Supplementary Information (SI) for Green Chemistry.
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Supporting Information

Electrochemical cascade of Knoevenagel condensation and reduction: Green strategy for intermolecular C(sp^3)-C(sp^3) bond formation

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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). [1] 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 ALUGRAMR Xtra SIL G/UV254). Column chromatography was performed using silica gel (0.060-0.200 mm, 60 A, CAS 7631-86-9, Acros).

Ethanol (water contents 5%), THF, DMSO, HFIP, AcOH, Methanol, NH_4OAc, NH_4ClO_4, NH_4I, $(NH_4)_2CO_3$, NaOAc, NaBr, $LiClO_4$ were purchased from commercial sources and were used as is.

Starting ketones and nitriles were purchased from commercial sources and were used as is.

General Experimental Procedure for Table 1.

Cyclohexanone **1b** (2.0-4.0 mmol, 196.3-392.6 mg) and solvent (10 mL) were placed in a 20 mL IKA electrolysis cell without any precautions to exclude air or moisture. Ethyl cyanoacetate **2a** (2.0-4.0 mmol, 226.3-452.5 mg) was then added. The electrolyte (5.0-10.0 mmol) was weighed on a weighing cup and added to the solution at once, after which glassy carbon electrodes (8 × 18 mm) were immediately placed and a direct current was applied. The mixture was electrolyzed using constant current conditions at 30-70 ˚C under magnetic stirring. After completion of electrolysis, the reaction mixture was concentrated under reduced pressure using a rotary evaporator (15-20 mmHg), (bath temperature ~ 50 °C). EtOAc (25 mL) and H_2O (25 mL) was added to the reaction mixture. The organic phase was separated, washed with H_2O (25 mL), and later with saturated aq. solution NaCl (25 mL), dried over Na_2SO_4. The organic phase was filtered off, concentrated under reduced pressure using a rotary evaporator (15-20 mmHg), (bath temperature, about 30-40 °C). The resulting crude mixture was analysed by NMR using 1,4-dinitrobenzene as an internal standard.

Entry 2: the target product **3ab** was isolated by column chromatography on SiO_2 (PE:EtOAc = from 20:1 to 2:1).

Calculation of the amount of electric current (3ab synthesis, Table 1, entry 2)

$Q = I \cdot t$

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

I - electric current, A

t - time, sec

$Q = I \cdot t = 0.1 \cdot 128 \cdot 60 = \ 768\ C$

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

N - number of electrons generated in the cell per 1 molecule of carbonyl compound, F/mol

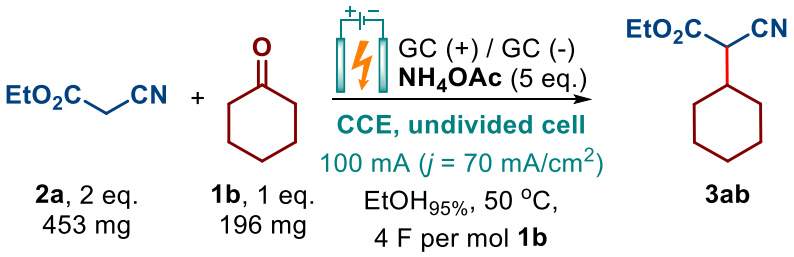
Q - amount of electricity passed, C (Coulomb)

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

n_r - amount of carbonyl compound, 2 mol

$$N = \frac{768}{96485 \cdot 0.002} = 3.98\ \frac{F}{mol} \approx 4\ \frac{F}{mol}$$

Calculation of E-factor values for the developed system:



E-factor = Kg (waste) / Kg (product)

Total amount of reactants: 453 mg + 196 mg + 771 mg (NH_4OAc) + 2.8 g $EtOH_{95\%}$ [8 g (initial) – 5.2 g (recycled)] = 4.22 g

Amount of final product: 270 mg

Amount of waste: 4.22 – 0.270 = 3.95 g

E-Factor = Amount of waste/Amount of product = 3.95 / 0.270 = 14.63

General procedure for recyclization of ethanol.

Note! To estimate the amount of ethanol which can be recycled these operations were run three times and after that mean value was taken into account of E-factor.

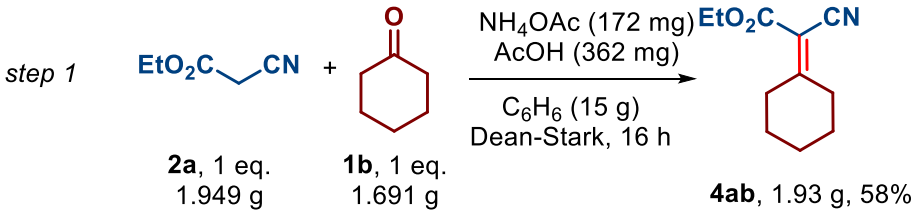
Cyclohexanone **1b** (2.0 mmol, 196.3 mg) and ethanol (5% water content by mass) (10 mL) were placed in a 20 mL IKA electrolysis cell without any precautions to exclude air or moisture. Ethyl cyanoacetate **2a** (4.0 mmol, 452.5 mg) was then added. The ammonium acetate (10.0 mmol) was weighed on a weighing cup and added to the solution at once, after which glassy carbon electrodes (8 × 18 mm) were immediately placed and a direct current was applied.

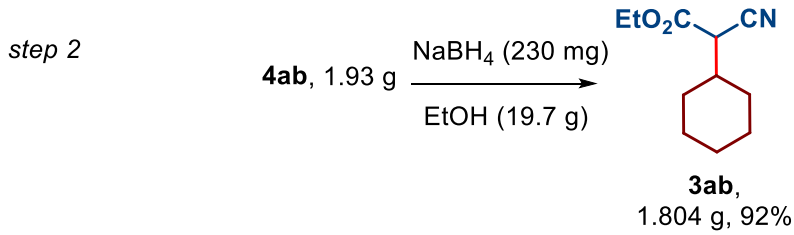
After completion of electrolysis, the reaction mixture was put into Claisen flask and ethanol was distilled off under reduced pressure with bath temperature ~ 50 °C until temperature of vapours starts to decrease (33-35 °C fraction). The average mass of the obtained ethanol was 5.2 g. The reside organic mixture was diluted with EtOAc (25 mL) and H_2O (25 mL). The organic phase was separated, washed with H_2O (25 mL), and later with saturated aq. solution NaCl (25 mL), dried over Na_2SO_4. The organic phase was filtered off, concentrated under reduced pressure using a rotary evaporator (15-20 mmHg), (bath temperature, about 30-40 °C). The target product **3ab** was isolated by column chromatography on SiO_2 (PE:EtOAc = from 20:1 to 2:1), 270 mg (69%) of ethyl 2-cyano-2-cyclohexylacetate **3ab** was obtained.

A mixture of recycled ethanol (5.2 g, 6.5 ml) and ethanol from a commercial source (2.8 g, 3.5 ml) could be used as a solvent in further experiment.

Calculation of E-factor values for the known two-step protocol:

[*J. Am. Chem. Soc.* 2020, 142, 25, 10914–10920]





E-factor = Kg (waste) / Kg (product)

Total amount of reactants: 1.949 g + 1.691 g + 0.172 g + 0.362 g + 15 g + 0.23 g + 19.7 g = 39.104 g

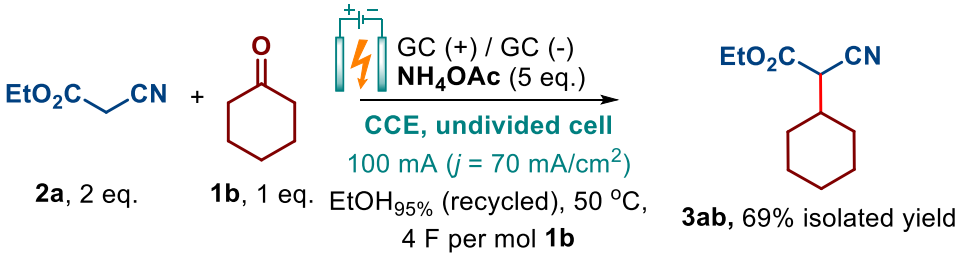
Amount of final product: 1.804 g

Amount of waste: 39.104 g – 1.804 g = 37.3 g

E-Factor = Amount of waste/Amount of product = 37.3 / 1.804 = 20.7

Calculation of Faradaic efficiency

(the current yield of product) for the developed system on example **3ab** synthesis:



FE (%) = [Q (prod) / Q (total)] * Chemical yield(%)

FE (%) = [2 / 4] * 69% = 34.5 %

General Experimental Procedure for Table 2

Cyclohexanone **1b** (2.0 mmol, 196.3 mg) and EtOH (5% water content by mass) (10 mL) were placed in a 20 mL IKA electrolysis cell without any precautions to exclude air or moisture. Ethyl cyanoacetate **2a** (4.0 mmol, 452.5 mg) was then added. NH_4OAc (10.0 mmol) was weighed on a weighing cup and added to the solution at once, after which IKA cap fitted with anode (8 × 18 mm) and cathode (8 × 18 mm) were immediately placed and a direct current was applied. The mixture was electrolyzed using constant current conditions at 50 ˚C under magnetic stirring for 128 min. with I = 100 mA (j = 70 mA/cm^2). After completion of electrolysis, the reaction mixture was concentrated under reduced pressure using a rotary evaporator (15-20 mmHg), (bath temperature ~ 50 °C). EtOAc (25 mL) and H_2O (25 mL) was added to the reaction mixture. The organic phase was separated, washed with H_2O (25 mL), and later with saturated aq. solution NaCl (25 mL), dried over Na_2SO_4. The organic phase was filtered off, concentrated under reduced pressure using a rotary evaporator (15-20 mmHg), (bath temperature, about 30-40 °C). The resulting crude mixture was analysed by NMR using 1,4-dinitrobenzene as an internal standard.

In the case of entry 6, after completion of electrolysis, the reaction mixture was concentrated under reduced pressure using a rotary evaporator (15-20 mmHg), (bath temperature ~ 50 °C). EtOAc (25 mL) and 1N HCl (25 mL) was added to the reaction mixture. The organic phase was separated, washed with H_2O (25 mL), and later with saturated aq. solution NaCl (25 mL), dried over Na_2SO_4. The organic phase was filtered off, concentrated under reduced pressure using a rotary evaporator (15-20 mmHg), (bath temperature, about 30-40 °C). The resulting crude mixture was analysed by NMR using 1,4-dinitrobenzene as an internal standard.

Experimental procedure for Scheme 4a

Ethyl 2-cyano-2-cyclohexylideneacetate **4ab** (2.0 mmol, 386.6 mg) and EtOH (5% water content by mass) (10 mL) were placed in a 20 mL IKA electrolysis cell without any precautions to exclude air or moisture. NH_4OAc (10.0 mmol) was weighed on a weighing cup and added to the solution at once, after which electrodes (8 × 18 mm) were immediately placed and a direct current was applied. The mixture was electrolyzed using constant current conditions at 50 ˚C under magnetic stirring for 128 min. with I = 100 mA (j = 70 mA/cm^2). After completion of electrolysis, the reaction mixture was concentrated under reduced pressure using a rotary evaporator (15-20 mmHg), (bath temperature ~ 50 °C). EtOAc (25 mL) and H_2O (25 mL) was added to the reaction mixture. The organic phase was separated, washed with H_2O (25 mL), and later with saturated aq. solution NaCl (25

mL), dried over Na_2SO_4. The organic phase was filtered off, concentrated under reduced pressure using a rotary evaporator (15-20 mmHg), (bath temperature, about 30-40 °C). Products were isolated by column chromatography on SiO_2 (PE:EtOAc = from 20:1 to 2:1).

Experimental procedures for Scheme 4b.

The first two experiments:

Cyclohexanone **1b** (2.0 mmol, 196.3 mg) and EtOH (5% water content by mass) (10 mL) were placed in a 20 mL IKA electrolysis cell without any precautions to exclude air or moisture. Ethyl cyanoacetate **2a** (4.0 mmol, 452.5 mg) and ethyl 2-cyano-2-cyclohexylideneacetate **4ab** (0–2 mmol, 0–386.6 mg) was then added. NH_4OAc (10.0 mmol) was weighed on a weighing cup and added to the solution at once, after which IKA cap fitted with glassy carbon electrodes (8 × 18 mm) were immediately placed and a direct current was applied. The mixture was electrolyzed using constant current conditions at 50 °C under magnetic stirring for 128 min. with I = 100 mA (j = 70 mA/cm^2). After completion of electrolysis, the reaction mixture was concentrated under reduced pressure using a rotary evaporator (15-20 mmHg), (bath temperature ~ 50 °C). EtOAc (25 mL) and H_2O (25 mL) was added to the reaction mixture. The organic phase was separated, washed with H_2O (25 mL), and later with saturated aq. solution NaCl (25 mL), dried over Na_2SO_4. The organic phase was filtered off, concentrated under reduced pressure using a rotary evaporator (15-20 mmHg), (bath temperature, about 30-40 °C). The resulting crude mixture was analysed by NMR using 1,4-dinitrobenzene as an internal standard.

The third experiment:

Ethyl 2-cyano-2-cyclohexylideneacetate **4ab** (2.0 mmol, 386.6 mg) and EtOH (5% water content by mass) (10 mL) were placed in a 20 mL IKA electrolysis cell without any precautions to exclude air or moisture. NH_4OAc (10.0 mmol) was weighed on a weighing cup and added to the solution at once, after which glassy carbon electrodes (8 × 18 mm) were immediately placed and a direct current was applied. The mixture was electrolyzed using constant current conditions at 50 °C under magnetic stirring for 128 min. with I = 100 mA (j = 70 mA/cm^2). After completion of electrolysis, the reaction mixture was concentrated under reduced pressure using a rotary evaporator (15-20 mmHg), (bath temperature ~ 50 °C). EtOAc (25 mL) and H_2O (25 mL) was added to the reaction mixture. The organic phase was separated, washed with H_2O (25 mL), and later with saturated aq. solution NaCl (25 mL), dried over Na_2SO_4. The organic phase was filtered off, concentrated under reduced pressure using a rotary evaporator (15-20 mmHg), (bath temperature, about 30-

40 °C). The resulting crude mixture was analysed by NMR using 1,4-dinitrobenzene as an internal standard.

Experimental procedures for Scheme 5a.

Cathode compartment experiment:

A divided cell was equipped with a glassy-carbon plate anode (8 mm×18 mm) and a glassy-carbon plate cathode (8 mm×18 mm) and connected to a DC regulated power supply. Ethyl 2-cyano-2-cyclohexylideneacetate **4ab** (1.0 mmol, 193.3 mg) and EtOH (5% water content by mass) (5 mL) were placed in a cathode compartment of the H-type divided electrochemical cell without any precautions to exclude air or moisture. NH_4OAc was weighed on a weighing cup and added to the solutions of cathode and anode compartments (5.0 mmol each), after which electrodes were immediately placed and a direct current was applied. Both compartments were electrolyzed using constant current conditions I = 20 mA at 50 °C under magnetic stirring (4 F/mol **4ab**).

After completion of electrolysis, the reaction mixture from cathode compartment was concentrated under reduced pressure using a rotary evaporator (15-20 mmHg), (bath temperature ~ 50 °C). EtOAc (25 mL) and H_2O (25 mL) was added to the reaction mixture. The organic phase was separated, washed with H_2O (25 mL), and later with saturated aq. solution NaCl (25 mL), dried over Na_2SO_4. The organic phase was filtered off, concentrated under reduced pressure using a rotary evaporator (15-20 mmHg), (bath temperature, about 30-40 °C). The resulting crude mixture was analysed by NMR using 1,4-dinitrobenzene as an internal standard.

Anode compartment experiment:

A divided cell was equipped with a glassy-carbon plate anode (8 mm×18 mm) and a glassy-carbon plate cathode (8 mm×18 mm) and connected to a DC regulated power supply. Ethyl 2-cyano-2-cyclohexylideneacetate **4ab** (1.0 mmol, 193.3 mg) and EtOH (5% water content by mass) (5 mL) were placed in anode compartment of the H-type divided electrochemical cell without any precautions to exclude air or moisture. NH_4OAc was weighed on a weighing cup and added to the solutions of cathode and anode compartments (5.0 mmol each), after which electrodes were immediately placed and a direct current was applied. Both compartments were electrolyzed using constant current conditions I = 20 mA at 50 °C under magnetic stirring (4 F/mol **4ab**).

After completion of electrolysis, the reaction mixture from anode compartment was concentrated under reduced pressure using a rotary evaporator (15-20 mmHg), (bath temperature ~ 50 °C). EtOAc (25 mL) and H_2O (25 mL) was added to the reaction mixture.

The organic phase was separated, washed with H_2O (25 mL), and later with saturated aq. solution NaCl (25 mL), dried over Na_2SO_4. The organic phase was filtered off, concentrated under reduced pressure using a rotary evaporator (15-20 mmHg), (bath temperature, about 30-40 °C). The resulting crude mixture was analysed by NMR using 1,4-dinitrobenzene as an internal standard.

Experimental procedures for Scheme 5b.

Cathode compartment experiment:

A divided cell was equipped with a glassy-carbon plate anode (8 mm×18 mm) and a glassy-carbon plate cathode (8 mm×18 mm) and connected to a DC regulated power supply. Cyclohexanone **1b** (1.0 mmol, 98.15 mg) and EtOH (5% water content by mass) (5 mL) were placed in a cathode compartment of the H-type divided electrochemical cell without any precautions to exclude air or moisture. Ethyl cyanoacetate **2a** (2.0 mmol, 226.23 mg) was then added. NH_4OAc was weighed on a weighing cup and added to the solutions of cathode and anode compartments (5.0 mmol each), after which electrodes were immediately placed and a direct current was applied. Both compartments were electrolyzed using constant current conditions I = 20 mA at 50 °C under magnetic stirring (4 F/mol **1b**).

After completion of electrolysis, the reaction mixture from cathode compartment was concentrated under reduced pressure using a rotary evaporator (15-20 mmHg), (bath temperature ~ 50 °C). EtOAc (25 mL) and H_2O (25 mL) was added to the reaction mixture. The organic phase was separated, washed with H_2O (25 mL), and later with saturated aq. solution NaCl (25 mL), dried over Na_2SO_4. The organic phase was filtered off, concentrated under reduced pressure using a rotary evaporator (15-20 mmHg), (bath temperature, about 30-40 °C). The resulting crude mixture was analysed by NMR using 1,4-dinitrobenzene as an internal standard.

Anode compartment experiment:

A divided cell was equipped with a glassy-carbon plate anode (8 mm×18 mm) and a glassy-carbon plate cathode (8 mm×18 mm) and connected to a DC regulated power supply. Cyclohexanone **1b** (1.0 mmol, 98.15 mg) and EtOH (5% water content by mass) (5 mL) were placed in an anode compartment of the H-type divided electrochemical cell without any precautions to exclude air or moisture. Ethyl cyanoacetate **2a** (2.0 mmol, 226.23 mg) was then added. NH_4OAc was weighed on a weighing cup and added to the solutions of cathode and anode compartments (5.0 mmol each), after which electrodes

were immediately placed and a direct current was applied. Both compartments were electrolyzed using constant current conditions I = 20 mA at 50 °C under magnetic stirring (4 F/mol **1b**).

After completion of electrolysis, the reaction mixture from anode compartment was concentrated under reduced pressure using a rotary evaporator (15-20 mmHg), (bath temperature ~ 50 °C). EtOAc (25 mL) and H_2O (25 mL) was added to the reaction mixture. The organic phase was separated, washed with H_2O (25 mL), and later with saturated aq. solution NaCl (25 mL), dried over Na_2SO_4. The organic phase was filtered off, concentrated under reduced pressure using a rotary evaporator (15-20 mmHg), (bath temperature, about 30-40 °C). The resulting crude mixture was analysed by NMR using 1,4-dinitrobenzene as an internal standard.

Experimental procedure for Scheme 5c.

Ethyl 2-cyano-2-cyclohexylacetate **3ab** (2.0 mmol, 390.5 mg) and EtOH (5% water content by mass) (10 mL) were placed in a 20 mL IKA electrolysis cell without any precautions to exclude air or moisture. NH_4OAc (10.0 mmol) was weighed on a weighing cup and added to the solution at once, after which glassy carbon electrodes (8 × 18 mm) were immediately placed and a direct current was applied. The mixture was electrolyzed using constant current conditions at 50 °C under magnetic stirring for 128 min. with I = 100 mA (j = 70 mA/cm^2). After completion of electrolysis, the reaction mixture was concentrated under reduced pressure using a rotary evaporator (15-20 mmHg), (bath temperature ~ 50 °C). EtOAc (25 mL) and H_2O (25 mL) was added to the reaction mixture. The organic phase was separated, washed with H_2O (25 mL), and later with saturated aq. solution NaCl (25 mL), dried over Na_2SO_4. The organic phase was filtered off, concentrated under reduced pressure using a rotary evaporator (15-20 mmHg), (bath temperature, about 30-40 °C). The resulting crude mixture was analysed by NMR using 1,4-dinitrobenzene as an internal standard.

General Experimental Procedure for Scheme 6.

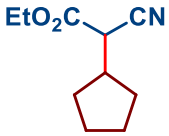
Note! *The target molecules are not visible under UV light; therefore, the thin-layer chromatography (TLC) plate was developed by staining with iodine (I_2) vapors in an iodine chamber at room temperature.*

Ketone **1a-o** (2.0 mmol) and EtOH (5% water content by mass) (10 mL) were placed in a 20 mL IKA electrolysis cell without any precautions to exclude air or moisture. Active methylene compound **2a-e** (4.0 mmol) was then added. NH_4OAc (10.0 mmol) was

weighed on a weighing cup and added to the solution at once, after which glassy carbon electrodes (8 × 18 mm) were immediately placed and a direct current was applied. The mixture was electrolyzed using constant current conditions at 50 ˚C under magnetic stirring for 128 min. with I = 100 mA (*j* = 70 mA/cm^2). After completion of electrolysis, the reaction mixture was concentrated under reduced pressure using a rotary evaporator (15-20 mmHg), (bath temperature ~ 50 °C). EtOAc (25 mL) and H_2O (25 mL) was added to the reaction mixture. The organic phase was separated, washed with H_2O (25 mL), and later with saturated aq. solution NaCl (25 mL), dried over Na_2SO_4. The organic phase was filtered off, concentrated under reduced pressure using a rotary evaporator (15-20 mmHg), (bath temperature, about 30-40 °C). The target product was isolated by column chromatography on SiO_2 (PE:EtOAc = from 20:1 to 2:1).

Characterization of the synthesized products 3

Ethyl 2-cyano-2-cyclopentylacetate, 3aa (known compound [2])

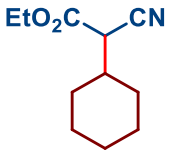


Yield 64% (232.0 mg, 1.28 mmol). Colorless oil. R_f = 0.55 (PE:EtOAc = 10:1).

^{1}H NMR (300.13 MHz, $CDCl_3$, δ): 4.24 (q, *J* = 7.1 Hz, 2H), 3.48 (d, *J* = 6.7 Hz, 1H), 2.48 (m, 1H), 1.97 – 1.79 (m, 2H), 1.78 – 1.66 (m, 2H), 1.65 – 1.54 (m, 2H), 1.52 – 1.36 (m, 2H), 1.30 (t, *J* = 7.1 Hz, 3H).

^{13}C{^{1}H} NMR (75.48 MHz, $CDCl_3$, δ): 166.20, 116.18, 62.66, 42.69, 40.31, 30.83, 29.89, 25.17, 14.12.

Ethyl 2-cyano-2-cyclohexylacetate, 3ab (known compound [2])

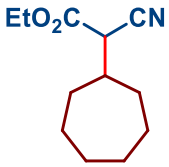


Yield 69% (269.5 mg, 1.38 mmol). Colorless oil. R_f = 0.39 (PE:EtOAc = 10:1).

^{1}H NMR (300.13 MHz, $CDCl_3$, δ): 4.25 (q, *J* = 7.1 Hz, 2H), 3.36 (d, *J* = 5.7 Hz, 1H), 2.14 – 1.93 (m, 1H), 1.87 – 1.60 (m, 5H), 1.38 – 1.09 (m, 8H).

^{13}C{^{1}H} NMR (75.48 MHz, $CDCl_3$, δ): 165.96,115.81, 62.66, 44.68, 38.93, 31.15, 29.48, 25.89, 25.69, 25.57, 14.16.

Ethyl 2-cyano-2-cycloheptylacetate, 3ac (known compound [2])

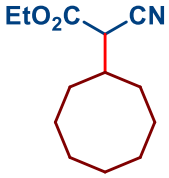


Yield 46% (192.5 mg, 0.92 mmol). Colorless oil. R_f = 0.33 (PE:EtOAc = 10:1).

^{1}H NMR (300.13 MHz, $CDCl_3$, δ): 4.25 (q, *J* = 7.1 Hz, 2H), 3.41 (d, *J* = 5.3 Hz, 1H), 2.31 – 2.17 (m, 1H), 1.83 – 1.42 (m, 12H), 1.31 (t, *J* = 7.1 Hz, 3H).

^{13}C{^{1}H} NMR (75.48 MHz, $CDCl_3$, δ): 166.14, 116.16, 62.69, 45.36, 40.62, 33.14, 31.04, 27.88, 27.66, 26.32, 26.15, 14.16.

Ethyl 2-cyano-2-cyclooctylacetate, 3ad



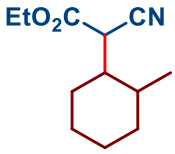
Yield 40% (178.7 mg, 0.80 mmol). Colorless oil. R_f = 0.37 (PE:EtOAc = 10:1).

^{1}H NMR (300.13 MHz, $CDCl_3$, δ): 4.26 (q, *J* = 7.1 Hz, 2H), 3.38 (d, *J* = 5.5 Hz, 1H), 2.43 – 2.25 (m, 1H), 1.80 – 1.43 (m, 14H), 1.31 (t, *J* = 7.1 Hz, 3H).

^{13}C{^{1}H} NMR (75.48 MHz, $CDCl_3$, δ): 166.22, 116.27, 62.69, 45.82, 38.64, 31.90, 29.68, 26.54, 26.37, 26.26, 25.69, 25.14, 14.18.

HRMS (ESI-TOF) m/z $[M+NH_4]^+$. Calcd for $[C_{13}H_{25}N_2O_2]^+$: 241.1911. Found: 241.1915.

Ethyl 2-cyano-2-(2-methylcyclohexyl)acetate, 3ae (known compound [2])

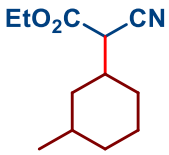


Yield 36% (mixture of diastereomers, 150.7 mg, 0.72 mmol). Colorless oil. R_f = 0.48 (PE:EtOAc = 10:1).

^{1}H NMR (300.13 MHz, $CDCl_3$, δ): 4.31 – 4.19 (m, 2H), 3.50 – 3.17 (m, 1H), 2.40 – 1.24 (m, 13H), 0.99 – 0.85 (m, 3H).

^{13}C{^{1}H} NMR (75.48 MHz, $CDCl_3$, δ): 166.12, 166.09, 166.07, 116.08, 115.81, 62.68, 62.66, 62.64, 44.65, 44.62, 44.48, 43.28, 43.21, 42.96, 39.62, 38.79, 38.66, 37.94, 37.85, 36.59, 35.64, 34.49, 34.28, 34.23, 34.21, 32.49, 32.28, 31.96, 31.91, 31.07, 30.58, 30.54, 30.17, 29.33, 29.20, 28.92, 28.14, 27.20, 27.15, 25.95, 25.67, 25.49, 25.04, 22.59, 22.36, 20.24, 20.17, 19.28, 19.27, 18.79, 14.16.

Ethyl 2-cyano-2-(3-methylcyclohexyl)acetate, 3af (known compound [2])

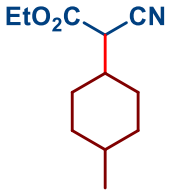


Yield 43% (mixture of diastereomers, ratio 1: 1 : 0.3 : 0.3, 180.0 mg, 0.86 mmol). Colorless oil. R_f = 0.43 (PE:EtOAc = 10:1).

^{1}H NMR (300.13 MHz, $CDCl_3$, δ): 4.31 – 4.19 (m, 2H), 3.47 – 3.33 (m, 1H), 2.39 – 1.86 (m, 2H), 1.84 – 1.10 (m, 11H), 1.08 – 0.79 (m, 3H).

^{13}C{^{1}H} NMR (75.48 MHz, $CDCl_3$, δ): 166.13, 166.08, 165.97, 116.09, 115.82, 62.70, 62.67, 62.65, 44.66, 44.62, 43.29, 43.22, 39.62, 38.82, 38.79, 37.85, 36.60, 35.64, 34.23, 34.21, 32.50, 32.29, 31.96, 31.92, 30.70, 30.18, 29.20, 28.92, 27.21, 27.16, 25.67, 25.49, 22.59, 20.25, 20.18, 19.28, 14.16.

Ethyl 2-cyano-2-(4-methylcyclohexyl)acetate, 3ag (known compound [2])

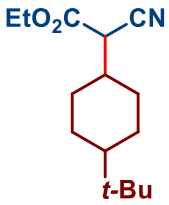


Yield 41% (mixture of diastereomers 2.3 : 1, 171.6 mg, 0.82 mmol). Colorless oil. R_f = 0.48 (PE:EtOAc = 10:1).

^{1}H NMR (300.13 MHz, $CDCl_3$, δ): 4.25 (q, *J* = 7.1 Hz, 2H), 3.45 (d, *J* = 7.5 Hz, 0.7H) + 3.36 (d, *J* = 5.6 Hz, 0.3H) = total 1H, 2.16 – 1.28 (m, 13H), 0.95 (d, *J* = 7.0 Hz, 2H) + 0.88 (d, *J* = 6.5 Hz, 1H) = total 3H.

^{13}C{^{1}H} NMR (75.48 MHz, $CDCl_3$, δ): δ 166.09, 166.00, 116.08, 115.80, 62.66, 44.49, 42.97, 38.68, 37.95, 34.51, 34.30, 31.97, 31.08, 30.60, 30.56, 29.35, 28.15, 25.97, 25.07, 22.36, 18.81, 14.16.

Ethyl 2-(4-(*tert*-butyl)cyclohexyl)-2-cyanoacetate, 3ah (known compound [2])



Isomer A: yield 32% (160.9 mg, 0.64 mmol). Colorless oil. R_f = 0.58 (PE:EtOAc = 10:1).

^{1}H NMR (300.13 MHz, $CDCl_3$, δ): 4.25 (q, *J* = 7.1 Hz, 2H), 3.61 (d, *J* = 11.5 Hz, 1H), 2.51 – 2.27 (m, 1H), 2.17 – 2.02 (m, 1H), 1.76 – 1.49 (m, 5H), 1.31 (t, *J* = 7.1 Hz, 3H), 1.17 – 0.95 (m, 3H), 0.84 (s, 9H).

^{13}C{^{1}H} NMR (75.48 MHz, $CDCl_3$, δ): 166.38, 116.76, 62.67, 47.82, 38.99, 34.88, 32.65, 28.98, 28.13, 27.49, 21.67, 21.33, 14.12.

Isomer B: yield 15% (75.4 mg, 0.30 mmol). Colorless oil. R_f = 0.51 (PE:EtOAc = 10:1).

^{1}H NMR (300.13 MHz, $CDCl_3$, δ): 4.23 (q, *J* = 7.1 Hz, 2H), 3.36 (d, *J* = 5.6 Hz, 1H), 2.03 – 1.90 (m, 1H), 1.88 – 1.75 (m, 4H), 1.35 – 1.14 (m, 5H), 1.11 – 0.95 (m, 3H), 0.82 (s, 9H).

^{13}C{^{1}H} NMR (75.48 MHz, $CDCl_3$, δ): 165.96, 115.75, 62.60, 47.23, 44.40, 38.89, 32.39, 31.45, 29.70, 27.52, 26.78, 26.56, 14.12.

Tert-butyl 4-(1-cyano-2-ethoxy-2-oxoethyl)piperidine-1-carboxylate, 3ai

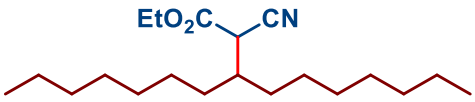
Yield 33% (195.6 mg, 0.66 mmol). Orange viscous oil. R_f = 0.25 (PE:EtOAc = 5:1).

^{1}H NMR (300.13 MHz, $CDCl_3$, δ): 4.32 – 4.09 (m, 4H), 3.40 (d, *J* = 6.0 Hz, 1H), 2.86 – 2.51 (m, 2H), 2.26 – 2.10 (m, 1H), 1.79 – 1.64 (m, 2H), 1.48 – 1.38 (m, 11H), 1.32 (t, *J* = 7.1 Hz, 3H).

^{13}C{^{1}H} NMR (75.48 MHz, $CDCl_3$, δ): 165.32, 154.62, 115.16, 79.94, 63.03, 43.76, 37.29, 30.02, 28.75, 28.52, 14.16.

HRMS (ESI-TOF) m/z $[M+Na]^+$. Calcd for $[C_{15}H_{24}N_2O_4Na]^+$: 319.1628. Found: 319.1626.

Ethyl 2-cyano-3-heptyldecanoate, 3aj



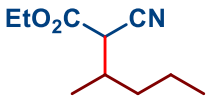
Yield 50% (323.5 mg, 0.50 mmol). Colorless oil. R_f = 0.52 (PE:EtOAc = 10:1).

^{1}H NMR (300.13 MHz, $CDCl_3$, δ): 4.26 (q, *J* = 7.1 Hz, 2H), 3.59 (d, *J* = 4.4 Hz, 1H), 2.17 – 2.00 (m, 1H), 1.57 – 1.40 (m, 3H), 1.36 – 1.20 (m, 24H), 0.92 – 0.83 (m, 6H).

^{13}C{^{1}H} NMR (75.48 MHz, $CDCl_3$, δ): 166.63, 115.78, 62.72, 42.15, 39.55, 31.93, 31.89, 31.74, 29.69, 29.58, 29.26, 29.23, 26.98, 26.82, 22.75, 14.20, 14.18.

HRMS (ESI-TOF) m/z $[M+NH_4]^+$. Calcd for $[C_{20}H_{41}N_2O_2]^+$: 341.3163. Found: 341.3167.

Ethyl 2-cyano-3-methylhexanoate, 3ak



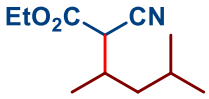
Yield 52% (mixture of diastereomers 1.5 : 1, 190.6 mg, 1.04 mmol). Colorless oil. R_f = 0.35 (PE:EtOAc = 10:1).

^{1}H NMR (300.13 MHz, $CDCl_3$, δ): 4.25 (q, *J* = 7.0 Hz, 2H), 3.51 (d, *J* = 4.5 Hz, 0.6H) + 3.41 (d, *J* = 5.3 Hz, 0.4H) = total 1H, 2.32 – 2.15 (m, 1H), 1.51 – 1.22 (m, 7H), 1.09 (d, *J* = 6.8 Hz, 1.2H) + 1.05 (d, *J* = 6.7 Hz, 1.8H) = total 3H, 0.97 – 0.85 (m, 3H).

^{13}C{^{1}H} NMR (75.48 MHz, $CDCl_3$, δ): 166.32, 166.02, 115.90, 115.42, 62.71, 62.67, 44.69, 43.84, 36.93, 35.44, 34.44, 34.33, 20.10, 20.01, 17.75, 16.44, 14.15, 13.9.

HRMS (ESI-TOF) m/z [M+Na]$^+$. Calcd for $[C_{10}H_{17}NO_2Na]^+$: 206.1151. Found: 206.1147.

Ethyl 2-cyano-3,5-dimethylhexanoate, 3al



Yield 52% (mixture of diastereomers 1.5 : 1, 205.2 mg, 1.04 mmol). Colorless oil. R_f = 0.35 (PE:EtOAc = 10:1).

^{1}H NMR (300.13 MHz, $CDCl_3$, δ): 4.31 – 4.19 (m, 2H), 3.47 (d, *J* = 4.4 Hz, 0.6H) + 3.41 (d, *J* = 5.0 Hz, 0.4H) = total 1H, 2.38 – 2.22 (m, 1H), 1.69 – 1.54 (m, 1H), 1.36 – 1.14 (m, 5H), 1.07 (d, *J* = 6.8 Hz, 1.2H) + 1.02 (d, *J* = 6.7 Hz, 1.8H) = total 3H, 0.93 – 0.81 (m, 6H).

^{13}C{^{1}H} NMR (75.48 MHz, $CDCl_3$, δ): 166.25, 165.92, 115.82, 115.38, 62.68, 62.62, 45.00, 43.98, 43.88, 42.31, 32.52, 32.30, 25.15, 25.13, 23.67, 22.74, 22.19, 21.32, 17.82, 16.41, 14.12.

HRMS (ESI-TOF) m/z [M+Na]$^+$. Calcd for $[C_{11}H_{19}NO_2Na]^+$: 220.1308. Found: 220.1312.

Ethyl 2-cyano-3-propylhexanoate, 3am

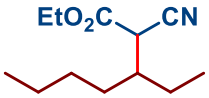
Yield 35% (147.9 mg, 0.70 mmol). Colorless oil. R_f = 0.43 (PE:EtOAc = 10:1).

^{1}H NMR (300.13 MHz, $CDCl_3$, δ): 4.24 (q, *J* = 7.1 Hz, 2H), 3.57 (d, *J* = 4.3 Hz, 1H), 2.20 – 2.01 (m, 1H), 1.55 – 1.19 (m, 11H), 0.99 – 0.80 (m, 6H).

^{13}C{^{1}H} NMR (75.48 MHz, $CDCl_3$, δ): 166.51, 115.68, 62.63, 42.05, 38.88, 34.04, 33.85, 20.02, 19.91, 14.09, 14.08, 13.97.

HRMS (ESI-TOF) m/z $[M+H]^+$. Calcd for $[C_{12}H_{22}NO_2]^+$: 212.1645. Found: 212.1637.

Ethyl 2-cyano-3-ethylheptanoate, 3an



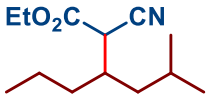
Yield 31% (131.0 mg, 0.62 mmol). Colorless oil. R_f = 0.42 (PE:EtOAc = 10:1).

^{1}H NMR (300.13 MHz, $CDCl_3$, δ): 4.25 (q, *J* = 7.1 Hz, 2H), 3.63 – 3.53 (m, 1H), 2.09 – 1.93 (m, 1H), 1.68 – 1.21 (m, 11H), 1.00 – 0.81 (m, 6H).

^{13}C{^{1}H} NMR (75.48 MHz, $CDCl_3$, δ): 166.59, 115.72, 62.70, 41.91, 41.76, 41.10, 40.97, 31.09, 29.06, 28.94, 24.75, 24.53, 22.76, 22.68, 14.13, 14.01, 13.98, 11.40, 11.26.

HRMS (ESI-TOF) m/z $[M+Na]^+$. Calcd for $[C_{12}H_{21}NO_2Na]^+$: 234.1465. Found: 234.1465.

Ethyl 2-cyano-5-methyl-3-propylhexanoate, 3ao



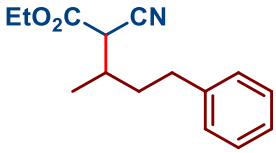
Yield 35% (157.7 mg, 0.70 mmol). Colorless oil. R_f = 0.47 (PE:EtOAc = 10:1).

^{1}H NMR (300.13 MHz, $CDCl_3$, δ): 4.23 (q, *J* = 7.1 Hz, 2H), 3.61 – 3.53 (m, 1H), 2.21 – 2.04 (m, 1H), 1.43 – 1.24 (m, 10H), 1.18 – 0.80 (m, 9H).

^{13}C{^{1}H} NMR (75.13 MHz, $CDCl_3$, δ): 166.46, 115.65, 62.59, 42.19, 42.11, 42.03, 41.18, 41.01, 38.86, 36.99, 36.77, 34.04, 33.84, 25.22, 25.15, 23.34, 23.12, 21.86, 21.79, 20.00, 19.89, 19.80, 14.06, 13.94.

HRMS (ESI-TOF) m/z $[M+NH_4]^+$. Calcd for $[C_{13}H_{27}N_2O_2]^+$: 243.2067. Found: 243.2066.

Ethyl 2-cyano-3-methyl-5-phenylpentanoate, 3ap



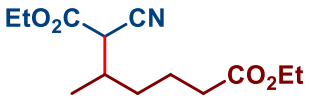
Yield 50% (mixture of diastereomers 1.5 : 1, 245.3 mg, 1.00 mmol). Colorless oil. R_f = 0.23 (PE:EtOAc = 10:1).

^{1}H NMR (300.13 MHz, $CDCl_3$, δ): 7.34 – 7.26 (m, 2H), 7.24 – 7.13 (m, 3H), 4.32 – 4.19 (m, 2H), 3.56 (d, *J* = 4.6 Hz, 0.6H) + 3.45 (d, *J* = 5.0 Hz, 0.4H) = total 1H, 2.84 – 2.50 (m, 2H), 2.38 – 2.19 (m, 1H), 1.94 – 1.62 (m, 2H), 1.35 – 1.26 (m, 3H), 1.19 (d, *J* = 6.8 Hz, 1.2H) + 1.13 (d, *J* = 6.7 Hz, 1.8H) = total 3H.

$^{13}C\{^{1}H\}$ NMR (75.13 MHz, $CDCl_3$, δ): 166.10, 141.19, 141.04, 128.70, 128.64, 128.42, 126.34, 126.26, 115.33, 62.85, 44.61, 43.88, 36.56, 34.95, 34.17, 34.15, 33.25, 33.07, 17.74, 16.48, 14.18, 14.15.

HRMS (ESI-TOF) m/z $[M+NH_4]^+$. Calcd for $[C_{15}H_{23}N_2O_2]^+$: 263.1754. Found: 263.1757.

Diethyl 2-cyano-3-methylheptanedioate, 3aq



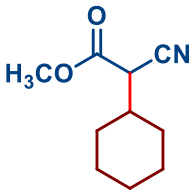
Yield 29% (mixture of diastereomers 1.5 : 1, 148.1 mg, 0.58 mmol). Slightly yellow oil. R_f = 0.40 (PE:EtOAc = 3:1).

^{1}H NMR (300.13 MHz, $CDCl_3$, δ): 4.28 – 4.19 (m, 2H), 4.14 – 4.05 (m, 2H), 3.52 (d, *J* = 4.5 Hz, 0.6H) + 3.41 (d, *J* = 5.2 Hz, 0.4H) = total 1H, 2.34 – 2.14 (m, 3H), 1.77 – 1.36 (m, 4H), 1.29 (t, *J* = 7.1 Hz, 3H), 1.25 – 1.19 (m, 3H), 1.09 (d, *J* = 6.9 Hz, 1.2H) + 1.05 (d, *J* = 6.7 Hz, 1.8H) = total 3H.

$^{13}C\{^{1}H\}$ NMR (75.13 MHz, $CDCl_3$, δ): 173.12, 166.01, 165.75, 115.63, 115.18, 62.73, 60.47, 60.41, 44.46, 43.72, 34.41, 34.31, 34.17, 34.05, 33.94, 32.67, 22.29, 22.25, 17.64, 16.32, 14.26, 14.08.

HRMS (ESI-TOF) m/z $[M+Na]^+$. Calcd for $[C_{13}H_{21}NO_4]^+$: 256.1543. Found: 256.1537.

Methyl 2-cyano-2-cyclohexylacetate, 3bb (known compound [3])

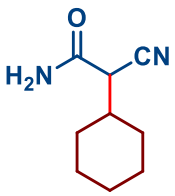


Yield 48% (174.0 mg, 0.96 mmol). Colorless oil. R_f = 0.22 (PE:EtOAc = 10:1).

^{1}H NMR (300.13 MHz, $CDCl_3$, δ): 3.79 (s, 3H), 3.38 (d, *J* = 5.7 Hz, 1H), 2.12 – 1.96 (m, 1H), 1.85 – 1.65 (m, 5H), 1.36 – 1.15 (m, 5H).

$^{13}C\{^{1}H\}$ NMR (75.48 MHz, $CDCl_3$, δ): 166.45, 115.71, 53.35, 44.49, 38.92, 31.13, 29.48, 25.87, 25.65, 25.51.

2-cyano-2-cyclohexylacetamide, 3cb



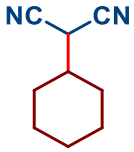
Yield 29% (96.4 mg, 0.58 mmol). White powder. R_f = 0.38 (PE:EtOAc = 1:1).

^{1}H NMR (300.13 MHz, DMSO-*d*$_6$, δ): 7.66 (s, 1H), 7.42 (s, 1H), 3.50 (d, *J* = 7.0 Hz, 1H), 1.92 – 1.58 (m, 6H), 1.32 – 1.01 (m, 5H).

^{13}C{^{1}H} NMR (75.48 MHz, DMSO-*d*$_6$, δ) 166.41, 117.78, 44.32, 37.96, 30.47, 29.27, 25.36, 25.23, 25.13.

HRMS (ESI-TOF) m/z [M+Na]$^+$. Calcd for [$C_9H_{14}N_2ONa$]$^+$: 189.0998. Found: 189.0997.

2-cyclohexylmalononitrile, 3db (known compound [4])

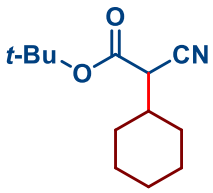


Yield 62% (141.0 mg, 0.62 mmol). Light green oil. R_f = 0.22 (PE:EtOAc = 10:1).

^{1}H NMR (300.13 MHz, $CDCl_3$, δ): 3.57 (d, *J* = 5.6 Hz, 1H), 2.03 – 1.68 (m, 6H), 1.39 – 1.14 (m, 5H).

^{13}C{^{1}H} NMR (75.48 MHz, $CDCl_3$, δ): 112.10, 39.46, 30.03, 29.45, 25.35, 25.16.

***Tert*-butyl 2-cyano-2-cyclohexylacetate, 3eb** (known compound [5])



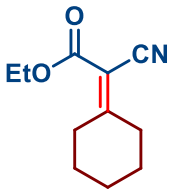
Yield 43% (191.8 mg, 0.86 mmol). Colourless oil. R_f = 0.43 (PE:EtOAc = 10:1).

^{1}H NMR (300.13 MHz, $CDCl_3$, δ): 3.25 (d, *J* = 5.8 Hz, 1H), 2.07 – 1.93 (m, 1H), 1.88 – 1.57 (m, 6H), 1.49 (s, 9H), 1.34 – 1.14 (m, 5H).

^{13}C{^{1}H} NMR (75.48 MHz, $CDCl_3$, δ): 164.95, 116.19, 83.87, 45.66, 38.93, 31.14, 29.43, 27.95, 25.94, 25.75, 25.64.

Characterization of alkene 4

Ethyl 2-cyano-2-cyclohexylideneacetate, 4ab



Yield 53% (204.8 mg, 1.06 mmol) (the reaction conditions from Table 1, entry 28). Yellowish oil. R_f = 0.39 (PE:EtOAc = 10:1).

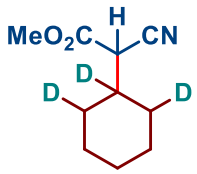
^{1}H NMR (300.13 MHz, $CDCl_3$, δ): 4.22 (q, *J* = 7.1 Hz, 2H), 3.09 – 2.83 (m, 2H), 2.76 – 2.51 (m, 2H), 1.91 – 1.50 (m, 6H), 1.30 (t, *J* = 7.1 Hz, 3H).

^{13}C{^{1}H} NMR (75.48 MHz, $CDCl_3$, δ): 179.91, 161.98, 115.56, 102.04, 61.67, 36.87, 31.57, 28.58, 28.25, 25.62, 14.07.

Experimental Procedure for Scheme 7.

NH_4OAc (10.0 mmol) was added to the flame dried 20 mL IKA electrolysis cell filled with argon *via* weighing cup and cell was closed with rubber sept. Methanol-D_1 (MeOD, 10 mL) was added *via* cannula. The same way cyclohexanone **1b** (2.0 mmol, 196.3 mg) and methyl cyanoacetate **2b** (4.0 mmol, 396.4 mg) were added. Then rubber sept was removed, after which glassy carbon electrodes (8 × 18 mm) were immediately placed and a direct current was applied. The mixture was electrolyzed using constant current conditions at 50 ˚C under magnetic stirring for 128 min. with I = 100 mA (*j* = 70 mA/cm^2). After completion of electrolysis, the reaction mixture was concentrated under reduced pressure using a rotary evaporator (15-20 mmHg), (bath temperature ~ 50 °C). EtOAc (25 mL) and H_2O (25 mL) was added to the reaction mixture. The organic phase was separated, washed with H_2O (25 mL), and later with saturated aq. solution NaCl (25 mL), dried over Na_2SO_4. The organic phase was filtered off, concentrated under reduced pressure using a rotary evaporator (15-20 mmHg), (bath temperature, about 30-40 °C). The desired product **3bb-*d*$_n$**. was isolated by chromatography on SiO_2 (PE:EtOAc = from 20:1 to 2:1).

Methyl 2-cyano-2-cyclohexylacetate-*d*$_n$, 3bb-*d*$_n$



Yield 50% (184.25 mg, 1.0 mmol). Colorless oil. R_f = 0.22 (PE:EtOAc = 10:1).

^{1}H NMR (300.13 MHz, $CDCl_3$, δ): 3.76 (s, 3H), 3.42 – 3.32 (m, 1H), 2.09 – 1.91 (m, 0.5H), 1.88 – 1.57 (m, 4H), 1.35 – 1.04 (m, 4H).

^{13}C{^{1}H} NMR (75.48 MHz, $CDCl_3$, δ): 166.35, 115.62, 53.22, 44.26 (d, *J* = 5.7 Hz), 38.85 – 38.40 (m), 31.09 – 30.13 (m), 29.42 – 28.47 (m), 25.81 – 25.26 (m).

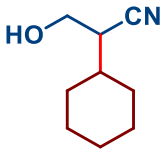
Experimental Procedure for Scheme 10.

Synthesis of compound 5

Target molecule **5** was synthesized *via* modified procedure [6].

In a round-bottomed flask, compound **3ab** (2.0 mmol, 390.5 mg) was dissolved in anhydrous THF (2.0 mL), and the reaction vessel was purged with argon. Subsequently, a solution of $Mg(BH_4)_2$ (6.0 mmol) in anhydrous THF (2.0 mL) was added dropwise to the reaction mixture at ambient temperature. The resulting mixture was stirred for 4 hours, after which the residual borohydride was quenched by the careful addition of 1 N HCl solution. The target aryl β-cyanoalcohol was then extracted with CH_2Cl_2 (25 mL). The combined organic extracts were dried over anhydrous $MgSO_4$ and filtered. Removal of the solvent under reduced pressure afforded product **5** (281.9 mg, 92% yield), which was utilized in the subsequent step without further purification.

2-cyclohexyl-3-hydroxypropanenitrile, 5



Yield 92% (281.9 mg, 1.84 mmol). Colourless oil. R_f = 0.11 (PE:EtOAc = 5:1).

^{1}H NMR (300.13 MHz, DMSO-d_6, δ): 5.20 (t, J = 5.4 Hz, 1H), 3.57 (t, J = 5.7 Hz, 2H), 2.68 (q, J = 6.2 Hz, 1H), 1.82 – 1.56 (m, 6H), 1.27 – 1.01 (m, 5H).

^{13}C{^{1}H} NMR (75.48 MHz, DMSO-d_6, δ): 120.83, 59.16, 40.56, 35.24, 30.66, 29.15, 25.64, 25.49, 25.33.

HRMS (ESI-TOF) m/z $[M+Na]^+$. Calcd for $[C_9H_{15}NONa]^+$: 176.1046. Found: 176.1047.

Synthesis of compound 6

A solution of methanesulfonyl chloride (1.0 equiv., 1.84 mmol, 210.8 mg) in EtOAc (9 mL) was added dropwise to a solution of alcohol **5** (1.84 mmol, 281.9 mg) and triethylamine (1.1 equiv., 2.02 mmol, 204.4 mg) in EtOAc (9 mL). The reaction mixture was stirred at ambient temperature for 4 hours. Complete conversion of alcohol **5** was confirmed by thin-layer chromatography (TLC). Subsequently, the reaction mixture was washed twice with brine. The organic layer was dried over anhydrous Na_2SO_4, filtered, and concentrated under reduced pressure using a rotary evaporator (15–20 mmHg) at a bath temperature of approximately 30–40 °C. The resulting product **6** (340.5 mg, 80% yield) was obtained as the sole product and employed in the following step without further purification.

2-cyano-2-cyclohexylethyl methanesulfonate, 6

Yield 80% (340.5 mg, 1.47 mmol). Colourless oil. R_f = 0.55 (PE:EtOAc = 1:1).

^{1}H NMR (300.13 MHz, $CDCl_3$, δ): 4.40 – 4.28 (m, 2H), 3.10 (s, 3H), 2.85 (dq, *J* = 7.2, 6.0 Hz, 1H), 1.89 – 1.66 (m, 6H), 1.32 – 1.16 (m, 5H).

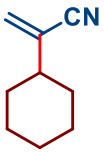
^{13}C{^{1}H} NMR (75.48 MHz, $CDCl_3$, δ): 118.21, 65.95, 38.59, 38.02, 36.50, 31.06, 29.68, 25.83, 25.75, 25.67.

HRMS (ESI-TOF) m/z $[M+H]^+$. Calcd for $[C_{10}H_{17}NO_3SNa]^+$: 254.0821. Found: 254.0821.

Synthesis of compound 7

DABCO (1.1 eq., 1.62 mmol, 181.7 mg) was added portion wise to a solution of **6** (1.47 mmol, 340.5 mg) in EtOAc (10 mL). The resulting reaction mixture was then refluxed for 6 h. Conversion of the starting material was monitored by thin-layer chromatography (TLC). Subsequently, the reaction mixture was allowed to cool to room temperature, and the precipitated crystals were filtered off. The filtrate was concentrated under reduced pressure (15–20 mmHg) using a rotary evaporator at a bath temperature of approximately 30–40 °C. The desired product **7** was isolated by chromatography on SiO_2 (PE:EtOAc = from 20:1 to 2:1).

2-cyclohexylacrylonitrile, 7



Yield 60% (119.3 mg, 0.88 mmol). Colourless oil. R_f = 0.69 (PE:EtOAc = 4:1).

^{1}H NMR (300.13 MHz, $CDCl_3$, δ): 5.78 (s, 1H), 5.67 (d, *J* = 1.3 Hz, 1H), 2.22 – 2.06 (m, 1H), 1.94 – 1.63 (m, 5H), 1.38 – 1.10 (m, 5H).

^{13}C{^{1}H} NMR (75.48 MHz, $CDCl_3$, δ): 129.23, 128.05, 118.51, 42.69, 31.42, 25.85, 25.65.

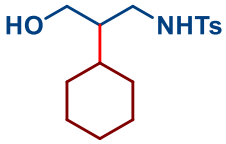
HRMS (ESI-TOF) m/z $[M+H]^+$. Calcd for $[C_9H_{14}N]^+$: 136.1121. Found: 136.1116.

Synthesis of compound 8

In a round-bottomed flask (argon gas atmosphere) $LiAlH_4$ (4.0 mmol, 42.0 mg) was added to the anhydrous THF (2.0 mL) and stirred at 5 °C for 10 minutes. Then solution **3ab** (2.0 mmol, 390.5 mg) in anhydrous THF (2.0 mL) was added *via* dropping funnel to the reaction vessel. The resulting mixture was let to warm up to room temperature and then refluxed for 4 hours. After reaction complete mixture was quenched by the careful addition of 6 N NaOH solution. The reaction mixture was extracted with CH_2Cl_2 (25 mL). The combined organic extracts were dried over anhydrous $MgSO_4$ and filtered. The reaction mixture was concentrated under reduced pressure (15–20 mmHg) using a rotary evaporator

at a bath temperature of approximately 30–40 °C. The resulted oil was dissolved in EtOAc (10 mL). Et_3N (2.2 equiv., 4.04 mmol, 408.8 mg) and p-toluenesulfonyl chloride (1.0 equiv., 2.0 mmol, 381.3 mg) was added to the reaction mixture. The reaction mixture was stirred at room temperature for 24 hours. Subsequently, the reaction mixture was washed twice with brine (25 mL). The organic layer was dried over anhydrous Na_2SO_4, filtered, and concentrated under reduced pressure using a rotary evaporator (15–20 mmHg) at a bath temperature of approximately 30–40 °C. The desired product 8 was isolated by chromatography on SiO_2 (PE:EtOAc = from 20:1 to 2:1).

N-(2-cyclohexyl-3-hydroxypropyl)-4-methylbenzenesulfonamide, 8



Yield 25% (155.8 mg, 0.50 mmol) – based on the started compound **3ab**. Yellow viscous oil. R_f = 0.44 (PE:EtOAc = 1:1).

^{1}H NMR (300.13 MHz, DMSO-d_6, δ): 7.67 (d, J = 8.1 Hz, 2H), 7.38 (d, J = 8.0 Hz, 2H), 7.31 (t, J = 5.9 Hz, 1H), 4.35 (t, J = 4.8 Hz, 1H), 3.45 – 3.36 (m, 1H), 3.32 – 3.20 (m, 1H), 2.89 – 2.56 (m, 2H), 2.37 (s, 3H), 1.66 – 0.83 (m, 12H).

^{13}C{^{1}H} NMR (75.48 MHz, DMSO-d_6, δ): 142.43, 137.69, 129.56, 126.56, 59.53, 45.54, 41.77, 35.94, 29.78, 28.92, 26.35, 26.30, 26.20, 20.95.

HRMS (ESI-TOF) m/z [M+H]$^+$. Calcd for $[C_{16}H_{26}NO_3S]^+$: 312.1628. Found: 312.1638.

CV study

Cyclic voltammetry (CV) was implemented on an IPC-Pro M computer-assisted potentiostat manufactured by «Econix» (scan rate error 1.0%). The starting potential was set to 0.25 mV, and the initial sweep was carried out in the positive (anode) region at a rate of 100 mV/s. Analyzed solutions were prepared in ethanol (water content 5% by mass) and contained *n*-Bu_4NBF_4 (0.1 M) as a supporting electrolyte and analyte (0.05 M). The experiments were performed in a 10 mL five neck glass conic electrochemical cell with a water jacket for thermostatting. CV curves were recorded using a three-electrode scheme. In a typical case, 10 mL of a solution was utilized. The working electrode was a disc glassy-carbon or platinum electrode (d = 3 mm, surface area ~0.07 cm^2). A platinum wire served as an auxiliary electrode. An $Ag/AgNO_3$ 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 15±0.5 °C and deaerated by bubbling argon. Electrochemical experiments were performed under an argon atmosphere. The working electrode was polished with figure-eight motions on a synthetic chamois leather pad using a Cr_2O_3-based polishing paste (~5 μm particle size) down to the mirror-like surface, and rinsed with ethanol. Polishing was carried before each recording of CV curve.

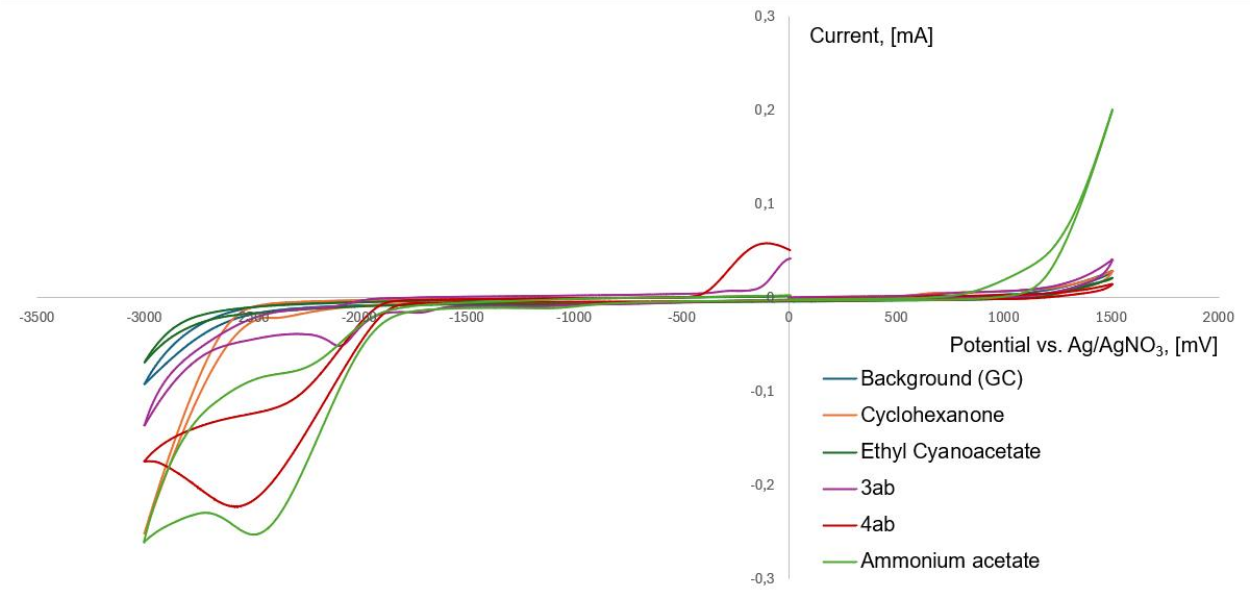


Figure S1. CV curve for on a working disc glassy-carbon electrode (d = 3 mm) under a scan rate of 0.1 V/s with a sweep in the positive (anode) region.

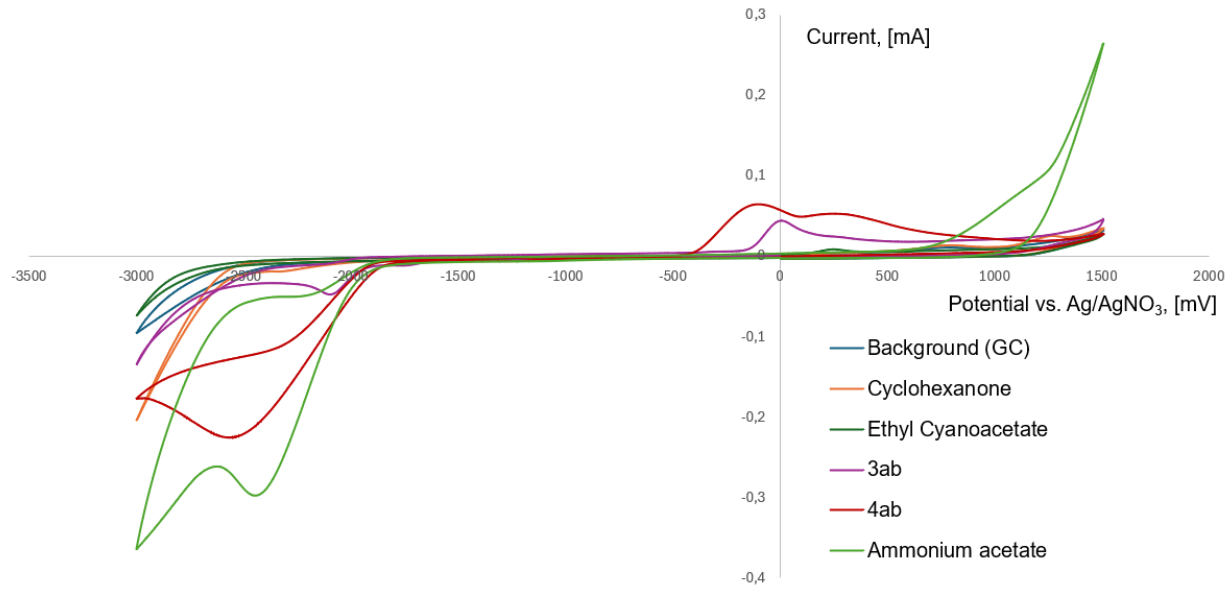


Figure S2. CV curve for on a working disc glassy-carbon electrode (d = 3 mm) under a scan rate of 0.1 V/s with a sweep in the negative (cathode) region.

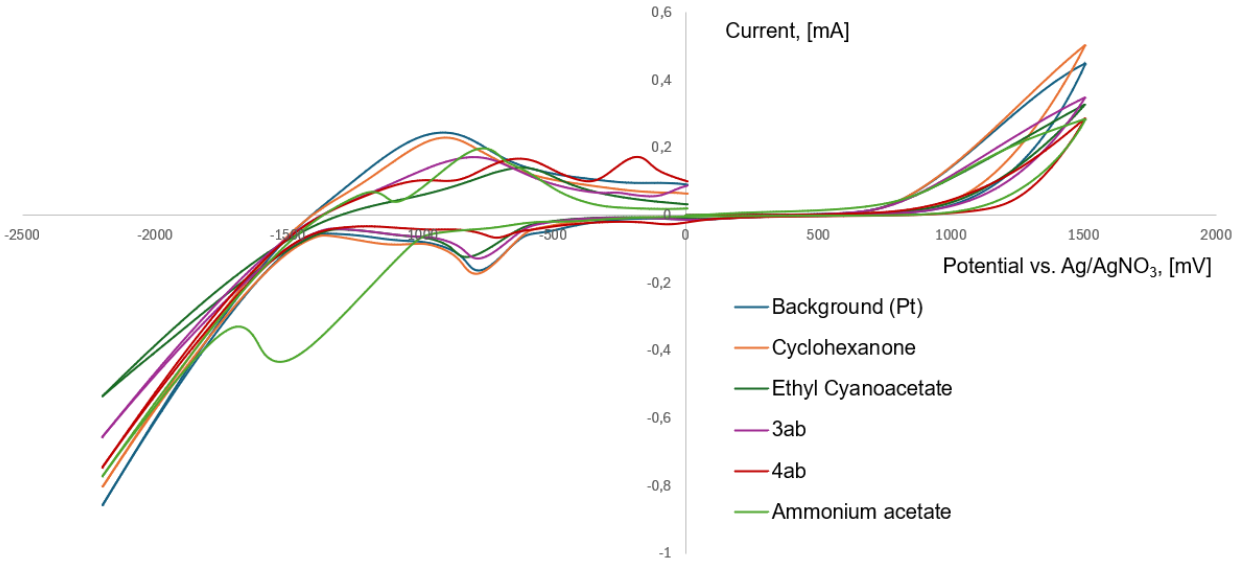


Figure S3. CV curve for on a working disc platinum electrode (d = 3 mm) under a scan rate of 0.1 V/s with a sweep in the positive (anode) region.

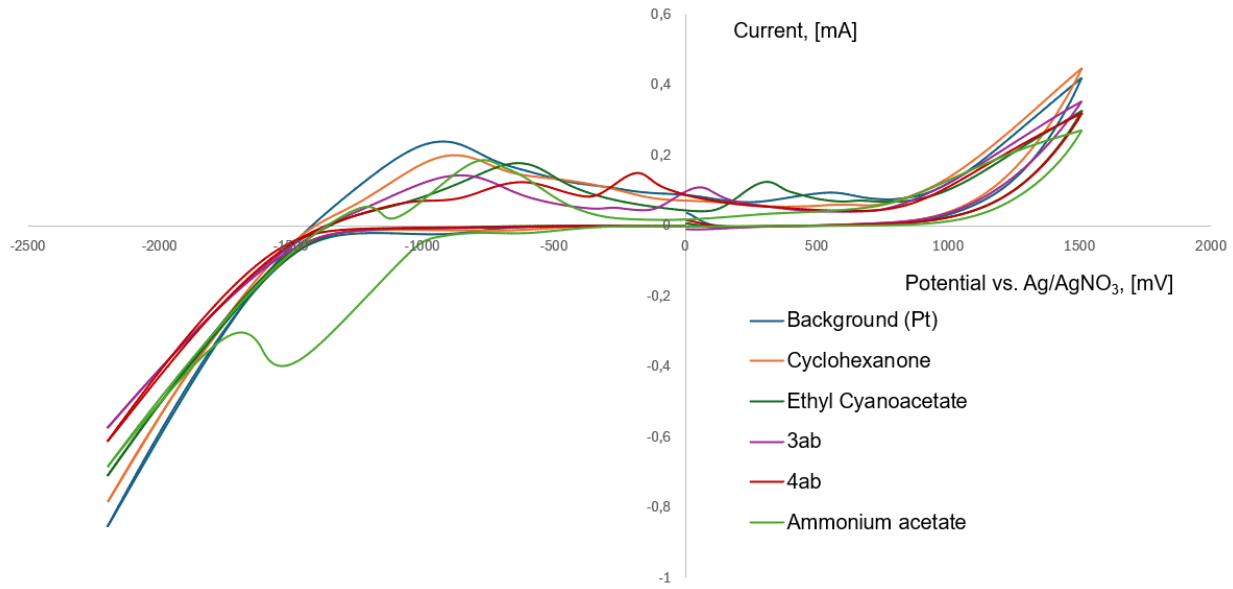


Figure S4. CV curve for on a working disc platinum electrode (d = 3 mm) under a scan rate of 0.1 V/s with a sweep in the negative (cathode) region.

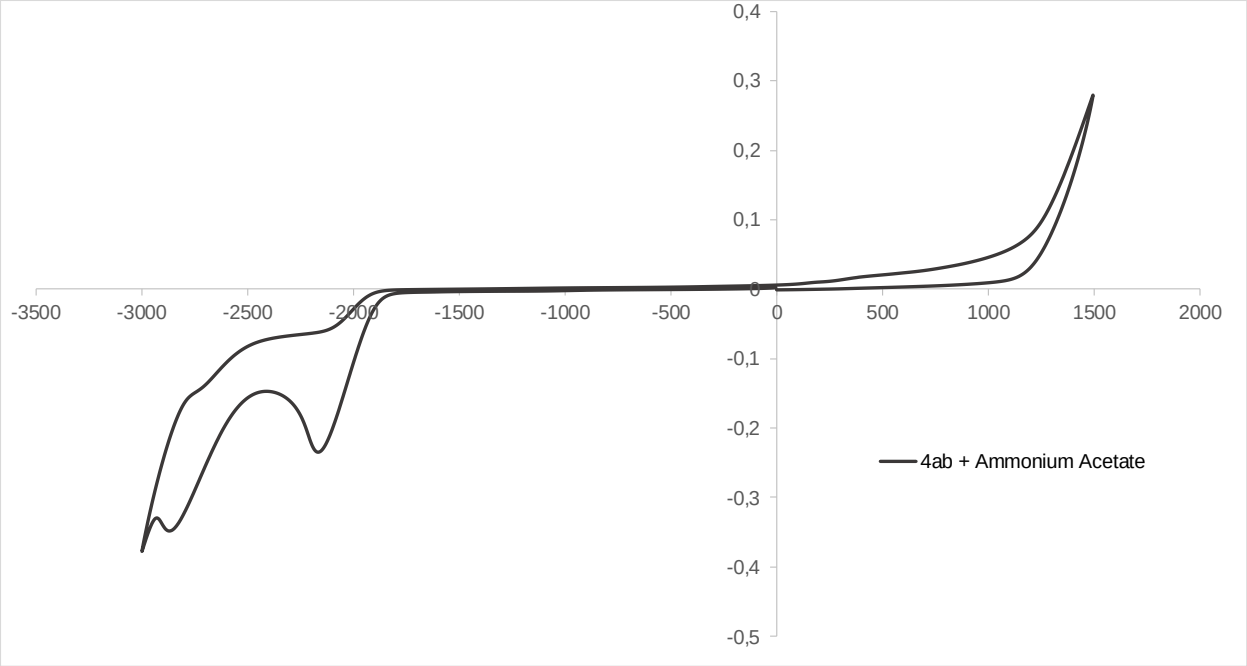
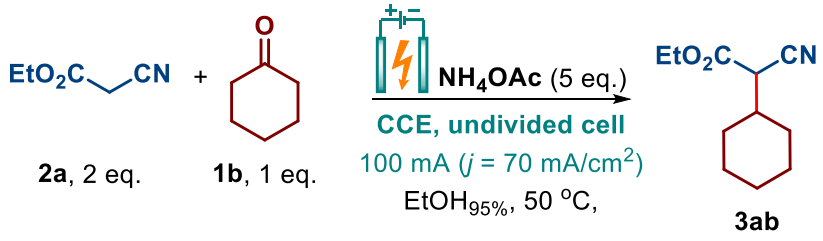


Figure S5. CV curve for on a working disc glassy-carbon electrode (d = 3 mm) under a scan rate of 0.1 V/s with a sweep in the negative (cathode) region.

Hydrogen detection with GC

The detection of produced hydrogen during electrolysis was carried out using gas chromatography modified with two columns. The use of a combination of a flame ionization detector and a Restek ShinCarbon ST, 80/100, 2 m, Din = 2 mm, 1/8 (USA) packed column made it possible to effectively separate and quantitatively determine the content of hydrocarbons of the C1 – C4 fraction in the reaction mixture, while the use of a CR-Alumina BOND/KCl capillary column 50 m, Din = 0.53 mm (Chromatec, Russia), connected to a thermal conductivity detector, made it possible to qualitatively evaluate changes in the concentrations of components such as hydrogen, nitrogen, carbon monoxide, as well as hydrocarbons of the C1 – C4 fraction.

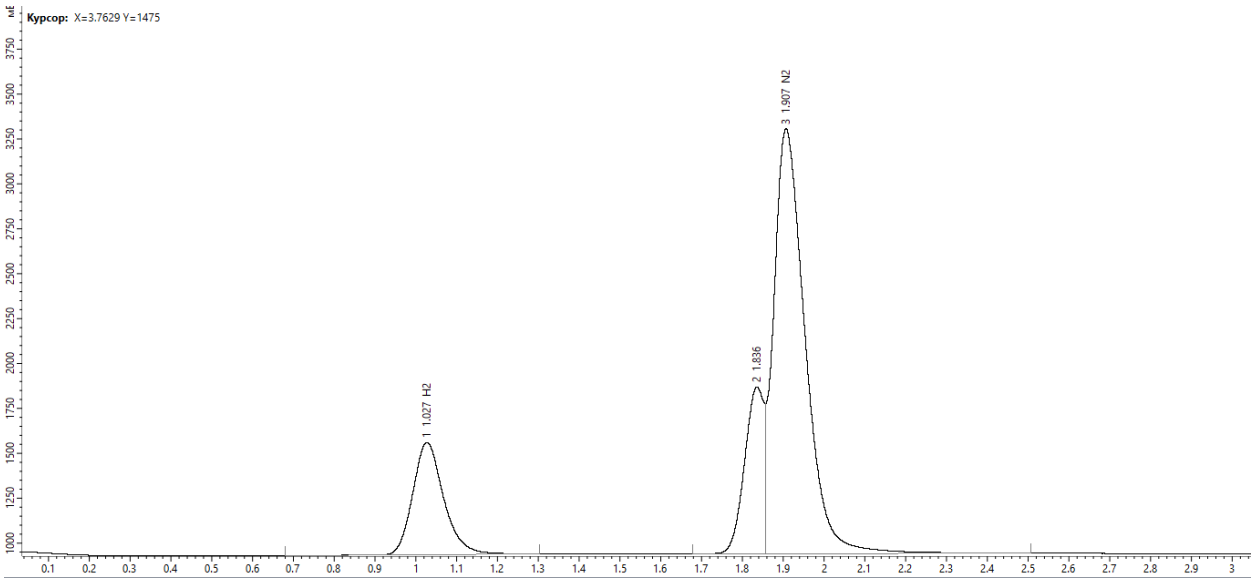
Electrolysis of the starting substrates was carried out under optimized conditions in two variants: with a glassy carbon cathode and with a platinum cathode. The other parameters were the same. The relative amount of hydrogen was measured in relation to the amounts of oxygen and nitrogen, which remained nearly constant under these conditions. Less hydrogen was produced using the glassy carbon cathode than the platinum cathode. These results are in line with our hypothesis that the glassy carbon cathode supports substrate reduction more effectively than the platinum cathode.

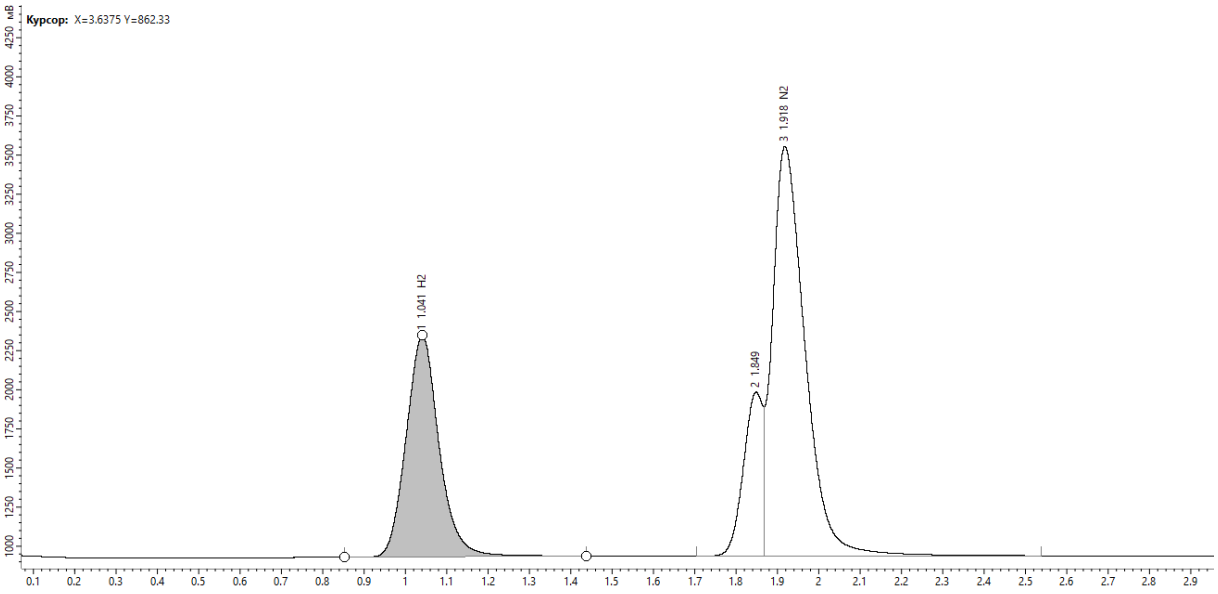


2a, 2 eq. **1b**, 1 eq. **3ab**

after 2 F per mol **1b**

ratio	H_2 / (O_2+N_2)
GC(+)/GC(-)	0.26
GC(+)/Pt(-)	0.54


Курсор: X=3.7629 Y=1475
мВ
1 1.027 H2
2 1.836
3 1.907 N2


Курсор: X=3.6375 Y=862.33
мВ
1 1.041 H2
2 1.849
3 1.918 N2

Parameter	Value		
Column thermostat	Rate °/min	T, °C	Exposure, min
		80	5
	15	180	8,3
thermal conductivity detector			
Temperature, °C	150		
Reference gas flow rate during purging, mL/min	20		
Reference gas flow rate, mL/min	15		
flame ionization detector			
Temperature, °C	150		
Purge gas flow rate, mL/min	30		
Hydrogen flow rate, mL/min	20		
Air flow rate, mL/min	200		
Injection port			
Sample injection mode:	With flow splitting and gas saving		
Flow splitting ratio	0,5		
Membrane purge flow rate, mL/min	0		
Pressure, kPa	47,375		
Temperature, °C	150		
Column 1 (CR-Alumina BOND/KCl)			
Carrier gas mode	Constant flow		
Flow rate, mL/min	5		
Carrier gas	Helium		
Column 2 (Restek ShinCarbon ST)			
Carrier gas mode	Constant pressure		
Flow rate, mL/min	15		
Carrier gas	Argon		
Valve 1, 2			
Temperature, °C	80		

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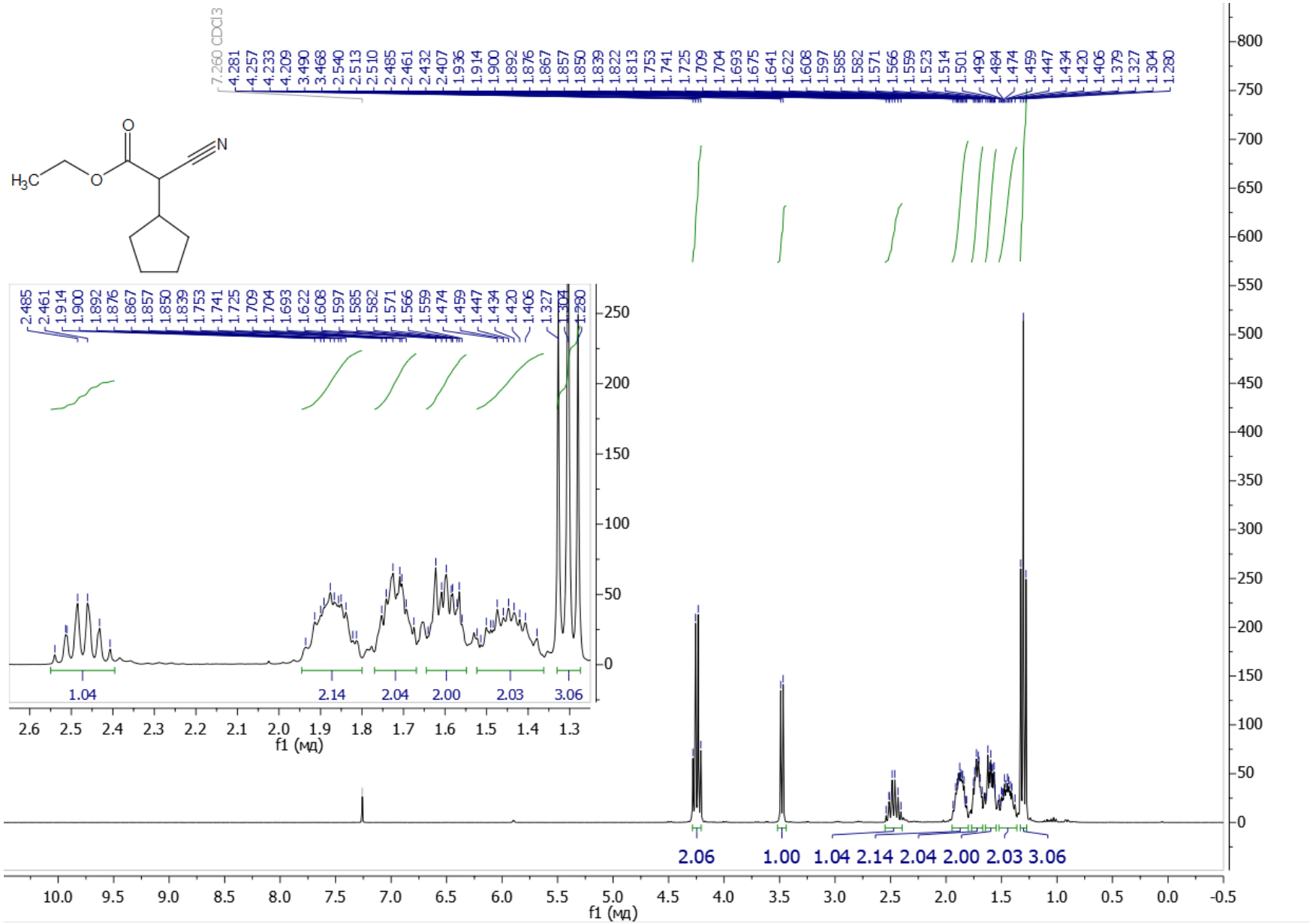
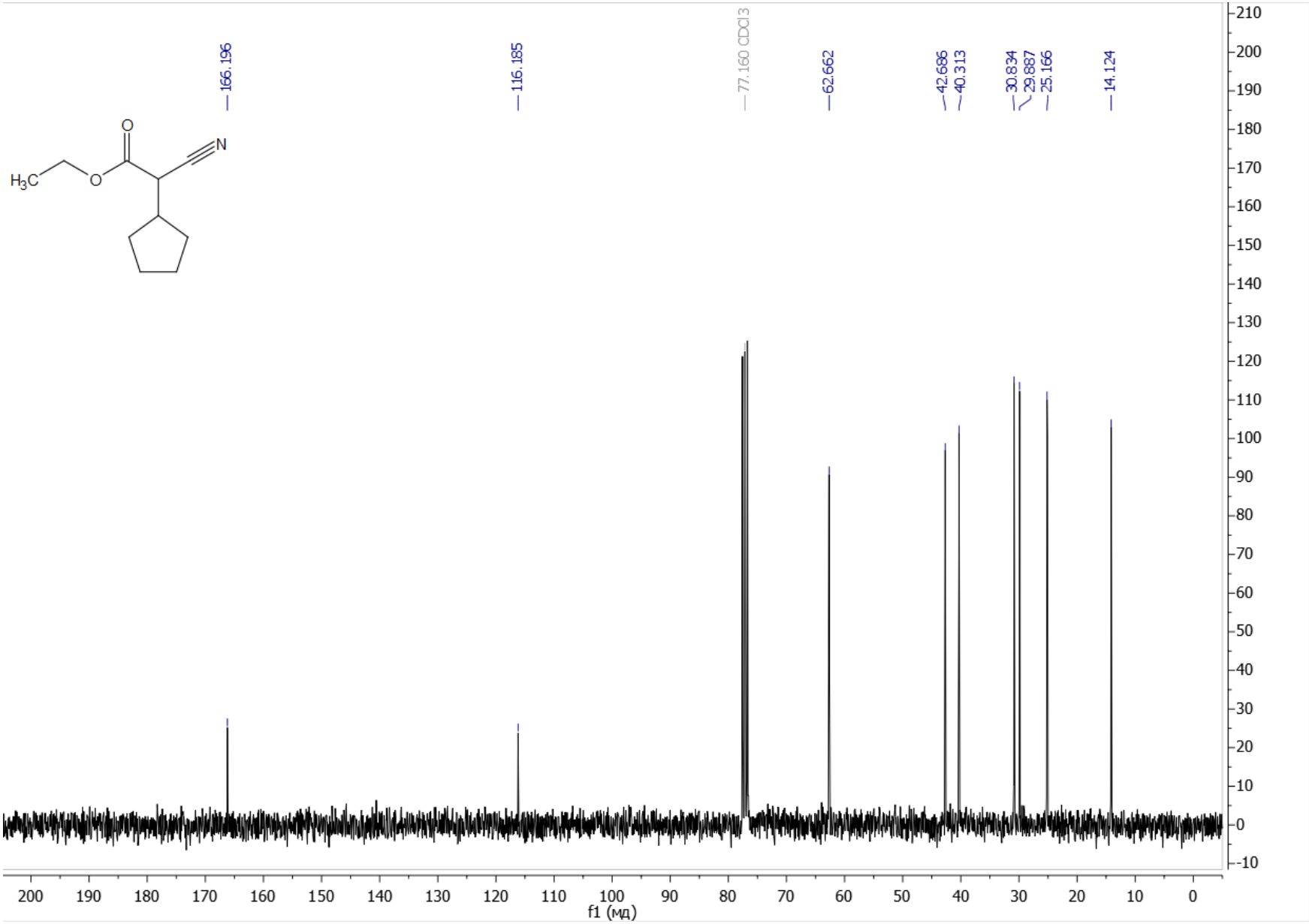
NMR spectra of the synthesized compounds

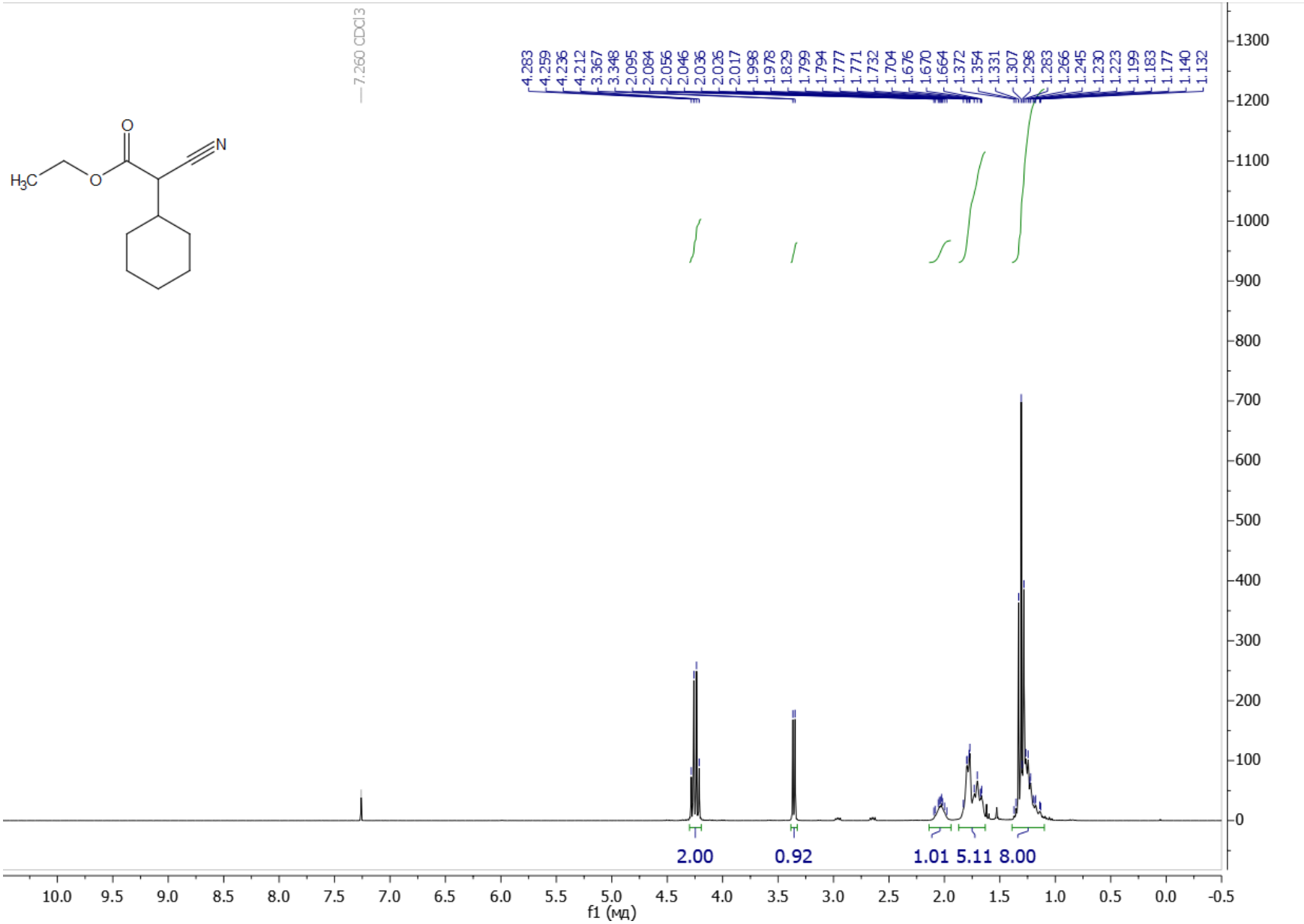
^{1}H NMR (300.13 MHz, $CDCl_3$) spectrum of ethyl 2-cyano-2-cyclopentylacetate, **3aa**



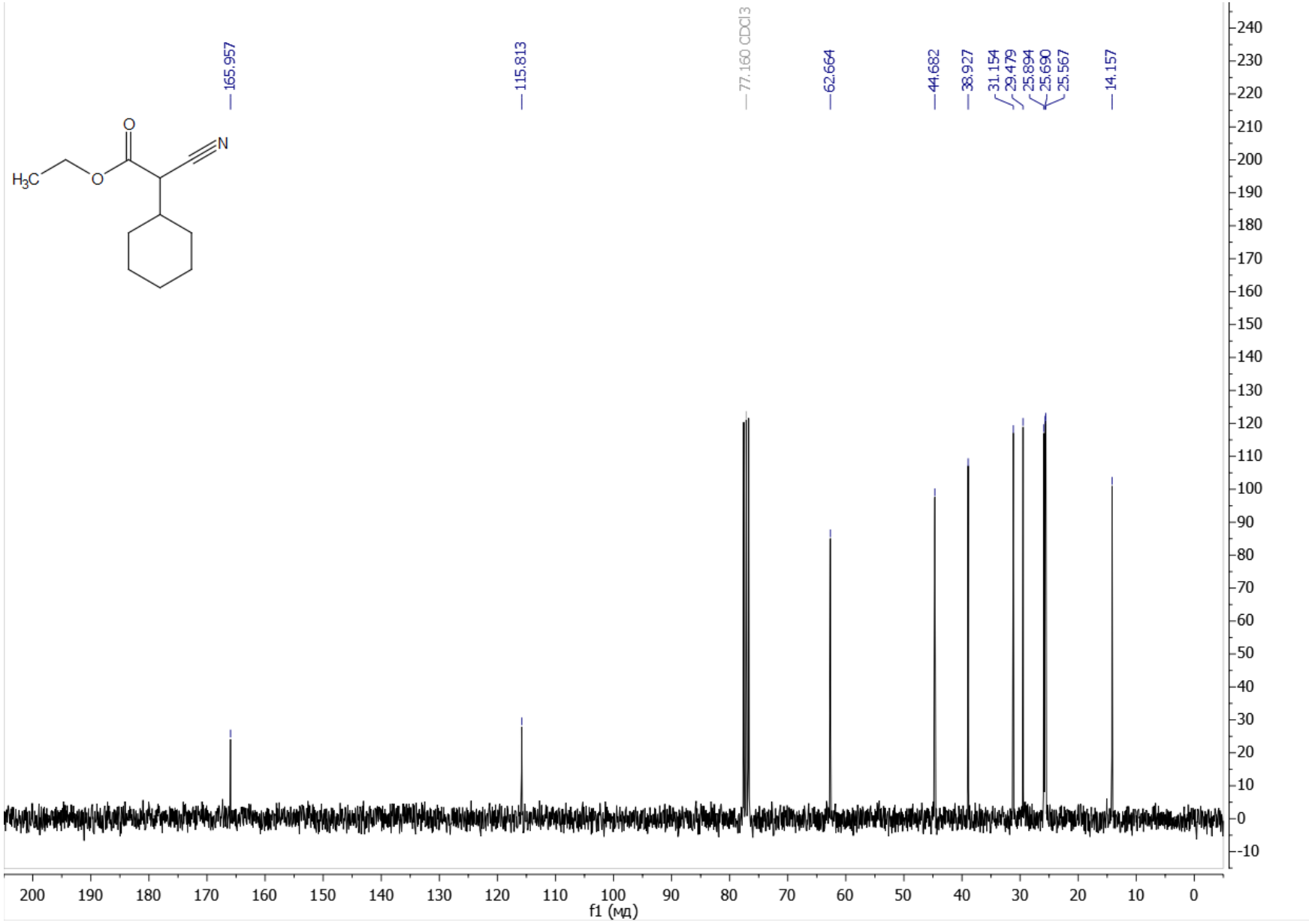
^{13}C NMR (75.48 MHz, $CDCl_3$) spectrum of ethyl 2-cyano-2-cyclopentylacetate, **3aa**



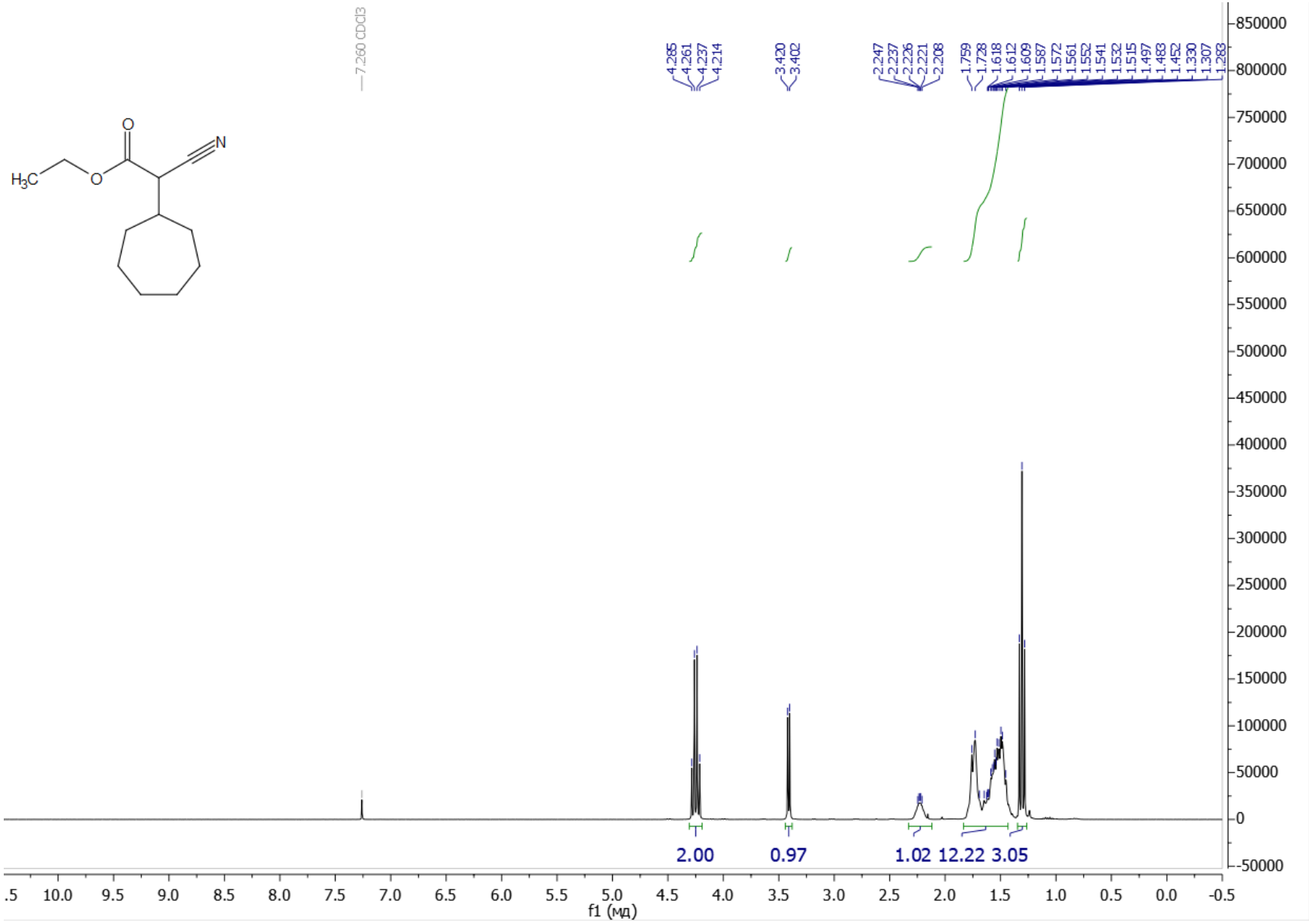
^{1}H NMR (300.13 MHz, $CDCl_3$) spectrum of ethyl 2-cyano-2-cyclohexylacetate, **3ab**



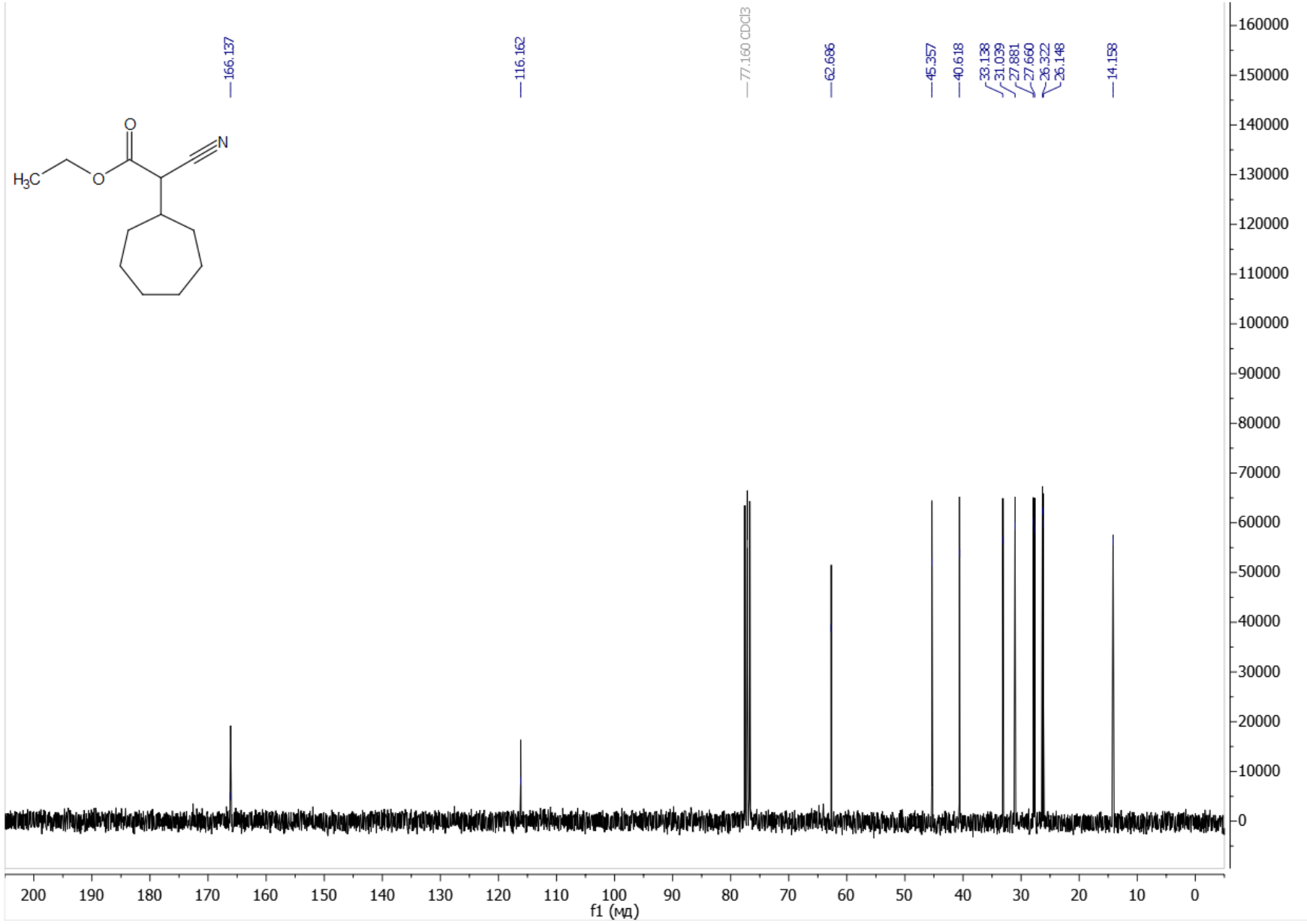
^{13}C NMR (75.48 MHz, $CDCl_3$) spectrum of ethyl 2-cyano-2-cyclohexylacetate, **3ab**



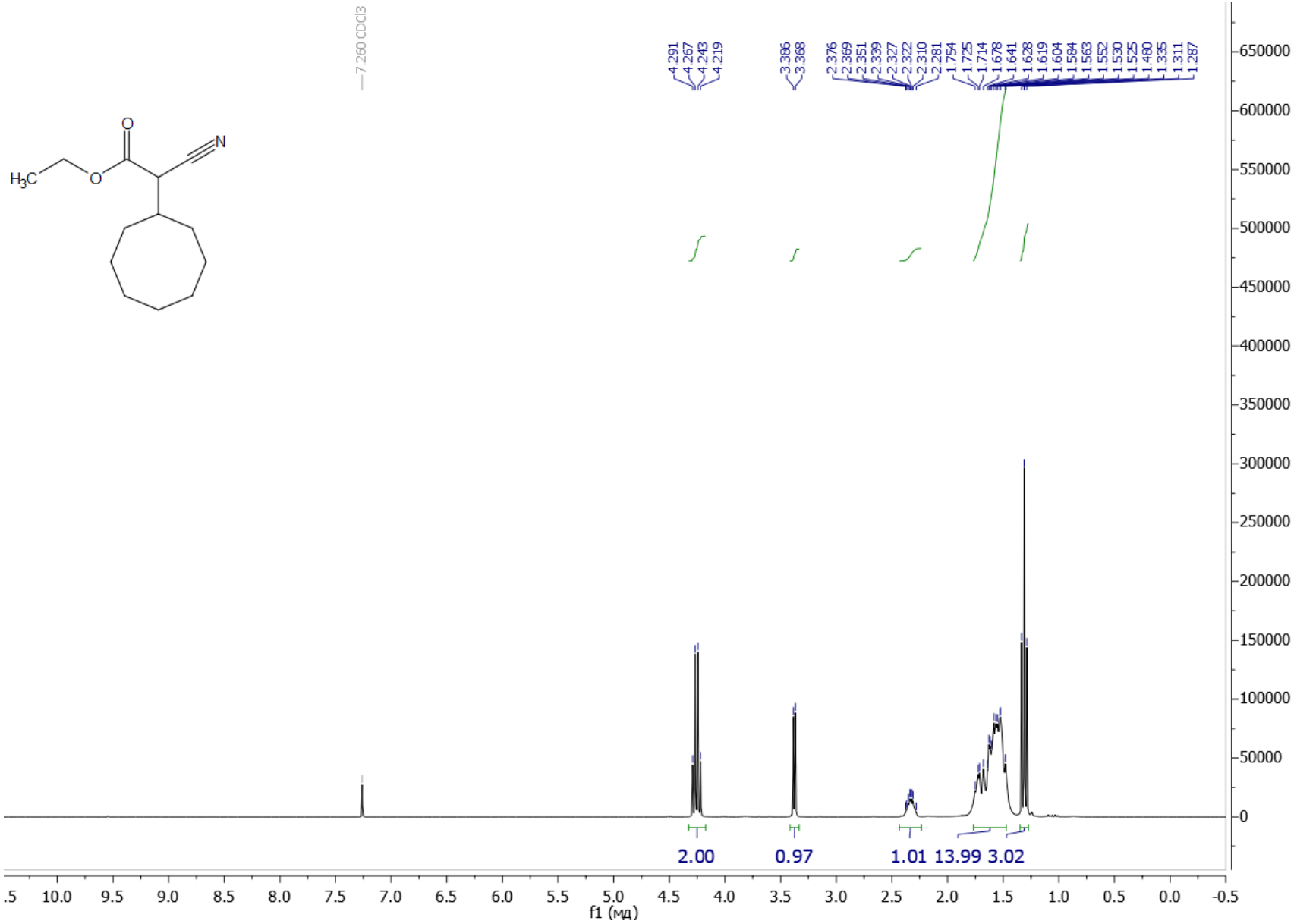
^{1}H NMR (300.13 MHz, $CDCl_3$) spectrum of ethyl 2-cyano-2-cycloheptylacetate, **3ac**



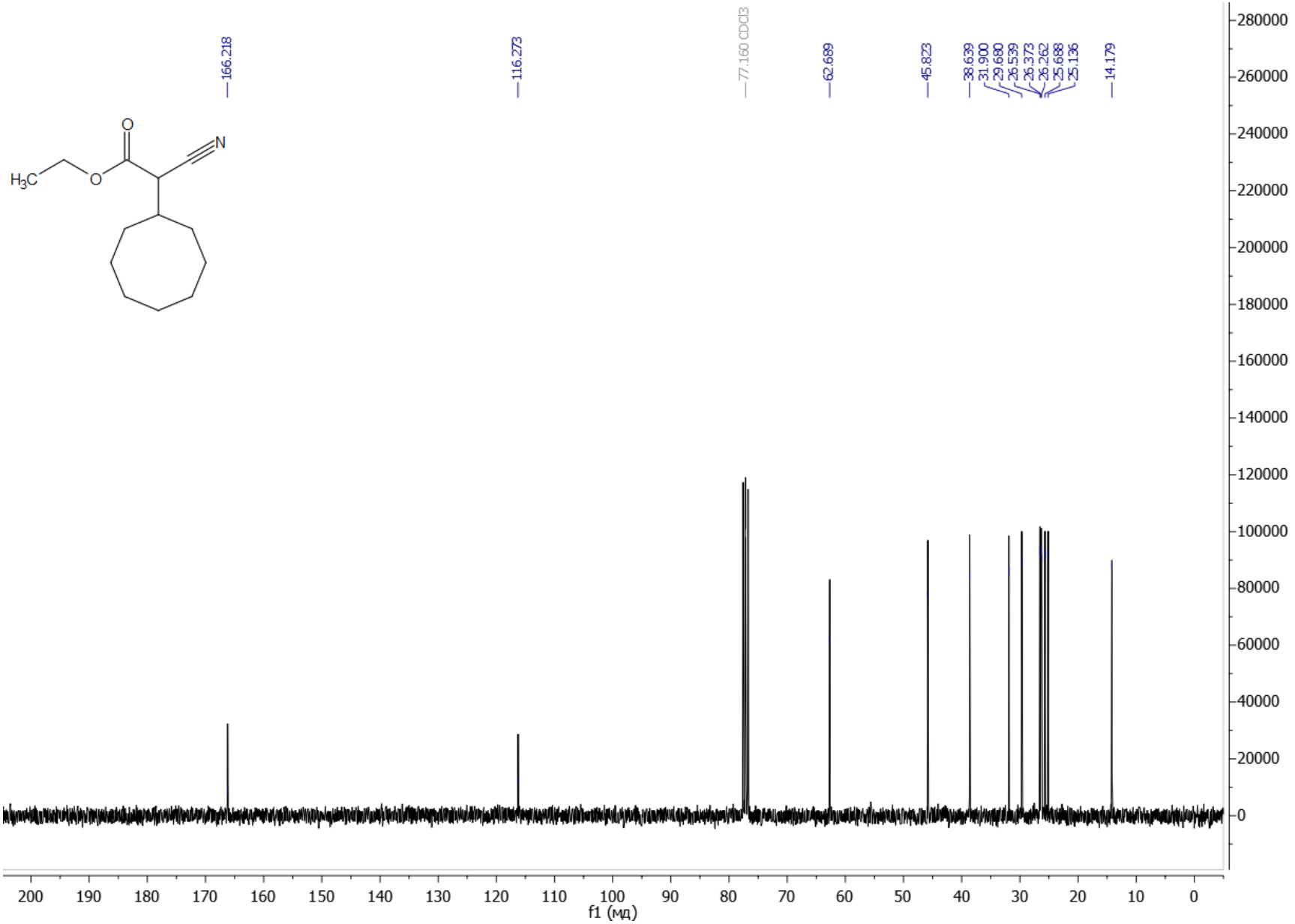
^{13}C NMR (75.48 MHz, $CDCl_3$) spectrum of ethyl 2-cyano-2-cycloheptylacetate, **3ac**



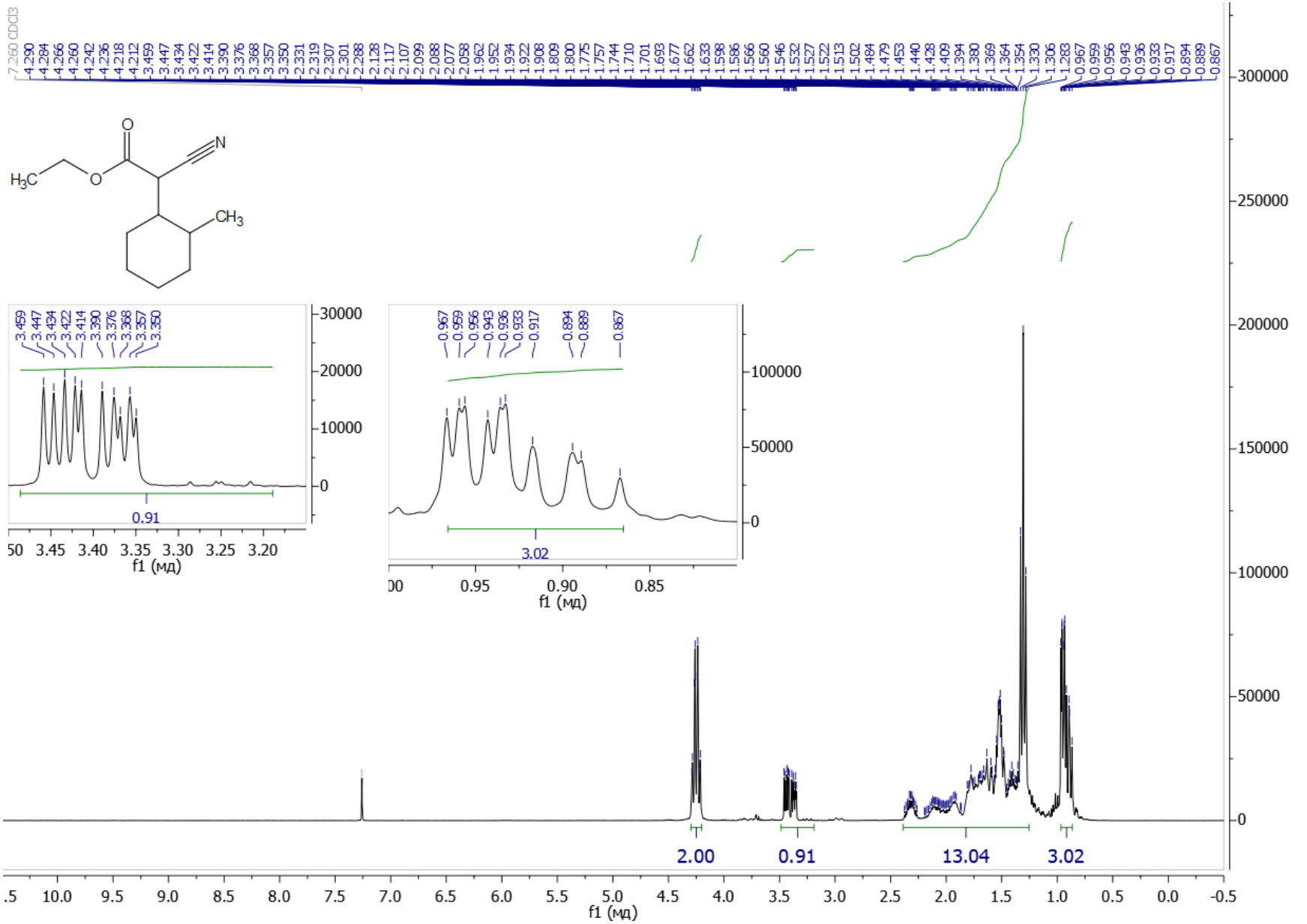
^{1}H NMR (300.13 MHz, $CDCl_3$) spectrum of ethyl 2-cyano-2-cyclooctylacetate, **3ad**



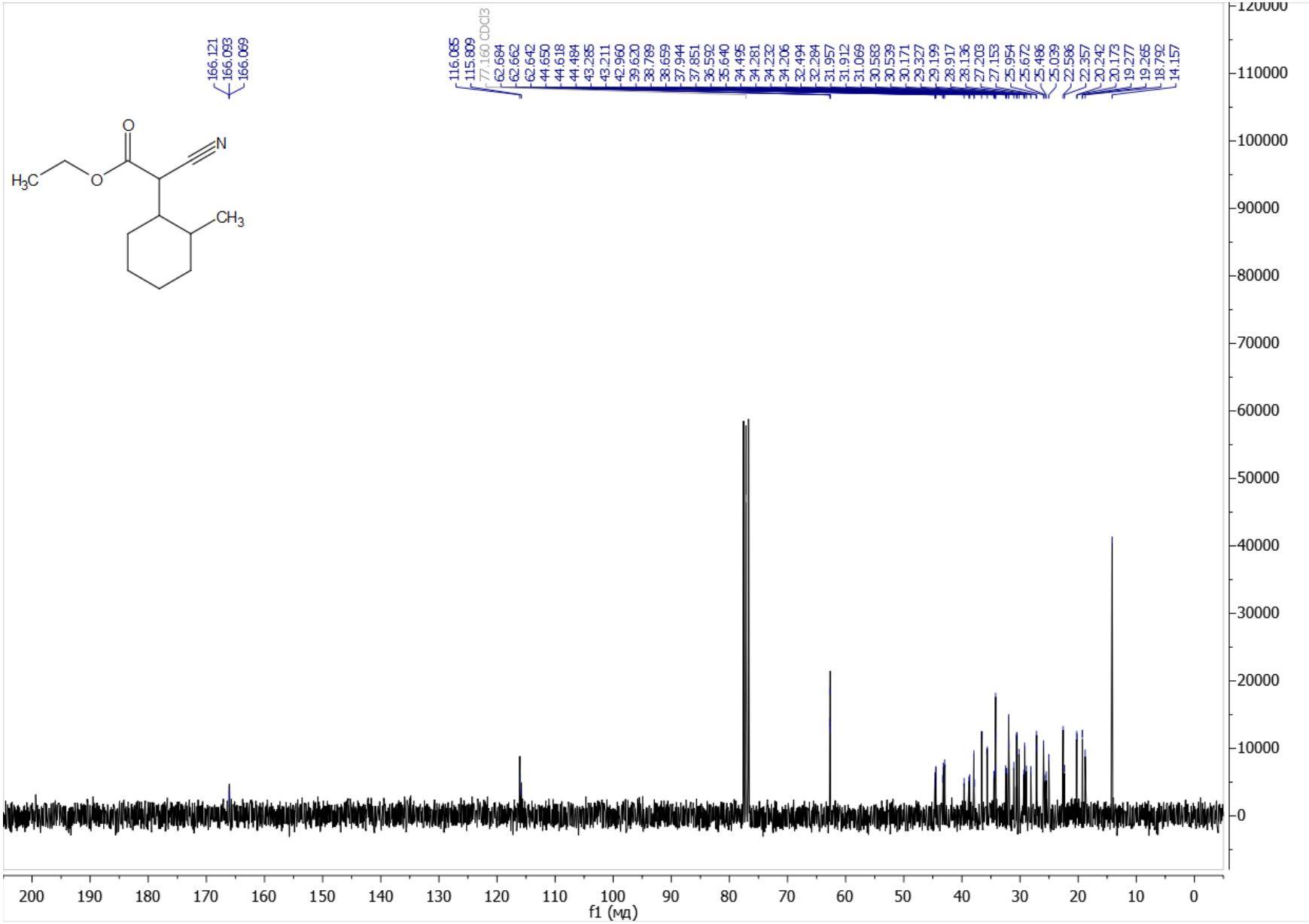
^{13}C NMR (75.48 MHz, $CDCl_3$) spectrum of ethyl 2-cyano-2-cyclooctylacetate, **3ad**



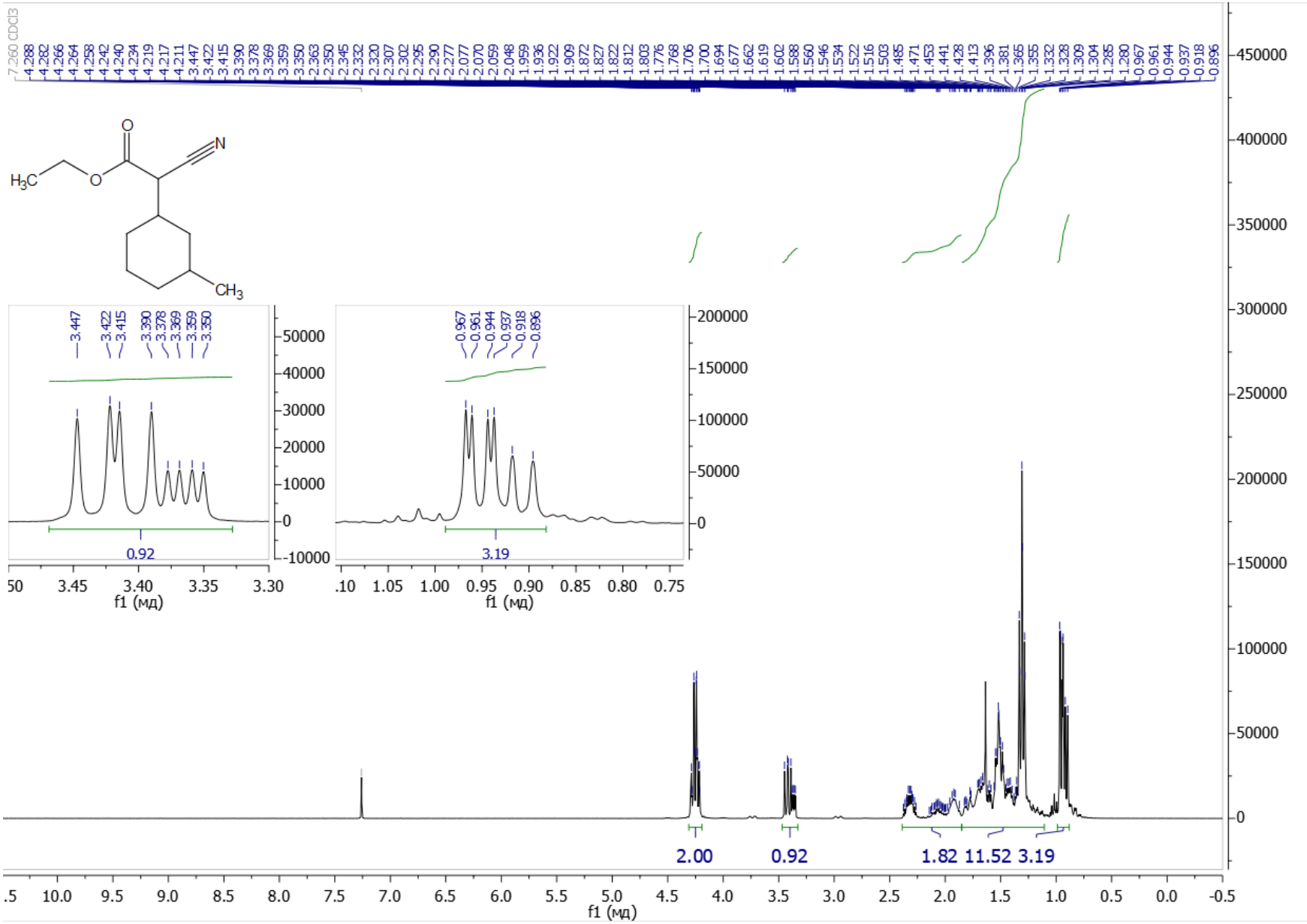
^{1}H NMR (300.13 MHz, $CDCl_3$) spectrum of ethyl 2-cyano-2-(2-methylcyclohexyl)acetate, **3ae**



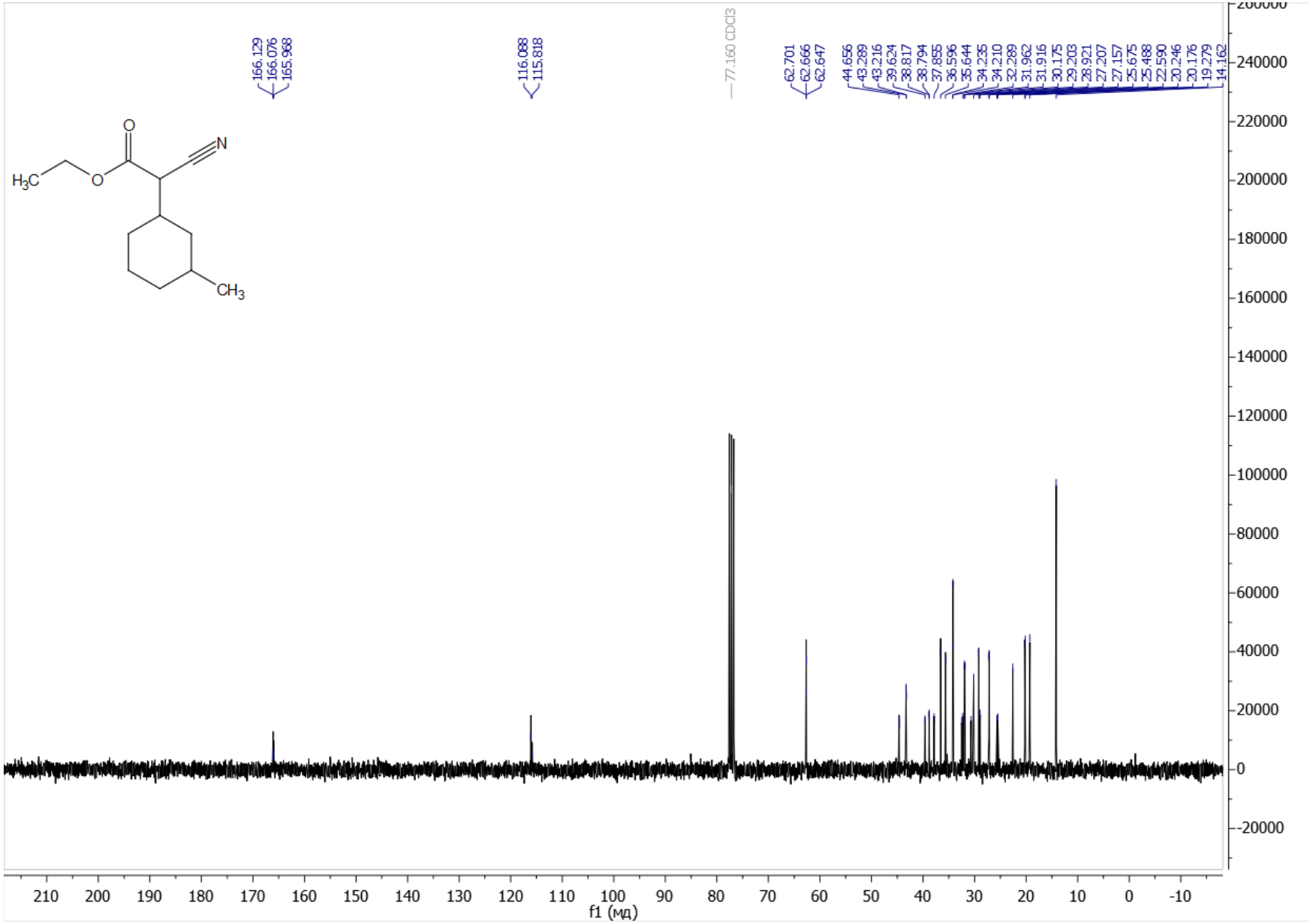
^{13}C NMR (75.48 MHz, $CDCl_3$) spectrum of ethyl 2-cyano-2-(2-methylcyclohexyl)acetate, **3ae**



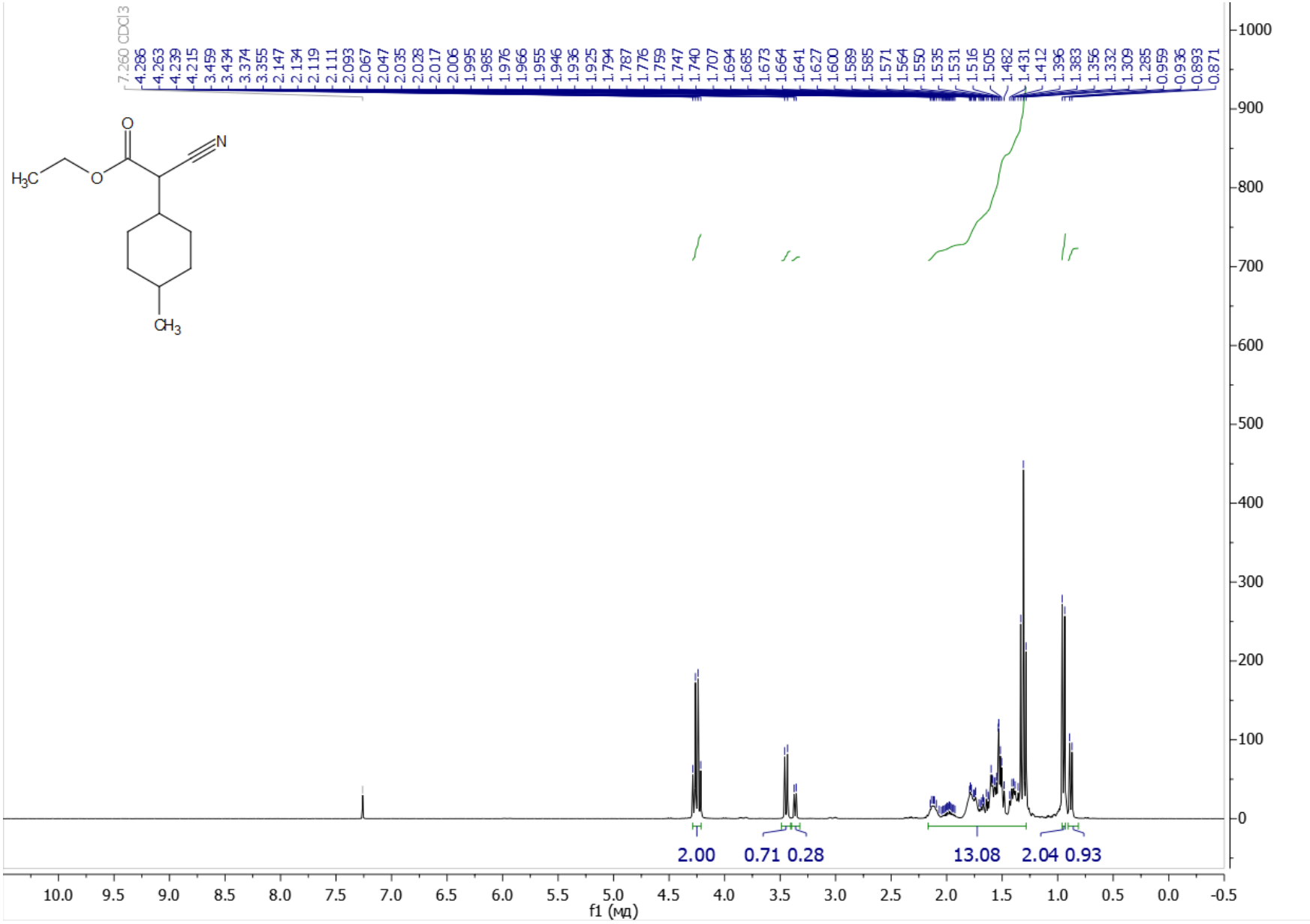
^{1}H NMR (300.13 MHz, $CDCl_3$) spectrum of ethyl 2-cyano-2-(3-methylcyclohexyl)acetate, **3af**



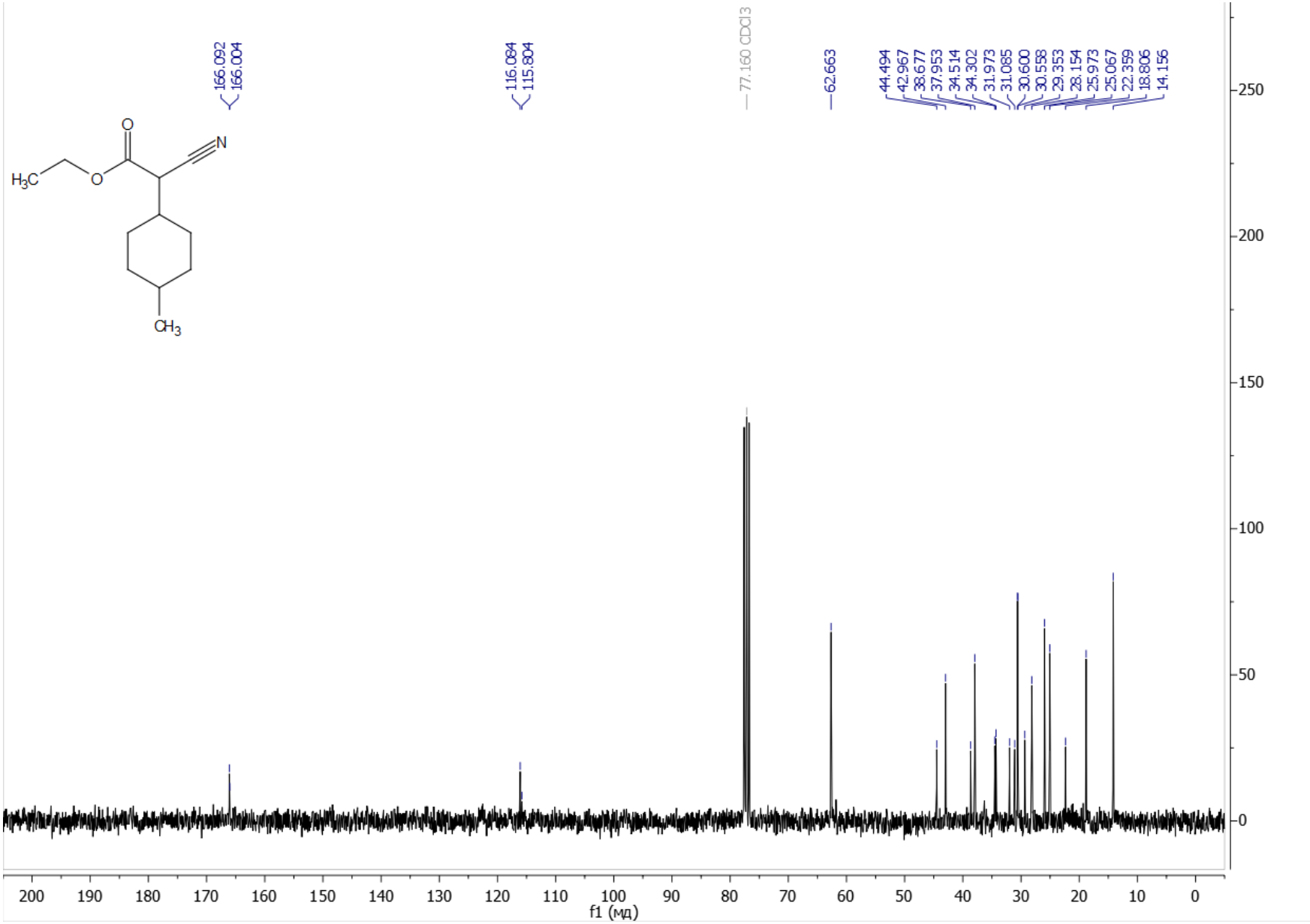
^{13}C NMR (75.48 MHz, $CDCl_3$) spectrum of ethyl 2-cyano-2-(3-methylcyclohexyl)acetate, **3af**



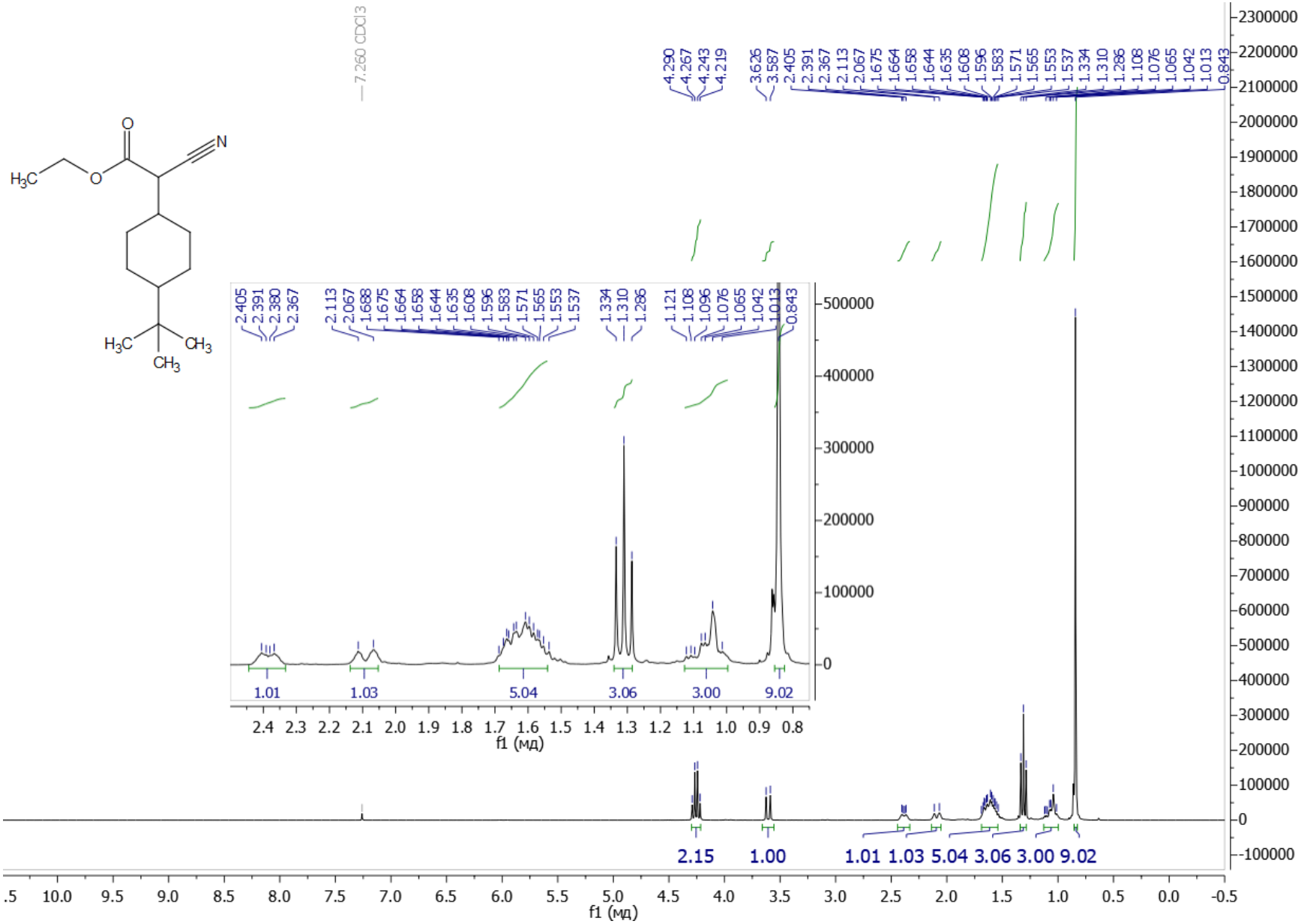
^{1}H NMR (300.13 MHz, $CDCl_3$) spectrum of ethyl 2-cyano-2-(4-methylcyclohexyl)acetate, **3ag**



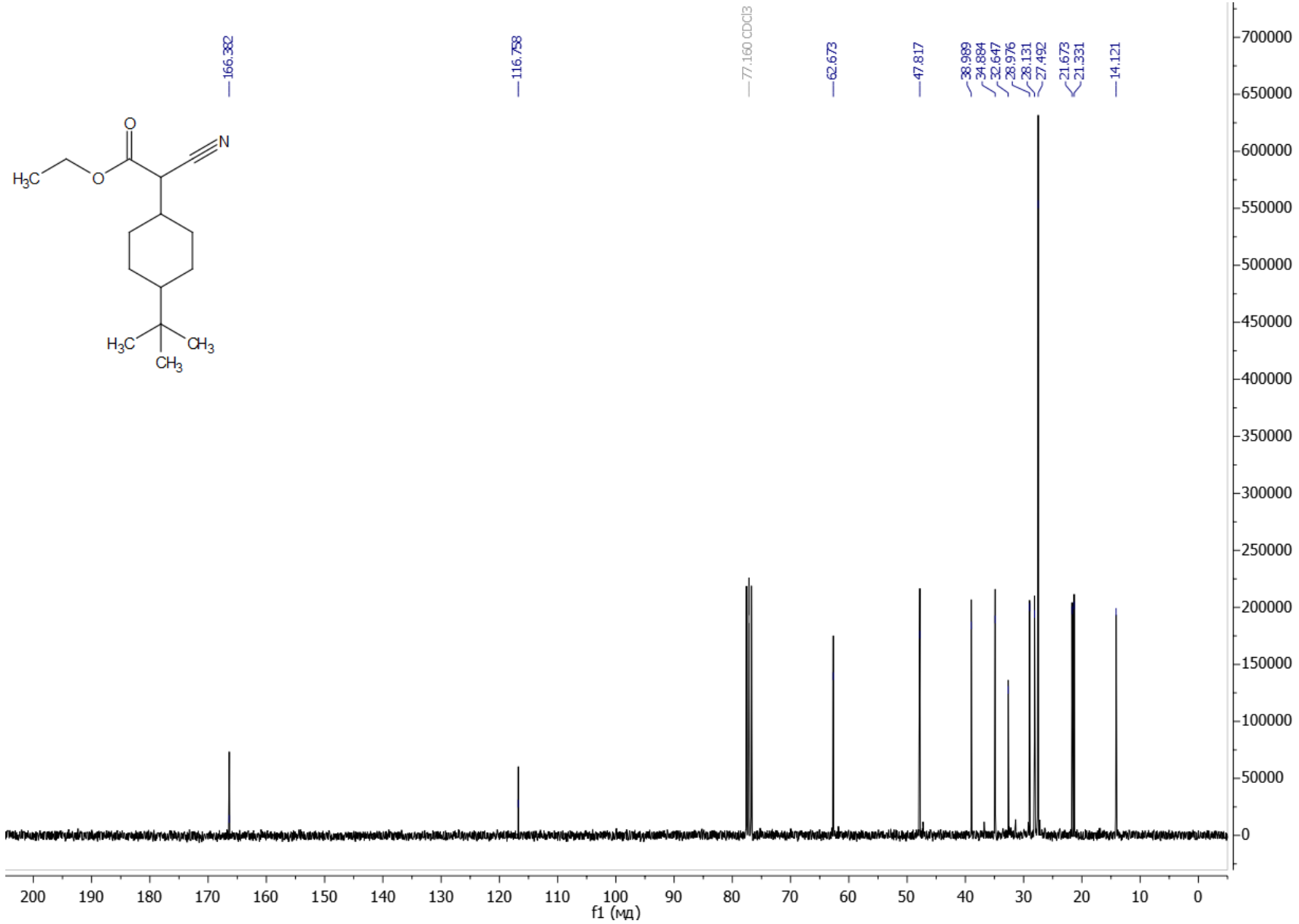
^{13}C NMR (75.48 MHz, $CDCl_3$) spectrum of ethyl 2-cyano-2-(4-methylcyclohexyl)acetate, **3ag**



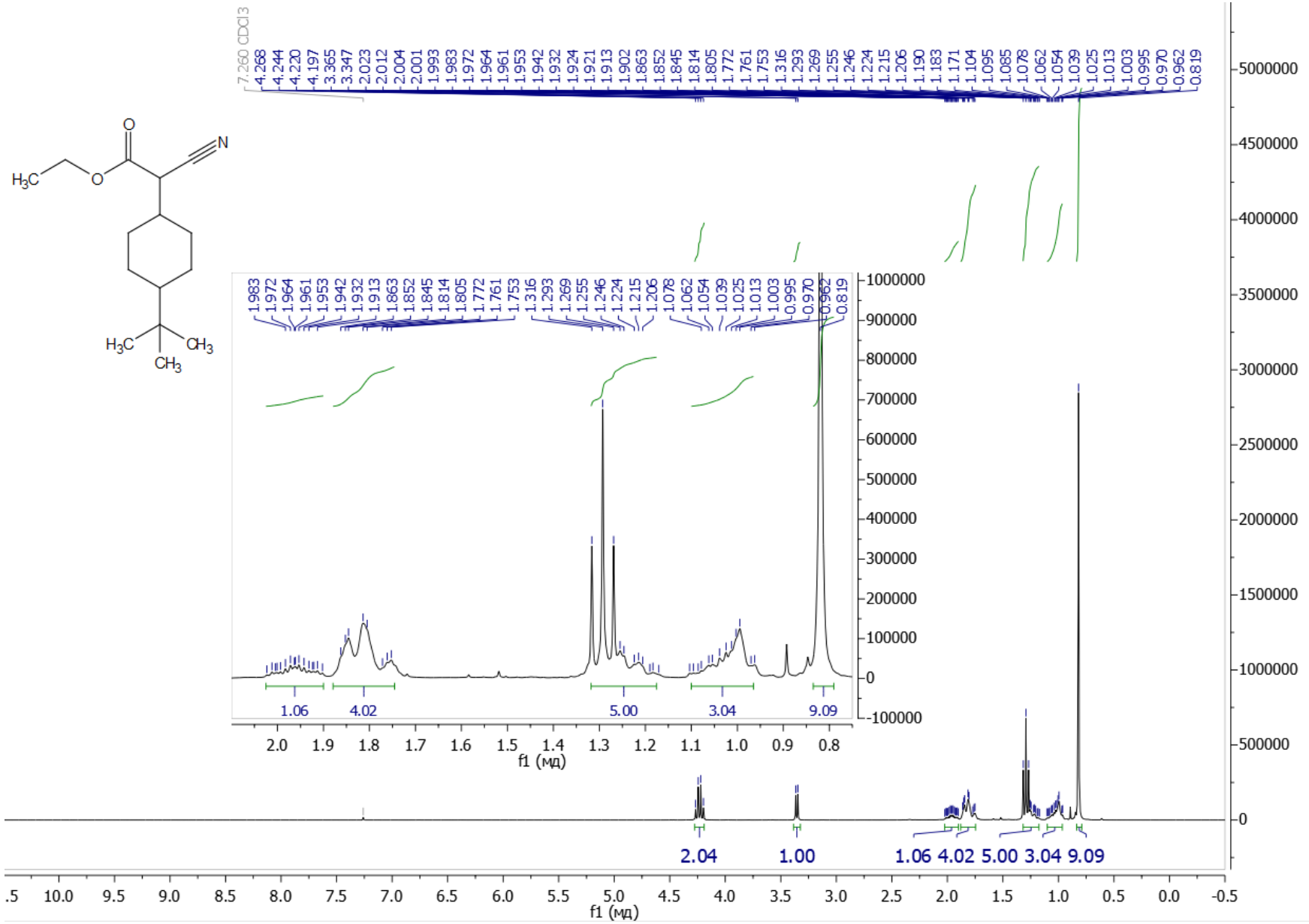
^{1}H NMR (300.13 MHz, $CDCl_3$) spectrum of ethyl 2-(4-(tert-butyl)cyclohexyl)-2-cyanoacetate, **3ah**[a]



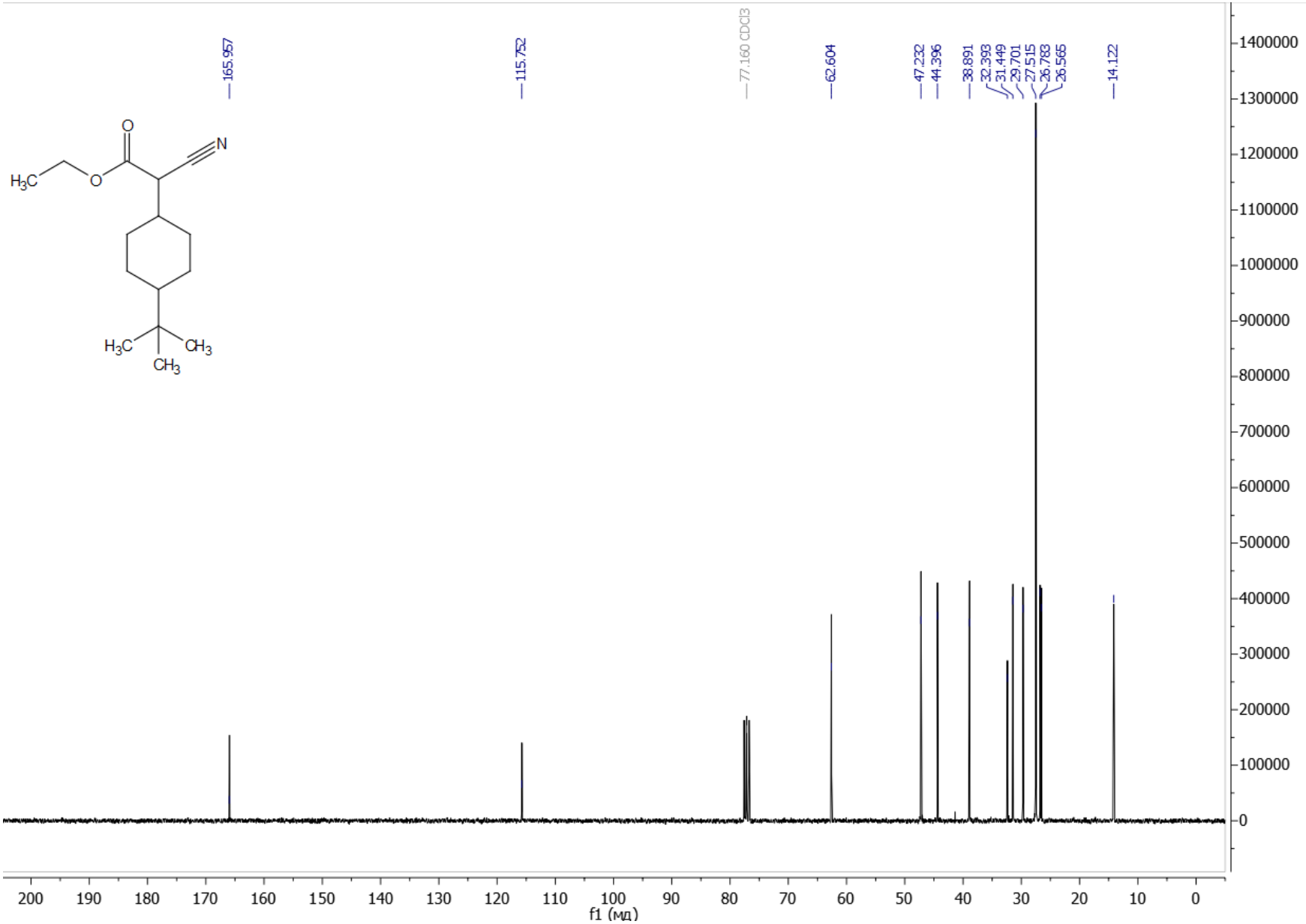
^{13}C NMR (75.48 MHz, $CDCl_3$) spectrum of ethyl 2-(4-(tert-butyl)cyclohexyl)-2-cyanoacetate, **3ah**[a]



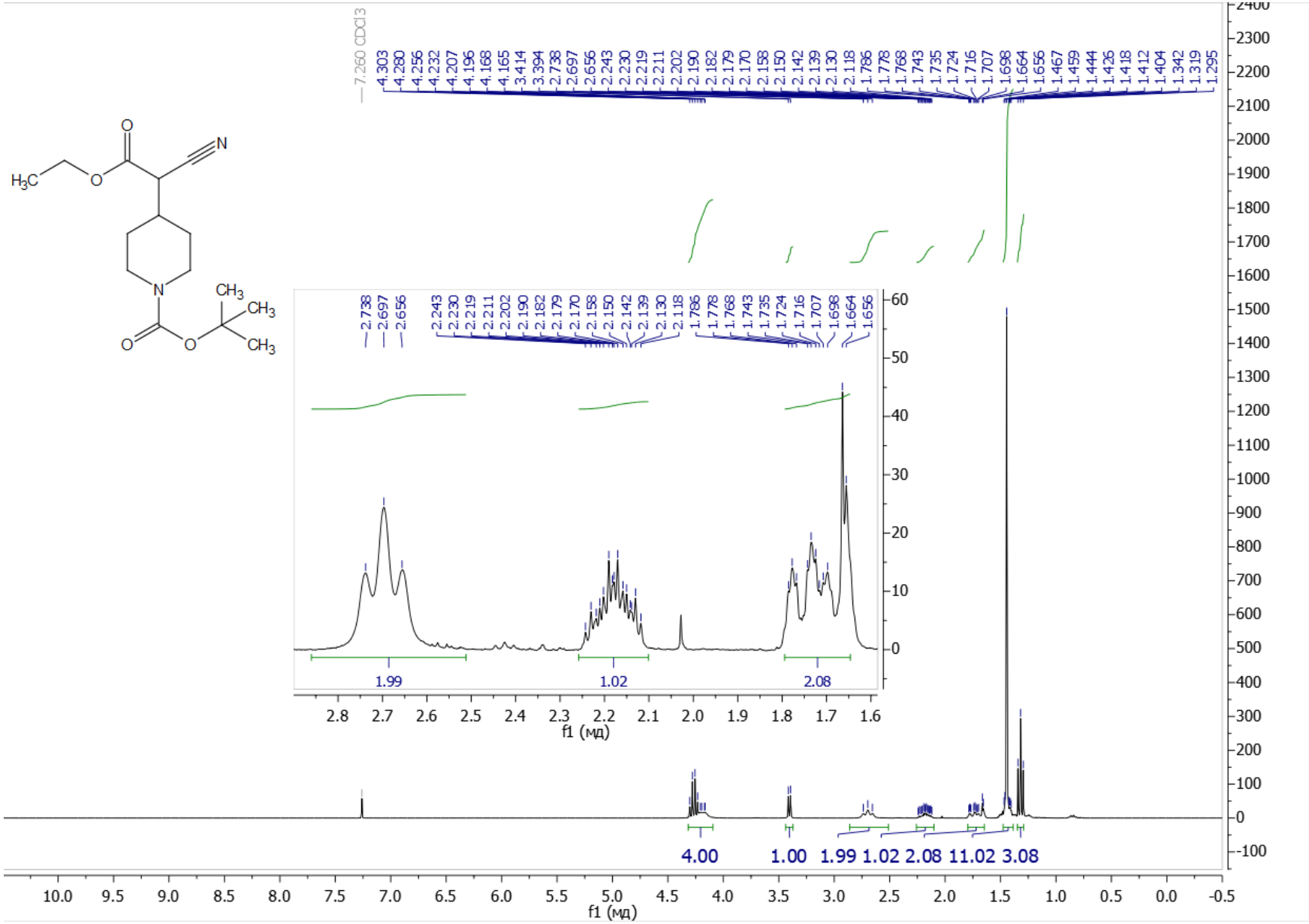
^{1}H NMR (300.13 MHz, $CDCl_3$) spectrum of ethyl 2-(4-(tert-butyl)cyclohexyl)-2-cyanoacetate, **3ah**^{b}



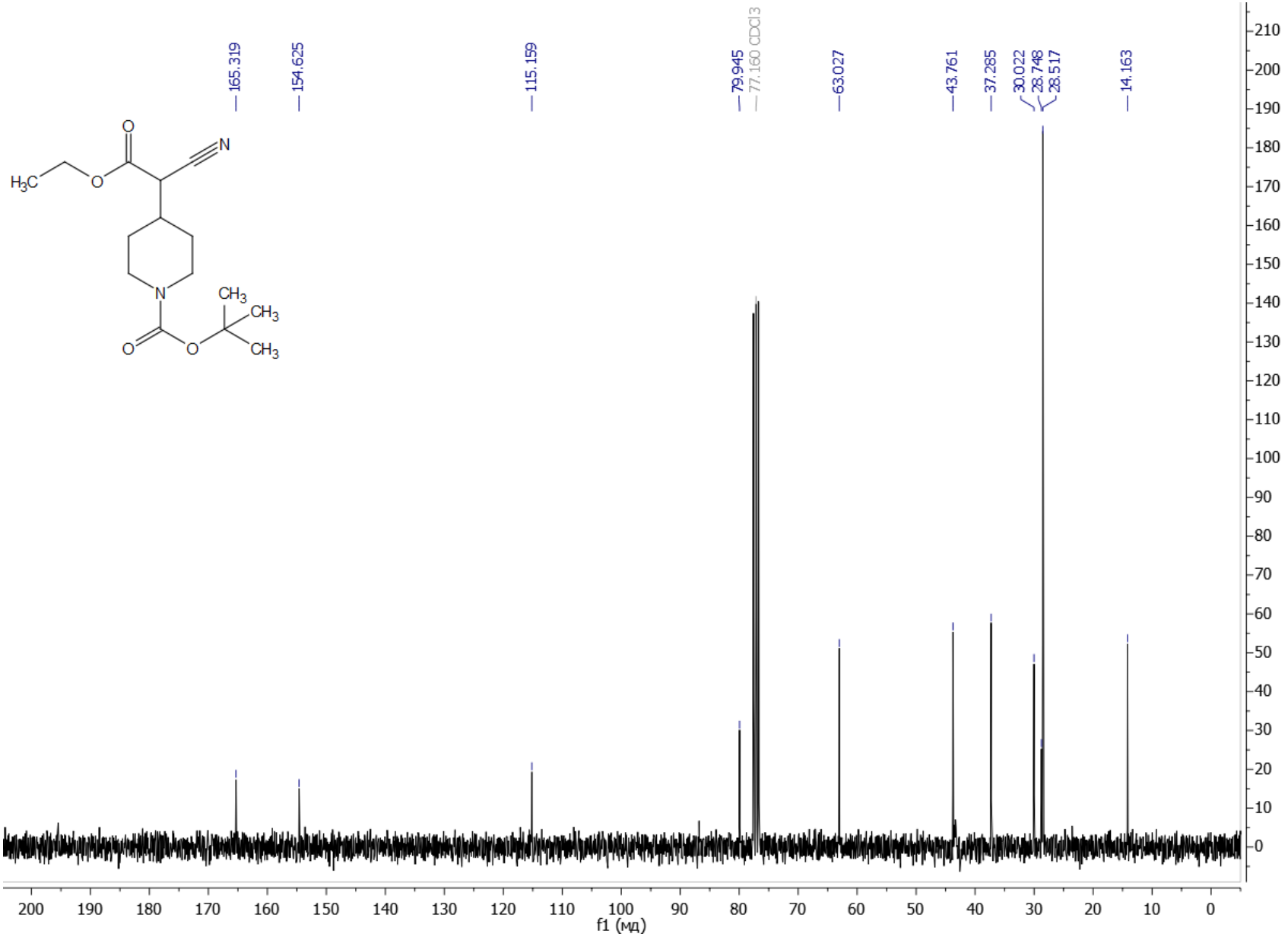
^{13}C NMR (75.48 MHz, $CDCl_3$) spectrum of ethyl 2-(4-(tert-butyl)cyclohexyl)-2-cyanoacetate, **3ah**[b]



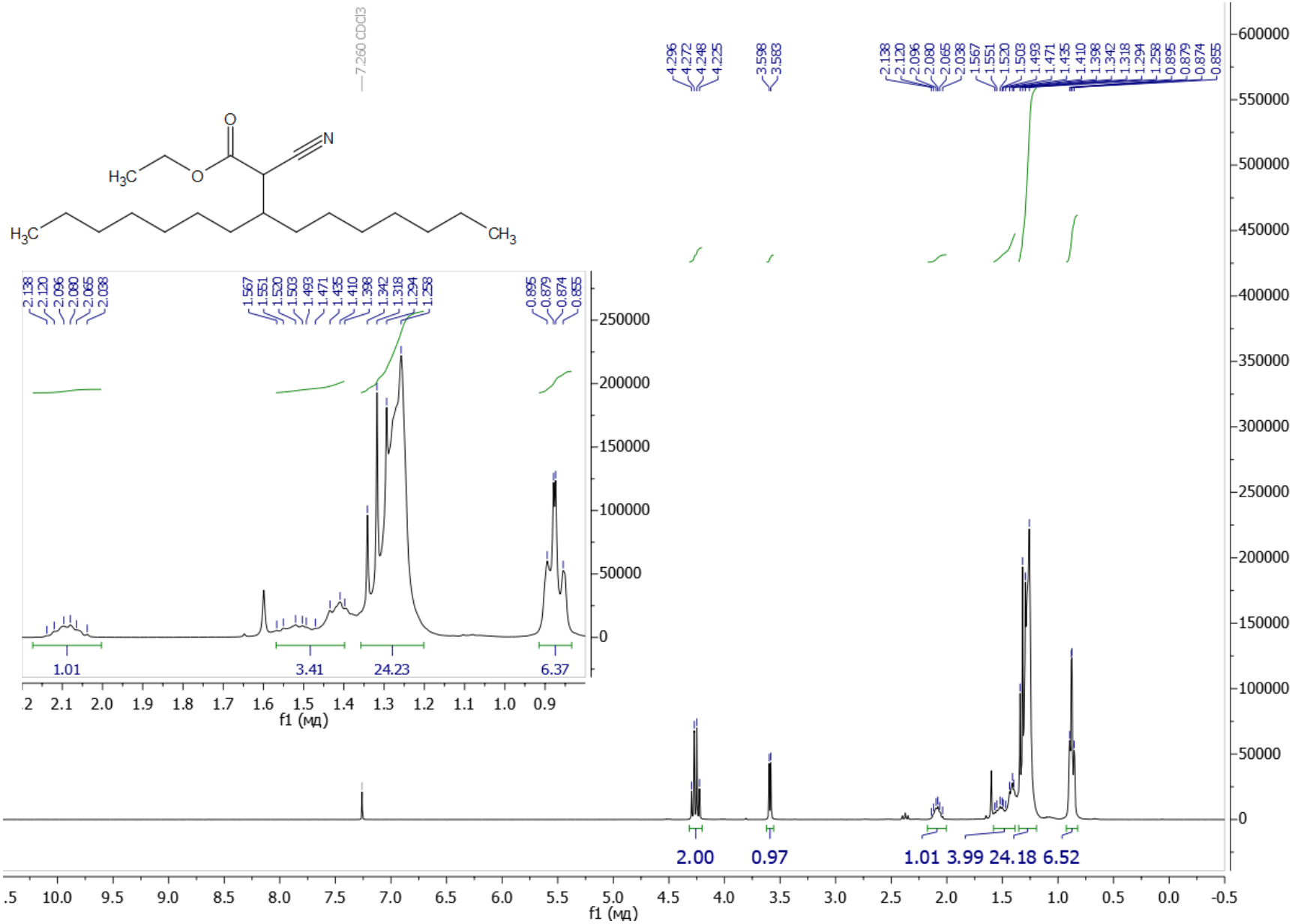
^{1}H NMR (300.13 MHz, $CDCl_3$) spectrum of *tert*-butyl 4-(1-cyano-2-ethoxy-2-oxoethyl)piperidine-1-carboxylate, **3ai**



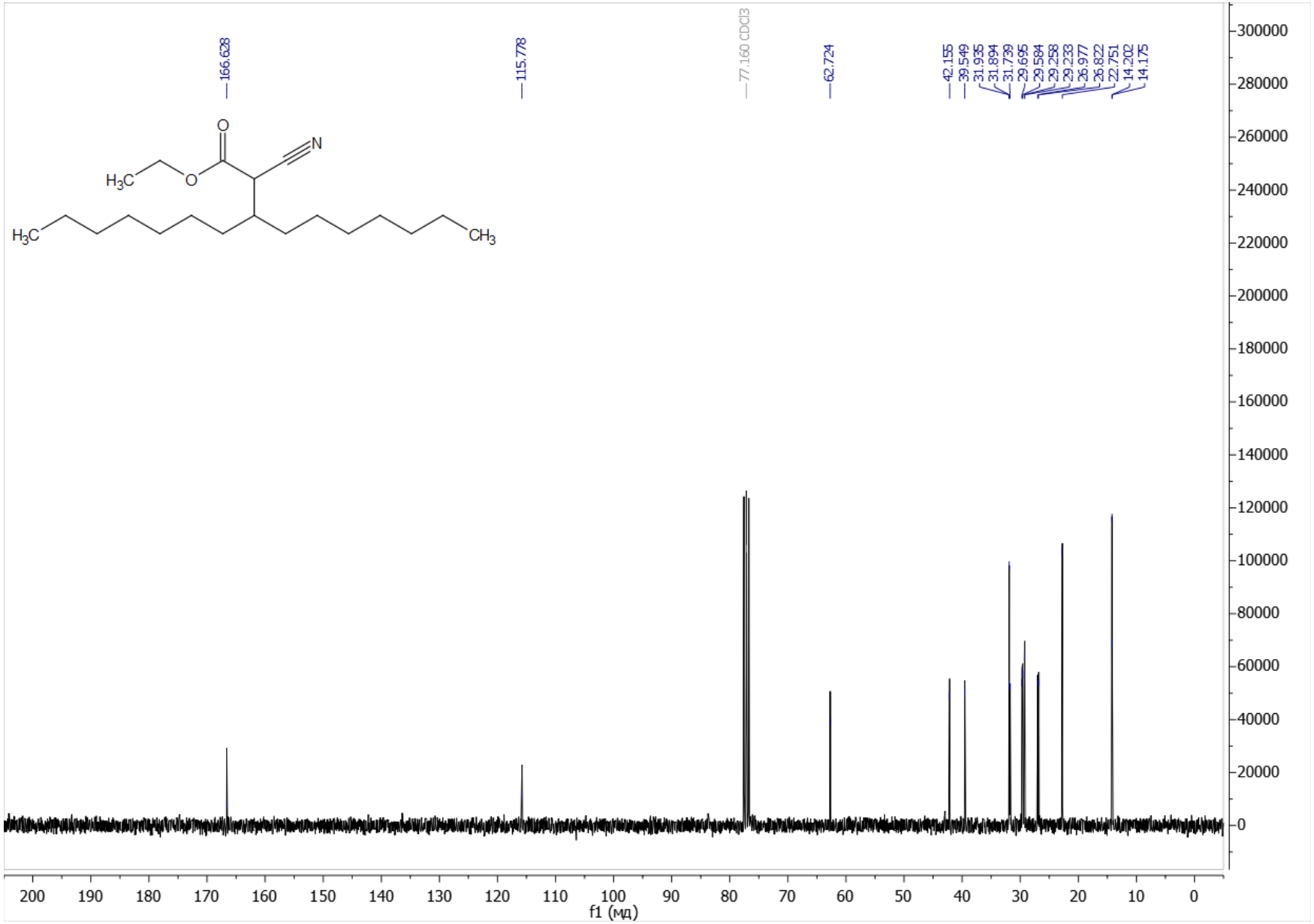
^{13}C NMR (75.48 MHz, $CDCl_3$) spectrum of *tert*-butyl 4-(1-cyano-2-ethoxy-2-oxoethyl)piperidine-1-carboxylate, **3ai**



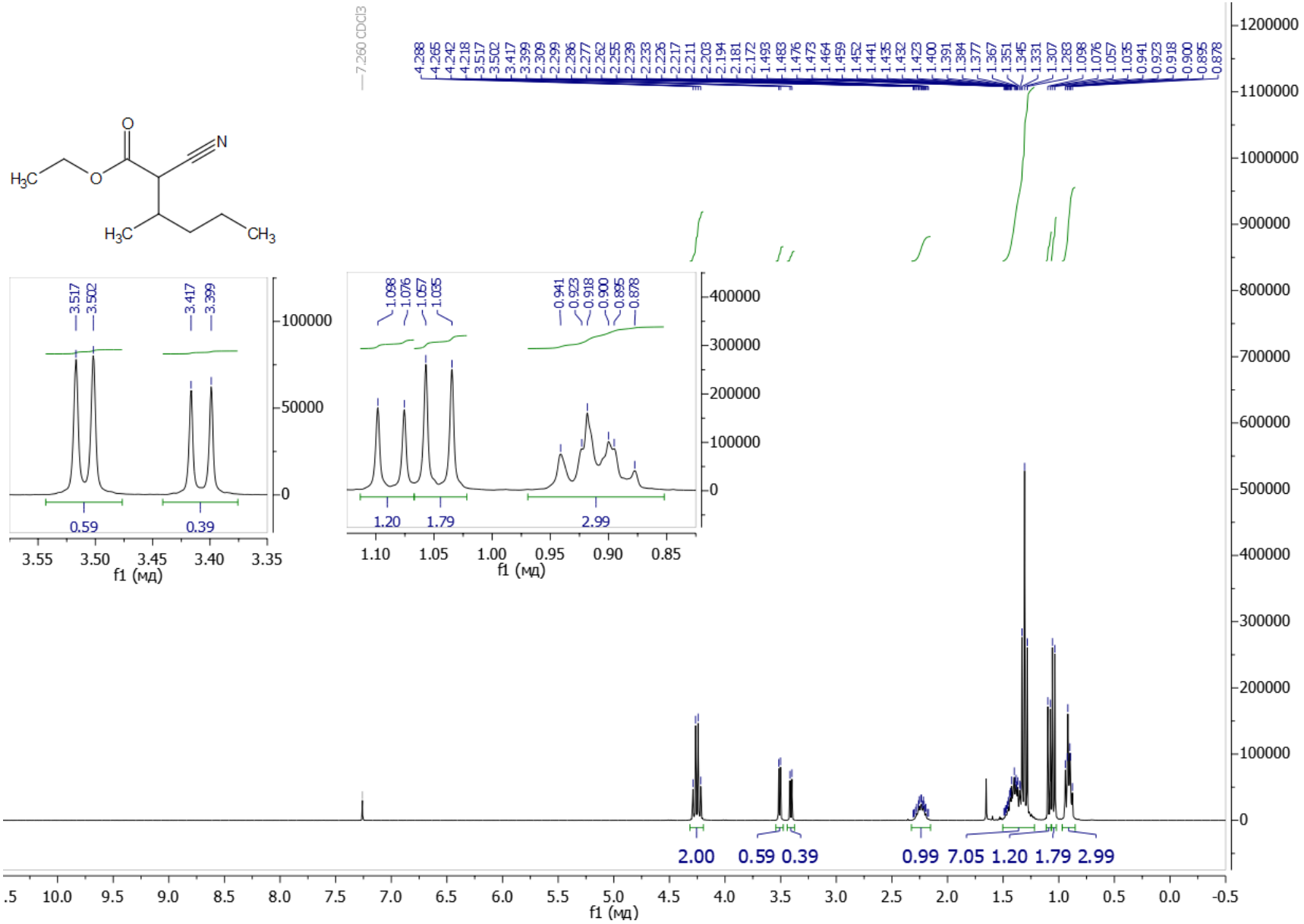
^{1}H NMR (300.13 MHz, $CDCl_3$) spectrum of ethyl 2-cyano-3-heptyldecanoate, **3aj**



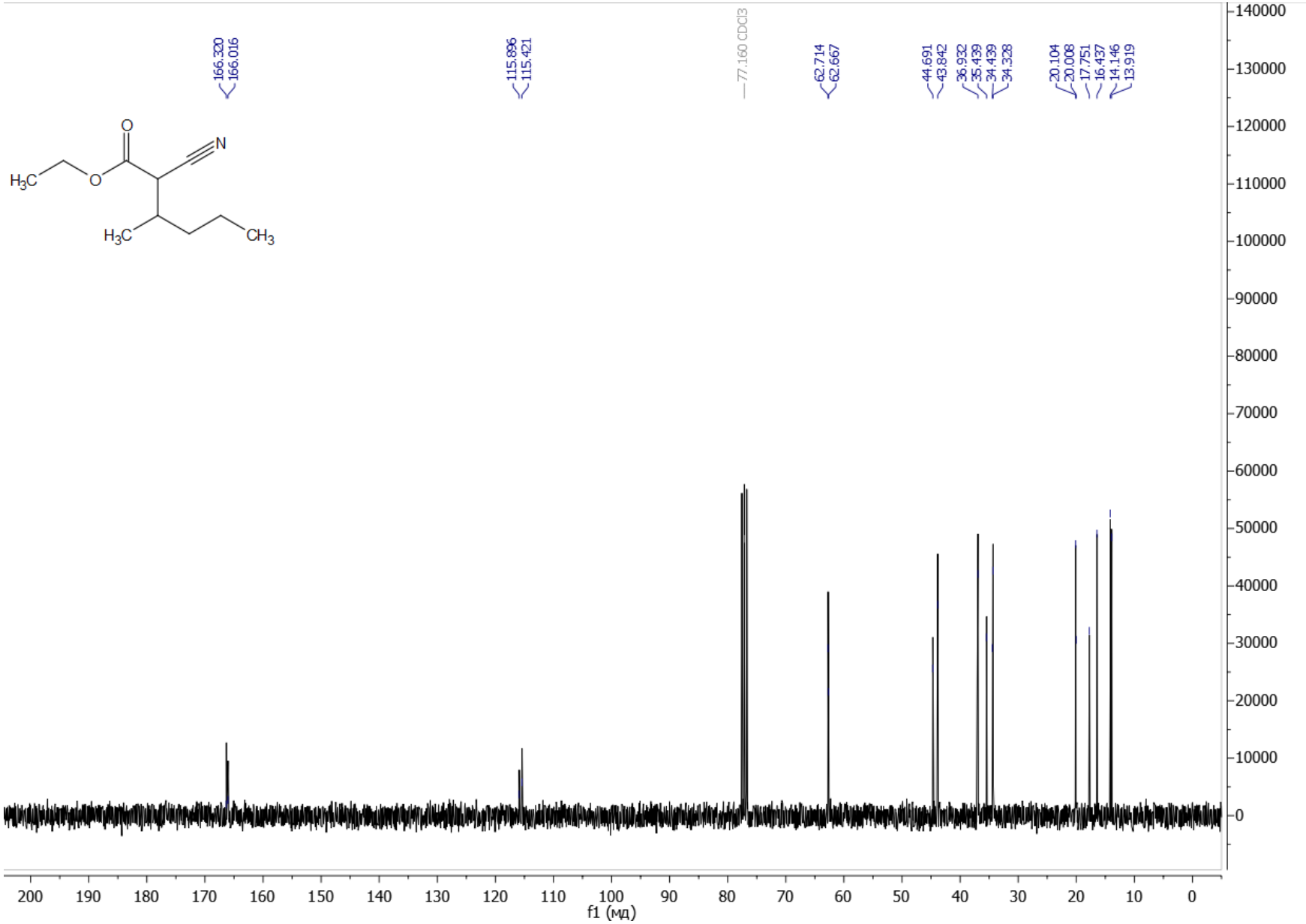
^{13}C NMR (75.48 MHz, $CDCl_3$) spectrum of ethyl 2-cyano-3-heptyldecanoate, **3aj**



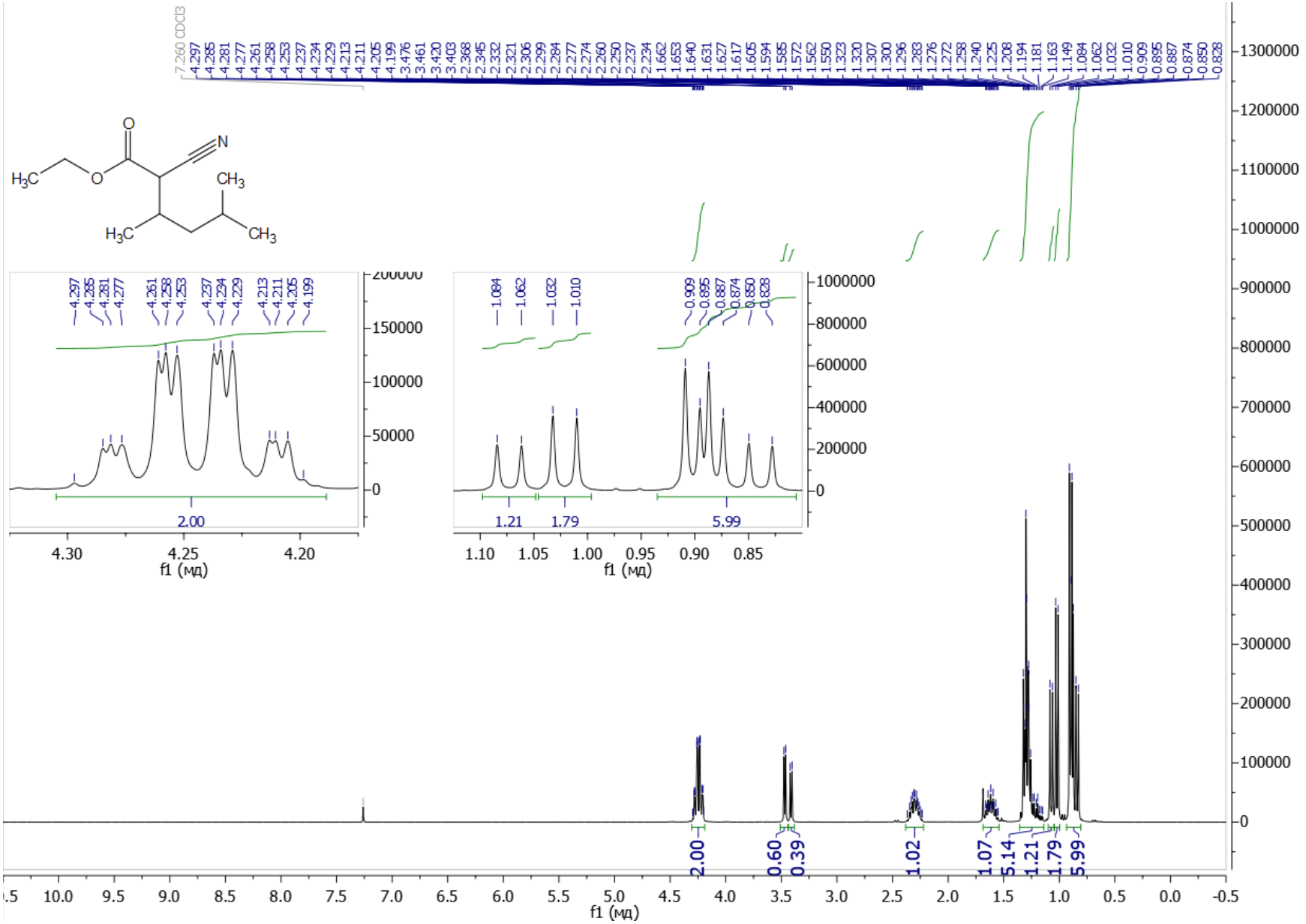
^{1}H NMR (300.13 MHz, $CDCl_3$) spectrum of ethyl 2-cyano-3-methylhexanoate, **3ak**



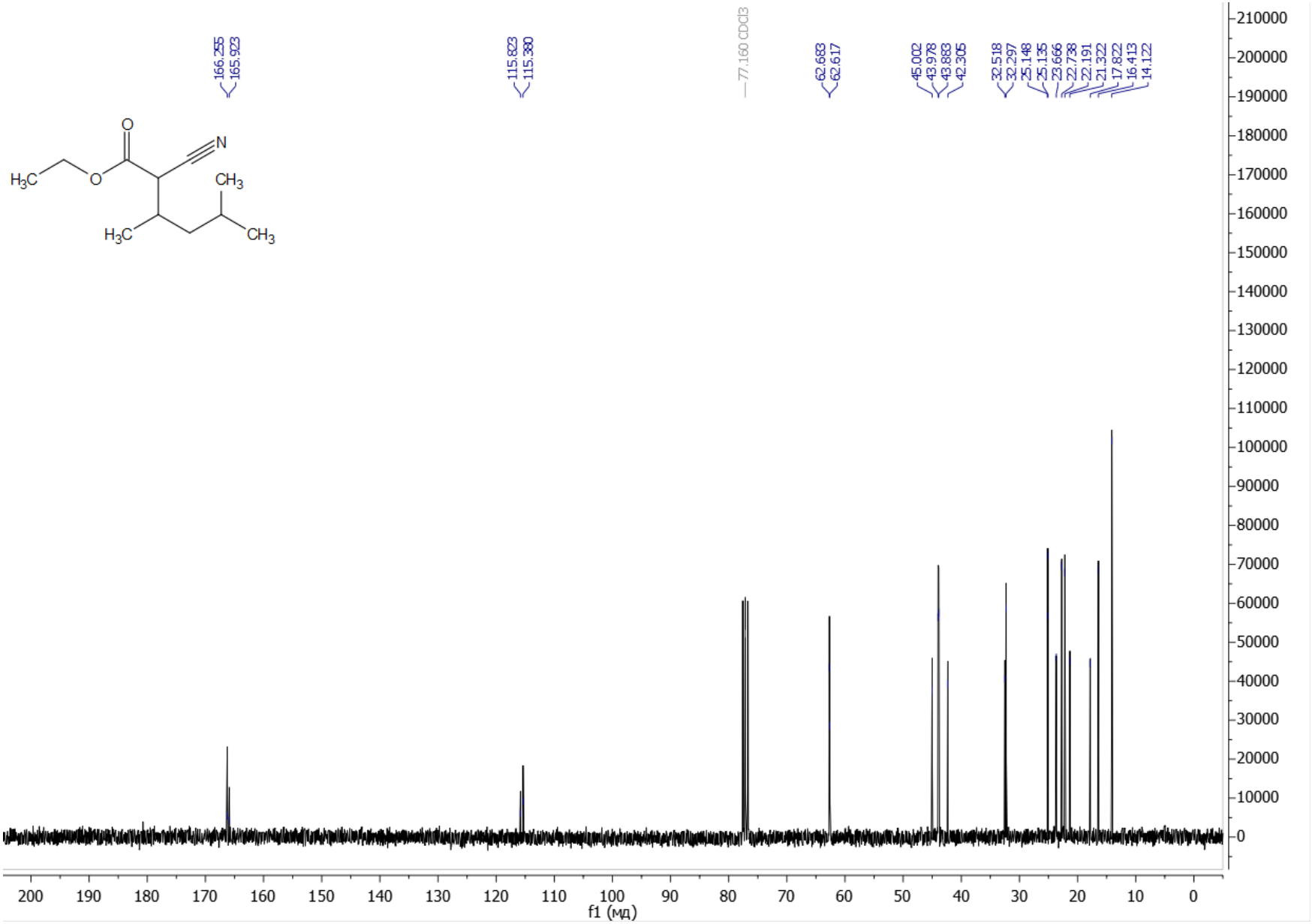
^{13}C NMR (75.48 MHz, $CDCl_3$) spectrum of ethyl 2-cyano-3-methylhexanoate, **3ak**



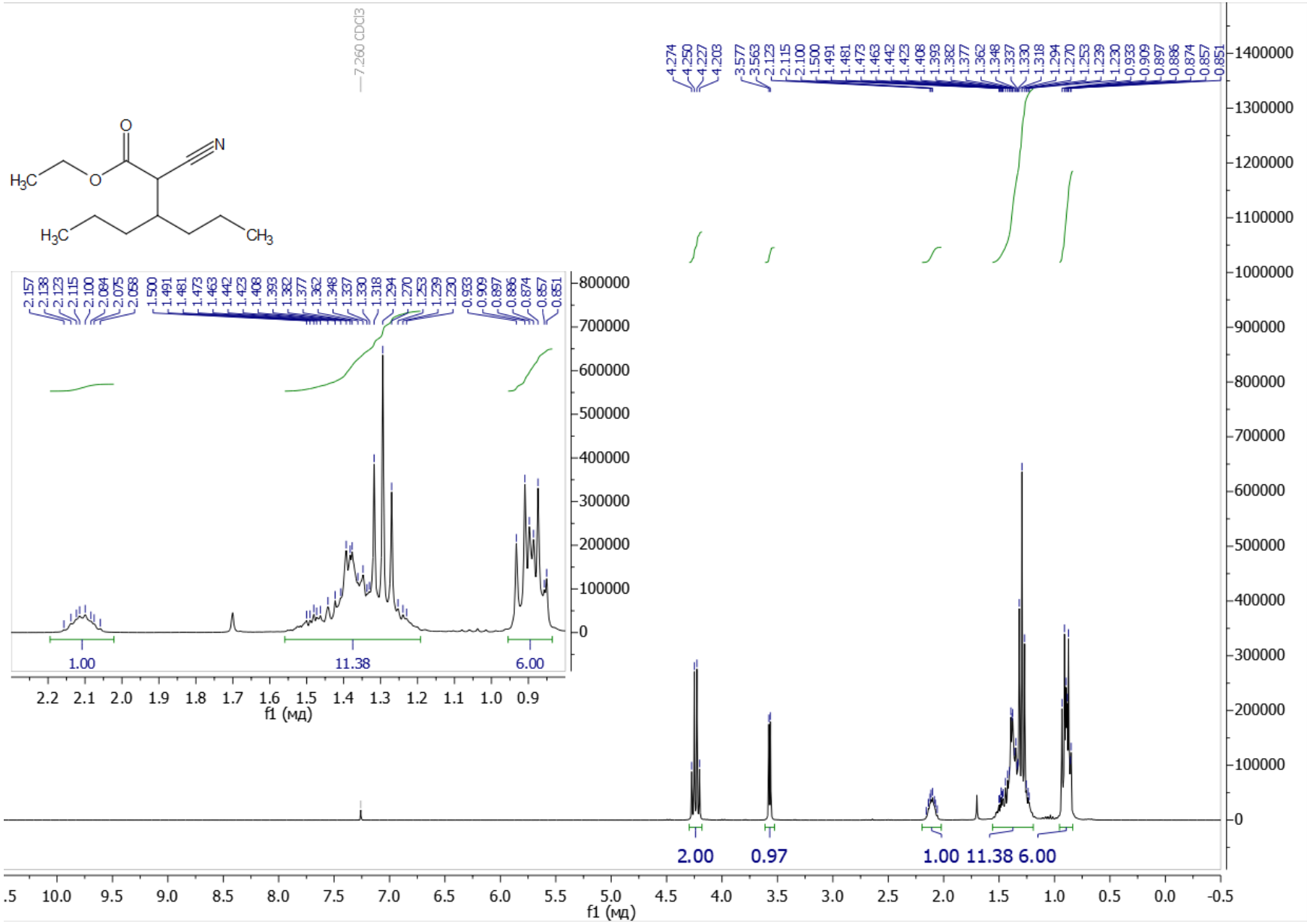
^{1}H NMR (300.13 MHz, $CDCl_3$) spectrum of ethyl 2-cyano-3,5-dimethylhexanoate, **3al**



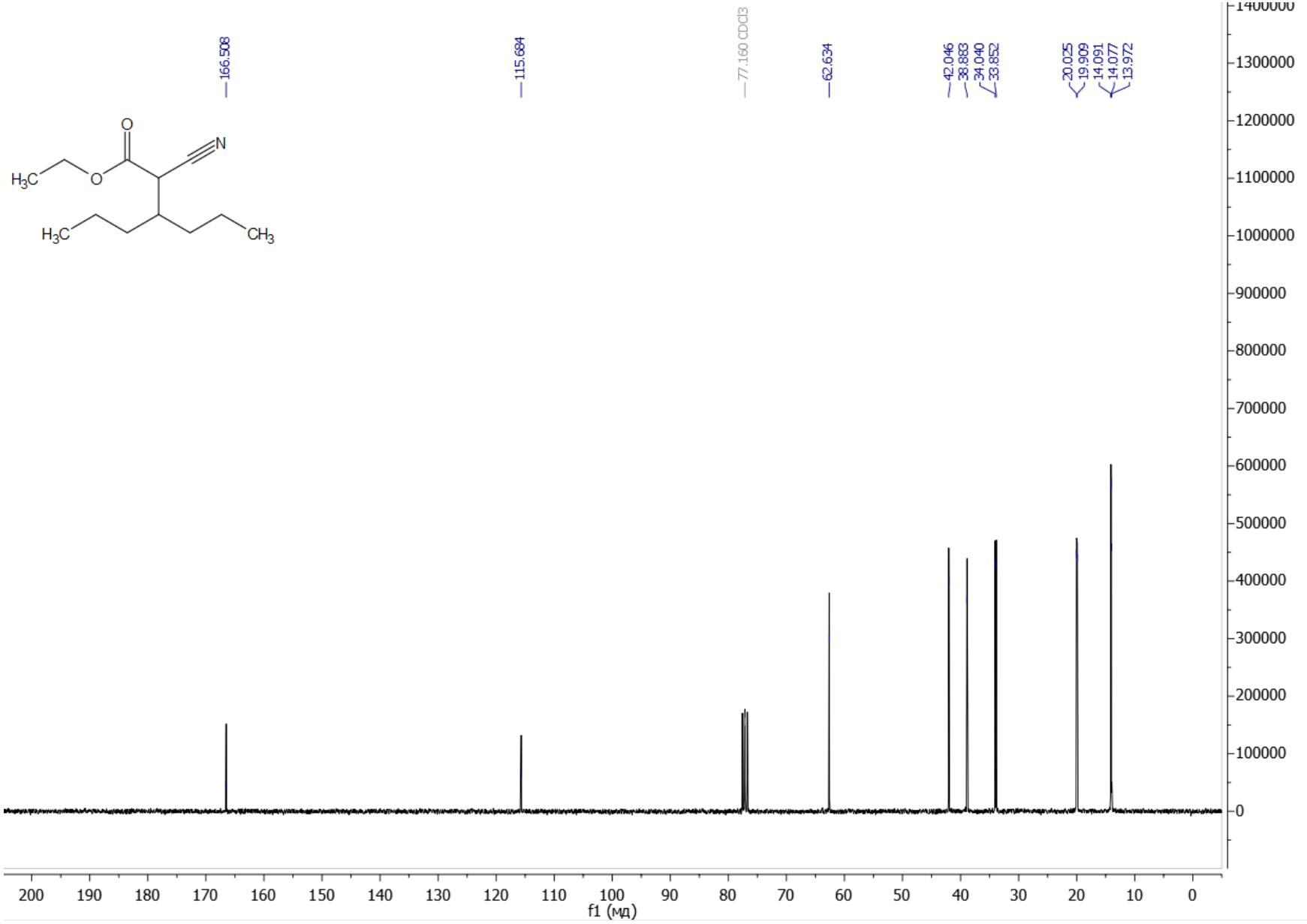
^{13}C NMR (75.48 MHz, $CDCl_3$) spectrum of ethyl 2-cyano-3,5-dimethylhexanoate, **3al**



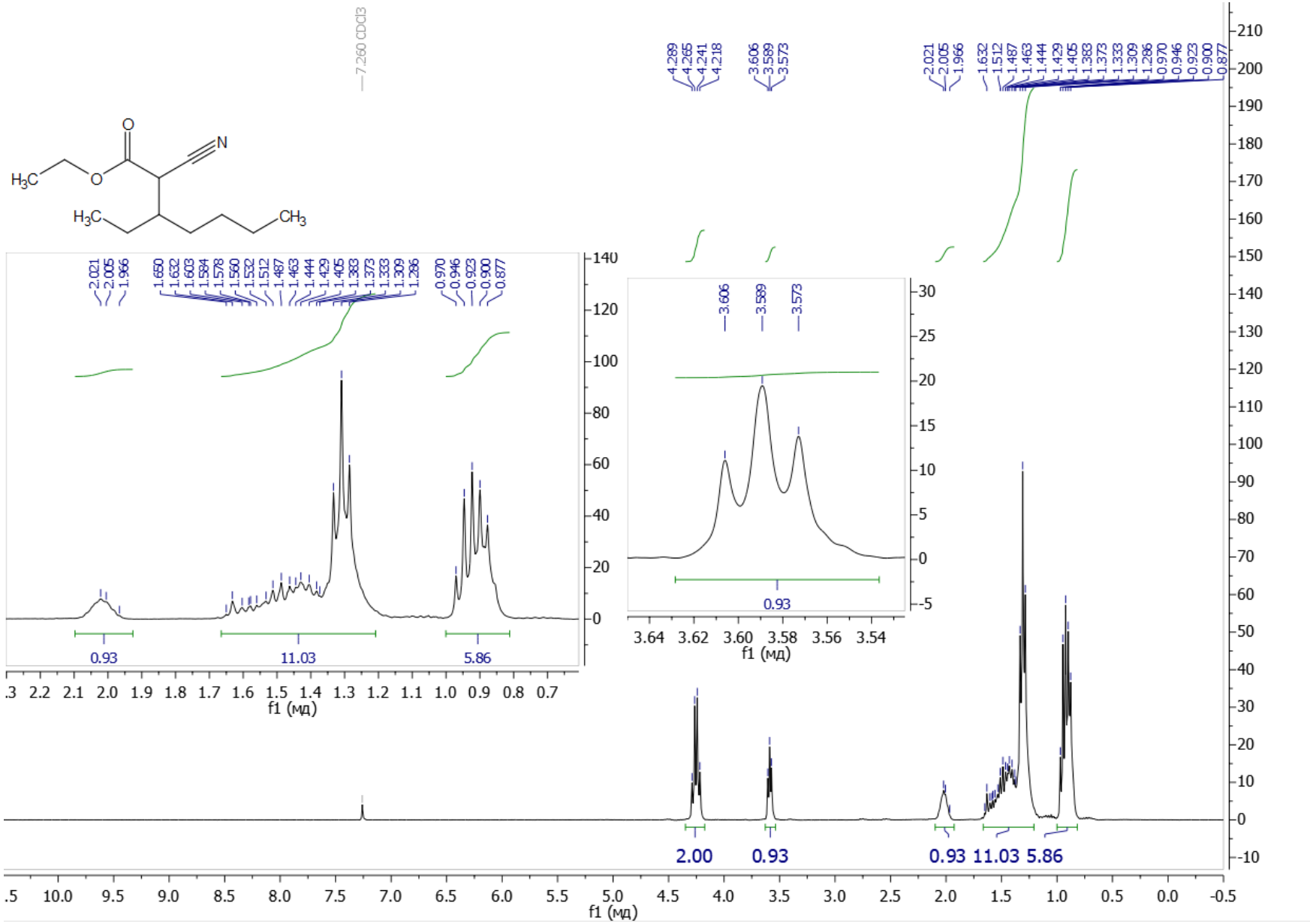
^{1}H NMR (300.13 MHz, $CDCl_3$) spectrum of ethyl 2-cyano-3-propylhexanoate, **3am**



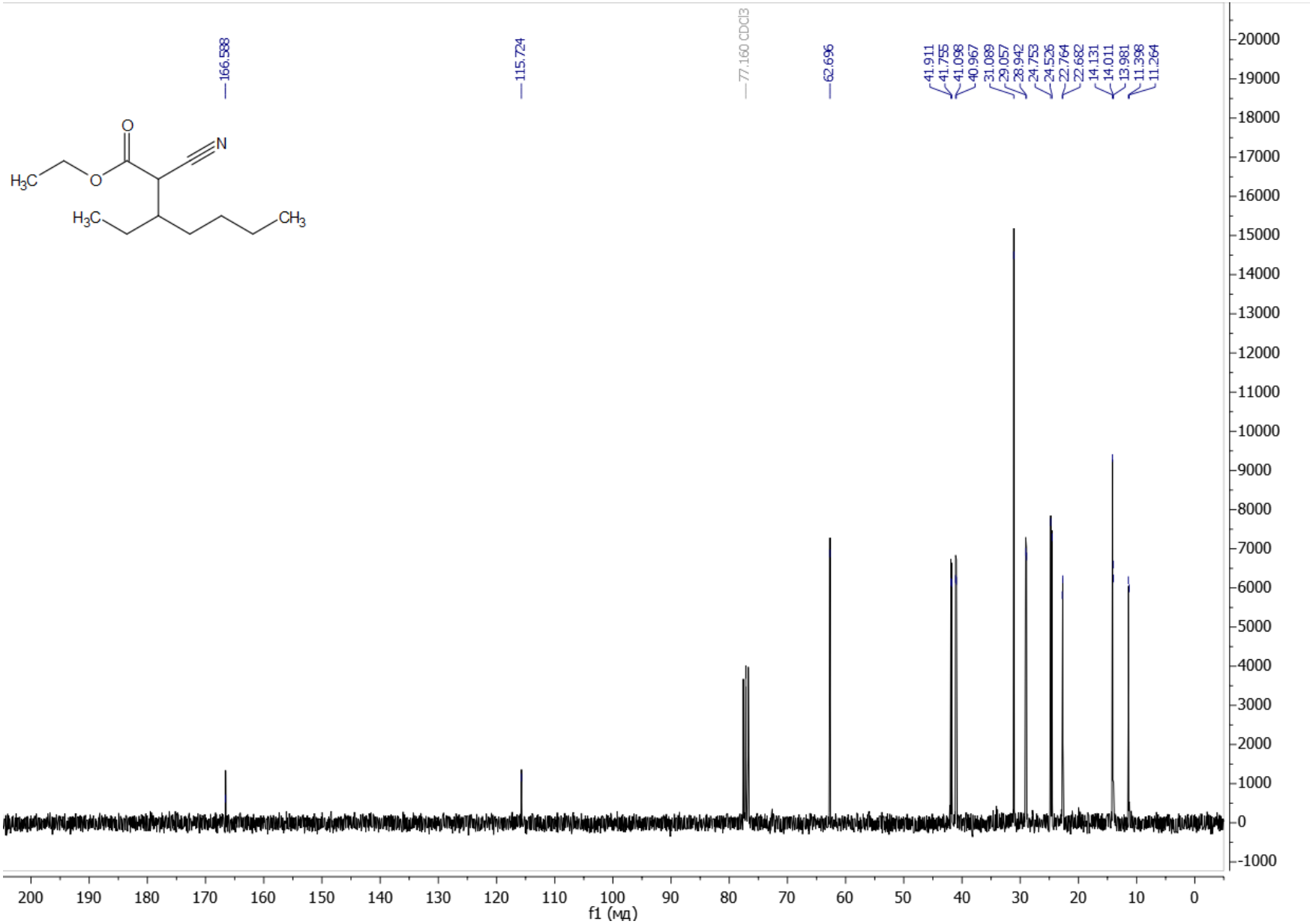
^{13}C NMR (75.48 MHz, $CDCl_3$) spectrum of ethyl 2-cyano-3-propylhexanoate, **3am**



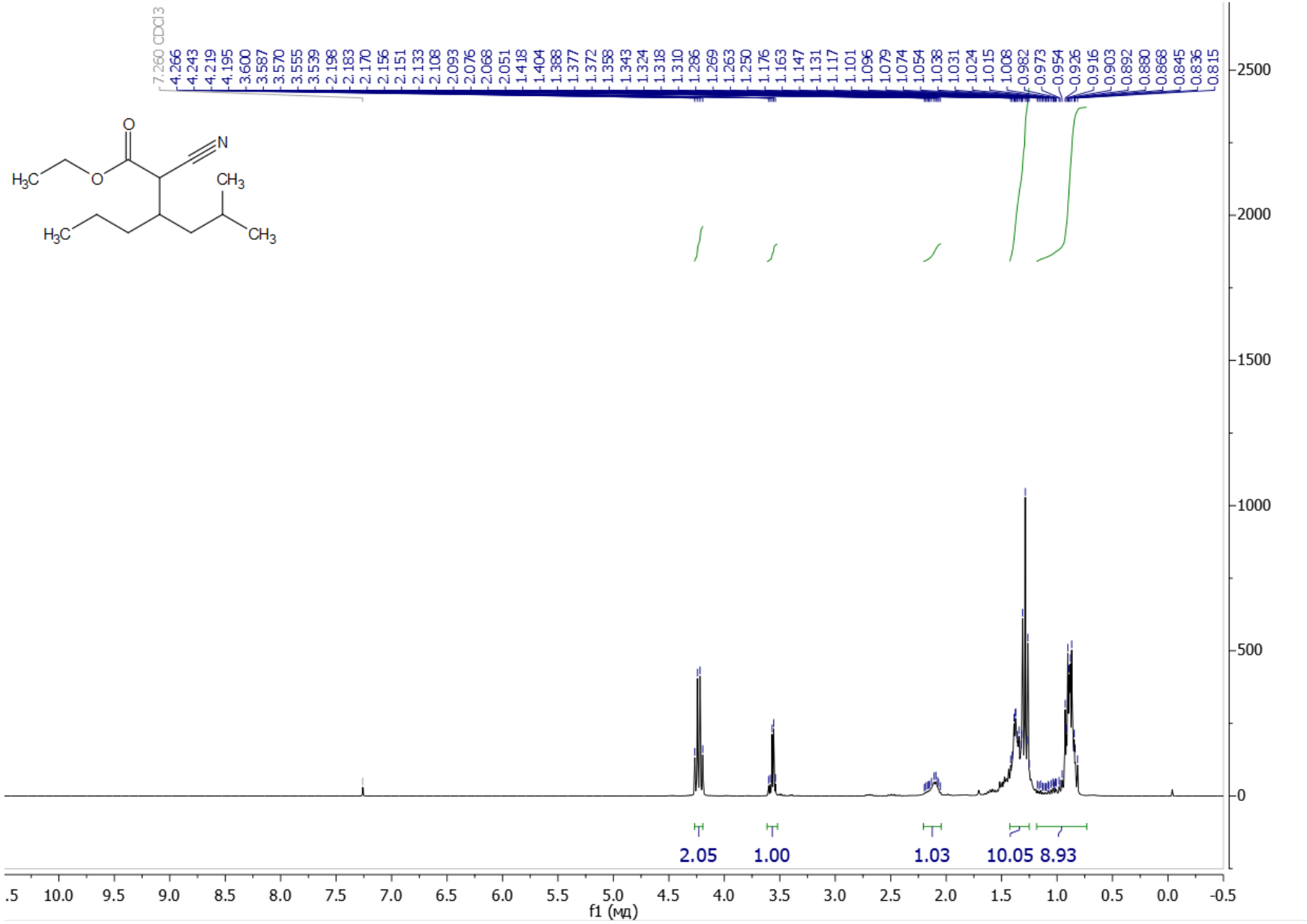
^{1}H NMR (300.13 MHz, $CDCl_3$) spectrum of ethyl 2-cyano-3-ethylheptanoate, **3an**



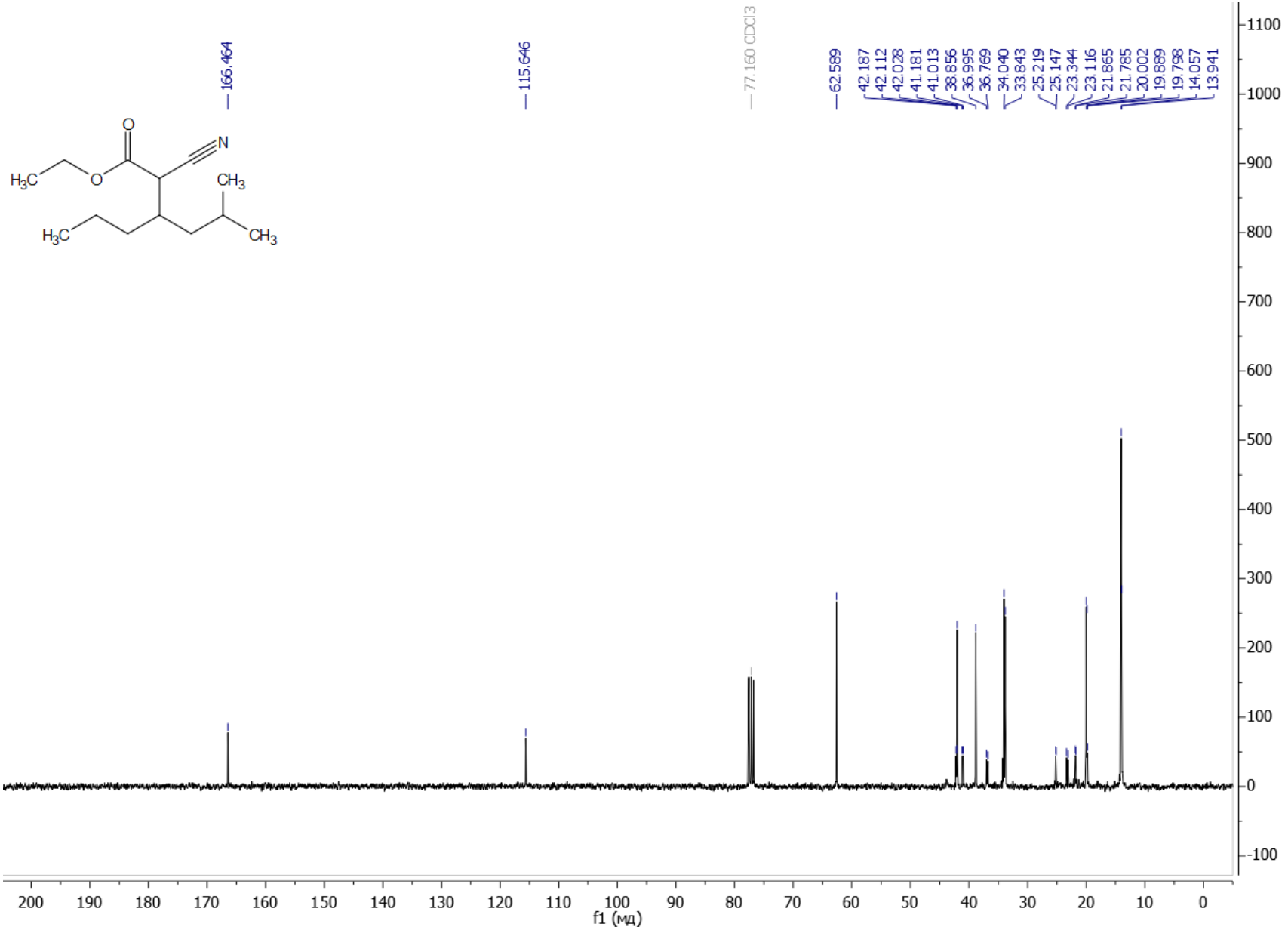
^{13}C NMR (75.48 MHz, $CDCl_3$) spectrum of ethyl 2-cyano-3-ethylheptanoate, **3an**



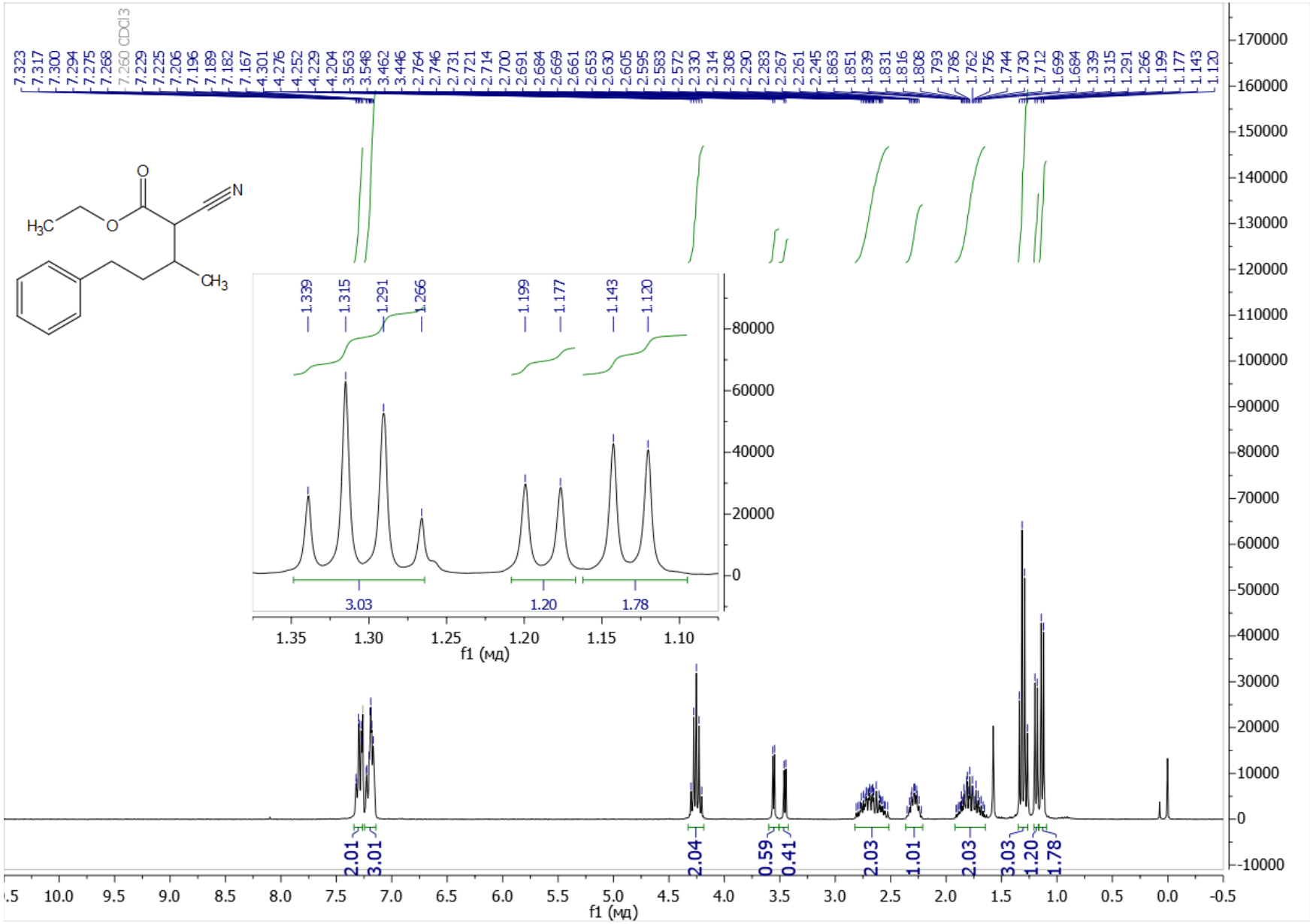
^{1}H NMR (300.13 MHz, $CDCl_3$) spectrum of ethyl 2-cyano-5-methyl-3-propylhexanoate, **3ao**



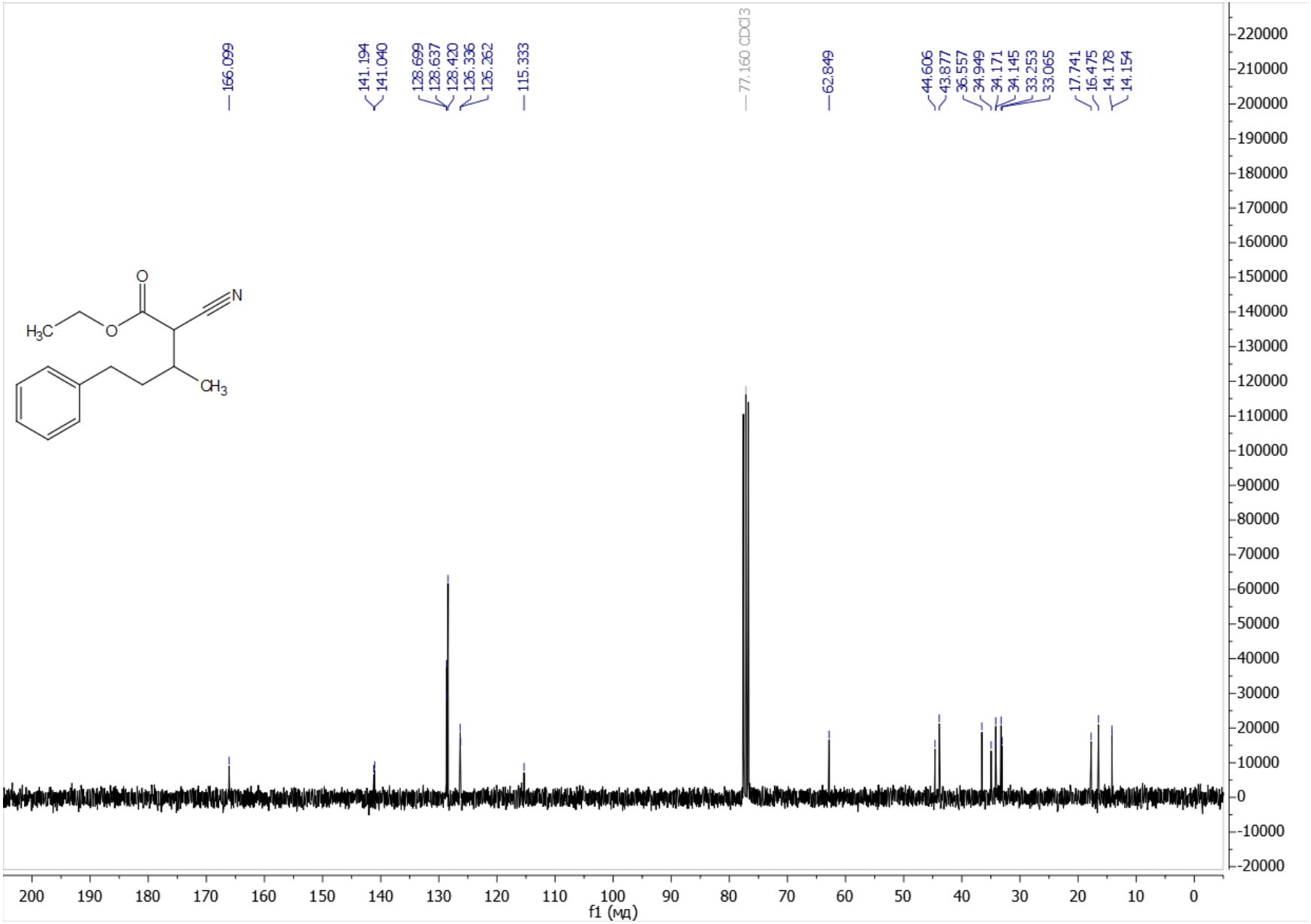
^{13}C NMR (75.48 MHz, $CDCl_3$) spectrum of ethyl 2-cyano-5-methyl-3-propylhexanoate, **3ao**



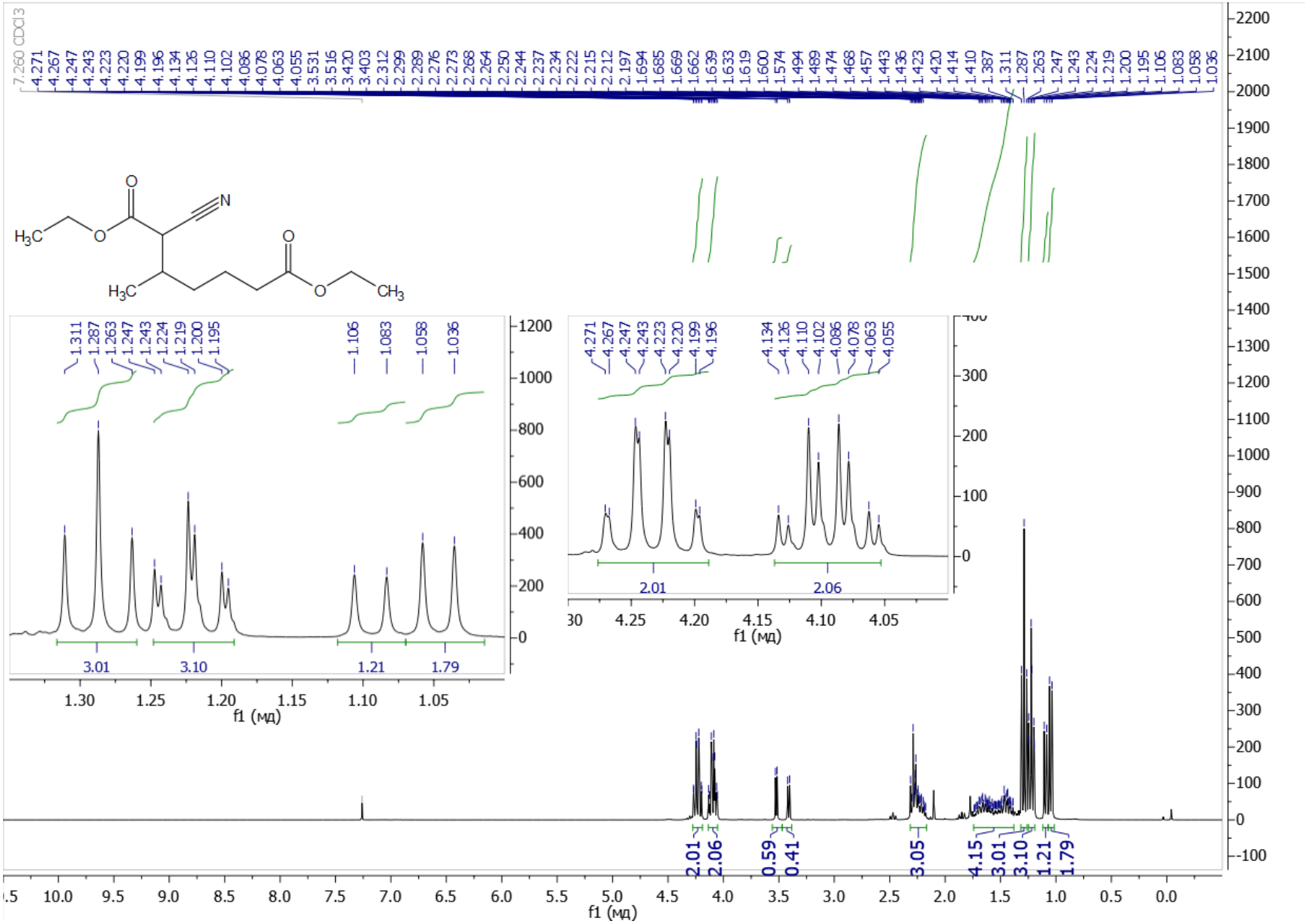
^{1}H NMR (300.13 MHz, $CDCl_3$) spectrum of ethyl 2-cyano-3-methyl-5-phenylpentanoate, **3ap**



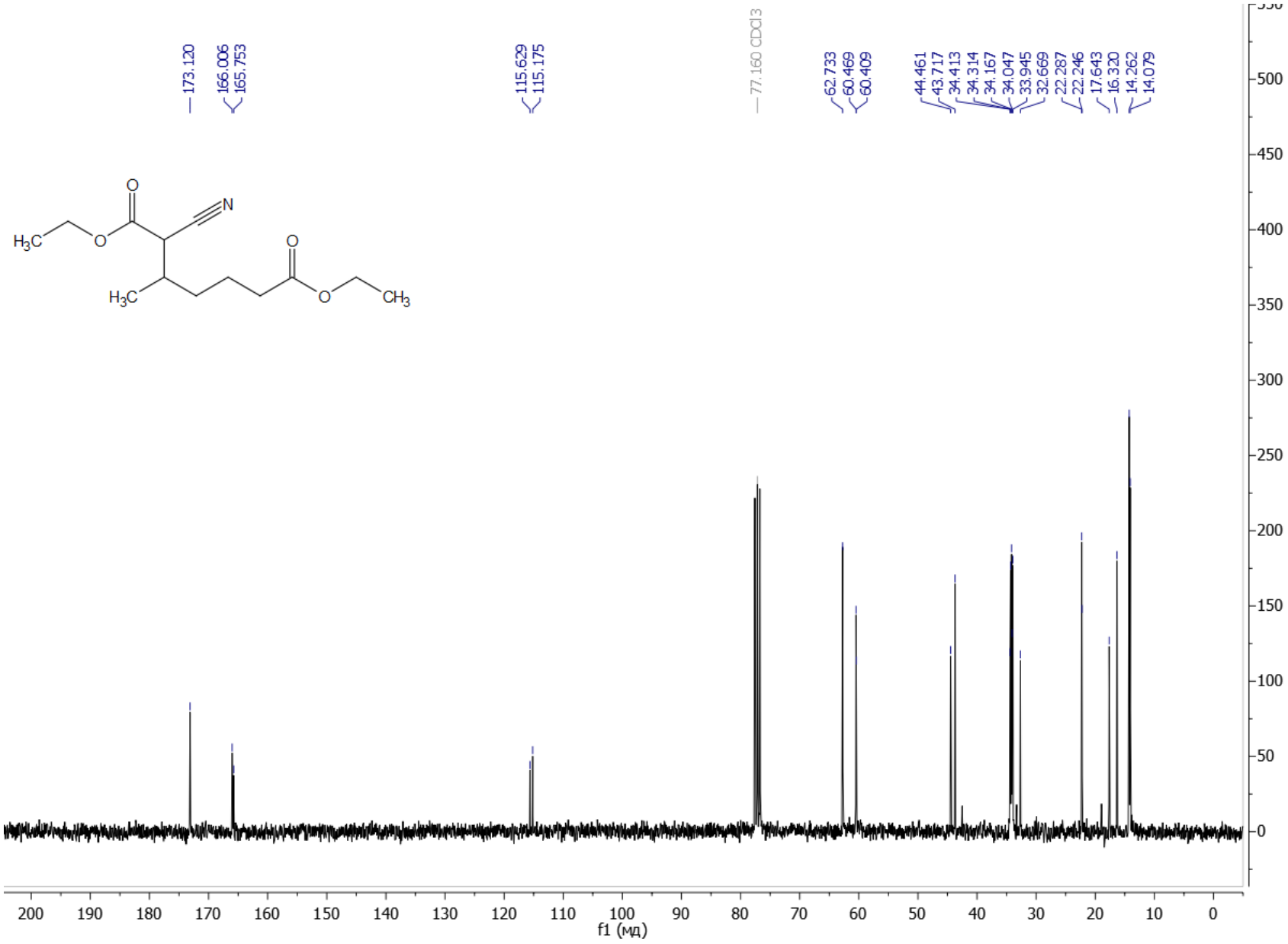
^{13}C NMR (75.48 MHz, $CDCl_3$) spectrum of ethyl 2-cyano-3-methyl-5-phenylpentanoate, **3ap**



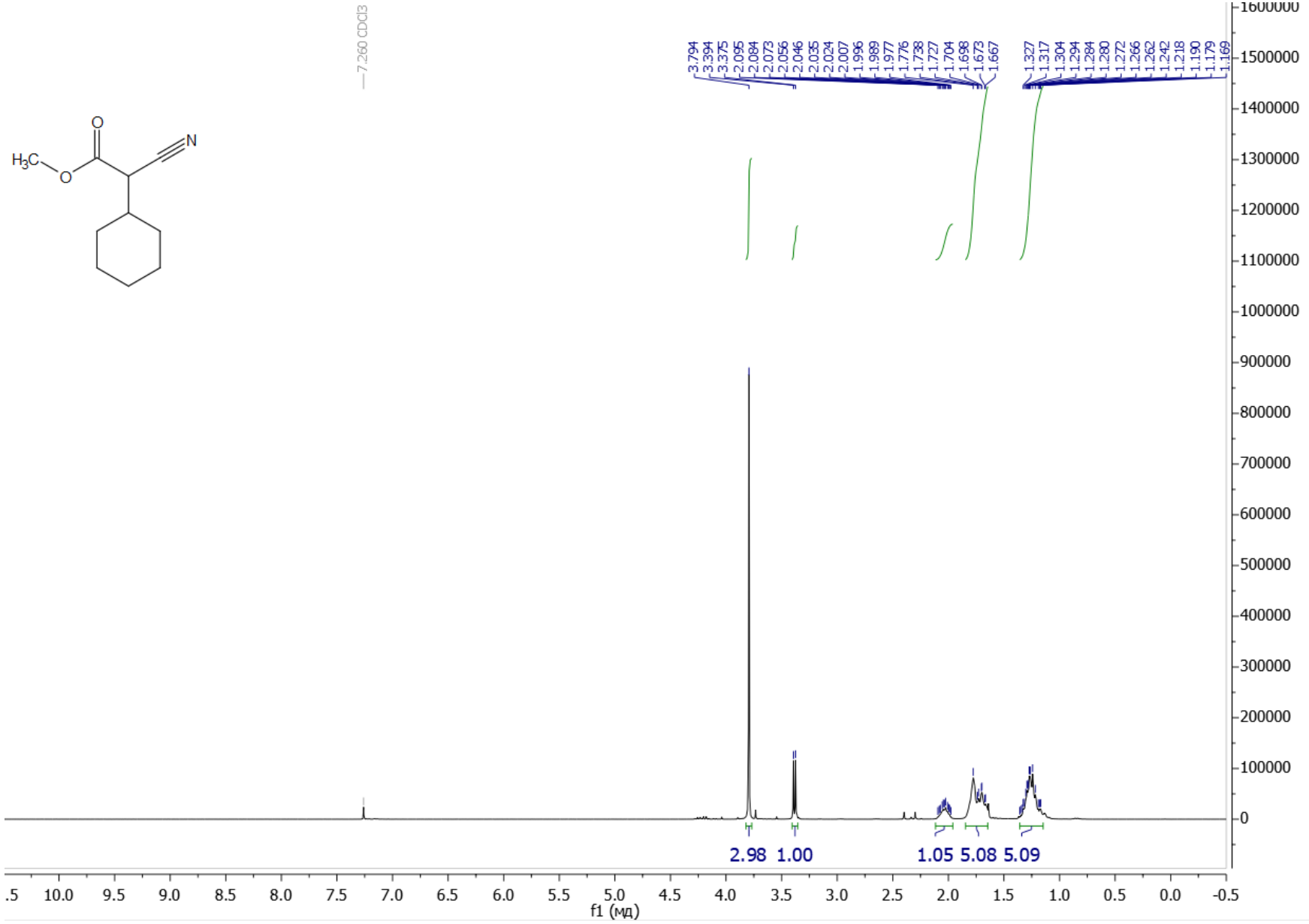
^{1}H NMR (300.13 MHz, $CDCl_3$) spectrum of diethyl 2-cyano-3-methylheptanedioate, **3aq**



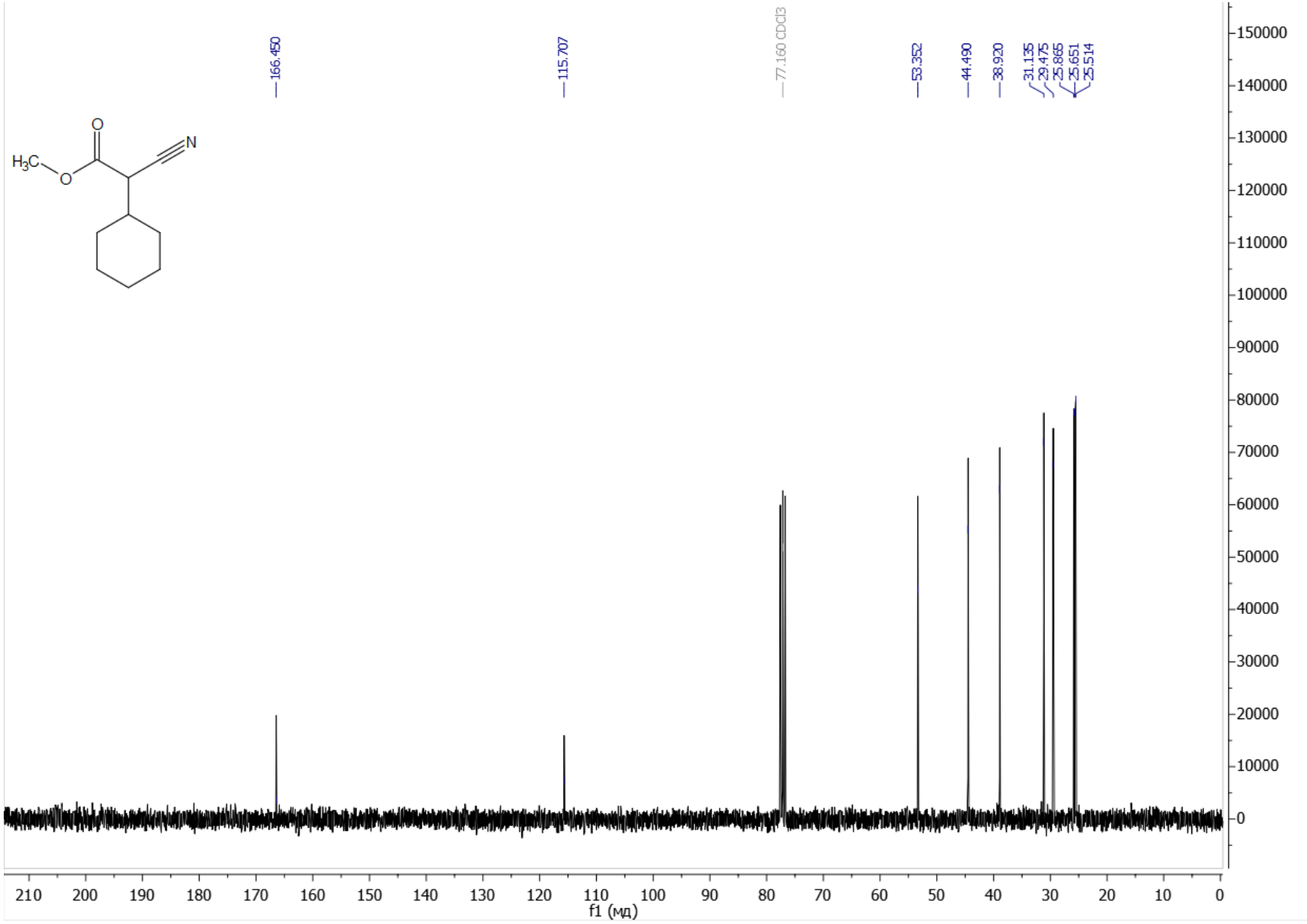
^{13}C NMR (75.48 MHz, $CDCl_3$) spectrum of diethyl 2-cyano-3-methylheptanedioate, **3aq**



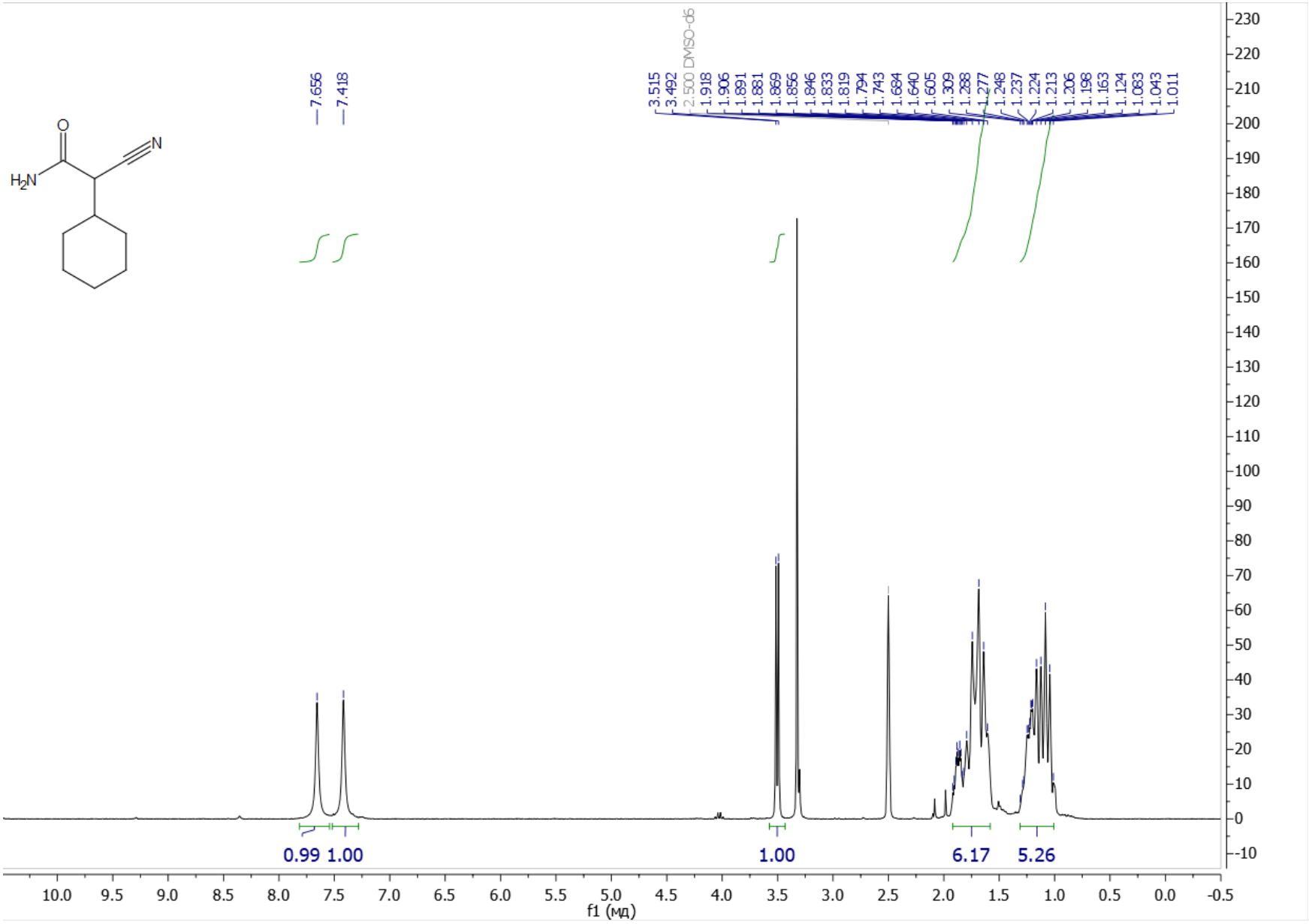
^{1}H NMR (300.13 MHz, $CDCl_3$) spectrum of methyl 2-cyano-2-cyclohexylacetate, **3bb**



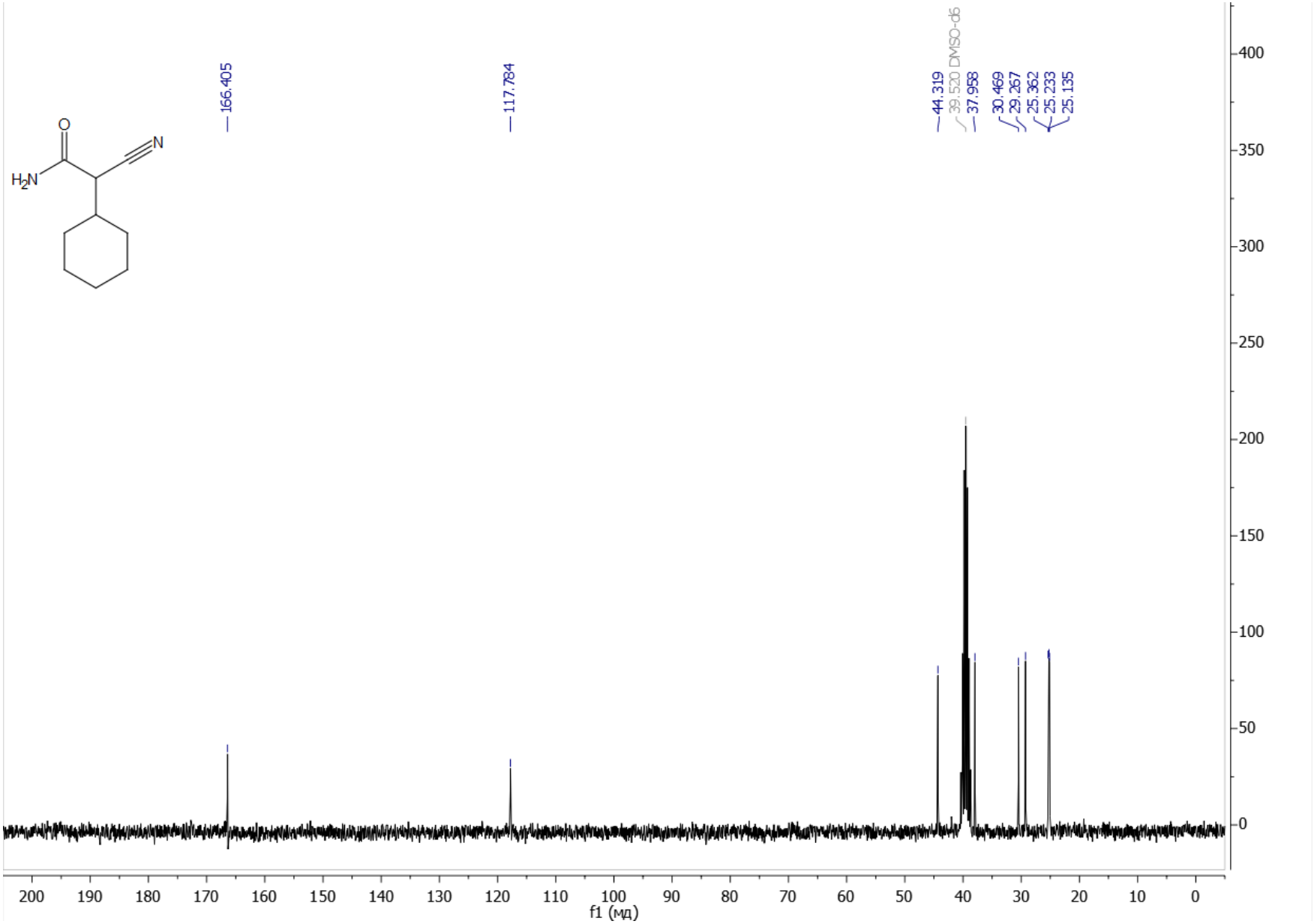
^{13}C NMR (75.48 MHz, $CDCl_3$) spectrum of methyl 2-cyano-2-cyclohexylacetate, **3bb**



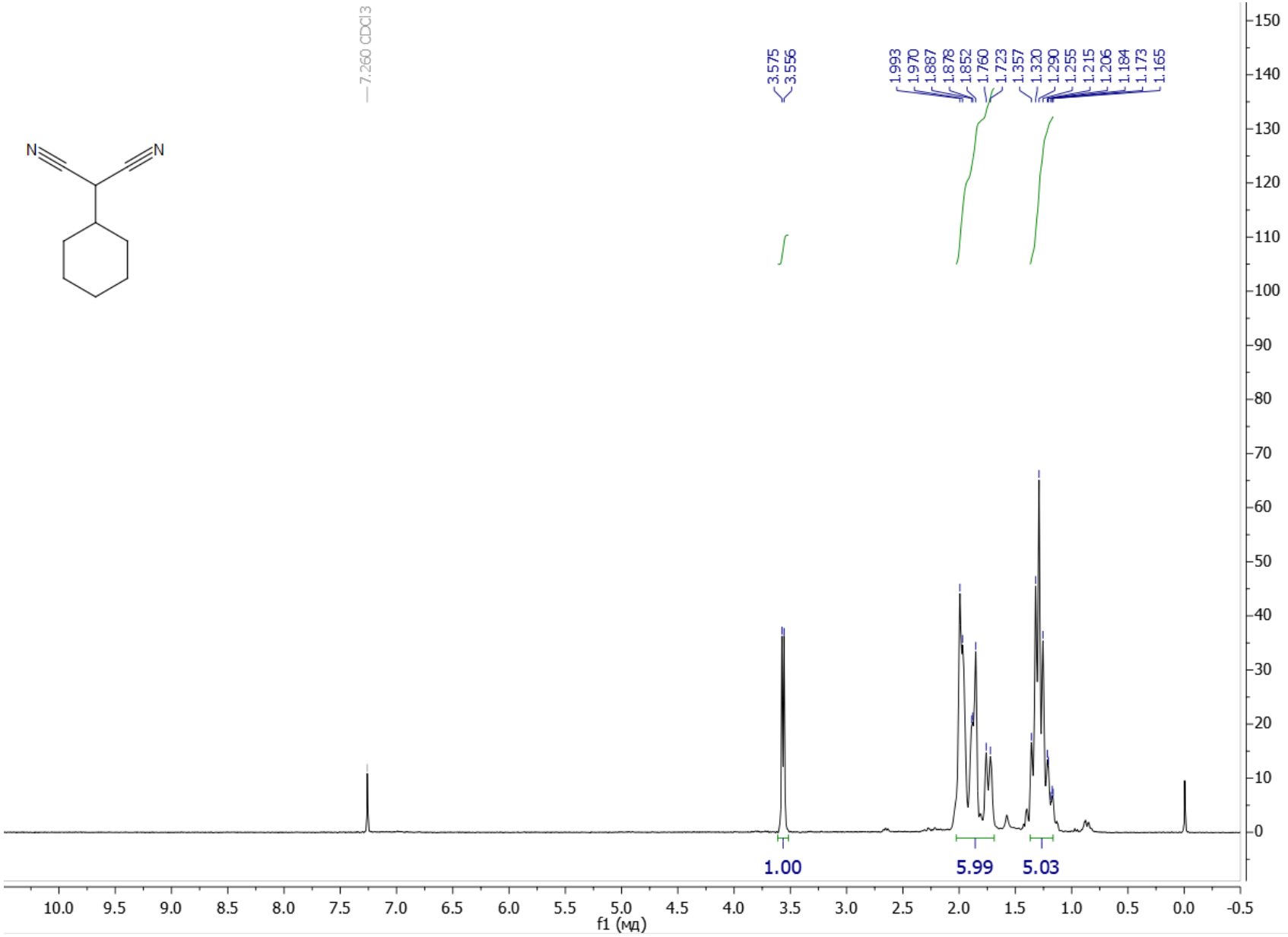
^{1}H NMR (300.13 MHz, DMSO-d_6) spectrum of 2-cyano-2-cyclohexylacetamide, **3cb**



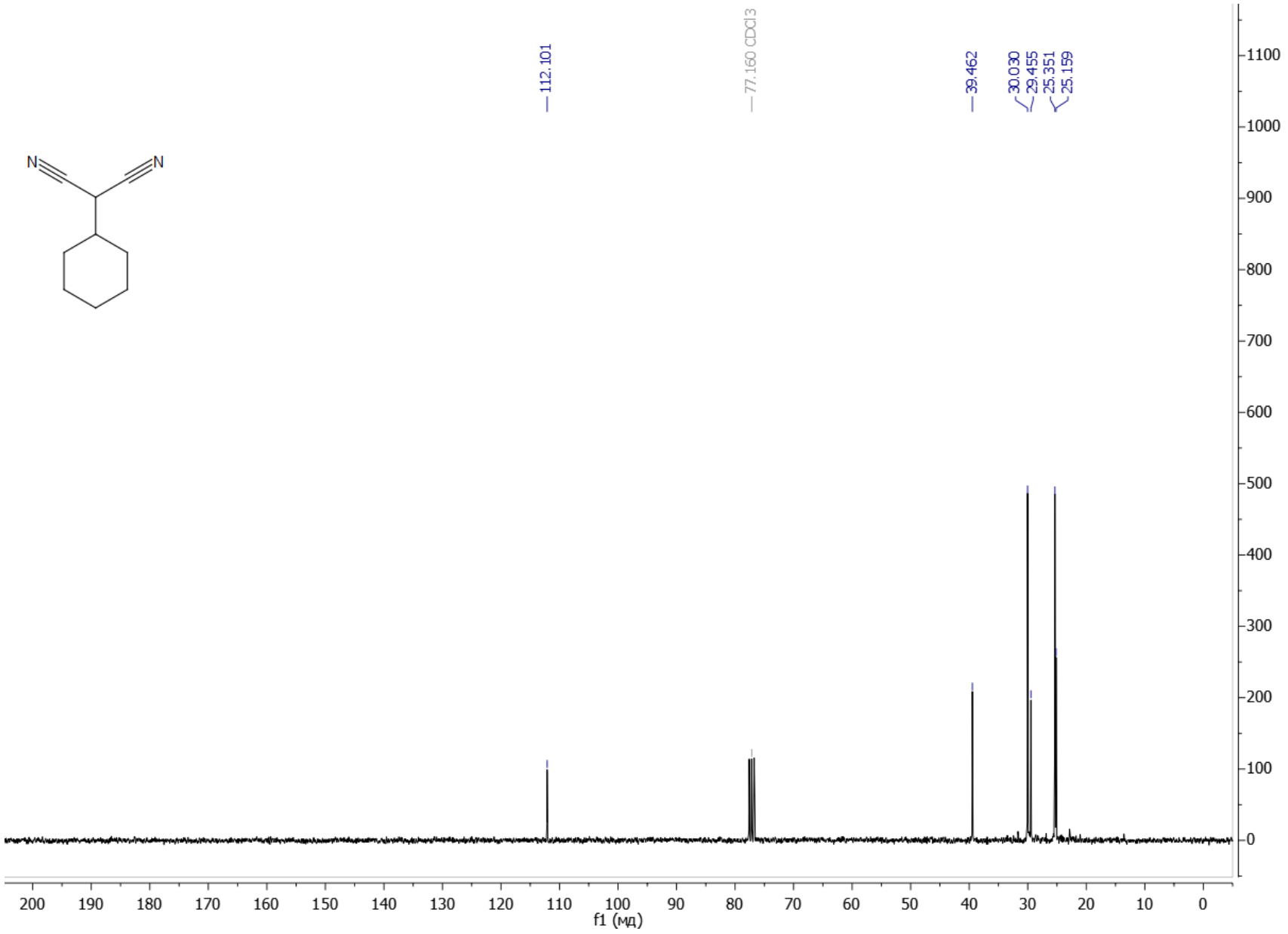
^{13}C NMR (75.48 MHz, DMSO-d_6) spectrum of 2-cyano-2-cyclohexylacetamide, **3cb**



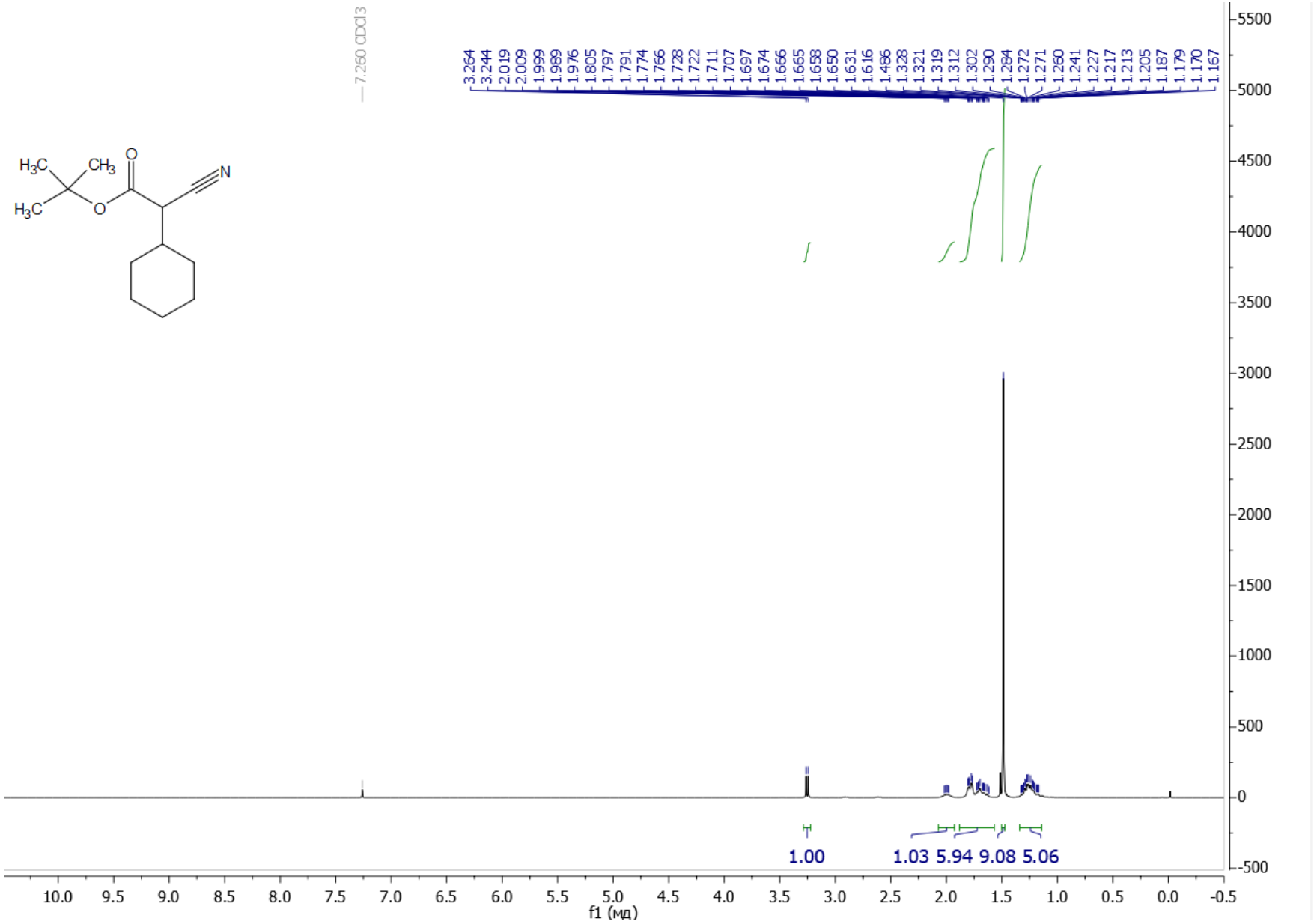
^{1}H NMR (300.13 MHz, $CDCl_3$) spectrum of 2-cyclohexylmalononitrile, **3eb**



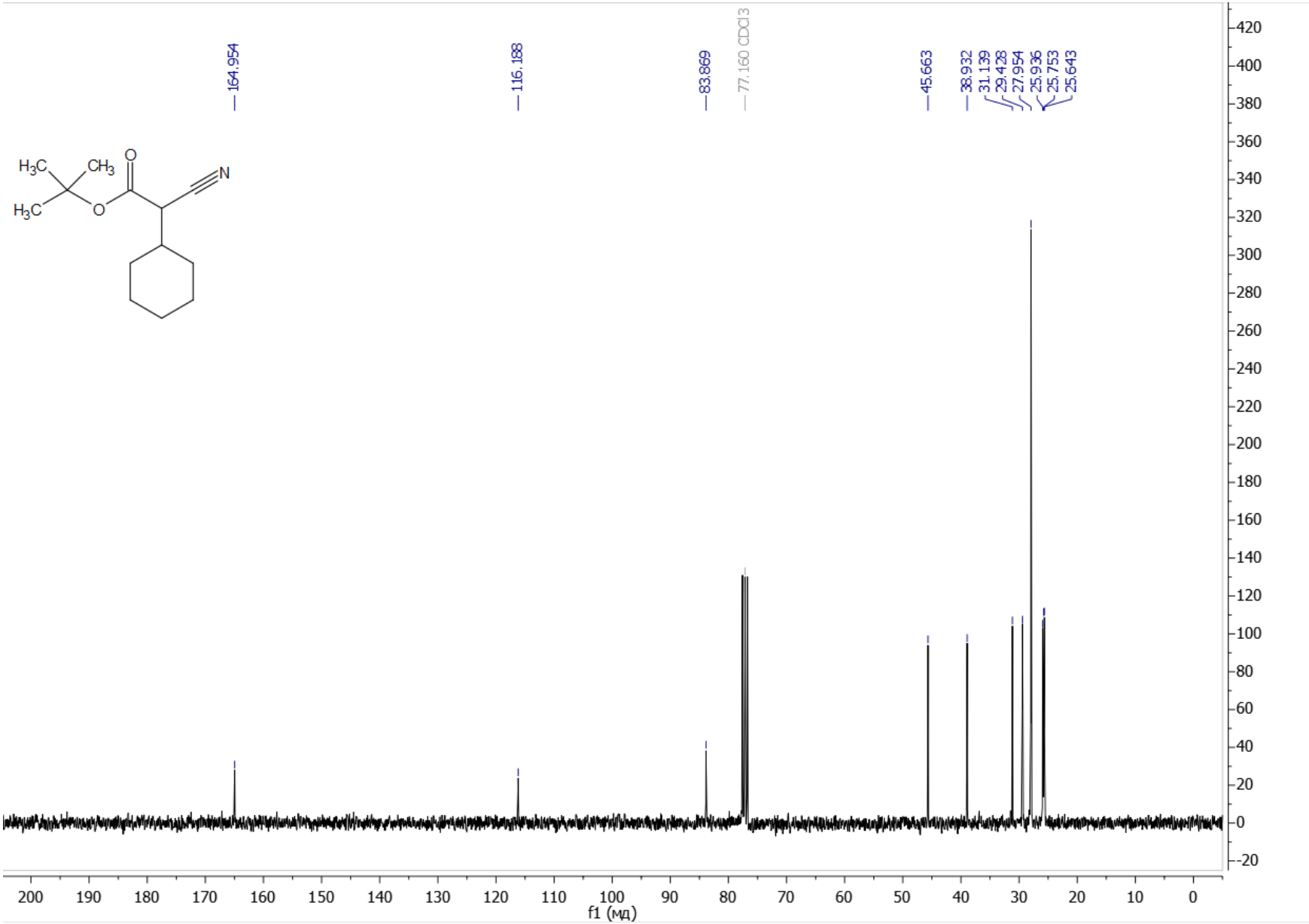
^{13}C NMR (75.48 MHz, $CDCl_3$) spectrum of 2-cyclohexylmalononitrile, **3eb**



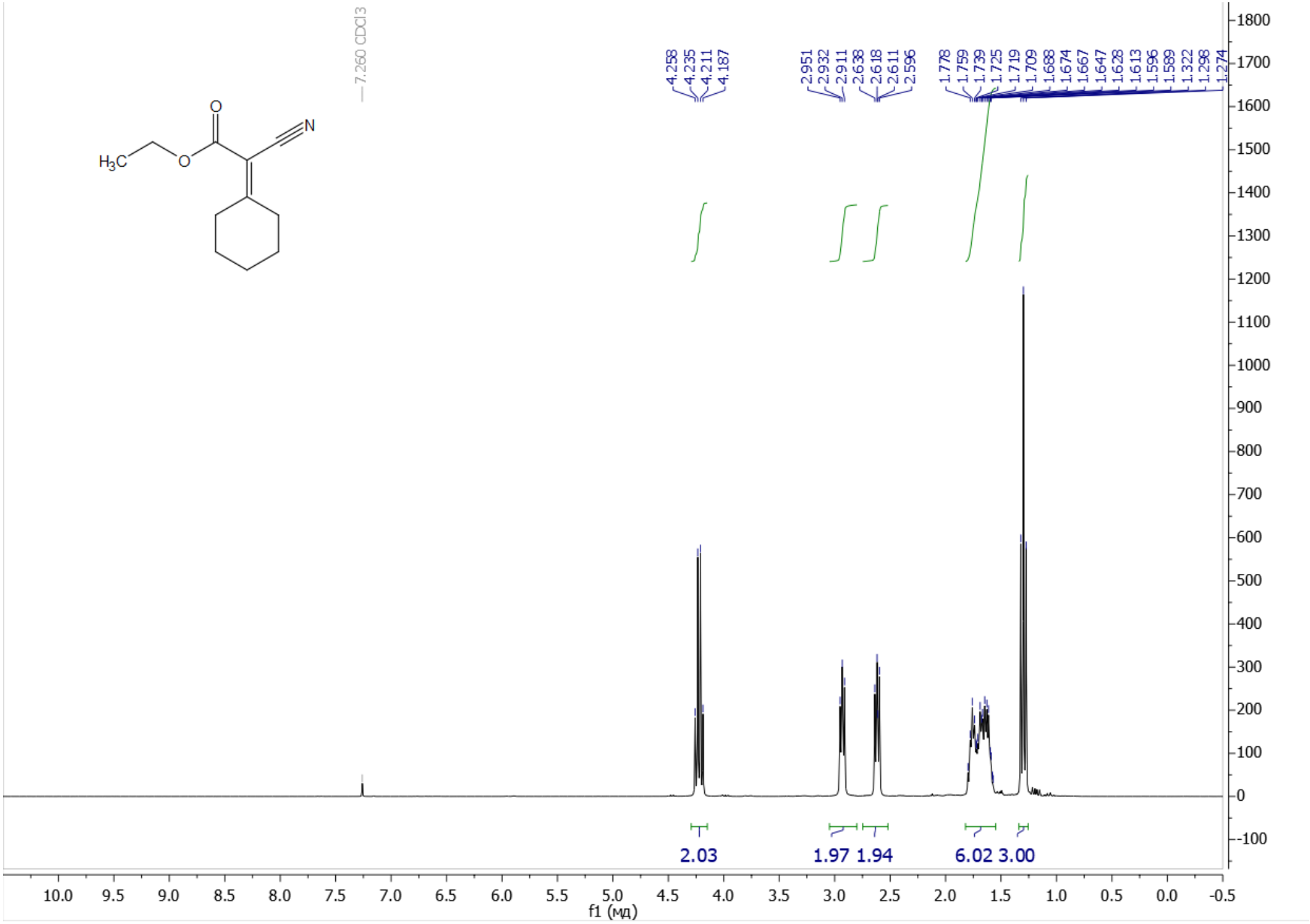
^{1}H NMR (300.13 MHz, $CDCl_3$) spectrum of *tert*-butyl 2-cyano-2-cyclohexylacetate, **3eb**



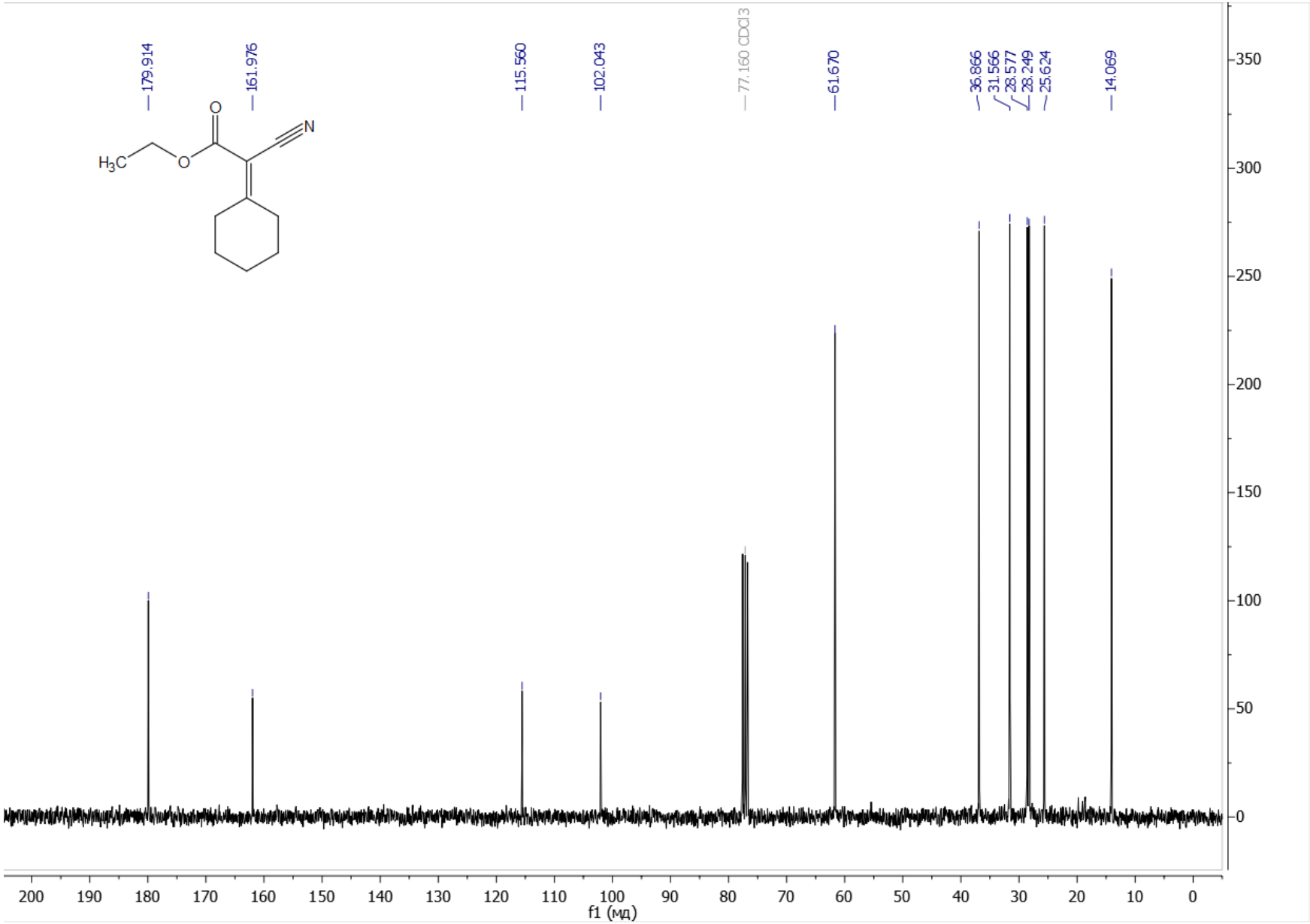
^{13}C NMR (75.48 MHz, $CDCl_3$) spectrum of *tert*-butyl 2-cyano-2-cyclohexylacetate, **3eb**



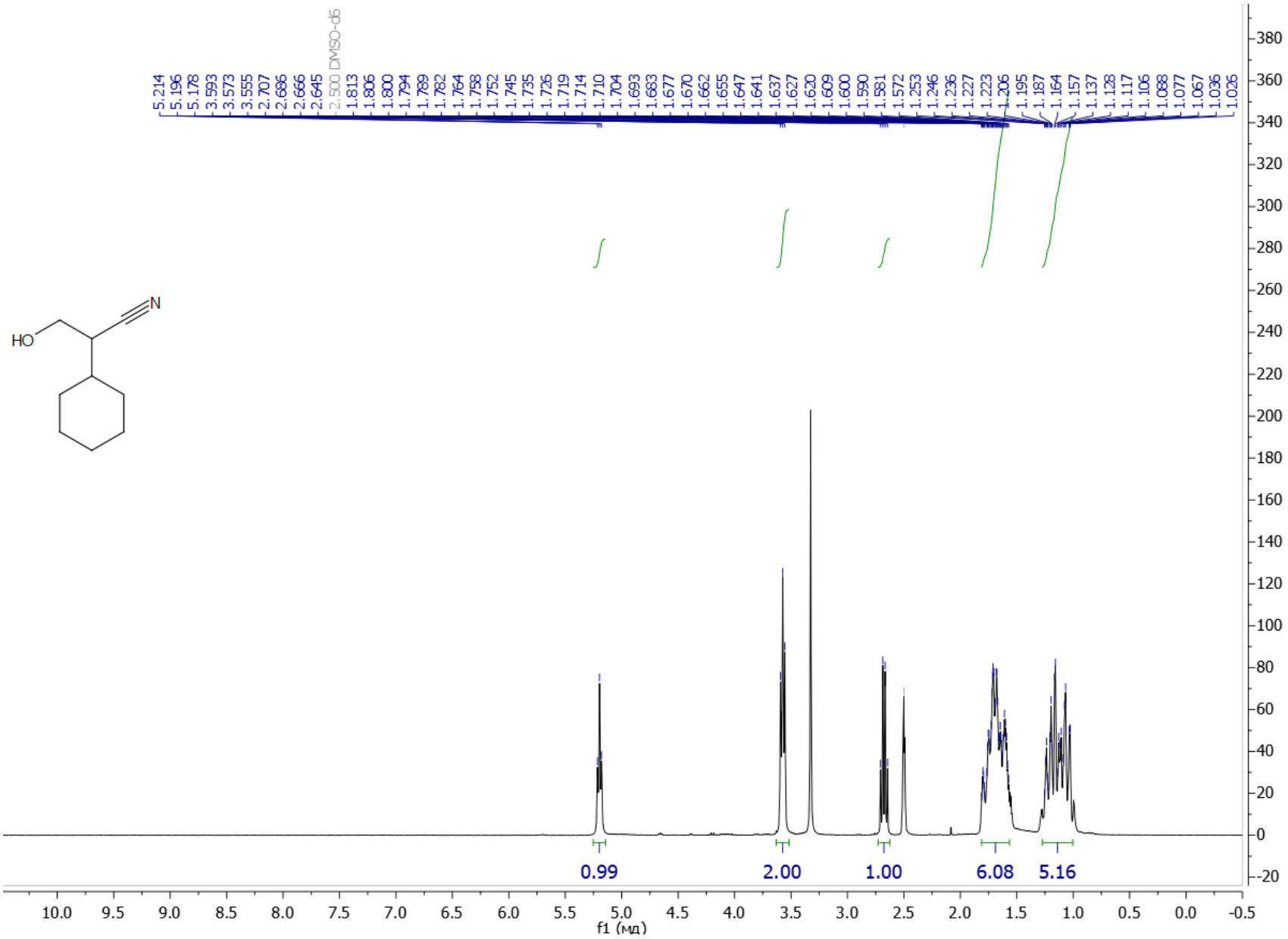
^{1}H NMR (300.13 MHz, $CDCl_3$) spectrum of ethyl 2-cyano-2-cyclohexylideneacetate, **4ab**



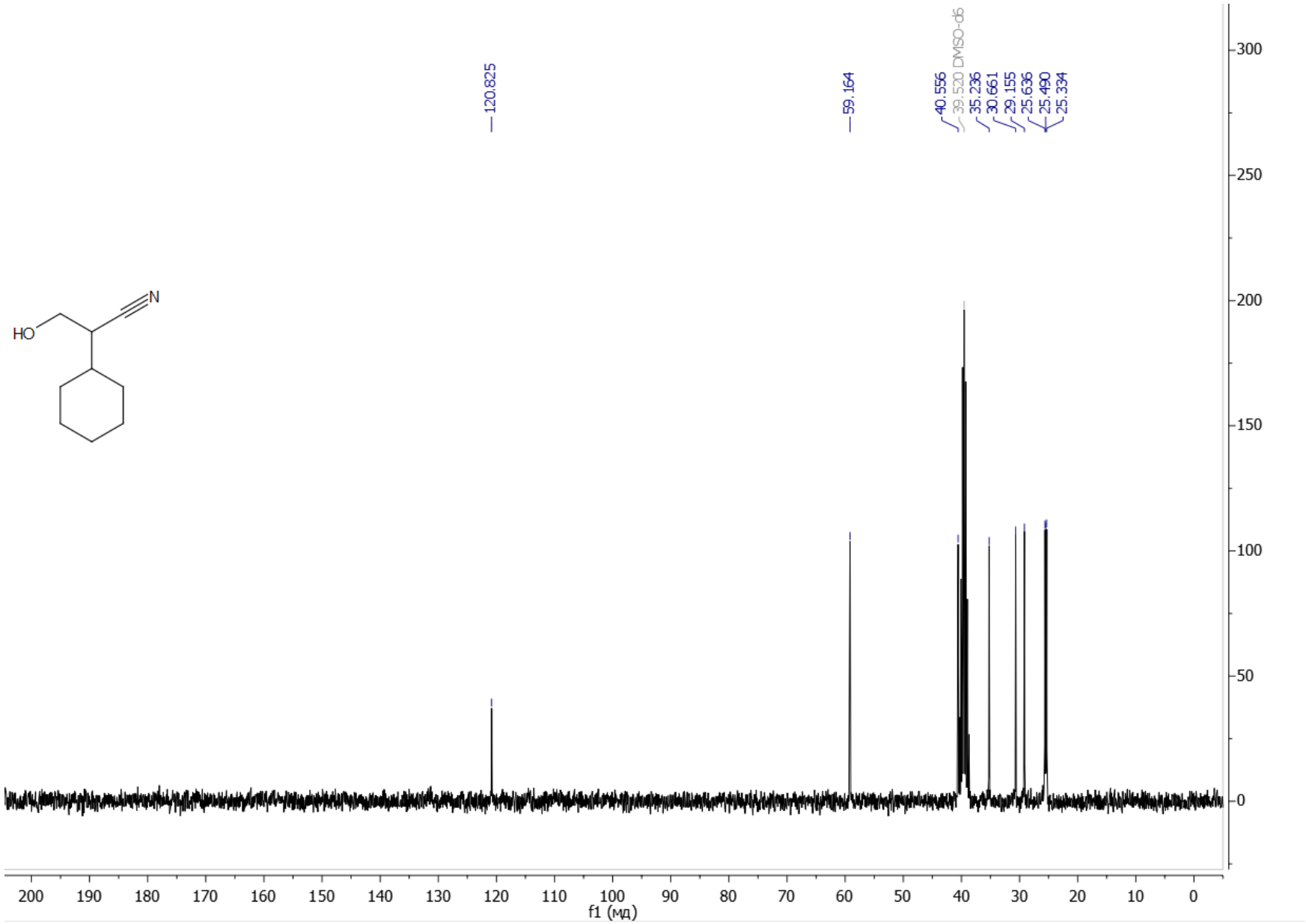
^{13}C NMR (75.48 MHz, $CDCl_3$) spectrum of ethyl 2-cyano-2-cyclohexylideneacetate, **4ab**



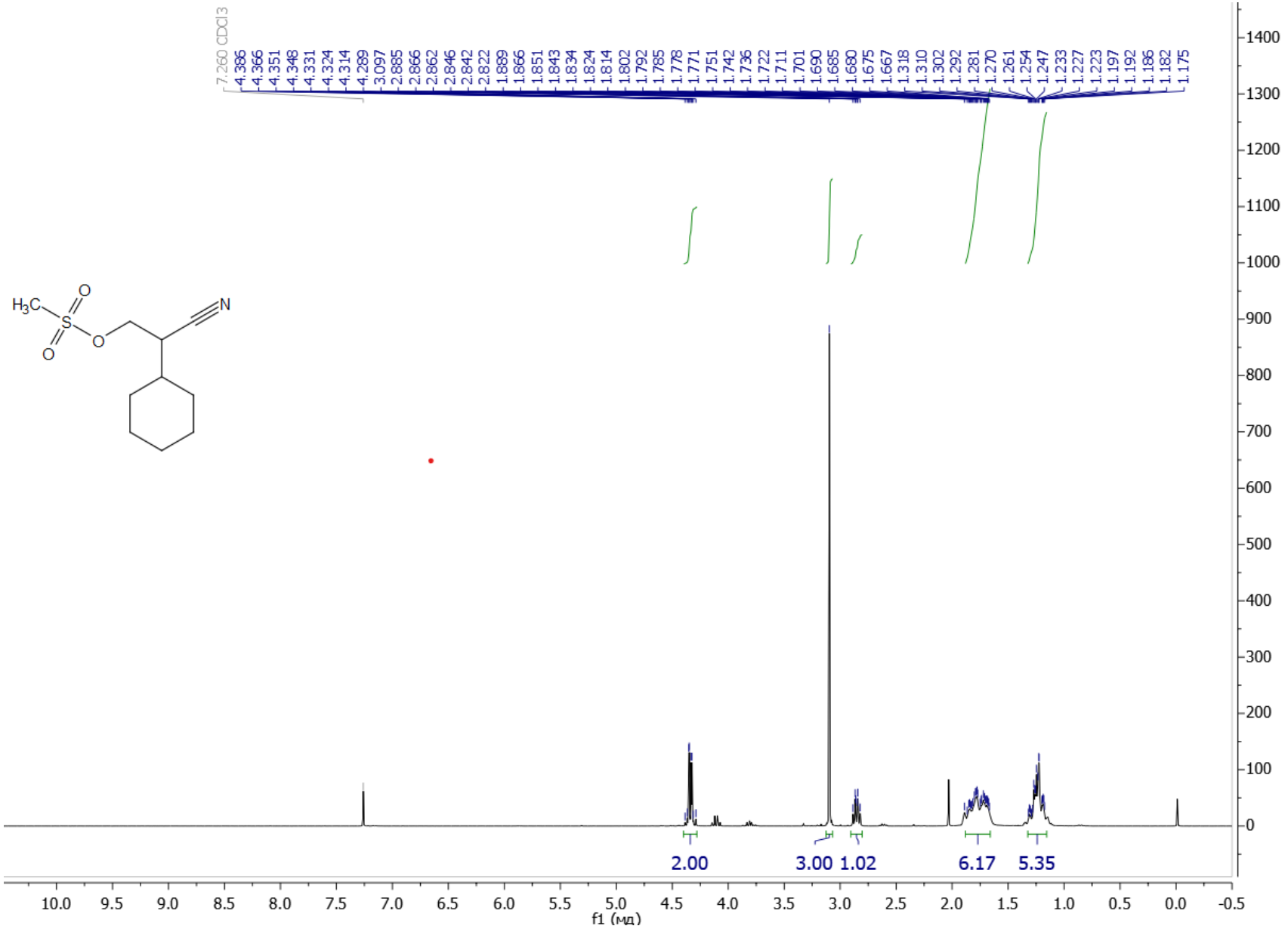
^{1}H NMR (300.13 MHz, DMSO-d_6) spectrum of 2-cyclohexyl-3-hydroxypropanenitrile, **5**



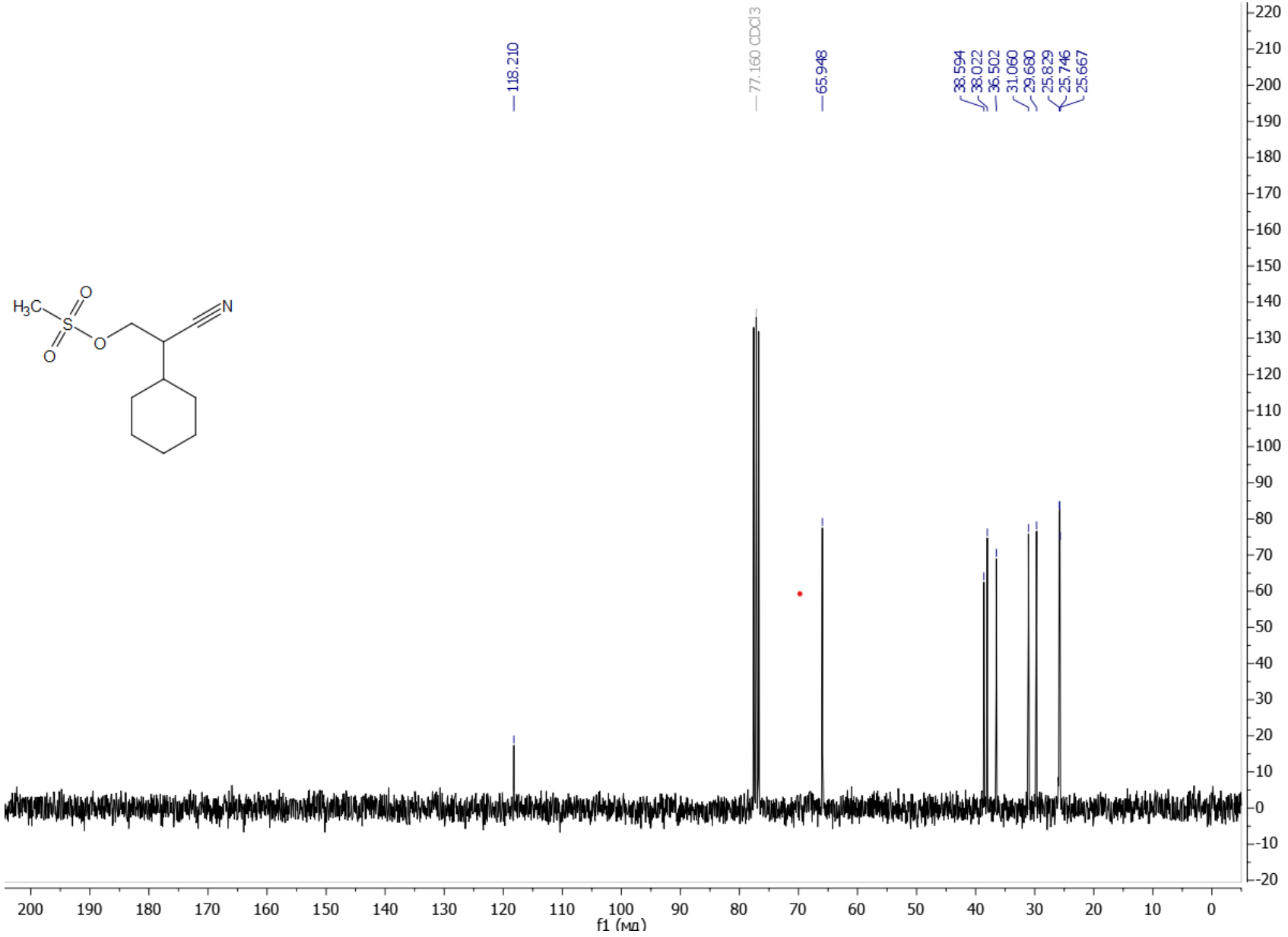
^{13}C NMR (75.48 MHz, DMSO-d_6) spectrum of 2-cyclohexyl-3-hydroxypropanenitrile, **5**



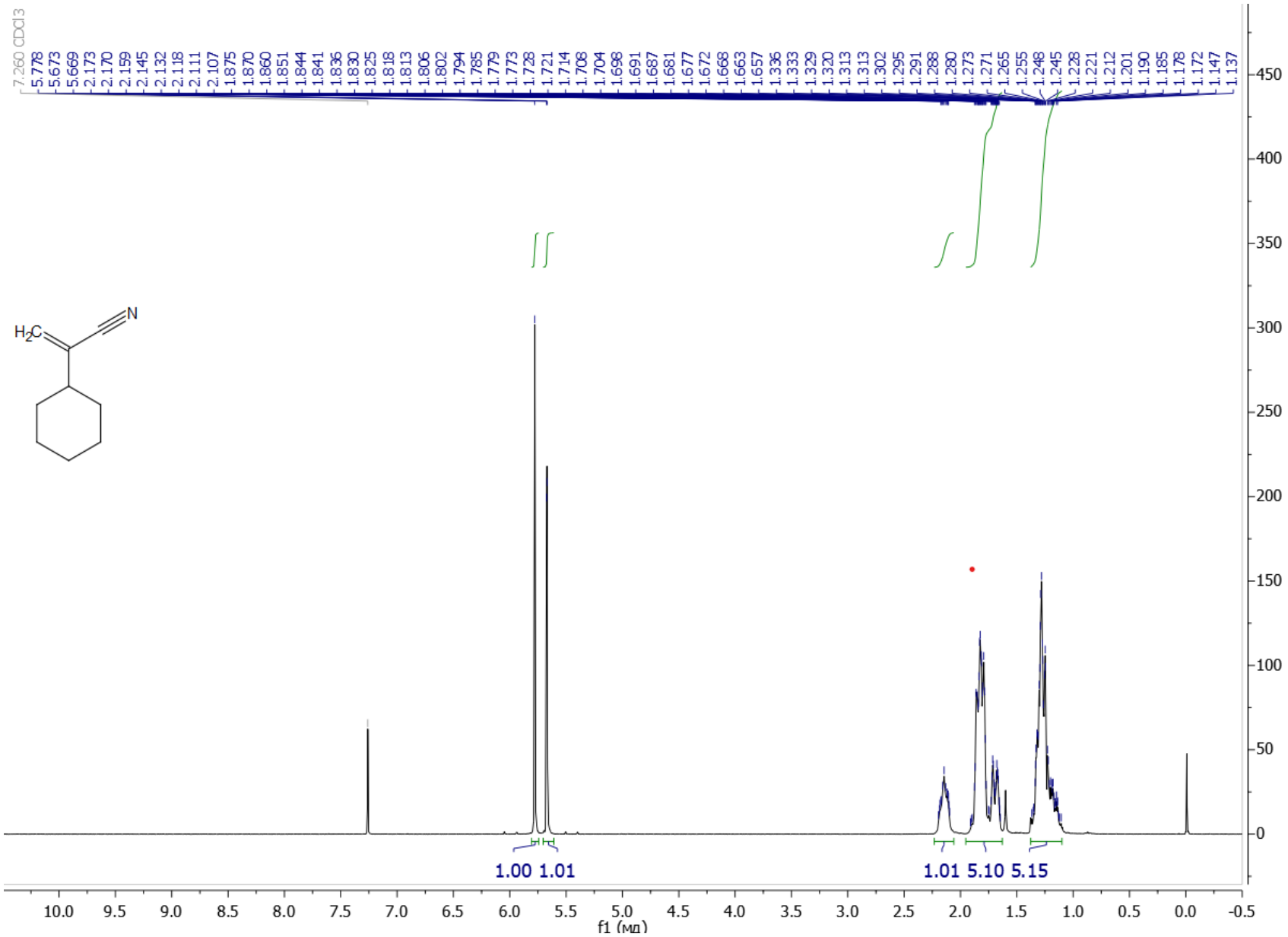
^{1}H NMR (300.13 MHz, $CDCl_3$) spectrum of 2-cyano-2-cyclohexylethyl methanesulfonate, **6**



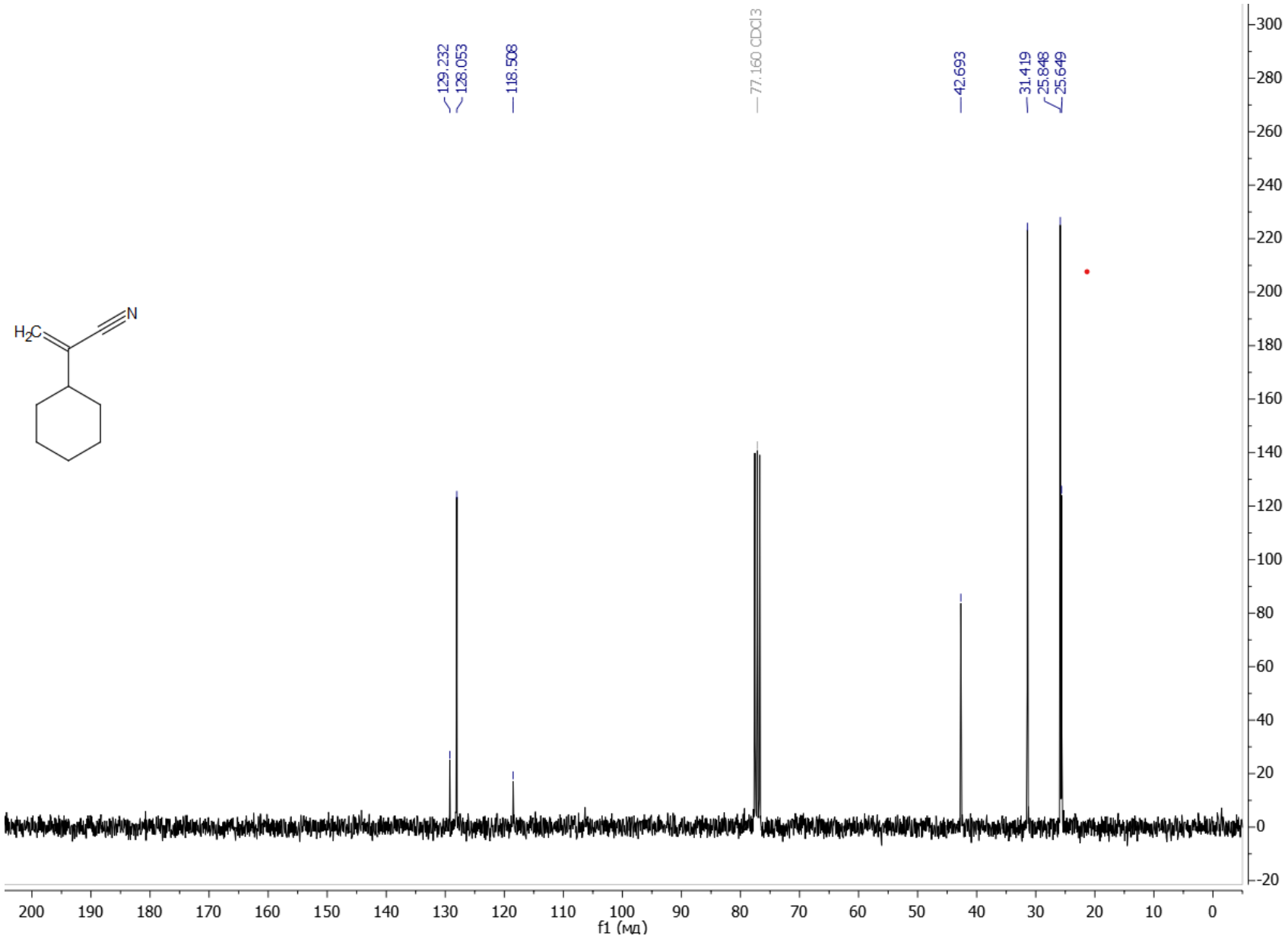
^{13}C NMR (75.48 MHz, $CDCl_3$) spectrum of 2-cyano-2-cyclohexylethyl methanesulfonate, **6**



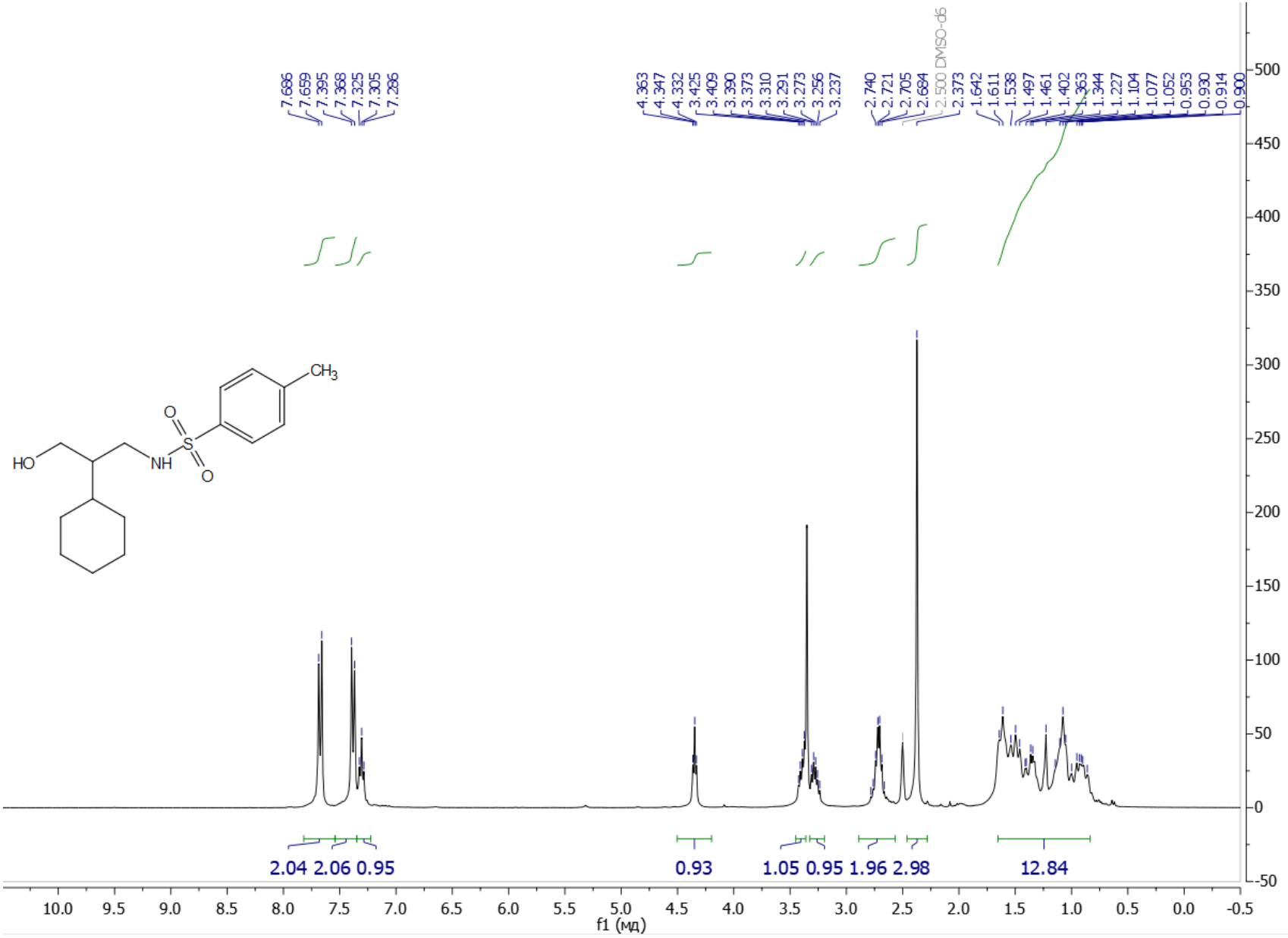
^{1}H NMR (300.13 MHz, $CDCl_3$) spectrum of 2-cyclohexylacrylonitrile, **7**



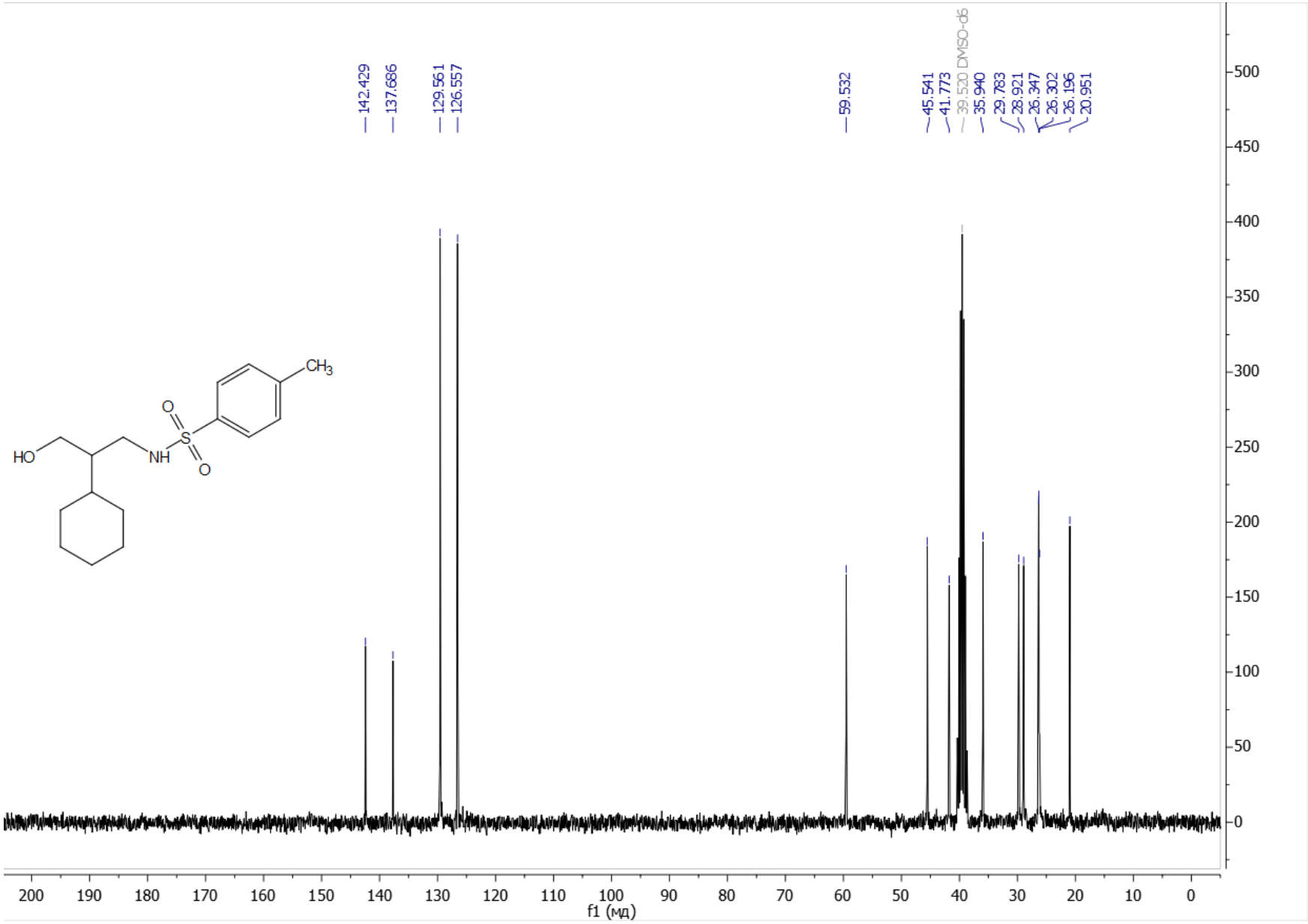
^{13}C NMR (75.48 MHz, $CDCl_3$) spectrum of 2-cyclohexylacrylonitrile, **7**



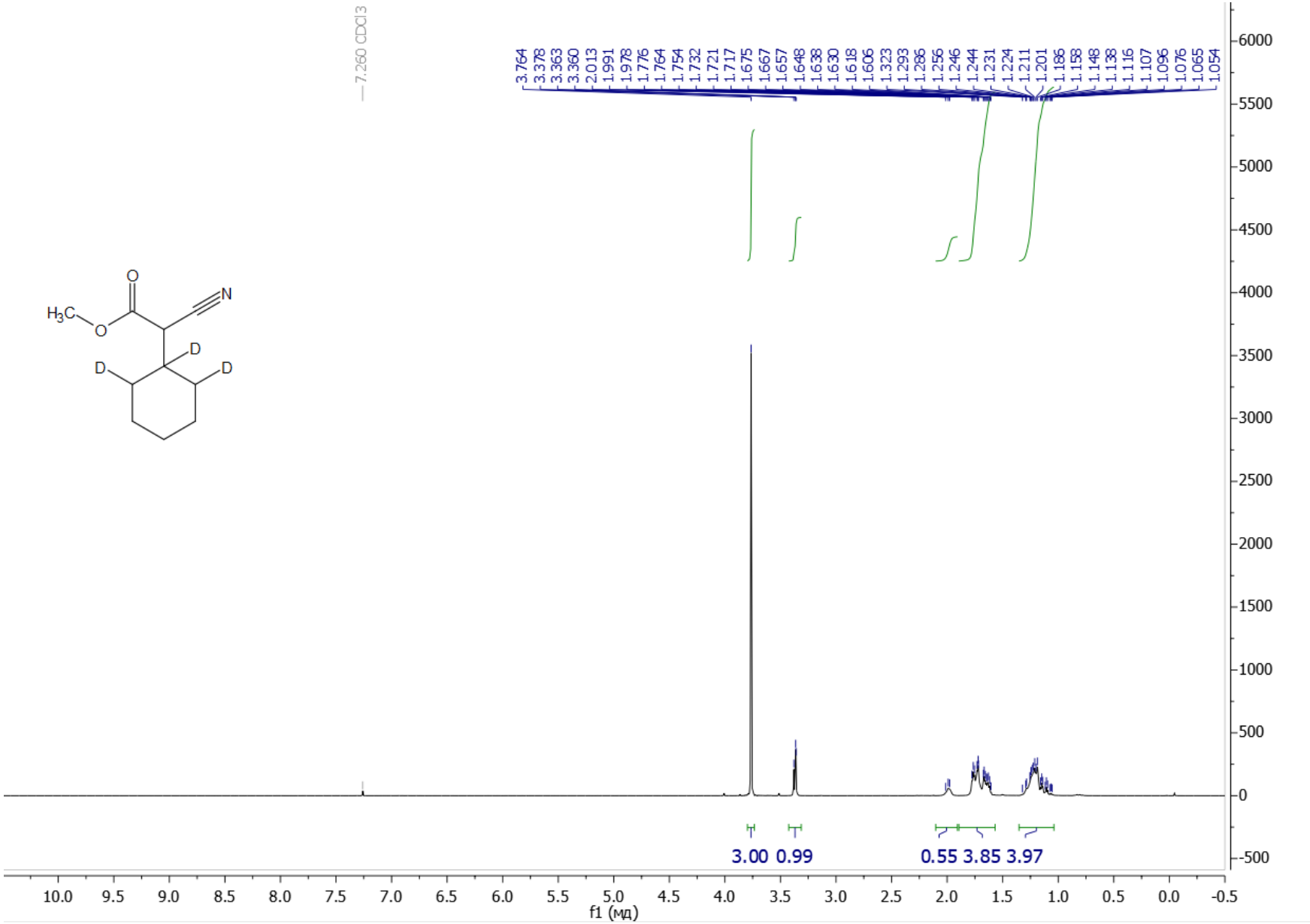
^{1}H NMR (300.13 MHz, DMSO-d_6) spectrum of N-(2-cyclohexyl-3-hydroxypropyl)-4-methylbenzenesulfonamide, **8**



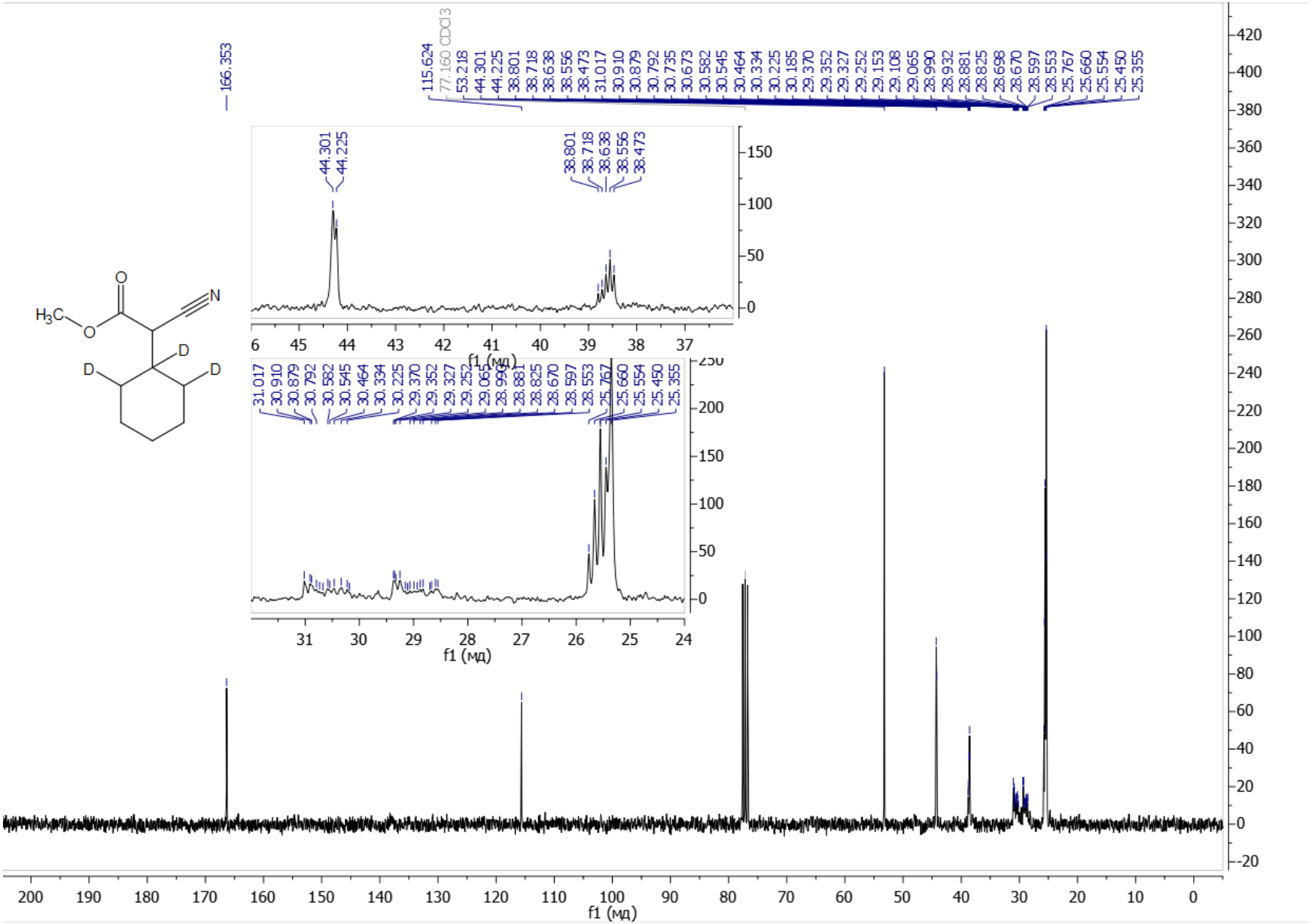
^{13}C NMR (75.48 MHz, DMSO-d_6) spectrum of N-(2-cyclohexyl-3-hydroxypropyl)-4-methylbenzenesulfonamide, **8**



^{1}H NMR (300.13 MHz, $CDCl_3$) spectrum of methyl 2-cyano-2-cyclohexylacetate-d_n, **3bb-d_n**



^{13}C NMR (75.48 MHz, $CDCl_3$) spectrum of methyl 2-cyano-2-cyclohexylacetate-d_n, **3bb-d_n**



HRMS spectra of the synthesized compounds

HRMS spectrum of ethyl 2-cyano-2-cyclooctylacetate, **3ad**

Display Report

Analysis Info

Analysis Name	D:\Data\Kolotyrkina\2025\Kirillov\0311008.d	Acquisition Date	11.03.2025 10:38:45
Method	tune_low.m	Operator	BDAL@DE
Sample Name	/VILV pil784_3d	Instrument / Ser#	micrOTOF 10248
Comment	C13H21NO2 clb added CH3CN		

Acquisition Parameter

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Focus	Not active			Set Dry Heater	180 °C
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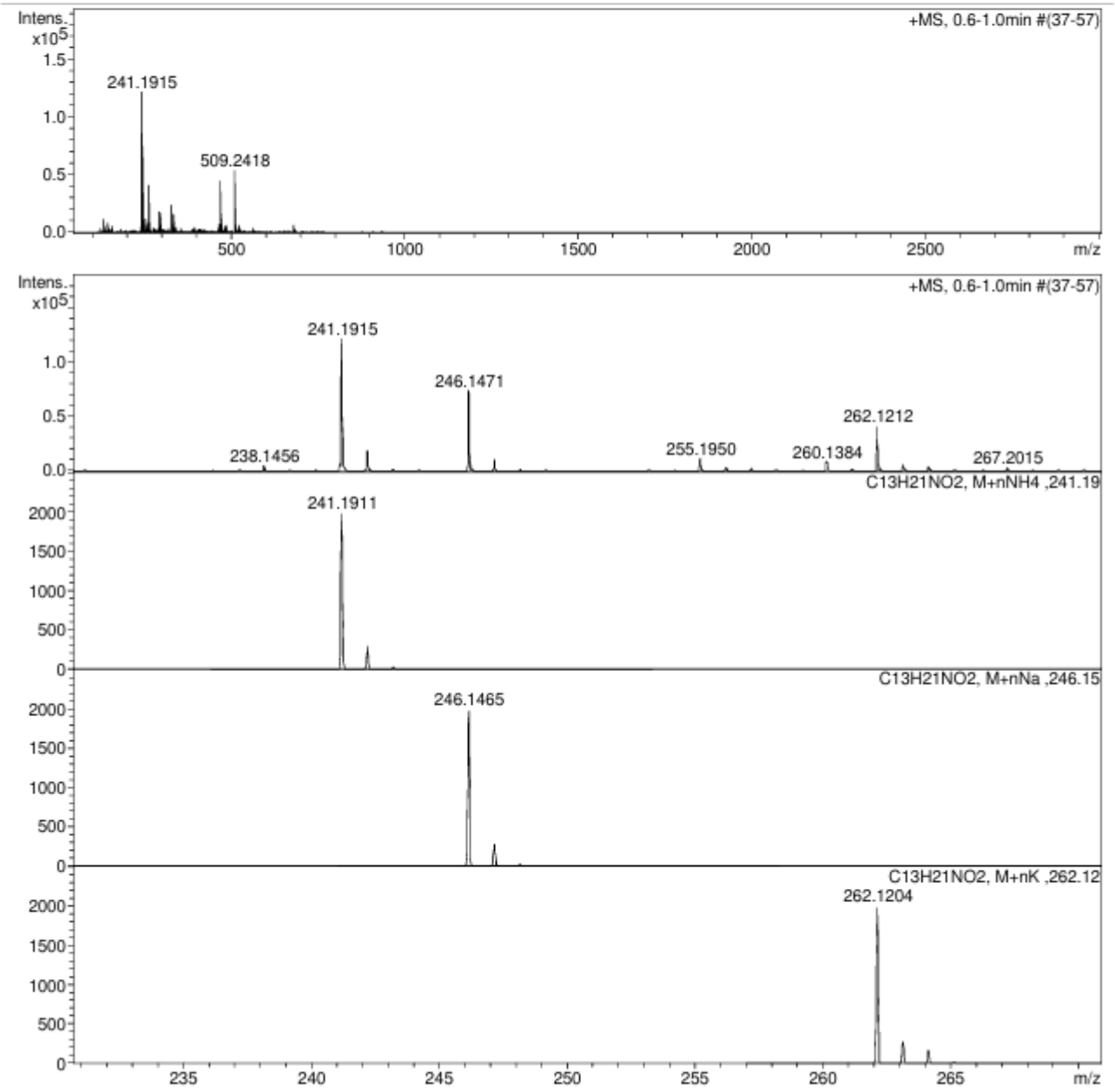
Scan End	3000 m/z	Set End Plate Offset	-500 V	Set Divert Valve	Waste



HRMS spectrum of *tert*-butyl 4-(1-cyano-2-ethoxy-2-oxoethyl)piperidine-1-carboxylate, **3ai**

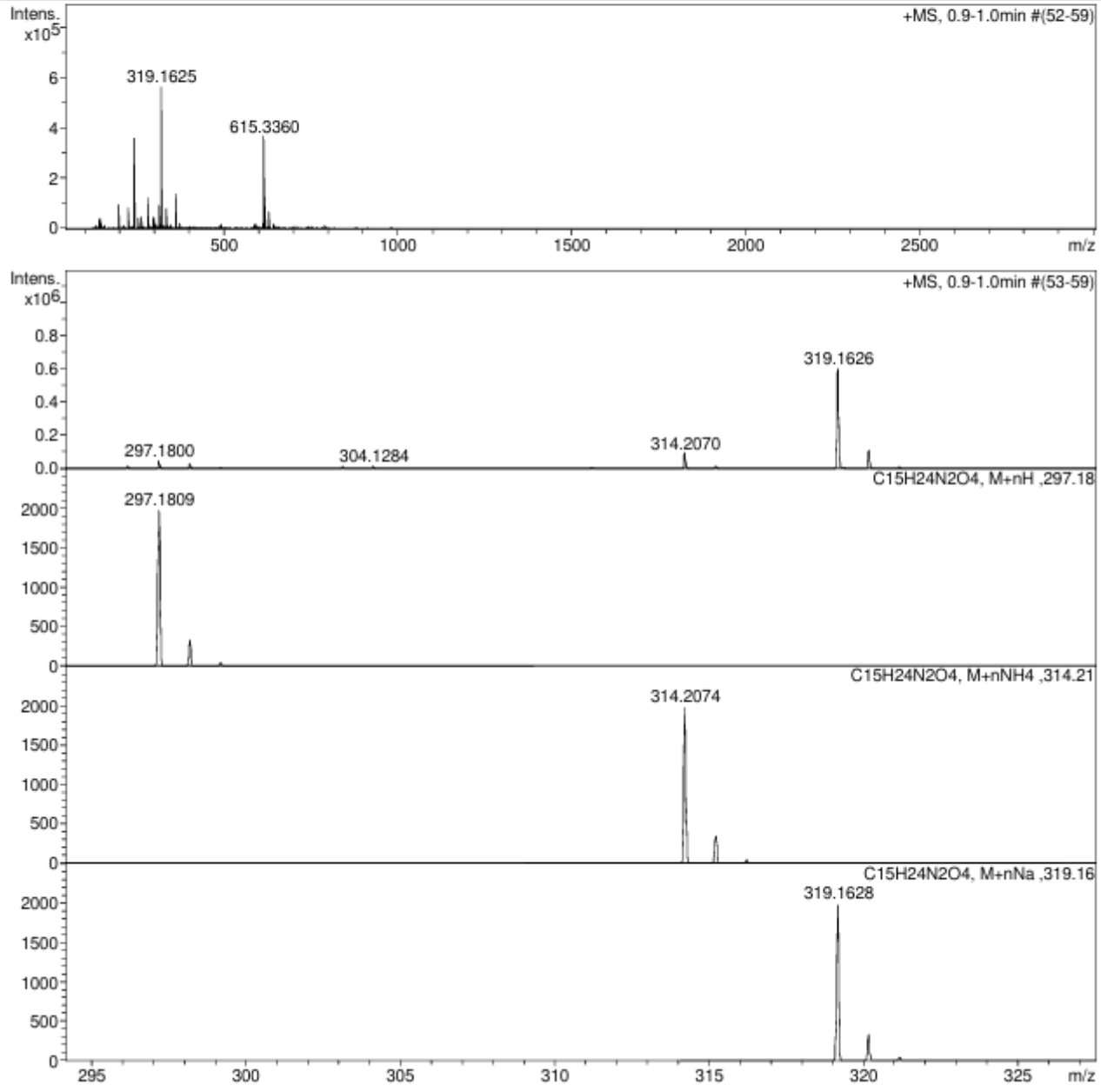
Display Report

Analysis Info

Analysis Name	D:\Data\Kolotyrkina\2025\Kirillov\0311007.d	Acquisition Date	11.03.2025 10:27:10
Method	tune_low.m	Operator	BDAL@DE
Sample Name	/VILV pil824_2i	Instrument / Ser#	micrOTOF 10248
Comment	C15H24N2O4 clb added CH3CN		

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 ethyl 2-cyano-3-heptyldecanoate, **3aj**

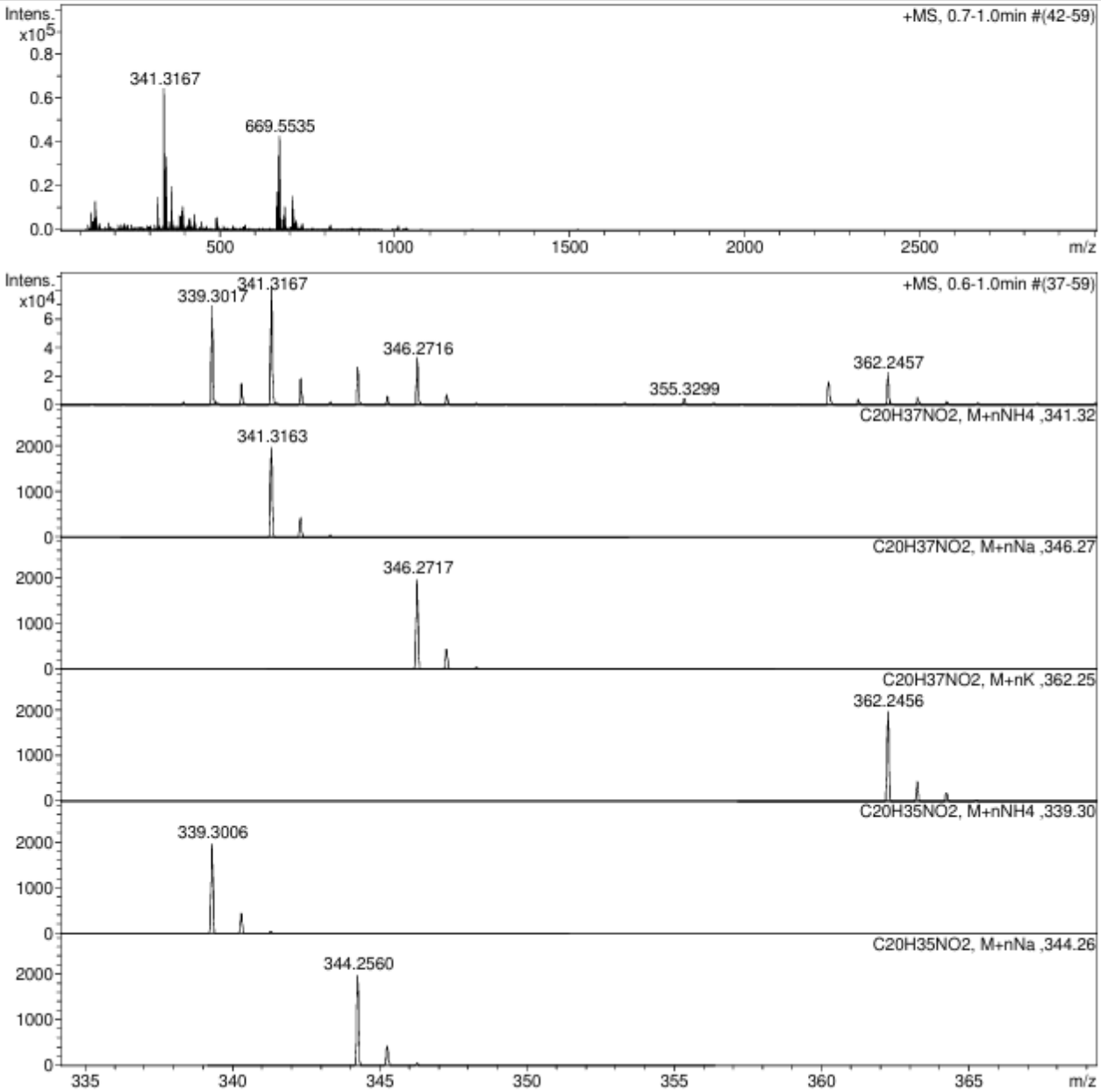
Display Report

Analysis Info

Analysis Name	D:\Data\Kolotyrkina\2025\Kirillov\0311006.d	Acquisition Date	11.03.2025 10:13:30
Method	tune_low.m	Operator	BDAL@DE
Sample Name	/VILV pil803_2j	Instrument / Ser#	micrOTOF 10248
Comment	C20H37NO2 clb added CH3CN		

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 ethyl 2-cyano-3-methylhexanoate, **3ak**

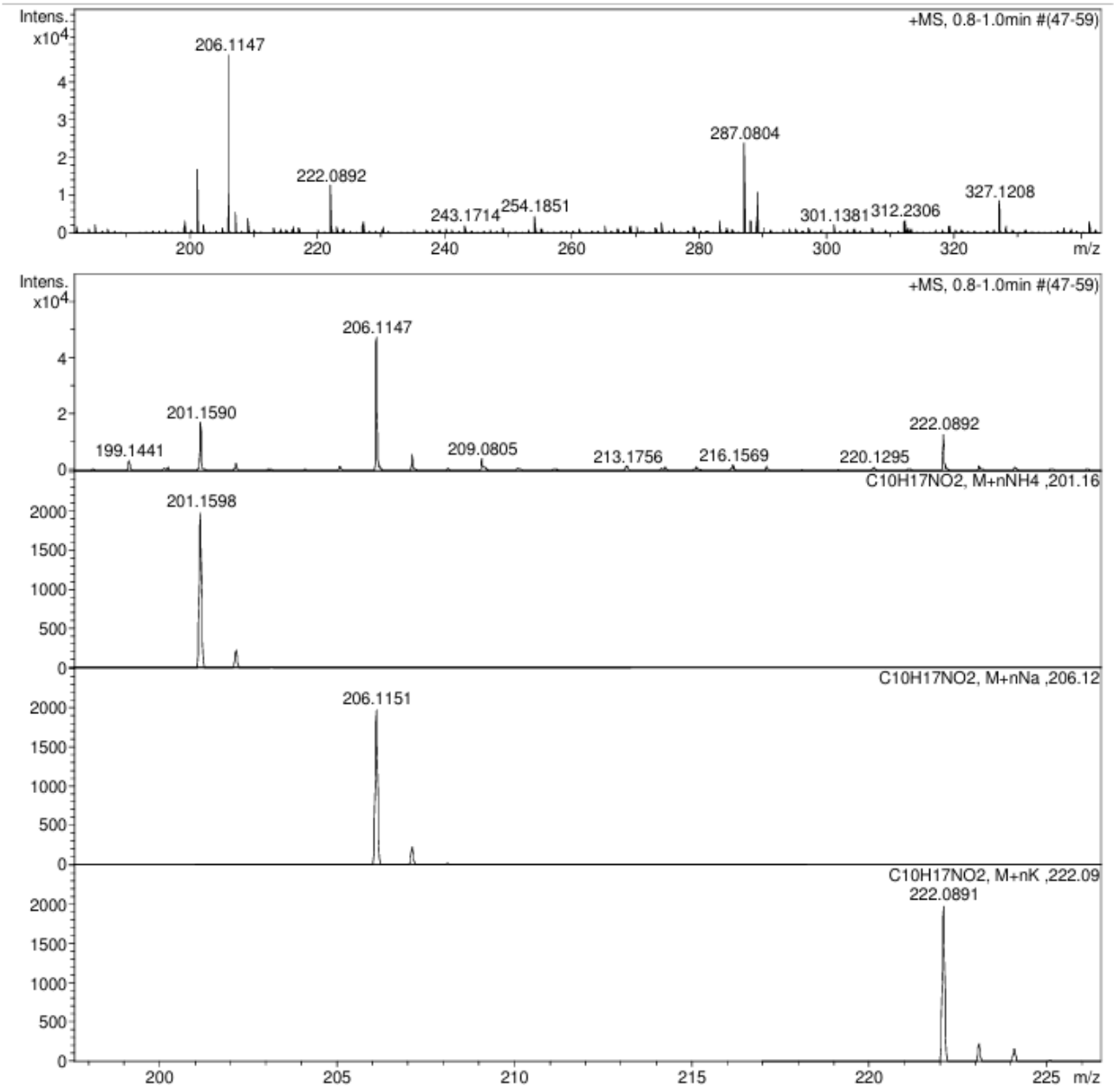
Display Report

Analysis Info

Analysis Name	D:\Data\Kolotyrkina\2025\Kirillov\0311005.d	Acquisition Date	11.03.2025 10:08:33
Method	tune_low.m	Operator	BDAL@DE
Sample Name	/VILV pil805_2k	Instrument / Ser#	micrOTOF 10248
Comment	C10H17NO2 clb added CH3CN		

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 ethyl 2-cyano-3,5-dimethylhexanoate, **3al**

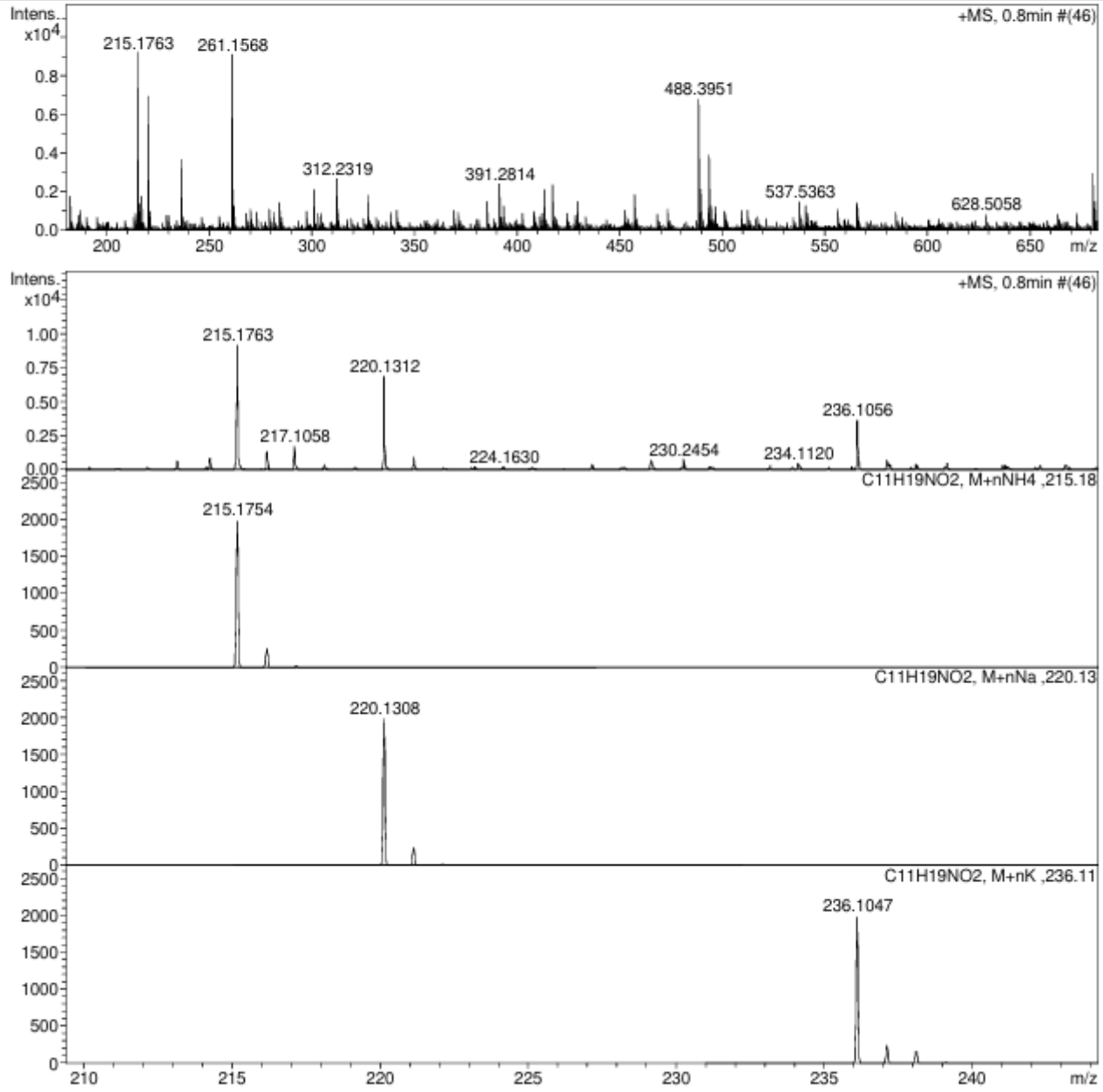
Display Report

Analysis Info

Analysis Name	D:\Data\Kolotyrkina\2025\Kirillov\0311004.d	Acquisition Date	11.03.2025 10:02:28
Method	tune_low.m	Operator	BDAL@DE
Sample Name	/VILV pil819_2R	Instrument / Ser#	micrOTOF 10248
Comment	C11H19NO2 clb added CH3CN		

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 ethyl 2-cyano-3-propylhexanoate, **3am**

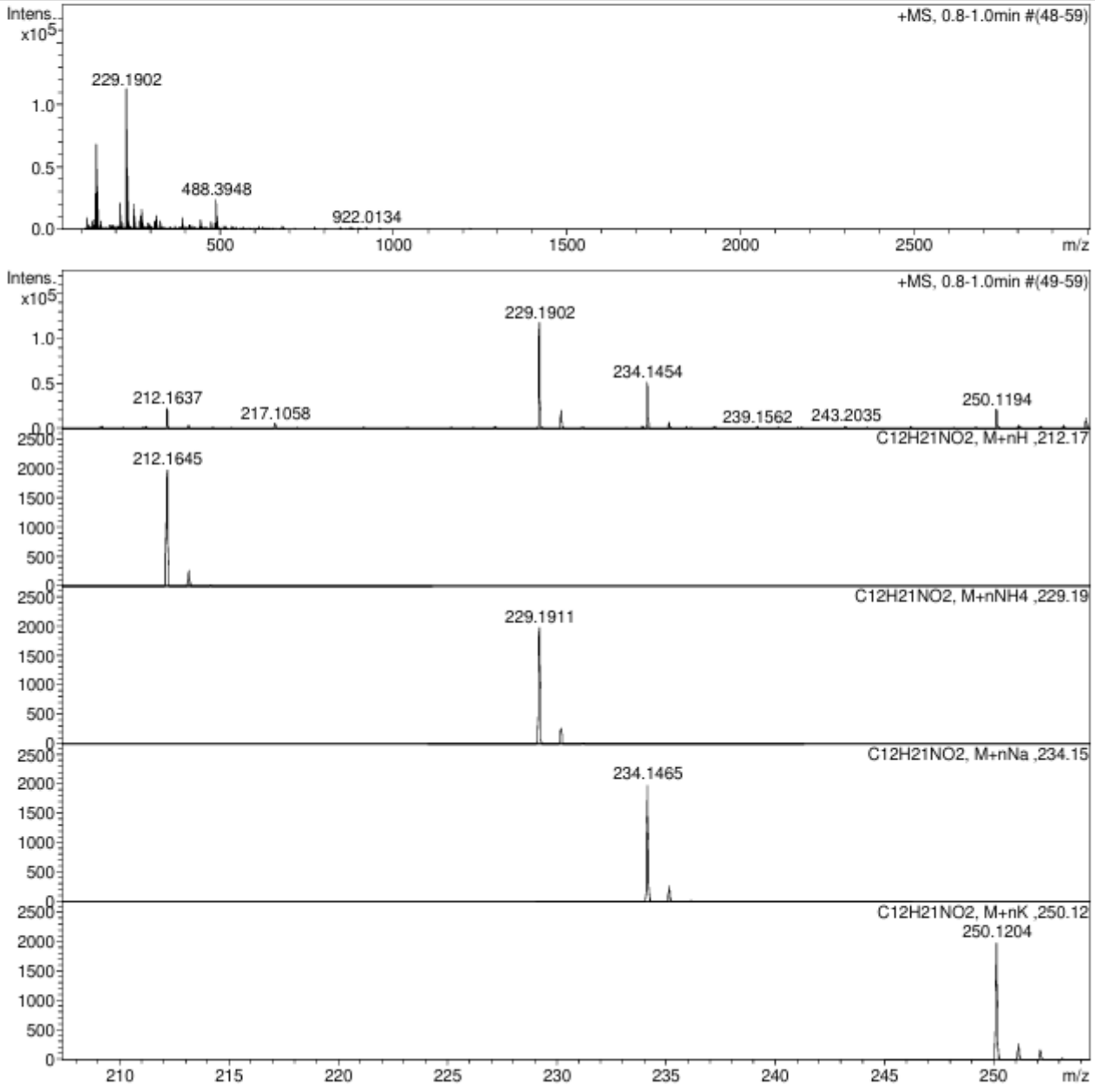
Display Report

Analysis Info

Analysis Name	D:\Data\Kolotyrkina\2025\Kirillov\0311003.d	Acquisition Date	11.03.2025 9:56:08
Method	tune_low.m	Operator	BDAL@DE
Sample Name	/VILV pil802_2m	Instrument / Ser#	micrOTOF 10248
Comment	C12H21NO2 clb added CH3CN		

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 ethyl 2-cyano-3-ethylheptanoate, **3an**

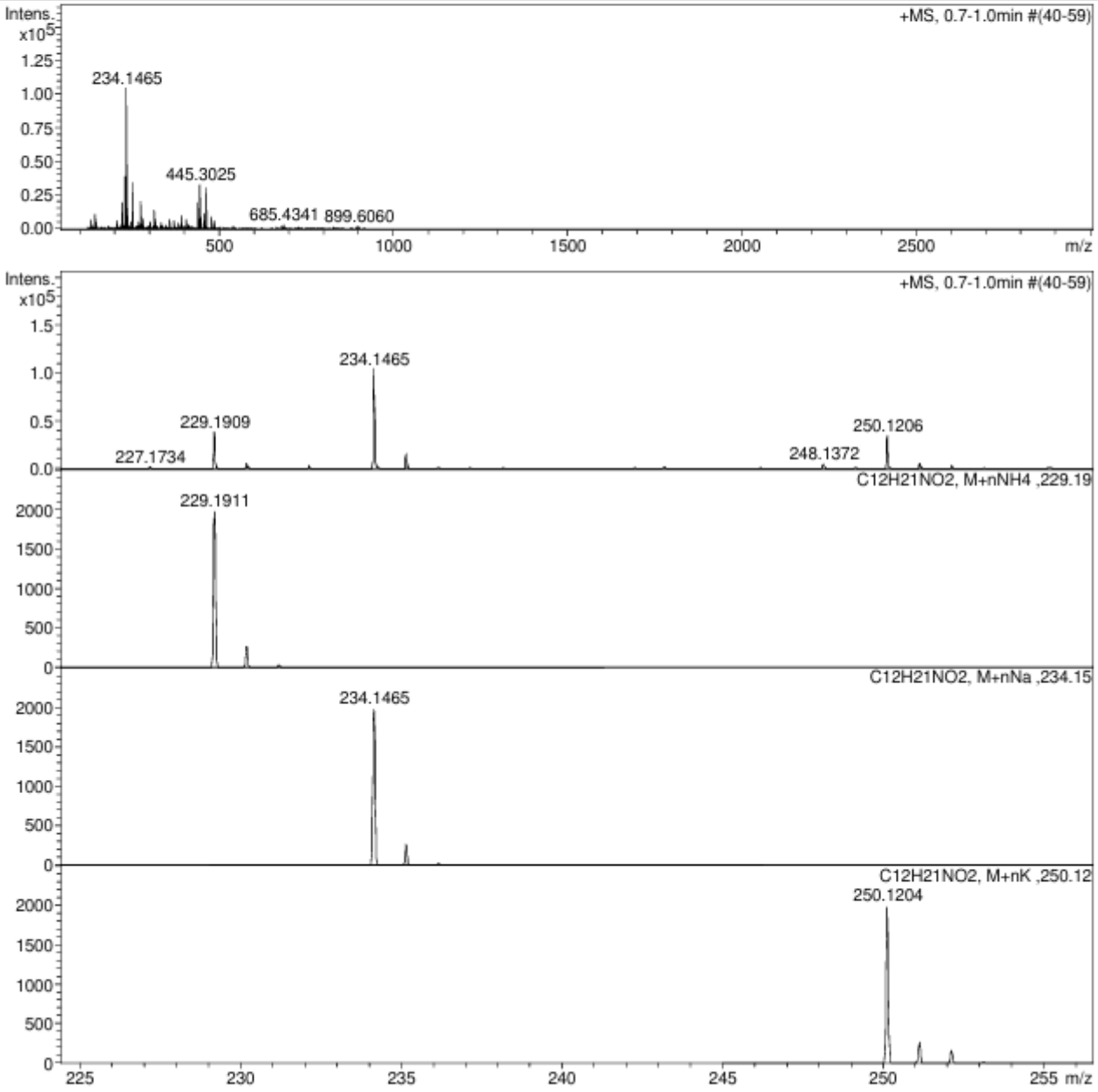
Display Report

Analysis Info

Analysis Name	D:\Data\Kolotyrkina\2025\Kirillov\0311002.d	Acquisition Date	11.03.2025 9:51:45
Method	tune_low.m	Operator	BDAL@DE
Sample Name	/VILV pil826_2m	Instrument / Ser#	micrOTOF 10248
Comment	C12H21NO2 clb added CH3CN		

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



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HRMS spectrum of ethyl 2-cyano-5-methyl-3-propylhexanoate, **3ao**

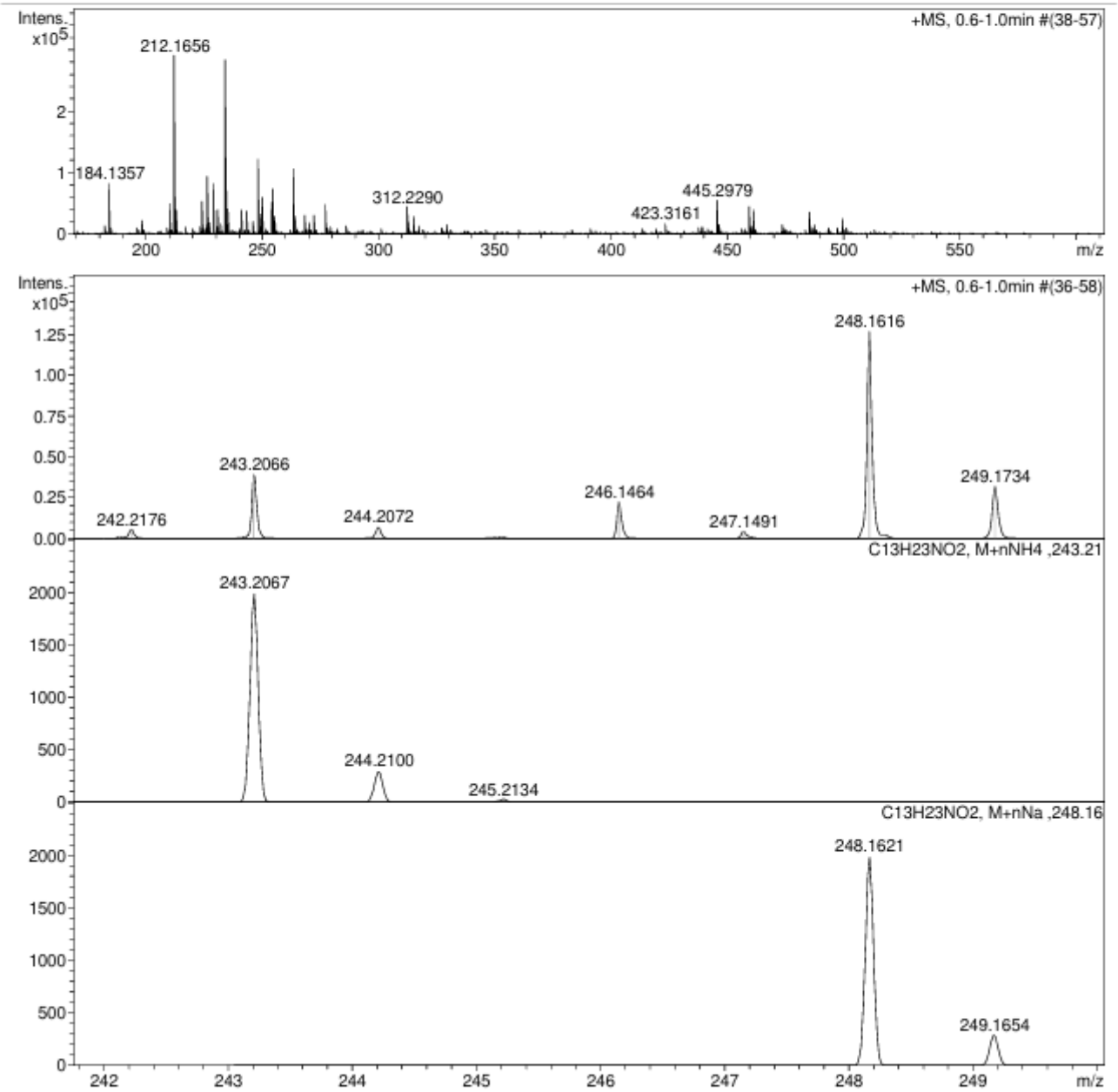
Display Report

Analysis Info

Analysis Name	D:\Data\Kolotyrkina\2025\Kirillov\0311009.d	Acquisition Date	11.03.2025 10:44:17
Method	tune_low.m	Operator	BDAL@DE
Sample Name	/VILV LT020	Instrument / Ser#	micrOTOF 10248
Comment	C13H23NO2 clb added CH3CN		

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 ethyl 2-cyano-3-methyl-5-phenylpentanoate, **3ap**

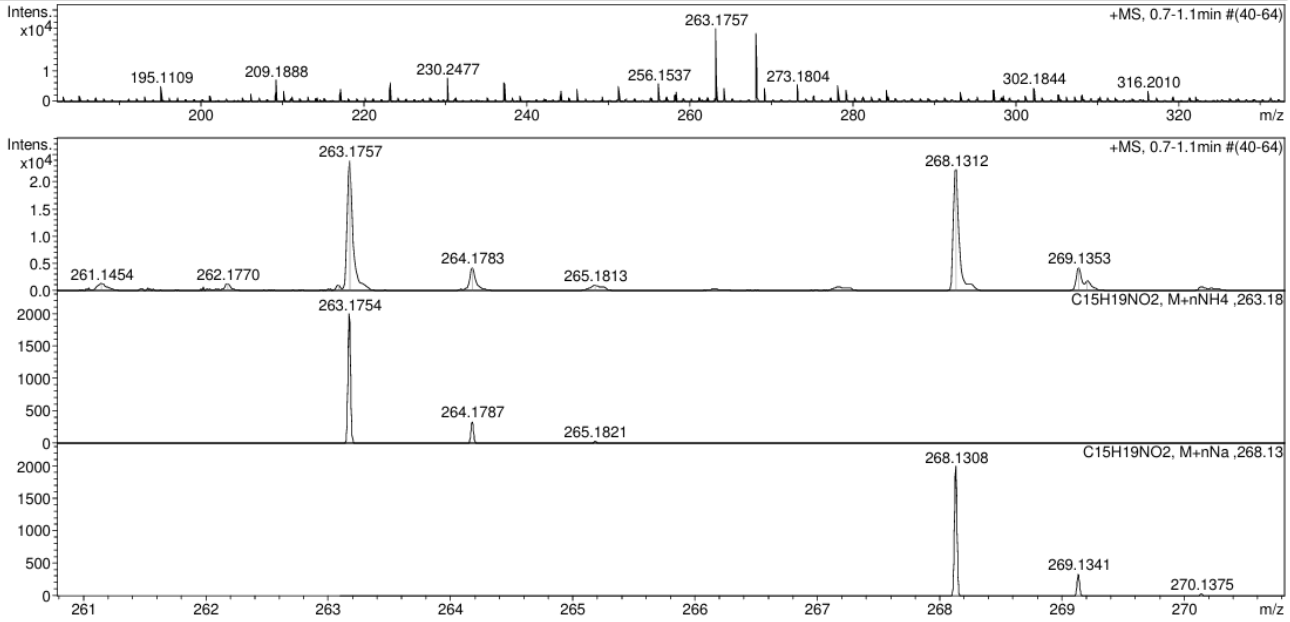
Display Report

Analysis Info

Analysis Name	D:\Data\Kolotyrkina\2025\Kirillov\0708007.d	Acquisition Date	08.07.2025 10:41:44
Method	tune_low.m	Operator	BDAL@DE
Sample Name	/VILV Zap	Instrument / Ser#	micrOTOF 10248
Comment	C15H19NO2 clb added CH3CN		

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 diethyl 2-cyano-3-methylheptanedioate, **3aq**

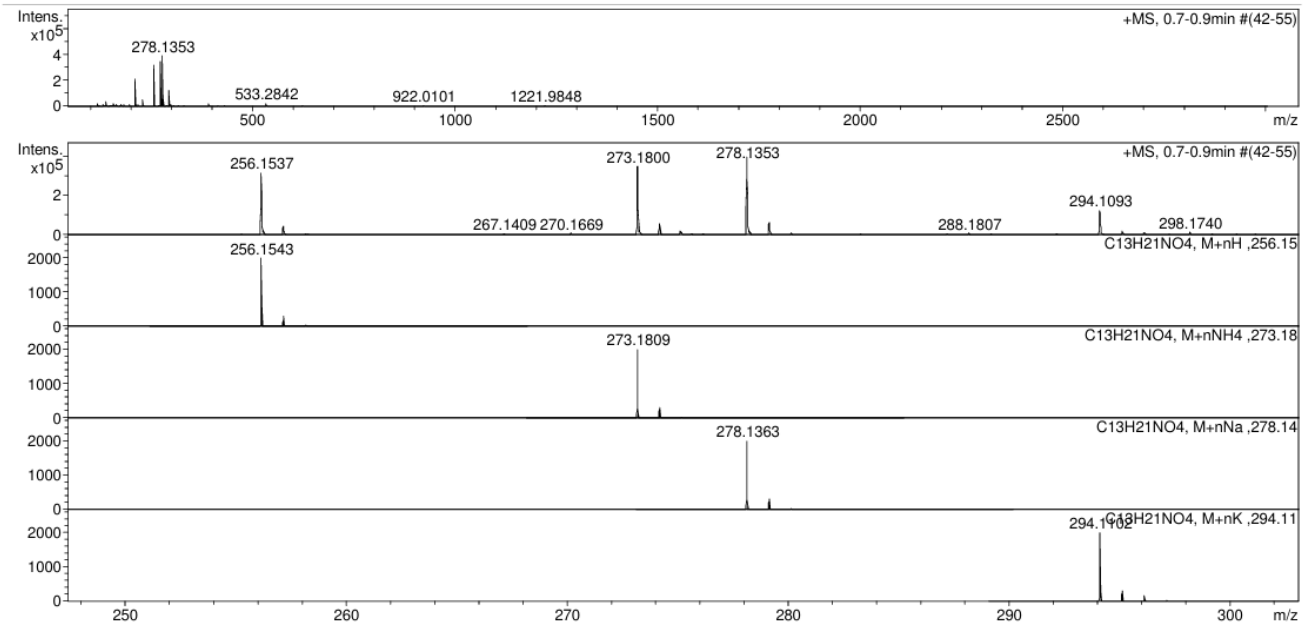
Display Report

Analysis Info

Analysis Name	D:\Data\Kolotyrkina\2025\Kirillov\0708006.d	Acquisition Date	08.07.2025 10:35:15
Method	tune_low.m	Operator	BDAL@DE
Sample Name	/VILV Zaq	Instrument / Ser#	micrOTOF 10248
Comment	C13H21NO4 clb added CH3CN		

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 2-cyano-2-cyclohexylacetamide, **3cb**

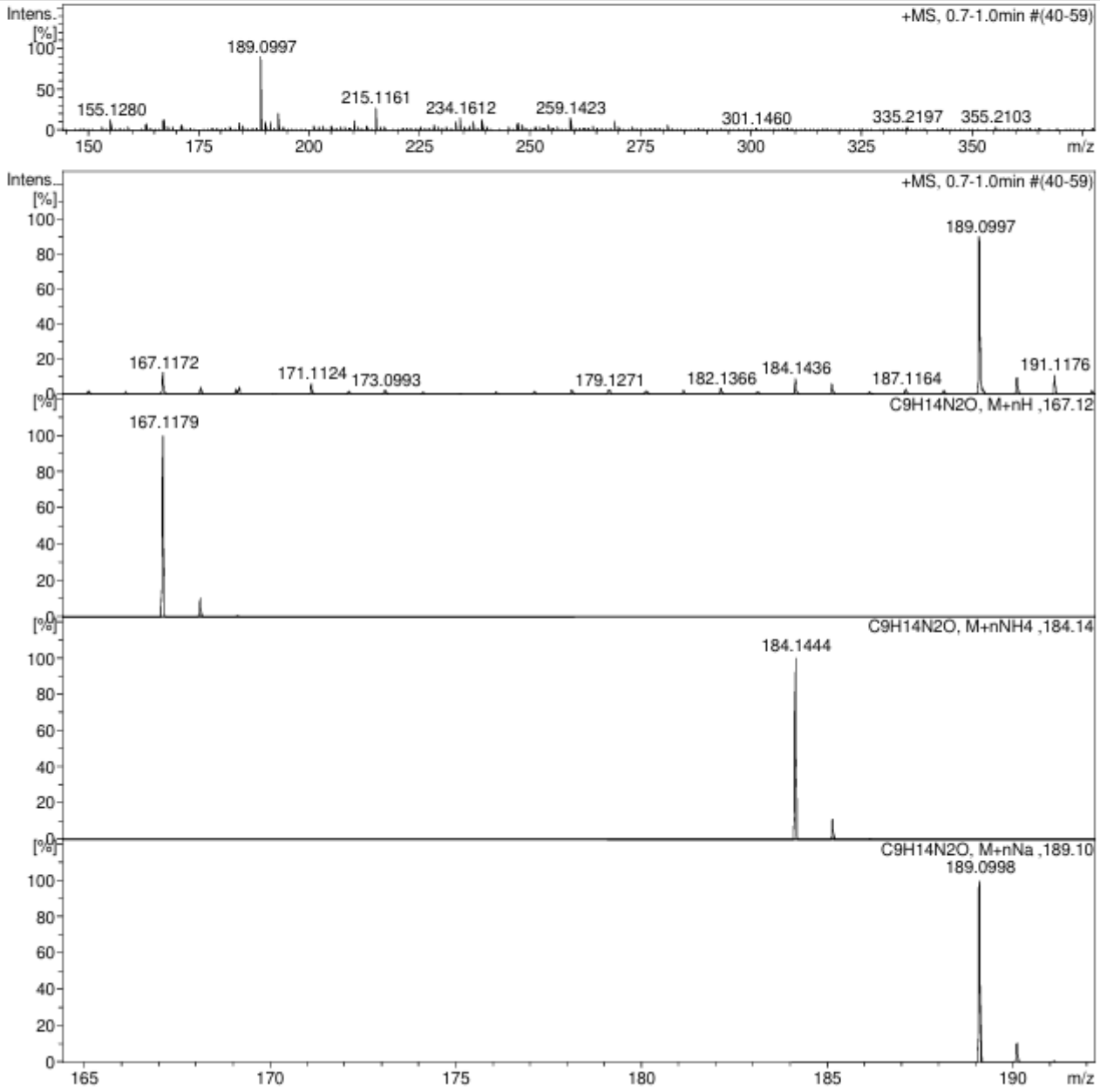
Display Report

Analysis Info

Analysis Name	D:\Data\Kolotyrkina\2025\Kirillov\0328032.d	Acquisition Date	28.03.2025 15:37:58
Method	tune_low.m	Operator	BDAL@DE
Sample Name	/VILV pil-3cb	Instrument / Ser#	micrOTOF 10248
Comment	C9H14N2O clb added CH3CN		

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 2-cyclohexyl-3-hydroxypropanenitrile, **5**

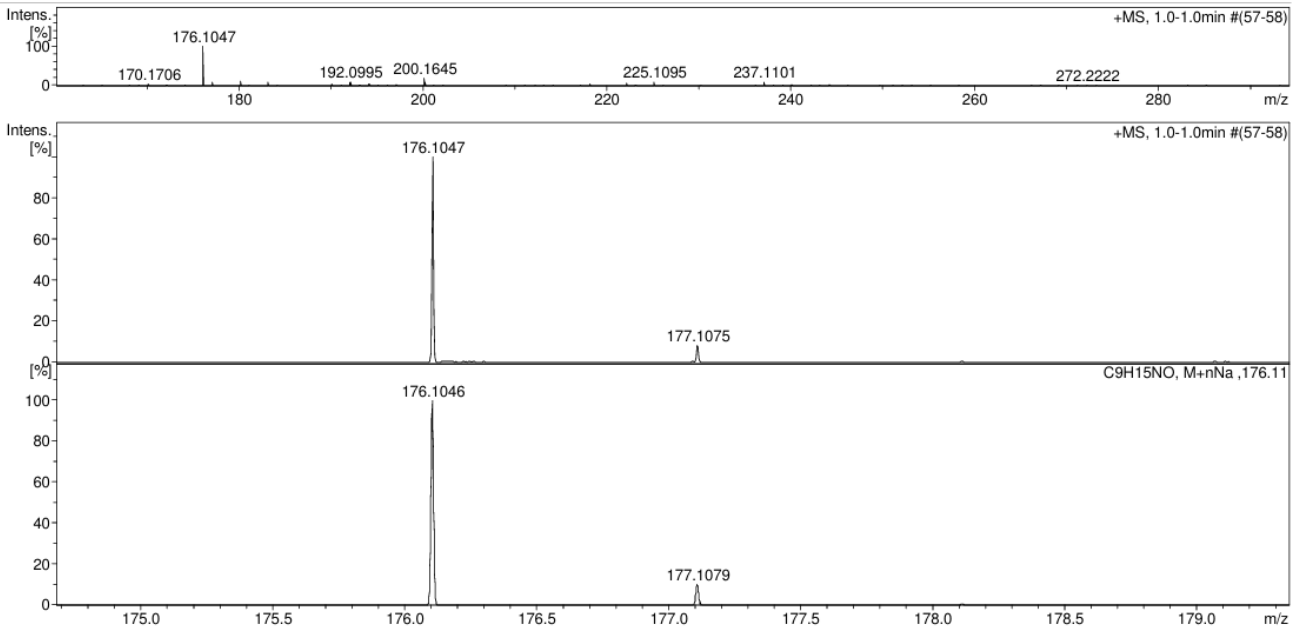
Display Report

Analysis Info

Analysis Name	D:\Data\Kolotyrkina\2025\Kirillov\0722012.d	Acquisition Date	22.07.2025 16:37:19
Method	tune_100-1200.m	Operator	BDAL@DE
Sample Name	/VILV pil1276_max	Instrument / Ser#	maXis 43
Comment	C9H15NO CH3OH		

Acquisition Parameter

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	1.0 Bar
Focus	Active			Set Dry Heater	200 °C
Scan Begin	50 m/z	Set Capillary	4500 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 2-cyano-2-cyclohexylethyl methanesulfonate, **6**

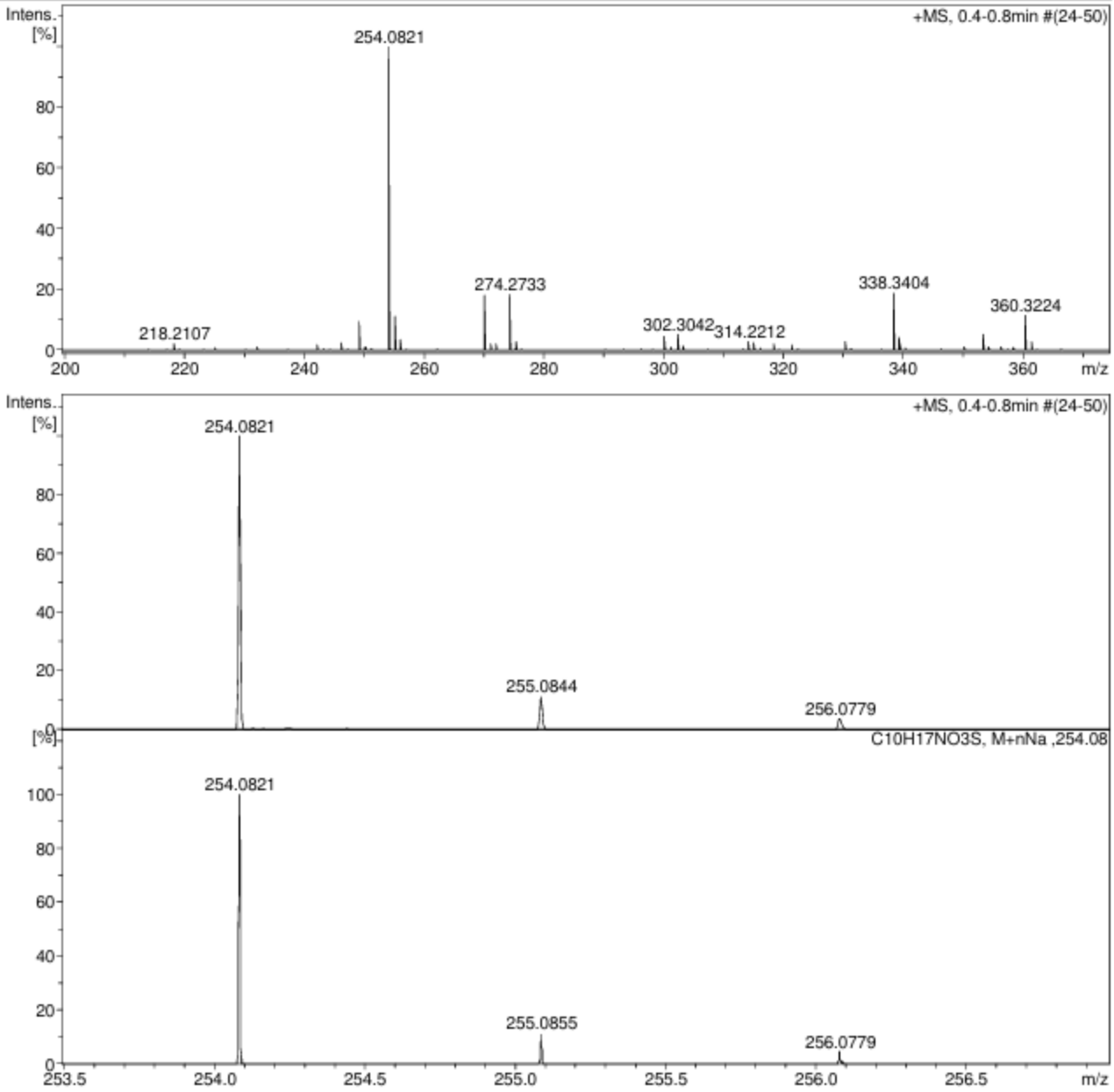
Display Report

Analysis Info

Analysis Name	D:\Data\Belyaev\lab13\pil1281-2.d	Acquisition Date	22.08.2025 14:29:41
Method	tune_100-1200.m	Operator	BDAL@DE
Sample Name	/VILV pil1281-2	Instrument / Ser#	maXis 43
Comment			

Acquisition Parameter

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	1.0 Bar
Focus	Active			Set Dry Heater	200 °C
Scan Begin	50 m/z	Set Capillary	4500 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 2-cyclohexylacrylonitrile, **7**

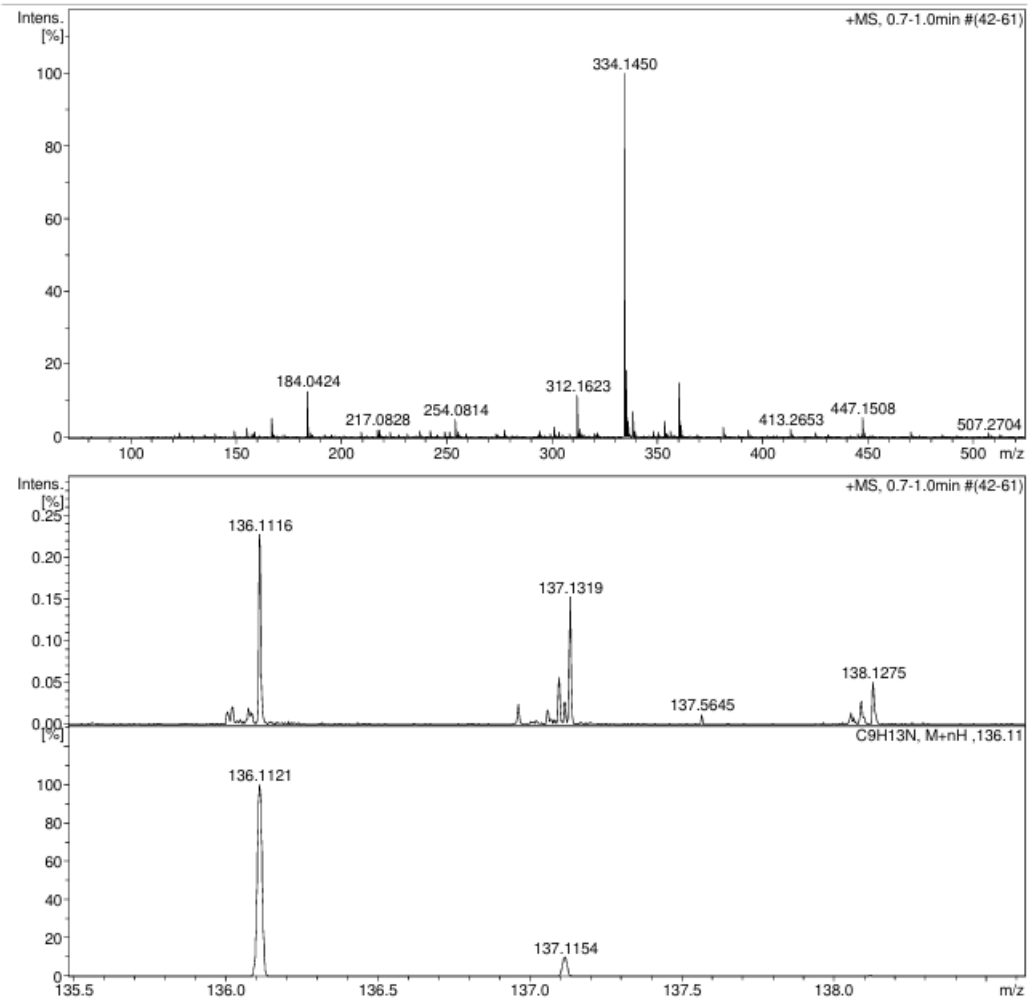
Display Report

Analysis Info

Analysis Name	D:\Data\Belyaev\lab7\pil1287.d	Acquisition Date	01.08.2025 21:39:02
Method	tune_100-1200.m	Operator	BDAL@DE
Sample Name	/VILV pil1287	Instrument / Ser#	maXis 43
Comment			

Acquisition Parameter

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	1.0 Bar
Focus	Active			Set Dry Heater	200 °C
Scan Begin	50 m/z	Set Capillary	4500 V	Set Dry Gas	4.0 l/min
Scan End	1500 m/z	Set End Plate Offset	-500 V	Set Divert Valve	Waste



HRMS spectrum of N-(2-cyclohexyl-3-hydroxypropyl)-4-methylbenzenesulfonamide, **8**

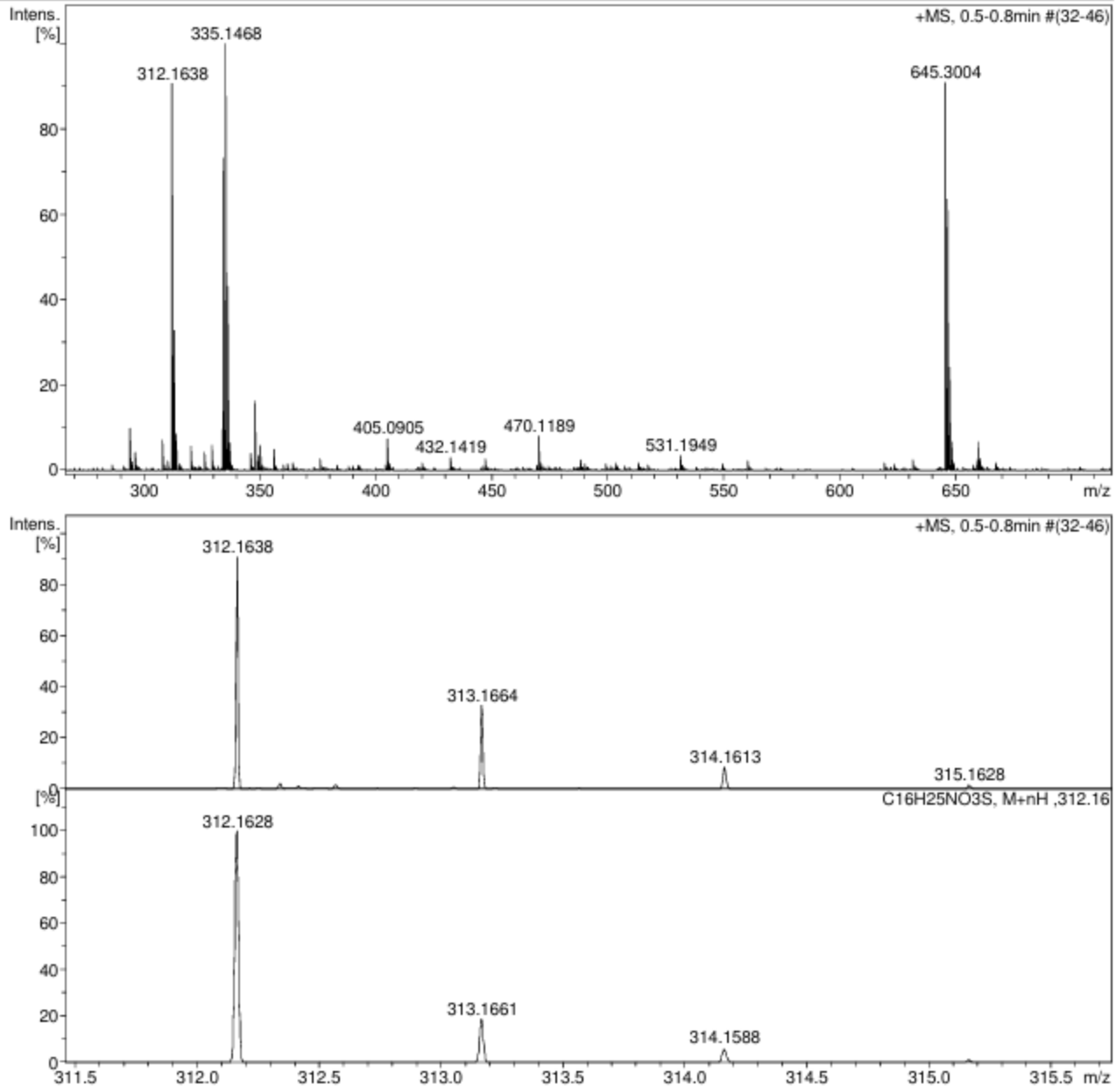
Display Report

Analysis Info

Analysis Name	D:\Data\Belyaev\lab7\pil1283.d	Acquisition Date	01.08.2025 21:27:13
Method	tune_100-1200.m	Operator	BDAL@DE
Sample Name	/VILV pil1283	Instrument / Ser#	maXis 43
Comment			

Acquisition Parameter

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	1.0 Bar
Focus	Active			Set Dry Heater	200 °C
Scan Begin	50 m/z	Set Capillary	4500 V	Set Dry Gas	4.0 l/min
Scan End	1500 m/z	Set End Plate Offset	-500 V	Set Divert Valve	Waste



HRMS spectrum of methyl 2-cyano-2-cyclohexylacetate-d_n, **3bb-d_n**

Display Report

Analysis Info

Analysis Name	D:\Data\Kolotyrkina\2025\Kirillov\0320007.d	Acquisition Date	20.03.2025 10:38:50
Method	tune_low.m	Operator	BDAL@DE
Sample Name	/VILV pil1185	Instrument / Ser#	micrOTOF 10248
Comment	C10H12D3NO2 clb added CH3CN		

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