Supporting Information

Direct Electrochemical Imidation of Aliphatic Amines via Anodic Oxidation

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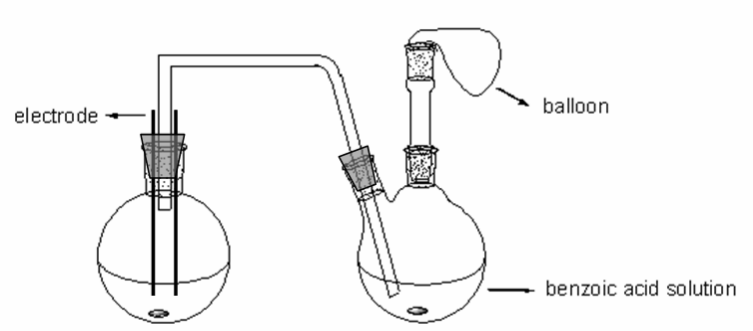
General Remarks: ^{1}H NMR and ^{13}C NMR were recorded on a Bruker AC-300 FT (^{1}H: 300 MHz, ^{13}C: 75 MHz) using TMS as internal reference. The chemical shifts (δ) and coupling constants (J) were expressed in ppm and Hz respectively. Infrared samples were recorded on a Perkin-Elmer 2000 FTIR spectrometer. HPLC analysis was carried out on an Agilent 1100 series HPLC with a multiple wavelength detector. Chiralpak OD columns was purchased from Daicel Chemical Industries, LTD. Hexane: 2-propanol = 80:20, flow rate = 0.45 mL/min, T = 23°C, UV = 254 nm.

1. Experimental Section.

Instruments: The instrument for electrolysis is dual display potentiostat (CJS-292) (made in China). Cyclic voltammetric (CV) experiments were carried out with a CHI400A system (USA CH Instruments) in a conventional three-electrode cell in the presence of supporting electrolyte. The anode electrode is Pt (1.0×1.0 cm^2) or a graphite (diameter 0.5 cm) while the cathode electrode is graphite (diameter 0.5 cm). A saturated calomel electrode (SCE) were used as the reference electrode. CH_3CN and CH_2Cl_2 were distilled from CaH_2. Et_3N, THF, hexane and 1, 4-dioxane were distilled from sodium/benzophenone. CCl_4 and $CHCl_3$ were dried over calcium chloride and distilled. ***Caution: Azides and diazoalkanes may be hazardous and/or explosive.***

Representative procedures for imidation of tertiary amines: An undivided cell was equipped with a magnet stirrer, two graphite electrode both as the working electrode and the counter electrode respectively. In the electrolytic cell a solution of TsN_3 (0.2 mmol), tertiary amine (0.4 mmol), Bu_4NPF_6 (0.3 mmol) in CH_3CN (6 mL) was added. In addition, for the safety, a gas tube was installed in the electrolytic cell and the terminal of this gas tube was inserted in the excess solution of benzoic acid (1.22g, 10 mmol, benzoic acid dissolved in CH_3CN) in a two-neck flask. One of the two necks was equipped with a buffer balloon for the safe. The electrolyte was allowed to stir and the electrolysis was carried out at a constant current of 4 mA (electrode square 1.6 cm^2) at ambient temperature (25±1℃) for 3 h until the quantity of the electricity 2.2 F/mol was passed.

Upon completion of the reaction, the solvent was removed with a rotary evaporator. The residue was washed with anhydrous ether (3×10 mL) and the insoluble Bu_4NPF_6 was filtered and dried for next use. The filtrate was combined and the solvent was removed with a rotary evaporator. The residue was purified by column chromatography on silica gel. The schematic diagram is listed as follows:



Representative procedures for imidation of secondary amines: An undivided cell was

equipped with a magnet stirrer, two graphite electrode both as the working electrode and the counter electrode respectively. In this cell a solution of TsN_3 (0.2 mmol), secondary amine (0.6 mmol), Bu_4NPF_6 (0.3 mmol) in CH_3CN (6 mL) was added. The electrolyte was allowed to stir and the electrolysis was carried out at a constant current of 6 mA (electrode square 1.6 cm^2) at ambient temperature (25±1℃) for 3 h until the quantity of the electricity 3.4 F/mol was passed. Upon completion of the reaction, the solvent was removed with a rotary evaporator. The residue was washed with anhydrous ether (3×10 mL) and the insoluble Bu_4NPF_6 was filtered and dried for next use. The filtrate was combined and the solvent was removed with a rotary evaporator. The residue was purified by column chromatography on silica gel (Ditto for the handling of diazoalkanes).

Representative procedures for imidation of primary amines: An undivided cell was equipped with a magnet stirrer, two graphite electrode both as the working electrode and the counter electrode respectively. In this cell a solution of TsN_3 (0.2 mmol), primary amine (1.0 mmol), Bu_4NPF_6 (0.3 mmol) in $CHCl_3$ (6 mL) was added. The electrolyte was allowed to stir and the electrolysis was carried out at a constant current of 7 mA (electrode square 1.6 cm^2) at ambient temperature (25±1℃) for 3 h until the quantity of the electricity 3.9 F/mol was passed. Upon completion of the reaction, the solvent was removed with a rotary evaporator. The residue was washed with anhydrous ether (3×10 mL) and the insoluble Bu_4NPF_6 was filtered and dried for next use. The filtrate was combined and the solvent was removed with a rotary evaporator. The residue was purified by column chromatography on silica gel (Ditto for the handling of diazoalkanes).

Experimental details for the capture of radical cation A:

An undivided cell was equipped with a magnet stirrer, two graphite electrode both as the working electrode and the counter electrode respectively. In this cell a solution of triethylamine (5 mmol), Bu_4NPF_6 (0.3 mmol) in $CHCl_3$ (6 mL) was added. The electrolyte was allowed to stir and the electrolysis was carried out at a constant current of 4 mA (electrode square 1.6 cm^2) at ambient temperature (25±1℃) for 0.5 h. Then the electrolyte was frozen by liquid nitrogen. The EPR measurements were performed with a Braker Elexsys X-band (9.7 GHz) E580 EPR spectrometer at room temperature (Ditto for the handling of diazoalkanes).
The X-band EPR measurement was conducted at room temperature and the result was shown in Figure 1. The characterized *g*- value of the radical was 2.0022. The EPR simulation demonstrated that the magnetic interactions between the unpaired electron and one ^{14}N and six equivalent ^{1}H (see **A** in Scheme 1) gave rise to the resolved isotropic hyperfine constants, A_{14N} (43.69 MHz) and A_{1H} (30.98 MHz) displayed in Figure 1.

Experimental details for the measurement of CV of Et_3N:

The cyclic voltammetry (CV) of Et_3N was measured in 0.05 M *n*-Bu_4NPF_6/CH_3CN. As shown in

Figure S1 (Supporting Information, SI), the value of +1.60 V (vs SCE) in trace **b** (green) indicated the oxidation potential of Et_3N in acetonitrile while trace **a** (red) showed that blank solution was not electroactive in the potential window of interest.

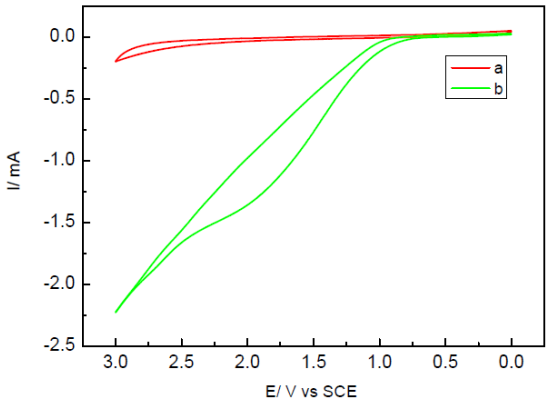
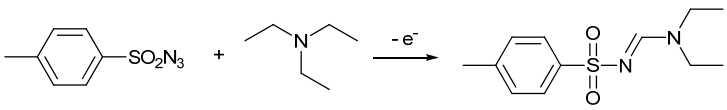


Figure S1. Cyclic voltammetry (CV) curves of a) 0.05 M *n*-Bu_4NPF_6/CH_3CN (red), b) 1mmol Et_3N, 0.05 M *n*-Bu_4NPF_6/CH_3CN (green), recorded at a glassy carbon electrode (diameter 4.0 mm), Scan rate: 100 mV s^{-1}, at room temperature.

Experimental details for the optimization of the reaction conditions:

The optimization of the reaction conditions was achieved by screening different solvents and electrodes, as shown in Table S1. When the platinum anode was replaced with a graphite anode, the reaction yield was hexane, and H_2O hardly gave the desired product and the use of MeOH and THF afforded the product with poor yields (Table S1, entries 8-14). In comparison with acetonitrile, the solvent $CHCl_3$ gave almost the same yield under the same condition while CH_2Cl_2 gave a moderate yield (Table S1, entries 3-7). Therefore CH_3CN and $CHCl_3$ should be the best solvents. Taking all factors into the consideration, the standard reaction condition was established as follows: CH_3CN as the solvent, Bu_4NPF_6 as the electrolyte, graphite as both anode and cathode.

Table S1. Synthesis of sulfonyl amidine under various conditions[a]



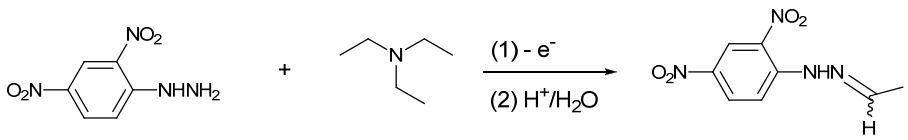
entry	solvent	anode	cathode	yield(%)[b]
1	CH_3CN	Pt	C	67
2	CH_3CN	C	C	96
3	CH_2Cl_2	Pt	C	45
4	CH_2Cl_2	C	C	68
5	CH_2Cl_2	Pt	Pt	39
6	$CHCl_3$	Pt	C	73
7	$CHCl_3$	C	C	94
8	1,4-dioxane	Pt	C	trace
9	1,4-dioxane	C	C	trace
10	CCl_4	C	C	N. D.
11	hexane	C	C	N. D.
12	THF	C	C	38
13	MeOH	C	C	33
14[c]	H_2O	C	C	trace

[a] Reaction conditions: the mixture of 0.2 mmol of TsN_3, 0.4 mmol of triethylamine, 0.3 mmol Bu_4NPF_6 and 6 mL of solvent was stirred and electrolyzed with constant current of 4 mA at room temperature for 3 h. [b] Isolated yield based on TsN_3. [c] Reaction conditions: the mixture of 0.2 mmol of TsN_3, 0.4 mmol of triethylamine, 0.3 mmol KNO_3 and 6 mL of H_2O was stirred and electrolyzed with constant current of 20 mA at room temperature for 3 h.

Experimental details for the investigation of reaction mechanism:

The electrolysis of TsN_3 and Et_3N was performed separately. After electrolysis of TsN_3 alone for 3 h, Et_3N was added and stirred for 1 h. Afterwards, TsN_3 could be recovered with a yield of 98% and no product was detected. In the parallel electrolysis of Et_3N for 3 h, the solution color was changed from colorless into yellow. When TsN_3 was added to this yellow solution and the mixture was stirred for 1 h, the corresponding sulfonyl amidine was obtained with a yield of 88%. These results showed that only Et_3N was electroactive substrate in this reaction.

Experimental details for the capture of enamine:



Scheme S1

An undivided cell was equipped with a magnet stirrer, two graphite electrode both as the working electrode and the counter electrode respectively. In this cell a solution of triethylamine (5 mmol), Bu_4NPF_6 (0.3 mmol) in CH_3CN (6 mL) was electrolyzed. The electrolyte was allowed to stir and the electrolysis was carried out at a constant current of 4 mA (electrode square 1.6 cm^2) at ambient temperature (25±1℃) for 2 h. Then 2, 4-dinitrobenzenehydrazine (0.100g, 0.5 mmol) was added and the reaction was quenched with 0.1 N and extracted with ethyl acetate (3×10 mL). The combined organic layers were washed with brine, dried over sodium sulfate. The filtrate was concentrated under reduced pressure. The residue was washed with anhydrous ether (3×10 mL) and the insoluble Bu_4NPF_6 was filtered and dried for next use. The filtrate was combined and the

solvent was removed with a rotary evaporator. The residue was purified by column chromatography on silica gel.

^{1}H-NMR ($CDCl_3$, 300 MHz, ppm): δ = 11.16 (s, 0.43 H), 11.03 (s, 0.95 H), 9.20-9.00 (m, 0.89 H), 8.40-8.20 (m, 1.22 H), 8.00-7.85 (m, 1.25 H), 7.70-7.40 (m, 1.05 H), 7.20-7.00 (m, 0.41 H), 2.14 (d, J = 5.4 Hz, 3 H), 2.08 (d, J = 5.7 Hz, 1.5 H).

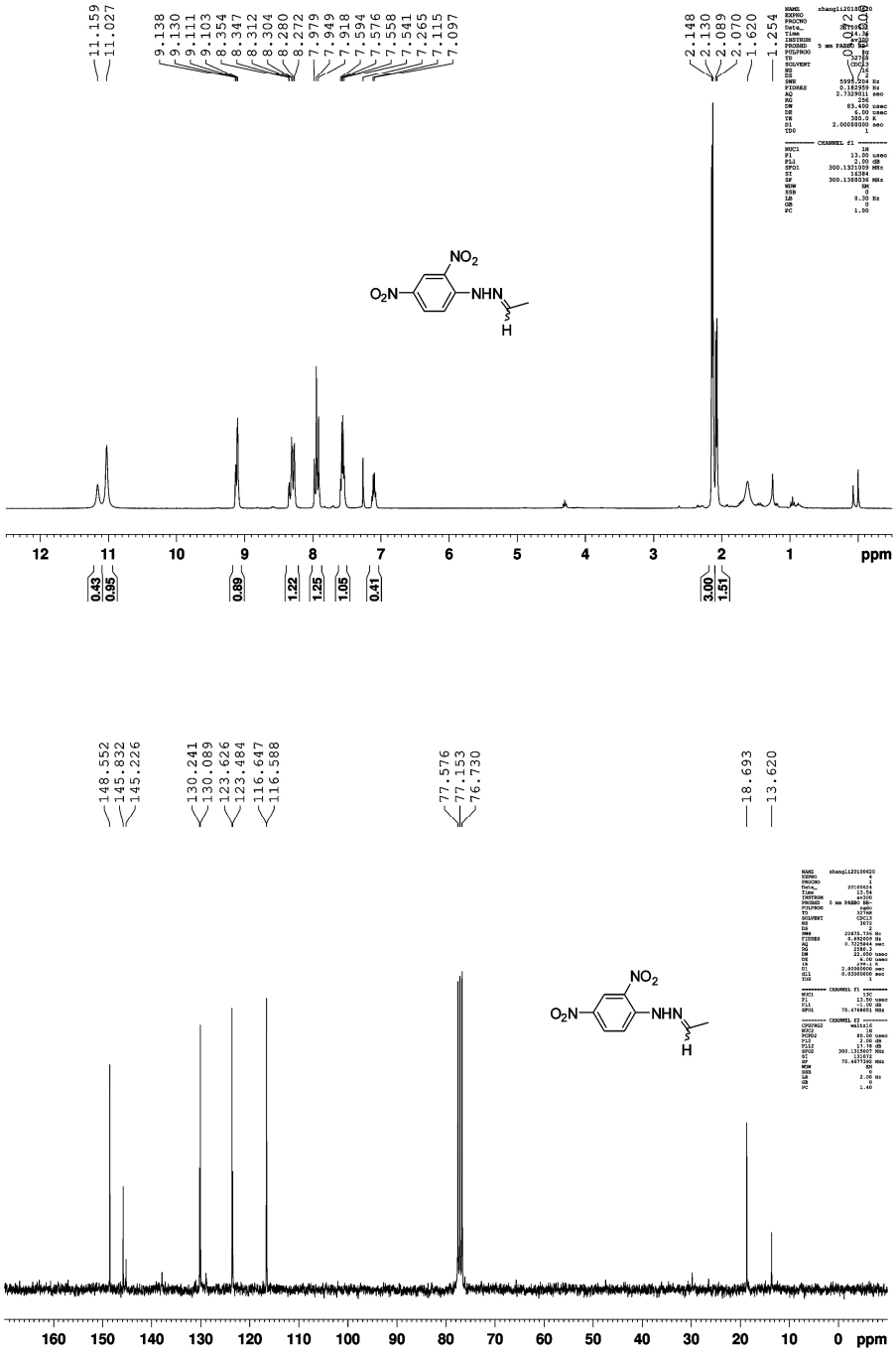
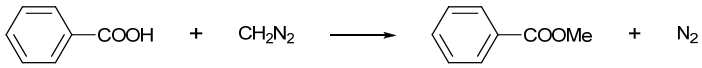


Figure S2

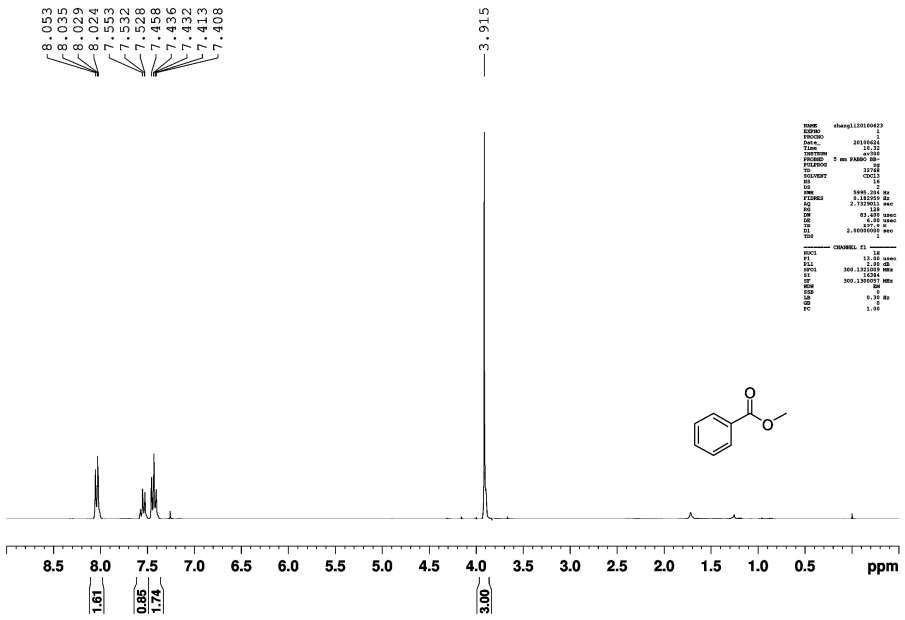
Experimental details for the capture of CH_2N_2:



Scheme S2

Caution: Azides and diazoalkanes may be hazardous and/or explosive. An undivided cell was equipped with a magnet stirrer, two graphite electrode both as the working electrode and the counter electrode respectively. In this cell a solution of TsN_3 (2 mmol), tertiary amine (4 mmol), Bu_4NPF_6 (0.3 mmol) in CH_3CN (6 mL) was added. In addition, for the safety, a gas tube was installed in the electrolytic cell and the terminal of this gas tube was inserted in the excess solution of benzoic acid (1.22g, 10 mmol, benzoic acid dissolved in CH_3CN) in a two-neck flask. One of the two necks was equipped with a buffer balloon for the safe. The electrolyte was allowed to stir and the electrolysis was carried out at a constant current of 4 mA (electrode square 1.6 cm^2) at ambient temperature (25±1℃) for 4 h. Upon completion of the reaction, the solvent was removed with a rotary evaporator. The residue was washed with anhydrous ether (3×10 mL). The filtrate was combined and the solvent was removed with a rotary evaporator. The residue was purified by column chromatography on silica gel.

^{1}H-NMR ($CDCl_3$, 300 MHz, ppm): δ = 8.10-7.90 (m, 2 H), 7.60-7.50 (m, 1 H), 7.50-7.40 (m, 2 H), 3.92 (s, 3 H).



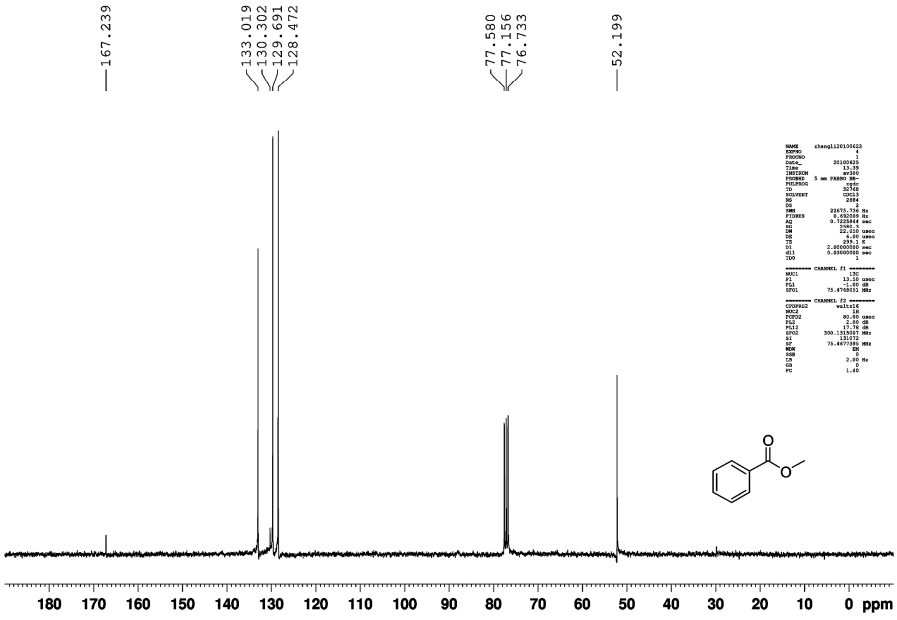
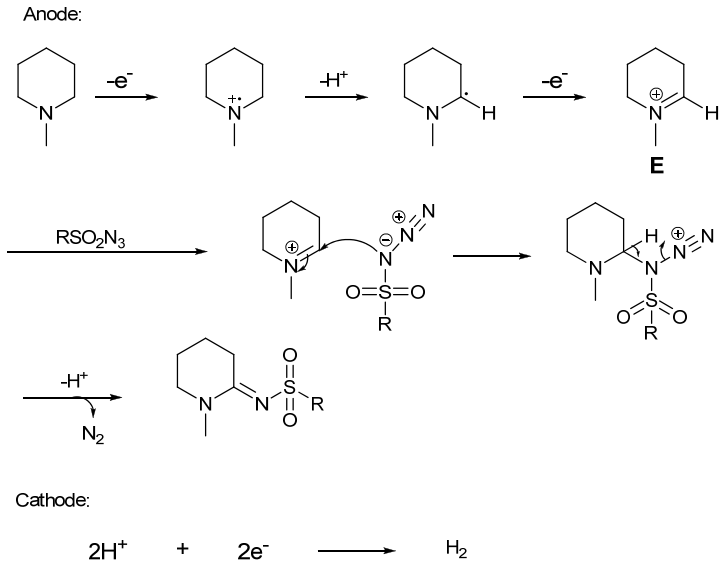


Figure S3

A representative mechanism for imidation of cyclic tertiary amine:

The mechanism of imidation of cyclic tertiary amine was also proposed as shown in Scheme S3. First, N-methylpiperidine is oxidized to form iminium ion **E** through the same route as triethylamine. It is easier for the cyclic hydrogen to be eliminated to produce iminium ion.[7a,8h] Then, iminium ion **E** is attacked by sulfonyl azide (nucleophile) with the release of one molecule of N_2 (Scheme S3, SI). At the cathode, the proton produced at the anode is reduced to hydrogen.



Scheme S3. Proposed mechanism for imidation of cyclic tertiary amine.

Experimental details for the measurment of CV of Et_2NH:

The cyclic voltammetry (CV) of Et_2NH was measured in 0.05 M *n*-Bu_4NPF_6/CH_3CN. As shown in Figure S4 (Supporting Information, SI), the value of +1.80 V (vs SCE) in trace **b** (green) indicated the oxidation potential of Et_2NH in acetonitrile while trace **a** (red) showed that blank solution was not electroactive in the potential window of interest.

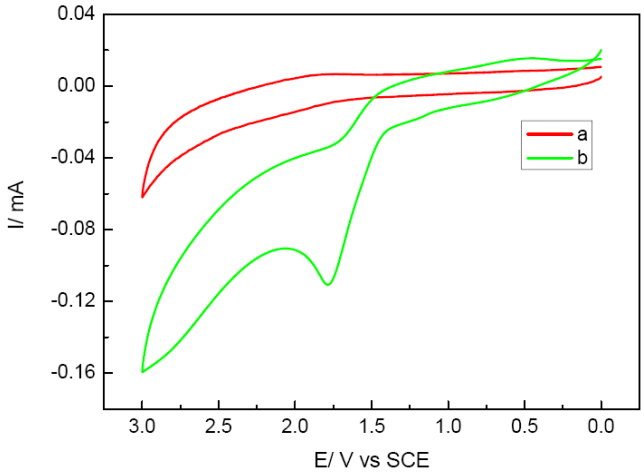


Figure S4. Cyclic voltammetry (CV) curves of a) 0.05 M *n*-Bu_4NPF_6/CH_3CN (red), b) 1mmol Et_2NH, 0.05 M *n*-Bu_4NPF_6/CH_3CN (green), recorded at a glassy carbon electrode (diameter 4.0 mm), Scan rate: 100 mV s^{-1}, at room temperature.

Experimental details for the capture of ethylamine:

An undivided cell was equipped with a magnet stirrer, two graphite electrode both as the working electrode and the counter electrode respectively. In this cell a solution of benzaldehyde (0.5 mmol), diethylamine (5 mmol), Bu_4NPF_6 (0.3 mmol) in CH_3CN (6 mL) was electrolyzed. The electrolyte was allowed to stir and the electrolysis was carried out at a constant current of 6 mA (electrode square 1.6 cm^2) at ambient temperature (25±1℃) for 4 h. Then the electrolyte was characterized by GC-MS.

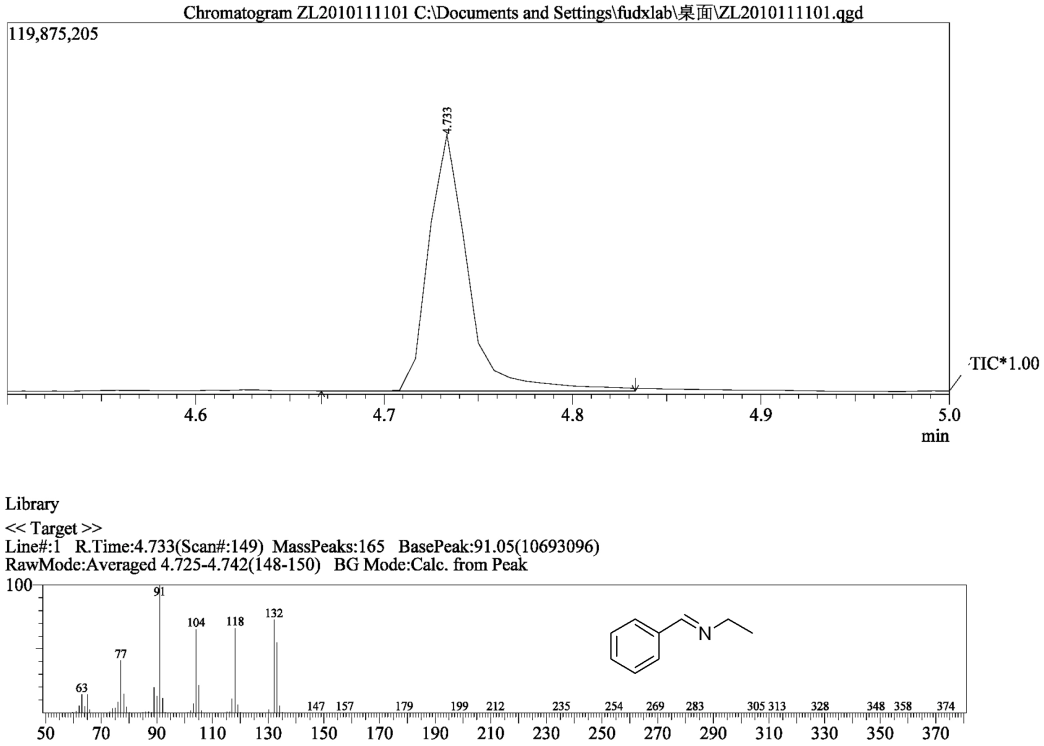
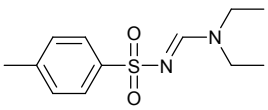


Figure S5

2. Characterization data of all products.

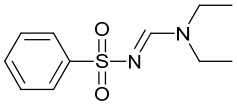
General Remarks: ^{1}H NMR and ^{13}C NMR were recorded on a Bruker AC-300 FT (^{1}H: 300 MHz, ^{13}C: 75 MHz) using TMS as internal reference. The chemical shifts (δ) and coupling constants (J) were expressed in ppm and Hz respectively. Infrared samples were recorded on a Perkin-Elmer 2000 FTIR spectrometer.

(E)-N, N-diethyl-N'-tosylformimidamide



^{1}H-NMR ($CDCl_3$, 300 MHz, ppm): δ = 8.14 (s, 1 H), 7.75 (d, J = 8.4 Hz, 2 H), 7.25 (d, J = 8.4 Hz, 2 H), 3.60-3.32 (m, 4 H), 2.39 (s, 3 H), 1.31-1.20 (t, J = 7.2 Hz, 3 H), 1.20-1.10 (t, J = 7.2 Hz, 3 H); ^{13}C-NMR ($CDCl_3$, 75 MHz, ppm): δ = 158.1, 142.4, 139.9, 129.4, 126.5, 47.2, 41.1, 29.8, 21.6, 14.6, 12.2; IR (liquid film, cm^{-1}): ν = 2978, 2937, 1610, 1451, 1345, 1298, 1283, 1148, 1087, 955, 875, 817, 768, 674.

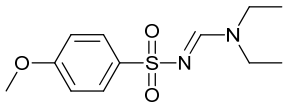
(E)-N, N-diethyl-N'-(phenylsulfonyl)formimidamide



^{1}H-NMR ($CDCl_3$, 300 MHz, ppm): δ = 8.16 (s, 1 H), 8.00-7.80 (m, 2 H), 7.60-7.40 (m, 3 H),

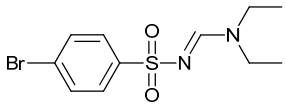
3.60-3.32 (m, 4 H), 1.30-1.24 (t, J = 7.2 Hz, 3 H), 1.20-1.10 (t, J = 7.2 Hz, 3 H); ^{13}C-NMR ($CDCl_3$, 75 MHz, ppm): δ = 158.3, 131.8, 128.9, 128.8, 126.5, 47.2, 41.1, 14.6, 12.2; IR (liquid film, cm^{-1}): v = 2978, 2938, 1612, 1447, 1345, 1299, 1149, 1088, 955, 875, 819, 770, 723, 690.

(E)-N, N-diethyl-N'-(4-methoxyphenylsulfonyl)formimidamide



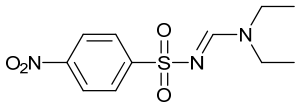
^{1}H-NMR ($CDCl_3$, 300 MHz, ppm): δ = 8.14 (s, 1 H), 7.81 (d, J = 9.0 Hz, 2 H), 6.92 (d, J = 9.0 Hz, 2 H), 3.84 (s, 3 H), 3.53-3.30 (m, 4 H), 1.30-1.20 (t, J = 7.2 Hz, 3 H), 1.20-1.10 (t, J = 7.2 Hz, 3 H); ^{13}C-NMR ($CDCl_3$, 75 MHz, ppm): δ = 158.0, 128.5, 113.9, 55.6, 47.1, 41.0, 14.6, 12.2; IR (liquid film, cm^{-1}): v = 2977, 2939, 1612, 1499, 1452, 1346, 1290, 1257, 1146, 1090, 1025, 955, 875, 768, 676.

(E)-N'-(4-bromophenylsulfonyl)-N, N-diethylformimidamide



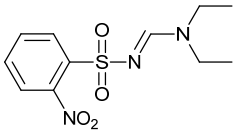
^{1}H-NMR ($CDCl_3$, 300 MHz, ppm): δ = 8.13 (s, 1 H), 7.75 (d, J = 8.4 Hz, 2 H), 7.59 (d, J = 8.4 Hz, 2 H), 3.53-3.30 (m, 4 H), 1.30-1.20 (t, J = 7.2 Hz, 3 H), 1.20-1.10 (t, J = 7.2 Hz, 3 H); ^{13}C-NMR ($CDCl_3$, 75 MHz, ppm): δ = 158.2, 132.0, 128.1, 47.3, 41.2, 14.6, 12.2; IR (liquid film, cm^{-1}): v = 2977, 2938, 1611, 1449, 1344, 1302, 1271, 1147, 1086, 877, 770, 740.

(E)-N, N-diethyl-N'-(4-nitrophenylsulfonyl)formimidamide



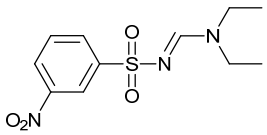
^{1}H-NMR ($CDCl_3$, 300 MHz, ppm): δ =8.31 (d, J = 9.0 Hz, 2 H), 8.16 (s, 1 H), 8.07 (d, J = 9.0 Hz, 2 H), 3.55-3.32 (m, 4 H), 1.33-1.25 (t, J = 7.2 Hz, 3 H), 1.20-1.10 (t, J = 7.2 Hz, 3 H); ^{13}C-NMR ($CDCl_3$, 75 MHz, ppm): δ = 158.5, 127.8, 124.1, 47.6, 41.4, 14.6, 12.2; IR (liquid film, cm^{-1}): v = 2980, 2940, 1613, 1528, 1449, 1345, 1295, 1150, 1086, 957, 881, 853, 732, 686.

(E)-N, N-diethyl-N'-(2-nitrophenylsulfonyl)formimidamide



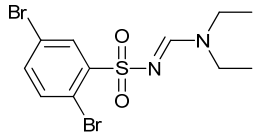
^{1}H-NMR ($CDCl_3$, 300 MHz, ppm): δ = 8.30-8.20 (m, 1 H), 8.11 (s, 1 H), 7.80-7.60 (m, 3 H), 3.60-3.32 (m, 4 H), 1.39-1.29 (t, J = 7.2 Hz, 3 H), 1.20-1.10 (t, J = 7.2 Hz, 3 H); ^{13}C-NMR ($CDCl_3$, 75 MHz, ppm): δ = 160.0, 132.8, 132.1, 130.9, 124.1, 47.5, 41.4, 14.5, 12.2; IR (liquid film, cm^{-1}): v = 2979, 2939, 1616, 1540, 1344, 1309, 1154, 1121, 880, 772, 607.

(E)-N,N-diethyl-N'-(3-nitrophenylsulfonyl)formimidamide



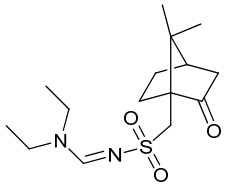
^{1}H-NMR ($CDCl_3$, 300 MHz, ppm): δ = 8.71 (s, 1 H), 8.40-8.34 (m, 1 H), 8.24 (d, J = 7.8 Hz, 1 H), 8.18 (s, 1 H), 7.73-7.60 (m, 1 H), 3.60-3.38 (m, 4 H), 1.35-1.27 (t, J = 7.2 Hz, 3 H), 1.20-1.10 (t, J = 7.2 Hz, 3 H); ^{13}C-NMR ($CDCl_3$, 75 MHz, ppm): δ = 158.5, 132.3, 130.2, 128.9, 126.4, 121.8, 47.5, 41.4, 14.5, 12.2; IR (liquid film, cm^{-1}): ν = 2979, 2939, 1613, 1530, 1351, 1156, 1118, 886, 664, 610, 587.

(E)-N'-(2,5-dibromophenylsulfonyl)-N,N-diethylformimidamide



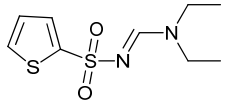
^{1}H-NMR ($CDCl_3$, 300 MHz, ppm): δ = 8.39 (s, 1 H), 8.29 (s, 1 H), 7.56-7.40 (m, 2 H), 3.60-3.40 (m, 4 H), 1.40-1.29 (t, J = 7.2 Hz, 3 H), 1.25-1.10 (t, J = 7.2 Hz, 3 H); ^{13}C-NMR ($CDCl_3$, 75 MHz, ppm): δ = 160.2, 136.3, 135.9, 133.5, 121.6, 47.5, 41.5, 14.7, 12.1; IR (liquid film, cm^{-1}): ν = 2978, 2937, 1614, 1445, 1343, 1303, 1153, 1023, 956, 880, 882, 766, 611, 592.

(E)-N'-(((1R,4R)-7,7-dimethyl-2-oxobicyclo[2.2.1]heptan-1-yl)methylsulfonyl)-N,N-diethylformimidamide



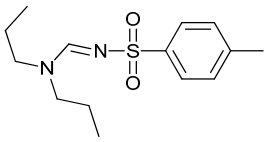
^{1}H-NMR ($CDCl_3$, 300 MHz, ppm): δ = 8.05 (s, 1 H), 3.55-3.41 (m, 4 H), 3.41-3.35 (m, 1 H), 3.10-2.90 (m, 1 H), 2.75-2.55 (m, 1 H), 2.40-2.20 (m, 1 H), 2.10-1.96 (m, 2 H), 1.95-1.80 (m, 1 H), 1.80-1.60 (m, 1 H), 1.50-1.35 (m, 1 H), 1.35-1.25 (t, J = 7.2 Hz, 3 H), 1.25-1.10 (t, J = 7.2 Hz, 3 H), 1.15 (s, 3 H), 0.85 (s, 3 H); ^{13}C-NMR ($CDCl_3$, 75 MHz, ppm): δ = 158.8, 58.6, 50.8, 48.1, 47.0, 42.8, 42.7, 40.8, 27.1, 24.8, 20.1, 19.8, 14.5, 12.1; IR (liquid film, cm^{-1}): ν = 2966, 2887, 1744, 1613, 1454, 1352, 1304, 1127, 955, 873, 766.

(E)-N,N-diethyl-N'-(thiophen-2-ylsulfonyl)formimidamide



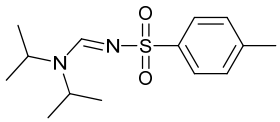
^{1}H-NMR ($CDCl_3$, 300 MHz, ppm): δ = 8.15 (s, 1 H), 7.62-7.53 (m, 1 H), 7.50-7.45 (m, 1 H), 7.05-7.00 (m, 1 H), 3.60-3.40 (m, 4 H), 1.40-1.25 (t, J = 7.2 Hz, 3 H), 1.23-1.10 (t, J = 7.2 Hz, 3 H); ^{13}C-NMR ($CDCl_3$, 75 MHz, ppm): δ = 158.4, 130.6, 130.4, 127.0, 47.4, 41.3, 14.6, 12.2; IR (liquid film, cm^{-1}): ν = 2977, 2938, 1611, 1451, 1341, 1298, 1133, 1088, 1014, 876, 672.

(E)-N,N-dipropyl-N'-tosylformimidamide



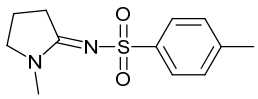
^{1}H-NMR ($CDCl_3$, 300 MHz, ppm): δ = 8.15 (s, 1 H), 7.75 (d, J = 8.4 Hz, 2 H), 7.25 (d, J = 8.4 Hz, 2 H), 3.40-3.20 (m, 4 H), 2.39 (s, 3 H), 1.68-1.50 (m, 4 H), 1.00-0.80 (m, 6 H); ^{13}C-NMR ($CDCl_3$, 75 MHz, ppm): δ = 159.0, 129.4, 126.5, 54.4, 48.0, 22.1, 21.6, 20.1, 11.3, 11.0; IR (liquid film, cm^{-1}): v = 2964, 2931, 1607, 1451, 1343, 1297, 1283, 1147, 1087, 909, 877, 674.

(E)-N,N-diisopropyl-N'-tosylformimidamide



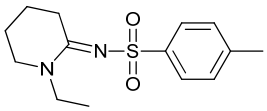
^{1}H-NMR ($CDCl_3$, 300 MHz, ppm): δ = 8.25 (s, 1 H), 7.75 (d, J = 8.4 Hz, 2 H), 7.25 (d, J = 8.4 Hz, 2 H), 4.60-4.40 (m, 1 H), 3.80-3.50 (m, 1 H), 2.39 (s, 3 H), 1.31 (d, J = 6.9 Hz, 6 H), 1.21 (d, J = 6.9 Hz, 6 H); ^{13}C-NMR ($CDCl_3$, 75 MHz, ppm): δ = 156.5, 129.4, 126.4, 48.6, 48.0, 23.7, 21.6, 19.8; IR (liquid film, cm^{-1}): v = 2976, 2933, 1602, 1341, 1282, 1146, 1088, 891, 839, 670.

(E)-4-methyl-N-(1-methylpyrrolidin-2-ylidene)benzenesulfonamide



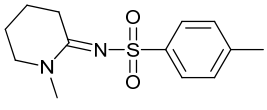
^{1}H-NMR ($CDCl_3$, 300 MHz, ppm): δ = 7.81 (d, J = 8.4 Hz, 2 H), 7.24 (d, J = 8.4 Hz, 2 H), 3.50-3.40 (t, J = 7.2 Hz, 2 H), 3.10-3.00 (t, J = 7.8 Hz, 2 H), 2.97 (s, 3 H), 2.39 (s, 3 H), 2.20-2.00 (m, 2 H); ^{13}C-NMR ($CDCl_3$, 75 MHz, ppm): δ = 170.0, 142.1, 140.7, 129.3, 126.6, 51.8, 32.1, 30.8, 21.6, 19.1; IR (liquid film, cm^{-1}): v = 2927, 1600, 1492, 1301, 1281, 1144, 1091, 1007, 902, 813, 670, 606, 580, 558.

(E)-N-(1-ethylpiperidin-2-ylidene)-4-methylbenzenesulfonamide



^{1}H-NMR ($CDCl_3$, 300 MHz, ppm): δ = 7.81 (d, J = 8.4 Hz, 2 H), 7.26 (d, J = 8.4 Hz, 2 H), 3.60-3.40 (m, 2 H), 3.38-3.10 (t, J = 6.0 Hz, 2 H), 3.10-3.00 (t, J = 6.0 Hz, 2 H), 2.40 (s, 3 H), 1.80-1.70 (m, 4 H), 1.18-1.00 (t, J = 7.2 Hz, 3 H); ^{13}C-NMR ($CDCl_3$, 75 MHz, ppm): δ = 165.3, 141.7, 129.8, 129.2, 126.6, 126.3, 48.3, 45.7, 28.7, 22.4, 21.5, 19.7, 11.5; IR (liquid film, cm^{-1}): v = 2937, 2871, 1562, 1482, 1261, 1141, 1093, 1066, 935, 815, 671, 584, 554.

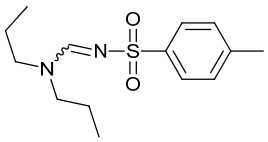
(E)-4-methyl-N-(1-methylpiperidin-2-ylidene)benzenesulfonamide



^{1}H-NMR ($CDCl_3$, 300 MHz, ppm): δ = 7.82 (d, J = 7.8 Hz, 2 H), 7.24 (d, J = 7.8 Hz, 2 H), 3.40-3.30 (m, 2 H), 3.03 (s, 3 H), 2.39 (s, 3 H), 1.90-1.60 (m, 6 H); ^{13}C-NMR ($CDCl_3$, 75 MHz,

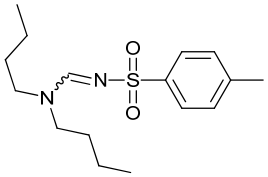
ppm): δ = 166.1, 141.8, 141.5, 129.6, 129.2, 126.6, 126.4, 50.9, 38.5, 28.7, 22.3, 21.4, 19.8; IR (liquid film, cm^{-1}): ν = 2950, 2870, 1571, 1483, 1273, 1142, 1089, 962, 823, 675.

(Z/E)-N,N-dipropyl-N'-tosylformimidamide



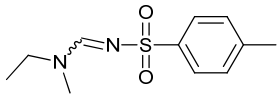
^{1}H-NMR ($CDCl_3$, 300 MHz, ppm): δ = 8.15 (s, 0.59 H), 7.81 (d, J = 8.4 Hz, 0.69 H), 7.75 (d, J = 8.4 Hz, 1.14 H), 7.30-7.20 (m, 1.83 H), 3.40-3.20 (m, 4 H), 2.39 (s, 3 H), 1.80-1.40 (m, 4 H), 1.00-0.70 (m, 6 H); ^{13}C-NMR ($CDCl_3$, 75 MHz, ppm): δ = 169.0, 162.9, 159.0, 142.3, 139.9, 129.7, 129.4, 129.1, 129.0, 126.5, 126.4, 54.4, 50.7, 49.3, 47.9, 24.4, 23.4, 22.4, 22.2, 21.5, 20.1, 11.9, 11.4; IR (liquid film, cm^{-1}): ν = 2965, 2935, 2876, 1670, 1609, 1545, 1297, 1283, 1147, 1087, 878, 676.

(Z/E)-N,N-dibutyl-N'-tosylformimidamide



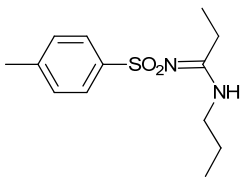
^{1}H-NMR ($CDCl_3$, 300 MHz, ppm): δ = 8.13 (s, 0.82 H), 7.82 (d, J = 8.4 Hz, 0.32 H), 7.75 (d, J = 8.4 Hz, 1.61 H), 7.30-7.20 (m, 1.94 H), 3.50-3.20 (m, 4 H), 2.36 (s, 3 H), 1.70-1.40 (m, 4 H), 1.40-1.10 (m, 4 H), 1.00-0.80 (m, 6 H); ^{13}C-NMR ($CDCl_3$, 75 MHz, ppm): δ = 158.8, 142.3, 140.0, 129.3, 129.0, 126.4, 126.1, 52.4, 47.1, 46.0, 30.8, 28.8, 21.5, 20.2, 20.0, 19.7, 13.8, 13.7, 13.6; IR (liquid film, cm^{-1}): ν = 2958, 2932, 2872, 1672, 1608, 1458, 1348, 1298, 1147, 1088, 892, 814.

(Z/E)-N-ethyl-N-methyl-N'-tosylformimidamide



^{1}H-NMR ($CDCl_3$, 300 MHz, ppm): δ = 8.18 (s, 0.68 H), 8.09 (s, 0.36 H), 7.80-7.70 (m, 2 H), 7.25 (d, J = 7.2 Hz, 2 H), 3.50-3.30 (m, 2 H), 3.08 (s, 1 H), 2.98 (s, 2 H), 2.40 (s, 3 H),1.28-1.20 (t, 2 H), 1.20-1.10 (m, 1 H); ^{13}C-NMR ($CDCl_3$, 75 MHz, ppm): δ = 158.7, 158.5, 142.5, 142.4, 139.8, 139.7, 129.4, 129.3, 126.6, 126.5, 49.6, 43.1, 38.9, 33.2, 21.6, 13.9, 11.3; IR (liquid film, cm^{-1}): ν = 2977, 2935, 1617, 1343, 1297, 1282, 1147, 1086, 905, 888, 673.

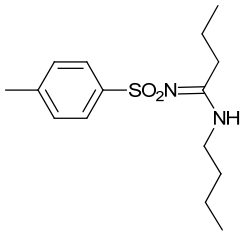
(Z/E)-N-propyl- N'-tosylpropionimidamide



^{1}H-NMR ($CDCl_3$, 300 MHz, ppm): δ = 8.25 (s, 0.39 H), 7.85-7.70 (m, 2.04 H), 7.30-7.20 (m, 2.22

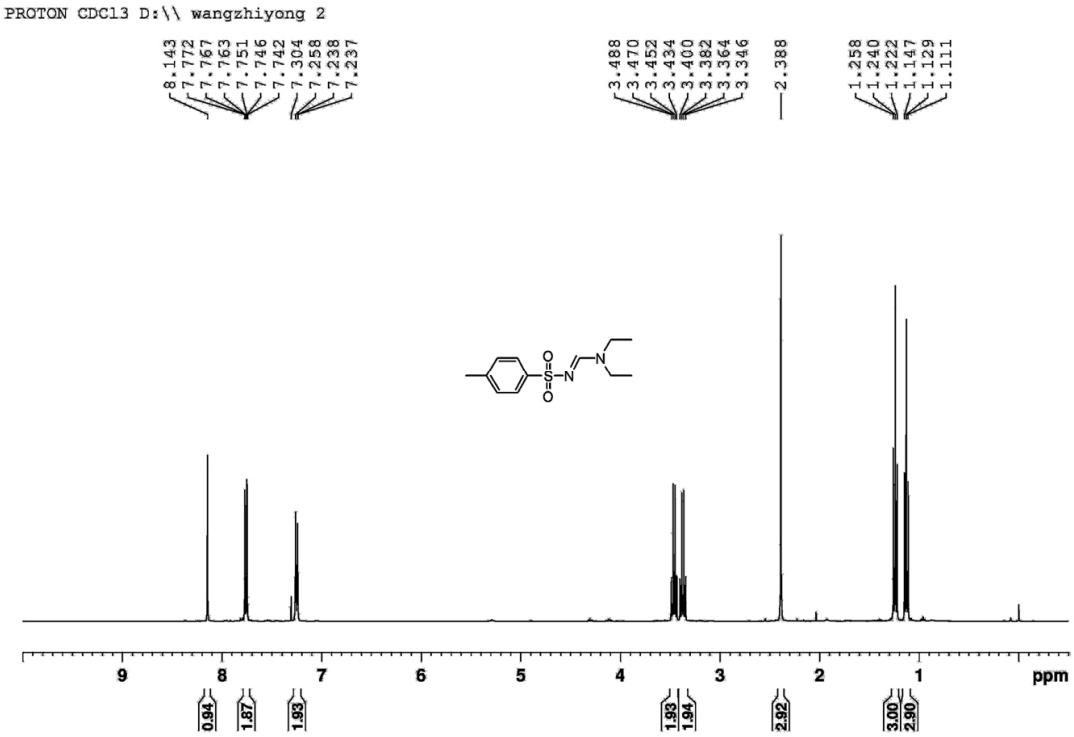
H), 5.90 (s, 0.57 H), 3.35-3.20 (m, 2 H), 2.92-2.80 (m, 1 H), 2.40 (s, 3 H), 2.40-2.30 (m, 1 H), 1.70-1.50 (m, 2.19 H), 1.30-1.20 (m, 1.87 H), 1.20-1.10 (m, 1.37 H) 1.00-0.90 (m, 1.46 H) 0.90-0.80 (m, 1.85 H); ^{13}C-NMR ($CDCl_3$, 75 MHz, ppm): δ = 169.7, 169.6, 142.7, 142.0, 141.3, 140.0, 129.4, 129.2, 126.4, 126.3, 45.5, 43.7, 27.5, 26.9, 23.1, 21.8, 21.6, 21.5, 11.5, 11.4, 11.2, 10.4; IR (liquid film, cm^{-1}): ν = 3313, 2966, 2934, 2877, 1558, 1271, 1142, 1088, 953, 694. HRMS calc. $C_{13}H_{20}N_2O_2S$ (M^+): 268.1245. Found: 268.1250.

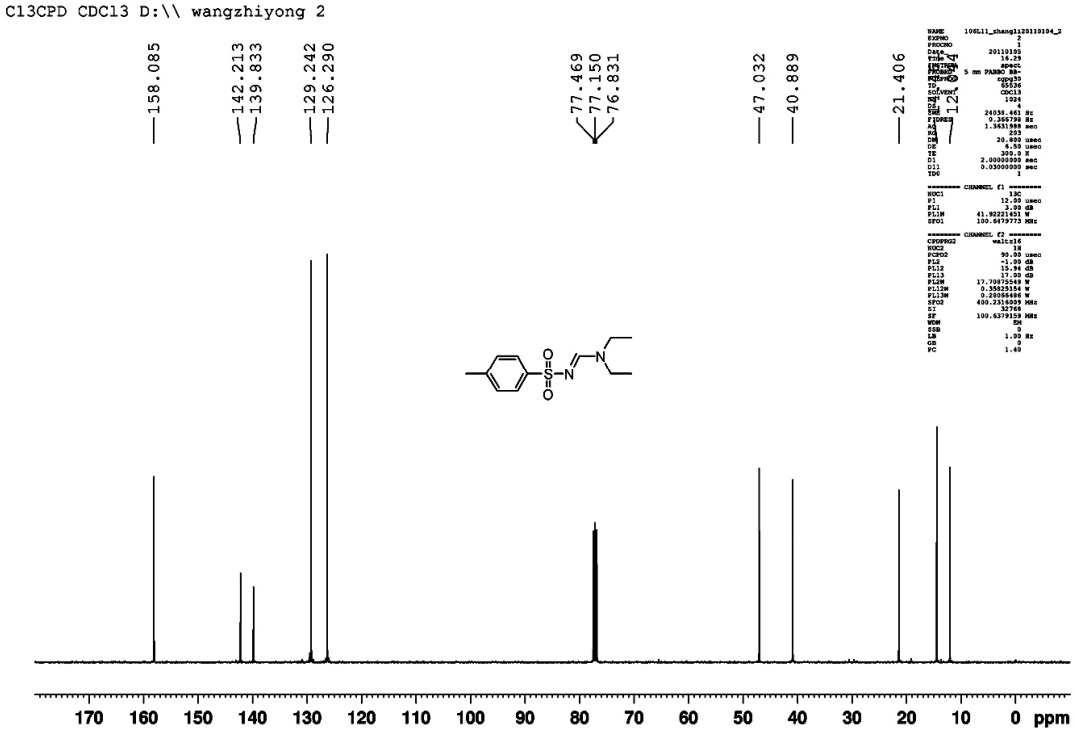
(Z/E)-N-butyl- N'-tosylbutyrimidamide

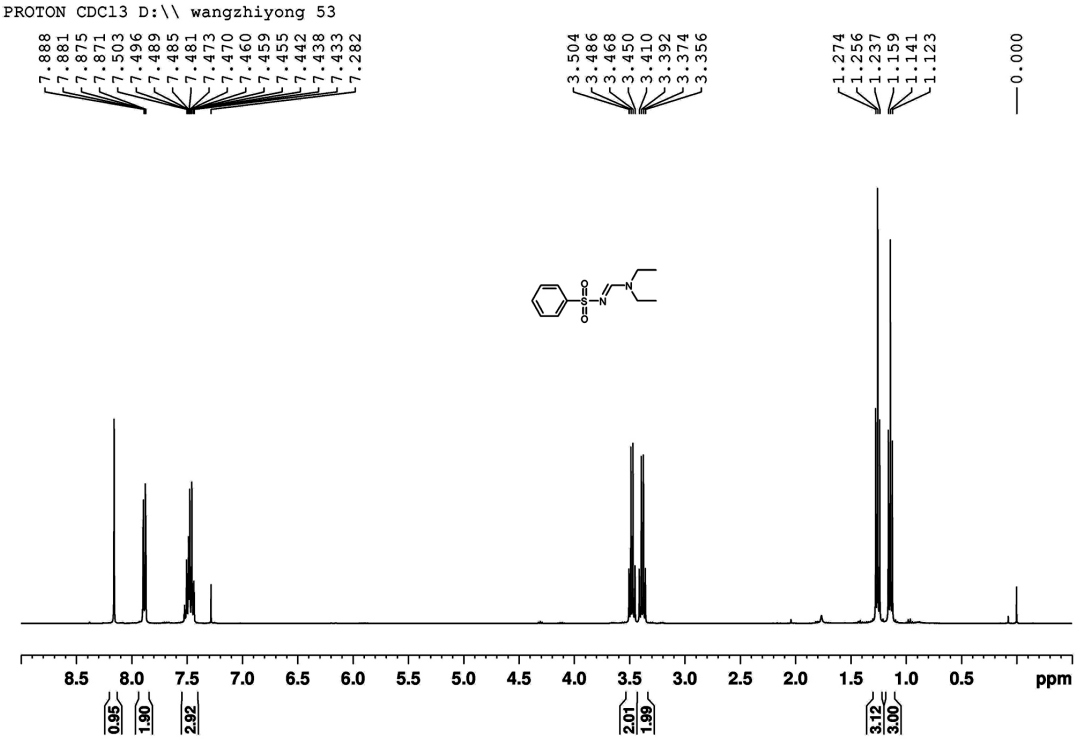


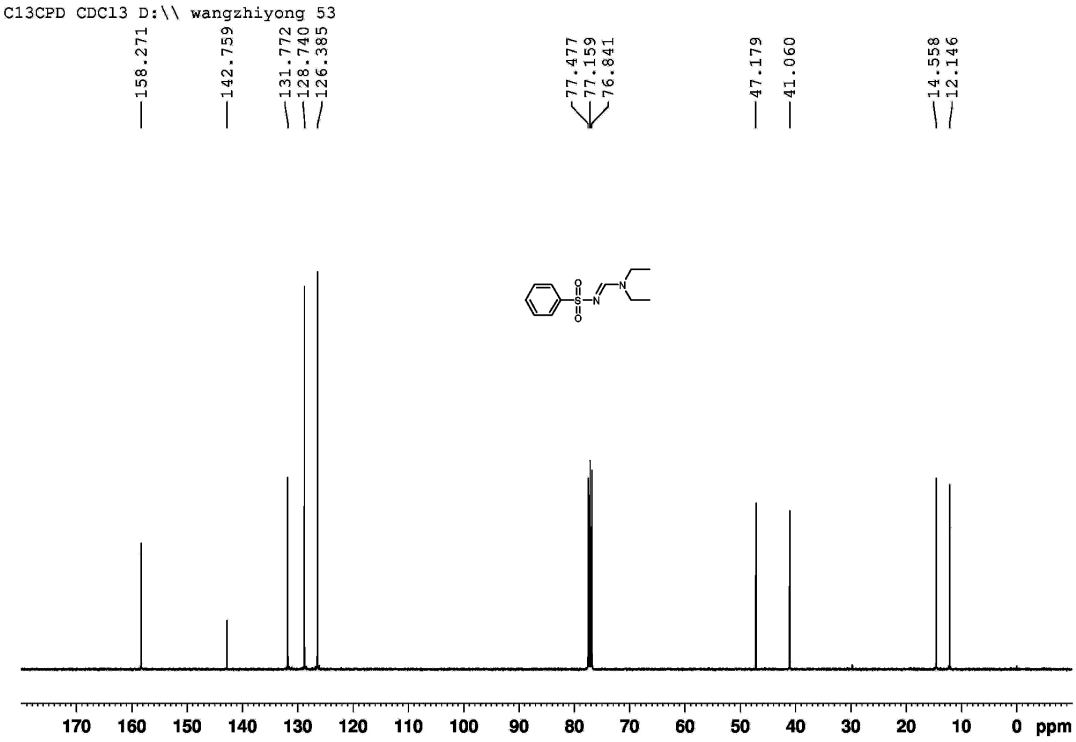
^{1}H-NMR ($CDCl_3$, 300 MHz, ppm): δ = 8.20 (s, 0.35 H), 7.85-7.70 (m, 1.87 H), 7.30-7.20 (m, 2.28 H), 5.67 (s, 0.43 H), 3.40-3.20 (m, 1.86 H), 2.80-2.60 (m, 0.97 H), 2.43 (s, 2.87 H), 2.30-2.20 (m, 0.88 H), 1.80-1.45 (m, 4.57 H), 1.45-1.35 (m, 0.96 H), 1.35-1.20 (m, 1.00 H) 1.10-0.80 (m, 5.97 H); ^{13}C-NMR ($CDCl_3$, 75 MHz, ppm): δ = 168.7, 168.5, 142.6, 141.9, 141.4, 140.0, 129.3, 129.2, 126.4, 43.7, 41.8, 36.2, 35.3, 31.9, 30.7, 21.6, 21.5, 20.8, 20.2, 19.9, 19.7, 13.9, 13.8, 13.7; IR (liquid film, cm^{-1}): ν = 3331, 2960, 2932, 2873, 1651, 1556, 1334, 1267, 1142, 1089, 814, 695. HRMS calc. $C_{15}H_{24}N_2O_2S$ (M^+): 296.1558. Found: 296.1553.

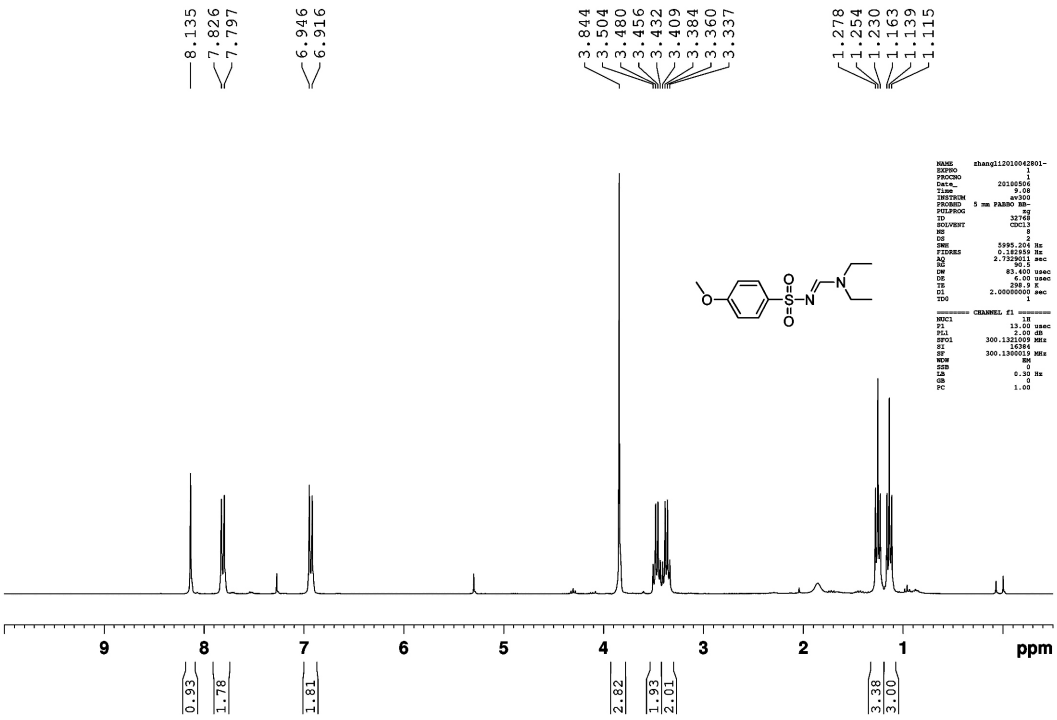
3. NMR Spectra of all products.


PROTON CDCl3 D:\\ wangzhiyong 2
8.143
7.772
7.767
7.763
7.751
7.746
7.742
7.304
7.258
7.238
7.237
3.488
3.470
3.452
3.434
3.400
3.382
3.364
3.346
2.388
1.258
1.240
1.222
1.147
1.129
1.111
ppm
0.94
1.87
1.93
1.93
1.94
2.92
3.00
2.90


C13CPD CDCl3 D:\\ wangzhiyong 2
158.085
142.213
139.833
129.242
126.290
77.469
77.150
76.831
47.032
40.889
21.406
ppm


PROTON CDCl3 D:\\ wangzhiyong 53
7.888
7.881
7.875
7.871
7.503
7.496
7.489
7.485
7.481
7.473
7.470
7.460
7.459
7.455
7.442
7.438
7.433
7.282
3.504
3.486
3.468
3.450
3.410
3.392
3.374
3.356
1.274
1.256
1.237
1.159
1.141
1.123
0.000
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm
0.95
1.90
2.92
2.01
1.99
3.12
3.00


C13CPD CDCl3 D:\\ wangzhiyong 53
158.271
142.759
131.772
128.740
126.385
77.477
77.159
76.841
47.179
41.060
14.558
12.146
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm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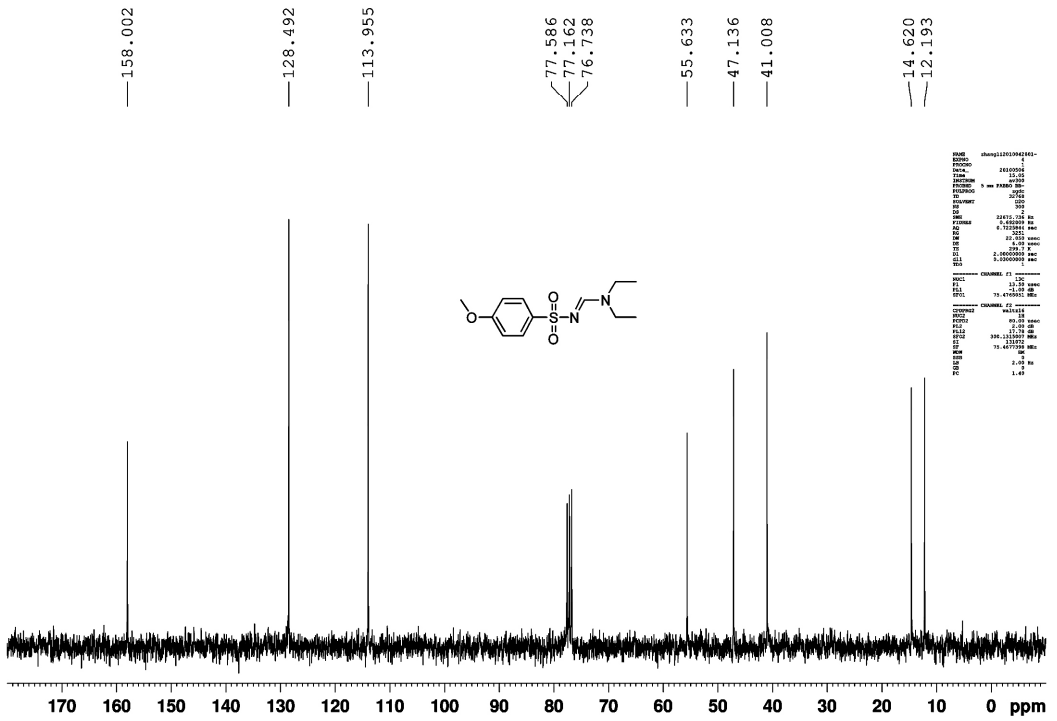
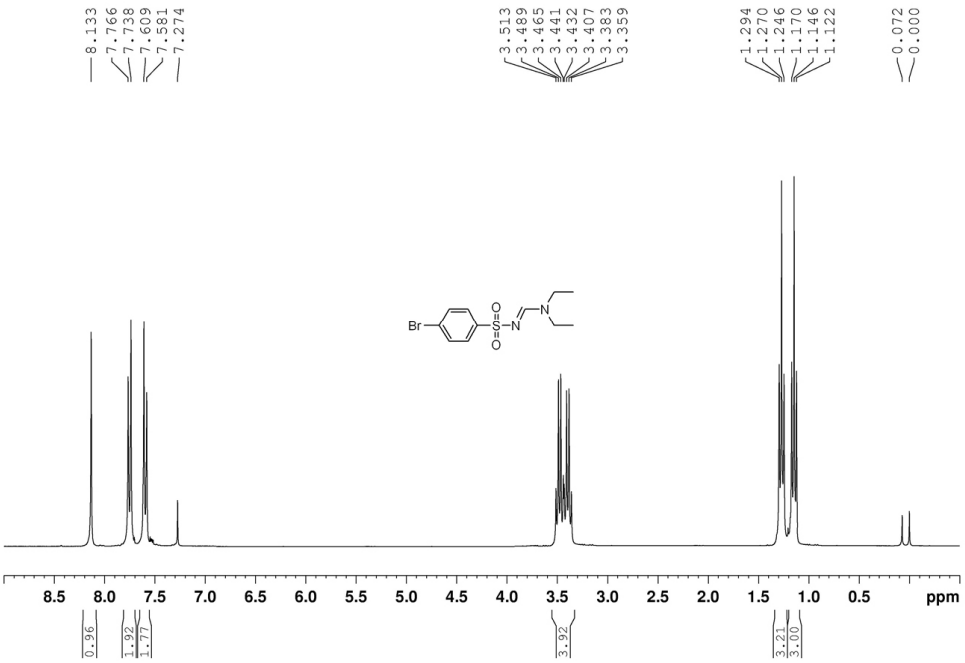
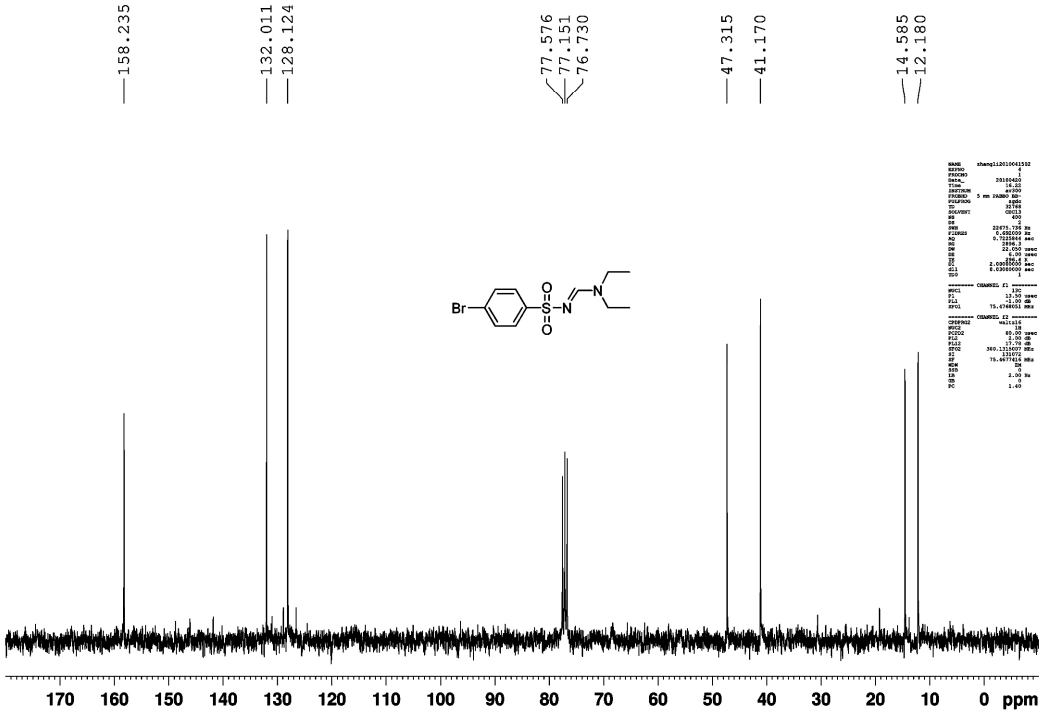
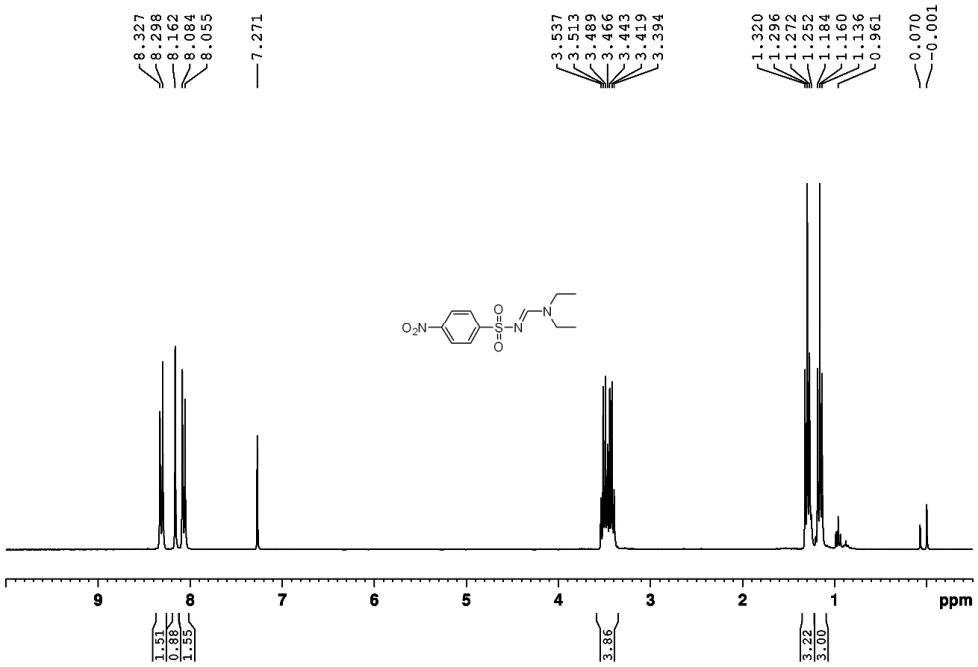
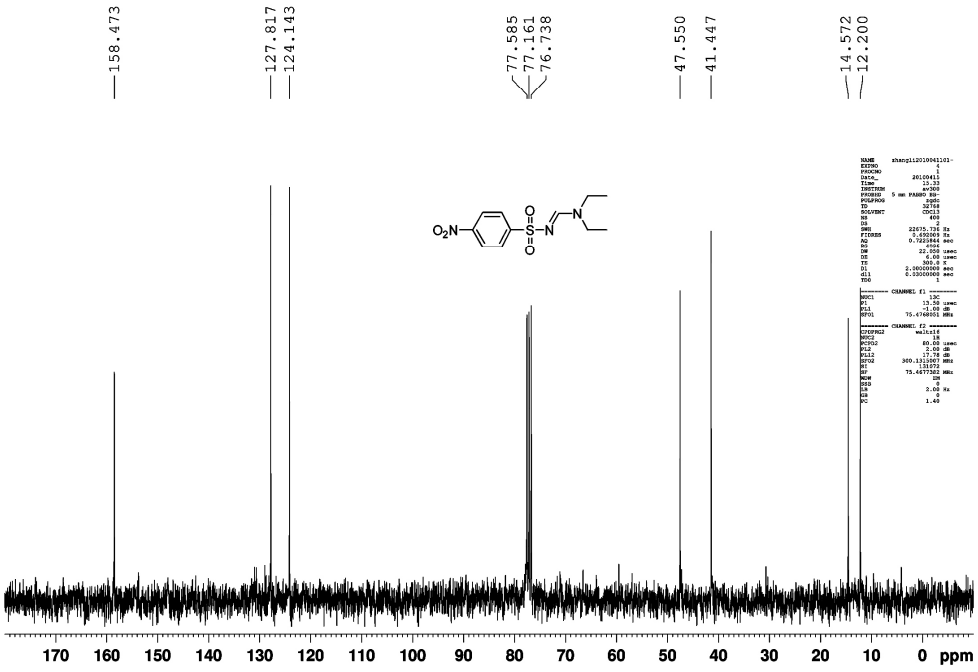
7.826
7.797
6.946
6.916
3.844
3.504
3.480
3.456
3.432
3.409
3.384
3.360
3.337
1.278
1.254
1.230
1.163
1.139
1.115
9
8
7
6
5
4
3
2
1
ppm
0.93
1.78
1.81
2.82
1.93
2.01
3.38
3.00


158.002
128.492
113.955
77.586
77.162
76.738
55.633
47.136
41.008
14.620
12.193
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm


8.133
7.766
7.738
7.609
7.581
7.274
3.513
3.489
3.465
3.441
3.432
3.407
3.383
3.359
1.294
1.270
1.246
1.170
1.146
1.122
0.072
0.000
Br
O
S
N
N
O
O
8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 ppm
0.96
1.92
1.77
3.92
3.21
3.00


158.235
132.011
128.124
77.576
77.151
76.730
47.315
41.170
14.585
12.180
Br
O
S
N
N
O
O
170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm


8.327
8.298
8.162
8.084
8.055
7.271
3.537
3.513
3.489
3.466
3.443
3.419
3.394
1.320
1.296
1.272
1.252
1.184
1.160
1.136
0.961
0.070
-0.001
9
8
7
6
5
4
3
2
1
ppm
1.51
0.88
1.55
3.86
3.22
3.00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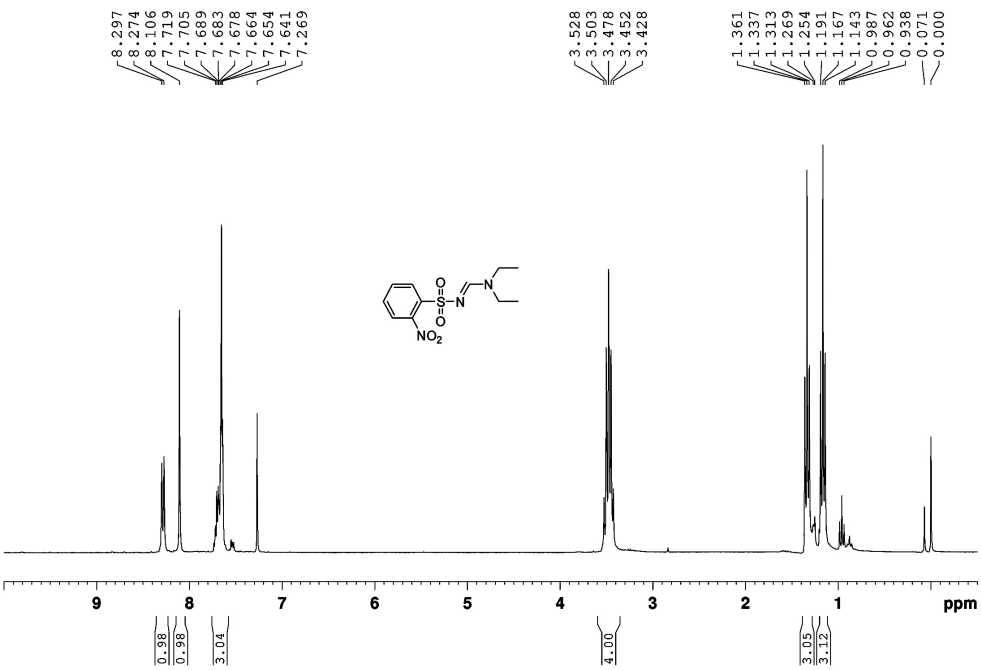
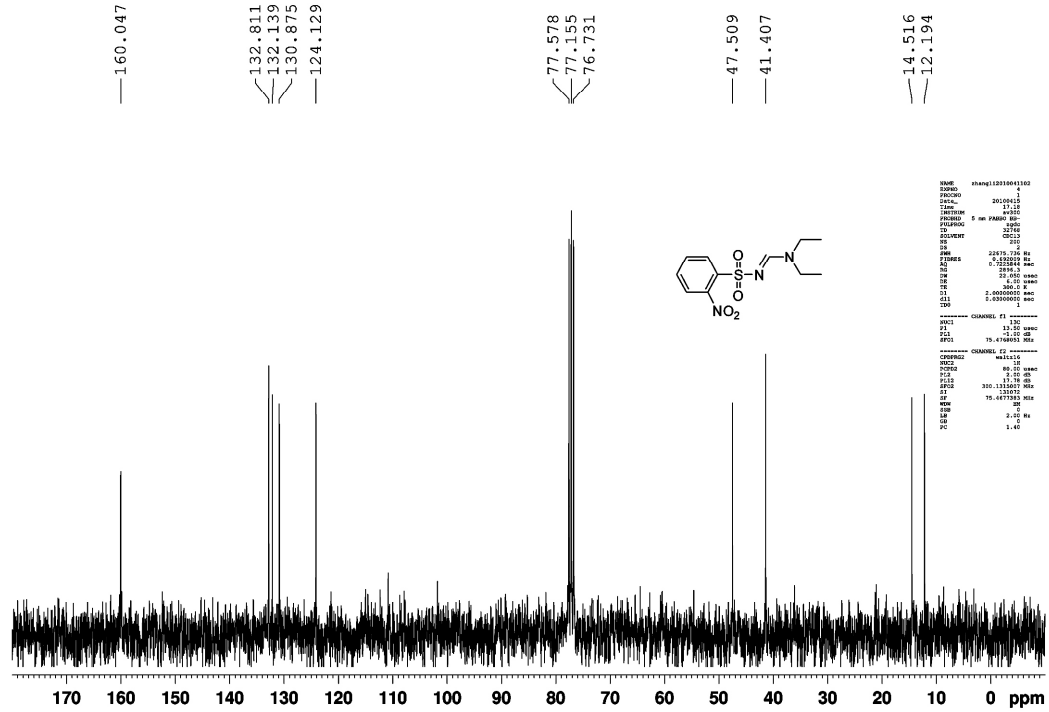
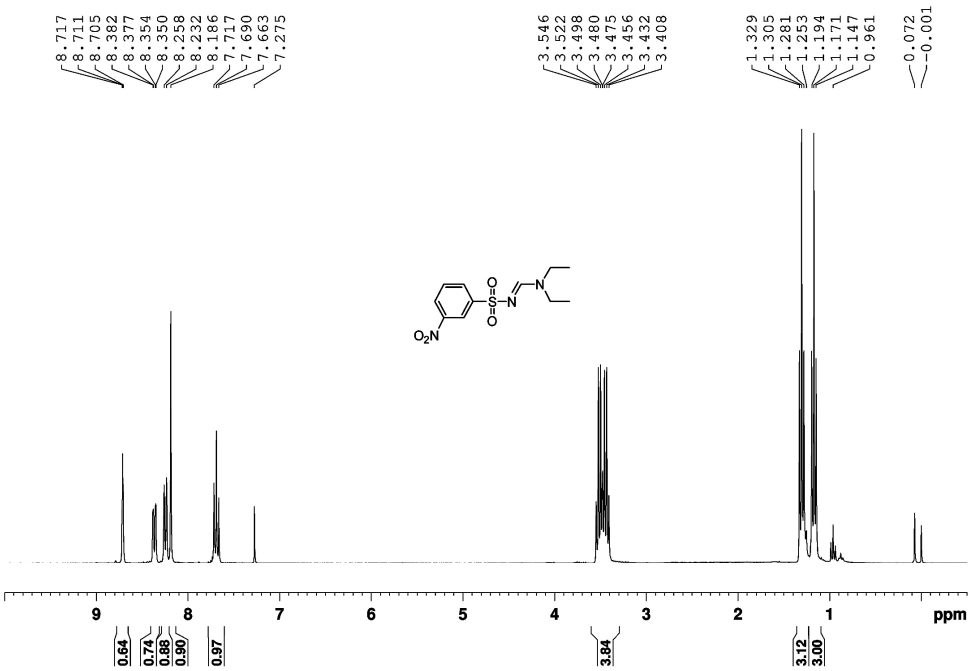
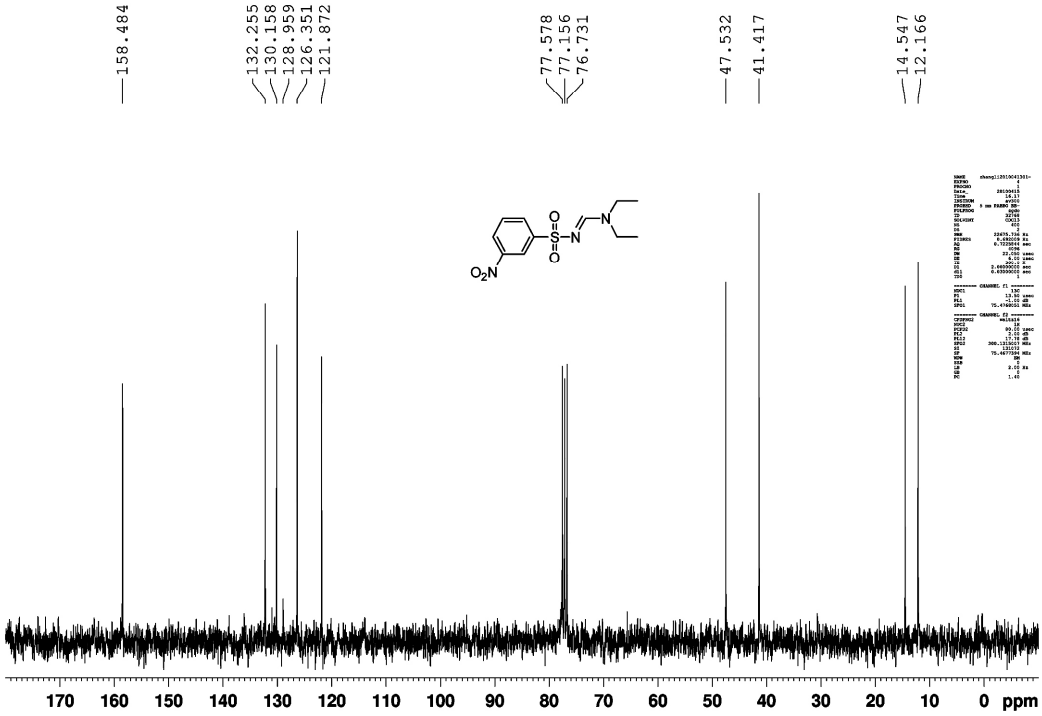
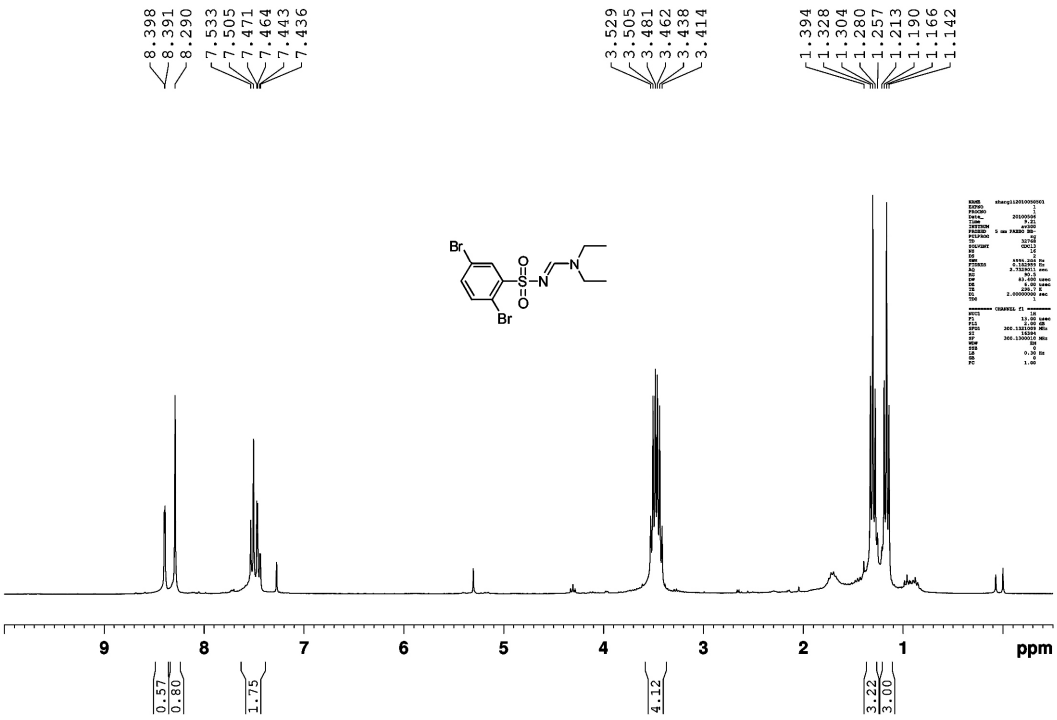
127.817
124.143
77.585
77.161
76.738
47.550
41.447
14.572
12.200
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm

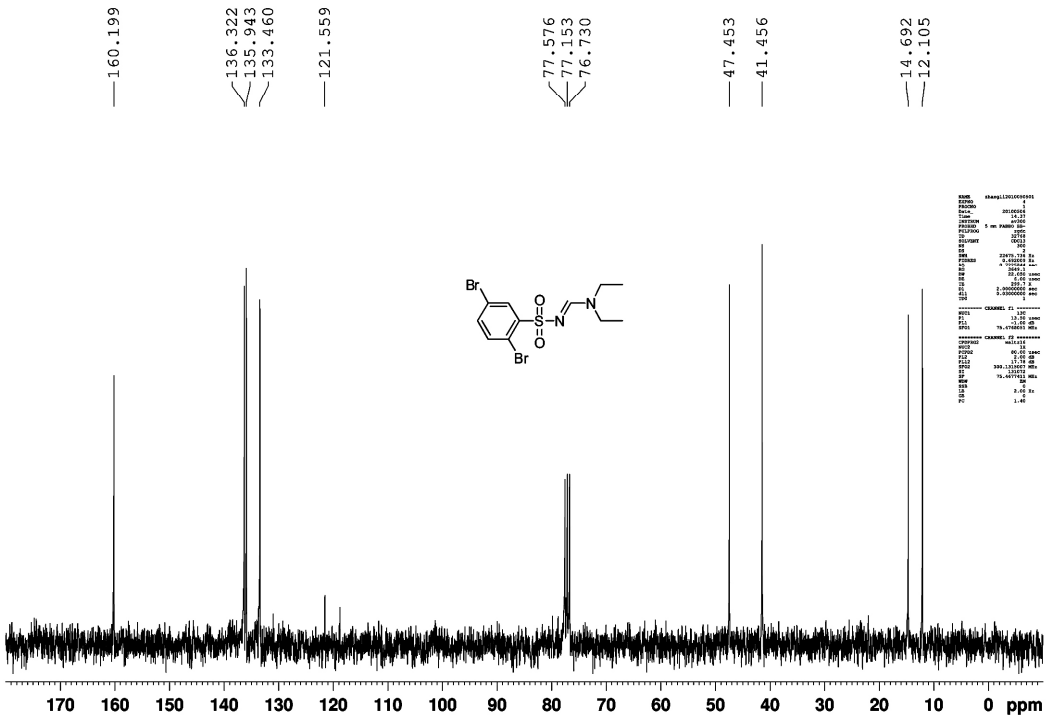

8.297
8.274
8.106
7.719
7.705
7.689
7.683
7.678
7.664
7.654
7.641
7.269
3.528
3.503
3.478
3.452
3.428
1.361
1.337
1.313
1.269
1.254
1.191
1.167
1.143
0.987
0.962
0.938
0.071
0.000
O
S
N
O
NO2
9
8
7
6
5
4
3
2
1
ppm
0.98
0.98
3.04
4.00
3.05
3.12

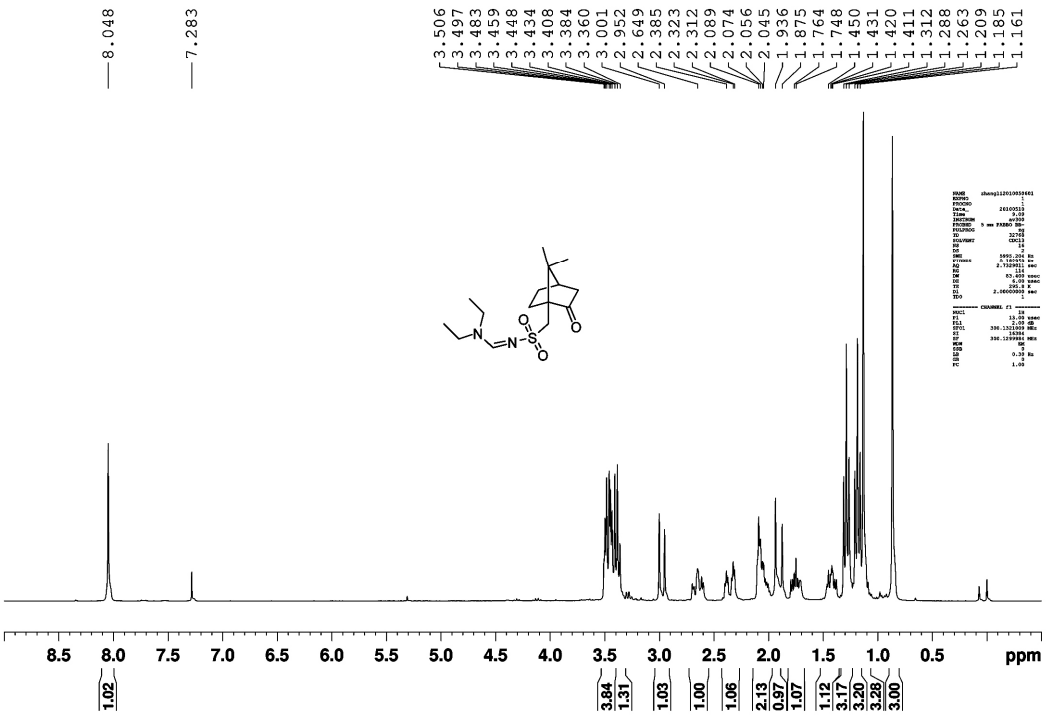

160.047
132.811
132.139
130.875
124.129
77.578
77.155
76.731
47.509
41.407
14.516
12.194
O
S
N
O
NO2
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm

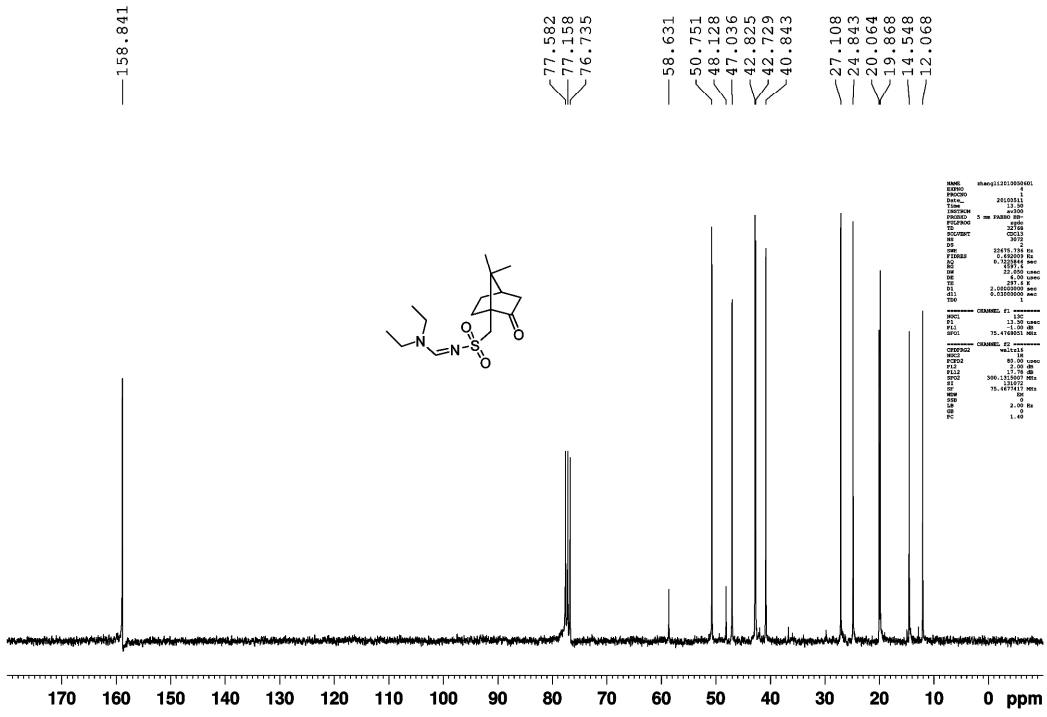

8.717
8.711
8.705
8.382
8.377
8.354
8.350
8.258
8.232
8.186
7.717
7.690
7.663
7.275
3.546
3.522
3.498
3.480
3.475
3.456
3.432
3.408
1.329
1.305
1.281
1.253
1.194
1.171
1.147
0.961
0.072
-0.001
O2N
9
8
7
6
5
4
3
2
1
ppm
0.64
0.74
0.88
0.90
0.97
3.84
3.12
3.00

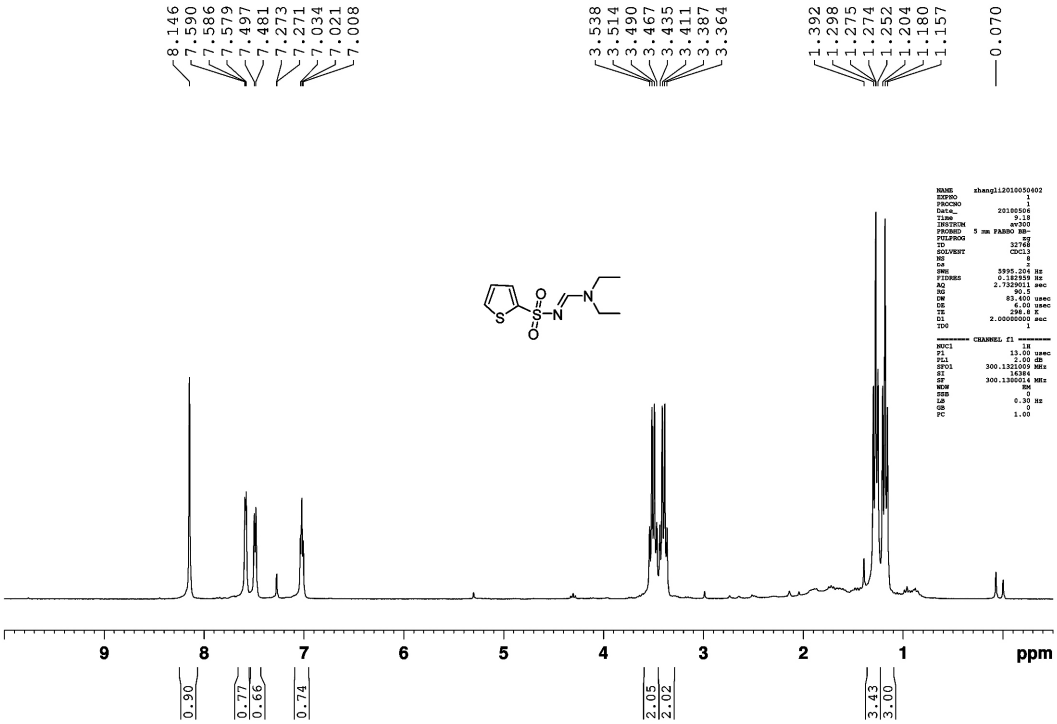

158.484
132.255
130.158
128.959
126.351
121.872
77.578
77.156
76.731
47.532
41.417
14.547
12.166
O2N
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm

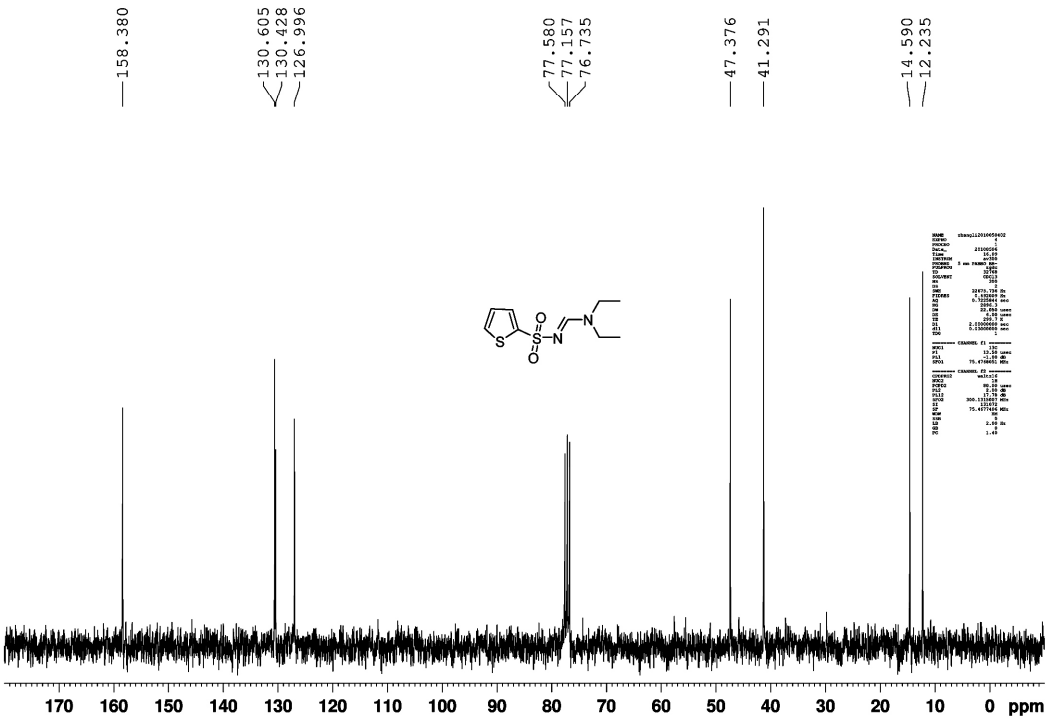

8.398
8.391
8.290
7.533
7.505
7.471
7.464
7.443
7.436
3.529
3.505
3.481
3.462
3.438
3.414
1.394
1.328
1.304
1.280
1.257
1.213
1.190
1.166
1.142
Br
O
S
N
O
Br
9
8
7
6
5
4
3
2
1
ppm
0.57
0.80
1.75
4.12
3.22
3.00

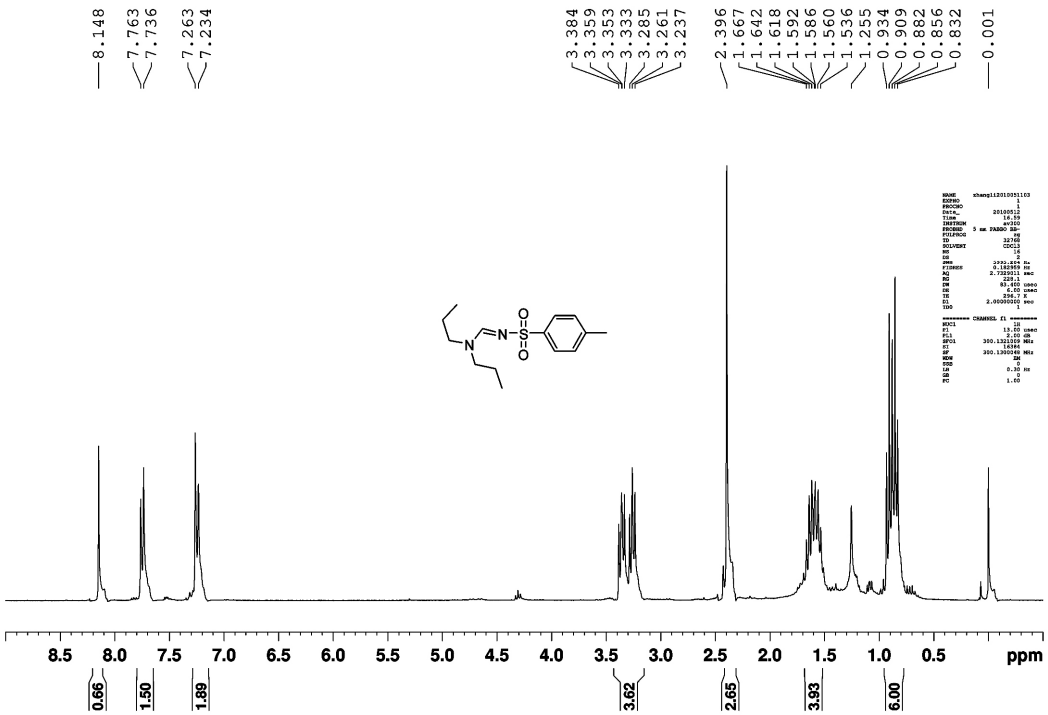

160.199
136.322
135.943
133.460
121.559
77.576
77.153
76.730
47.453
41.456
14.692
12.105
Br
O
S
N
O
Br
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm

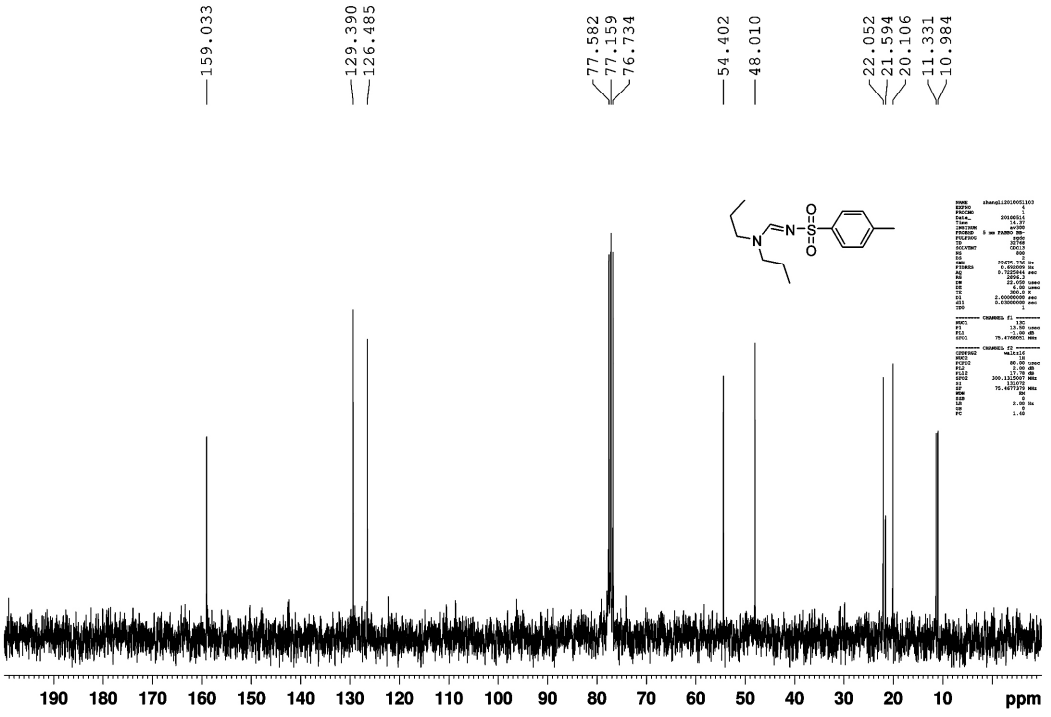

8.048
7.283
3.506
3.497
3.483
3.459
3.448
3.434
3.408
3.384
3.360
3.001
2.952
2.649
2.385
2.323
2.312
2.089
2.074
2.056
2.045
1.936
1.875
1.764
1.748
1.450
1.431
1.420
1.411
1.312
1.288
1.263
1.209
1.185
1.161
8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 ppm
1.02
3.84
1.31
1.03
1.00
1.06
2.13
0.97
1.07
1.12
3.17
3.20
3.28
3.00


158.841
77.582
77.158
76.735
58.631
50.751
48.128
47.036
42.825
42.729
40.843
27.108
24.843
20.064
19.868
14.548
12.068
170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm


8.146
7.590
7.586
7.579
7.497
7.481
7.273
7.271
7.034
7.021
7.008
3.538
3.514
3.490
3.467
3.435
3.411
3.387
3.364
1.392
1.298
1.275
1.274
1.252
1.204
1.180
1.157
0.070
9
8
7
6
5
4
3
2
1
ppm
0.90
0.77
0.66
0.74
2.05
2.02
3.43
3.00


158.380
130.605
130.428
126.996
77.580
77.157
76.735
47.376
41.291
14.590
12.235
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm


8.148
7.763
7.736
7.263
7.234
3.384
3.359
3.353
3.333
3.285
3.261
3.237
2.396
1.667
1.642
1.618
1.592
1.586
1.560
1.536
1.255
0.934
0.909
0.882
0.856
0.832
0.001
8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 ppm
0.66
1.50
1.89
3.62
2.65
3.93
6.00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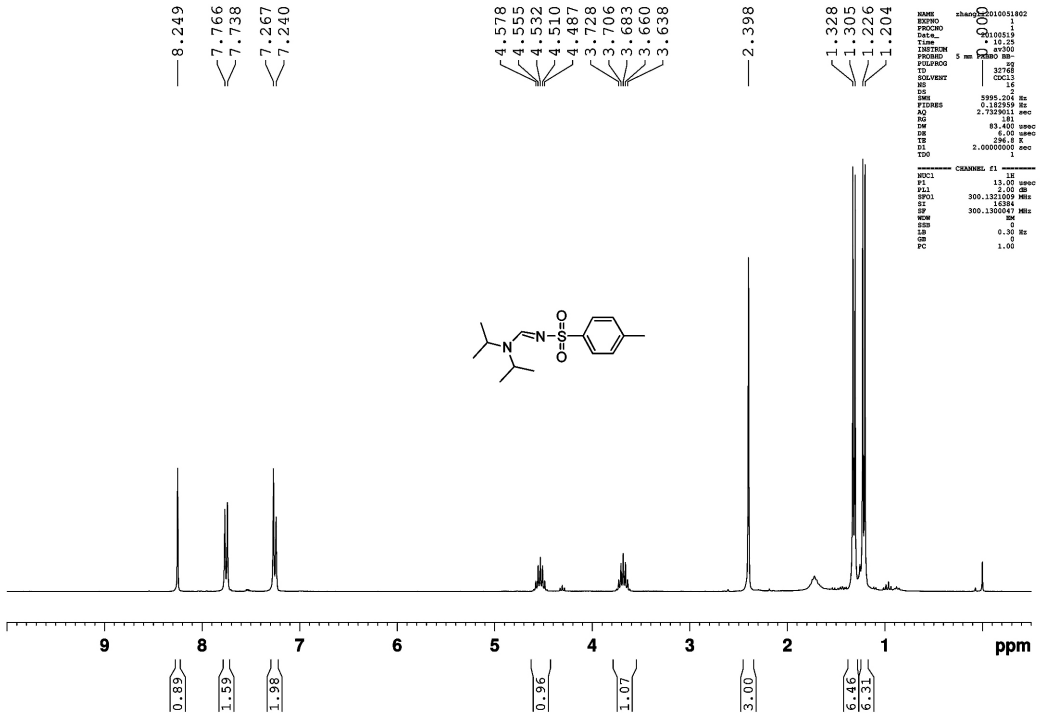
129.390
126.485
77.582
77.159
76.734
54.402
48.010
22.052
21.594
20.106
11.331
10.984
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm


8.249
7.766
7.738
7.267
7.240
4.578
4.555
4.532
4.510
4.487
3.728
3.706
3.683
3.660
3.638
2.398
1.328
1.305
1.226
1.204
9
8
7
6
5
4
3
2
1
ppm
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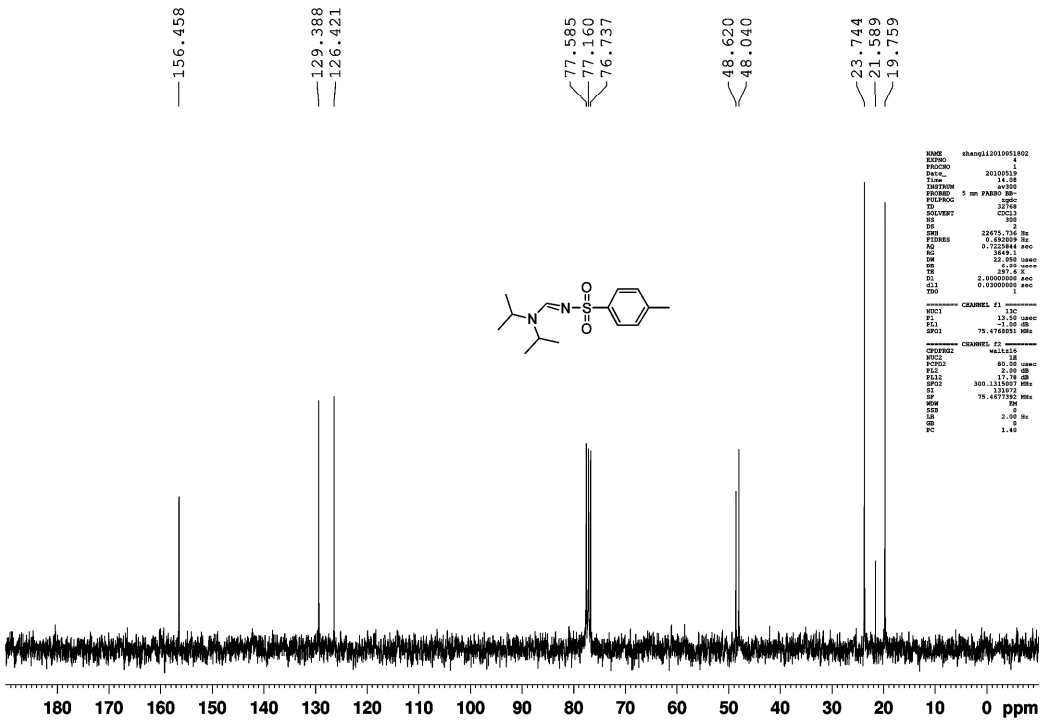
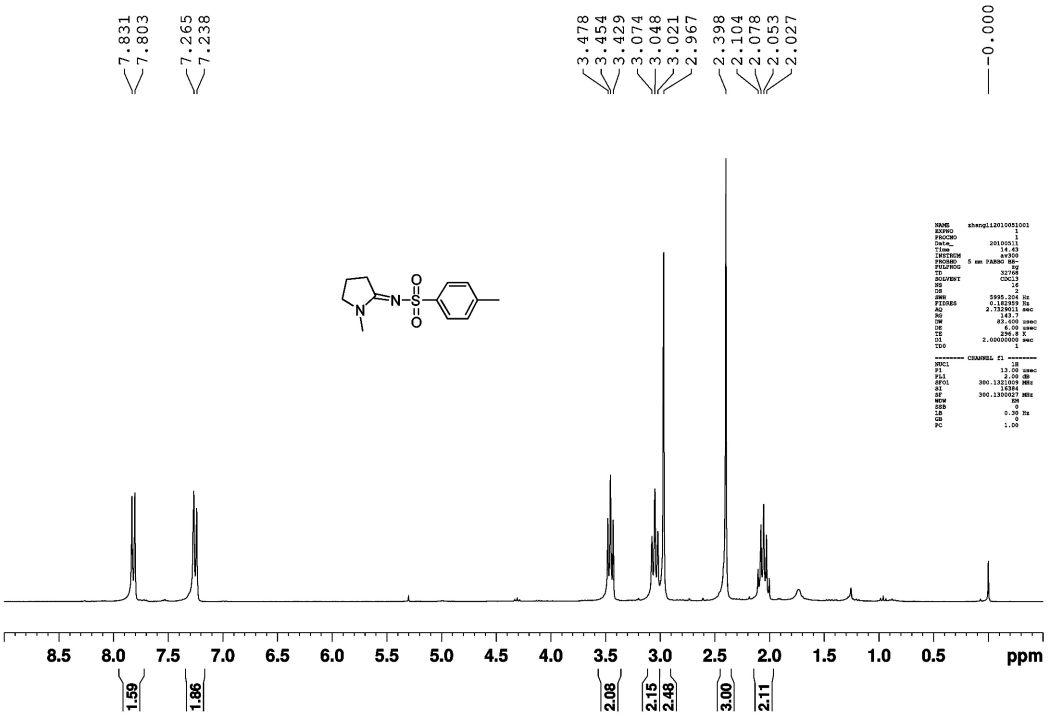
1.98
0.96
1.07
3.00
6.46
6.31


156.458
129.388
126.421
77.585
77.160
76.737
48.620
48.040
23.744
21.589
19.759
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm


7.831
7.803
7.265
7.238
3.478
3.454
3.429
3.074
3.048
3.021
2.967
2.398
2.104
2.078
2.053
2.027
0.000
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm
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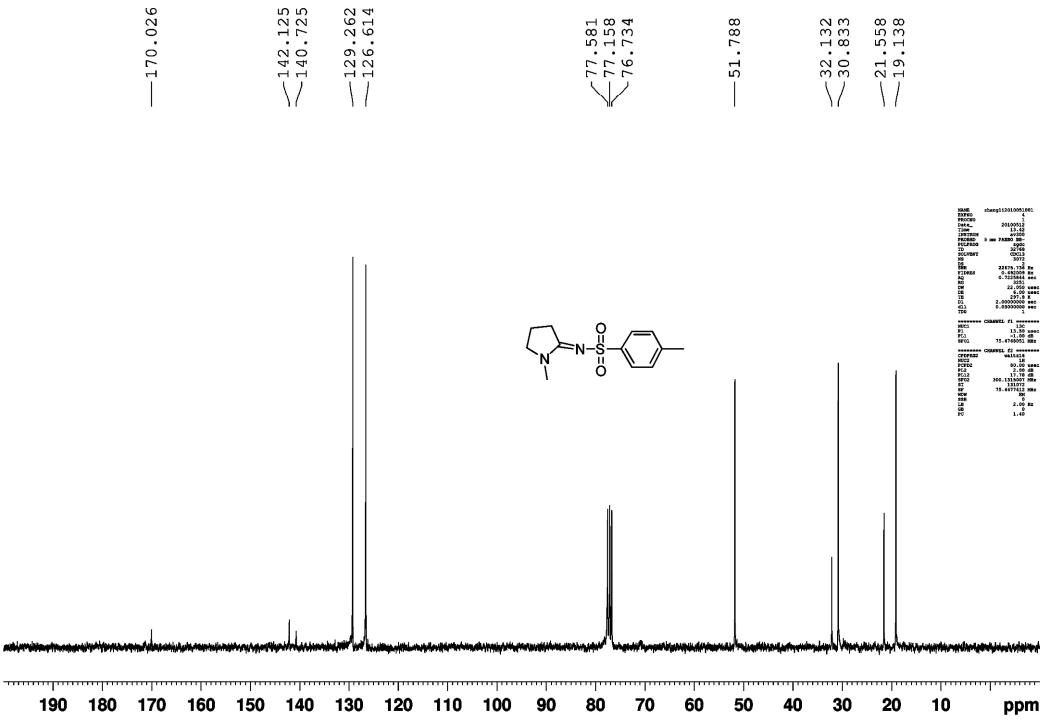
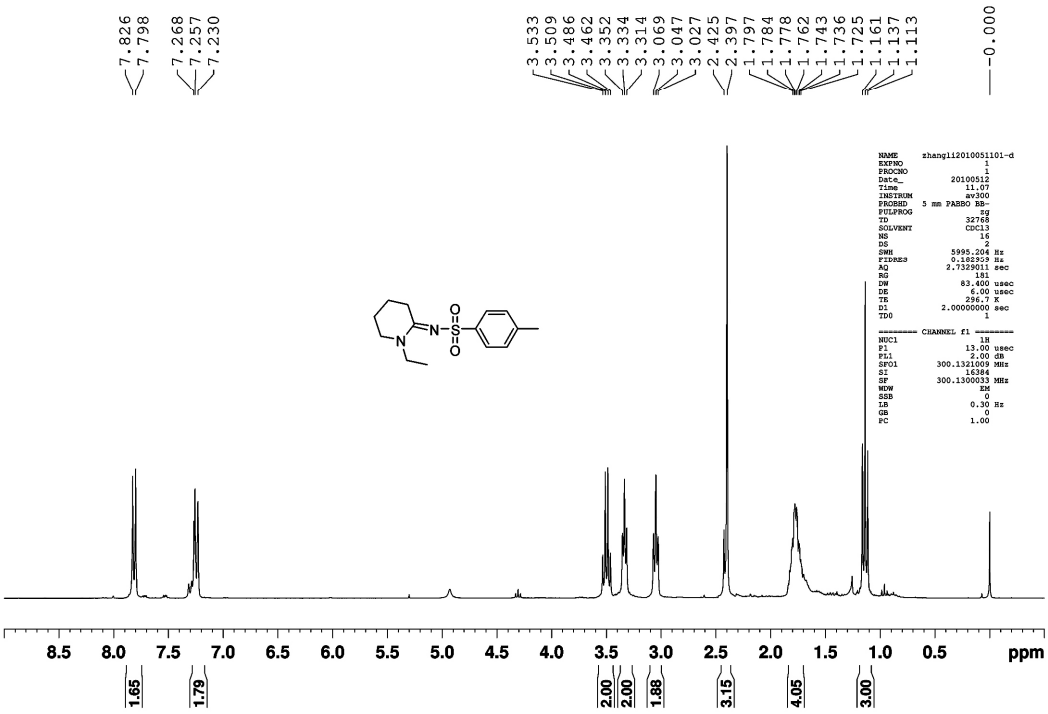
1.86
2.08
2.15
2.48
3.00
2.11


170.026
142.125
140.725
129.262
126.614
77.581
77.158
76.734
51.788
32.132
30.833
21.558
19.138
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm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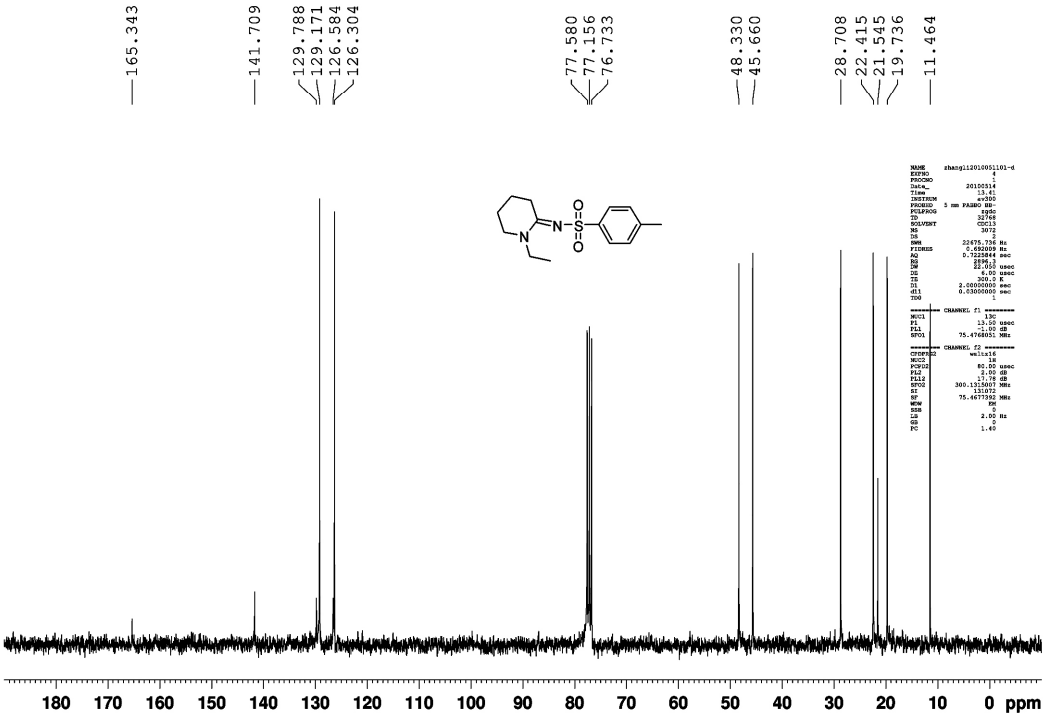
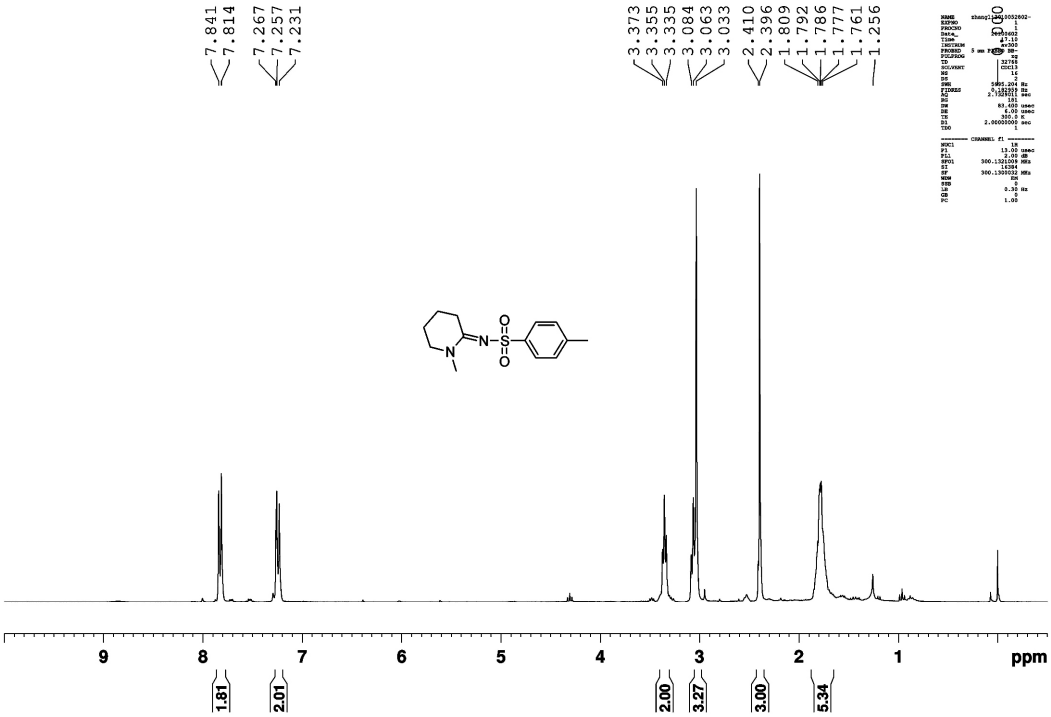
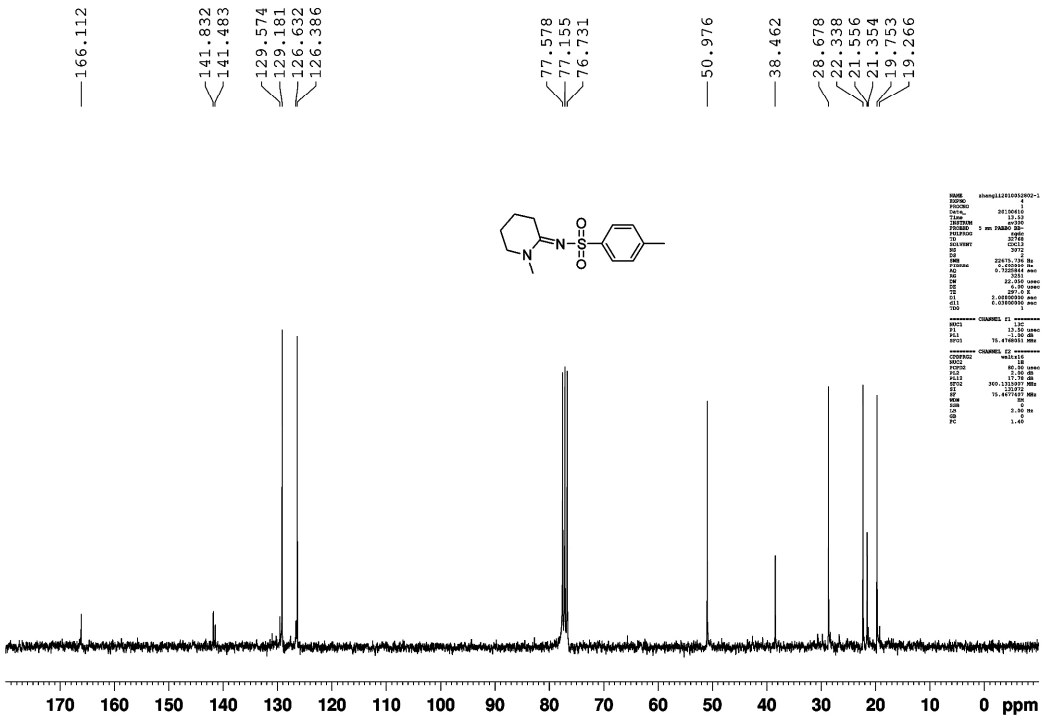
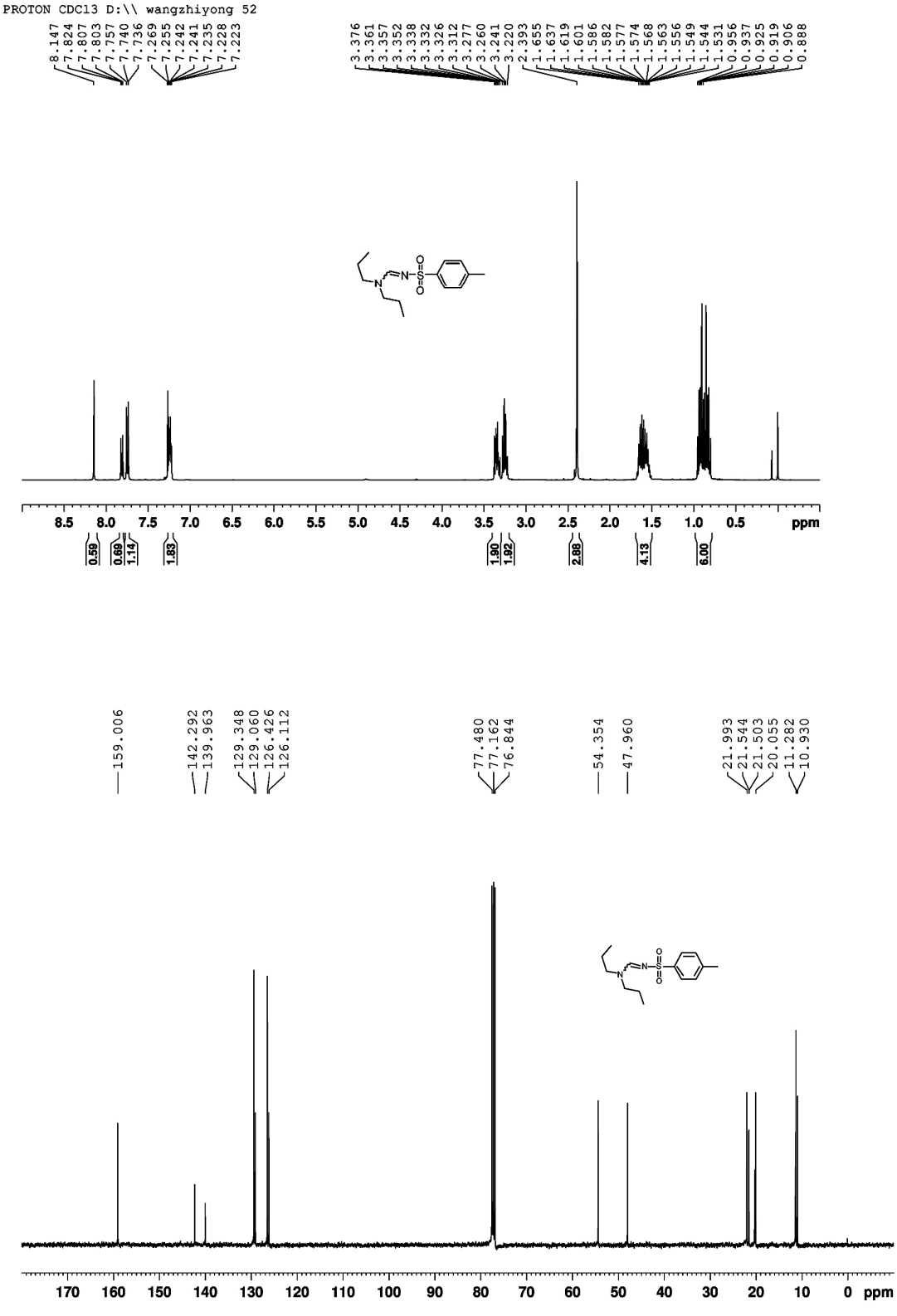
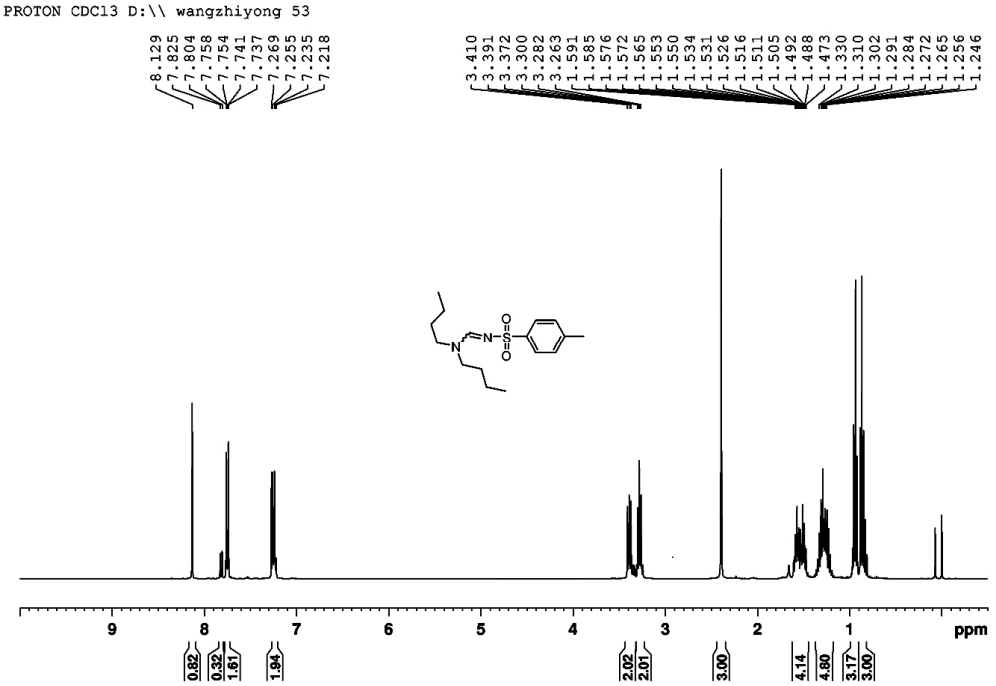
7.798
7.268
7.257
7.230
3.533
3.509
3.486
3.462
3.352
3.334
3.314
3.069
3.047
3.027
2.425
2.397
1.797
1.784
1.778
1.762
1.743
1.736
1.725
1.161
1.137
1.113
0.000
1.65
1.79
2.00
2.00
1.88
3.15
4.05
3.00
ppm


165.343
141.709
129.788
129.171
126.584
126.304
77.580
77.156
76.733
48.330
45.660
28.708
22.415
21.545
19.736
11.464
ppm


7.841
7.814
7.267
7.257
7.231
3.373
3.355
3.335
3.084
3.063
3.033
2.410
2.396
1.809
1.792
1.786
1.777
1.761
1.256
9
8
7
6
5
4
3
2
1
ppm
1.81
2.01
2.00
3.27
3.00
5.34


166.112
141.832
141.483
129.574
129.181
126.632
126.386
77.578
77.155
76.731
50.976
38.462
28.678
22.338
21.556
21.354
19.753
19.266
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm


PROTON CDCl3 D:\\ wangzhiyong 52
8.147
7.824
7.807
7.803
7.757
7.740
7.736
7.269
7.255
7.242
7.241
7.235
7.228
7.223
3.376
3.361
3.357
3.352
3.338
3.332
3.326
3.312
3.277
3.260
3.241
3.220
2.393
1.655
1.637
1.619
1.601
1.586
1.582
1.577
1.574
1.568
1.563
1.556
1.549
1.544
1.531
0.956
0.937
0.925
0.919
0.906
0.888
ppm
0.59
0.69
1.14
1.83
1.90
1.92
2.88
4.13
6.00
159.006
142.292
139.963
129.348
129.060
126.426
126.112
77.480
77.162
76.844
54.354
47.960
21.993
21.544
21.503
20.055
11.282
10.930
ppm


PROTON CDCl3 D:\\ wangzhiyong 53
8.129
7.825
7.804
7.758
7.754
7.741
7.737
7.269
7.255
7.235
7.218
3.410
3.391
3.372
3.300
3.282
3.263
1.591
1.585
1.576
1.572
1.565
1.553
1.550
1.534
1.531
1.526
1.516
1.511
1.505
1.492
1.488
1.473
1.330
1.310
1.302
1.291
1.284
1.272
1.265
1.256
1.246
9
8
7
6
5
4
3
2
1
ppm
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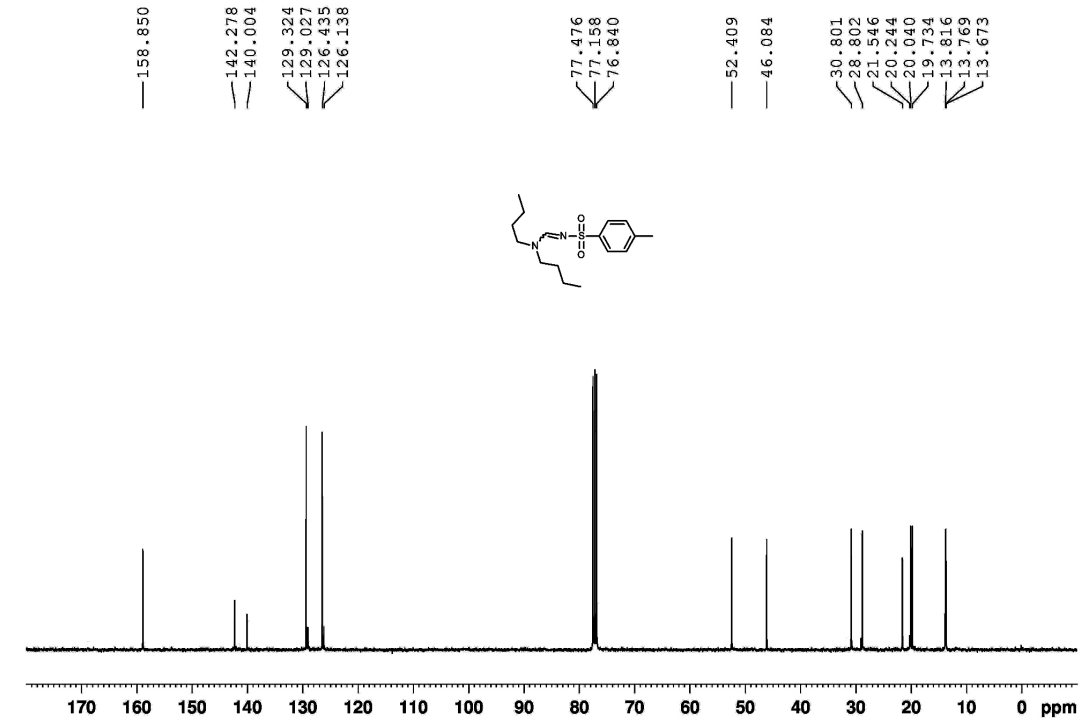
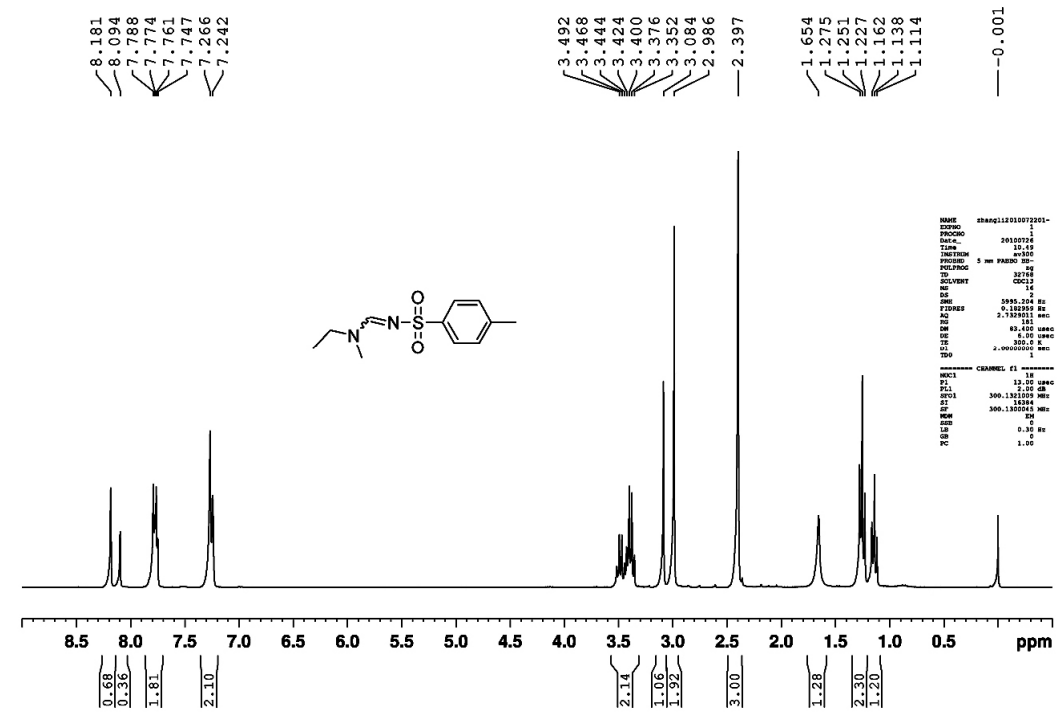
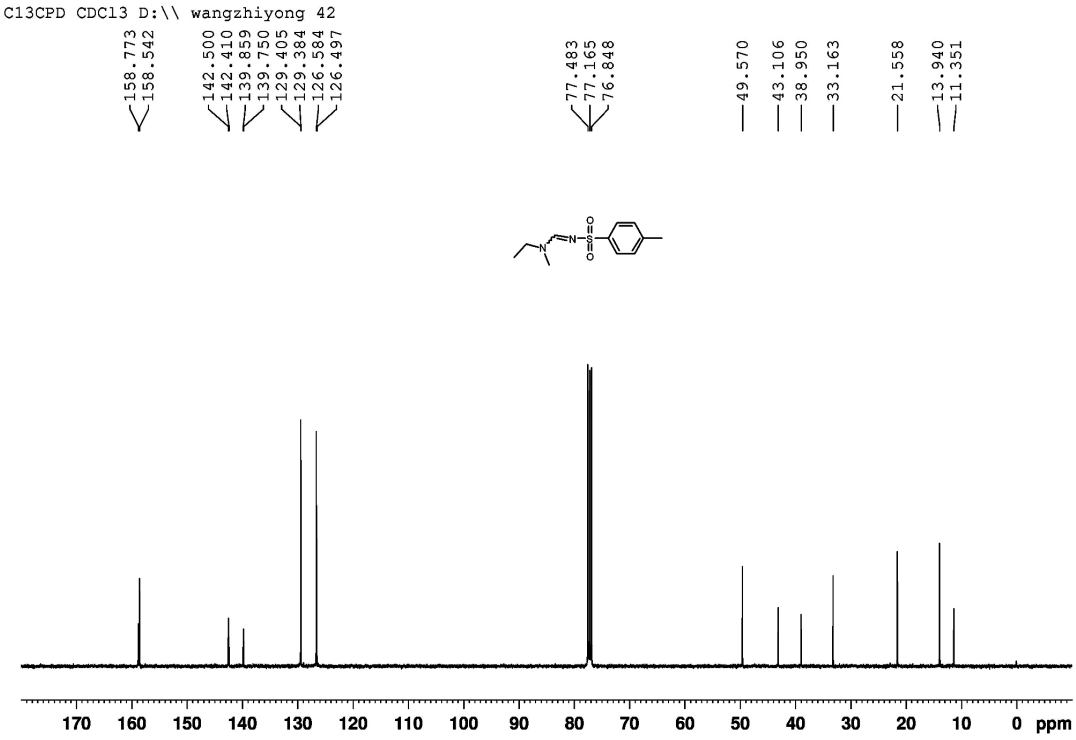
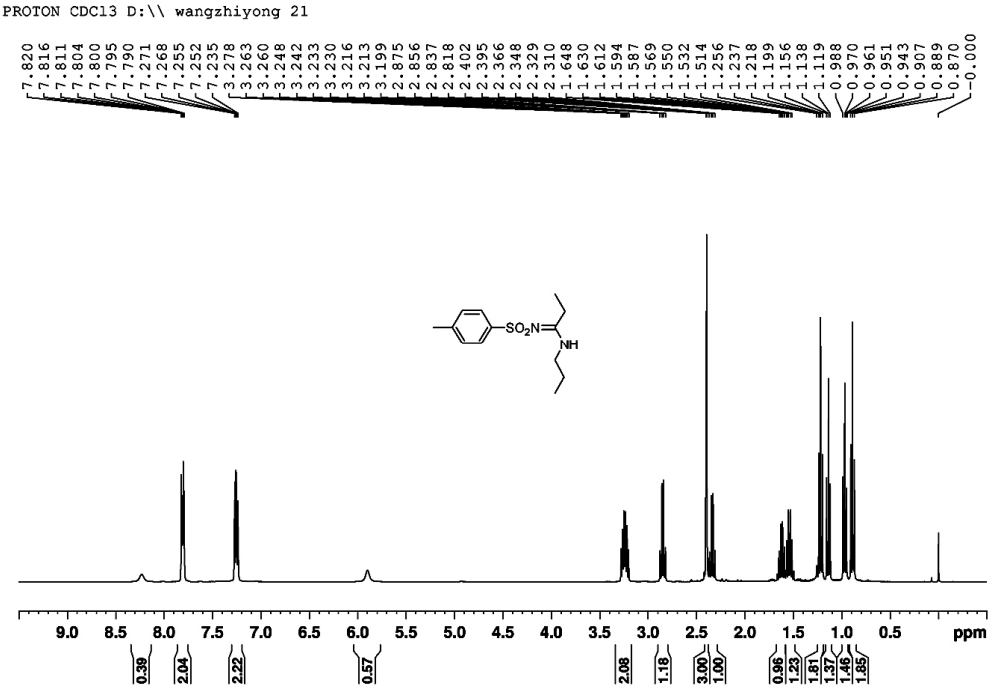
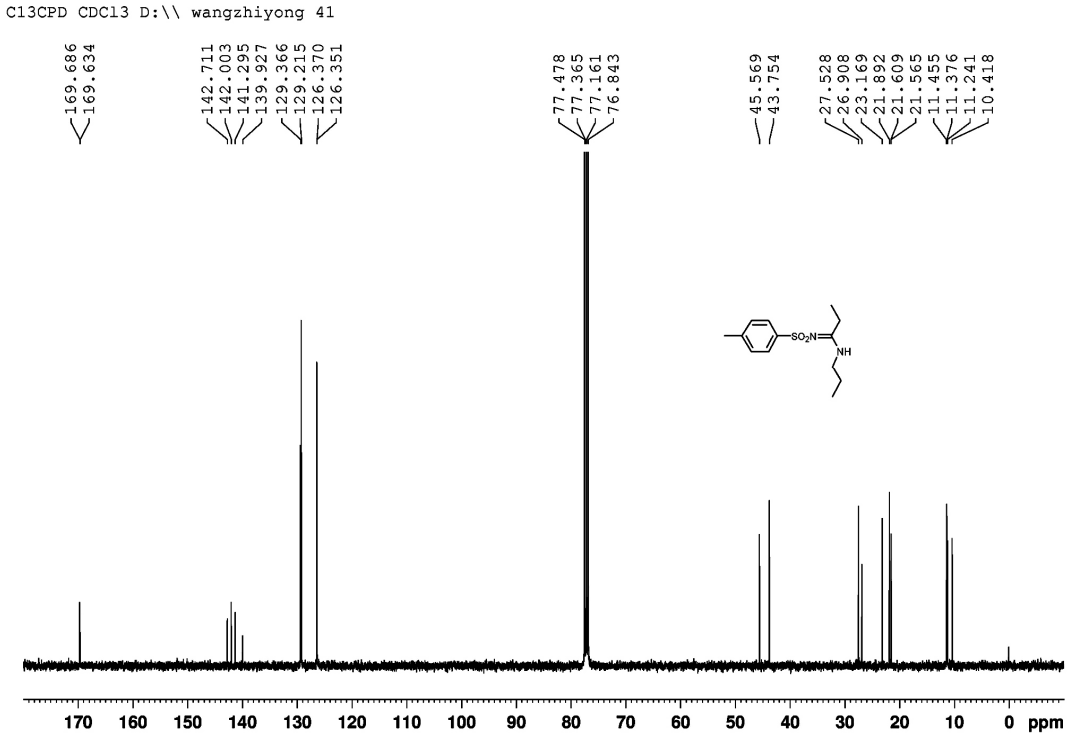
0.32
1.61
1.94
2.02
2.01
3.00
4.14
4.80
3.17
3.00

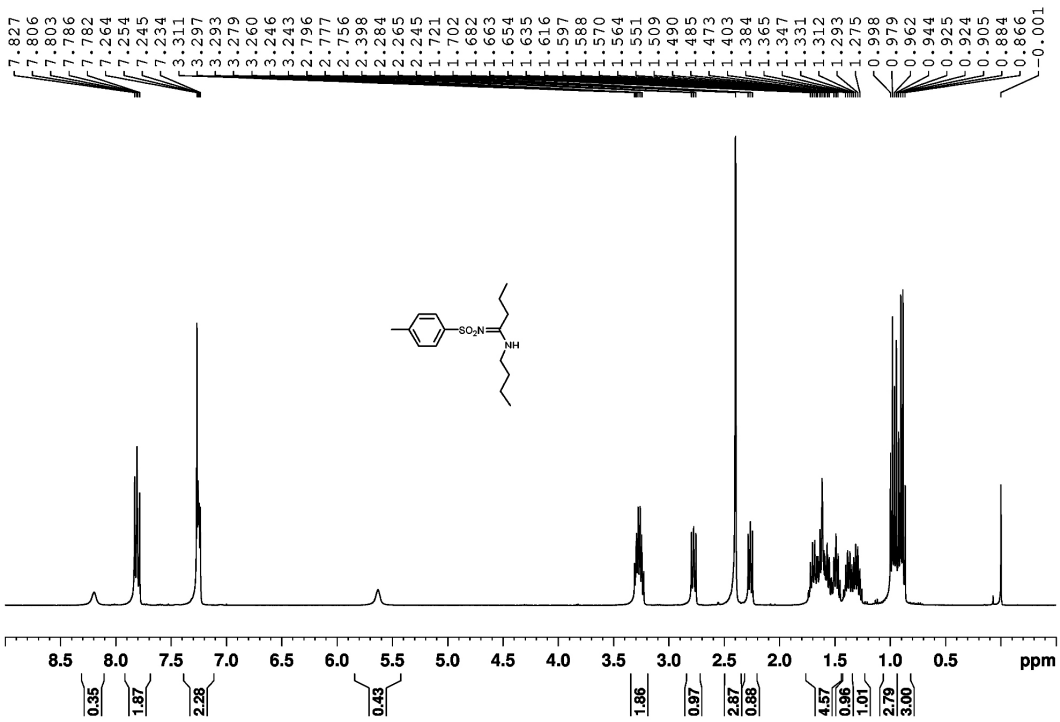

158.850
142.278
140.004
129.324
129.027
126.435
126.138
77.476
77.158
76.840
52.409
46.084
30.801
28.802
21.546
20.244
20.040
19.734
13.816
13.769
13.673
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm

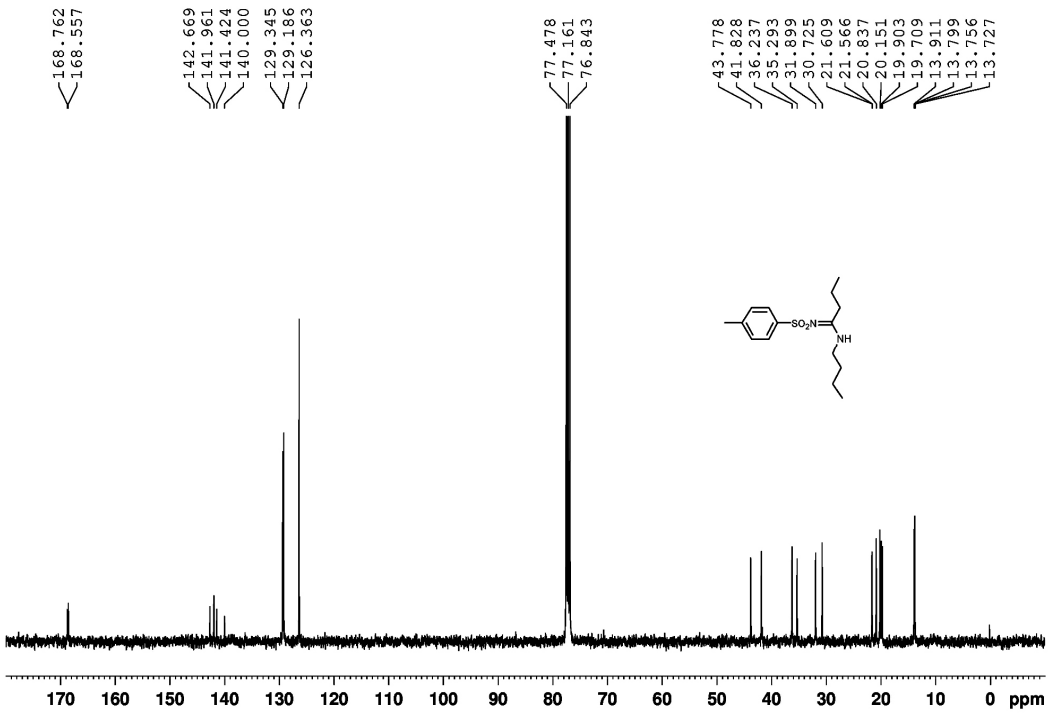

8.181
8.094
7.788
7.774
7.761
7.747
7.266
7.242
3.492
3.468
3.444
3.424
3.400
3.376
3.352
3.084
2.986
2.397
1.654
1.275
1.251
1.227
1.162
1.138
1.114
-0.001
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm
0.68
0.36
1.81
2.10
2.14
1.06
1.92
3.00
1.28
2.30
1.20


C13CPD CDCl3 D:\\ wangzhiyong 42
158.773
158.542
142.500
142.410
139.859
139.750
129.405
129.384
126.584
126.497
77.483
77.165
76.848
49.570
43.106
38.950
33.163
21.558
13.940
11.351
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm

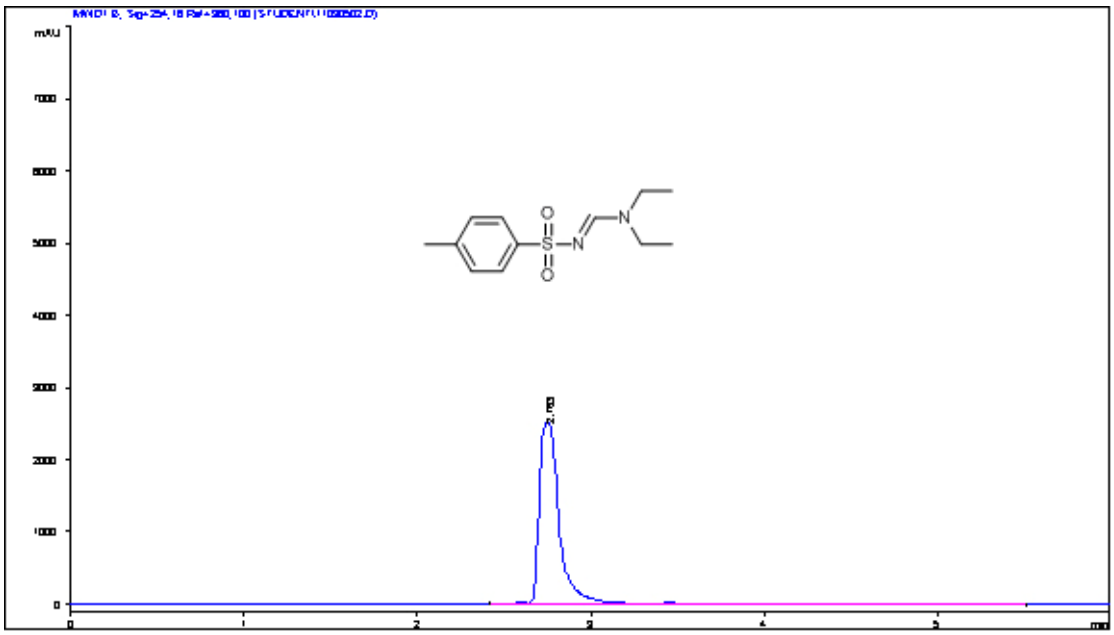

PROTON CDCl3 D:\\ wangzhiyong 21
7.820
7.816
7.811
7.804
7.800
7.795
7.790
7.271
7.268
7.255
7.252
7.235
3.278
3.263
3.260
3.248
3.242
3.233
3.230
3.216
3.213
3.199
2.875
2.856
2.837
2.818
2.402
2.395
2.366
2.348
2.329
2.310
1.648
1.630
1.612
1.594
1.587
1.569
1.550
1.532
1.514
1.256
1.237
1.218
1.199
1.156
1.138
1.119
0.988
0.970
0.961
0.951
0.943
0.907
0.889
0.870
-0.000
SO2N
NH
9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 ppm
0.39
2.04
2.22
0.57
2.08
1.18
3.00
1.00
0.96
1.23
1.61
1.37
1.46
1.85

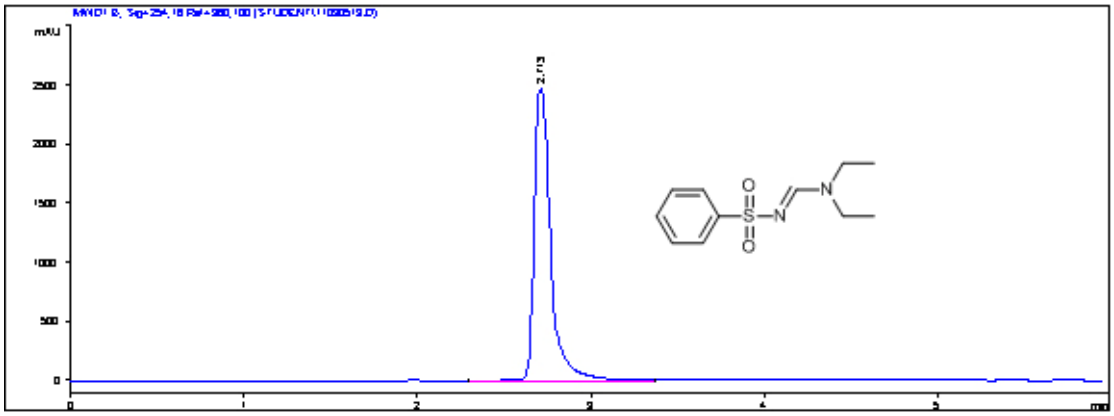

C13CPD CDCl3 D:\\ wangzhiyong 41
169.686
169.634
142.711
142.003
141.295
139.927
129.366
129.215
126.370
126.351
77.478
77.365
77.161
76.843
45.569
43.754
27.528
26.908
23.169
21.892
21.609
21.565
11.455
11.376
11.241
10.418
SO2N
NH
170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

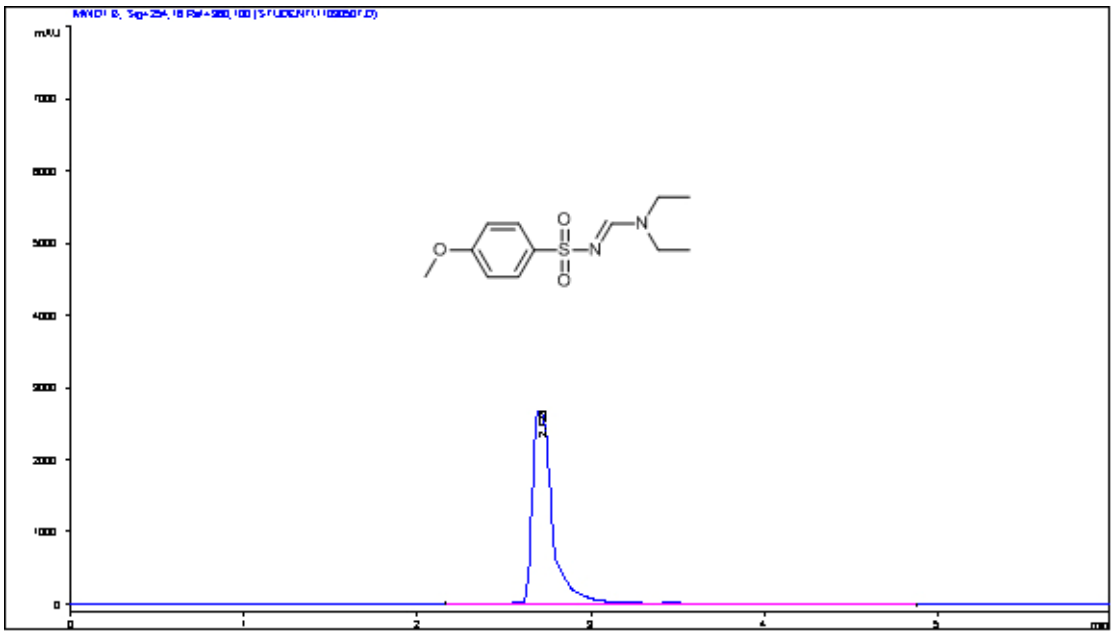

7.827
7.806
7.803
7.786
7.782
7.264
7.254
7.245
7.234
3.311
3.297
3.293
3.279
3.260
3.246
3.243
2.796
2.777
2.756
2.398
2.284
2.265
2.245
1.721
1.702
1.682
1.663
1.654
1.635
1.616
1.597
1.588
1.570
1.564
1.551
1.509
1.490
1.485
1.473
1.403
1.384
1.365
1.347
1.331
1.312
1.293
1.275
0.998
0.979
0.962
0.944
0.925
0.924
0.905
0.884
0.866
-0.001
SO2N
NH
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm
0.35
1.87
2.28
0.43
1.86
0.97
2.87
0.88
4.57
0.96
1.01
2.79
3.00

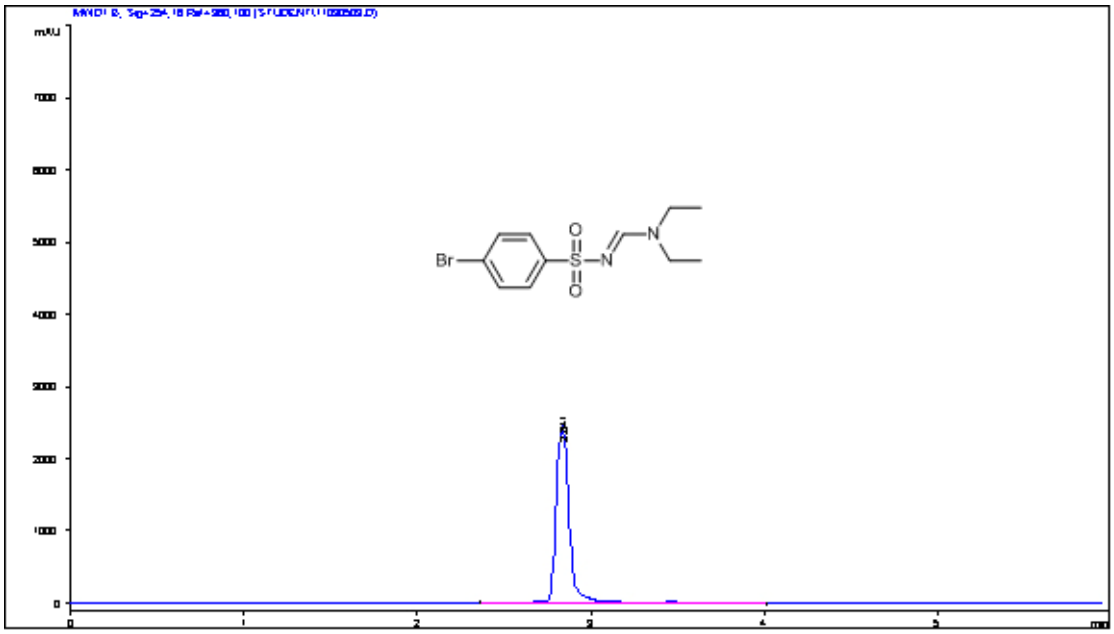

168.762
168.557
142.669
141.961
141.424
140.000
129.345
129.186
126.363
77.478
77.161
76.843
43.778
41.828
36.237
35.293
31.899
30.725
21.609
21.566
20.837
20.151
19.903
19.709
13.911
13.799
13.756
13.727
SO2N
NH
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm

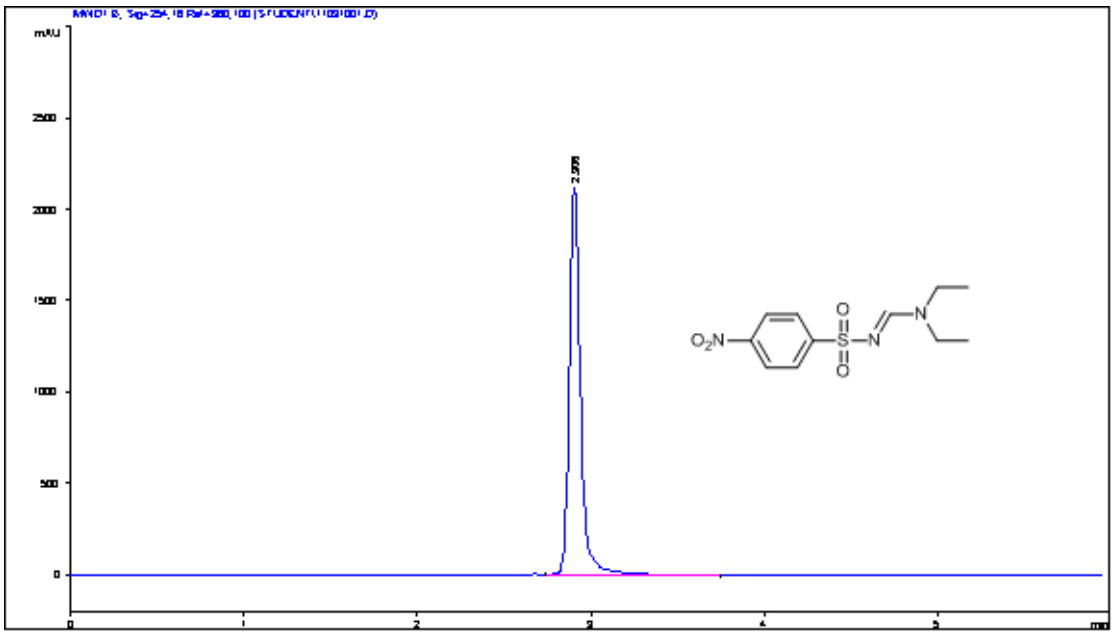
4. HPLC Spectra of all products.

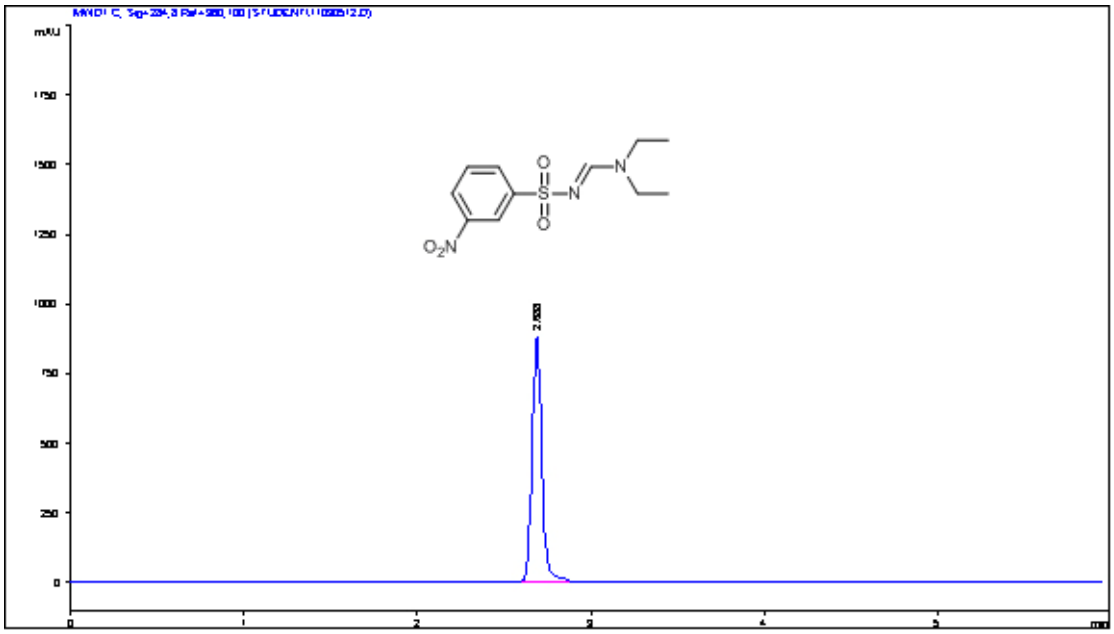


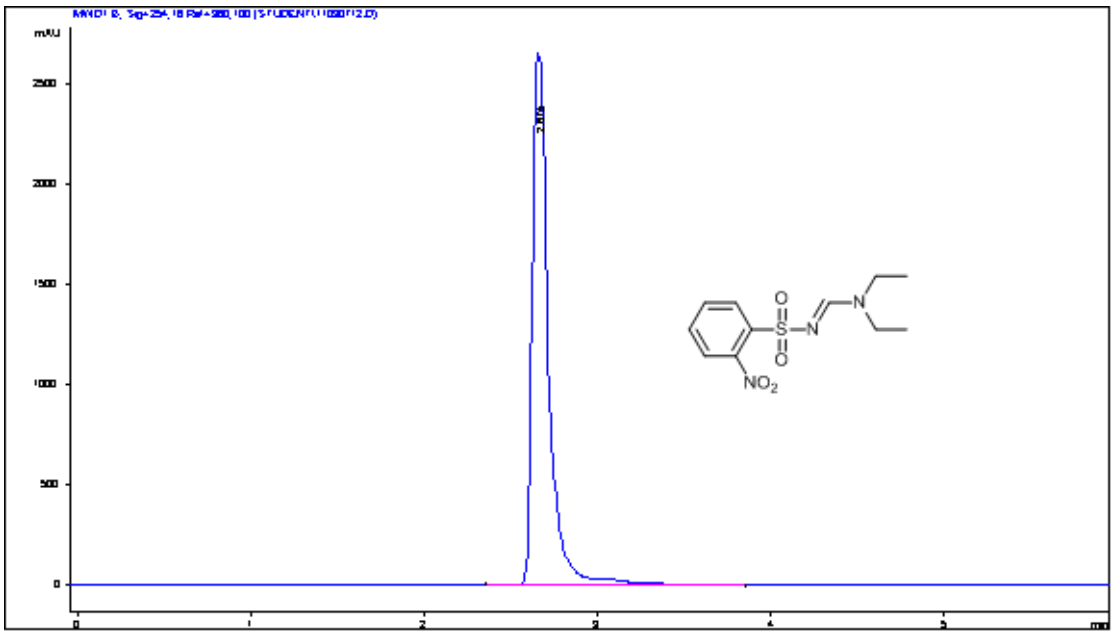


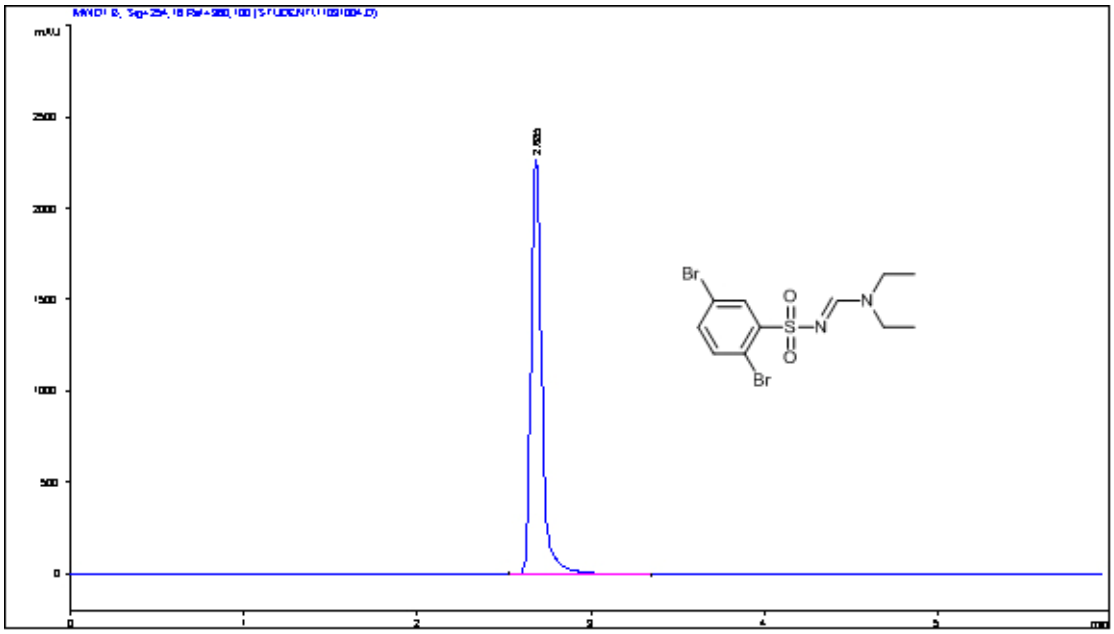

mAU
O
S
N
O

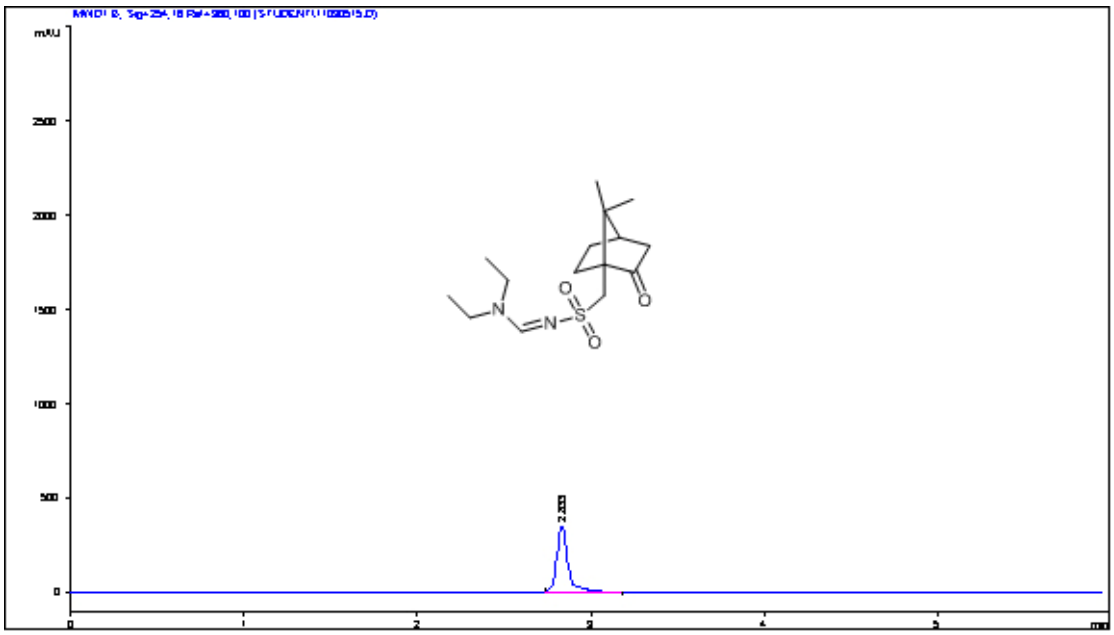

mAU
Br
O
S
N
O

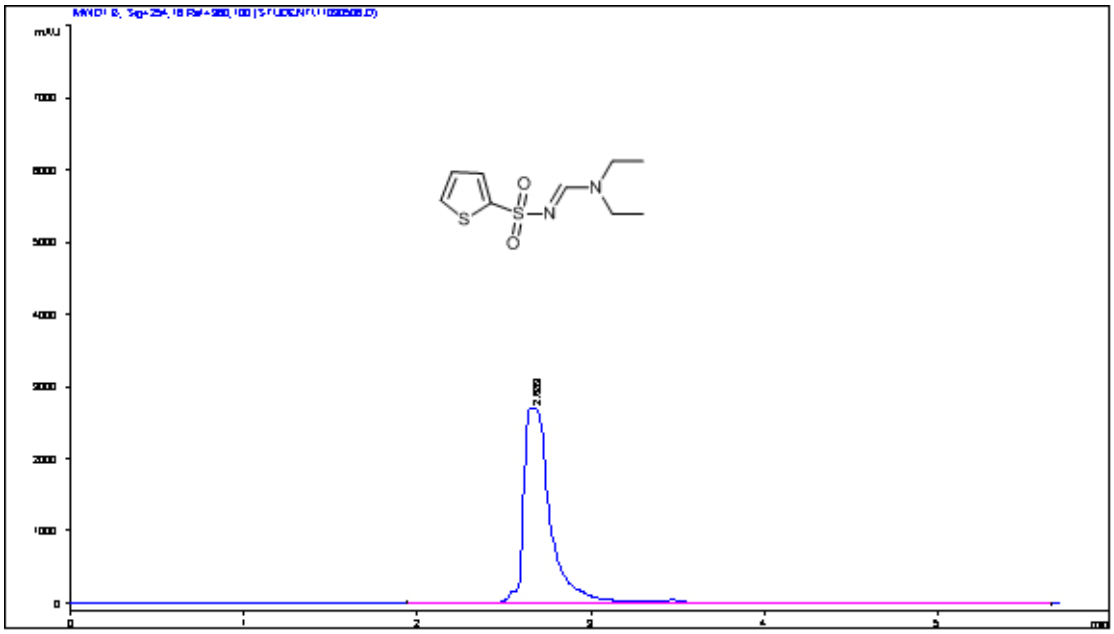

mAU
2500
2000
1500
1000
500
0
2.955
O_2N
S
O
N
N
0
1
2
3
4
5
min

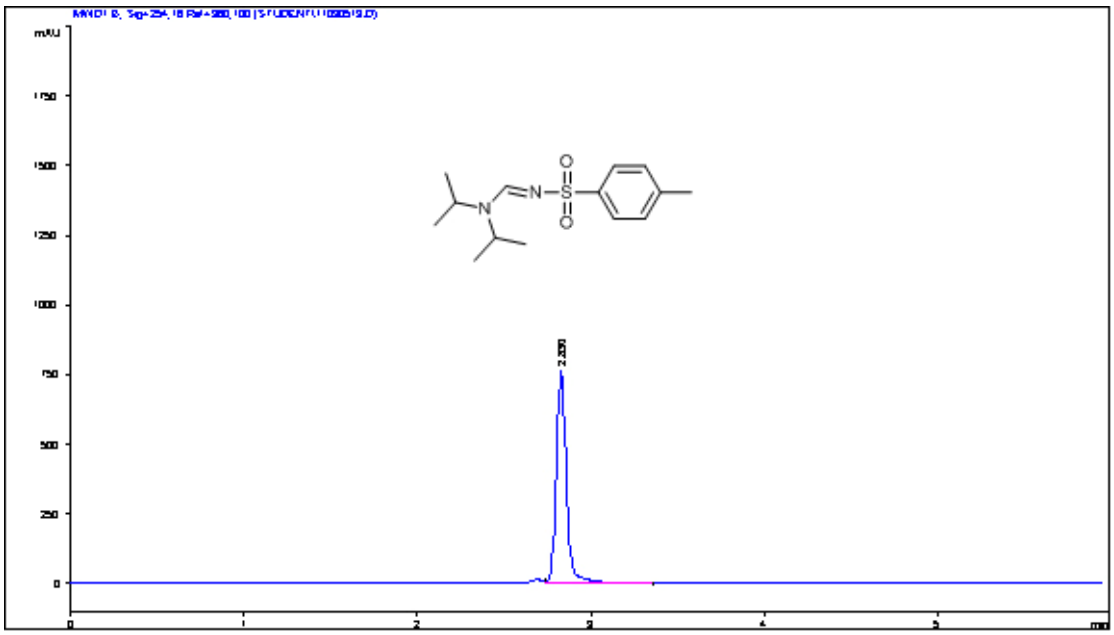

mAU
1750
1500
1250
1000
750
500
250
0
2.653
O_2N
S
O
N
N
0
1
2
3
4
5
min

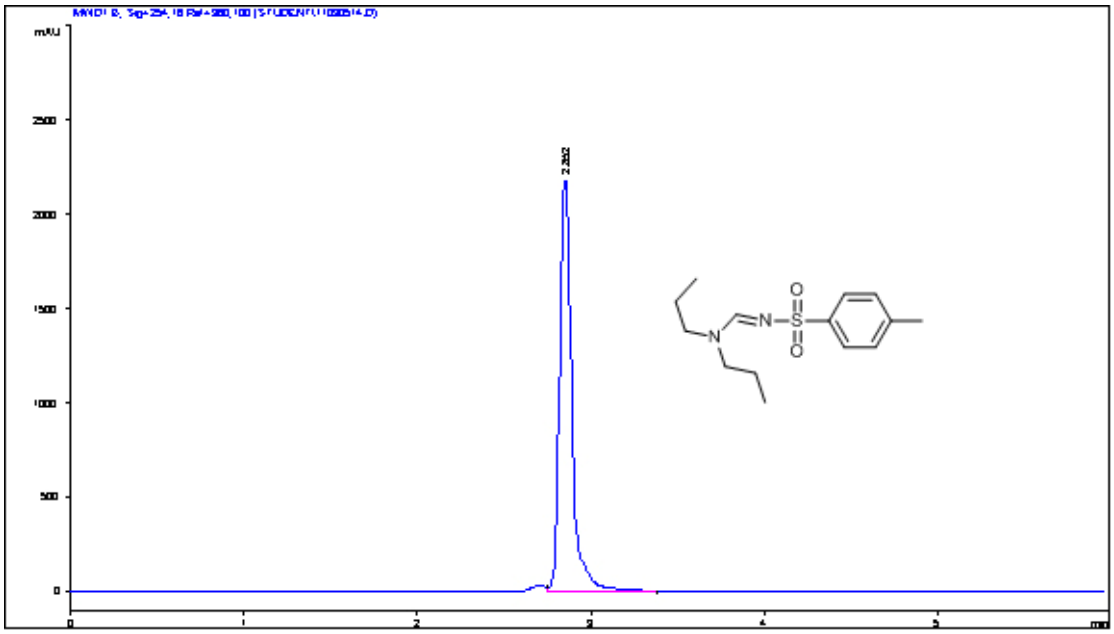

mAU
2500
2000
1500
1000
500
0
O
S
N
N
NO2


mAU
2500
2000
1500
1000
500
0
Br
Br
O
S
N
N


mAU
2500
2000
1500
1000
500
0
2.333
0
1
2
3
4
5
min


mAU
7000
6000
5000
4000
3000
2000
1000
0
2.409
0
1
2
3
4
5
min


mAU
2.359


mAU
2.352