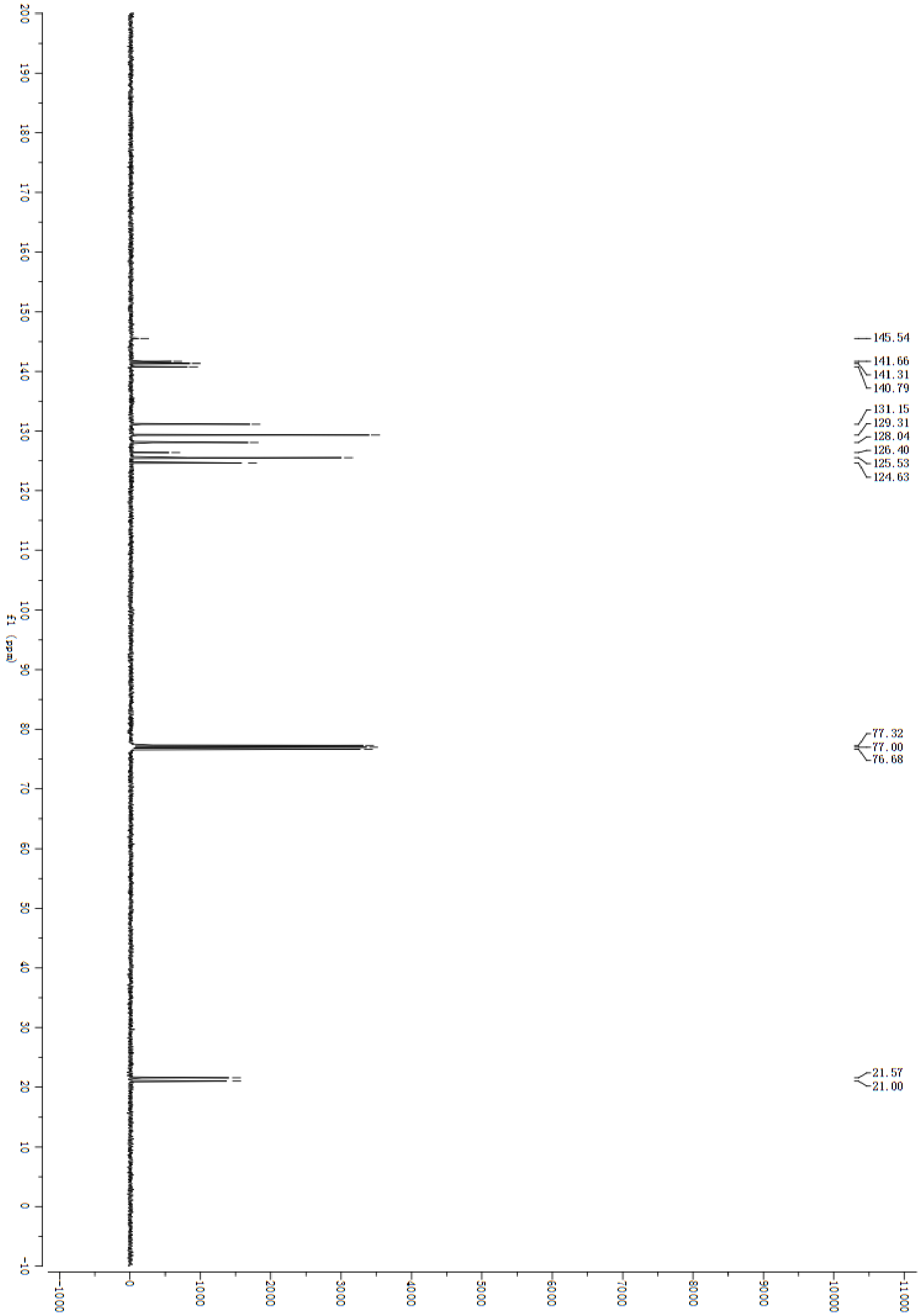
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77.32
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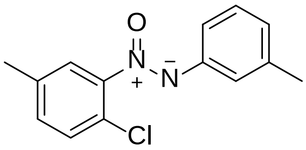
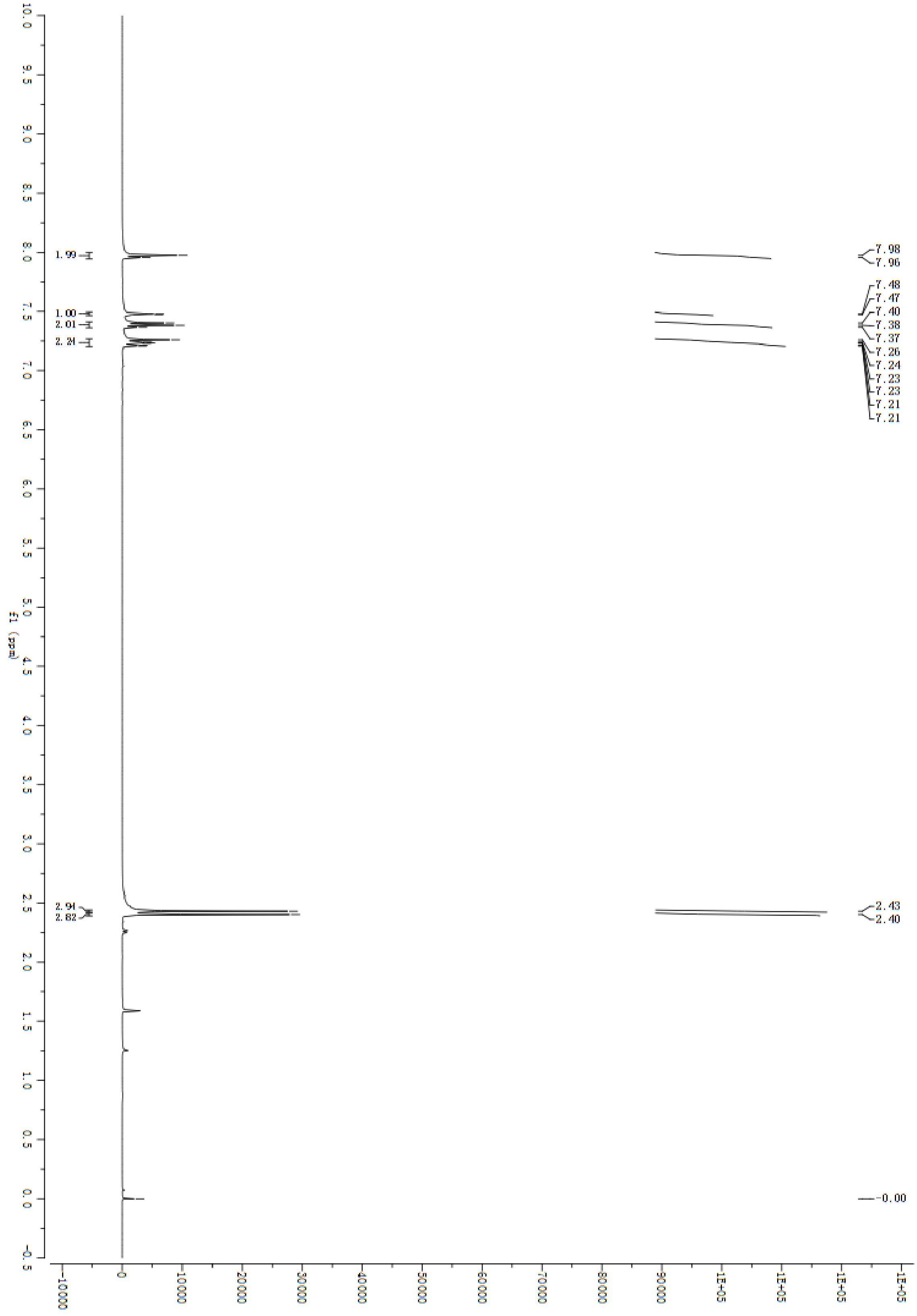
21.57
21.00
f1 (ppm)

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7.38
7.37
7.26
7.24
7.23
7.23
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7.21
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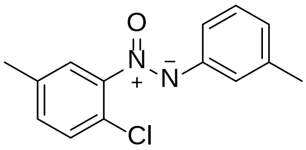
2.40
-0.00
1.99
1.00
2.01
2.24
2.94
2.82
f1 (ppm)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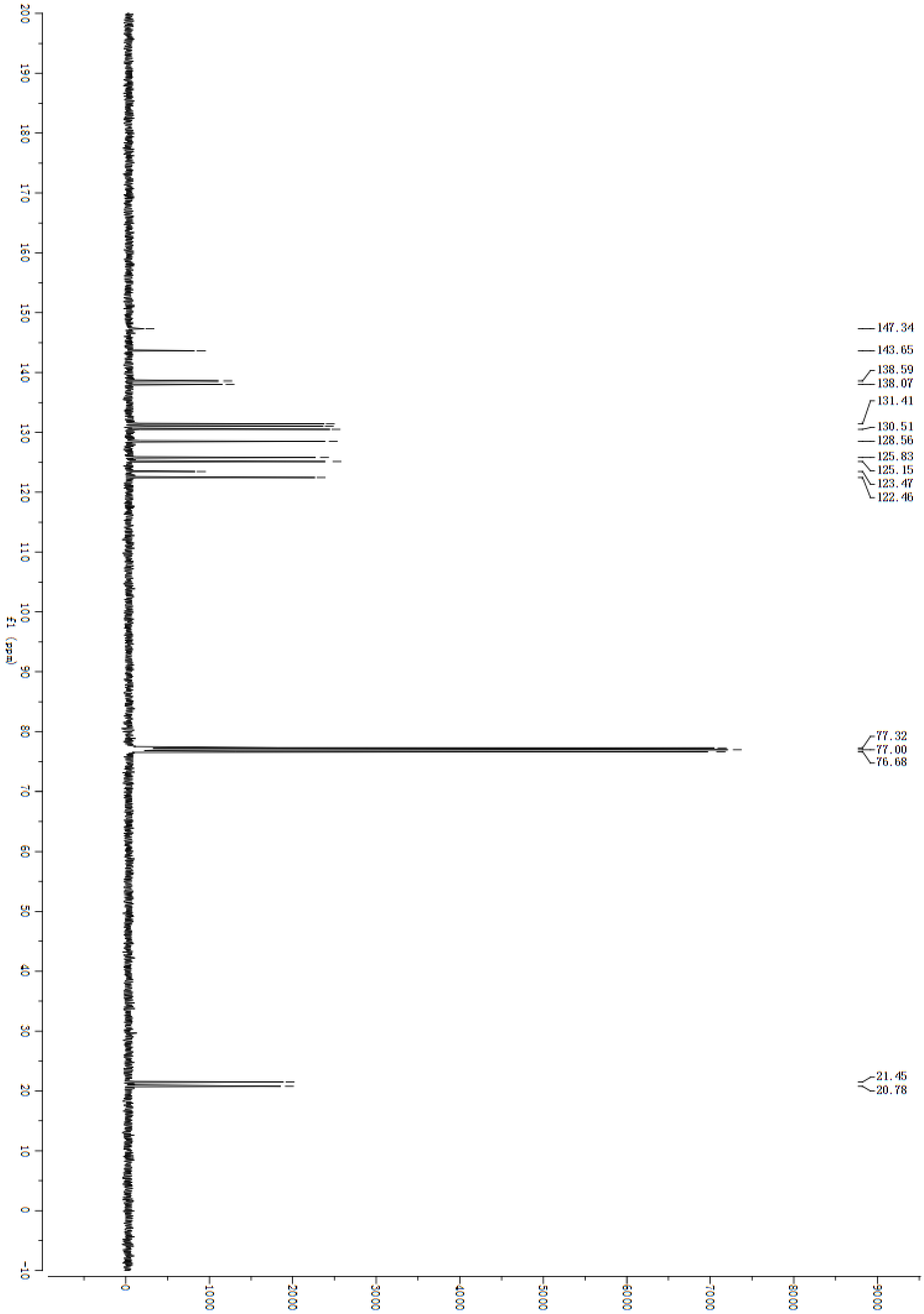
N
+
N
−
Cl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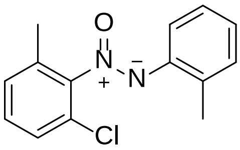
76.68
21.45
20.78
f1 (ppm)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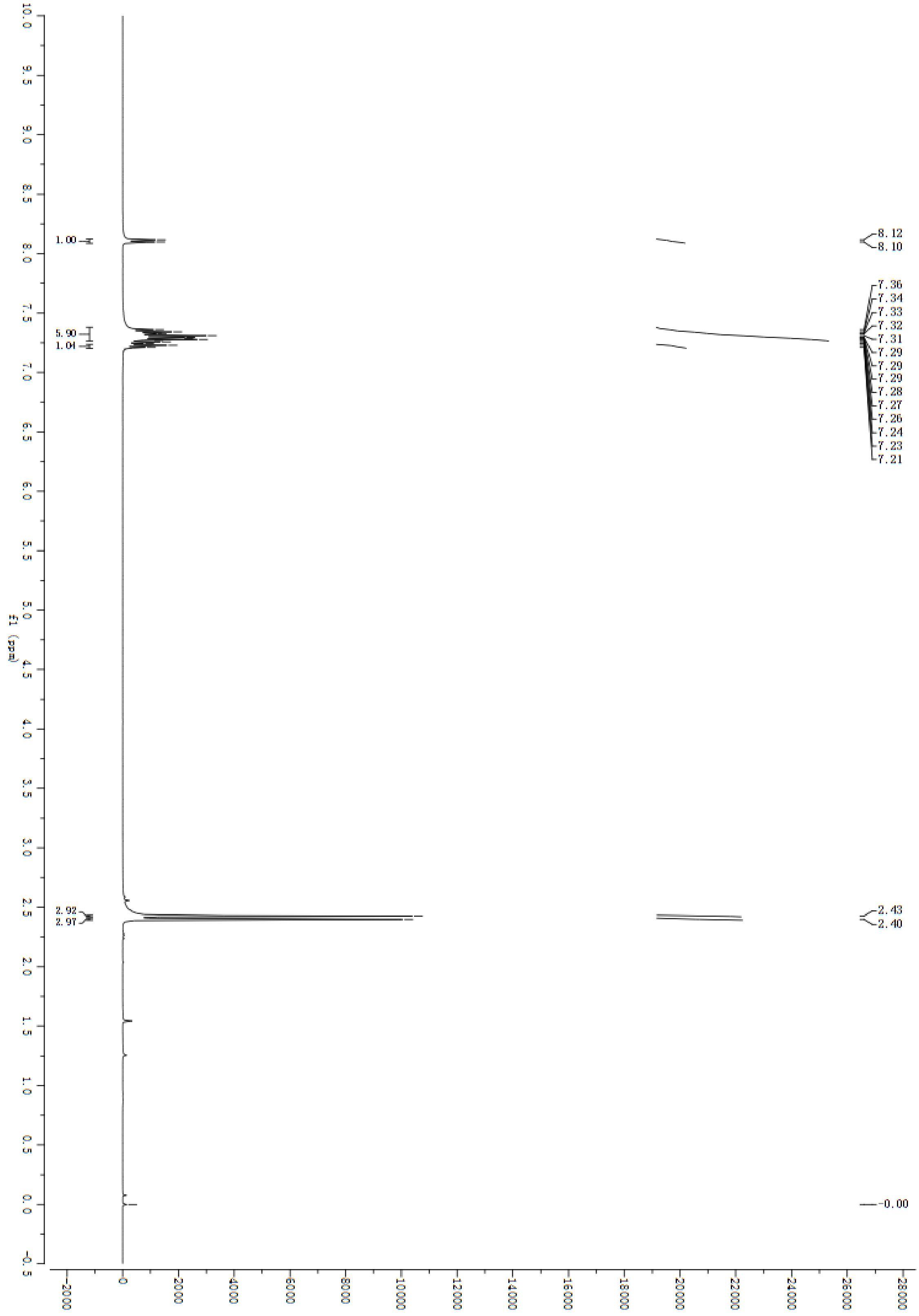
+
N
Cl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7.29
7.28
7.27
7.26
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7.23
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1.04
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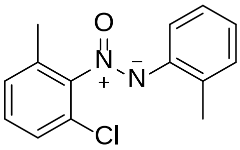
2.97
f1 (ppm)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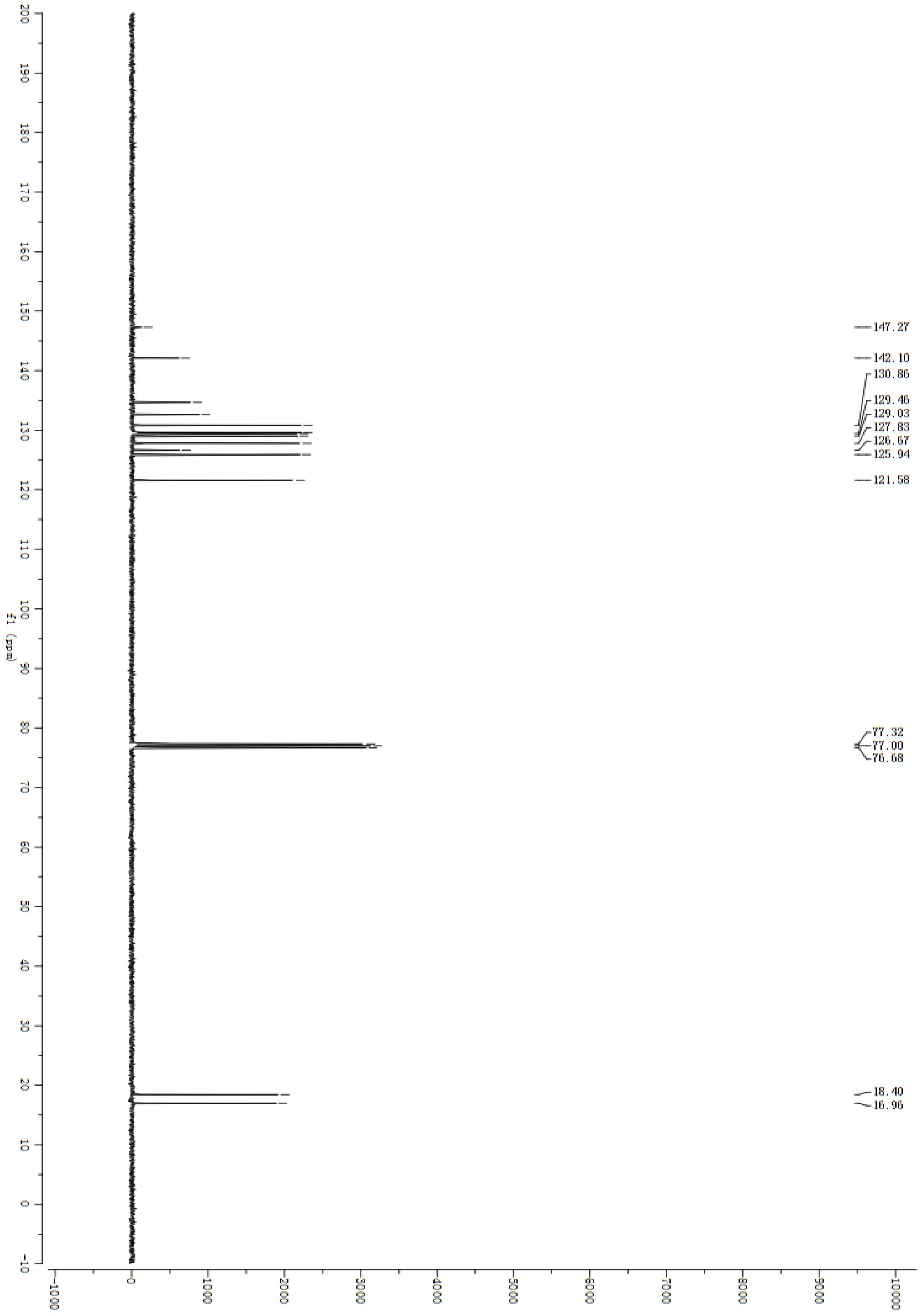
+
N
Cl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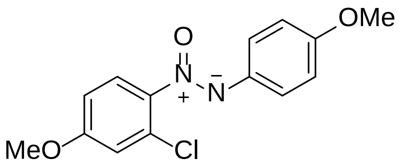
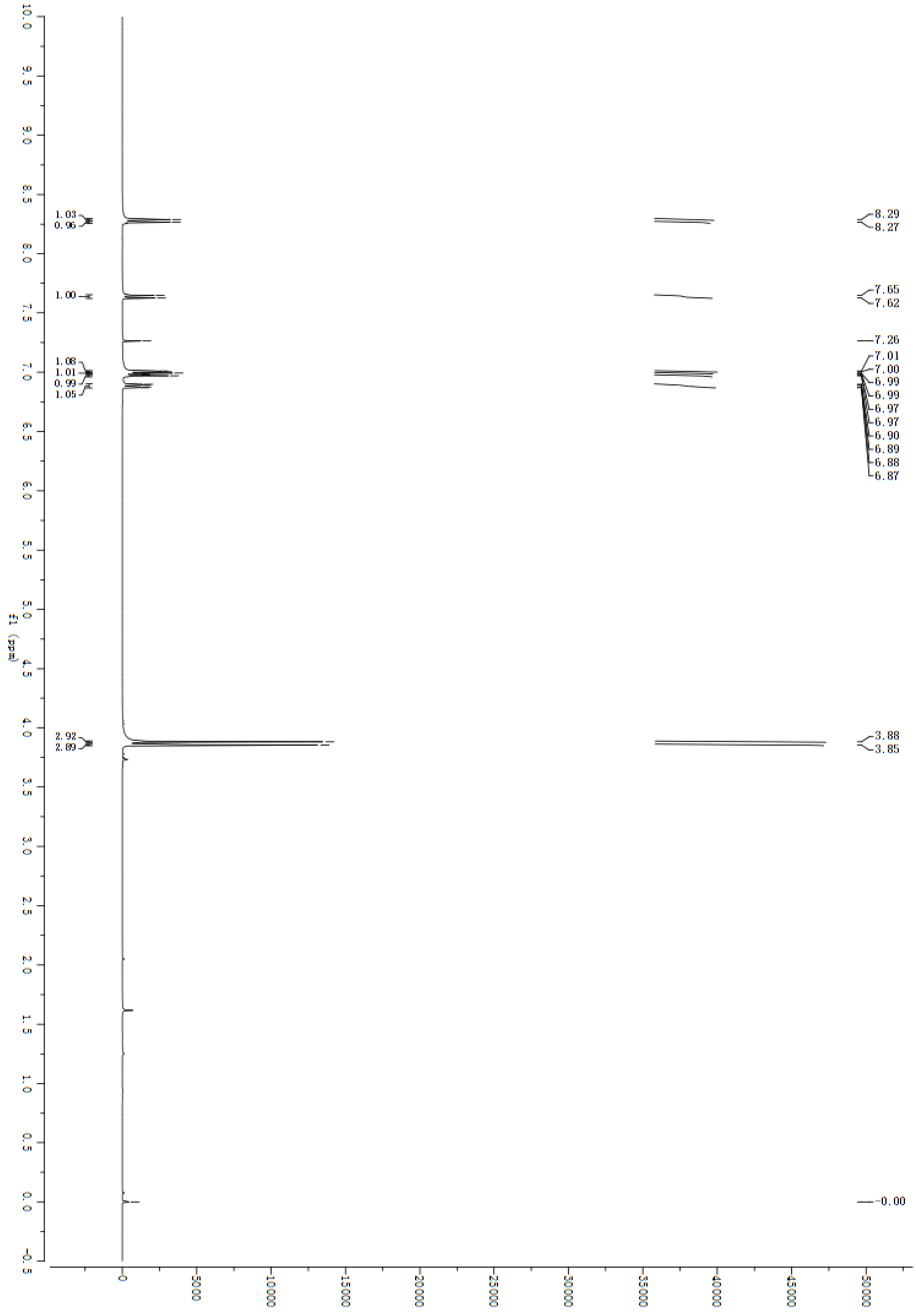

OMe
O
N
+
−
N
MeO
Cl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6.99
6.97
6.97
6.90
6.89
6.88
6.87
3.88
3.85
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0.96
1.00
1.08
1.01
0.99
1.05
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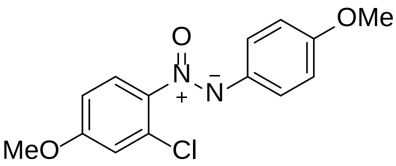
2.89
f1 (ppm)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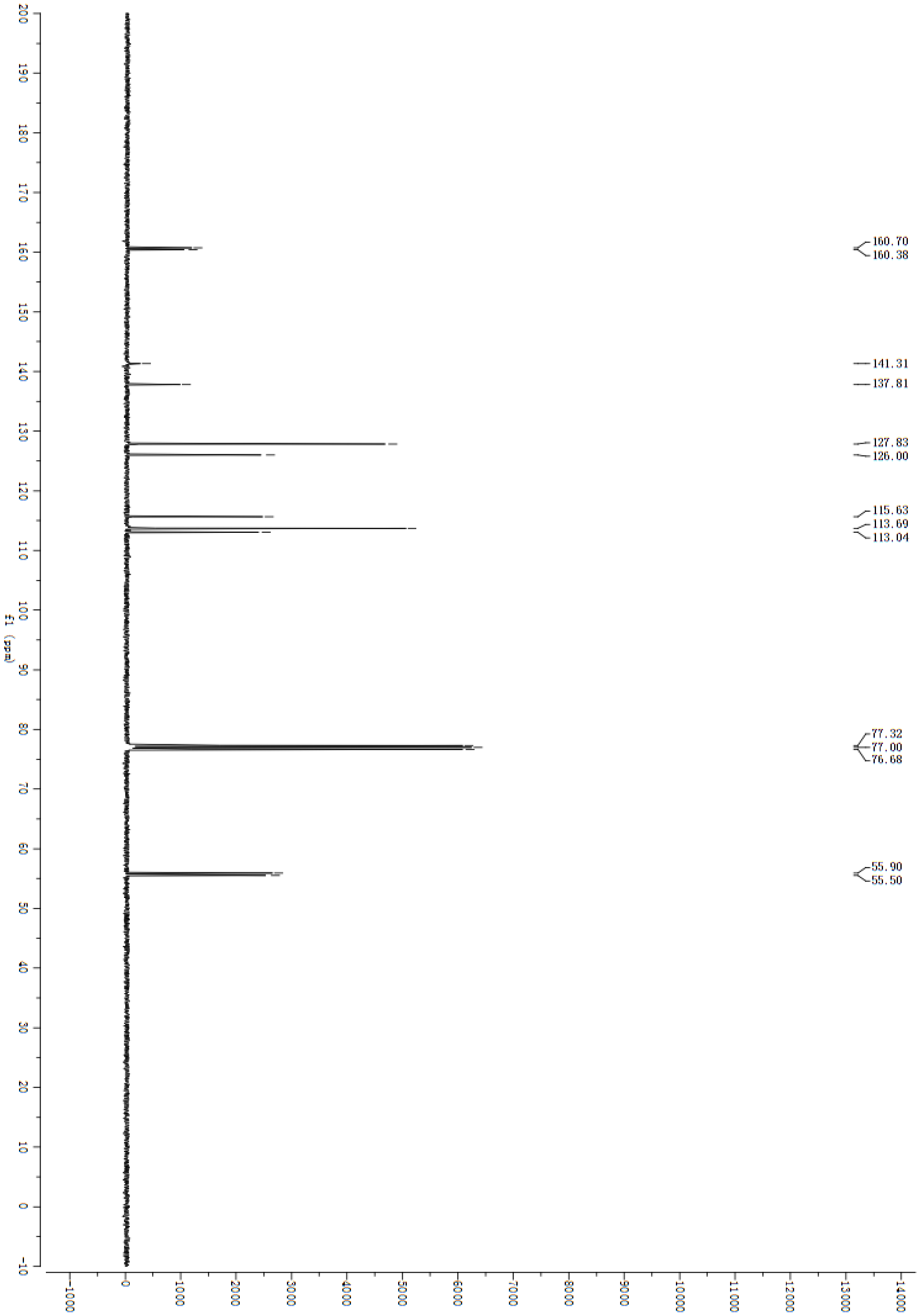
O
N
N
MeO
Cl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76.68
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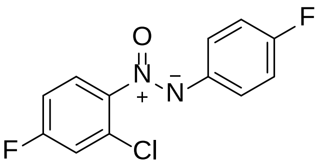
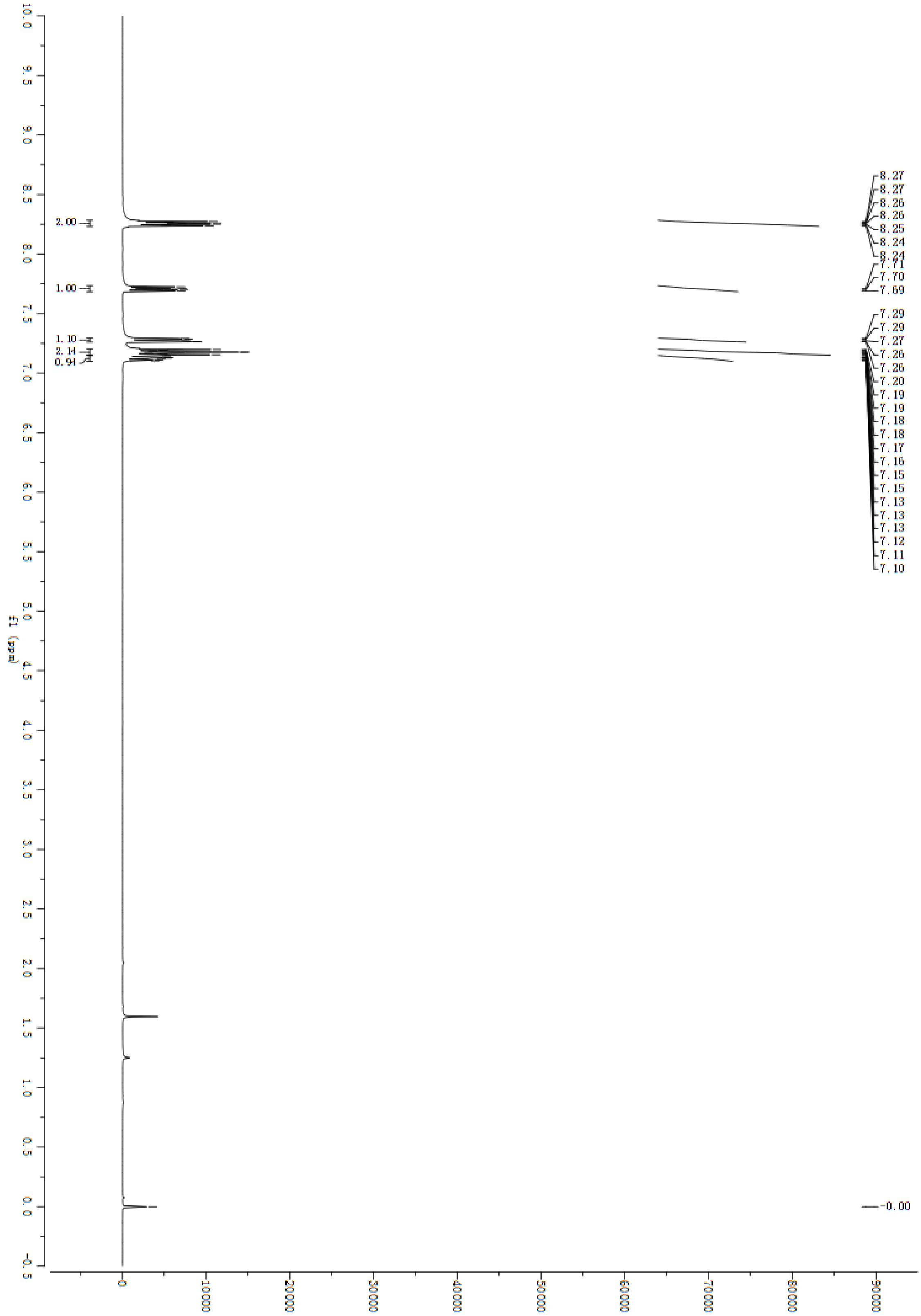
55.50
f1 (ppm)

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8.26
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7.26
7.26
7.20
7.19
7.19
7.18
7.18
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7.13
7.13
7.12
7.11
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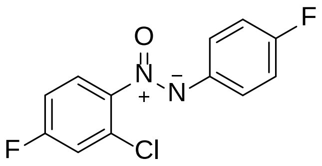
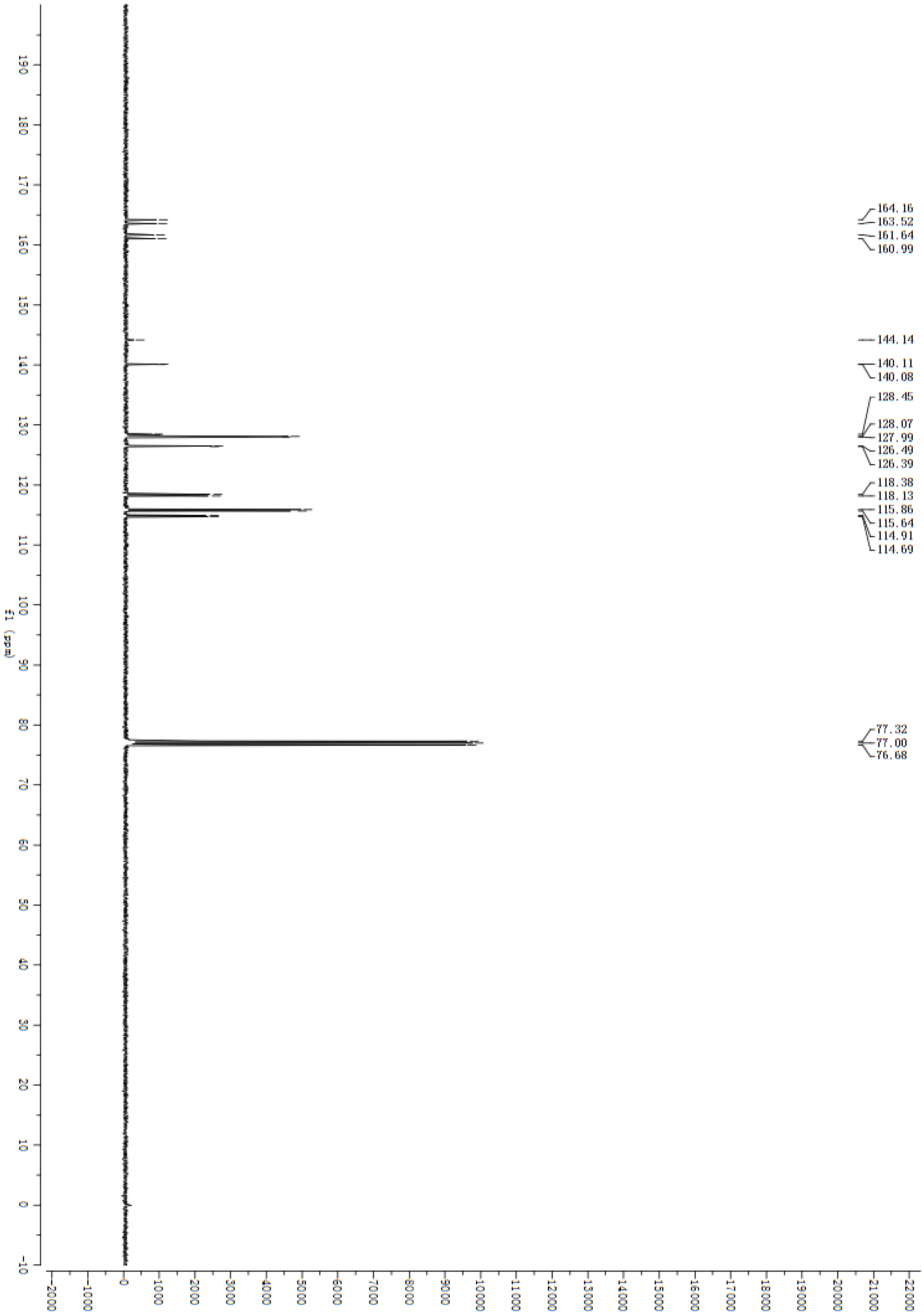
-0.00
f1 (ppm)

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160.99
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128.45
128.07
127.99
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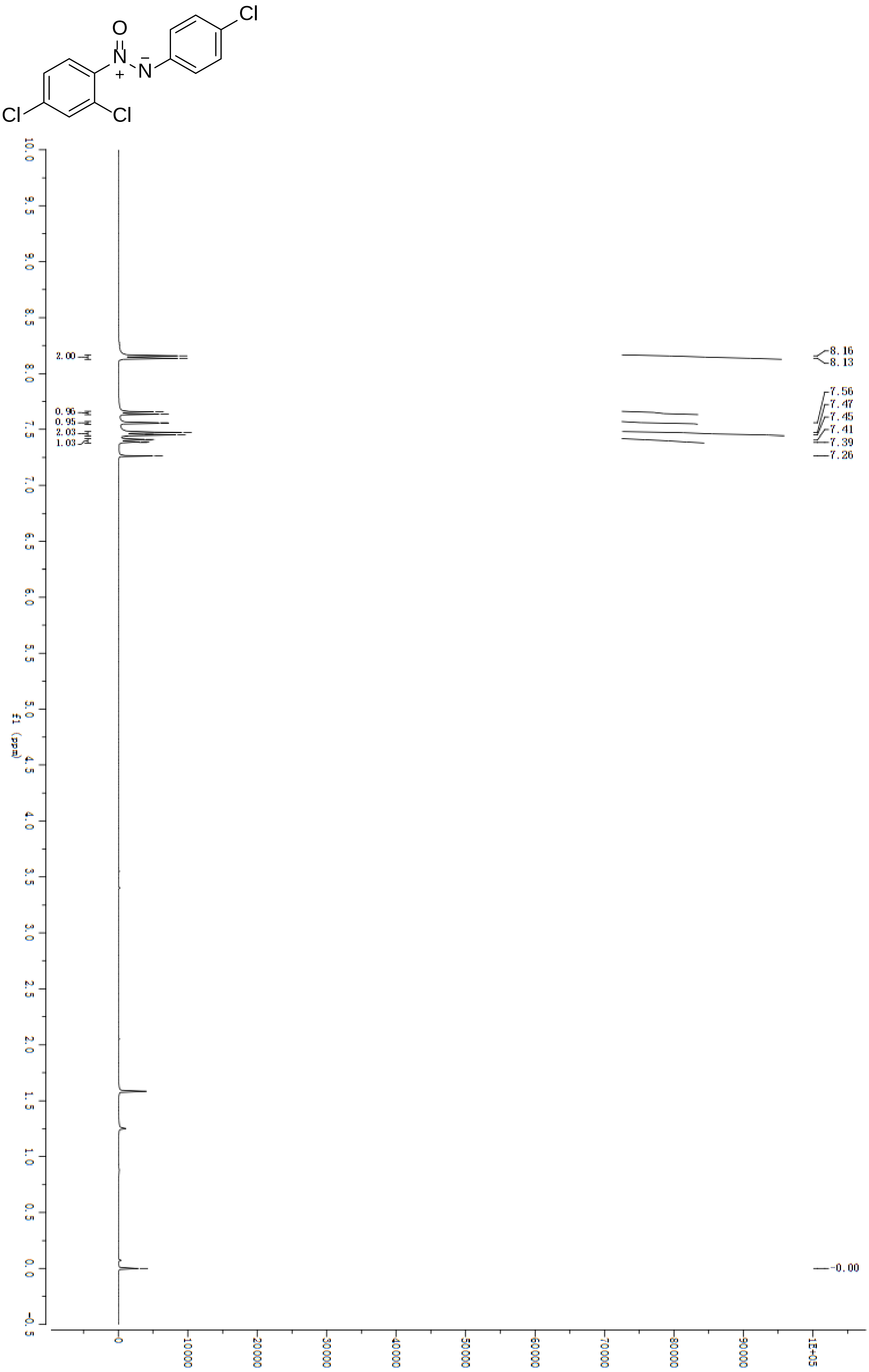
77.00
76.68
f1 (ppm)



Cl
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N
Cl
Cl
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0.96
0.95
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7.39
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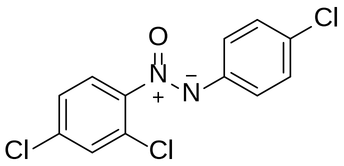
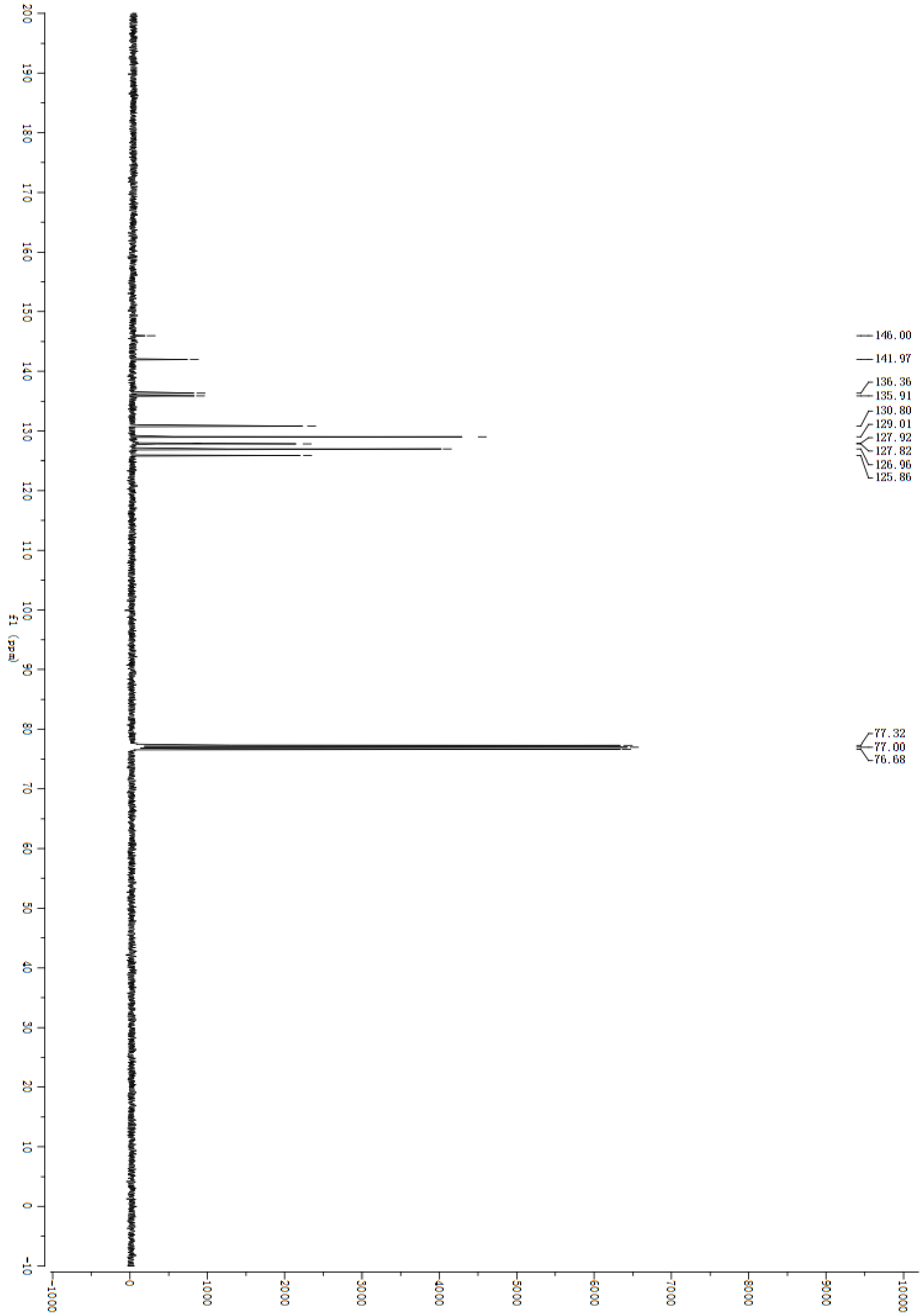
f1 (ppm)





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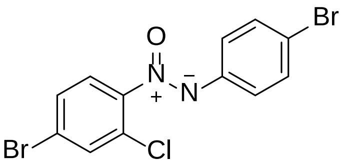
127.82
126.96
125.86
77.32
77.00
76.68
f1 (ppm)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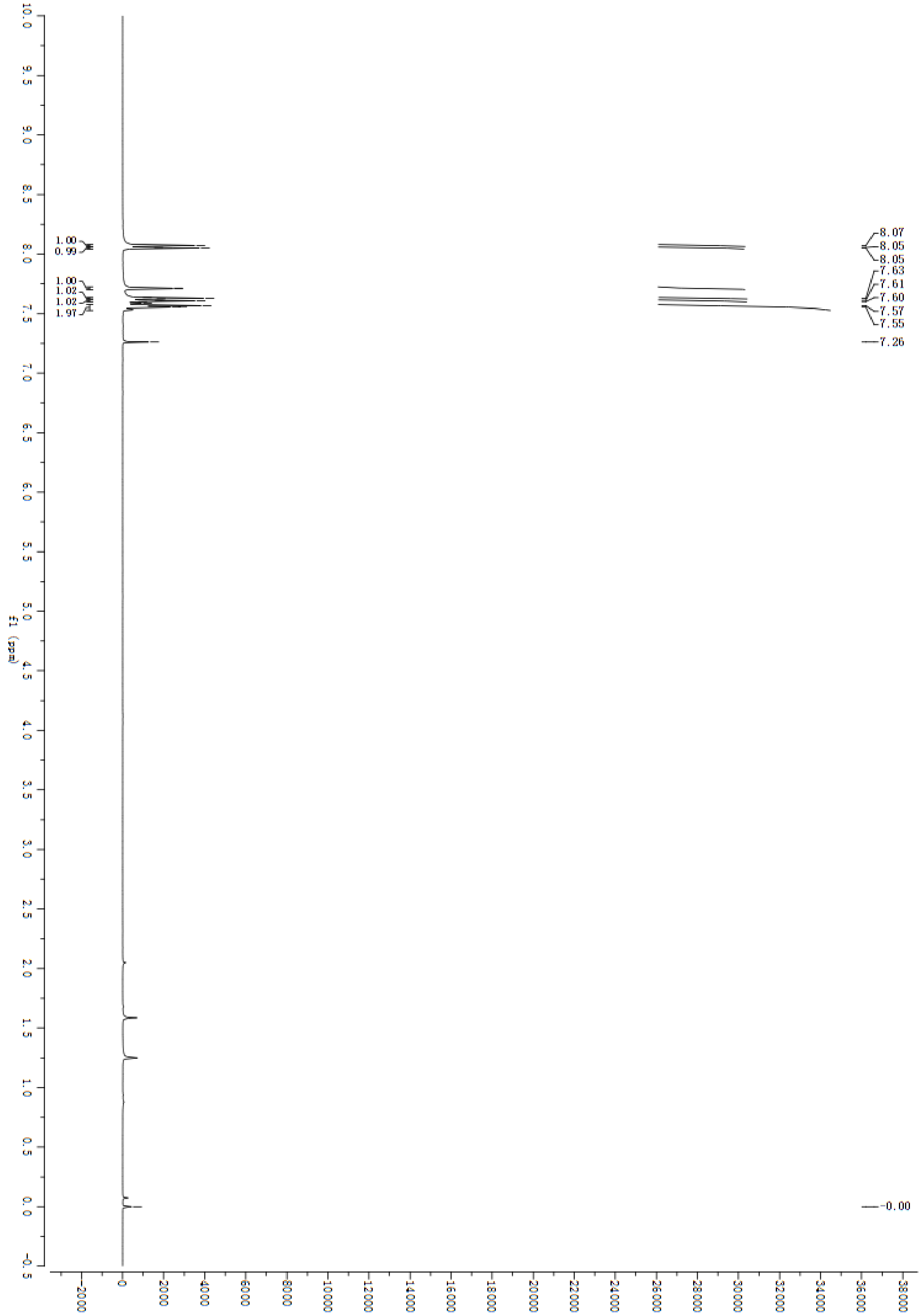
O
N
N
Br
Cl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8.05
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7.26
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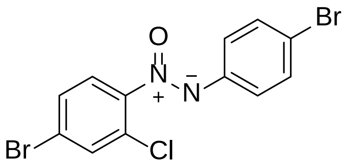
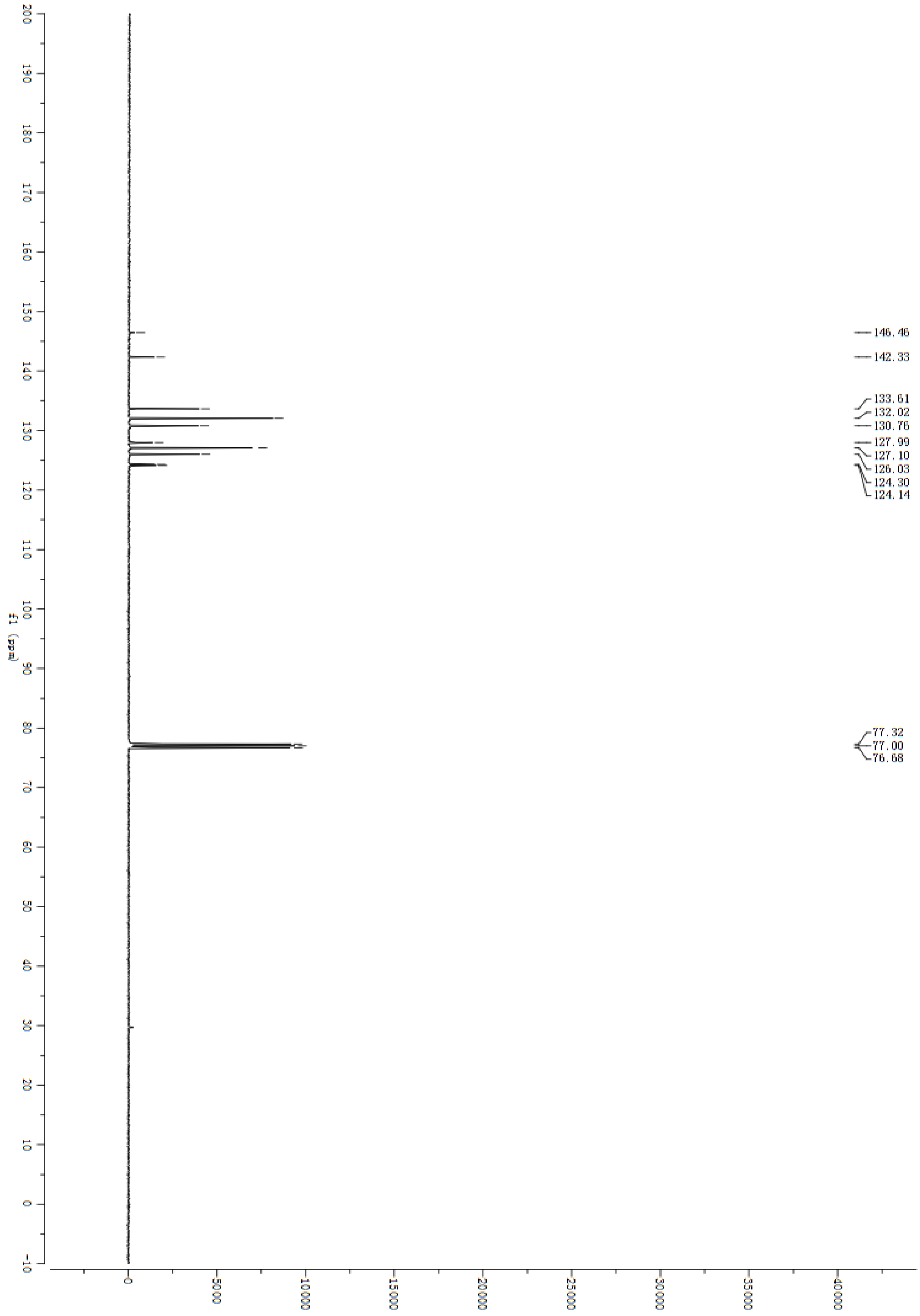
1.00
0.99
1.00
1.02
1.02
1.97
f1 (ppm)

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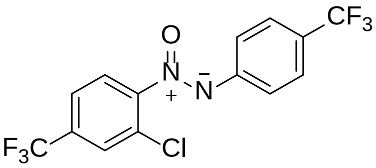
127.10
126.03
124.30
124.14
77.32
77.00
76.68
f1 (ppm)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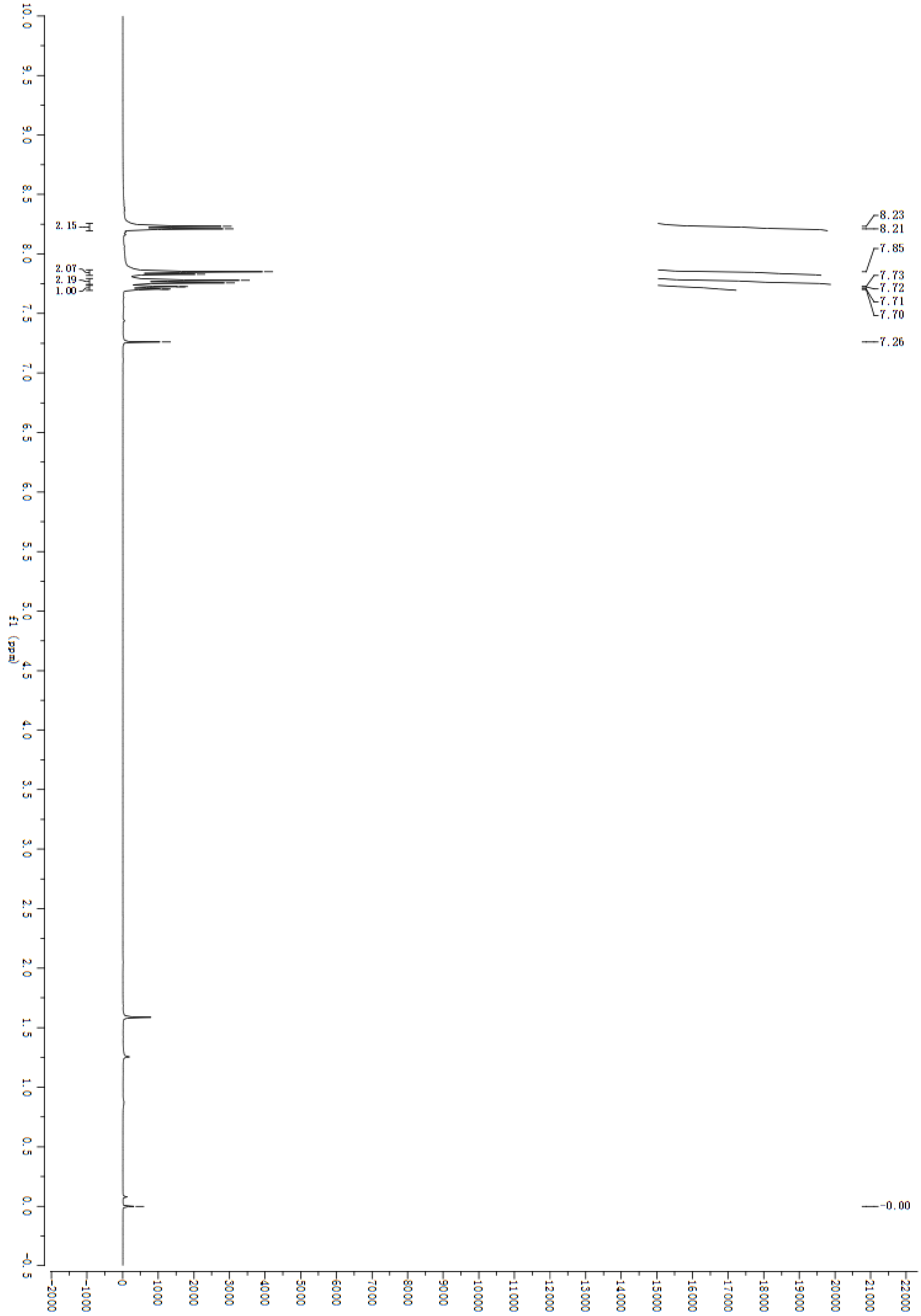
CF3
F3C
Cl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8.21
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7.73
7.72
7.71
7.70
7.26
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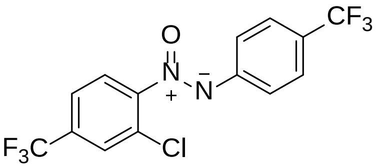
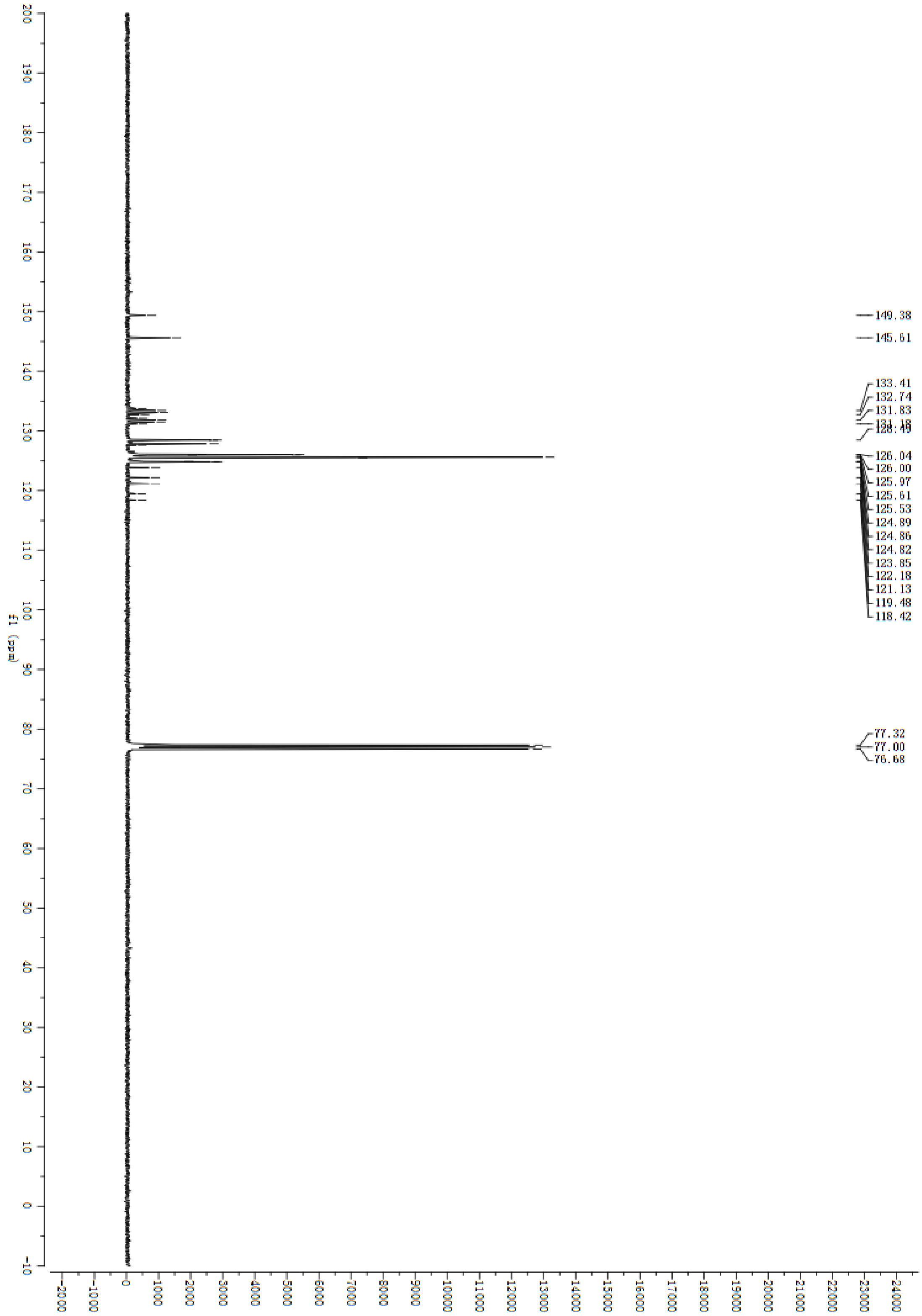
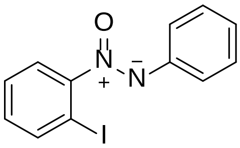
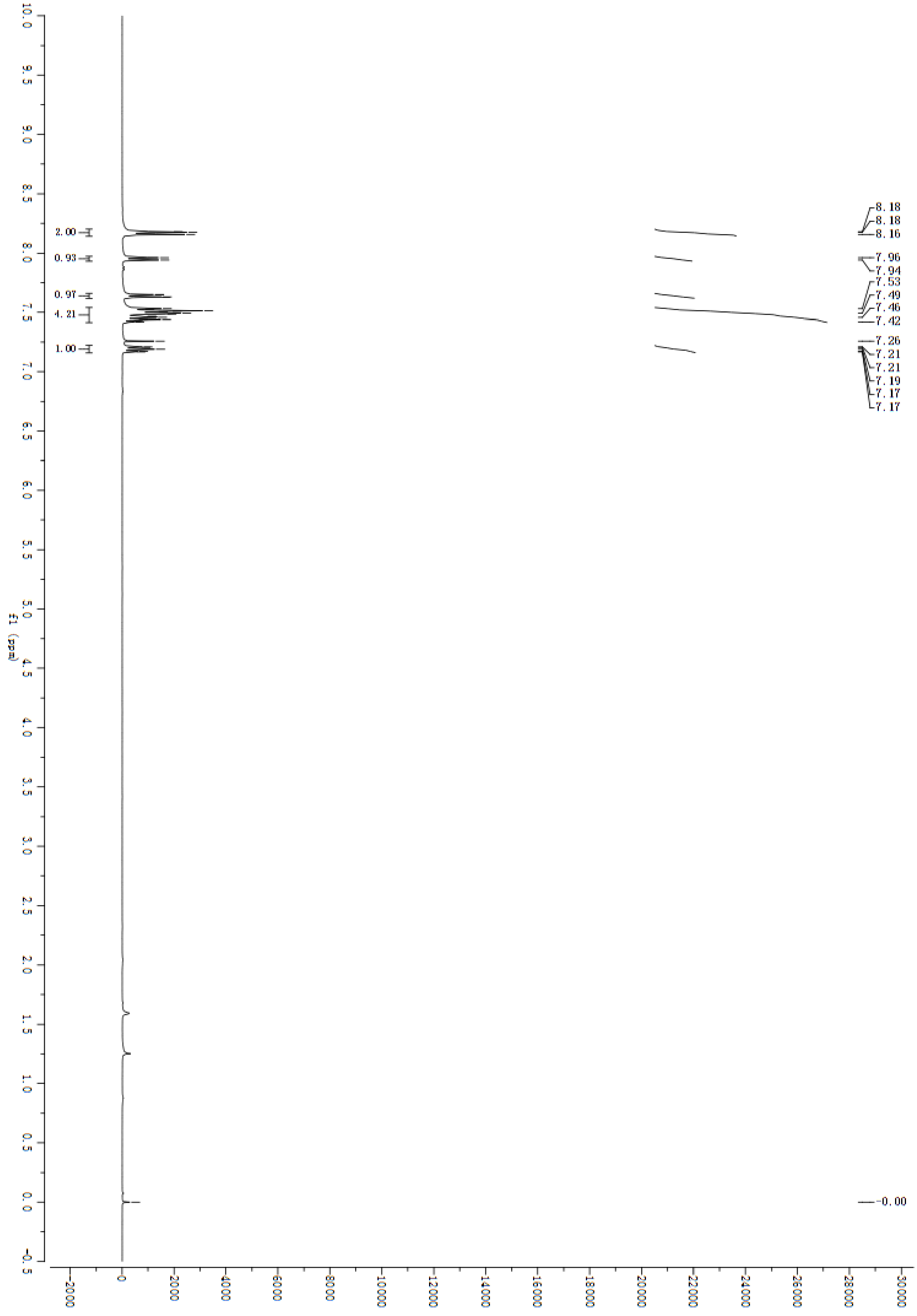
2.15
2.07
2.19
1.00
f1 (ppm)





149.38
145.61
133.41
132.74
131.83
126.04
126.00
125.97
125.61
125.53
124.89
124.86
124.82
123.85
122.18
121.13
119.48
118.42
77.32
77.00
76.68
f1 (ppm)

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7.49
7.46
7.42
7.26
7.21
7.21
7.19
7.17
7.17
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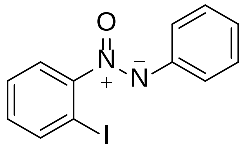
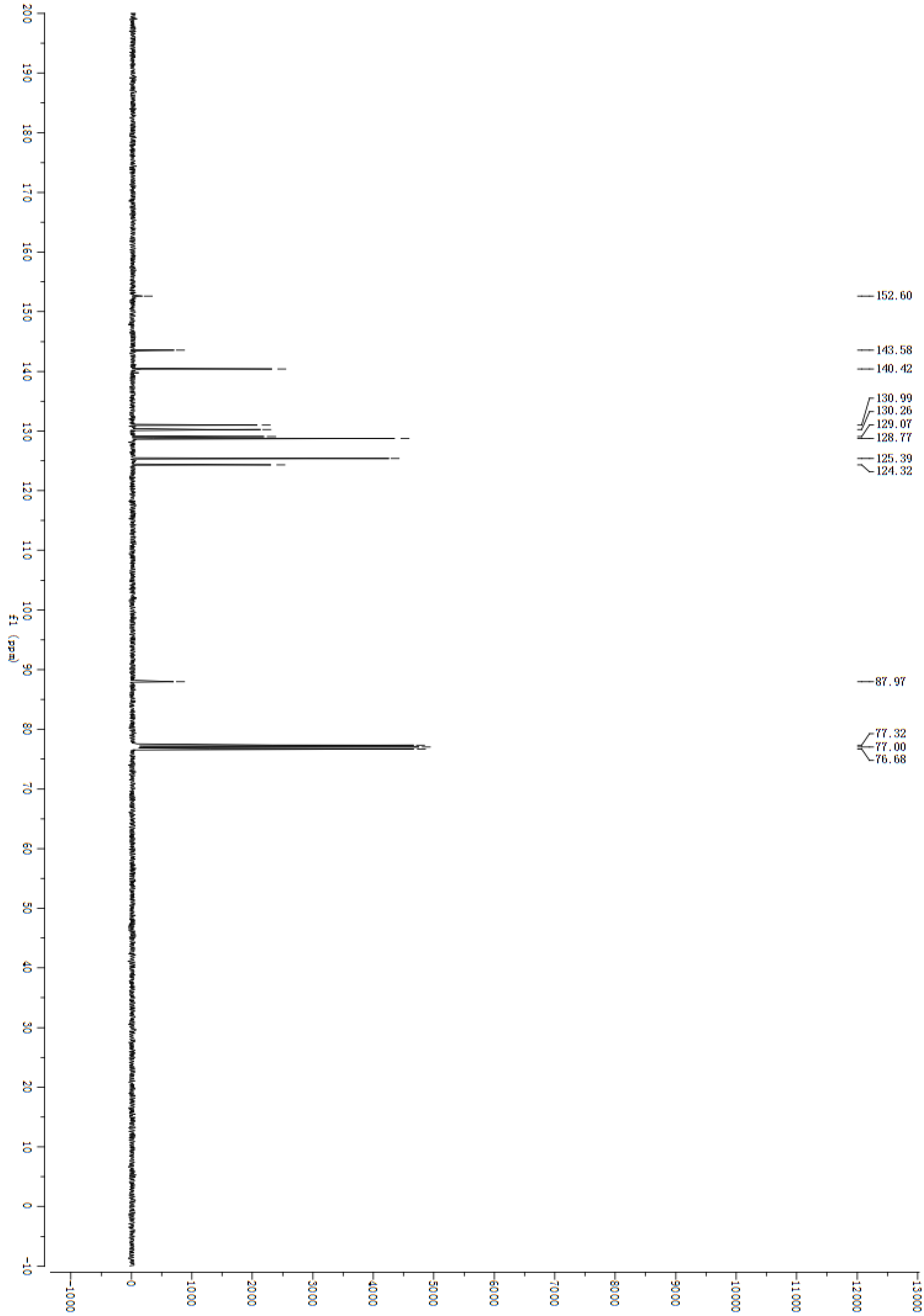
2.00
0.93
0.97
4.21
1.00
f1 (ppm)

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143.58
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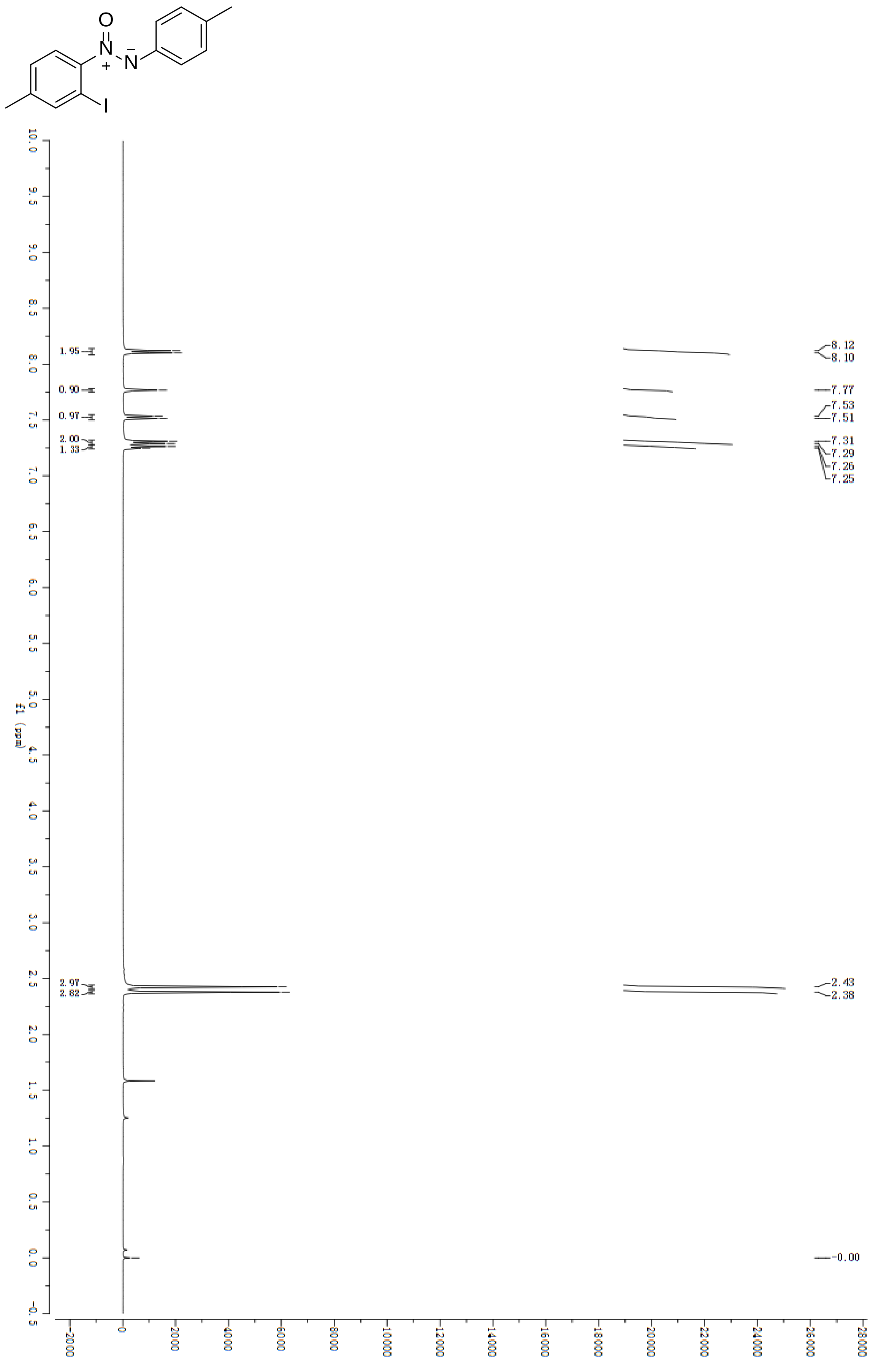
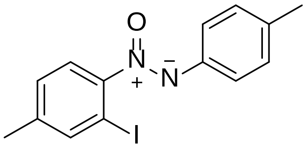
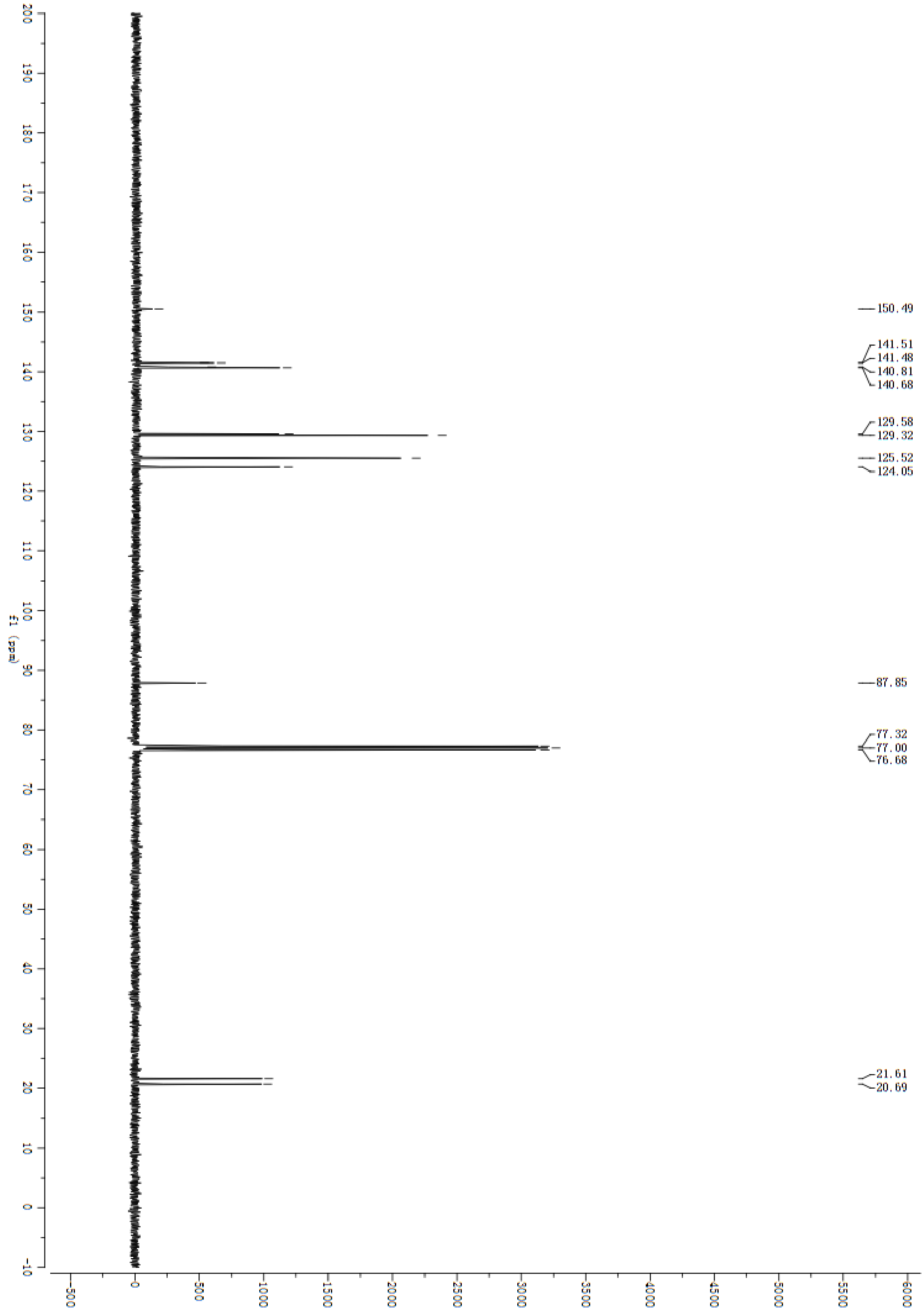
130.26
129.07
128.77
125.39
124.32
87.97
77.32
77.00
76.68
f1 (ppm)



8.12
8.10
7.77
7.53
7.51
7.31
7.29
7.26
7.25
2.43
2.38
-0.00
1.95
0.90
0.97
2.00
1.33
2.97
2.82
f1 (ppm)

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77.32
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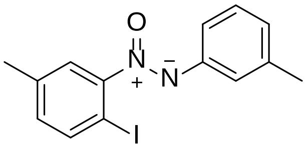
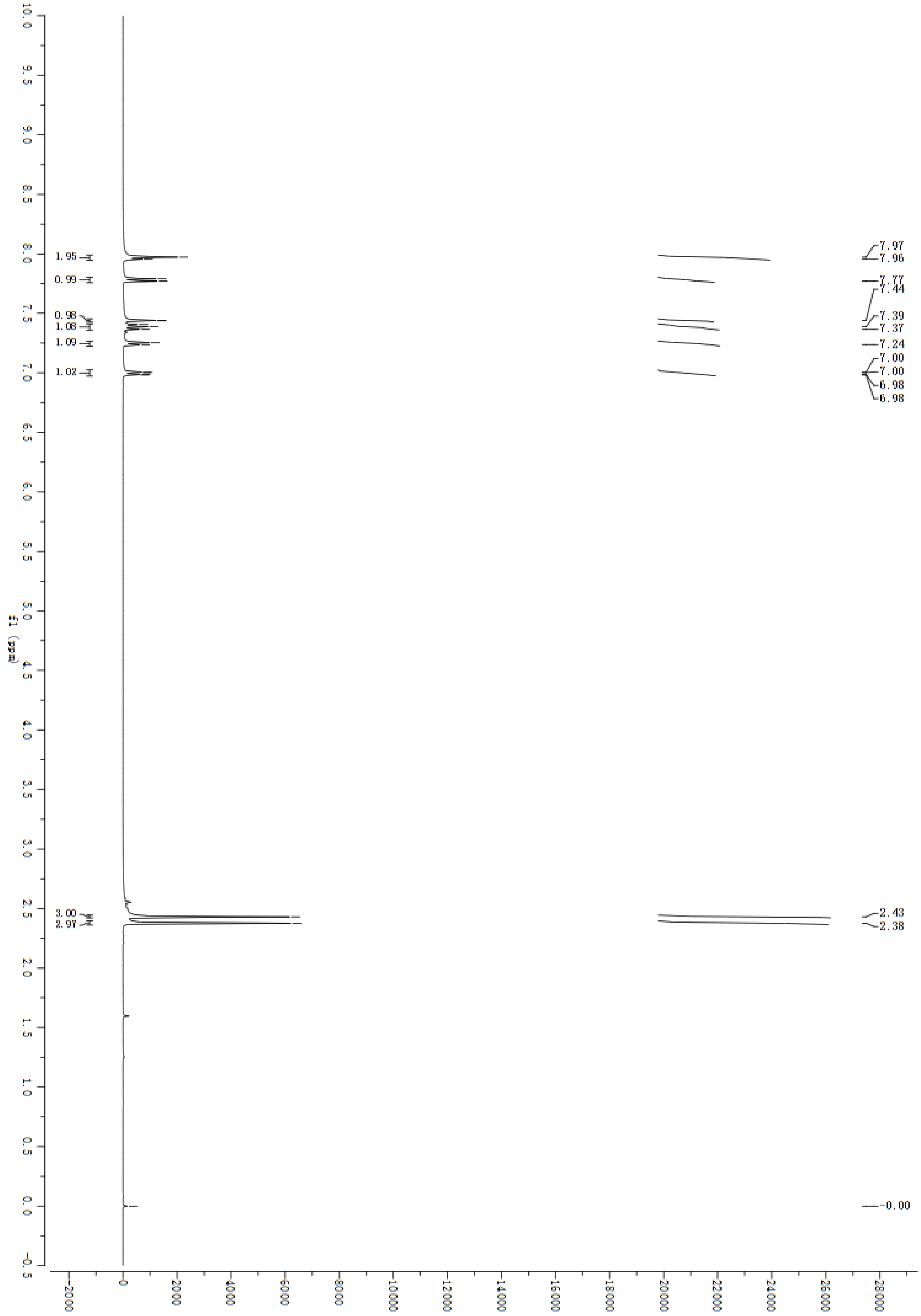
21.61
20.69
f1 (ppm)


O
N
+
N
I


f1 (ppm)
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0.98
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1.09
1.02
3.00
2.97
7.97
7.96
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7.44
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7.37
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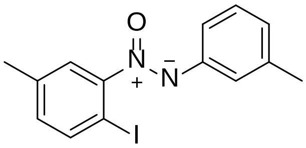
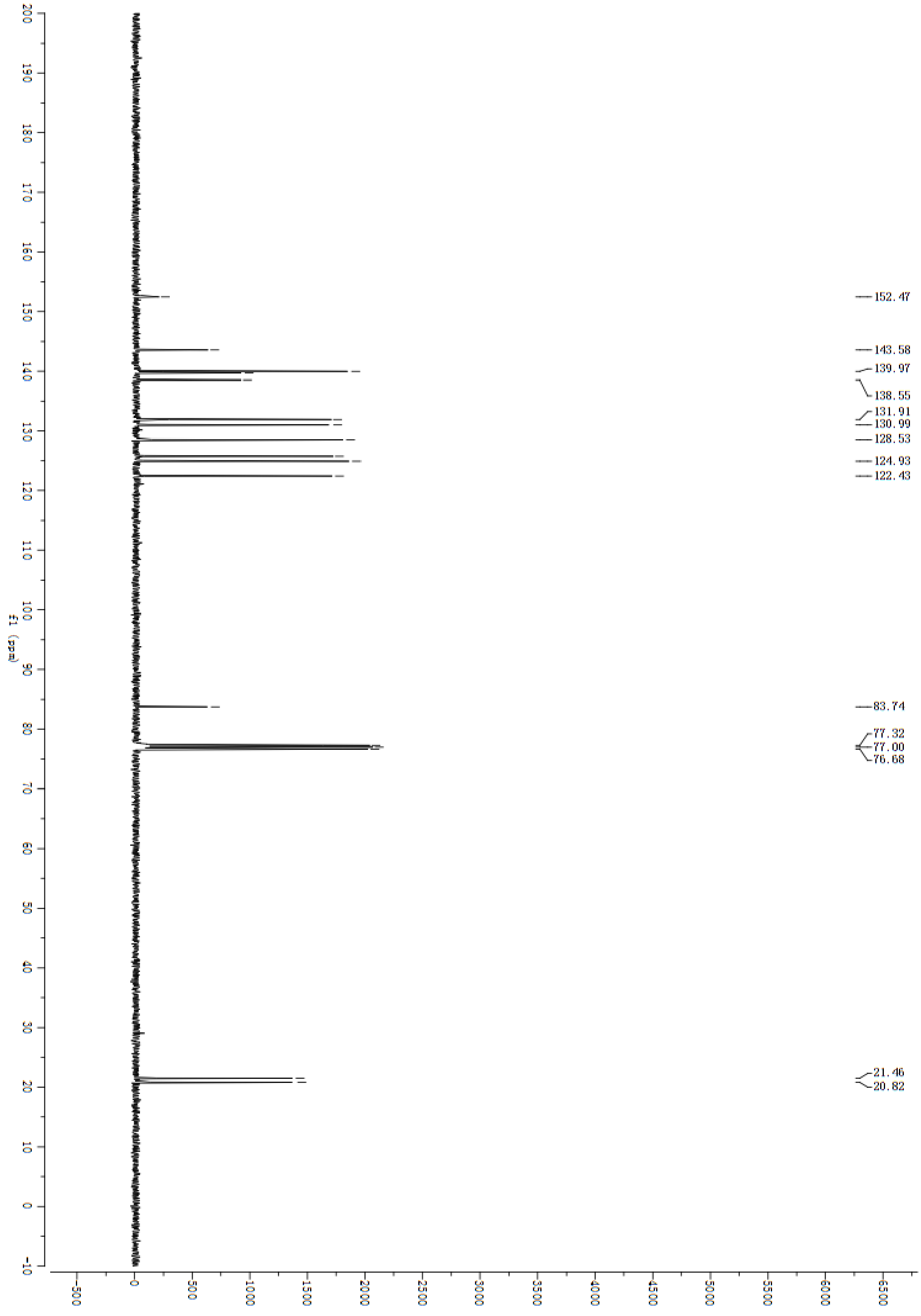
7.00
7.00
6.98
6.98
2.43
2.38
-0.00


O
N
+
N
I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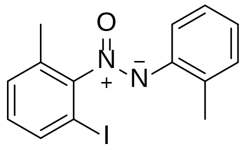
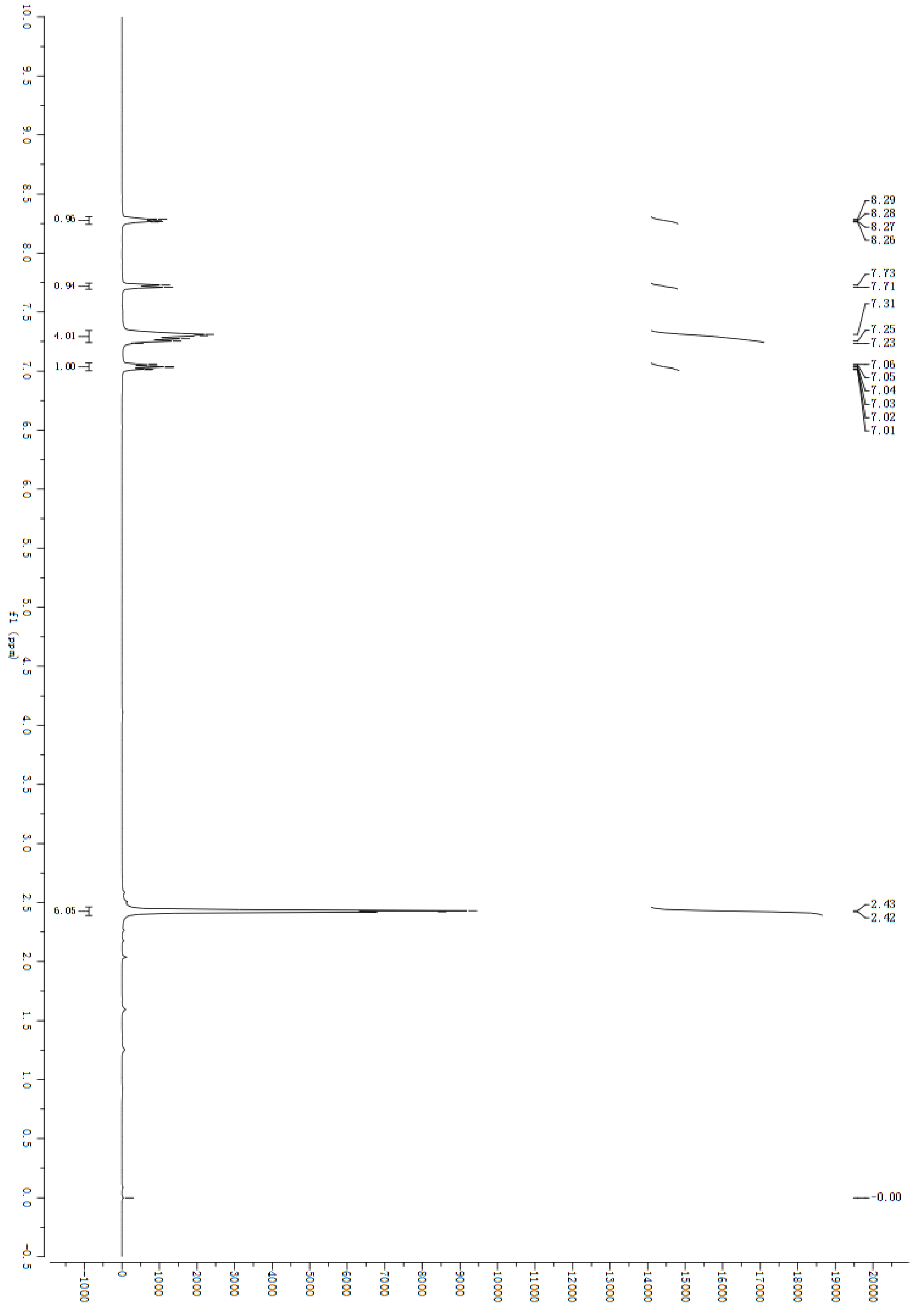
21.46
20.82
f1 (ppm)

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7.25
7.23
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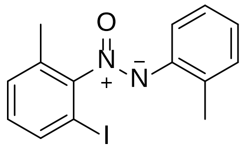
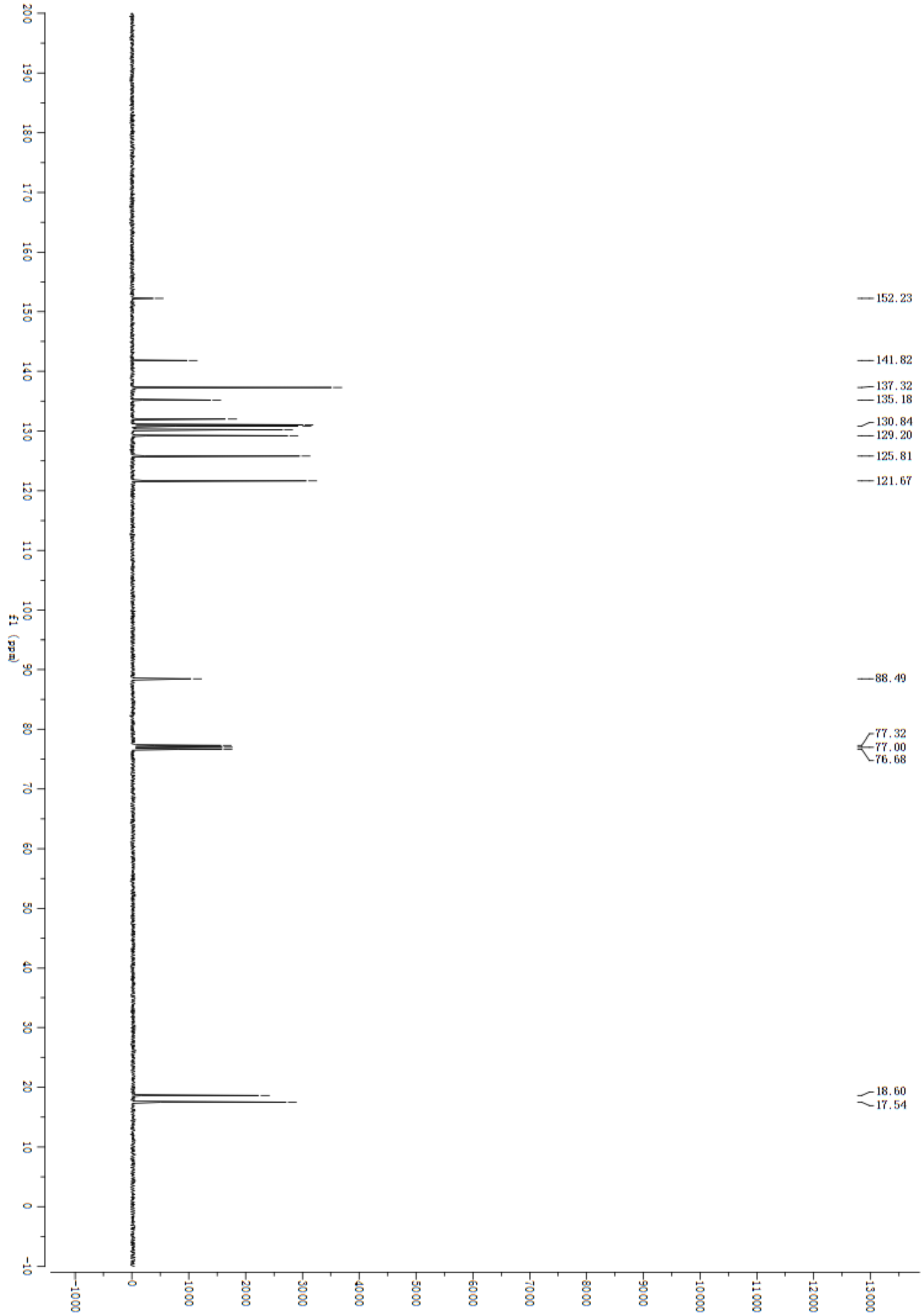
7.05
7.04
7.03
7.02
7.01
2.43
2.42
-0.00
0.96
0.94
4.01
1.00
6.05
f1 (ppm)


O
N
+
N
I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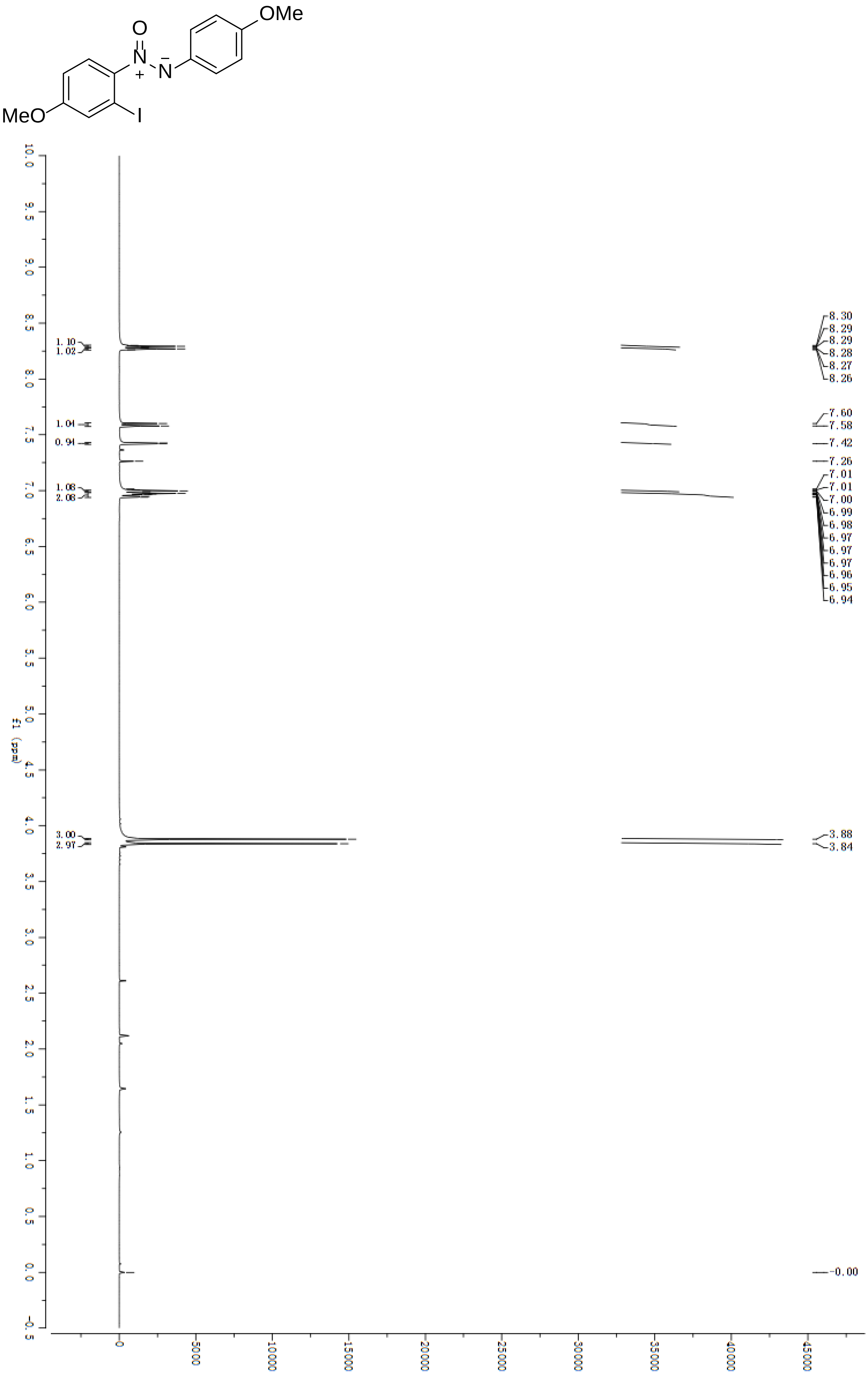
77.32
77.00
76.68
18.60
17.54
f1 (ppm)



OMe
O
N
N
MeO
I
8.30
8.29
8.29
8.28
8.27
8.26
7.60
7.58
7.42
7.26
7.01
7.01
7.00
6.99
6.98
6.97
6.97
6.97
6.96
6.95
6.94
3.88
3.84
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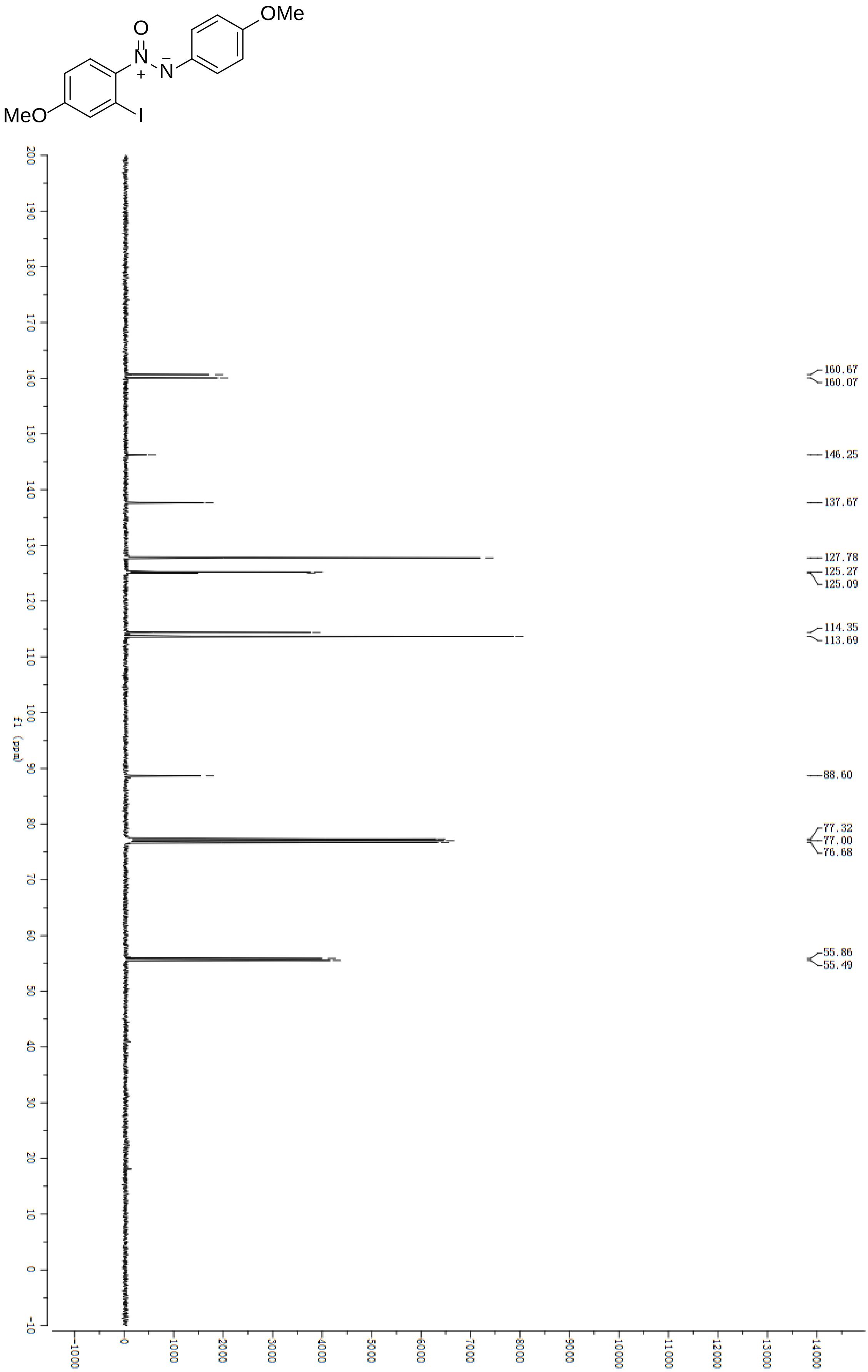
1.10
1.02
1.04
0.94
1.08
2.08
3.00
2.97
f1 (ppm)


OMe
MeO
I
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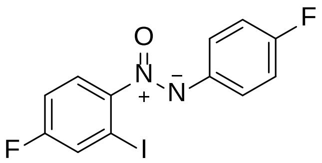
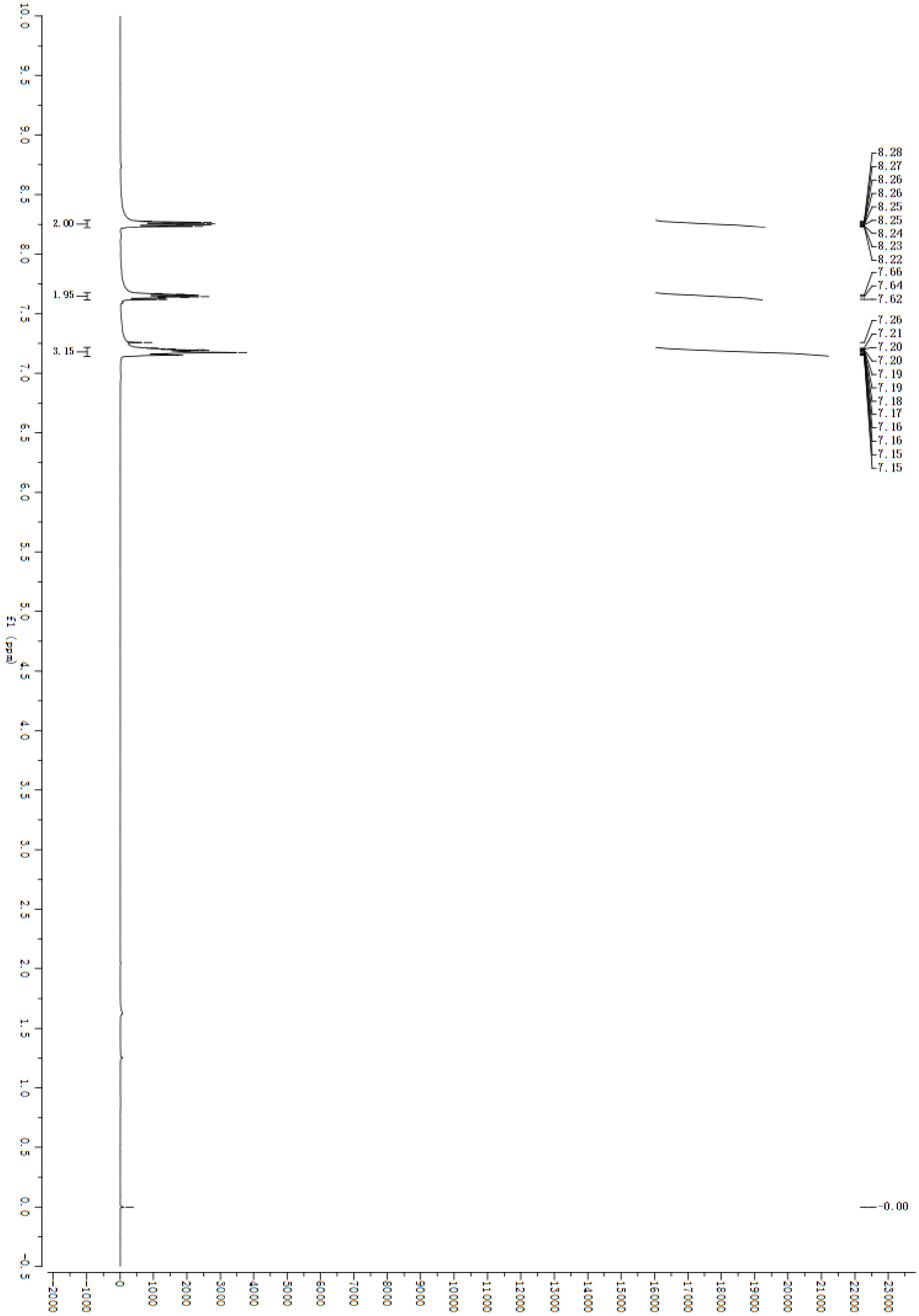
77.32
77.00
76.68
55.86
55.49
f1 (ppm)


O
N
+
–
N
F
F
I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3.15
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8.25
8.25
8.24
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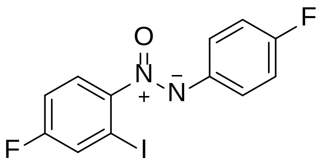
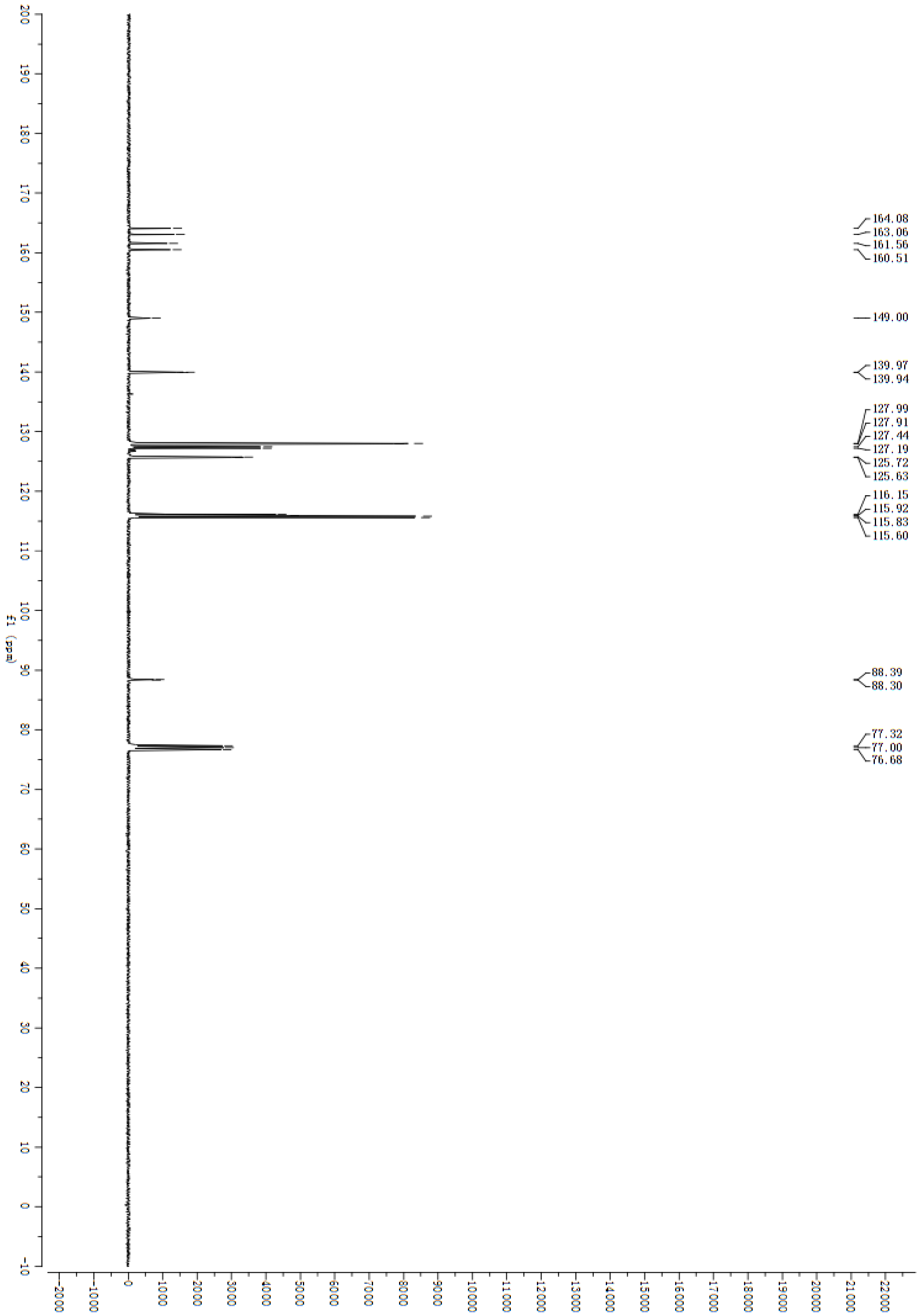
8.22
7.66
7.64
7.62
7.26
7.21
7.20
7.20
7.19
7.19
7.18
7.17
7.16
7.16
7.15
7.15
-0.00
f1 (ppm)





164.08
163.06
161.56
160.51
149.00
139.97
139.94
127.99
127.91
127.44
127.19
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115.92
115.83
115.60
88.39
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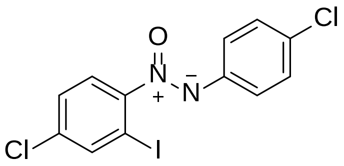
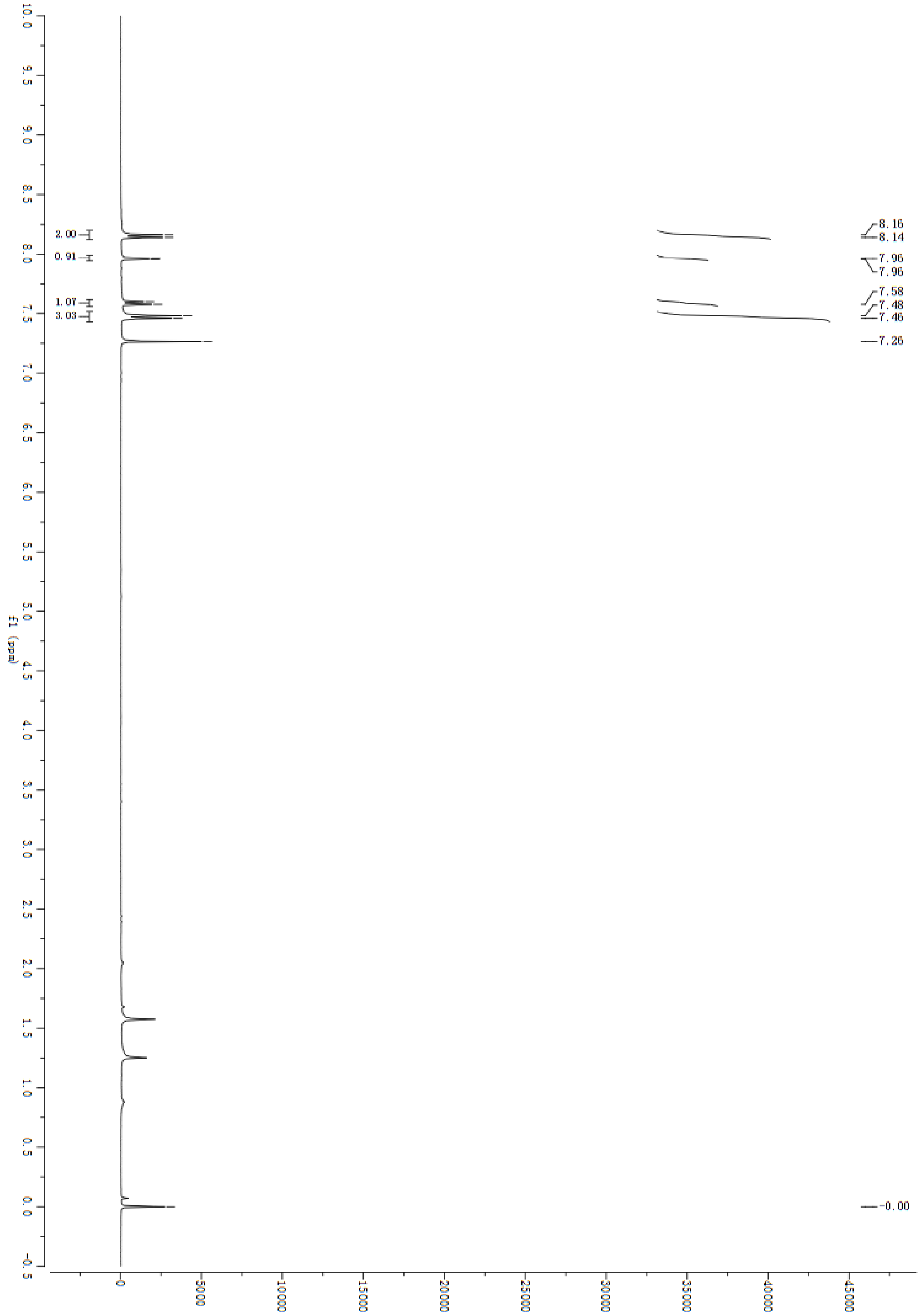
77.00
76.68
f1 (ppm)

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8.14
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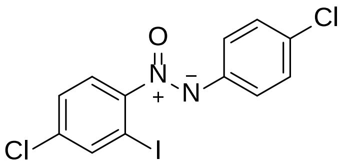
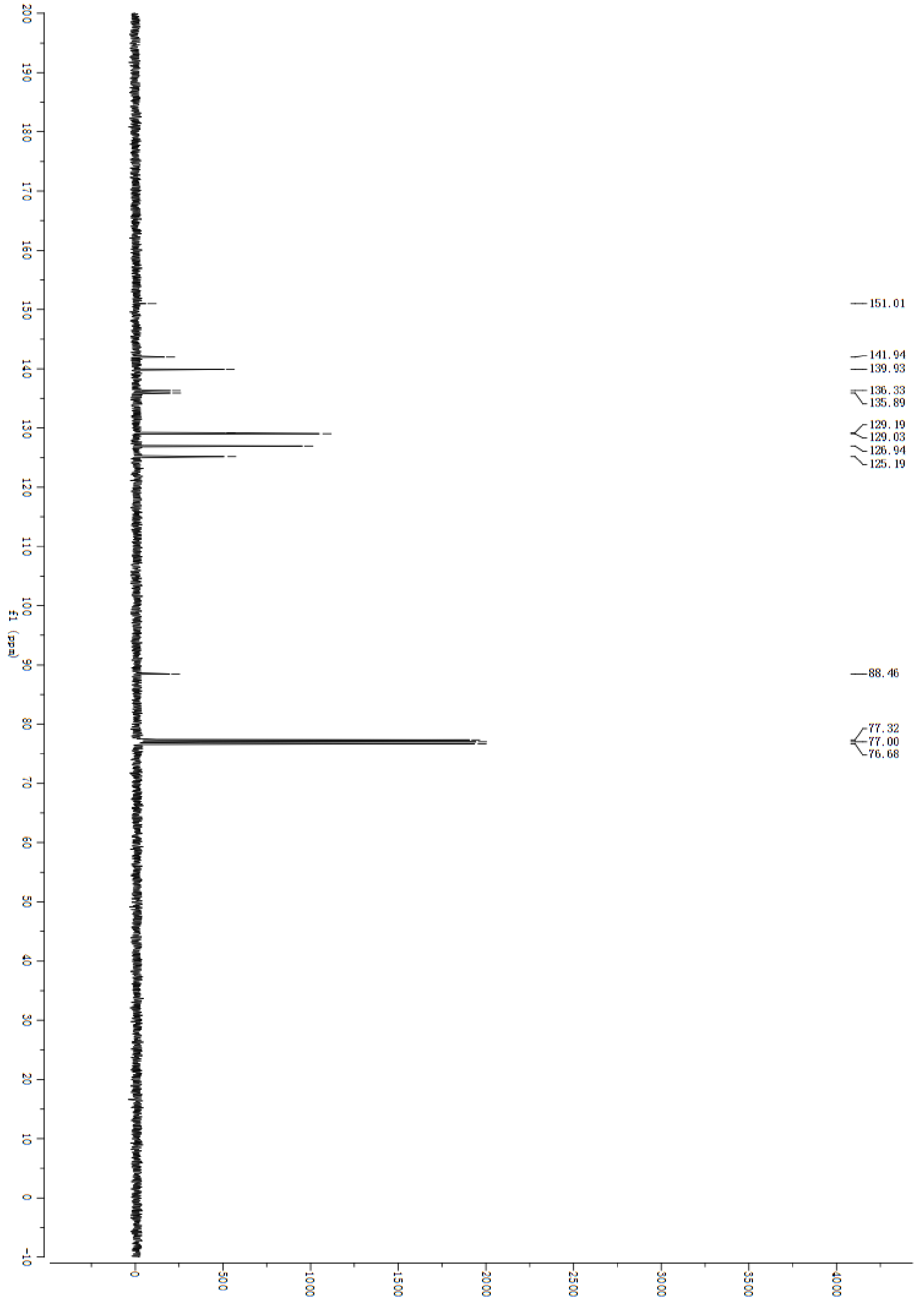
7.96
7.58
7.48
7.46
7.26
-0.00
2.00
0.91
1.07
3.03
f1 (ppm)


O
N
N
Cl
Cl
I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126.94
125.19
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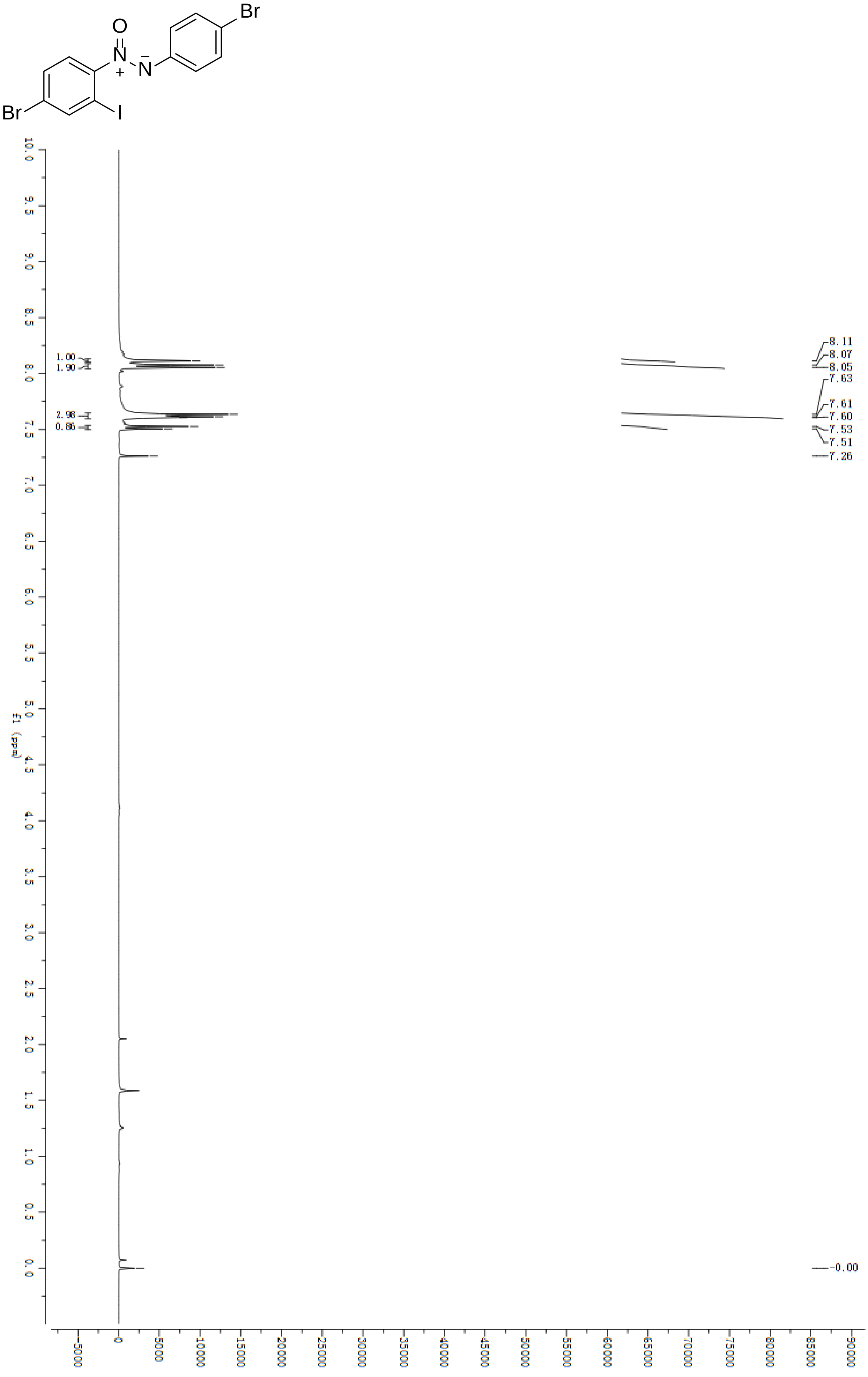
77.00
76.68
f1 (ppm)



Br
O
N
N
Br
I
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2.98
0.86
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8.05
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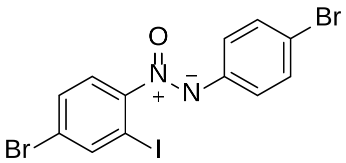
7.61
7.60
7.53
7.51
7.26
-0.00
f1 (ppm)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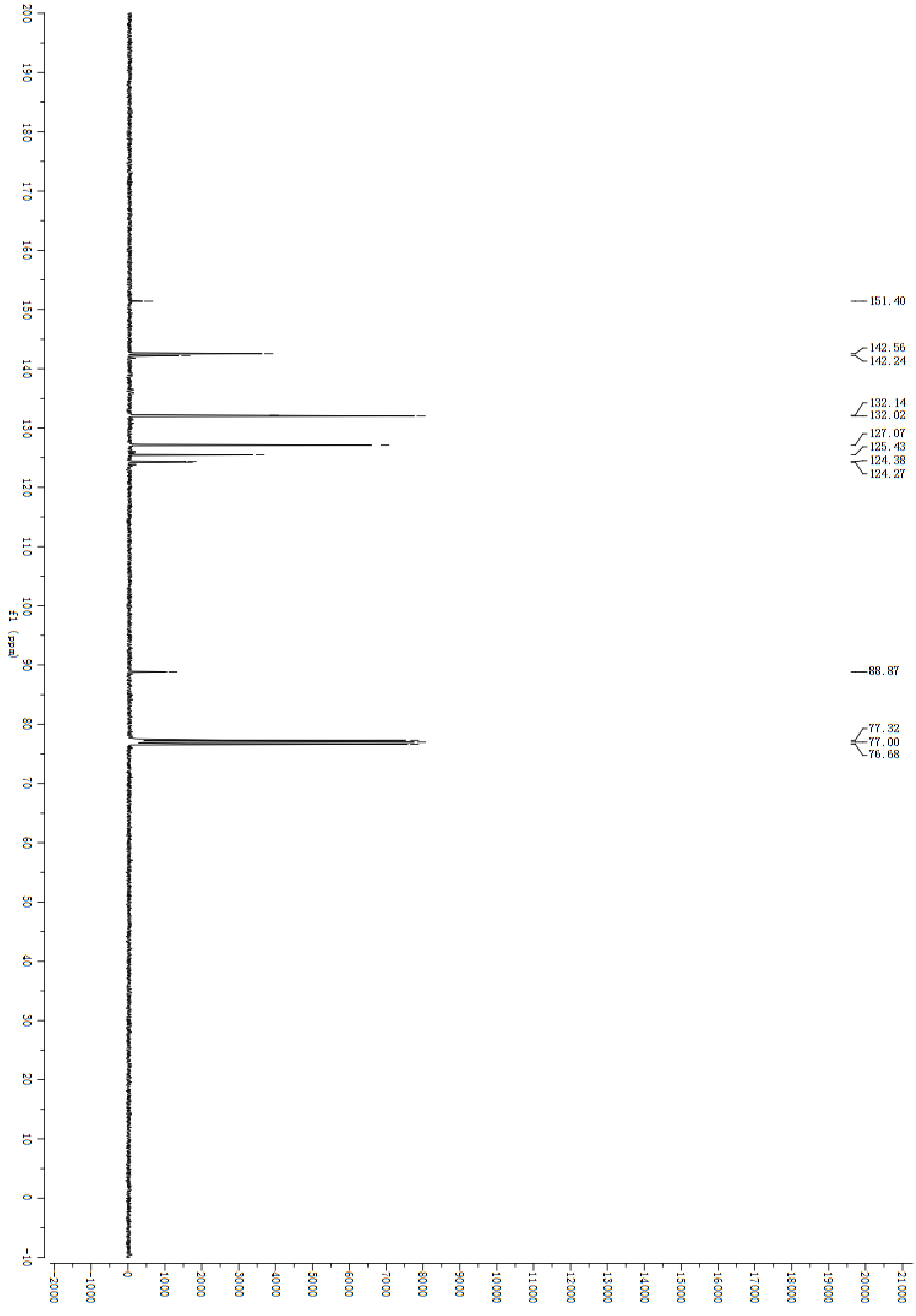
O
N
+
−
N
Br
I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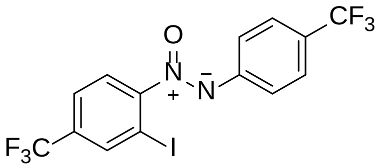
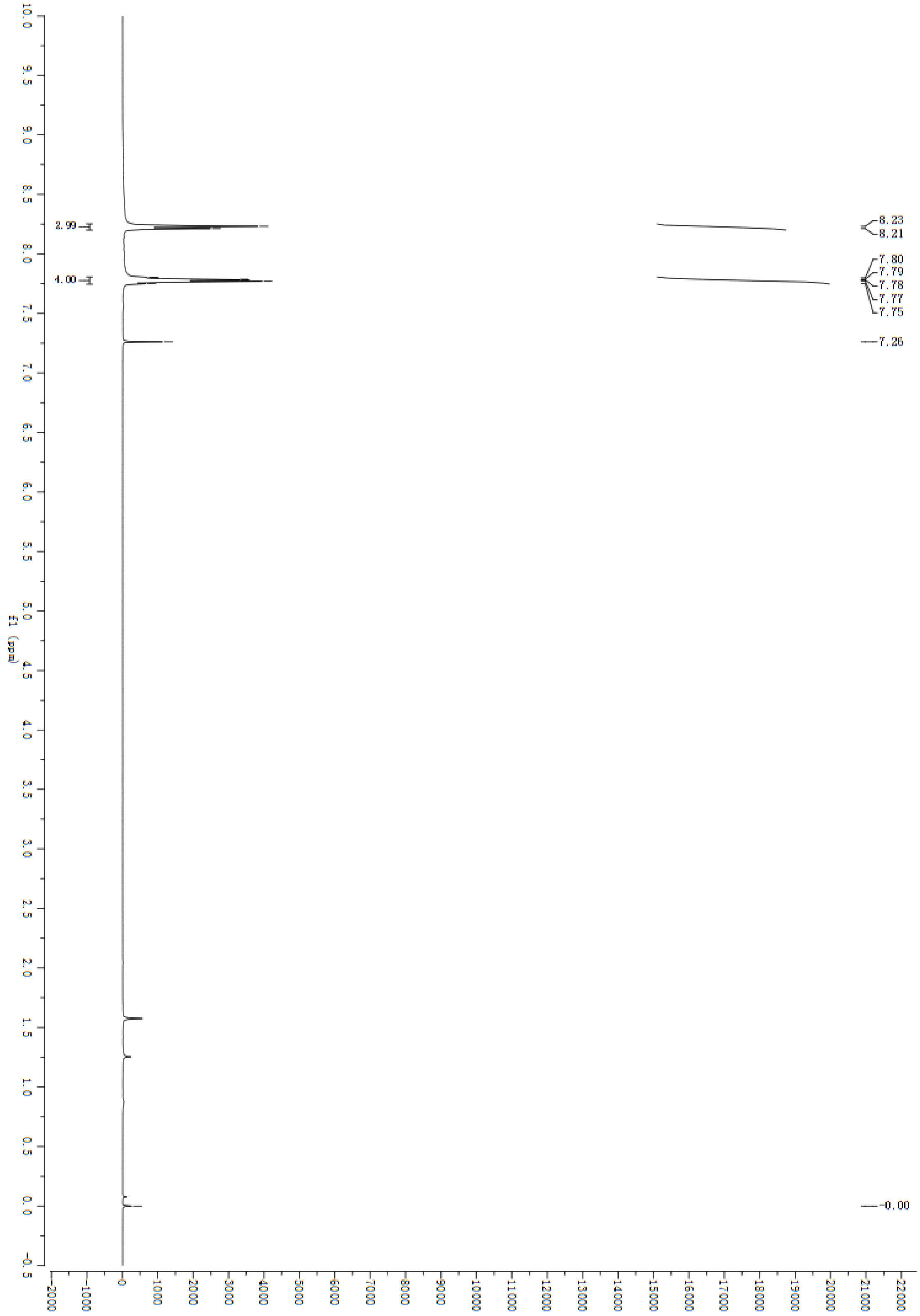
76.68
f1 (ppm)





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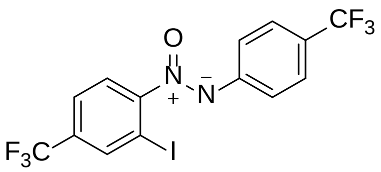
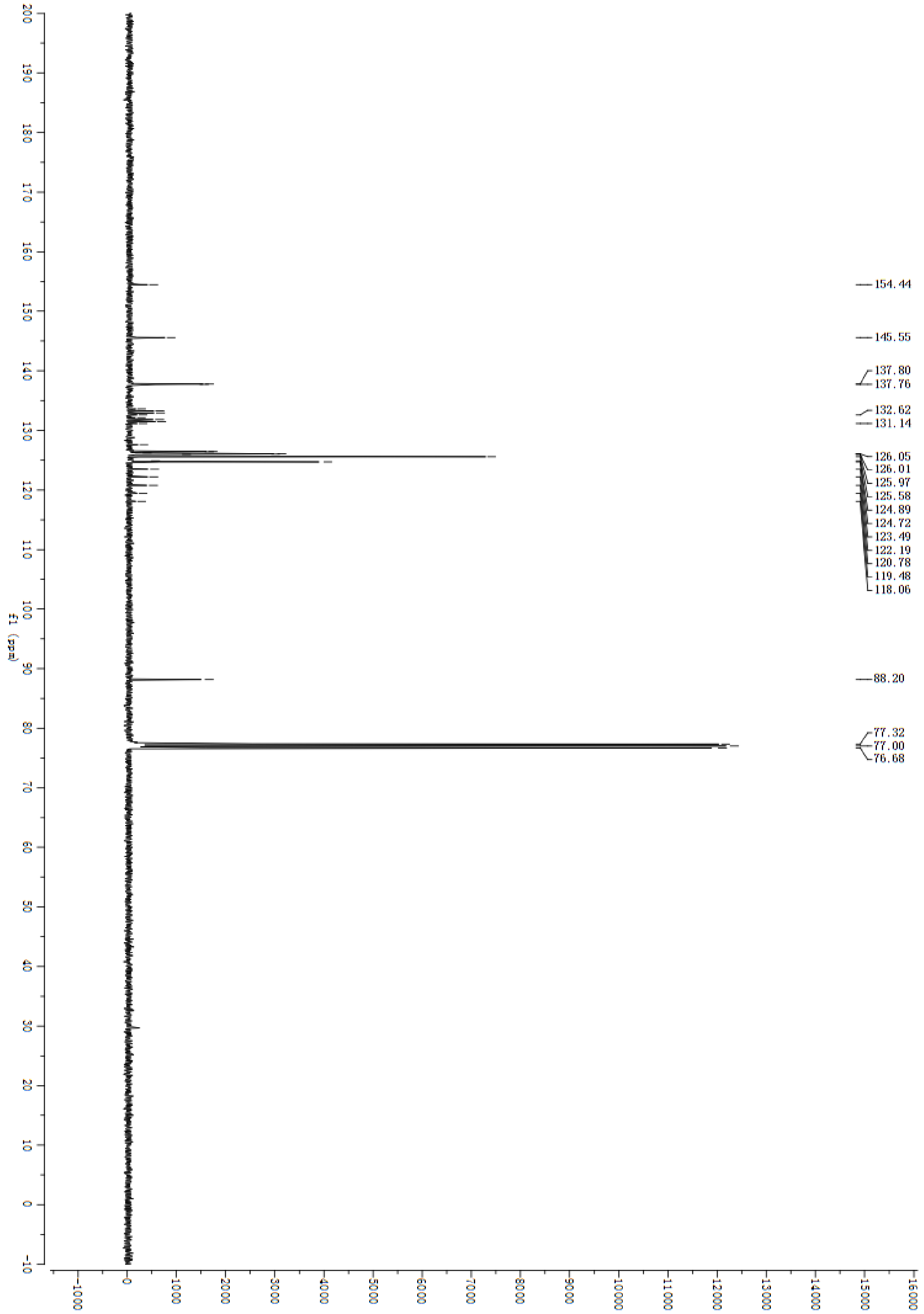
7.78
7.77
7.75
7.26
-0.00
2.99
4.00
f1 (ppm)


O
N
+
N
−
CF3
F3C
I


154.44
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126.01
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125.58
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122.19
120.78
119.48
118.06
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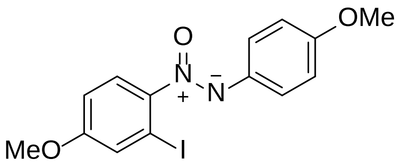
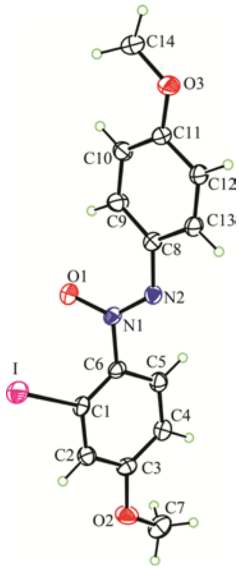
77.32
77.00
76.68
f1 (ppm)



Crystal structure.

X-ray single-crystal for *ortho*-iodinated azoxybenzene **4ea** (CCDC 1054759)



5. References

[1] Christin, G.; Beate, P.; Elisabeth, I.; Karola, R.-B. *Synthesis*, **2008**, 1889.