Supporting Information

Electrochemical thiocyanation of barbituric acids

Oleg V. Bityukov,[1] Andrey S. Kirillov,[1] Pavel Yu. Serdyuchenko,[1,3] Maria A. Kuznetsova,[2] Valentina N. Demidova,[2] Vera A. Vil',[1] Alexander O. Terent'ev*[1]*

[1] N. D. Zelinsky Institute of Organic Chemistry, Russian Academy of Sciences, 47 Leninsky prosp., 119991 Moscow, Russian Federation
[phone +7 (499) 1356428; fax, +7 (499) 1355328, e-mail, alterex@yandex.ru]
[2] All-Russian Research Institute for Phytopathology, B. Vyazyomy, 143050, Moscow Region, Russian Federation
[3] D. I. Mendeleev University of Chemical Technology of Russia, 9 Miusskaya Square, Moscow 125047, Russian Federation

Table of contents

General materials and methods

^{1}H and ^{13}C NMR spectra were recorded on Bruker AVANCE II 300 spectrometer (300.13 and 75.48 MHz, respectively) in $CDCl_3$. Chemical shifts were reported in parts per million (ppm), and the residual solvent peak was used as an internal reference: ^{1}H ($CDCl_3$ δ=7.25 ppm), ^{13}C ($CDCl_3$ δ=77.00 ppm). Multiplicity was indicated as follows: s (singlet), d (doublet), t (triplet), q (quartet), sept (septet), m (multiplet).

High resolution mass spectra (HR-MS) were measured on a Bruker micrOTOF II instrument using electrospray ionization (ESI). The measurements were performed in a positive ion mode (interface capillary voltage - 4500 V); mass range from m/z 50 to m/z 3000 Da; external calibration with Electrospray Calibrant Solution (Fluka). A syringe injection was used for all acetonitrile solutions (flow rate 3 μL/min). Nitrogen was applied as a dry gas; interface temperature was set at 180 °C.

The TLC analysis was carried out on standard silica gel chromatography plates (DC-Fertigfolien ALUGRAM^{R} Xtra SIL G/UV_{254}). Column chromatography was performed using silica gel (0.060-0.200 mm, 60 A, CAS 7631-86-9, Acros).

Acetic acid, KSCN, NH_4SCN, CH_3CN, CH_3OH, THF, DMSO, petroleum ether (PE, 40/70), ethyl acetate (EA), *p*-TsOH·H_2O were purchased from commercial sources and was used as is.

Starting α-substituted barbituric acids **1** were prepared accordingly literature procedures.[1]

Calculation of the amount of electric current

$$Q = I \cdot t$$

Q — amount of passed electric current, C (Coulomb)

I — electric current, A

t — time, sec

Q = I · t = 0.32 · 15 · 60 = 288 C

$$N = \frac{Q}{F \cdot n_r}$$

N — number of electrons generated in the cell per 1 molecule of α-substituted barbituric acid, F/mol

Q — amount of passed electric current, C (Coulomb)

F — Faraday constant, F = 96485 $\mathrm{C \cdot mol^{-1}}$

n_r — amount of α-substituted barbituric acid, mol

N = 288 / (96485 · 0.001) = 2.98 F/mol ≈ 3.0 F/mol.

The reaction setup

Figure S1. Glassy carbon anode (4.5 cm^2) and platinum plate cathode (4.5 cm^2)

Figure S2. Undivided electrochemical cell equipped with glassy carbon anode (4.5 cm^2) and platinum plate cathode (4.5 cm^2) and connected to a DC regulated power supply with the reaction mixture during electrolysis under constant current conditions.

Figure S3. Undivided electrochemical cell equipped with glassy carbon anode (4.5 cm^2) and platinum plate cathode (4.5 cm^2) with the reaction mixture at the beginning of the reaction.

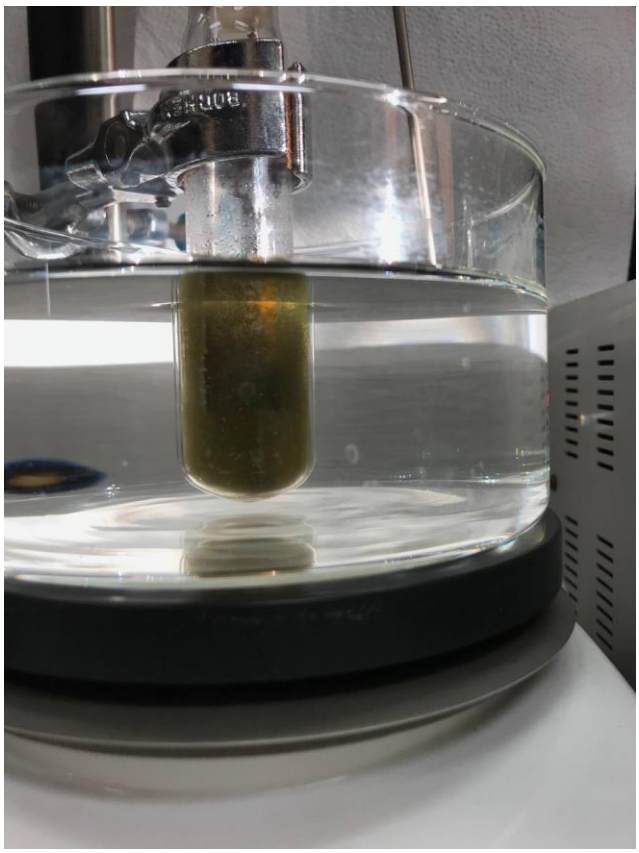
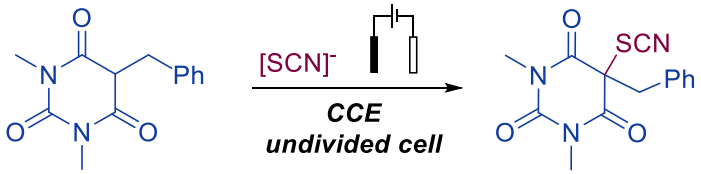


Figure S4. Undivided electrochemical cell equipped with glassy carbon anode (4.5 cm^2) and platinum plate cathode (4.5 cm^2) with the reaction mixture at the end of the reaction.

Table S1. Detailed optimization of the reaction conditions.[a]



1a **2a**

Entry	Electrolyte (molar ratio: mol per mol **1a**)	Solvent	T, °C	j_{anode}, mA/cm^2	Electricity passed, F/mol **1a**	Yield **2a**, %[b]
1	NH_4SCN (2 eq.)	CH_3OH	50	9	3	10
2	NH_4SCN (2 eq.)	THF	50	9	3	6
3	NH_4SCN (2 eq.)	DMSO	50	9	3	trace
4	NH_4SCN (2 eq)	CH_3CN	50	9	3	42
5	KSCN (2 eq.)	CH_3CN	50	9	3	5
6[c]	NH_4SCN (2 eq.)	CH_3CN	50	9	3	39
7	NH_4SCN (2 eq.)	CH_3CN	50	9	2	38
8	NH_4SCN (2 eq.)	CH_3CN	50	9	4	41
9	NH_4SCN (2 eq.)	CH_3CN	50	-	0	0
10	NH_4SCN (2 eq.)	CH_3CN	20-25	9	3	53
11	NH_4SCN (2 eq.)	CH_3CN	20-25	18	3	60
12	NH_4SCN (2 eq.)	CH_3CN	20-25	27	3	46
13	NH_4SCN (2 eq.)	CH_3CN	20-25	36	3	57
14	NH_4SCN (2 eq.)	CH_3CN	20-25	44	3	58
15	NH_4SCN (2 eq.)	CH_3CN	20-25	53	3	73
16	NH_4SCN (2 eq.)	CH_3CN	20-25	62	3	83
17	NH_4SCN (2 eq.)	CH_3CN	20-25	71	3	76
18	NH_4SCN (4 eq.)	CH_3CN	20-25	71	3	80
19[d]	NH_4SCN (4 eq.)	CH_3CN	20-25	9	3	87
20[d]	NH_4SCN (4 eq.)	CH_3CN	20-25	18	3	68
21[d]	NH_4SCN (4 eq.)	CH_3CN	20-25	27	3	71
22[d]	NH_4SCN (4 eq.)	CH_3CN	20-25	36	3	78
23[d]	NH_4SCN (4 eq.)	CH_3CN	20-25	44	3	79
24[d]	NH_4SCN (4 eq.)	CH_3CN	20-25	53	3	83
25[d]	NH_4SCN (4 eq.)	CH_3CN	20-25	62	3	86
26[d]	NH_4SCN (4 eq.)	CH_3CN	20-25	71	3	95
27[e]	NH_4SCN (4 eq.)	CH_3CN	20-25	71	3	71
28[f]	NH_4SCN (4 eq.)	CH_3CN	20-25	71	3	66
29[g]	NH_4SCN (4 eq.)	CH_3CN	20-25	71	3	24
30[h]	NH_4SCN (4 eq.)	CH_3CN	20-25	71	3	75

[a] **Reaction conditions:** undivided cell, platinum plate cathode (4.5 cm^2) and glassy carbon anode (4.5 cm^2), constant current = 40-320 mA (j_{anode} ≈ 9-71 mA/cm^2), **1a** (1.0 mmol, 246.3 mg), supporting electrolyte (2.0-4.0 eq., 2.0-4.0 mmol), solvent (15.0 mL), 20-50 °C, air atmosphere. [b] Isolated yields. [c] Platinum plate (4.5 cm^2) anode and

cathode. [d] Added AcOH (4.0 eq., 4.0 mmol, 240 mg). [e] Added AcOH (4.0 eq., 4.0 mmol, 240 mg), Glassy carbon anode and cathode. [f] Added HCOOH (4.0 eq., 4.0 mmol, 184 mg). [g] Added *p*-TsOH·H_2O (4.0 eq., 4 mmol, 760 mg). [h] Added AcOH (4.0 eq., 4.0 mmol, 240 mg), glassy carbon anode and steel cathode.

Experimental Procedures for Table 1. Optimization of the reaction conditions.

Experimental Procedure for Table 1, entries 1-2, 4.

An undivided cell was equipped with a glassy carbon anode (4.5 cm^2) and a platinum plate cathode (4.5 cm^2) and connected to a DC regulated power supply. The solution of α-benzyl barbituric acid **1a** (1.0 mmol, 246.3 mg) and NH_4SCN (2 eq., 2.0 mmol, 152.2 mg) in the solvent (CH_3OH, THF or CH_3CN) (15 mL) was electrolyzed using constant current conditions I = 40 mA (j_{anode} = 9 mA/cm^2) at 50 °C under magnetic stirring (3 F/mol, 120 min). The reaction mixture was concentrated under reduced pressure using a rotary evaporator (15-20 mmHg), (bath temperature, ca. 20-25 °C). Product **2a** was isolated by chromatography on SiO_2 (PE:EtOAc = 5:1).

Experimental Procedure for Table 1, entry 3.

An undivided cell was equipped with a glassy carbon anode (4.5 cm^2) and a platinum plate cathode (4.5 cm^2) and connected to a DC regulated power supply. The solution of α-benzyl barbituric acid **1a** (1.0 mmol, 246.3 mg) and NH_4SCN (2 eq., 2.0 mmol, 152.2 mg) in DMSO (15 mL) was electrolyzed using constant current conditions I = 40 mA (j_{anode} = 9 mA/cm^2) at 50 °C under magnetic stirring (3 F/mol, 120 min). After that time, CH_2Cl_2 (30 mL) was added. The mixture was washed with brine (2 × 10 mL), dried over $MgSO_4$, filtered, and concentrated under reduced pressure using a rotary evaporator (15-20 mmHg) (bath temperature, ca. 20-25 °C). Product **2a** was isolated by chromatography on SiO_2 (PE:EtOAc = 5:1).

Experimental Procedure for Table 1, entry 5.

An undivided cell was equipped with a glassy carbon anode (4.5 cm^2) and a platinum plate cathode (4.5 cm^2) and connected to a DC regulated power supply. The solution of α-benzyl barbituric acid **1a** (1.0 mmol, 246.3 mg) and NH_4SCN (2 eq., 2.0 mmol, 152.2 mg) in CH_3CN (15 mL) was stirred for 15 minutes at 50 °C. The reaction mixture was concentrated under reduced pressure using a rotary evaporator (15-20 mmHg), (bath temperature, ca. 20-25 °C). Product **2a** was not detected.

Experimental Procedure for Table 1, entries 6-8.

An undivided cell was equipped with a glassy carbon anode (4.5 cm^2) and a platinum plate cathode (4.5 cm^2) and connected to a DC regulated power supply. The solution of

α-benzyl barbituric acid **1a** (1.0 mmol, 246.3 mg) and NH_4SCN (2 eq., 2.0 mmol, 152.2 mg) in CH_3CN (15 mL) was electrolyzed using constant current conditions I = 40-320 mA (j_{anode} = 9-71 mA/cm^2) at 20-25 °C under magnetic stirring (3 F/mol, 15-120 min). The reaction mixture was concentrated under reduced pressure using a rotary evaporator (15-20 mmHg), (bath temperature, ca. 20-25 °C). Product **2a** was isolated by chromatography on SiO_2 (PE:EtOAc = 5:1).

Experimental Procedure for Table 1, entry 9.

An undivided cell was equipped with a glassy carbon anode (4.5 cm^2) and a platinum plate cathode (4.5 cm^2) and connected to a DC regulated power supply. The solution of α-benzyl barbituric acid **1a** (1.0 mmol, 246.3 mg) and NH_4SCN (4 eq., 4.0 mmol, 304.4 mg) in CH_3CN (15 mL) was electrolyzed using constant current conditions I = 320 mA (j_{anode} = 71 mA/cm^2) at 20-25 °C under magnetic stirring (3 F/mol, 15 min). The reaction mixture was concentrated under reduced pressure using a rotary evaporator (15-20 mmHg), (bath temperature, ca. 20-25 °C). Product **2a** was isolated by chromatography on SiO_2 (PE:EtOAc = 5:1).

Experimental Procedure for Table 1, entries 10-11.

An undivided cell was equipped with a glassy carbon anode (4.5 cm^2) and a platinum plate cathode (4.5 cm^2) and connected to a DC regulated power supply. The solution of α-benzyl barbituric acid **1a** (1.0 mmol, 246.3 mg), NH_4SCN (4 eq., 4.0 mmol, 304.4 mg) and acetic acid (4.0 mmol, 240 mg) in CH_3CN (15 mL) was electrolyzed using constant current conditions I = 40 or 320 mA (j_{anode} = 9 or 71 mA/cm^2) at 20-25 °C under magnetic stirring (3 F/mol, 15 or 120 min). The combined organic phases were concentrated under reduced pressure using a rotary evaporator (15-20 mmHg), (bath temperature, ca. 20-25 °C). Product **2a** was isolated by chromatography on SiO_2 (PE:EtOAc = 5:1).

Experimental Procedure for Table 1, entries 12-13.

An undivided cell was equipped with a glassy carbon anode (4.5 cm^2) and a platinum plate cathode (4.5 cm^2) and connected to a DC regulated power supply. The solution of α-benzyl barbituric acid **1a** (1.0 mmol, 246.3 mg), NH_4SCN (4 eq., 4.0 mmol, 304.4 mg) and HCOOH (4.0 mmol, 184 mg) (entry 12) or *p*-TsOH·H_2O (4.0 mmol, 760 mg) (entry 13) in CH_3CN (15 mL) was electrolyzed using constant current conditions I = 320 mA (j_{anode} = 71 mA/cm^2) at 20-25 °C under magnetic stirring (3 F/mol, 15 min). The combined organic phases were concentrated under reduced pressure using a rotary evaporator (15-20 mmHg), (bath temperature, ca. 20-25 °C). Product **2a** was isolated by chromatography on SiO_2 (PE:EtOAc = 5:1).

Experimental Procedure for Table 1, entries 14-16.

An undivided cell was equipped with a glassy carbon anode (4.5 cm^2) and cathode (4.5 cm^2) (entry 14) or glassy carbon anode (4.5 cm^2) and stainless steel cathode (4.5 cm^2) (entry 15) or platinum anode (4.5 cm^2) and cathode (4.5 cm^2) (entry 16) and connected to a DC regulated power supply. The solution of α-benzyl barbituric acid **1a** (1.0 mmol, 246.3 mg), NH_4SCN (4 eq., 4.0 mmol, 304.4 mg) and acetic acid (4.0 mmol, 240 mg) in CH_3CN (15 mL) was electrolyzed using constant current conditions I = 320 mA (j_{anode} = 71 mA/cm^2) at 20-25 °C under magnetic stirring (3 F/mol, 15 min). The combined organic phases were concentrated under reduced pressure using a rotary evaporator (15-20 mmHg), (bath temperature, ca. 20-25 °C). Product **2a** was isolated by chromatography on SiO_2 (PE:EtOAc = 5:1).

Experimental Procedure for Table 1, entries 17-18.

An undivided cell was equipped with a glassy carbon anode (4.5 cm^2) and a platinum plate cathode (4.5 cm^2) and connected to a DC regulated power supply. The solution of α-benzyl barbituric acid **1a** (1.0 mmol, 246.3 mg), KSCN (4 eq., 4.0 mmol, 388.7 mg) (entry 17) or NaSCN (4 eq., 4.0 mmol, 324.3 mg) (entry 18) and acetic acid (4.0 mmol, 240 mg) in CH_3CN (15 mL) was electrolyzed using constant current conditions I = 320 mA (j_{anode} = 71 mA/cm^2) at 20-25 °C under magnetic stirring (3 F/mol, 15 min). The combined organic phases were concentrated under reduced pressure using a rotary evaporator (15-20 mmHg), (bath temperature, ca. 20-25 °C). Product **2a** was isolated by chromatography on SiO_2 (PE:EtOAc = 5:1).

Experimental Procedures for Scheme 3.

The influence of the current density on 2a yield (top).

An undivided cell was equipped with a glassy carbon anode (4.5 cm^2) and a platinum plate cathode (4.5 cm^2) and connected to a DC regulated power supply. The solution of α-benzyl barbituric acid **1a** (1.0 mmol, 246.3 mg), NH_4SCN (4 eq., 4 mmol, 304.4 mg) and acetic acid (4.0 mmol, 240 mg) in CH_3CN (15 mL) was electrolyzed using constant current conditions I = 22-900 mA (j_{anode} = 5-200 mA/cm^2) at 20-25 °C under magnetic stirring (3 F/mol, 5-214 min). The reaction mixture was concentrated under reduced pressure using a rotary evaporator (15-20 mmHg), (bath temperature, ca. 20-25 °C). Product **2a** was isolated by chromatography on SiO_2 (PE:EtOAc).

The recovery of 2a (bottom).

An undivided cell was equipped with a glassy carbon anode (4.5 cm^2) and a platinum plate cathode (4.5 cm^2) and connected to a DC regulated power supply. The solution of

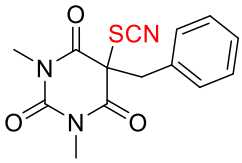
compound **2a** (1.0 mmol, 303.3 mg), NH_4SCN (4 eq., 4.0 mmol, 304.4 mg) or *n*-Bu_4NClO_4 (1 eq., 1 mmol, 341.9 mg) in CH_3CN (15 mL) was electrolyzed using constant current conditions I = 45-675 mA (j_{anode} = 10-150 mA/cm^2) at 20-25 °C under magnetic stirring (3 F/mol, 7-107 min). The reaction mixture was concentrated under reduced pressure using a rotary evaporator (15-20 mmHg), (bath temperature, ca. 20-25 °C). Compound **2a** was isolated by chromatography on SiO_2 (PE:EtOAc).

Experimental Procedure for Scheme 4.

Electrochemical synthesis of α-thiocyanobarbiturates 2a-u from different α-substituted barbituric acids 1a-u.

An undivided cell was equipped with a glassy carbon anode (4.5 cm^2) and a platinum plate cathode (4.5 cm^2) and connected to a DC regulated power supply. The solution of α-substituted barbituric acids **1a-u** (1.0 mmol, 156.1-315.1 mg), NH_4SCN (4 eq., 4.0 mmol, 304.4 mg) and acetic acid (4.0 mmol, 240 mg) in CH_3CN (15 mL) was electrolyzed using constant current conditions I = 320 mA (j_{anode} = 71 mA/cm^2) at 20-25 °C under magnetic stirring (3 F/mol, 15 min). The reaction mixture was concentrated under reduced pressure using a rotary evaporator (15-20 mmHg), (bath temperature, ca. 20-25 °C). Products **2a-u** were isolated by chromatography on SiO_2 (PE:EtOAc).

5-Benzyl-1,3-dimethyl-5-thiocyanatopyrimidine-2,4,6(1*H*,3*H*,5*H*)-trione, 2a



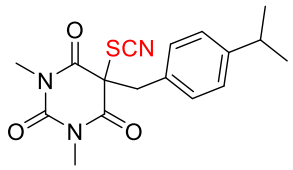
Yield was 80% (0.80 mmol, 242.4 mg). Yellow solid (mp = 80-81 °C). R_f = 0.37 (PE:EtOAc =5:1);

^{1}H NMR (300.13 MHz, $CDCl_3$) δ 7.29-7.24 (m, 3H), 7.08-7.06 (m, 2H), 3.64 (s, 2H), 3.21 (s, 6H);

^{13}C NMR (75.48 MHz, $CDCl_3$) δ 165.44, 148.85, 132.08, 129.66, 129.07, 128.97, 107.81, 59.75, 43.66, 29.34;

HRMS (ESI-TOF) m/z [M + Na]$^+$: Calcd for $[C_{14}H_{13}N_3O_3SNa]^+$: 326.0570. Found: 326.0568.

5-(4-Isopropylbenzyl)-1,3-dimethyl-5-thiocyanatopyrimidine-2,4,6(1*H*,3*H*,5*H*)-trione, 2b



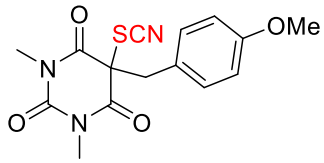
Yield was 70% (0.70 mmol, 202.5 mg). Yellow oil. R_f = 0.45 (PE:EtOAc = 5:1);

^{1}H NMR (300.13 MHz, $CDCl_3$) δ 7.11 (d, *J* = 8.1 Hz, 2H), 6.96 (d, *J* = 8.3 Hz, 2H), 3.59 (s, 2H), 3.20 (s, 6H), 2.84 (sept, *J* = 6.7 Hz, 1H), 1.19 (d, *J* = 7.0 Hz, 6H);

^{13}C NMR (75.48 MHz, $CDCl_3$) δ 165.59, 149.88, 148.98, 129.69, 129.34, 127.14, 107.97, 59.87, 43.39, 33.84, 29.42, 23.89;

HRMS (ESI-TOF) m/z [M + Na]$^+$: Calcd for $[C_{17}H_{19}N_3NaO_3S]^+$: 368.1039. Found: 368.1040.

5-(4-Methoxybenzyl)-1,3-dimethyl-5-thiocyanatopyrimidine-2,4,6(1*H*,3*H*,5*H*)-trione, 2c [2]



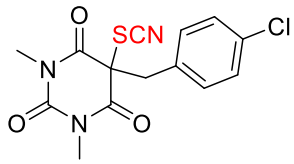
Yield was 40% (0.40 mmol, 134.7 mg). Yellow solid (mp = 108-109 °C). R_f = 0.62 (PE:EtOAc = 2:1);

^{1}H NMR (300.13 MHz, $CDCl_3$) δ 6.98 (d, *J* = 8.8 Hz, 2H), 6.76 (d, *J* = 8.8 Hz, 2H), 3.76 (s, 3H), 3.57 (s, 2H), 3.22 (s, 6H);

^{13}C NMR (75.48 MHz, $CDCl_3$) δ 165.63, 159.94, 149.00, 131.01, 123.92, 114.46, 107.90, 59.52, 55.34, 42.78, 29.45;

HRMS (ESI-TOF) m/z [M + Na]$^+$: Calcd for $[C_{15}H_{15}N_3NaO_4S]^+$: 356.0675. Found: 356.0678.

5-(4-Chlorobenzyl)-1,3-dimethyl-5-thiocyanatopyrimidine-2,4,6(1*H*,3*H*,5*H*)-trione, 2d



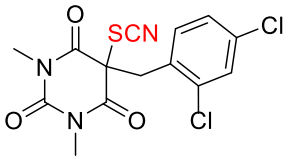
Yield was 74% (0.74 mmol, 249.1 mg). Yellow solid (mp = 78-79 °C). R_f = 0.67 (PE:EtOAc = 2:1);

^{1}H NMR (300.13 MHz, $CDCl_3$) δ 7.24 (d, *J* = 8.4 Hz, 2H), 7.04 (d, *J* = 8.4 Hz, 2H), 3.62 (s, 2H), 3.25 (s, 6H);

^{13}C NMR (75.48 MHz, $CDCl_3$) δ 165.21, 148.88, 135.12, 131.45, 130.78, 129.39, 107.41, 58.54, 41.61, 29.57;

HRMS (ESI-TOF) m/z [M + Na]$^+$: Calcd for $[C_{14}H_{12}ClN_3NaO_3S]^+$: 360.0180. Found: 360.0175.

5-(2,4-Dichlorobenzyl)-1,3-dimethyl-5-thiocyanatopyrimidine-2,4,6(1*H*,3*H*,5*H*)-trione, 2e



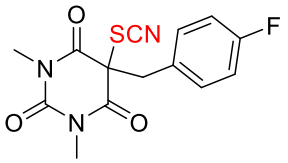
Yield was 56% (0.56 mmol, 207.4 mg). White solid (mp = 146-147 °C). R_f = 0.22 (PE:EtOAc = 5:1);

^{1}H NMR (300.13 MHz, $CDCl_3$) δ 7.39 (d, *J* = 2.1 Hz, 1H), 7.21 (dd, *J* = 8.3, 2.1 Hz, 1H), 7.08 (d, *J* = 8.3 Hz, 1H), 3.81 (s, 2H), 3.24 (s, 6H);

^{13}C NMR (75.48 MHz, $CDCl_3$) δ 165.02, 149.05, 135.90, 135.63, 132.39, 130.19, 128.76, 127.71, 108.31, 61.16, 41.05, 29.89;

HRMS (ESI-TOF) m/z [M + Na]$^+$: Calcd for $[C_{14}H_{11}Cl_2N_3NaO_3S]^+$: 395.9761. Found: 395.9759.

5-(4-Fluorobenzyl)-1,3-dimethyl-5-thiocyanatopyrimidine-2,4,6(1*H*,3H,5H)-trione, 2f



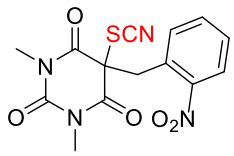
Yield was 85% (0.85 mmol, 274.7 mg). Yellow solid (mp = 89-90 °C). R_f = 0.17 (PE:EtOAc = 5:1);

^{1}H NMR (300.13 MHz, $CDCl_3$) δ 7.10-7.05 (m, 2H), 6.98-6.93 (m, 2H), 3.62 (s, 2H), 3.25 (s, 6H);

^{13}C NMR (75.48 MHz, $CDCl_3$) δ 165.35, 162.91 (d, *J* = 249.1 Hz), 148.90, 131.83 (d, *J* = 8.2 Hz), 128.09 (d, *J* = 3.5 Hz), 116.24 (d, *J* = 21.3 Hz), 107.55, 58.90, 41.87, 29.54;

HRMS (ESI-TOF) m/z [M + Na]$^+$: Calcd for $[C_{14}H_{12}FN_3NaO_3S]^+$: 344.0476. Found: 344.0489.

1,3-Dimethyl-5-(2-nitrobenzyl)-5-thiocyanatopyrimidine-2,4,6(1*H*,3*H*,5*H*)-trione, 2g



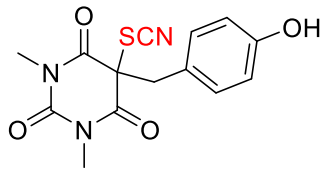
Yield was 45% (0.45 mmol, 155.7 mg). Yellow solid (mp = 142-143 °C). R_f = 0.39 (PE:EtOAc = 2:1);

^{1}H NMR (300.13 MHz, $CDCl_3$) δ 8.01 (dd, *J* = 7.7, 1.6 Hz, 1H), 7.63 (td, *J* = 7.7, 1.6 Hz, 1H), 7.54 (td, *J* = 7.7, 1.6 Hz, 1H), 7.42 (dd, *J* = 7.7, 1.6 Hz, 1H), 4.08 (s, 2H), 3.29 (s, 6H);

^{13}C NMR (75.48 MHz, $CDCl_3$) δ 165.08, 149.51, 149.10, 133.64, 133.13, 130.15, 127.45, 125.92, 107.90, 59.59, 38.79, 29.86;

HRMS (ESI-TOF) m/z $[M + K]^+$: Calcd for $[C_{14}H_{12}N_4KO_5S]^+$: 387.0160. Found: 387.0158.

5-(4-Hydroxybenzyl)-1,3-dimethyl-5-thiocyanatopyrimidine-2,4,6(1*H*,3*H*,5*H*)-trione, 2h



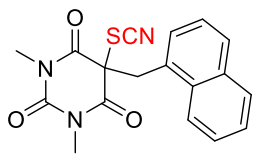
Yield was 83% (0.83 mmol, 265.1 mg). Yellow solid (mp = 155-156 °C). R_f = 0.31 (PE:EtOAc = 2:1);

^{1}H NMR (300.13 MHz, DMSO-d_6) δ 9.58 (s br., 1H), 6.79 (d, *J* = 8.6 Hz, 2H), 6.67 (d, *J* = 8.6 Hz, 2H), 3.45 (s, 2H), 3.01 (s, 6H);

^{13}C NMR (75.48 MHz, DMSO-d_6) δ 166.28, 157.54, 149.28, 130.58, 121.98, 115.39, 110.40, 61.50, 45.64, 28.86;

HRMS (ESI-TOF) m/z $[M + Na]^+$: Calcd for $[C_{14}H_{13}N_3NaO_4S]^+$: 342.0519. Found: 342.0512.

1,3-Dimethyl-5-(naphthalen-1-ylmethyl)-5-thiocyanatopyrimidine-2,4,6(1*H*,3*H*,5*H*)-trione, 2i



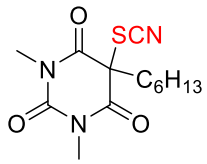
Yield was 67% (0.67 mmol, 235.5 mg). Yellow solid (mp = 152-153 °C). R_f = 0.24 (PE:EtOAc = 5:1);

^{1}H NMR (300.13 MHz, $CDCl_3$) δ 7.85-7.77 (m, 3H), 7.55-7.45 (m, 2H), 7.36-7.30 (m, 1H), 7.17-7.15 (m, 1H), 4.11 (s, 2H), 2.78 (s, 6H);

^{13}C NMR (75.48 MHz, $CDCl_3$) δ 165.62, 148.41, 133.62, 131.22, 129.97, 129.08, 128.37, 127.95, 126.95, 126.32, 124.77, 122.78, 108.73, 62.54, 42.52, 29.12;

HRMS (ESI-TOF) m/z [M + Na]$^+$: Calcd for $[C_{18}H_{15}N_3NaO_3S]^+$: 376.0726. Found: 376.0717.

5-Hexyl-1,3-dimethyl-5-thiocyanatopyrimidine-2,4,6(1*H*,3*H*,5*H*)-trione, 2j



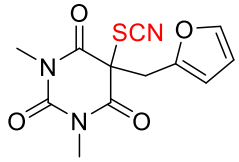
Yield was 87% (0.87 mmol, 258.6 mg). Colorless oil. R_f = 0.29 (PE:EtOAc = 5:1);

^{1}H NMR (300.13 MHz, $CDCl_3$) δ 3.40 (s, 6H), 2.32-2.26 (m, 2H), 1.33-1.12 (m, 8H), 0.85 (t, *J* = 6.8 Hz, 3H);

^{13}C NMR (75.48 MHz, $CDCl_3$) δ 165.74, 149.53, 107.61, 57.45, 35.86, 31.22, 29.70, 28.94, 25.65, 22.45, 14.02;

HRMS (ESI-TOF) m/z [M + Na]$^+$: Calcd for $[C_{13}H_{19}N_3NaO_3S]^+$: 320.1039. Found: 320.1031.

5-(Furan-2-ylmethyl)-1,3-dimethyl-5-thiocyanatopyrimidine-2,4,6(1*H*,3*H*,5*H*)-trione, 2k



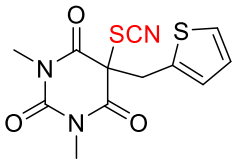
Yield was 69% (0.69 mmol, 201.8 mg). Yellow oil. R_f = 0.25 (PE:EtOAc = 5:1);

^{1}H NMR (300.13 MHz, $CDCl_3$) δ 7.28 (dd, *J* = 1.9, 0.9 Hz, 1H), 6.29 (dd, *J* = 3.3, 1.9 Hz, 1H), 6.16 (dd, *J* = 3.3, 0.9 Hz, 1H), 3.73 (s, 2H), 3.31 (s, 6H);

^{13}C NMR (75.48 MHz, $CDCl_3$) δ 165.11, 149.22, 146.59, 143.39, 111.05, 110.31, 107.39, 57.07, 36.04, 29.63;

HRMS (ESI-TOF) m/z [M + Na]$^+$: Calcd for $[C_{12}H_{11}N_3NaO_4S]^+$: 316.0362. Found: 316.0368.

1,3-Dimethyl-5-thiocyanato-5-(thiophen-2-ylmethyl)pyrimidine-2,4,6(1*H*,3*H*,5*H*)-trione, 2l



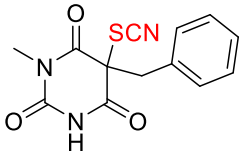
Yield was 65% (0.65 mmol, 202.3 mg). Colorless oil. R_f = 0.35 (PE:EtOAc = 5:1);

^{1}H NMR (300.13 MHz, $CDCl_3$) δ 7.20 (dd, *J* = 5.1, 1.3 Hz, 1H), 6.89 (dd, *J* = 5.1, 3.5 Hz, 1H), 6.84-6.83 (m, 1H), 3.86 (s, 2H), 3.28 (s, 6H);

^{13}C NMR (75.48 MHz, $CDCl_3$) δ 165.20, 149.08, 133.09, 129.48, 127.46, 127.10, 107.25, 57.67, 36.63, 29.59;

HRMS (ESI-TOF) m/z [M + Na]$^+$: Calcd for $[C_{12}H_{11}N_3NaO_3S_2]^+$: 332.0134. Found: 332.0141.

5-Benzyl-1-methyl-5-thiocyanatopyrimidine-2,4,6(1*H*,3*H*,5*H*)-trione, 2m



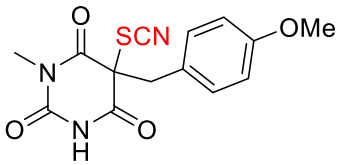
Yield was 86% (0.86 mmol, 249.7 mg). Yellow solid (mp = 123-124 °C). R_f = 0.51 (PE:EtOAc = 2:1);

^{1}H NMR (300.13 MHz, $CDCl_3$) δ 9.05 (s, 1H), 7.30-7.24 (m, 3H), 7.13-7.10 (m, 2H), 3.63 (s, 2H), 3.20 (s, 3H);

^{13}C NMR (75.48 MHz, $CDCl_3$) δ 166.14, 164.99, 147.96, 131.86, 129.98, 129.26, 129.02, 107.51, 59.06, 42.39, 28.78.

HRMS (ESI-TOF) m/z [M + Na]$^+$: Calcd for $[C_{13}H_{11}N_3NaO_3S]^+$: 312.0413. Found: 312.0409.

5-(4-Methoxybenzyl)-1-methyl-5-thiocyanatopyrimidine-2,4,6(1*H*,3*H*,5*H*)-trione, 2n



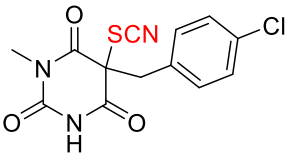
Yield was 89% (0.89 mmol, 283.6 mg). Yellow solid (mp = 124-125 °C). R_f = 0.46 (PE:EtOAc = 2:1);

^{1}H NMR (300.13 MHz, $CDCl_3$) δ 8.62 (s, 1H), 7.03 (d, *J* = 8.8 Hz, 2H), 6.79 (d, *J* = 8.8 Hz, 2H), 3.77 (s, 3H), 3.57 (s, 2H), 3.22 (s, 3H);

^{13}C NMR (75.48 MHz, $CDCl_3$) δ 166.27, 164.94, 159.95, 147.77, 131.29, 123.69, 114.65, 107.54, 58.90, 55.37, 41.65, 28.86;

HRMS (ESI-TOF) m/z [M + Na]$^+$: Calcd for [$C_{14}H_{13}N_3NaO_4S$]$^+$: 342.0519. Found: 342.0510.

5-(4-Chlorobenzyl)-1-methyl-5-thiocyanatopyrimidine-2,4,6(1*H*,3*H*,5*H*)-trione, 2o



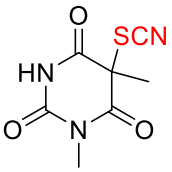
Yield was 51% (0.51 mmol, 166.4 mg). White solid (mp = 106-107 °C). R_f = 0.16 (PE:EtOAc = 5:1);

^{1}H NMR (300.13 MHz, $CDCl_3$) δ 9.08 (s, 1H), 7.25 (d, *J* = 8.7 Hz, 2H), 7.08 (d, *J* = 8.6 Hz, 2H), 3.62 (s, 2H), 3.24 (s, 3H);

^{13}C NMR (75.48 MHz, $CDCl_3$) δ 165.82, 164.66, 147.92, 135.12, 131.62, 130.49, 129.48, 107.16, 58.09, 40.67, 28.91;

HRMS (ESI-TOF) m/z [M + Na]$^+$: Calcd for [$C_{13}H_{10}ClN_3NaO_3S$]$^+$: 346.0024. Found: 346.0023.

1,5-Dimethyl-5-thiocyanatopyrimidine-2,4,6(1*H*,3*H*,5*H*)-trione, 2p



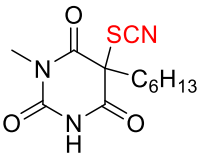
Yield was 91% (0.91 mmol, 193.5 mg). Yellow solid (mp = 115-116 °C). R_f = 0.35 (PE:EtOAc = 2:1);

^{1}H NMR (300.13 MHz, $CDCl_3$) δ 9.31 (s br., 1H), 3.36 (s, 3H), 1.91 (s, 3H);

^{13}C NMR (75.48 MHz, $CDCl_3$) δ 166.66, 165.62, 148.83, 107.57, 53.36, 29.13, 20.60;

HRMS (ESI-TOF) m/z [M + Na]$^+$: Calcd for [$C_7H_7N_3NaO_3S$]$^+$: 236.0100. Found: 236.0105.

5-Hexyl-1-methyl-5-thiocyanatopyrimidine-2,4,6(1*H*,3*H*,5*H*)-trione, 2q



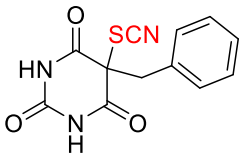
Yield was 18% (0.18 mmol, 50.9 mg). Colorless oil. R_f = 0.27 (PE:EtOAc = 5:1);

^{1}H NMR (300.13 MHz, $CDCl_3$) δ 9.42 (br.s, 1H), 3.38 (s, 3H), 2.31-2.26 (m, 2H), 1.31-1.19 (m, 8H), 0.85 (t, *J* =6.7 Hz, 3H);

^{13}C NMR (75.48 MHz, $CDCl_3$) δ 166.41, 165.28, 148.66, 107.49, 57.50, 35.59, 31.17, 29.03, 28.90, 25.56, 22.42, 14.00;

HRMS (ESI-TOF) m/z [M + Na]$^+$: Calcd for $[C_{12}H_{17}N_3NaO_3S]^+$: 306.0883. Found: 306.0878.

5-Benzyl-5-thiocyanatopyrimidine-2,4,6(1*H*,3*H*,5*H*)-trione, 2r



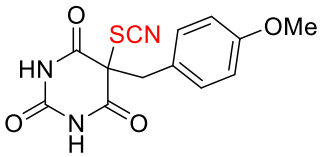
Yield was 40% (0.40 mmol, 110.7 mg). White solid (mp = 184-185 °C). R_f = 0.40 (PE:EtOAc = 2:1);

^{1}H NMR (300.13 MHz, DMSO-d_6) δ 11.95 (s, 2H), 7.33-7.31 (m, 3H), 7.11-7.08 (m, 2H), 3.50 (s, 2H);

^{13}C NMR (75.48 MHz, DMSO-d_6) δ 167.18, 148.46, 132.59, 129.88, 128.75, 128.33, 109.93, 58.98, 42.29;

HRMS (ESI-TOF) m/z [M + Na]$^+$: Calcd for $[C_{12}H_9N_3NaO_3S]^+$: 298.0257. Found: 298.0256.

5-(4-Methoxybenzyl)-5-thiocyanatopyrimidine-2,4,6(1*H*,3*H*,5*H*)-trione, 2s



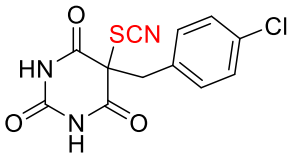
Yield was 35% (0.35 mmol, 106.2 mg). Yellow solid (mp = 197-198 °C). R_f = 0.21 (PE:EtOAc = 2:1);

^{1}H NMR (300.13 MHz, DMSO-d_6) δ 11.94 (s, 2H), 7.01 (d, J = 8.8 Hz, 2H), 6.87 (d, J = 8.8 Hz, 2H), 3.72 (s, 3H), 3.43 (s, 2H);

^{13}C NMR (75.48 MHz, DMSO-d_6) δ 167.29, 159.11, 148.54, 131.14, 124.30, 114.12, 109.94, 58.88, 55.11, 41.59;

HRMS (ESI-TOF) m/z [M + Na]$^+$: Calcd for $[C_{13}H_{11}N_3NaO_4S]^+$: 328.0362. Found: 328.0362.

5-(4-Chlorobenzyl)-5-thiocyanatopyrimidine-2,4,6(1*H*,3*H*,5*H*)-trione, 2t



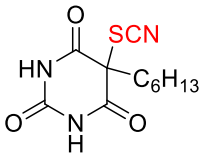
Yield was 65% (0.65 mmol, 203.4 mg). Yellow solid (mp = 173-174 °C). R_f = 0.34 (PE:EtOAc = 2:1);

^{1}H NMR (300.13 MHz, DMSO-d_6) δ 11.98 (s, 2H), 7.41 (d, J = 8.5 Hz, 2H), 7.12 (d, J = 8.5 Hz, 2H), 3.51 (s, 2H);

^{13}C NMR (75.48 MHz, DMSO-d_6) δ 167.01, 148.50, 133.08, 131.82, 131.66, 128.70, 109.87, 58.88, 41.24;

HRMS (ESI-TOF) m/z [M + Na]$^+$: Calcd for [$C_{12}H_8ClN_3NaO_3S$]$^+$: 331.9867. Found: 331.9871.

5-Hexyl-5-thiocyanatopyrimidine-2,4,6(1*H*,3*H*,5*H*)-trione, 2u



Yield was 22% (0.22 mmol, 58.1 mg). Yellow solid (mp = 89-90 °C). R_f = 0.67 (PE:EtOAc = 2:1);

^{1}H NMR (300.13 MHz, $CDCl_3$) δ 9.53 (s, 2H), 2.31-2.26 (m, 2H), 1.33-1.25 (m, 8H), 0.86 (t, J = 6.7 Hz, 3H);

^{13}C NMR (75.48 MHz, $CDCl_3$) δ 166.19, 148.21, 107.68, 57.55, 35.44, 31.19, 28.95, 25.48, 22.46, 14.04;

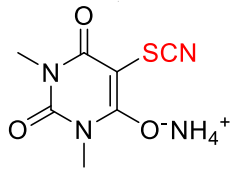
HRMS (ESI-TOF) m/z [M + Na]$^+$: Calcd for [$C_{11}H_{15}N_3NaO_3S$]$^+$: 292.0726. Found: 292.0725.

Experimental Procedures for Scheme 5.

Electrochemical synthesis of ammonium salts α-thiocyanobarbiturates 4a-b from different α-unsubstituted barbituric acids 3a-b.

An undivided cell was equipped with a glassy carbon anode (4.5 cm^2) and a platinum plate cathode (4.5 cm^2) and connected to a DC regulated power supply. The solution of α-unsubstituted barbituric acids **3a-b** (1.0 mmol, 142.1-156.1 mg) and NH_4SCN (4 eq., 4.0 mmol, 304.4 mg) in CH_3CN (15 mL) was electrolyzed using constant current conditions I = 320 mA (j_{anode} = 71 mA/cm^2) at 20-25 °C under magnetic stirring (3 F/mol, 15 min). After completion of the reaction, the pure compound **4a-b** was obtained by filtration of the reaction mixture and washing of the residue with CH_3CN (5 mL).

Ammonium 1,3-dimethyl-2,6-dioxo-5-thiocyanato-1,2,3,6-tetrahydropyrimidin-4-olate, 4a



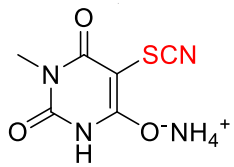
Yield was 76% (0.76 mmol, 174.9 mg). Orange solid;

^{1}H NMR (300.13 MHz, DMSO-d_6) δ 7.10 (s, 4H), 3.09 (s, 6H);

^{13}C NMR (75.48 MHz, DMSO-d_6) δ 162.16, 152.44, 115.06, 66.17, 27.72;

HRMS (ESI-TOF) m/z [M-NH_4]$^-$: Calcd for [$C_7H_6N_3O_3S$]$^-$: 212.0135. Found: 212.0131.

Ammonium 1-methyl-2,6-dioxo-5-thiocyanato-1,2,3,6-tetrahydropyrimidin-4-olate, 4b



Yield was 75% (0.75 mmol, 161.6 mg). Yellow solid;

^{1}H NMR (300.13 MHz, DMSO-d_6) δ 9.95 (s, 1H), 7.14 (s, 4H), 3.02 (s, 3H);

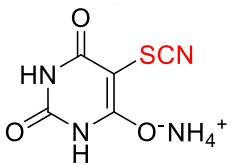
^{13}C NMR (75.48 MHz, DMSO-d_6) δ 163.54, 162.73, 151.99, 115.13, 65.82, 26.94;

HRMS (ESI-TOF) m/z [M-NH_4]$^-$: Calcd for [$C_6H_4N_3O_3S$]$^-$: 197.9979. Found: 197.9976.

Electrochemical synthesis of ammonium salt α-thiocyanobarbiturate 4c from α-unsubstituted barbituric acid 3c.

An undivided cell was equipped with a glassy carbon anode (4.5 cm^2) and a platinum plate cathode (4.5 cm^2) and connected to a DC regulated power supply. The solution of α-unsubstituted barbituric acid **3c** (1.0 mmol, 128.1 mg) and NH_4SCN (4 eq., 4.0 mmol, 304.4 mg) in CH_3OH (15 mL) was electrolyzed using constant current conditions I = 320 mA (j_{anode} = 71 mA/cm^2) at 20-25 °C under magnetic stirring (3 F/mol, 15 min). After completion of the reaction, the pure compound **4c** was obtained by filtration of the reaction mixture and washing of the residue with CH_3OH (5 mL).

Ammonium 2,6-dioxo-5-thiocyanato-1,2,3,6-tetrahydropyrimidin-4-olate, 4c



Yield was 72% (0.72 mmol, 146.1 mg). White solid;

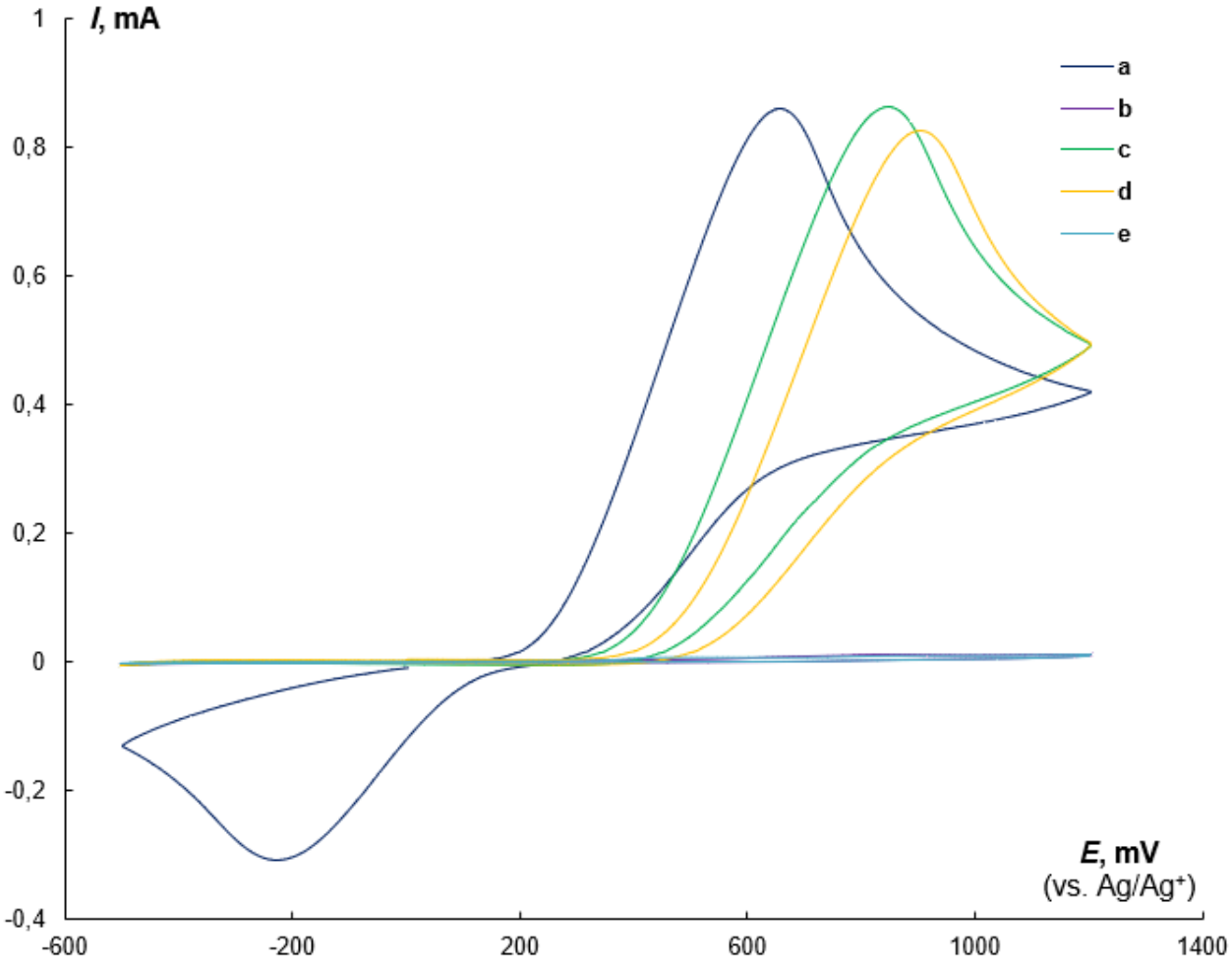
^{1}H NMR (300.13 MHz, DMSO-d_6) δ 9.75 (s, 2H), 7.15 (s, 4H);

^{13}C NMR (75.48 MHz, DMSO-d_6) δ 164.22, 151.66, 115.19, 65.28;

HRMS (ESI-TOF) m/z $[M\text{-}NH_4]^-$: Calcd for $[C_5H_2N_3O_3S]^-$: 183.9822. Found: 183.9824.

Experimental procedure for Figure 1. Cyclic voltammetry

Cyclic voltammetry (CV) was implemented on an IPC-Pro M computer-assisted potentiostat manufactured by Econix (scan rate error 1.0 %; potential setting 0.25 mV; scan rate 100 mV s^{-1}). The experiments were performed in a 2 mL five-neck glass conic electrochemical cell with a water jacket for thermostatting. CV curves were recorded using a three-electrode scheme. The working electrode was a disc glassy-carbon electrode (d = 3 mm). A platinum wire served as an auxiliary electrode. An Ag/Ag^+ electrode was used as the reference electrode and was linked to the solution by a porous glass diaphragm. The solutions were kept under thermally controlled conditions at 25±0.5 °C and deaerated by bubbling argon. Electrochemical experiments were performed under an argon atmosphere. The working electrode was polished before recording each CV curve. In a typical case, 2 mL of solution was utilized. The compound concentration was 0.05 M.



(a) NH_4SCN (0.05 M), (b) **1a** (0.05 M), (c) mixture **1a** (0.05 M) with NH_4SCN (0.05 M), (d) mixture **1a** (0.05 M) with NH_4SCN (0.05 M) and AcOH (0.05 M) in 0.1 M *n*-Bu_4NBF_4 in CH_3CN, (e) 0.1 M *n*-Bu_4NBF_4 in CH_3CN.

Experimental procedures for Scheme 6. Control experiments.

a) ***Divided cell (without AcOH):***

A divided cell was equipped with a glassy carbon anode (2.25 cm^2) and a platinum plate cathode (2.25 cm^2) and connected to a DC regulated power supply. The solution of α-benzyl barbituric acid **1a** (0.5 mmol, 123.1 mg), NH_4SCN (4.0 eq., 2.0 mmol, 152.2 mg) in CH_3CN (7 mL) (anodic compartment) and solution of NH_4SCN (4.0 eq., 2.0 mmol, 152.2 mg) in CH_3CN (7 mL) (cathode compartment) were electrolyzed using constant current conditions I = 30 mA (13 mA/cm^2) at 20-25 °C under magnetic stirring (3 F/mol, 80 min). The combined organic phases (anodic and cathode compartments) were concentrated under reduced pressure using a rotary evaporator (15-20 mmHg), (bath temperature, ca. 20-25 °C). Product **2a** was isolated by chromatography on SiO_2 (PE:EtOAc = from 10:1 to 5:1).

Divided cell (4.0 eq. AcOH):

A divided cell was equipped with a glassy carbon anode (2.25 cm^2) and a platinum plate cathode (2.25 cm^2) and connected to a DC regulated power supply. The solution of α-benzyl barbituric acid **1a** (0.5 mmol, 123.1 mg), NH_4SCN (4.0 eq., 2.0 mmol, 152.2 mg), acetic acid (2.0 mmol, 120 mg) in CH_3CN (7 mL) (anodic compartment) and solution of NH_4SCN (4 eq., 2 mmol, 152.2 mg) in CH_3CN (7 mL) (cathode compartment) were electrolyzed using constant current conditions I = 30 mA (13 mA/cm^2) at 20-25 °C under magnetic stirring (3 F/mol, 80 min). The combined organic phases (anodic and cathode compartments) were concentrated under reduced pressure using a rotary evaporator (15-20 mmHg), (bath temperature, ca. 20-25 °C). Product **2a** was isolated by chromatography on SiO_2 (PE:EtOAc = from 10:1 to 5:1).

Undivided cell (without AcOH):

An undivided cell was equipped with a glassy carbon anode (4.5 cm^2) and a platinum plate cathode (4.5 cm^2) and connected to a DC regulated power supply. The solution of α-benzyl barbituric acid **1a** (1.0 mmol, 246.3 mg) and NH_4SCN (4.0 eq., 4.0 mmol, 304.4 mg) in CH_3CN (15 mL) was electrolyzed using constant current conditions I = 60 mA (13 mA/cm^2) at 20-25 °C under magnetic stirring (3 F/mol, 80 min). The reaction mixture was concentrated under reduced pressure using a rotary evaporator (15-20 mmHg), (bath temperature, ca. 20-25 °C). Product **2a** was isolated by chromatography on SiO_2 (PE:EtOAc = from 10:1 to 5:1).

Undivided cell (4.0 eq. AcOH):

An undivided cell was equipped with a glassy carbon anode (4.5 cm^2) and a platinum plate cathode (4.5 cm^2) and connected to a DC regulated power supply. The

solution of α-benzyl barbituric acid **1a** (1.0 mmol, 246.3 mg), NH_4SCN (4 eq., 4.0 mmol, 304.4 mg) and acetic acid (4.0 mmol, 240 mg) in CH_3CN (15 mL) was electrolyzed using constant current conditions I = 60 mA (13 mA/cm^2) at 20-25 °C under magnetic stirring (3 F/mol, 80 min). The reaction mixture was concentrated under reduced pressure using a rotary evaporator (15-20 mmHg), (bath temperature, ca. 20-25 °C). Product **2a** was isolated by chromatography on SiO_2 (PE:EtOAc = from 10:1 to 5:1).

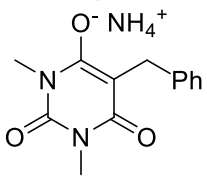
b) ***Without AcOH:***

An undivided cell was equipped with a glassy carbon anode (4.5 cm^2) and a platinum plate cathode (4.5 cm^2) and connected to a DC regulated power supply. The solution of ammonium 5-benzyl-1,3-dimethyl-2,6-dioxo-1,2,3,6-tetrahydropyrimidin-4-olate **5** (1.0 mmol, 263.3 mg) and NH_4SCN (4.0 eq., 4.0 mmol, 304.4 mg) in CH_3CN (15 mL) was electrolyzed using constant current conditions I = 320 mA (71 mA/cm^2) at 20-25 °C under magnetic stirring (3 F/mol, 15 min). The reaction mixture was concentrated under reduced pressure using a rotary evaporator (15-20 mmHg), (bath temperature, ca. 20-25 °C). Product **2a** was isolated by chromatography on SiO_2 (PE:EtOAc = from 10:1 to 5:1).

b) ***5.0 eq. AcOH:***

An undivided cell was equipped with a glassy carbon anode (4.5 cm^2) and a platinum plate cathode (4.5 cm^2) and connected to a DC regulated power supply. The solution of ammonium 5-benzyl-1,3-dimethyl-2,6-dioxo-1,2,3,6-tetrahydropyrimidin-4-olate **5** (1.0 mmol, 263.3 mg), NH_4SCN (4.0 eq., 4.0 mmol, 304.4 mg) and acetic acid (5.0 mmol, 300 mg) in CH_3CN (15 mL) was electrolyzed using constant current conditions I = 320 mA (71 mA/cm^2) at 20-25 °C under magnetic stirring (3 F/mol, 15 min). The reaction mixture was concentrated under reduced pressure using a rotary evaporator (15-20 mmHg), (bath temperature, ca. 20-25 °C). Product **2a** was isolated by chromatography on SiO_2 (PE:EtOAc = from 10:1 to 5:1).

Ammonium 5-benzyl-1,3-dimethyl-2,6-dioxo-1,2,3,6-tetrahydropyrimidin-4-olate, 5



Preparation of ammonium 5-benzyl-1,3-dimethyl-2,6-dioxo-1,2,3,6-tetrahydropyrimidin-4-olate (**5**): the solution of α-benzyl barbituric acid **1a** (1.0 mmol, 246.3 mg) in CH_3CN (15 mL) was flushed with NH_3 with stirring at 20-25 °C. After completion of the reaction,

pure compound **5** was obtained by filtration of the reaction mixture and washing the residue with CH_3CN (5 mL).

White solid;

^1H NMR (300.13 MHz, DMSO-d_6) δ 7.21-7.09 (m, 8H), 7.03-6.98 (m, 1H), 3.45 (s, 2H), 3.05 (s, 6H);

^{13}C NMR (75.48 MHz, DMSO-d_6) δ 162.26, 152.92, 145.17, 128.26, 127.35, 124.36, 84.96, 30.23, 27.03.

Experimental procedure for Scheme 6, c

An undivided cell was equipped with a glassy carbon anode (4.5 cm^2) and a platinum plate cathode (4.5 cm^2) and connected to a DC regulated power supply. The solution of α-benzyl barbituric acid **1a** (1.0 mmol, 246.3 mg), NH_4SCN (4.0 eq., 4.0 mmol, 304.4 mg) and acetic acid (4.0 mmol, 240 mg) in CH_3CN (15 mL) was electrolyzed using constant current conditions I = 320 mA (j_{anode} = 71 mA/cm^2) at 20-25 °C under magnetic stirring (3 F/mol, 15 min). pH of the reaction mixture was measurement every 90 sec.

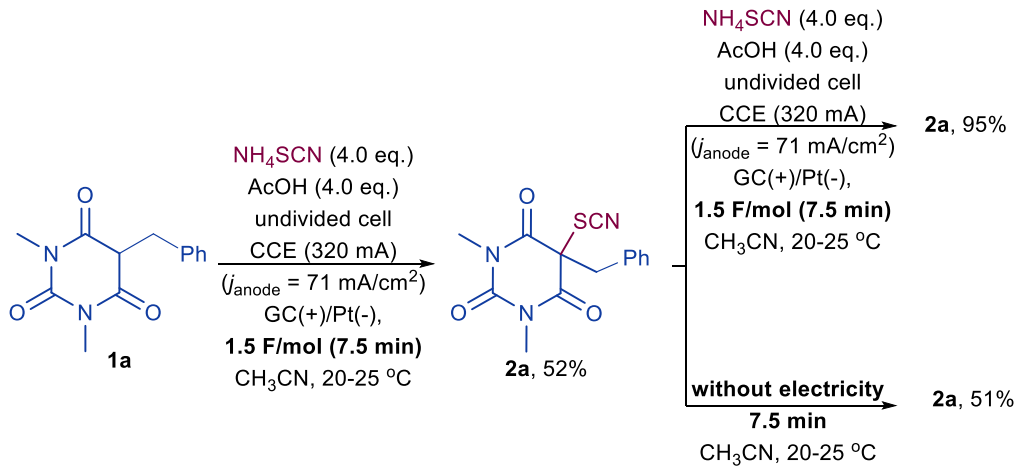
Experimental procedure for Scheme 6, d

An undivided cell was equipped with a glassy carbon anode (4.5 cm^2) and a platinum plate cathode (4.5 cm^2) and connected to a DC regulated power supply. The solution of α-benzyl barbituric acid **1a** (1.0 mmol, 246.3 mg), NH_4SCN (4.0 eq., 4.0 mmol, 304.4 mg), acetic acid (4.0 mmol, 240 mg) and 2,6-di-*tert*-butyl-4-methylphenol (BHT) (4.0 mmol, 881.4 mg) or (2,2,6,6-tetramethylpiperidin-1-yl)oxyl (TEMPO) (4.0 mmol, 625.0 mg) in CH_3CN (15 mL) was electrolyzed using constant current conditions I = 320 mA (j_{anode} = 71 mA/cm^2) at 20-25 °C under magnetic stirring (3 F/mol, 15 min). The reaction mixture was concentrated under reduced pressure using a rotary evaporator (15-20 mmHg), (bath temperature, ca. 20-25 °C). Product **2a** and substrate **1a** were isolated by chromatography on SiO_2 (PE:EtOAc = from 10:1 to 2:1).

Experimental procedure for Scheme 6, e

Bromine (2.0 mmol, 319.6 mg, 103 μL) was added to the solution of potassium thiocyanate (4.0 mmol, 388.8 mg) in acetic acid (3 mL) at 20-25 °C under stirring. Later, the solution of α-benzyl barbituric acid **1a** (1.0 mmol, 246.3 mg) in CH_3CN (15 mL) was added to the resulting solution of thiocyanogen at 20-25 °C under stirring. The reaction mixture was stirring for 15 min at 20-25 °C. Product **2a** was isolated by chromatography on SiO_2 (PE:EtOAc = from 10:1 to 5:1).

Control ON/OFF experiment



Scheme S1.

An undivided cell was equipped with a glassy carbon anode (4.5 cm^2) and a platinum plate cathode (4.5 cm^2) and connected to a DC regulated power supply. The solution of α-substituted barbituric acids **1a** (1.0 mmol, 246.3 mg), NH_4SCN (4.0 eq., 4.0 mmol, 304.4 mg) and acetic acid (4.0 mmol, 240 mg) in CH_3CN (15 mL) was electrolyzed using constant current conditions I = 320 mA (j_{anode} = 71 mA/cm^2) at 20-25 °C under magnetic stirring (1.5 F/mol, 7.5 min).

The reaction mixture was divided in half.

Aliquot 1. The half of the reaction mixture was concentrated under reduced pressure using a rotary evaporator (15-20 mmHg), (bath temperature, ca. 20-25 °C). Product **2a** (0.26 mmol, 78.9 mg) was isolated by chromatography on SiO_2 (PE:EtOAc).

Aliquot 2. The other half of the reaction mixture was standing for additional 7.5 min at 20-25 °C under magnetic stirring. Later, it was concentrated under reduced pressure using a rotary evaporator (15-20 mmHg), (bath temperature, ca. 20-25 °C). Product **2a** (0.25 mmol, 77.2 mg) was isolated by chromatography on SiO_2 (PE:EtOAc).

Bioassay of fungicidal activity

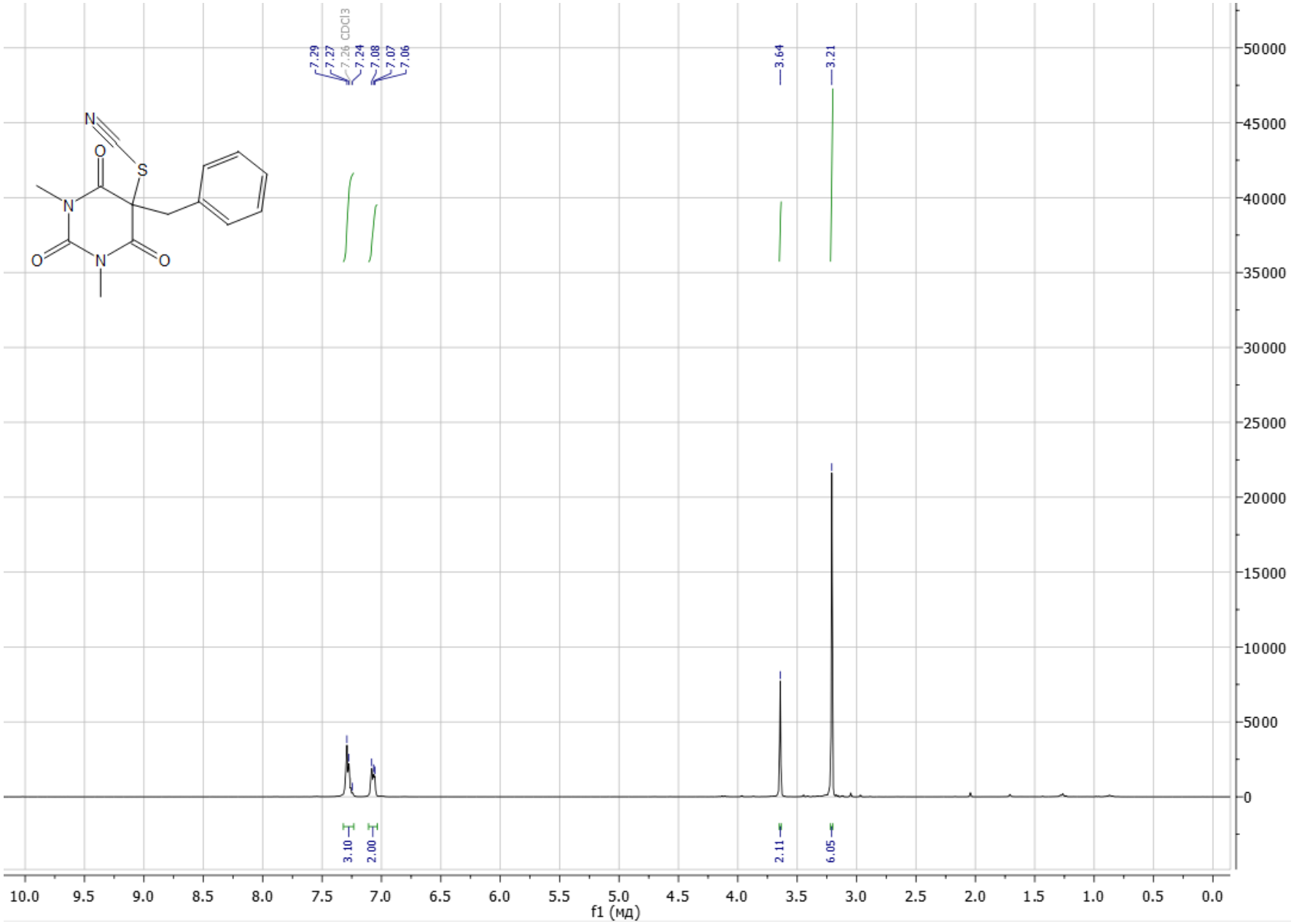
The antifungal activities were tested according to the conventional procedure [3] with six phytopathogenic fungi from different taxonomic classes: *Fusarium culmorum (F.c.), Rhizoctonia solani (R.s.), Alternaria solani (A.s.), Phytophthora infestans (P.i.), and Colletotrichum coccodes (C.c.)*. The effect of the chemicals on mycelial radial growth was determined by dissolving concentration 3 mg×mL^{-1} in acetone and suspending aliquots in potato-saccharose agar at 50 °C to give the concentration 30 μg×mL^{-1}. The final acetone concentration of both fungicide-containing and control samples was 10 mL×L^{-1}. Petri dishes containing 15 mL of the agar medium were inoculated by placing 2-mm micelial agar discs on the agar surface. Plates were incubated at 25 °C and radial growth was measured after 5 days. The mixed 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 5 days of culture. The growth inhibition rates were calculated with the following equation: I = [(DC – DT)/DC]×100%. Here I is the growth inhibition rates (%), DC is the control settlement diameter (mm), and DT is the treatment group fungi settlement diameter (mm). The results are summarized in Table 2.

References

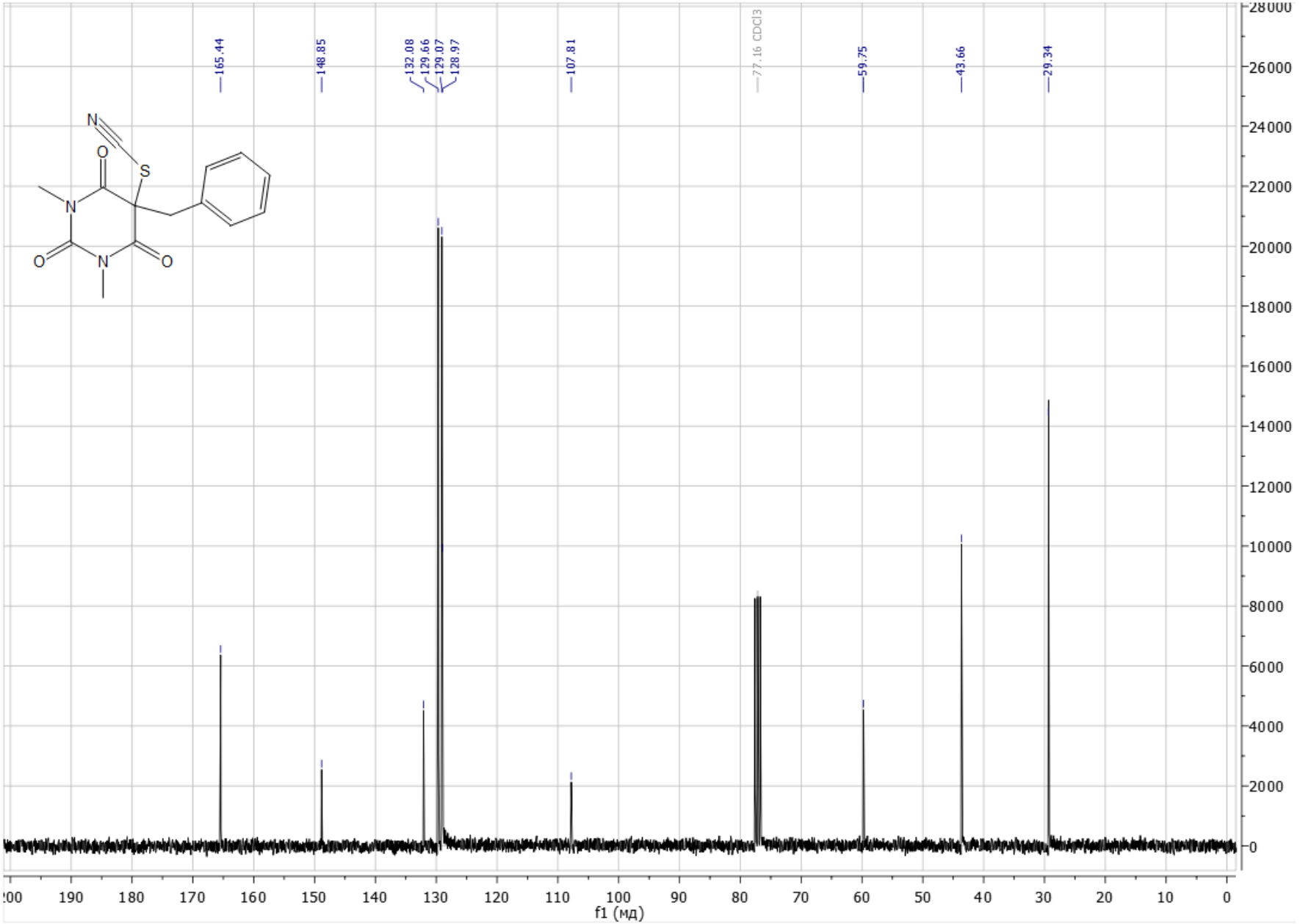
1. A. A. Al-Turkistani, O. A. Al-Deeb, N. R. El-Brollosy and A. A. El-Emam, *Molecules*, 2011, **16**.
2. M. Y. Sharipov, I. B. Krylov, I. D. Karpov, O. V. Vasilkova, A.-M. V. Oleksiienko and A. O. Terent'ev, *Chem. Heterocycl. Compd.*, 2021, **57**, 531-537.
3. (a) Metodicheskie rekomendatsii po opredeleniyu fungitsidnoi aktivnosti novykh soedinenii, NIITEKhIM, Cherkassy, 1984, pp. 32 (in Russian); (b) S. V. Popkov, L. V. Kovalenko, M. M. Bobylev, O. Y. Molchanov, M. Z. Krimer, V. P. Tashchi and Y. G. Putsykin, *Pesticide Science*, 1997, **49**, 125-129; (c) H. Itoh, H. Kajino, T. Tsukiyama, J. Tobitsuka, H. Ohta, Y. Takahi, M. Tsuda and H. Takeshiba, *Bioorg. Med. Chem.*, 2002, **10**, 4029-4034.

Copies of ^{1}H NMR, ^{13}C NMR and HRMS spectra

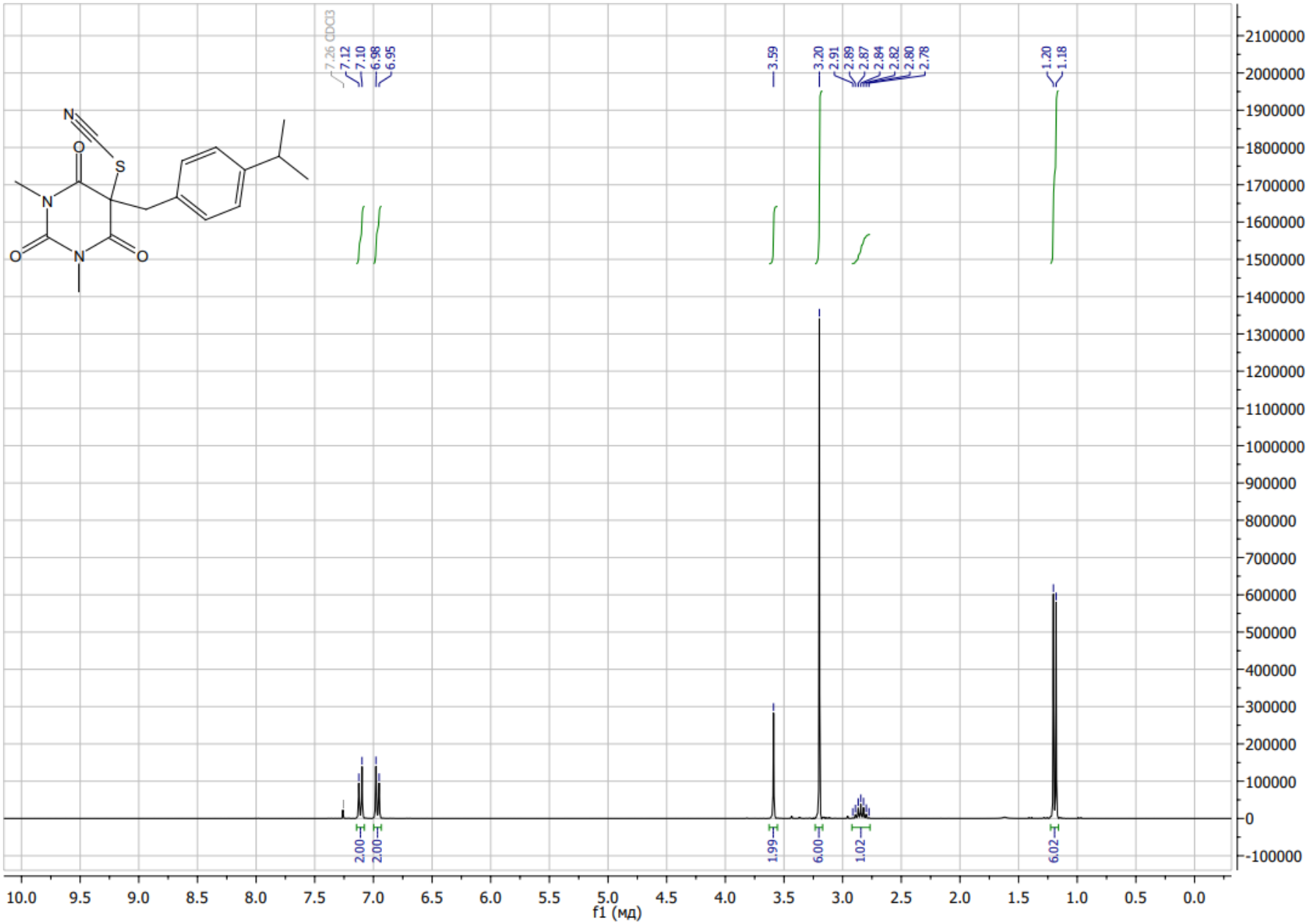
^{1}H NMR ($CDCl_3$) spectrum of 2a



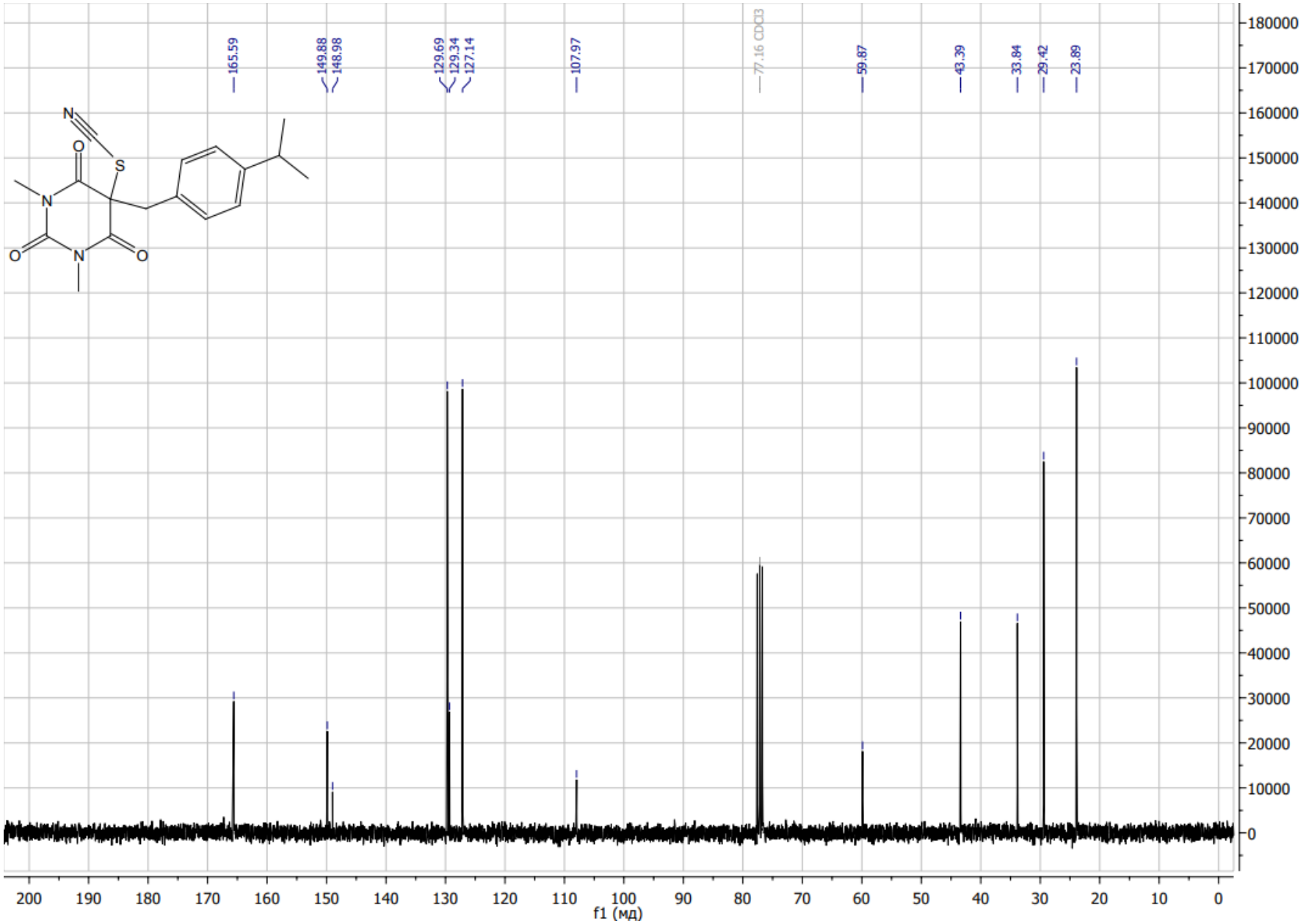
^{13}C NMR ($CDCl_3$) spectrum of 2a



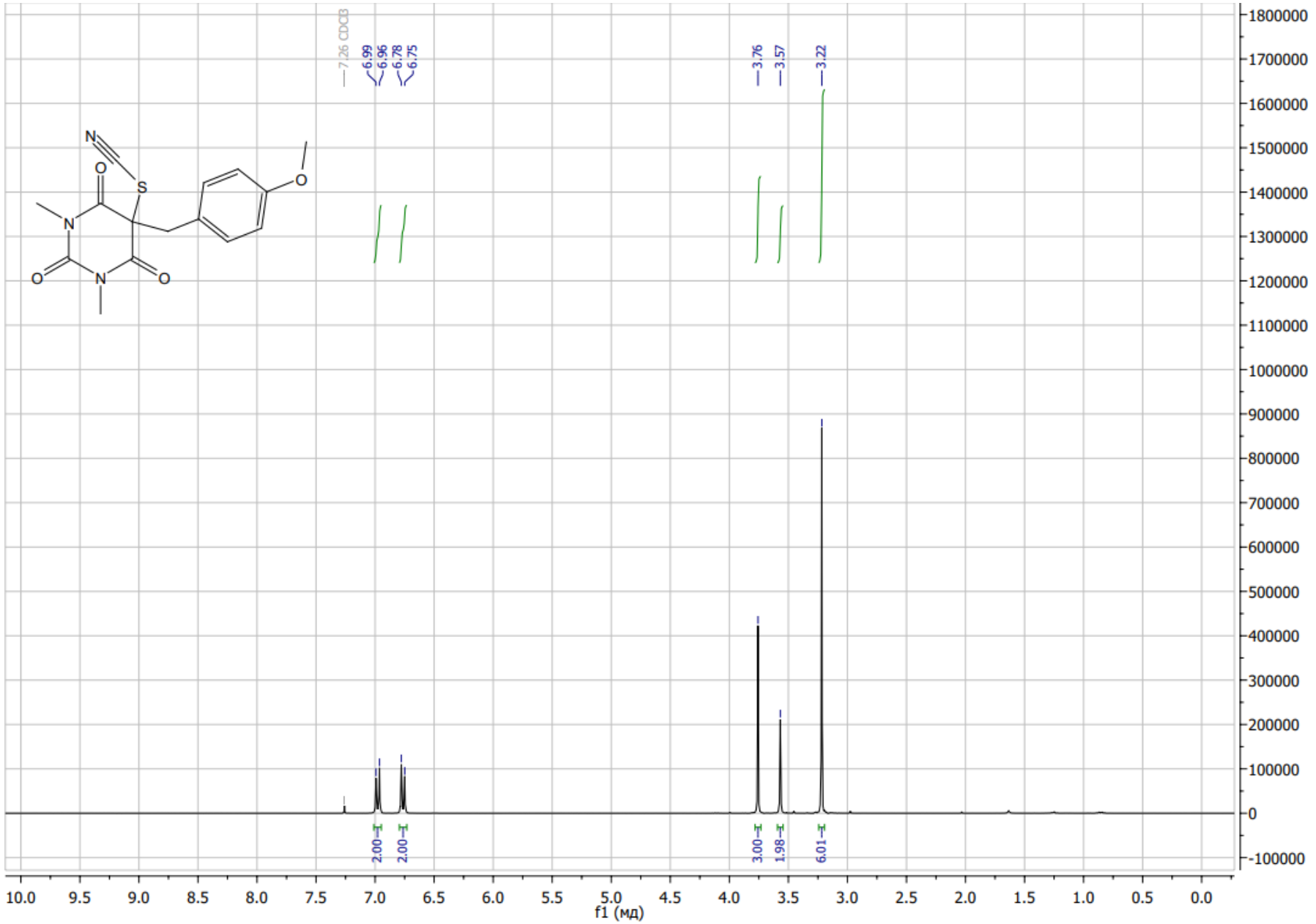
^{1}H NMR ($CDCl_3$) spectrum of 2b



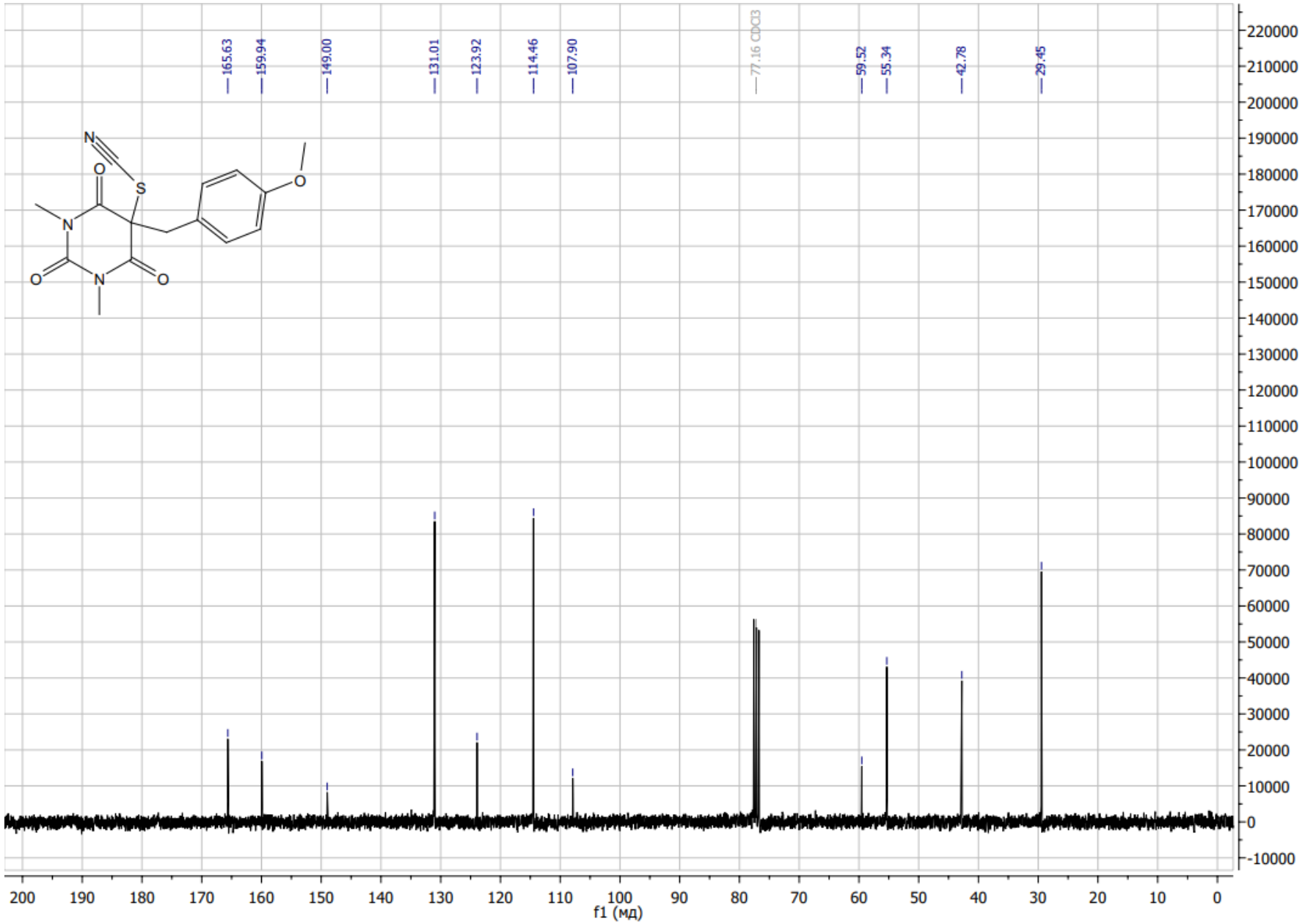
^{13}C NMR ($CDCl_3$) spectrum of 2b



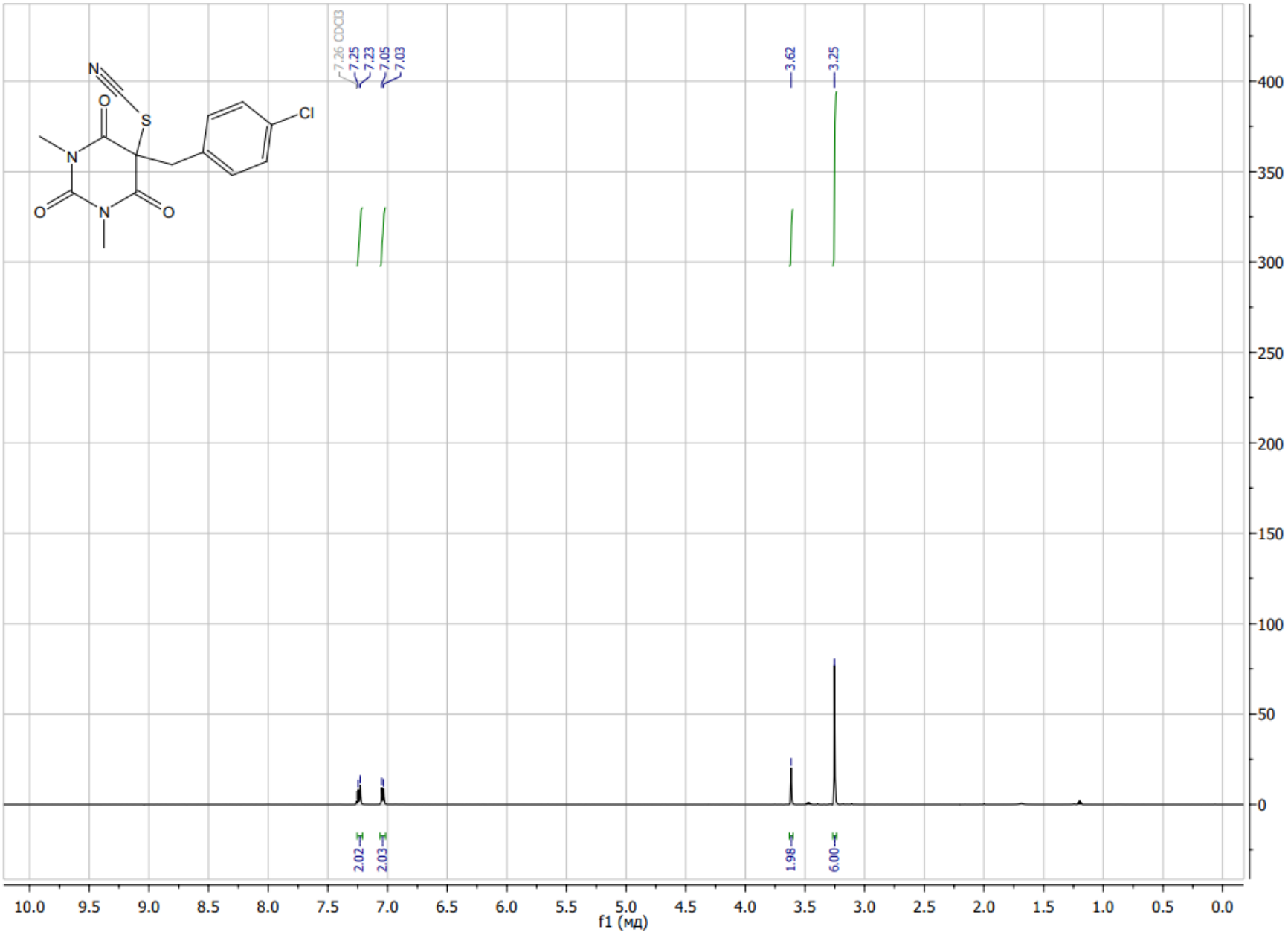
^{1}H NMR ($CDCl_3$) spectrum of 2c



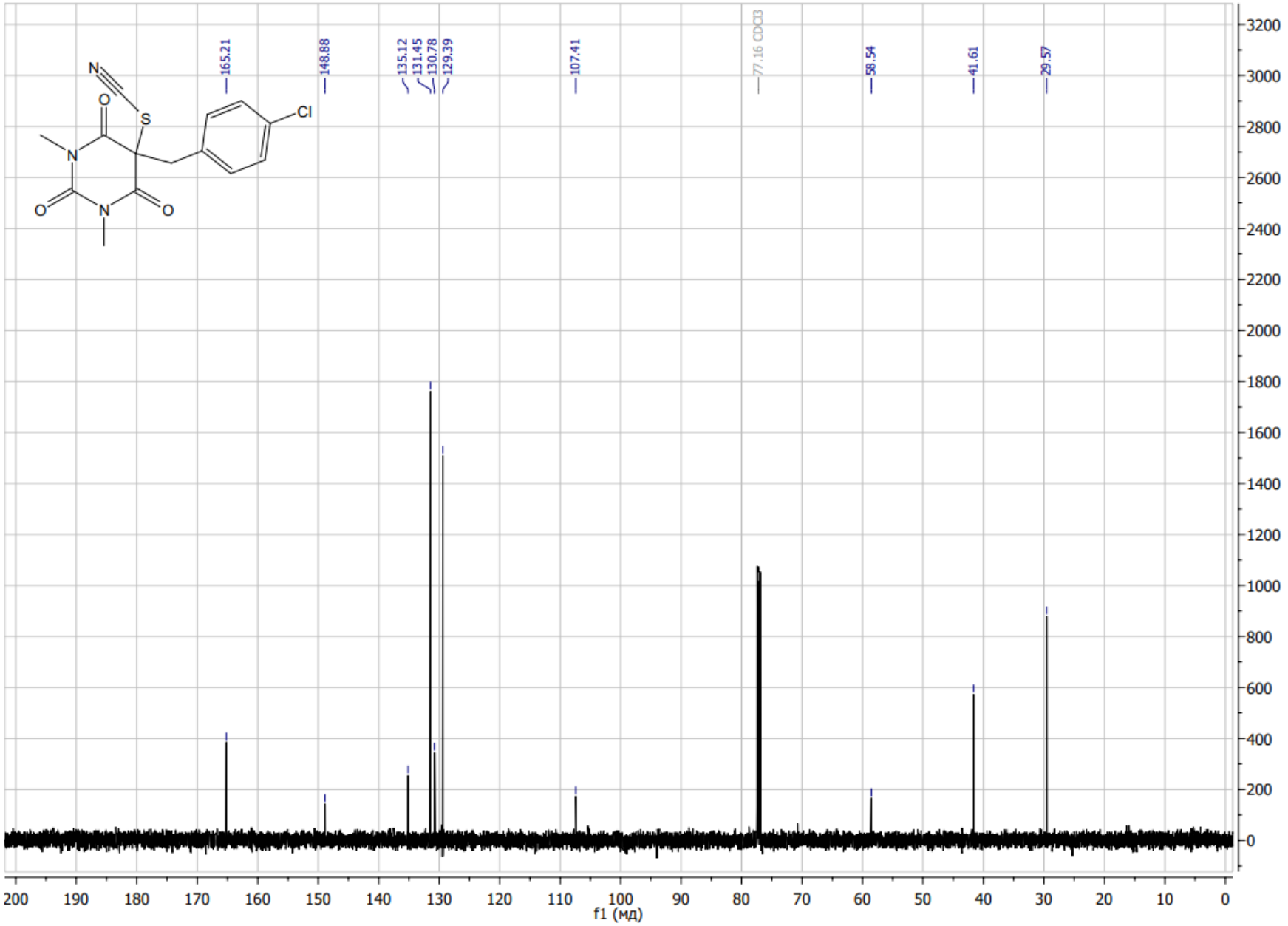
^{13}C NMR ($CDCl_3$) spectrum of 2c



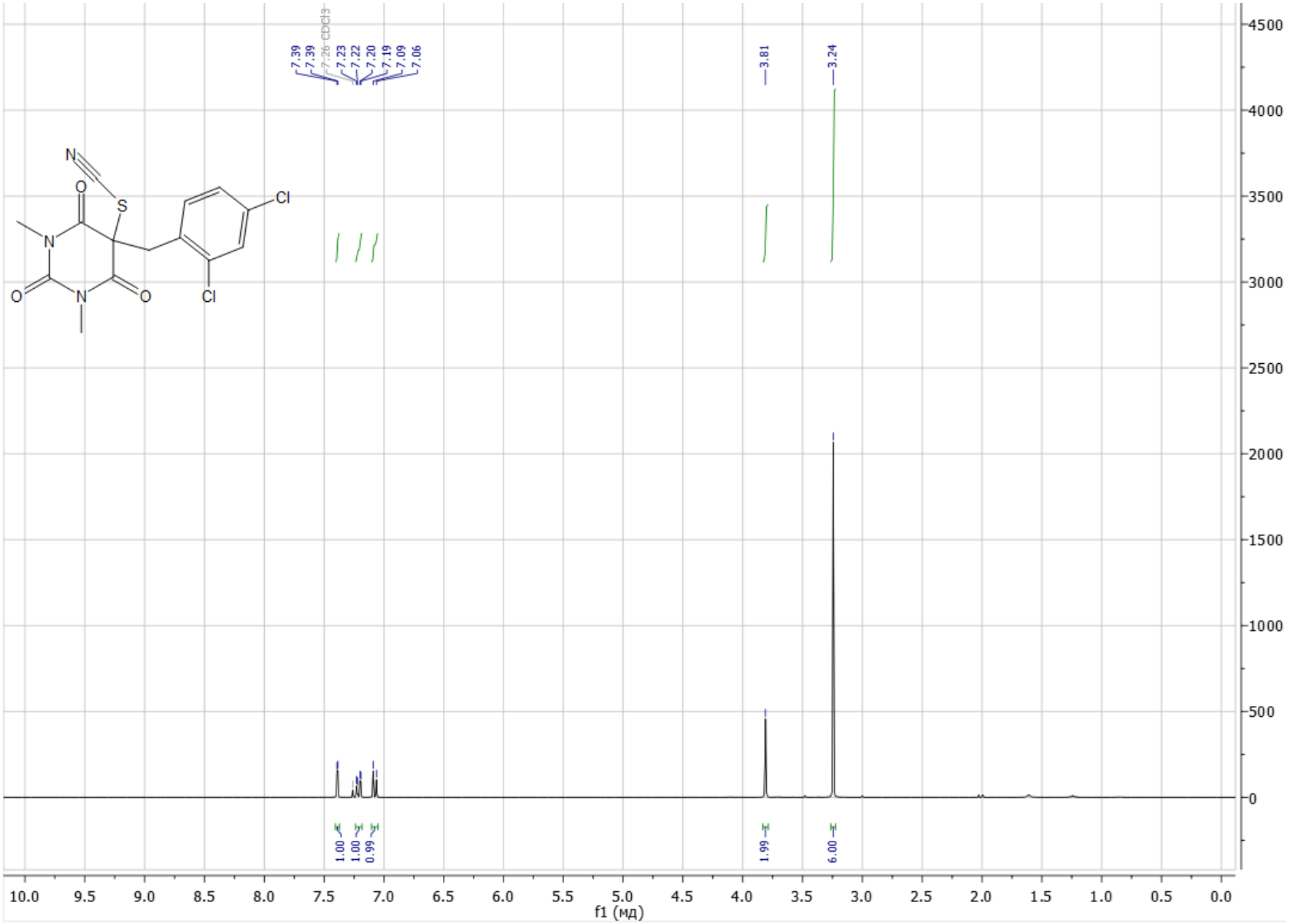
^{1}H NMR ($CDCl_3$) spectrum of 2d



^{13}C NMR ($CDCl_3$) spectrum of 2d



^{1}H NMR ($CDCl_3$) spectrum of 2e



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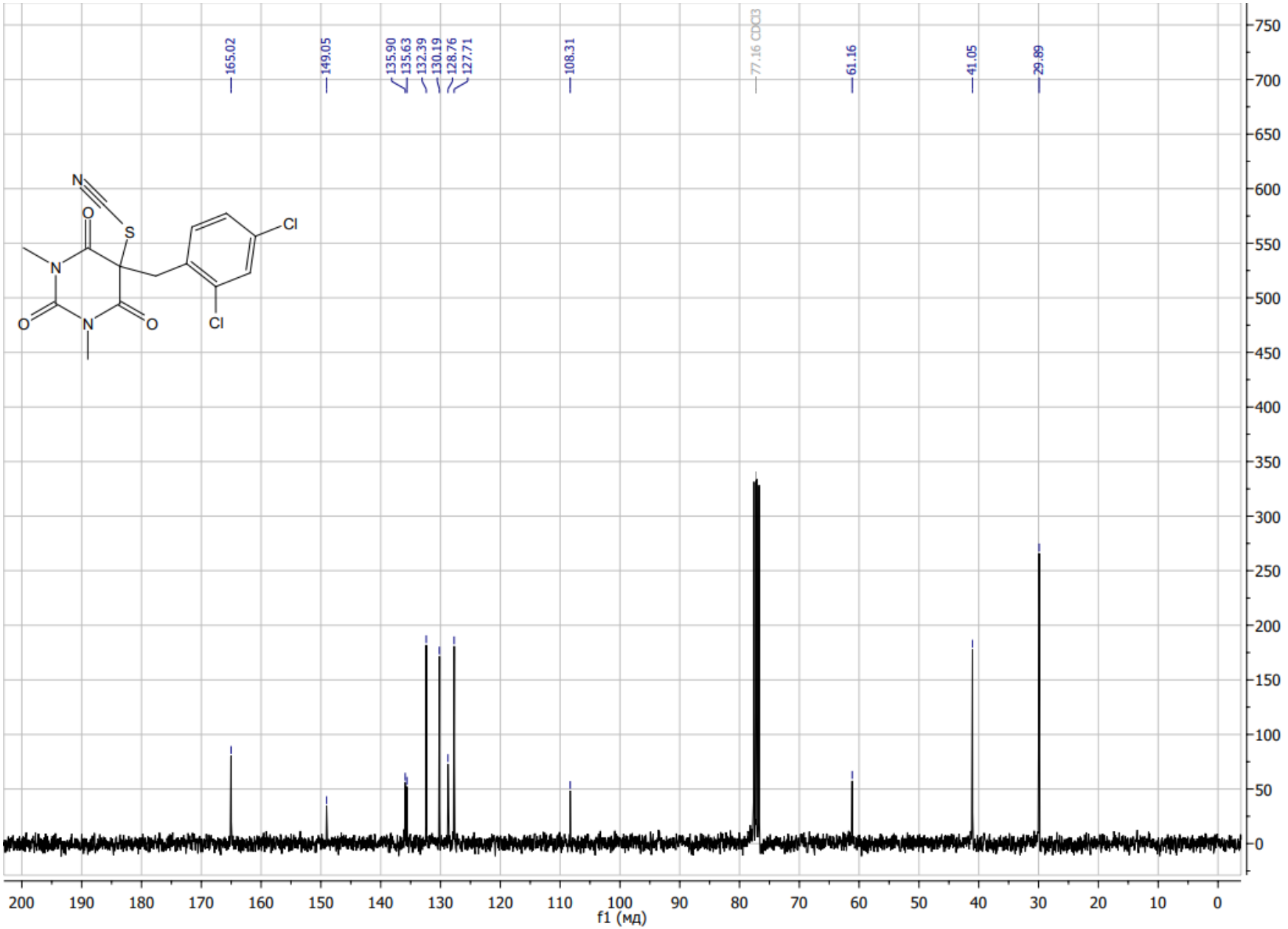
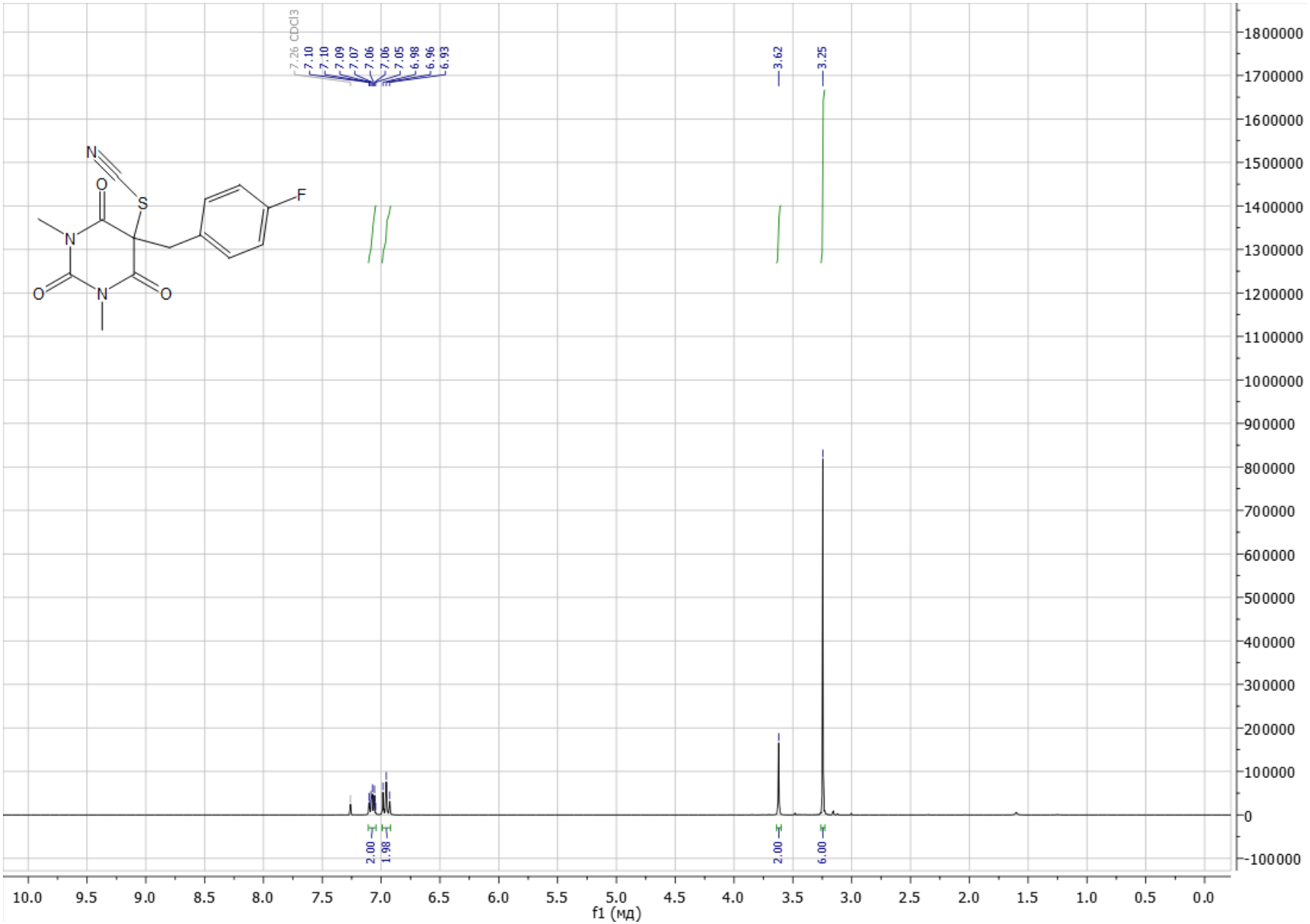
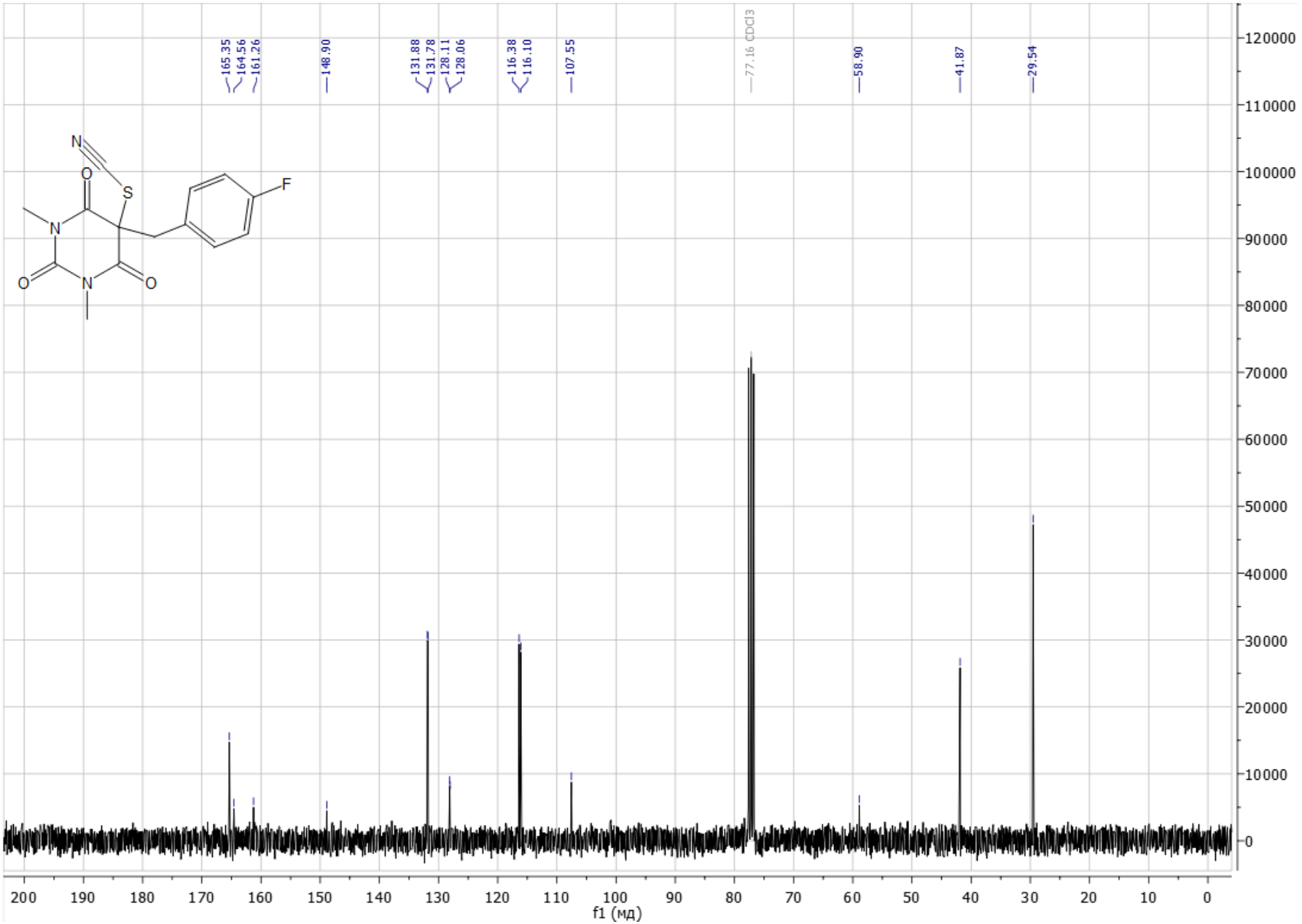


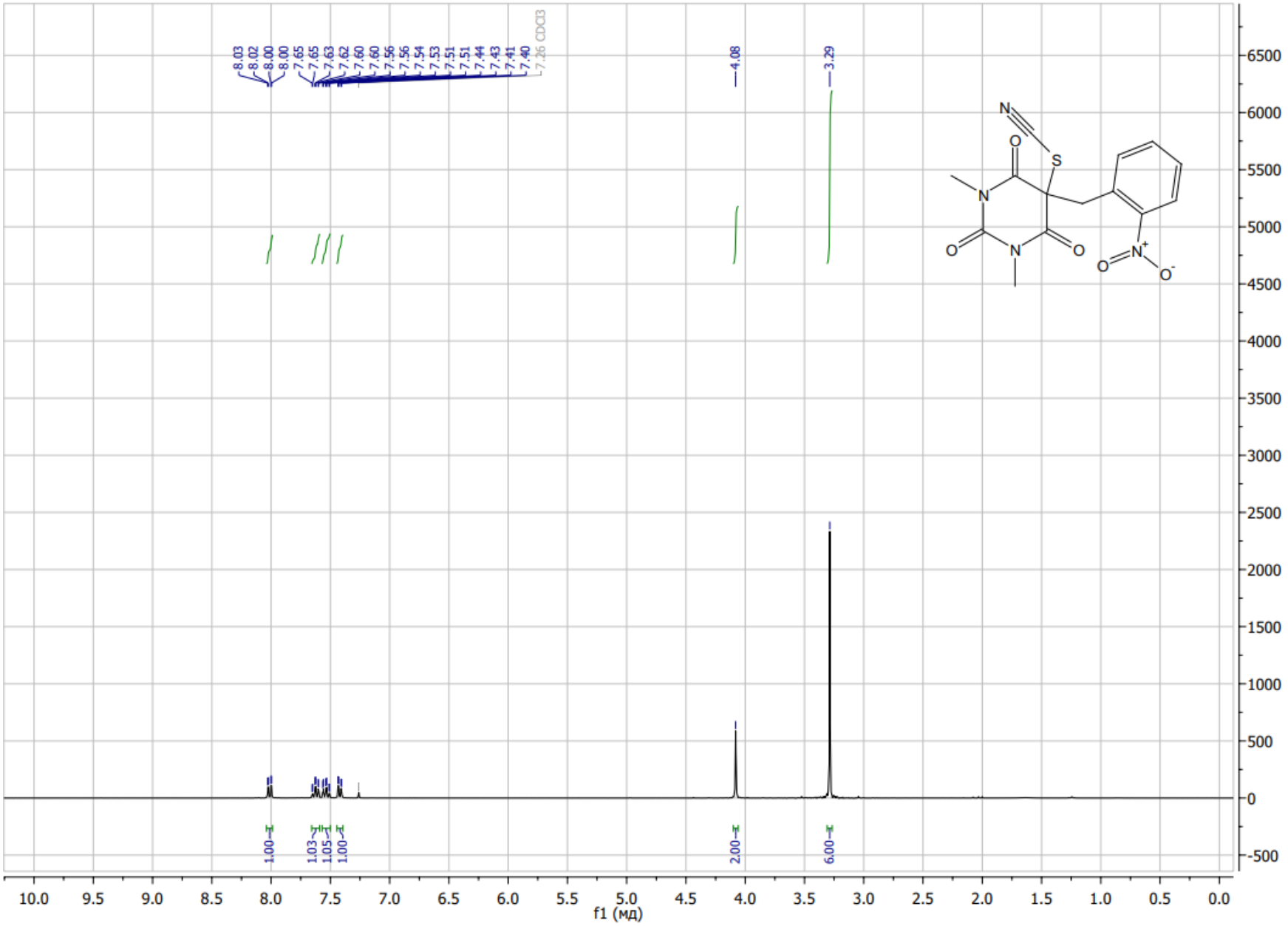
^{1}H NMR ($CDCl_3$) spectrum of 2f



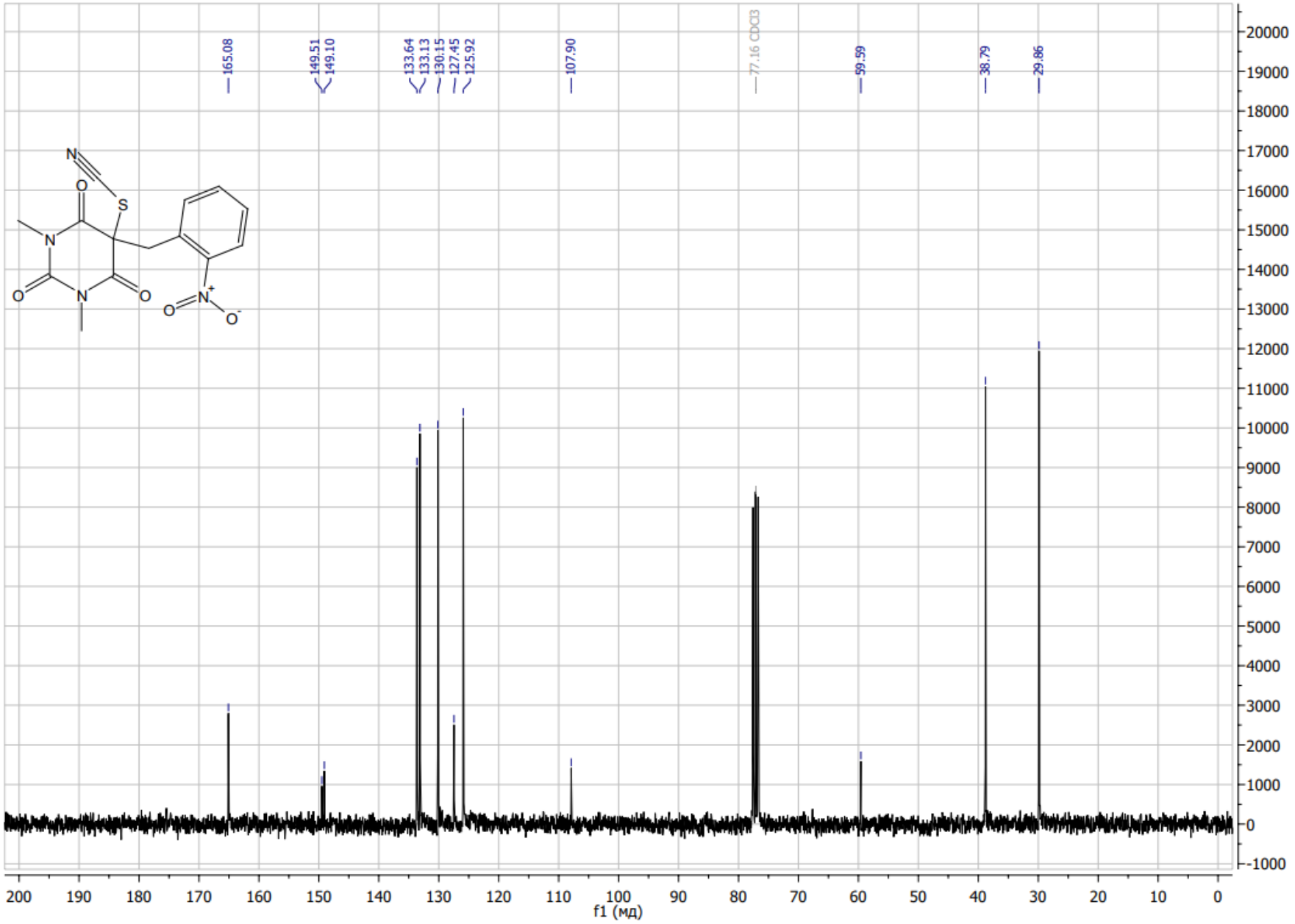
^{13}C NMR ($CDCl_3$) spectrum of 2f



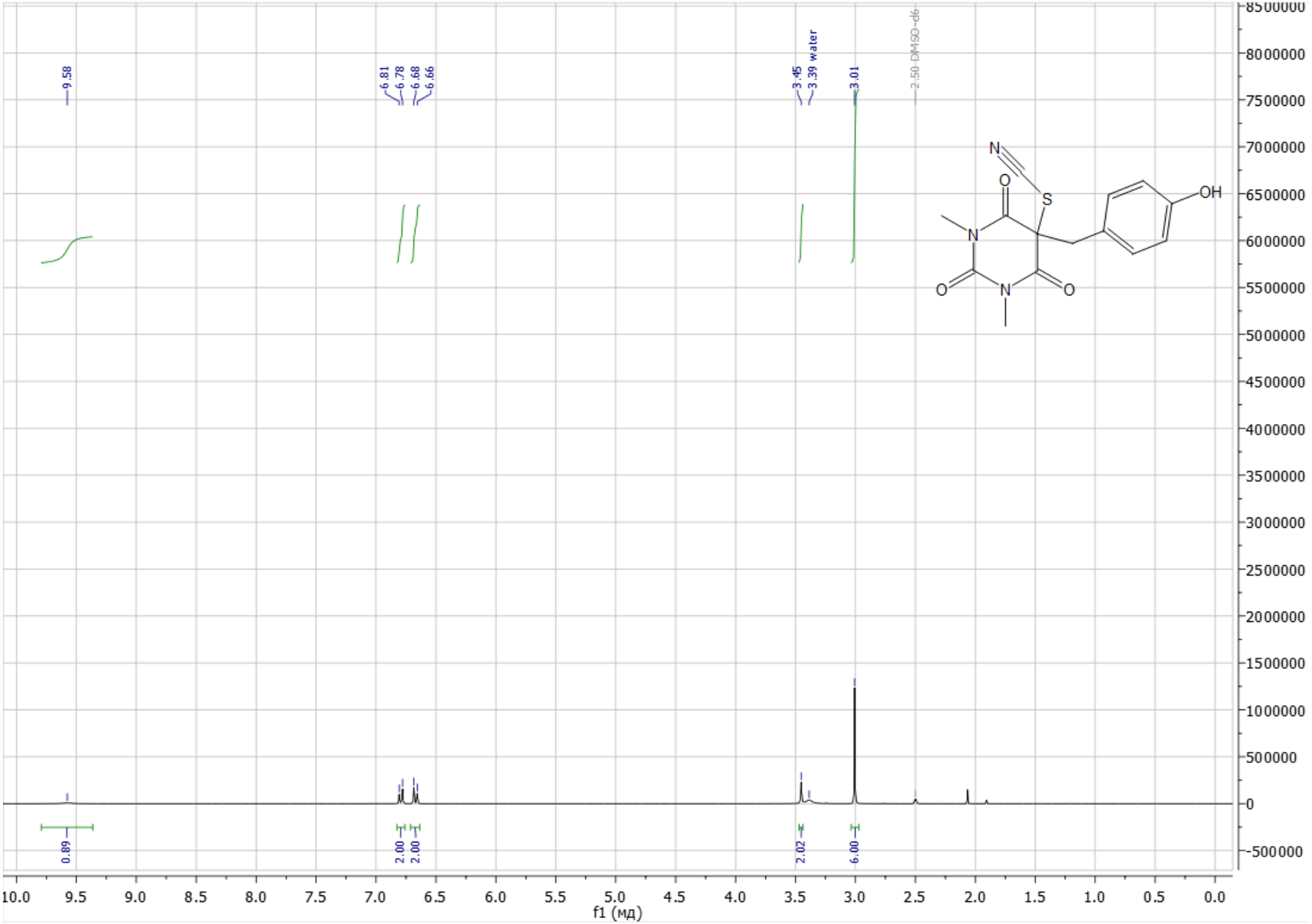
^{1}H NMR ($CDCl_3$) spectrum of 2g



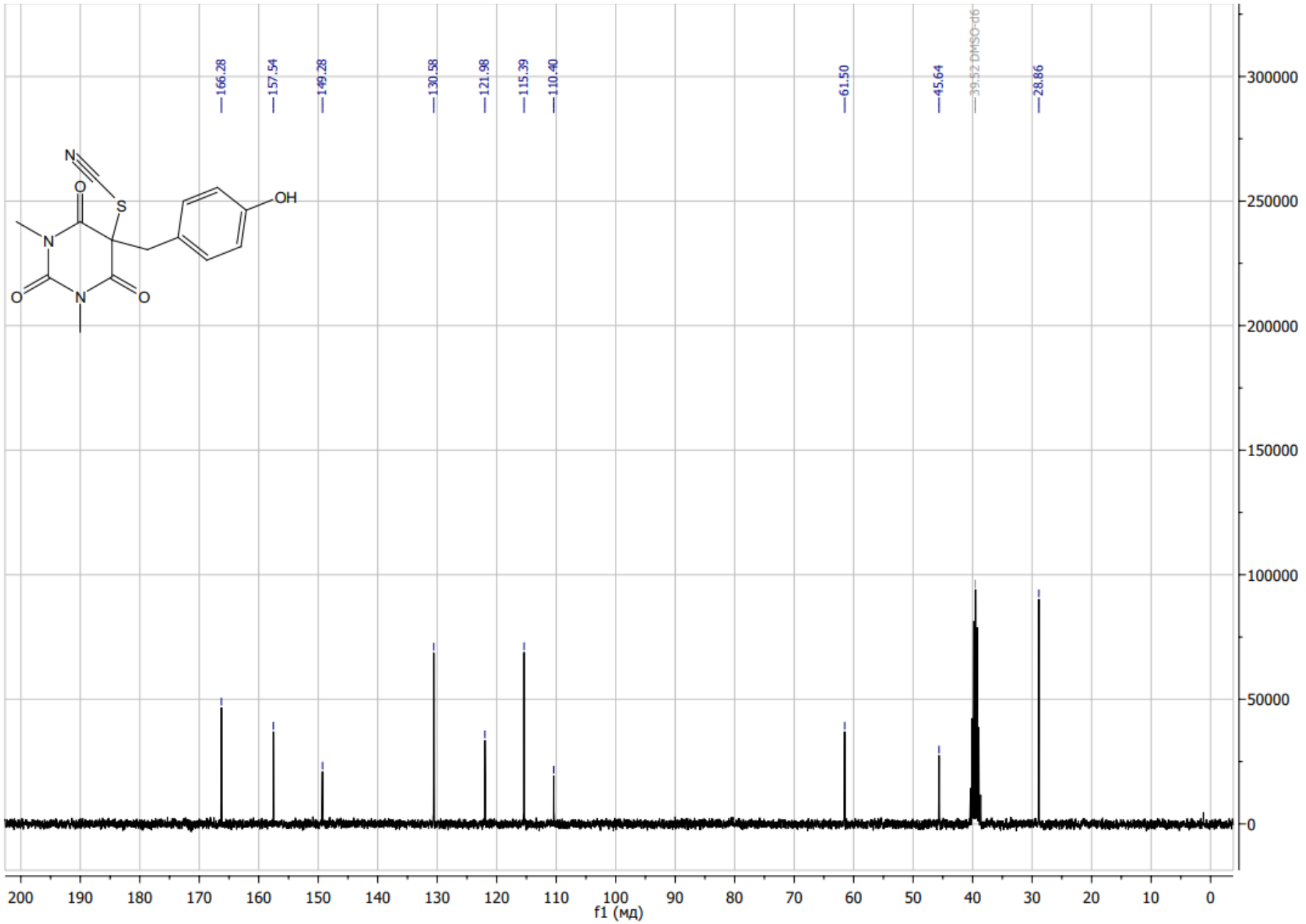
^{13}C NMR ($CDCl_3$) spectrum of 2g



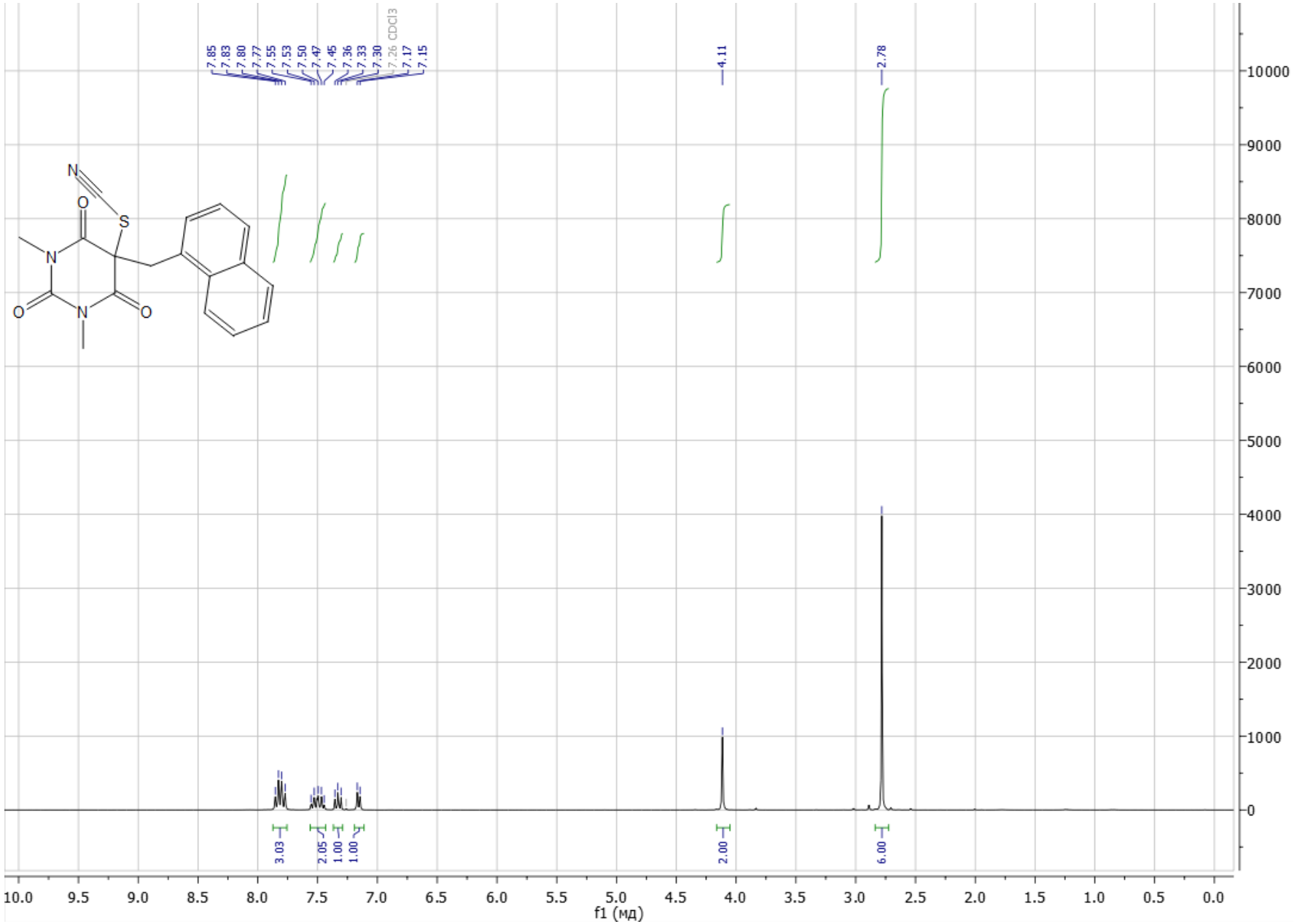
^{1}H NMR (DMSO-d_6) spectrum of 2h



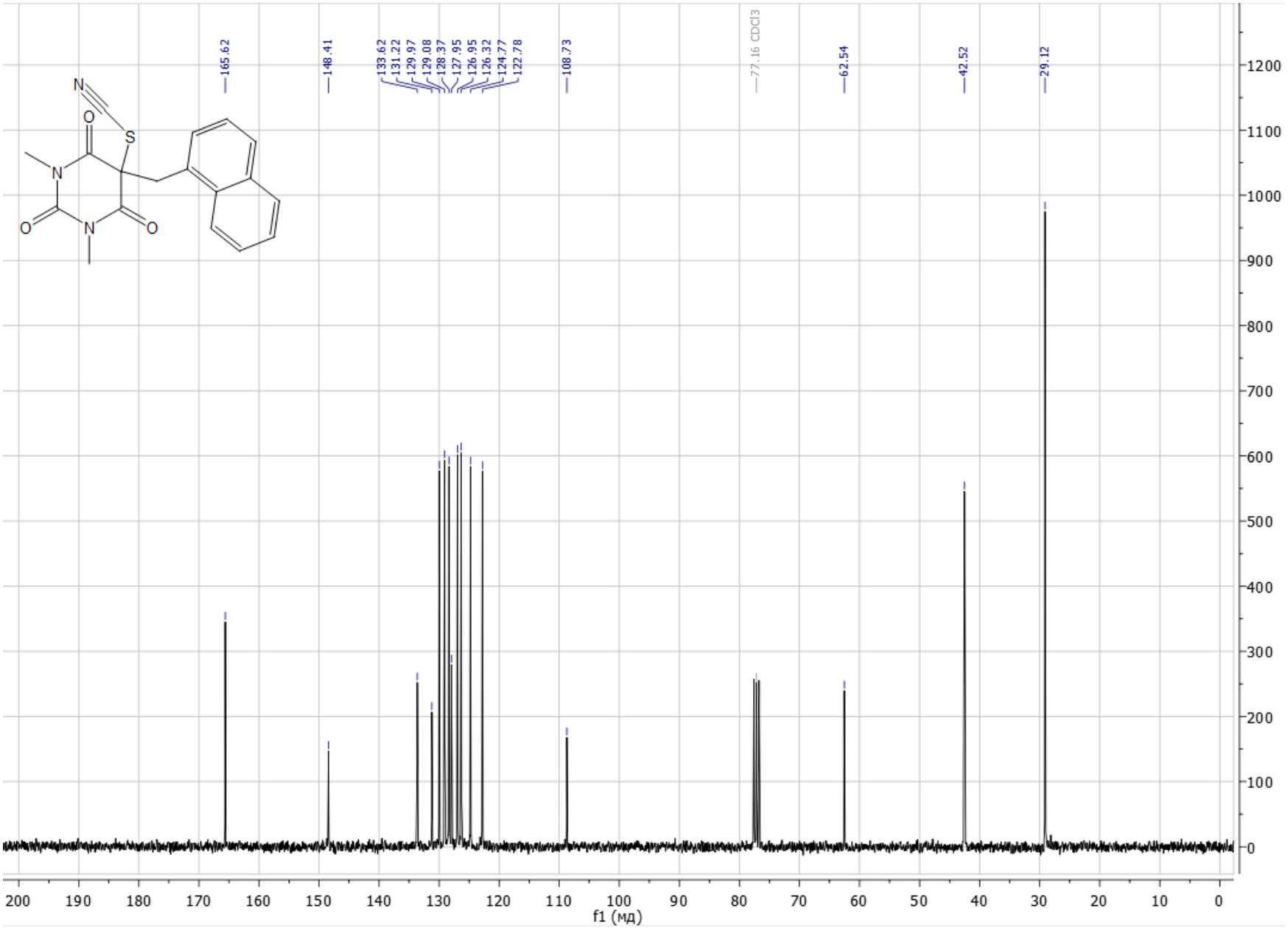
^{13}C NMR (DMSO-d_6) spectrum of 2h



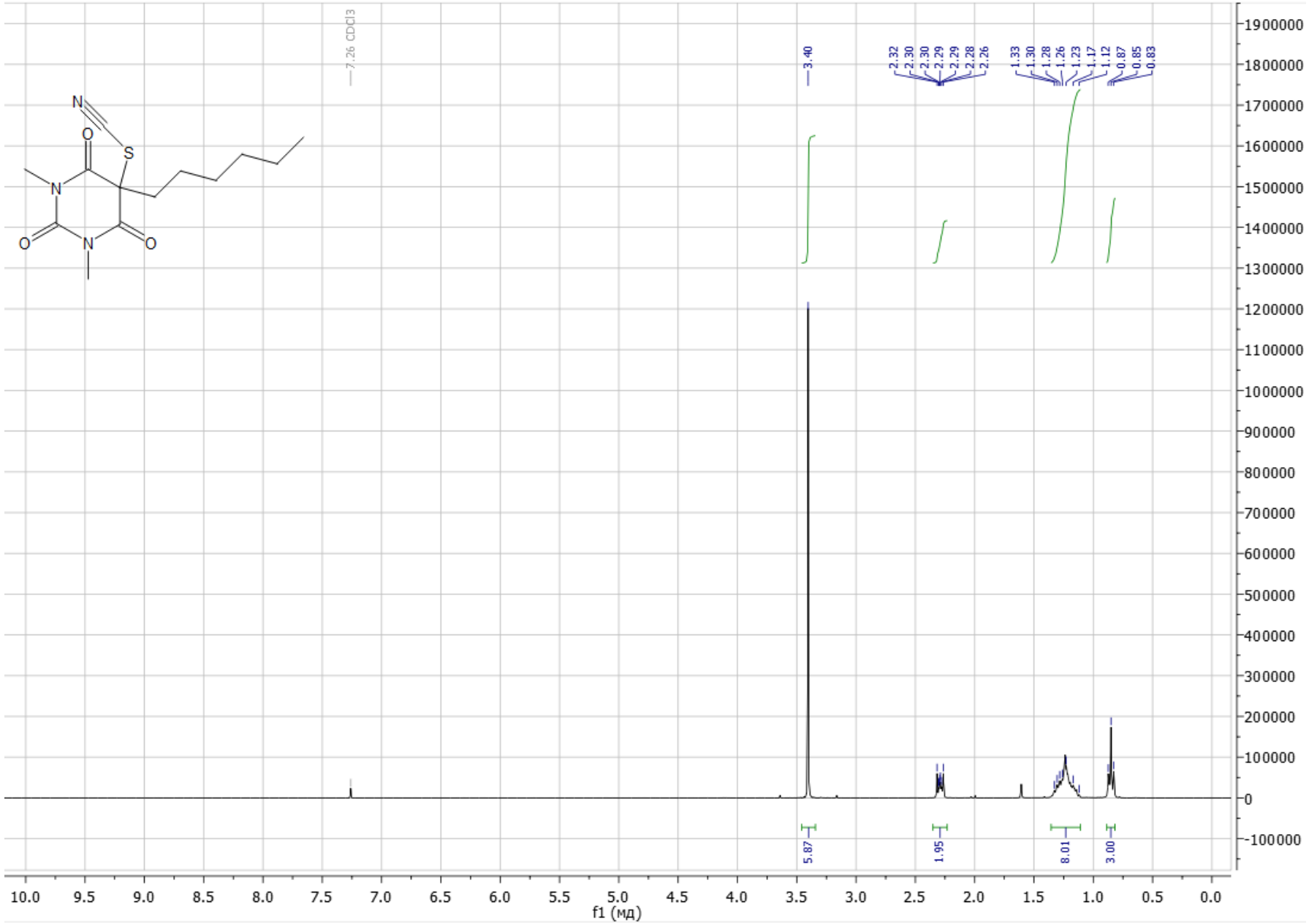
^{1}H NMR ($CDCl_3$) spectrum of 2i



^{13}C NMR ($CDCl_3$) spectrum of 2i



^{1}H NMR ($CDCl_3$) spectrum of 2j



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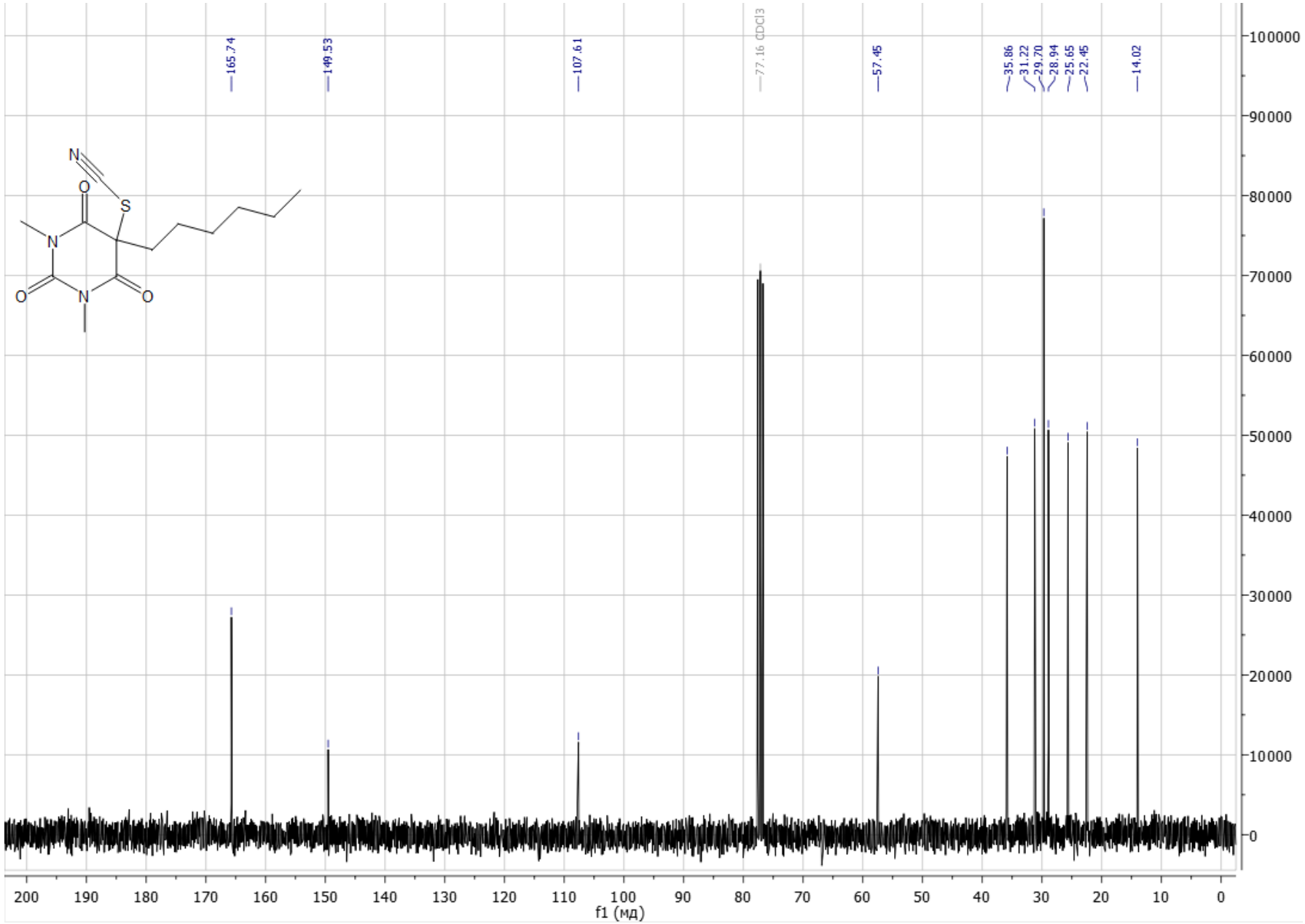

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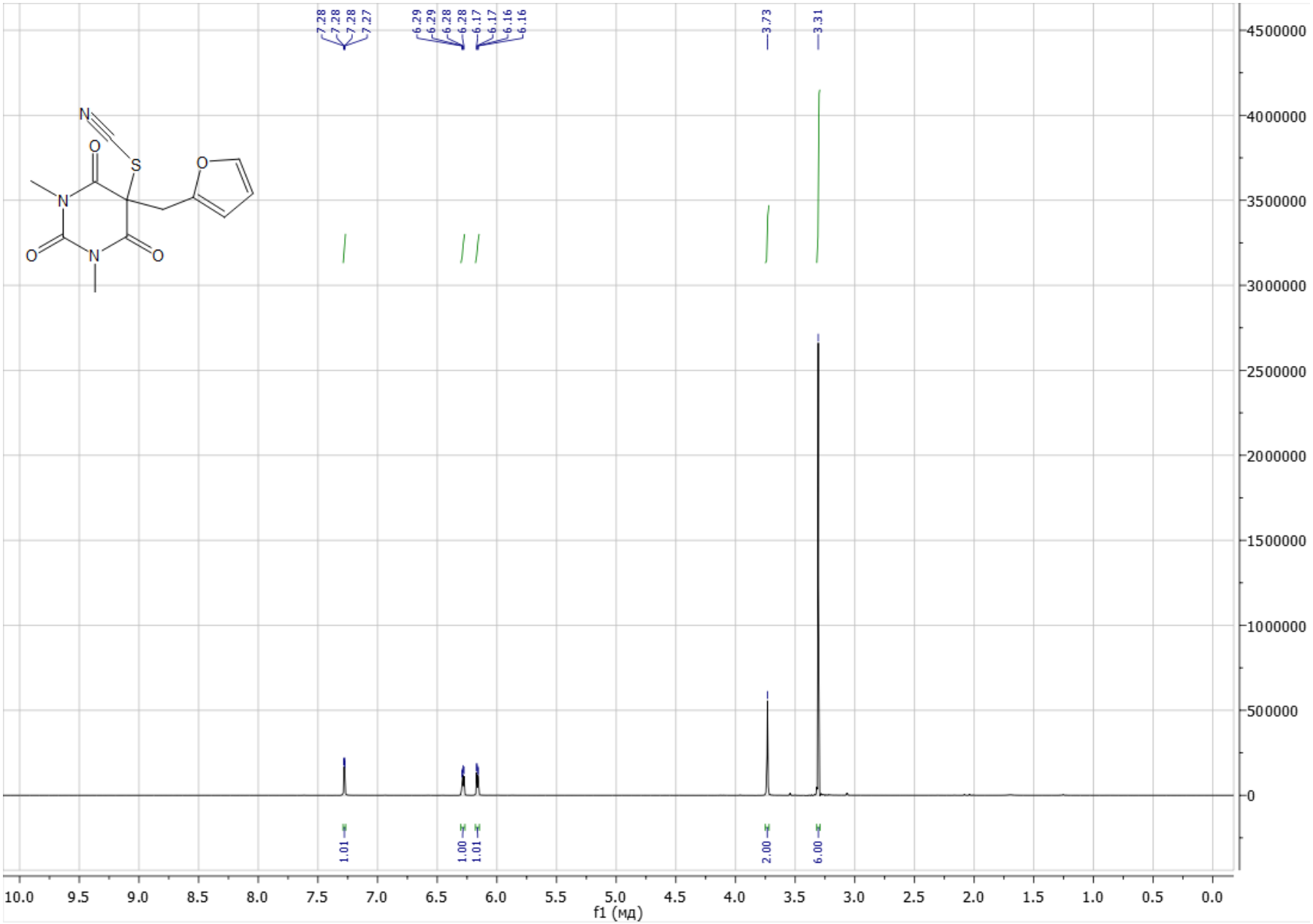

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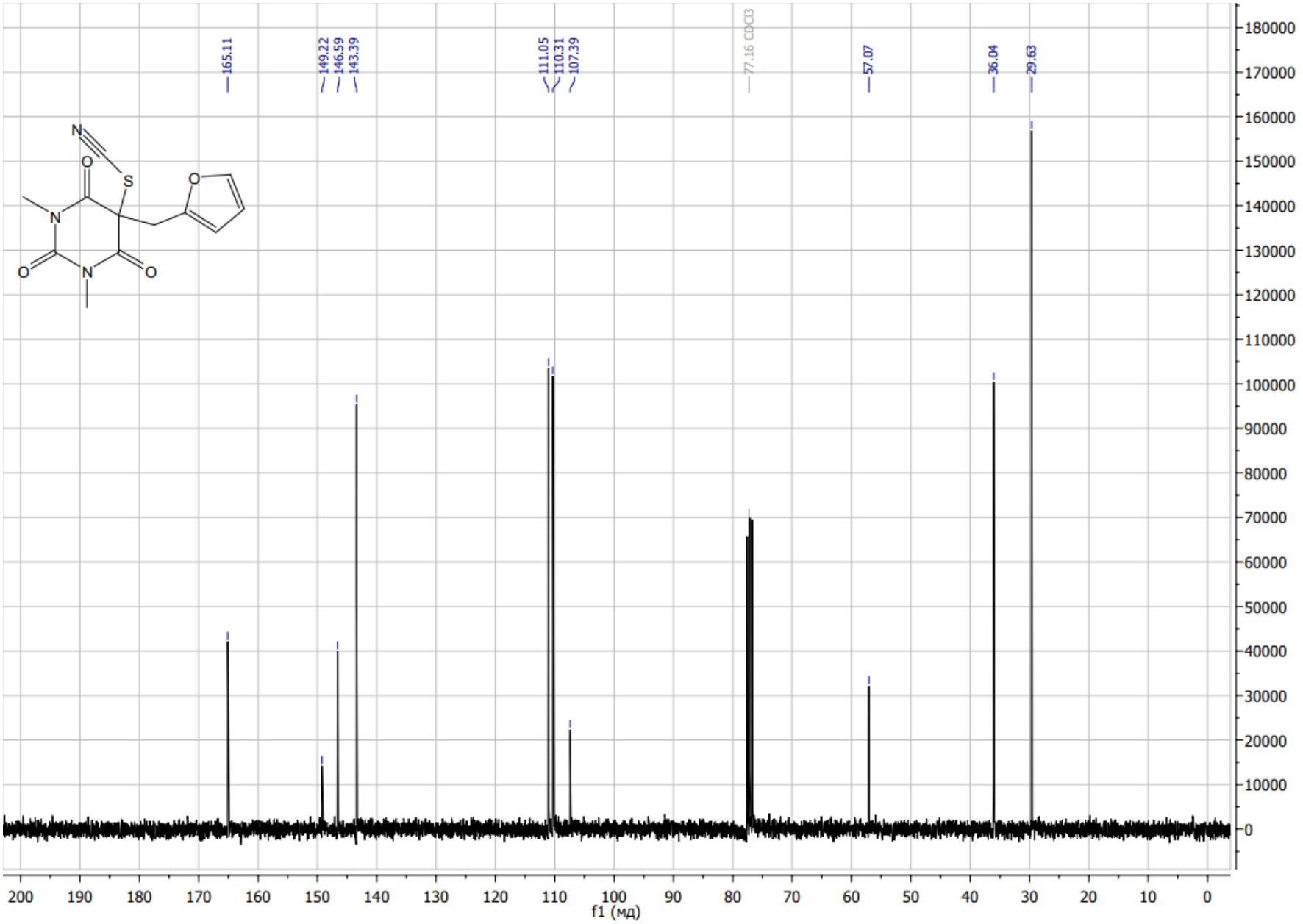
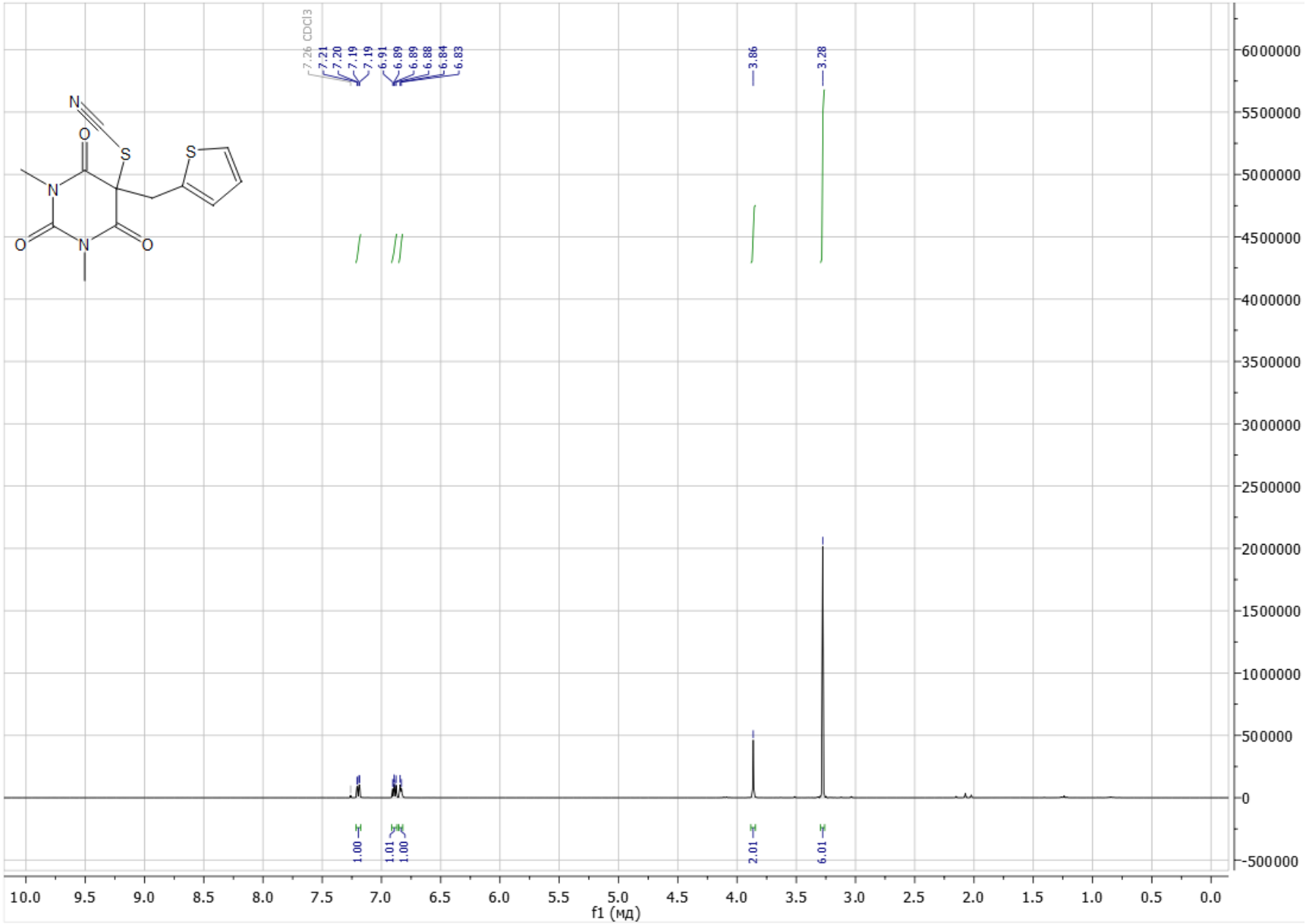


^{1}H NMR ($CDCl_3$) spectrum of 2l



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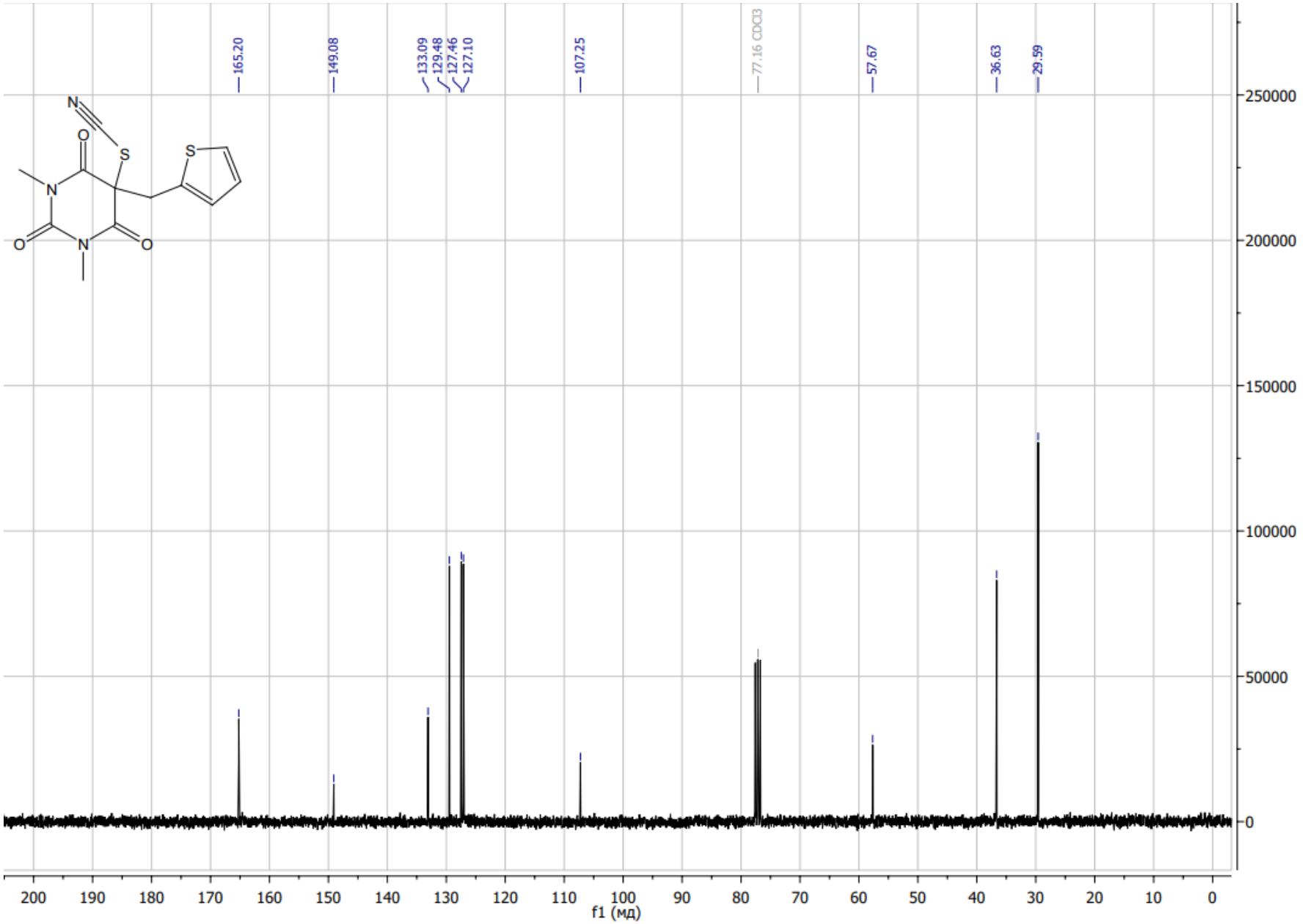
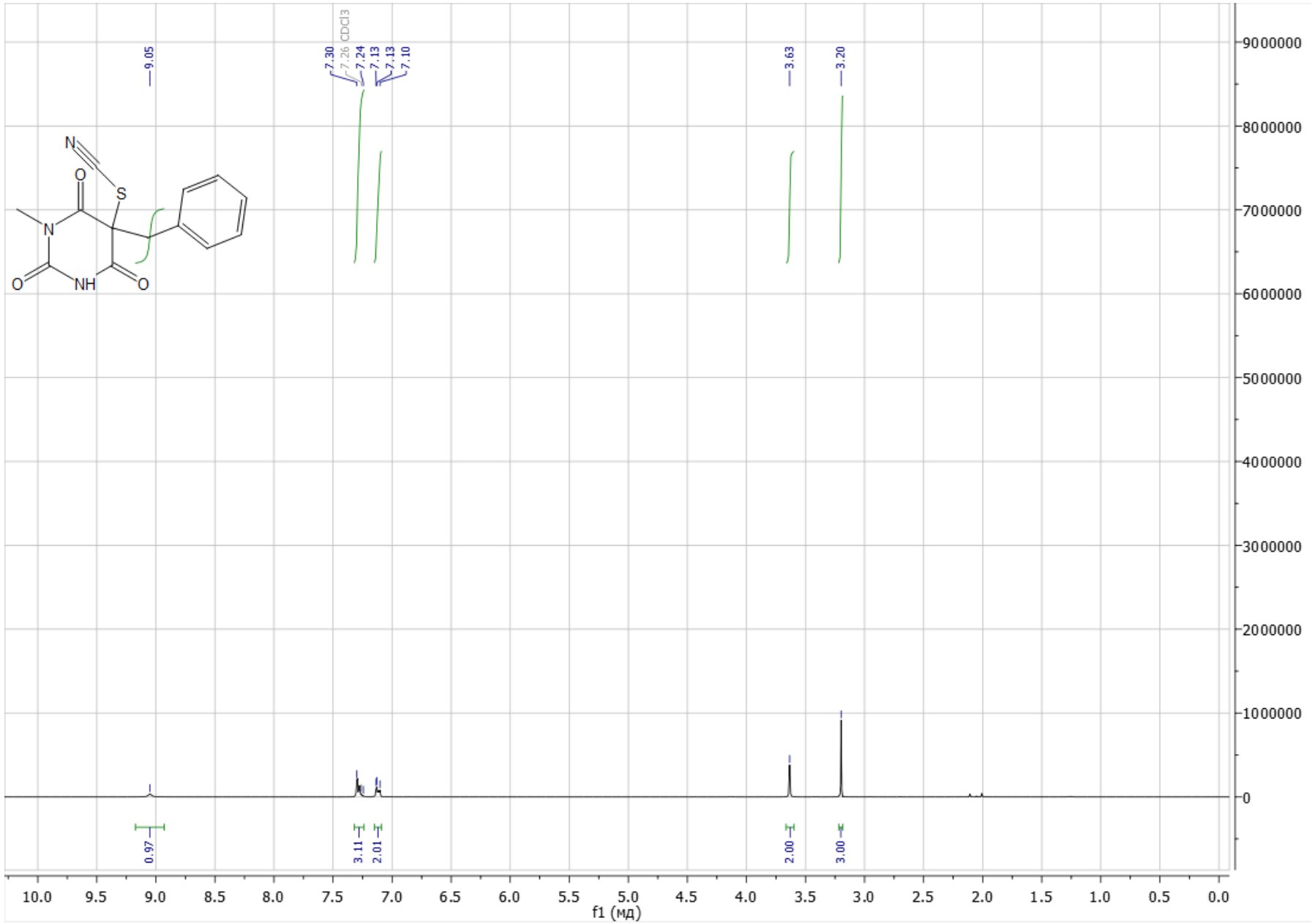
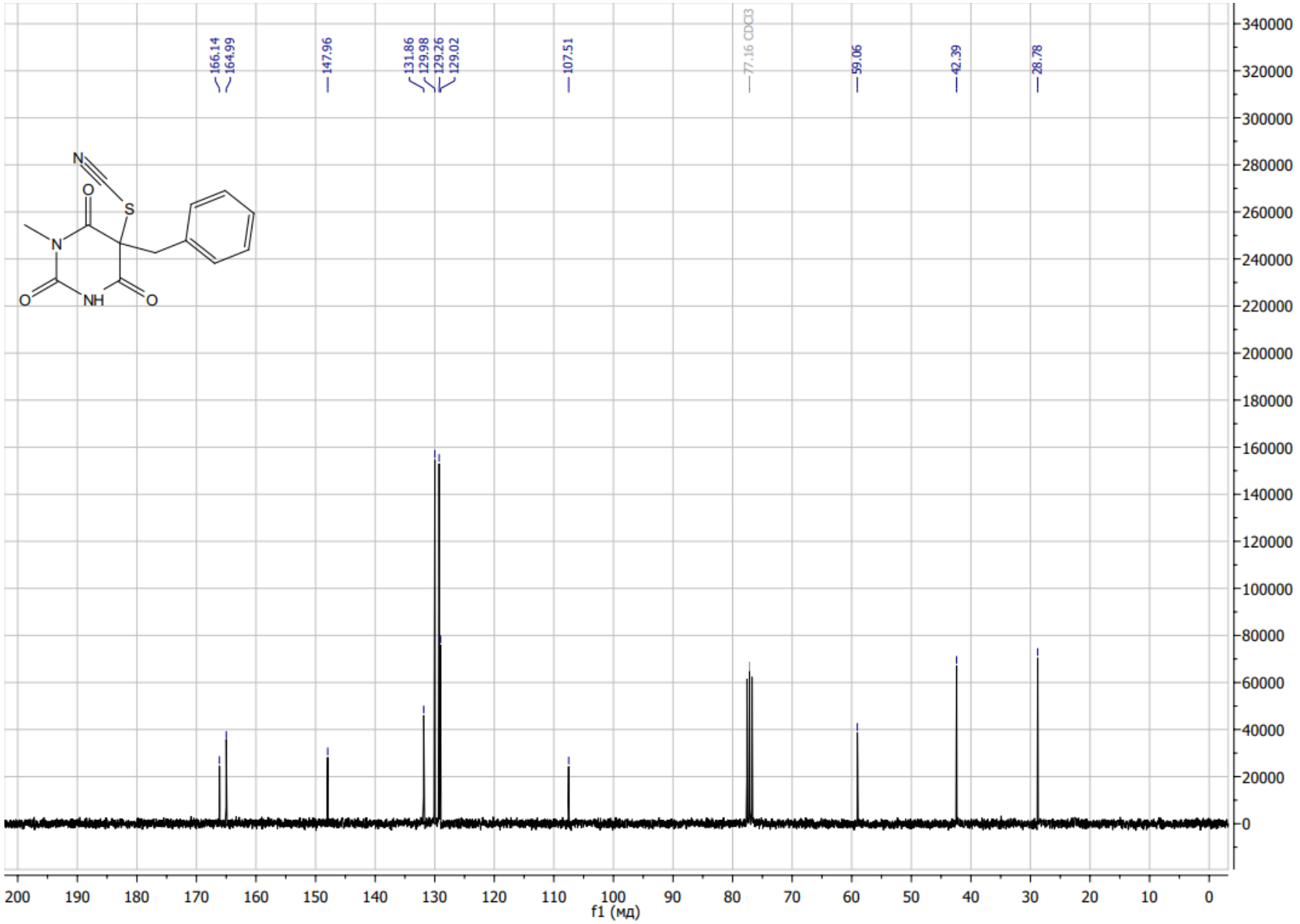


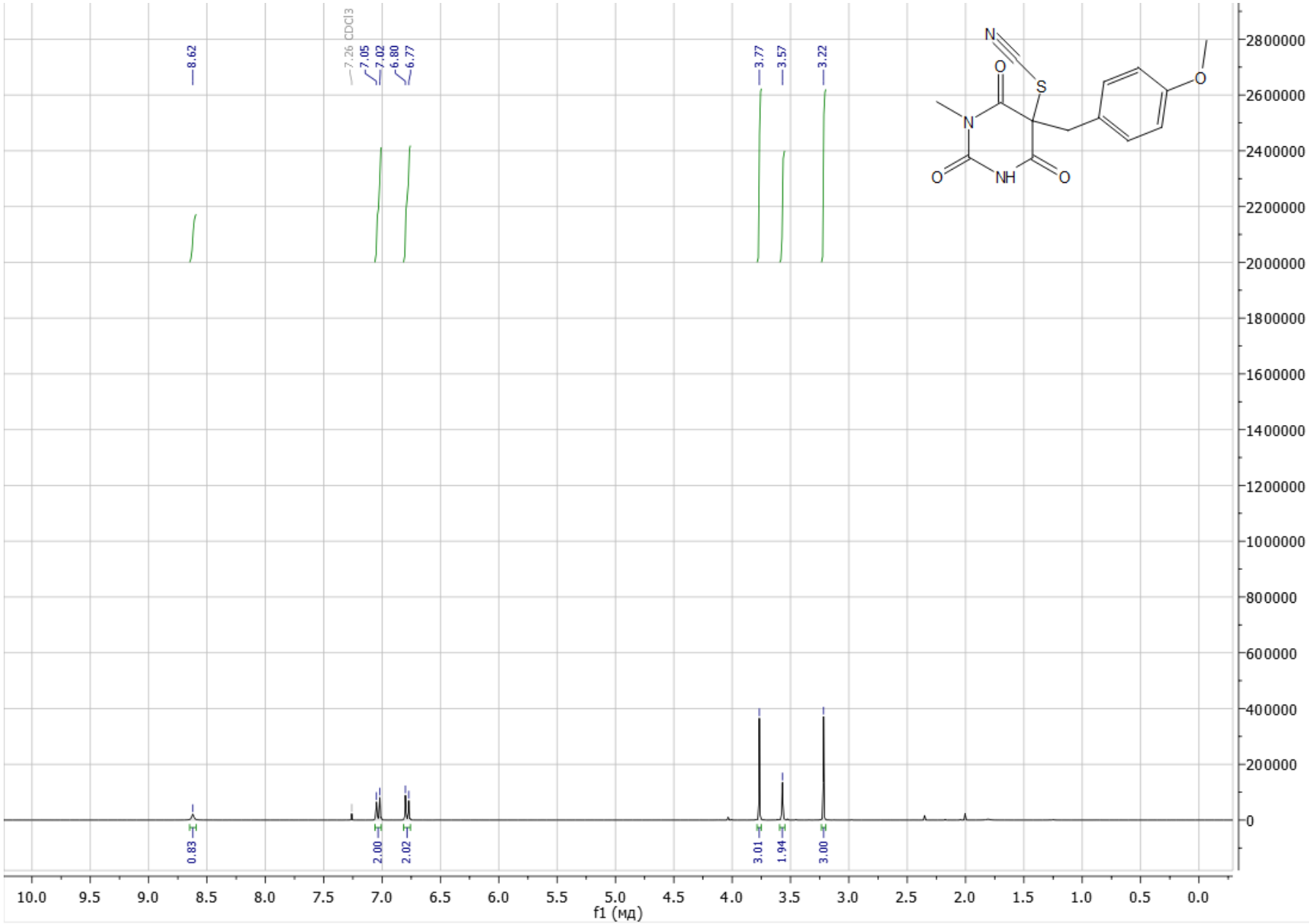
^{1}H NMR ($CDCl_3$) spectrum of 2m



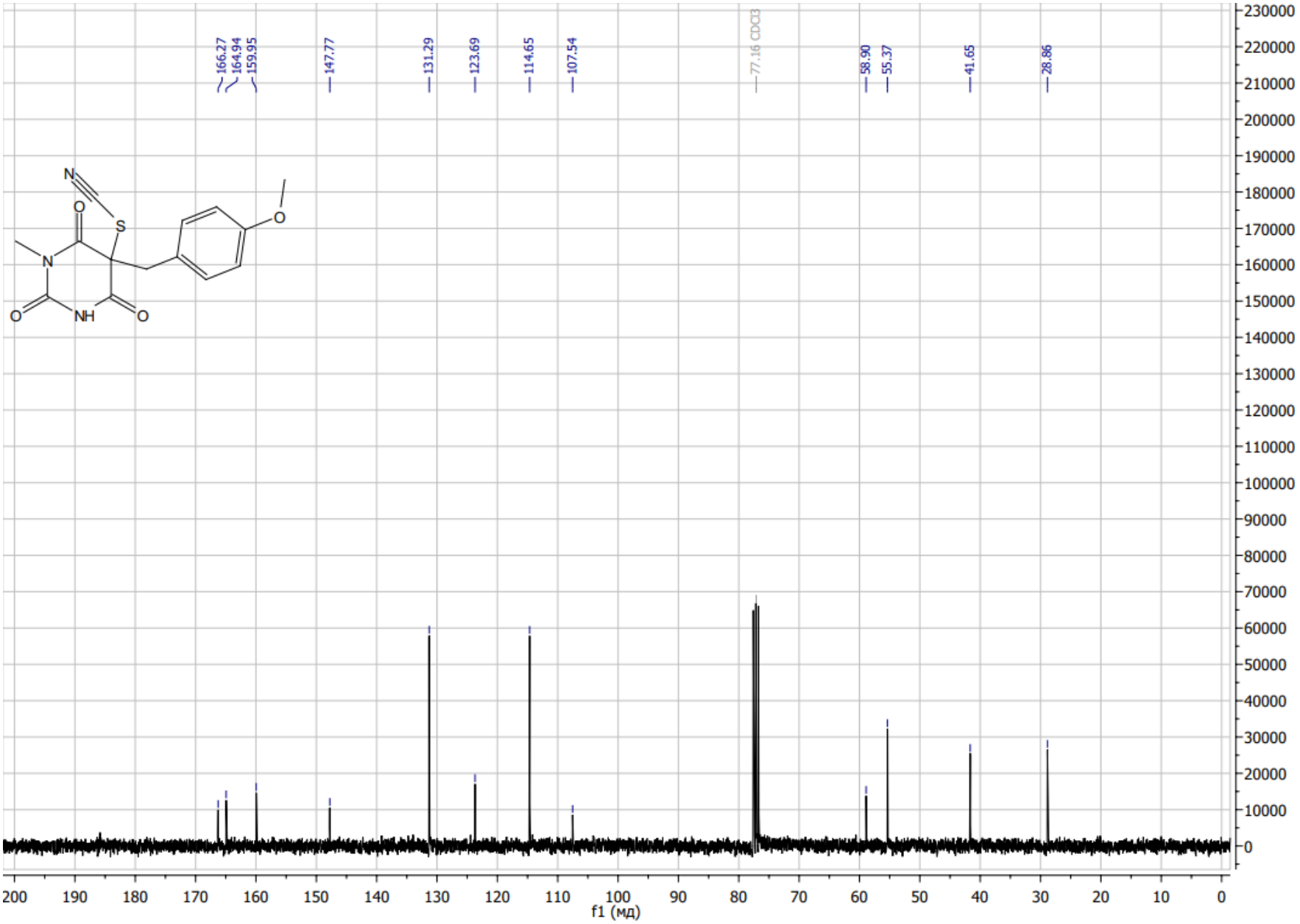
^{13}C NMR ($CDCl_3$) spectrum of 2m



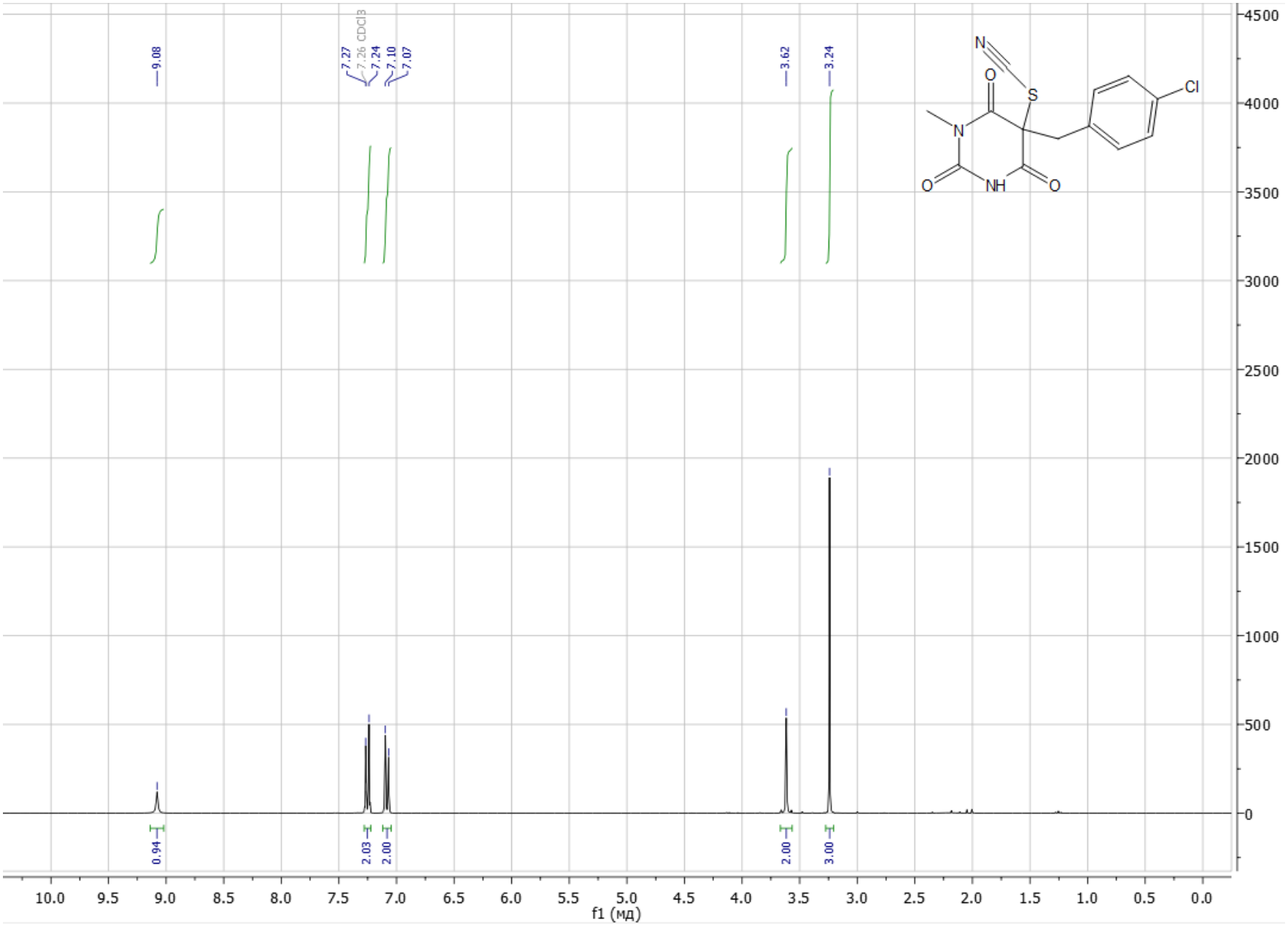
^{1}H NMR ($CDCl_3$) spectrum of 2n



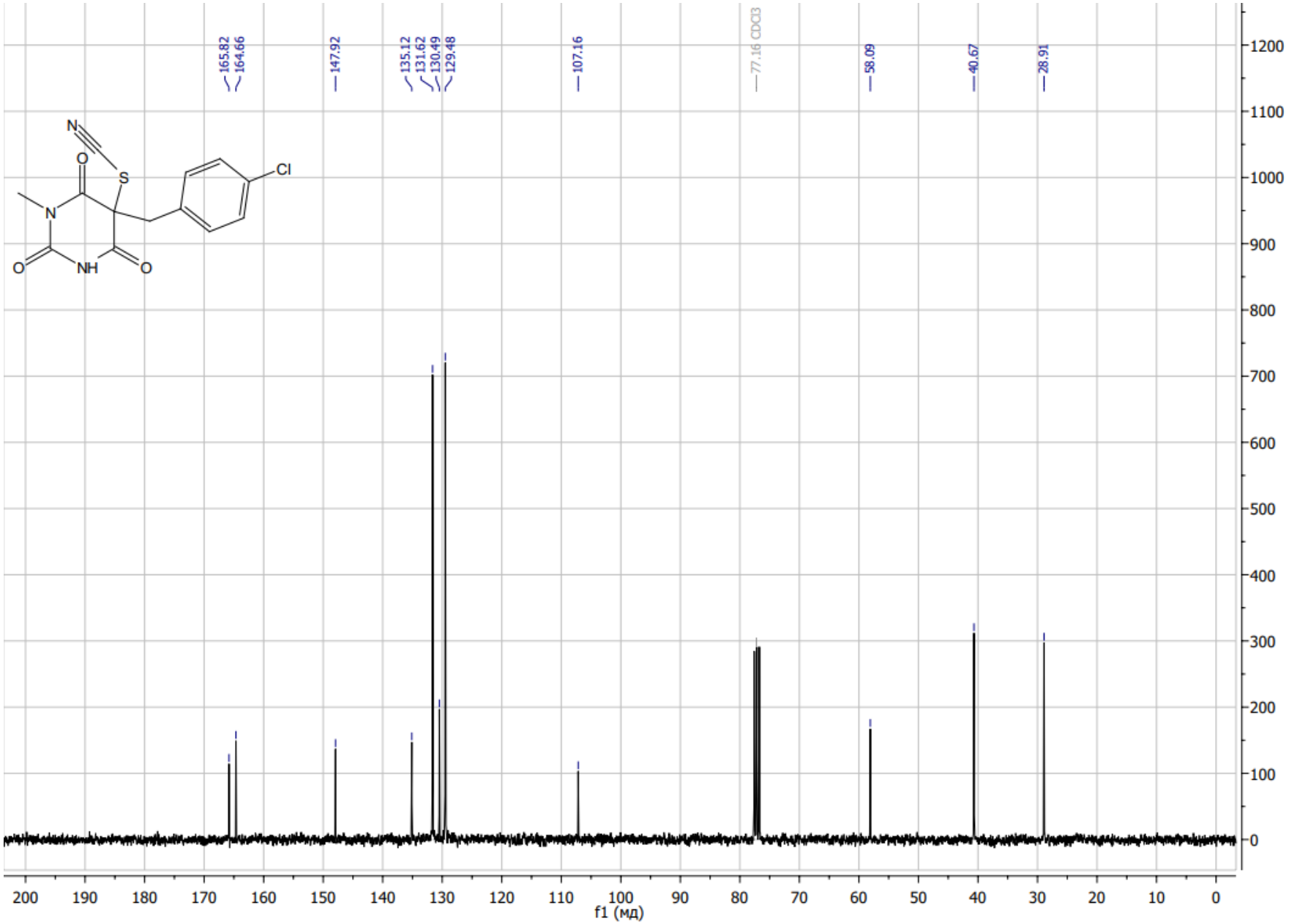
^{13}C NMR ($CDCl_3$) spectrum of 2n



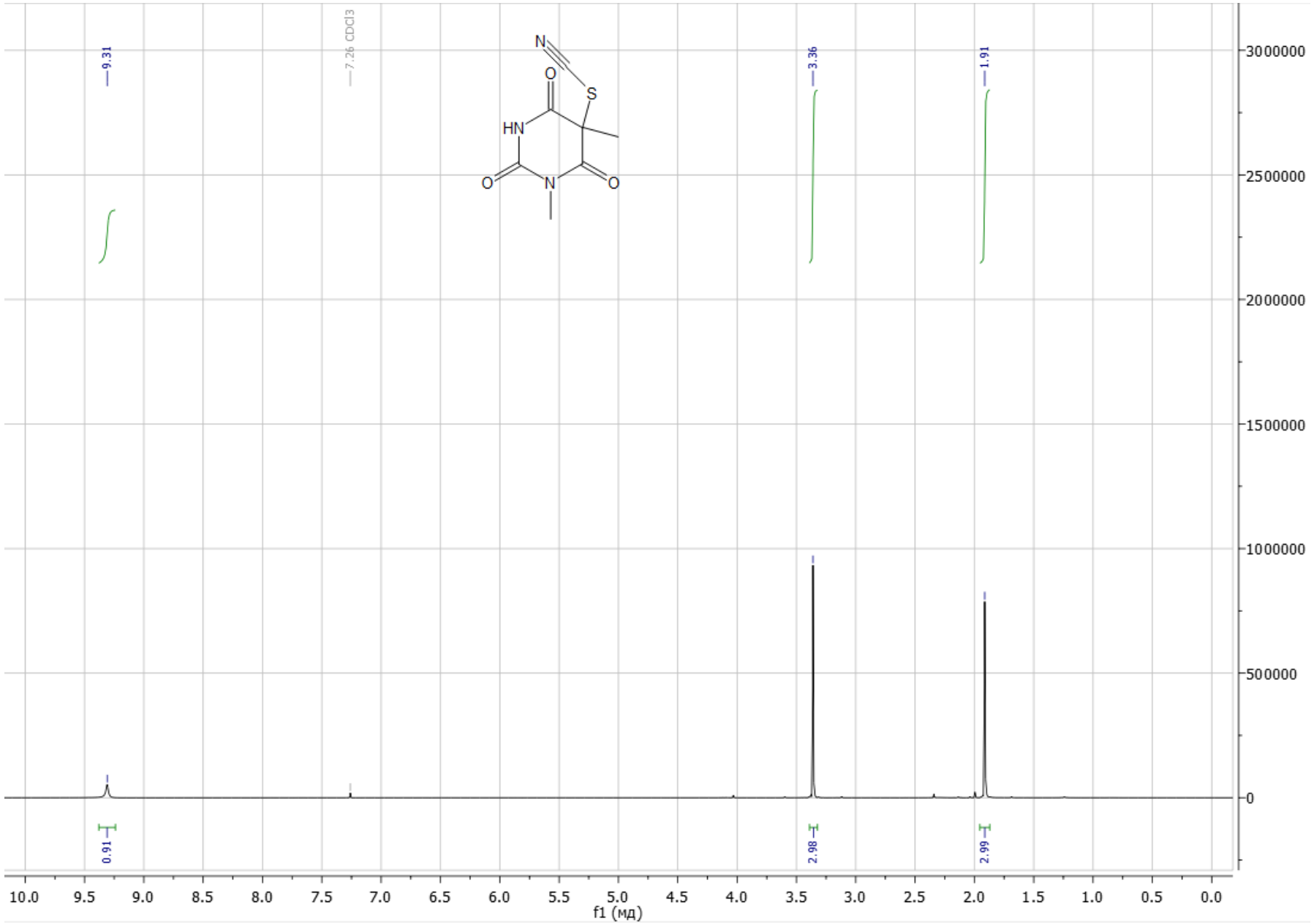
^{1}H NMR ($CDCl_3$) spectrum of 2o



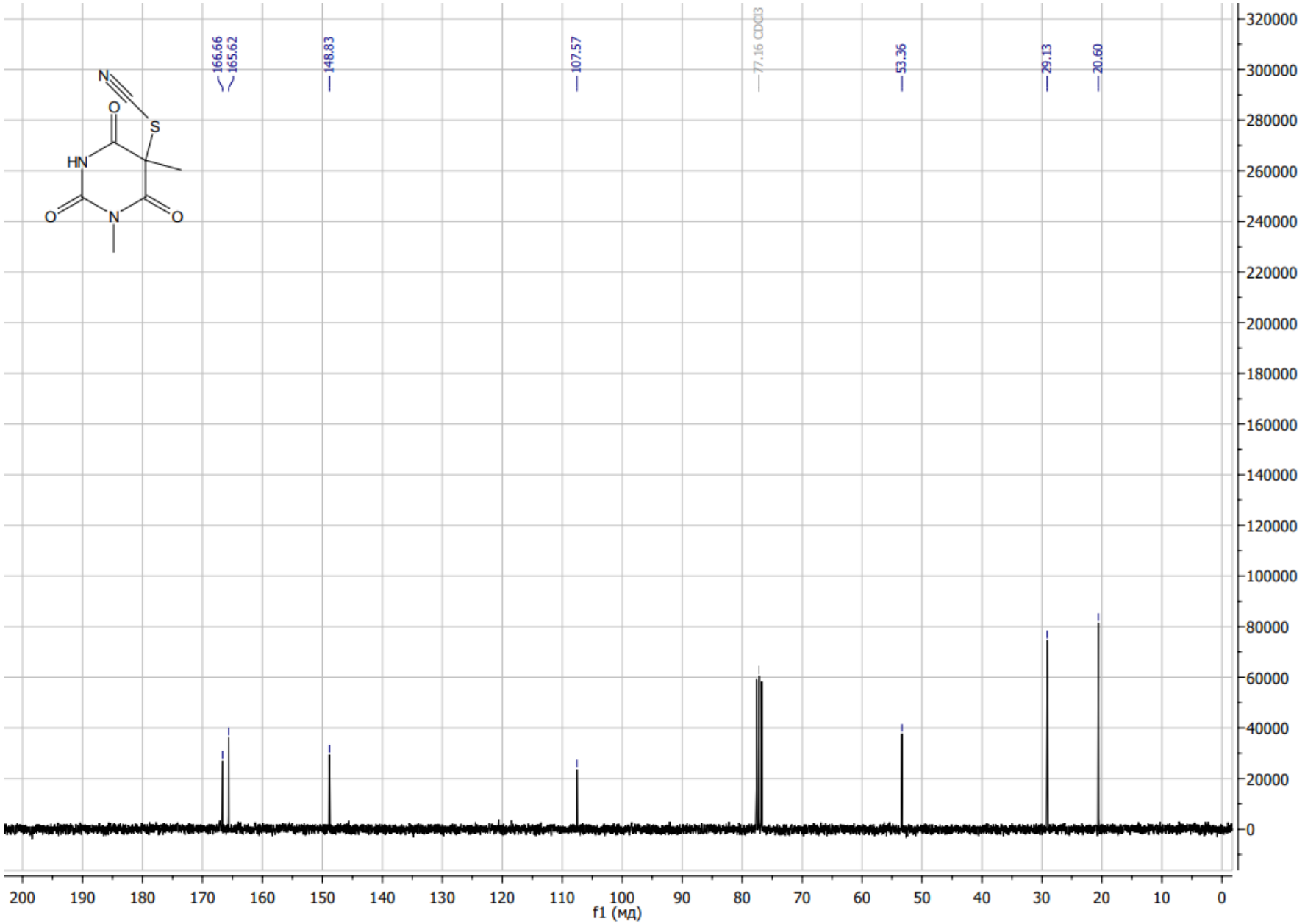
^{13}C NMR ($CDCl_3$) spectrum of 2o



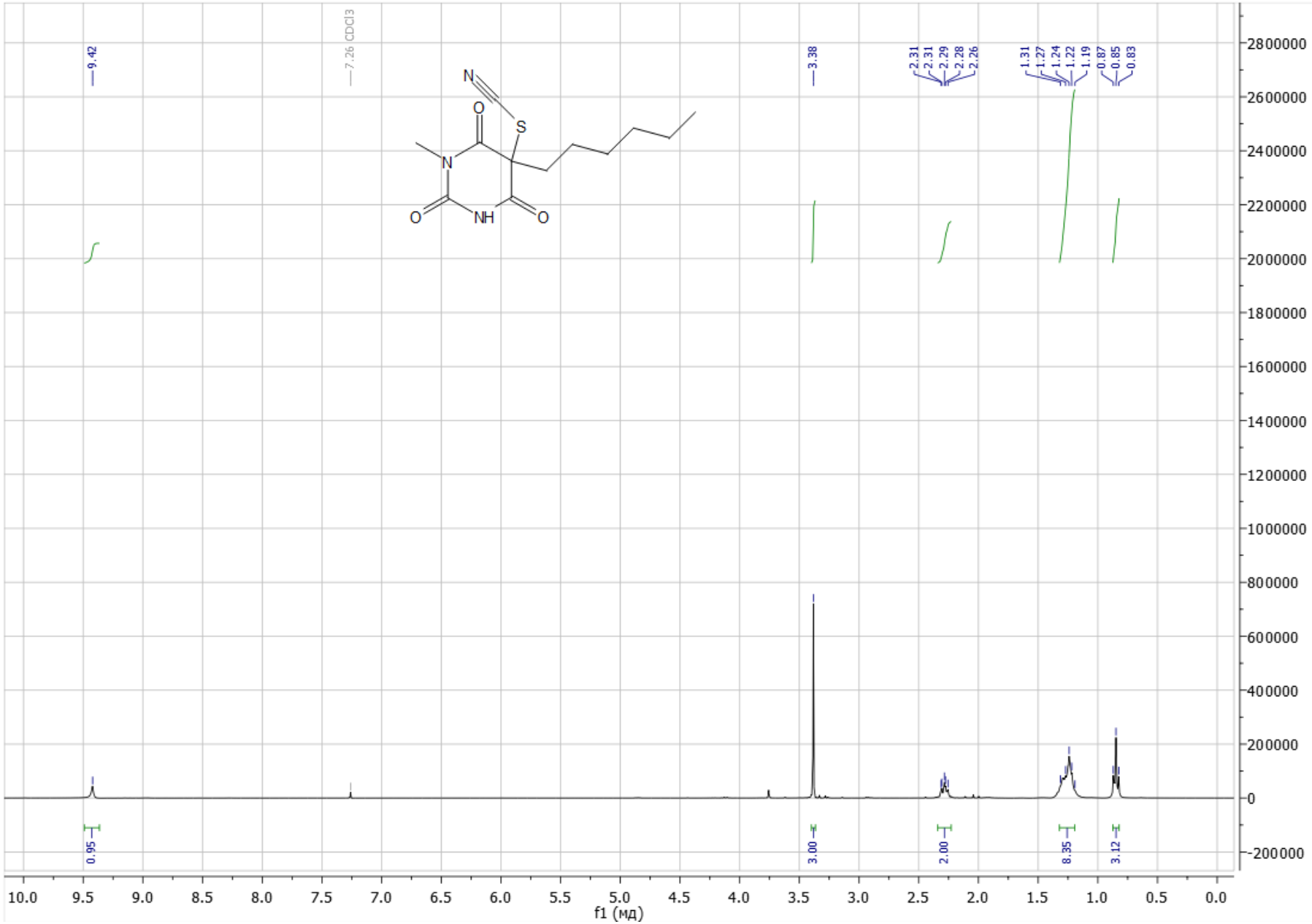
^{1}H NMR ($CDCl_3$) spectrum of 2p



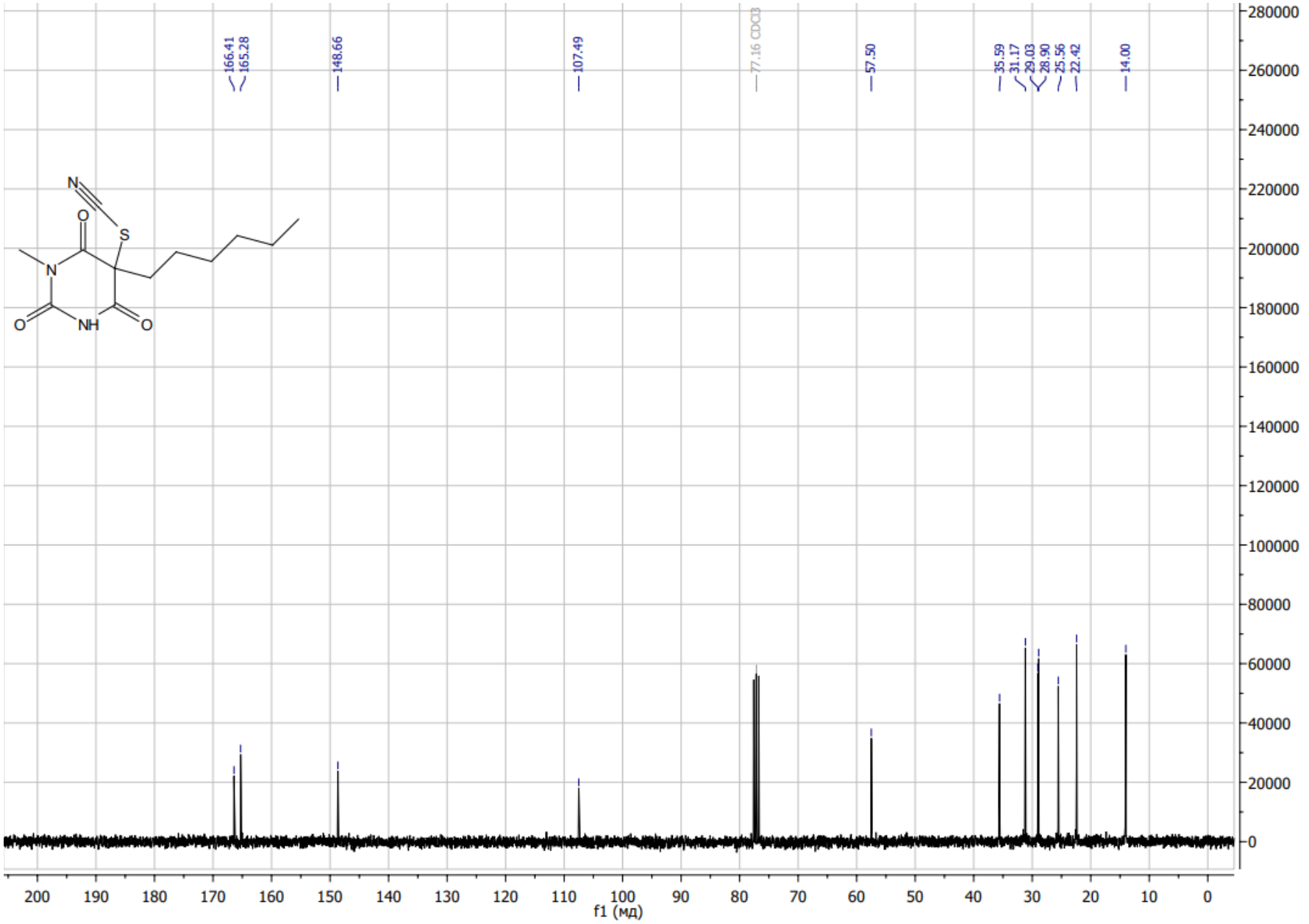
^{13}C NMR ($CDCl_3$) spectrum of 2p



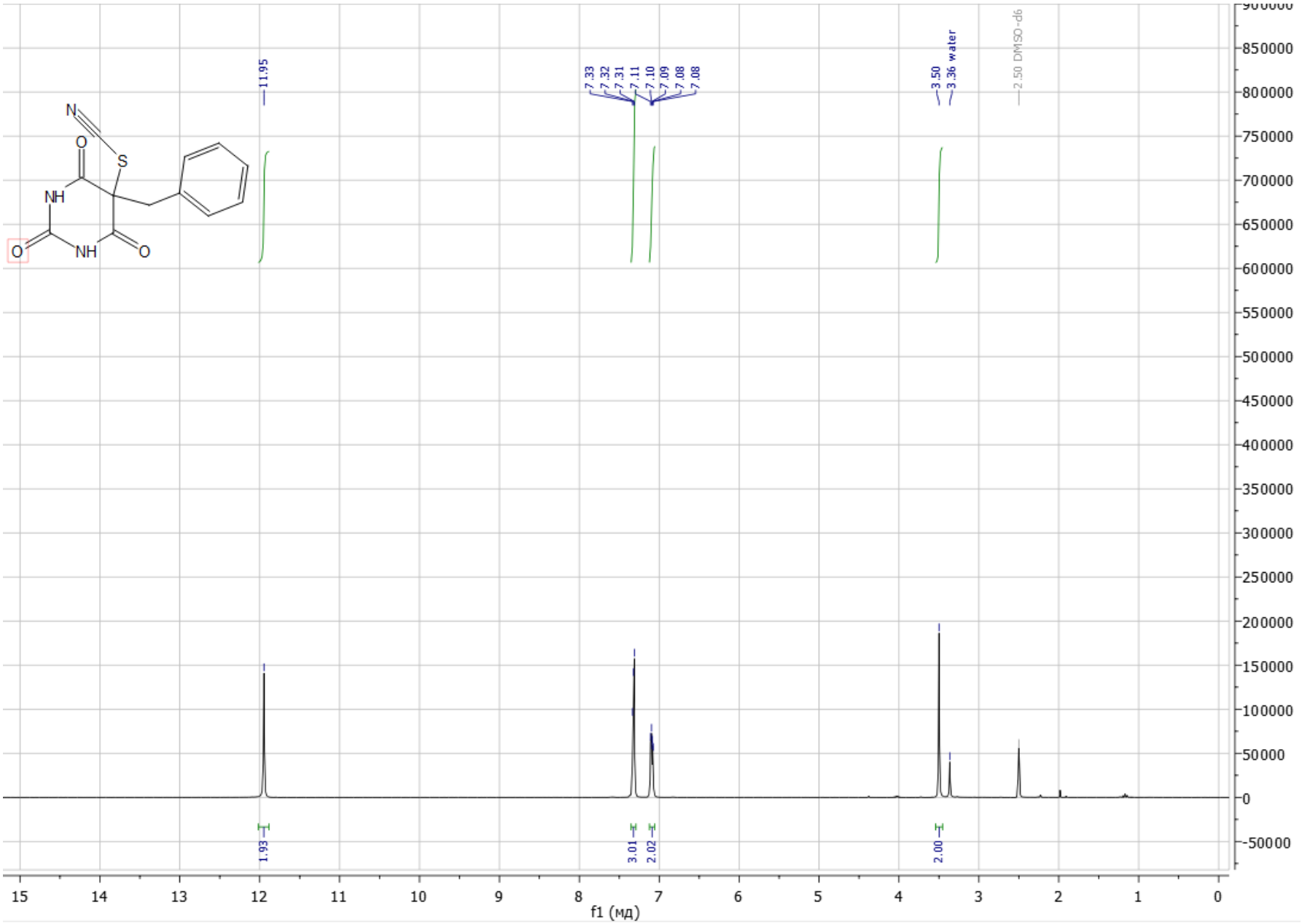
^{1}H NMR ($CDCl_3$) spectrum of 2q



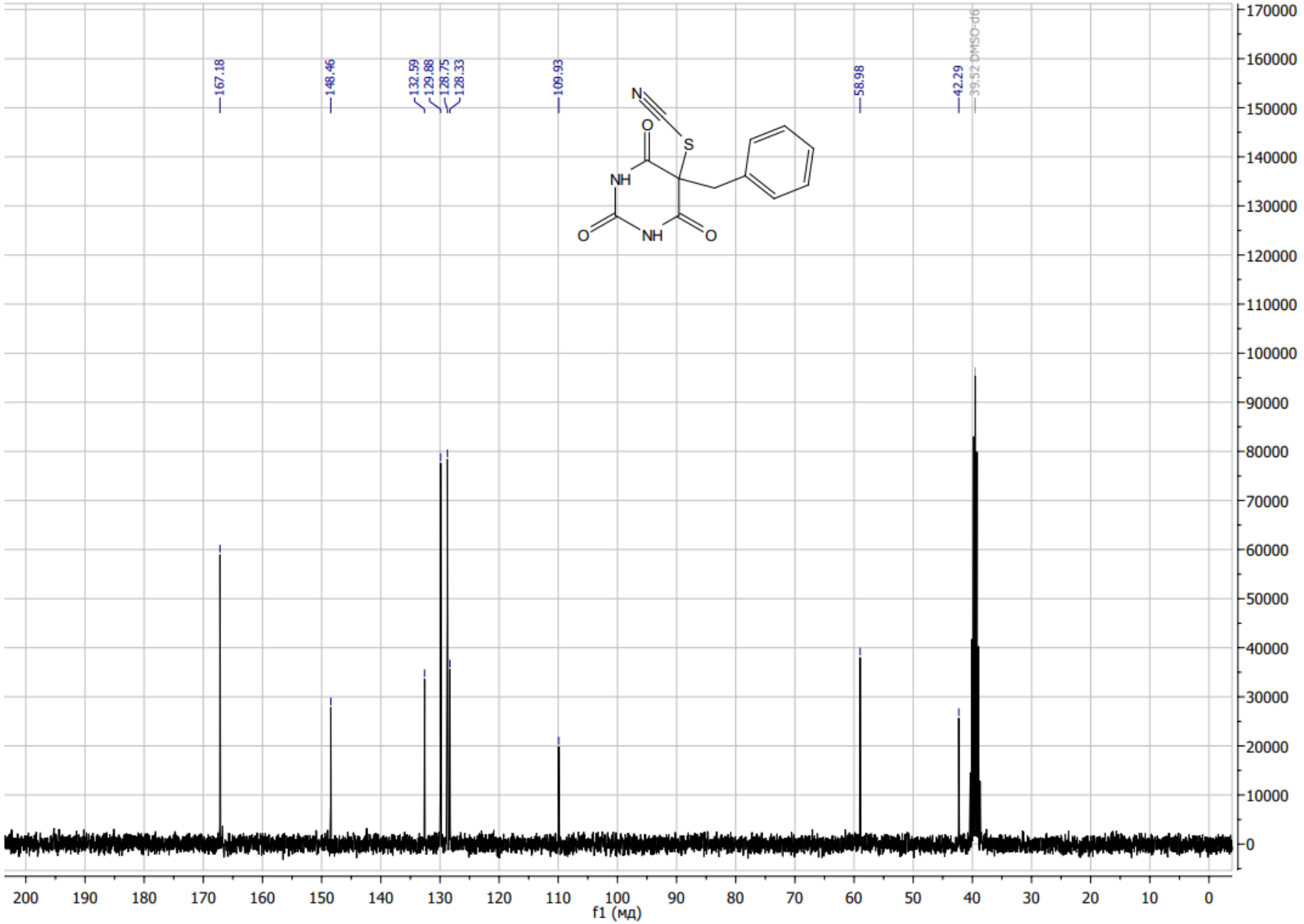
^{13}C NMR ($CDCl_3$) spectrum of 2q



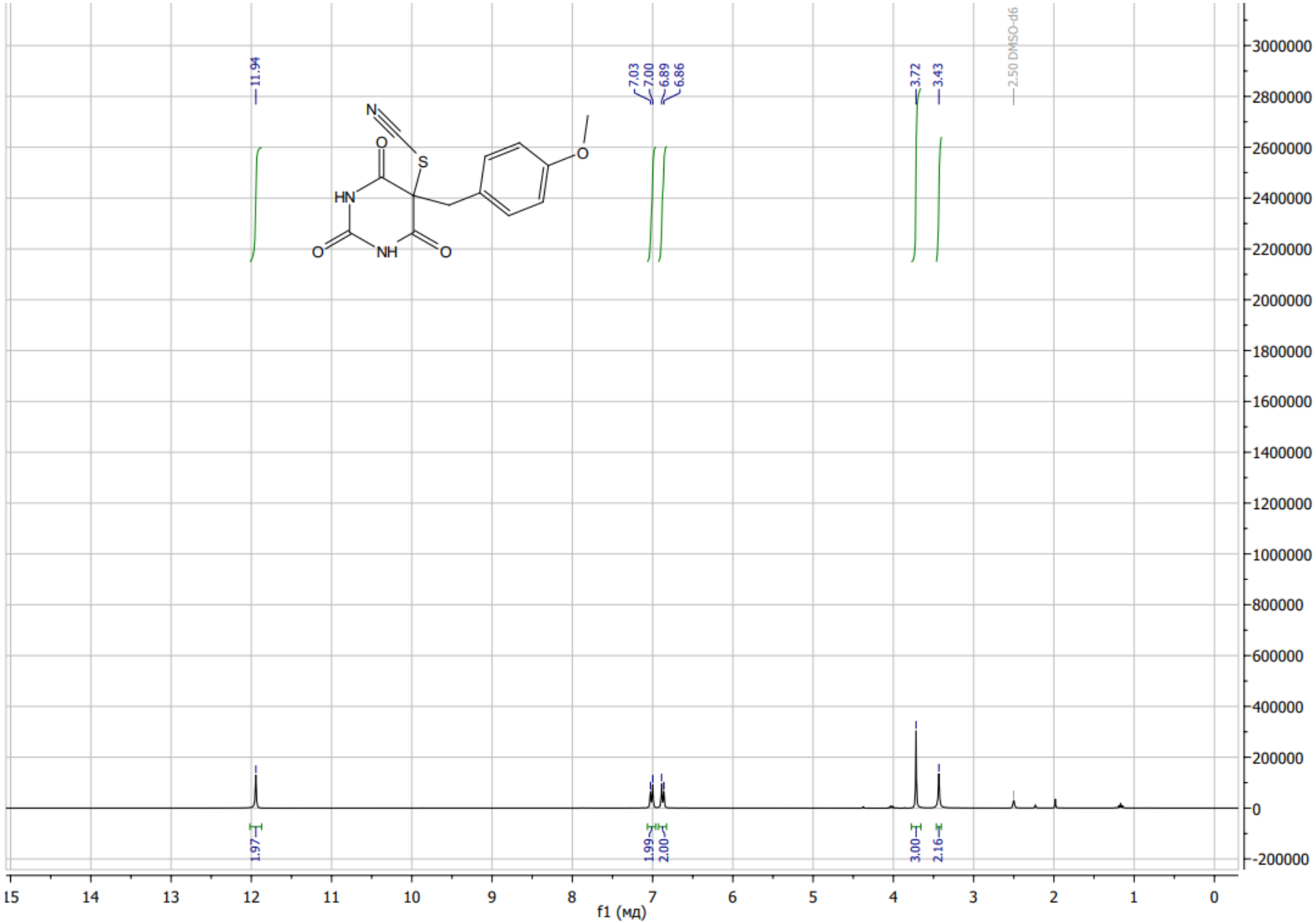
^{1}H NMR (DMSO-d_6) spectrum of 2r



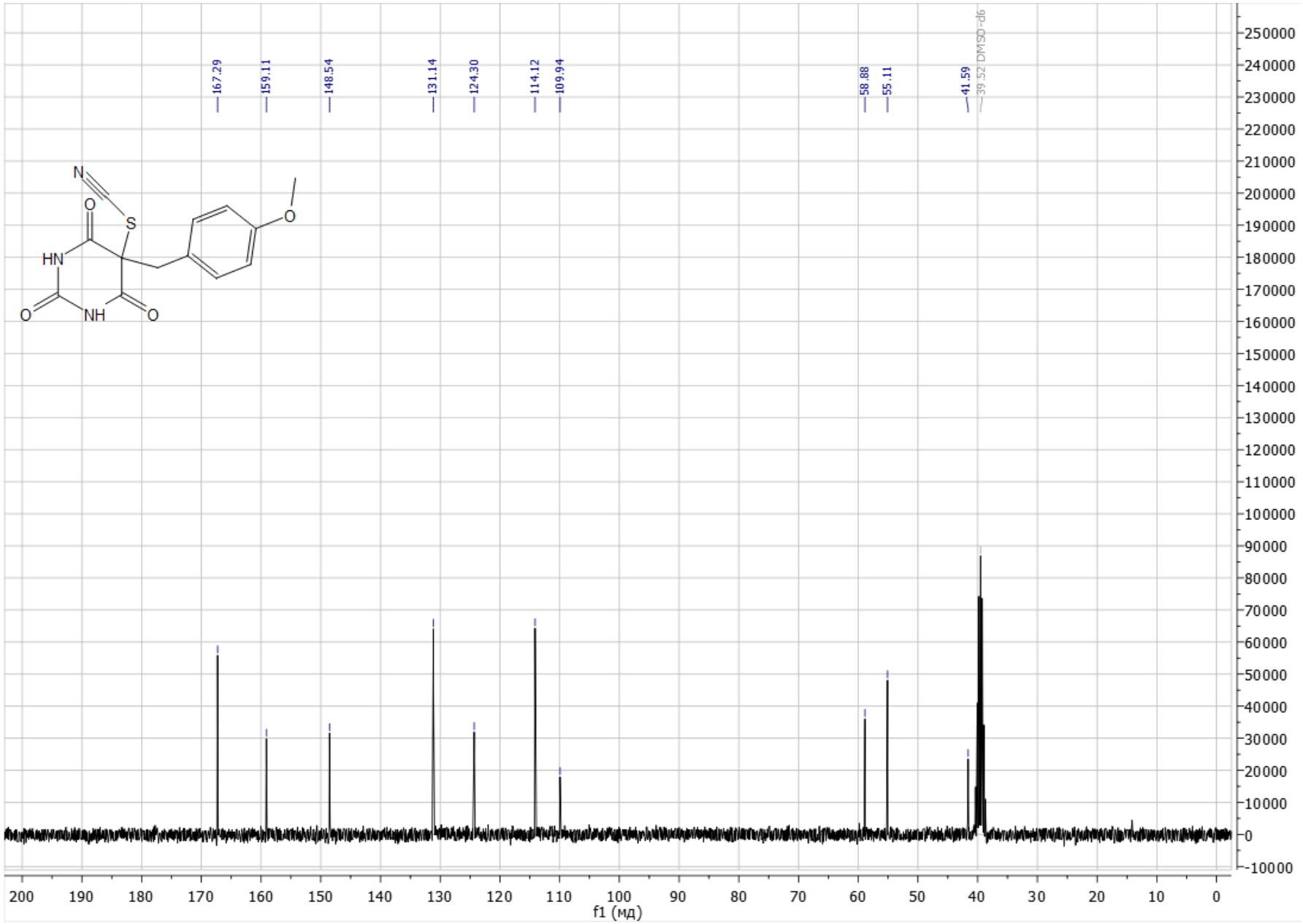
^{13}C NMR (DMSO-d_6) spectrum of 2r



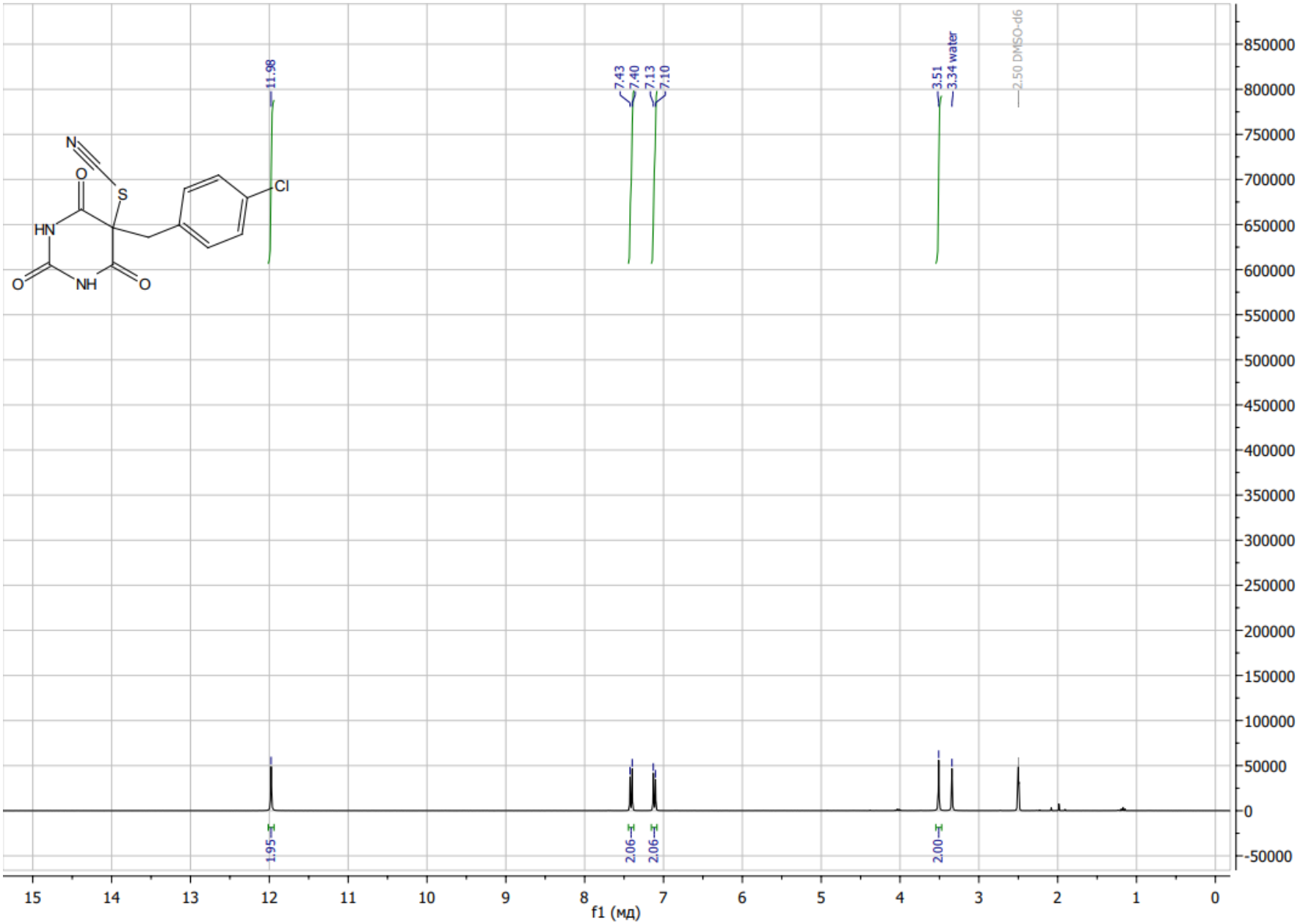
^{1}H NMR (DMSO-*d*$_6$) spectrum of 2s



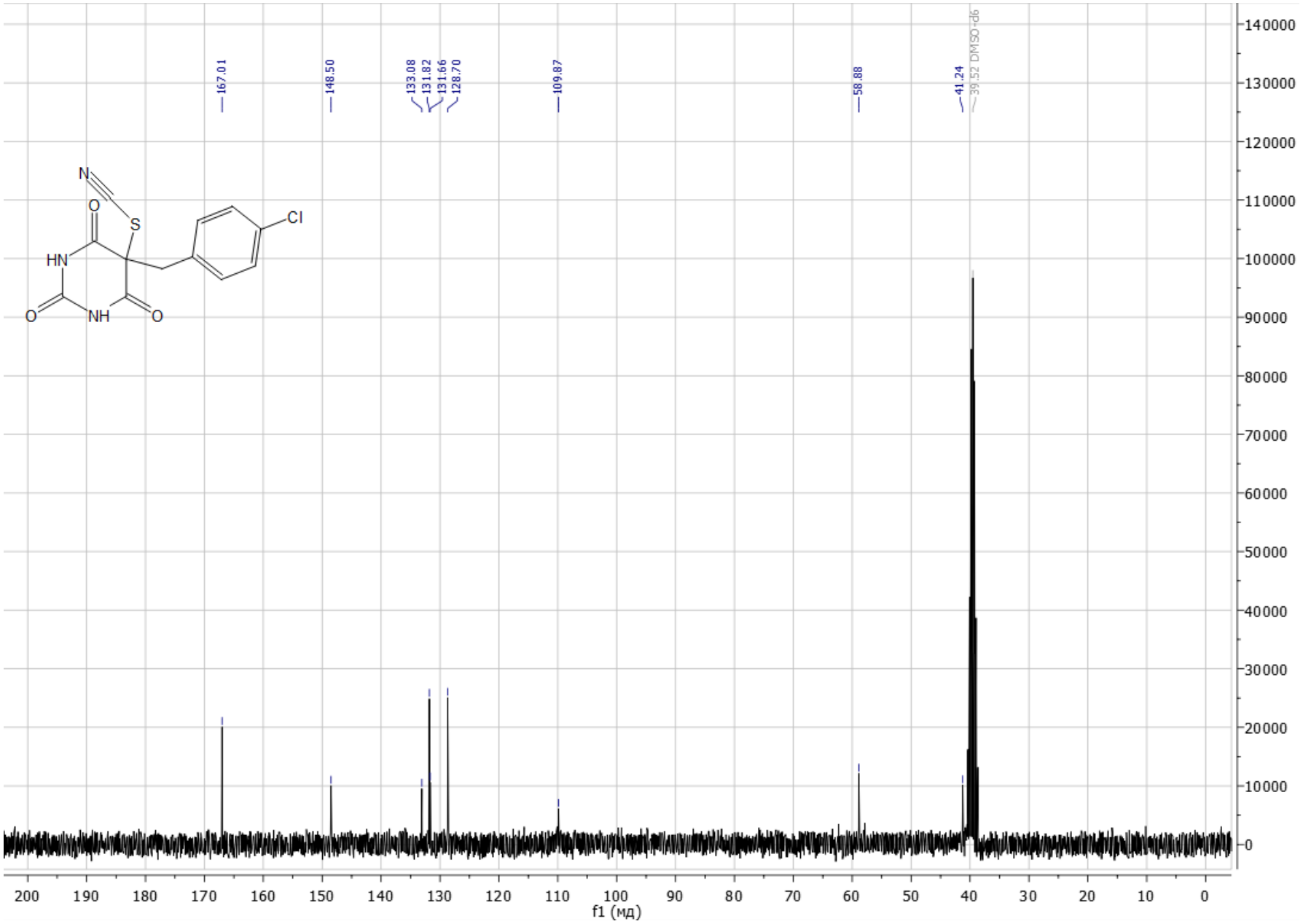
^{13}C NMR (DMSO-d_6) spectrum of 2s



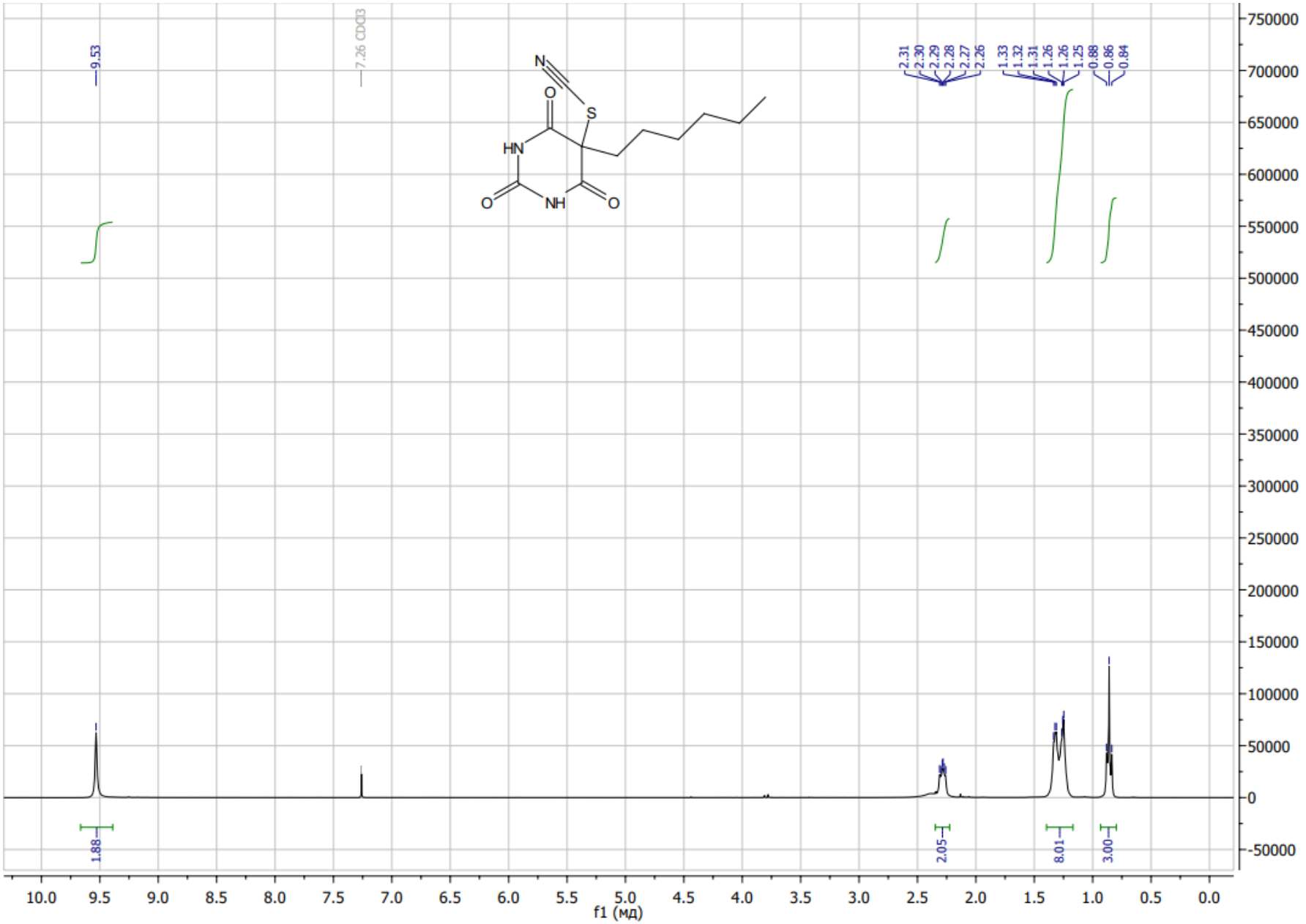
^{1}H NMR (DMSO-d_6) spectrum of 2t



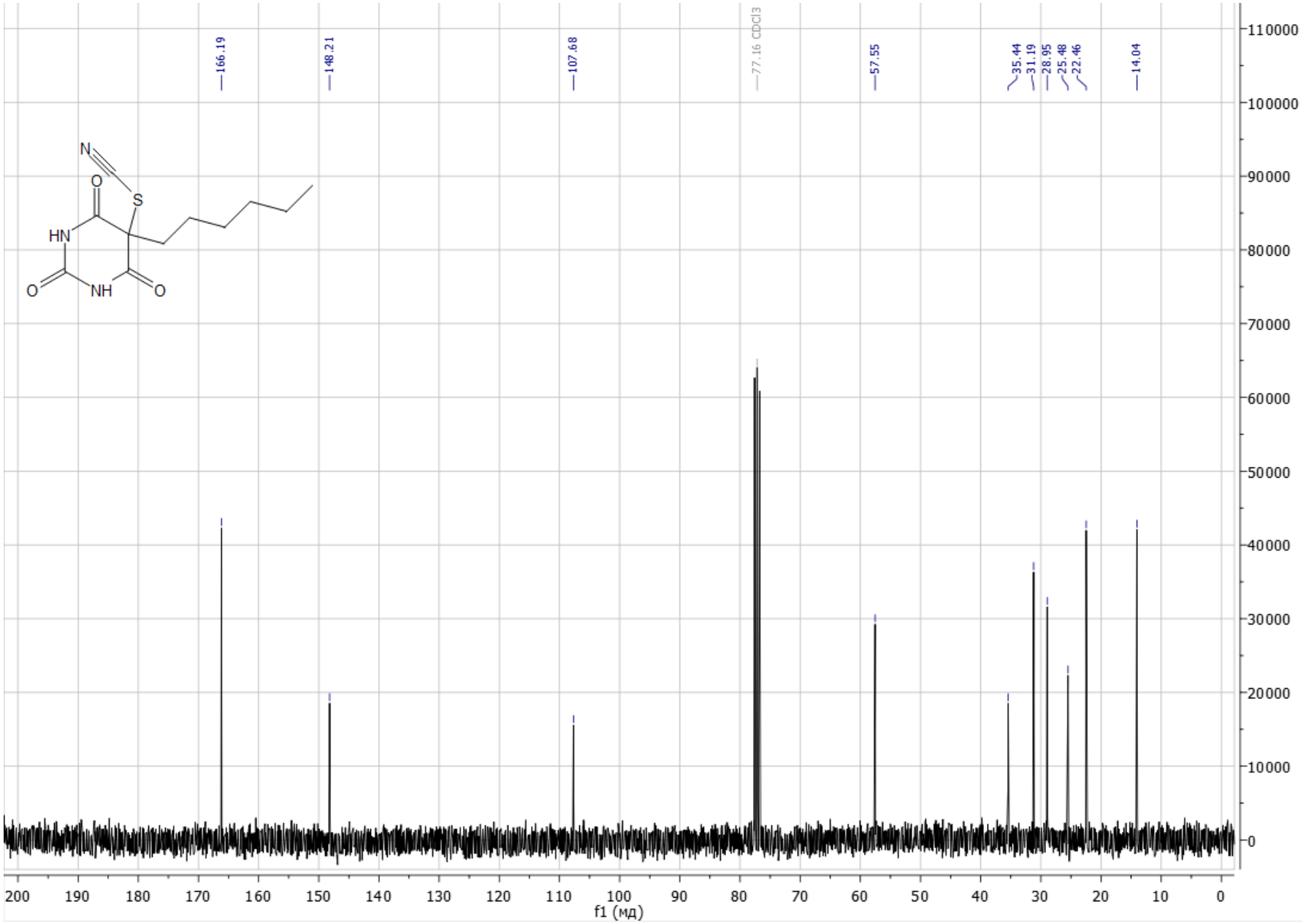
^{13}C NMR (DMSO-d_6) spectrum of 2t



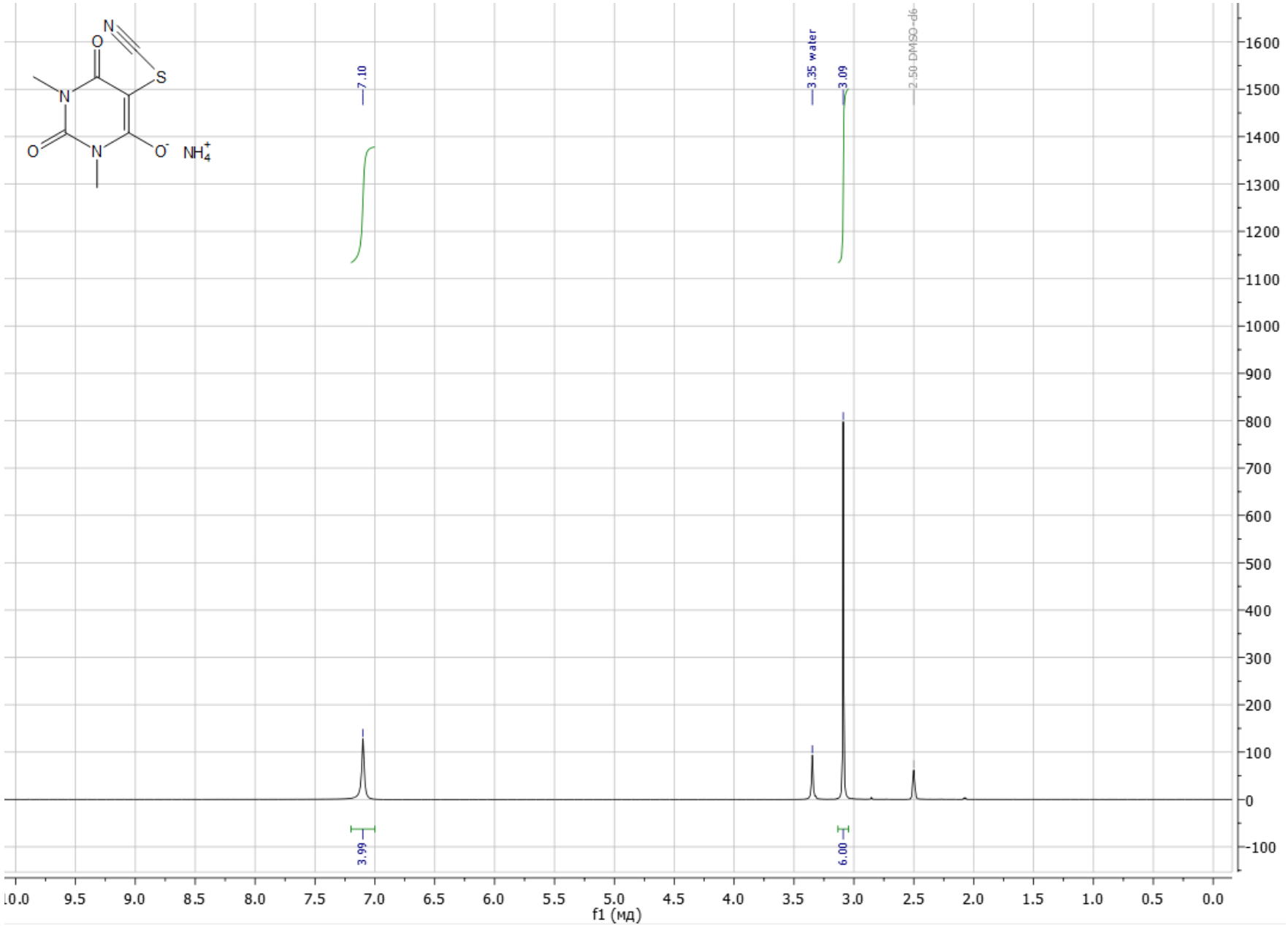
^{1}H NMR ($CDCl_3$) spectrum of 2u



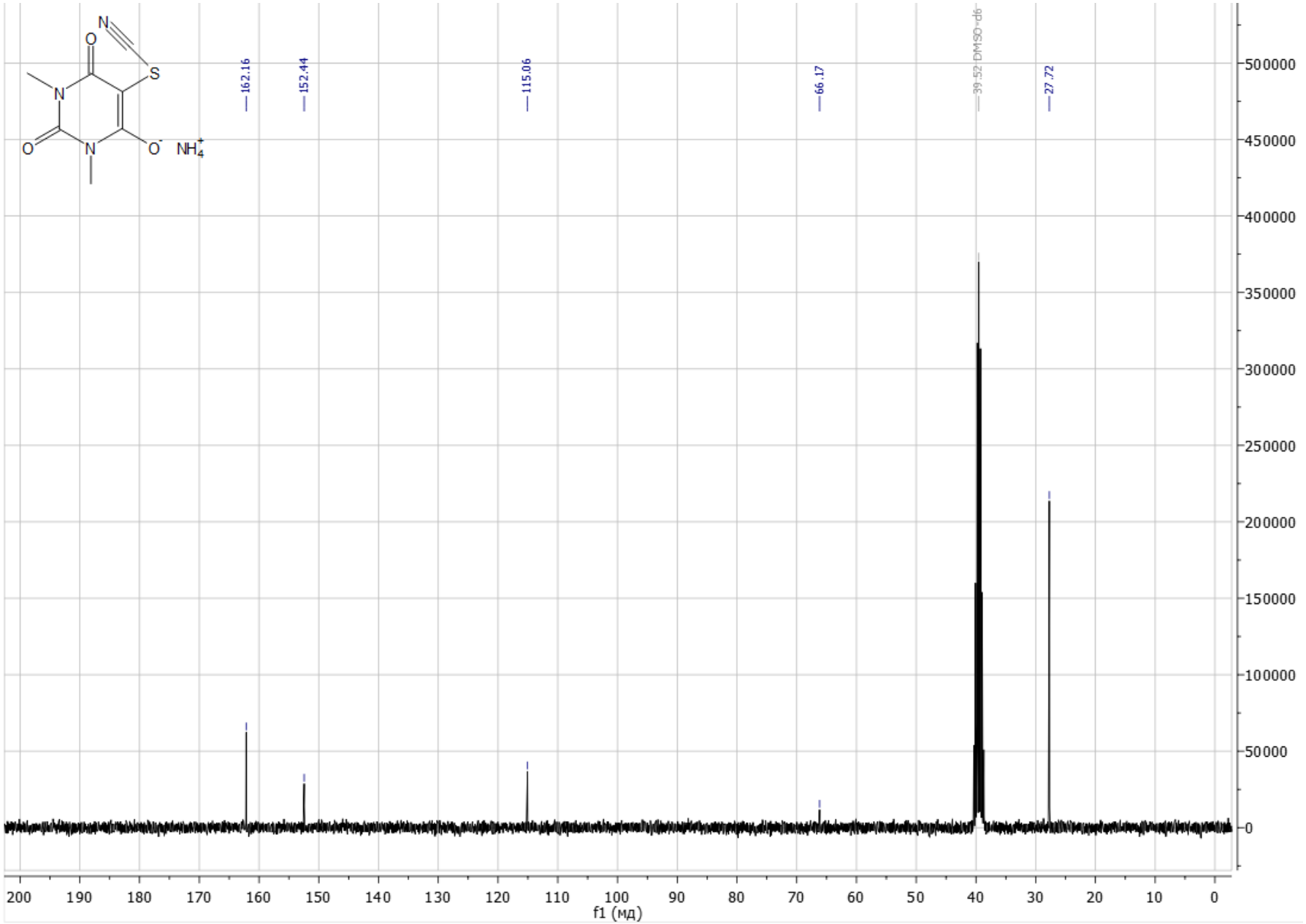
^{13}C NMR ($CDCl_3$) spectrum of 2u



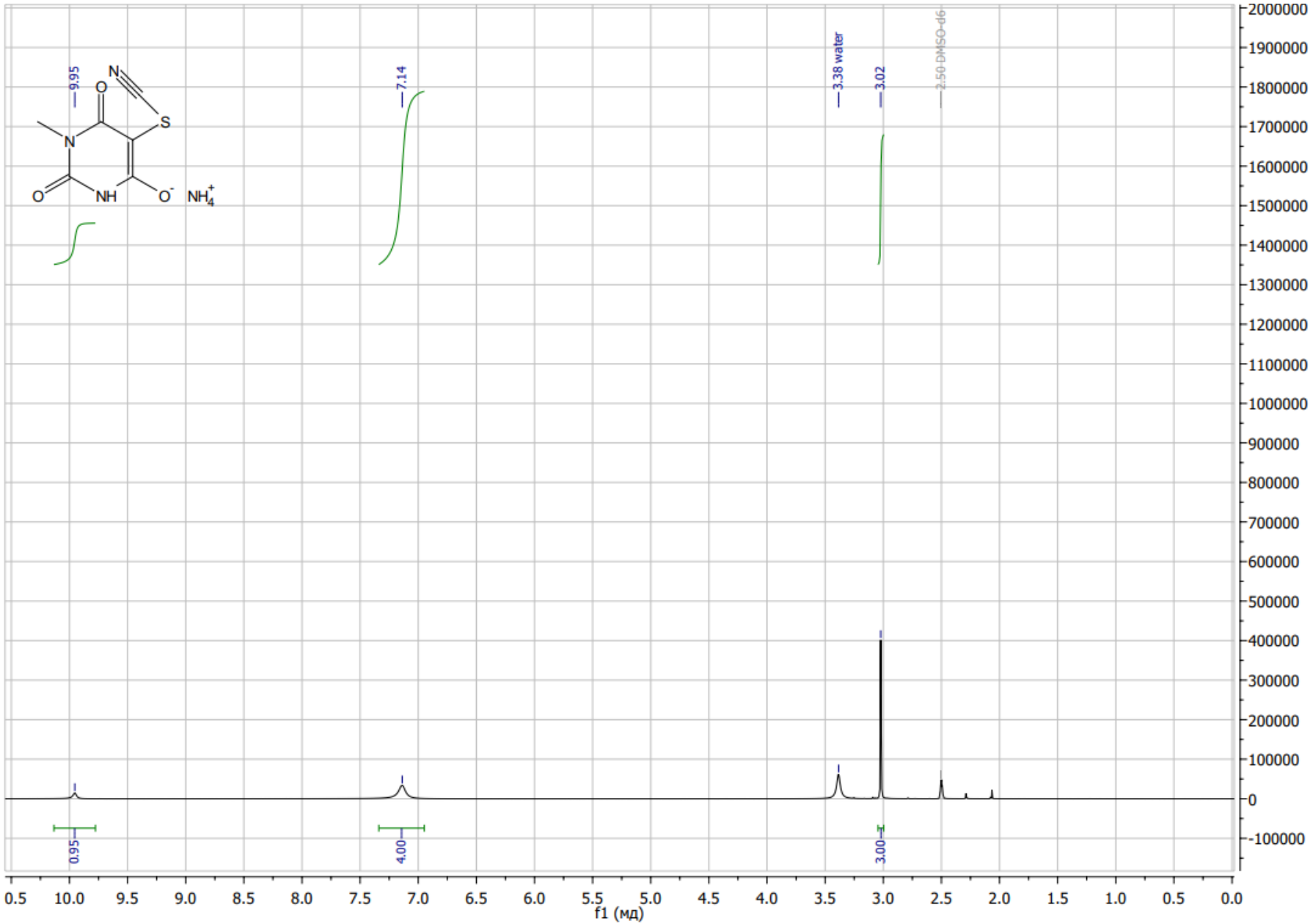
^{1}H NMR (DMSO-*d*$_6$) spectrum of 4a



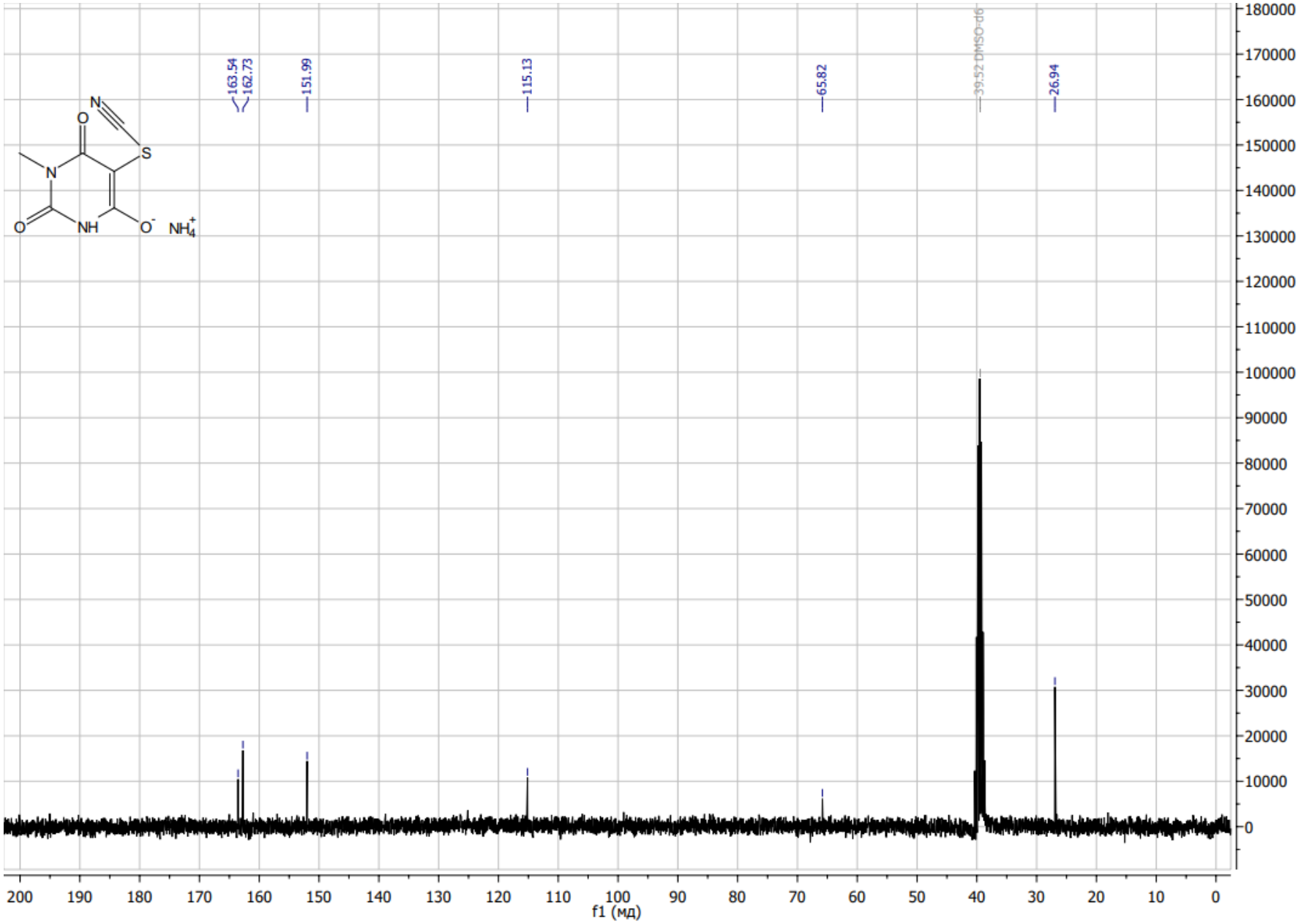
^{13}C NMR (DMSO-d_6) spectrum of 4a



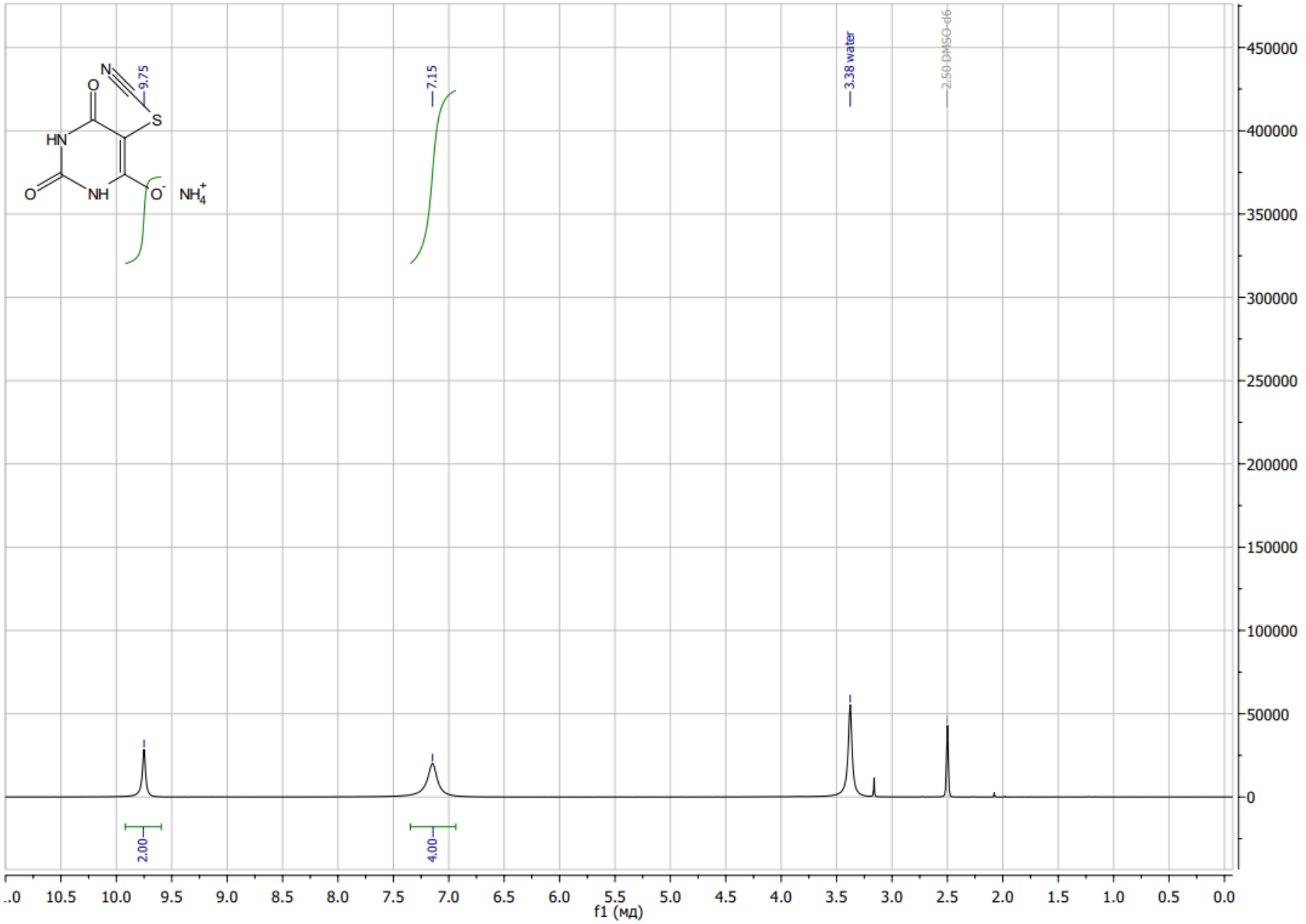
^{1}H NMR (DMSO-d_6) spectrum of 4b



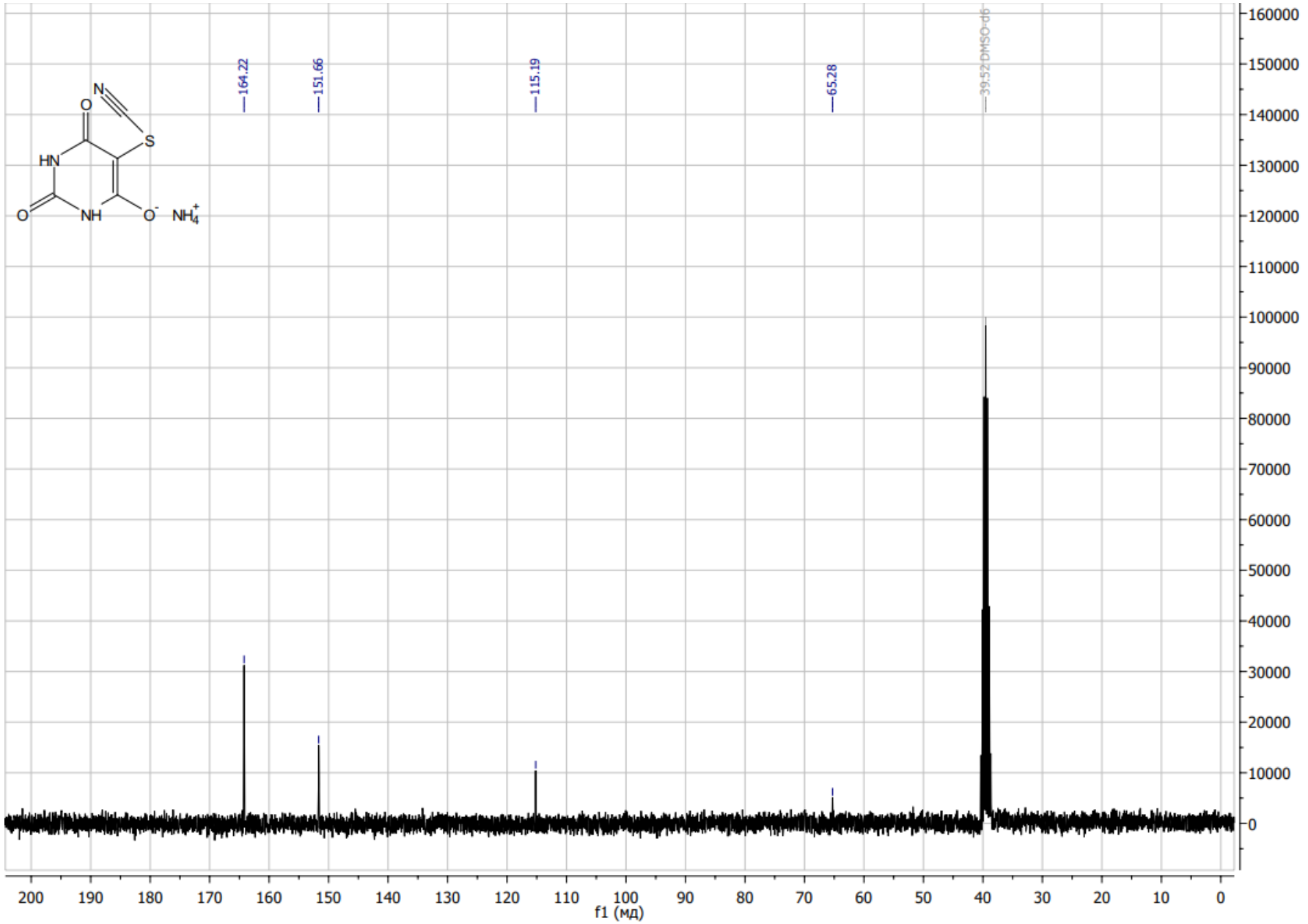
^{13}C NMR (DMSO-d_6) spectrum of 4b



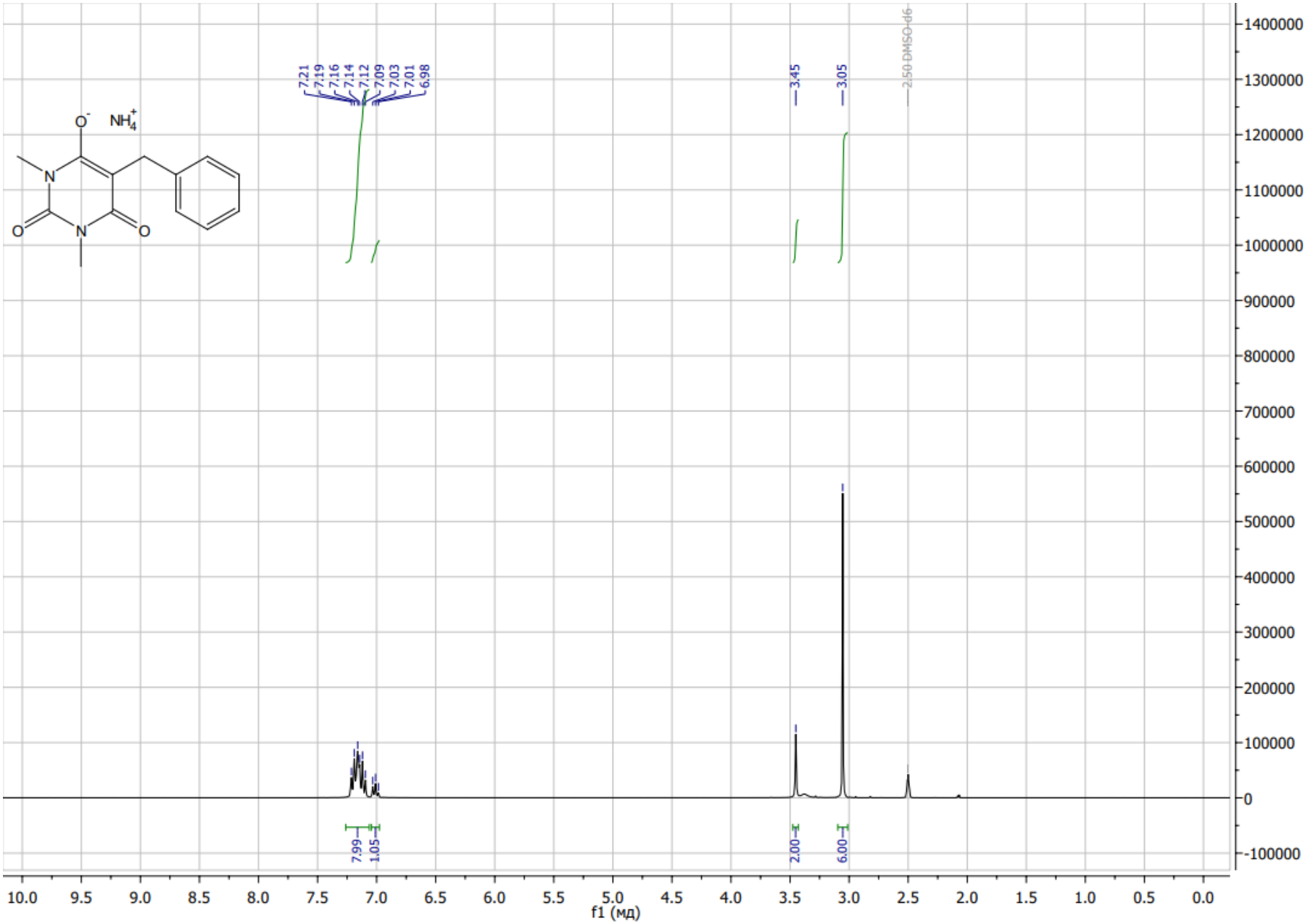
^{1}H NMR (DMSO-d_6) spectrum of 4c



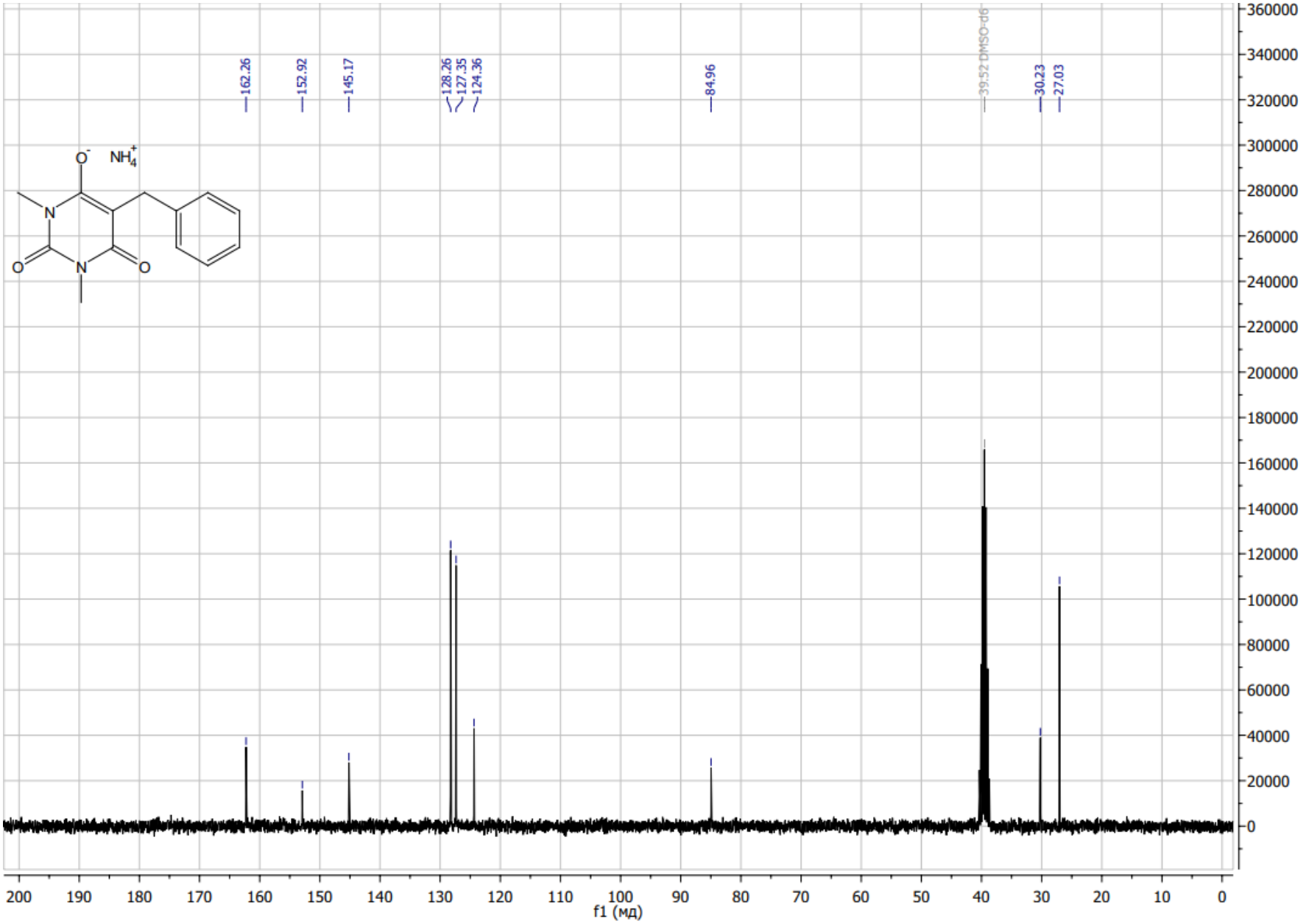
^{13}C NMR (DMSO-d_6) spectrum of 4c



^{1}H NMR (DMSO-*d*$_6$) spectrum of 5



^{13}C NMR (DMSO-d_6) spectrum of 5



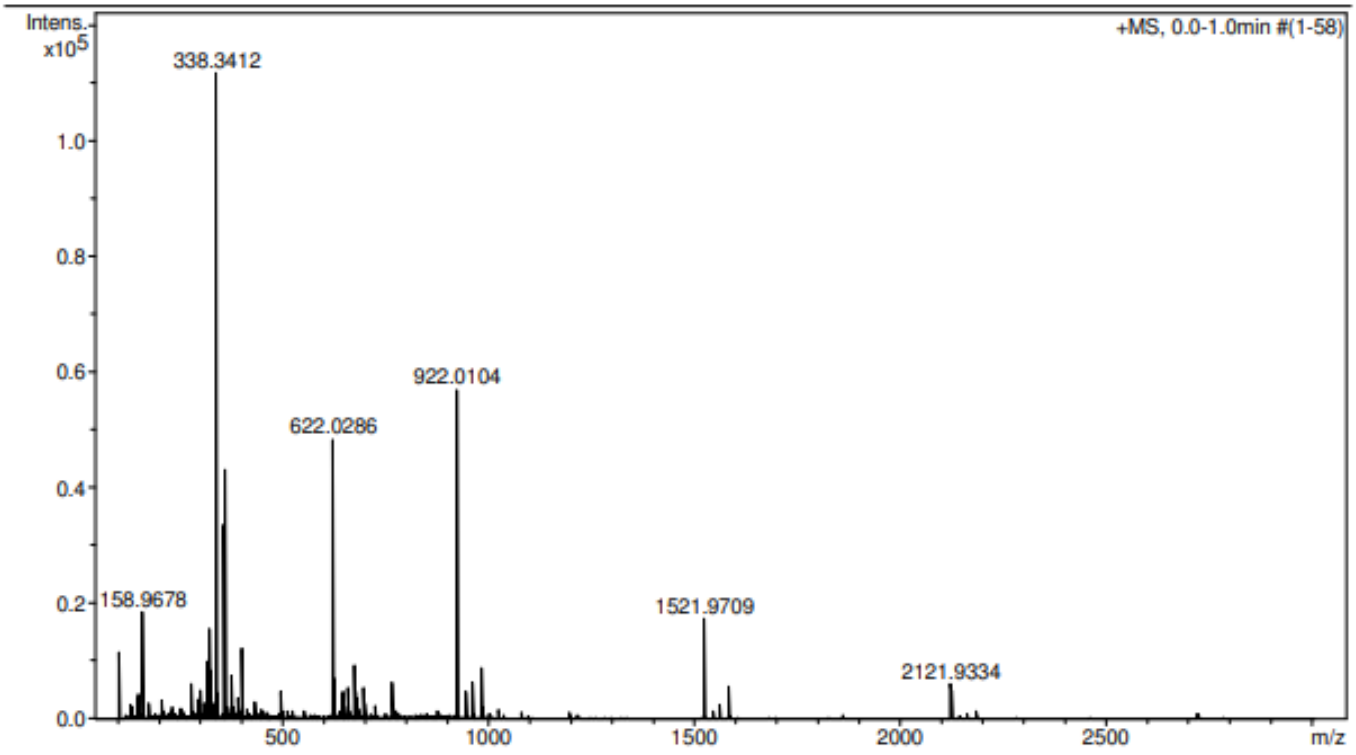
HRMS spectrum of 2a

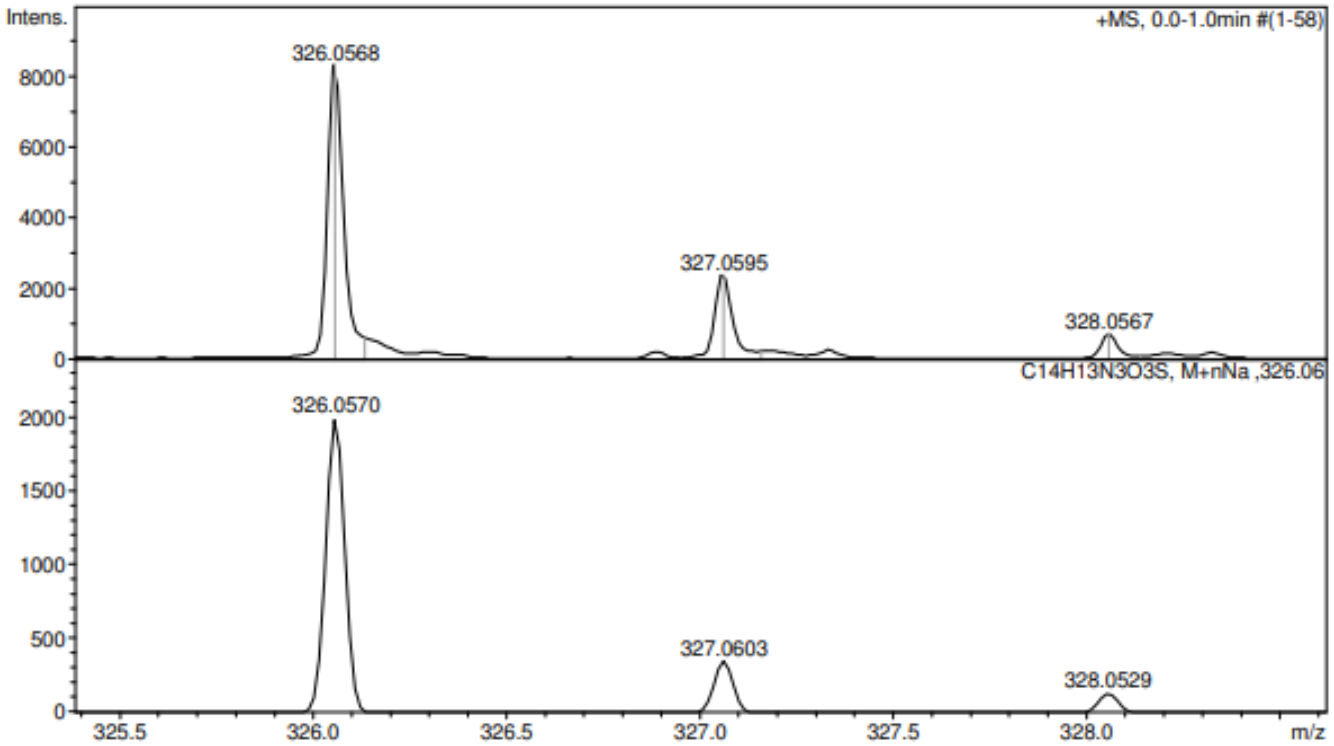
Analysis Info

Analysis Name	D:\Data\Chizhov\Terentiev\Bityukov\pil86_&clblow.d	Acquisition Date	03.02.2020 15:36:15
Method	tune_low.m	Operator	BDAL@DE
Sample Name	/TERN Pil86	Instrument / Ser#	micrOTOF 10248
Comment	CH3CN 100 %, dil. 200, calibrant added		

Acquisition Parameter

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	0.4 Bar
Focus	Not active			Set Dry Heater	180 °C
Scan Begin	50 m/z	Set Capillary	4500 V	Set Dry Gas	4.0 l/min
Scan End	3000 m/z	Set End Plate Offset	-500 V	Set Divert Valve	Waste





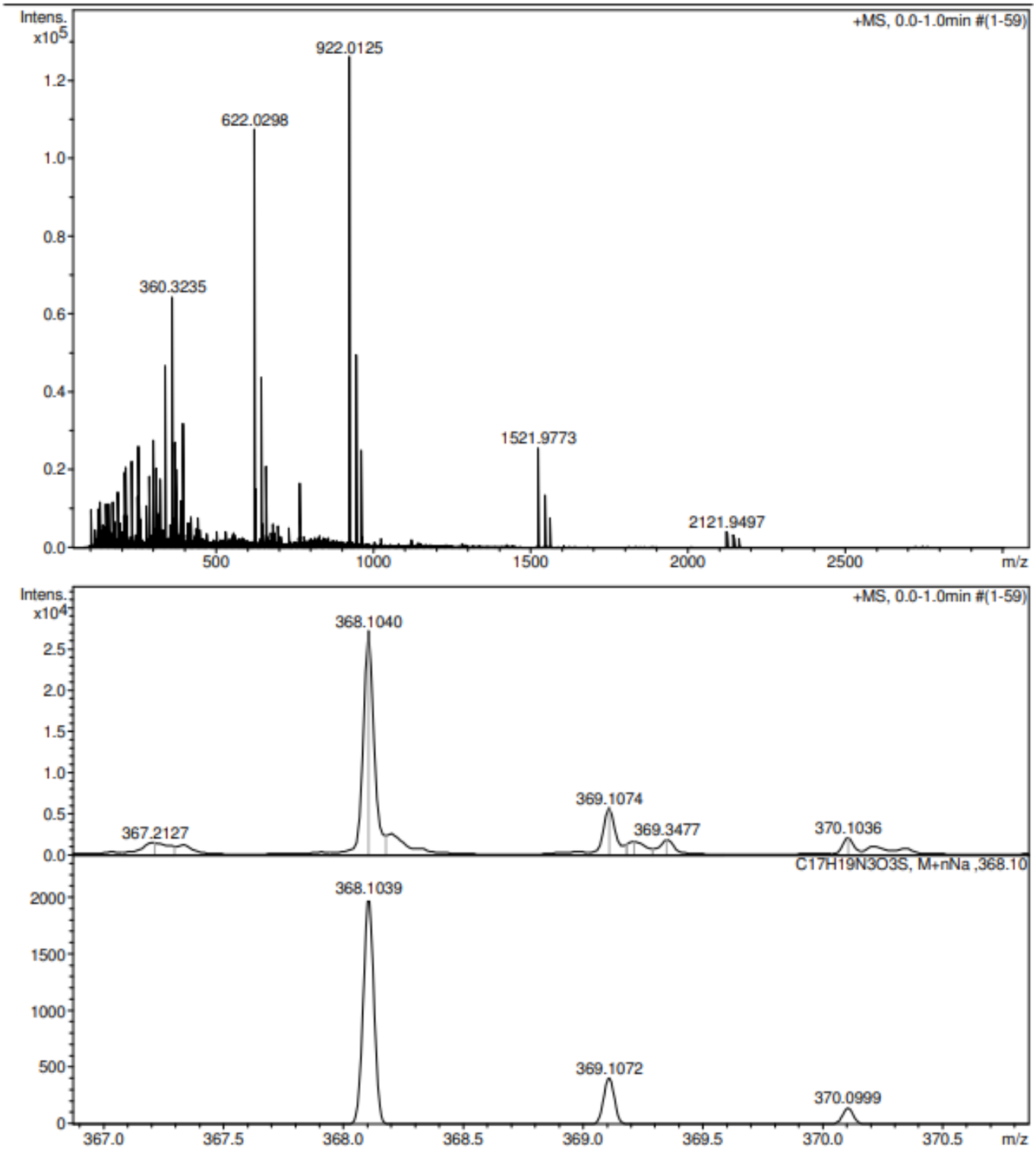
HRMS spectrum of 2b

Analysis Info

Analysis Name	D:\Data\Chizhov\Terentiev\Bityukov\ov2234_&clblow.d	Acquisition Date	14.05.2021 14:36:36
Method	tune_low.m	Operator	BDAL@DE
Sample Name	/TERN ov2234	Instrument / Ser#	micrOTOF 10248
Comment	CH3OH 100 %, dil. 2000, calibrant added		

Acquisition Parameter

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	0.4 Bar
Focus	Not active			Set Dry Heater	180 °C
Scan Begin	50 m/z	Set Capillary	4500 V	Set Dry Gas	4.0 l/min
Scan End	3000 m/z	Set End Plate Offset	-500 V	Set Divert Valve	Waste



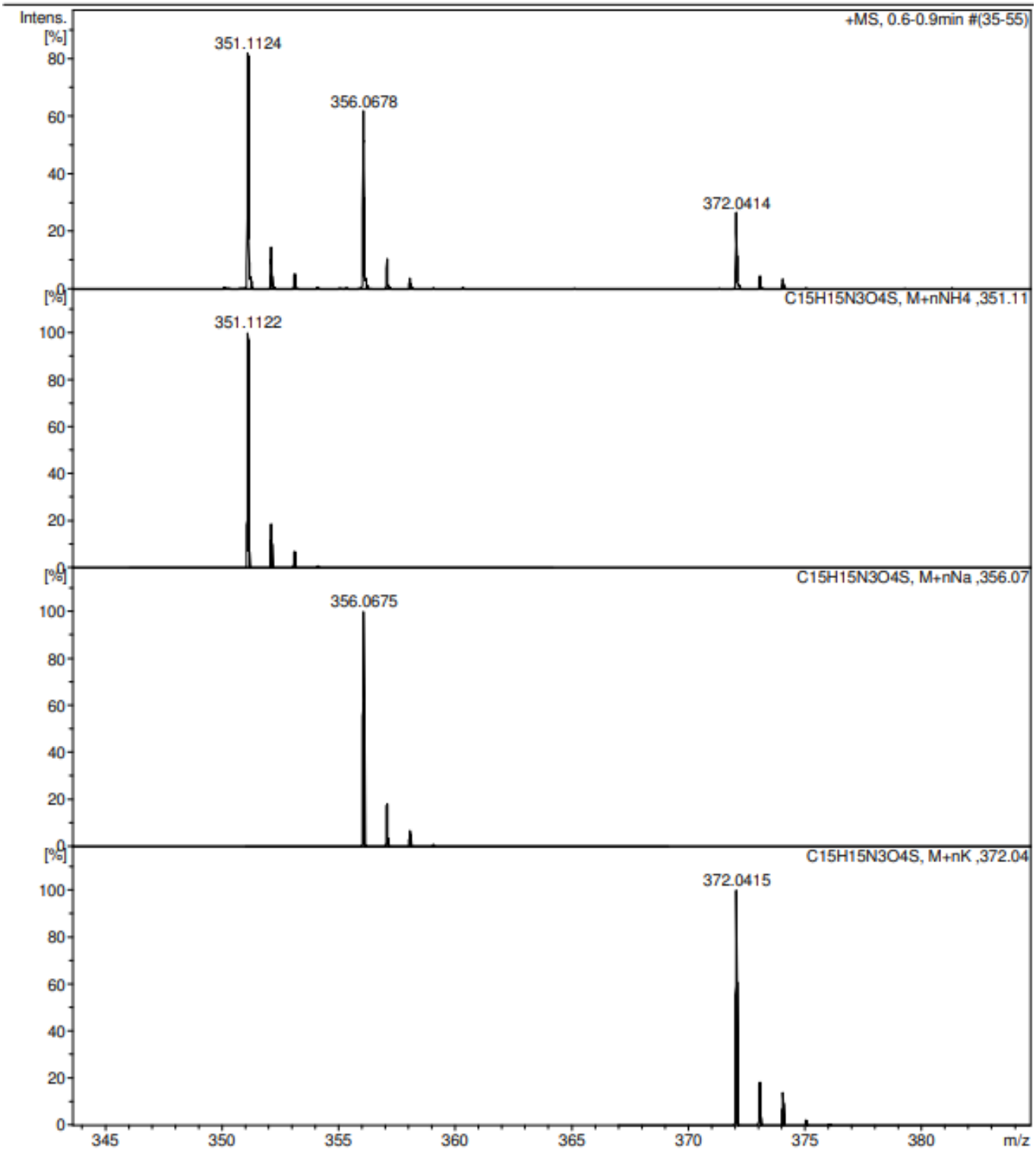
HRMS spectrum of 2c

Analysis Info

Analysis Name	D:\Data\Kolotyrkina\2021\Kirillov\0420053.d	Acquisition Date	20.04.2021 18:22:15
Method	tune_50-1600.m	Operator	BDAL@DE
Sample Name	/TERN ov2206	Instrument / Ser#	micrOTOF 10248
Comment	C15H15N3O4S mH 334.0856 calibrant added CH3CN		

Acquisition Parameter

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	1.0 Bar
Focus	Not active			Set Dry Heater	200 °C
Scan Begin	50 m/z	Set Capillary	4500 V	Set Dry Gas	4.0 l/min
Scan End	1600 m/z	Set End Plate Offset	-500 V	Set Divert Valve	Waste



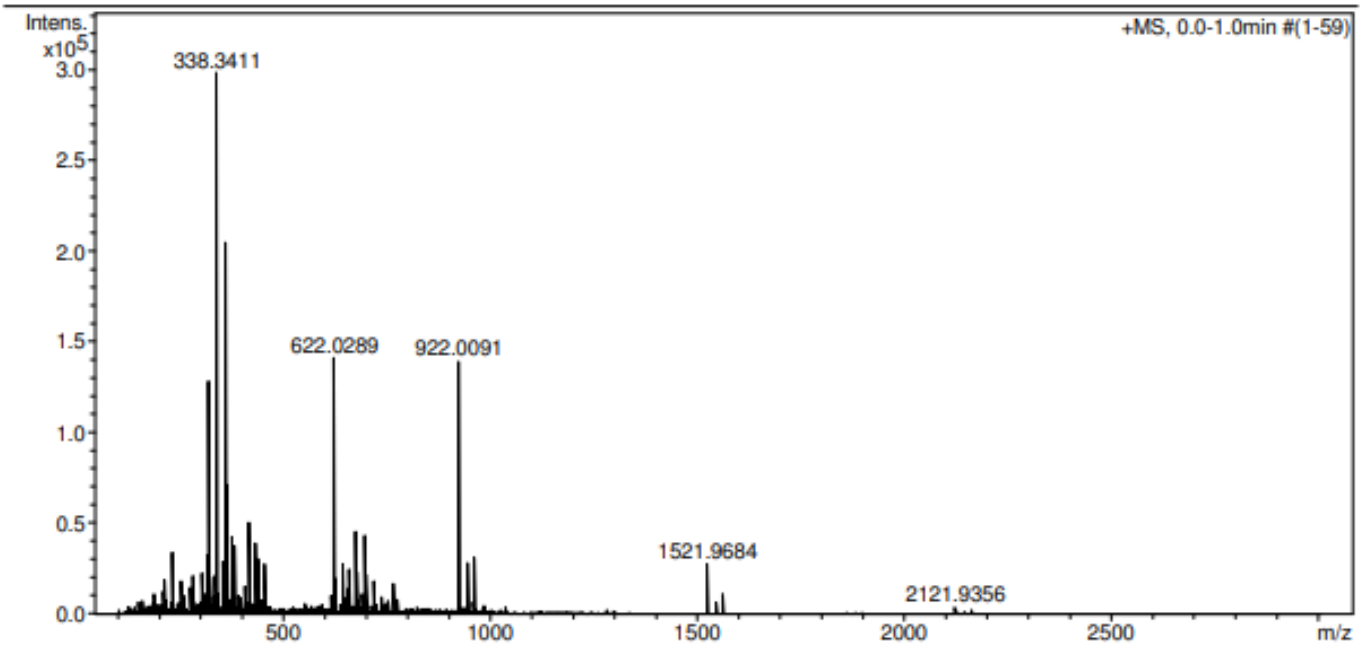
HRMS spectrum of 2d

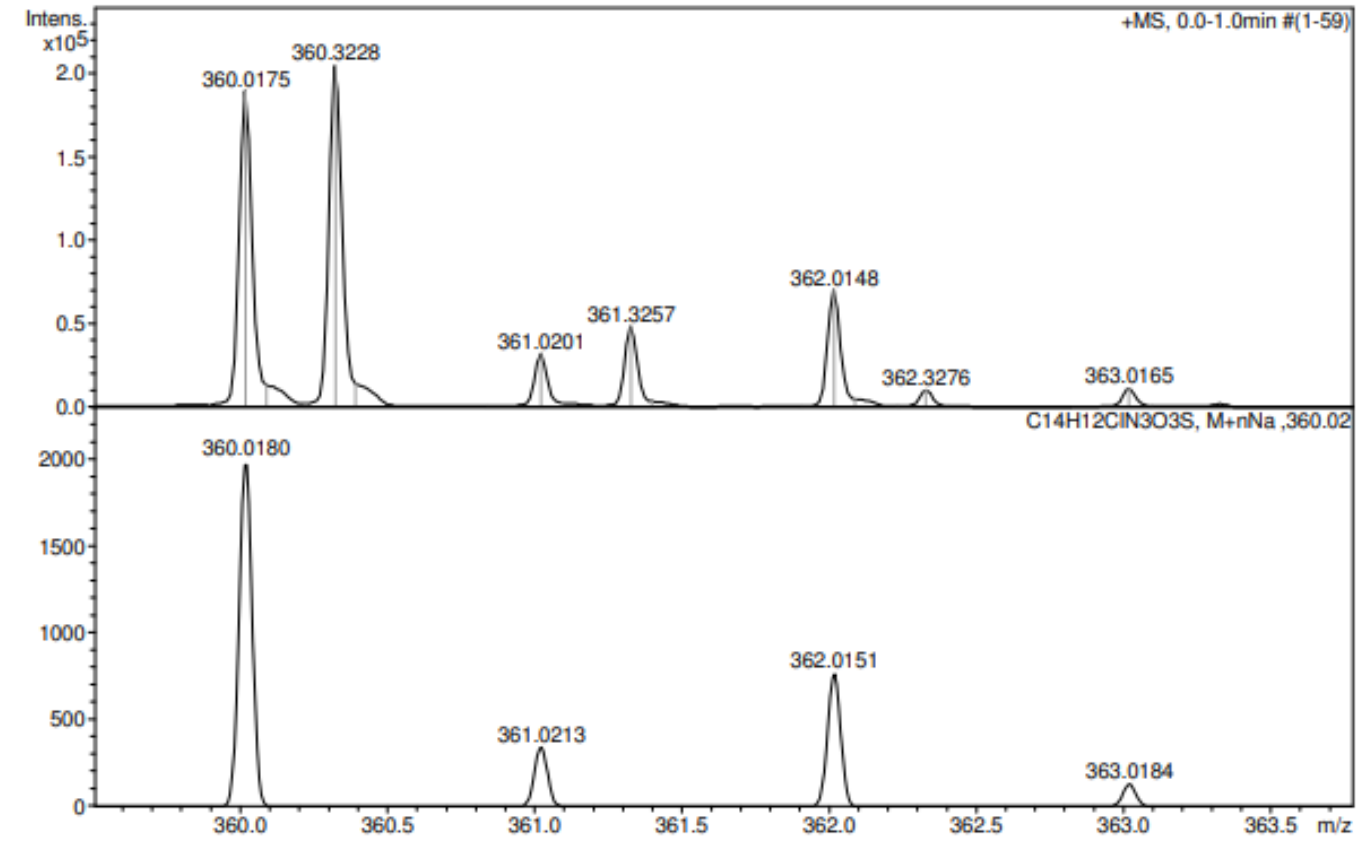
Analysis Info

Acquisition Date	17.06.2021 15:50:30		
Analysis Name	D:\Data\Chizhov\Terentiev\Bityukov\Jun_17_2021\pil279_&clblow%.d		
Method	tune_low.m	Operator	BDAL@DE
Sample Name	/TERN Pil279	Instrument / Ser#	micrOTOF 10248
Comment	CH3OH 100 %, dil. 20, calibrant added		

Acquisition Parameter

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	0.4 Bar
Focus	Not active			Set Dry Heater	180 °C
Scan Begin	50 m/z	Set Capillary	4500 V	Set Dry Gas	4.0 l/min
Scan End	3000 m/z	Set End Plate Offset	-500 V	Set Divert Valve	Waste





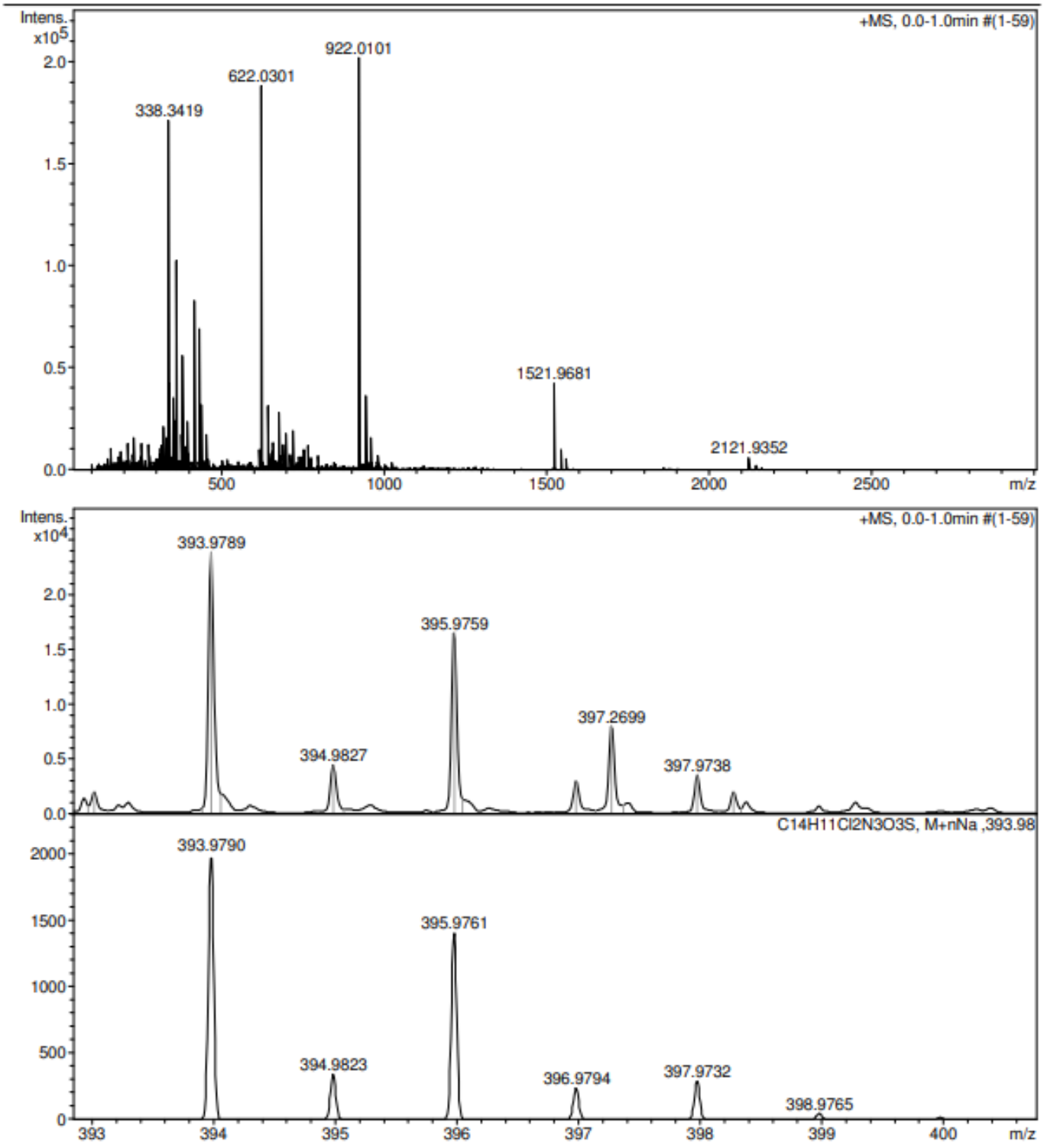
HRMS spectrum of 2e

Analysis Info

Analysis Name	D:\Data\Chizhov\Terentiev\Bityukov\Jun_17_2021\ps26_&clblow%.d	Acquisition Date	17.06.2021 15:39:59
Method	tune_low.m	Operator	BDAL@DE
Sample Name	/TERN PS26	Instrument / Ser#	micrOTOF 10248
Comment	CH3OH 100 %, dil. 200, calibrant added		

Acquisition Parameter

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	0.4 Bar
Focus	Not active			Set Dry Heater	180 °C
Scan Begin	50 m/z	Set Capillary	4500 V	Set Dry Gas	4.0 l/min
Scan End	3000 m/z	Set End Plate Offset	-500 V	Set Divert Valve	Waste



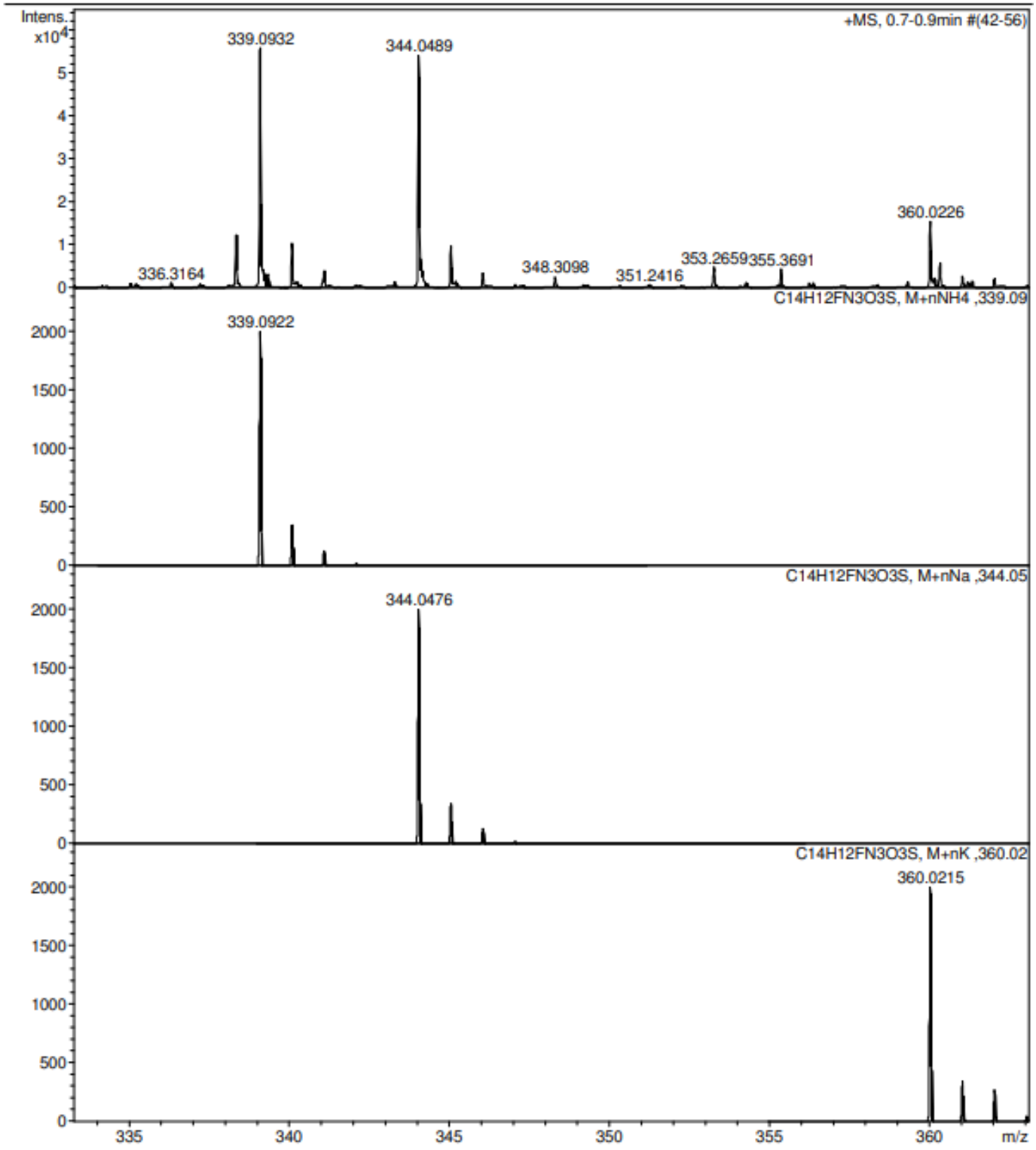
HRMS spectrum of 2f

Analysis Info

Analysis Name	D:\Data\Kolotyrkina\2021\Kirillov\0420050.d	Acquisition Date	20.04.2021 18:03:02
Method	tune_50-1600.m	Operator	BDAL@DE
Sample Name	/TERN ov2211	Instrument / Ser#	micrOTOF 10248
Comment	C14H12FN3O3S mH 322.0656 calibrant added CH3CN		

Acquisition Parameter

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	1.0 Bar
Focus	Not active			Set Dry Heater	200 °C
Scan Begin	50 m/z	Set Capillary	4500 V	Set Dry Gas	4.0 l/min
Scan End	1600 m/z	Set End Plate Offset	-500 V	Set Divert Valve	Waste



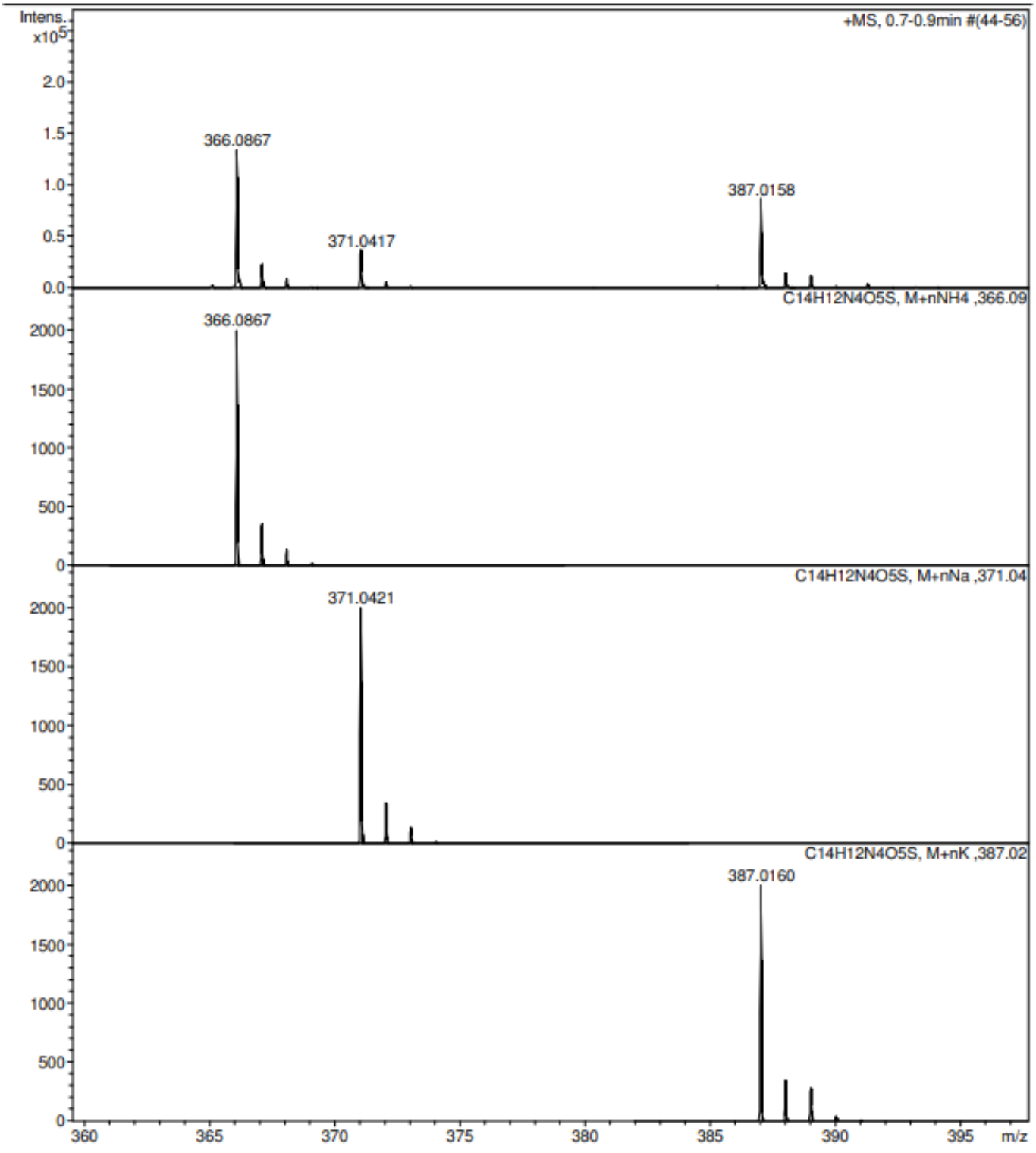
HRMS spectrum of 2g

Analysis Info

Analysis Name	D:\Data\Kolotyrkina\2021\Bitukov\0429044.d	Acquisition Date	29.04.2021 17:06:21
Method	tune_50-1600.m	Operator	BDAL@DE
Sample Name	/TERN OV2233	Instrument / Ser#	micrOTOF 10248
Comment	C14H12N4O5S mH 349.0601 clb added CH3CN		

Acquisition Parameter

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	1.0 Bar
Focus	Not active			Set Dry Heater	200 °C
Scan Begin	50 m/z	Set Capillary	4500 V	Set Dry Gas	4.0 l/min
Scan End	1600 m/z	Set End Plate Offset	-500 V	Set Divert Valve	Waste



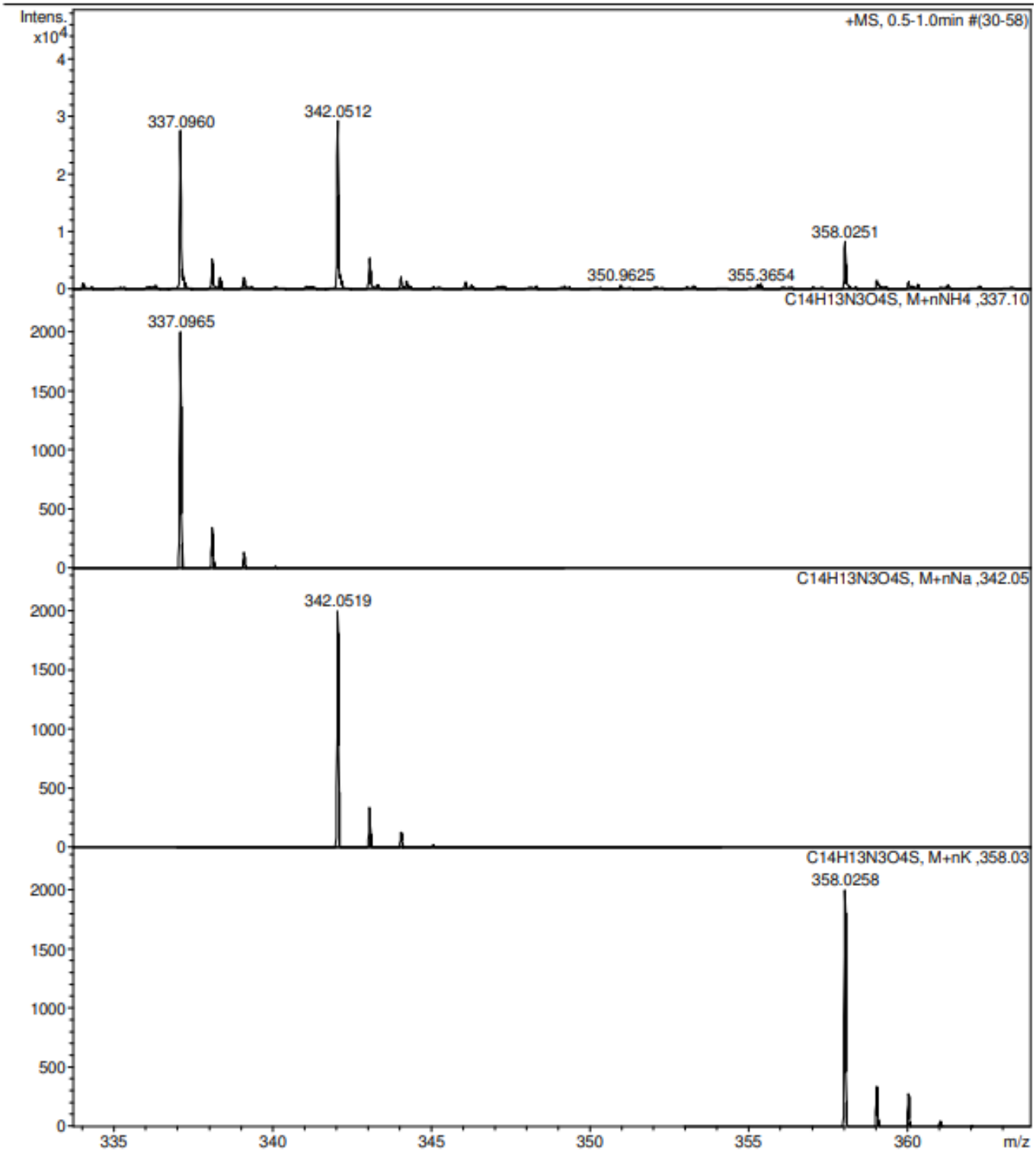
HRMS spectrum of 2h

Analysis Info

Analysis Name	D:\Data\Kolotyrkina\2021\Bitukov\04290434.d	Acquisition Date	29.04.2021 16:47:48
Method	tune_50-1600.m	Operator	BDAL@DE
Sample Name	/TERN OV2228	Instrument / Ser#	micrOTOF 10248
Comment	C14H13N3O4S mH 320.0699 clb added CH3CN		

Acquisition Parameter

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	1.0 Bar
Focus	Not active			Set Dry Heater	200 °C
Scan Begin	50 m/z	Set Capillary	4500 V	Set Dry Gas	4.0 l/min
Scan End	1600 m/z	Set End Plate Offset	-500 V	Set Divert Valve	Waste



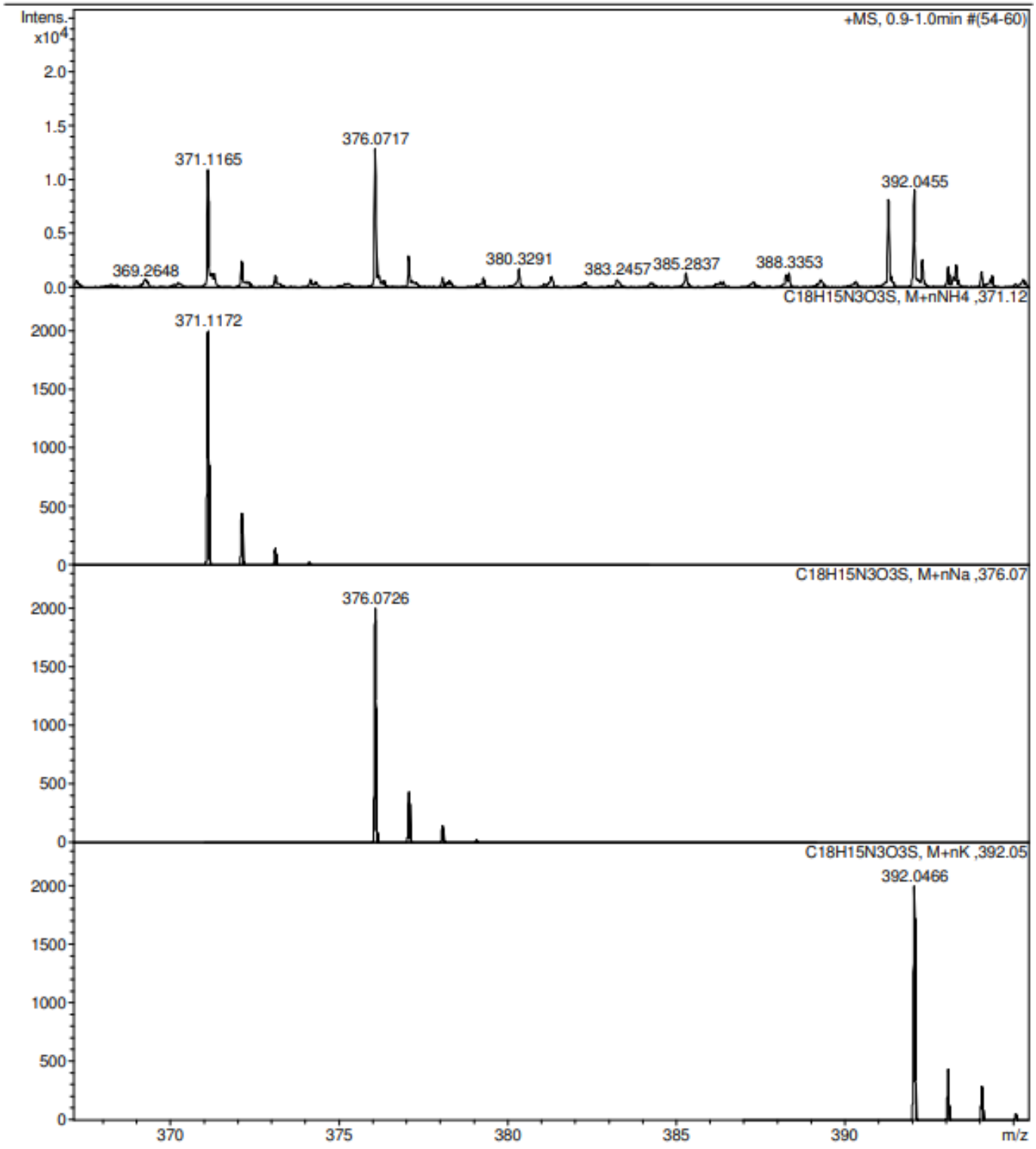
HRMS spectrum of 2i

Analysis Info

Analysis Name	D:\Data\Kolotyrkina\2021\Kirillov\0420051.d	Acquisition Date	20.04.2021 18:11:10
Method	tune_50-1600.m	Operator	BDAL@DE
Sample Name	/TERN ov2209	Instrument / Ser#	micrOTOF 10248
Comment	C18H15N3O3S mH 354.0906 calibrant added CH3CN		

Acquisition Parameter

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	1.0 Bar
Focus	Not active			Set Dry Heater	200 °C
Scan Begin	50 m/z	Set Capillary	4500 V	Set Dry Gas	4.0 l/min
Scan End	1600 m/z	Set End Plate Offset	-500 V	Set Divert Valve	Waste



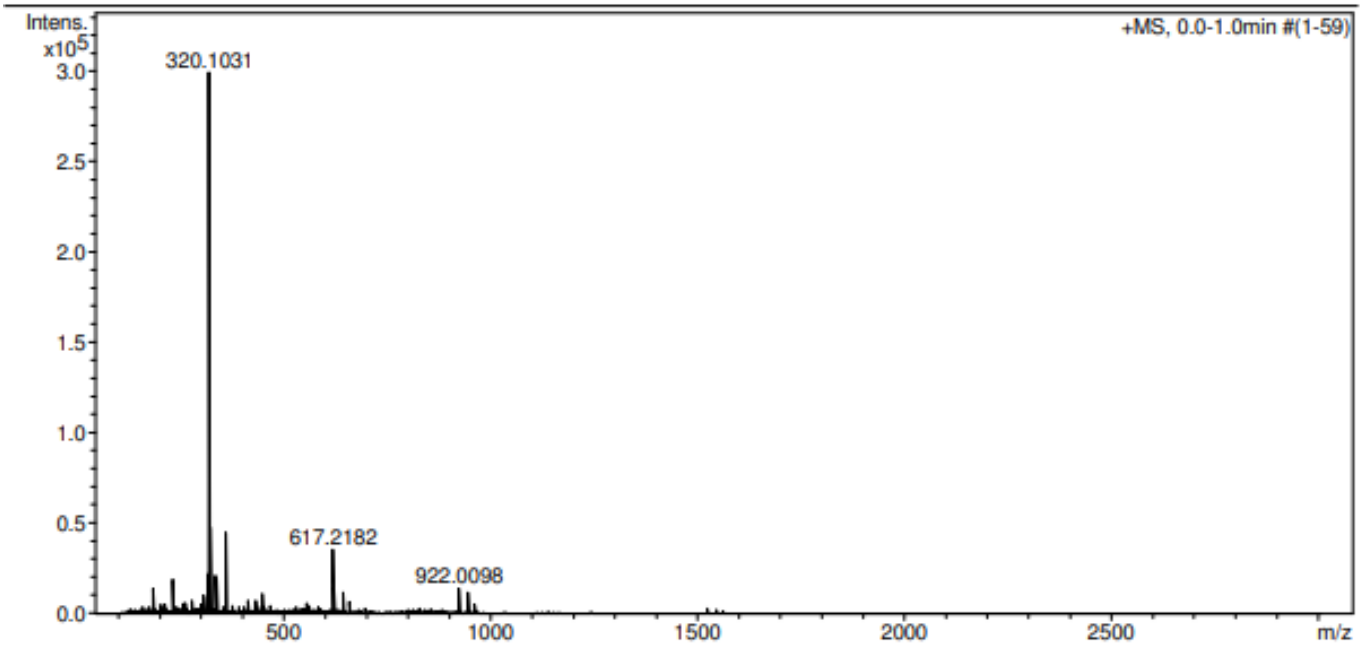
HRMS spectrum of 2j

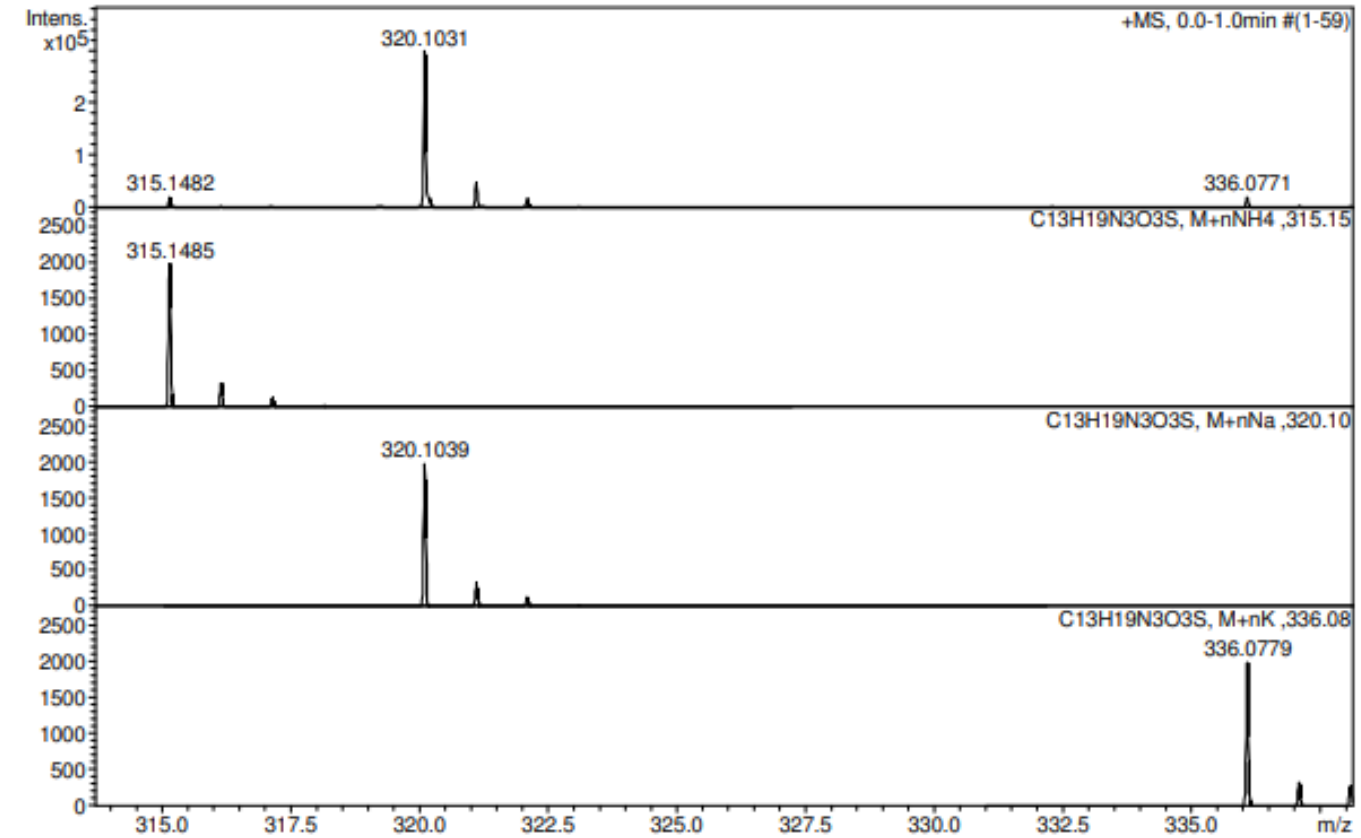
Analysis Info

Acquisition Date	17.06.2021 15:08:38		
Analysis Name	D:\Data\Chizhov\Terentiev\Bityukov\Jun_17_2021\ov2269_&clblow.d		
Method	tune_low.m	Operator	BDAL@DE
Sample Name	/TERN OV2269	Instrument / Ser#	micrOTOF 10248
Comment	CH3OH 100 %, dil. 20000, calibrant added		

Acquisition Parameter

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	0.4 Bar
Focus	Not active			Set Dry Heater	180 °C
Scan Begin	50 m/z	Set Capillary	4500 V	Set Dry Gas	4.0 l/min
Scan End	3000 m/z	Set End Plate Offset	-500 V	Set Divert Valve	Waste





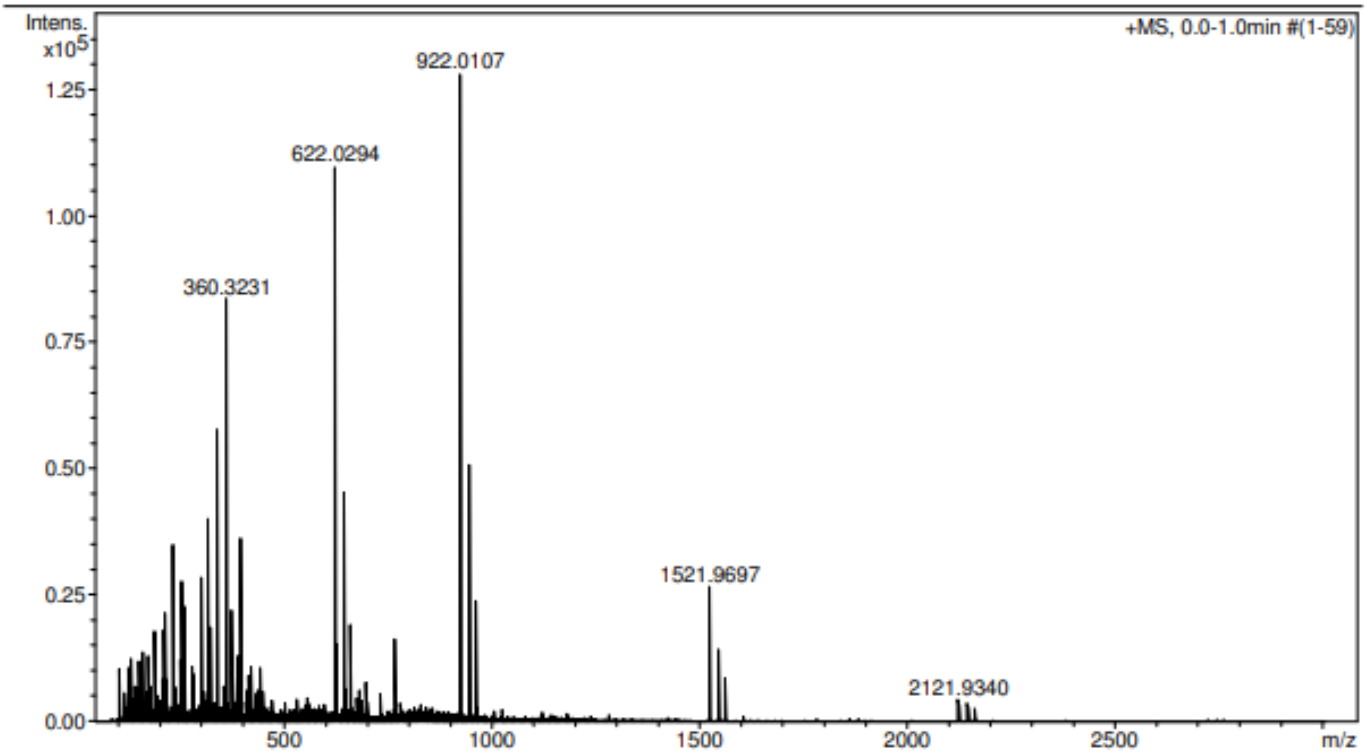
HRMS spectrum of 2k

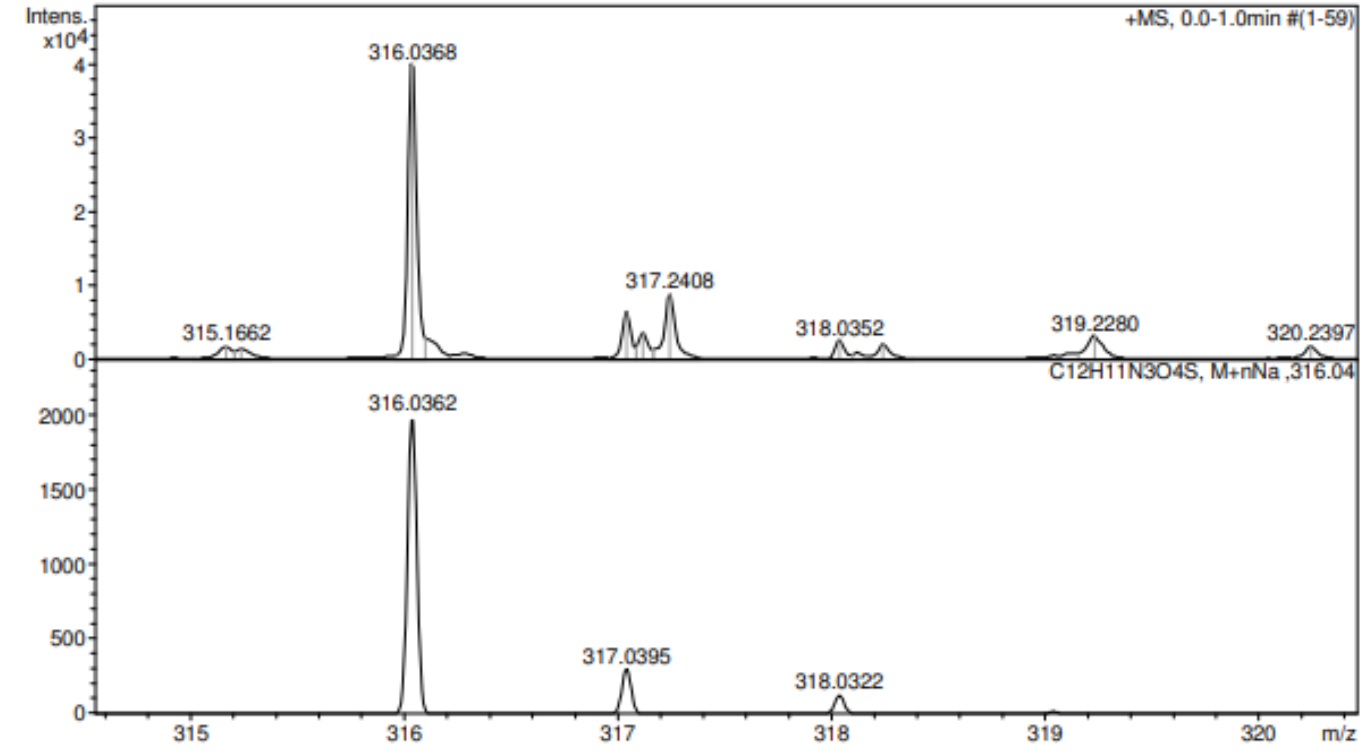
Analysis Info

Analysis Name	D:\Data\Chizhov\Terentiev\Bityukov\ov2231_&clblow.d	Acquisition Date	14.05.2021 14:24:02
Method	tune_low.m	Operator	BDAL@DE
Sample Name	/TERN ov2231	Instrument / Ser#	micrOTOF 10248
Comment	CH3OH 100 %, dil. 2000, calibrant added		

Acquisition Parameter

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	0.4 Bar
Focus	Not active			Set Dry Heater	180 °C
Scan Begin	50 m/z	Set Capillary	4500 V	Set Dry Gas	4.0 l/min
Scan End	3000 m/z	Set End Plate Offset	-500 V	Set Divert Valve	Waste





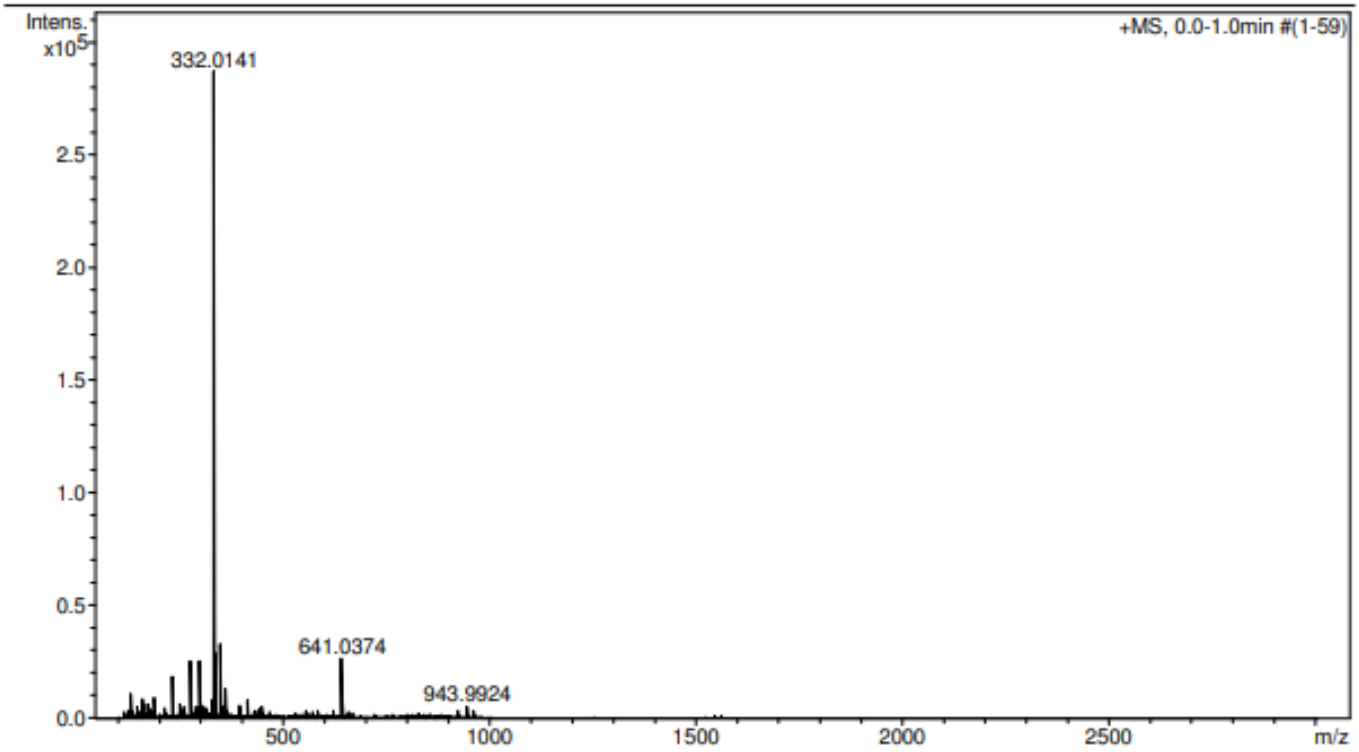
HRMS spectrum of 2l

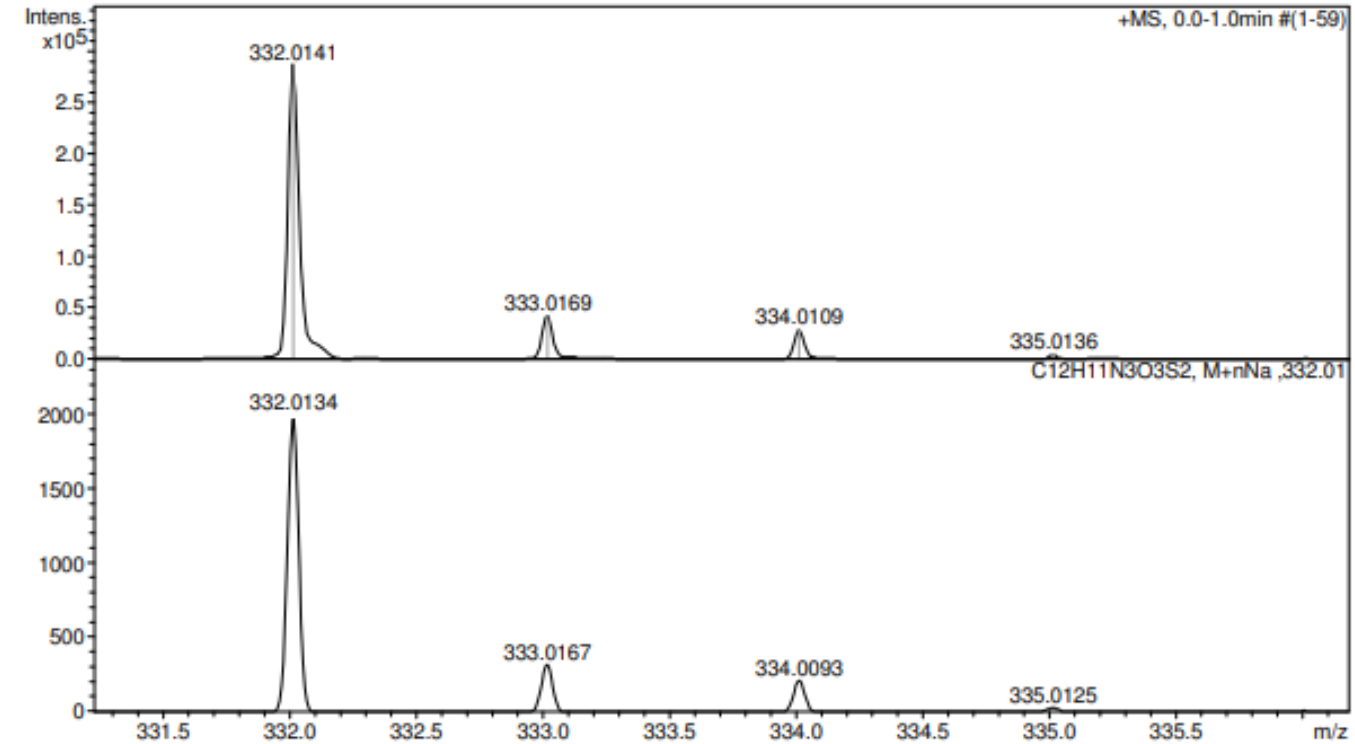
Analysis Info

Analysis Name	D:\Data\Chizhov\Terentiev\Bityukov\ov2232_&clblow.d	Acquisition Date	14.05.2021 14:31:52
Method	tune_low.m	Operator	BDAL@DE
Sample Name	/TERN ov2232	Instrument / Ser#	micrOTOF 10248
Comment	CH3OH 100 %, dil. 20, calibrant added		

Acquisition Parameter

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	0.4 Bar
Focus	Not active			Set Dry Heater	180 °C
Scan Begin	50 m/z	Set Capillary	4500 V	Set Dry Gas	4.0 l/min
Scan End	3000 m/z	Set End Plate Offset	-500 V	Set Divert Valve	Waste





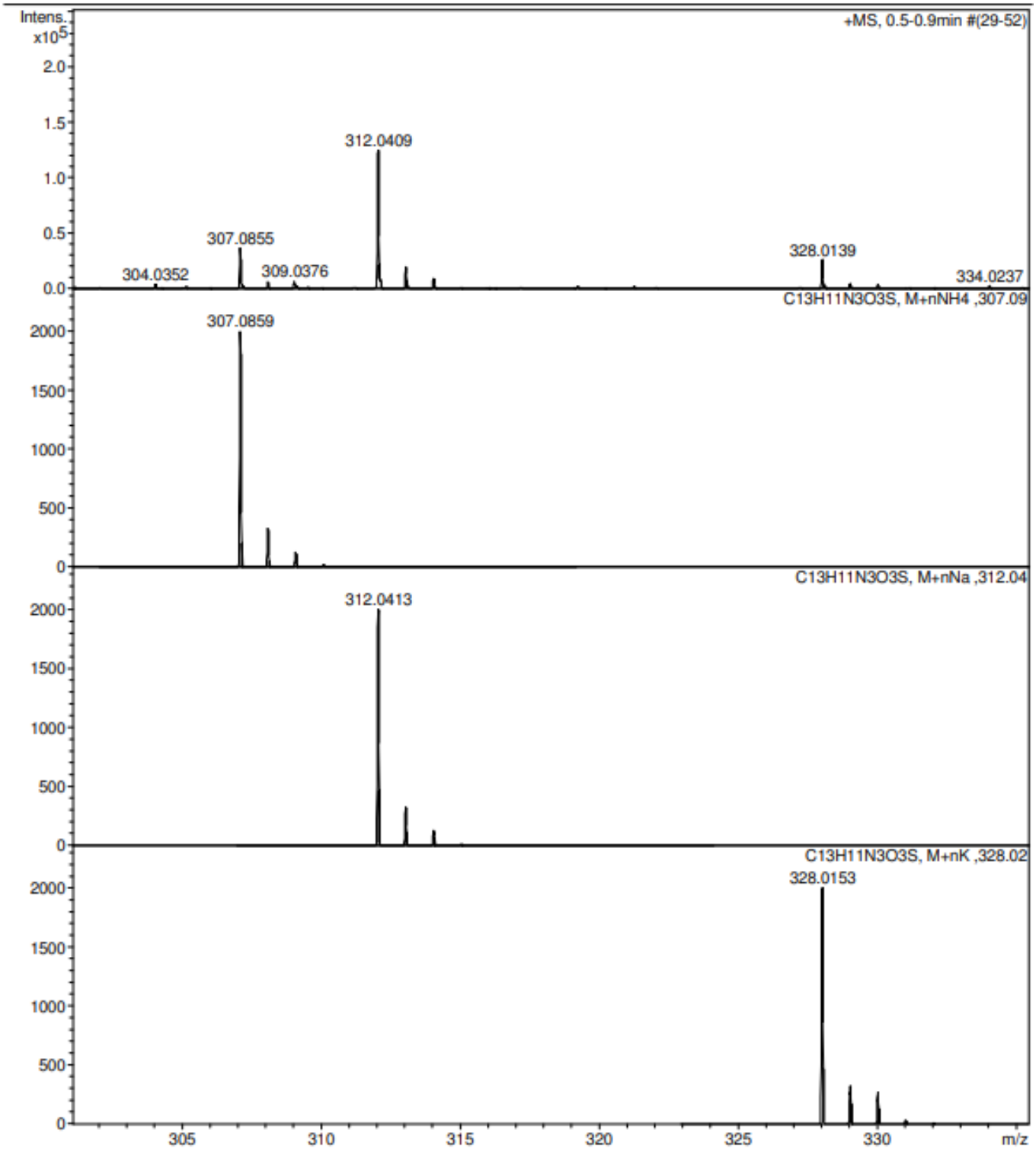
HRMS spectrum of 2m

Analysis Info

Analysis Name	D:\Data\Kolotyrkina\2021\Bitukov\0429043.d	Acquisition Date	29.04.2021 16:52:46
Method	tune_50-1600.m	Operator	BDAL@DE
Sample Name	/TERN OV2229	Instrument / Ser#	micrOTOF 10248
Comment	C13H11N3O3S mH 290.0593 clb added CH3CN		

Acquisition Parameter

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	1.0 Bar
Focus	Not active			Set Dry Heater	200 °C
Scan Begin	50 m/z	Set Capillary	4500 V	Set Dry Gas	4.0 l/min
Scan End	1600 m/z	Set End Plate Offset	-500 V	Set Divert Valve	Waste



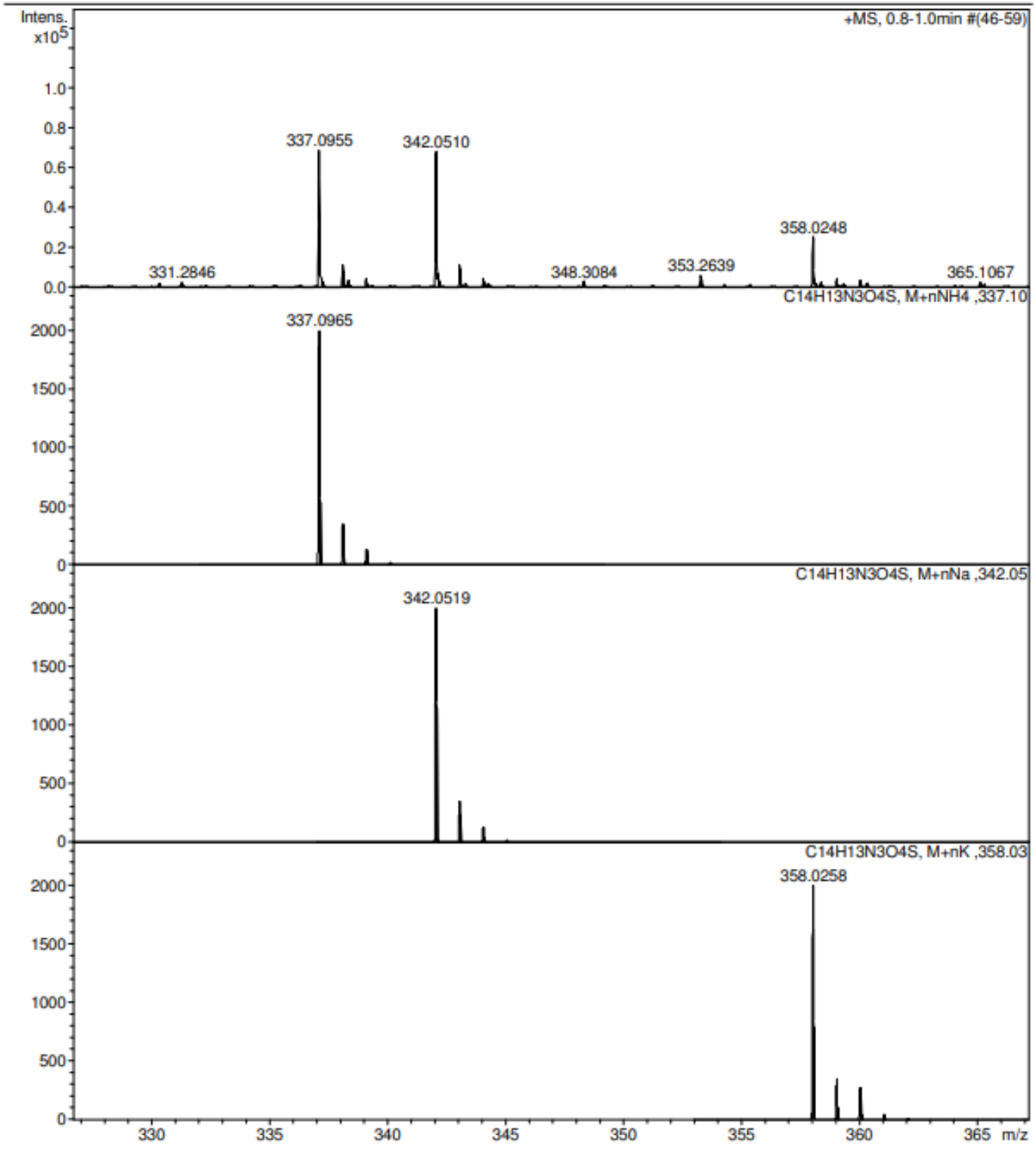
HRMS spectrum of 2n

Analysis Info

Analysis Name	D:\Data\Kolotyrkina\2021\Kirillov\0420052.d	Acquisition Date	20.04.2021 18:16:59
Method	tune_50-1600.m	Operator	BDAL@DE
Sample Name	/TERN ov2207	Instrument / Ser#	micrOTOF 10248
Comment	C14H13N3O4S mH 320.0699 calibrant added CH3CN		

Acquisition Parameter

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	1.0 Bar
Focus	Not active			Set Dry Heater	200 °C
Scan Begin	50 m/z	Set Capillary	4500 V	Set Dry Gas	4.0 l/min
Scan End	1600 m/z	Set End Plate Offset	-500 V	Set Divert Valve	Waste



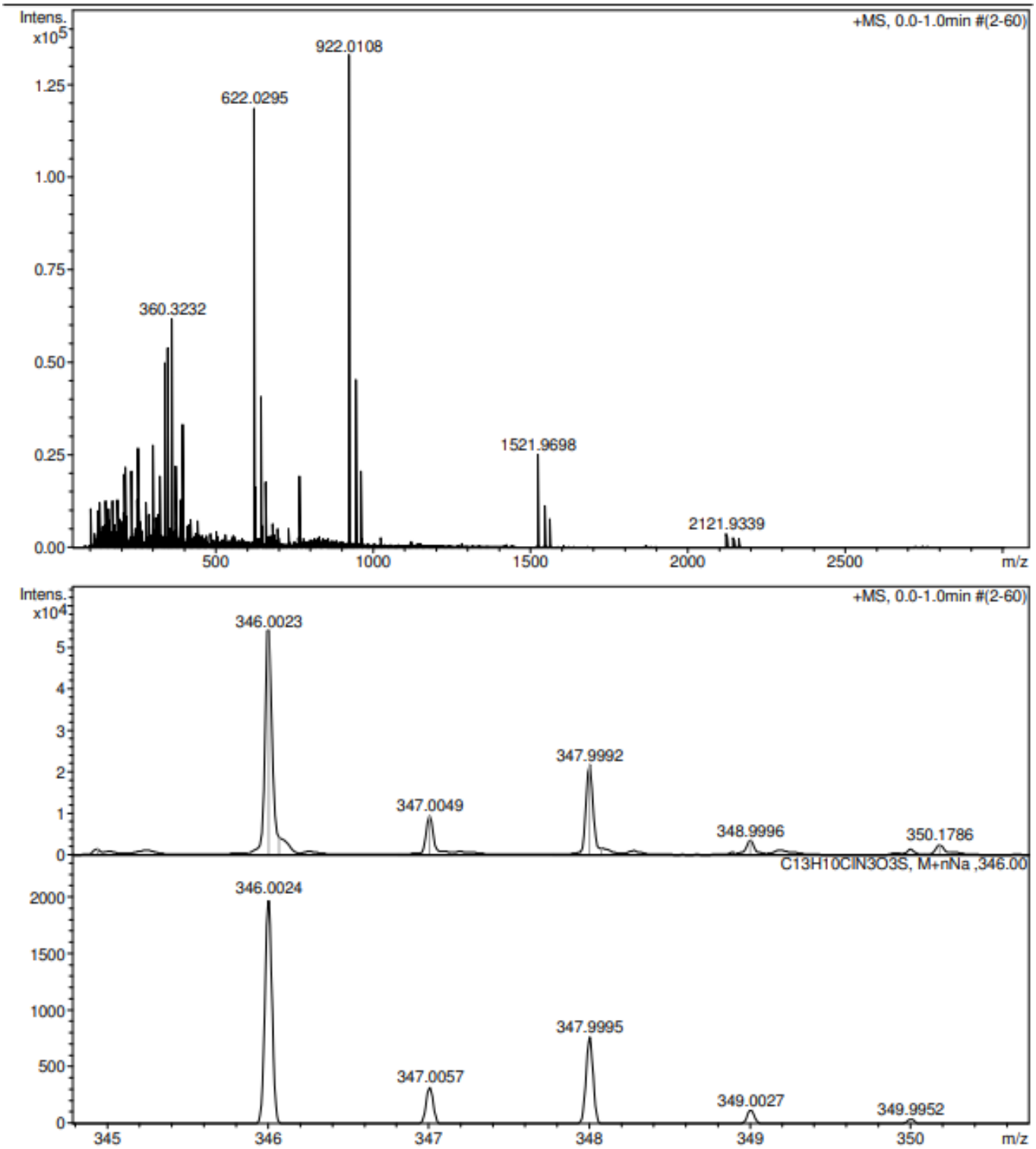
HRMS spectrum of 2o

Analysis Info

Analysis Name	D:\Data\Chizhov\Terentiev\Bityukov\ov2236_&clblow.d	Acquisition Date	14.05.2021 14:42:32
Method	tune_low.m	Operator	BDAL@DE
Sample Name	/TERN ov2236	Instrument / Ser#	micrOTOF 10248
Comment	CH3OH 100 %, dil. 20, calibrant added		

Acquisition Parameter

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	0.4 Bar
Focus	Not active			Set Dry Heater	180 °C
Scan Begin	50 m/z	Set Capillary	4500 V	Set Dry Gas	4.0 l/min
Scan End	3000 m/z	Set End Plate Offset	-500 V	Set Divert Valve	Waste



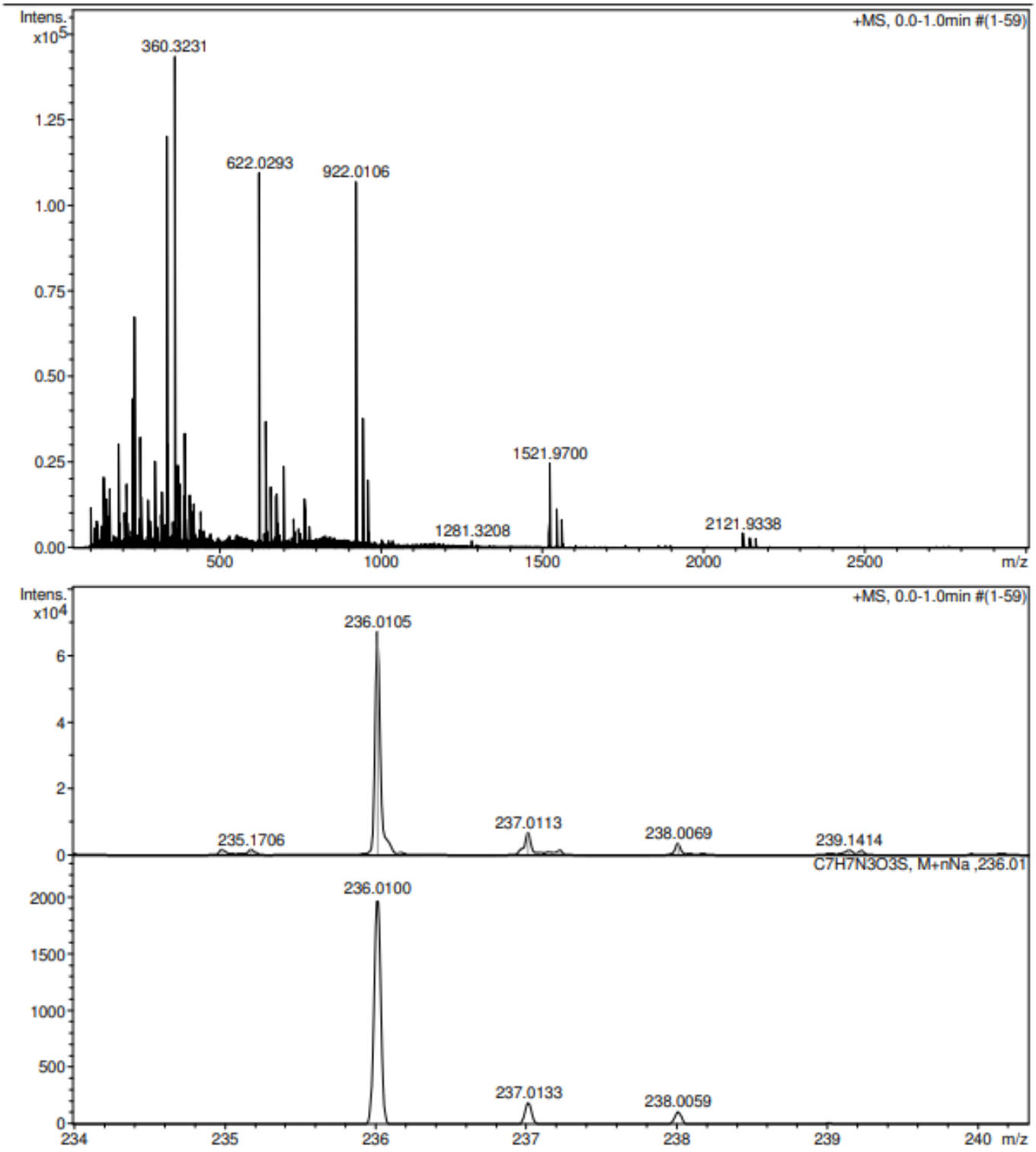
HRMS spectrum of 2p

Analysis Info

Analysis Name	D:\Data\Chizhov\Terentiev\Bityukov\ov2227_&clblow.d	Acquisition Date	14.05.2021 14:12:05
Method	tune_low.m	Operator	BDAL@DE
Sample Name	/TERN ov2227	Instrument / Ser#	micrOTOF 10248
Comment	CH3OH 100 %, dil. 200, calibrant added		

Acquisition Parameter

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	0.4 Bar
Focus	Not active			Set Dry Heater	180 °C
Scan Begin	50 m/z	Set Capillary	4500 V	Set Dry Gas	4.0 l/min
Scan End	3000 m/z	Set End Plate Offset	-500 V	Set Divert Valve	Waste



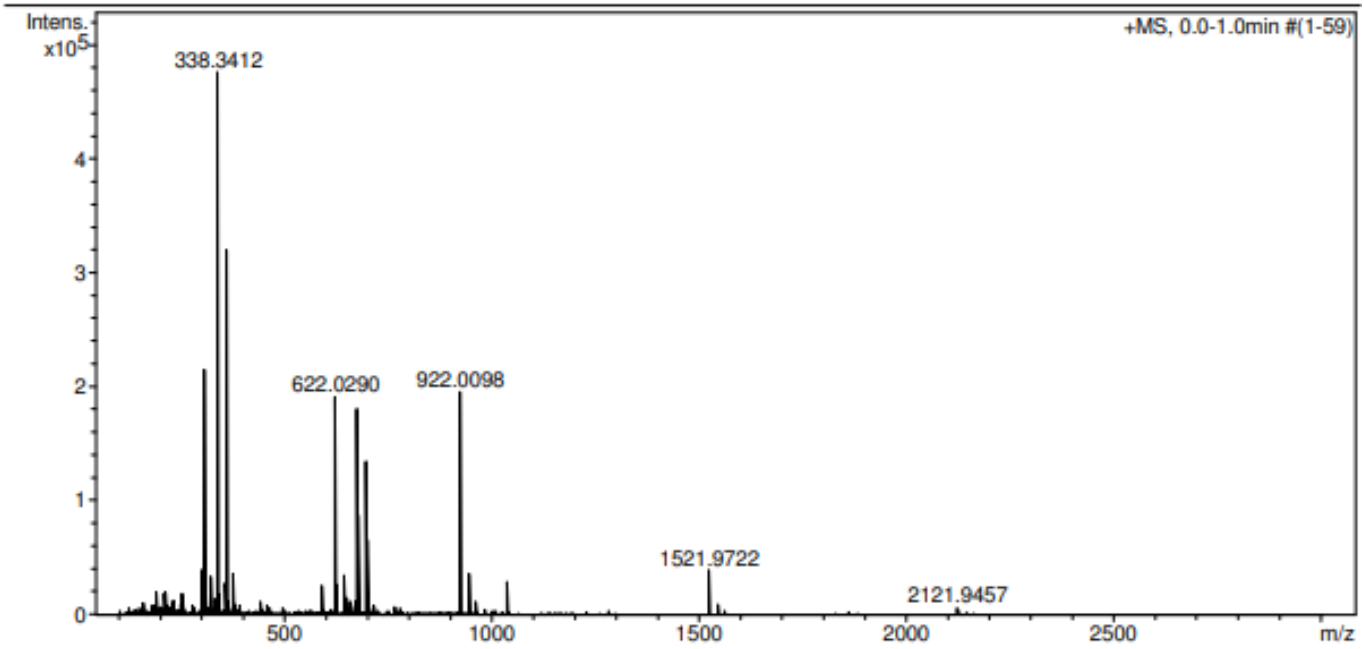
HRMS spectrum of 2q

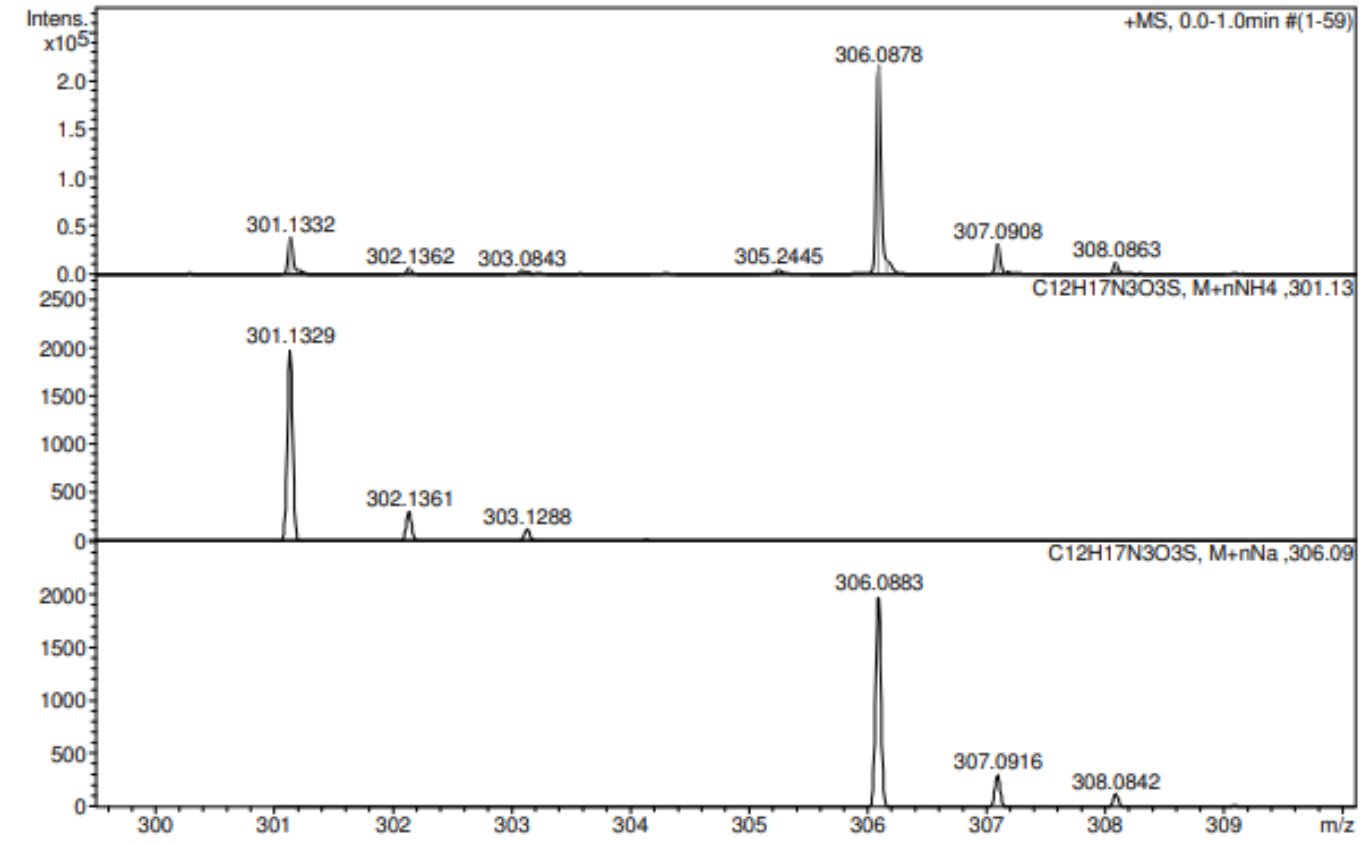
Analysis Info

Analysis Name	D:\Data\Chizhov\Terentiev\Bityukov\Jun_17_2021\ov2243_&clblow.d	Acquisition Date	17.06.2021 15:03:25
Method	tune_low.m	Operator	BDAL@DE
Sample Name	/TERN OV2243	Instrument / Ser#	micrOTOF 10248
Comment	CH3OH 100 %, dil. 2000, calibrant added		

Acquisition Parameter

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	0.4 Bar
Focus	Not active			Set Dry Heater	180 °C
Scan Begin	50 m/z	Set Capillary	4500 V	Set Dry Gas	4.0 l/min
Scan End	3000 m/z	Set End Plate Offset	-500 V	Set Divert Valve	Waste





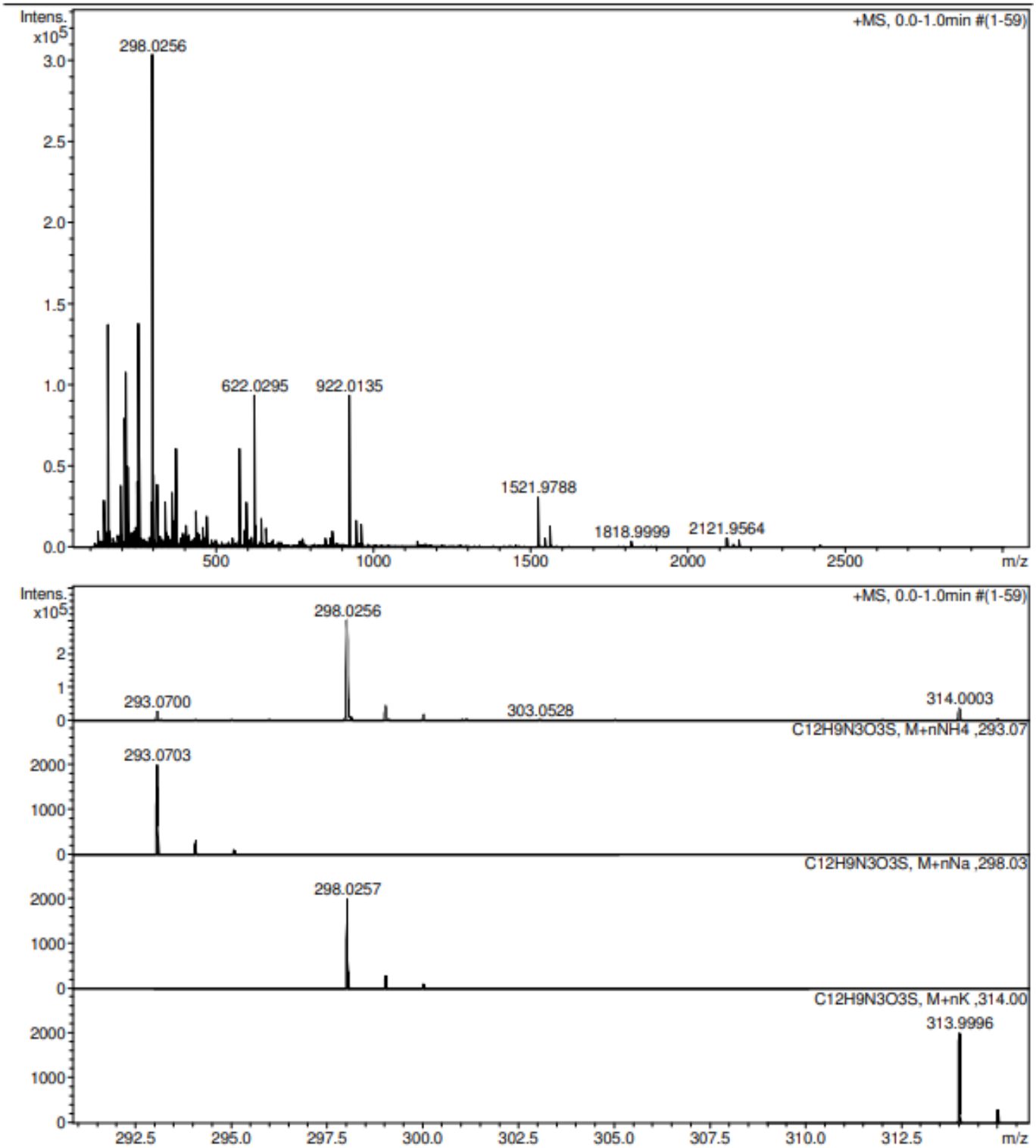
HRMS spectrum of 2r

Analysis Info

Analysis Name	D:\Data\Chizhov\Terentiev\Bityukov\ov2297_&clblow.d	Acquisition Date	17.01.2022 17:06:24
Method	tune_low.m	Operator	BDAL@DE
Sample Name	/TERN ov2297	Instrument / Ser#	micrOTOF 10248
Comment	CH3OH 100 %, dil. 200, calibrant added		

Acquisition Parameter

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	0.4 Bar
Focus	Not active			Set Dry Heater	180 °C
Scan Begin	50 m/z	Set Capillary	4500 V	Set Dry Gas	4.0 l/min
Scan End	3000 m/z	Set End Plate Offset	-500 V	Set Divert Valve	Waste



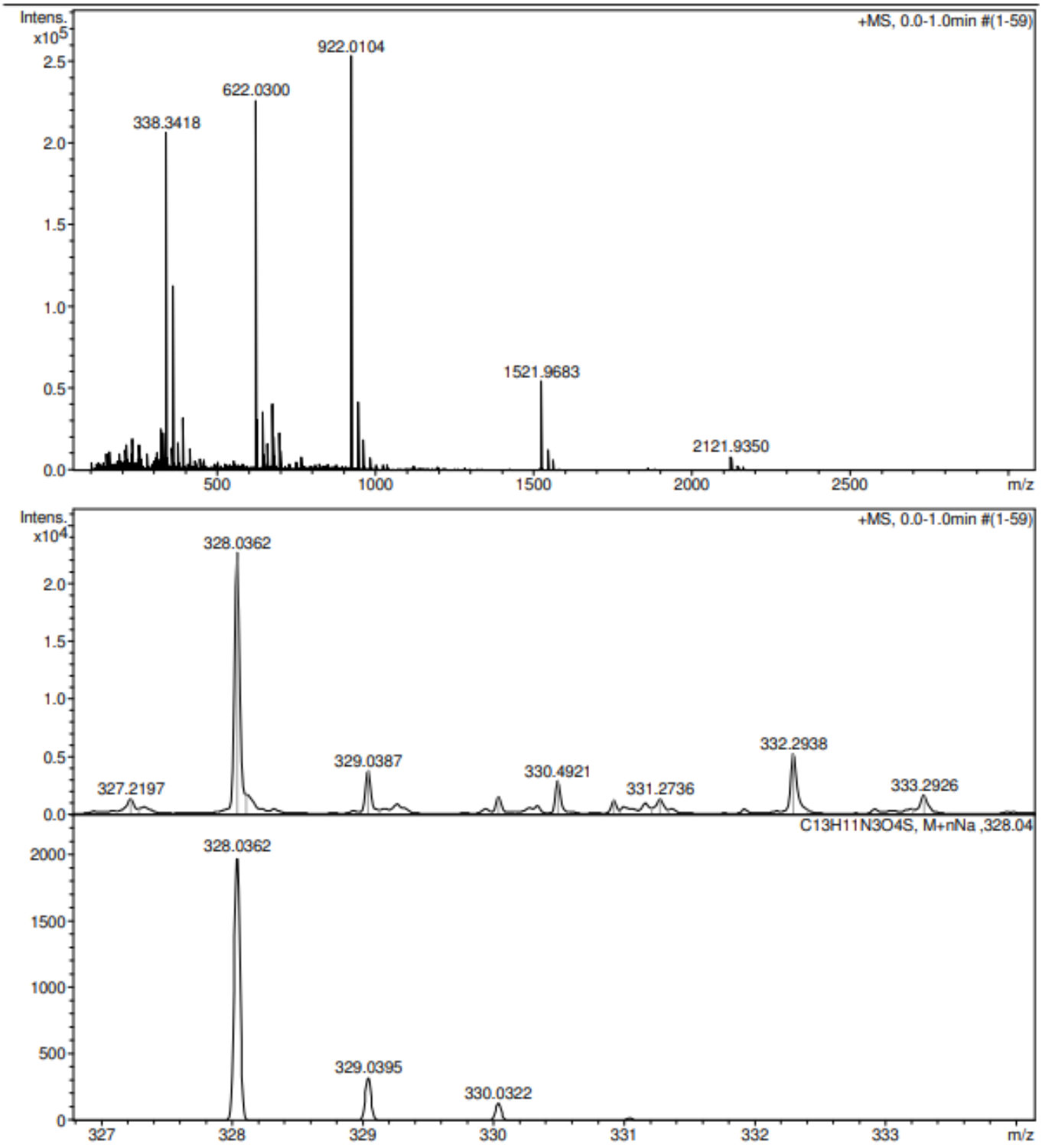
HRMS spectrum of 2s

Analysis Info

Analysis Name	D:\Data\Chizhov\Terentiev\Bityukov\Jun_17_2021\ov2225_&clblow.d	Acquisition Date	17.06.2021 14:59:17
Method	tune_low.m	Operator	BDAL@DE
Sample Name	/TERN OV2225	Instrument / Ser#	micrOTOF 10248
Comment	CH3OH 100 %, dil. 2000, calibrant added		

Acquisition Parameter

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	0.4 Bar
Focus	Not active			Set Dry Heater	180 °C
Scan Begin	50 m/z	Set Capillary	4500 V	Set Dry Gas	4.0 l/min
Scan End	3000 m/z	Set End Plate Offset	-500 V	Set Divert Valve	Waste



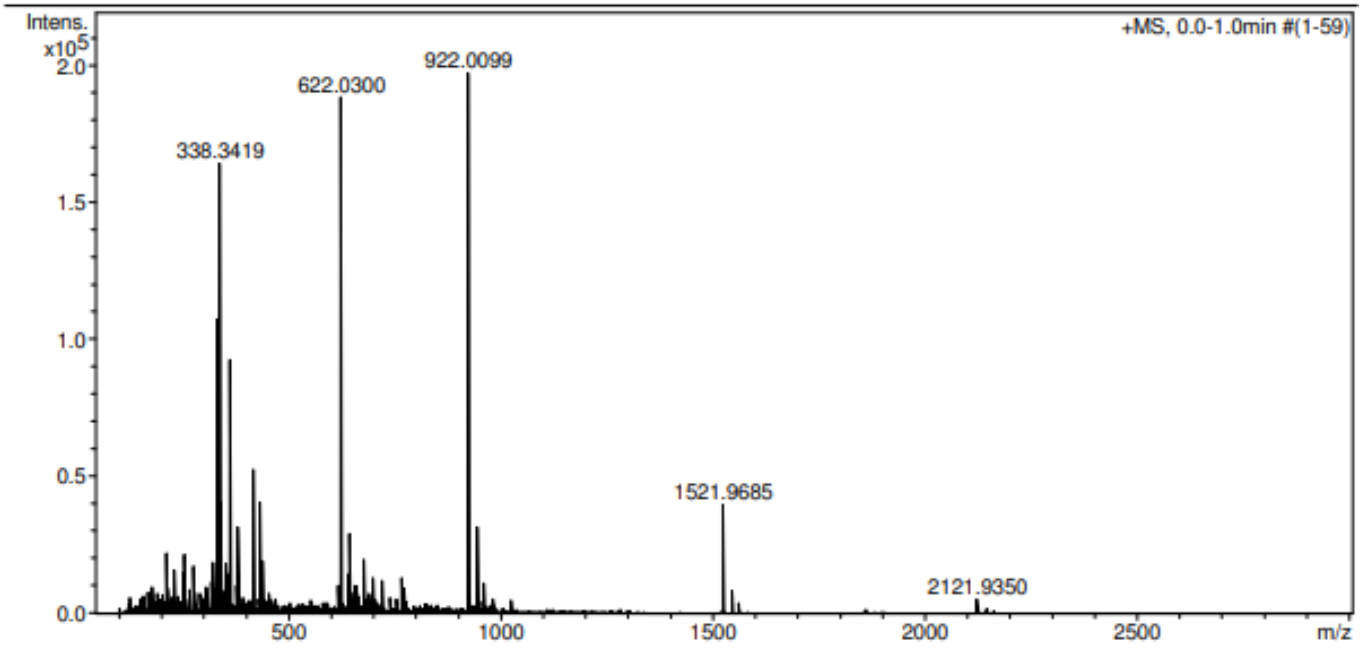
HRMS spectrum of 2t

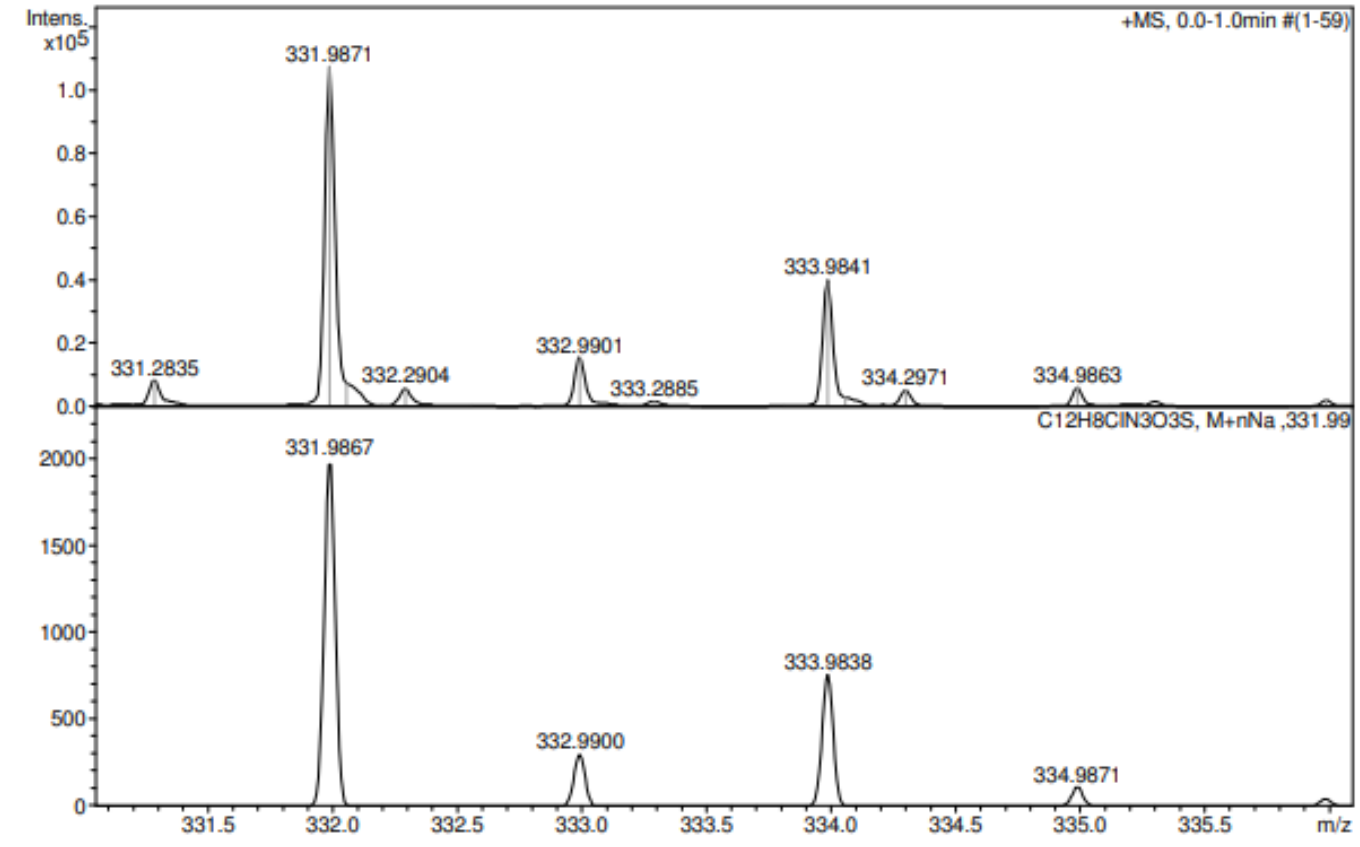
Analysis Info

Analysis Name	D:\Data\Chizhov\Terentiev\Bityukov\Jun_17_2021\pil283_&clblow.d	Acquisition Date	17.06.2021 15:24:12
Method	tune_low.m	Operator	BDAL@DE
Sample Name	/TERN Pil283	Instrument / Ser#	micrOTOF 10248
Comment	CH3OH 100 %, dil. 200, calibrant added		

Acquisition Parameter

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	0.4 Bar
Focus	Not active			Set Dry Heater	180 °C
Scan Begin	50 m/z	Set Capillary	4500 V	Set Dry Gas	4.0 l/min
Scan End	3000 m/z	Set End Plate Offset	-500 V	Set Divert Valve	Waste





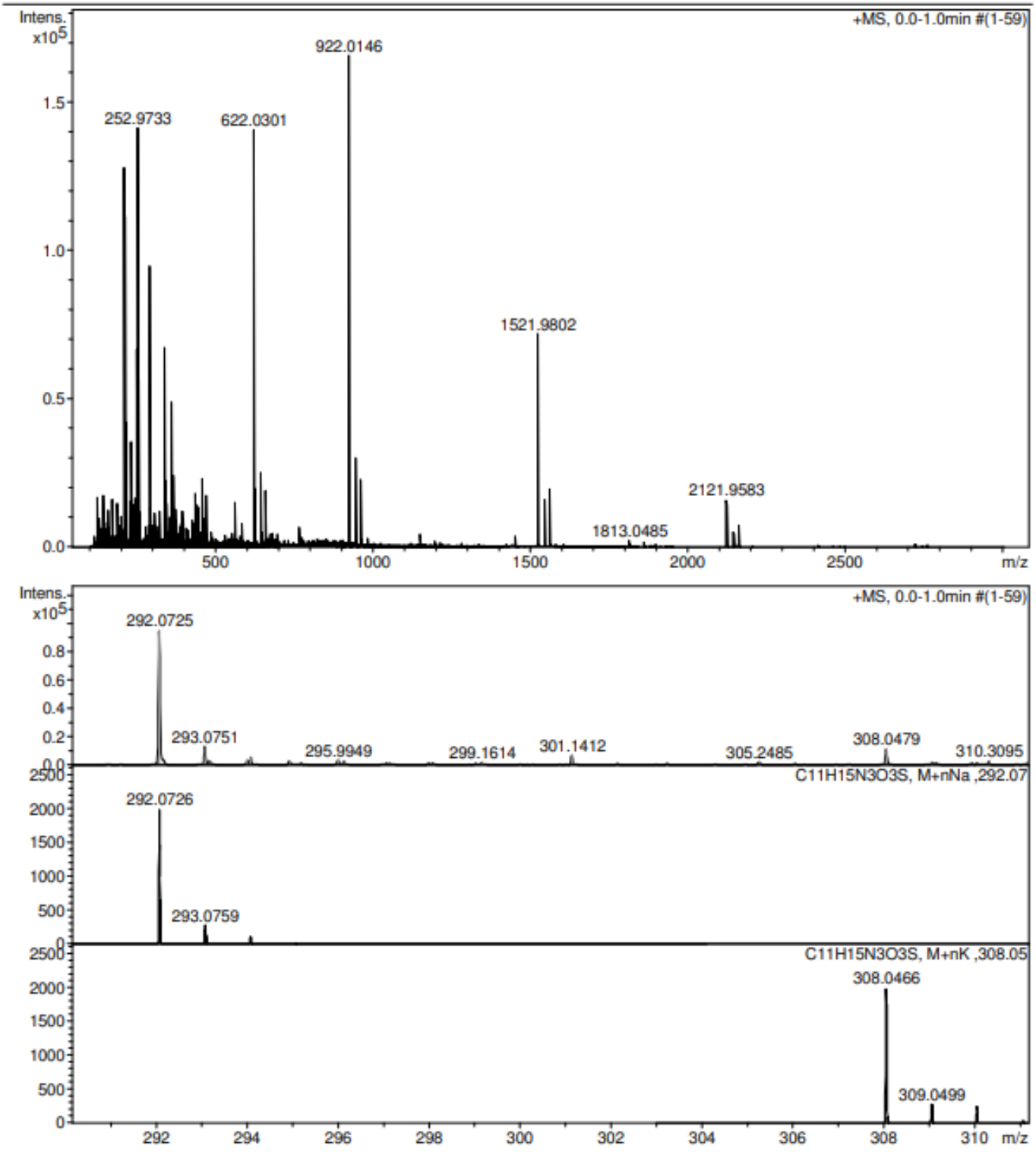
HRMS spectrum of 2u

Analysis Info

Analysis Name	D:\Data\Chizhov\Terentiev\Bityukov\pil286_&clblow.d	Acquisition Date	17.01.2022 17:01:16
Method	tune_low.m	Operator	BDAL@DE
Sample Name	/TERN pil286	Instrument / Ser#	micrOTOF 10248
Comment	CH3OH 100 %, dil. 200, calibrant added		

Acquisition Parameter

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	0.4 Bar
Focus	Not active			Set Dry Heater	180 °C
Scan Begin	50 m/z	Set Capillary	4500 V	Set Dry Gas	4.0 l/min
Scan End	3000 m/z	Set End Plate Offset	-500 V	Set Divert Valve	Waste



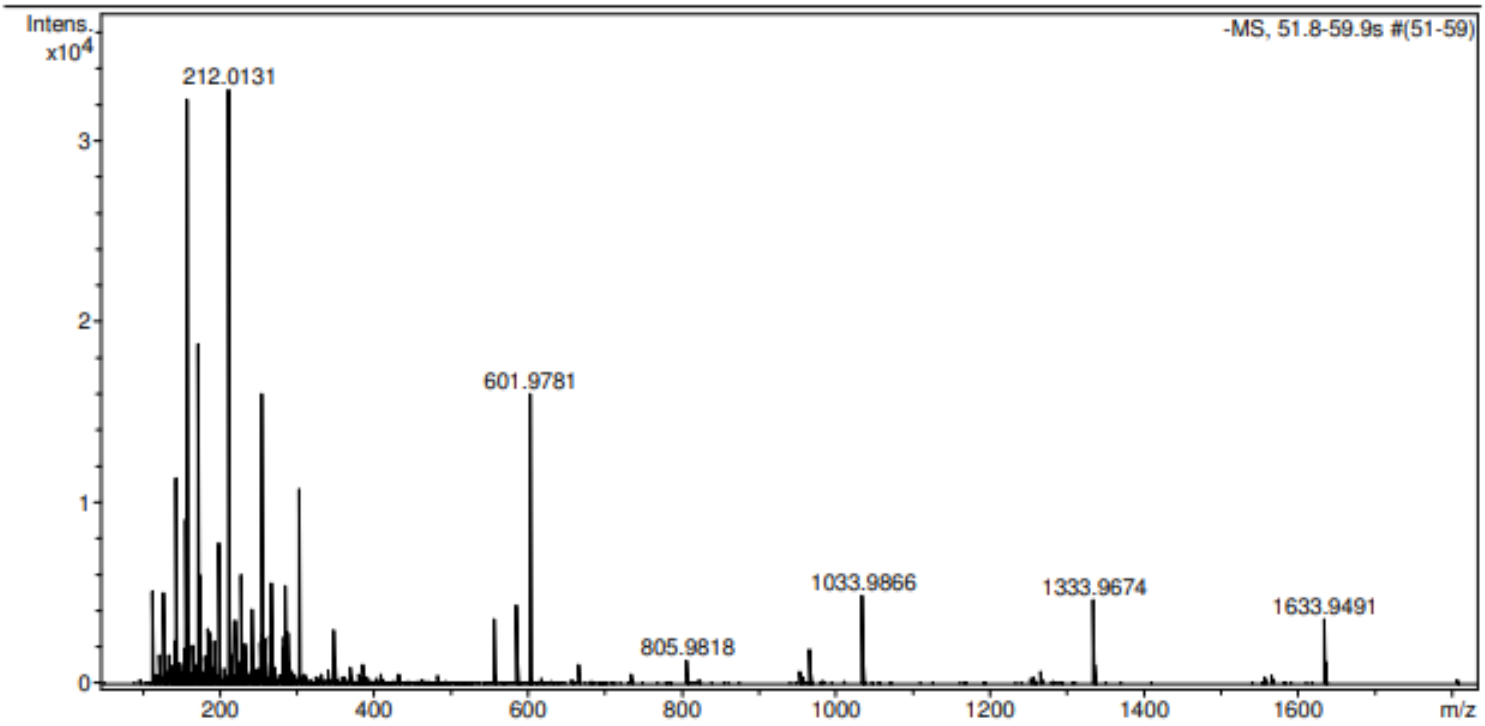
HRMS spectrum of 4a

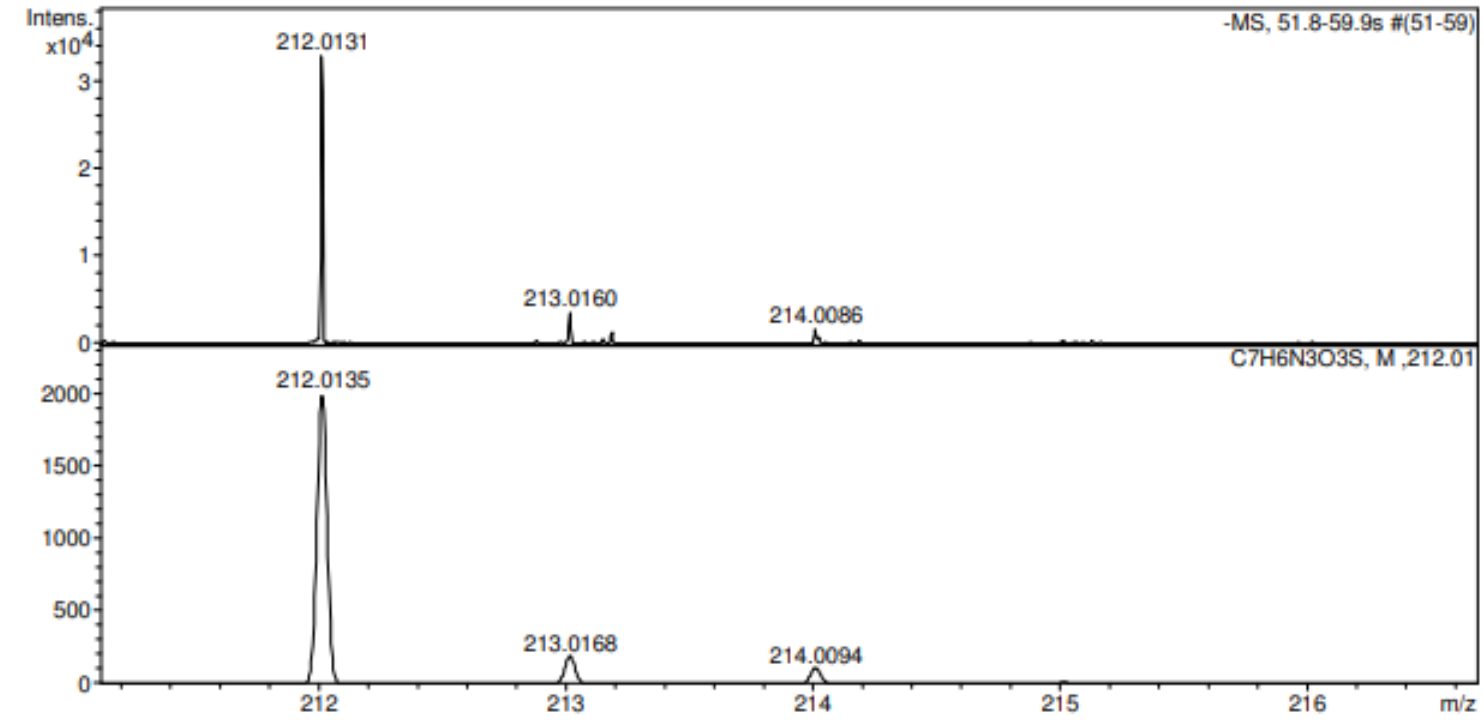
Analysis Info

Analysis Name	D:\Data\Burykina\OV2284negrep.d	Acquisition Date	20.01.2022 13:27:43
Method	tune_100-1200.m	Operator	BDAL@DE
Sample Name	OV2284	Instrument / Ser#	maXis 43
Comment	H2O 100%		

Acquisition Parameter

Source Type	ESI	Ion Polarity	Negative	Set Nebulizer	1.0 Bar
Focus	Active			Set Dry Heater	200 °C
Scan Begin	50 m/z	Set Capillary	4000 V	Set Dry Gas	4.0 l/min
Scan End	1800 m/z	Set End Plate Offset	-500 V	Set Divert Valve	Waste





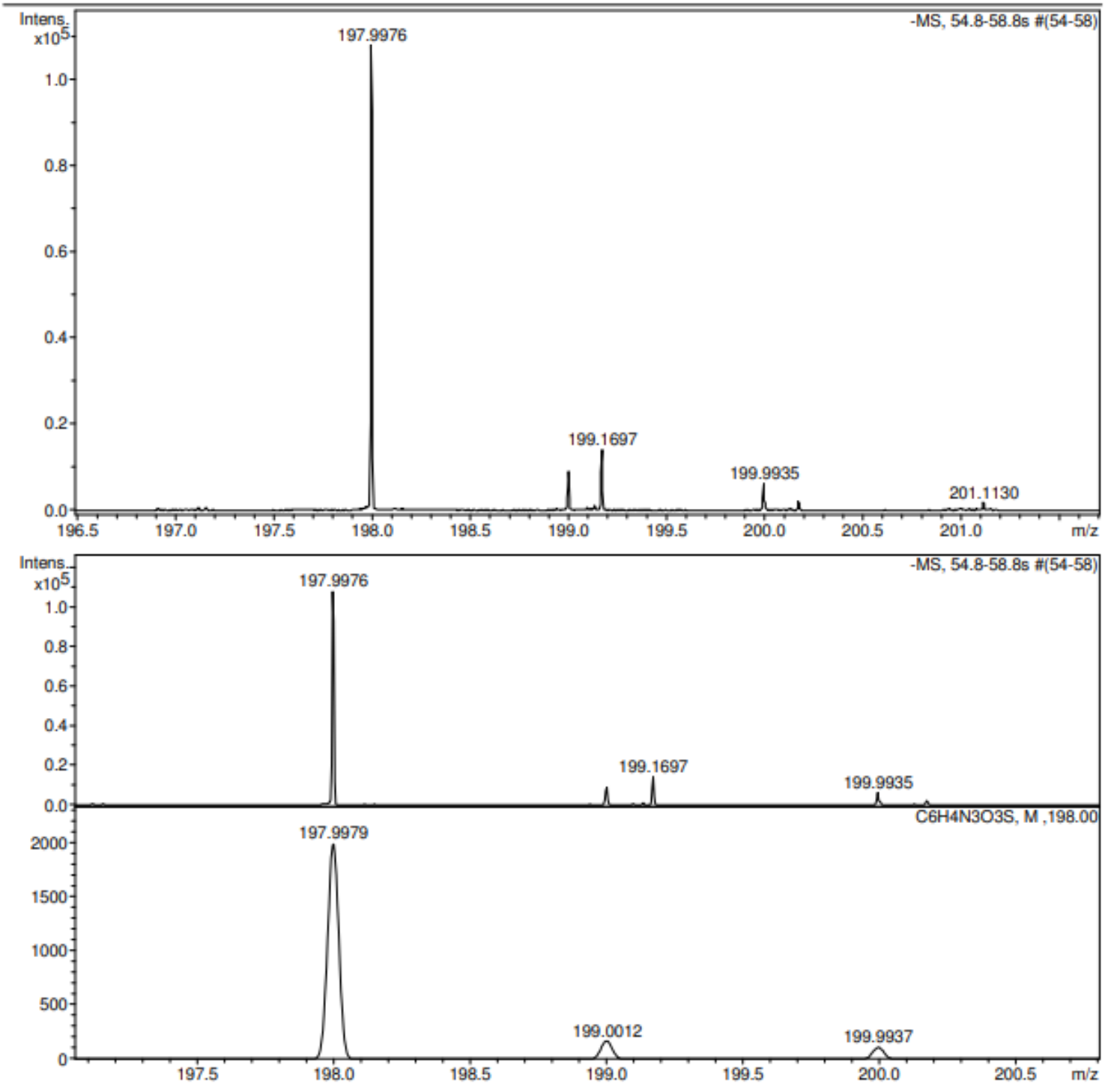
HRMS spectrum of 4b

Analysis Info

Analysis Name	D:\Data\Burykina\OV2295neg.d	Acquisition Date	20.01.2022 13:36:36
Method	tune_100-1200.m	Operator	BDAL@DE
Sample Name	/TERN OV2295	Instrument / Ser#	maXis 43
Comment	H2O 100%		

Acquisition Parameter

Source Type	ESI	Ion Polarity	Negative	Set Nebulizer	1.0 Bar
Focus	Active			Set Dry Heater	200 °C
Scan Begin	50 m/z	Set Capillary	4000 V	Set Dry Gas	4.0 l/min
Scan End	1800 m/z	Set End Plate Offset	-500 V	Set Divert Valve	Waste



Bruker Compass DataAnalysis 4.0 printed: 20.01.2022 13:41:47 Page 1 of 1

HRMS spectrum of 4c

Analysis Info

Analysis Name	D:\Data\Kolotyrkina\2022\Bitikov\0120010.d	Acquisition Date	20.01.2022 15:48:23
Method	tune_50-1600_neg.m	Operator	BDAL@DE
Sample Name	/TERN 2433	Instrument / Ser#	micrOTOF 10248
Comment	C5H2N3O3S m 182.9733 clb added H2O		

Acquisition Parameter

Source Type	ESI	Ion Polarity	Negative	Set Nebulizer	1.0 Bar
Focus	Not active			Set Dry Heater	200 °C
Scan Begin	50 m/z	Set Capillary	3200 V	Set Dry Gas	4.0 l/min
Scan End	1650 m/z	Set End Plate Offset	-500 V	Set Divert Valve	Waste