Supplementary Information (SI) for ChemComm.
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Supplementary Information

Electrochemical Synthesis of Nitrosation Compounds Using CH_3NO_2 as A Nitroso Reagent

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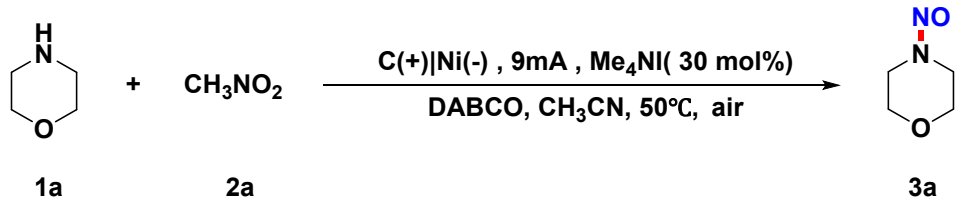
1、General Information

Unless otherwise noted, materials were obtained from commercial suppliers and used without further purification. NMR spectra were recorded on a Bruker AV-500 (^{1}H: 500 MHz, ^{13}C: 125 MHz, ^{19}F NMR: 470 MHz) spectrometer using TMS as internal reference. Chemical shifts (δ) and coupling constants (J) were expressed in ppm and Hz, respectively. GC-MS was Shimadzu QP-5050 GC-MS system. Commercially available compounds were used without further purification. All substances were known available compounds. High resolution mass spectra (HRMS) were measured using electrospray ionization (ESI) and the time-of-flight (TOF) mass analyzer. The anode electrode and cathode electrode are carbon electrode and nickel electrode, respectively. These electrodes are commercially available from GaossUnion, China.

2、Experimenetal Procedure



1a 2a 3a

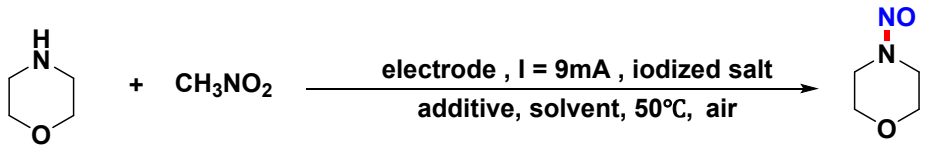
Typical synthesis steps of *N*-nitrosomorpholine (3a): A mixture of morpholine (0.3 mmol), nitromethane (0.45 mmol), Me_4NI(0.09 mmol) and DABCO (0.2 mmol) and CH_3CN = 6 ml were added to an undivided electrolytic cell. The electrolytic cell was equipped with a carbon electrode as anode and a nickel electrode as cathode. The reaction mixture was stirred and electrolyzed at a constant current of 9 mA under 50℃ for corresponding time. When the reaction was finished, the solution was extracted with EtOAc (3×10 mL). The combined organic layer was dried with Na_2SO_4, filtered. The solvent was removed with a rotary evaporator. The residue was purified by column chromatography on silica gel (PE/EtOAc = 3:1) to afford the desired product.

Gram-scale synthesis of *N*-nitrosomorpholine (3a): A mixture of morpholine (10 mmol), nitromethane (15 mmol), Me_4NI(3 mmol) and DABCO (6 mmol) and CH_3CN = 200 ml were added to an undivided electrolytic cell. The electrolytic cell was equipped with a carbon electrode as anode and a nickel electrode as cathode. The reaction mixture was stirred and electrolyzed at a constant current of 27 mA under 50℃ for 29 h. When the reaction was completed, the solution was extracted with EtOAc (3×100 mL). The combined organic layer was dried with Na_2SO_4, filtered. The

solvent was removed with a rotary evaporator. The residue was purified by column chromatography on silica gel (PE/EtOAc = 3:1) to afford the desired product.

3、Optimization of Reaction Conditions

(1) Table S1. Optimization of reaction conditions for nitrosation of secondary amine.[a]



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

entry	electrode	electrolyte	solvent	additive	Yield(%)[b]
1	Pt(+)|C(-)	KI	CH_3CN	\	67
2	Pt(+)|C(-)	**Me_4NI**	CH_3CN	\	75
3	Pt(+)|C(-)	TBAI	CH_3CN	\	63
4	C(+)|C(-)	Me_4NI	CH_3CN	\	72
5	C(+)|Pt(-)	Me_4NI	CH_3CN	\	65
6	**C(+)|Ni(-)**	Me_4NI	CH_3CN	\	81
7	C(+)|Ni(-)	Me_4NI	DCM	\	38
8	C(+)|Ni(-)	Me_4NI	THF	\	46
9	C(+)|Ni(-)	Me_4NI	DMF	\	76
10	**C(+)|Ni(-)**	**Me_4NI**	**CH_3CN**	**DABCO**	**94**
11	C(+)|Ni(-)	Me_4NI	CH_3CN	DMAP	75
12	C(+)|Ni(-)	Me_4NI	CH_3CN	KOH	78
13	C(+)|Ni(-)	Me_4NI	CH_3CN	K_2CO_3	86
14	C(+)|Ni(-)	Me_4NI	CH_3CN	TFEA	88
15[c]	C(+)|Ni(-)	Me_4NI	CH_3CN	DABCO	56
16[d]	C(+)|Ni(-)	Me_4NI	CH_3CN	DABCO	54
17	C(+)|Ni(-)	\	CH_3CN	DABCO	trace
18[e]	C(+)|Ni(-)	Me_4NI	CH_3CN	DABCO	trace

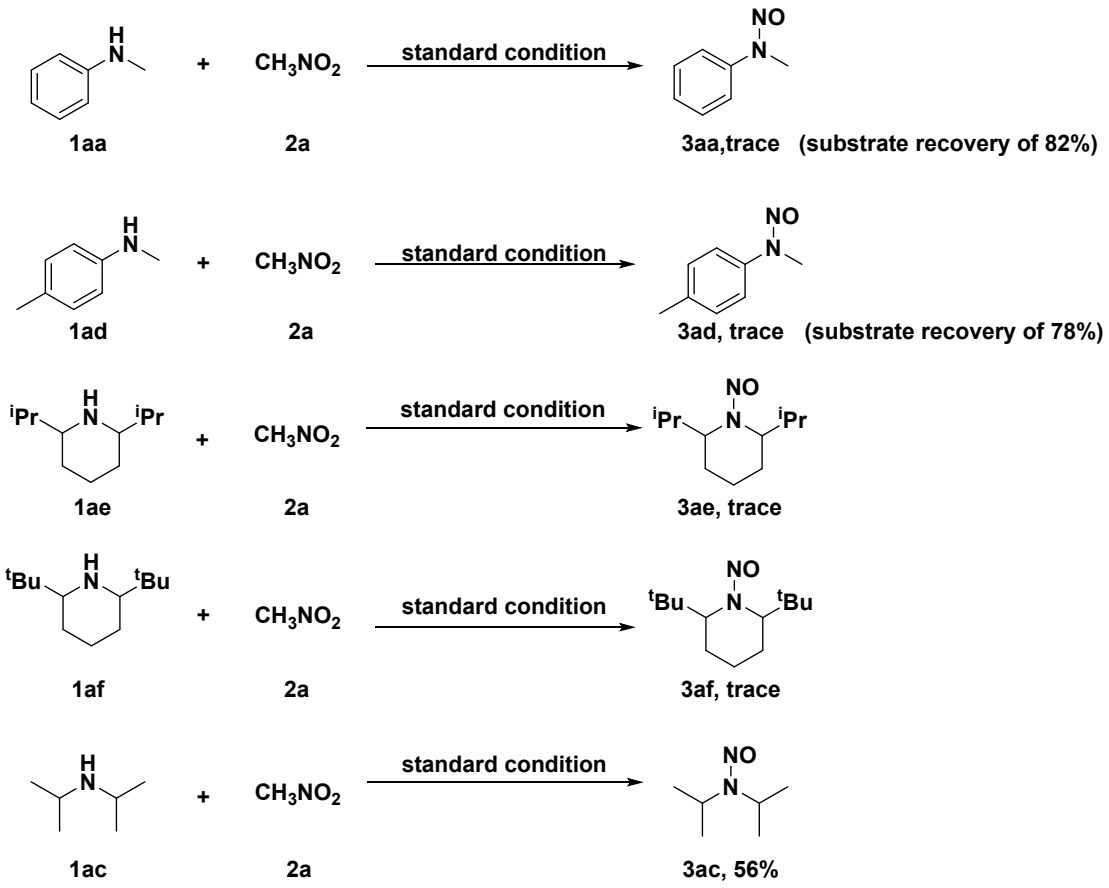
[a] Reaction conditions: **1a** (0.3 mmol), **2a** (0.45 mmol), iodized salt (0.09 mmol), additive (0.2 mmol), solvent (6 mL), undivided cell, 9 mA, 6 h, 50 °C, air, 6.7F [b] Yield of isolated product. [c/d] the reaction temperature is 25℃/ 70℃. [e] Without electricity.

(2) Calculation of Faraday Efficiency in Model Reaction

= (m*n*F) / (I*t) = (0.3*0.94*2*96485) / (9*6*60*60) = 28%

4、Scope of Amines for N-Nitrosation

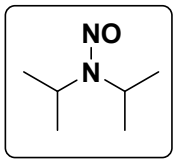
（1）Scheme S2: Partial substrate expansion.



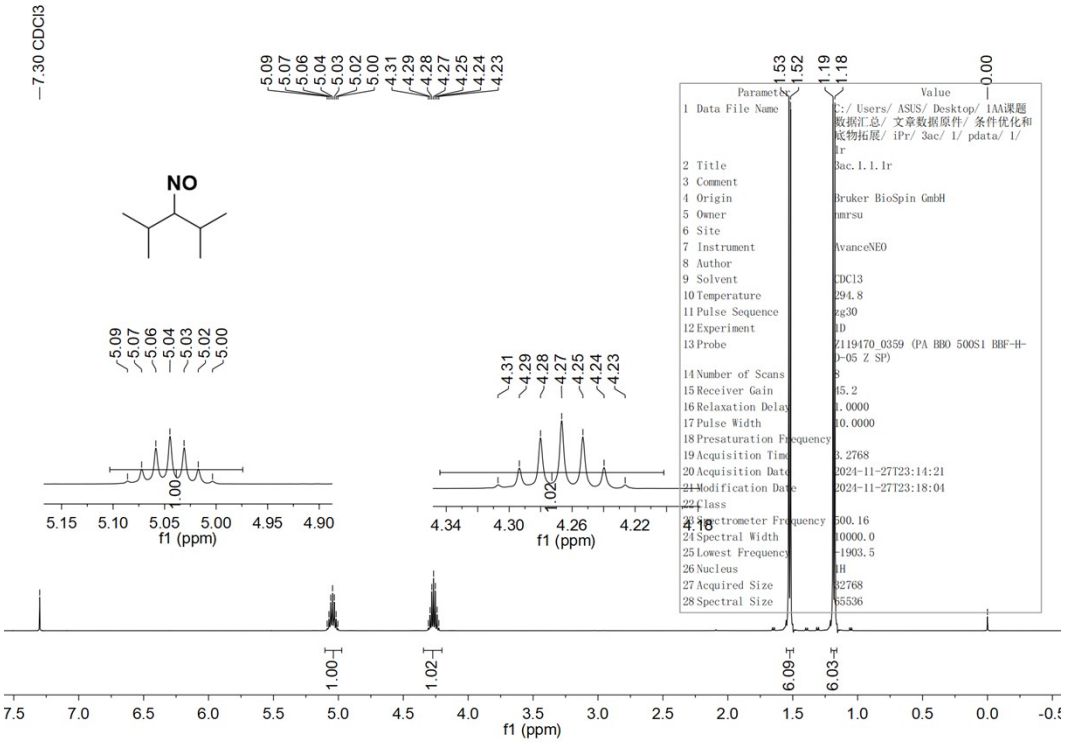
In the study of the substrate applicability range of secondary amines, the reactivity of some aromatic secondary amines is poor, such as **3aa** and **3ad**, which did not obtain the target product under standard conditions, but obtained the corresponding raw materials with high recovery rates. At the same time, when we increase the steric hindrance of the substrate, the change in yield is more significant. When the substituents are isopropyl and tert butyl, the reaction is almost impossible to proceed **(3ae-3af)**. When the substituent of the chain like secondary amine is changed to isopropyl, the target product (**3ac**) is obtained with a yield of 56%.

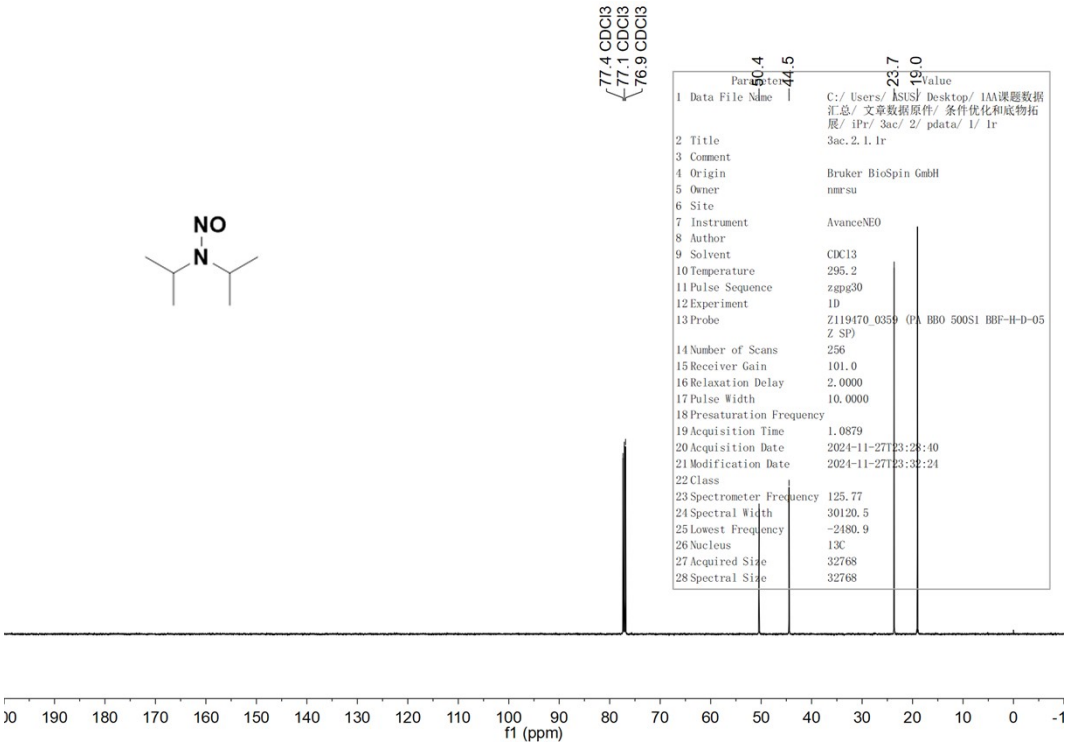
（2）NMR data and spectra of 3ac

N,N-diisopropylnitrous amide (3ac)



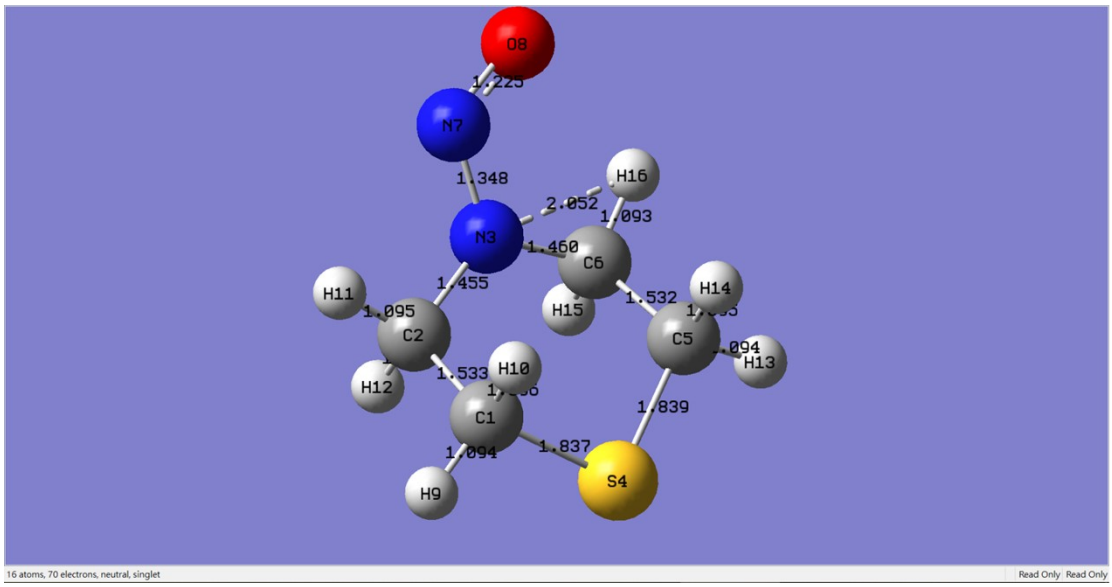
The title compound was prepared according to the general working procedure and purified by column chromatography (petroleum ether/ethyl acetate = 8:1) to give the product as a yellow oil. 56% yield, 21.8mg. ^{1}H NMR (500 MHz, Chloroform-*d*) δ 5.04 (hept, *J* = 6.9 Hz, 1H), 4.27 (hept, *J* = 6.8 Hz, 1H), 1.52 (d, *J* = 6.8 Hz, 6H), 1.18 (d, *J* = 6.9 Hz, 6H). ^{13}C NMR (125 MHz, Chloroform-*d*) δ 50.4, 44.5, 23.7, 19.0.



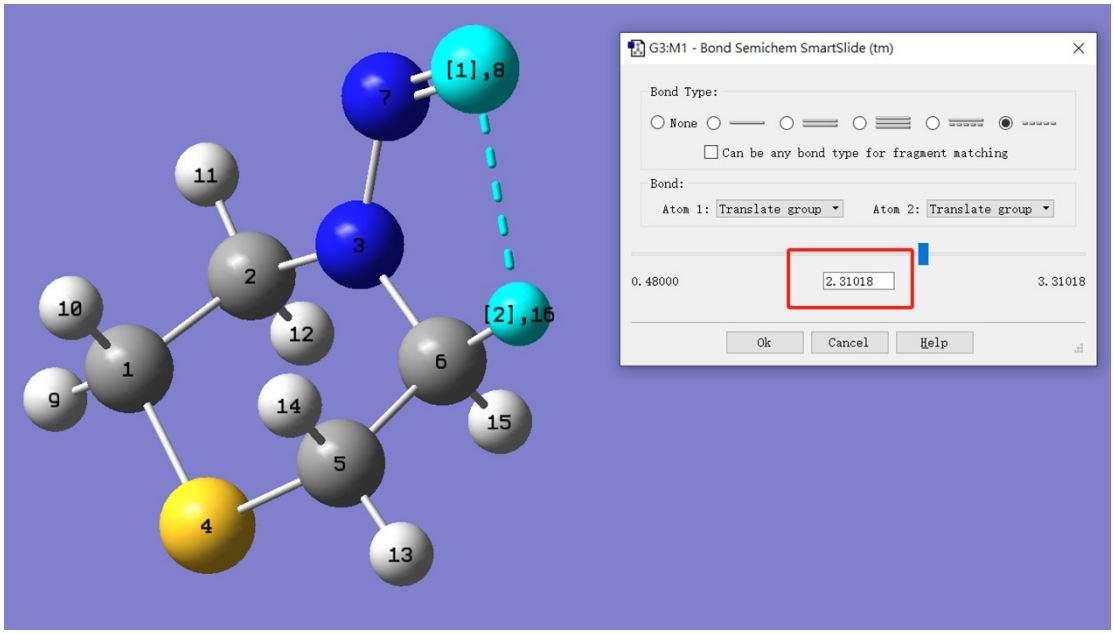


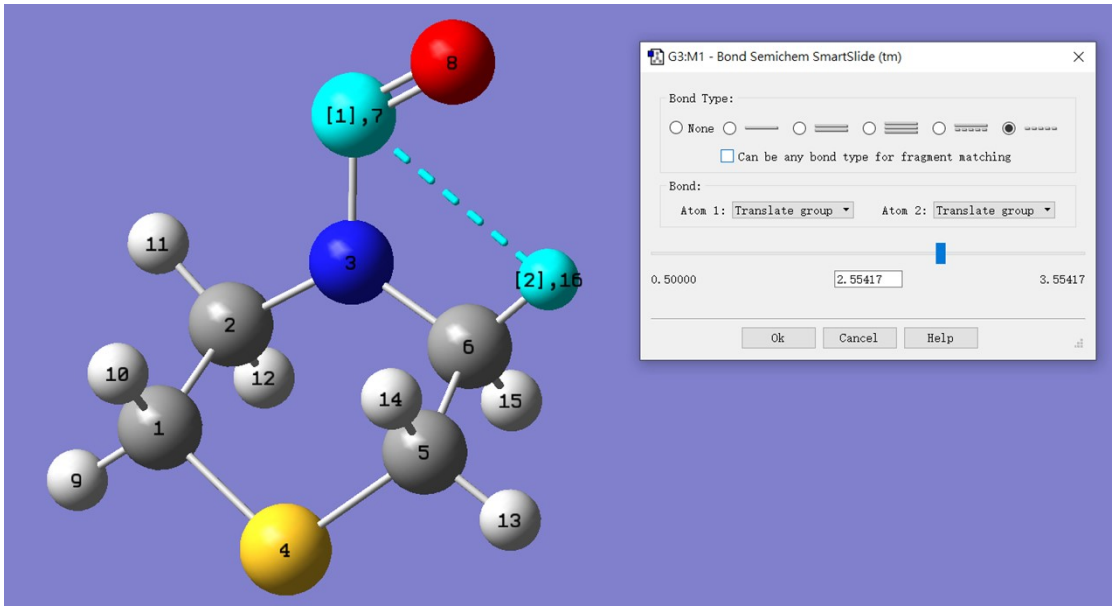
（3）DFT calculations (Gaussian 16W) of 3e.

(a) Fig. S3: Stereoscopic configuration diagram of 3e.



(b) Fig. S4: The formation of the hydrogen bong of *N-N=O* group with the adjacen Hydrogens.





(c) Scheme S3: All data on distances between atoms.

Distance matrix (angstroms):

		1	2	3	4	5
1	C	0.000000				
2	C	1.532676	0.000000			
3	N	2.472299	1.454688	0.000000		

4	S	1.836879	2.800897	3.147151	0.000000	
5	C	2.757362	3.047430	2.471056	1.838590	0.000000
6	C	3.055601	2.525590	1.460338	2.806674	1.531831
7	N	3.446502	2.381320	1.347716	4.374917	3.513576
8	O	4.429463	3.488967	2.181203	5.039068	3.769282
9	H	1.094097	2.168130	3.411641	2.387513	3.725950
10	H	1.095647	2.169947	2.772015	2.434574	2.994813
11	H	2.176178	1.094516	2.051601	3.774074	4.005325
12	H	2.166345	1.097513	2.092115	3.023157	3.472848
13	H	3.724790	4.035018	3.412031	2.387280	1.094077
14	H	2.994497	3.435700	2.772917	2.434165	1.095278
15	H	3.478781	2.818785	2.089495	3.032393	2.170300
16	H	4.008520	3.397491	2.052231	3.779519	2.178578

		6	7	8	9	10
6	C	0.000000				
7	N	2.473177	0.000000			
8	O	2.689200	1.225110	0.000000		
9	H	4.042953	4.339651	5.408428	0.000000	
10	H	3.441522	3.416595	4.320962	1.774779	0.000000
11	H	3.402417	2.366618	3.589400	2.552128	2.478575
12	H	2.824994	3.111932	4.142225	2.469004	3.073425
13	H	2.166847	4.430671	4.499523	4.599093	4.012458
14	H	2.170135	3.488358	3.628012	4.013241	2.817158
15	H	1.096750	3.179706	3.444993	4.295059	4.124398
16	H	1.092970	2.507965	2.194382	5.041563	4.207651

		11	12	13	14	15
11	H	0.000000				
12	H	1.783259	0.000000			
13	H	5.038400	4.289119	0.000000		
14	H	4.207947	4.120646	1.772948	0.000000	
15	H	3.761553	2.676366	2.476002	3.076539	0.000000
16	H	4.103370	3.765947	2.558515	2.479559	1.785354

		16
16	H	0.000000

Distance matrix (angstroms):

		1	2	3	4	5
1	C	0.000000				
2	C	1.532676	0.000000			
3	N	2.472299	1.454688	0.000000		
4	S	1.836879	2.800897	3.147151	0.000000	
5	C	2.757362	3.047430	2.471056	1.838590	0.000000
6	C	3.055601	2.525590	1.460338	2.806674	1.531831
7	N	3.446502	2.381320	1.347716	4.374917	3.513576
8	O	4.429463	3.488967	2.181203	5.039068	3.769282
9	H	1.094097	2.168130	3.411641	2.387513	3.725950
10	H	1.095647	2.169947	2.772015	2.434574	2.994813
11	H	2.176178	1.094516	2.051601	3.774074	4.005325
12	H	2.166345	1.097513	2.092115	3.023157	3.472848
13	H	3.724790	4.035018	3.412031	2.387280	1.094077
14	H	2.994497	3.435700	2.772917	2.434165	1.095278
15	H	3.478781	2.818785	2.089495	3.032393	2.170300
16	H	4.008520	3.397491	2.052231	3.779519	2.178578

N3-H16

		6	7	8	9	10
6	C	0.000000				
7	N	2.473177	0.000000			
8	O	2.689200	1.225110	0.000000		
9	H	4.042953	4.339651	5.408428	0.000000	
10	H	3.441522	3.416595	4.320962	1.774779	0.000000
11	H	3.402417	2.366618	3.589400	2.552128	2.478575
12	H	2.824994	3.111932	4.142225	2.469004	3.073425
13	H	2.166847	4.430671	4.499523	4.599093	4.012458
14	H	2.170135	3.488358	3.628012	4.013241	2.817158
15	H	1.096750	3.179706	3.444993	4.295059	4.124398
16	H	1.092970	2.507965	2.194382	5.041563	4.207651

N7-H16 O8-H16

		11	12	13	14	15
11	H	0.000000				
12	H	1.783259	0.000000			

5、Mechanistic Experiments

Cyclic Voltammetry Studies

Cyclic voltammetry data were measured with a Shanghai Chenhua potentiostat (CHI760E).

Working electrode: The working electrode is a 3 mm diameter Pt disk working electrode. Polished with 0.3 μm aluminum oxide and then sonicated in distilled water before drying.

Reference electrode: The reference electrode consisted of a silver wire covered with silver chloride immersed in a saturated solution of potassium chloride.

Counter electrode: The counter electrode is a platinum wire that was polished with sand paper.

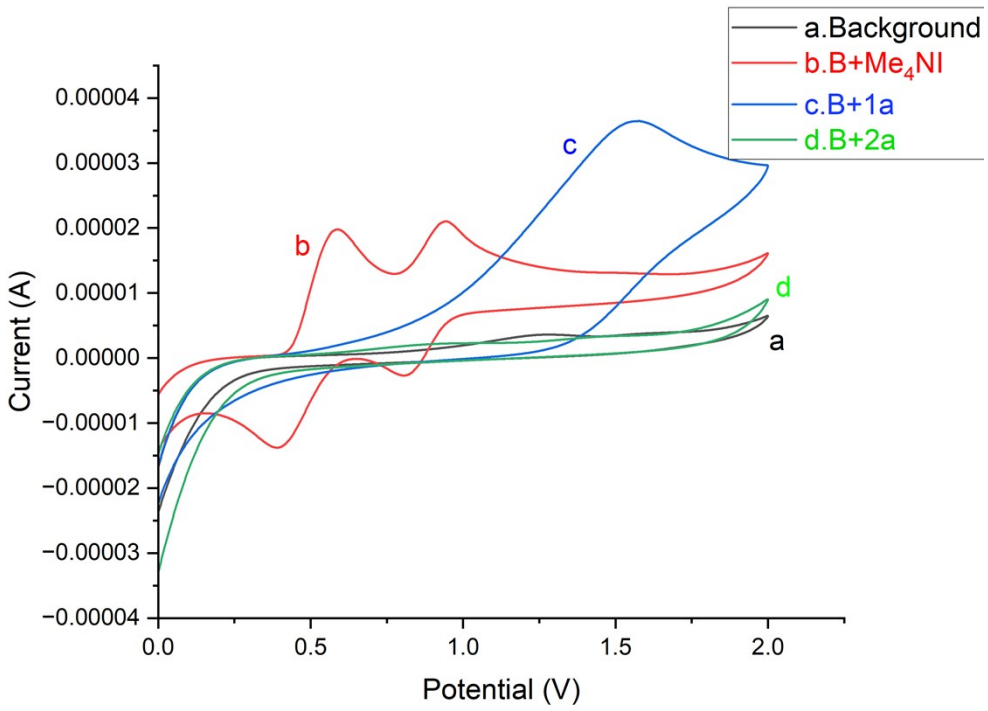
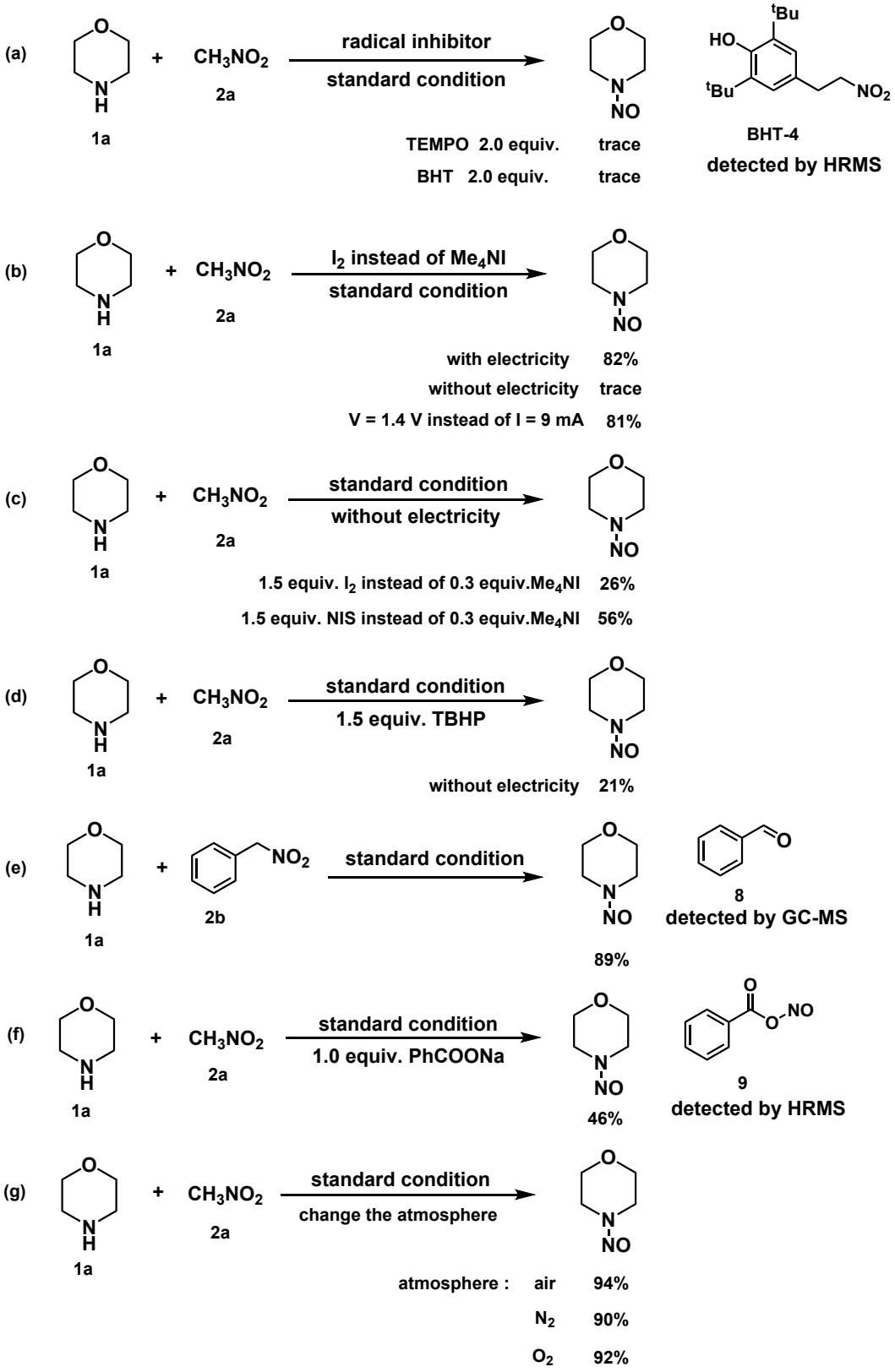


Fig 2. Cyclic voltammograms of **1a, 2a** and Me_4NI in 0.1 M NH_4BF_4/CH_3CN using a Pt disk as the working electrode, and Pt wire and Ag/AgCl as the counter and reference electrodes, respectively, at a scan rate of 100 mV/s: background (curve **a**), Me_4NI (0.002 M) (curve **b**), **1a** (0.005 M) (curve **c**) and **2a** (0.005 M) (curve **d**).

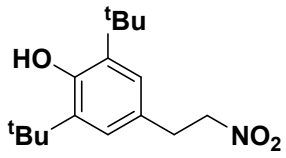
6 Control Experiments

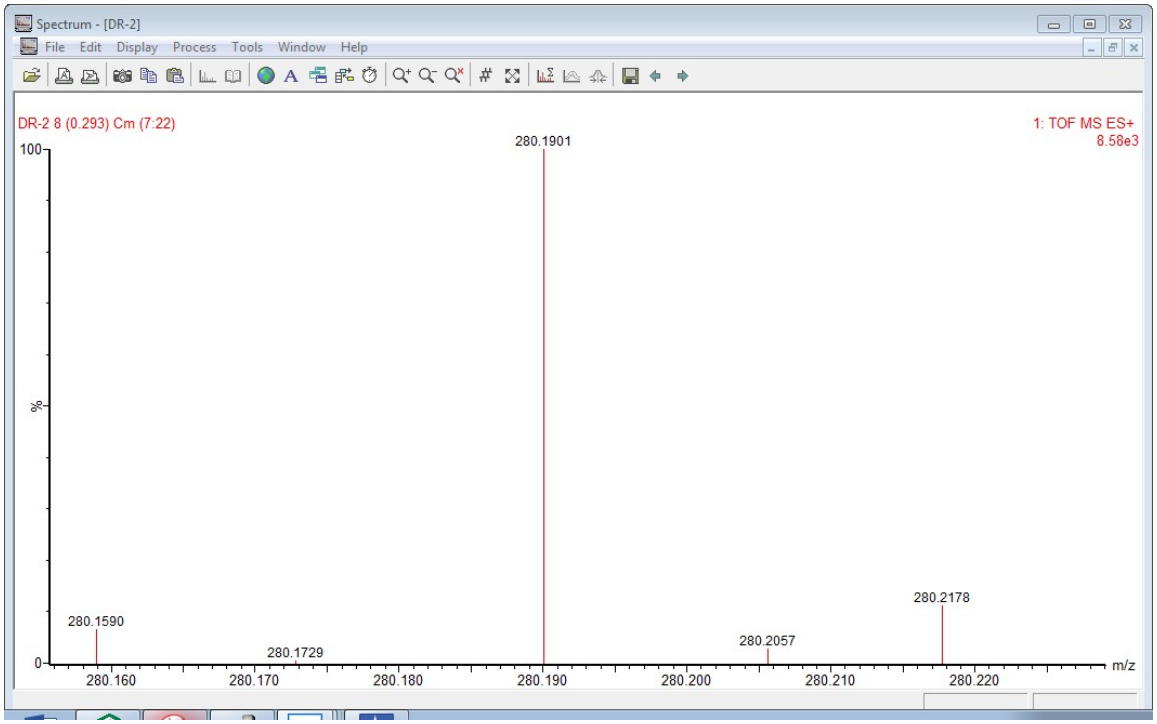
Scheme S4: Control experiments.



Scheme S4a: for BHT-4: A mixture of morpholine (0.3 mmol), nitromethane (0.45 mmol), Me_4NI (0.09 mmol) and DABCO (0.2 mmol), butylated hydroxytoluene (BHT, 0.6 mmol) and CH_3CN = 6 mL were added to an undivided cell. The cell was equipped with a carbon electrode as anode and a nickel electrode as cathode. The reaction mixture was stirred and electrolyzed at a constant current of 9 mA under 50℃ for corresponding time.

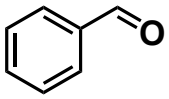
HRMS (ESI) m/z calcd for $C_{16}H_{25}NO_2[M+H]^+$ 280.1907, found 280.1901.

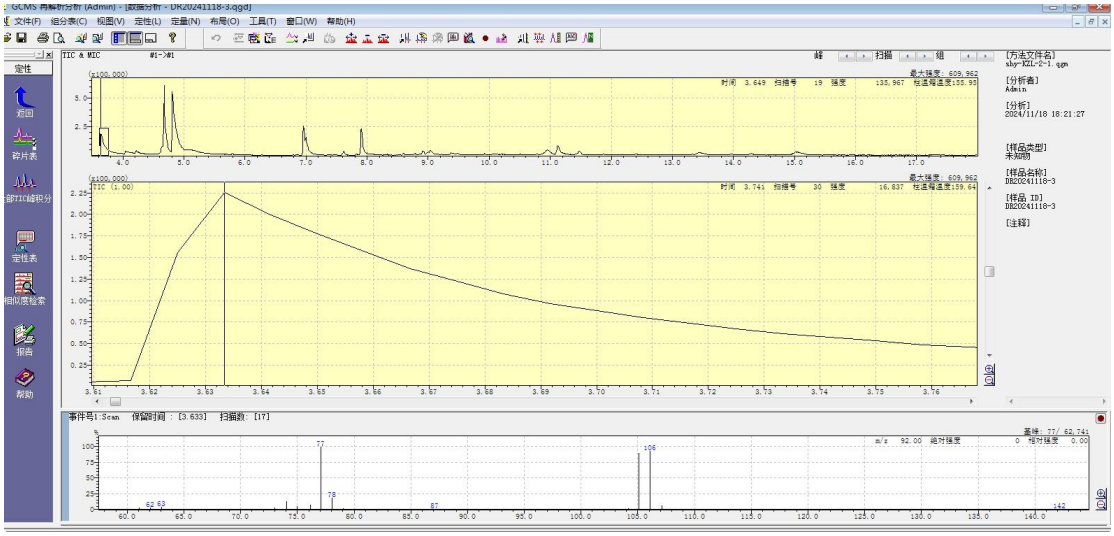


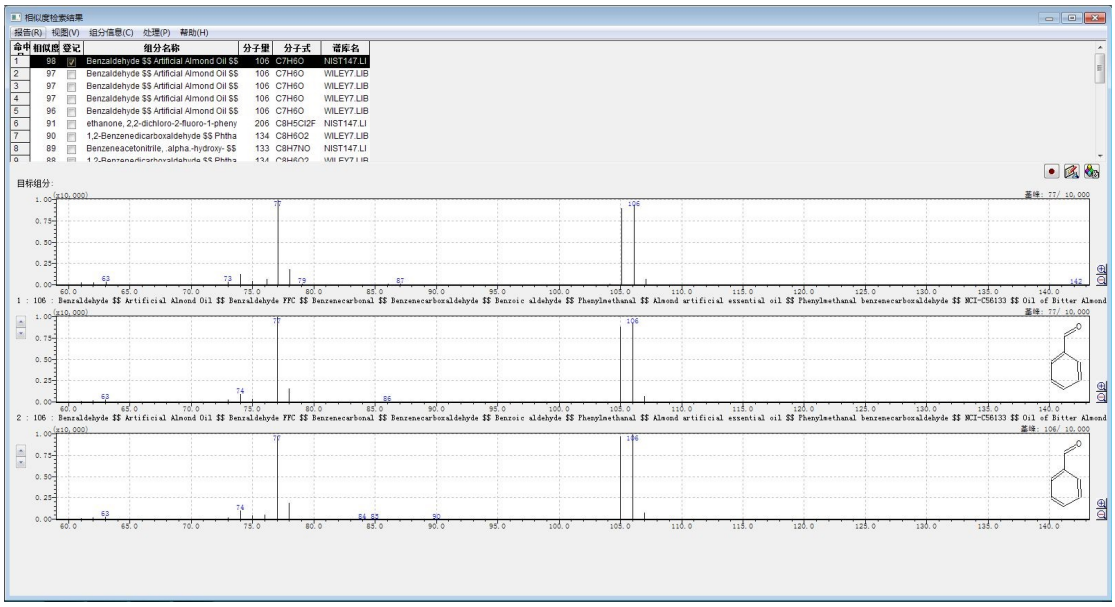


Scheme S4e: for benzaldehyde (8): A mixture of morpholine (0.3 mmol), (nitromethyl)benzene (0.45 mmol), Me_4NI (0.09 mmol), DABCO (0.2 mmol) and CH_3CN = 6 mL were added to an undivided cell. The cell was equipped with a carbon electrode as anode and a nickel electrode as cathode. The reaction mixture was stirred and electrolyzed at a constant current of 9 mA under 50℃ for corresponding time.

GC-MS: m/z calcd for C_7H_6O 106, found 106.

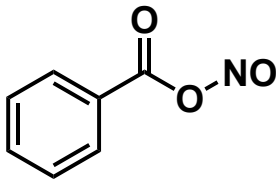


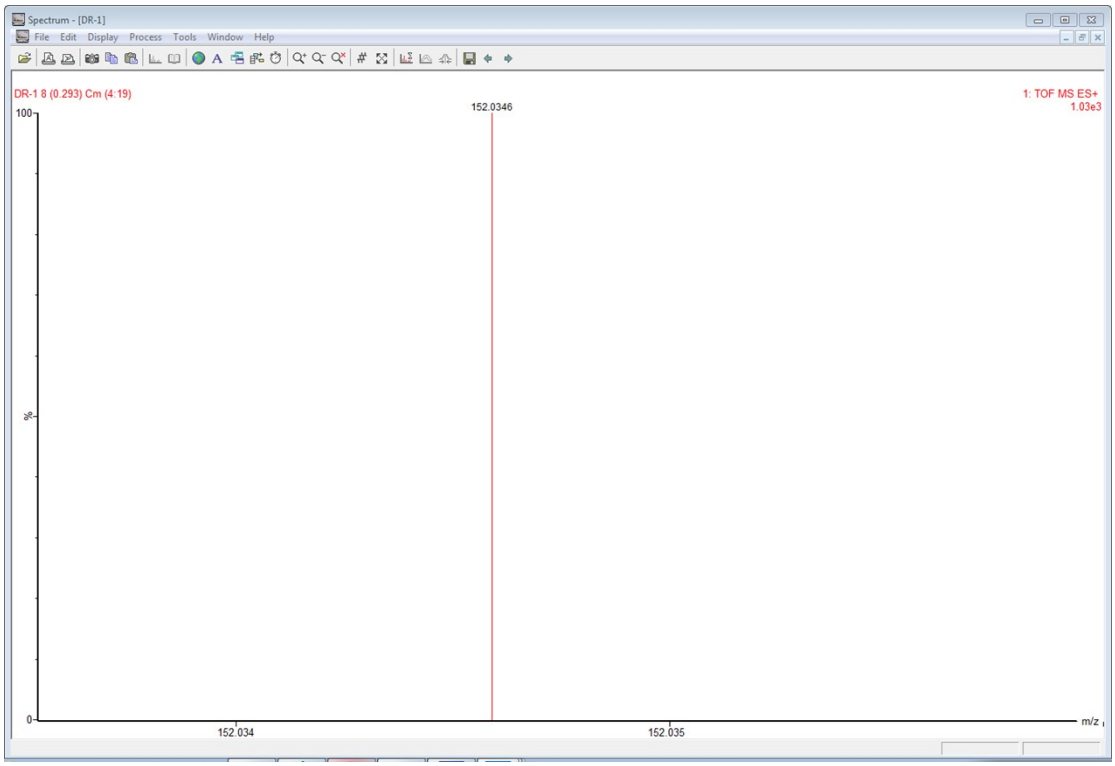




Scheme S4f: for benzoic nitrous anhydride (9): A mixture of morpholine (0.3 mmol), nitromethane (0.45 mmol), Me_4NI (0.09 mmol) and DABCO (0.2 mmol), sodium benzoate (PhCOONa, 0.3 mmol) and CH_3CN = 6 mL were added to an undivided cell. The cell was equipped with a carbon electrode as anode and a nickel electrode as cathode. The reaction mixture was stirred and electrolyzed at a constant current of 9 mA under 50℃ for corresponding time.

HRMS (ESI) m/z calcd for $C_7H_6NO_3[M+H]^+$ 152.0342, found 152.0346.

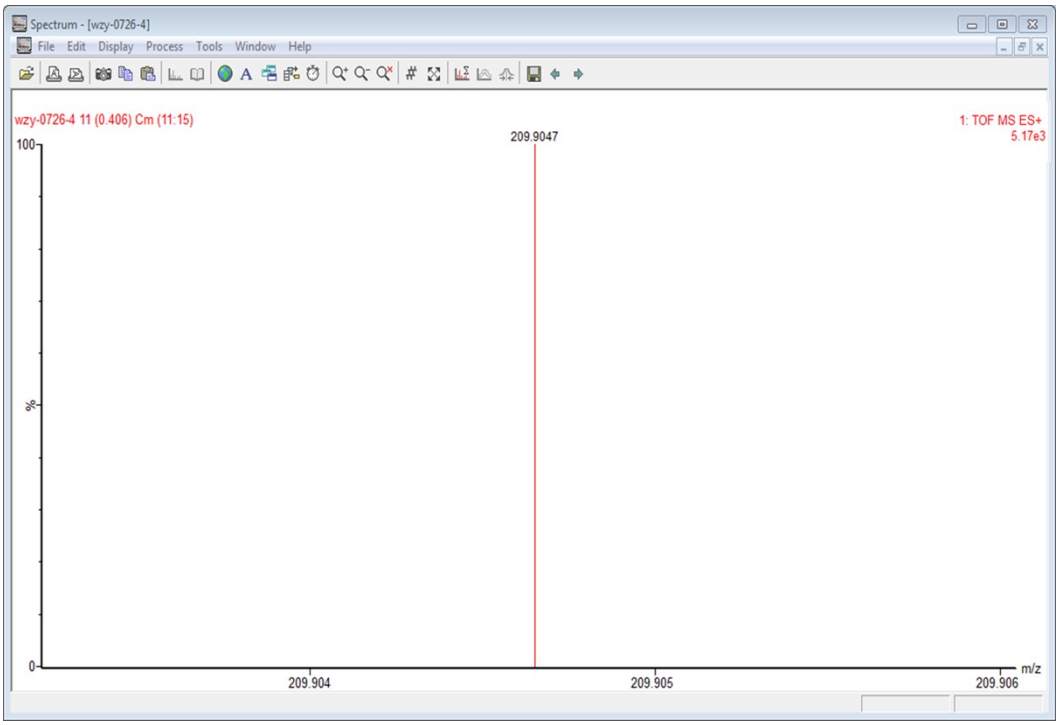



Spectrum - [DR-1]
File Edit Display Process Tools Window Help
DR-1 8 (0.293) Cm (4:19)
1: TOF MS ES+
1.03e3
152.0346
100
%
0
152.034
152.035
m/z

7、Proposed possible reaction mechanism

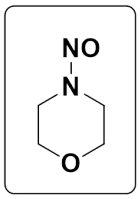
Scheme 5-5: iodo-(nitro)-methane (substance 5): A mixture of morpholine (0.3 mmol), nitromethane (0.45 mmol), Me_4NI (0.09 mmol) and DABCO (0.2 mmol) and CH_3CN = 6 ml were added to an undivided cell. The cell was equipped with a carbon electrode as anode and a nickel electrode as cathode. The reaction mixture was stirred and electrolyzed at a constant current of 9 mA under 50℃ for corresponding time.

HRMS (ESI) m/z calcd for $CH_3NO_2[M+Na]^+$ 209.9023, found 209.9047.



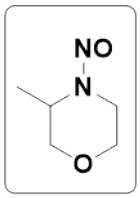
8、Detail Descriptions for Products

4-nitrosomorpholine(3a)[S1]



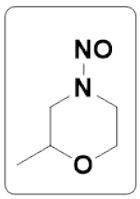
The title compound was prepared according to the general working procedure and purified by column chromatography (petroleum ether/ethyl acetate = 3:1) to give the product as a yellow oil. 94% yield, 32.7mg. ^{1}H NMR (500 MHz, Acetone-*d*6) δ 4.26 (m, 2H), 3.85 (m, 2H), 3.82 – 3.76 (m, 2H), 3.65 – 3.57 (m, 2H). ^{13}C NMR (125 MHz, Acetone-*d*6) δ 67.0, 65.5, 49.6, 40.0.

3-methyl-4-nitrosomorpholine(3b)[S3]



The title compound was prepared according to the general working procedure and purified by column chromatography (petroleum ether/ethyl acetate = 4:1) to give the product as a yellow oil. 92% yield, 35.9mg. ^{1}H NMR (500 MHz, Acetone-*d*6) δ 4.93 - 4.60 (m, 1H), 4.51 - 4.02 (m,1H), 3.93 - 3.86 (dd, *J* = 11.5, 3.5 Hz, 1H), 3.76 – 3.43 (m, 4H), 1.50 (dd, *J* = 6.8, 0.9 Hz, 2H), 1.15 (d, *J* = 6.9 Hz, 1H). ^{13}C NMR (125 MHz, Acetone-*d*6) δ 72.6, 70.9, 67.9, 66.7, 56.2, 46.9, 45.4, 38.6, 16.0, 14.2.

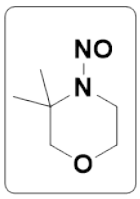
2-methyl-4-nitrosomorpholine(3c)[S3]



The title compound was prepared according to the general working procedure and purified by column chromatography (petroleum ether/ethyl acetate = 4:1) to give the product as a yellow oil. 94% yield, 36.7mg. ^{1}H NMR (500 MHz, Acetone-*d*6) δ 4.83 – 4.72 (m, 1H), 4.68 – 4.55 (m, 1H), 4.10 - 3.94 (ddd, *J* = 11.7, 4.3, 1.2Hz, 1H), 3.85 (2.74) (m, 1H), 3.73 -3.62 (m, 1H), 3.50 (2.42) (ddd, *J* = 13.3, 10.6, 1.2 Hz, 1H), 3.41 – 3.30 (m, 1H), 1.27 (d, *J* = 6.1 Hz, 2H), 1.18 (d, *J* = 6.2 Hz, 1H). ^{13}C NMR (125 MHz, Acetone-*d*6) δ 72.7, 71.3, 66.5, 65.0, 55.1, 48.9, 45.2, 39.2, 18.2, 17.8.

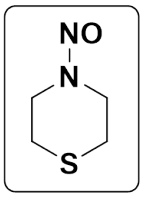
^{1}H NMR (600 MHz, Acetone-*d*6, -80℃) δ 4.87 (dd, *J* = 31.6, 13.6 Hz, 1H), 4.73 (4.63) (d, *J* = 13.0 Hz, 1H), 4.14 (3.99) (dd, *J* = 11.6, 3.8 Hz, 1H), 3.88 (2.77) (td, *J* = 12.6, 3.8 Hz, 1H), 3.78 – 3.66 (3.43 – 3.33) (m, 2H), 3.57 – 3.53 (2.56 – 2.39) (m, 1H), 1.28 (1.20) (d, *J* = 6.2 Hz, 3H).

3,3-dimethyl-4-nitrosomorpholine(3d)[S3]



The title compound was prepared according to the general working procedure and purified by column chromatography (petroleum ether/ethyl acetate = 6:1) to give the product as a yellow oil. 84% yield, 36.3mg. ^{1}H NMR (500 MHz, Methanol-*d*4) δ 3.82 (dd, *J* = 6.0, 5.0 Hz, 2H), 3.74 – 3.69 (dd, *J* = 6.0, 5.0 Hz, 2H), 3.61 (s, 2H), 1.57 (s, 6H). ^{13}C NMR (125 MHz, Methanol-*d*4) δ 77.3, 67.3, 61.5, 38.3, 23.9.

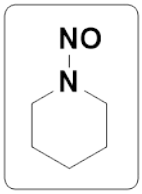
4-nitrosothiomorpholin(3e)[S1]



The title compound was prepared according to the general working procedure and purified by column chromatography (petroleum ether/ethyl acetate = 6:1) to give the product as a yellow oil. 93% yield, 36.8mg. ^{1}H NMR (500 MHz, Chloroform-*d*) δ 4.56 – 4.47 (m, 2H), 4.13 – 4.00 (m, 2H), 2.95 – 2.82 (m, 2H), 2.63 – 2.55 (m, 2H). ^{13}C NMR (125 MHz, Chloroform-*d*) δ 52.4, 41.3, 28.9, 27.3.

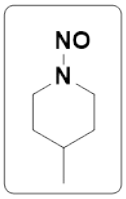
^{1}H NMR (600 MHz, Chloroform-*d*, -60℃) δ 4.66 – 4.45 (m, 2H), 4.15 (s, 2H), 3.12 – 2.86 (t, *J* = 5.4 Hz, 2H), 2.66 (t, *J* = 5.4 Hz, 2H).

1-nitrosopiperidine(3f)[S1]



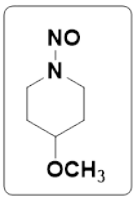
The title compound was prepared according to the general working procedure and purified by column chromatography (petroleum ether/ethyl acetate = 6:1) to give the product as a yellow oil. 90% yield, 30.1mg. ^{1}H NMR (500 MHz, Chloroform-*d*) δ 4.22 – 4.14 (m, 2H), 3.81 – 3.74 (m, 2H), 1.84 – 1.71 (m, 4H), 1.56 (m, 2H). ^{13}C NMR (125 MHz, Chloroform-*d*) δ 49.9, 38.8, 25.4, 23.7, 23.1.

4-methyl-1-nitrosopiperidine(3g)[S1]



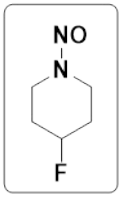
The title compound was prepared according to the general working procedure and purified by column chromatography (petroleum ether/ethyl acetate = 4:1) to give the product as a yellow oil. 88% yield, 33.8mg. ^{1}H NMR (500 MHz, Chloroform-*d*) δ 4.99 (m, 1H), 4.72 (m, 1H), 3.67 (td, *J* = 12.9, 3.6 Hz, 1H), 2.57 (td, *J* = 13.1, 3.9 Hz, 1H), 2.00 – 1.59 (m, 3H), 1.36 (m, 1H), 1.10 –0.97(m, 1H), 1.01 (d, *J* = 6.6 Hz, 3H). ^{13}C NMR (125 MHz, Chloroform-*d*) δ 50.1, 39.0, 34.4, 32.7, 30.9, 21.3.

4-methoxy-1-nitrosopiperidine(3h)[S1]



The title compound was prepared according to the general working procedure and purified by column chromatography (petroleum ether/ethyl acetate = 3:1) to give the product as a yellow oil. 96% yield, 41.5mg. ^{1}H NMR (500 MHz, Methanol-*d*4) δ 4.31 (ddd, *J* = 12.9, 8.4, 4.3 Hz, 1H), 4.17 (ddd, *J* = 13.3, 6.9, 4.5 Hz, 1H), 3.91 – 3.77 (m, 2H), 3.64 (tt, *J* = 6.6, 3.2 Hz, 1H), 3.39 (s, 3H), 2.06 – 1.98 (m, 1H), 1.86 (m, 1H), 1.76 (m, 1H), 1.62 (m, 1H). ^{13}C NMR (125 MHz, Methanol-*d*4) δ 74.2, 54.9, 46.4, 35.5, 30.4, 28.7.

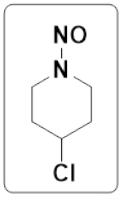
4-fluoro-1-nitrosopiperidine(3i)



The title compound was prepared according to the general working procedure and purified by column chromatography (petroleum ether/ethyl acetate = 4:1) to give the product as a yellow oil. 90% yield, 35.6mg. ^{1}H NMR (500 MHz, Chloroform-*d*) δ 5.06 (4.97) (tt, *J* = 5.2, 2.5 Hz, 1H), 4.51 (m, 2H), 4.20 (ddd, *J* = 14.0, 10.8, 3.9 Hz, 1H), 3.32 (ddd, *J* = 14.5,

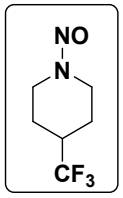
11.0, 4.2 Hz, 1H), 2.19 (m, 1H), 2.10 – 1.89 (m, 2H), 1.81 – 1.62 (m, 1H). ^{13}C NMR (125 MHz, Chloroform-*d*) δ 87.5, 86.1, 45.3, 45.2, 34.3, 34.3, 31.5, 31.4, 29.8, 29.7. ^{19}F NMR (470 MHz, Chloroform-*d*) δ -187.1.
HRMS (ESI) m/z calcd for $C_{16}H_{25}NO_2[M+Na]^+$ 155.0591, found 155.0578.

4-chloro-1-nitrosopiperidine(3j)[S1]



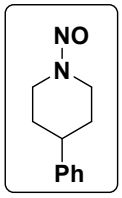
The title compound was prepared according to the general working procedure and purified by column chromatography (petroleum ether/ethyl acetate = 3:1) to give the product as a yellow oil. 92% yield, 40.8mg. ^{1}H NMR (500 MHz, Chloroform-*d*) δ 4.45 (tt, *J* = 6.1, 3.4 Hz, 1H), 4.39 (d, *J* = 4.7 Hz, 1H), 4.38 (d, *J* = 5.2 Hz, 1H) 4.23 (dt, *J* = 13.9, 5.1 Hz, 1H), 3.66 (ddd, *J* = 13.7, 9.1, 4.4 Hz, 1H), 2.25 (m, 1H), 2.13 (m, 1H), 2.01 – 1.85 (m, 2H). ^{13}C NMR (125 MHz, Chloroform-*d*) δ 55.5, 46.2, 35.2, 34.8, 33.3, 33.2.

1-nitroso-4-(trifluoromethyl)piperidine(3k)[S3]



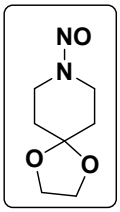
The title compound was prepared according to the general working procedure and purified by column chromatography (petroleum ether/ethyl acetate = 6:1) to give the product as a yellow oil. 95% yield, 51.9mg. ^{1}H NMR (500 MHz, Chloroform-*d*) δ 5.15 – 5.05 (m, 1H), 4.90 (d, J = 13.5 Hz, 1H), 3.72 (td, *J* = 13.1, 3.6 Hz, 1H), 2.59 (td, *J* = 13.3, 3.9 Hz, 1H), 2.44 (m, 1H), 2.19 (m, 1H), 2.03 – 1.96 (m, 1H), 1.77 (qd, *J* = 12.6, 4.8 Hz, 1H), 1.45 (qd, *J* = 12.7, 5.0 Hz, 1H).^{13}C NMR (125 MHz, Chloroform-*d*) δ 125.6 (d, *J* = 278.5 Hz), 39.3 (q, *J* = 27.7 Hz), 36.2, 24.1 (q, *J* = 2.7 Hz), 22.5 (q, *J* = 2.8 Hz). ^{19}F NMR (470 MHz, Chloroform-*d*) δ -73.5.

1-nitroso-4-phenylpiperidine(3l)[S3]



The title compound was prepared according to the general working procedure and purified by column chromatography (petroleum ether/ethyl acetate = 8:1) to give the product as a light yellow solid. 68% yield, 38.7mg. ^{1}H NMR (500 MHz, Acetone-*d*6) δ 7.35 – 7.19 (m, 5H), 5.11 (m, 1H), 4.83 (m, 1H), 3.92 – 3.81 (m, 1H), 3.03 (tt, *J* = 12.3, 3.4 Hz, 1H), 2.71 – 2.61 (m, 1H), 2.14 – 2.02 (m, 1H), 1.96 – 1.79 (m, 2H), 1.50 (m, 1H). ^{13}C NMR (125 MHz, Acetone-*d*6) δ 205.4, 145.1, 128.5, 126.8, 126.5, 49.8, 42.0, 38.5, 33.6, 32.0.

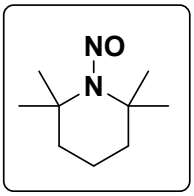
8-nitroso-1,4-dioxa-8-azaspiro[4.5]decane(3m)[S3]



The title compound was prepared according to the general working procedure and purified by column chromatography (petroleum ether/ethyl acetate = 4:1) to give the product as a white solid. 97% yield, 50.1mg. ^{1}H NMR (500 MHz, Acetone-*d*6) δ 4.33 – 4.27 (m, 2H), 4.01 (t, *J* = 1.4 Hz, 4H), 3.84 – 3.78 (m, 2H), 1.91 (ddd, *J* = 7.1, 5.4, 1.2 Hz, 2H), 1.63 (ddd, *J* = 7.4, 5.6, 1.2 Hz, 2H). ^{13}C NMR (125 MHz, Acetone-*d*6) δ 106.6,

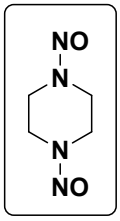
64.4, 47.2, 36.0, 35.0, 33.4.

2,2,6,6-tetramethyl-1-nitrosopiperidine(3n)[S1]



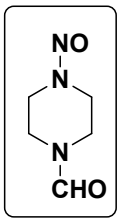
The title compound was prepared according to the general working procedure and purified by column chromatography (petroleum ether/ethyl acetate = 8:1) to give the product as a yellow oil. 60% yield, 28.9mg. ^{1}H NMR (500 MHz, Chloroform-*d*) δ 1.84 – 1.79 (m, 2H), 1.71 – 1.65 (m, 2H), 1.61 (s, 8H), 1.41 (s, 6H). ^{13}C NMR (125 MHz, Chloroform-*d*) δ 61.1, 59.7, 40.5, 37.8, 30.8, 25.0, 15.2.

1,4-dinitrosopiperazine(3o) [S1]



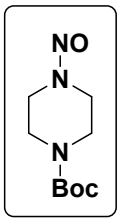
The title compound was prepared according to the general working procedure and purified by column chromatography (petroleum ether/ethyl acetate = 1:1) to give the product as a white solid. 60% yield, 25.9mg. ^{1}H NMR (500 MHz, Chloroform-*d*) δ 4.56 (d, *J* = 1.5 Hz, 2H), 4.42 – 4.39 (m, 2H), 4.05 (td, *J* = 5.8, 1.4 Hz, 2H), 3.82 (d, *J* = 1.5 Hz, 2H). ^{13}C NMR (125 MHz, Chloroform-*d*) δ 49.6, 47.1, 40.5, 37.8.

4-nitrosopiperazine-1-carbaldehyde(3p)[S3]



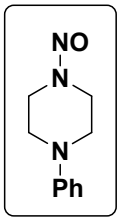
The title compound was prepared according to the general working procedure and purified by column chromatography (petroleum ether/ethyl acetate = 2:1) to give the product as a yellow oil. 84% yield, 36.0mg. ^{1}H NMR (500 MHz, Chloroform-*d*) δ 8.10 (d, *J* = 18.5 Hz, 1H), 4.30 – 4.27 (m, 1H), 4.24 (dd, *J* = 6.3, 4.6 Hz, 1H), 3.82 (dd, *J* = 6.4, 4.7 Hz, 1H), 3.76 (ddd, *J* = 15.9, 6.2, 4.7 Hz, 2H), 3.64 – 3.57 (m, 1H), 3.49 (dd, *J* = 6.5, 4.7 Hz, 1H), 3.36 (dd, *J* = 6.4, 4.6 Hz, 1H). ^{13}C NMR (125 MHz, Chloroform-*d*) δ 161.0, 160.9, 50.0, 48.8, 45.7, 44.0, 40.2, 39.9, 38.8, 38.6.

tert-butyl 4-nitrosopiperazine-1-carboxylate(3q)[S4]



The title compound was prepared according to the general working procedure and purified by column chromatography (petroleum ether/ethyl acetate = 6:1) to give the product as a white solid. 77% yield, 49.7mg. ^{1}H NMR (400 MHz, Chloroform-*d*) δ 4.29 – 4.24 (m, 2H), 3.81 (t, *J* = 5.5 Hz, 2H), 3.68 (dd, *J* = 6.3, 4.5 Hz, 2H), 3.45 (t, *J* = 5.5 Hz, 2H), 1.49 (s, 9H). ^{13}C NMR (125 MHz, Chloroform-*d*) δ 154.3, 80.9, 49.3, 48.8, 46.1, 39.7, 28.3.

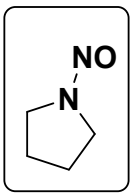
1-nitroso-4-phenylpiperazine(3r)[S4]



The title compound was prepared according to the general working procedure and purified by column chromatography (petroleum ether/ethyl acetate = 4:1) to give the product as a white solid. 73% yield, 41.8mg. ^{1}H NMR (500 MHz, Acetone-*d*6) δ 7.31 – 7.22 (m, 2H), 7.03 (d, *J* =

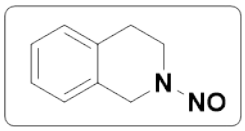
8.0 Hz, 2H), 6.87 (t, *J* = 7.3 Hz, 1H), 4.43 – 4.37 (m, 2H), 3.97 – 3.86 (m, 2H), 3.49 – 3.43 (m, 2H), 3.25 – 3.18 (m, 2H). ^{13}C NMR (125 MHz, Acetone-*d*6) δ 151.7, 120.0, 121.0, 117.6, 50.8, 49.8, 49.0, 40.0.

1-nitrosopyrrolidine(3s)[S4]



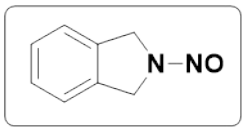
The title compound was prepared according to the general working procedure and purified by column chromatography (petroleum ether/ethyl acetate = 4:1) to give the product as a yellow oil. 82% yield, 24.6mg. ^{1}H NMR (500 MHz, Chloroform-d) δ 4.27 (t, *J* = 6.7 Hz, 2H), 3.59 (t, *J* = 7.1 Hz, 2H), 2.12 – 1.96 (m, 4H). ^{13}C NMR (125 MHz, Chloroform-*d*) δ 48.8, 44.2, 23.0, 21.6.

2-nitroso-1,2,3,4-tetrahydroisoquinoline(3t)[S4]



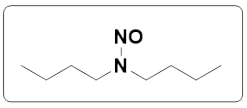
The title compound was prepared according to the general working procedure and purified by column chromatography (petroleum ether/ethyl acetate = 6:1) to give the product as a yellow oil. 84% yield, 40.8mg. ^{1}H NMR (500 MHz, Chloroform-*d*) δ 7.29 – 7.14 (m, 4H), 4.83(5.39) (s, 2H), 4.54(3.88) (t, *J* = 5.9 Hz, 2H), 3.10 (2.96) (t, *J* = 5.9 Hz, 2H). ^{13}C NMR (125 MHz, Chloroform-*d*) δ 135.0(133.9), 132.4, 130.0, 128.7, 128.1, 128.0, 127.3, 127.3, 127.2, 126.2, 51.3, 47.8, 44.5, 40.8, 29.8, 27.4.

2-nitrosoisoindoline(3u)[S2]



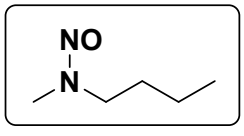
The title compound was prepared according to the general working procedure and purified by column chromatography (petroleum ether/ethyl acetate = 3:1) to give the product as a white grey solid. 45% yield, 20.0mg. ^{1}H NMR (500 MHz, Chloroform-*d*) δ 7.36 (m, 4H), 5.63 (s, 2H), 4.90 (s, 2H). ^{13}C NMR (125 MHz, Chloroform-*d*) δ 134.0, 133.2, 128.4, 128.1, 123.7, 123.1, 54.7, 51.4.

N,N-dibutylnitrous amide(3v) [S4]



The title compound was prepared according to the general working procedure and purified by column chromatography (petroleum ether/ethyl acetate = 8:1) to give the product as a yellow oil. 71% yield, 33.7mg. ^{1}H NMR (500 MHz, Chloroform-*d*) δ 4.07 (t, *J* = 7.3 Hz, 2H), 3.57 – 3.50 (m, 2H), 1.73 (tt, *J* = 7.6, 6.6 Hz, 2H), 1.51 – 1.43 (m, 2H), 1.39 (m, 2H), 1.30 (m, 2H), 0.97 (t, *J* = 7.4 Hz, 3H), 0.92 (t, *J* = 7.4 Hz, 3H). ^{13}C NMR (125 MHz, Chloroform-*d*) δ 52.1, 43.5, 30.4, 28.2, 20.5, 19.8, 13.7, 13.6.

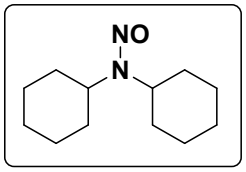
N-butyl-*N*-methylnitrous amide (3w)[S4]



The title compound was prepared according to the general working procedure and purified by column chromatography

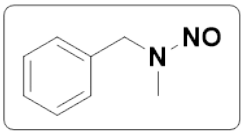
(petroleum ether/ethyl acetate = 6:1) to give the product as a yellow oil. 79% yield, 27.5mg. ^{1}H NMR (500 MHz, Chloroform-*d*) δ 4.15(3.59) (t, *J* = 7.2 Hz, 2H), 3.05(3.75) (s, 3H), 1.73(1.28) (p, *J* = 7.4 Hz, 2H), 1.37(1.48) (h, *J* = 7.4 Hz, 2H), 0.97 (0.92) (t, *J* = 7.4 Hz, 3H). ^{13}C NMR (125 MHz, Chloroform-*d*) δ 53.2, 44.5, 38.9, 31.1, 29.9, 27.6, 20.1, 19.5, 13.5, 13.4.

N,N-dicyclohexylnitrous amide(3x)[S4]



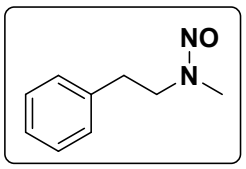
The title compound was prepared according to the general working procedure and purified by column chromatography (petroleum ether/ethyl acetate = 6:1) to give the product as a white solid. 73% yield, 46.0mg. ^{1}H NMR (500 MHz, Chloroform-*d*) δ 4.87 (tt, *J* = 12.0, 3.8 Hz, 1H), 3.72 (tt, *J* = 11.3, 4.4 Hz, 1H), 1.99 – 1.85 (m, 6H), 1.80 (d, *J* = 13.1 Hz, 2H), 1.75 – 1.65 (m, 2H), 1.59 (dd, *J* = 12.0, 3.6 Hz, 2H), 1.39 (m, 6H), 1.26 (m, 1H), 1.15 (m, 1H). ^{13}C NMR (125 MHz, Chloroform-*d*) δ 58.6, 52.2, 34.4, 29.4, 26.1, 25.5, 25.4, 25.2.

N-benzyl-*N*-methylnitrous amide(3y)[S1]



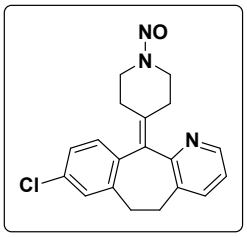
The title compound was prepared according to the general working procedure and purified by column chromatography (petroleum ether/ethyl acetate = 6:1) to give the product as a yellow oil. 76% yield, 34.2mg. ^{1}H NMR (500 MHz, Chloroform-*d*) δ 7.42 – 7.23 (7.18 – 7.10) (m, 5H), 5.30 (4.80) (s, 2H), 3.69 (2.94) (s, 3H). ^{13}C NMR (125 MHz, Chloroform-*d*) δ 134.5, 133.8, 129.1, 128.9, 128.6, 128.4, 128.1, 128.0, 57.6, 47.8, 38.4, 30.9.

N-methyl-*N*-phenethylnitrous amide(3z)[S5]



The title compound was prepared according to the general working procedure and purified by column chromatography (petroleum ether/ethyl acetate = 6:1) to give the product as a yellow oil. 72% yield, 35.4mg. ^{1}H NMR (500 MHz, Chloroform-*d*) δ 7.39 – 7.10 (m, 5H), 4.37 (3.78)(t, *J* = 7.2 Hz, 2H), 3.04 (2.80) (t, *J* = 7.2 Hz 2H), 2.98 (3.56) (s, 3H). ^{13}C NMR (125 MHz, Chloroform-*d*) δ 138.0, 137.3, 128.8, 128.7, 128.7, 127.0, 126.8, 55.1, 47.1, 39.8, 35.1, 32.0, 31.8.

8-chloro-11-(1-nitrosopiperidin-4-ylidene)-6,11-dihydro-5H-benzo[5,6]cyclohepta[1,2-b]pyridine(3ab)[S2]



The title compound was prepared according to the general working procedure and purified by column chromatography (petroleum ether/ethyl acetate = 2:1) to give the product as a orange solid. 78% yield, 79.3mg. ^{1}H NMR (500 MHz, Chloroform-*d*) δ 8.41 (ddd, *J* = 9.8, 4.9, 1.7 Hz, 1H), 7.47 (td, *J* =

7.6, 1.7 Hz, 1H), 7.22 – 7.07 (m, 4H), 4.53 – 4.38 (m, 1H), 4.21 – 4.02 (m, 2H), 3.59 – 3.47 (m, 1H), 3.44 – 3.27 (m, 2H),2.93 – 2.76 (m, 2H), 2.71 – 2.33 (m, 4H). ^{13}C NMR (125 MHz, Chloroform-*d*) δ 156.4, 156.1, 146.7 (d, *J* = 3.5 Hz), 139.6, 138.0 (d, *J* = 6.8 Hz), 137.5, 137.3, 136.2, 136.1, 135.1, 135.0, 133.4 (d, *J* = 2.8 Hz), 133.3, 130.1 (d, *J* = 12.8 Hz), 129.1 (d, *J* = 5.3 Hz), 126.4 (d, *J* = 4.6 Hz), 122.6, 49.9 (d, *J* = 18.6 Hz), 40.3 (d, *J* = 11.7 Hz), 31.6 – 31.4 (m), 31.0, 30.7, 28.8, 28.6.
HRMS (ESI) m/z calcd for $C_{19}H_{18}N_3OCl[M+Na]^+$ 340.1211, found 340.1216.

9、Supporting Reference

[S1] R. Ali, R. Babaahmadi, M. Didsbury, R. Stephens, R. L. Melen and T. Wirth, *Chem. Eur. J.* 2023, **29**, e202300957.

[S2] Y. Wang, S. You, M. Ruan, F. Wang, C. Ma, C. Lu, G. Yang, Z. Chen, M. Gao, *Eur. J. Org. Chem.* 2021, **22**, 3289–3293.

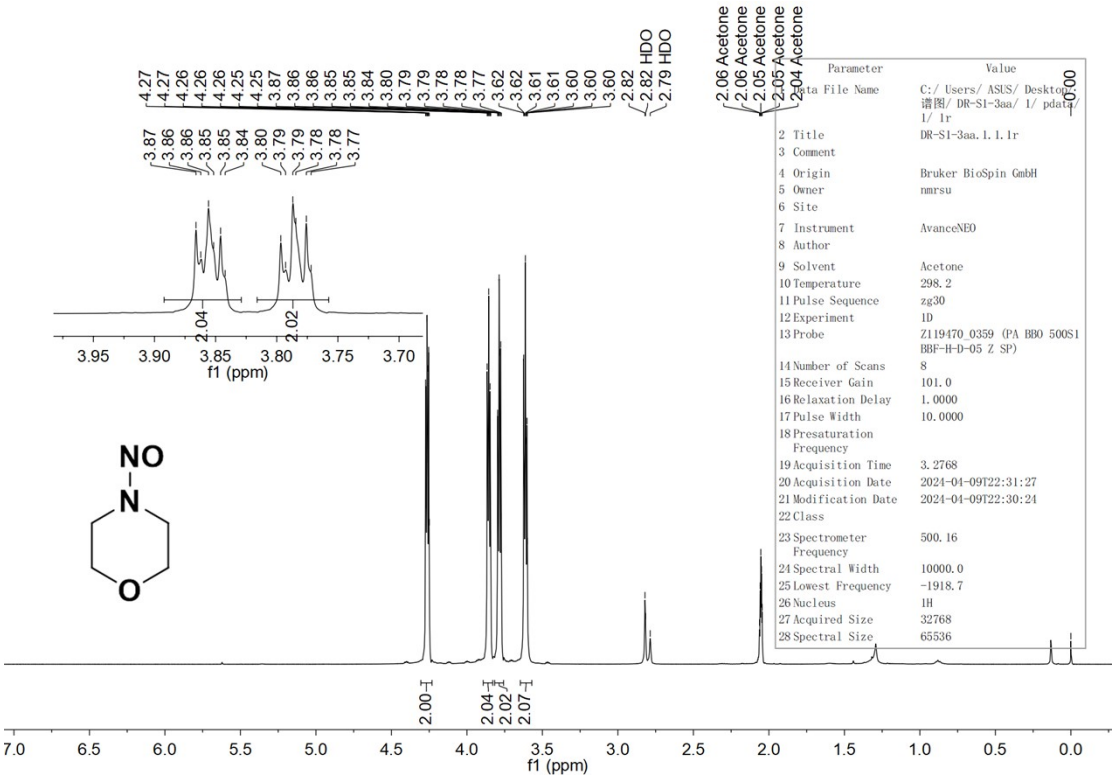
[S3] J. P. Zhao, L. J. Ding, P. C. Wang, Y. Liu, M. J. Huang, X. L. Zhou and M. Lu, *Adv. Synth. Catal.* 2020, **362**, 5036– 5043.

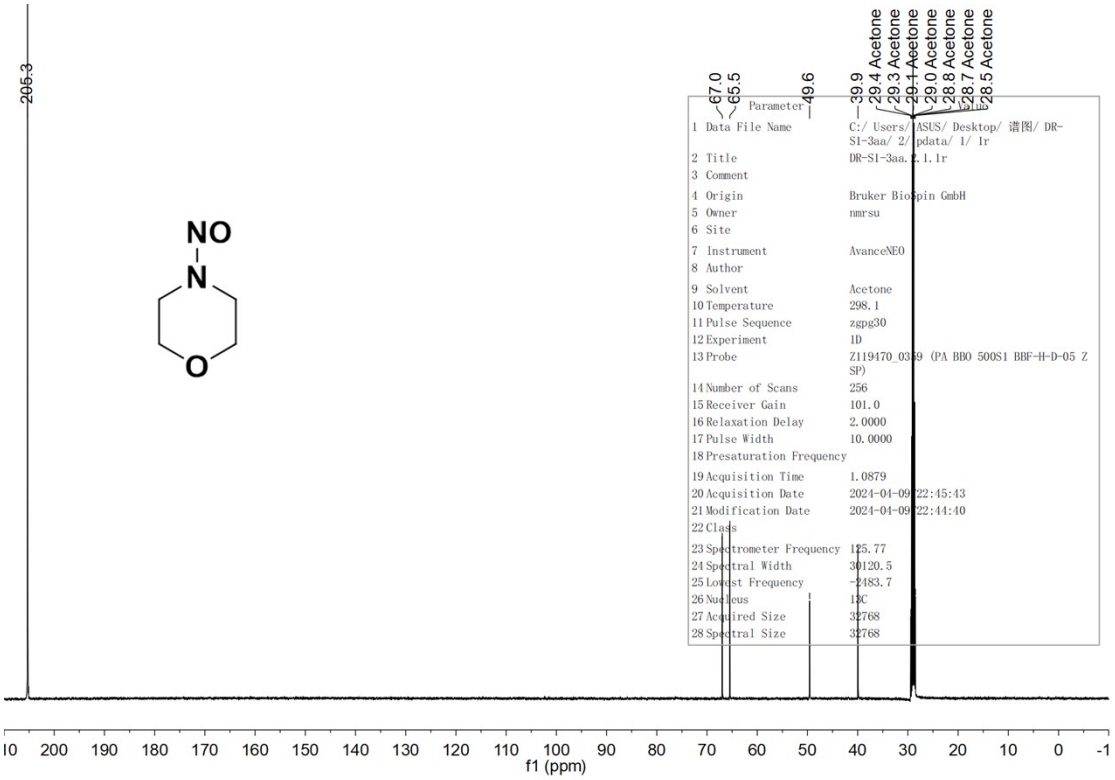
[S4] J. Zhang, J. W. Jiang, Y. L. Li and X. B. Wan, *J. Org. Chem.* 2013, **78**, 11366–11372.

[S5] Y. P. Yu, J. M. Ostresh and R. A. Houghten, *J. Org. Chem. 2003, **68**, 183-186.*

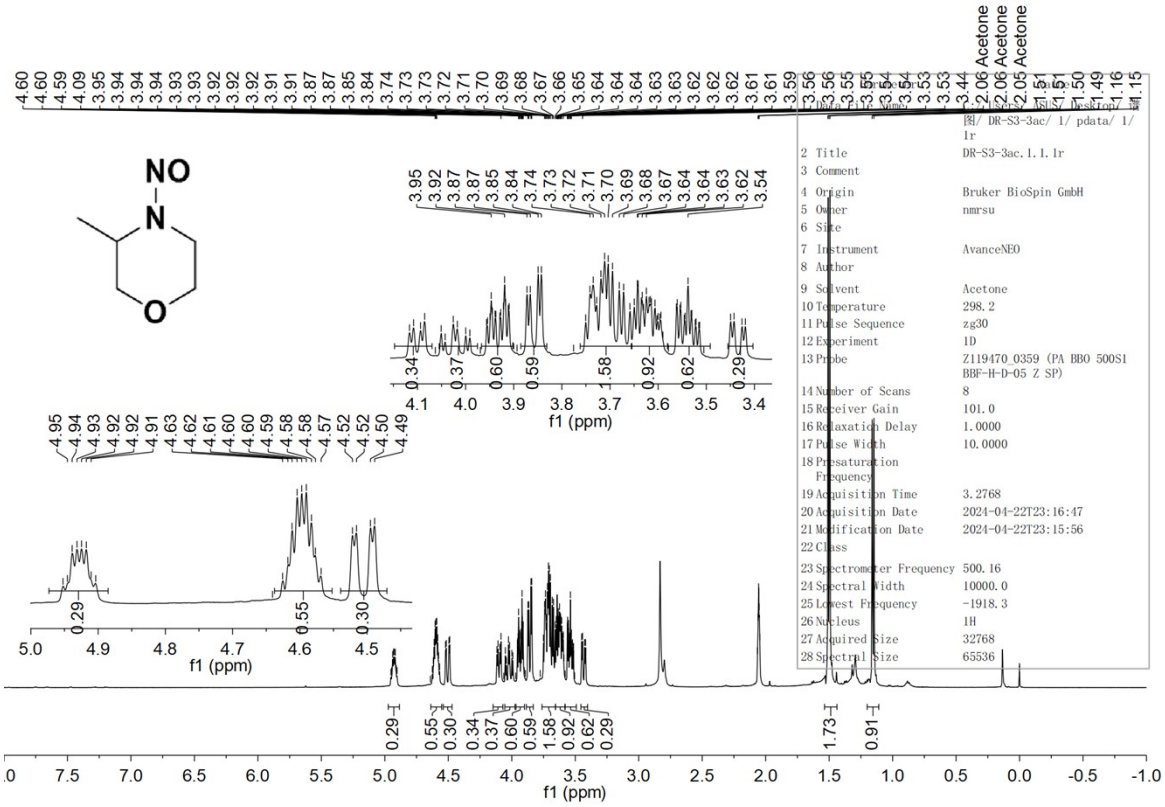
10、Copies of Product NMR Spectra

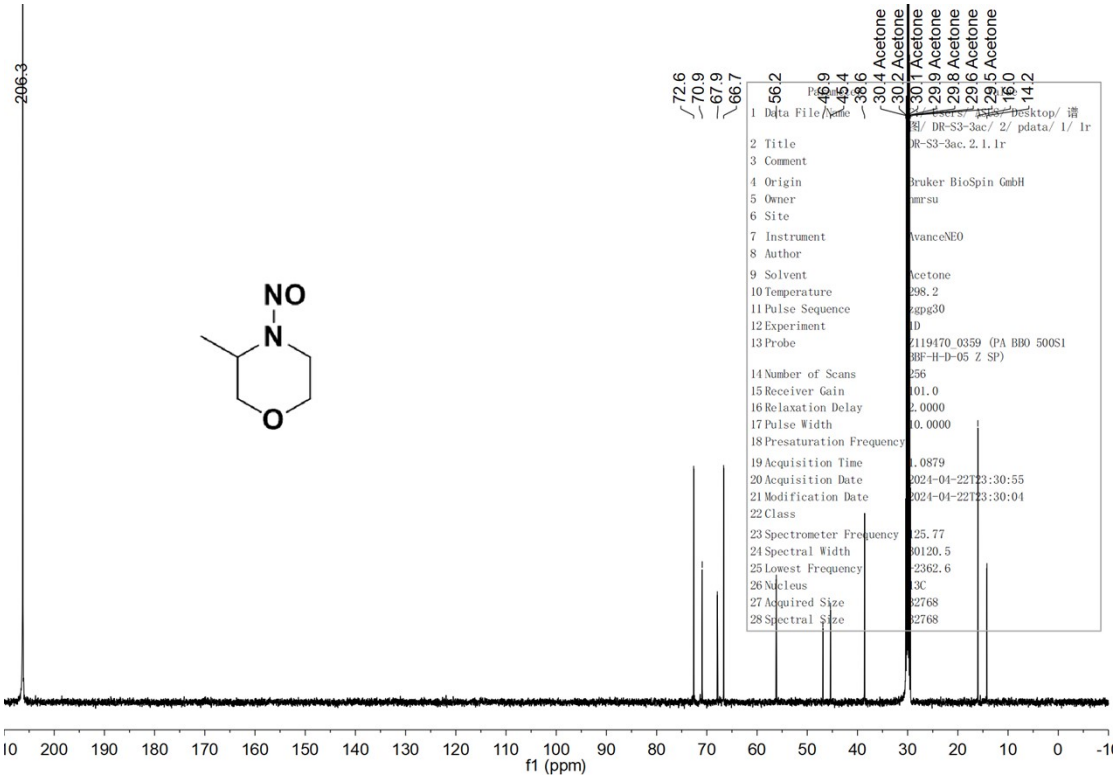
4-nitrosomorpholine(3a)



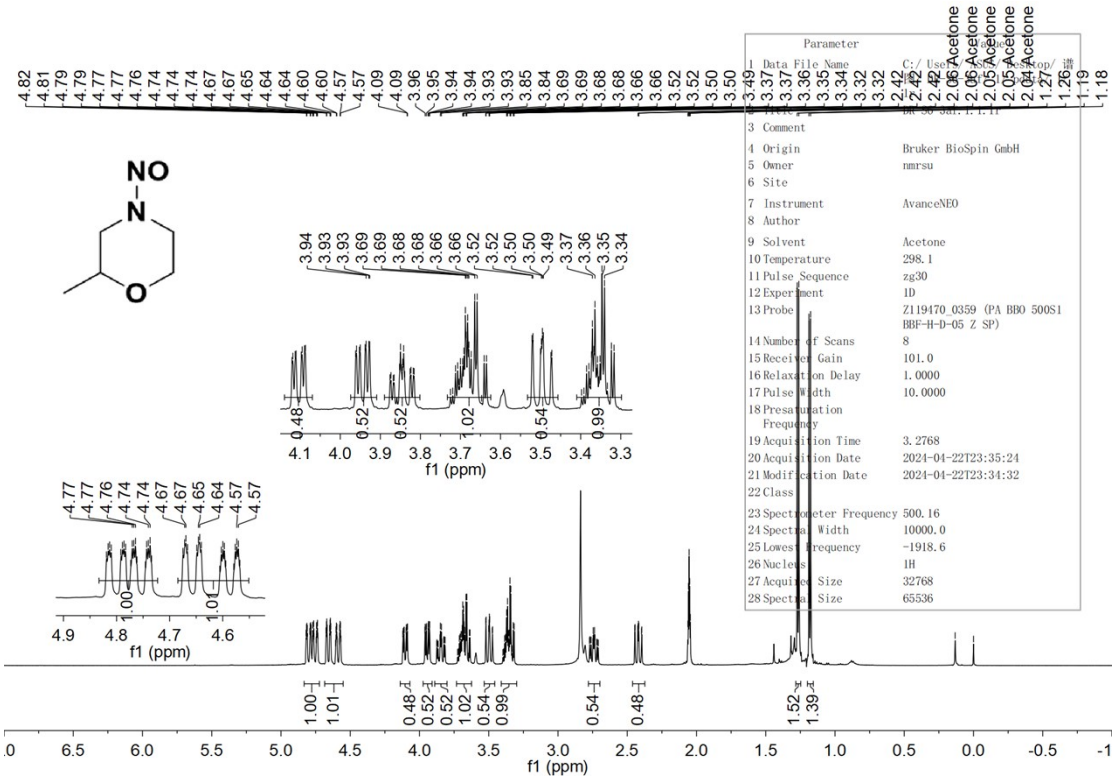


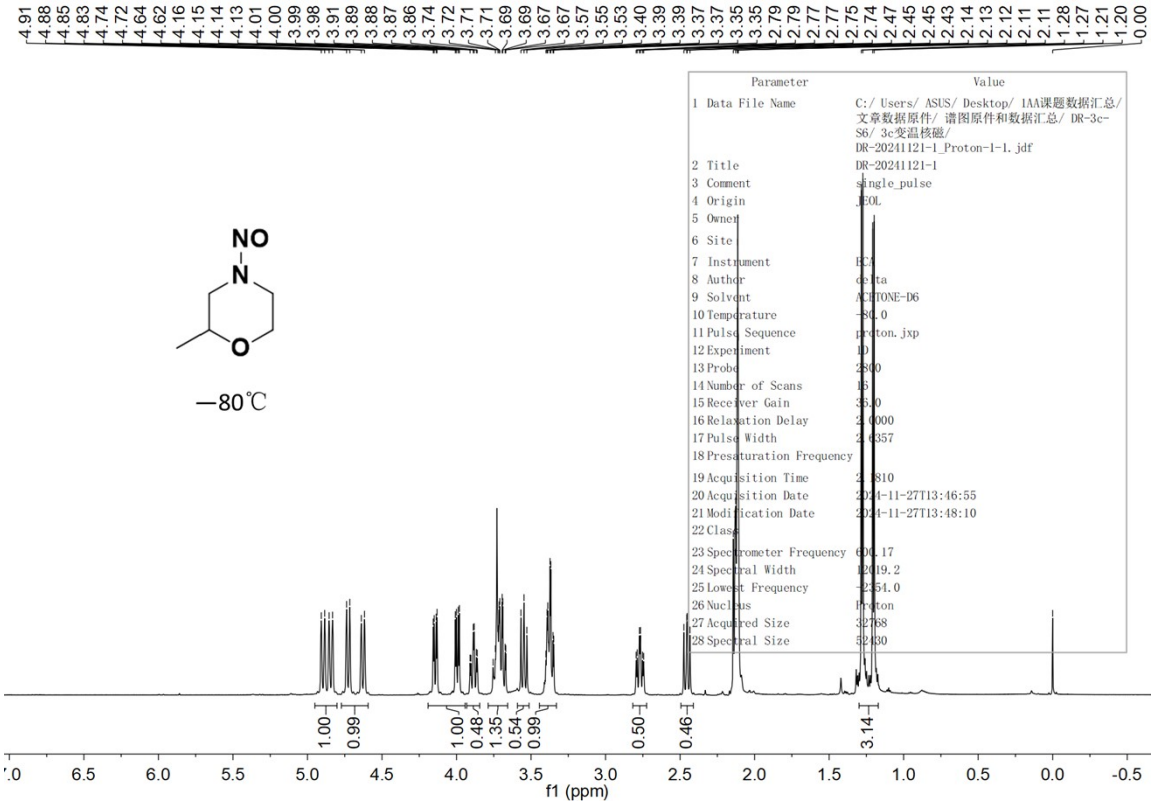
3-methyl-4-nitrosomorpholine(3b)

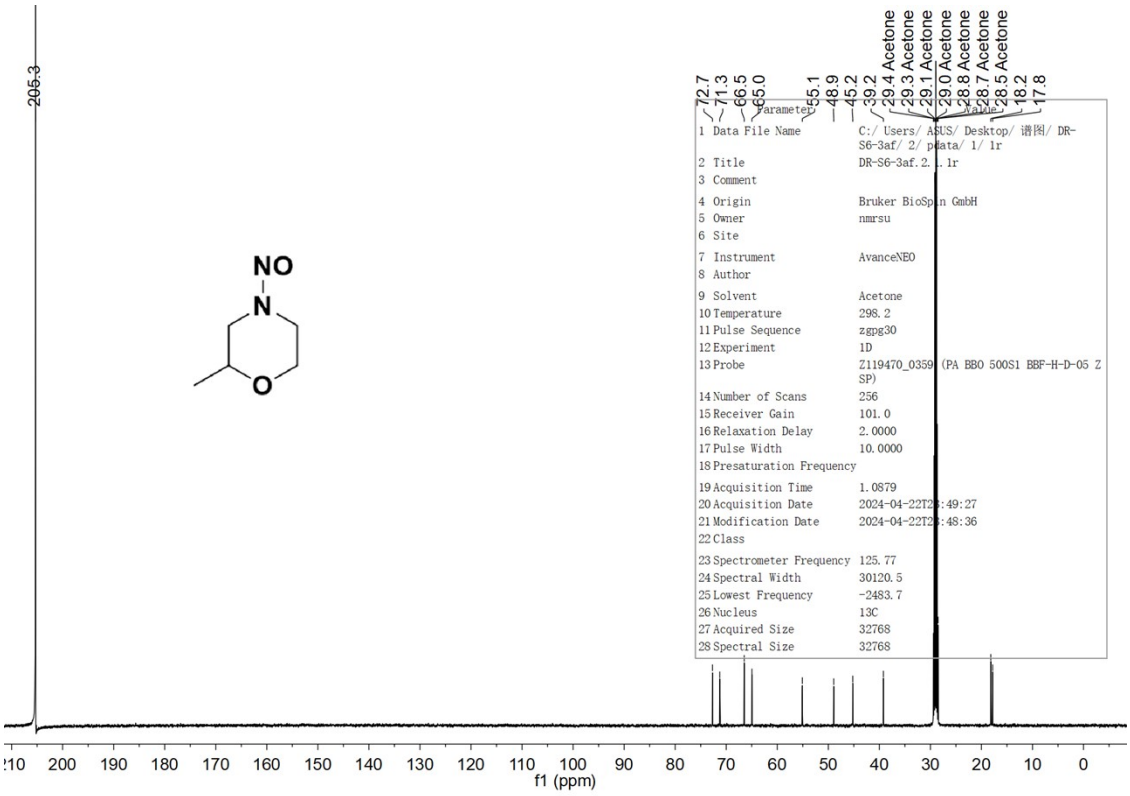




2-methyl-4-nitrosomorpholine(3c)







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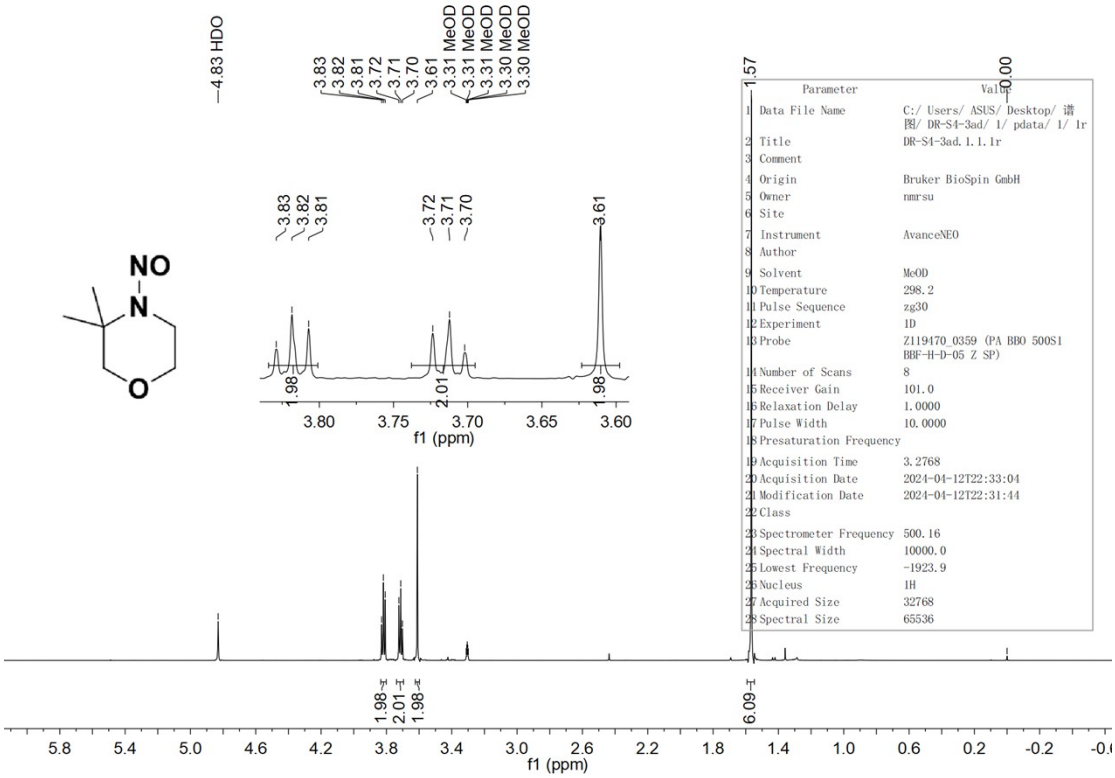
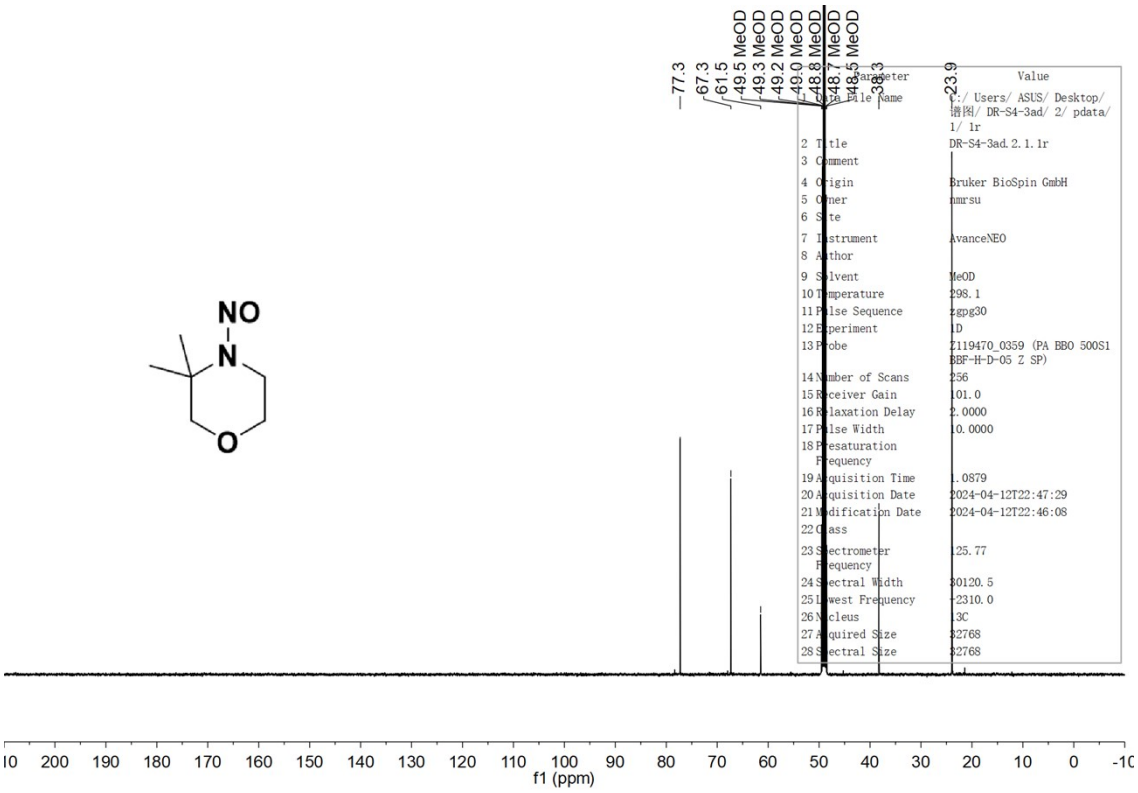
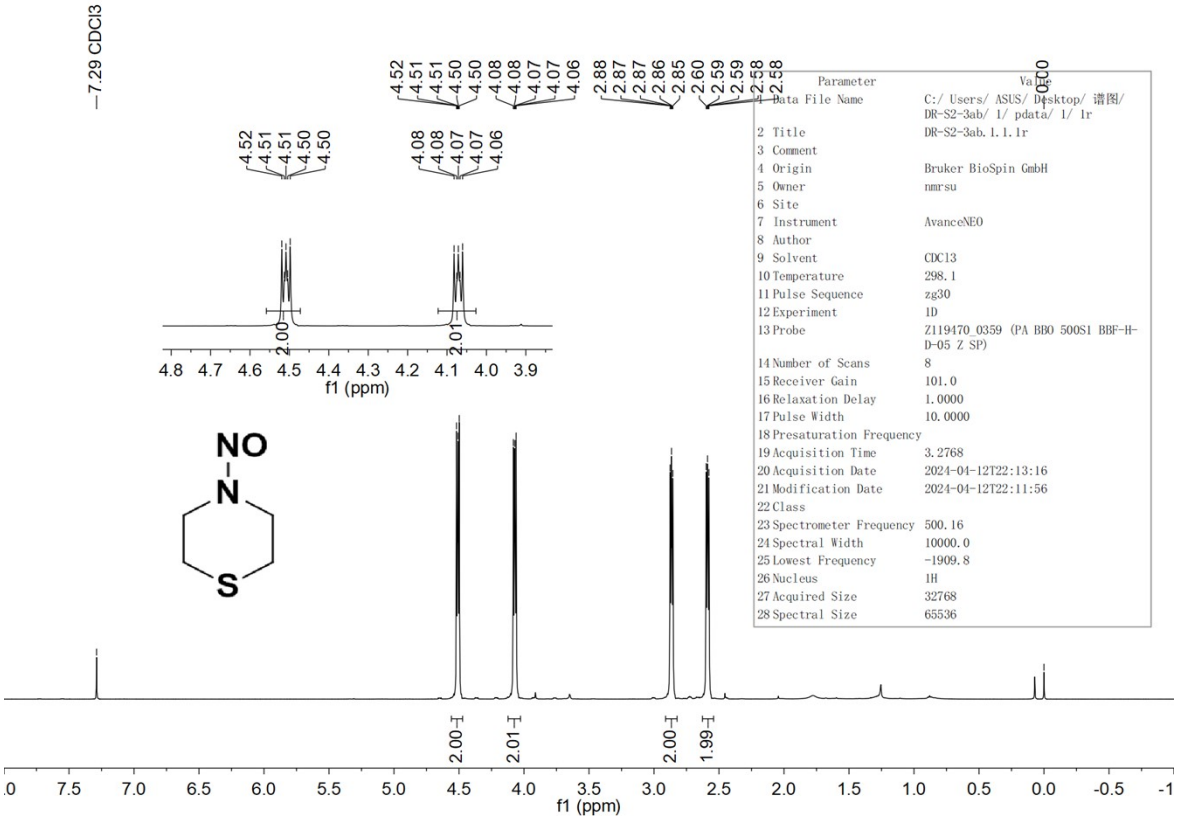




4-nitrosothiomorpholin（3e）



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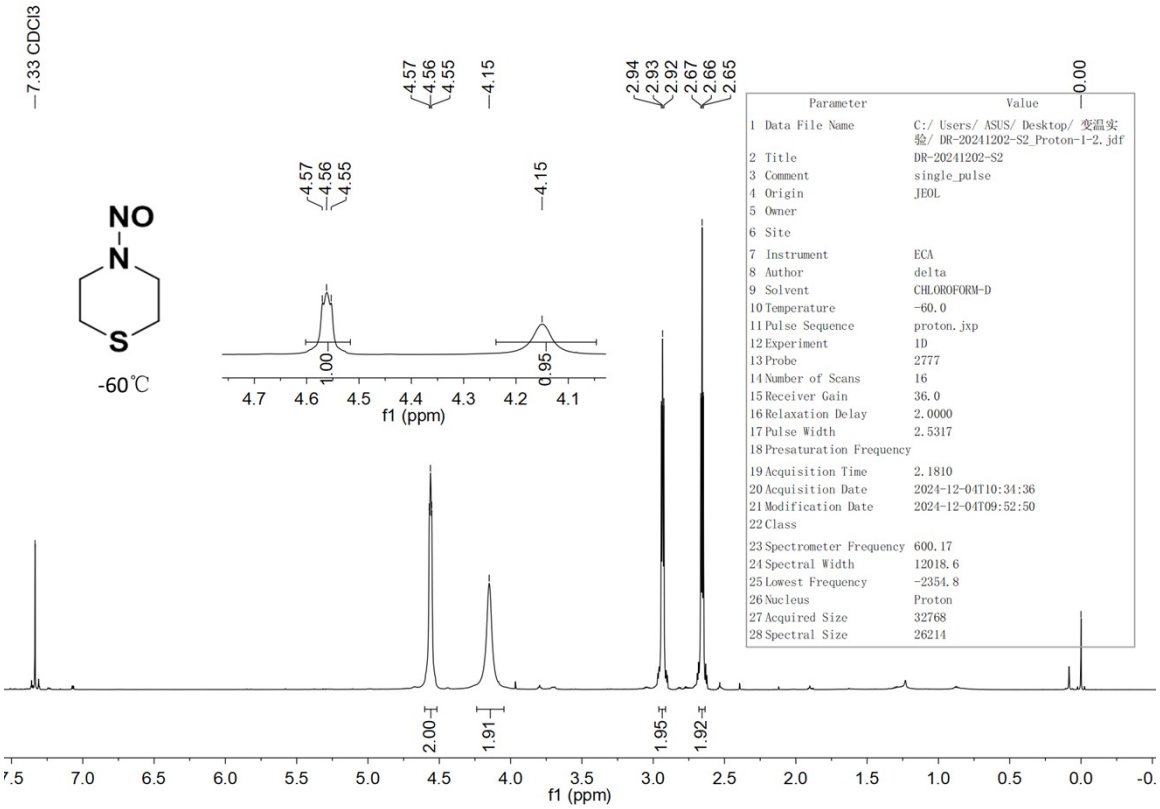
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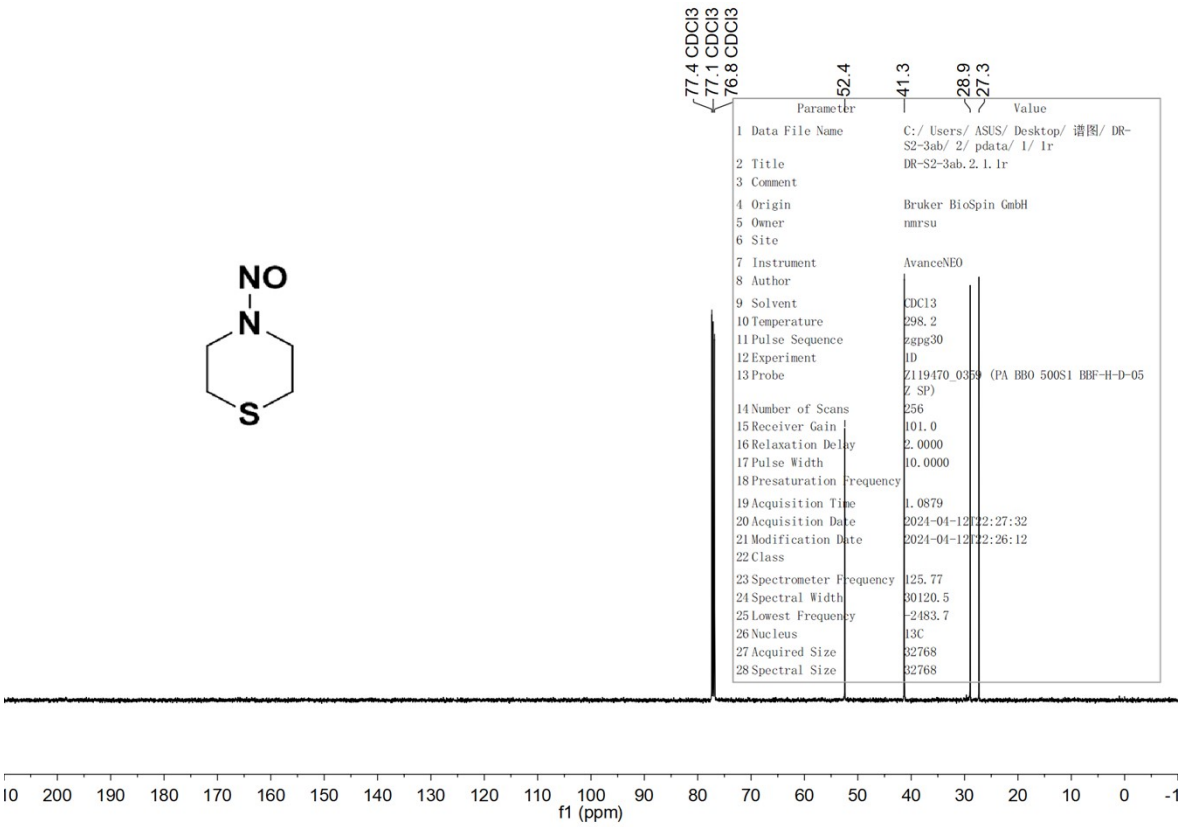
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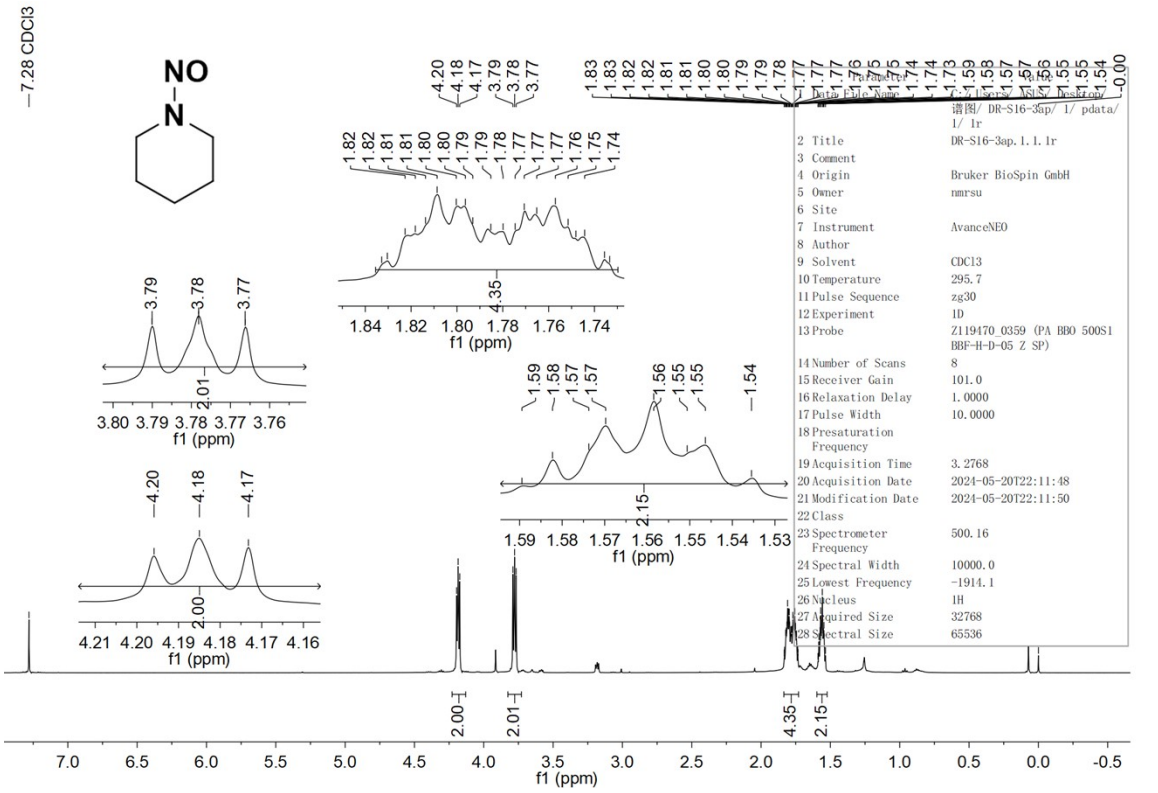
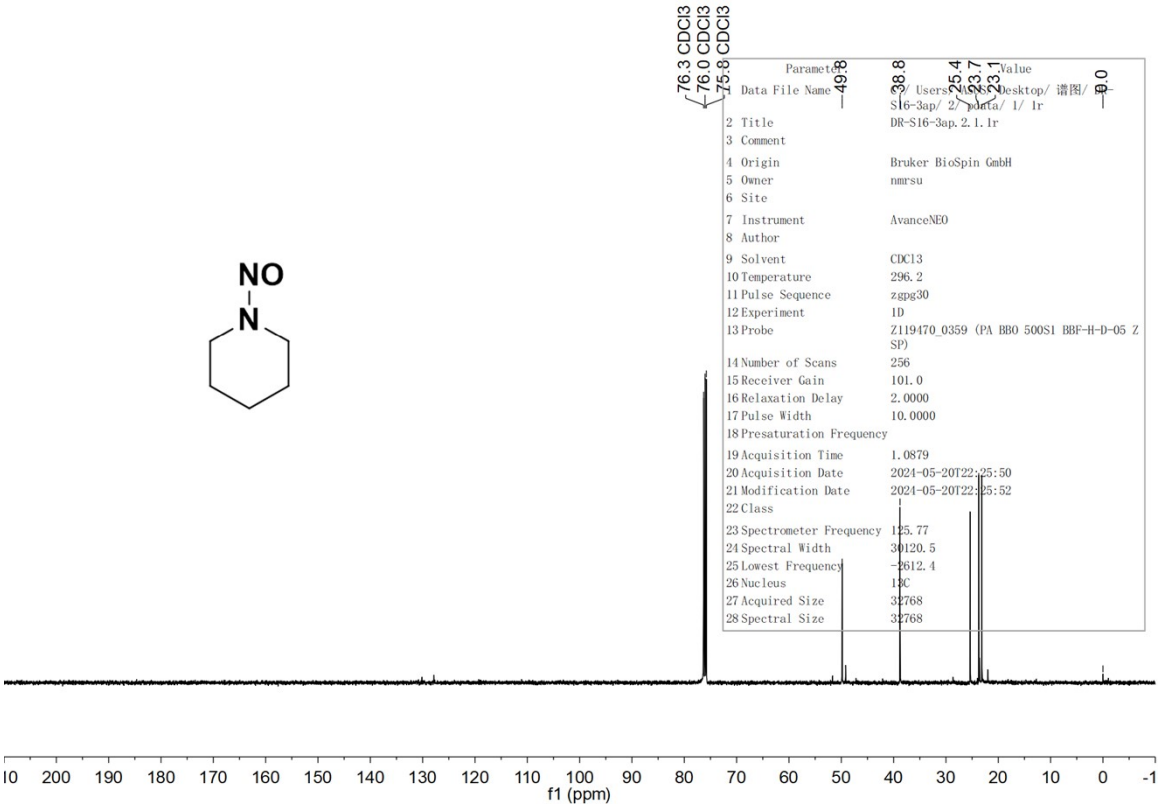
1-nitrosopiperidine(3f)





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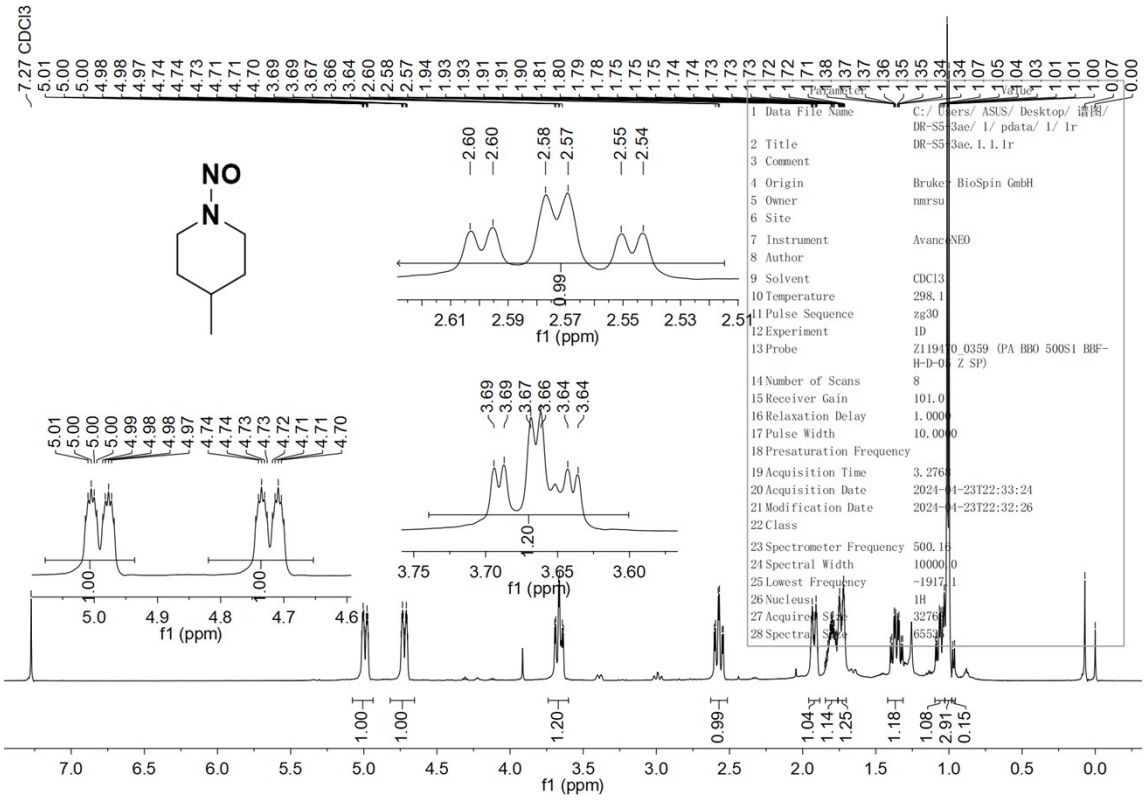

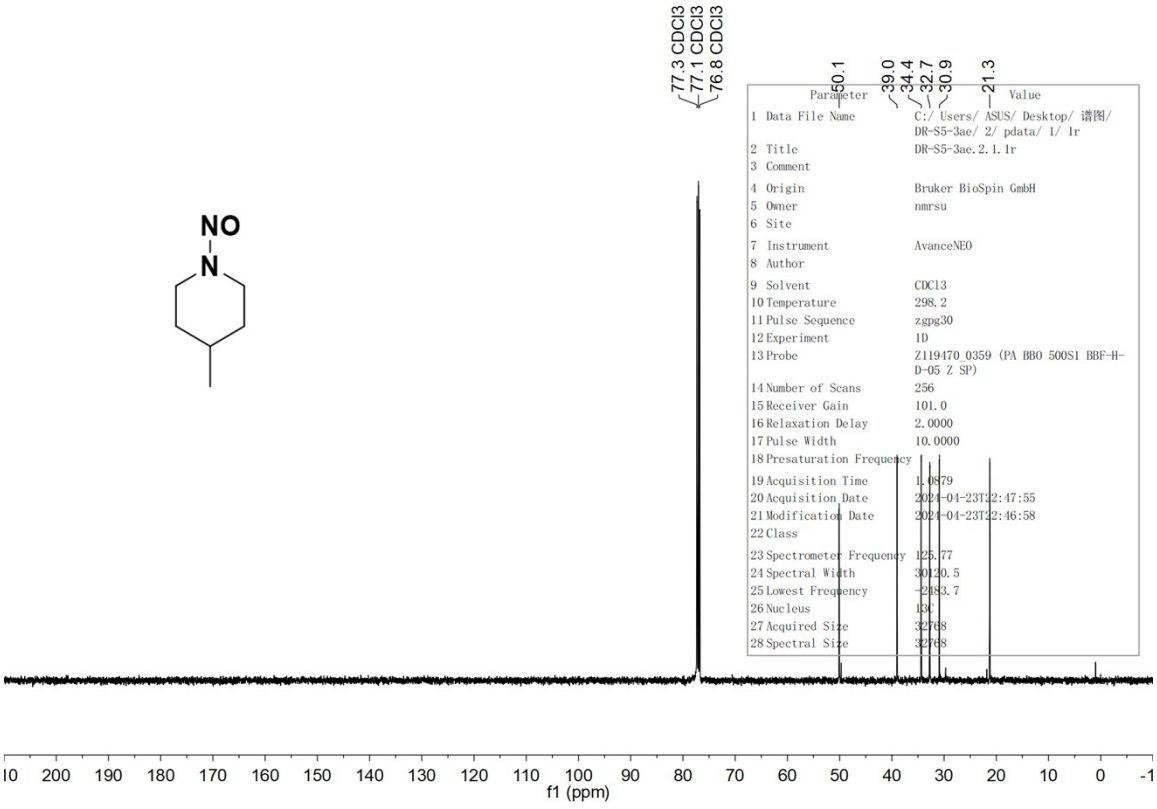

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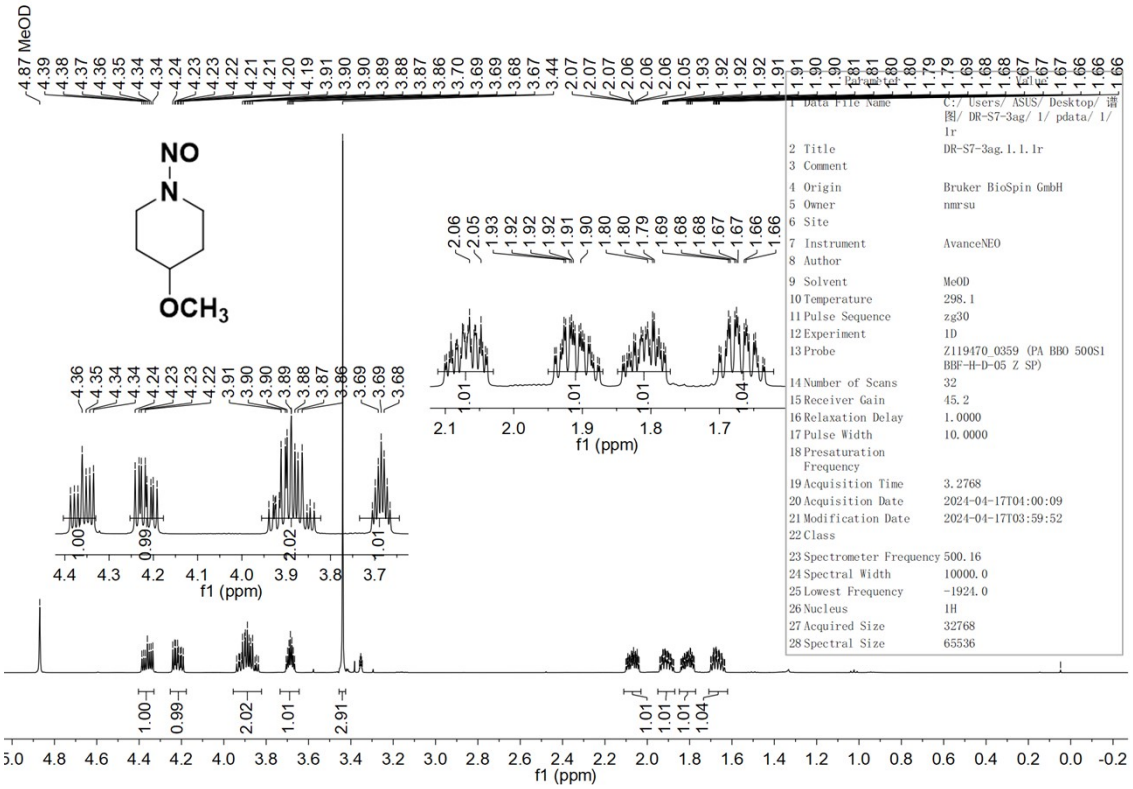

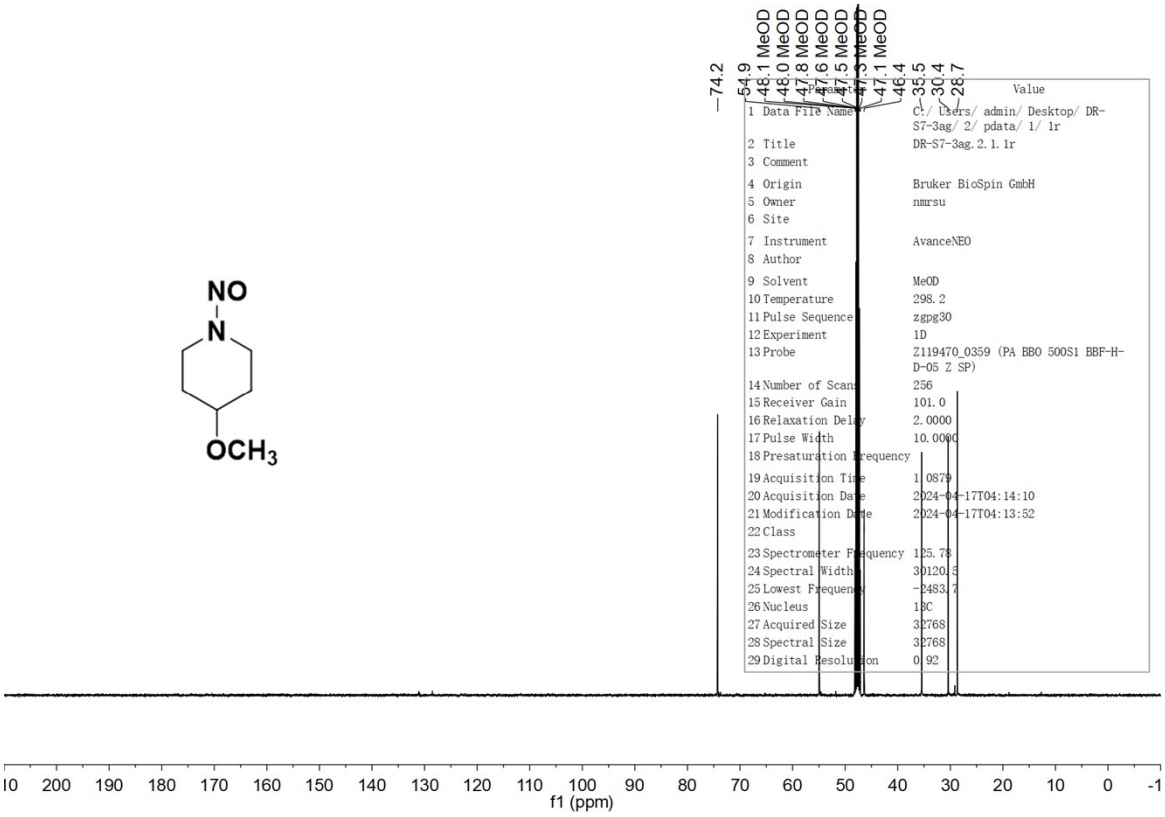

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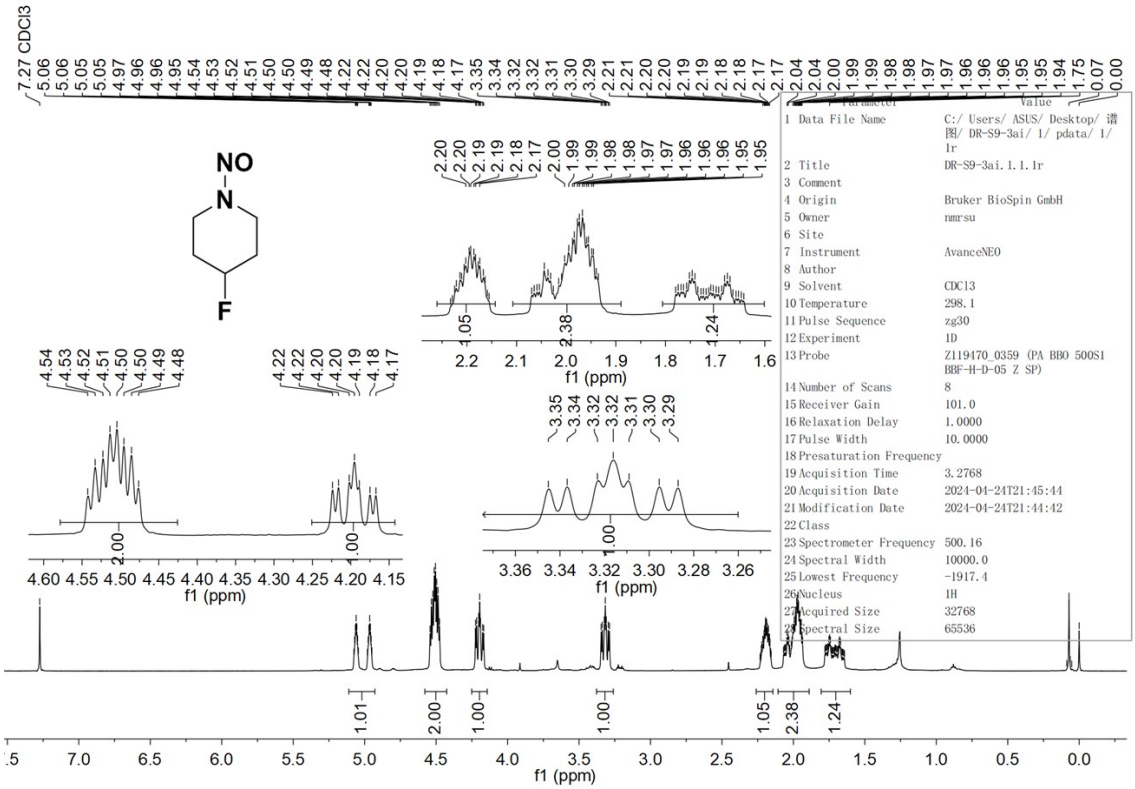

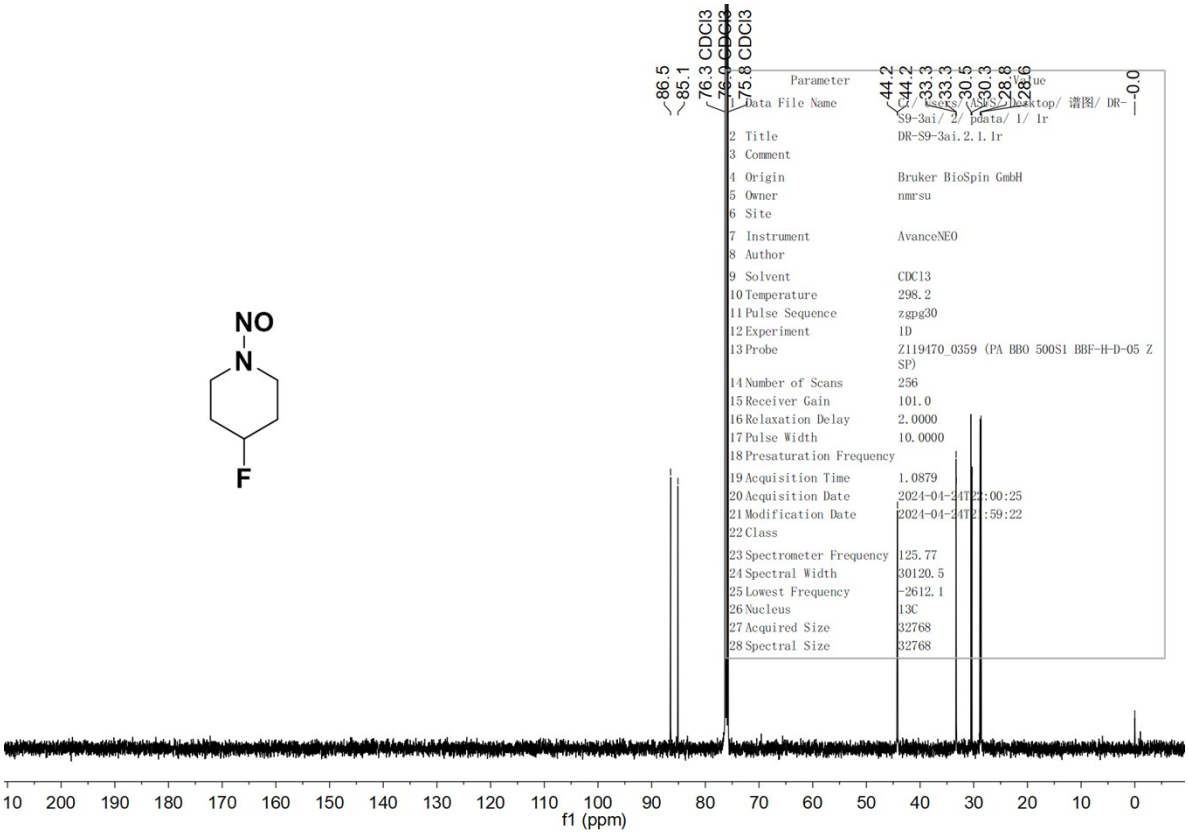

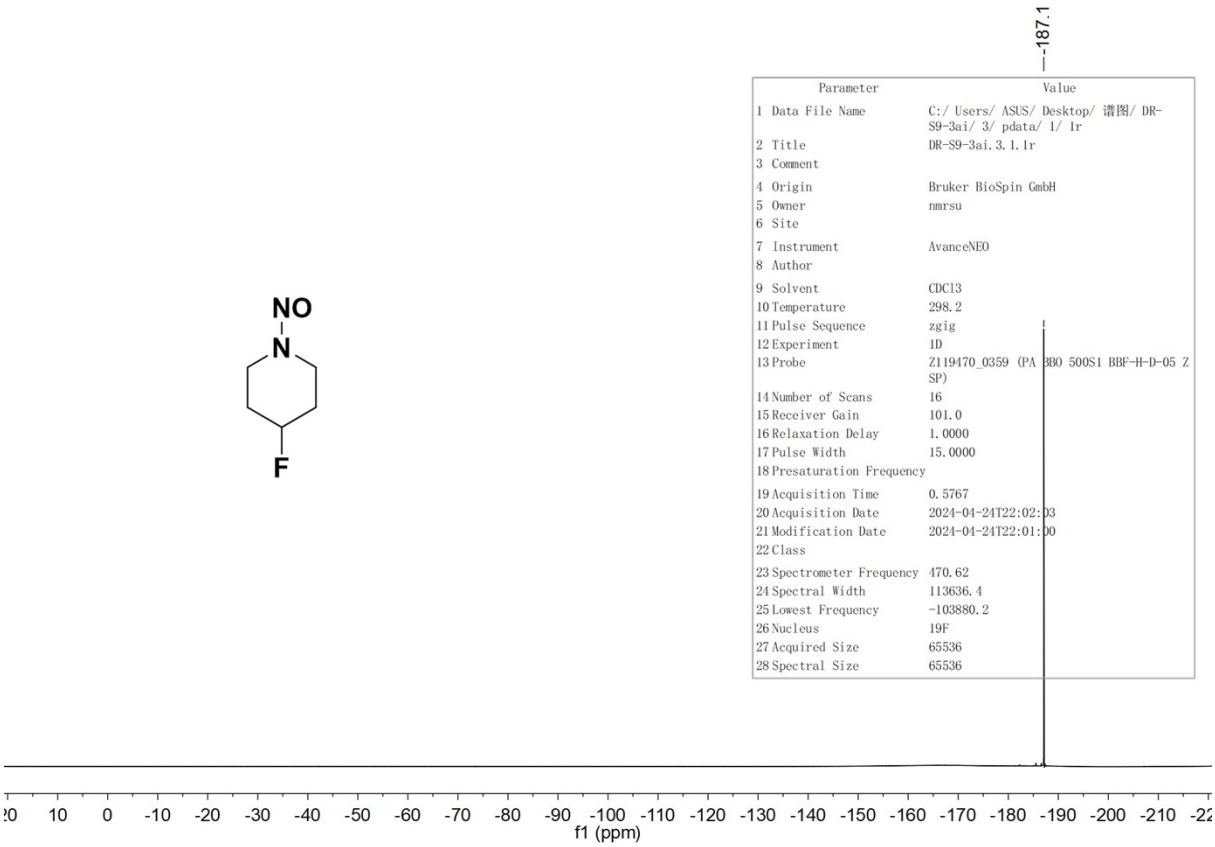

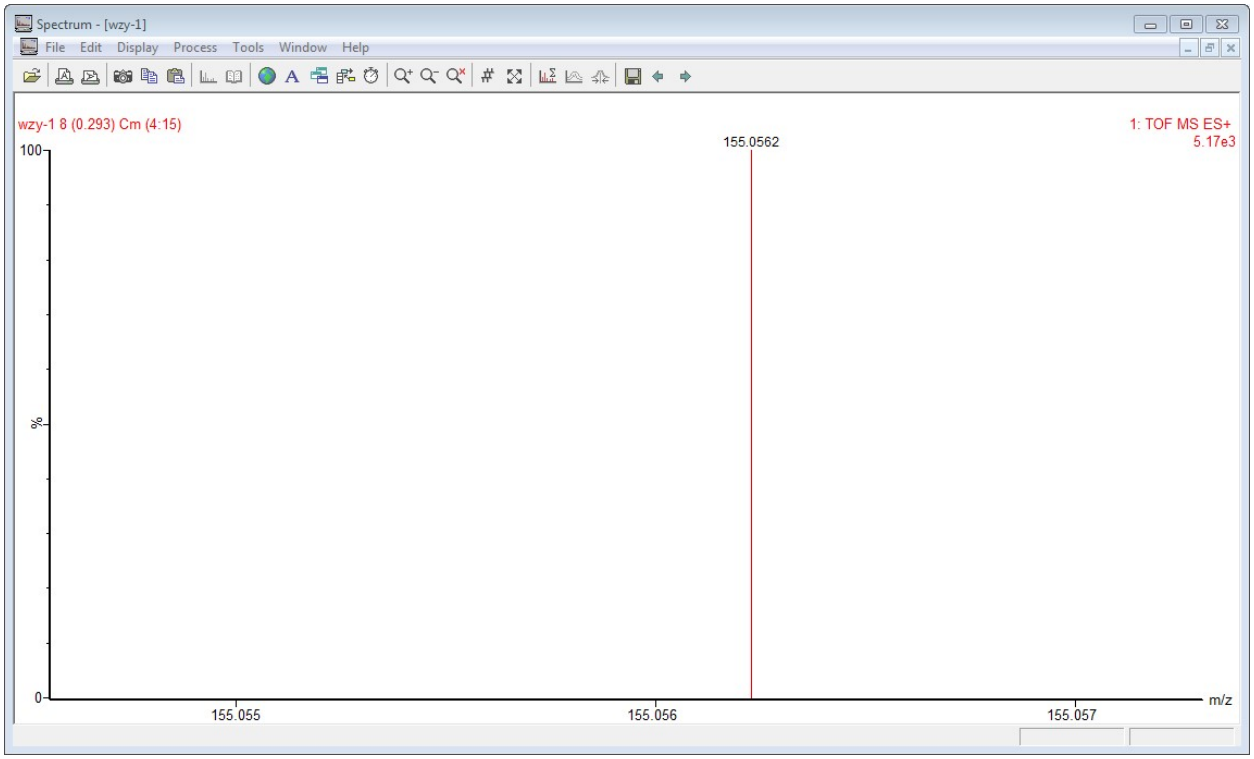

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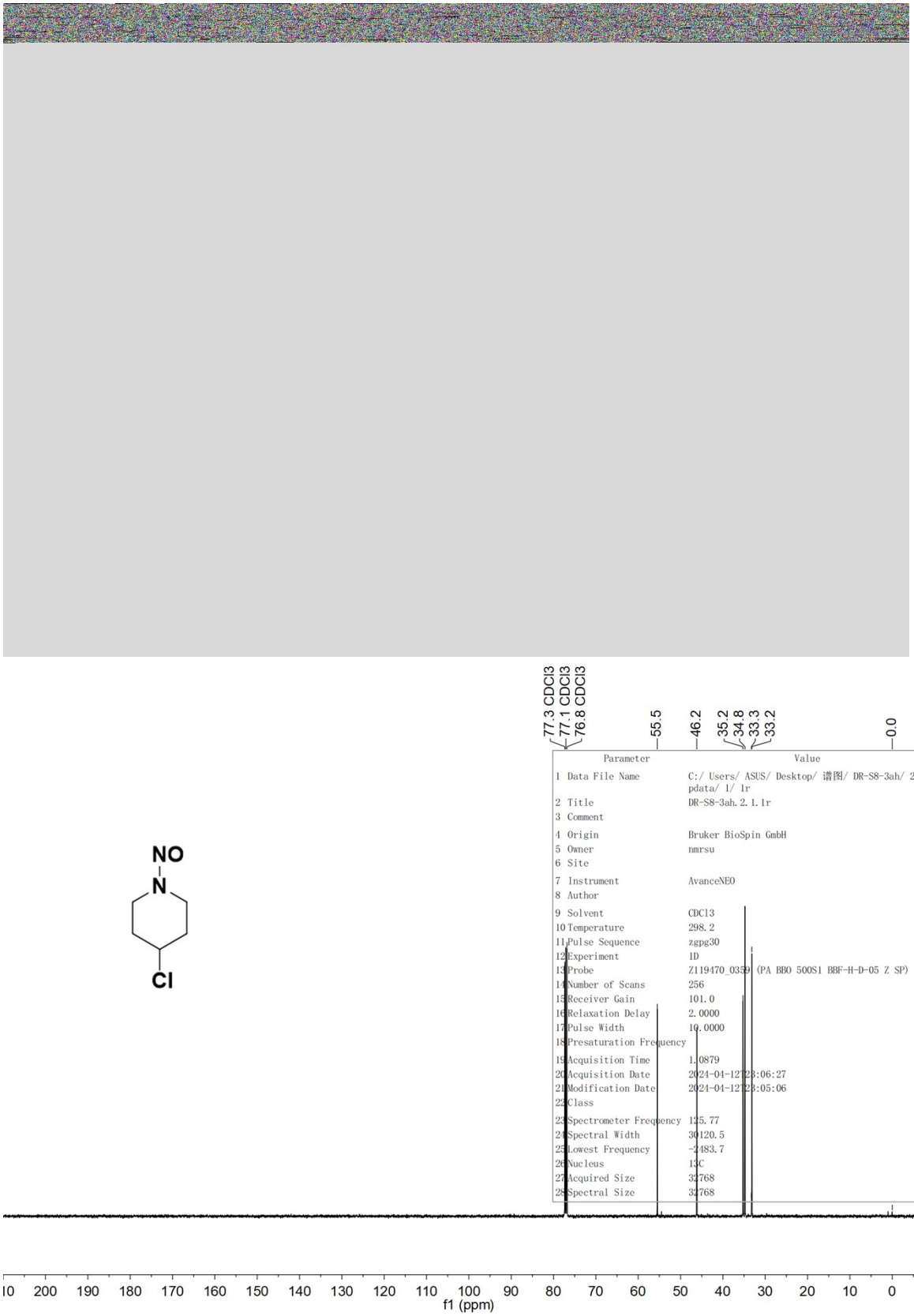

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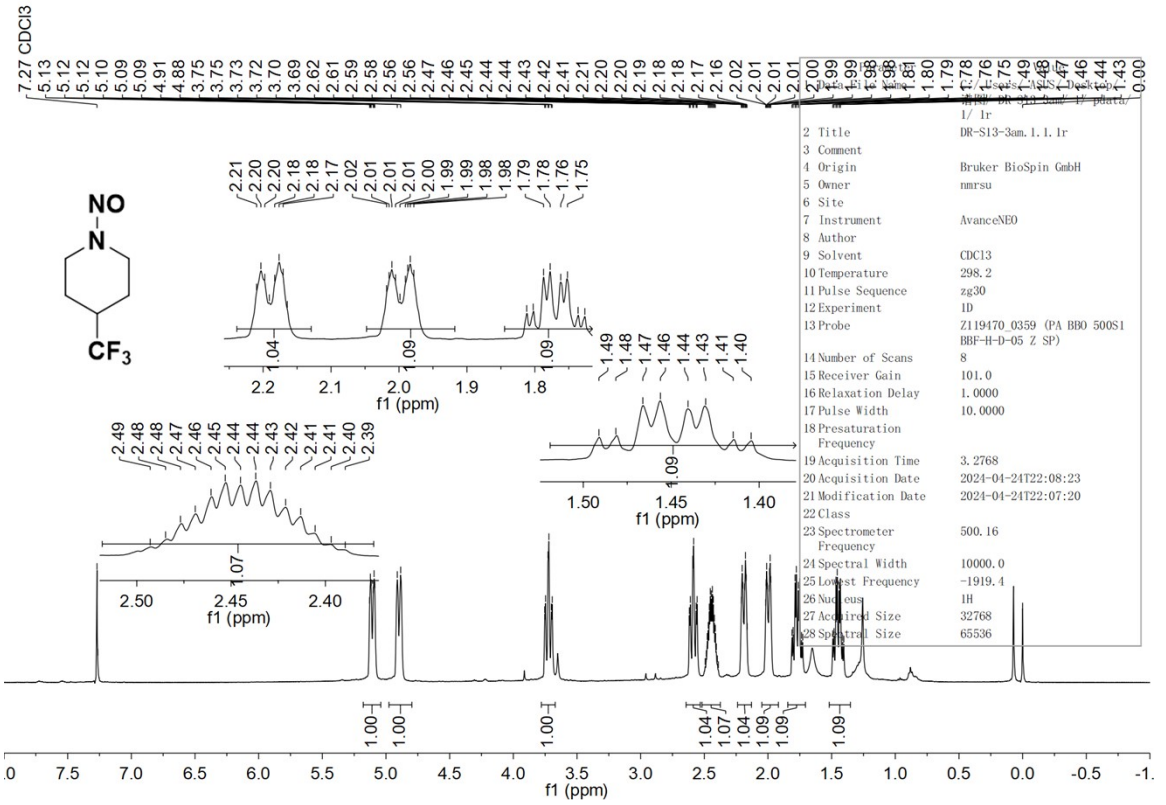
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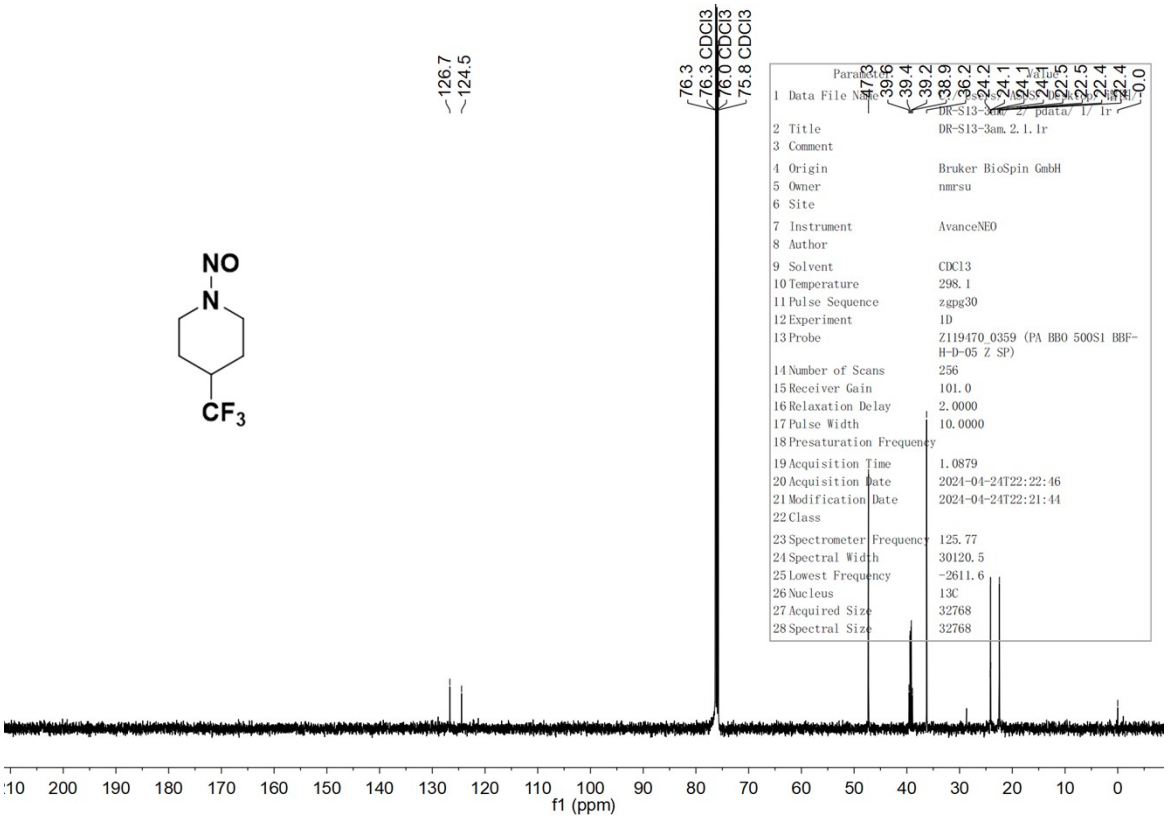
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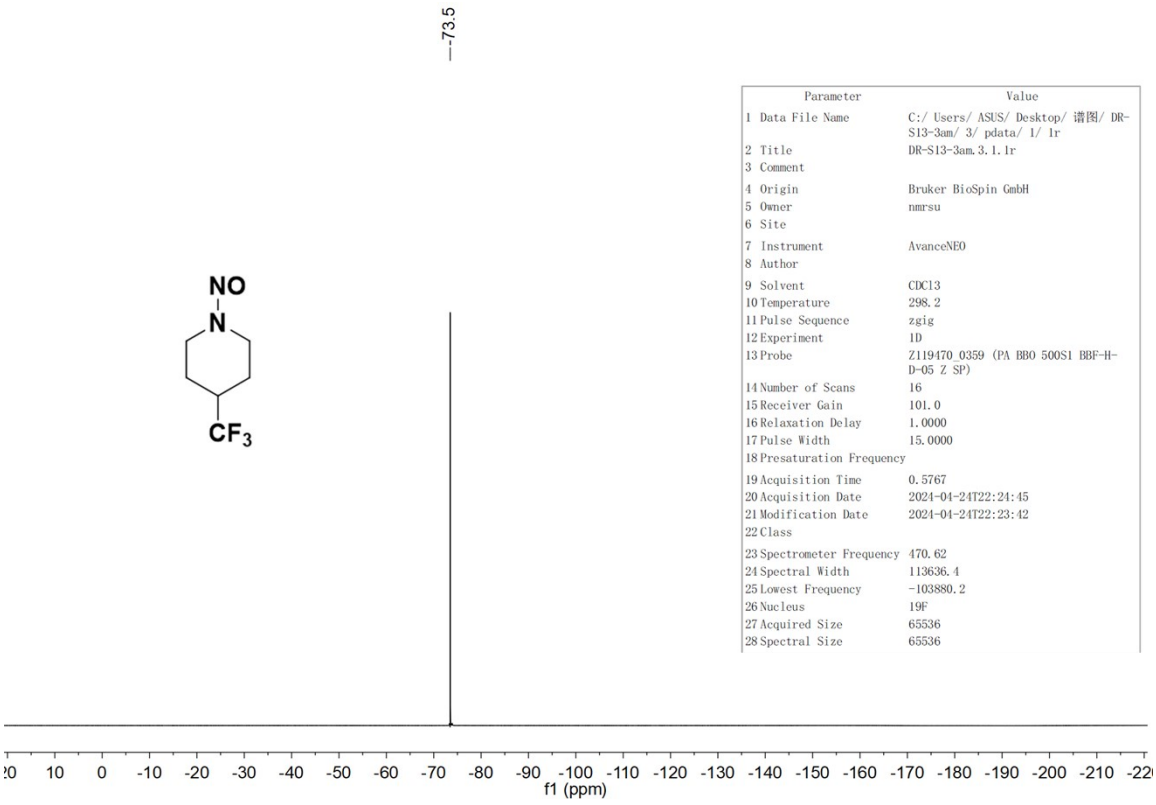

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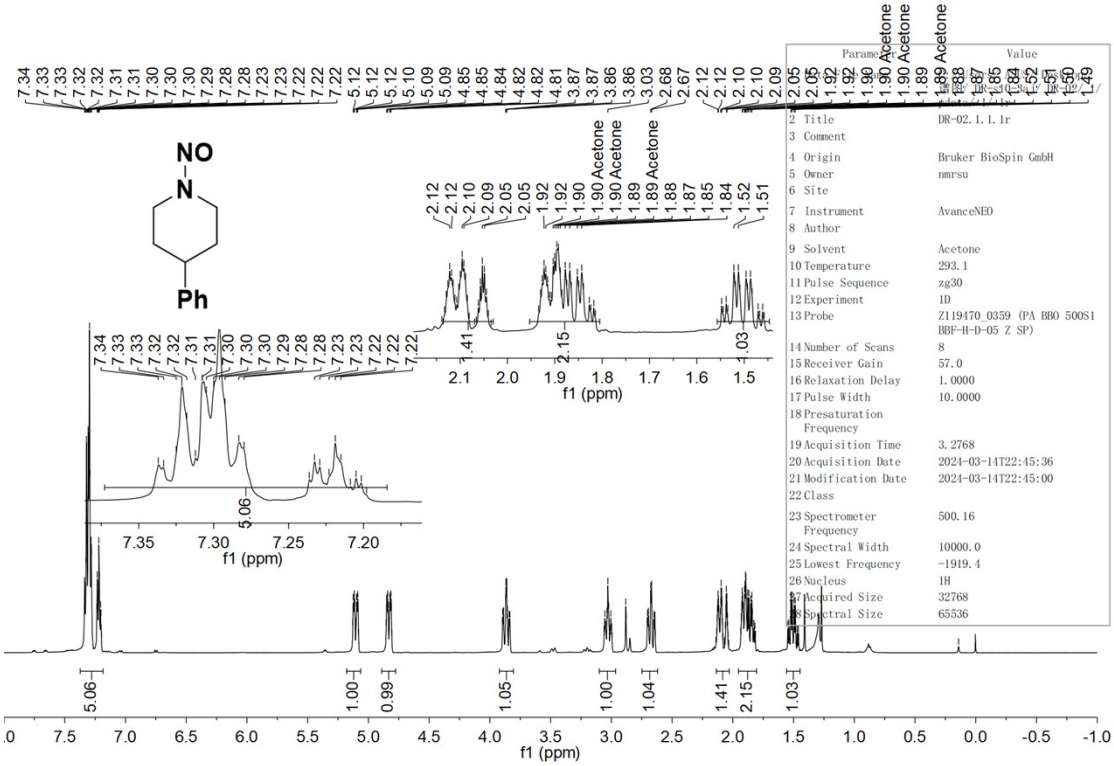
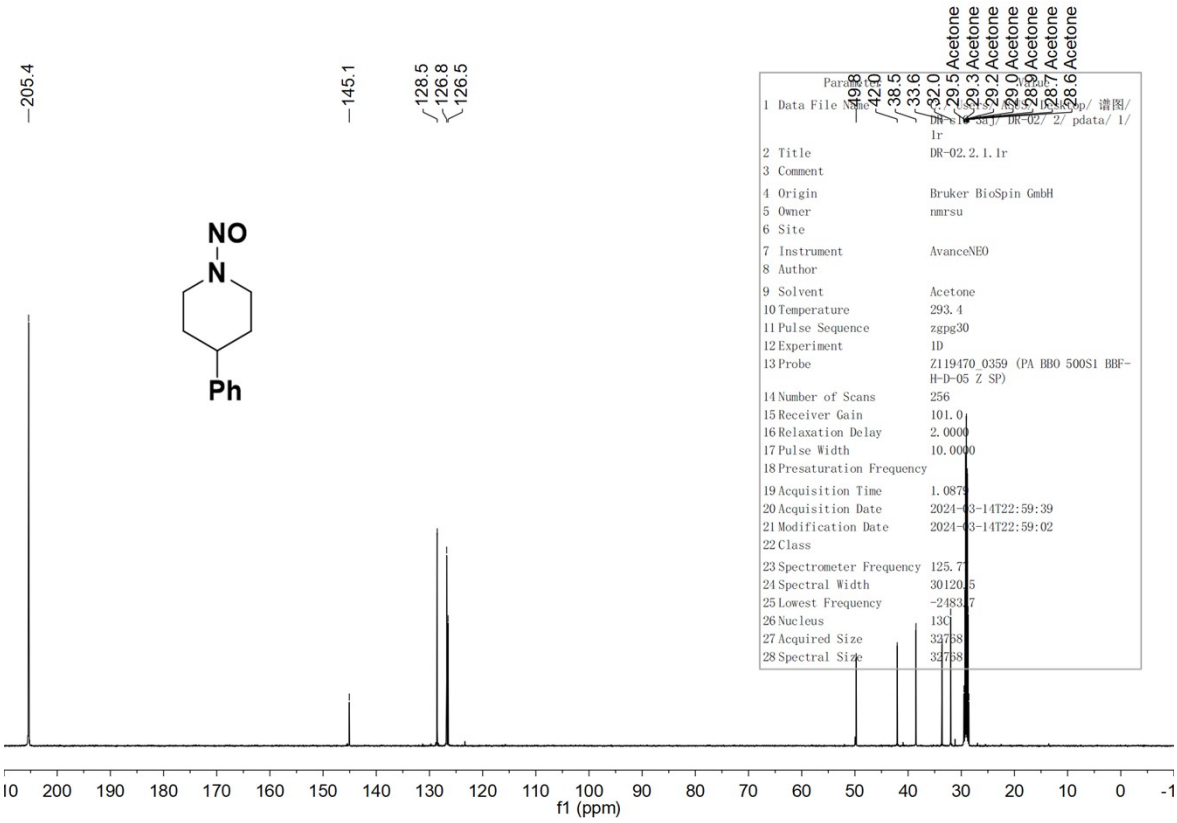
22 Class	
23 Spectrometer Frequency	470.62
24 Spectral Width	113636.4
25 Lowest Frequency	-103880.2
26 Nucleus	19F
27 Acquired Size	65536
28 Spectral Size	65536

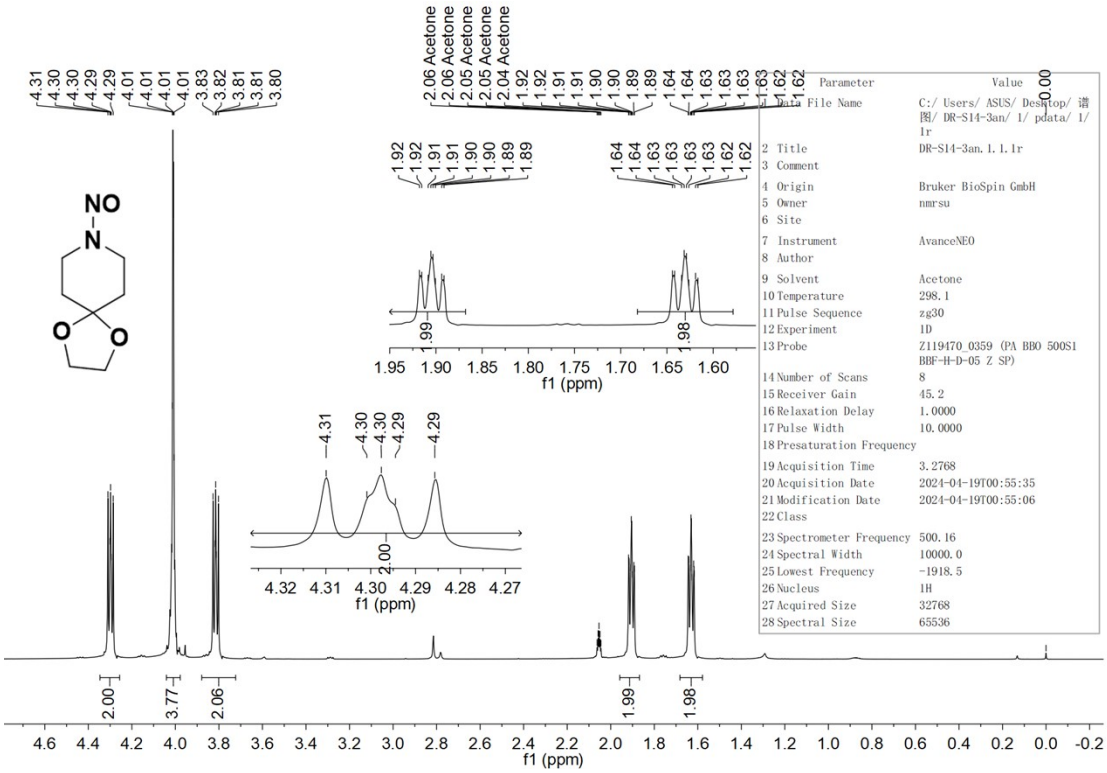
1-nitroso-4-phenylpiperidine(3l)

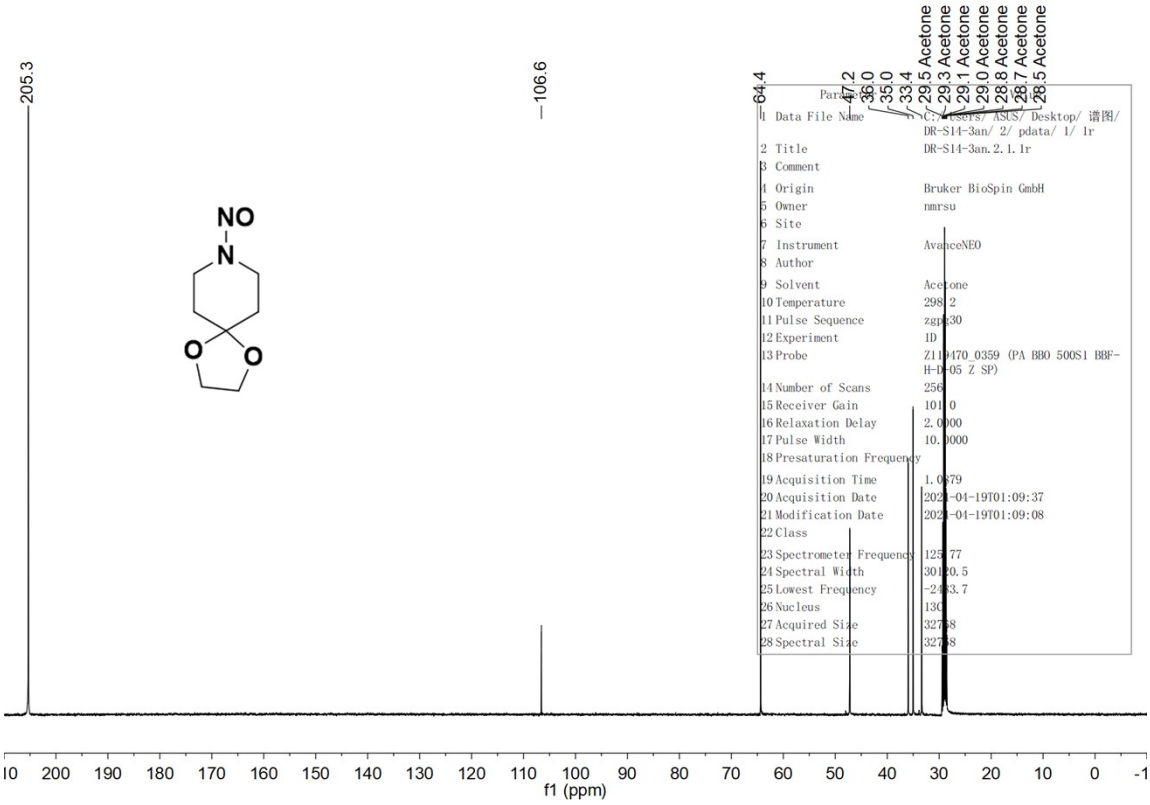


Parameter	Value
2 Title	DR-02.1.1.1r
3 Comment	
4 Origin	Bruker BioSpin GmbH
5 Owner	nmrsu
6 Site	
7 Instrument	AvanceNEO
8 Author	
9 Solvent	Acetone
10 Temperature	293.1
11 Pulse Sequence	zg30
12 Experiment	1D
13 Probe	Z119470_0359 (PA BBO 500S1 BBF-H-D-05 Z SP)
14 Number of Scans	8
15 Receiver Gain	57.0
16 Relaxation Delay	1.0000
17 Pulse Width	10.0000
18 Presaturation Frequency	
19 Acquisition Time	3.2768
20 Acquisition Date	2024-03-14T22:45:36
21 Modification Date	2024-03-14T22:45:00
22 Class	
23 Spectrometer Frequency	500.16
24 Spectral Width	10000.0
25 Lowest Frequency	-1919.4
26 Nucleus	1H
27 Acquired Size	32768
28 Spectral Size	65536

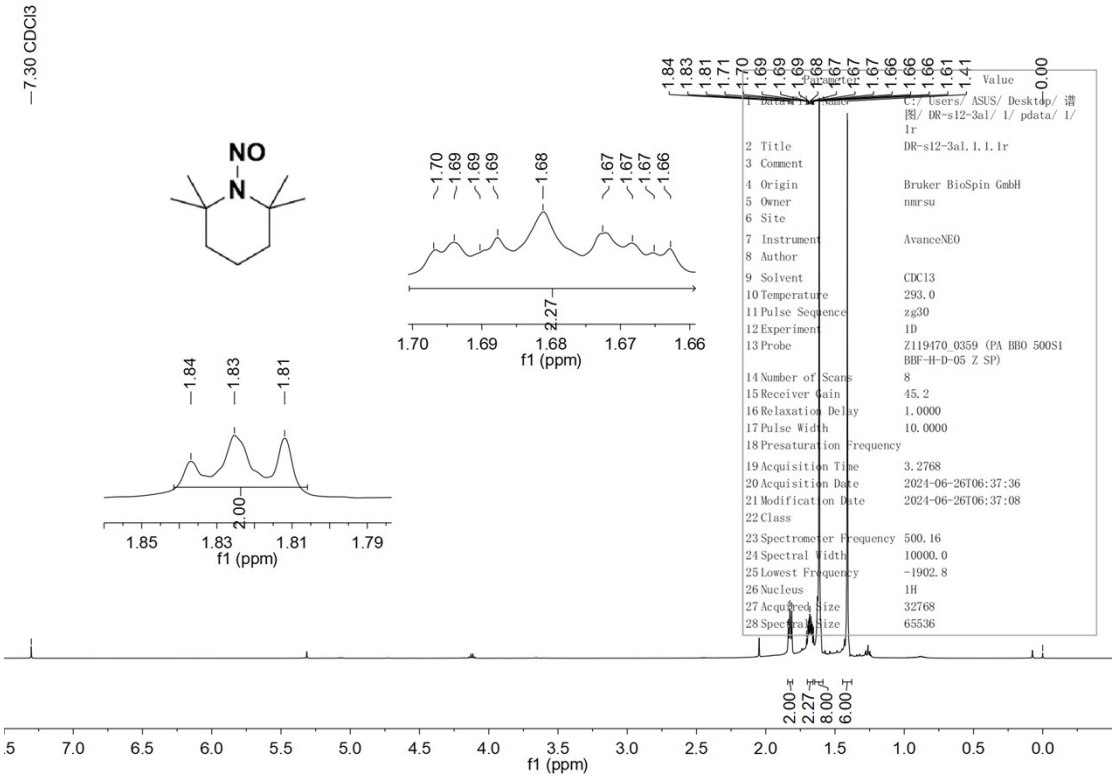


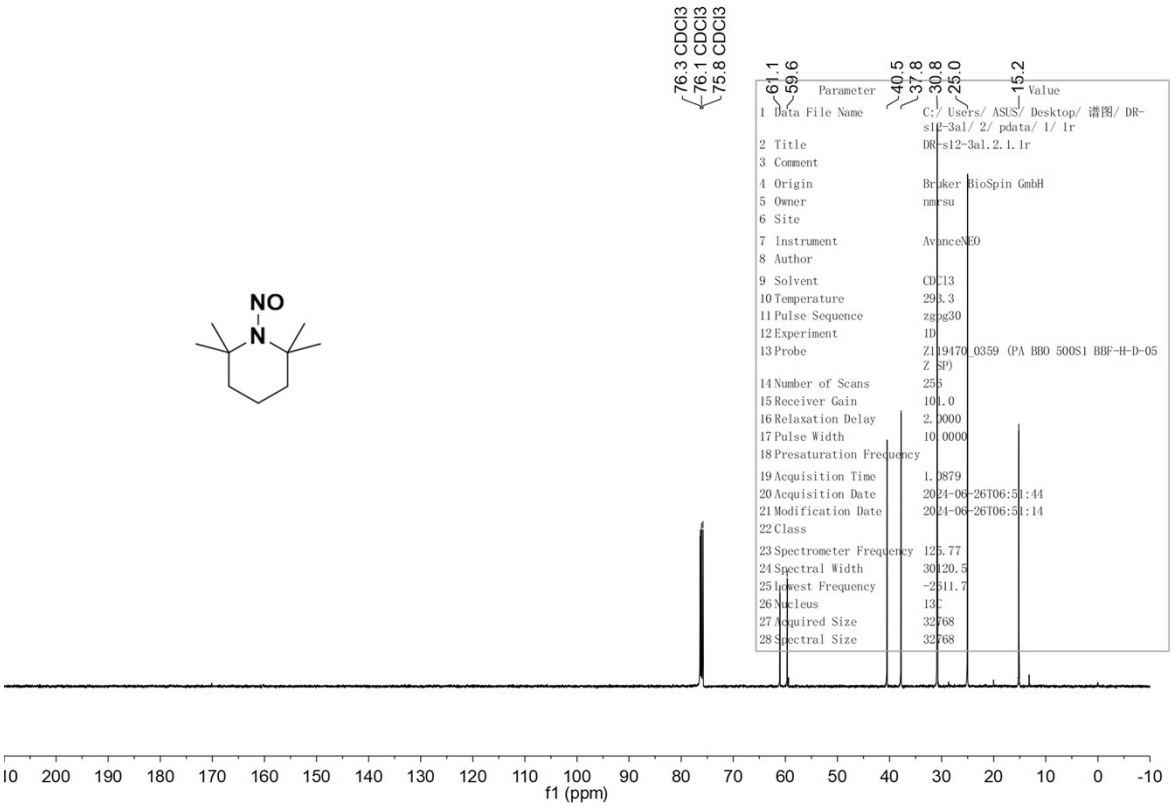
8-nitroso-1,4-dioxa-8-azaspiro[4.5]decane(3m)





2,2,6,6-tetramethyl-1-nitrosopiperidine（3n）





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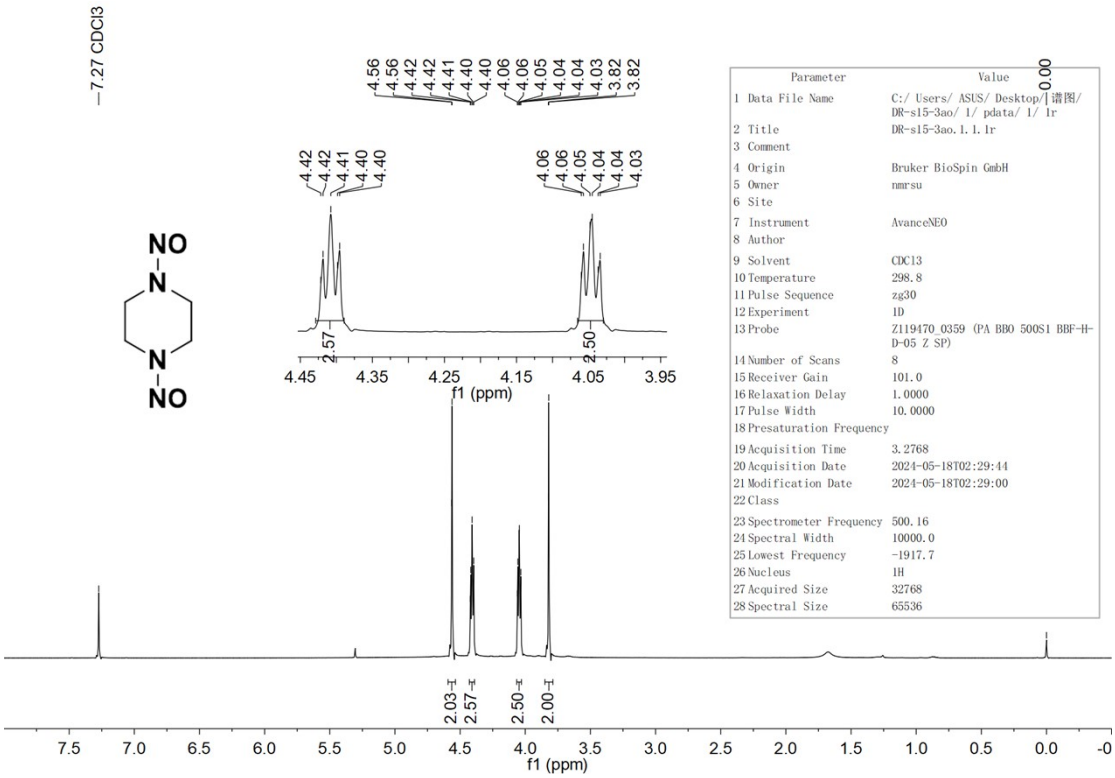
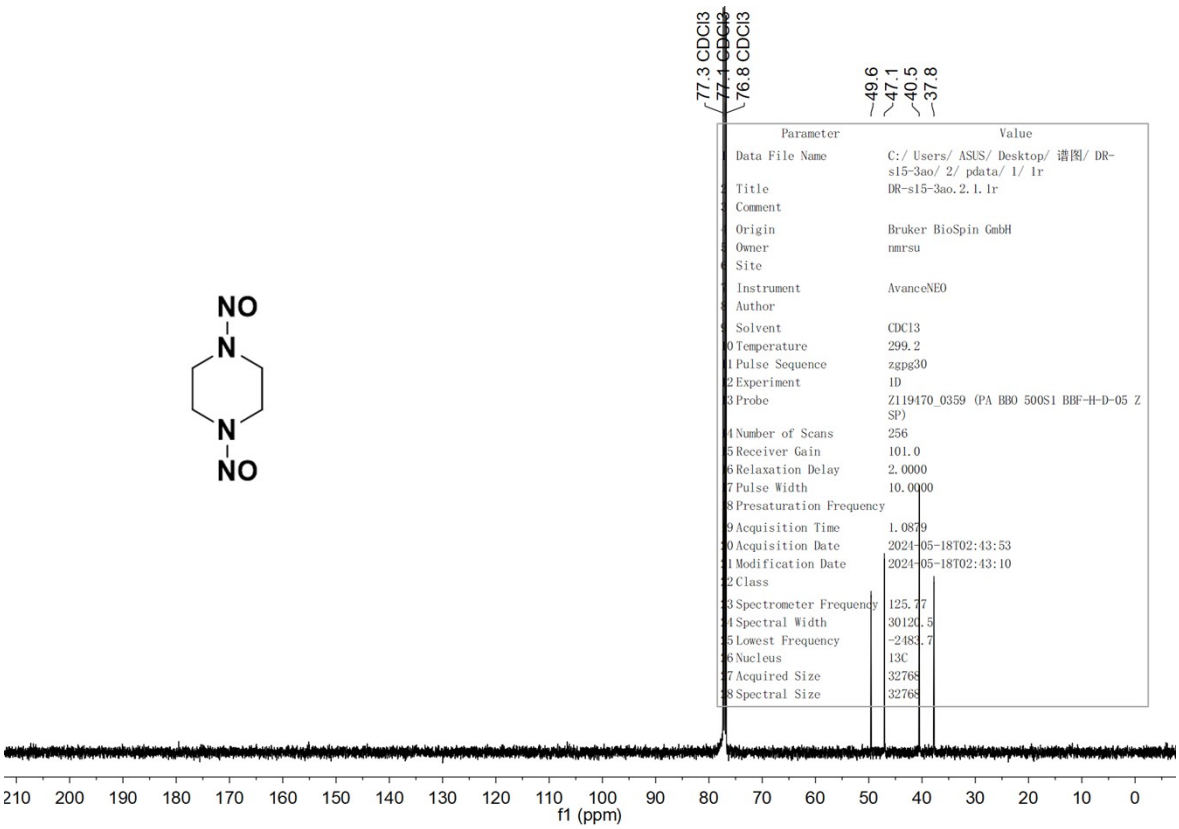
1 Data File Name	C:/ Users/ ASUS/ Desktop/ 谱图/ DR-s12-3al/ 2/ pdata/ 1/ 1r
2 Title	DR-s12-3al.2.1.1r
3 Comment	
4 Origin	Bruker BioSpin GmbH
5 Owner	nmrsu
6 Site	
7 Instrument	AvanceNEO
8 Author	
9 Solvent	CDCl3
10 Temperature	298.3
11 Pulse Sequence	zgpg30
12 Experiment	1D
13 Probe	Z119470_0359 (PA BBO 500S1 BBF-H-D-05 Z SP)
14 Number of Scans	256
15 Receiver Gain	101.0
16 Relaxation Delay	2.0000
17 Pulse Width	10.0000
18 Presaturation Frequency	
19 Acquisition Time	1.0879
20 Acquisition Date	2024-06-26T06:51:44
21 Modification Date	2024-06-26T06:51:14
22 Class	
23 Spectrometer Frequency	125.77
24 Spectral Width	30120.5
25 Lowest Frequency	-2511.7
26 Nucleus	13C
27 Acquired Size	32768
28 Spectral Size	32768

1,4-dinitrosopiperazine(3o)

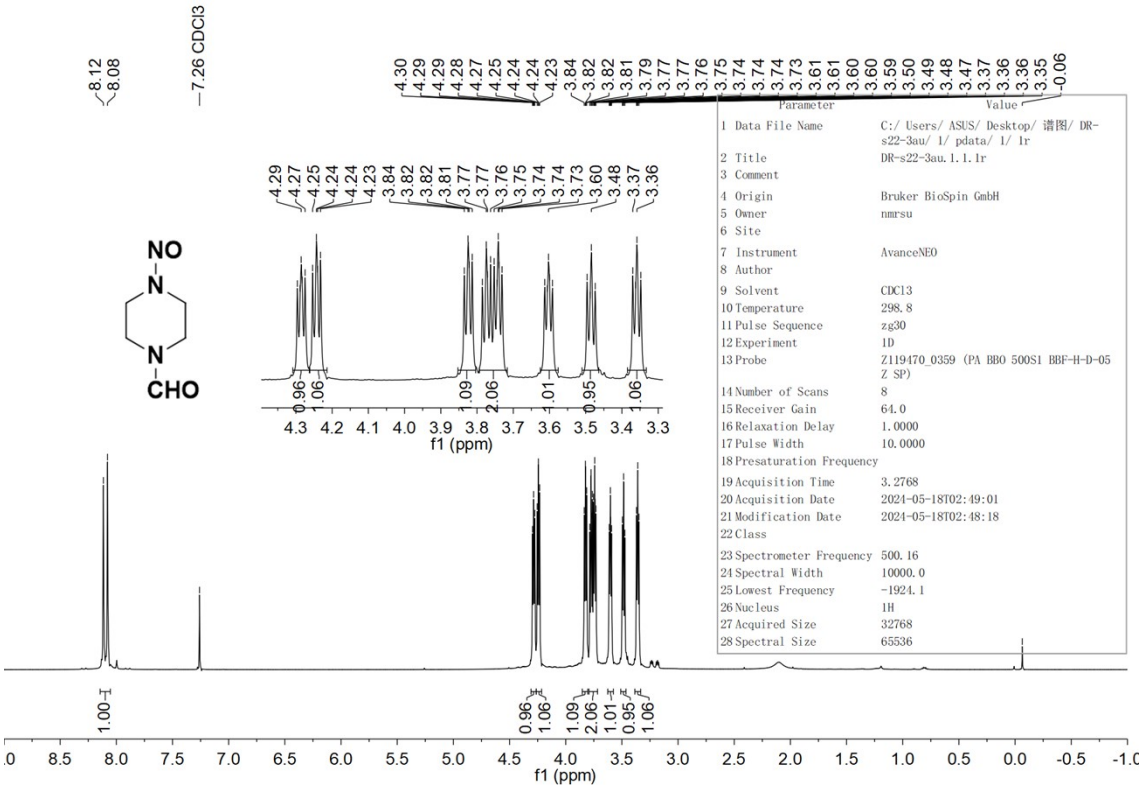


Parameter	Value
1 Data File Name	C:/ Users/ ASUS/ Desktop/谱图/ DR-s15-3ao/ 1/ pdata/ 1/ 1r
2 Title	DR-s15-3ao.1.1.1r
3 Comment	
4 Origin	Bruker BioSpin GmbH
5 Owner	nmrsu
6 Site	
7 Instrument	AvanceNEO
8 Author	
9 Solvent	CDCl3
10 Temperature	298.8
11 Pulse Sequence	zg30
12 Experiment	1D
13 Probe	Z119470_0359 (PA BBO 500S1 BBF-H-D-05 Z SP)
14 Number of Scans	8
15 Receiver Gain	101.0
16 Relaxation Delay	1.0000
17 Pulse Width	10.0000
18 Presaturation Frequency	
19 Acquisition Time	3.2768
20 Acquisition Date	2024-05-18T02:29:44
21 Modification Date	2024-05-18T02:29:00
22 Class	
23 Spectrometer Frequency	500.16
24 Spectral Width	10000.0
25 Lowest Frequency	-1917.7
26 Nucleus	1H
27 Acquired Size	32768
28 Spectral Size	65536

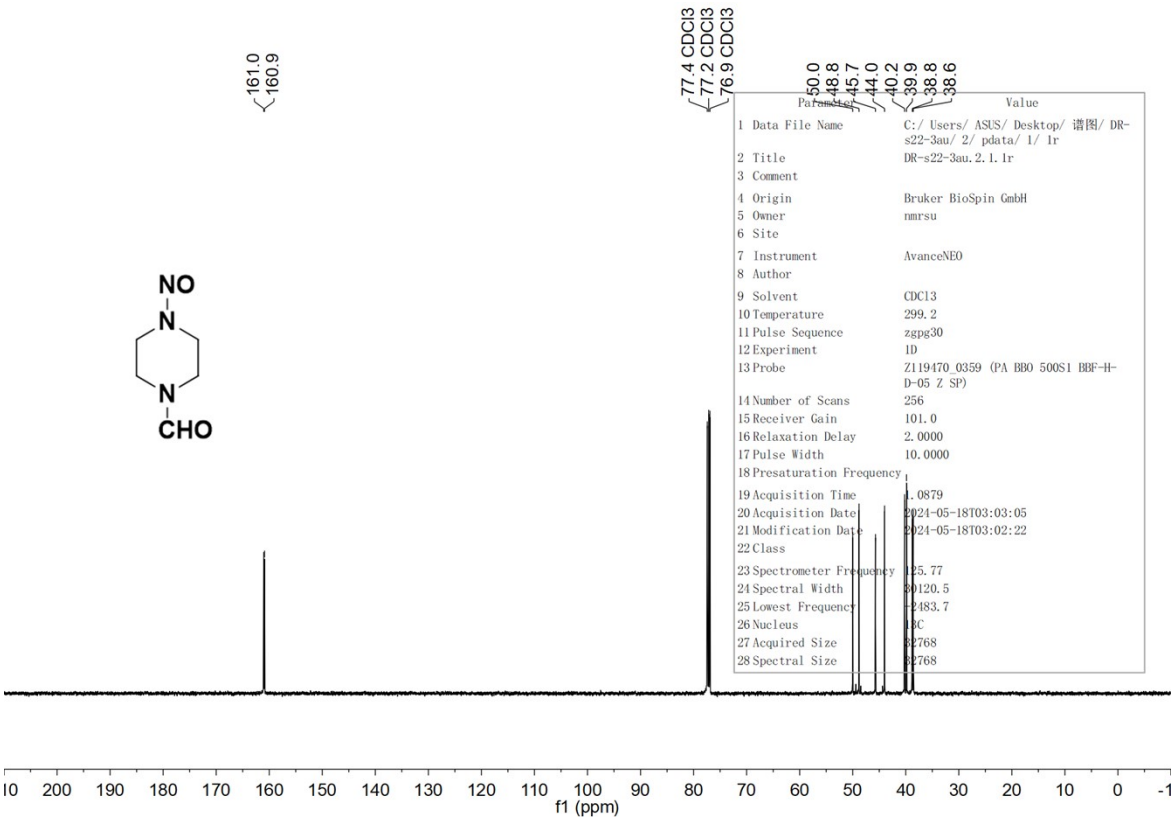


Parameter	Value
Data File Name	C:/ Users/ ASUS/ Desktop/ 谱图/ DR-s15-3ao/ 2/ pdata/ 1/ 1r
Title	DR-s15-3ao.2.1.1r
Comment	
Origin	Bruker BioSpin GmbH
Owner	nmrsu
Site	
Instrument	AvanceNEO
Author	
Solvent	CDCl3
Temperature	299.2
Pulse Sequence	zgpg30
Experiment	1D
Probe	Z119470_0359 (PA BBO 500S1 BBF-H-D-05 Z SP)
Number of Scans	256
Receiver Gain	101.0
Relaxation Delay	2.0000
Pulse Width	10.0000
Presaturation Frequency	
Acquisition Time	1.0879
Acquisition Date	2024-05-18T02:43:53
Modification Date	2024-05-18T02:43:10
Class	
Spectrometer Frequency	125.77
Spectral Width	30120.5
Lowest Frequency	-2483.7
Nucleus	13C
Acquired Size	32768
Spectral Size	32768

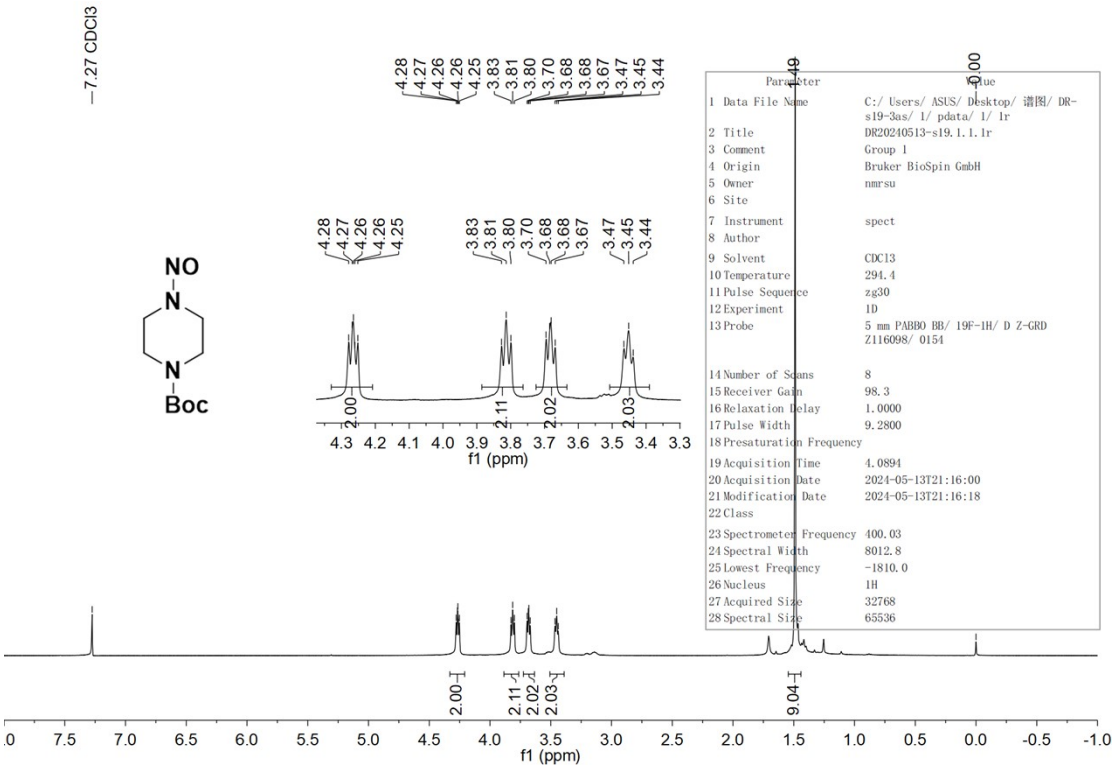
4-nitrosopiperazine-1-carbaldehyde(3p)

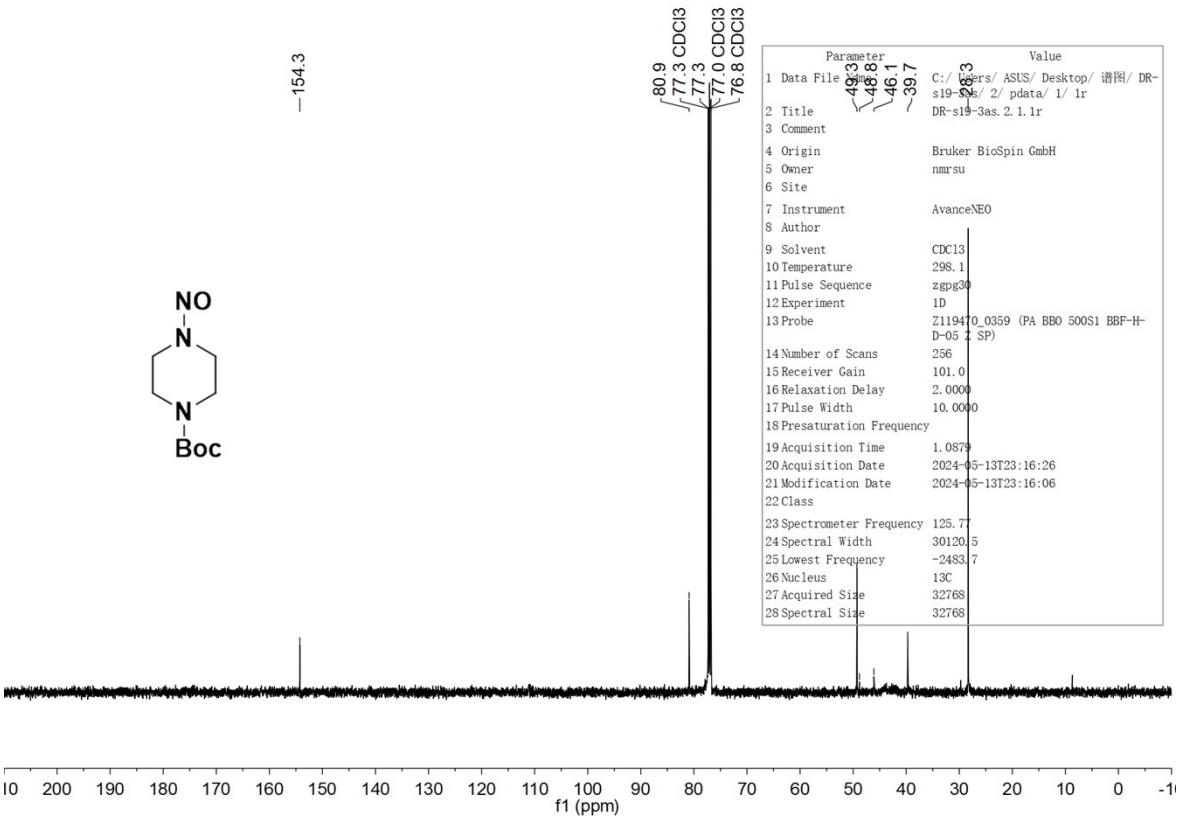


Parameter	Value
Data File Name	C:/ Users/ ASUS/ Desktop/ 谱图/ DR-s22-3au/ 1/ pdata/ 1/ 1r
Title	DR-s22-3au.1.1.1r
Comment	
Origin	Bruker BioSpin GmbH
Owner	nmrsu
Site	
Instrument	AvanceNEO
Author	
Solvent	CDCl3
Temperature	298.8
Pulse Sequence	zg30
Experiment	1D
Probe	Z119470_0359 (PA BBO 500S1 BBF-H-D-05 Z SP)
Number of Scans	8
Receiver Gain	64.0
Relaxation Delay	1.0000
Pulse Width	10.0000
Presaturation Frequency	
Acquisition Time	3.2768
Acquisition Date	2024-05-18T02:49:01
Modification Date	2024-05-18T02:48:18
Class	
Spectrometer Frequency	500.16
Spectral Width	10000.0
Lowest Frequency	-1924.1
Nucleus	1H
Acquired Size	32768
Spectral Size	65536

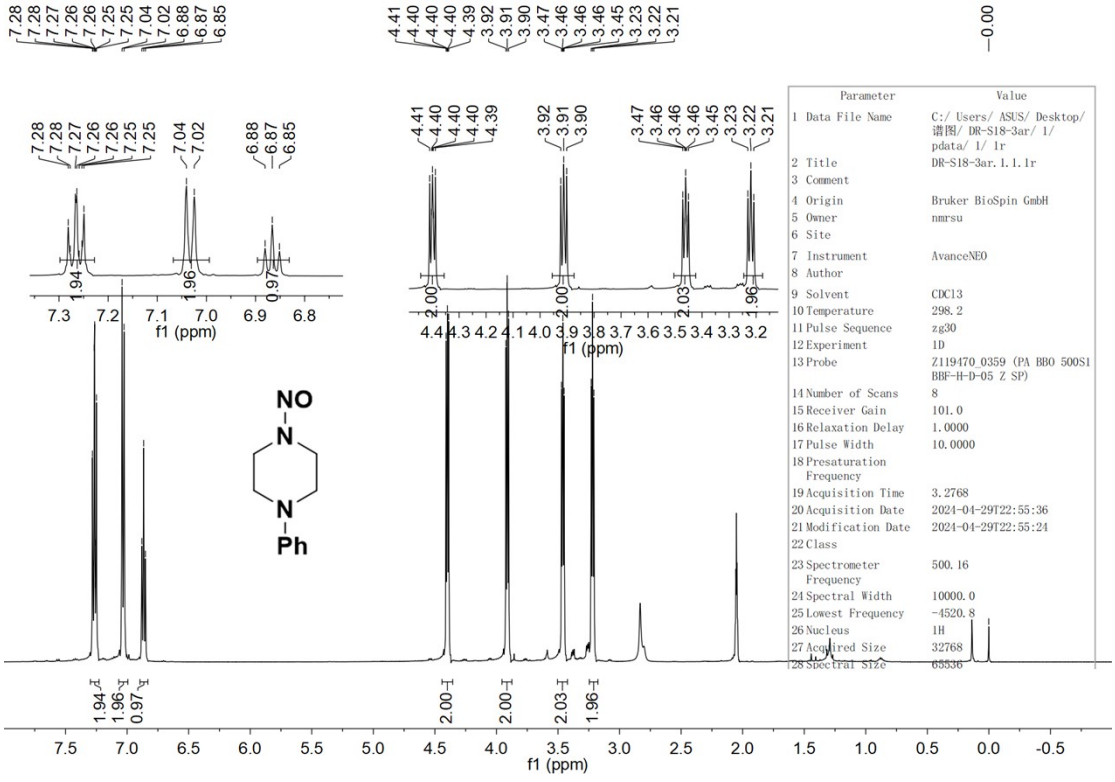


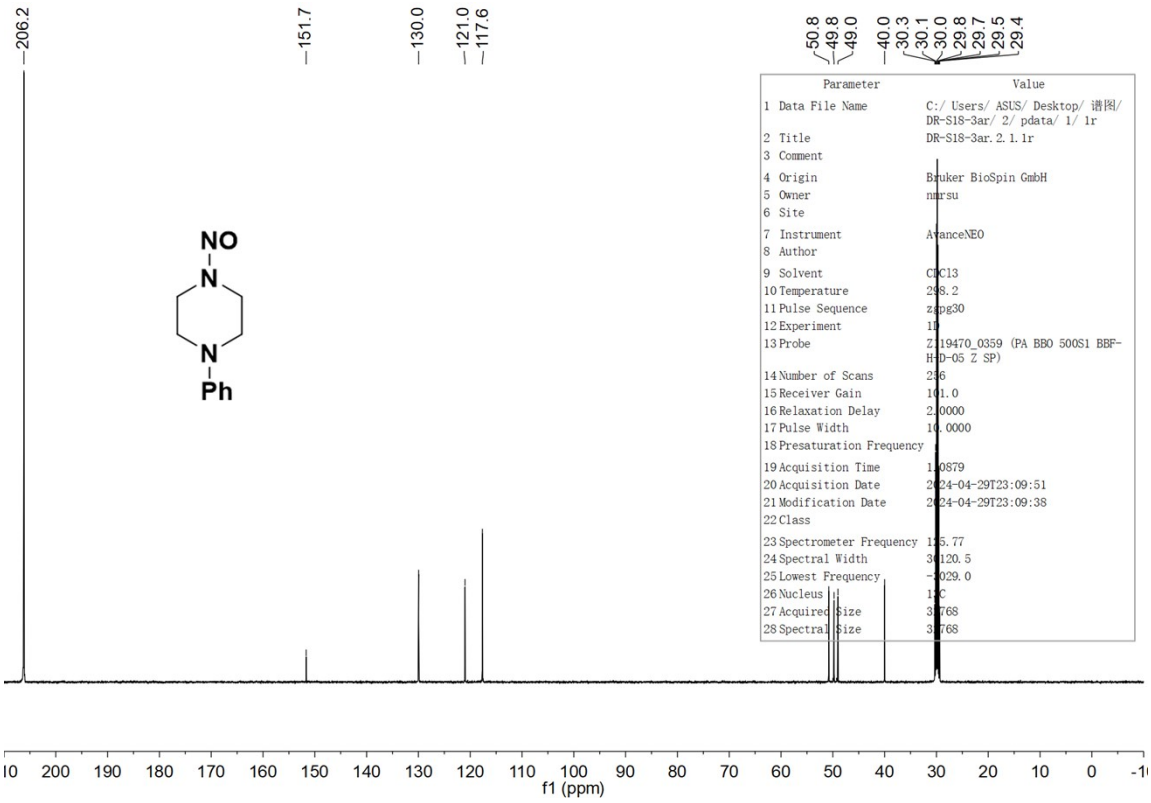
tert-butyl 4-nitrosopiperazine-1-carboxylate(3q)





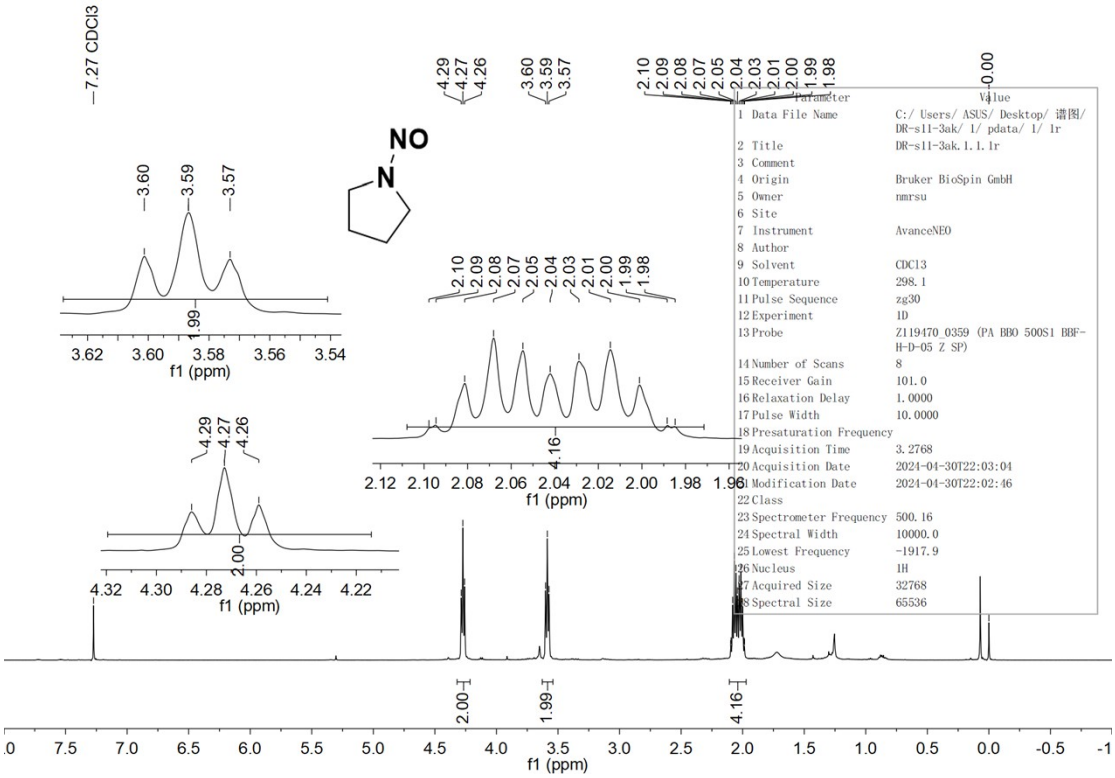
1-nitroso-4-phenylpiperazine(3r)



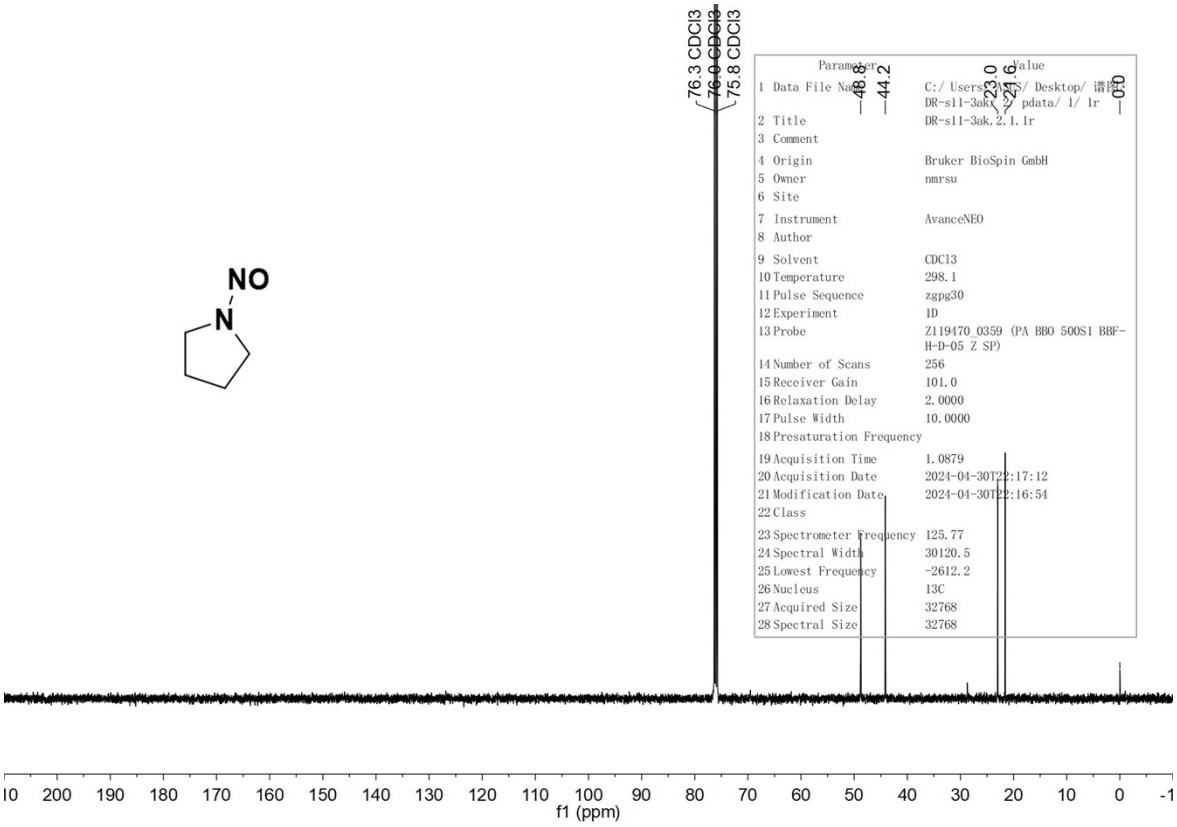


Parameter	Value
1 Data File Name	C:/ Users/ ASUS/ Desktop/ 谱图/ DR-S18-3ar/ 2/ pdata/ 1/ 1r
2 Title	DR-S18-3ar.2.1.1r
3 Comment	
4 Origin	Bruker BioSpin GmbH
5 Owner	nmrsu
6 Site	
7 Instrument	AvanceNEO
8 Author	
9 Solvent	CDCl3
10 Temperature	298.2
11 Pulse Sequence	zgpg30
12 Experiment	1D
13 Probe	Z119470_0359 (PA BBO 500S1 BBF-H-D-05 Z SP)
14 Number of Scans	236
15 Receiver Gain	101.0
16 Relaxation Delay	2.0000
17 Pulse Width	10.0000
18 Presaturation Frequency	
19 Acquisition Time	1.0879
20 Acquisition Date	2024-04-29T23:09:51
21 Modification Date	2024-04-29T23:09:38
22 Class	
23 Spectrometer Frequency	125.77
24 Spectral Width	30120.5
25 Lowest Frequency	-1029.0
26 Nucleus	13C
27 Acquired Size	32768
28 Spectral Size	32768

1-nitrosopyrrolidine(3s)

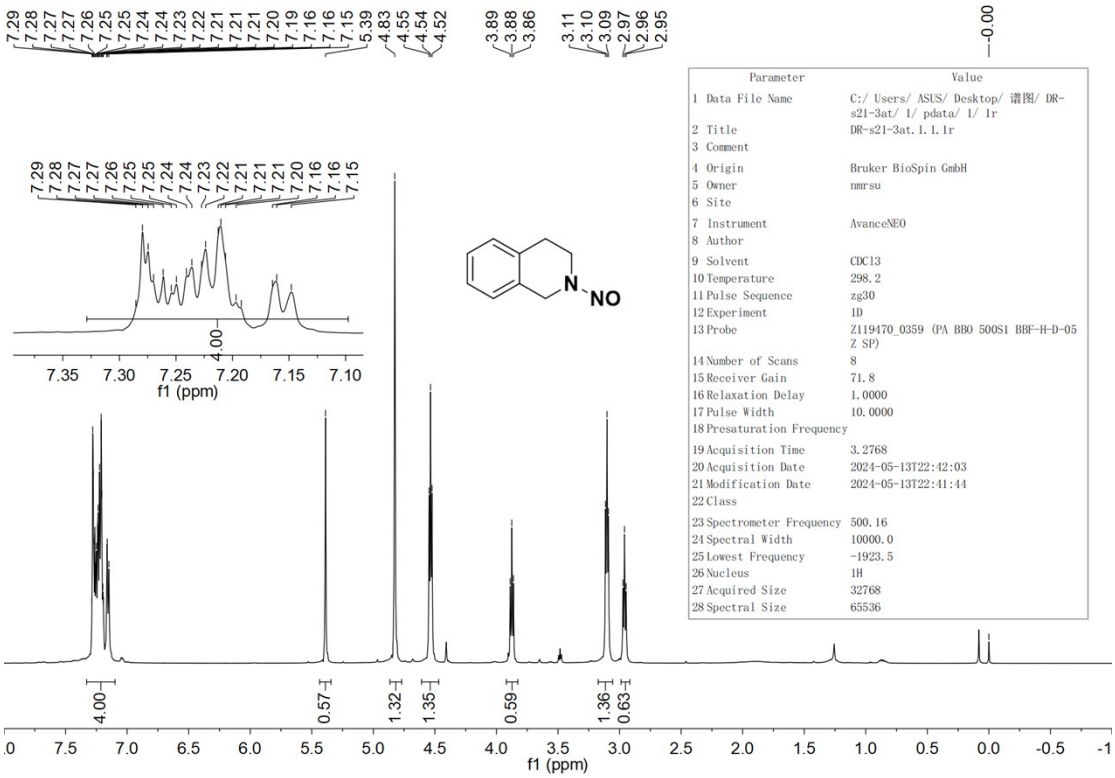


Parameter	Value
1 Data File Name	C:/ Users/ ASUS/ Desktop/ 谱图/ DR-s11-3ak/ 1/ pdata/ 1/ 1r
2 Title	DR-s11-3ak.1.1.1r
3 Comment	
4 Origin	Bruker BioSpin GmbH
5 Owner	nmrsu
6 Site	
7 Instrument	AvanceNEO
8 Author	
9 Solvent	CDCl3
10 Temperature	298.1
11 Pulse Sequence	zg30
12 Experiment	1D
13 Probe	Z119470_0359 (PA BBO 500S1 BBF-H-D-05 Z SP)
14 Number of Scans	8
15 Receiver Gain	101.0
16 Relaxation Delay	1.0000
17 Pulse Width	10.0000
18 Presaturation Frequency	
19 Acquisition Time	3.2768
20 Acquisition Date	2024-04-30T22:03:04
21 Modification Date	2024-04-30T22:02:46
22 Class	
23 Spectrometer Frequency	500.16
24 Spectral Width	10000.0
25 Lowest Frequency	-1917.9
26 Nucleus	1H
27 Acquired Size	32768
28 Spectral Size	65536

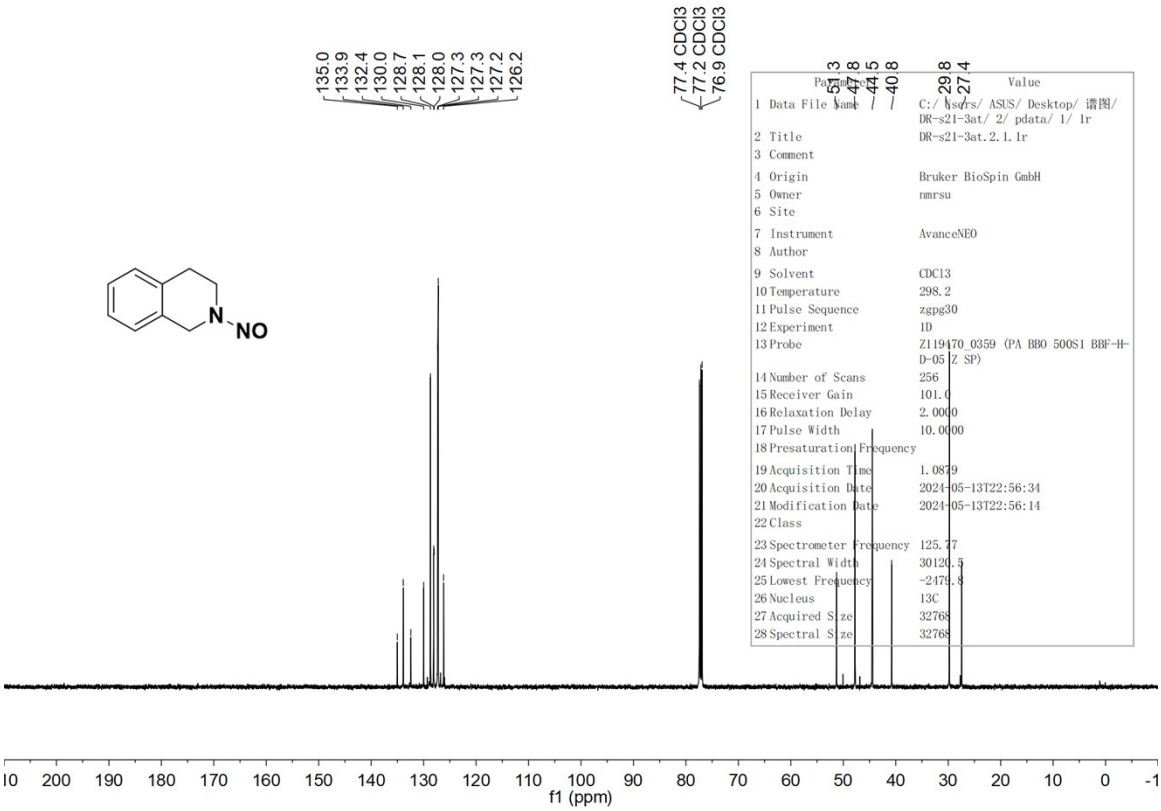


Parameter	Value
1 Data File Name	C:/ Users/ ASUS/ Desktop/ 谱图/ DR-s11-3ak/ 1/ pdata/ 1/ 1r
2 Title	DR-s11-3ak.2.1.1r
3 Comment	
4 Origin	Bruker BioSpin GmbH
5 Owner	nmrsu
6 Site	
7 Instrument	AvanceNEO
8 Author	
9 Solvent	CDCl3
10 Temperature	298.1
11 Pulse Sequence	zgpg30
12 Experiment	1D
13 Probe	Z119470_0359 (PA BBO 500S1 BBF-H-D-05 Z SP)
14 Number of Scans	256
15 Receiver Gain	101.0
16 Relaxation Delay	2.0000
17 Pulse Width	10.0000
18 Presaturation Frequency	
19 Acquisition Time	1.0879
20 Acquisition Date	2024-04-30T22:17:12
21 Modification Date	2024-04-30T22:16:54
22 Class	
23 Spectrometer Frequency	125.77
24 Spectral Width	30120.5
25 Lowest Frequency	-2612.2
26 Nucleus	13C
27 Acquired Size	32768
28 Spectral Size	32768

2-nitroso-1,2,3,4-tetrahydroisoquinoline(3t)



Parameter	Value
1 Data File Name	C:/ Users/ ASUS/ Desktop/ 谱图/ DR-s21-3at/ 1/ pdata/ 1/ 1r
2 Title	DR-s21-3at.1.1.1r
3 Comment	
4 Origin	Bruker BioSpin GmbH
5 Owner	nmrsu
6 Site	
7 Instrument	AvanceNEO
8 Author	
9 Solvent	CDCl3
10 Temperature	298.2
11 Pulse Sequence	zg30
12 Experiment	1D
13 Probe	Z119470_0359 (PA BBO 500S1 BBF-H-D-05 Z SP)
14 Number of Scans	8
15 Receiver Gain	71.8
16 Relaxation Delay	1.0000
17 Pulse Width	10.0000
18 Presaturation Frequency	
19 Acquisition Time	3.2768
20 Acquisition Date	2024-05-13T22:42:03
21 Modification Date	2024-05-13T22:41:44
22 Class	
23 Spectrometer Frequency	500.16
24 Spectral Width	10000.0
25 Lowest Frequency	-1923.5
26 Nucleus	1H
27 Acquired Size	32768
28 Spectral Size	65536



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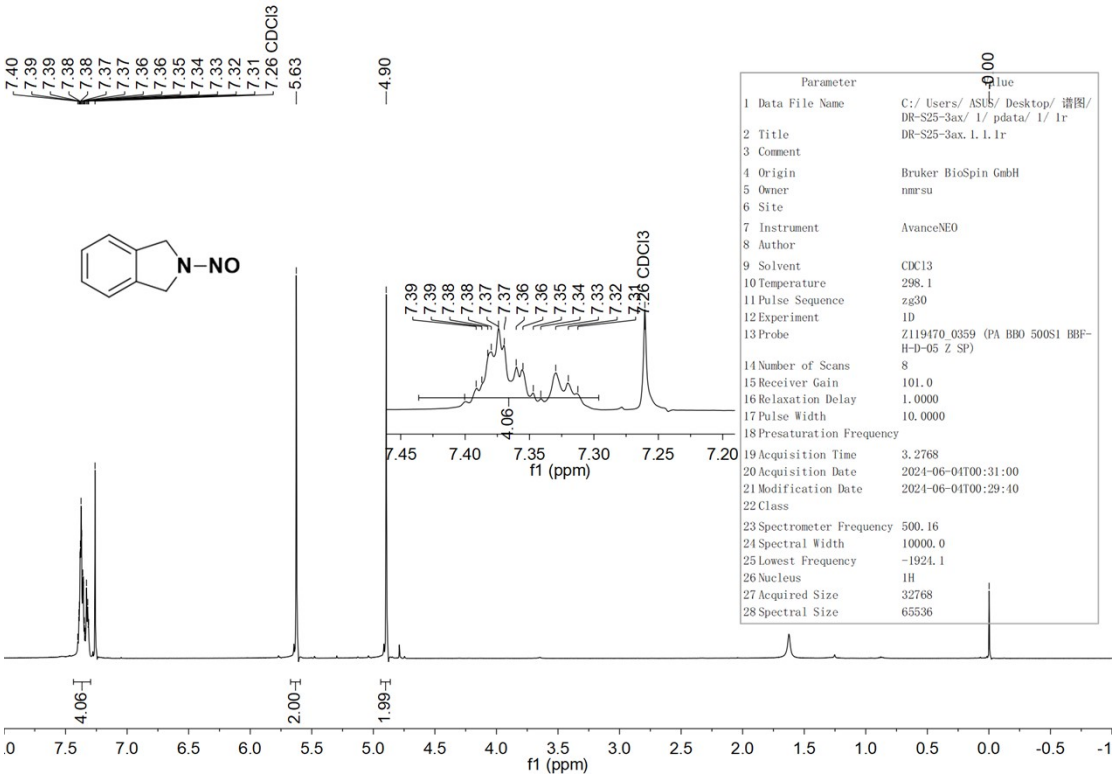
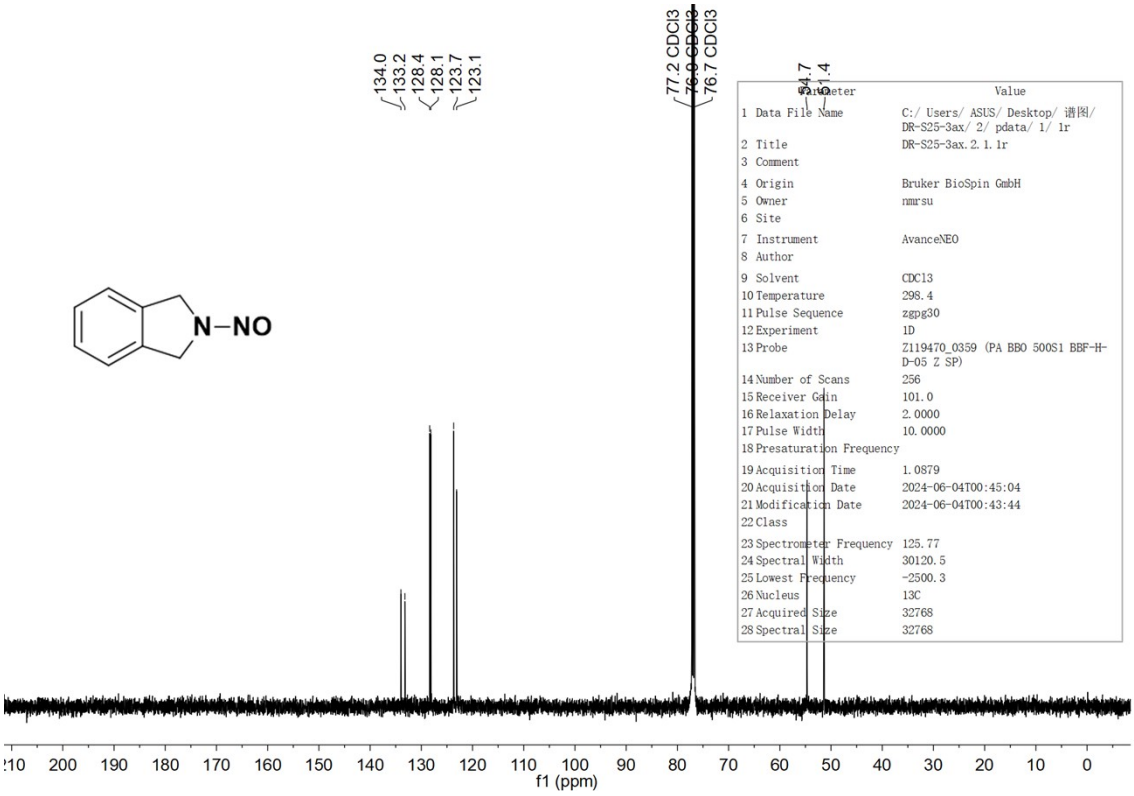
1 Data File Name	C:/ Users/ ASUS/ Desktop/ 谱图/ DR-s21-3at/ 2/ pdata/ 1/ 1r
2 Title	DR-s21-3at.2.1.1r
3 Comment	
4 Origin	Bruker BioSpin GmbH
5 Owner	nmrsu
6 Site	
7 Instrument	AvanceNEO
8 Author	
9 Solvent	CDCl3
10 Temperature	298.2
11 Pulse Sequence	zgpg30
12 Experiment	1D
13 Probe	Z119470_0359 (PA BBO 500S1 BBF-H-D-05 Z SP)
14 Number of Scans	256
15 Receiver Gain	101.0
16 Relaxation Delay	2.0000
17 Pulse Width	10.0000
18 Presaturation Frequency	
19 Acquisition Time	1.0879
20 Acquisition Date	2024-05-13T22:56:34
21 Modification Date	2024-05-13T22:56:14
22 Class	
23 Spectrometer Frequency	125.77
24 Spectral Width	30120.5
25 Lowest Frequency	-2479.8
26 Nucleus	13C
27 Acquired Size	32768
28 Spectral Size	32768

2-nitrosoisoindoline(3u)

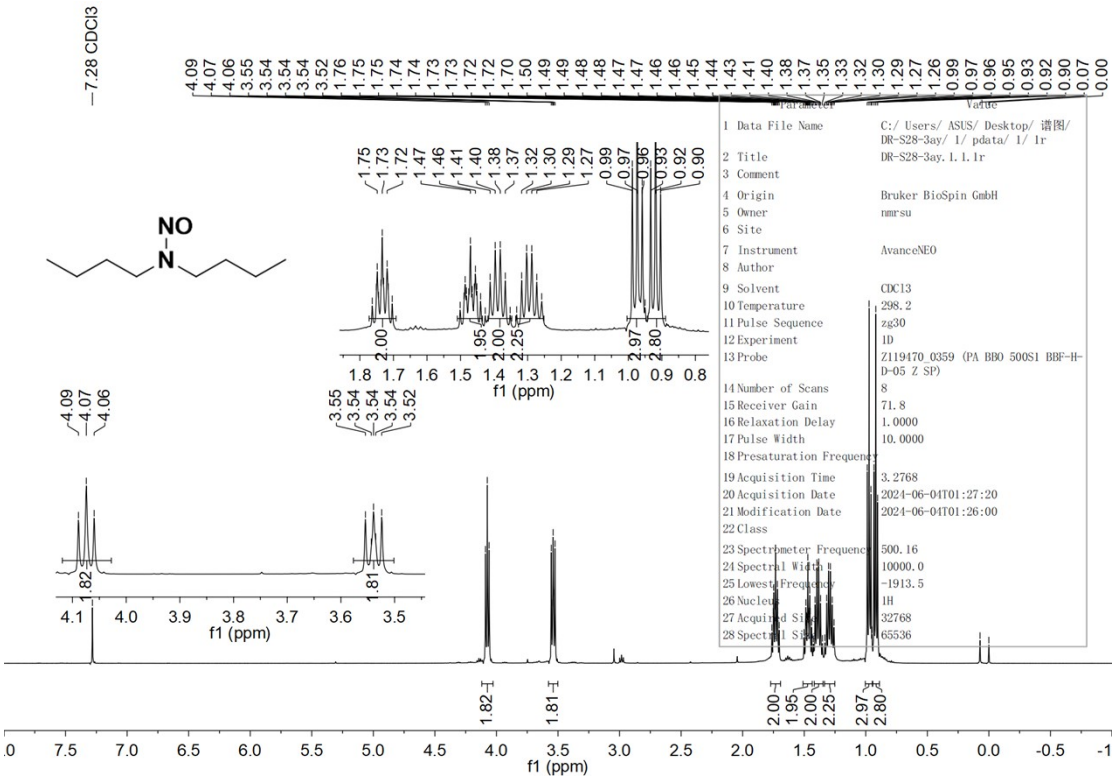


Parameter	Value
1 Data File Name	C:/ Users/ ASUS/ Desktop/ 谱图/ DR-S25-3ax/ 1/ pdata/ 1/ 1r
2 Title	DR-S25-3ax.1.1.1r
3 Comment	
4 Origin	Bruker BioSpin GmbH
5 Owner	nmrsu
6 Site	
7 Instrument	AvanceNEO
8 Author	
9 Solvent	CDCl3
10 Temperature	298.1
11 Pulse Sequence	zg30
12 Experiment	1D
13 Probe	Z119470_0359 (PA BBO 500S1 BBF-H-D-05 Z SP)
14 Number of Scans	8
15 Receiver Gain	101.0
16 Relaxation Delay	1.0000
17 Pulse Width	10.0000
18 Presaturation Frequency	
19 Acquisition Time	3.2768
20 Acquisition Date	2024-06-04T00:31:00
21 Modification Date	2024-06-04T00:29:40
22 Class	
23 Spectrometer Frequency	500.16
24 Spectral Width	10000.0
25 Lowest Frequency	-1924.1
26 Nucleus	1H
27 Acquired Size	32768
28 Spectral Size	65536

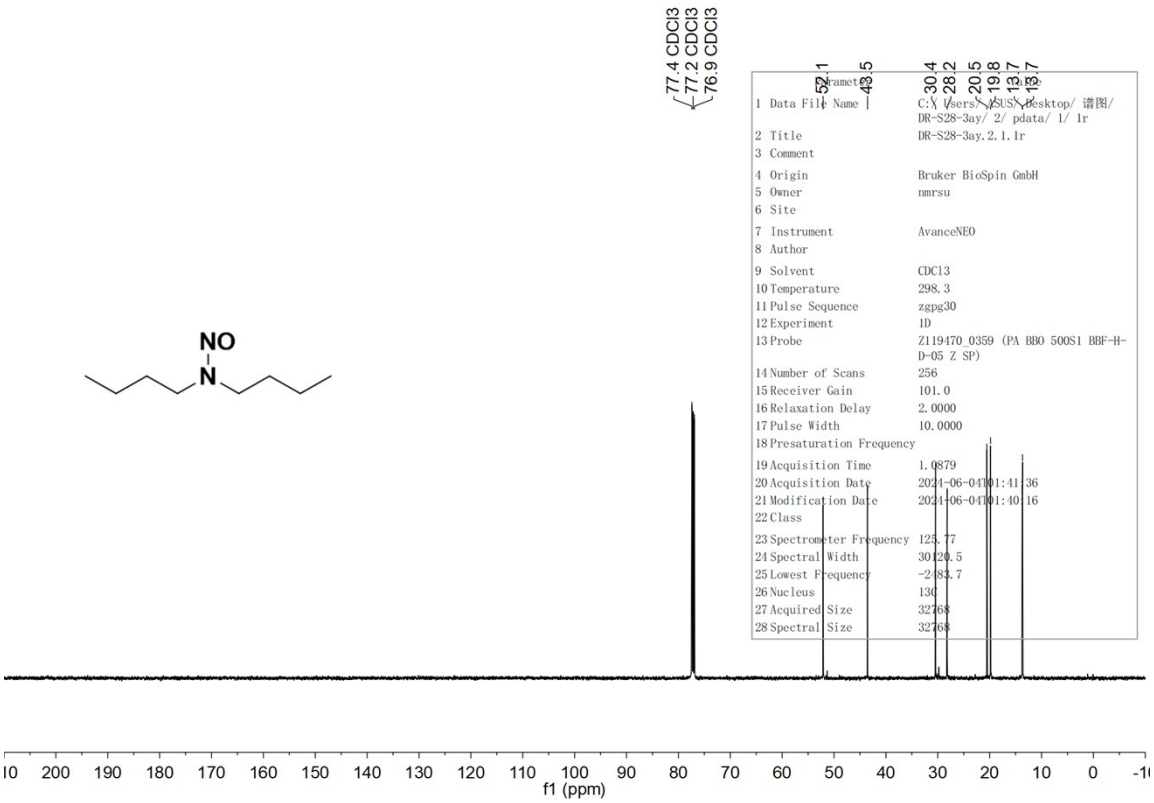


	Parameter	Value
1	Data File Name	C:/ Users/ ASUS/ Desktop/ 谱图/ DR-S25-3ax/ 2/ pdata/ 1/ 1r
2	Title	DR-S25-3ax. 2. 1. 1r
3	Comment	
4	Origin	Bruker BioSpin GmbH
5	Owner	nmrsu
6	Site	
7	Instrument	AvanceNEO
8	Author	
9	Solvent	CDCl3
10	Temperature	298. 4
11	Pulse Sequence	zgpg30
12	Experiment	1D
13	Probe	Z119470_0359 (PA BBO 500S1 BBF-H-D-05 Z SP)
14	Number of Scans	256
15	Receiver Gain	101. 0
16	Relaxation Delay	2. 0000
17	Pulse Width	10. 0000
18	Presaturation Frequency	
19	Acquisition Time	1. 0879
20	Acquisition Date	2024-06-04T00:45:04
21	Modification Date	2024-06-04T00:43:44
22	Class	
23	Spectrometer Frequency	125. 77
24	Spectral Width	30120. 5
25	Lowest Frequency	-2500. 3
26	Nucleus	13C
27	Acquired Size	32768
28	Spectral Size	32768

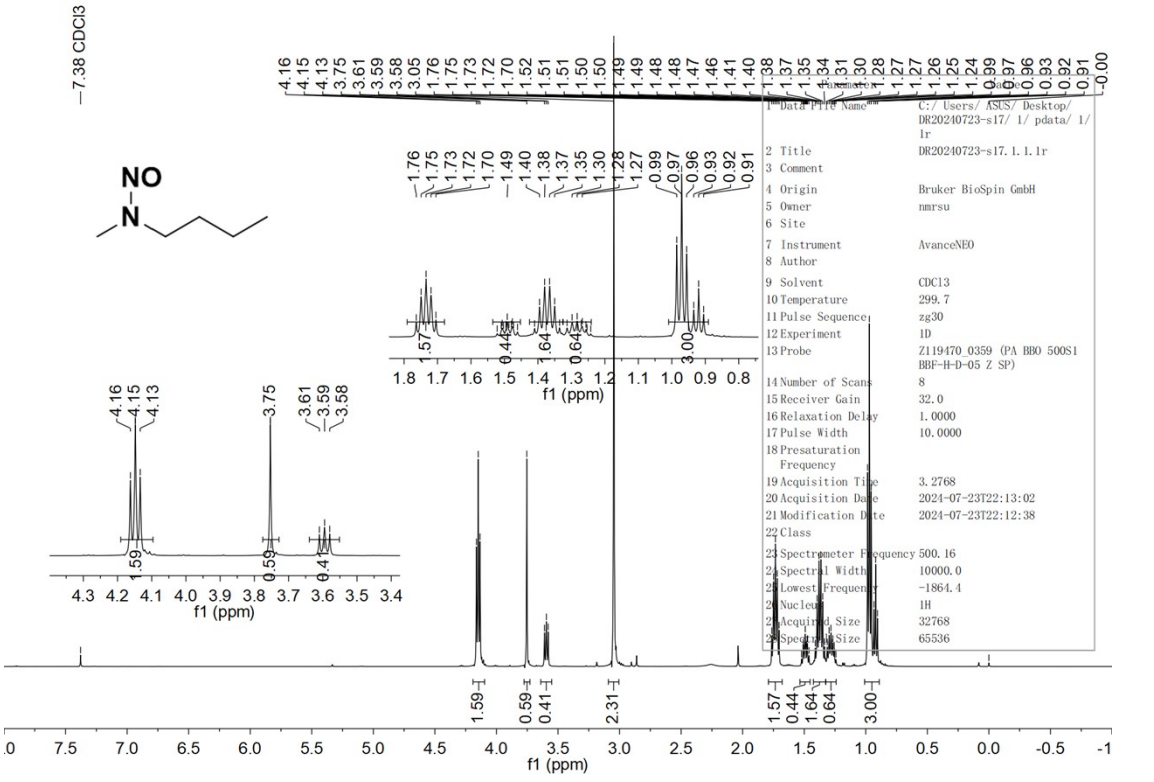
N,N-dibutylnitrous amide(3v)

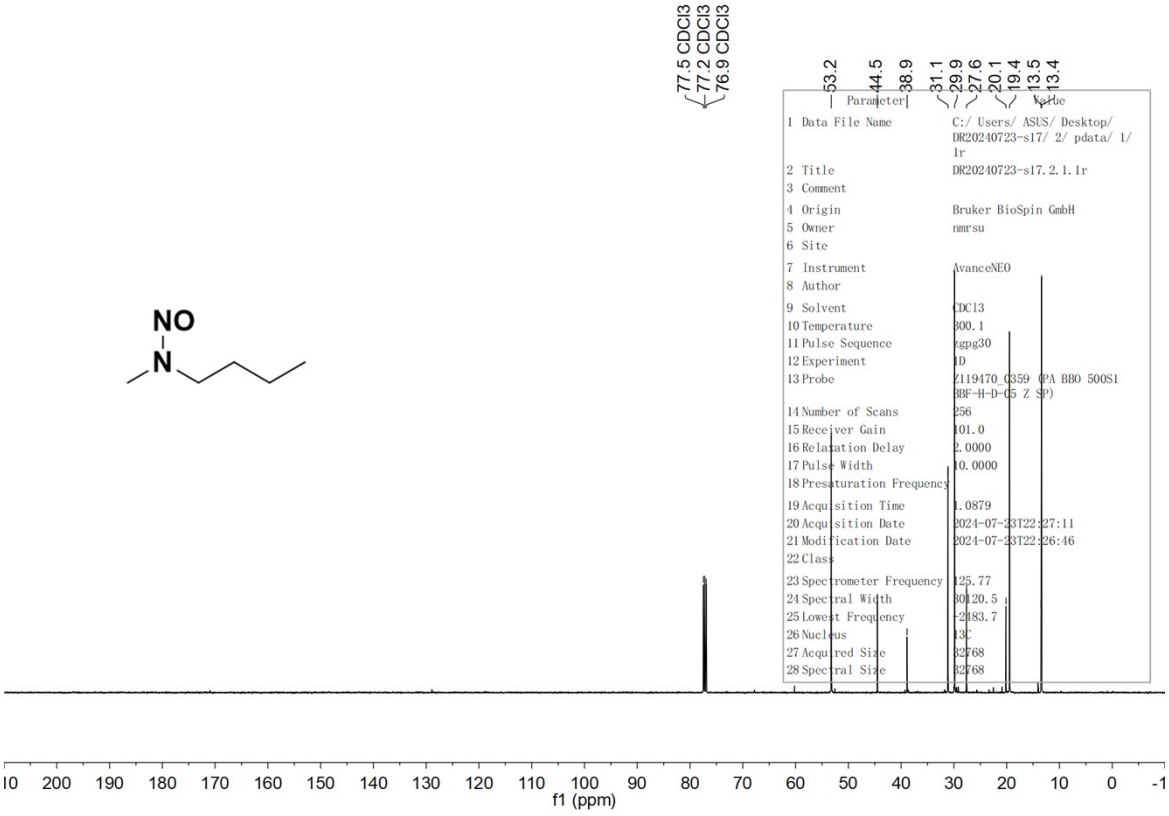


	Parameter	Value
1	Data File Name	C:/ Users/ ASUS/ Desktop/ 谱图/ DR-S28-3ay/ 1/ pdata/ 1/ 1r
2	Title	DR-S28-3ay. 1. 1. 1r
3	Comment	
4	Origin	Bruker BioSpin GmbH
5	Owner	nmrsu
6	Site	
7	Instrument	AvanceNEO
8	Author	
9	Solvent	CDCl3
10	Temperature	298. 2
11	Pulse Sequence	zg30
12	Experiment	1D
13	Probe	Z119470_0359 (PA BBO 500S1 BBF-H-D-05 Z SP)
14	Number of Scans	8
15	Receiver Gain	71. 8
16	Relaxation Delay	1. 0000
17	Pulse Width	10. 0000
18	Presaturation Frequency	
19	Acquisition Time	3. 2768
20	Acquisition Date	2024-06-04T01:27:20
21	Modification Date	2024-06-04T01:26:00
22	Class	
23	Spectrometer Frequency	500. 16
24	Spectral Width	10000. 0
25	Lowest Frequency	-1913. 5
26	Nucleus	1H
27	Acquired Size	32768
28	Spectral Size	65536

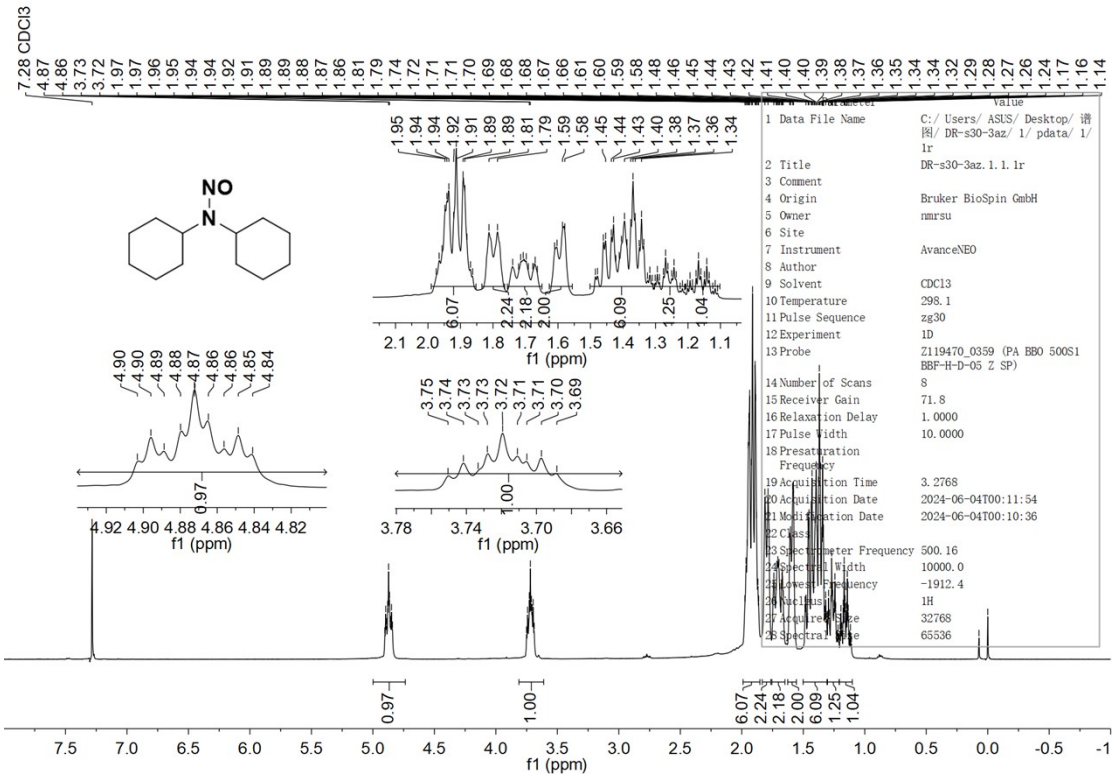


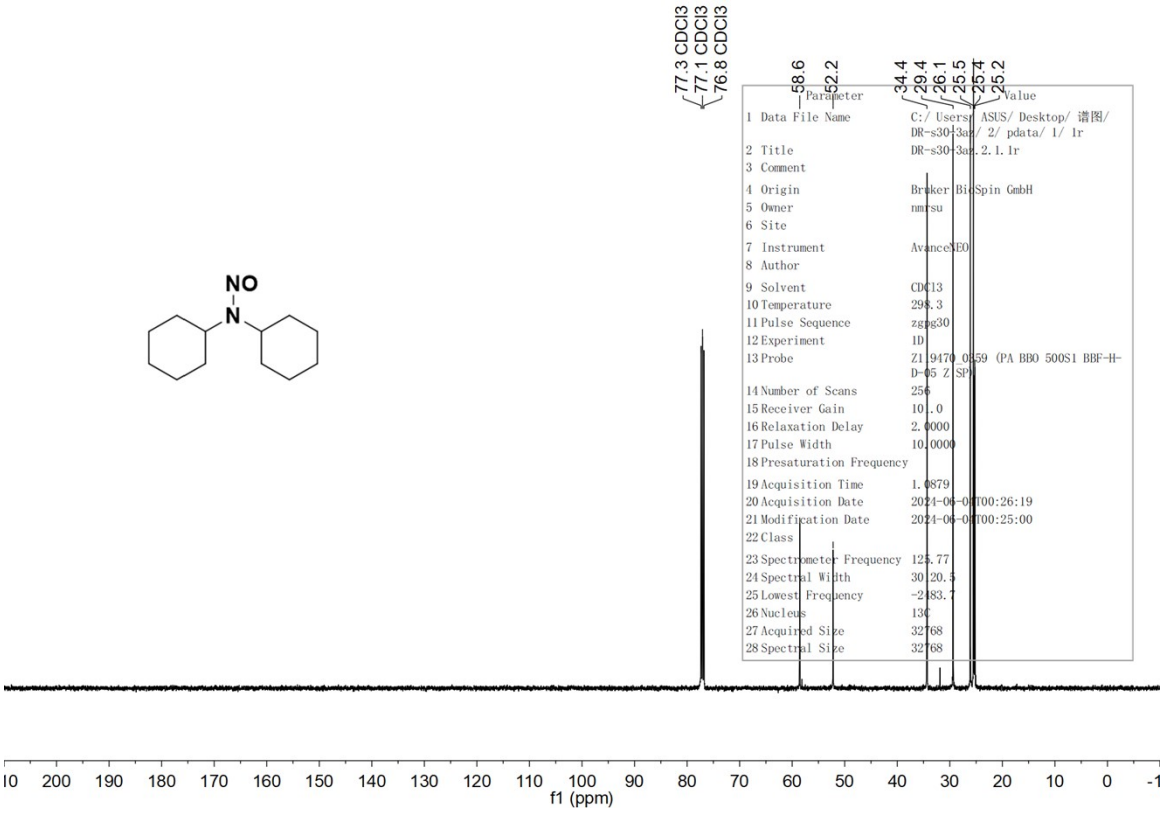
N-butyl-*N*-methylnitrous amide (3w)



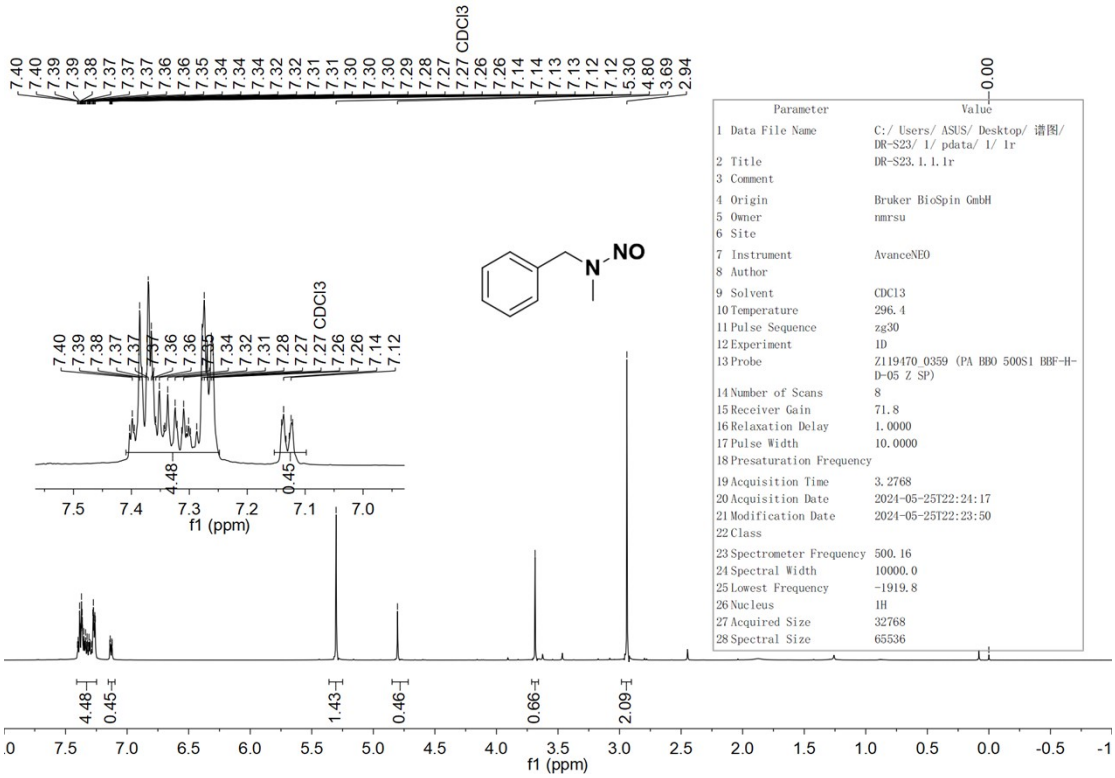


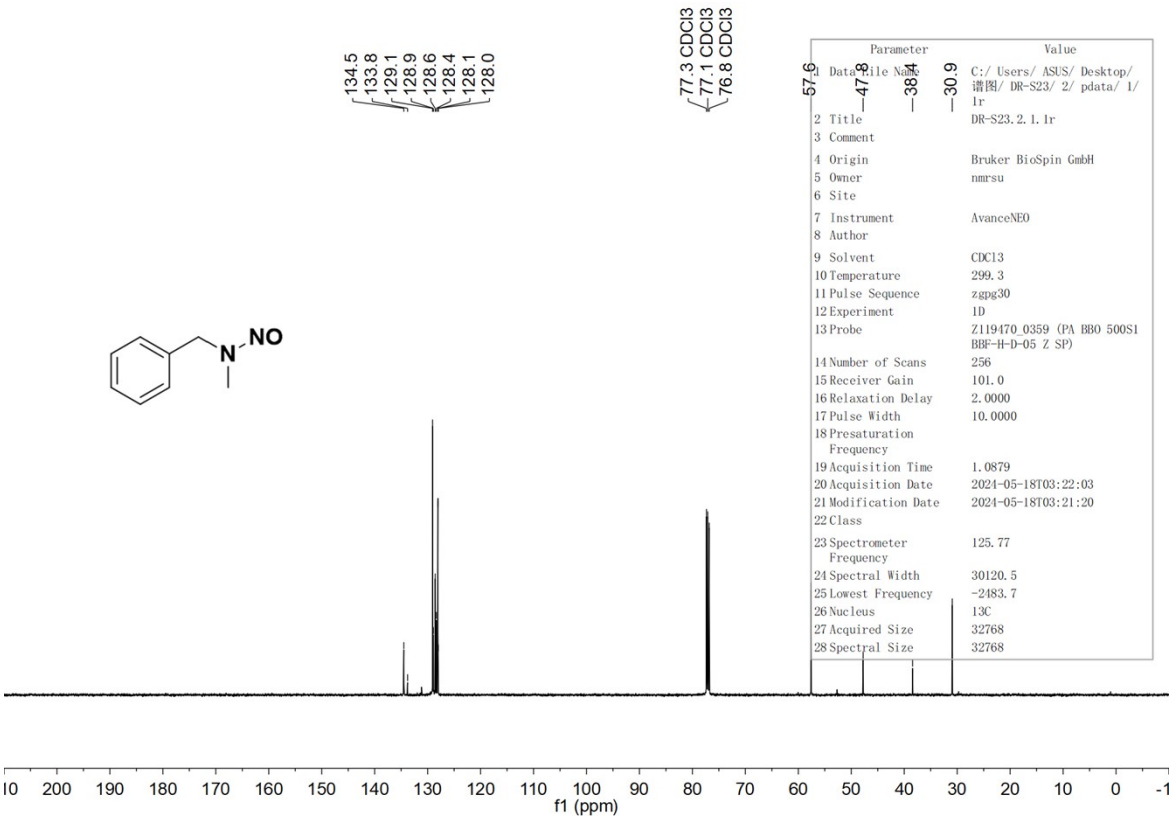
N,N-dicyclohexylnitrous amide(3x)





N-benzyl-*N*-methylnitrous amide(3y)





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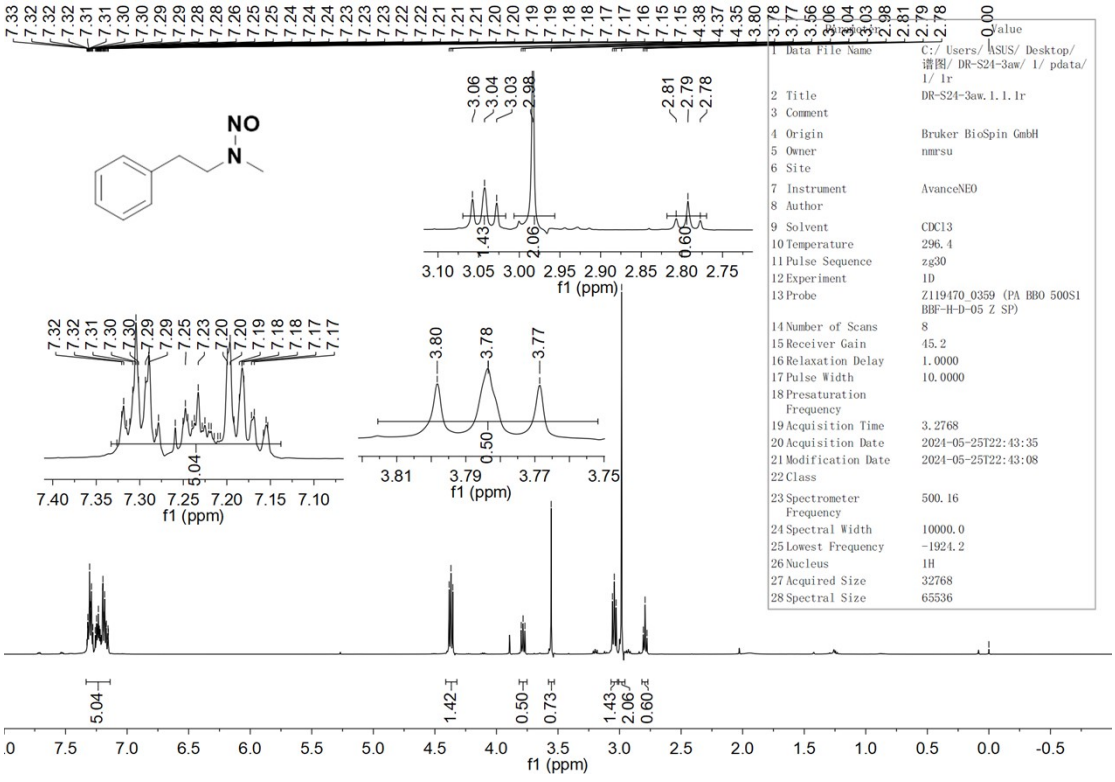
1 Data File Name	C:/ Users/ ASUS/ Desktop/ 谱图/ DR-S23/ 2/ pdata/ 1/ 1r
2 Title	DR-S23.2.1.1r
3 Comment	
4 Origin	Bruker BioSpin GmbH
5 Owner	nmrsu
6 Site	
7 Instrument	AvanceNEO
8 Author	
9 Solvent	CDCl3
10 Temperature	299.3
11 Pulse Sequence	zgpg30
12 Experiment	1D
13 Probe	Z119470_0359 (PA BBO 500S1 BBF-H-D-05 Z SP)
14 Number of Scans	256
15 Receiver Gain	101.0
16 Relaxation Delay	2.0000
17 Pulse Width	10.0000
18 Presaturation Frequency	
19 Acquisition Time	1.0879
20 Acquisition Date	2024-05-18T03:22:03
21 Modification Date	2024-05-18T03:21:20
22 Class	
23 Spectrometer Frequency	125.77
24 Spectral Width	30120.5
25 Lowest Frequency	-2483.7
26 Nucleus	13C
27 Acquired Size	32768
28 Spectral Size	32768

N-methyl-*N*-phenethylnitrous amide(3z)



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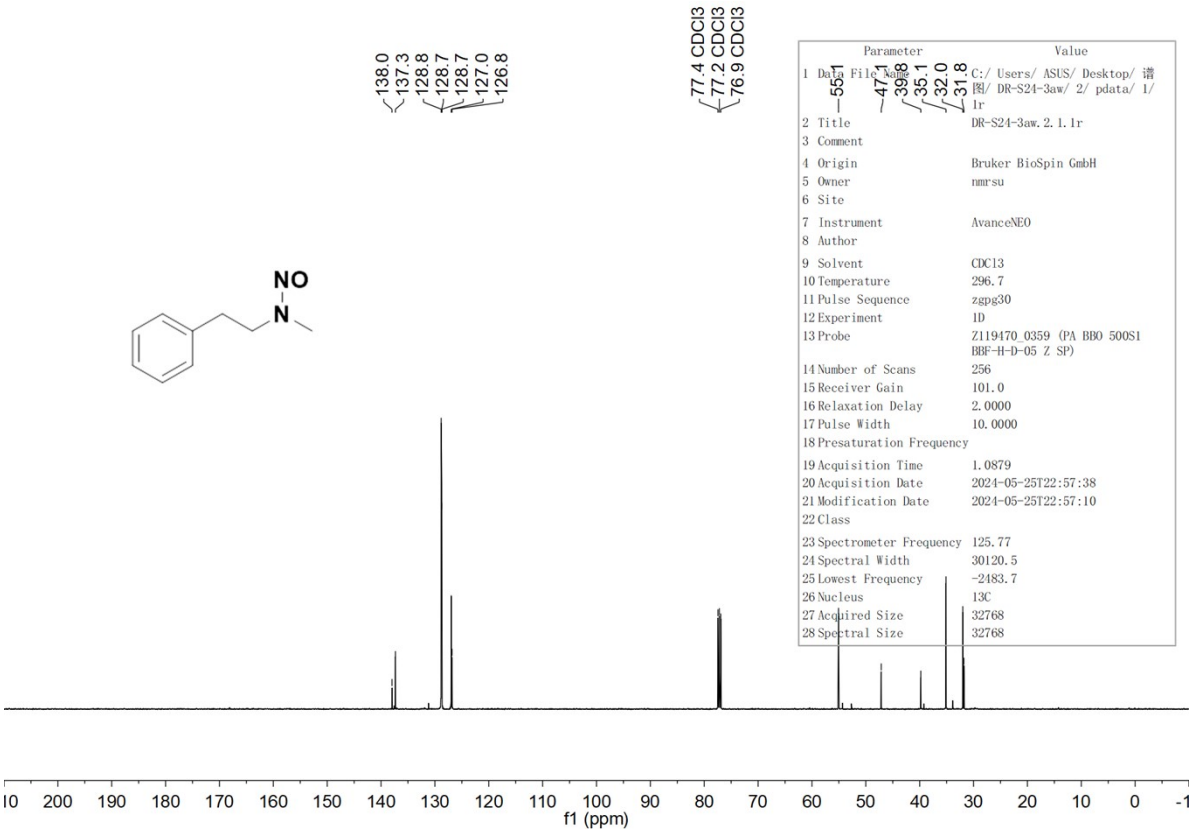
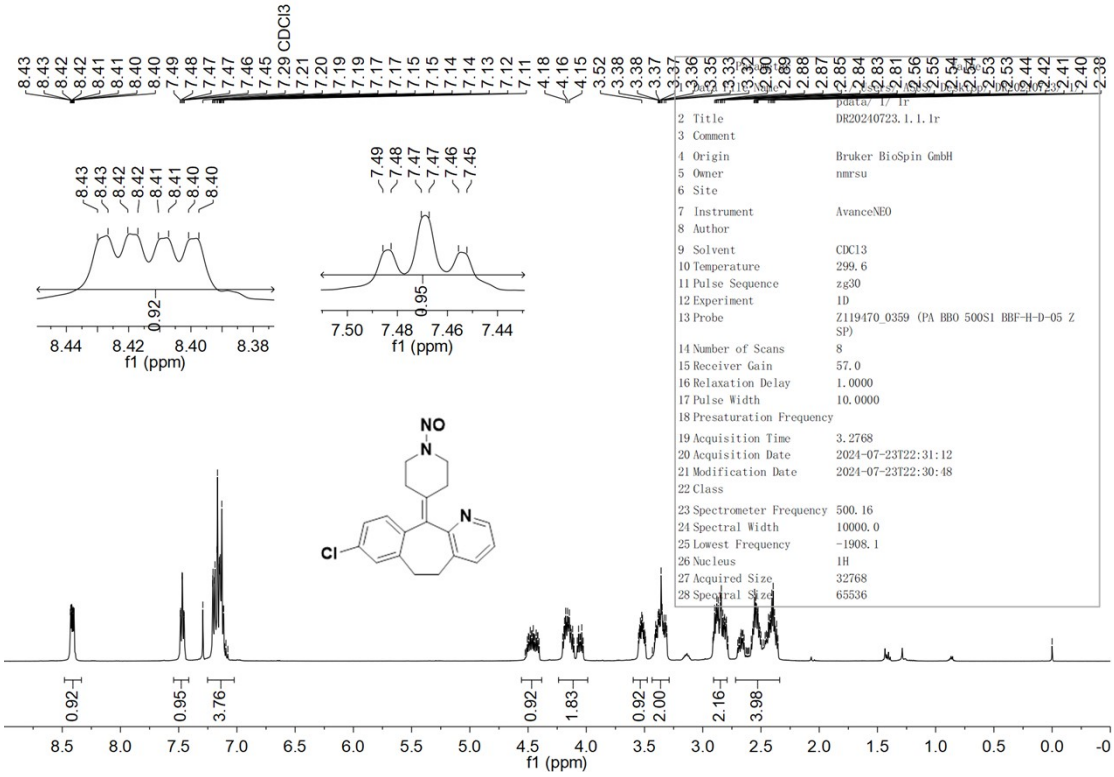
3 Comment	
4 Origin	Bruker BioSpin GmbH
5 Owner	nmrsu
6 Site	
7 Instrument	AvanceNEO
8 Author	
9 Solvent	CDCl3
10 Temperature	296.4
11 Pulse Sequence	zg30
12 Experiment	1D
13 Probe	Z119470_0359 (PA BBO 500S1 BBF-H-D-05 Z SP)
14 Number of Scans	8
15 Receiver Gain	45.2
16 Relaxation Delay	1.0000
17 Pulse Width	10.0000
18 Presaturation Frequency	
19 Acquisition Time	3.2768
20 Acquisition Date	2024-05-25T22:43:35
21 Modification Date	2024-05-25T22:43:08
22 Class	
23 Spectrometer Frequency	500.16
24 Spectral Width	10000.0
25 Lowest Frequency	-1924.2
26 Nucleus	1H
27 Acquired Size	32768
28 Spectral Size	65536



Parameter	Value
1 Data File Name	C:/ Users/ ASUS/ Desktop/ 谱图/ DR-S24-3aw/ 2/ pdata/ 1/ 1r
2 Title	DR-S24-3aw.2.1.1r
3 Comment	
4 Origin	Bruker BioSpin GmbH
5 Owner	nmrsu
6 Site	
7 Instrument	AvanceNEO
8 Author	
9 Solvent	CDCl3
10 Temperature	296.7
11 Pulse Sequence	zgpg30
12 Experiment	1D
13 Probe	Z119470_0359 (PA BBO 500S1 BBF-H-D-05 Z SP)
14 Number of Scans	256
15 Receiver Gain	101.0
16 Relaxation Delay	2.0000
17 Pulse Width	10.0000
18 Presaturation Frequency	
19 Acquisition Time	1.0879
20 Acquisition Date	2024-05-25T22:57:38
21 Modification Date	2024-05-25T22:57:10
22 Class	
23 Spectrometer Frequency	125.77
24 Spectral Width	30120.5
25 Lowest Frequency	-2483.7
26 Nucleus	13C
27 Acquired Size	32768
28 Spectral Size	32768

8-chloro-11-(1-nitrosopiperidin-4-ylidene)-6,11-dihydro-5*H*-benzo[5,6]cyclohepta[1,2-b]pyridine(3ab)



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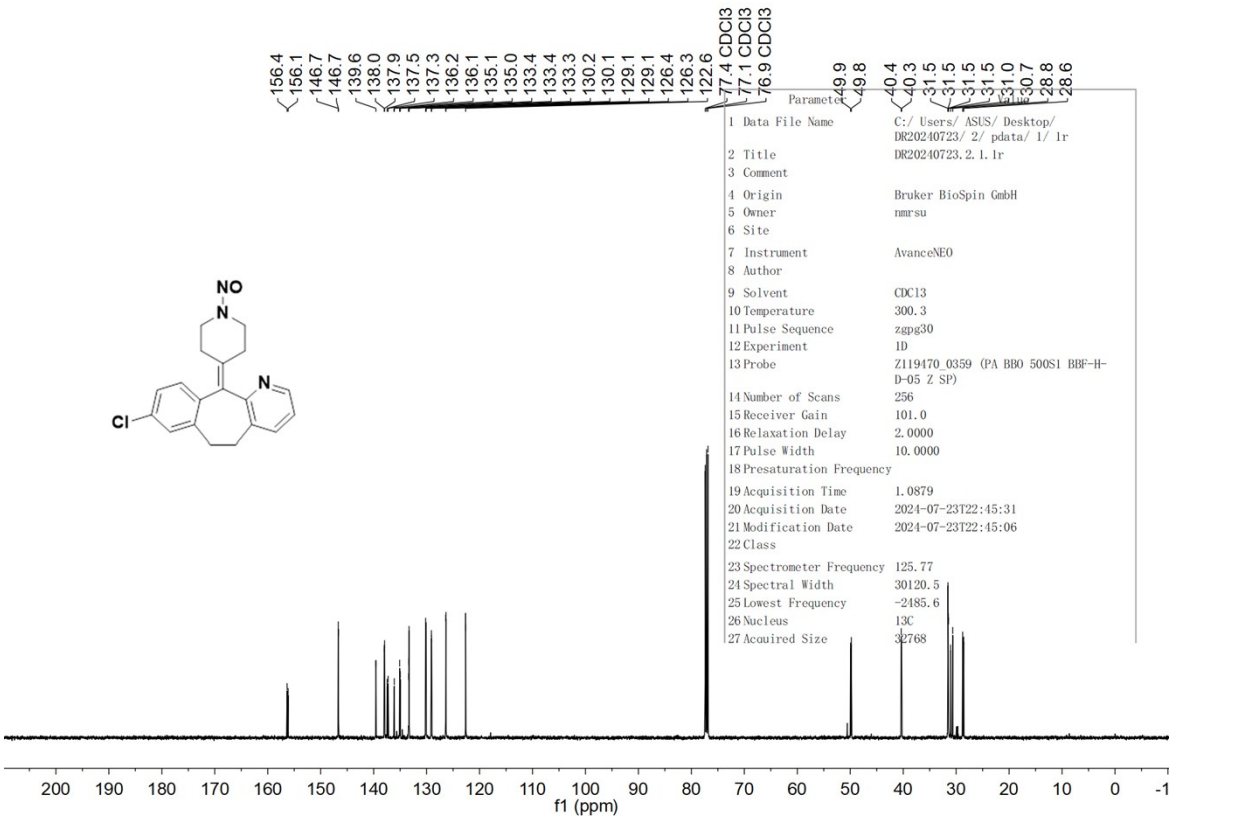
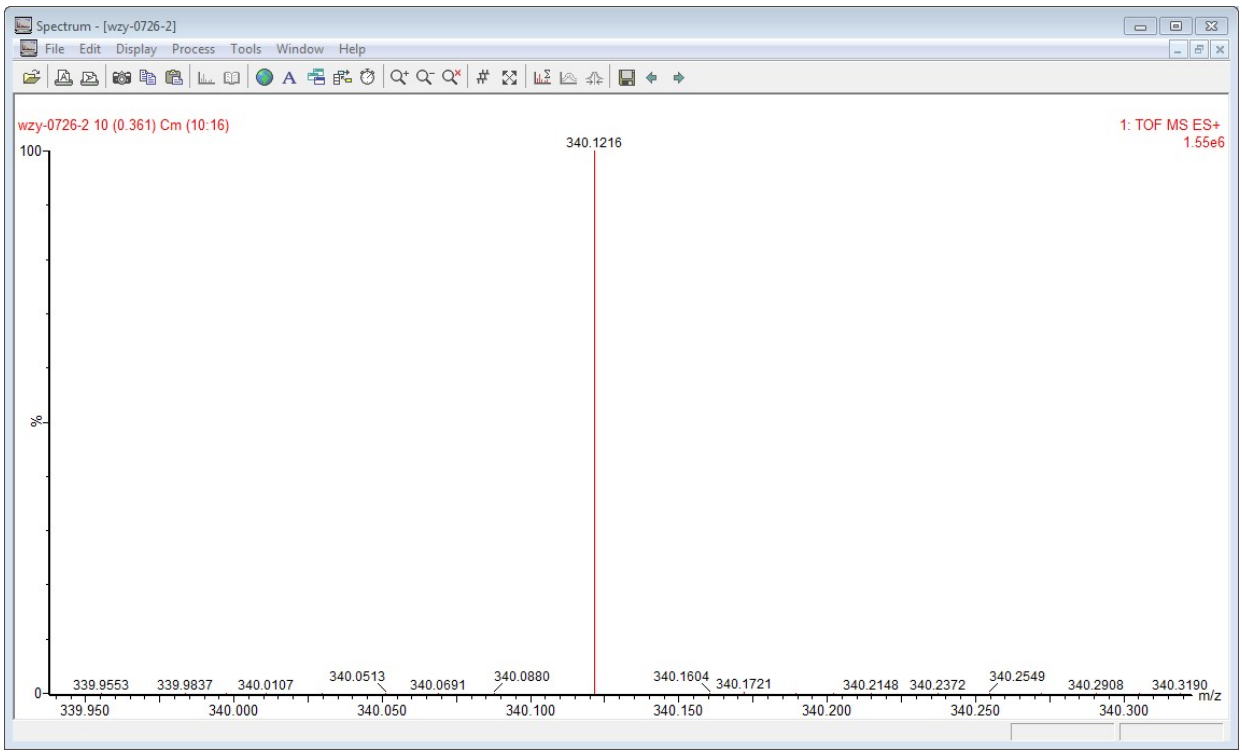
2 Title	DR20240723.1.1.1r
3 Comment	
4 Origin	Bruker BioSpin GmbH
5 Owner	nmrsu
6 Site	
7 Instrument	AvanceNEO
8 Author	
9 Solvent	CDCl3
10 Temperature	299.6
11 Pulse Sequence	zg30
12 Experiment	1D
13 Probe	Z119470_0359 (PA BBO 500S1 BBF-H-D-05 Z SP)
14 Number of Scans	8
15 Receiver Gain	57.0
16 Relaxation Delay	1.0000
17 Pulse Width	10.0000
18 Presaturation Frequency	
19 Acquisition Time	3.2768
20 Acquisition Date	2024-07-23T22:31:12
21 Modification Date	2024-07-23T22:30:48
22 Class	
23 Spectrometer Frequency	500.16
24 Spectral Width	10000.0
25 Lowest Frequency	-1908.1
26 Nucleus	1H
27 Acquired Size	32768
28 Spectral Size	65536


156.4
156.1
146.7
146.7
139.6
138.0
137.9
137.5
137.3
136.2
136.1
135.1
135.0
133.4
133.4
133.3
130.2
130.1
129.1
129.1
126.4
126.3
122.6
77.4 CDCl3
77.1 CDCl3
76.9 CDCl3
49.9
49.8
40.4
40.3
31.5
31.5
31.5
31.5
31.0
30.7
28.8
28.6
NO
N
N
Cl
Parameter Value
1 Data File Name C:/ Users/ ASUS/ Desktop/ DR20240723/ 2/ pdata/ 1/ 1r
2 Title DR20240723.2.1.1r
3 Comment
4 Origin Bruker BioSpin GmbH
5 Owner nmrsu
6 Site
7 Instrument AvanceNEO
8 Author
9 Solvent CDCl3
10 Temperature 300.3
11 Pulse Sequence zgpg30
12 Experiment 1D
13 Probe Z119470_0359 (PA BBO 500S1 BBF-H-D-05 Z SP)
14 Number of Scans 256
15 Receiver Gain 101.0
16 Relaxation Delay 2.0000
17 Pulse Width 10.0000
18 Presaturation Frequency
19 Acquisition Time 1.0879
20 Acquisition Date 2024-07-23T22:45:31
21 Modification Date 2024-07-23T22:45:06
22 Class
23 Spectrometer Frequency 125.77
24 Spectral Width 30120.5
25 Lowest Frequency -2485.6
26 Nucleus 13C
27 Acquired Size 32768
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -1
f1 (ppm)


Spectrum - [wzy-0726-2]
File Edit Display Process Tools Window Help
wzy-0726-2 10 (0.361) Cm (10:16)
1: TOF MS ES+
1.55e6
340.1216
100
%
0
339.9553
339.9837
340.0107
340.0513
340.0691
340.0880
340.1604
340.1721
340.2148
340.2372
340.2549
340.2908
340.3190
339.950
340.000
340.050
340.100
340.150
340.200
340.250
340.300
m/z