Electronic Supplementary Material (ESI) for New Journal of Chemistry.

General information

Unless otherwise stated, all the reagents were purchased from commercial suppliers and used without further purification. All the aldehydes liquid under standard conditions were filtrated through silica gel pads prior to being introduced in reactions. DMF used in the experiments contained 0.3 mass % of water.

^{1}H, ^{13}C, and ^{19}F spectra were recorded in $CDCl_3$ on Bruker Avance 300, Bruker Avance 400, or Varian Inova 400 spectrometers. Chemical shifts are reported in parts per million relative to $CHCl_3$ (7.26 and 77.16 ppm for ^{1}H and ^{13}C respectively). The following abbreviations were used to designate chemical shift multiplicities: s = singlet, d = doublet, dd = doublet of doublets, t = triplet, q = quartet, hept - septet, m = multiplet.

High-resolution mass spectra (HRMS) were registered on a TripleTOF 5600+ AM Sciex mass spectrometer using electrospray ionization (ESI). The voltage on the capillary was 5500 V in positive ion registration mode, 4500 V in negative ion registration mode; the range of scanned masses, m/z 50-1000. The flux of the ion source gas was 15 arb, of the curtain gas – 25 arb. The sample was injected with an infusion pump.

GC-MS analyses were performed using a gas chromatograph (Chromatec) equipped with an AS-2M (3D) autosampler and a CR-5ms capillary column (30 m x 250 μm) and EI-mass detector. The instrument was set to an injection volume of 0.2 μL, an inlet split ratio of 75:1, and inlet and detector temperatures of 250 °C and 250 °C, respectively. Helium was used as a carrier gas in isobaric conditions (60.9 kPa).

Comparison between commercial dimethylamine solutions and DMF in terms of safety

According to Sigma's Safety Data Sheets, DMF has lower categories of hazard in flammability, acute toxicity and eye irritation. The table below provides the classification of these substances according to Regulation (EC) No 1272/2008, which assigns substances numeric categories from 1 to 4, with 1 meaning the highest danger and 4 - -the lowest.

Table S1. Classification of the substances according to Regulation (EC) No 1272/2008.[1]

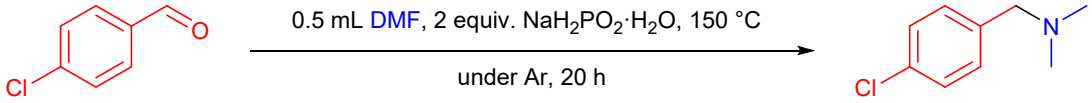
	DMF	Me_2NH/THF	Me_2NH/MeOH
Flammable liquids	3	2	2
Acute toxicity, Inhalation	4	-	3
Skin irritation	-	2	2
Eye damage	2	1	1

Optimization data

General protocol of a typical optimization experiment

The reactions were set up in 10 mL Schlenk tubes. The Schlenk tube was connected to the Schlenk line and flushed three times with argon. Then the tube was charged with $NaH_2PO_2 \cdot H_2O$, p-chlorobenzaldehyde, water (if stated), DMSO (if stated), sealed and heated for a time specified in the tables below. Then the tubes were cooled, opened and an aliquot of the internal standard (N,N-dimethylaniline) in DMF was added into the Schlenk tube. Then a portion of the reaction mixture was analyzed with GC-FID. The calibration curve (product concentration) - (Peak of product: Peak of standard relation) had been obtained using NMR pure product and N,N-dimethylaniline.

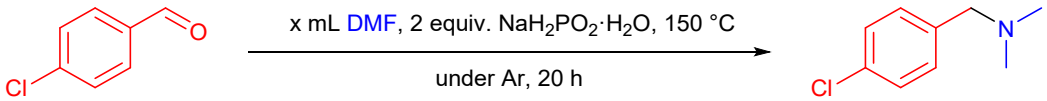
Table S2. Temperature optimization



Entry[a]	Temperature, °C	Yield, %
1	150	53
2	130	11
3	110	traces
4	90	traces
5	70	traces

[a]Reaction conditions: 0.285 mmol p-chlorobenzaldehyde, 0.594 mmol $NaH_2PO_2 \cdot H_2O$, 0.5 mL DMF.

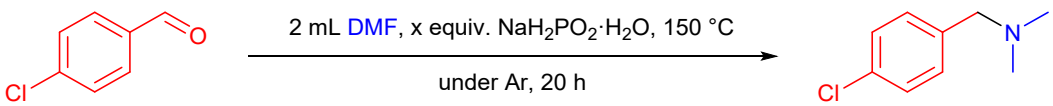
Table S3. Concentration optimization



Entry[a]	Solvent volume, mL	Concentration, M	Yield, %
1	0.25	1.13	33
2	0.5	0.58	53
3	1	0.29	55
4	2	0.14	62
5	5	0.057	62
6	10	0.029	69

[a]Reaction conditions: 0.285 mmol p-chlorobenzaldehyde, 0.594 mmol $NaH_2PO_2 \cdot H_2O$.

Table S4. Hypophosphite load optimization

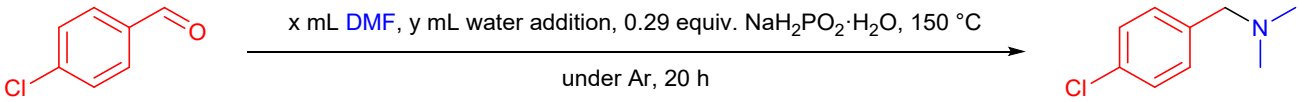


Entry[a]	Hypophosphite load, equiv.	Yield, %
1	0	16
2	0.13	45

3	0.26	91
4	0.53	75
5	1	64
6	2	62

[a]Reaction conditions: 0.285 mmol p-chlorobenzaldehyde, 2mL DMF

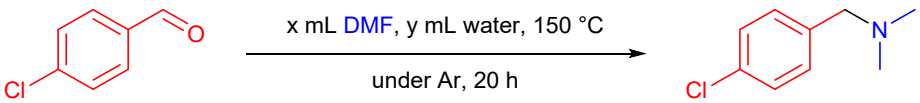
Table S5. Water quantity optimization



Entry[a]	DMF volume, mL	Water volume, µL	Water quantity, mmol[b]	Water quantity, equiv.	Yield, %
1	5	0	0.99	1.4	58
2	5	2	1.1	1.5	71
3	5	4	1.2	1.7	73
4	5	6	1.3	1.8	95
5	5	8	1.4	2.0	94
6	2	14	1.5	2.1	91

[a]Reaction conditions: 0.711 mmol p-chlorobenzaldehyde, 0.208 mmol $NaH_2PO_2·H_2O$. [b]The values provided in the column refer to the total quantities of water in the solvent, $NaH_2PO_2·H_2O$ and added to the reaction mixture.

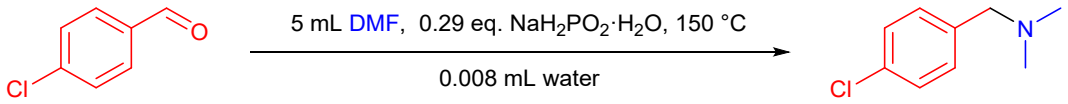
Table S6. Control experiments: reactions without hypophosphite



Entry[a]	DMF volume, mL	Water volume, mL	Water quantity, equiv.	Yield, %
1	0.5	0.014	1.2	24
2	1	0.014	1.3	23
3	2	0.014	1.5	11
4	5	0	1.1	5
5	5	0.008	1.7	8
6	2	0.012	1.4	18
7	2	0.017	1.8	11
8[b]	2	0.014	1.5	72

[a]Reaction conditions: 0.711 m mol p-chlorobenzaldehyde. [b]180 °C.

Table S7. Reaction time influence

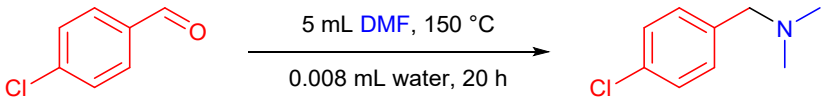


Entry[a]	Time, h	Yield, %

1	1	7
2	2	10
3	3	15
4	4	20
5	5	22
6	8	55
7	12	90
8	16	90

[a]Reaction conditions: 0.711 mmol p-chlorobenzaldehyde, 0.208 mmol $NaH_2PO_2 \cdot H_2O$, 5 mL DMF, 8 µL water.

Table S8. Atmosphere influence

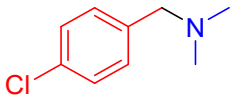


Entry[a]	Atmosphere	Yield, %
1	argon	94
2	air	90

[a]Reaction conditions: 0.711 mmol p-chlorobenzaldehyde, 0.208 mmol $NaH_2PO_2 \cdot H_2O$, 5 mL DMF, 8 µL water.

Substrate scope investigation

1-(4-chlorophenyl)-N,N-dimethylmethanamine (1)



A 10 mL Schlenk tube was charged with 22 mg (0.208 mmol, 0.29 eq) of $NaH_2PO_2 \cdot H_2O$, 100 mg (0.711 mmol) of p-chlorobenzaldehyde, 14 µL of H_2O, 2 mL DMF, sealed under air, and heated to 150 °C for 20 h. The water content in the reaction mixture is estimated to be 1.5 mmol. Then the tube was cooled and the reaction mixture was placed into a separation funnel and diluted with 40 mL of aqueous brine solution. The solution was extracted with ether (3 * 20 mL), then the combined organic fraction was washed with brine (3 * 20 mL) to get rid of the remaining DMF, dried over Na_2SO_4. The solvent was removed using a rotary evaporator to give 98 mg (81 %) of product as a transparent yellowish oil.

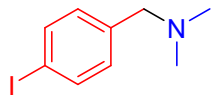
External GC calibration was obtained. GC yield of the product - 92 %.

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.35 – 7.24 (m, 4H), 3.43 (s, 2H), 2.27 (s, 6H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 137.4, 132.8, 130.5, 128.5, 63.6, 45.3.

The spectra are in agreement with literature data.[2]

1-(4-iodophenyl)-N,N-dimethylmethanamine (2)



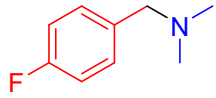
A 10 mL Schlenk tube was charged with 22 mg (0.208 mmol, 0.29 eq) of $NaH_2PO_2 \cdot H_2O$, 166 mg (0.711 mmol, 1 eq) of p-iodobenzaldehyde, 14 µL of H_2O, 2 mL DMF, sealed under air, and heated to 150 °C for 20 h. Then the tube was cooled and the reaction mixture was placed into a separation funnel and diluted with 40 mL of aqueous brine solution. The solution was extracted with ether (3 * 20 mL), then the combined organic fraction was washed with brine (3 * 20 mL) to get rid of the remaining DMF, dried over Na_2SO_4. The solvent was removed using a rotary evaporator to give 125 mg (67 %) of product as a transparent yellowish oil.

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.63 (d, J = 8.0 Hz, 2H), 7.06 (d, J = 8.0 Hz, 2H), 3.35 (s, 2H), 2.21 (s, 6H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 138.7, 137.4, 131.1, 92.5, 63.8, 45.4.

The spectra are in agreement with literature data.[3]

1-(4-fluorophenyl)-N,N-dimethylmethanamine (3)



A 100 mL Schlenk tube was charged with 497 mg (4.69 mmol, 0.29 eq) of $NaH_2PO_2 \cdot H_2O$, 1.73 mL (2.0 g, 16.1 mmol, 1 eq) of p-fluorobenzaldehyde, 300 µL of H_2O, 20 mL DMF, sealed under air, and heated to 150 °C for 20 h. The water content in the reaction mixture is estimated to be 24.5 mmol. Then the tube was cooled and the reaction mixture was placed into a separation funnel and diluted with 100 mL of aqueous brine solution. The solution was extracted with ether (3 * 50 mL), then the combined organic fraction was washed with brine (3 * 50 mL) to get rid of the remaining DMF, and then washed with aqueous acid (3 * 50 mL, 10 % v/v concentrated HCl in water). The acidic fraction was combined, washed with ether (3 * 50 mL), then solid NaOH was added to pH 10. The resulting liquid was extracted with ether (3 * 50 mL). These three ether fractions were combined, dried over Na_2SO_4, and the solvent was removed using a rotary evaporator to give 1.58 g (64 %) of product as a transparent yellowish oil.

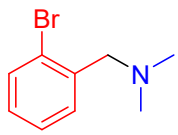
^{1}H NMR (300 MHz, $CDCl_3$) δ 7.23 (m app as t, J = 6.4 Hz, 2H), 6.97 (m app as t, J = 8.7 Hz, 2H), 3.37 (s, 2H), 2.20 (s, 6H).

^{19}F NMR (282 MHz, $CDCl_3$) δ -115.64.

^{13}C NMR (101 MHz, Chloroform-d) δ 162.1 (d, J = 244.8 Hz), 134.6 (d, J = 3.2 Hz), 130.7 (d, J = 8.0 Hz), 115.1 (d, J = 21.1 Hz), 63.6, 45.3.

The spectra are in agreement with literature data.[4]

1-(2-bromophenyl)-N,N-dimethylmethanamine (4)



A 10 mL Schlenk tube was charged with 22 mg (0.208 mmol, 0.29 eq) of $NaH_2PO_2 \cdot H_2O$, 83.4 µL (132.1 mg, 0.711 mmol, 1 eq) of o-bromobenzaldehyde, 14 µL of H_2O, 2 mL DMF, sealed under air, and heated

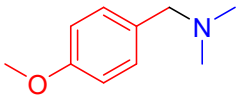
to 150 °C for 20 h. The water content in the reaction mixture is estimated to be 1.5 mmol. Then the tube was cooled and the reaction mixture was placed into a separation funnel and diluted with 40 mL of aqueous brine solution. The solution was extracted with ether (3 * 20 mL), then the combined organic fraction was washed with brine (3 * 20 mL) to get rid of the remaining DMF, and then washed with aqueous acid (3 * 20 mL, 10 % v/v concentrated HCl in water). The acidic fraction was combined, washed with ether (3 * 20 mL), then solid NaOH was added to pH 10. The resulting liquid was extracted with ether (3 * 20 mL). The solvent was removed using a rotary evaporator to give 110 mg (72 %) of product as a transparent yellowish oil.

^{1}H NMR (300 MHz, Chloroform-d) δ 7.54 (dd, J = 7.7, 1.3 Hz, 1H), 7.42 (dd, J = 7.7, 1.8 Hz, 1H), 7.28 (ddd, J = 7.7, 7.7, 1.3 Hz, 1H), 7.11 (ddd, J = 7.7, 7.7, 1.8 Hz, 1H), 3.52 (s, 2H), 2.30 (s, 6H).

^{13}C NMR (75 MHz, Chloroform-d) δ 138.3, 132.9, 131.1, 128.6, 127.3, 124.9, 63.4, 45.7.

The spectra are in agreement with literature data.[5]

1-(4-methoxyphenyl)-N,N-dimethylmethanamine (5)



A 10 mL Schlenk tube was charged with 22 mg (0.208 mmol, 0.29 eq) of $NaH_2PO_2{\cdot}H_2O$, 90.8 µL (97.1 mg, 0.711 mmol) of p-methoxybenzaldehyde, 14 µL of H_2O, 2 mL DMF, sealed under air, and heated to 150 °C for 20 h. The water content in the reaction mixture is estimated to be 1.5 mmol. Then the tube was cooled and the reaction mixture was placed into a separation funnel and diluted with 40 mL of aqueous brine solution. The solution was extracted with ether (3 * 20 mL), then the combined organic fraction was washed with brine (3 * 20 mL) to get rid of the remaining DMF, dried over Na_2SO_4. The solvent was removed using a rotary evaporator to give 84 mg (71 %) of product as a transparent yellowish oil.

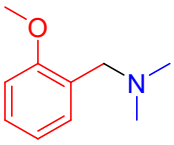
External GC calibration was obtained. GC yield of the product - 81 %.

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.21 (d, J = 8.1 Hz, 2H), 6.85 (d, J = 8.1 Hz, 2H), 3.79 (s, 3H), 3.35 (s, 2H), 2.22 (s, 6H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 158.8, 131.0, 130.3, 113.6, 63.8, 55.3, 45.3.

The spectra are in agreement with literature data.[2]

1-(2-methoxyphenyl)-N,N-dimethylmethanamine (6)



A 10 mL Schlenk tube was charged with 22 mg (0.208 mmol, 0.29 eq) of $NaH_2PO_2{\cdot}H_2O$, 97 mg (0.711 mmol, 1 eq) of o-methoxybenzaldehyde, 14 µL of H_2O, 2 mL DMF, sealed under air, and heated to 150 °C for 20 h. The water content in the reaction mixture is estimated to be 1.5 mmol. Then the tube was cooled and the reaction mixture was placed into a separation funnel and diluted with 40 mL of aqueous brine solution. The solution was extracted with ether (3 * 20 mL), then the combined organic fraction was washed with brine (3 * 20 mL) to get rid of the remaining DMF, and dried over Na_2SO_4. The solvent

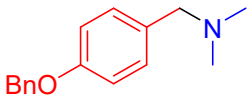
was removed using a rotary evaporator to give 83 mg (70 % yield) of product as a transparent yellowish oil.

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.31 – 7.20 (m, 2H), 6.92 (dd, J = 7.4, 7.4 Hz, 1H), 6.87 (d, J = 8.1 Hz, 1H), 3.83 (s, 3H), 3.45 (s, 2H), 2.27 (s, 6H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 157.9, 131.0, 128.3, 126.7, 120.2, 110.4, 57.9, 55.4, 45.5.

The spectra are in agreement with literature data.[4]

1-(4-benzyloxyphenyl)-N,N-dimethylmethanamine (7)



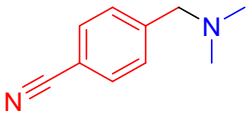
A 10 mL Schlenk tube was charged with 22 mg (0.208 mmol, 0.29 eq) of $NaH_2PO_2{\cdot}H_2O$, 151 mg (0.711 mmol, 1 eq) of p-benzyloxybenzaldehyde, 14 µL of H_2O, 2 mL DMF, sealed under air, and heated to 150 °C for 20 h. The water content in the reaction mixture is estimated to be 1.5 mmol. Then the tube was cooled and the reaction mixture was placed into a separation funnel and diluted with 40 mL of aqueous brine solution. The solution was extracted with ether (3 * 20 mL), then the combined organic fraction was washed with brine (3 * 20 mL) to get rid of the remaining DMF, dried over Na_2SO_4. The solvent was removed using a rotary evaporator to give 148 mg (86 %) of product as a transparent yellowish oil.

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.47 – 7.42 (m, 2H), 7.39 (m app as t, J = 7.5 Hz, 2H), 7.36 – 7.29 (m, 1H), 7.23 (d, J = 8.8 Hz, 2H), 6.95 (d, J = 8.8 Hz, 2H), 5.06 (s, 2H), 3.37 (s, 2H), 2.23 (s, 6H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 158.0, 137.2, 131.3, 130.4, 128.6, 128.0, 127.6, 114.6, 70.1, 63.8, 45.3.

The spectra are in agreement with literature data.[6]

4-((dimethylamino)methyl)benzonitrile (8)



A 100 mL Schlenk tube was charged with 470 mg (4.43 mmol, 0.29 eq) of $NaH_2PO_2{\cdot}H_2O$, 2 g (15.3 mmol, 1 eq) of p-cyanobenzaldehyde, 283 µL of H_2O, 20 mL DMF, sealed under air, and heated to 150 °C for 20 h. The water content in the reaction mixture is estimated to be 23.3 mmol. Then the tube was cooled and the reaction mixture was placed into a separation funnel and diluted with 200 mL of aqueous brine solution. The solution was extracted with ether (3 * 50 mL), then the combined organic fraction was washed with brine (3 * 50 mL) to get rid of the remaining DMF, and then washed with aqueous acid (3 * 50 mL, 10 % v/v concentrated HCl in water). The acidic fraction was combined, washed with ether (3 * 50 mL), then solid NaOH was added to pH 10. The resulting liquid was extracted with ether (3 * 50 mL). The solvent was removed using a rotary evaporator to give 1.0 g (41 %) of the product as a transparent yellowish oil.

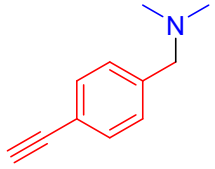
External GC calibration was obtained. GC yield of the product - 53 %.

^{1}H NMR (300 MHz, $CDCl_3$) δ 7.60 (d, J = 8.5 Hz, 2H), 7.43 (d, J = 8.5 Hz, 2H), 3.48 (s, 2H), 2.25 (s, 6H).

^{13}C NMR (101 MHz, Chloroform-d) δ 144.6, 132.1, 129.6, 119.0, 110.9, 63.8, 45.4.

The spectra are in agreement with literature data.[2]

1-(4-ethynylphenyl)-N,N-dimethylmethanamine (9)



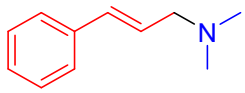
A 10 mL Schlenk tube was charged with 22 mg (0.208 mmol, 0.29 eq) of $NaH_2PO_2 \cdot H_2O$, 93 mg (0.711 mmol, 1 eq) of p-ethynylbenzaldehyde, 14 µL of H_2O, 2 mL DMF, sealed under air, and heated to 150 °C for 20 h. The water content in the reaction mixture is estimated to be 1.5 mmol. Then the tube was cooled and the reaction mixture was placed into a separation funnel and diluted with 40 mL of aqueous brine solution. The solution was extracted with ether (3 * 20 mL), then the combined organic fraction was washed with brine (3 * 20 mL) to get rid of the remaining DMF, and then washed with aqueous acid (3 * 20 mL, 10 % v/v concentrated HCl in water). The acidic fraction was combined, washed with ether (3 * 20 mL), then solid NaOH was added to pH 10. The resulting liquid was extracted with ether (3 * 20 mL). These three ether fractions were combined, dried over Na_2SO_4, and the solvent was removed using a rotary evaporator to give 82 mg (72 %) of product as a transparent yellowish oil.

1H NMR (400 MHz, $CDCl_3$) δ 7.43 (d, J = 7.9 Hz, 2H), 7.27 (d, J = 7.9 Hz, 2H), 3.42 (s, 2H), 3.06 (s, 1H), 2.24 (s, 6H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 139.8, 132.2, 129.2, 120.9, 83.8, 77.10, 64.1, 45.4.

The spectra are in agreement with literature data.[7]

(E)-N,N-dimethyl-3-phenylprop-2-en-1-amine (10)



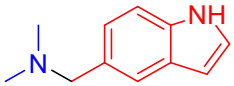
A 10 mL Schlenk tube was charged with 22 mg (0.208 mmol, 0.29 eq) of $NaH_2PO_2 \cdot H_2O$, 89.8 µL (94.2 mg, 0.711 mmol, 1 eq) of *trans*-cinnamaldehyde, 14 µL of H_2O, 2 mL DMF, sealed under air, and heated to 150 °C for 20 h. The water content in the reaction mixture is estimated to be 1.5 mmol. Then the tube was cooled and the reaction mixture was placed into a separation funnel and diluted with 40 mL of aqueous brine solution. The solution was extracted with ether (3 * 20 mL), then the combined organic fraction was washed with brine (3 * 20 mL) to get rid of the remaining DMF, and then washed with aqueous acid (3 * 20 mL, 10 % v/v concentrated HCl in water). The acidic fraction was combined, washed with ether (3 * 20 mL), then solid NaOH was added to pH 10. The resulting liquid was extracted with ether (3 * 20 mL), and the resulting ether fraction was dried over Na_2SO_4. After evaporation of the solvent, 73 mg of crude product was obtained, which was 10 : 1 mixture of the target product and N,N-dimethyl-3-phenylpopanamine. The product (58 mg of a yellowish oil, 50 % yield) was isolated via column chromatography of the mixture (Acros Organic silica gel 0.06–0.2 mm, 6 % MeOH-DCM, product R_f = 0.25).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.43 – 7.35 (m, 2H), 7.31 (m app as t, J = 7.6 Hz, 2H), 7.25 – 7.17 (m, 1H), 6.52 (d, J = 15.8 Hz, 1H), 6.27 (dt, J = 15.8, 6.7 Hz, 1H), 3.08 (d, J = 6.7 Hz, 2H), 2.28 (s, 6H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 137.0, 133.1, 128.7, 127.7, 126.9, 126.5, 62.1, 45.2.

The spectra are in agreement with literature data.[5]

1-(1H-indol-5-yl)-N,N-dimethylmethanamine (11)



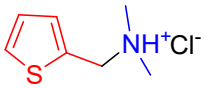
A 10 mL Schlenk tube was charged with 22 mg (0.208 mmol, 0.29 eq) of $NaH_2PO_2{\cdot}H_2O$, 104 mg (0.711 mmol, 1 eq) of 5-formylindole, 14 µL of H_2O, 2 mL DMF, sealed under air, and heated to 150 °C for 20 h. The water content in the reaction mixture is estimated to be 1.5 mmol. Then the tube was cooled and the reaction mixture was placed into a separation funnel and diluted with 40 mL of aqueous brine solution. The solution was extracted with ether (3 * 20 mL), then the combined organic fraction was washed with brine (3 * 20 mL) to get rid of the remaining DMF, and then washed with aqueous acid (3 * 20 mL, 10 % v/v concentrated HCl in water). The acidic fraction was combined, washed with ether (3 * 20 mL), then solid NaOH was added to pH 10. The resulting liquid was extracted with ether (3 * 20 mL). These three ether fractions were combined, dried over Na_2SO_4, and the solvent was removed using a rotary evaporator to give 64 mg (51 %) of product as a transparent yellowish oil.

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.64 (s, 1H), 7.56 (s, 1H), 7.32 (d, J = 8.3 Hz, 1H), 7.21 – 7.14 (m, 2H), 6.52 (s, 1H), 3.58 (s, 2H), 2.30 (s, 6H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 135.4, 129.5, 127.9, 124.7, 123.7, 121.5, 111.1, 102.5, 64.8, 45.2.

The spectra are in agreement with literature data.[8]

N,N-dimethyl-1-(thiophen-2-yl)methanamine hydrochloride (12)



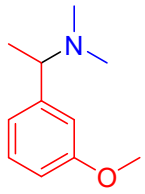
A 10 mL Schlenk tube was charged with 22 mg (0.208 mmol, 0.29 eq) of $NaH_2PO_2{\cdot}H_2O$, 66.8 µL (80 mg, 0.711 mmol, 1 eq) of 2-formylthiophene, 14 µL of H_2O, 2 mL DMF, sealed under air, and heated to 150 °C for 20 h. The water content in the reaction mixture is estimated to be 1.5 mmol. Then the tube was cooled and the reaction mixture was placed into a separation funnel and diluted with 40 mL of aqueous brine solution. The solution was extracted with ether (3 * 20 mL), then the combined organic fraction was washed with brine (3 * 20 mL) to get rid of the remaining DMF. Then HCl in ether was added, the ether was evaporated with a rotary evaporator, and the residue was dried in vacuo under 50 °C. 115 mg (86 % yield) of product were isolated as a white solid.

^{1}H NMR (400 MHz, DMSO) δ 11.17 (s, 1H), 7.69 (d, J = 5.0 Hz, 1H), 7.40 (d, J = 3.4 Hz, 1H), 7.13 (dd, J = 5.0, 3.4 Hz, 1H), 4.49 (d, J = 5.0 Hz, 2H), 2.67 (d, J = 4.7 Hz, 6H).

^{13}C NMR (101 MHz, DMSO) δ 132.2, 131.0, 129.2, 127.6, 52.8, 40.9.

HRMS (ESI (+), acetonitrile; m/z): calcd. for $C_7H_{12}SN^+$ $[M+H]^+$ 142.0685. Found: 142.0691.

1-(3-methoxyphenyl)-N,N-dimethylethan-1-amine (13)



A 10 mL Schlenk tube was charged with 22 mg (0.208 mmol, 0.29 eq) of $NaH_2PO_2 \cdot H_2O$, 100.8 µL (0.711 mmol, 1 eq) of *m*-methoxyacetophenone, 14 µL of H_2O, 2 mL of DMF, sealed under air, and heated to 180 °C for 20 h. The water content in the reaction mixture is estimated to be 1.5 mmol. Then the tube was cooled and the reaction mixture was placed into a separation funnel and diluted with 30 mL of aqueous brine solution. The solution was extracted with ether (3 * 10 mL), then the combined organic fraction was washed with brine (3 * 30 mL), and the ether fraction was dried with Na_2SO_4 and the solvent was removed using a rotary evaporator to give a yellowish oil.

The oil was dissolved in 10% HCl solution, washed with ether (3 * 10 mL), then solid NaOH was added to pH 10 to the aqueous layer. The layer was extracted with ether (3 * 10 mL), then the combined organic fraction was washed with brine (3 * 30 mL), the ether fraction was dried with Na_2SO_4 and the solvent was removed using a rotary evaporator to give 100 mg (78 %) of the product as a transparent yellowish oil.

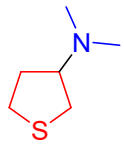
External GC calibration was obtained. GC yield of the product - 78 %.

^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.39 – 7.18 (m, 1H), 7.01 – 6.75 (m, 3H), 3.84 (s, 3H), 3.23 (q, *J* = 6.7 Hz, 1H), 2.24 (s, 6H), 1.39 (d, *J* = 6.7 Hz, 3H).

^{13}C NMR (101 MHz, Chloroform-*d*) δ 159.7, 146.1, 129.2, 120.0, 113.0, 112.3, 66.2, 55.3, 43.5, 20.6.

The spectra are in agreement with the literature data.[9]

N,N-dimethyltetrahydrothiophen-3-amine (14)



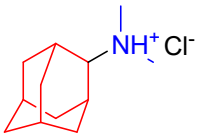
A 100 mL Schlenk tube was charged with 604 mg (5.70 mmol, 0.29 eq) of $NaH_2PO_2 \cdot H_2O$, 1.75 mL (2.0 g, 19.6 mmol, 1 eq) of 3-oxothiolane, 365 µL of H_2O, 20 mL DMF, sealed under air, and heated to 150 °C for 20 h. The water content in the reaction mixture is estimated to be 29.1 mmol. Then the tube was cooled and the reaction mixture was placed into a separation funnel and diluted with 100 mL of aqueous brine solution. The solution was extracted with ether (3 * 50 mL), then the combined organic fraction was washed with brine (3 * 50 mL) to get rid of the remaining DMF, and then washed with aqueous acid (3 * 50 mL, 10 % v/v concentrated HCl in water). The acidic fraction was combined, washed with ether (3 * 50 mL), then solid NaOH was added to pH 10. The resulting liquid was extracted with ether (3 * 50 mL). These three ether fractions were combined, dried over Na_2SO_4, and the solvent was removed using a rotary evaporator to give 0.83 g (31 %) of product as a dark-brown oil.

^{1}H NMR (300 MHz, $CDCl_3$) δ 2.97 – 2.78 (m, 4H), 2.78 – 2.67 (m, 1H), 2.33 (s, 6H), 2.30 – 2.19 (m, 1H), 1.94 – 1.74 (m, 1H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 70.6, 44.3, 33.8, 33.2, 28.6.

HRMS (ESI (+), acetonitrile; m/z): calcd. for $C_6H_{14}SN^+$ $[M+H]^+$ 132.0841. Found: 132.0841.

N,N-dimethyladamantan-2-amine hydrochloride (15)



A 10 mL Schlenk tube was charged with 22 mg (0.208 mmol, 0.29 eq) of $NaH_2PO_2 \cdot H_2O$, 107 mg (0.711 mmol, 1 eq) of 2-adamantanone, 14 µL of H_2O, 2 mL DMF, sealed under air, and heated to 150 °C for 20 h. The water content in the reaction mixture is estimated to be 1.5 mmol. Then the tube was cooled and the reaction mixture was placed into a separation funnel and diluted with 40 mL of aqueous brine solution. The solution was extracted with ether (3 * 20 mL), then the combined organic fraction was washed with brine (3 * 20 mL) to get rid of the remaining DMF. Then HCl in ether was added, the ether was evaporated with a rotary evaporator, and the residue was dried in vacuo under 50 °C. 159 mg (99 % yield) of product were isolated as a white solid.

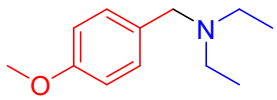
^{1}H NMR (400 MHz, DMSO) δ 9.94 (s, 1H), 3.20 (m app as d, J = 9.6 Hz, 1H), 2.72 (d, J = 4.8 Hz, 6H), 2.29 (m app as s, 2H), 2.19 (m app as d, J = 13.4 Hz, 2H), 1.90 – 1.76 (m, 4H), 1.76 – 1.64 (m app as s, 4H), 1.50 (m app as d, J = 13.3 Hz, 2H).

^{1}H NMR (300 MHz, D_2O) δ 3.18 (s, 1H), 2.80 (s, 6H), 2.29 (s, 2H), 1.90 (m app as d, J = 12.7 Hz, 2H), 1.83 – 1.75 (m, 2H), 1.72 – 1.62 (m, 8H).

^{13}C NMR (101 MHz, D_2O) δ 71.6, 41.2, 36.13, 36.07, 29.7, 27.7, 25.9, 25.8.

HRMS (ESI (+), acetonitrile; m/z): calcd. for $C_{12}H_{22}N^+$ $[M+H]^+$ 180.1747. Found: 180.1744.

N-ethyl-N-(4-methoxybenzyl)ethanamine (16)



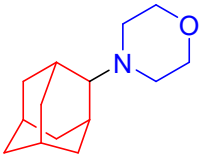
A 10 mL Schlenk tube was charged with 22 mg (0.208 mmol, 0.29 eq) of $NaH_2PO_2 \cdot H_2O$, 90.8 µL (97.1 mg, 0.711 mmol) of *p*-methoxybenzaldehyde, 14 µL of H_2O, 2 mL of N,N-diethylformamide, sealed under air, and heated to 150 °C for 20 h. The water content in the reaction mixture is estimated to be 1.5 mmol. Then the tube was cooled and the reaction mixture was placed into a separation funnel and diluted with 40 mL of aqueous brine solution. The solution was extracted with ether (3 * 20 mL), then the combined organic fraction was washed with brine (3 * 20 mL), and HCl in ether was added to the organic fraction. The ether then was removed with a rotary evaporator, and the residue was dried in vacuo under 80 °C for 4 h. The crystals were dissolved in water, washed with ether (3 * 20 mL), then solid NaOH was added to the aqueous layer. The layer was extracted with ether (3 * 20 mL), the ether fraction was dried with Na_2SO_4 and the solvent was removed using a rotary evaporator to give 68 mg (48 %) of the product as a transparent yellowish oil.

^{1}H NMR (300 MHz, $CDCl_3$) δ 7.22 (d, J = 8.6 Hz, 2H), 6.83 (d, J = 8.6 Hz, 2H), 3.78 (s, 3H), 3.49 (s, 2H), 2.49 (q, J = 7.1 Hz, 4H), 1.02 (t, J = 7.1 Hz, 6H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 158.6, 131.9, 130.1, 113.6, 56.9, 55.3, 46.6, 11.8.

The spectra are in agreement with literature data.[10]

4-(adamantan-2-yl)-morpholine (17)



A 10 mL Schlenk tube was charged with 22 mg (0.208 mmol, 0.29 eq) of $NaH_2PO_2 \cdot H_2O$, 107 mg (0.711 mmol, 1 eq) of 2-adamantanone, 14 µL of H_2O, 2 mL of N-formylmorpholine, sealed under air, and heated to 150 °C for 20 h. The water content in the reaction mixture is estimated to be 1.5 mmol. Then the tube was cooled and the reaction mixture was placed into a separation funnel and diluted with 30 mL of aqueous brine solution. The solution was extracted with ether (3 * 10 mL), then the combined organic fraction was washed with brine (3 * 30 mL), and the ether fraction was dried with Na_2SO_4 and the solvent was removed using a rotary evaporator to give colorless oil.

The oil was dissolved in 10% HCl solution, washed with ether (3 * 10 mL), then solid NaOH was added to pH 10 to the aqueous layer. The layer was extracted with ether (3 * 10 mL), then the combined organic fraction was washed with brine (3 * 30 mL), the ether fraction was dried with Na_2SO_4 and the solvent was removed using a rotary evaporator to give 134.1 mg (85 %) of the product as a colorless oil.

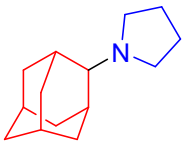
External GC calibration was obtained. GC yield of the product - 95 %.

^{1}H NMR (400 MHz, Chloroform-*d*) δ 3.71 (m app. as t, *J* = 4.6 Hz, 4H), 2.40 (s, 4H), 2.14 – 1.92 (m, 5H), 1.88 – 1.47 (m, 8H), 1.40 (m app. as d, *J* = 12.4 Hz, 2H).

^{13}C NMR (101 MHz, Chloroform-*d*) δ 68.1, 67.7, 50.3, 37.9, 37.3, 31.4, 28.7, 27.6, 27.5.

The spectra are in agreement with the literature data.[11]

1-(adamantan-2-yl)-pyrrolidine (18)



A 10 mL Schlenk tube was charged with 22 mg (0.208 mmol, 0.29 eq) of $NaH_2PO_2 \cdot H_2O$, 107 mg (0.711 mmol, 1 eq) of 2-adamantanone, 14 µL of H_2O, 2 mL of N-formylpyrrolidine, sealed under air, and heated to 150 °C for 20 h. Then the tube was cooled and the reaction mixture was placed into a separation funnel and diluted with 30 mL of aqueous brine solution. The solution was extracted with ether (3 * 10 mL), then the combined organic fraction was washed with brine (3 * 30 mL), and the ether fraction was dried with Na_2SO_4 and the solvent was removed using a rotary evaporator to give yellowish oil.

The oil was dissolved in 10% HCl solution, washed with ether (3 * 10 mL), then solid NaOH was added to pH 10 to the aqueous layer. The layer was extracted with ether (3 * 10 mL), then the combined organic fraction was washed with brine (3 * 30 mL), the ether fraction was dried with Na_2SO_4 and the solvent was removed using a rotary evaporator to give 45 mg (31 %) of the product as a transparent yellowish oil.

External GC calibration was obtained. GC yield of the product - 88 %.

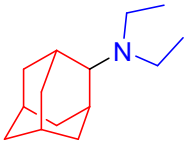
^{1}H NMR (400 MHz, Chloroform-*d*) δ 2.49 (s, 4H), 2.14 (m app. as d, *J* = 12.1 Hz, 2H), 2.08 (s, 1H), 1.94 (s, 2H), 11.87 – 1.61 (m, 12H), 1.42 (m app. as d, *J* = 12.1 Hz, 2H).

^{13}C NMR (101 MHz, Chloroform-*d*) δ 70.5, 51.7, 38.2, 37.4, 31.67, 31.62, 27.74, 27.66, 23.6.

The spectra are in agreement with the literature data.[11]

The large discrepancy between the preparative and GC yields could be explained by evaporation of the product when removing the solvent.

N,N-diethyladamantan-2-amine (19)



A 10 mL Schlenk tube was charged with 22 mg (0.208 mmol, 0.29 eq) of $NaH_2PO_2 \cdot H_2O$, 107 mg (0.711 mmol, 1 eq) of 2-adamantanone, 14 µL of H_2O, 2 mL of N,N-diethylformamide, sealed under air, and heated to 150 °C for 20 h. The water content in the reaction mixture is estimated to be 1.5 mmol. Then the tube was cooled and the reaction mixture was placed into a separation funnel and diluted with 30 mL of aqueous brine solution. The solution was extracted with ether (3 * 10 mL), then the combined organic fraction was washed with brine (3 * 30 mL), and the ether fraction was dried with Na_2SO_4 and the solvent was removed using a rotary evaporator to give yellowish oil.

The oil was dissolved in 10% HCl solution, washed with ether (3 * 10 mL), then solid NaOH was added to pH 10 to the aqueous layer. The layer was extracted with ether (3 * 10 mL), then the combined organic fraction was washed with brine (3 * 30 mL), the ether fraction was dried with Na_2SO_4 and the solvent was removed using a rotary evaporator to give 42.4 mg (31%) of the product as transparent yellowish oil.

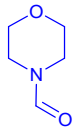
External GC calibration was obtained. GC yield of the product – 40 %.

^{1}H NMR (400 MHz, Chloroform-*d*) δ 2.64 (q, *J* = 7.0 Hz, 4H), 2.46 (s, 1H), 2.07 (m app. as d, *J* = 12.1 Hz, 2H), 1.99 (s, 2H), 1.91 – 1.73 (m, 4H), 1.73 – 1.62 (m, 4H), 1.40 (m app. as d, *J* = 12.0 Hz, 2H), 0.93 (t, *J* = 7.0 Hz, 6H).

^{13}C NMR (101 MHz, Chloroform-*d*) δ 63.3, 41.8, 38.1, 37.6, 31.6, 29.7, 27.8, 27.6, 10.9.

HRMS (ESI (+), acetonitrile; m/z): calcd. for $C_{14}H_{26}N^+$ $[M+H]^+$ 208.2060. Found: 208.2061.

N-formylmorpholine



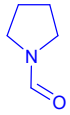
A 100 mL bomb tube was charged with 15mL (0.243 mol, 1.4 eq) of methyl formate, 15 mL (0.173 mol, 1 eq) of morpholine, sealed under air, and heated to 150 °C for 20 h. The product distilled under 150°C on a diaphragm pump. Pure fraction contained 15.7 g (79 %) of the product as colorless oil.

^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.01 (s, 1H), 3.64 (t, *J* = 4.8 Hz, 2H), 3.61 (t, *J* = 4.8 Hz, 2H), 3.50 (t, *J* = 4.9 Hz, 2H), 3.35 (t, *J* = 4.9 Hz, 2H).

^{13}C NMR (101 MHz, Chloroform-*d*) δ 160.8, 67.2, 66.4, 45.7, 40.5.

The spectra are in agreement with the literature data.[12]

N-formylpyrrolidine



A 100 mL bomb tube was charged with 15mL (0.243 mol, 1.33 eq) of methyl formate, 15 mL (0.183 mol, 1 eq) of pyrrolidine, sealed under air, and heated to 150 °C for 20 h. The product distilled under 150°C on a diaphragm pump. Pure fraction contained 12.0 g (66 %) of the product as colorless oil.

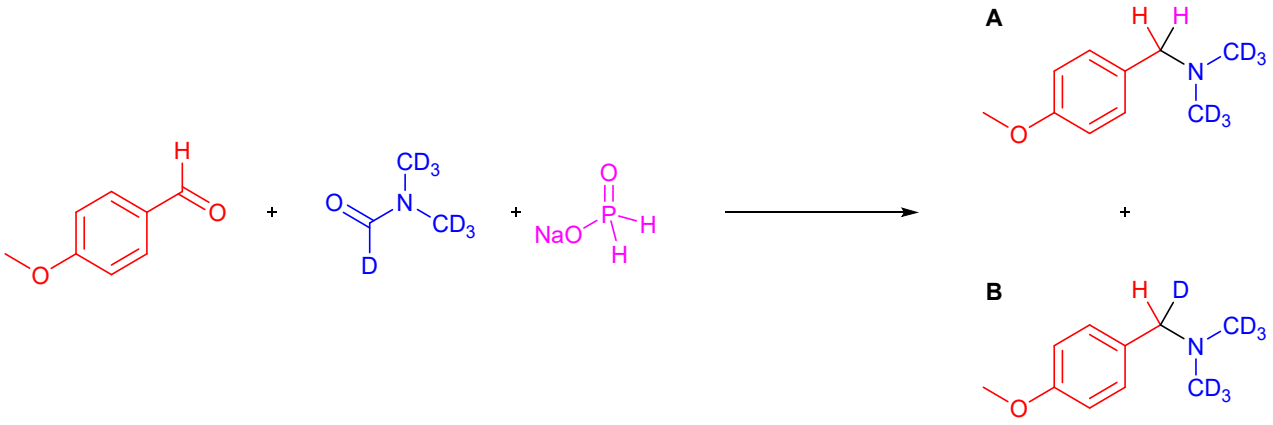
^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.22 (s, 1H), 3.46 (t, *J* = 6.4 Hz, 2H), 3.38 (t, *J* = 6.7 Hz, 2H), 2.07 – 1.68 (m, 4H).

^{13}C NMR (101 MHz, Chloroform-*d*) δ 160.8, 46.0, 43.0, 24.8, 24.1.

The spectra are in agreement with the literature data.[13]

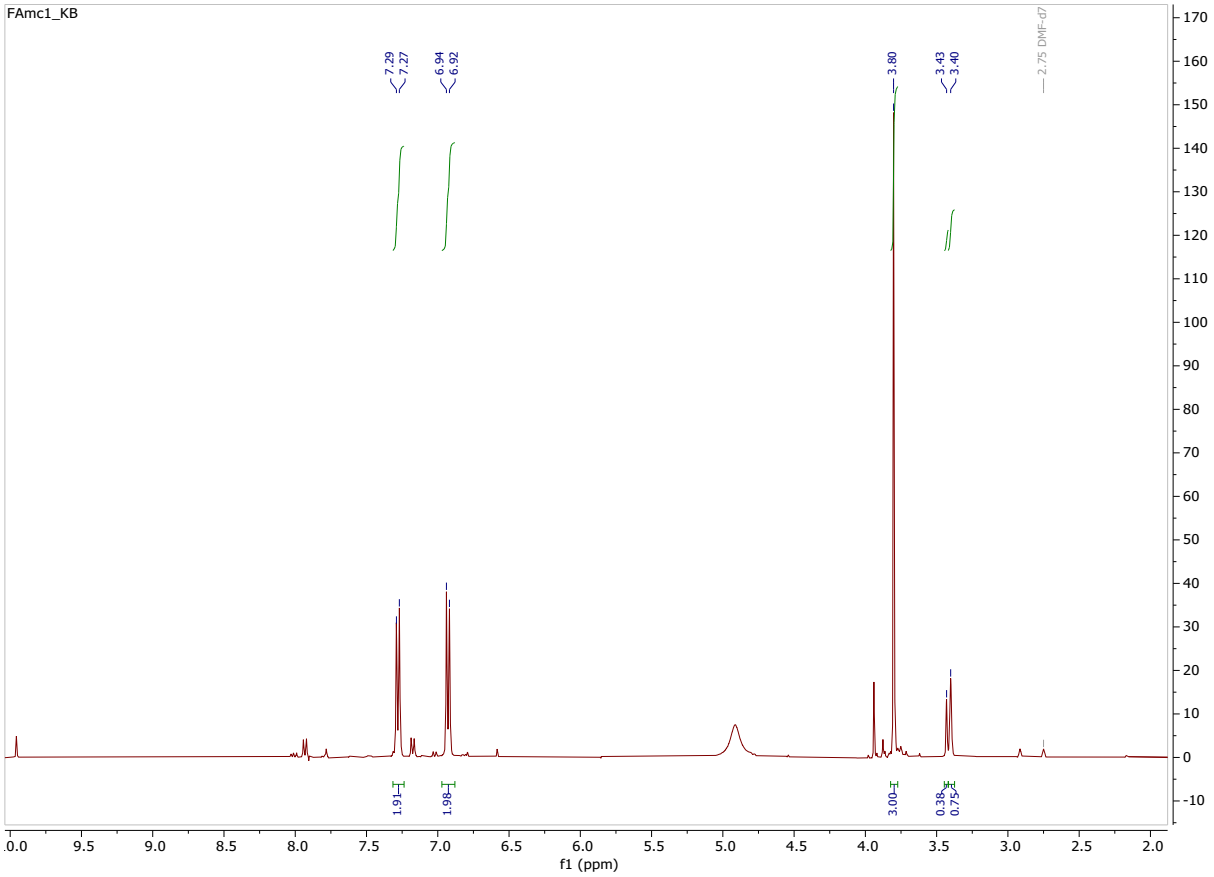
Mechanistic experiment

The purpose of the experiment was to determine whether the main reductant in the system is hypophosphite or the formate formed by DMF decomposition.



A high-pressure NMR tube was charged with 3.3 mg (0.031 mmol, 0.29 equiv.) of $NaH_2PO_2 \cdot H_2O$, 13.6 µL (14.6 mg, 0.107 mmol) of p-methoxybenzaldehyde, 2 µL of H_2O, 0.3 mL of DMF-D7, sealed and heated to 150 °C for 20 h. Then the tube was cooled to room temperature and the ^{1}H-NMR spectrum was registered (Figure S1).

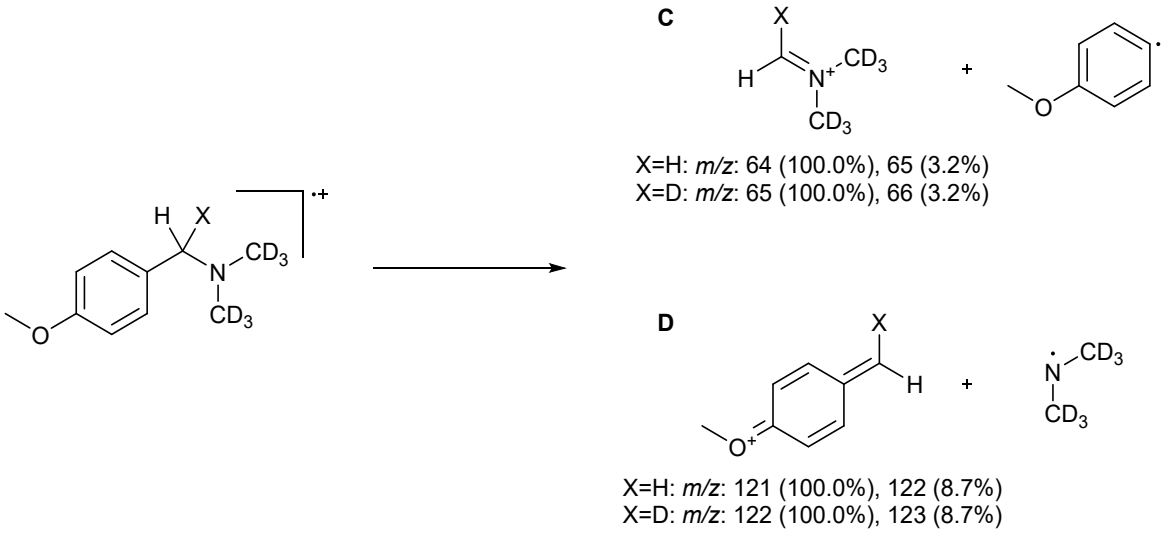
Figure S1. 1H-NMR spectrum of the reaction mixture of the reaction set up in DMF-D7.



In the spectrum of the formed product, there are two signals corresponding to the benzylic hydrogens at 3.43 and 3.40 ppm, their intensities relate as 1 : 2. It follows that products **A** and **B** formed either in 1 : 1 (if 3.43 peak corresponds to **B**, 3.40 – to **A**) or in 1 : 4 ratio (in another case).

Then the content of the NMR-tube was transferred to a GC vial and diluted with DCM to 1 mL. 200 µL of the solution were transferred to another GC vial and diluted with DCM to 1 mL. The resulting solution was analyzed by GC-MS. The chromatogram contained the main peak of the product and the minor peak of the starting aldehyde.

Analysis of the molecular ions of **A** and **B** was hindered by the fast hydrogen radical abstraction from the molecular cation-radical leading to strong $[M-1]^+$ peak. In the case of **B**, that is further complicated by the competition between protium- and deuterium-radical abstractions. Thus, we analysed the main fragment ions (the spectrum is provided in Table S9 below).



The fraction of undeuterated ions **C** χ_H(**C**) was calculated using the following expression ($I_{m/z}$ stands for *m/z* ion abundance):

$$\chi_H(C) = \frac{I_{64} * 100\ \%}{(I_{65} - 0.032 * I_{64}) + I_{64}}$$

The similar procedure was applied to **D**:

$$\chi_H(D) = \frac{I_{121} * 100\ \%}{(I_{122} - 0.087 * I_{121}) + I_{121}}$$

Thus, we obtained the estimations of the undeuterated product fraction listed below:

	υ(**A**)/(υ(**A**)+υ(**B**)), %
NMR	20
Ion **C** peaks in MS	25
Ion **D** peaks in MS	21
Average	22

Thus, we estimate that at most 25 % of the black hydrogens in the product on the scheme below come from hypophosphite. The main source of hydrogen is probably the formate formed by DMF decomposition.

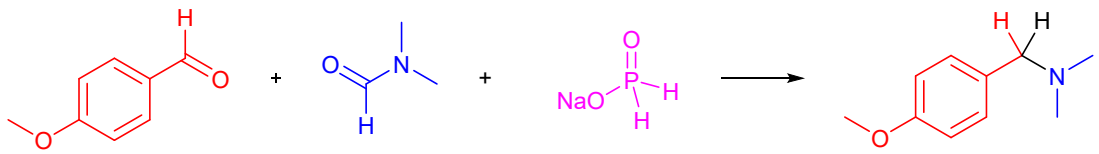
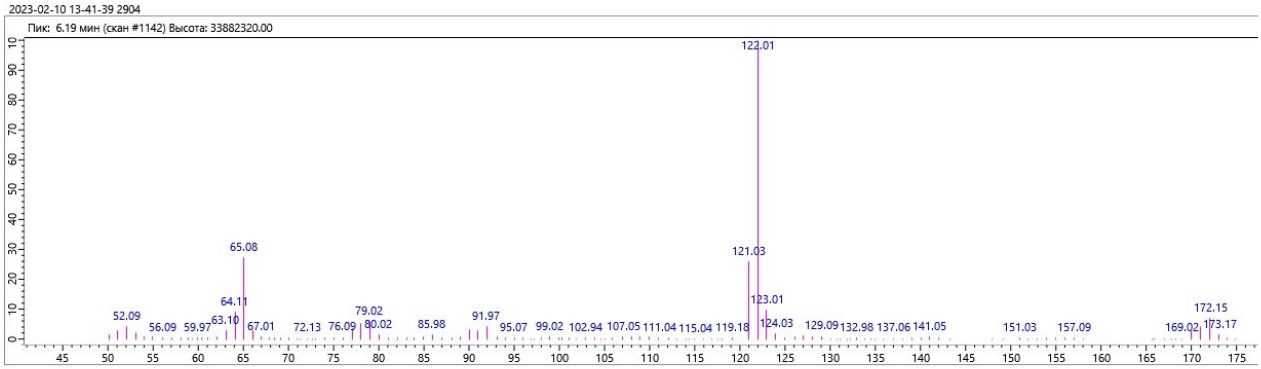


Table S9. EI-MS spectrum of the product of the reaction set up in DMF-D7.

m/z	Abundance	m/z	Abundance	m/z	Abundance	m/z	Abundance
50.18	418603.63	83.99	95146.11	118.07	16691.72	165.97	477.02
51.08	886311.94	85.01	196121.81	119.18	48021.32	167.15	7351.60
52.09	1389196	85.98	441072.22	**121.03**	**8719294.00**	167.95	9387.86
53.08	722290.75	87.03	57088.13	**122.01**	**33882320.00**	168.39	6156.92
54.04	251362.47	88.14	51973.21	123.01	3250501.25	169.02	20906.96
54.95	223426.56	89.08	217805.83	124.03	572264.19	170.06	881173.44
56.09	83731.82	90.03	1052052.5	125.01	94068.64	171.12	1407980.63
57.1	39058.57	91	898898.25	126.13	198587.05	172.15	2345688.00
58.12	43021.67	91.97	1399354.88	127.10	273502.78	173.17	387840.75
58.96	28355.85	93.11	183846.22	128.09	228075.48	174.08	35254.52
59.37	18964.3	94.09	74538.42	129.09	250954.94	175.00	132.35
59.97	58958.79	95.07	68108.77	130.11	12183.50	192.83	101.13
60.4	25509.05	96.04	34519.38	130.79	2245.51	206.97	4137.61
61.07	129220.09	96.88	15251.76	131.13	3385.95	207.73	343.53
62.04	243588.14	97.31	8593.53	131.96	8817.64	208.27	689.90
63.1	911007.06	98.01	61028.98	132.30	6710.69	209.16	1210.55
64.11	**2989344.25**	99.02	164226.63	132.98	46802.15	223.03	170.41
65.08	**9242329**	99.93	94495.83	133.95	7391.68	231.86	395.91
66.05	867544.25	100.28	70167.4	134.61	2972.45	270.94	143.50
67.01	172321.36	101.12	39393.85	135.08	16795.36	280.75	149.91
67.95	100865.13	101.96	10613.9	136.00	23118.73	297.62	211.41
68.51	97286.41	102.94	65799.91	137.06	13585.73	341.15	498.41
69.19	58197.3	104	31660.55	138.09	18454.96	485.85	84.56
70.06	41045.53	104.67	11778.39	139.10	31545.07	521.29	13.32
71	13329.74	105.06	8836.39	140.10	69887.20		
71.33	7127.3	105.96	104330.26	141.05	231318.95		
72.13	20071.55	107.05	231005.38	142.07	28636.31		
72.63	3008.47	108.06	202368.52	143.31	5591.79		
73.07	20401.65	109.03	203887.05	147.97	1330.54		
74.04	64779.05	110.06	155392.09	149.25	1396.90		
75.04	55079.54	111.04	59683.76	151.03	68504.29		
76.09	133073.22	112.17	28743.14	151.96	8969.38		
77.06	1117192	113.12	21256.08	152.97	20796.96		
78.03	1686415.63	114.09	4687.94	154.07	28043.05		
79.02	1983232.13	114.41	3753.78	155.11	93095.75		
80.02	381791.56	115.04	7270.03	156.14	100348.56		
81.09	139237.14	116.06	6990.11	157.09	112183.43		
82.1	128446.04	116.98	2514.99	158.08	12540.37		
83.12	101869.2	117.59	5073.02	165.71	855.61		

Figure S2. EI-MS spectrum of the product of the reaction set up in DMF-D7.



Bioassays of fungicidal activities

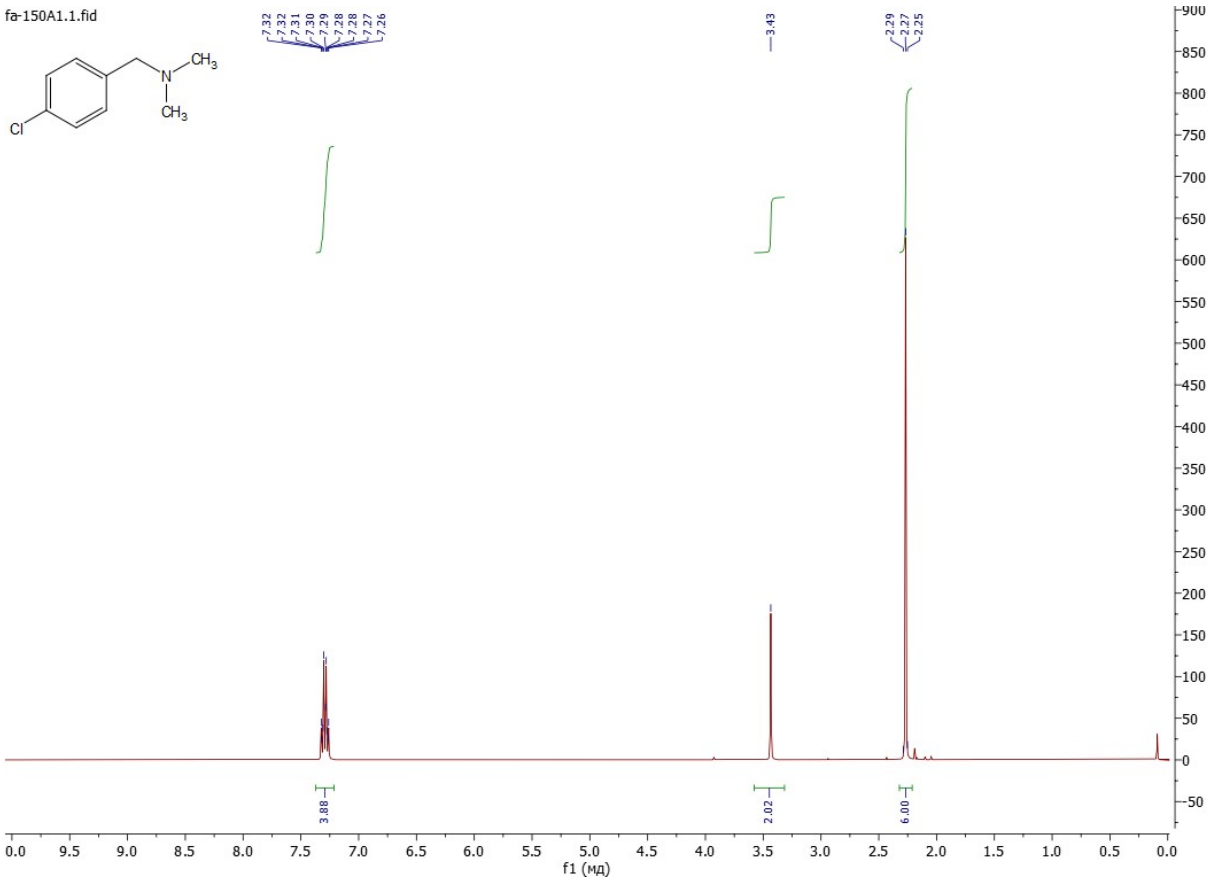
The effect of the chemicals on mycelial radial growth was determined by dissolving the compounds at 3 mg/mL in acetone or at 2 mg/mL in acetone/water mixture (2:1), which was used in cases of poor solubility of the chemical in acetone. Then aliquots were suspended in potato-saccharose agar at 50 ℃ to give the concentration 30 μg/mL. The final solvent concentration was 10 mL/L. Petri dishes containing 15 mL of the agar medium were inoculated by placing 2-mm mycelial agar discs on the agar surface. Plates were incubated at 25 ℃ and radial growth was measured after 72 h. The agar medium without a sample was used as the blank control. Three replicates of each test were carried out. The mycelium elongation diameter (mm) of fungi settlements was measured after 72 h of culture. The growth inhibition rates (***I***) were calculated using the following equation:

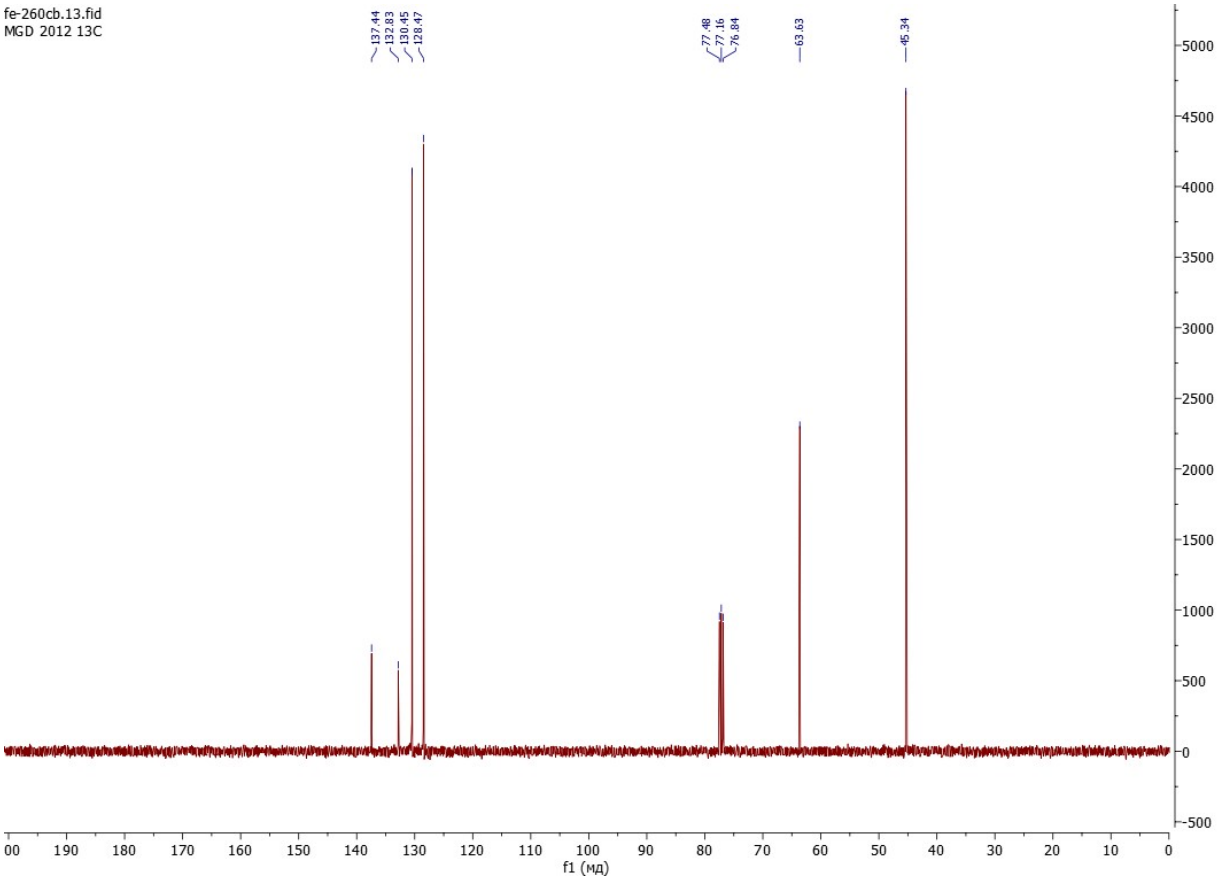
$$I = \frac{Control\ settlement\ diameter\ (mm)\ -\ Test\ settlement\ diameter\ (mm)}{Control\ settlement\ diameter\ (mm)} * 100\%$$

Table S10. The results of the assay of the antifungal activity of the synthesized amides.

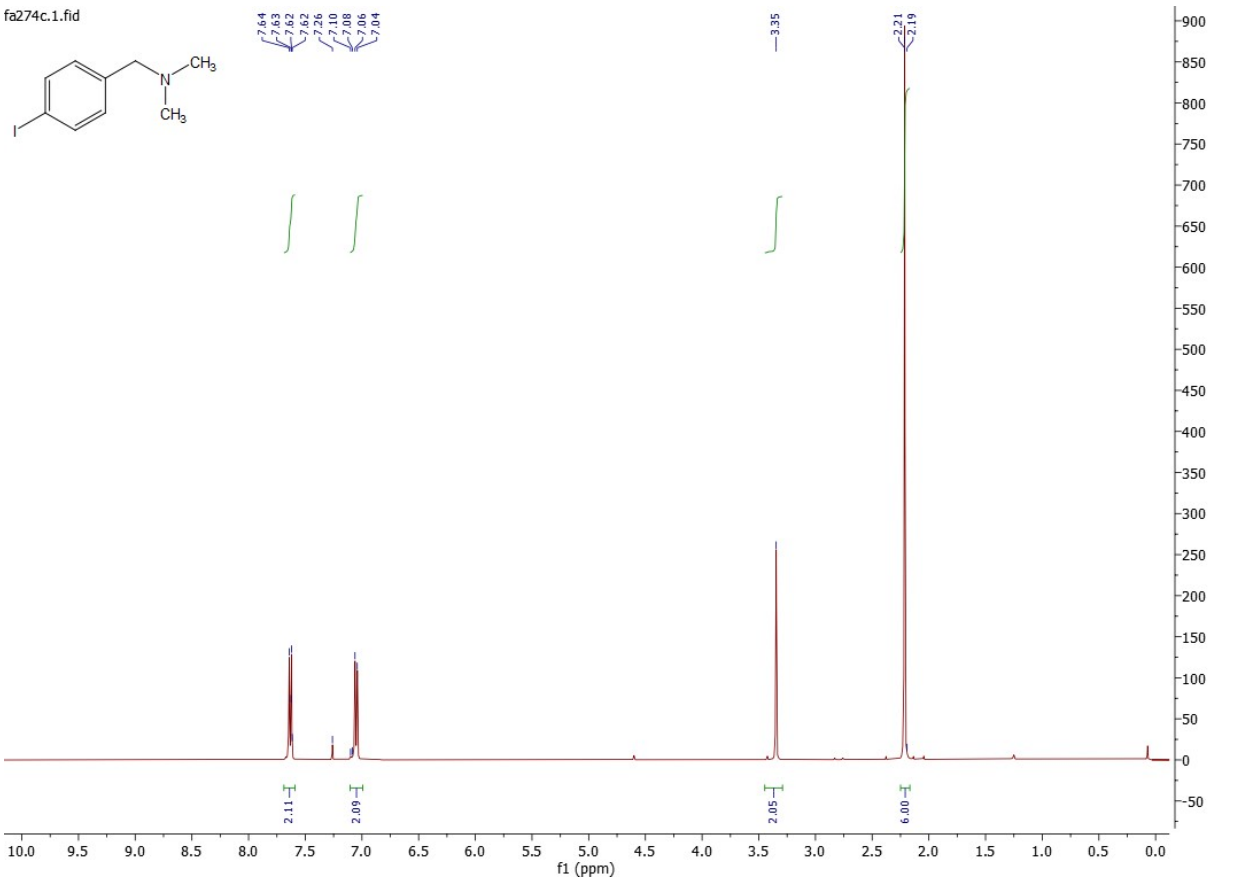
Amine	Mycelium growth inhibition by the product, %/ Mycelium growth inhibition by triadimefon, %			
	R.s.	***V.i.***	***P.c.***	***S.s.***
1	9/,41	18/ 64	22/,32	5/,60
2	28/,41	6/,64	21/,32	0/,60
3	13/,41	19/,64	0/,32	−2/,60
4	9/,41	10/,64	13/,32	1/,60
5	11/,41	19/ 64	27/,32	16/,60
6	9/,41	12/,64	3/,32	1/,60
7	20/,41	21/,64	9/,32	10/,60
8	23/,41	26/,64	28/,32	0/,60
10	21/,41	23/,64	0/,32	0/,60
12	26/,41	12/,64	21/,32	1/,60
13	11/,41	30/,64	26/,32	1/,60
14	24/,41	17/,64	1/,32	1/,60
15	10/,41	6/,64	5/,32	5/,60
17	−13/,41	17/,64	24/,32	49/,60
18	3/,41	31/,64	28/,32	13/,60
19	9/,41	23/,64	5/,32	9/,60

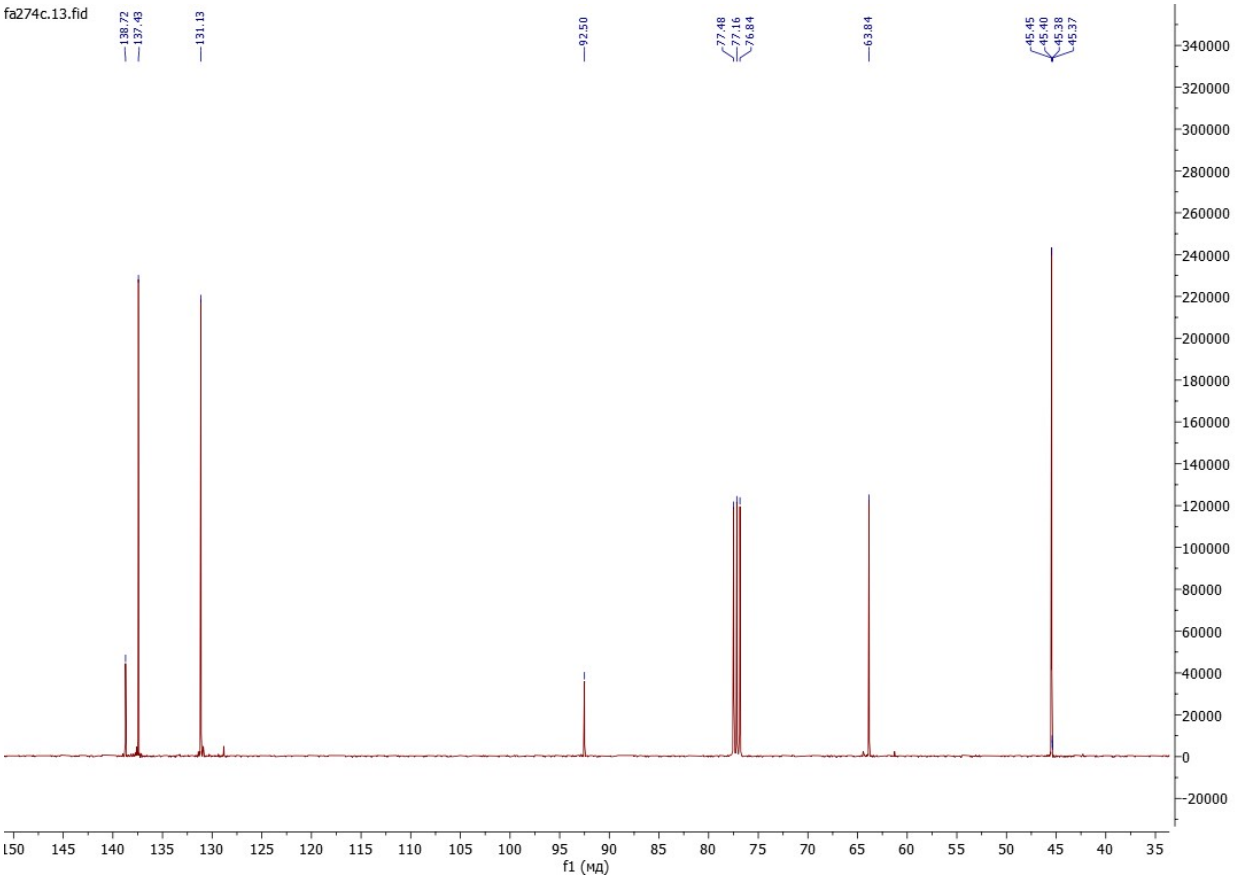
1-(4-chlorophenyl)-N,N-dimethylmethanamine (1)



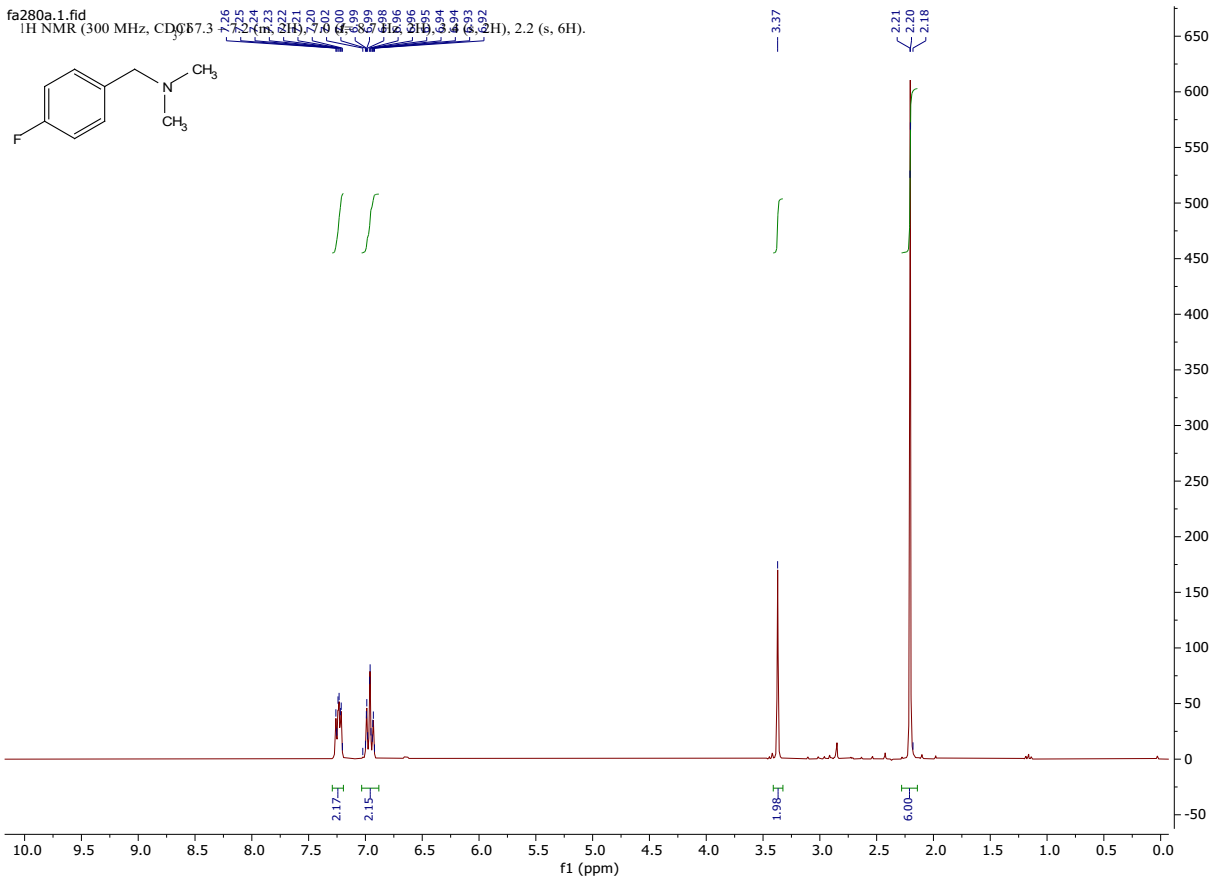


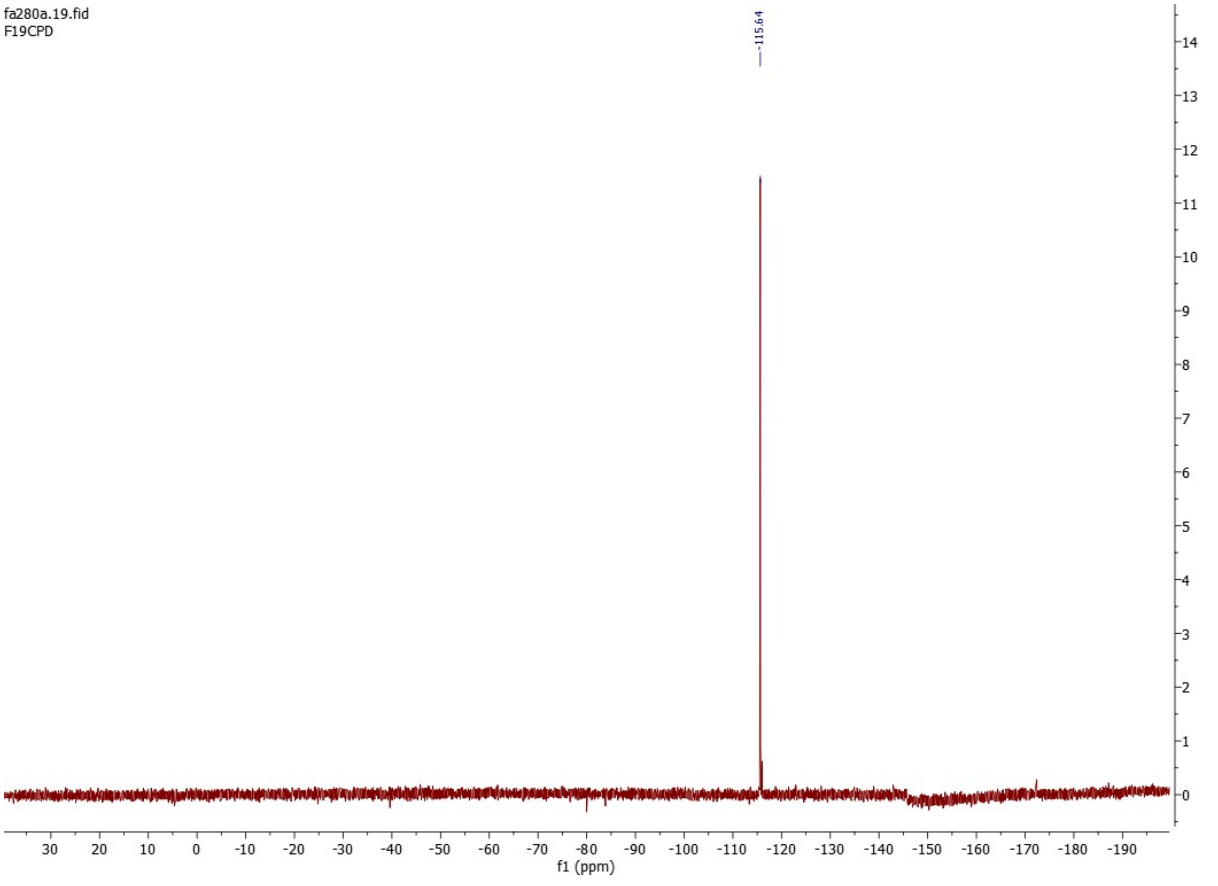
1-(4-iodophenyl)-N,N-dimethylmethanamine (2)

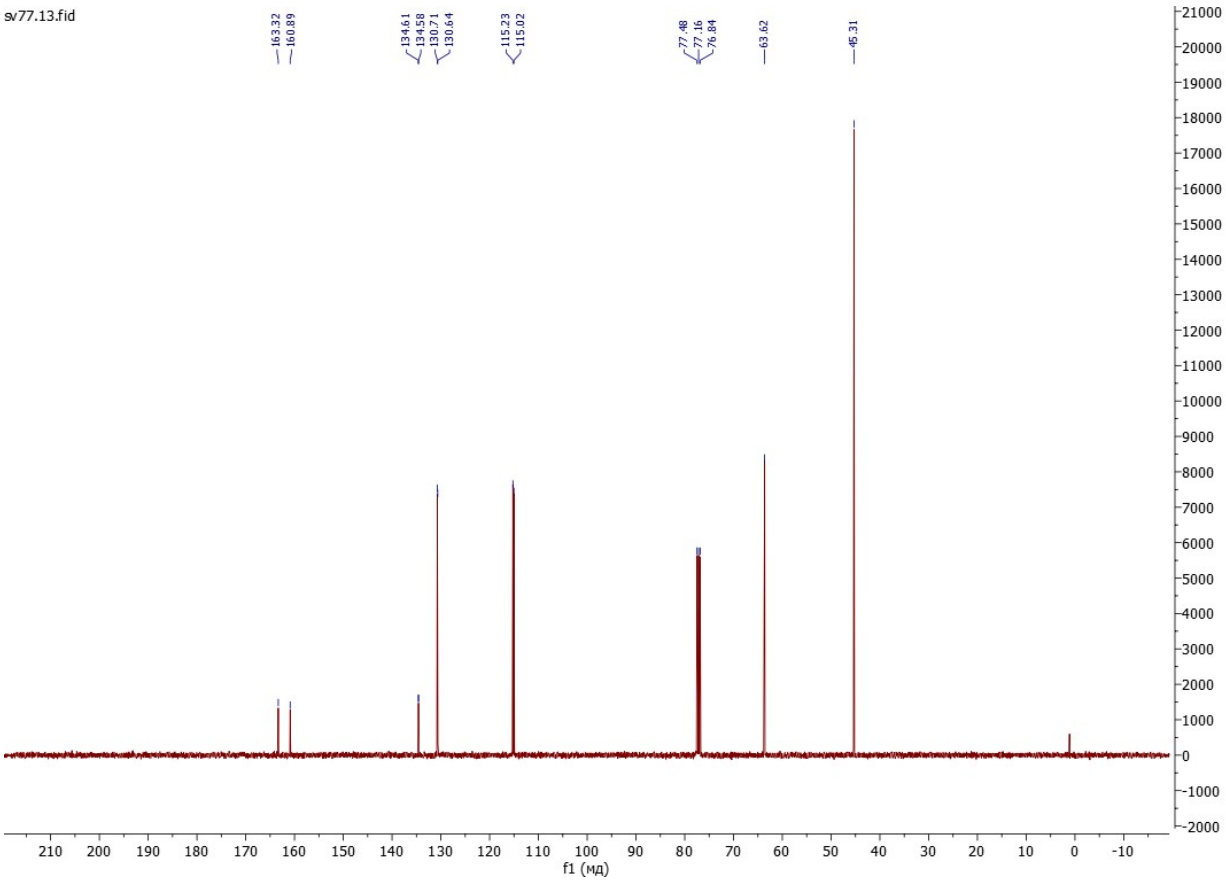




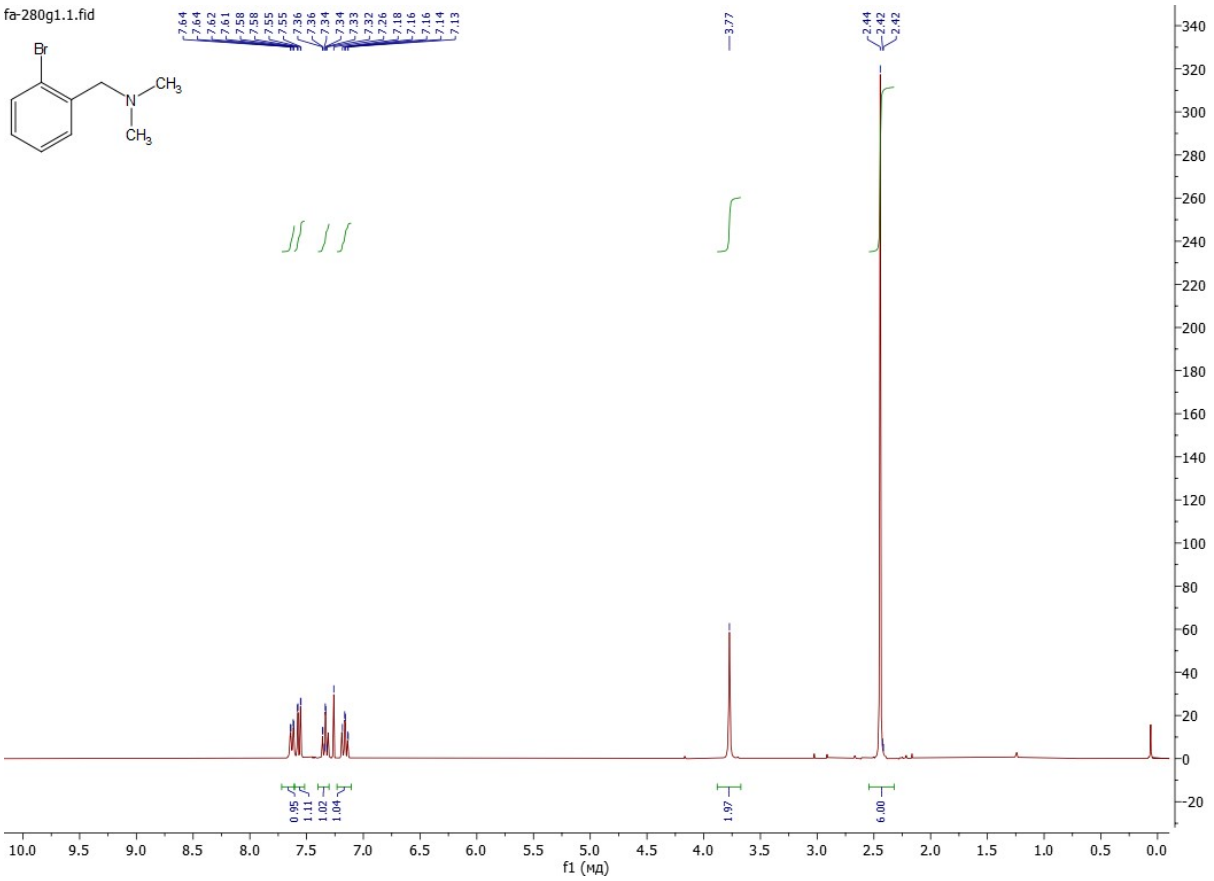
1-(4-fluorophenyl)-N,N-dimethylmethanamine (3)

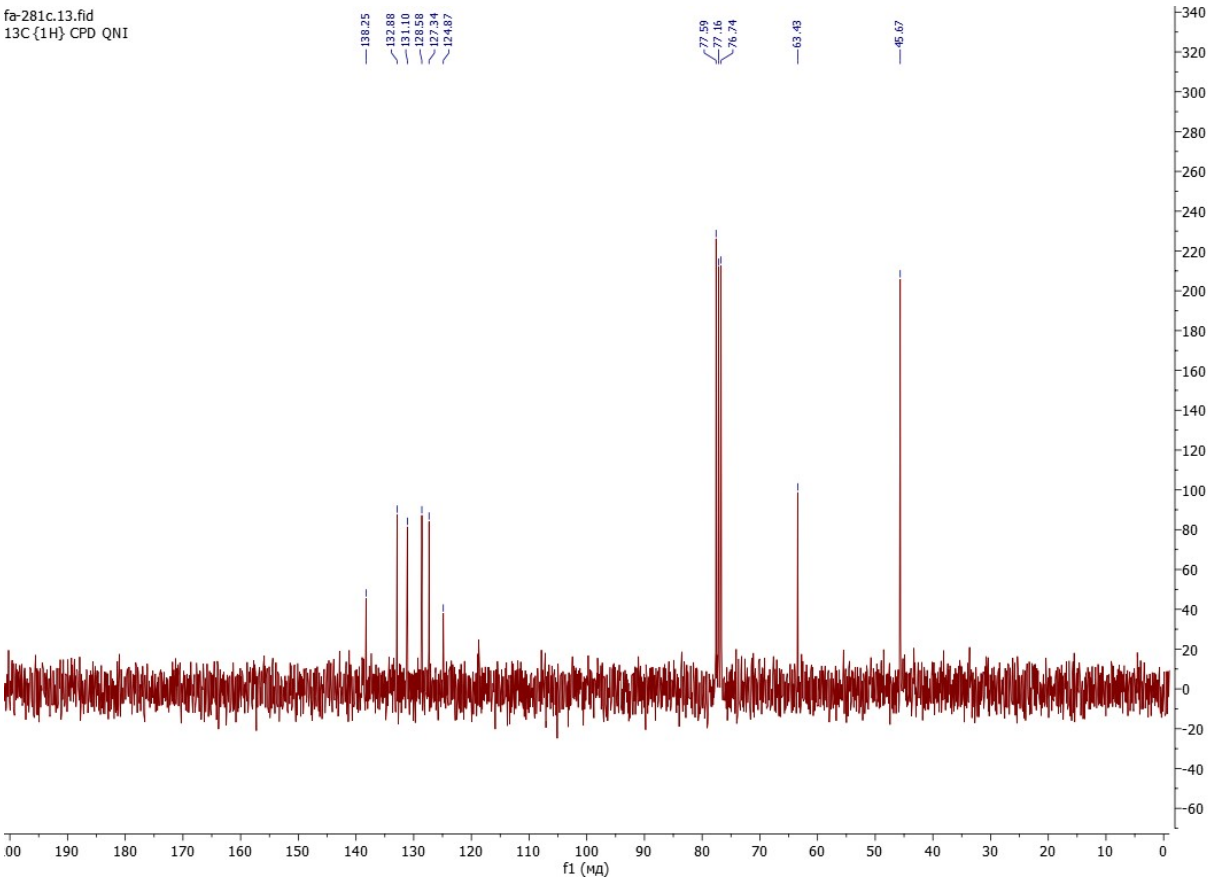

fa280a.1.fid
1H NMR (300 MHz, CD3OD) δ: 7.3 - 7.2 (m, 2H), 7.0 (t, 2H), 6.4 (s, 2H), 2.2 (s, 6H).
7.26
7.25
7.24
7.23
7.22
7.21
7.20
7.02
7.00
6.99
6.98
6.96
6.95
6.94
6.93
6.92
3.37
2.21
2.20
2.18
CH3
N
CH3
F
2.17
2.15
1.98
6.00
f1 (ppm)


fa280a.19.fid
F19CPD
-115.64
f1 (ppm)

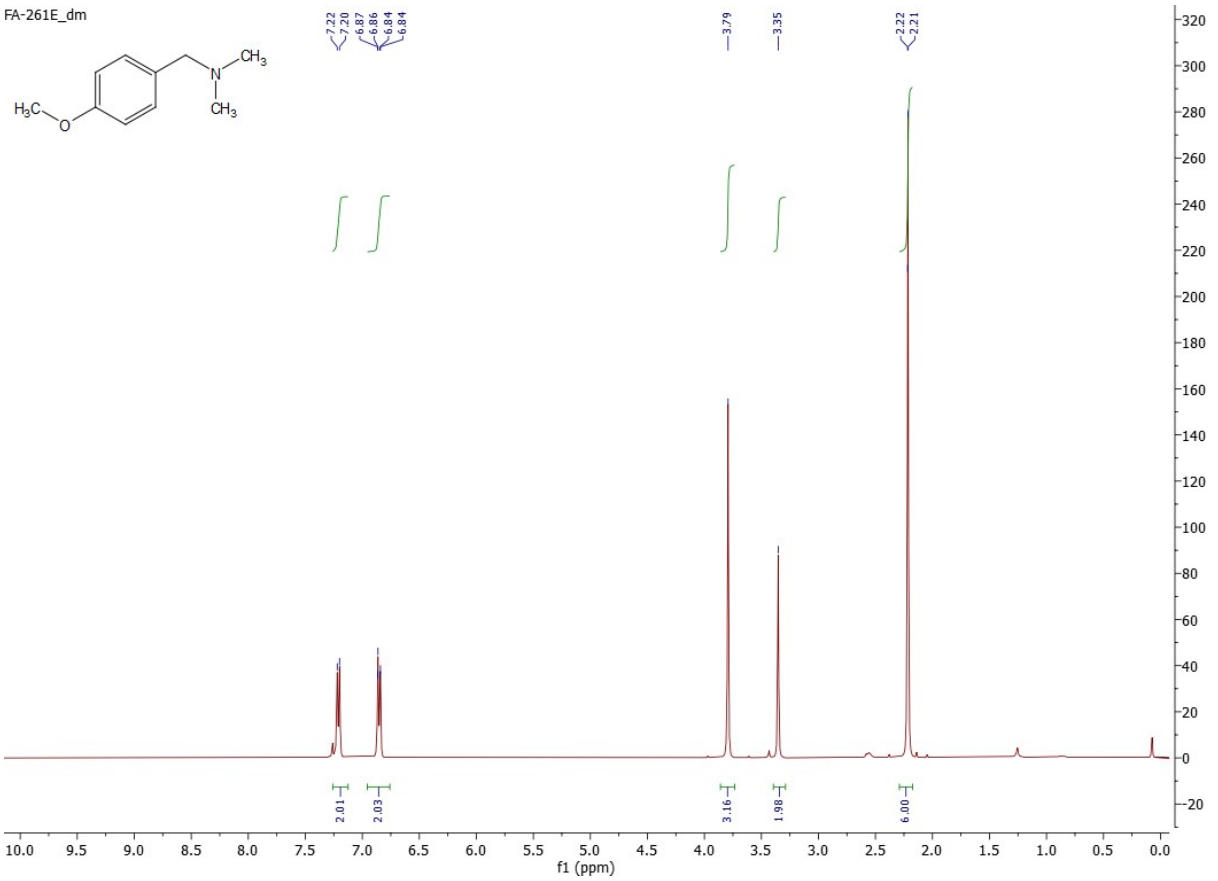

sv77.13.fid
163.32
160.89
134.61
134.58
130.71
130.64
115.23
115.02
77.48
77.16
76.84
63.62
45.31
f1 (мд)

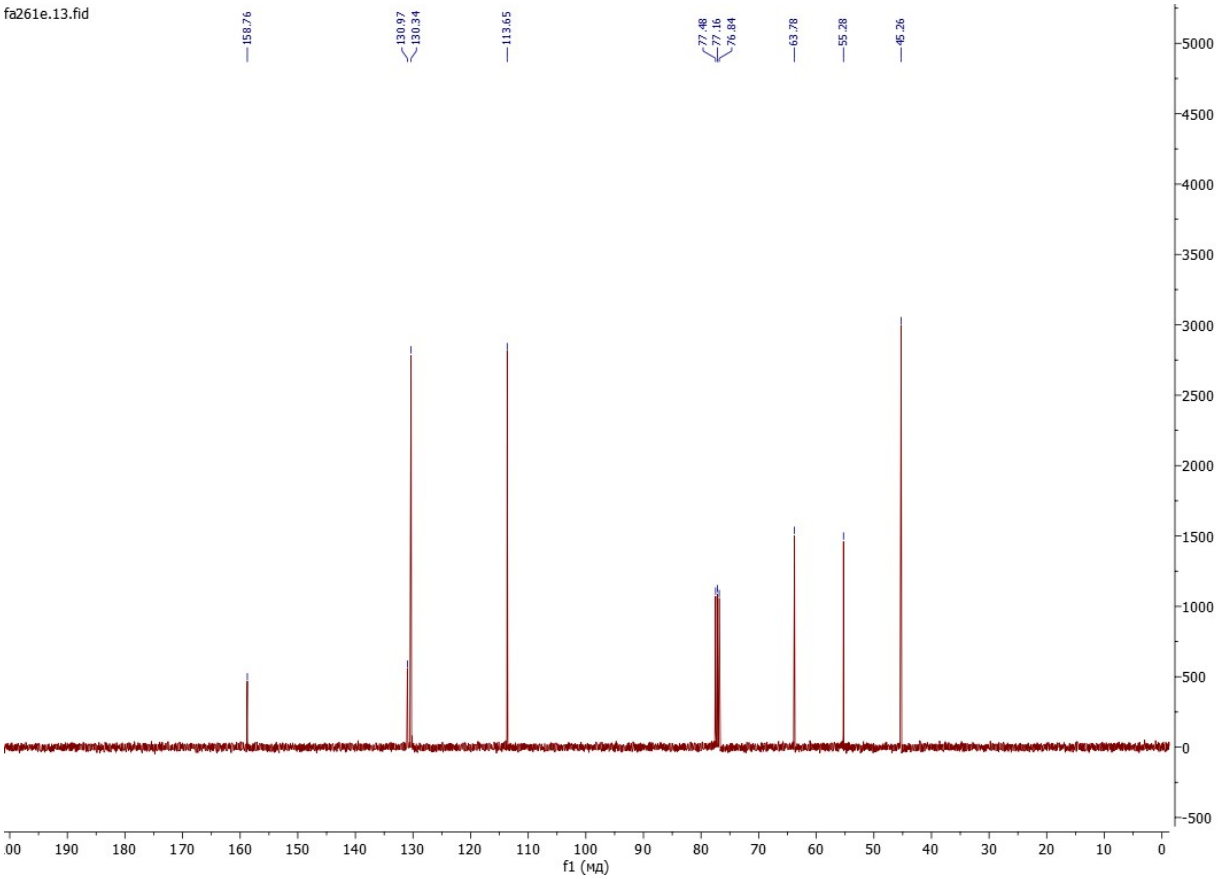
1-(2-bromophenyl)-N,N-dimethylmethanamine (4)



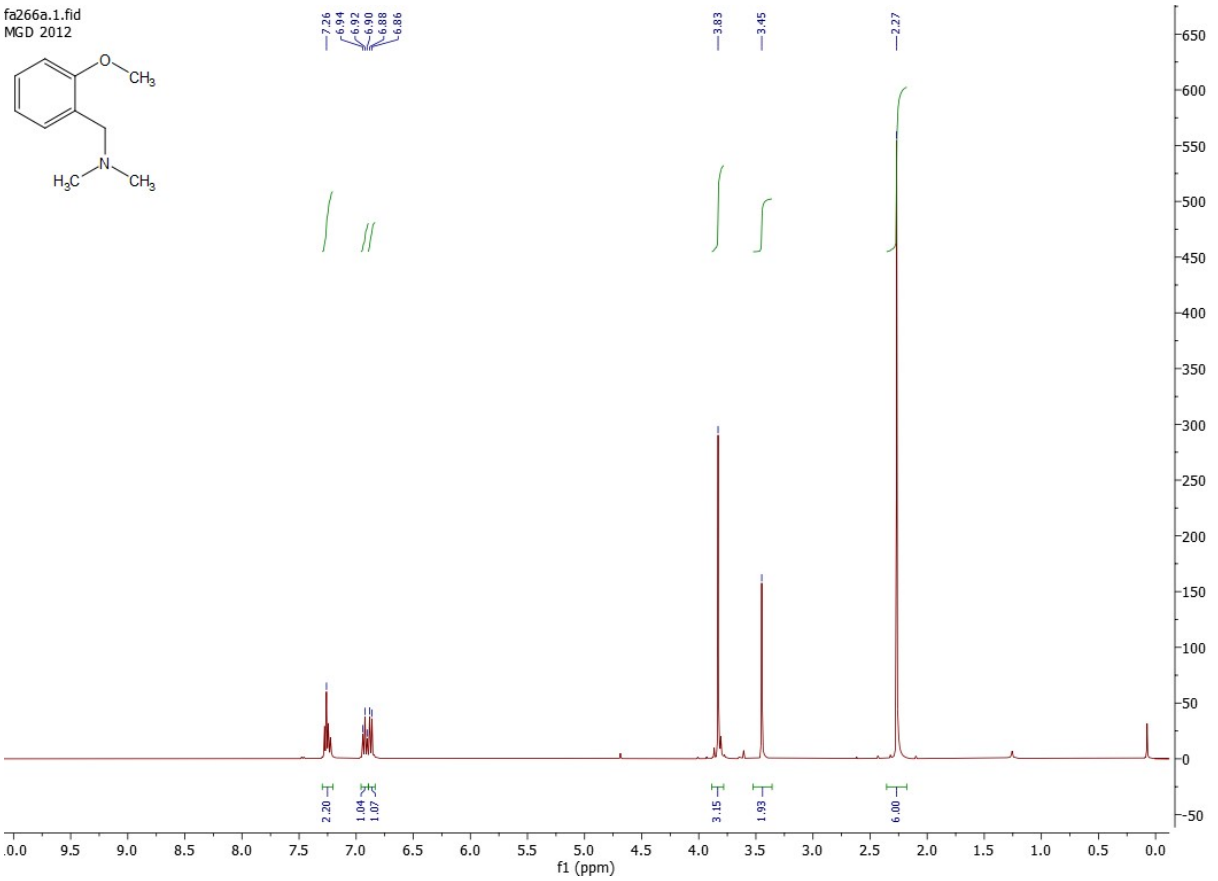


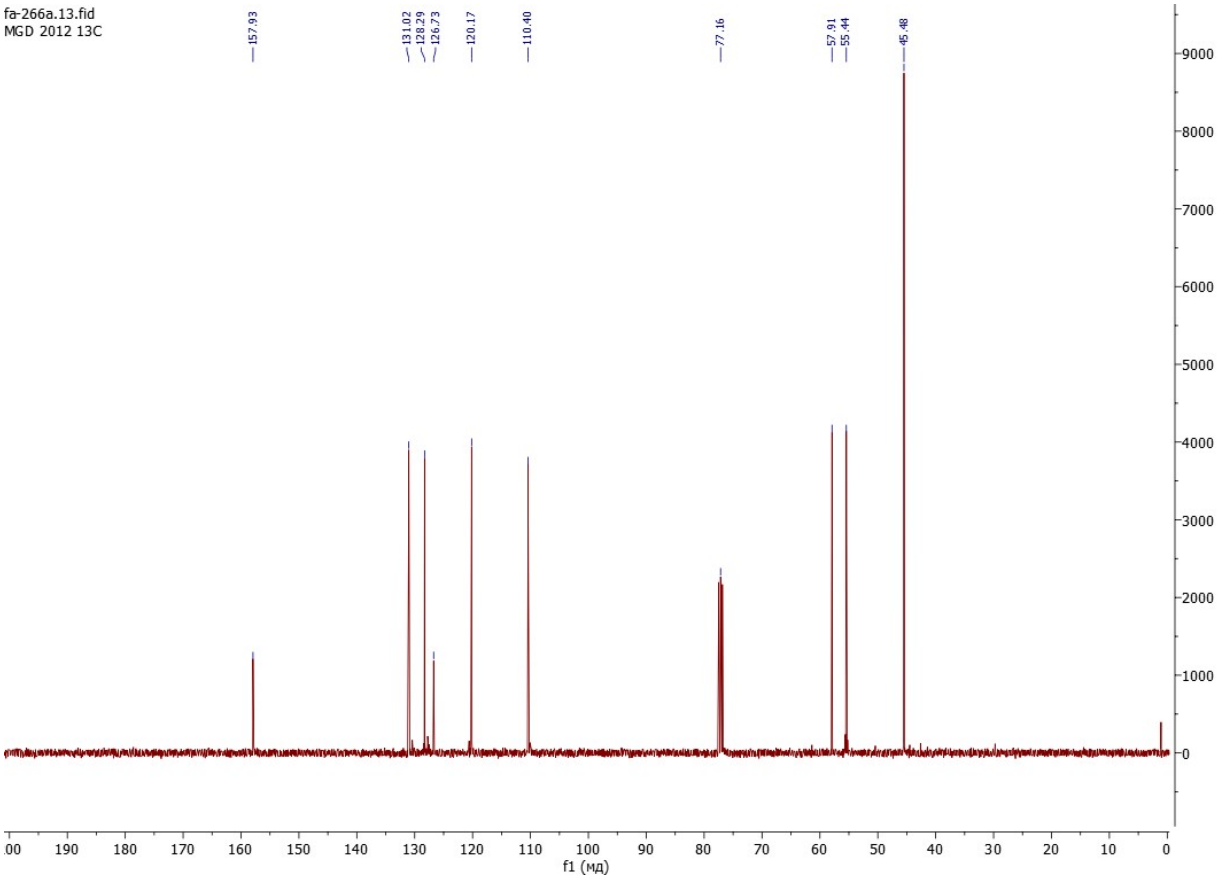
1-(4-methoxyphenyl)-N,N-dimethylmethanamine (5)



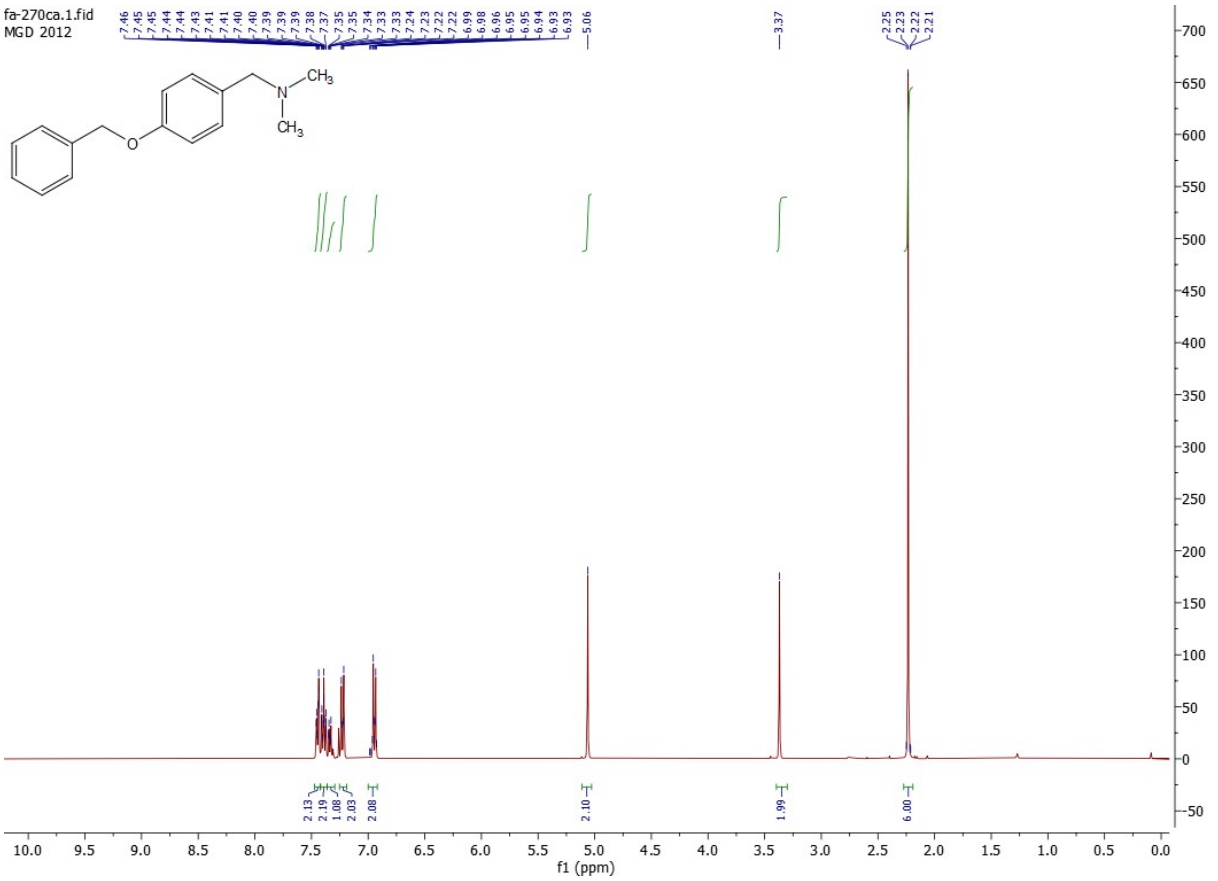


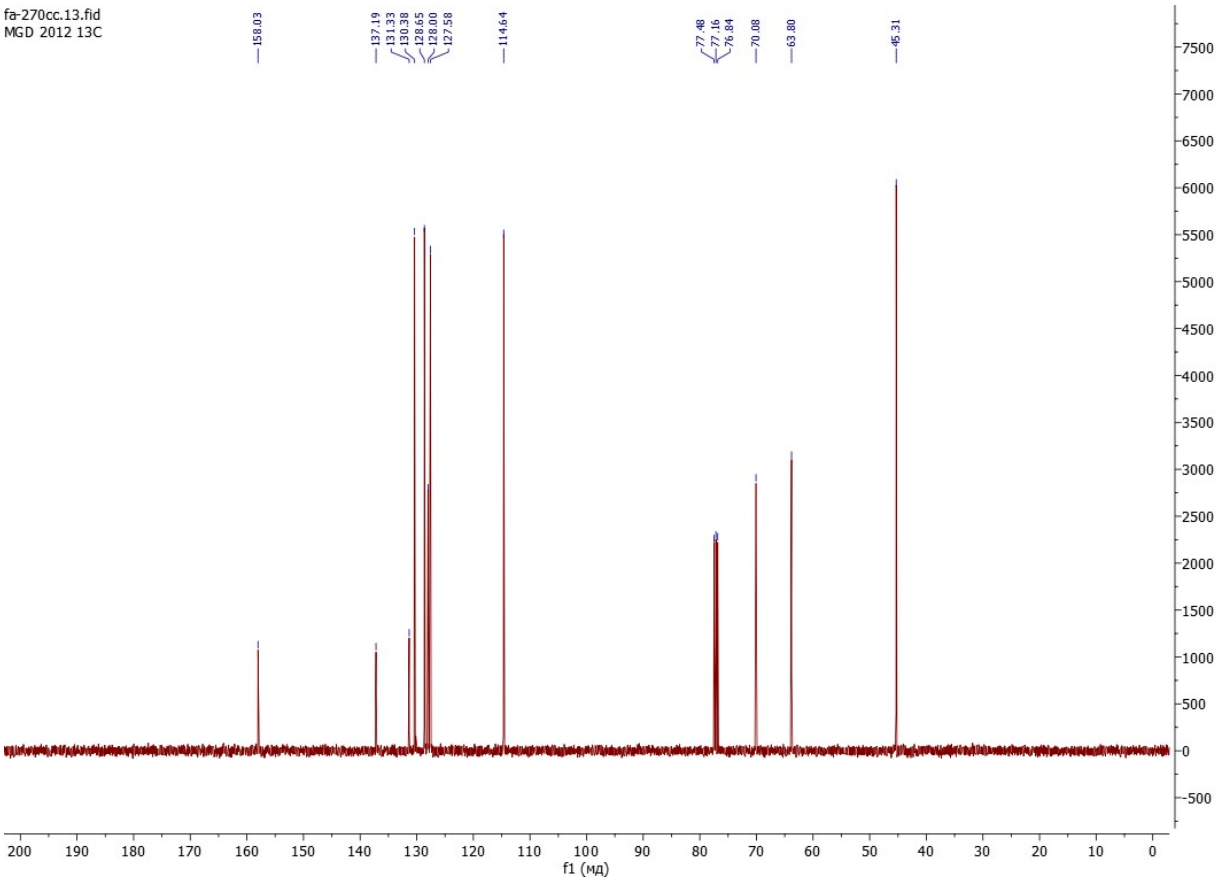
1-(2-methoxyphenyl)-N,N-dimethylmethanamine (6)



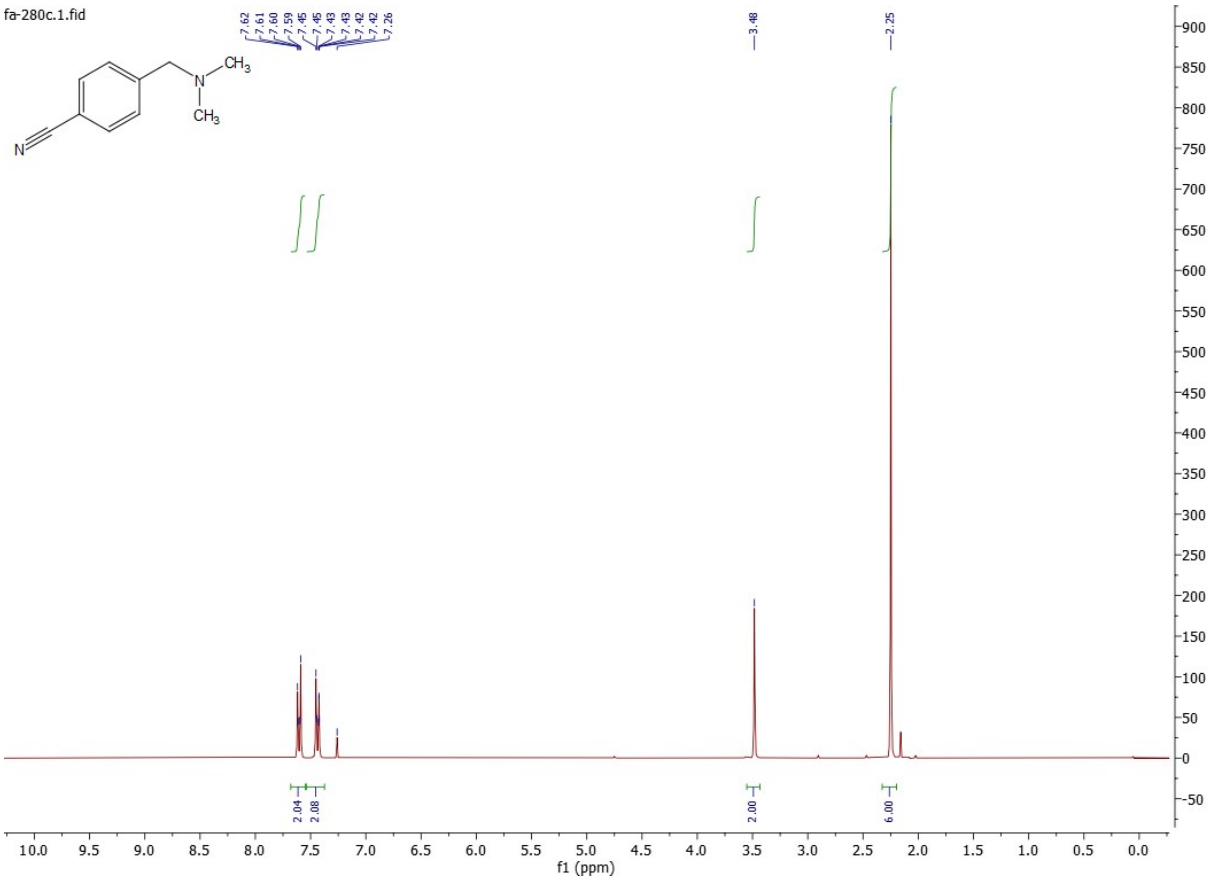


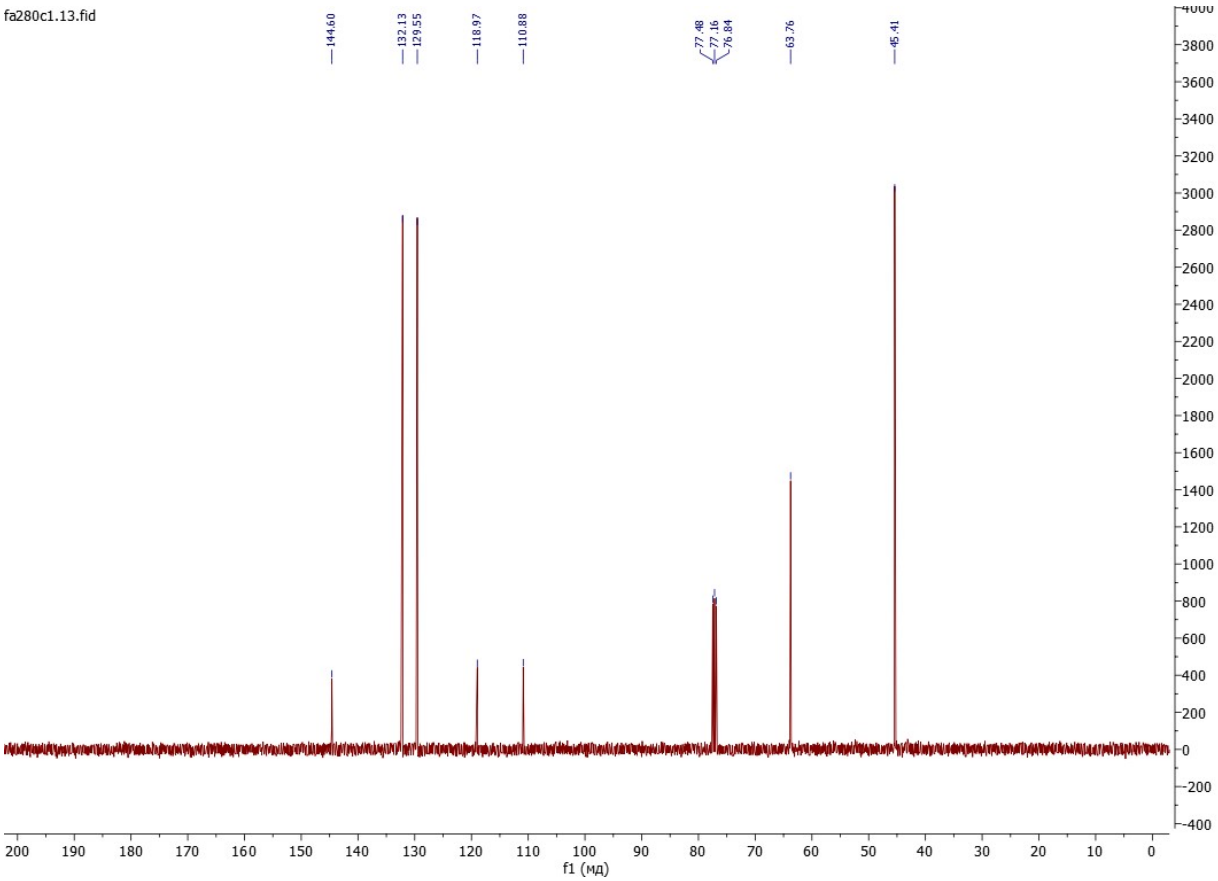
1-(4-benzyloxyphenyl)-N,N-dimethylmethanamine (7)



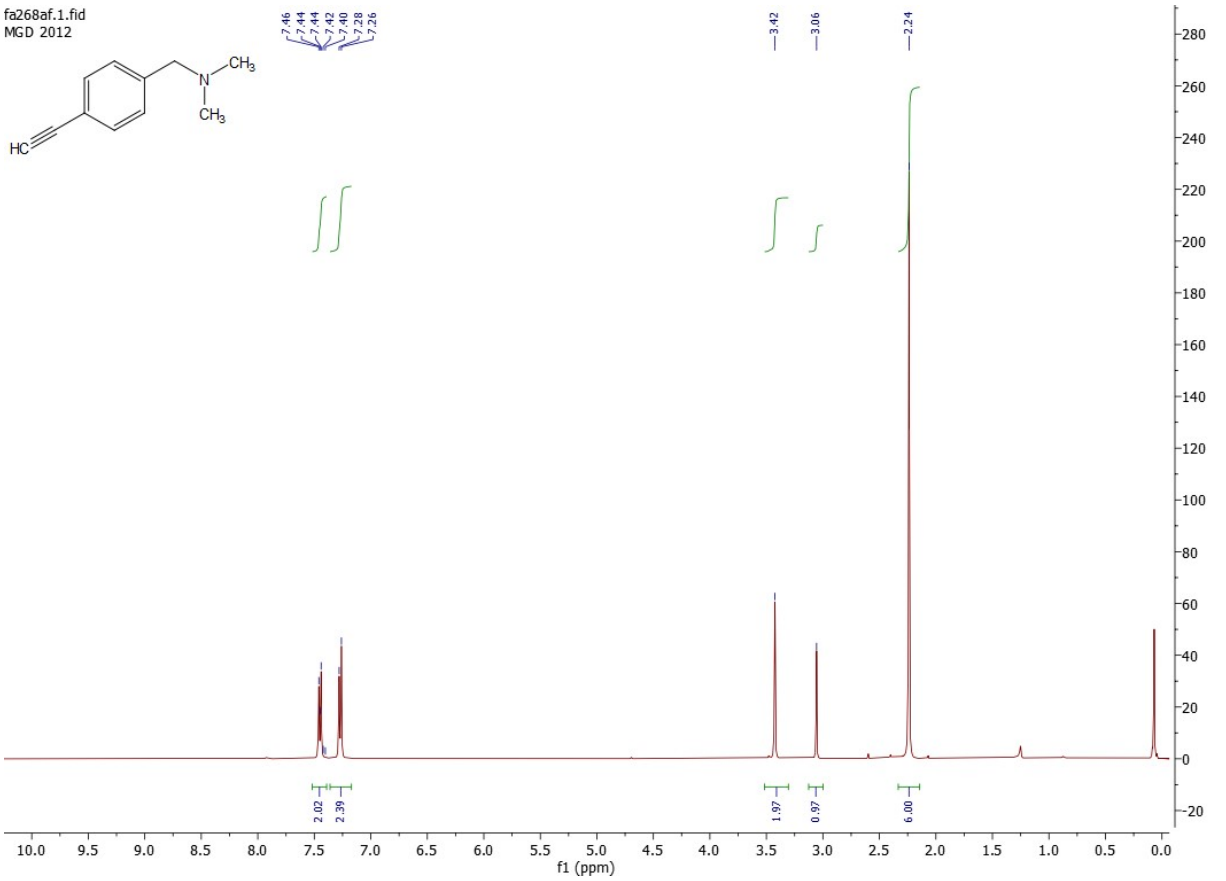


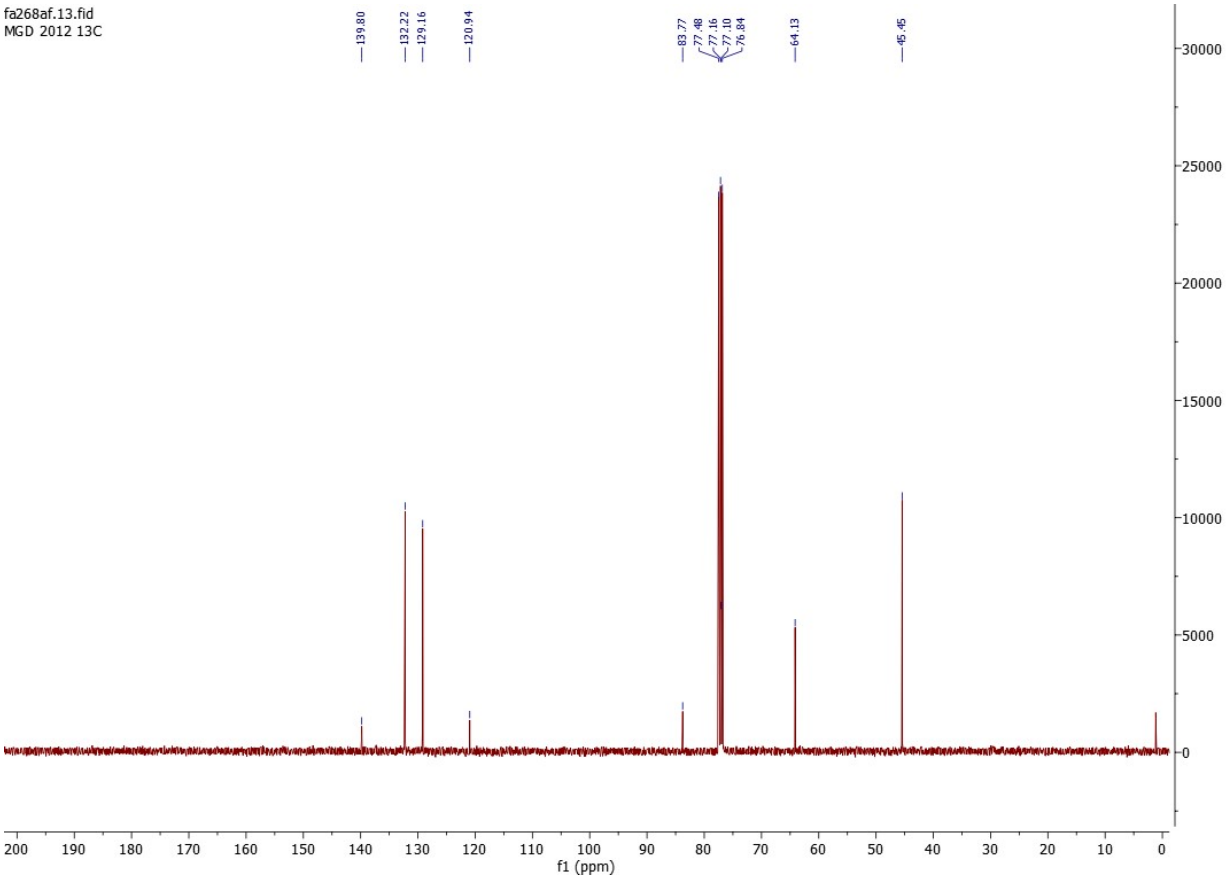
4-((dimethylamino)methyl)benzonitrile (8)



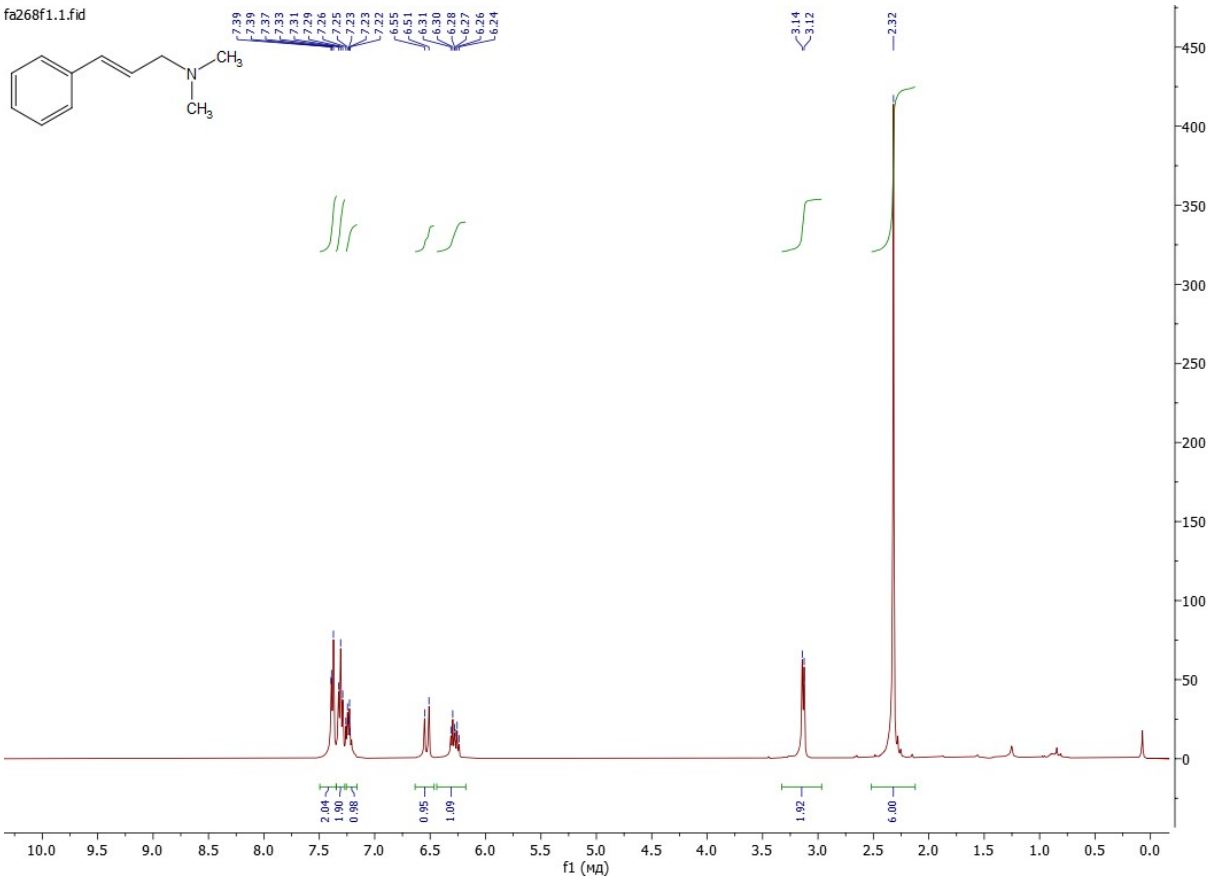


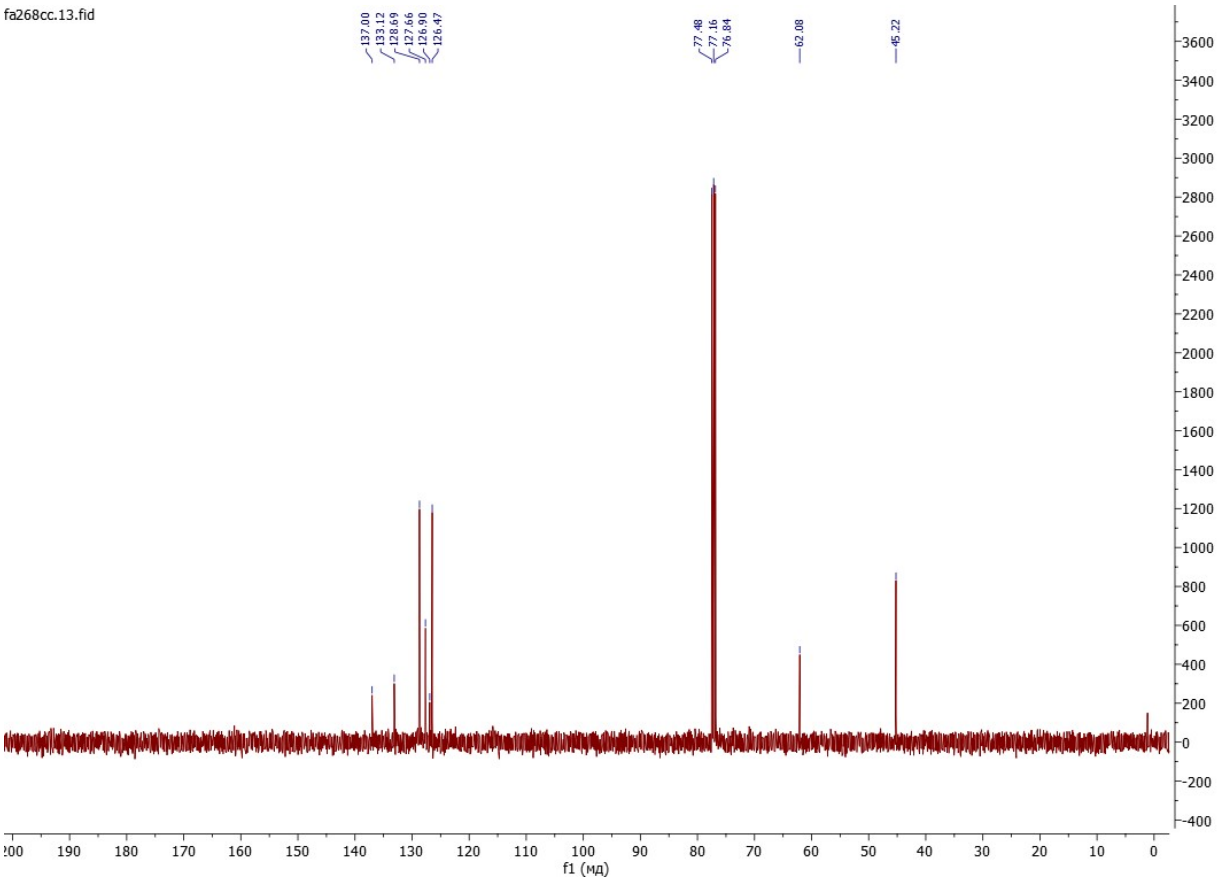
1-(4-ethynylphenyl)-N,N-dimethylmethanamine (9)



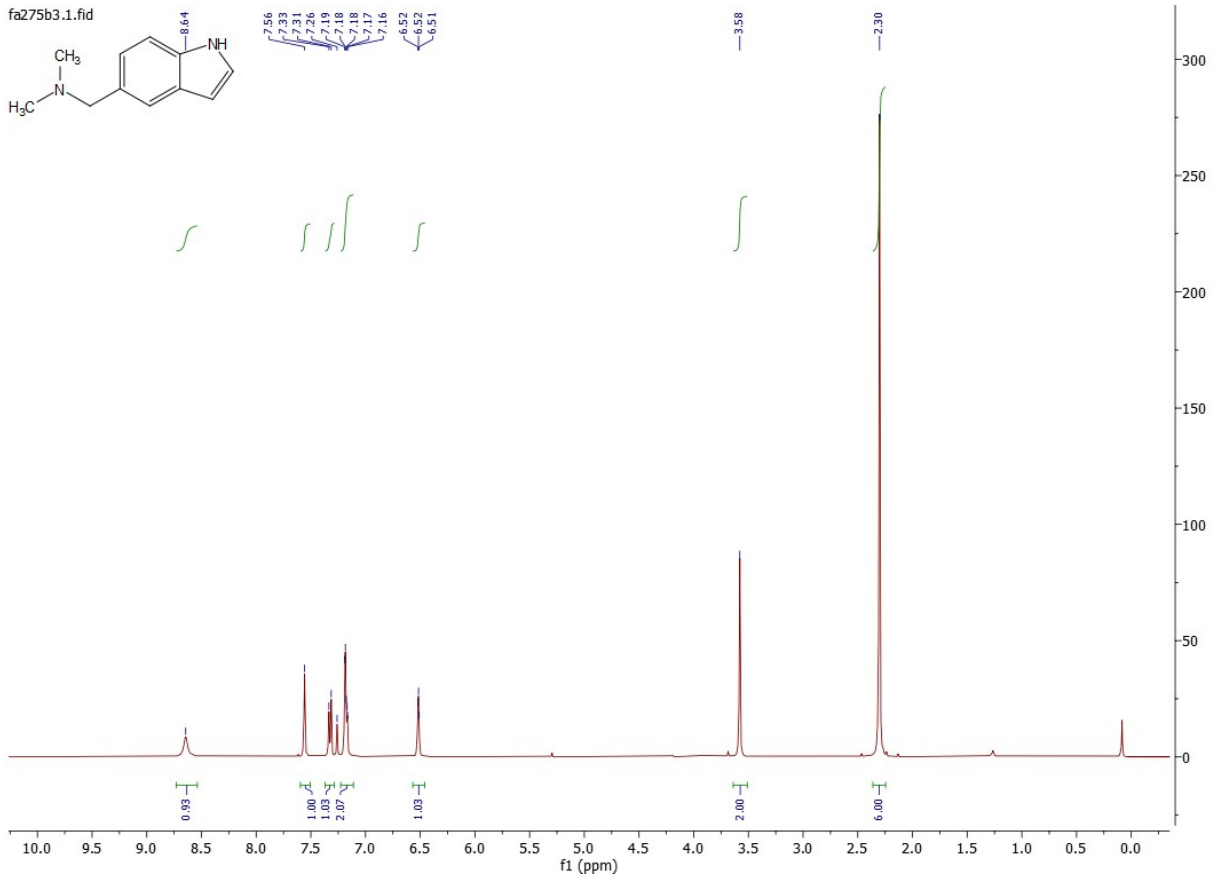


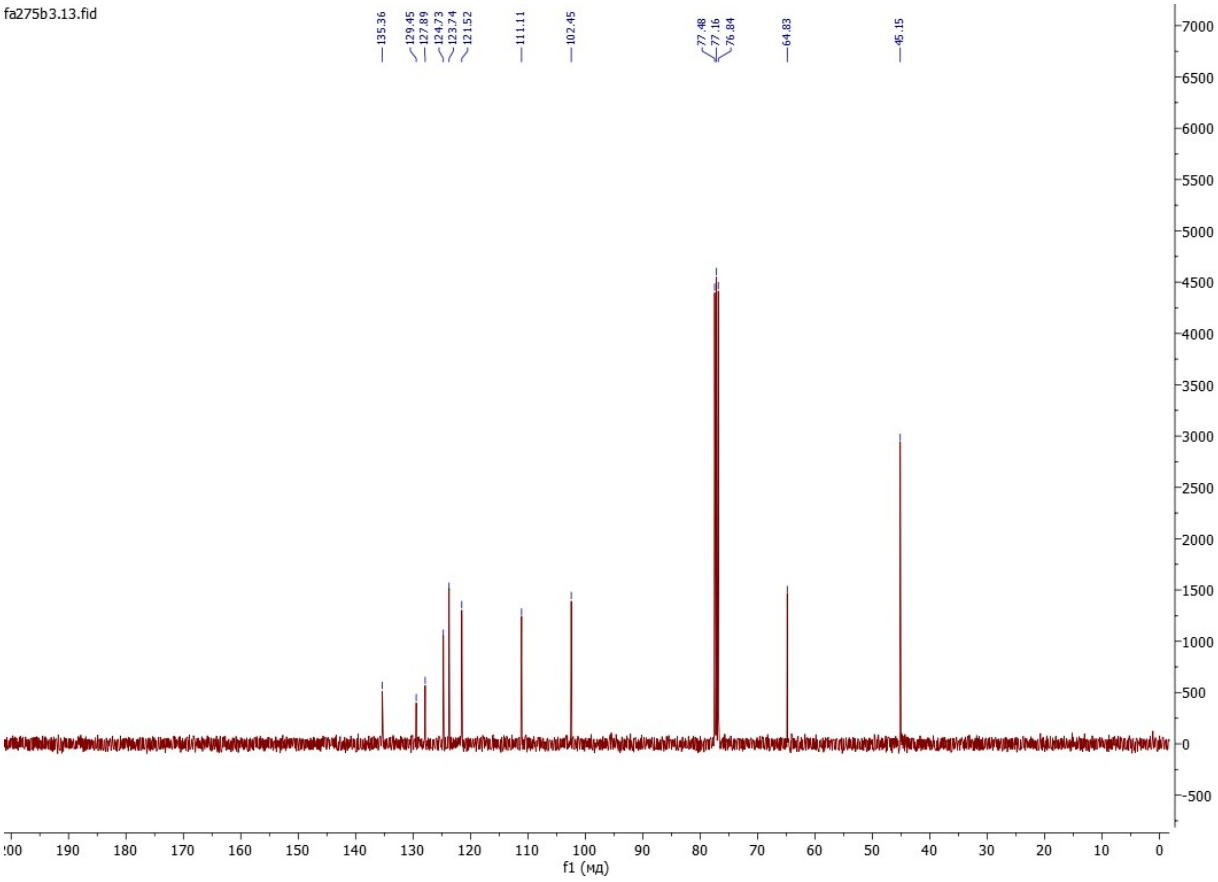
(E)-N,N-dimethyl-3-phenylprop-2-en-1-amine (10)



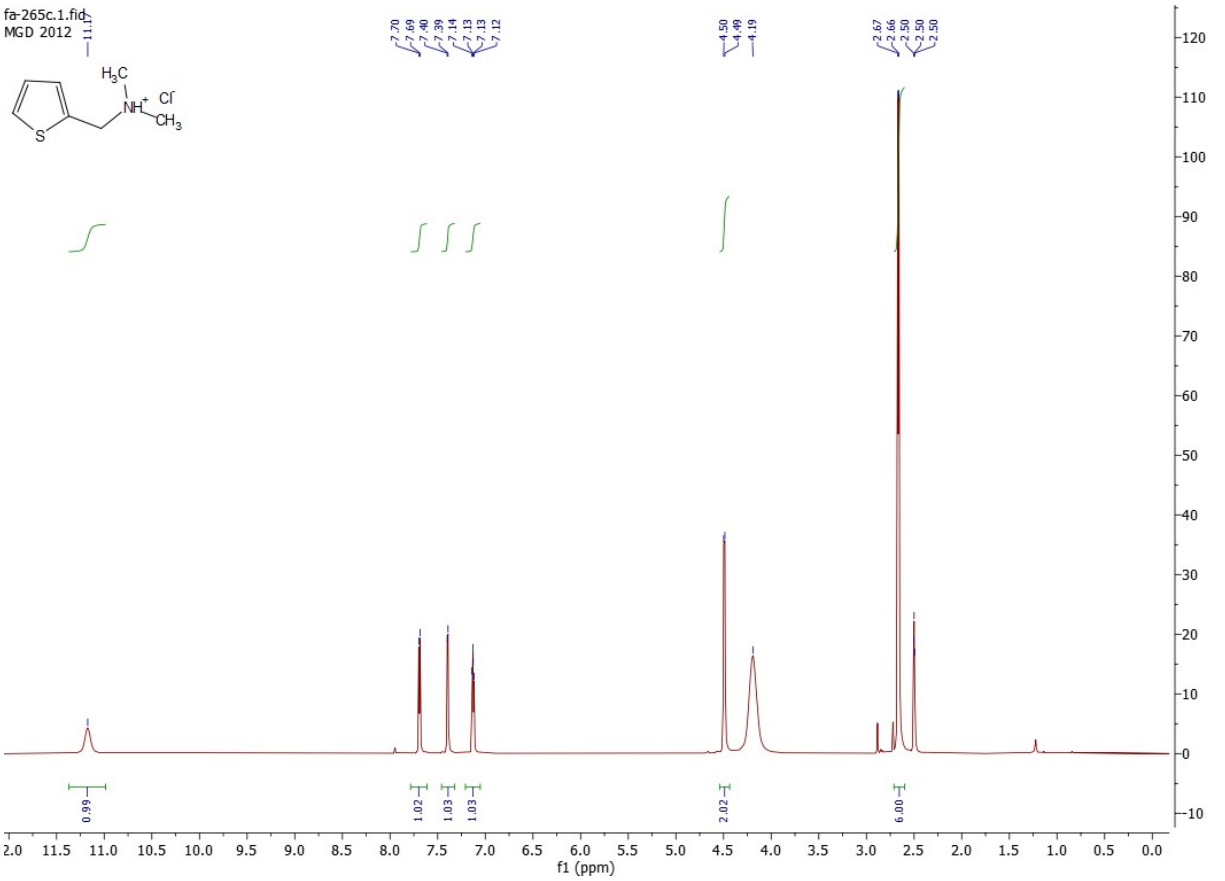


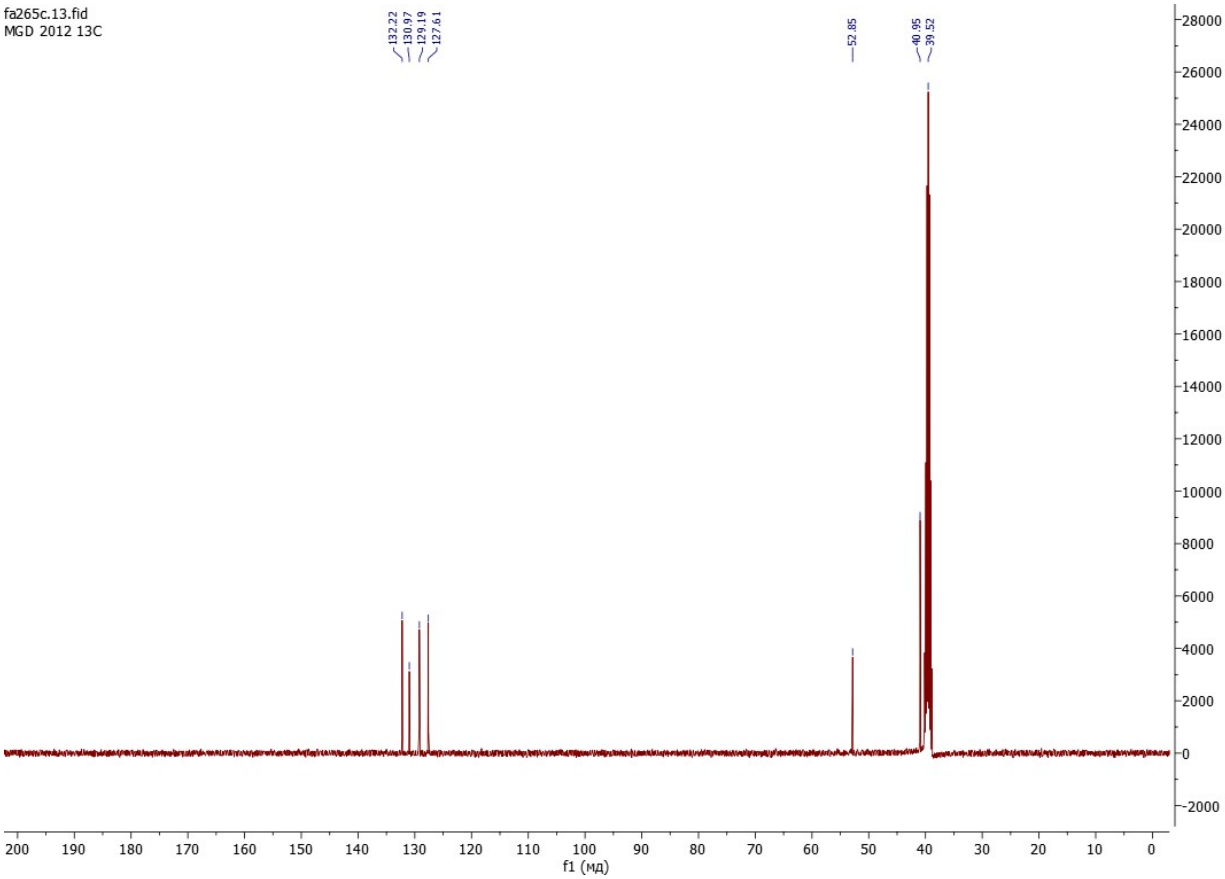
1-(1H-indol-5-yl)-N,N-dimethylmethanamine (11)

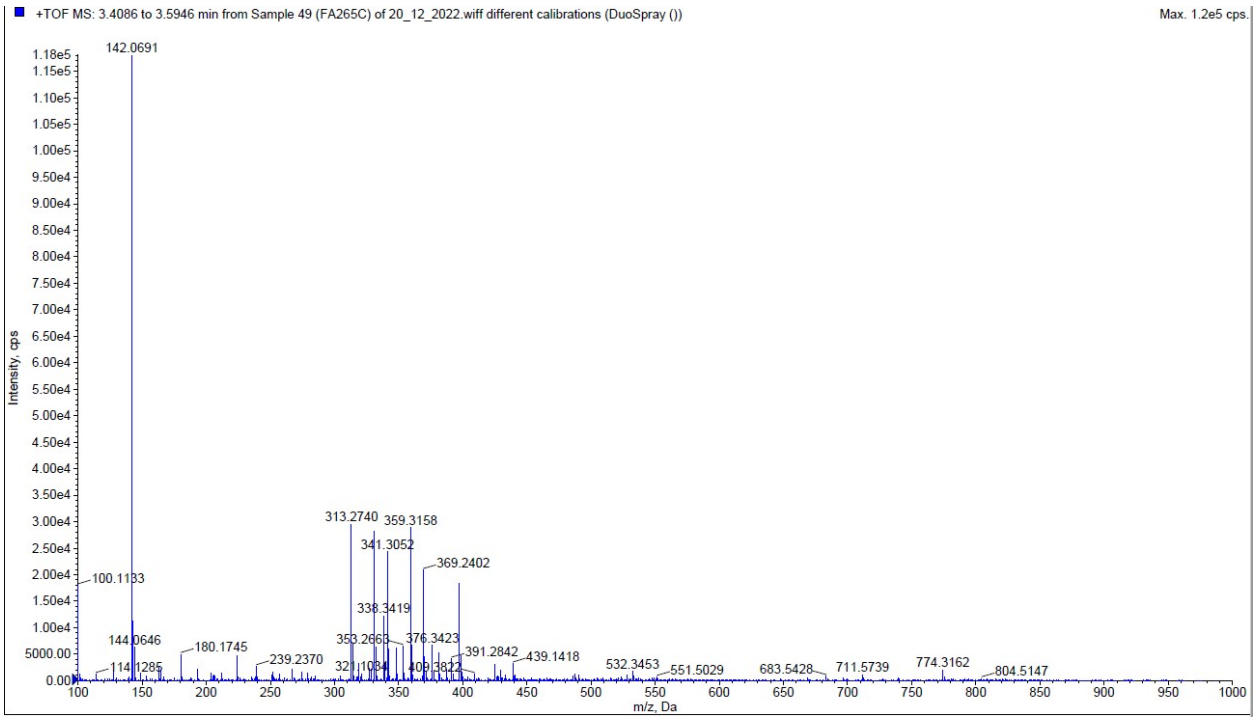




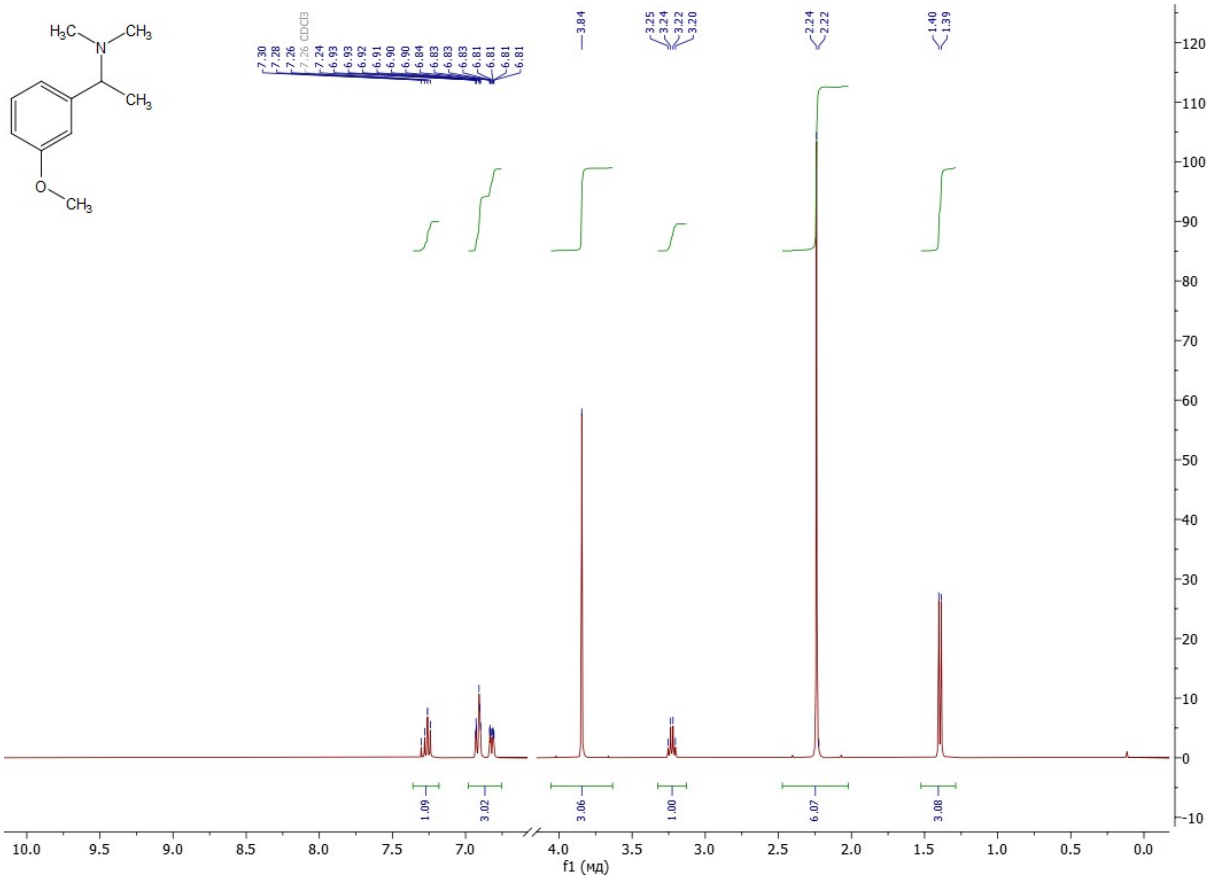
N,N-dimethyl-1-(thiophen-2-yl)methanamine hydrochloride (12)

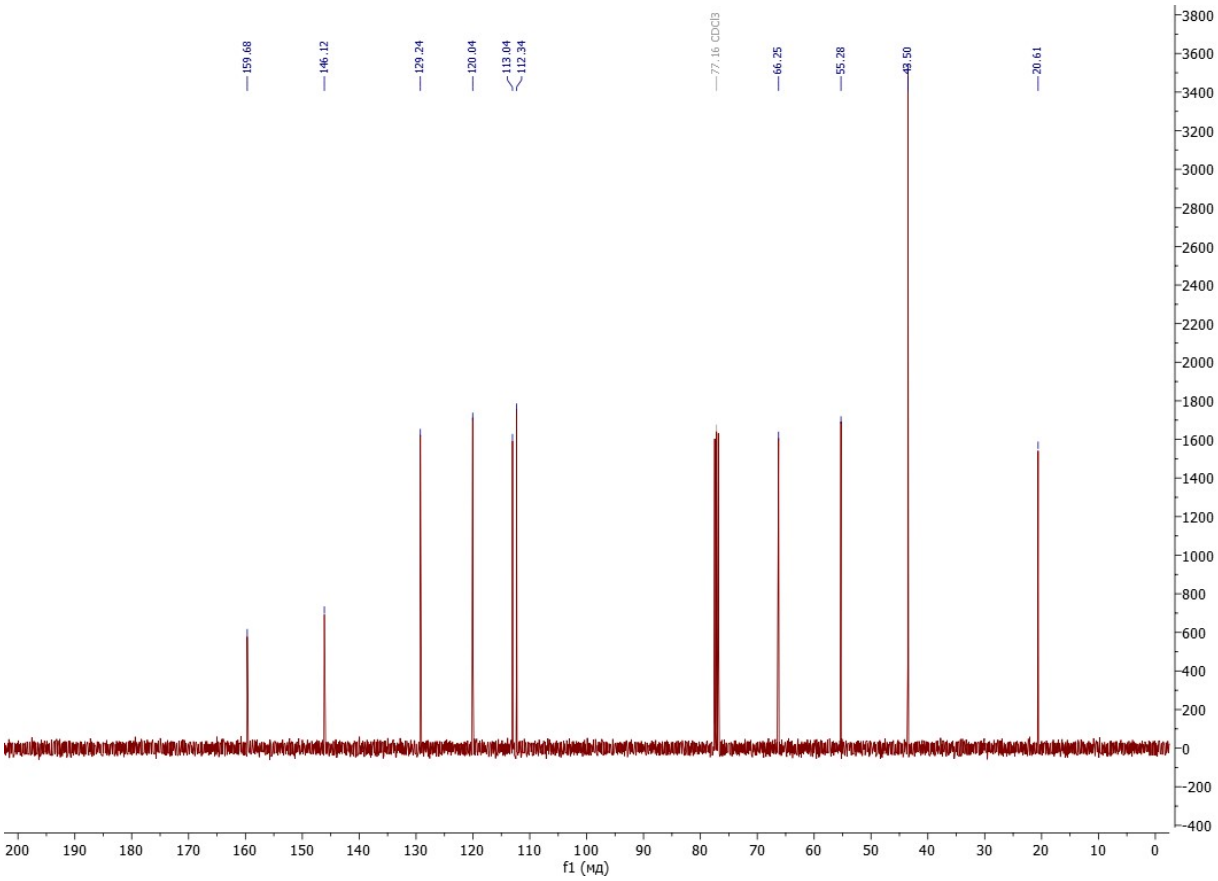




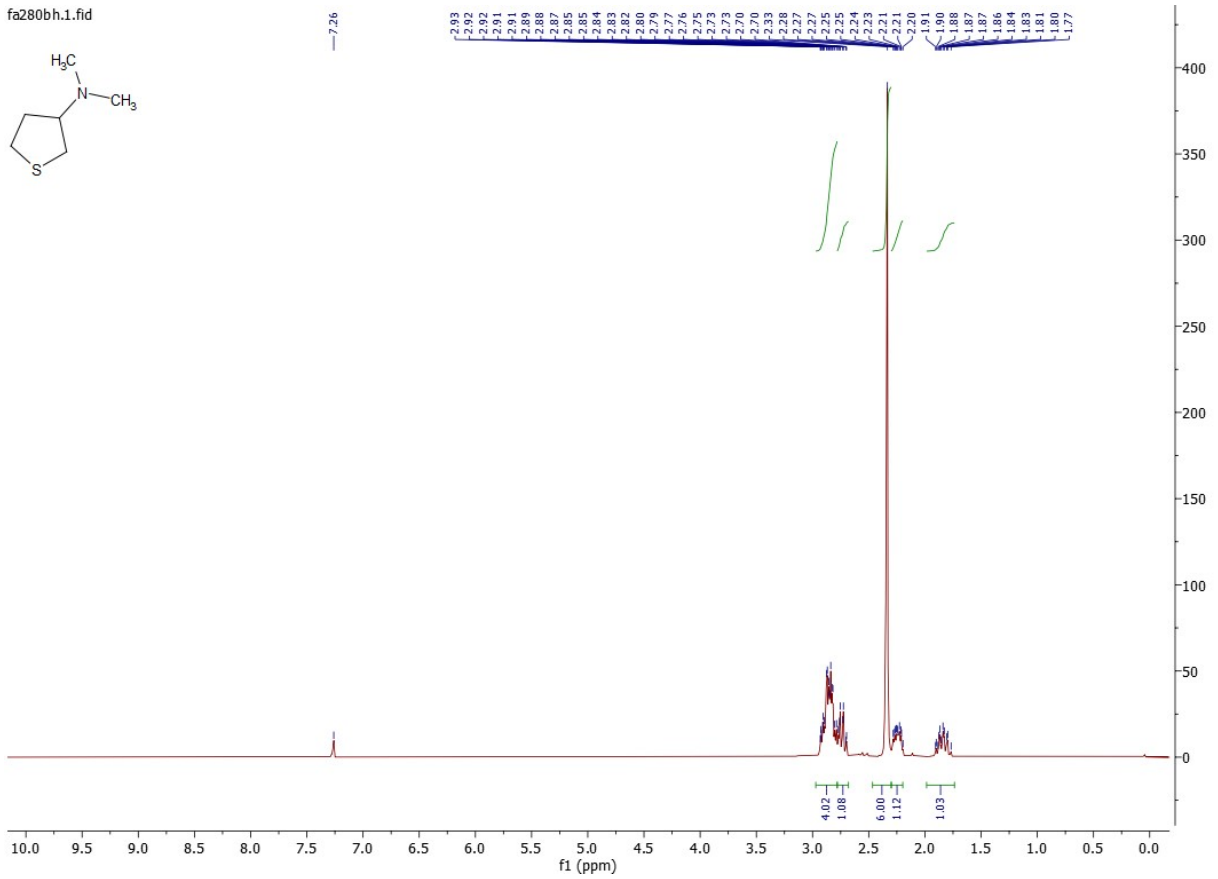

+TOF MS: 3.4086 to 3.5946 min from Sample 49 (FA265C) of 20_12_2022.wiff different calibrations (DuoSpray ())
Max. 1.2e5 cps.
Intensity, cps
m/z, Da
142.0691
313.2740
359.3158
341.3052
369.2402
100.1133
338.3419
144.0646
180.1745
353.2663
376.3423
391.2842
439.1418
239.2370
532.3453
551.5029
683.5428
711.5739
774.3162
804.5147

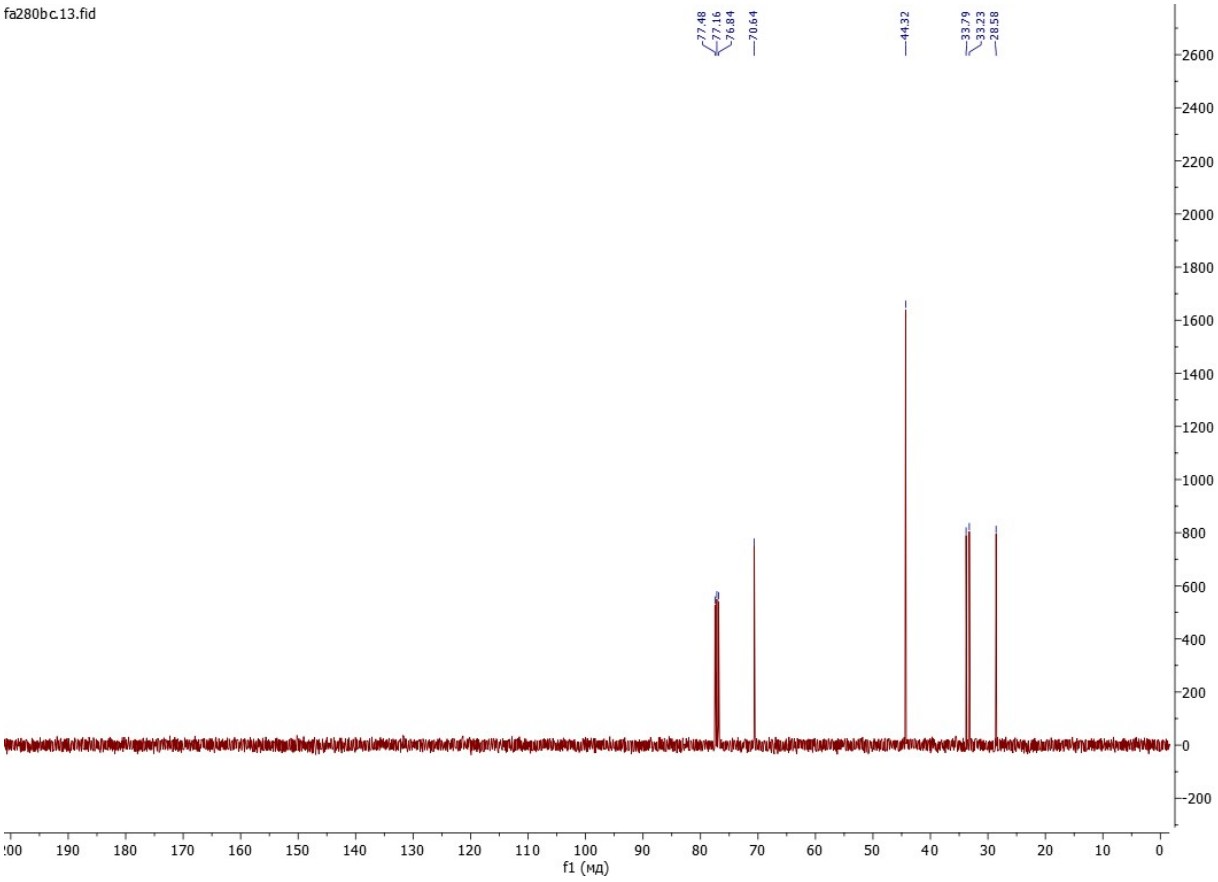
1-(3-methoxyphenyl)-N,N-dimethylethan-1-amine (13)

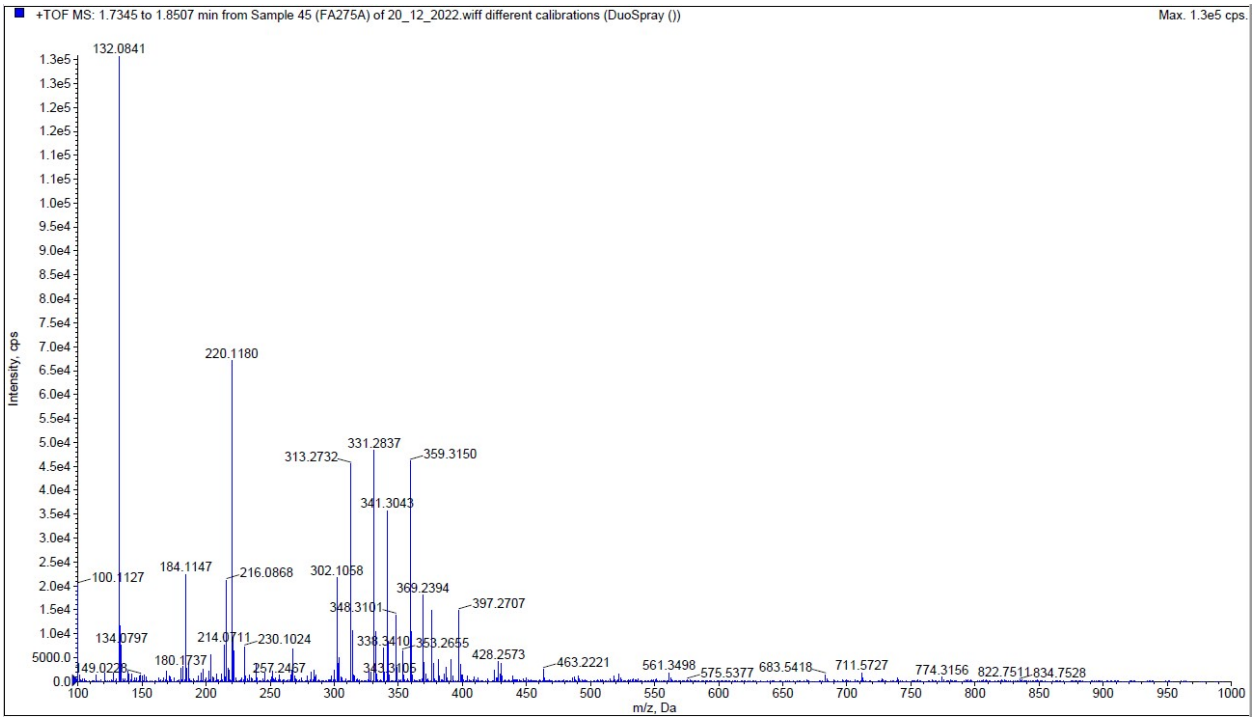




N,N-dimethyltetrahydrothiophen-3-amine (14)

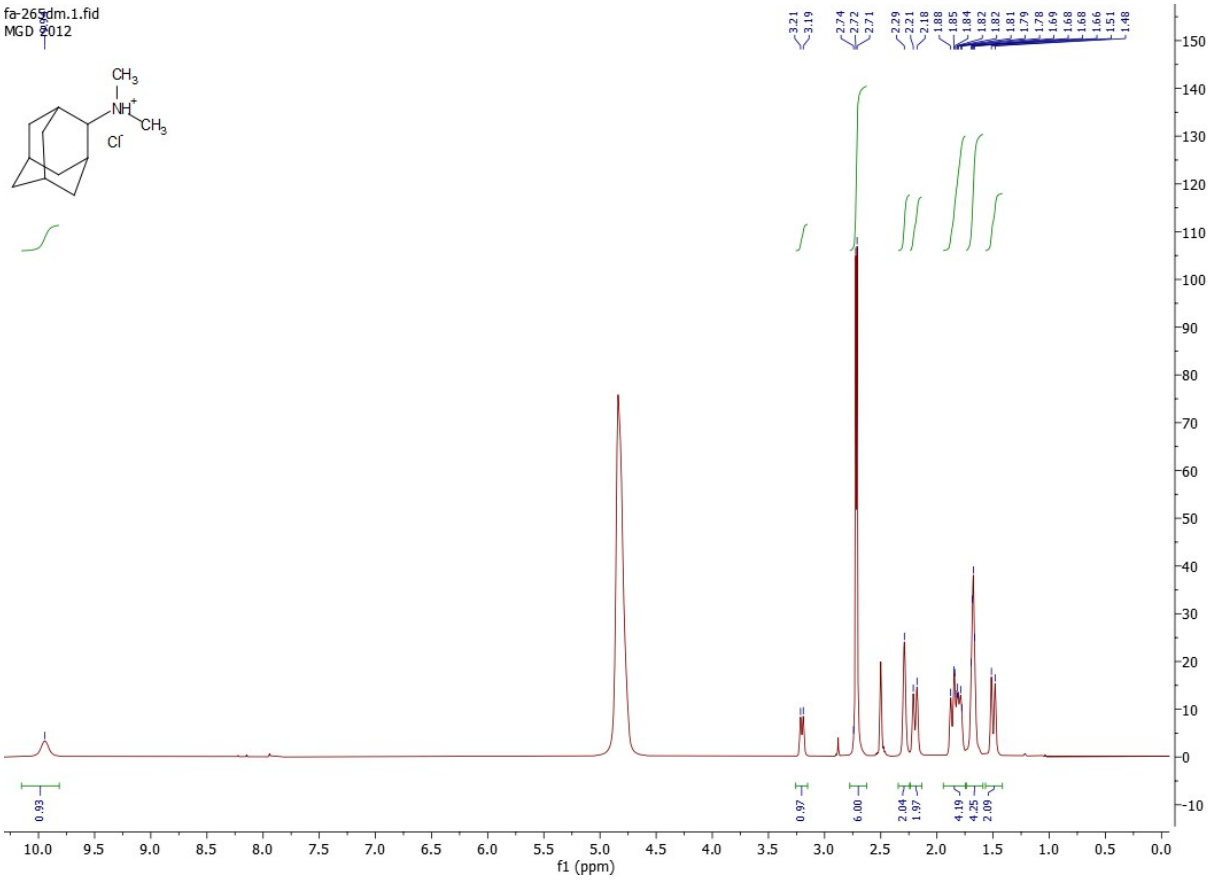




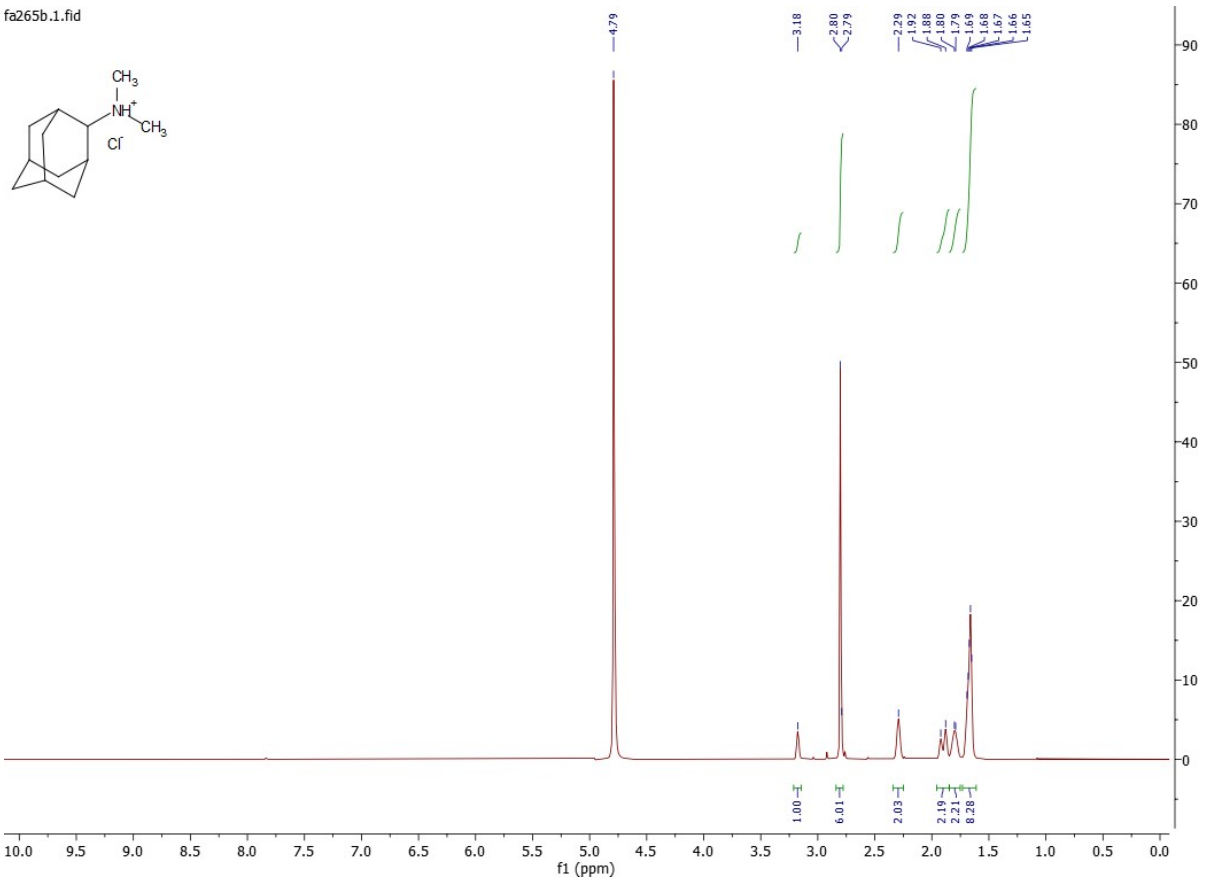

+TOF MS: 1.7345 to 1.8507 min from Sample 45 (FA275A) of 20_12_2022.wiff different calibrations (DuoSpray ())
Max. 1.3e5 cps.
Intensity, cps
m/z, Da
132.0841
220.1180
331.2837
313.2732
359.3150
341.3043
184.1147
216.0868
302.1058
100.1127
369.2394
348.3101
397.2707
134.0797
214.0711
230.1024
338.3410
353.2655
428.2573
180.1737
149.0228
257.2457
343.3105
463.2221
561.3498
575.5377
683.5418
711.5727
774.3156
822.7511
834.7528

N,N-dimethyladamantan-2-amine hydrochloride (15)

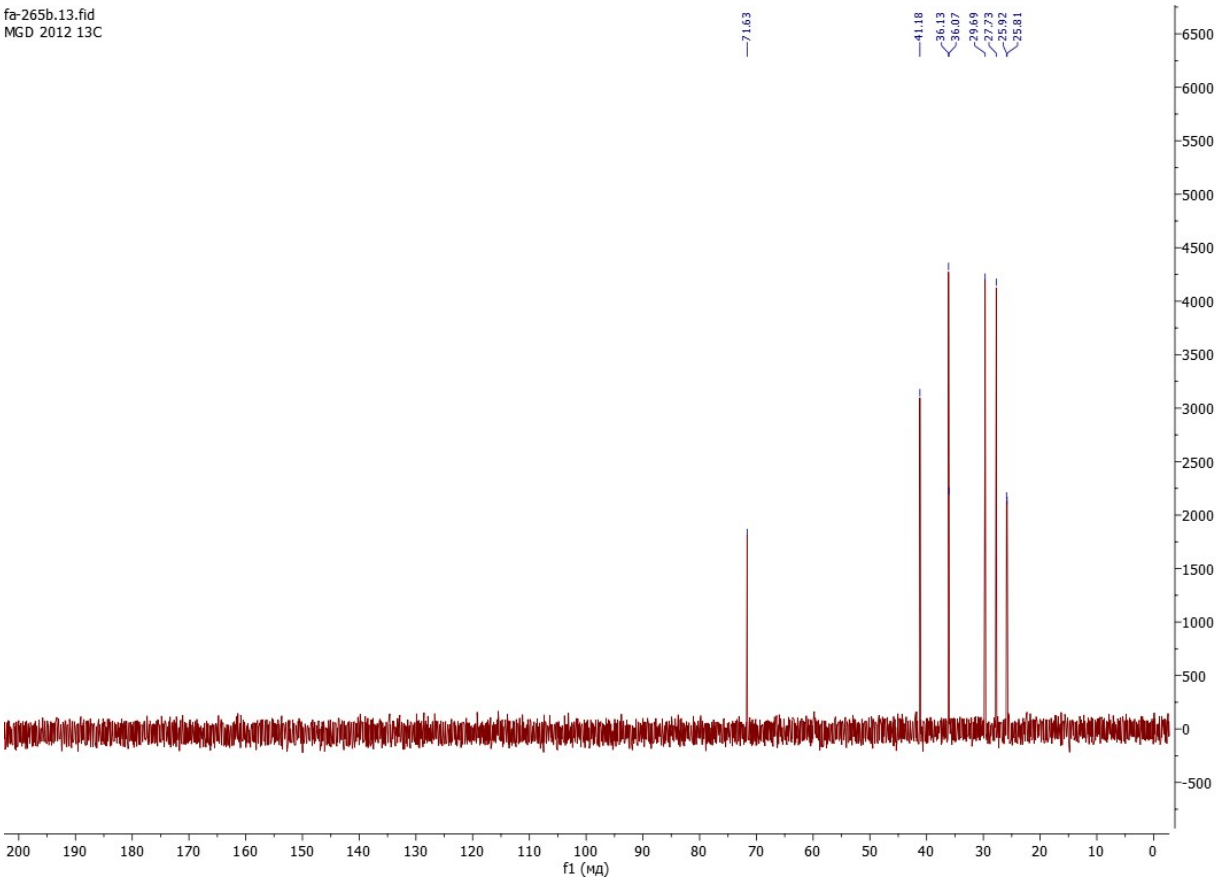
^{1}H-NMR in DMSO:

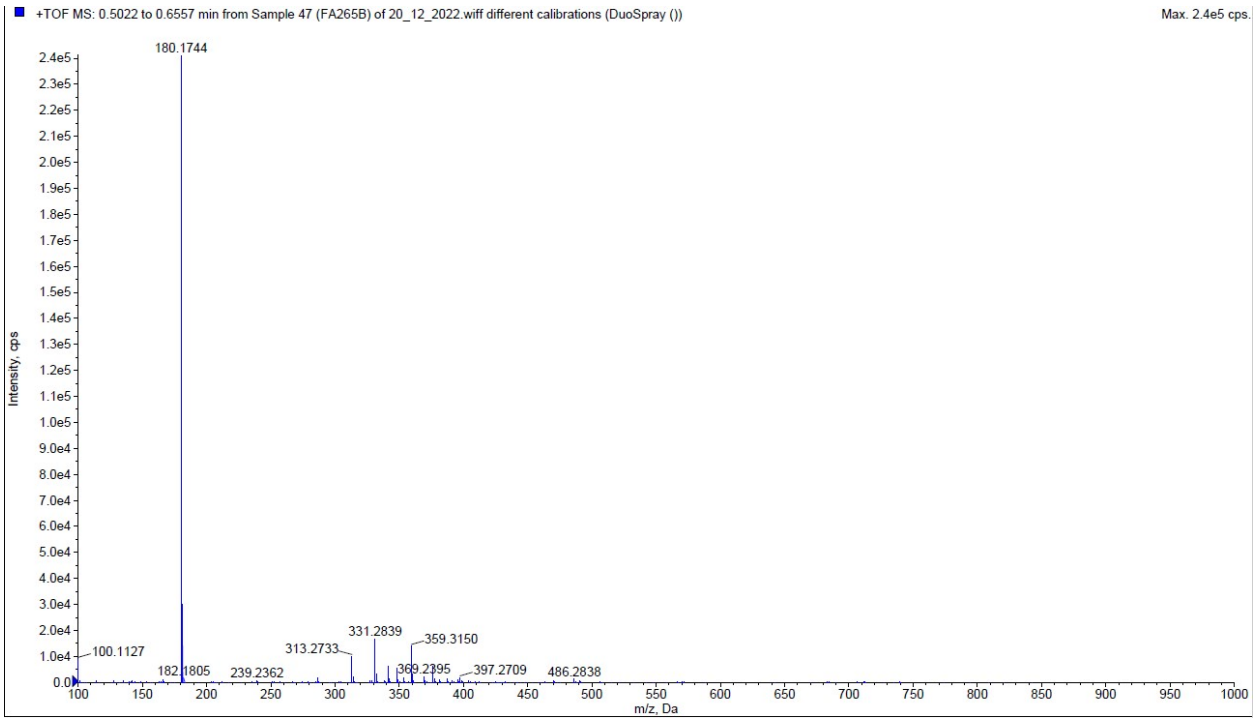


^{1}H-NMR in D_2O:

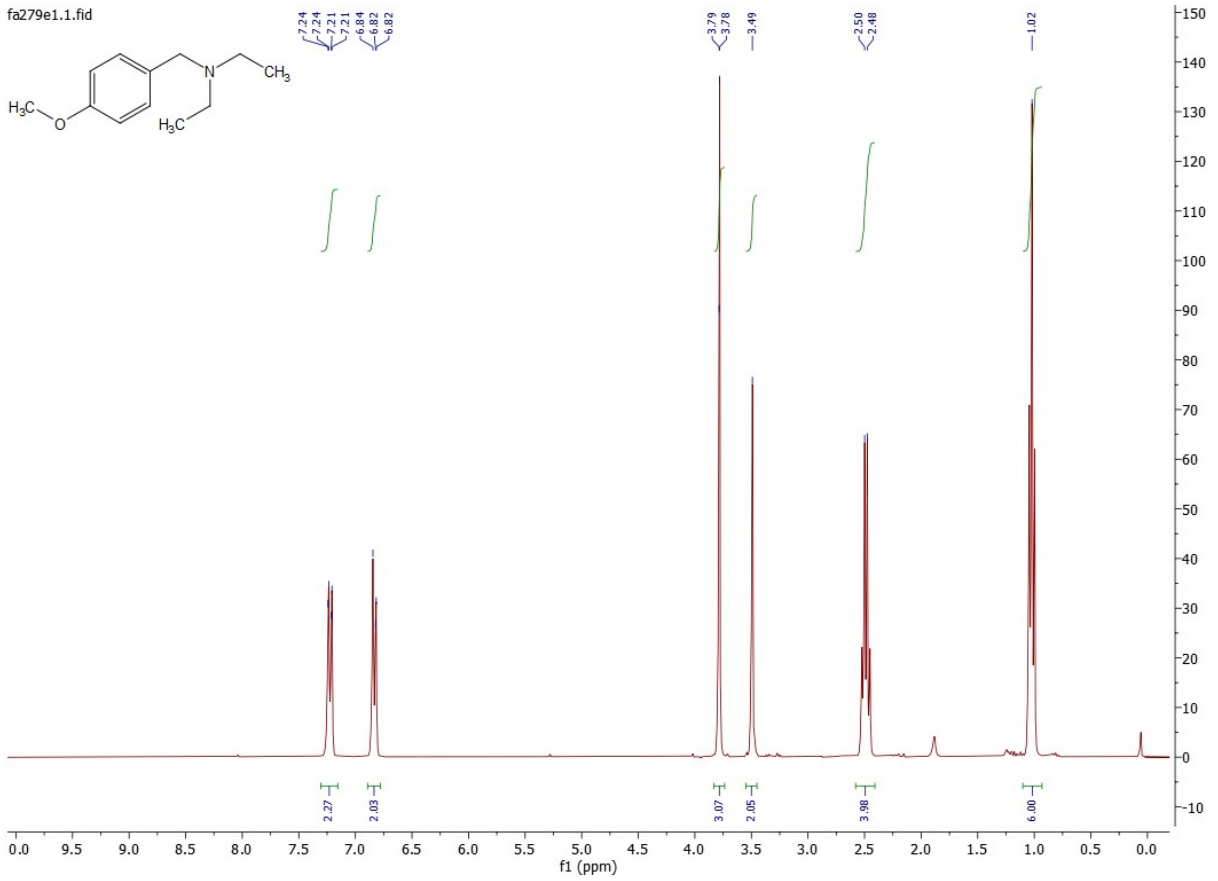


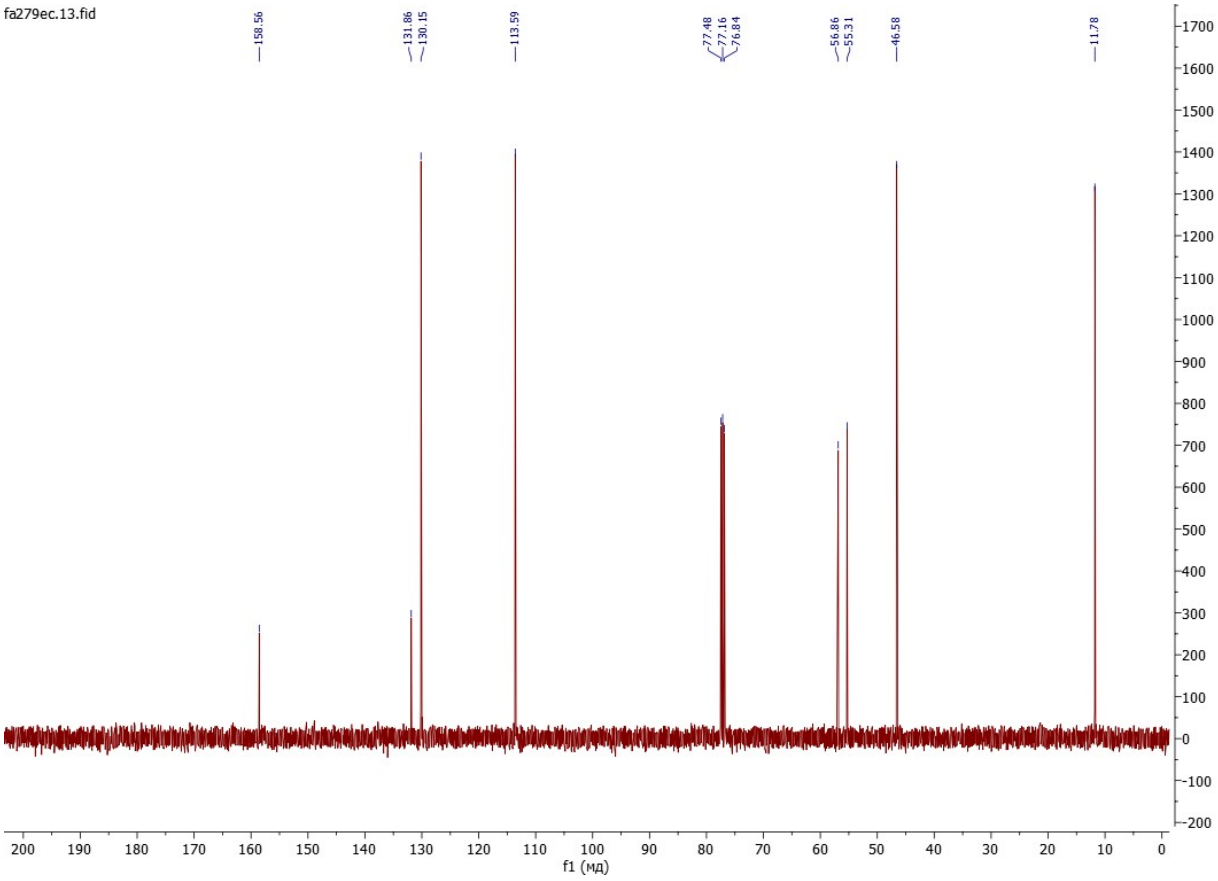
^{13}C-NMR in D_2O:



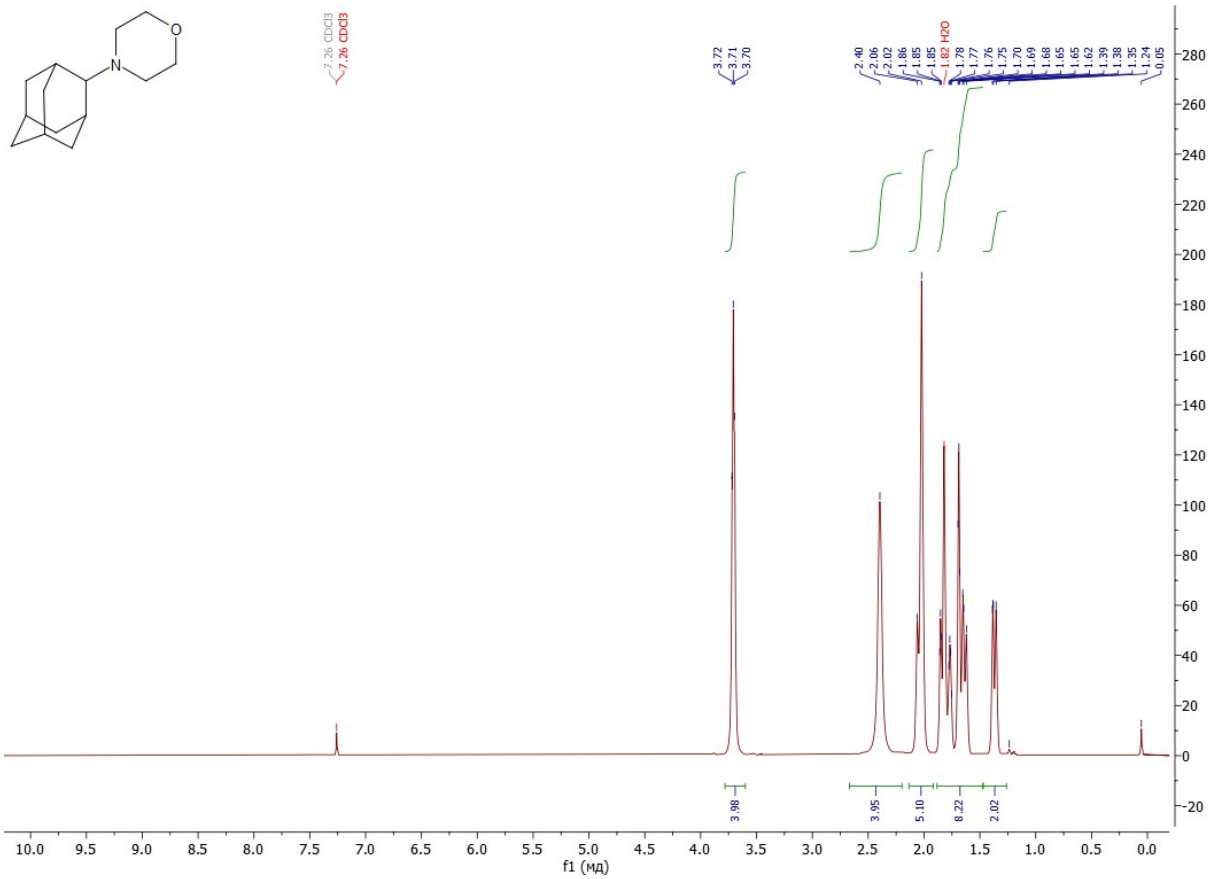

+TOF MS: 0.5022 to 0.6557 min from Sample 47 (FA265B) of 20_12_2022.wiff different calibrations (DuoSpray ())
Max. 2.4e5 cps.
Intensity, cps
m/z, Da
180.1744
331.2839
359.3150
313.2733
100.1127
182.1805
239.2362
369.2395
397.2709
486.2838

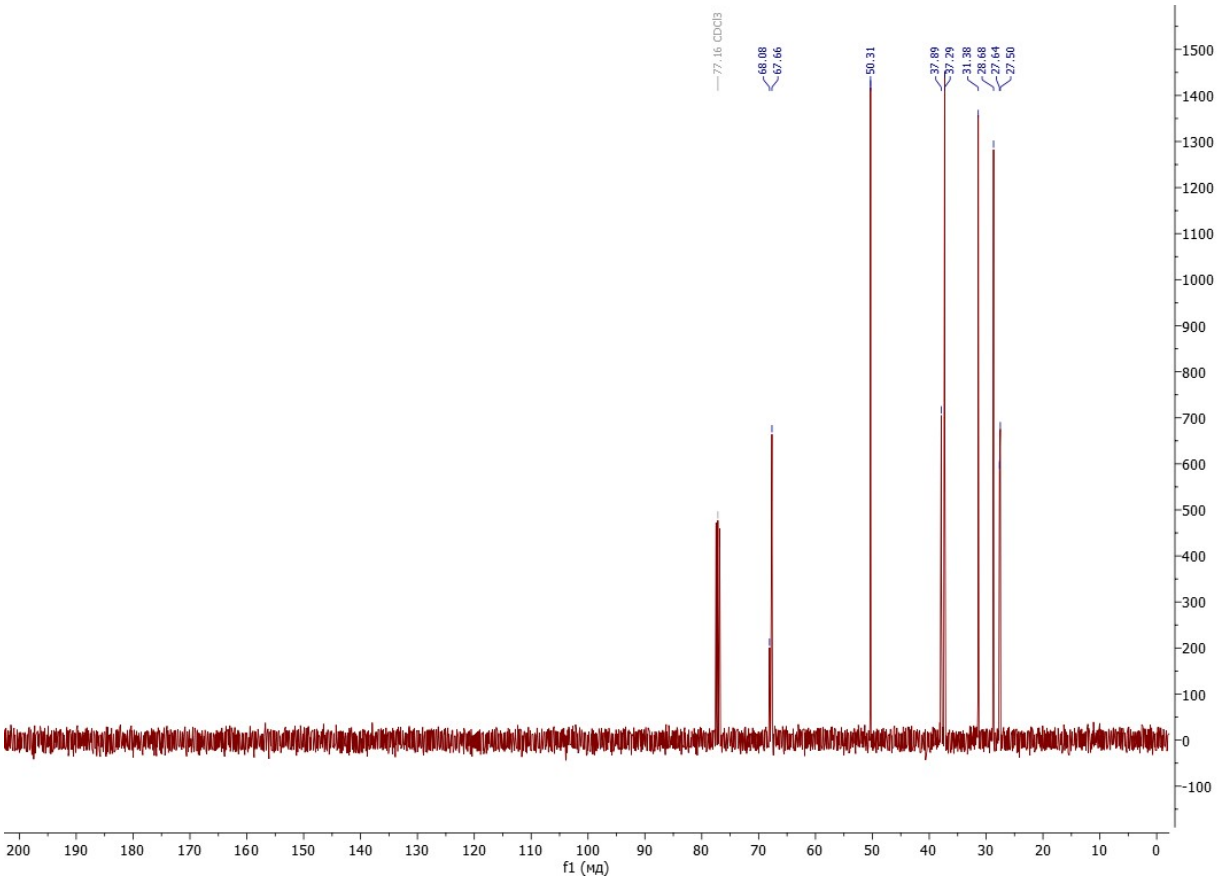
N-ethyl-N-(4-methoxybenzyl)ethanamine (16)



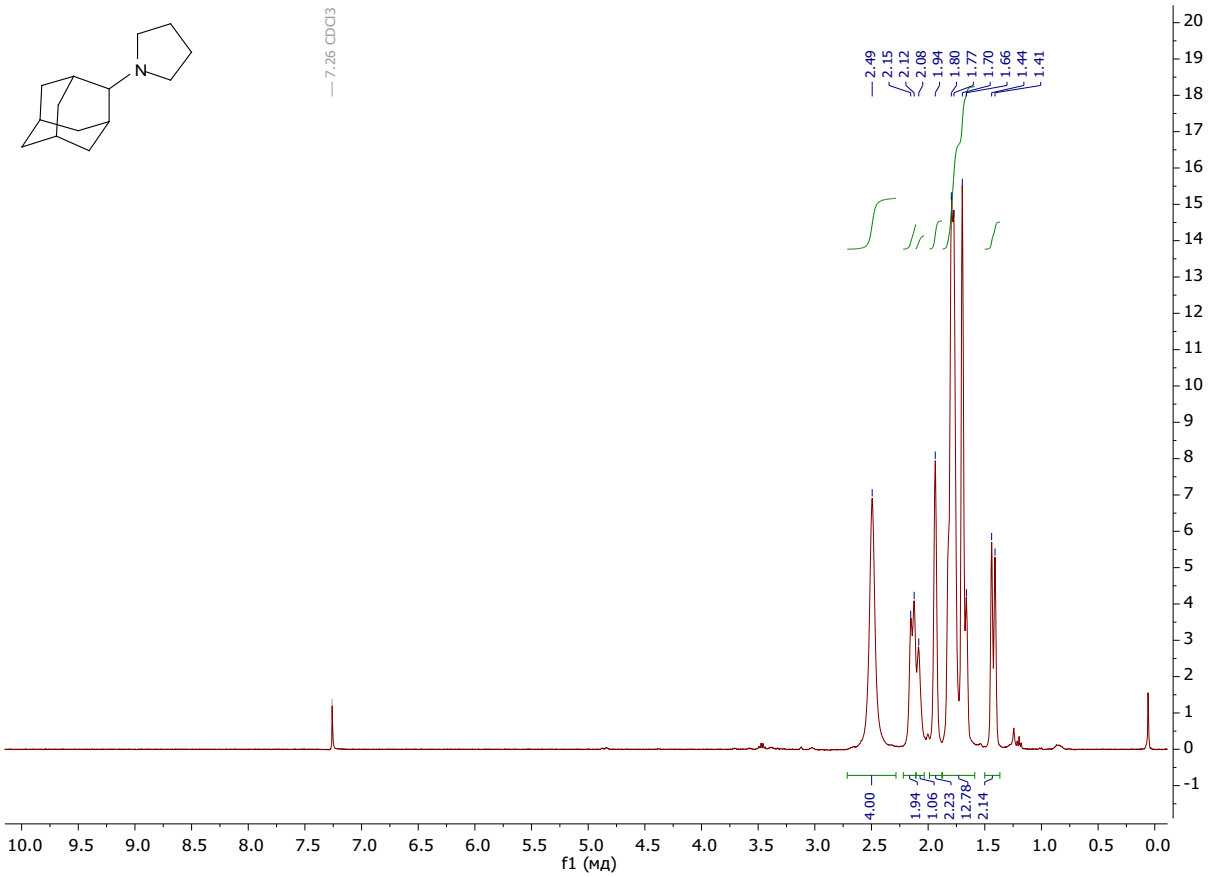


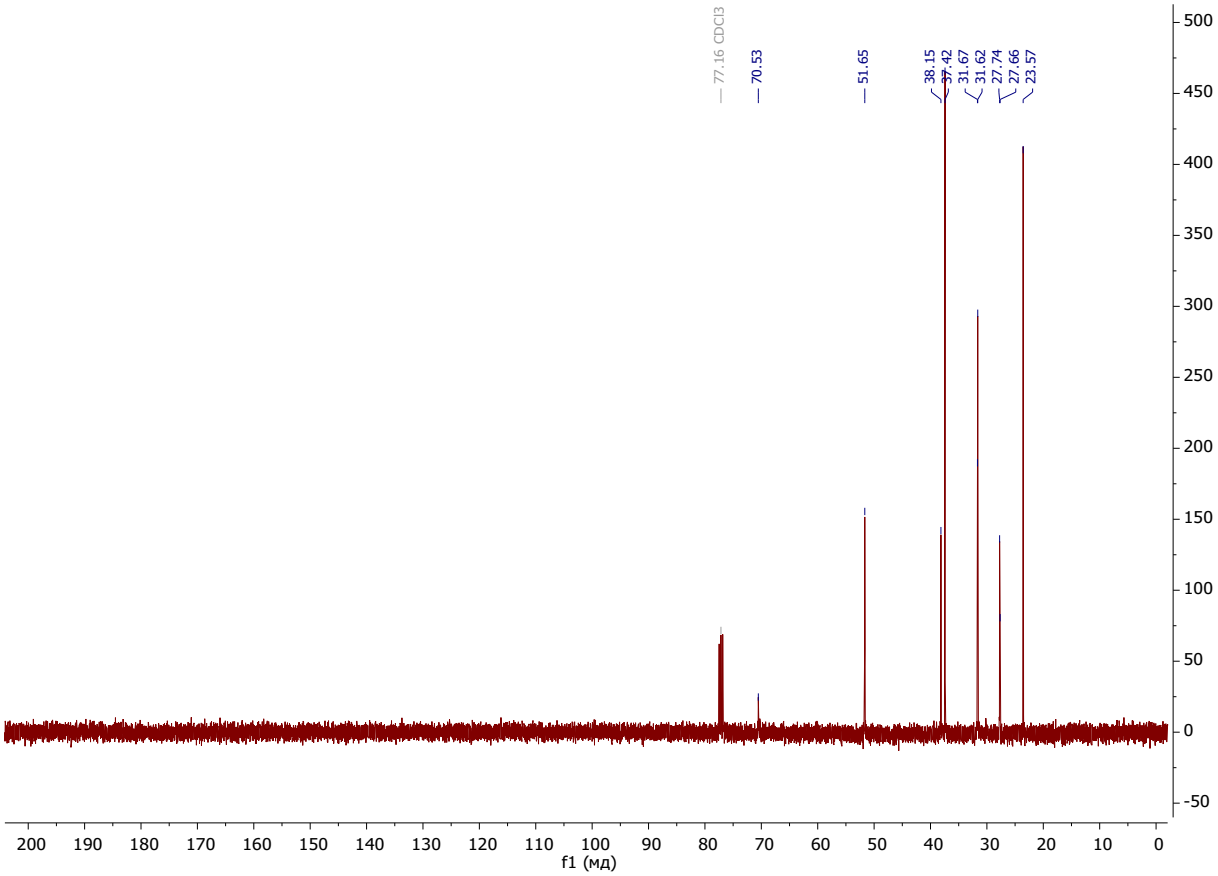
4-(adamantan-2-yl)-morpholine (17)



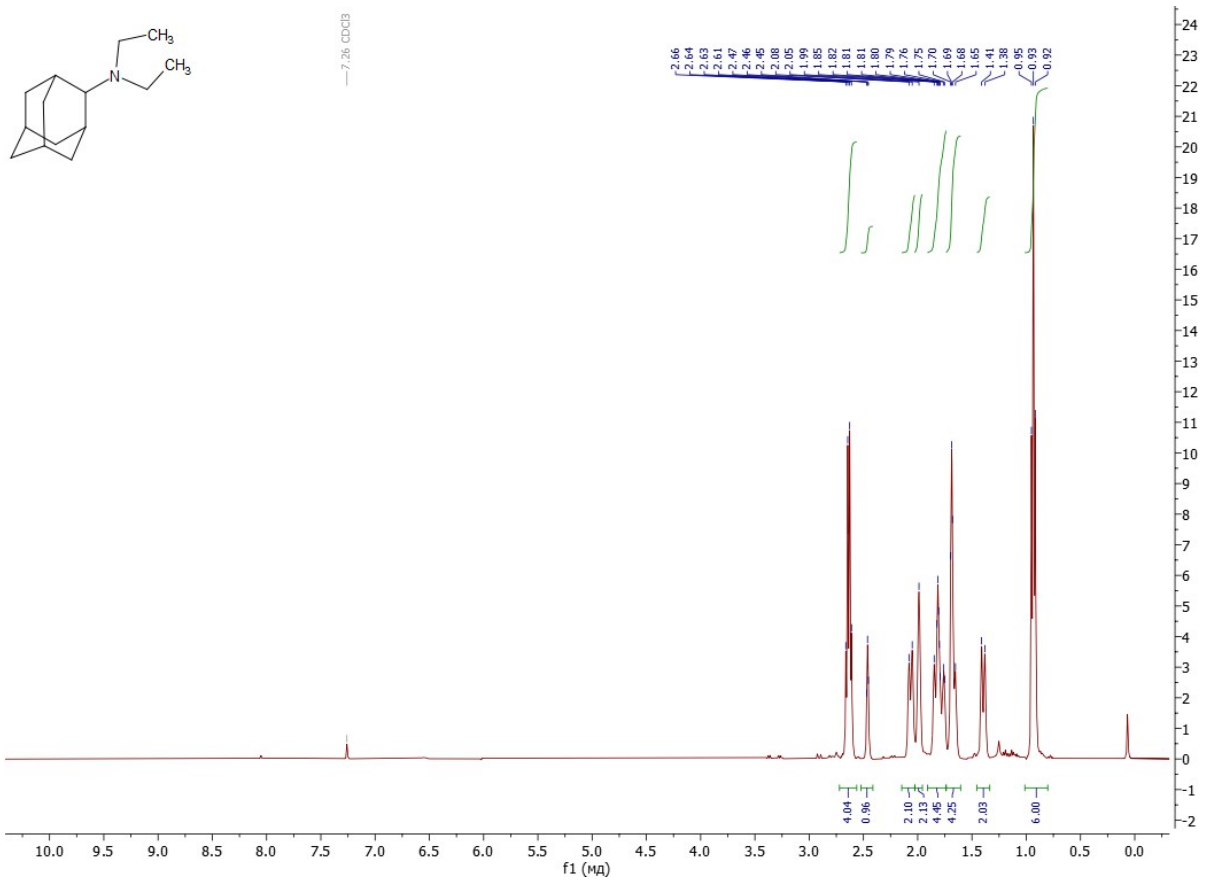


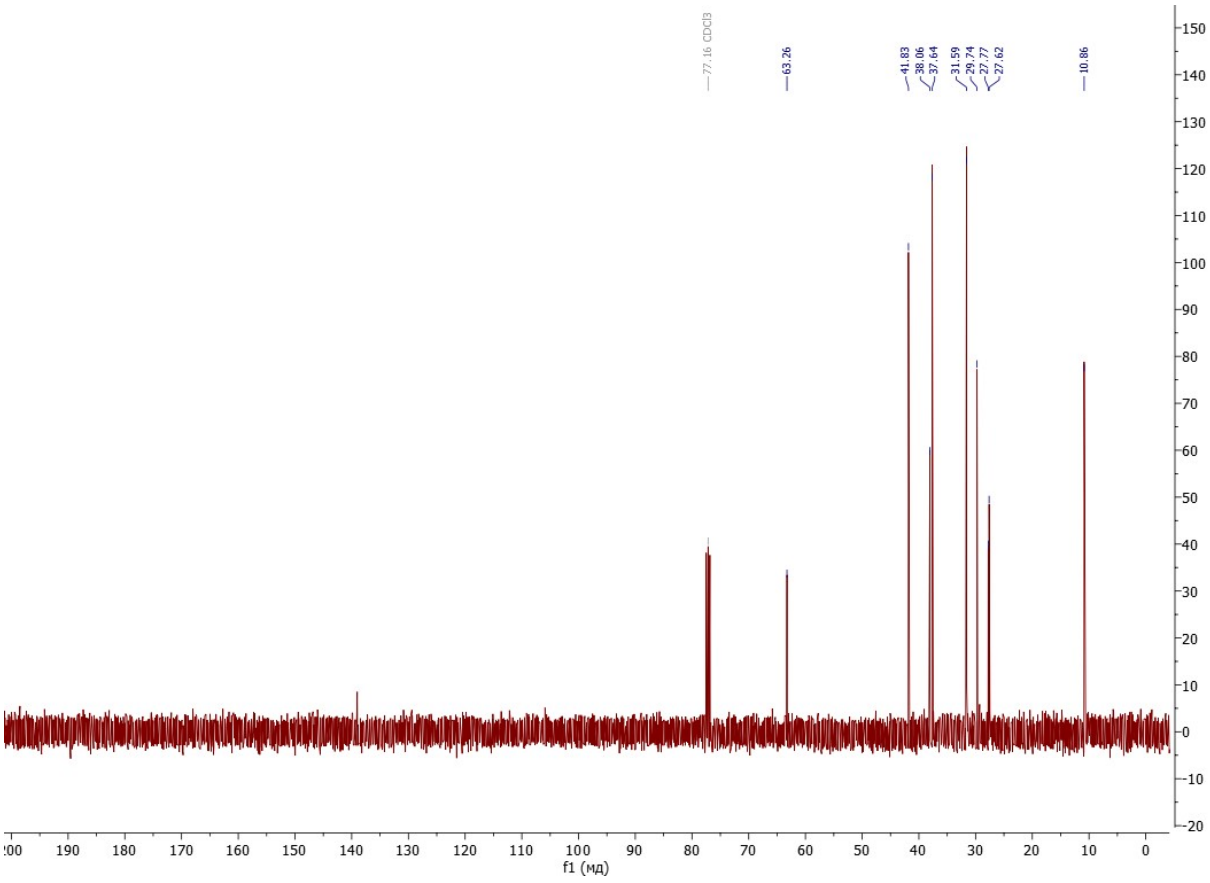
1-(adamantan-2-yl)-pyrrolidine (18)

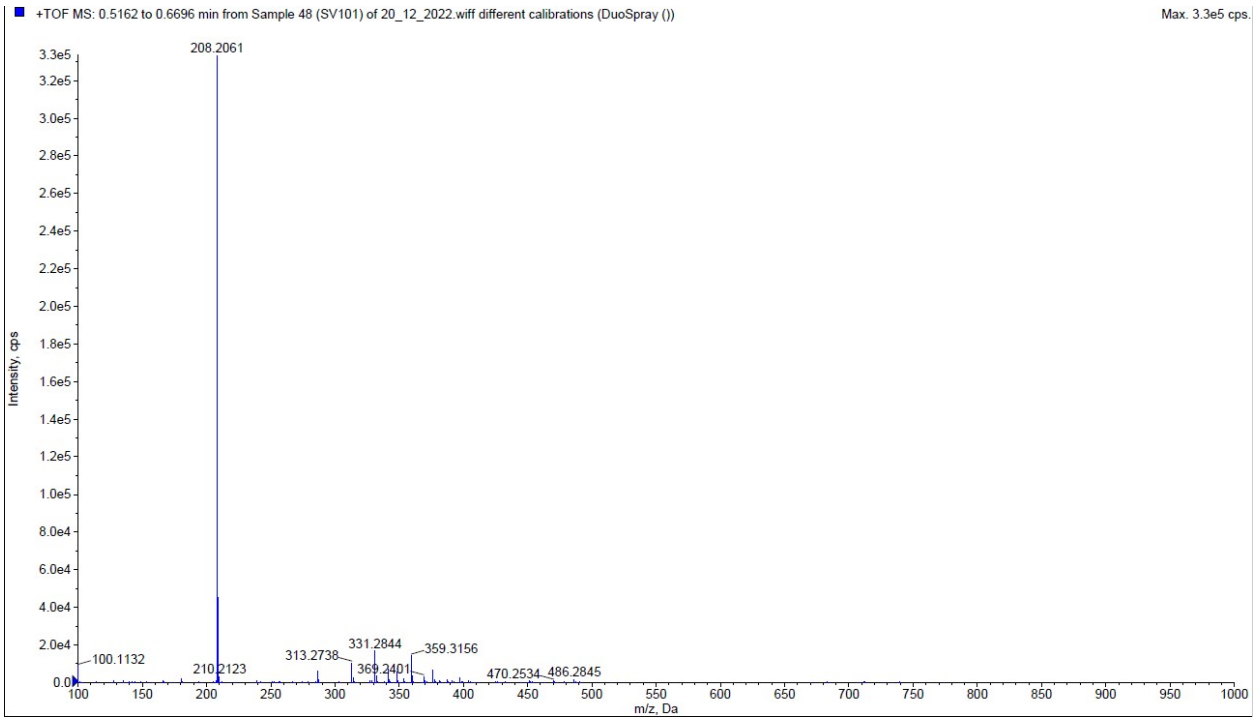




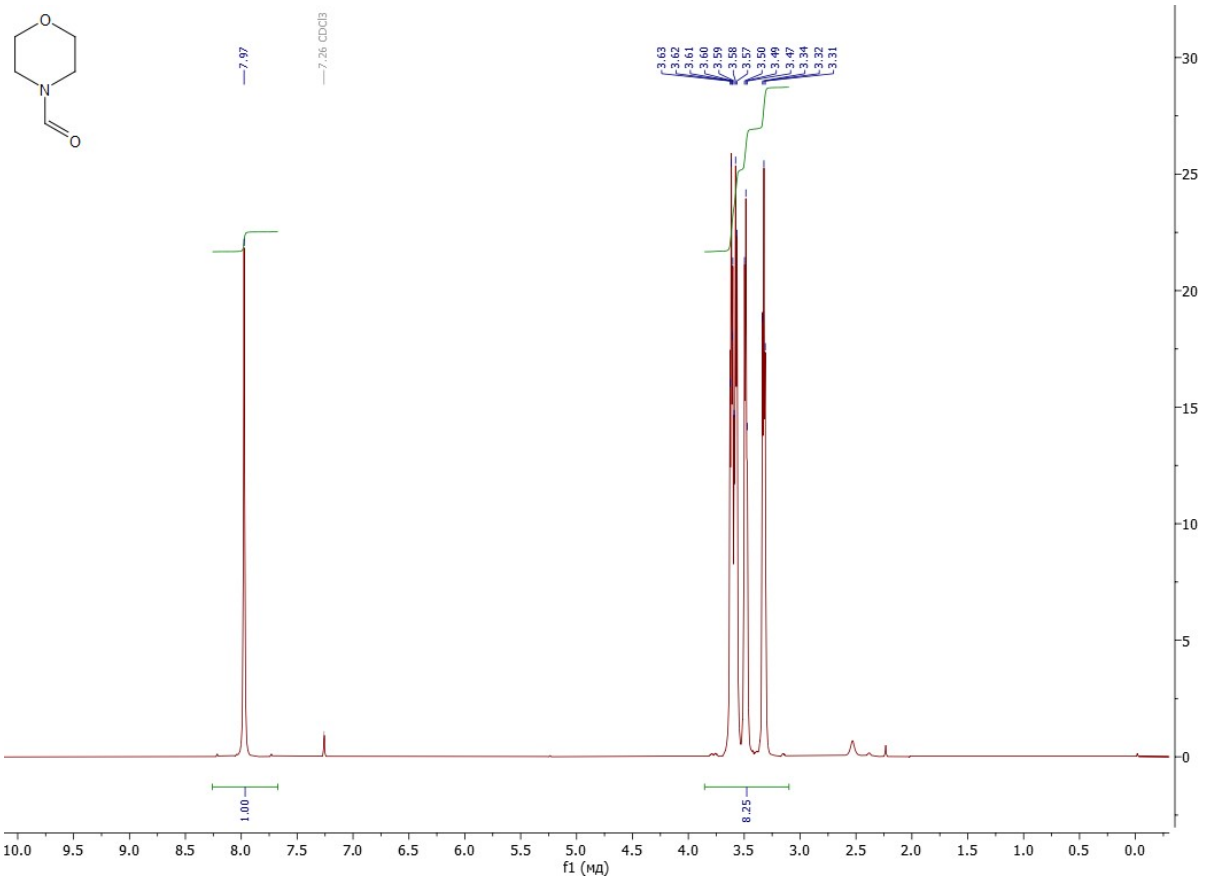
N,N-diethyladamantan-2-amine (19)

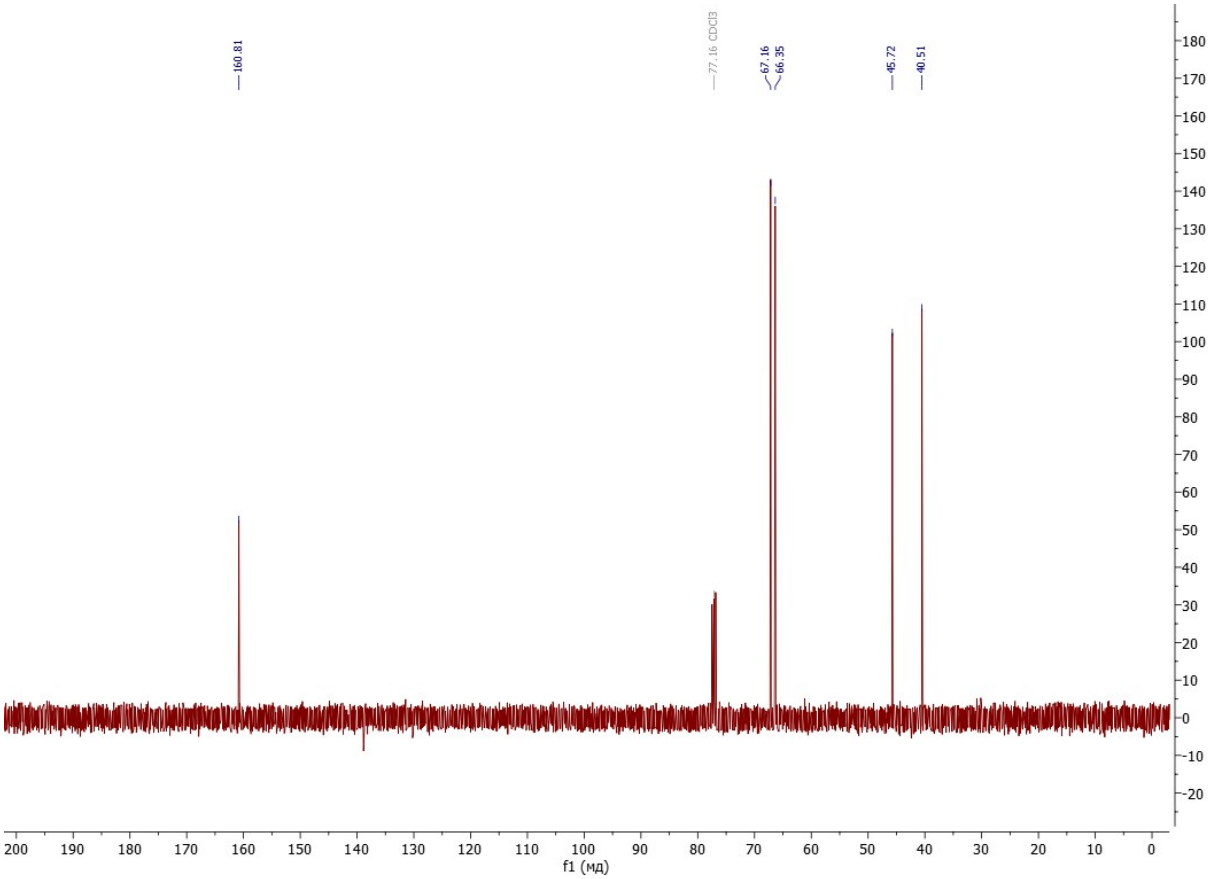




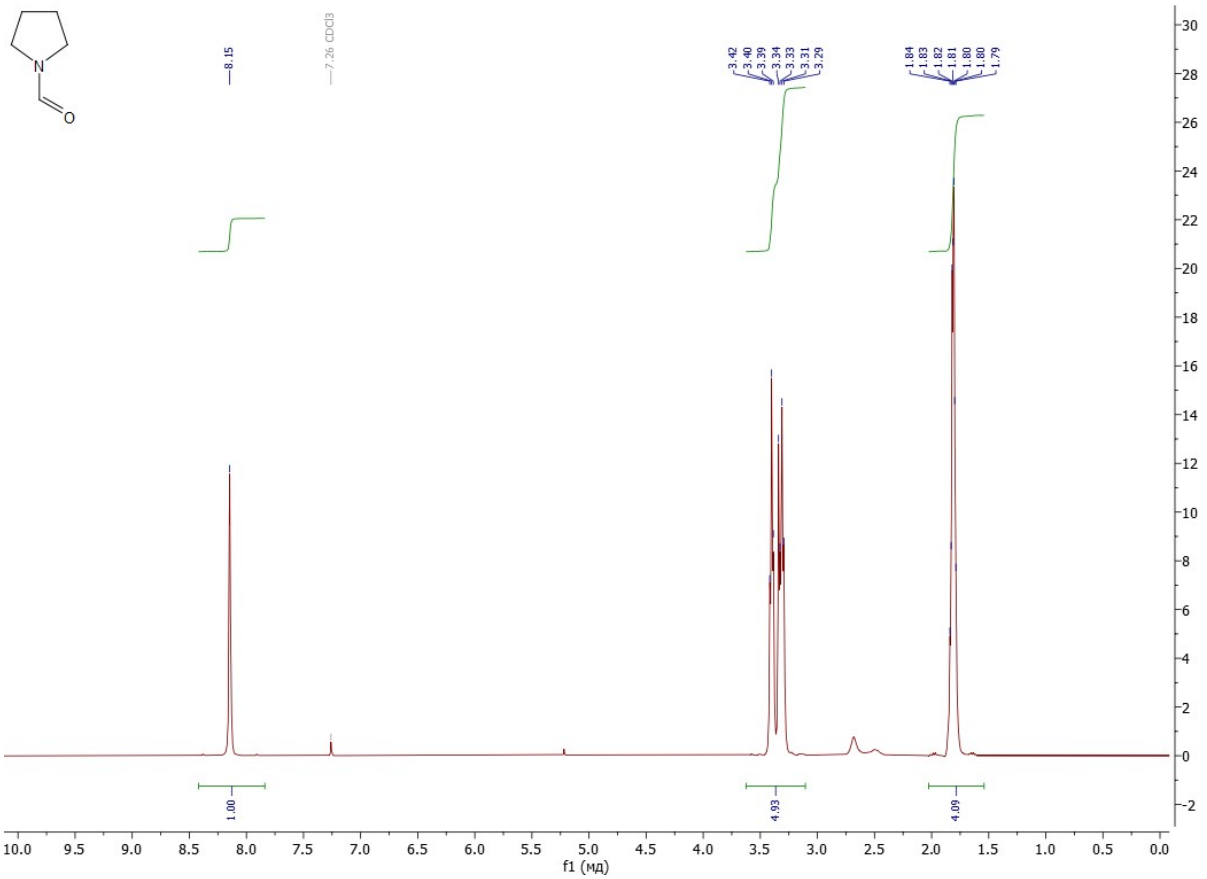

+TOF MS: 0.5162 to 0.6696 min from Sample 48 (SV101) of 20_12_2022.wiff different calibrations (DuoSpray ())
Max. 3.3e5 cps.
Intensity, cps
m/z, Da
208.2061
100.1132
210.2123
313.2738
331.2844
359.3156
369.2401
470.2534
486.2845

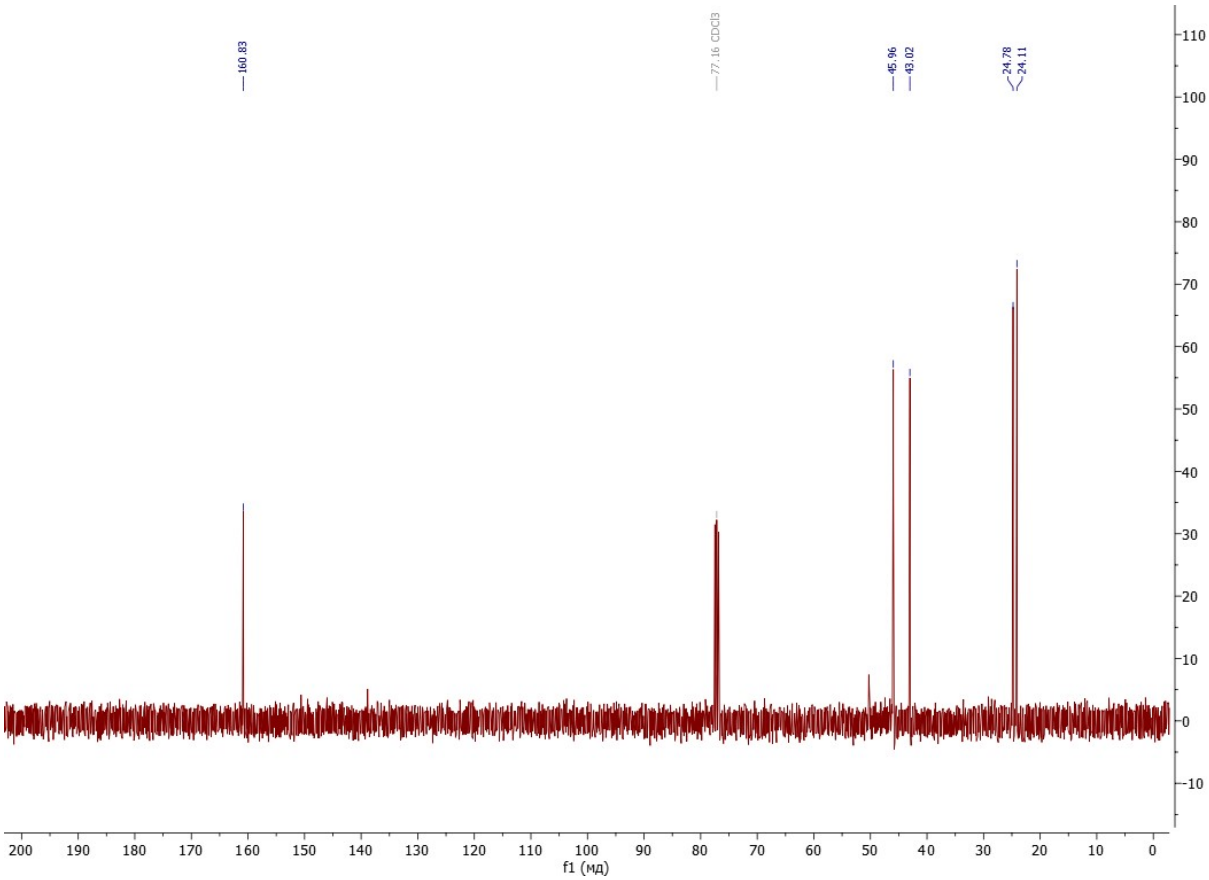
N-formylmorpholine





N-formylpyrrolidine





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