

Oxidative C–O coupling as a new idea in the ‘click-like chemistry’: malonyl peroxides for the conjugation of two molecules

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General Experimental Part

Caution: Although we have encountered no difficulties in working with malonyl peroxides, precautions such as the use of safety shield, fume hood should be taken, the use of redox-active transition-metal salts, heating and vigorous shaking should be avoided!

NMR spectra were recorded on a commercial instrument (300 MHz for ^1H ; 126, 75 or 50 MHz for ^{13}C) in CDCl_3 or $\text{DMSO-}d_6$. The chemical shifts of CHCl_3 are 7.26 ppm for ^1H NMR and 77.16 ppm for ^{13}C NMR (central peak). The chemical shifts of $\text{DMSO-}d_6$ are 2.50 ppm for ^1H NMR (also signal of H_2O is observed at 3.30 ppm) and 39.52 ppm for ^{13}C NMR (central peak). IR spectra were recorded on a FT-IR spectrometer. High resolution mass spectra (HRMS) were measured using electrospray ionization (ESI) [S1]. The measurements were done in a positive ion mode (interface capillary voltage 4500 V); the mass ratio was from m/z 50 to m/z 3000 Da; external/internal calibration was done with Electrospray Calibrant Solution. A syringe injection was used for solutions in MeCN, MeOH or H_2O (flow rate $3 \mu\text{l min}^{-1}$). Nitrogen was applied as a dry gas; interface temperature was set at $180 \text{ }^\circ\text{C}$. The TLC analyses were carried out on standard silica-gel chromatography plates. The melting points were determined on a Kofler hot-stage apparatus. Chromatography was performed on silica gel (63-200 mesh).

Starting compounds. Solvents: methanol, dichloromethane, chloroform, petroleum ether (PE) (40/70), ethyl acetate (EA), tetrahydrofuran (THF) were purified before use by distillation. Diethyl cyclopropane-1,1-dicarboxylate, 1,1-cyclobutanedicarboxylic acid, hydrazine monohydrate, hydroxylamine hydrochloride, phenylbutazone, 1,3-dimethylbarbituric acid, benzaldehyde, $\text{CF}_3\text{CH}_2\text{OH}$, AcOH were used as purchased. Ethyl 2-acetylhexanoate [S2], ethyl 2-acetyloctanoate [S3], ethyl 2-benzyl-3-oxobutanoate [S4], were prepared according to the literature data.

Synthesis of malonyl peroxides

Malonyl peroxides **2a,b** were prepared according to the literature data [S5,S6].

Spirocyclopropylmalonyl peroxide, **2a**

According to a literature procedure [S5], diethyl cyclopropane-1,1-dicarboxylate (10.0 g, 0.054 mol) gave the desired malonyl peroxide **2a** as colorless needles in 85% yield (5.9 g, 0.046 mol). mp $90\text{--}91 \text{ }^\circ\text{C}$. (lit. [S5] mp $89\text{--}91 \text{ }^\circ\text{C}$). ^1H NMR (300 MHz, CDCl_3) δ 2.10 (s, 4H). ^{13}C NMR (75 MHz, CDCl_3) δ 172.3, 23.7, 19.9.

Spirocyclobutylmalonyl peroxide, 2b

Following the general procedure [S6], 1,1-cyclobutanedicarboxylic acid (5.0 g, 35.0 mmol) gave the title compound as colorless needles in 71% yield (3.5 g, 24.9 mmol). mp 63-64 °C (lit. mp 63 °C).

¹H NMR (300 MHz, CDCl₃) δ 2.71 (t, *J* = 8.1 Hz, 4H), 2.43-2.30 (m, 2H).

¹³C NMR (75 MHz, CDCl₃) δ 174.1, 40.6, 29.0, 16.4.

Synthesis of starting heterocycles

4-Butyl-5-methyl-1,2-dihydro-3H-pyrazol-3-one, 1a

Following the general procedure [S7], ethyl 2-acetylhexanoate (1862.5 mg, 10.0 mmol) was treated with hydrazine monohydrate (N₂H₄ 64-65%) (750.9 mg, 15.0 mmol) to afford **1a** (1341.6 mg, 8.7 mmol, 87%) as a colorless powder. mp 189 °C (decomp).

¹H NMR (300 MHz, DMSO-*d*₆) δ 10.30 (s, 2H), 2.18 (t, *J* = 7.3 Hz, 2H), 2.03 (s, 3H), 1.42-1.31 (m, 2H), 1.42-1.18 (m, 2H), 0.87 (t, *J* = 7.2 Hz, 3H).

¹³C NMR (75 MHz, DMSO-*d*₆) δ 159.8, 136.4, 100.6, 32.1, 21.8, 21.1, 13.9, 9.9.

4-Hexyl-5-methyl-1,2-dihydro-3H-pyrazol-3-one, 1b

Following the general procedure [S7], ethyl 2-acetyloctanoate (2143.1 mg, 10.0 mmol) was treated with hydrazine monohydrate (N₂H₄ 64-65%) (750.9 mg, 15.0 mmol) to afford **1b** (1476.4 mg, 8.1 mmol, 81%) as a colorless powder. mp 186-188 °C.

¹H NMR (300 MHz, DMSO-*d*₆) δ 10.12 (s, 2H), 2.16 (t, *J* = 7.3 Hz, 2H), 2.02 (s, 3H), 1.43-1.17 (m, 8H), 0.85 (t, *J* = 6.7 Hz, 3H).

¹³C NMR (75 MHz, DMSO-*d*₆) δ 159.7, 136.3, 100.8, 31.2, 29.8, 28.4, 22.1, 21.3, 14.0, 9.8.

4-Benzyl-5-methyl-1,2-dihydro-3H-pyrazol-3-one, 1c.[S8]

Following the general procedure [S7], ethyl 2-benzyl-3-oxobutanoate (2202.7 mg, 10.0 mmol) was treated with hydrazine monohydrate (N₂H₄ 64-65%) (750.9 mg, 15.0 mmol) to afford **1c** (1562.1 mg, 8.3 mmol, 83 %) as a colorless powder. mp 229 °C (lit. [S8] mp 232°C).

¹H NMR (300 MHz, DMSO-*d*₆) δ 7.27-7.07 (m, 5H), 3.56 (s, 2H), 2.01 (s, 3H).

¹³C NMR (75 MHz, DMSO-*d*₆) δ 159.7, 141.9, 137.0, 128.1, 128.0, 125.4, 100.0, 27.3, 10.0.

4-Benzyl-3-methylisoxazol-5(2H)-one, 8

Following the general procedure [S9], ethyl 2-benzyl-3-oxobutanoate (2.2 g, 10.0 mmol) gave the title compound **8** as a colorless powder in 80% yield (1.5 g, 8.0 mmol, tautomeric ratio imine/enamine = 2:3, determined by proton NMR in CDCl₃ at room temperature) after drying under vacuum.

¹H NMR (300 MHz, CDCl₃) δ 7.37-7.15 (m, 5H), 3.61 (t, *J* = 5.6 Hz, 0.4H_{imine}), 3.56 (s, 1.2H_{enamine}), 3.26 (dd, *J* = 14.3, 5.6 Hz, 0.4H_{imine}), 3.20 (dd, *J* = 14.3, 5.6 Hz, 0.4H_{imine}), 2.07 (s, 1.8H_{enamine}), 2.00 (s, 1.2H_{imine}).

¹³C NMR (75 MHz, CDCl₃) δ 177.8, 174.1, 166.1, 161.9, 139.0, 135.2, 129.9, 129.2, 129.0, 128.7, 128.3, 127.9, 126.5, 100.3, 48.7, 33.6, 27.7, 14.3, 11.0.

5-Benzyl-1,3-dimethylpyrimidine-2,4,6(1H,3H,5H)-trione 14 [S10]

Colorless powder, mp 125-126 °C.

¹H NMR (300 MHz, CDCl₃) δ 7.25-7.21 (m, 3H), 7.05-7.00 (m, 2H), 3.77 (t, *J* = 4.7 Hz, 1H), 3.46 (d, *J* = 4.7 Hz, 2H), 3.12 (s, 6H).

¹³C NMR (75 MHz, CDCl₃) δ 168.4, 151.1, 135.3, 129.0, 128.7, 127.9, 50.9, 33.0, 28.3.

General Experimental Procedure for Scheme 1.

Malonyl peroxide **2a,b** (140.9-156.3 mg, 1.10 mmol) was added with stirring to the solution of 3H-pyrazol-3-one **1a-c** (154.2-188.2 mg, 1.00 mmol) in CF₃CH₂OH (2 ml). The reaction mixture was stirred at 20-25 °C for 24 h, afterwards concentrated under a water-jet vacuum. Subsequently, THF (2 ml) was added, cooled to 0 °C, and ethyl chloroformate (173.6 mg, 1.6 mmol) and triethylamine (161.9 mg, 1.6 mmol) were added while stirring. After keeping at 0-10 °C for 30 min, the appropriate amine **4a-d** or alcohol **5** (2.00 mmol) (146.3 mg *n*-butylamine **4a**, 360.5 mg 2-amino-6-methoxybenzothiazole **4b**, 316.4 mg 1-(8-quinolinyl)methanamine **4c**, 174.2 mg morpholine **4d**, 318.4 mg quinoline-8-methanol **5**) was added. The mixture was stirred at 20-25 °C for 12 h. After that, the mixture was diluted with EtOAc (50 ml), and the organic layer was washed with H₂O (10 ml), 5 % aqueous NaHCO₃ solution (10 ml), 5 % aqueous HCl solution (10 ml), and H₂O (10 ml), dried over MgSO₄, filtered, and concentrated under a water-jet vacuum. The product **6a-e** or **7** was isolated by column chromatography on SiO₂ eluting with CH₂Cl₂-EtOAc in a linear gradient of EtOAc from 5 to 50 vol %.

4-Butyl-3-methyl-5-oxo-4,5-dihydro-1H-pyrazol-4-yl 1-(*N*-butylcarbamoyl)cyclopropane-1-carboxylate, 6a

Yield: 82% (276.7 mg, 0.82 mmol), colorless powder, mp 83-84 °C. R_f = 0.65 (PE:EtOAc = 1:1+2% AcOH).

¹H NMR (300 MHz, DMSO-*d*₆) δ 11.19 (s, 1H), 8.13 (s, 1H), 3.15-3.06 (m, 2H), 1.89 (s, 3H), 1.90-1.63 (m, 2H), 1.48-1.05 (m, 12H), 0.86 (t, *J* = 7.3 Hz, 3H), 0.84 (t, *J* = 7.3 Hz, 3H).

¹³C NMR (75 MHz, DMSO-*d*₆) δ 173.5, 170.2, 166.2, 156.3, 80.3, 38.7, 32.1, 31.1, 27.5, 22.6, 21.9, 19.5, 17.3, 17.3, 13.6, 12.7.

HRMS (ESI) m/z $[M+Na]^+$. Calcd for $[C_{17}H_{27}N_3NaO_4]^+$: 360.1894. Found 360.1881.

Anal. Calcd for $C_{17}H_{27}N_3O_4$ C: 60.51 %, H: 8.07 %, N: 12.45 %. Found C: 60.58 %, H: 8.12 %, N: 12.52 %.

IR (KBr): 3355, 3238, 2964, 2936, 2875, 1735, 1716, 1645, 1541, 1355, 1152, 1101, 756 cm^{-1} .

4-Hexyl-3-methyl-5-oxo-4,5-dihydro-1H-pyrazol-4-yl 1-[N-(6-methoxybenzo[d]thiazol-2-yl)carbamoyl]cyclopropane-1-carboxylate, 6b

Yield: 76% (359.2 mg, 0.76 mmol), yellow powder, mp 90-91 °C. R_f = 0.61 (CH_2Cl_2 :MeOH = 10:1).

1H NMR (300 MHz, DMSO- d_6) δ 12.58 (s, 1H), 11.21 (s, 1H), 7.66-7.55 (m, 2H), 7.03 (dd, J = 8.8, 2.3 Hz, 1H), 3.80 (s, 3H), 1.97 (s, 3H), 1.67-1.57 (m, 3H), 1.52-1.47 (m, 1H), 1.27-1.04 (m, 10H), 0.69 (t, J = 6.6 Hz, 3H).

^{13}C NMR (75 MHz, DMSO- d_6) δ 173.4, 168.9, 156.5, 156.2, 142.6, 132.8, 121.2, 115.0, 114.6, 104.7, 80.4, 55.6, 32.5, 30.8, 29.3, 28.4, 21.8, 20.5, 16.9, 13.7, 13.0.

HRMS (ESI) m/z $[M+Na]^+$. Calcd for $[C_{23}H_{28}N_4NaO_5S]^+$: 495.1673. Found 495.1673.

Anal. Calcd for $C_{23}H_{28}N_4O_5S$ C: 58.46 %, H: 5.97 %, N: 11.86 %, S: 6.79 %. Found C: 58.82 %, H: 5.91 %, N: 11.47 %, S: 6.46 %.

IR (KBr): 3288, 2954, 2930, 1718, 1606, 1549, 1471, 1437, 1265, 1145, 1058, 828, 568 cm^{-1} .

4-Hexyl-3-methyl-5-oxo-4,5-dihydro-1H-pyrazol-4-yl 1-[N-(quinolin-8-ylmethyl)carbamoyl]cyclobutane-1-carboxylate, 6c

Yield: 64% (297.3 mg, 0.64 mmol), colorless powder, mp 98-99 °C. R_f = 0.67 (CH_2Cl_2 :MeOH = 10:1).

1H NMR (300 MHz, DMSO- d_6) δ 11.21 (s, 1H), 8.95 (s, 1H), 8.52-8.48 (m, 1H), 8.38 (d, J = 7.8 Hz, 1H), 7.88 (d, J = 7.4 Hz, 1H), 7.69-7.54 (m, 3H), 5.10-4.89 (m, 2H), 2.72-2.65 (m, 1H), 2.59-2.34 (m, 4H), 1.93-1.58 (m, 6H), 1.35-1.08 (m, 8H), 0.91-0.76 (m, 3H).

^{13}C NMR (126 MHz, DMSO- d_6) δ 173.7, 170.6, 169.7, 156.3, 149.7, 145.4, 136.6, 136.4, 127.7, 126.8, 126.4, 126.1, 121.4, 80.3, 52.8, 39.0, 32.4, 30.9, 29.0, 28.7, 28.3, 21.9, 20.3, 15.7, 13.9, 12.6.

HRMS (ESI) m/z $[M+Na]^+$. Calcd for $[C_{26}H_{32}N_4NaO_4]^+$: 487.2316. Found 487.2319.

Anal. Calcd for $C_{26}H_{32}N_4O_4$ C: 67.22 %, H: 6.94 %, N: 12.06 %. Found C: 67.28 %, H: 7.01 %, N: 12.08 %.

IR (KBr): 3435, 3348, 2925, 1742, 1729, 1664, 1528, 1104, 799, 618, 564 cm^{-1} .

4-Benzyl-3-methyl-5-oxo-4,5-dihydro-1H-pyrazol-4-yl 1-(morpholin-4-ylcarbonyl)-cyclopropane-1-carboxylate, 6d

Yield: 71% (273.6 mg, 0.71 mmol), colorless powder, mp 91-92 °C. $R_f = 0.21$ (CH_2Cl_2 :MeOH = 10:1).

^1H NMR (300 MHz, $\text{DMSO-}d_6$) δ 11.06 (s, 1H), 7.31-7.24 (m, 3H), 7.20-7.13 (m, 2H), 3.66-3.41 (m, 8H), 3.17 (d, $J = 13.4$ Hz, 1H), 3.01 (d, $J = 13.4$ Hz, 1H), 1.81 (s, 3H), 1.49-1.34 (m, 4H).

^{13}C NMR (75 MHz, $\text{DMSO-}d_6$) δ 172.9, 169.1, 164.7, 155.3, 131.7, 130.2, 128.0, 127.4, 81.1, 65.8, 65.7, 45.9, 42.2, 38.6, 27.5, 16.4, 16.1, 13.4.

HRMS (ESI) m/z $[\text{M}+\text{Na}]^+$. Calcd for $[\text{C}_{20}\text{H}_{23}\text{N}_3\text{NaO}_5]^+$: 408.1530. Found 408.1518.

Anal. Calcd for $\text{C}_{20}\text{H}_{23}\text{N}_3\text{O}_5$ C: 62.33 %, H: 6.01 %, N: 10.90 %. Found C: 62.52 %, H: 6.04 %, N: 10.66 %.

IR (KBr): 3435, 3271, 2923, 1735, 1629, 1438, 1384, 1307, 1152, 1114, 1034, 702, 599 cm^{-1} .

4-Benzyl-3-methyl-5-oxo-4,5-dihydro-1H-pyrazol-4-yl 1-[N-(quinolin-8-ylmethyl)-carbamoyl]cyclopropane-1-carboxylate, 6e

Yield: 85% (388.0 mg, 0.85 mmol), yellow powder, mp 89-90 °C. $R_f = 0.52$ (CH_2Cl_2 :MeOH = 10:1).

^1H NMR (300 MHz, $\text{DMSO-}d_6$) δ 11.06 (s, 1H), 8.96-8.87 (m, 2H), 8.38 (dd, $J = 8.1, 1.6$ Hz, 1H), 7.88 (d, $J = 8.1$ Hz, 1H), 7.68-7.53 (m, 4H), 7.28-7.25 (m, 2H), 7.15-7.12 (m, 1H), 5.00-4.93 (m, 2H), 3.07 (d, $J = 13.2$ Hz, 1H), 2.97 (d, $J = 13.2$ Hz, 1H), 2.08 (s, 3H), 1.88-1.85 (m, 2H), 1.57-1.52 (m, 2H).

^{13}C NMR (75 MHz, $\text{DMSO-}d_6$) δ 173.0, 170.5, 170.1, 166.6, 155.4, 149.8, 145.5, 136.4, 136.3, 131.6, 130.2, 128.0, 127.8, 127.4, 127.1, 127.0, 126.8, 126.2, 121.5, 80.8, 38.5, 30.7, 27.5, 18.0, 15.8, 13.3.

HRMS (ESI) m/z $[\text{M}+\text{Na}]^+$. Calcd for $[\text{C}_{26}\text{H}_{24}\text{N}_4\text{NaO}_4]^+$: 479.1690. Found 479.1675.

Anal. Calcd for $\text{C}_{26}\text{H}_{24}\text{N}_4\text{O}_4$ C: 68.41 %, H: 5.30 %, N: 12.27 %. Found C: 68.53 %, H: 5.38 %, N: 12.15 %.

IR (KBr): 3375, 3255, 1802, 1721, 1655, 1530, 1499, 1337, 1142, 1085, 792, 701 cm^{-1} .

4-Benzyl-3-methyl-5-oxo-4,5-dihydro-1H-pyrazol-4-yl (quinolin-8-yl)methyl cyclopropane-1,1-dicarboxylate, 7

Yield: 67% (306.5 mg, 0.67 mmol), colorless powder, mp 66-67 °C. $R_f = 0.68$ (CH_2Cl_2 :MeOH = 10:1).

¹H NMR (300 MHz, DMSO-*d*₆) δ 10.96 (s, 1H), 8.98 (d, *J* = 4.5 Hz, 1H), 8.42 (d, *J* = 7.3 Hz, 1H), 8.01 (d, *J* = 8.1 Hz, 1H), 7.90 (d, *J* = 7.1 Hz, 1H), 7.68-7.57 (m, 2H), 7.25-7.21 (m, 3H), 7.05-6.98 (m, 2H), 5.91 (d, *J* = 13.1 Hz, 1H), 5.83 (d, *J* = 13.1 Hz, 1H), 2.87 (d, *J* = 13.2 Hz, 1H), 2.82 (d, *J* = 13.2 Hz, 1H), 1.61-1.37 (m, 7H).

¹³C NMR (75 MHz, DMSO-*d*₆) δ 172.7, 168.2, 167.0, 154.7, 150.2, 145.3, 136.4, 133.1, 131.3, 129.9, 128.7, 128.5, 128.0, 127.7, 127.4, 126.2, 121.7, 81.1, 63.5, 38.3, 27.5, 16.5, 16.4, 12.7.

HRMS (ESI) *m/z* [M+Na]⁺. Calcd for [C₂₆H₂₃N₃NaO₅]⁺: 480.1530. Found 480.1519.

Anal. Calcd for C₂₆H₂₃N₃O₅: C: 68.26 %, H: 5.07 %, N: 9.19 %. Found C: 68.07 %, H: 5.21 %, N: 9.05 %.

IR (KBr): 3327, 3033, 1800, 1734, 1500, 1318, 1198, 1126, 1076, 701, 570 cm⁻¹.

Experimental Procedure for Scheme 2.

Malonyl peroxide **2a** (140.9 mg, 1.10 mmol) was added with stirring to a solution of isoxazol-5(2*H*)-one **8** (189.2 mg, 1.00 mmol) in CF₃CH₂OH (2 ml). The mixture was stirred at 20-25 °C for 24 h, afterwards concentrated under a water-jet vacuum. Subsequently, THF (2 ml) was added, cooled to 0 °C, and ethyl chloroformate (173.6 mg, 1.6 mmol) and triethylamine (161.9 mg, 1.6 mmol) were added while stirring. After 30 min at 0-10 °C, 1-(quinolin-8-yl)methanamine **4c** (316.4 mg, 2.00 mmol) was added. The mixture was stirred at 20-25 °C for 12 h. After that, the mixture was diluted with EtOAc (50 ml), and the organic layer was washed with H₂O (10 ml), 5 % aqueous NaHCO₃ solution (10 ml), 5 % aqueous HCl solution (10 ml), and H₂O (10 ml), dried over MgSO₄, filtered, and concentrated under a water-jet vacuum. The product **10** was isolated by column chromatography on SiO₂ eluting with CH₂Cl₂-EtOAc in a linear gradient of EtOAc from 5 to 50 vol %.

4-Benzyl-3-methyl-5-oxo-4,5-dihydroisoxazol-4-yl 1-[*N*-(quinolin-8-ylmethyl)carbamoyl]-cyclopropane-1-carboxylate, 10

Yield: 51% (233.3 mg, 0.51 mmol), colorless powder, mp 153-154 °C. R_f = 0.29 (CH₂Cl₂:MeOH = 10:1).

¹H NMR (300 MHz, DMSO-*d*₆) δ 8.95-8.92 (m, 1H), 8.91-8.84 (m, 1H), 8.38 (d, *J* = 8.1 Hz, 1H), 7.88 (d, *J* = 7.9 Hz, 1H), 7.67 (d, *J* = 6.8 Hz, 1H), 7.62-7.54 (m, 2H), 7.36-7.28 (m, 3H), 7.20-7.12 (m, 2H), 4.99 (d, *J* = 5.6 Hz, 2H), 3.31 (d, *J* = 14.7 Hz, 1H), 3.23 (d, *J* = 14.7 Hz, 1H), 2.08 (s, 3H), 1.67-1.51 (m, 4H).

¹³C NMR (75 MHz, DMSO-*d*₆) δ 172.9, 170.8, 166.0, 164.7, 149.7, 145.5, 136.4, 136.2, 130.3, 130.2, 128.4, 128.0, 127.8, 127.1, 126.2, 121.5, 79.5, 38.0, 27.8, 18.1, 11.2.

HRMS (ESI) *m/z* [M+Na]⁺. Calcd for [C₂₆H₂₃N₃NaO₅]⁺: 480.1530. Found 480.1518.

Anal. Calcd for C₂₆H₂₃N₃O₅ C: 68.26 %, H: 5.07 %, N: 9.19 %. Found C: 68.29 %, H: 5.01 %, N: 9.17 %.

IR (KBr): 3398, 3026, 2939, 1799, 1721, 1651, 1529, 1346, 1129, 854, 708, 557 cm⁻¹.

Experimental Procedure for Scheme 3.

Malonyl peroxide **2b** (156.3 mg, 1.10 mmol) was added with stirring to a solution of phenylbutazone **11** (308.4 mg, 1.00 mmol) in CF₃CH₂OH (2 ml). The mixture was stirred at 20–25 °C for 24 h, afterwards concentrated under a water-jet vacuum. Subsequently, THF (2 ml) was added, cooled to 0 °C, and ethyl chloroformate (173.6 mg, 1.6 mmol) and triethylamine (161.9 mg, 1.6 mmol) were added while stirring. After 30 min at 0–10 °C, (1-phenyl-1*H*-pyrazol-5-yl)methanamine **4e** (346.4 mg, 2.00 mmol) was added. The mixture was stirred at 20–25 °C for 12 h. After that, the mixture was diluted with EtOAc (50 ml), and the organic layer was washed with H₂O (10 ml), 5 % aqueous NaHCO₃ solution (10 ml), 5% aqueous HCl solution (10 ml), and H₂O (10 ml), dried over MgSO₄, filtered, and concentrated under a water-jet vacuum. The product **13** was isolated by column chromatography on SiO₂ eluting with CH₂Cl₂-EtOAc in a linear gradient of EtOAc from 5 to 50 vol %.

4-Butyl-3,5-dioxo-1,2-diphenylpyrazolidin-4-yl 1-[*N*-(1-phenyl-1*H*-pyrazol-5-ylmethyl)carbamoyl]cyclobutane-1-carboxylate, 13

Yield: 73% (442.2 mg, 0.73 mmol), colorless powder, mp 67-68 °C. R_f = 0.72 (CH₂Cl₂:MeOH = 10:1).

¹H NMR (300 MHz, DMSO-*d*₆) δ 8.65 (t, *J* = 5.5 Hz, 1H), 7.59-7.52 (m, 5H), 7.46-7.38 (m, 9H), 7.30-7.22 (m, 2H), 6.48 (m, 1H), 4.41 (d, *J* = 5.5 Hz, 2H), 2.50-2.38 (m, 4H), 2.08-2.00 (m, 2H), 1.92-1.70 (m, 2H), 1.45-1.20 (m, 4H), 0.83 (t, *J* = 7.1 Hz, 3H).

¹³C NMR (75 MHz, DMSO-*d*₆) δ 171.7, 168.8, 167.1, 140.7, 139.6, 135.2, 129.2, 129.1, 127.7, 127.4, 124.5, 123.6, 123.4, 106.5, 75.8, 52.1, 35.4, 33.2, 28.7, 23.4, 21.8, 15.5, 13.5.

HRMS (ESI) *m/z* [M+Na]⁺. Calcd for [C₃₅H₃₅N₅NaO₅]⁺: 628.2530. Found 628.2517.

Anal. Calcd for C₃₅H₃₅N₅O₅ C: 69.41 %, H: 5.82 %, N: 11.66 %. Found C: 69.04 %, H: 6.05 %, N: 11.29 %.

IR (KBr): 3351, 3065, 2957, 2872, 1768, 1732, 1677, 1597, 1502, 1292, 1102, 759, 691 cm⁻¹.

Experimental Procedure for Scheme 4.

Malonyl peroxide **2a** (140.9 mg, 1.10 mmol) was added with stirring to a solution of barbituric derivative **14** (246.3 mg, 1.00 mmol) in CF₃CH₂OH (2 ml). The mixture was stirred at 20-25 °C for 24 h, afterwards concentrated under a water-jet vacuum. Subsequently, THF (2 ml) was added, cooled to 0 °C, and ethyl chloroformate (173.6 mg, 1.6 mmol) and triethylamine (161.9 mg, 1.6 mmol) were added while stirring. After 30 min at 0–10 °C, (1-phenyl-1*H*-pyrazol-5-yl)methanamine **4e** (346.4 mg, 2.00 mmol) was added. The mixture was stirred at 20-25 °C for 12 h. After that, the mixture was diluted with EtOAc (50 ml), and the organic layer was washed with H₂O (10 ml), 5 % aqueous NaHCO₃ solution (10 ml), 5 % aqueous HCl solution (10 ml), and H₂O (10 ml), dried over MgSO₄, filtered, and concentrated under a water-jet vacuum. The product **16** was isolated by column chromatography on SiO₂ eluting with CH₂Cl₂-EtOAc in a linear gradient of EtOAc from 5 to 50 vol %.

5-Benzyl-1,3-dimethyl-2,4,6-trioxohexahydropyrimidin-5-yl 1-[*N*-(1-phenyl-1*H*-pyrazol-5-ylmethyl)carbamoyl]cyclopropane-1-carboxylate, **16**

Yield: 73% (386.6 mg, 0.73 mmol), colorless oil. R_f = 0.62 (CH₂Cl₂:MeOH = 10:1).

¹H NMR (300 MHz, CDCl₃) δ 8.26-8.22 (m, 1H), 7.61 (s, 1H), 7.47-7.35 (m, 5H), 7.30-7.26 (m, 3H), 6.97-6.91 (m, 2H), 6.38 (m, 1H), 4.52 (d, *J* = 5.3 Hz, 2H), 3.30 (s, 2H), 3.09 (s, 6H), 1.89-1.78 (m, 4H).

¹³C NMR (75 MHz, CDCl₃) δ 172.86, 167.26, 166.90, 149.47, 140.15, 139.57, 129.93, 129.41, 129.36, 129.05, 128.84, 128.32, 125.22, 125.12, 106.88, 78.76, 43.88, 35.69, 28.64, 26.28, 21.30.

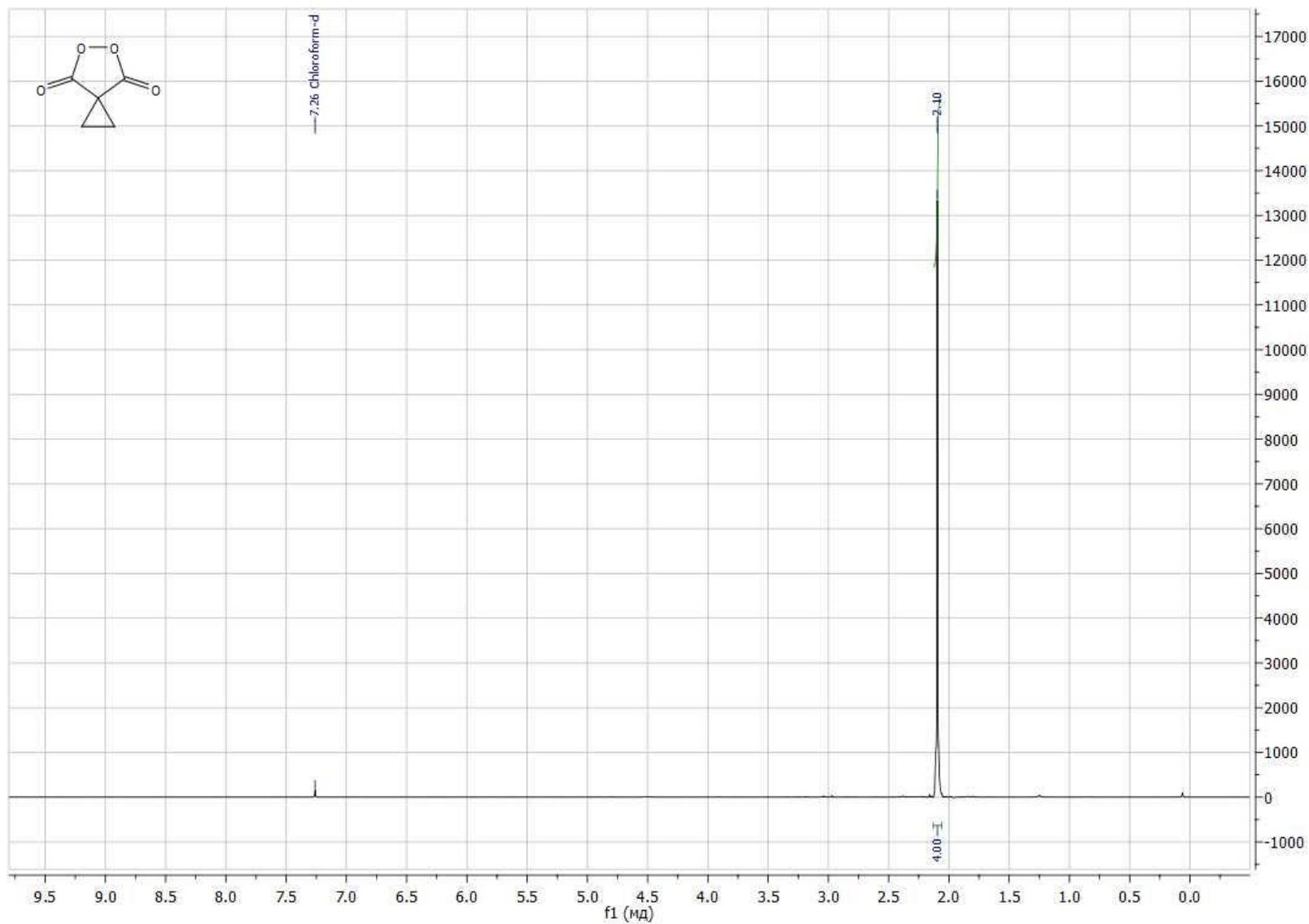
HRMS (ESI) *m/z* [M+H]⁺. Calcd for [C₂₈H₂₈N₅O₆]⁺: 530.2034. Found 530.2023.

Anal. Calcd for C₂₈H₂₇N₅O₆: C: 63.51 %, H: 5.14 %, N: 13.23 %. Found C: 63.48 %, H: 5.01 %, N: 13.52 %.

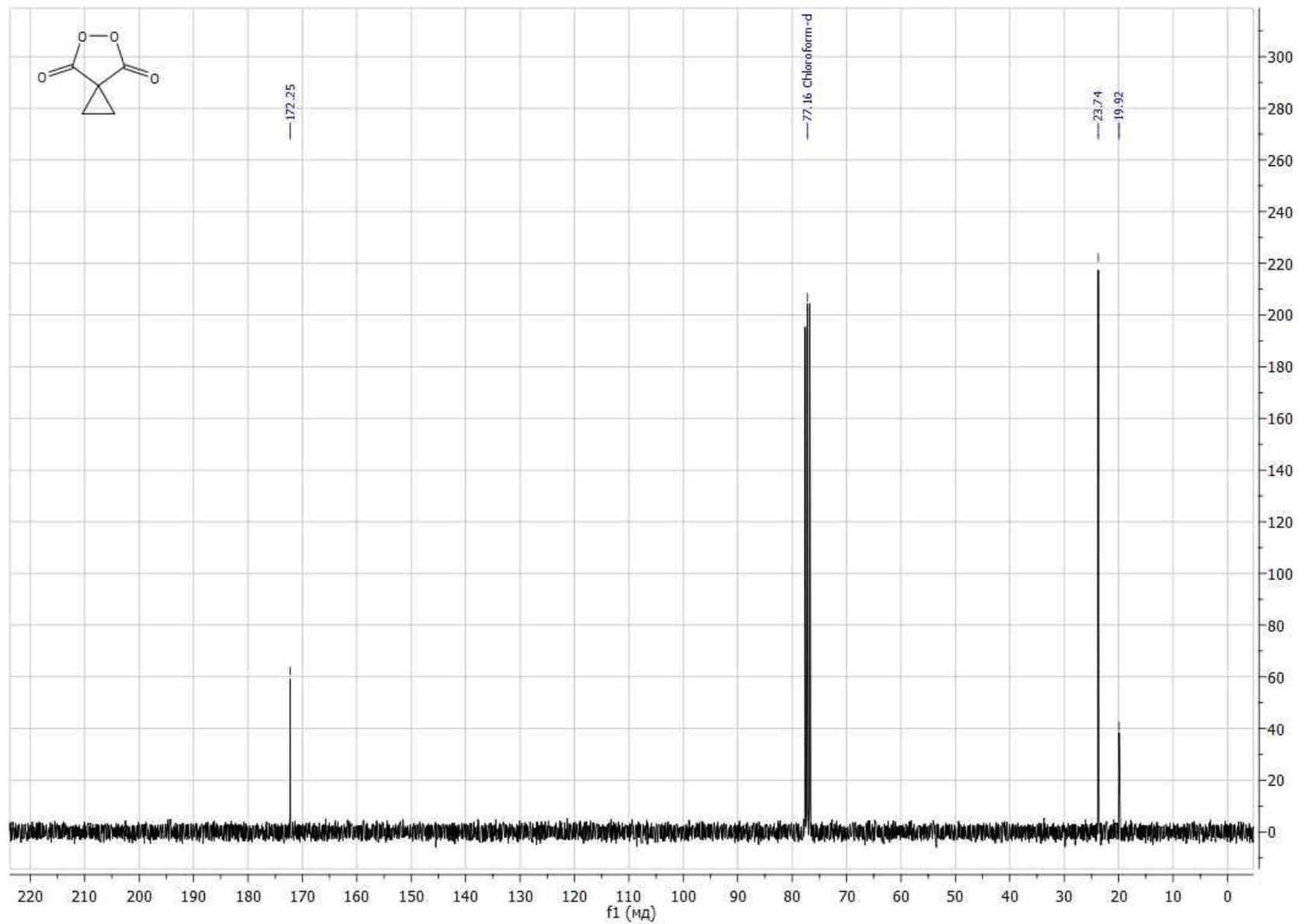
IR (CHCl₃): 3371, 2929, 1693, 1531, 1503, 1446, 1379, 1136, 1046, 766, 752 cm⁻¹.

NMR spectra of starting compounds

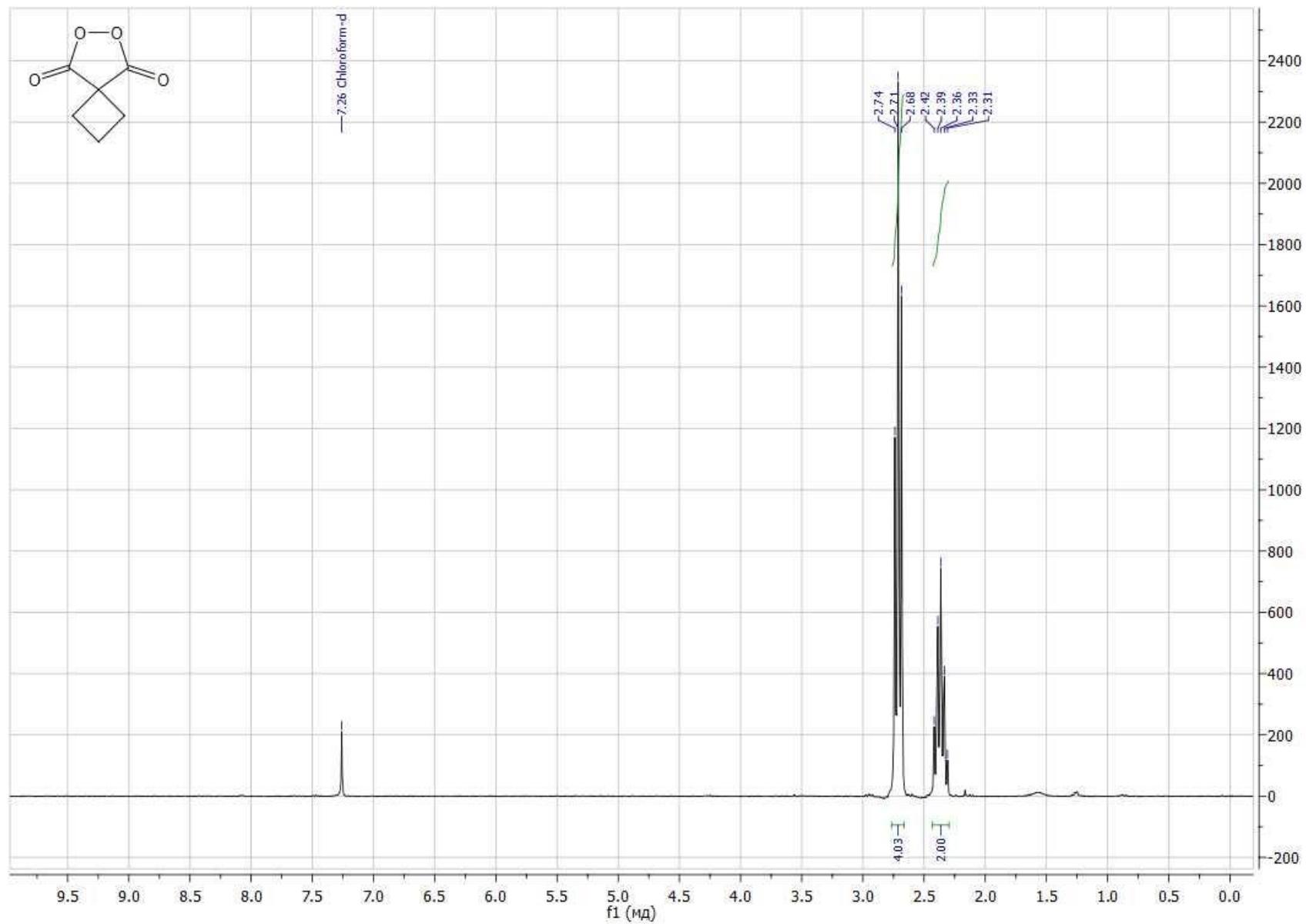
¹H NMR of spirocyclopropylmalonyl peroxide, **2a**



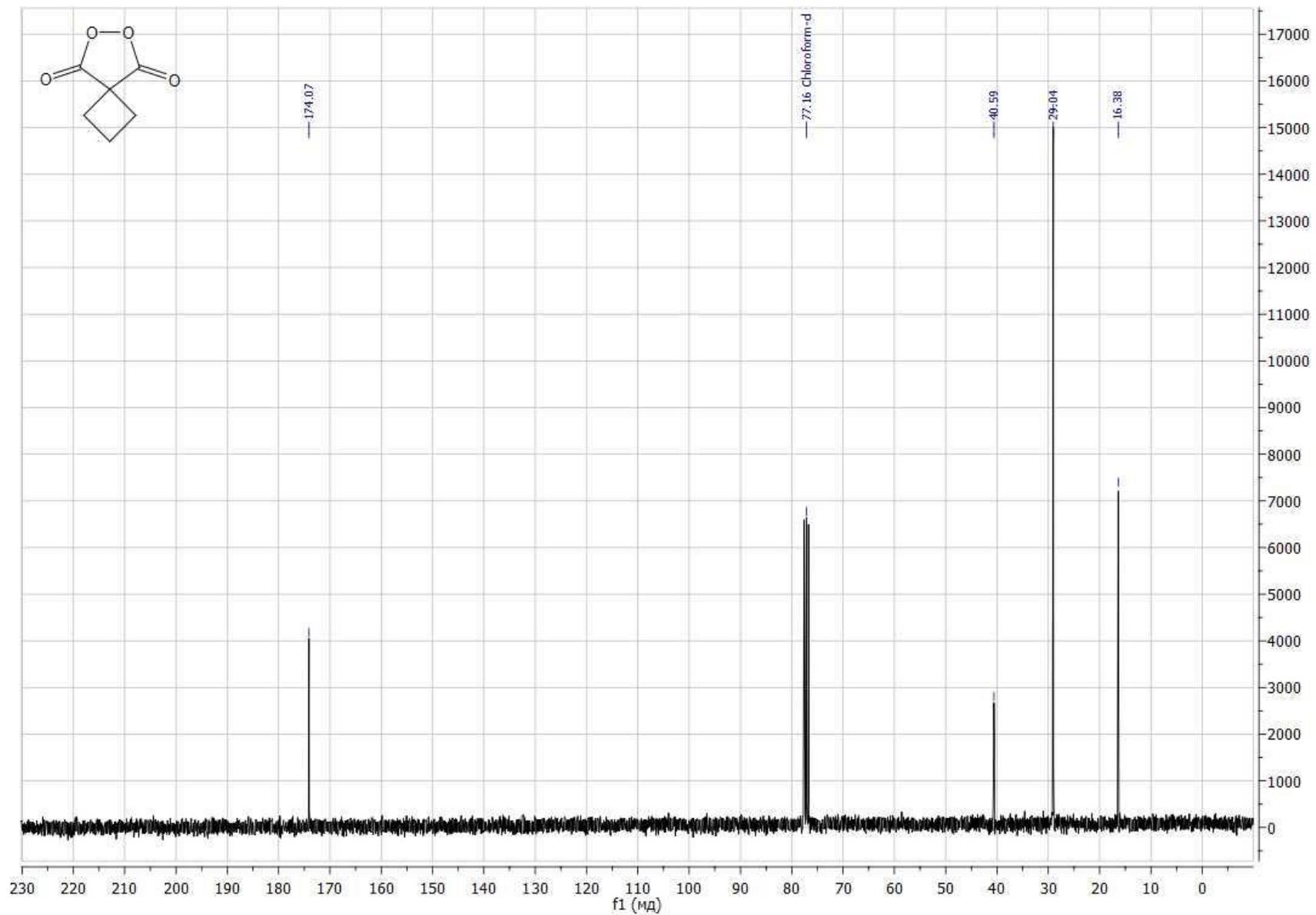
¹³C NMR of spirocyclopropylmalonyl peroxide, **2a**



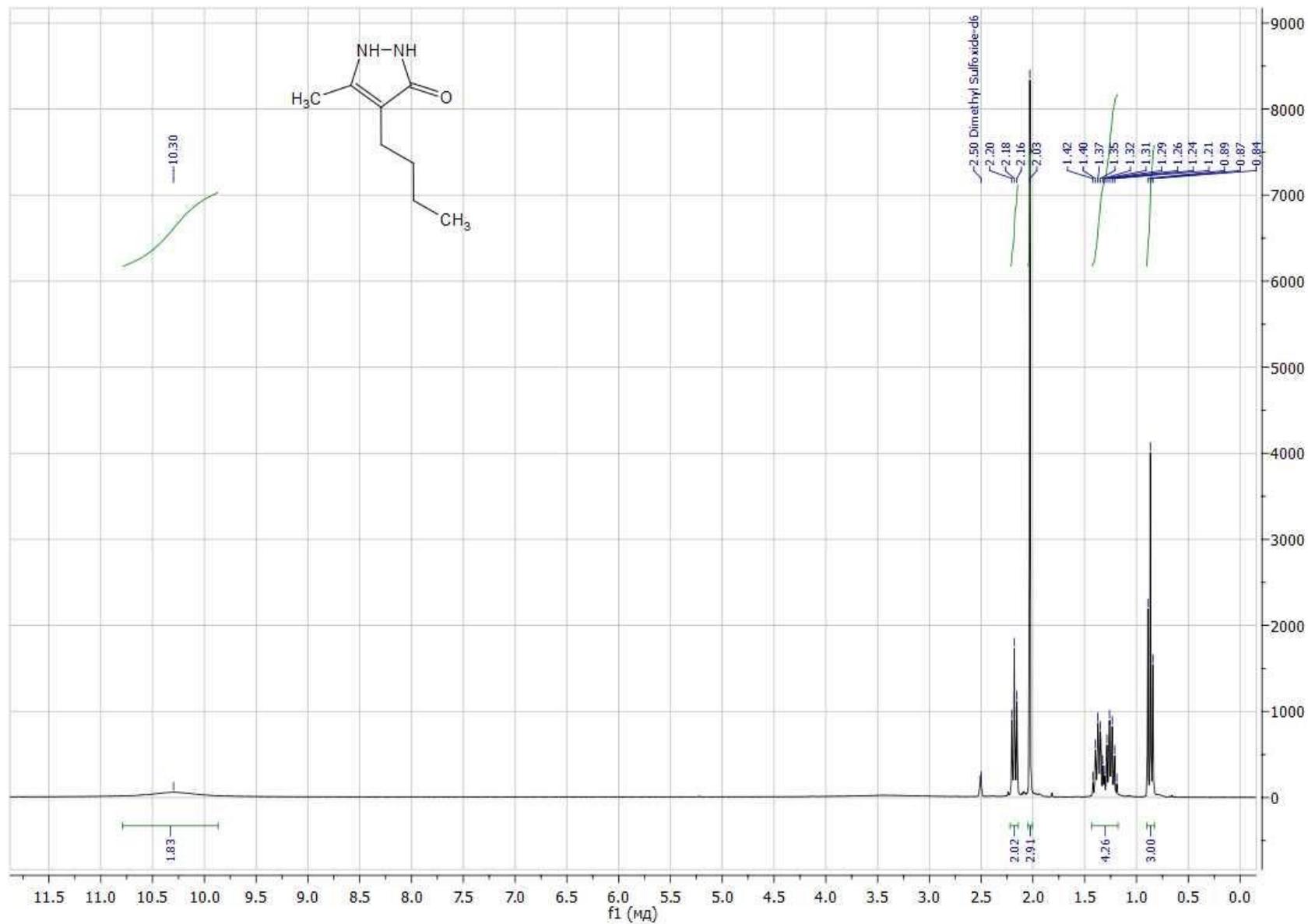
¹H NMR of spirocyclobutylmalonyl peroxide, **2b**



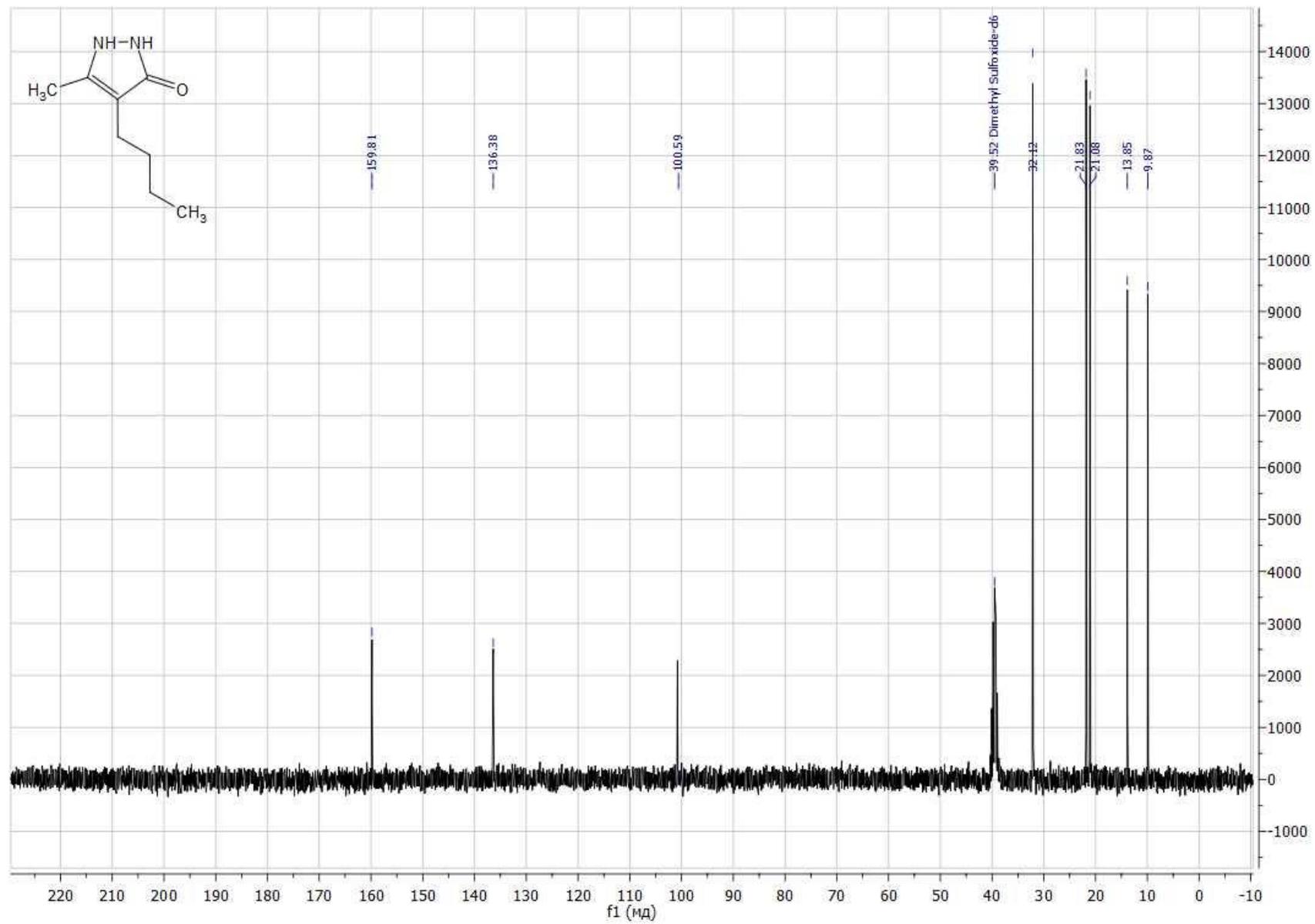
¹³C NMR of spirocyclobutylmalonyl peroxide, **2b**



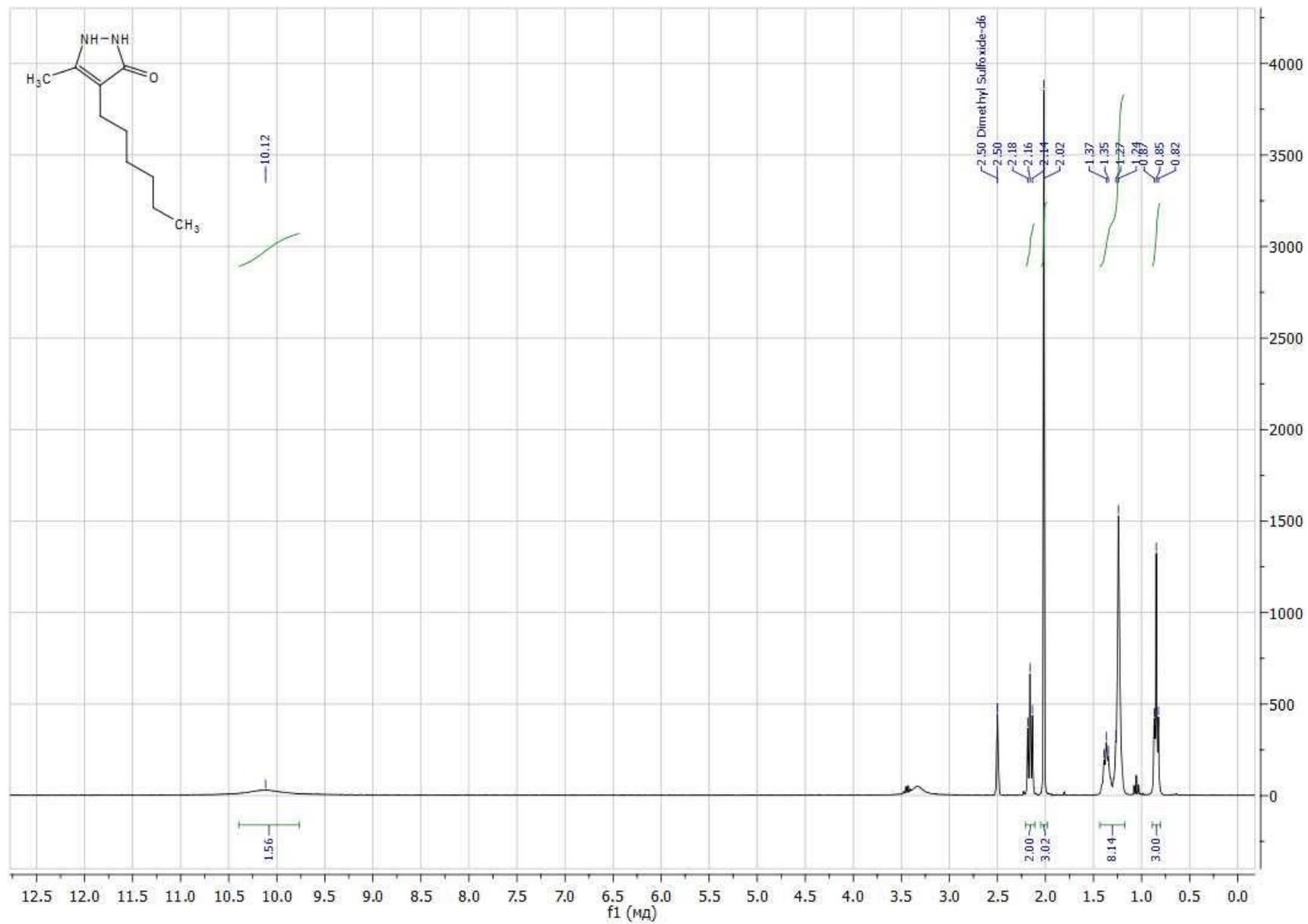
¹H NMR of 4-butyl-5-methyl-1,2-dihydro-3H-pyrazol-3-one, **1a**



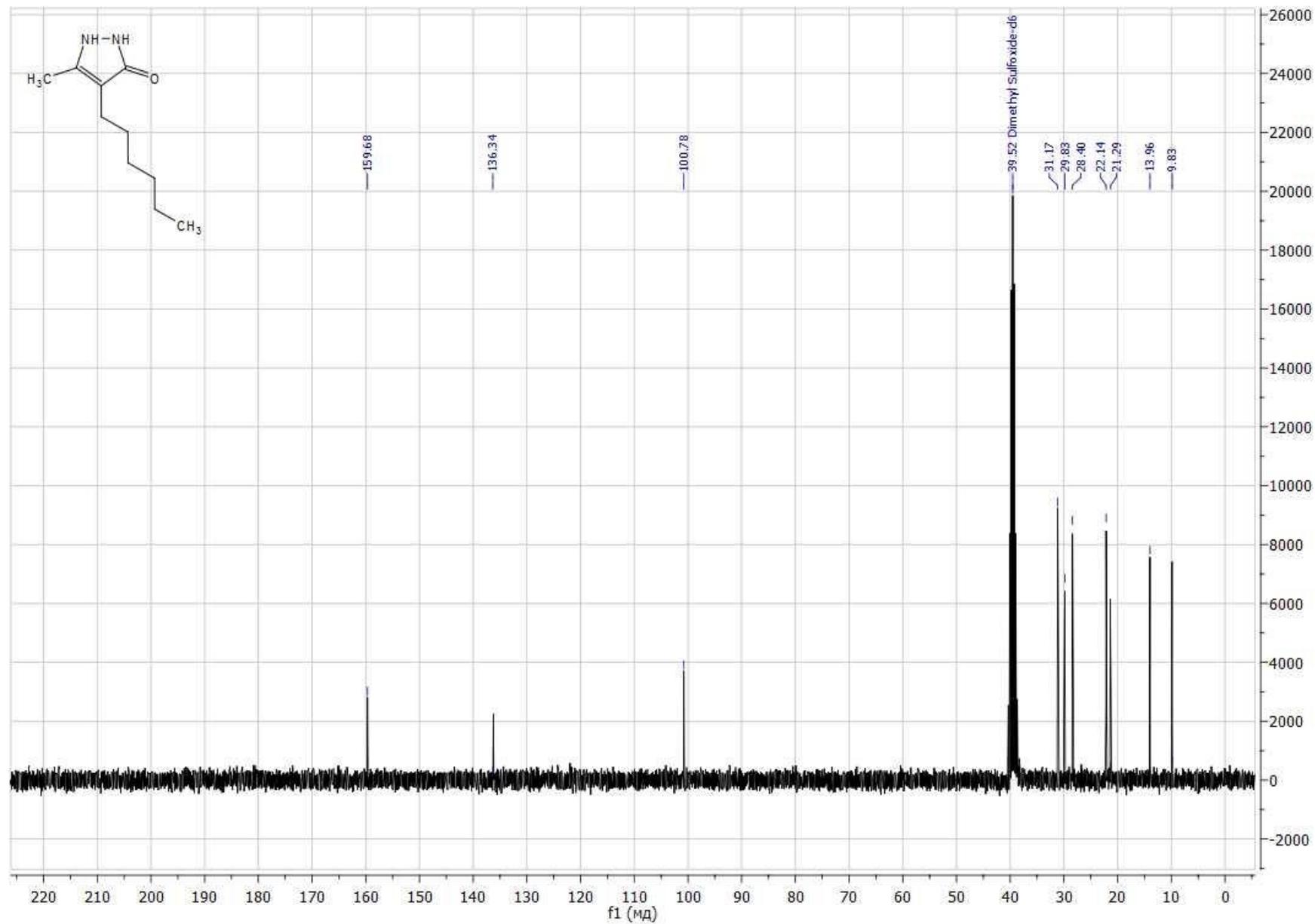
¹³C NMR of 4-butyl-5-methyl-1,2-dihydro-3H-pyrazol-3-one, **1a**



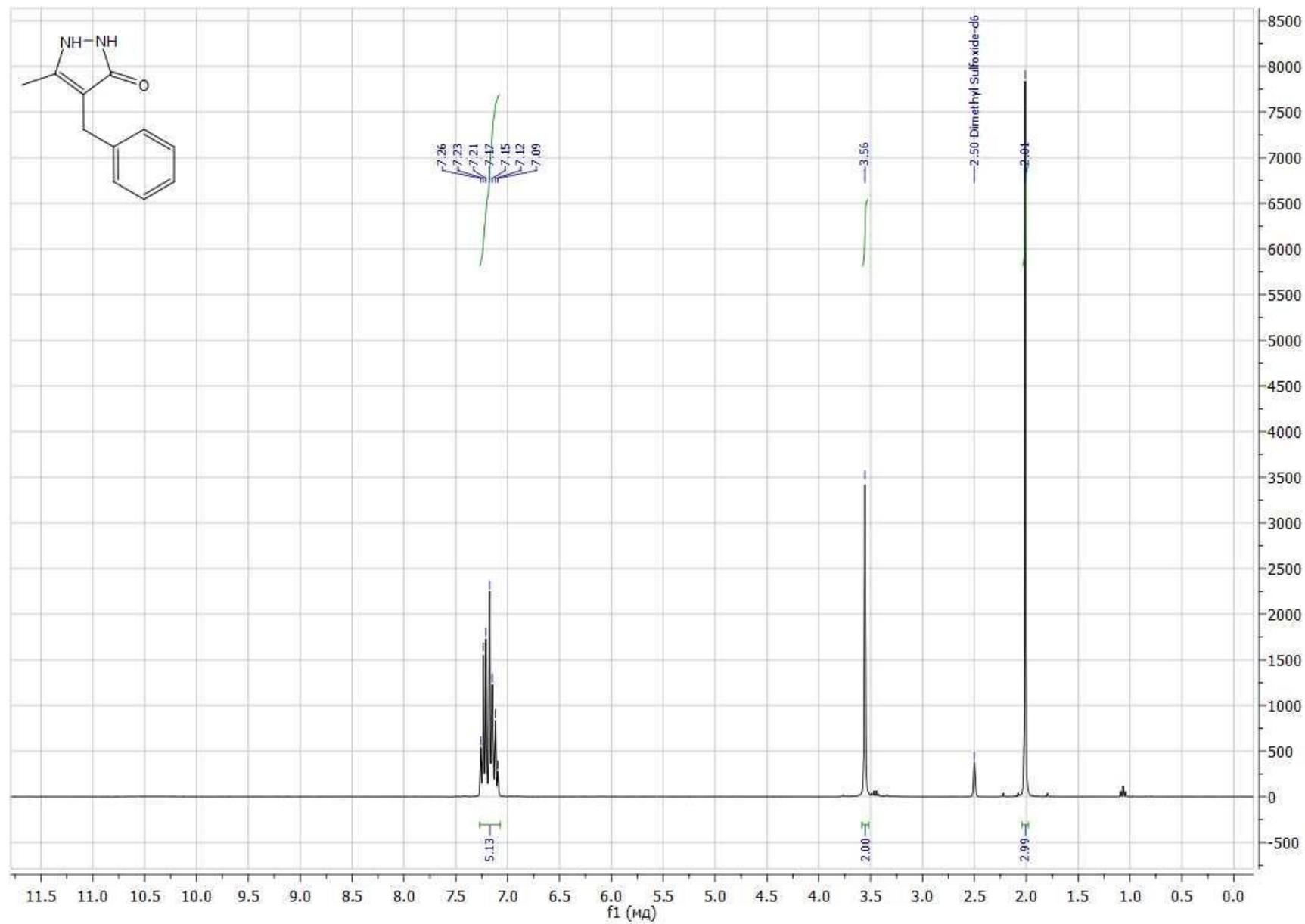
¹H NMR of 4-hexyl-5-methyl-1,2-dihydro-3H-pyrazol-3-one, **1b**



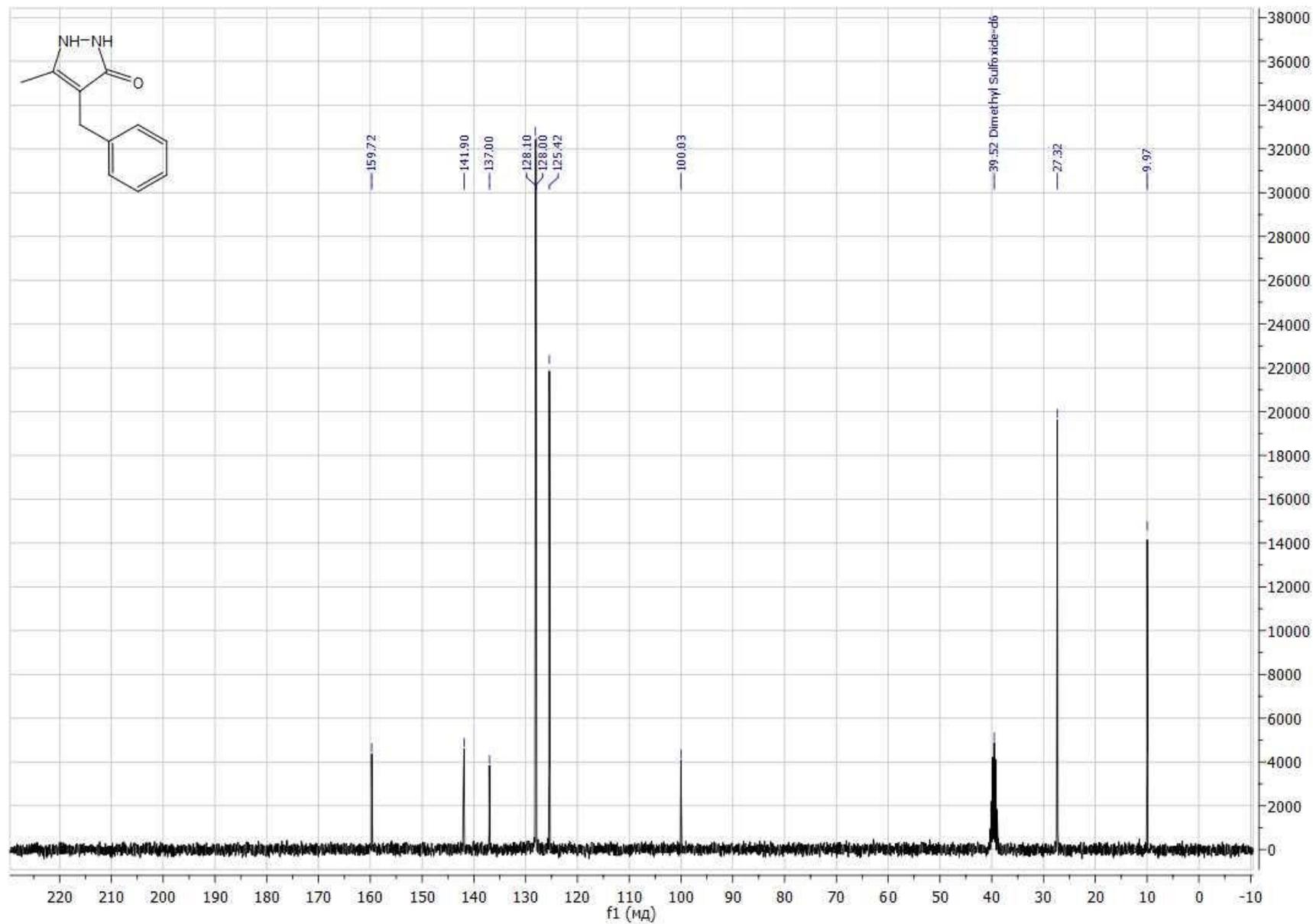
¹³C NMR of 4-hexyl-5-methyl-1,2-dihydro-3H-pyrazol-3-one, **1b**



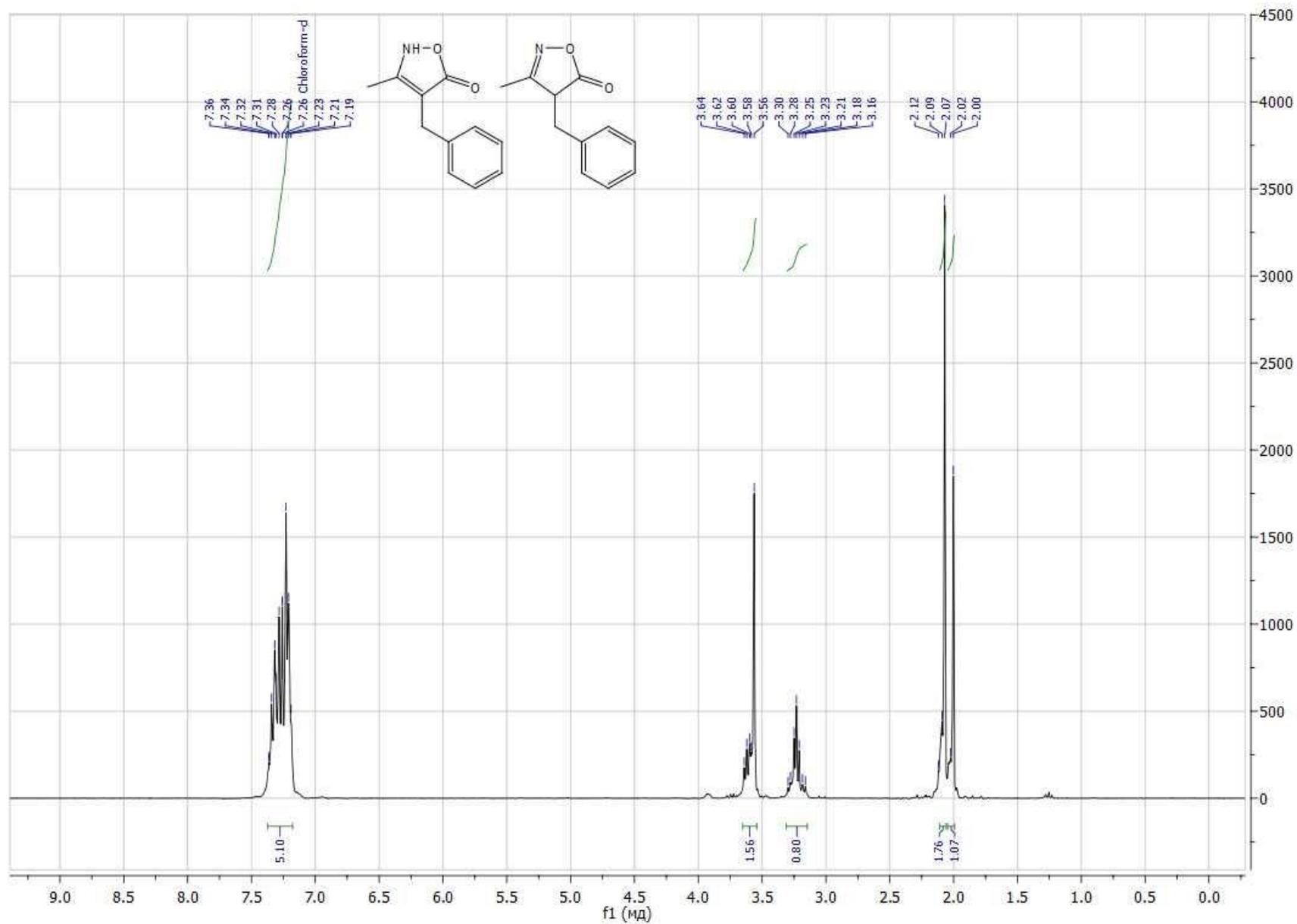
¹H NMR of 4-benzyl-5-methyl-1,2-dihydro-3H-pyrazol-3-one, **1c**



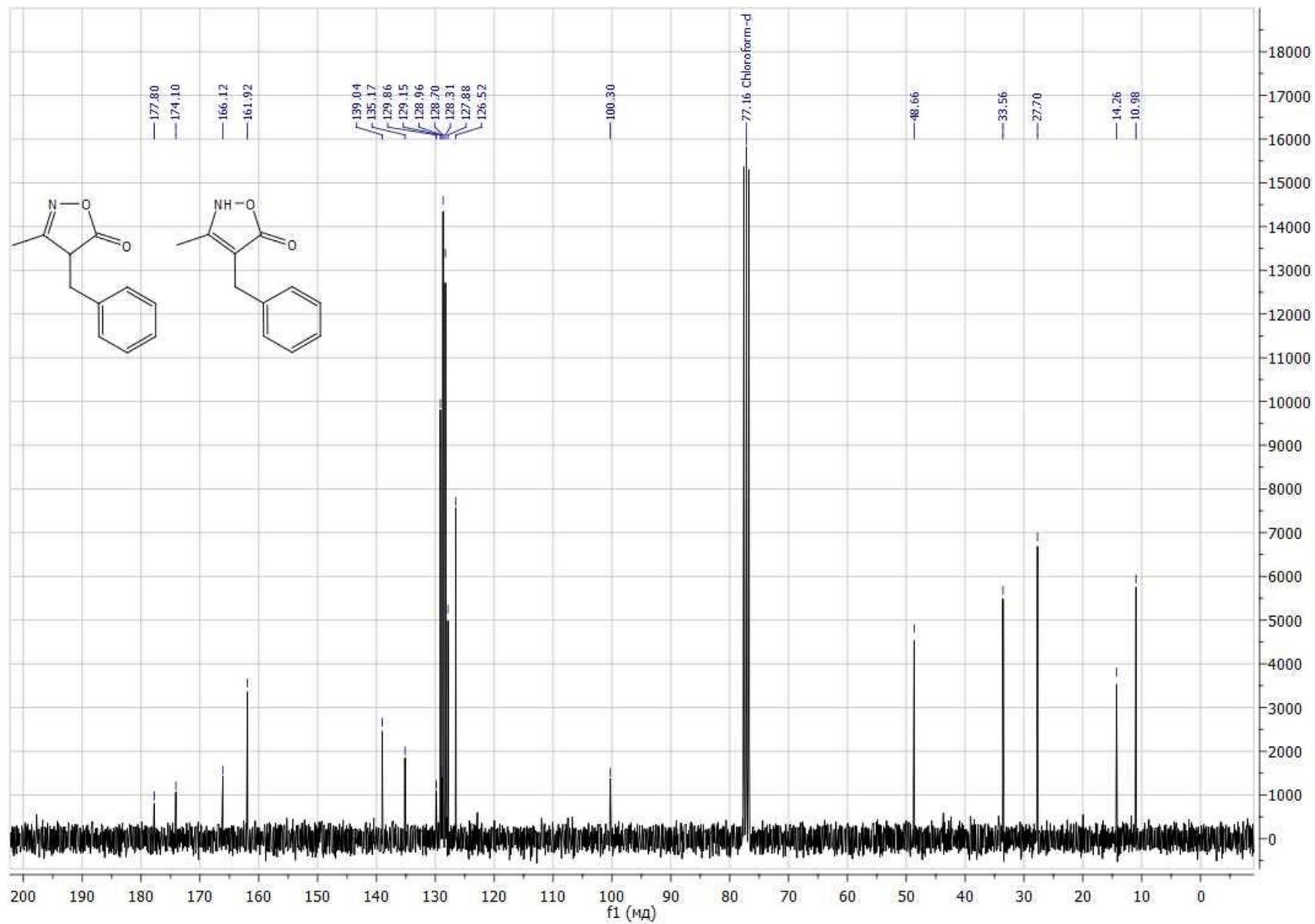
¹³C NMR of 4-benzyl-5-methyl-1,2-dihydro-3H-pyrazol-3-one, **1c**



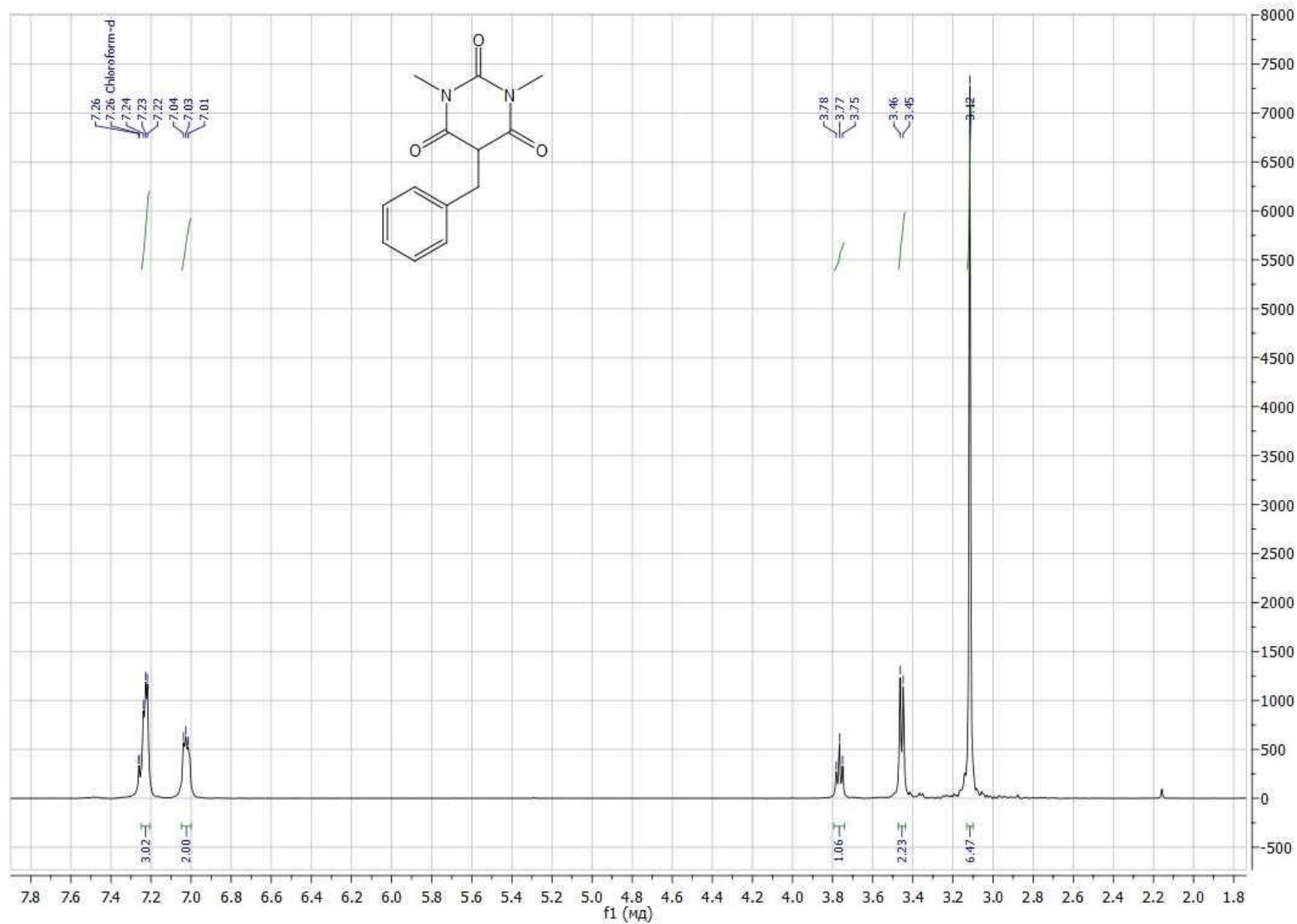
¹H NMR of 4-benzyl-3-methylisoxazol-5(2H)-one, **8**



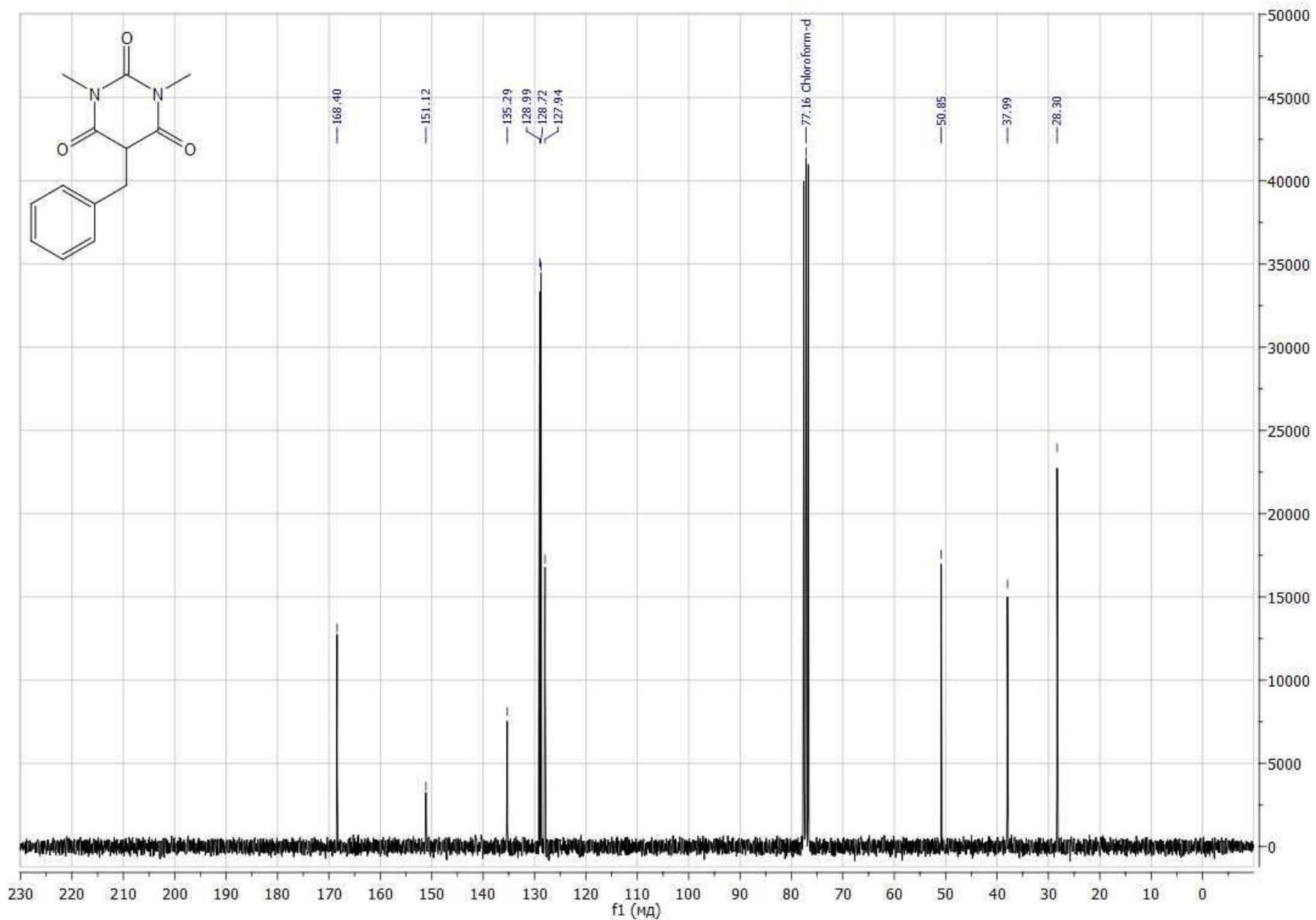
¹³C NMR of 4-benzyl-3-methylisoxazol-5(2H)-one, **8**



¹H NMR of 5-benzyl-1,3-dimethylpyrimidine-2,4,6(1*H*,3*H*,5*H*)-trione, **14**

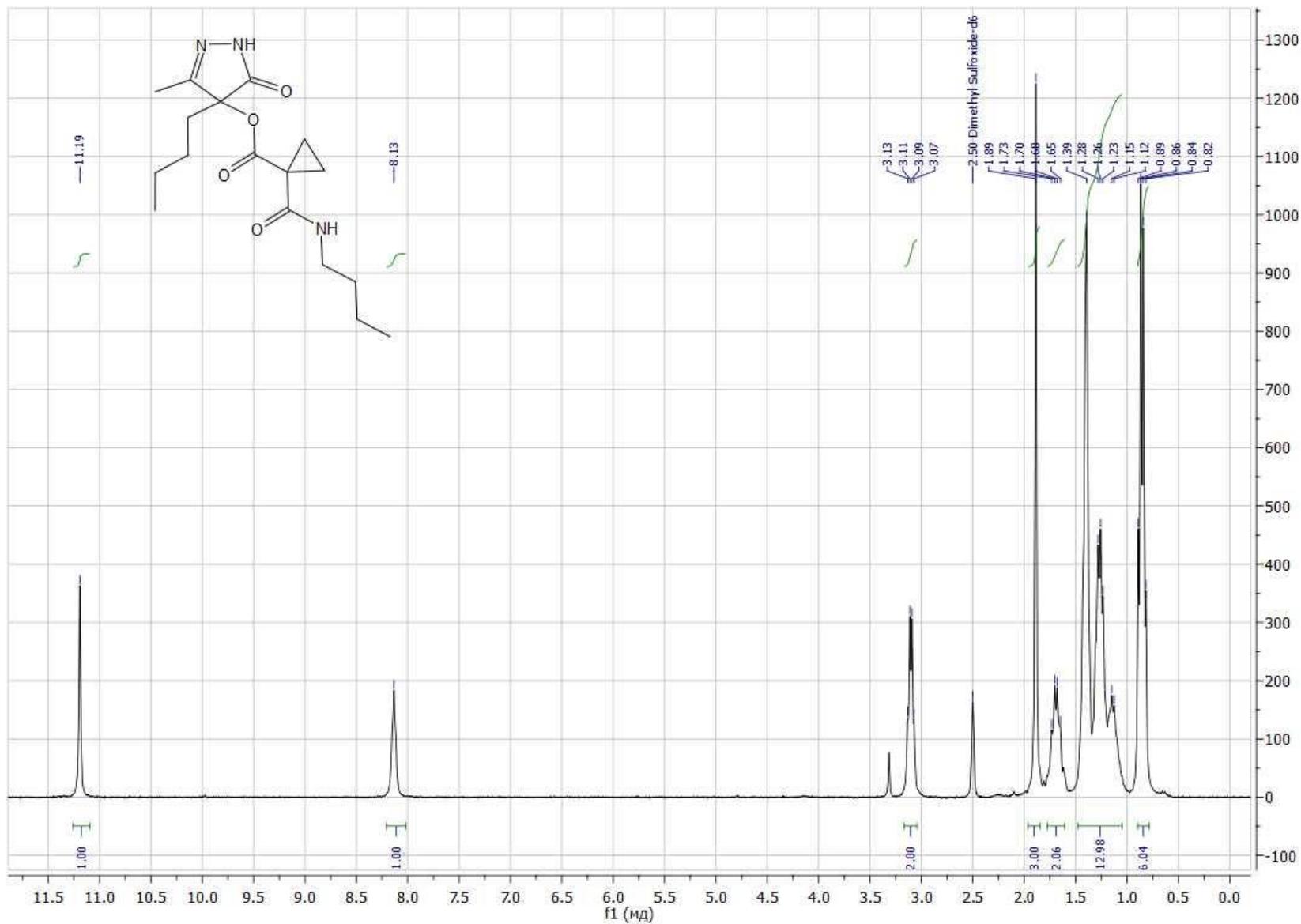


^{13}C NMR of 5-benzyl-1,3-dimethylpyrimidine-2,4,6(1*H*,3*H*,5*H*)-trione, **14**

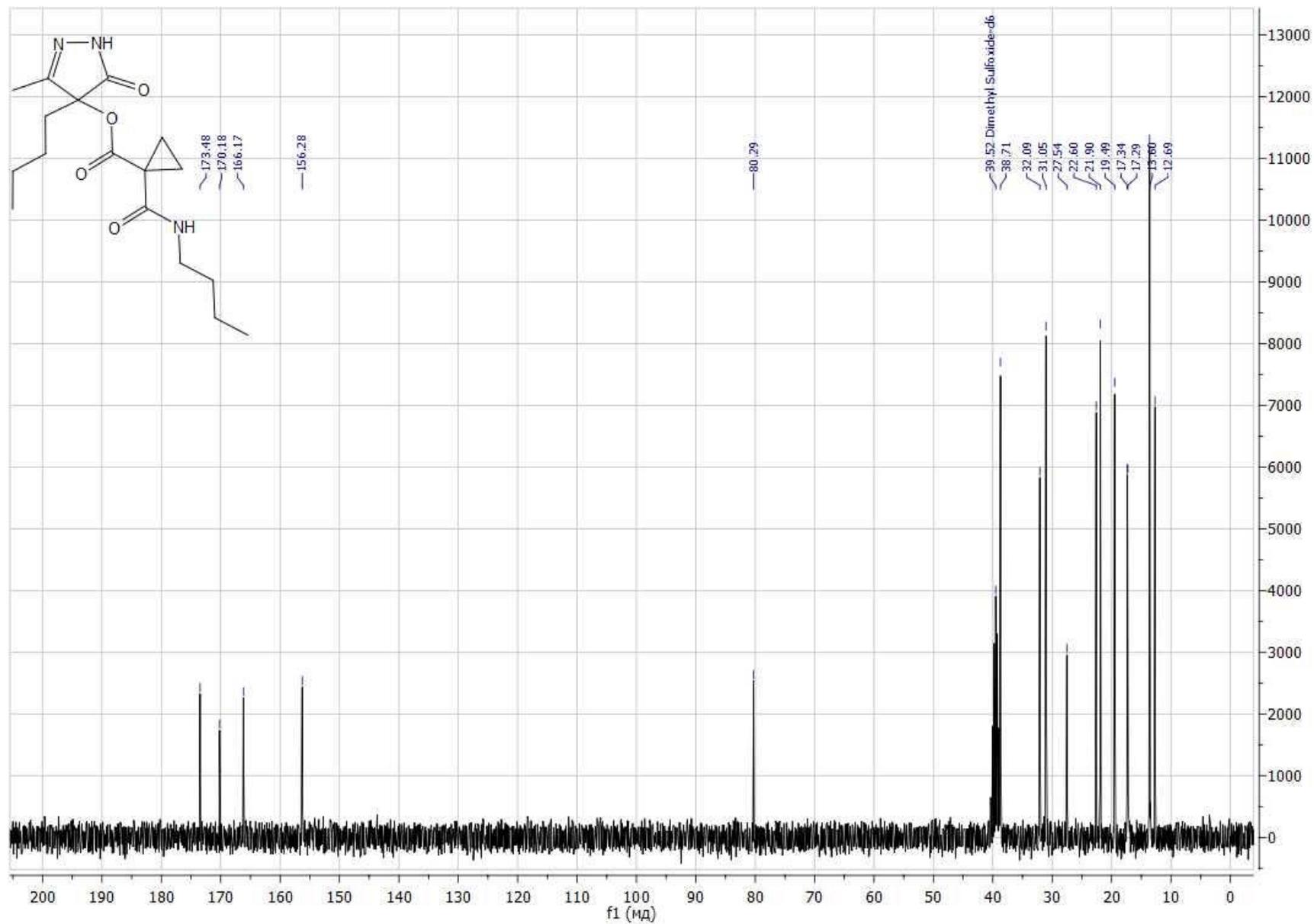


NMR spectra of hybrid compounds 6a-e, 7, 10, 13 and 16

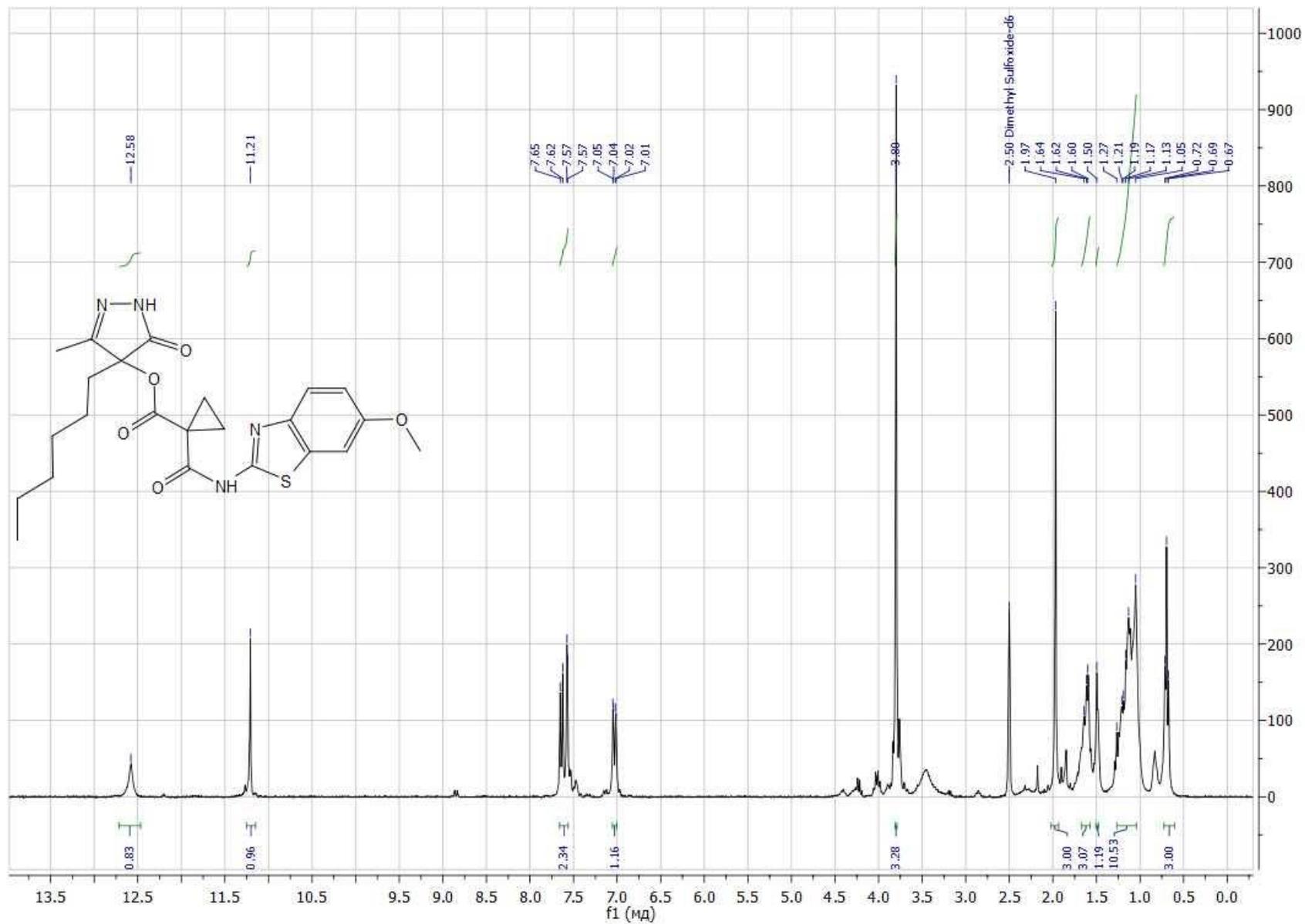
¹H NMR of 4-butyl-3-methyl-5-oxo-4,5-dihydro-1H-pyrazol-4-yl 1-(N-butylcarbamoyl)cyclopropane-1-carboxylate, **6a**



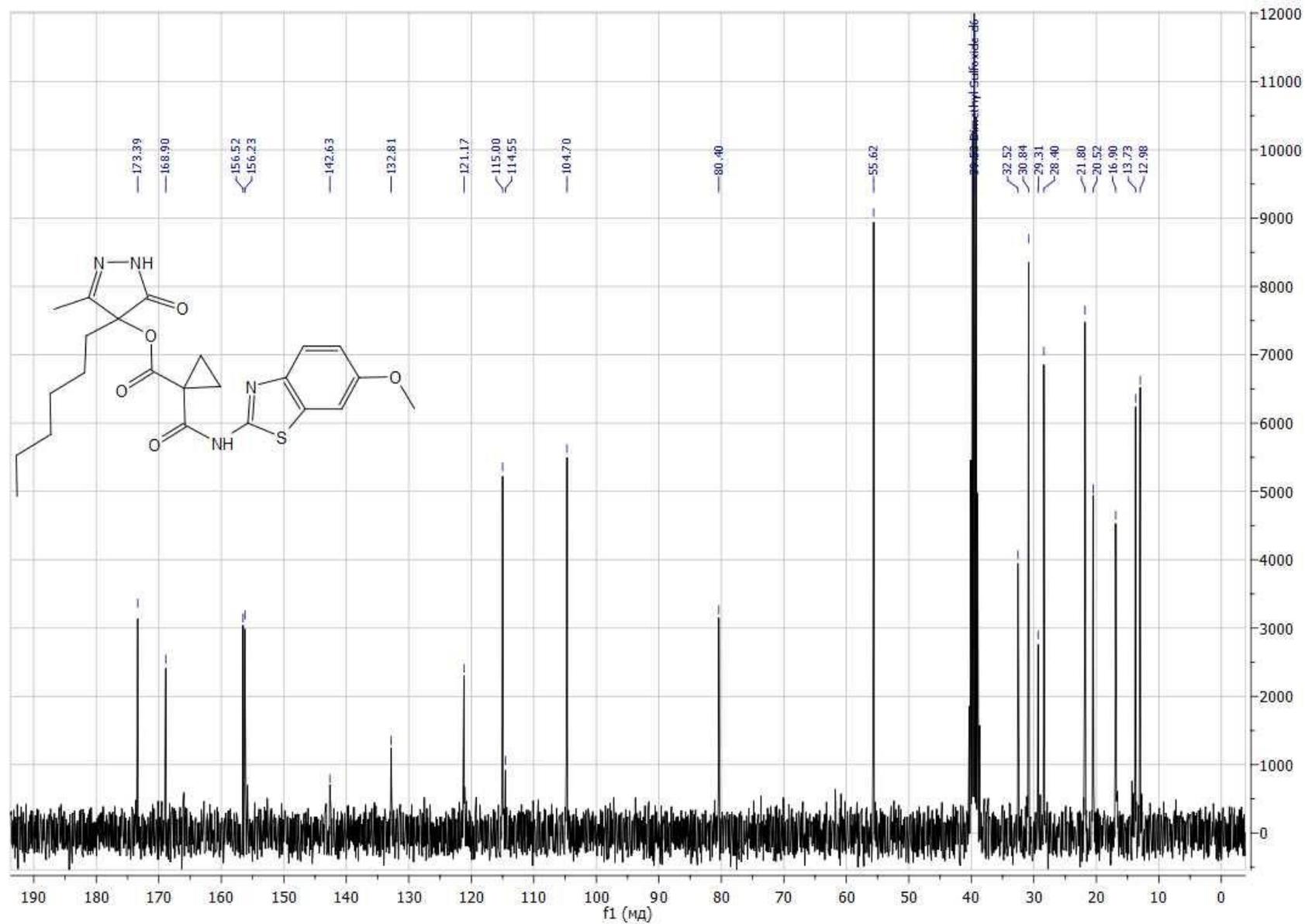
¹³C NMR of 4-butyl-3-methyl-5-oxo-4,5-dihydro-1H-pyrazol-4-yl 1-(N-butylcarbamoyl)cyclopropane-1-carboxylate, **6a**



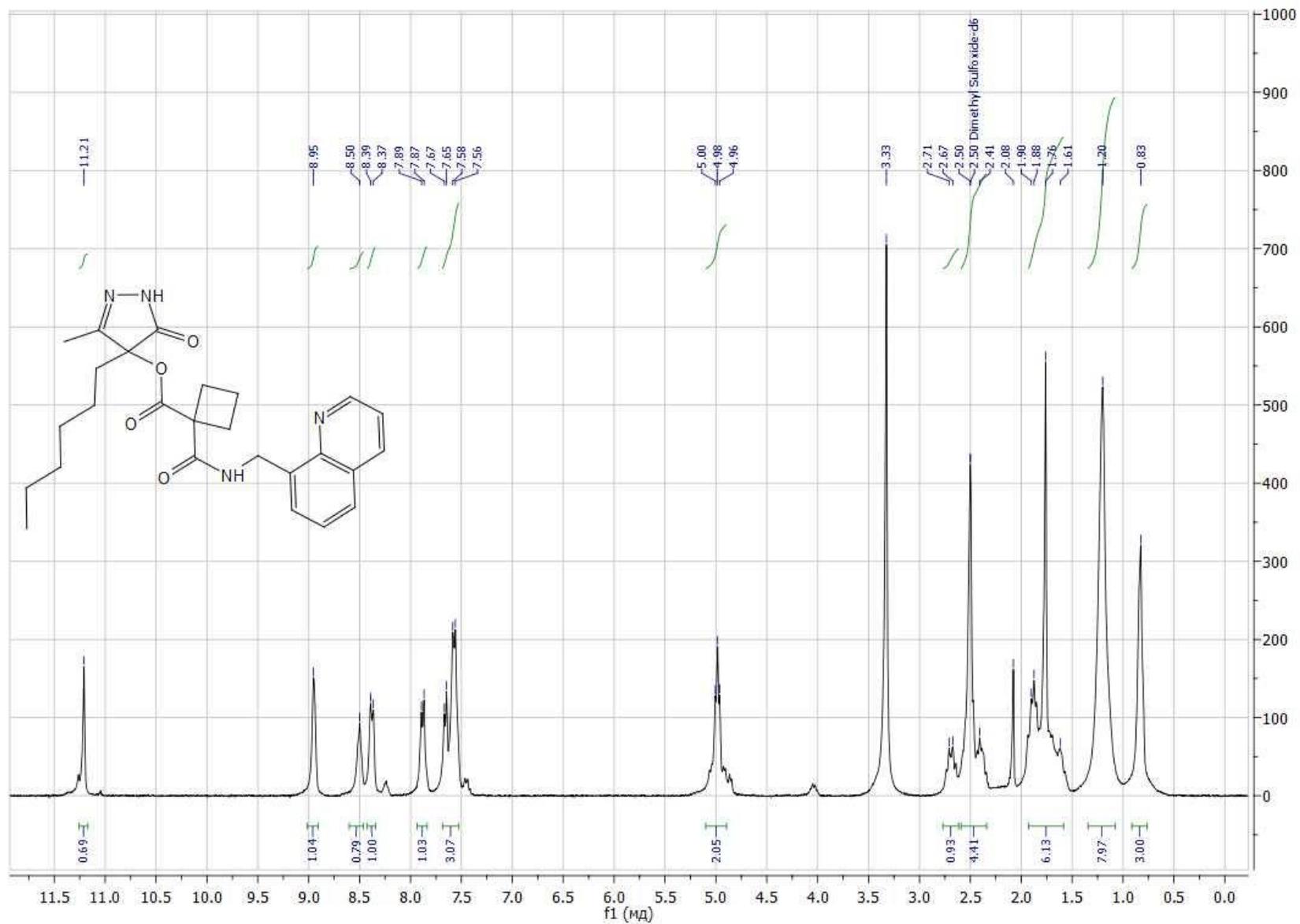
¹H NMR of 4-hexyl-3-methyl-5-oxo-4,5-dihydro-1H-pyrazol-4-yl 1-[N-(6-methoxybenzo[d]thiazol-2-yl)carbamoyl]cyclopropane-1-carboxylate, **6b**



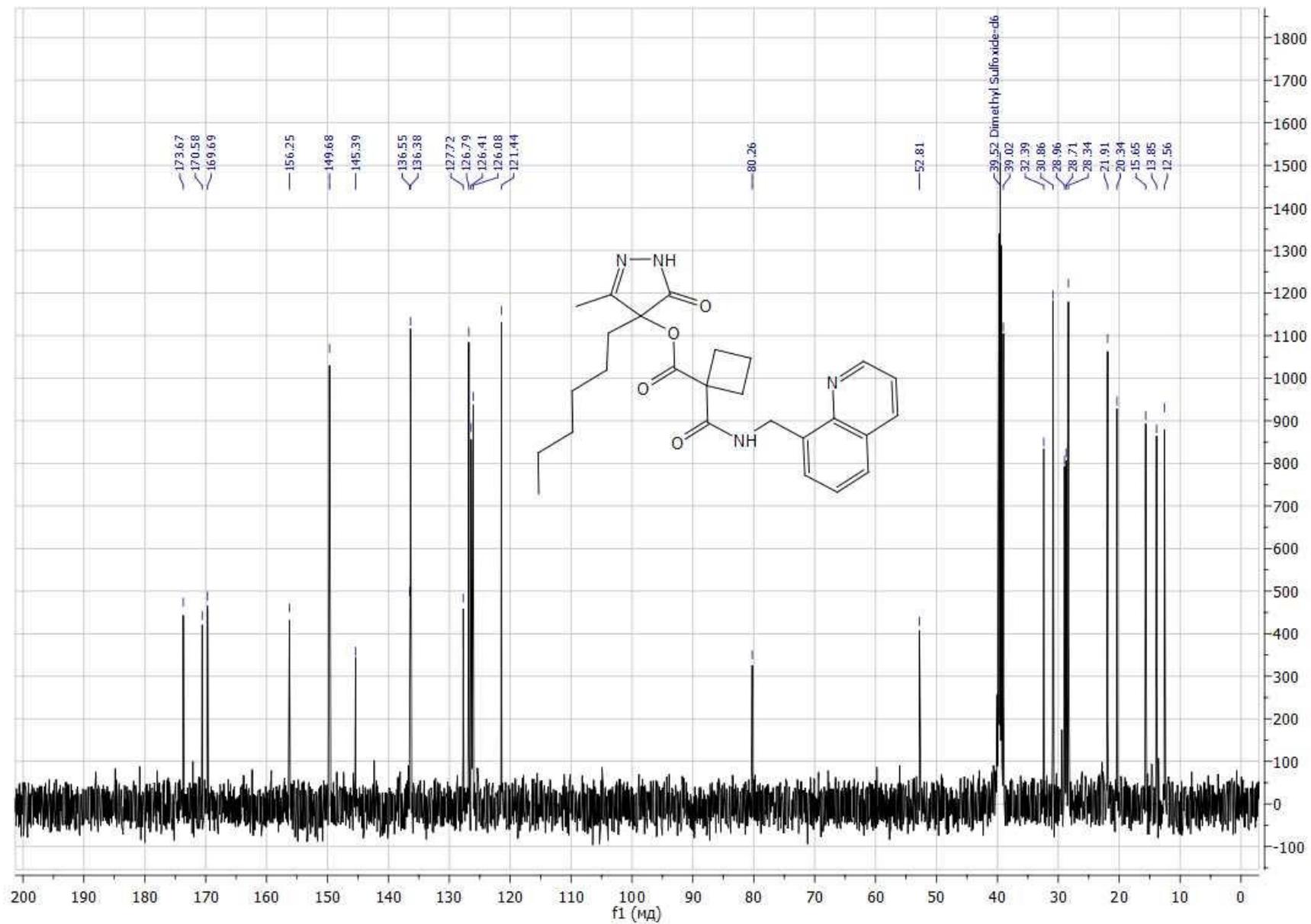
¹³C NMR of 4-hexyl-3-methyl-5-oxo-4,5-dihydro-1H-pyrazol-4-yl 1-[N-(6-methoxybenzo[d]thiazol-2-yl)carbamoyl]cyclopropane-1-carboxylate, **6b**



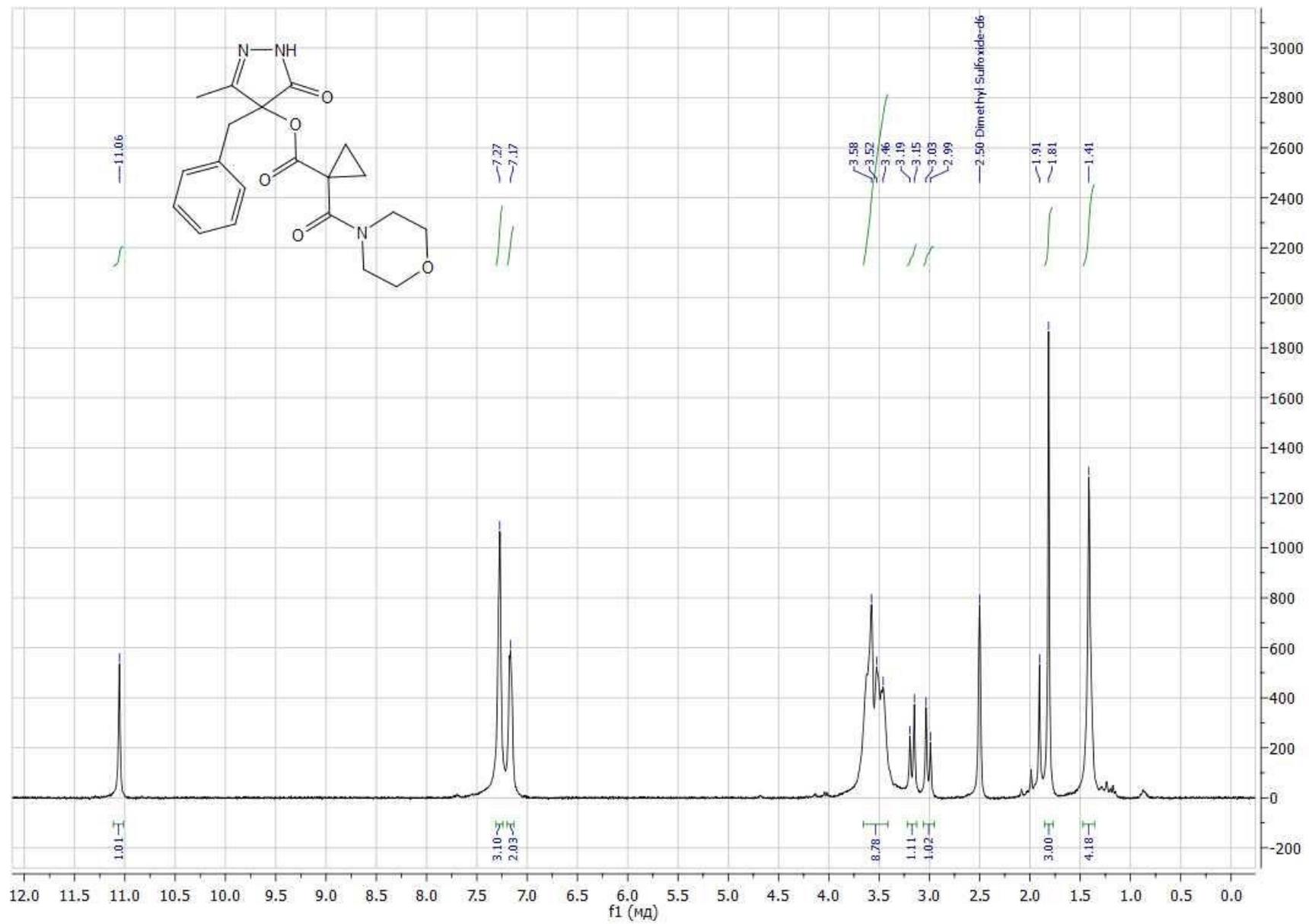
¹H NMR of 4-hexyl-3-methyl-5-oxo-4,5-dihydro-1H-pyrazol-4-yl 1-[N-(quinolin-8-ylmethyl)carbamoyl]cyclobutane-1-carboxylate, **6c**



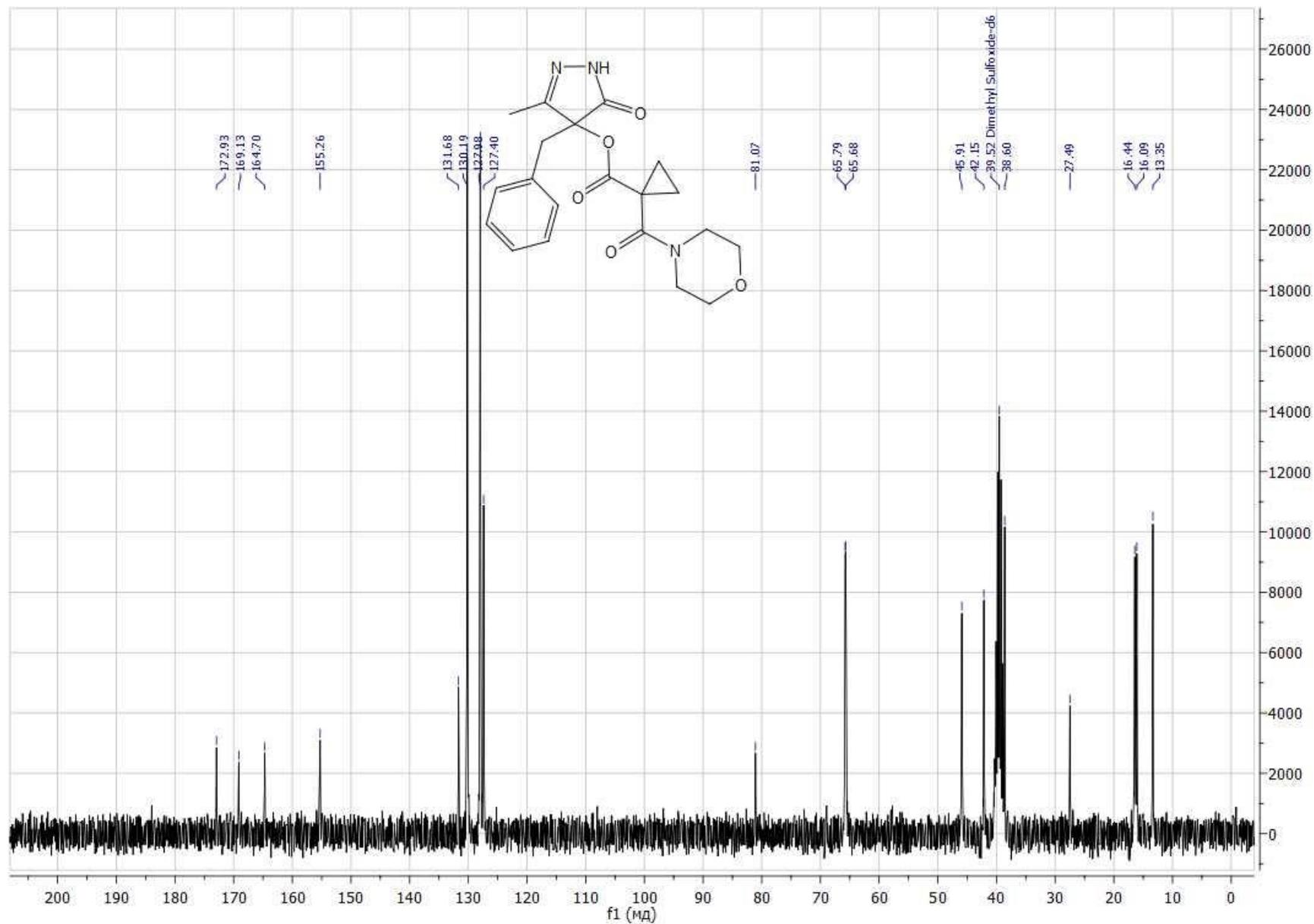
^{13}C NMR of 4-hexyl-3-methyl-5-oxo-4,5-dihydro-1H-pyrazol-4-yl 1-[*N*-(quinolin-8-ylmethyl)carbamoyl]cyclobutane-1-carboxylate, **6c**



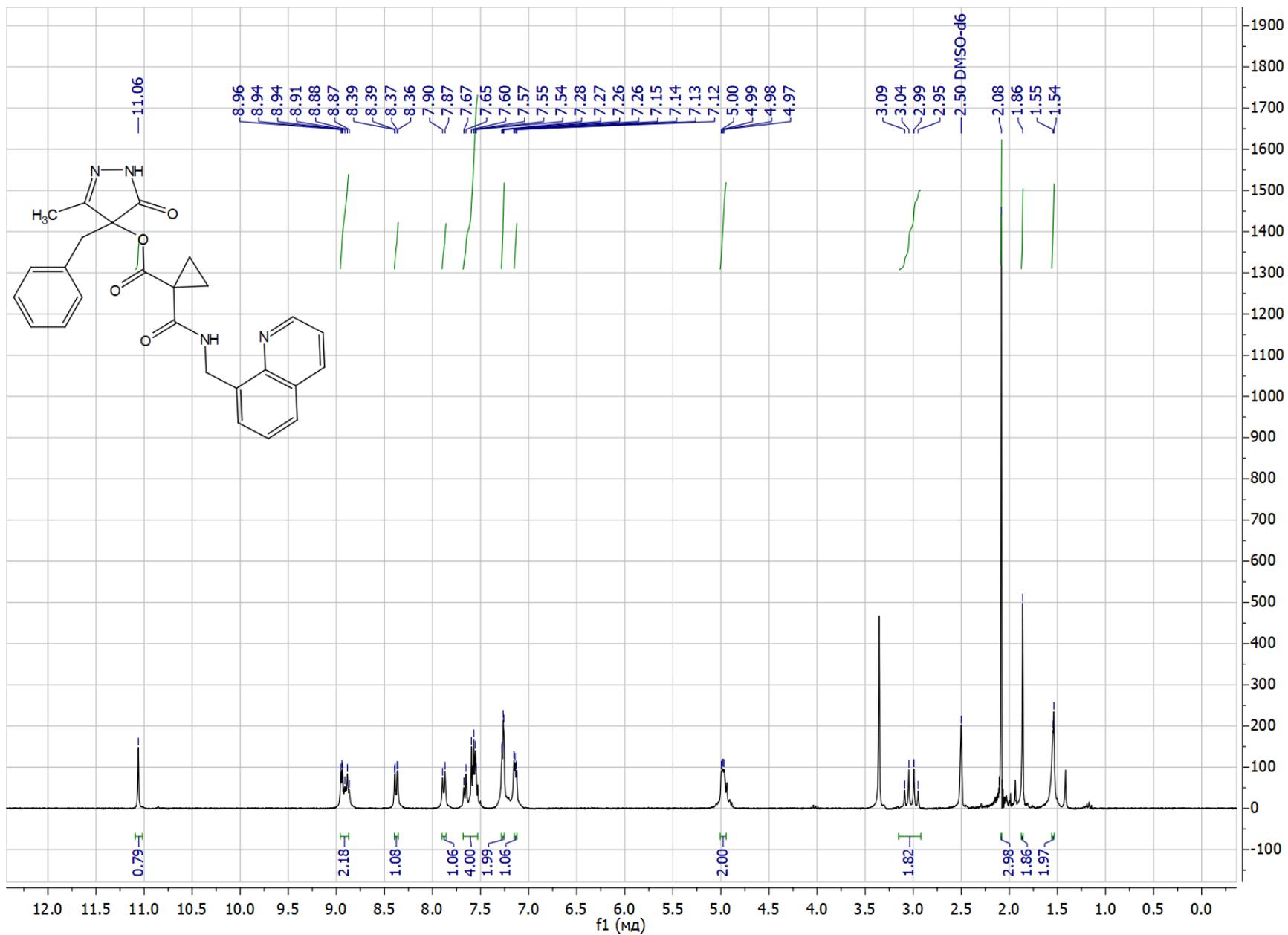
^1H NMR of 4-benzyl-3-methyl-5-oxo-4,5-dihydro-1*H*-pyrazol-4-yl 1-(morpholin-4-ylcarbonyl)cyclopropane-1-carboxylate, **6d**



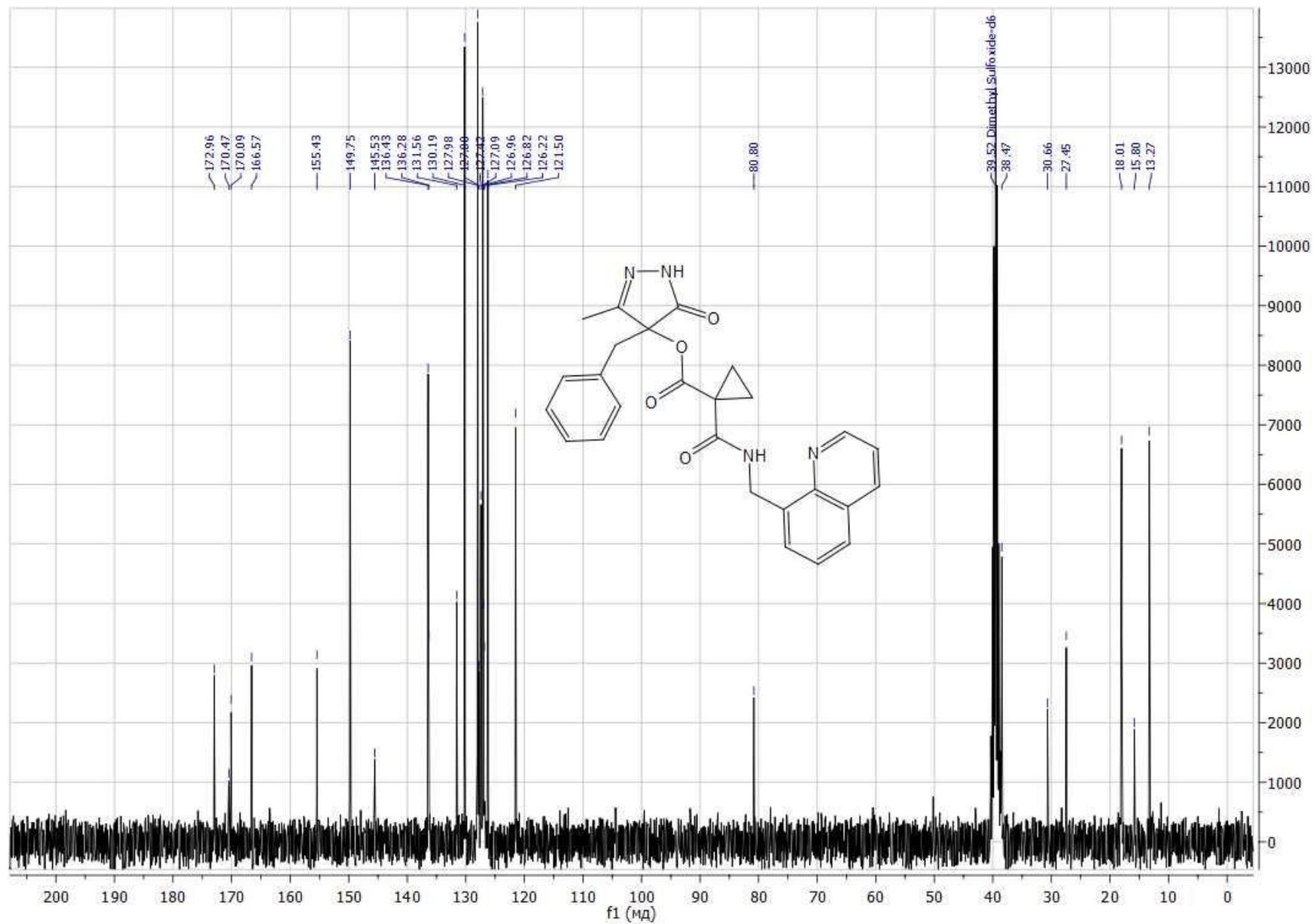
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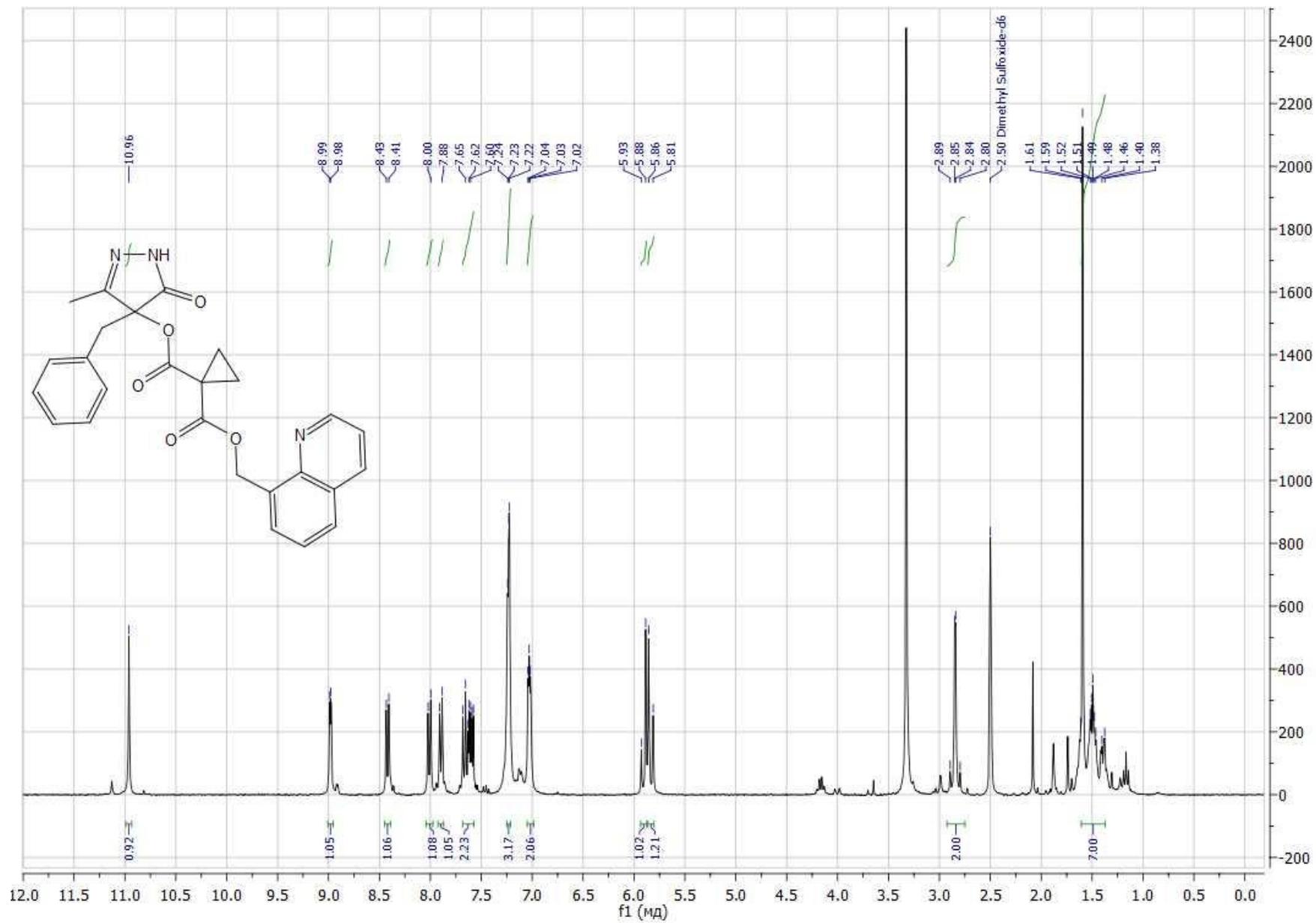
¹H NMR of 4-benzyl-3-methyl-5-oxo-4,5-dihydro-1H-pyrazol-4-yl 1-[N-(quinolin-8-ylmethyl)carbamoyl]cyclopropane-1-carboxylate, **6e**



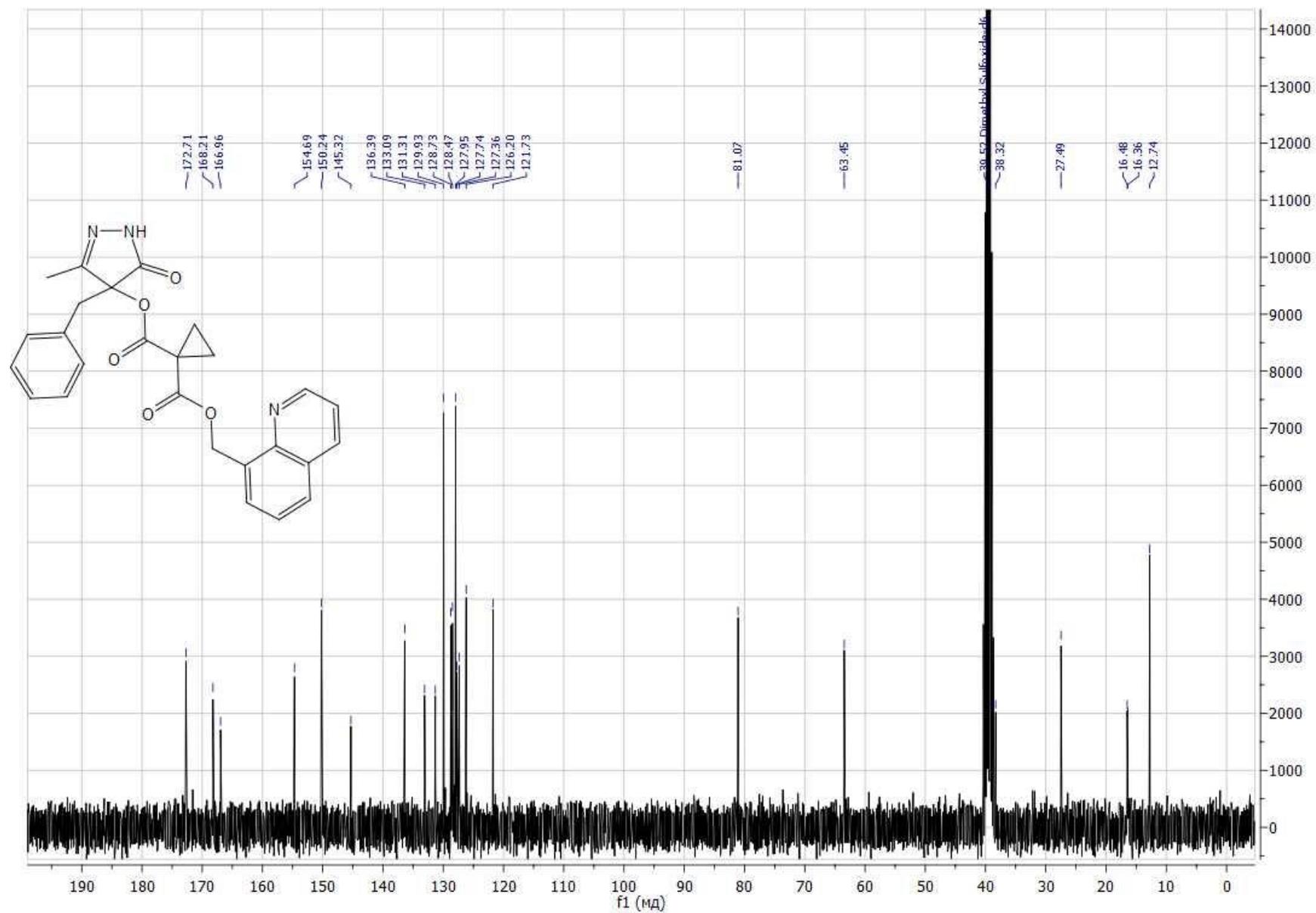
¹³C NMR of 4-benzyl-3-methyl-5-oxo-4,5-dihydro-1H-pyrazol-4-yl 1-[N-(quinolin-8-ylmethyl)carbamoyl]cyclopropane-1-carboxylate, **6e**



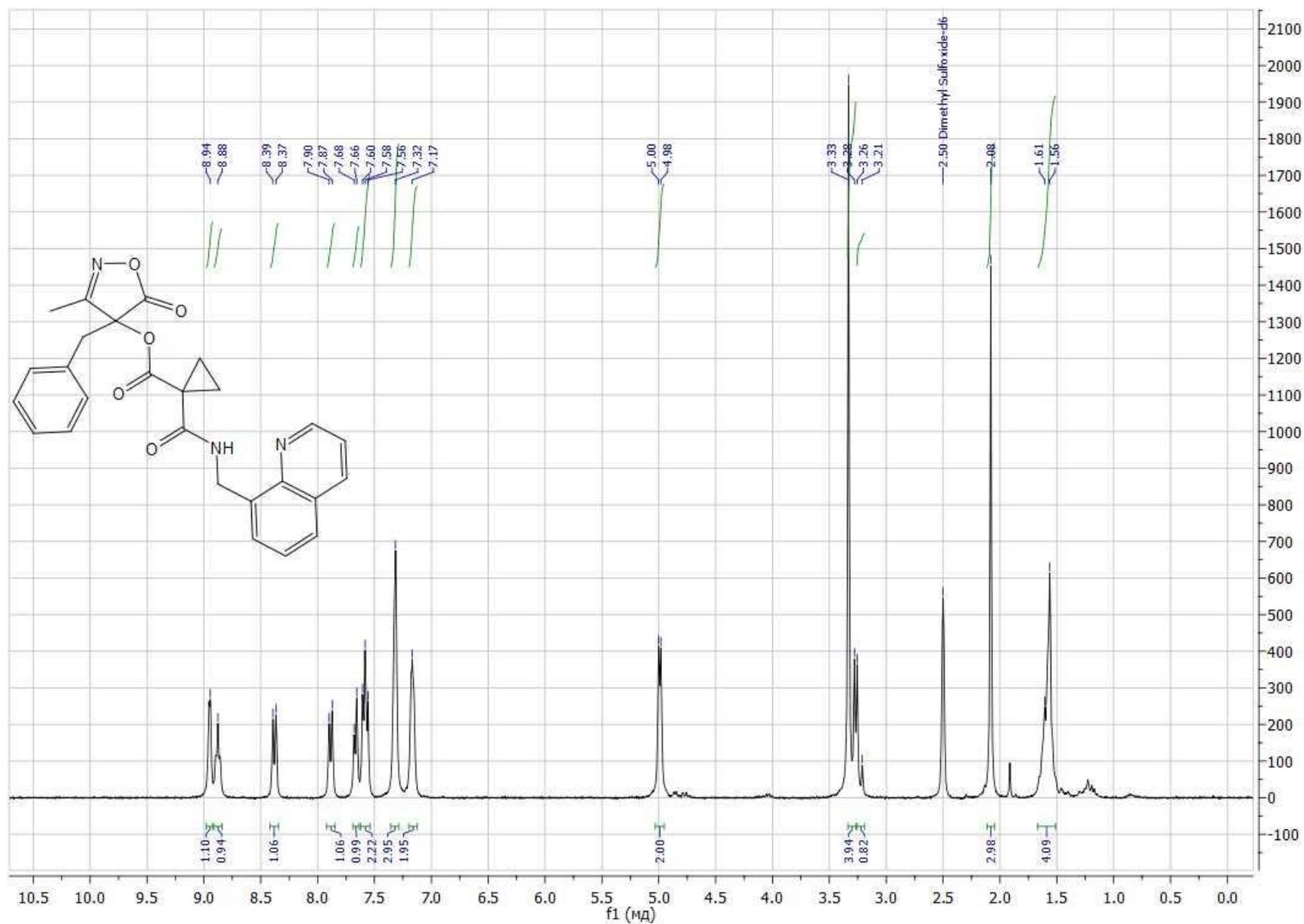
¹H NMR of 4-benzyl-3-methyl-5-oxo-4,5-dihydro-1H-pyrazol-4-yl (quinolin-8-yl)methyl cyclopropane-1,1-dicarboxylate, **7**



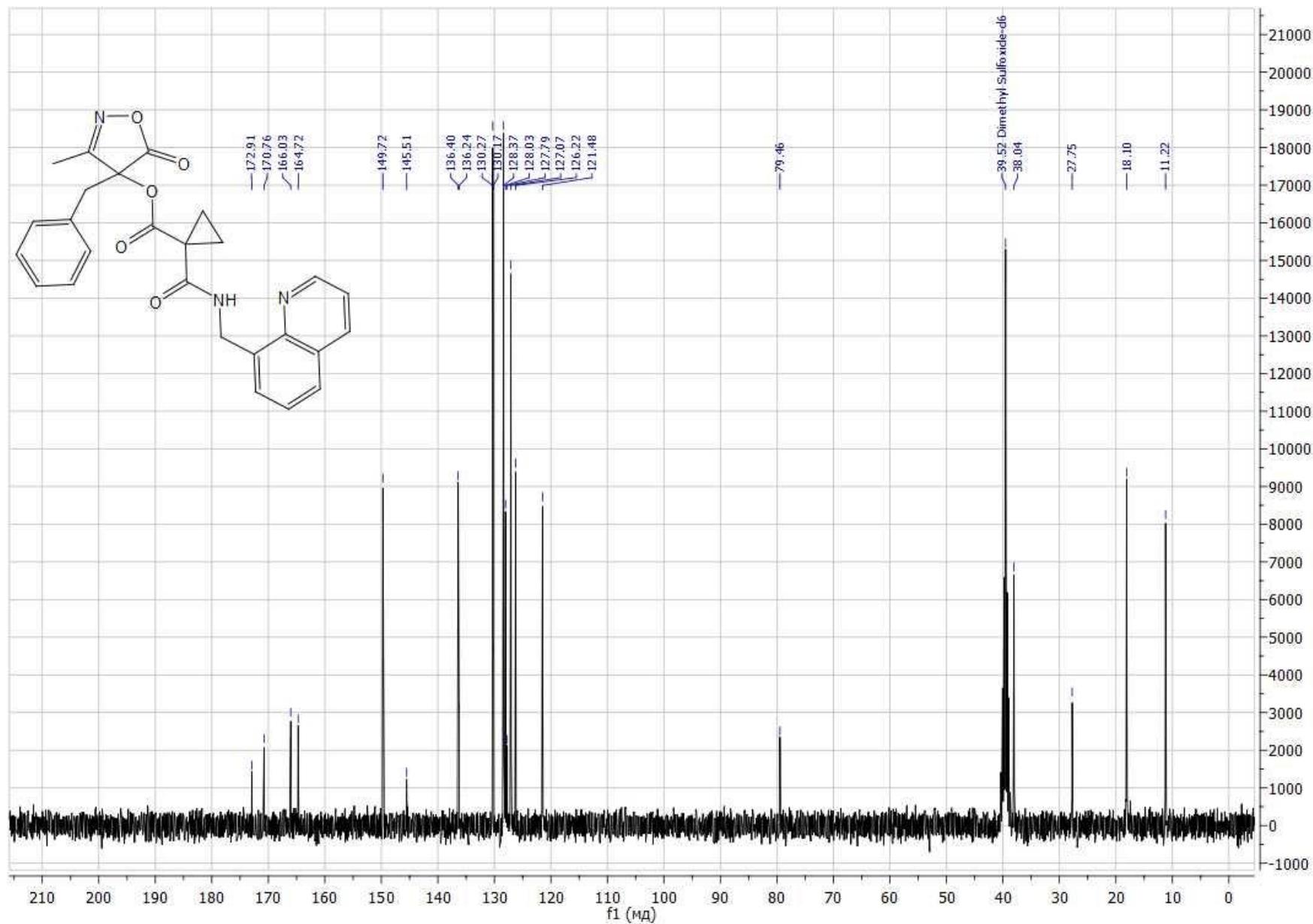
¹³C NMR of 4-benzyl-3-methyl-5-oxo-4,5-dihydro-1H-pyrazol-4-yl (quinolin-8-yl)methyl cyclopropane-1,1-dicarboxylate, **7**



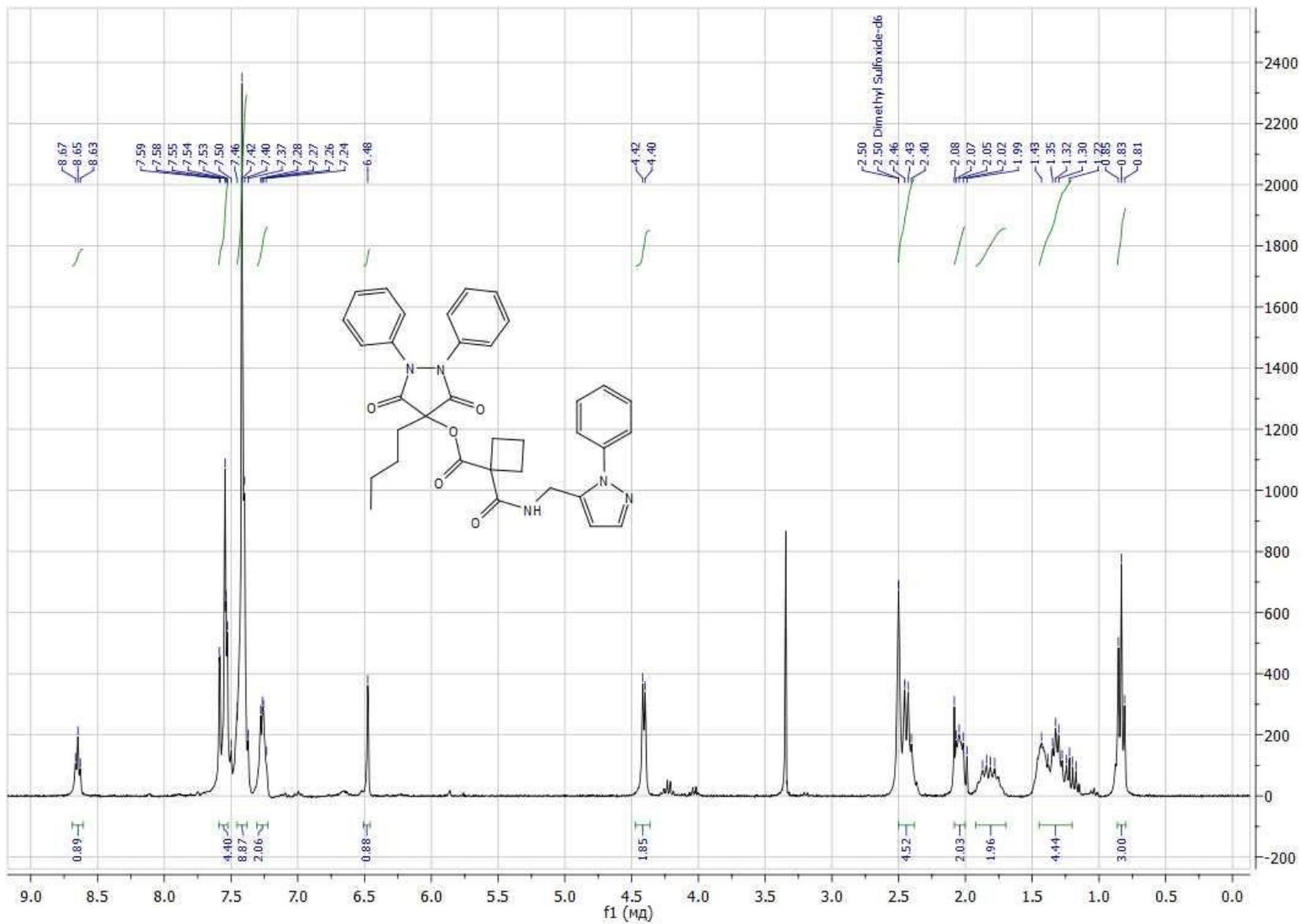
¹H NMR of 4-benzyl-3-methyl-5-oxo-4,5-dihydroisoxazol-4-yl 1-[N-(quinolin-8-ylmethyl)carbamoyl]cyclopropane-1-carboxylate, **10**



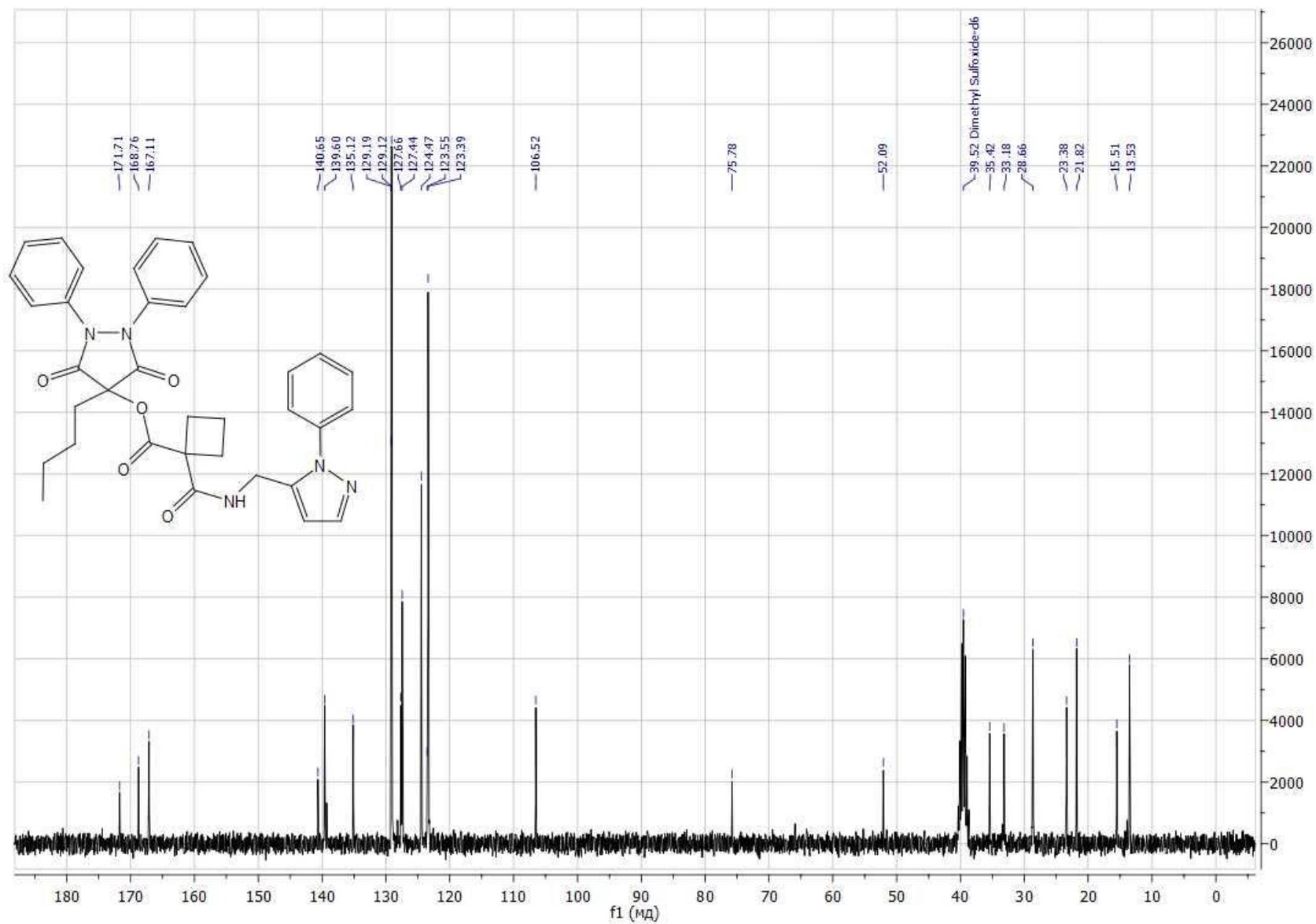
¹³C NMR of 4-benzyl-3-methyl-5-oxo-4,5-dihydroisoxazol-4-yl 1-[N-(quinolin-8-ylmethyl)carbamoyl]cyclopropane-1-carboxylate, **10**



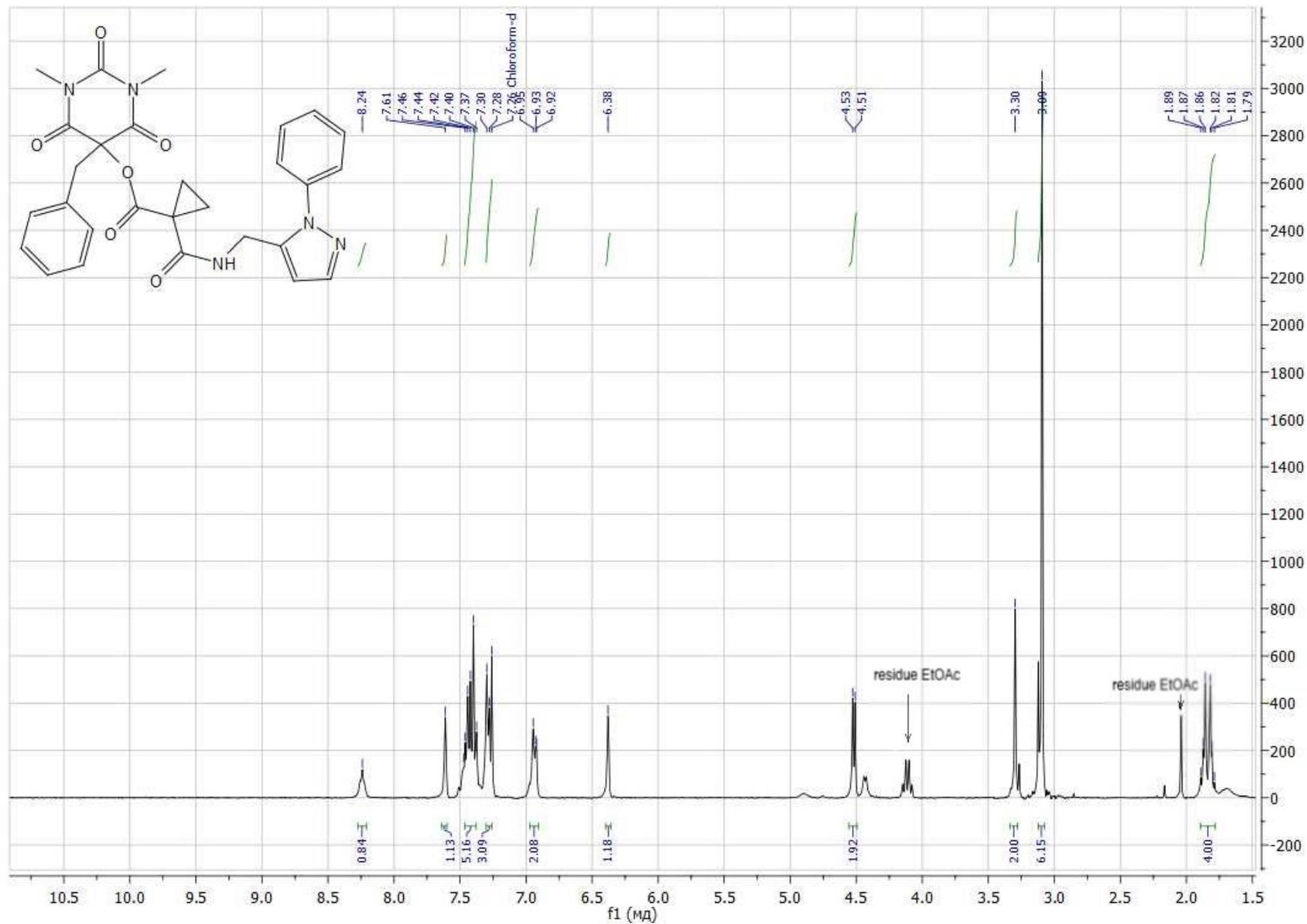
¹H NMR of 4-butyl-3,5-dioxo-1,2-diphenylpyrazolidin-4-yl 1-[N-(1-phenyl-1H-pyrazol-5-ylmethyl)carbamoyl]cyclobutane-1-carboxylate, **13**



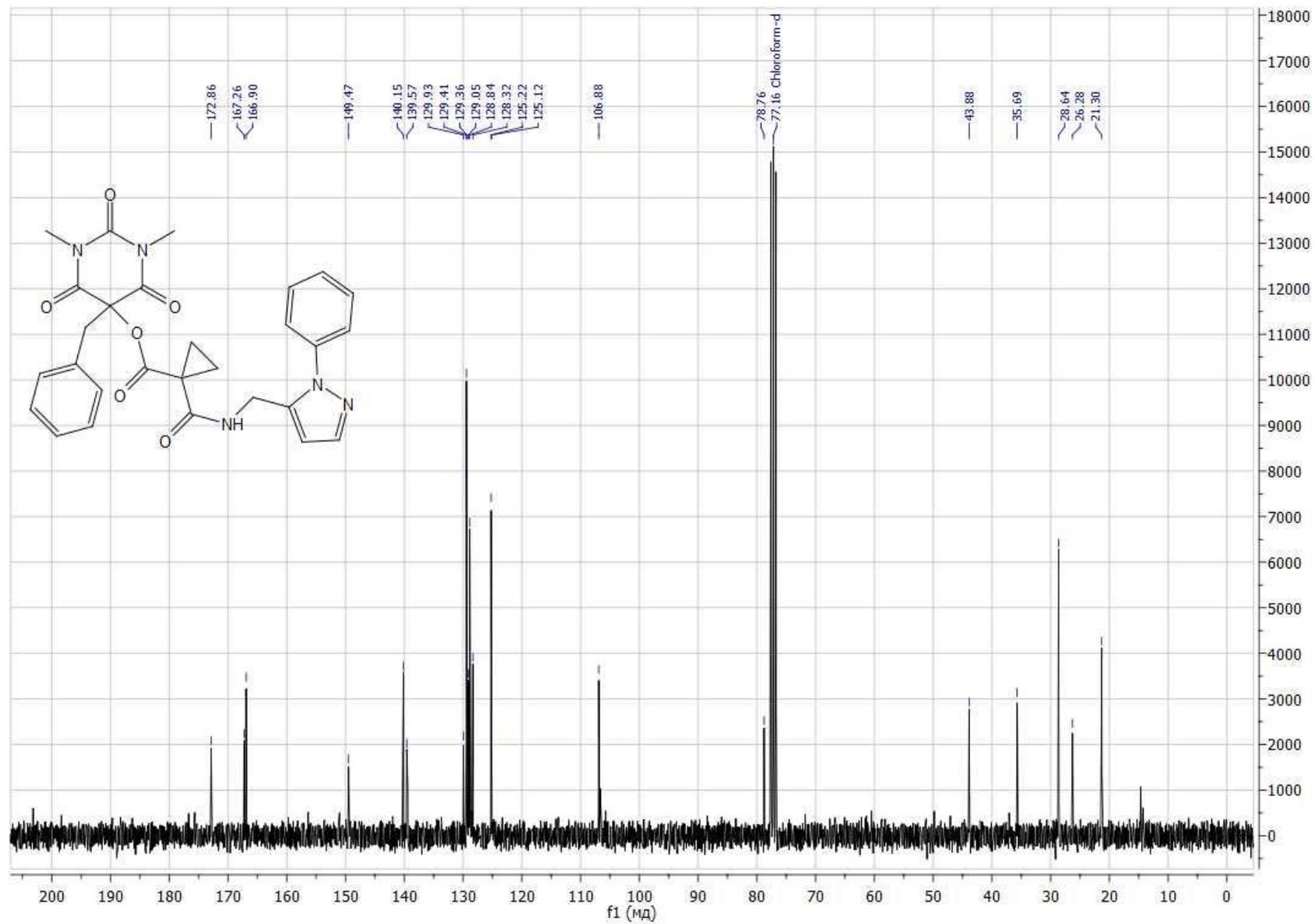
¹³C NMR of 4-butyl-3,5-dioxo-1,2-diphenylpyrazolidin-4-yl 1-[N-(1-phenyl-1H-pyrazol-5-ylmethyl)carbamoyl]cyclobutane-1-carboxylate, **13**



¹H NMR of 5-benzyl-1,3-dimethyl-2,4,6-trioxohexahydropyrimidin-5-yl 1-[N-(1-phenyl-1H-pyrazol-5-ylmethyl)carbamoyl]cyclobutane-1-carboxylate, **16**



^{13}C NMR of 5-benzyl-1,3-dimethyl-2,4,6-trioxohexahydropyrimidin-5-yl 1-[N-(1-phenyl-1*H*-pyrazol-5-ylmethyl)carbamoyl]cyclobutane-1-carboxylate, **16**



HRMS spectra of hybrid compounds 6a-e, 7, 10, 13 and 16

HRMS of 4-butyl-3-methyl-5-oxo-4,5-dihydro-1H-pyrazol-4-yl
1-(N-butylcarbamoyl)cyclopropane-1-carboxylate, **6a**

Display Report

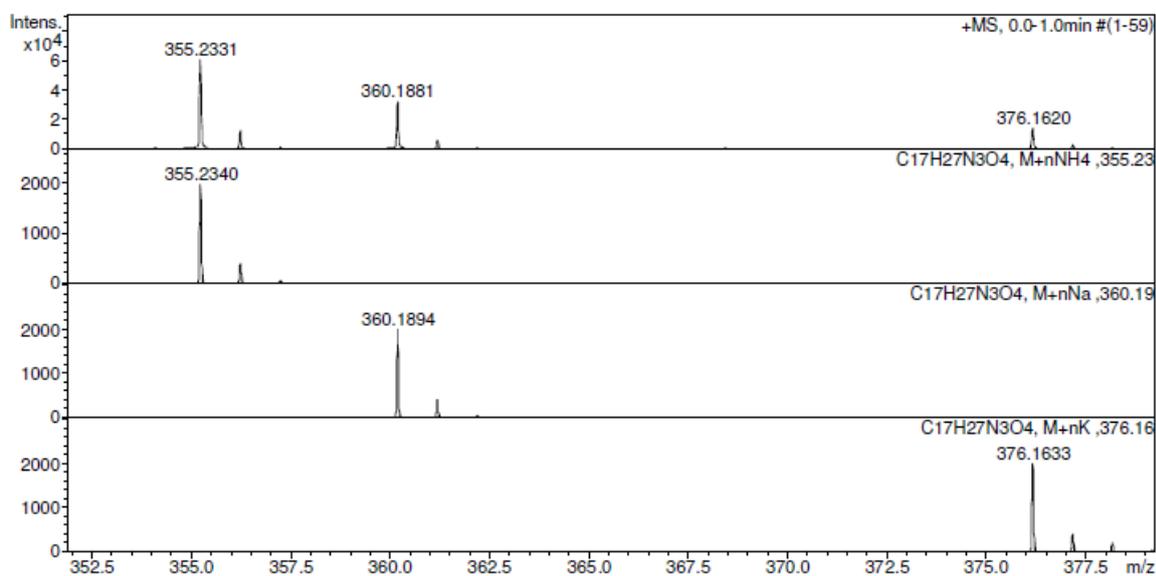
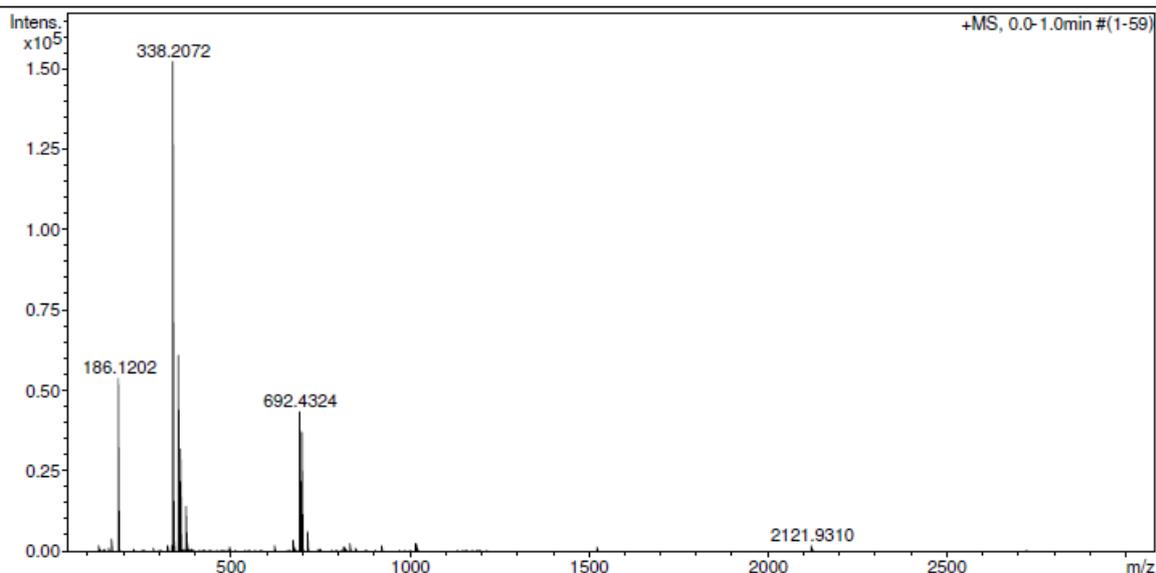
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Operator BDAL@DE
Instrument / Ser# micrOTOF 10248

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HRMS of 4-hexyl-3-methyl-5-oxo-4,5-dihydro-1H-pyrazol-4-yl
1-[N-(6-methoxybenzo[d]thiazol-2-yl)carbamoyl]cyclopropane-1-carboxylate, **6b**

Display Report

Analysis Info

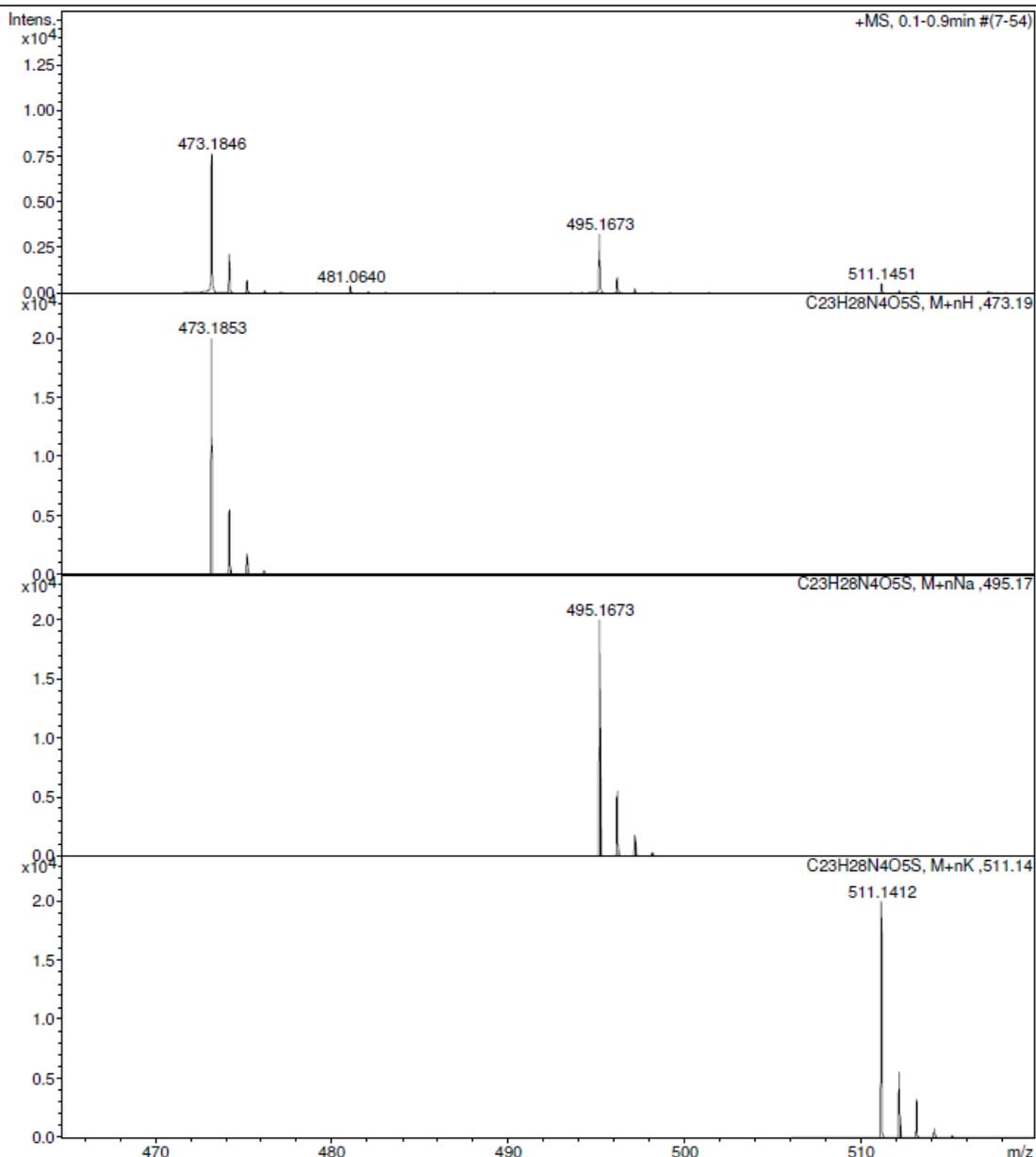
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HRMS of 4-hexyl-3-methyl-5-oxo-4,5-dihydro-1*H*-pyrazol-4-yl
1-[*N*-(quinolin-8-ylmethyl)carbamoyl]cyclobutane-1-carboxylate, **6c**

Display Report

Analysis Info

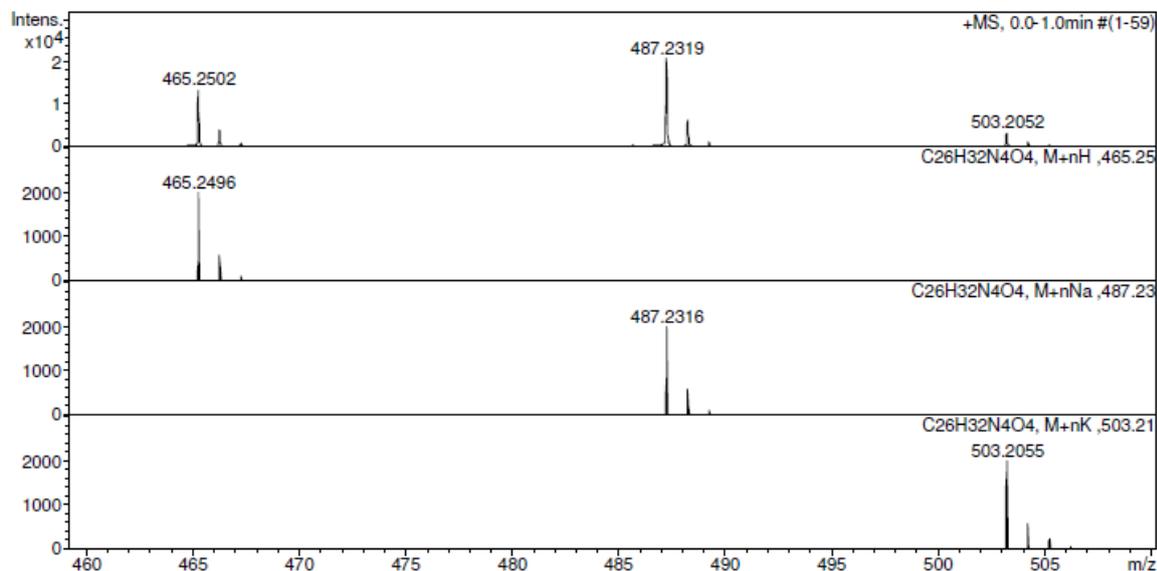
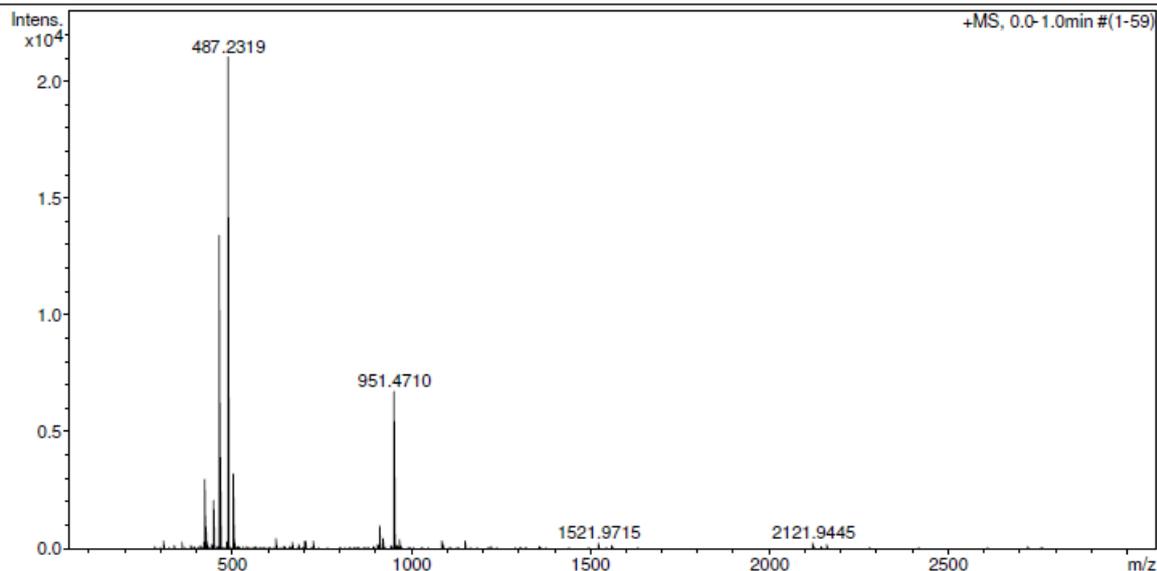
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Operator BDAL@DE
Instrument / Ser# micrOTOF 10248

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HRMS of 4-benzyl-3-methyl-5-oxo-4,5-dihydro-1H-pyrazol-4-yl
1-(morpholin-4-ylcarbonyl)cyclopropane-1-carboxylate, **6d**

Display Report

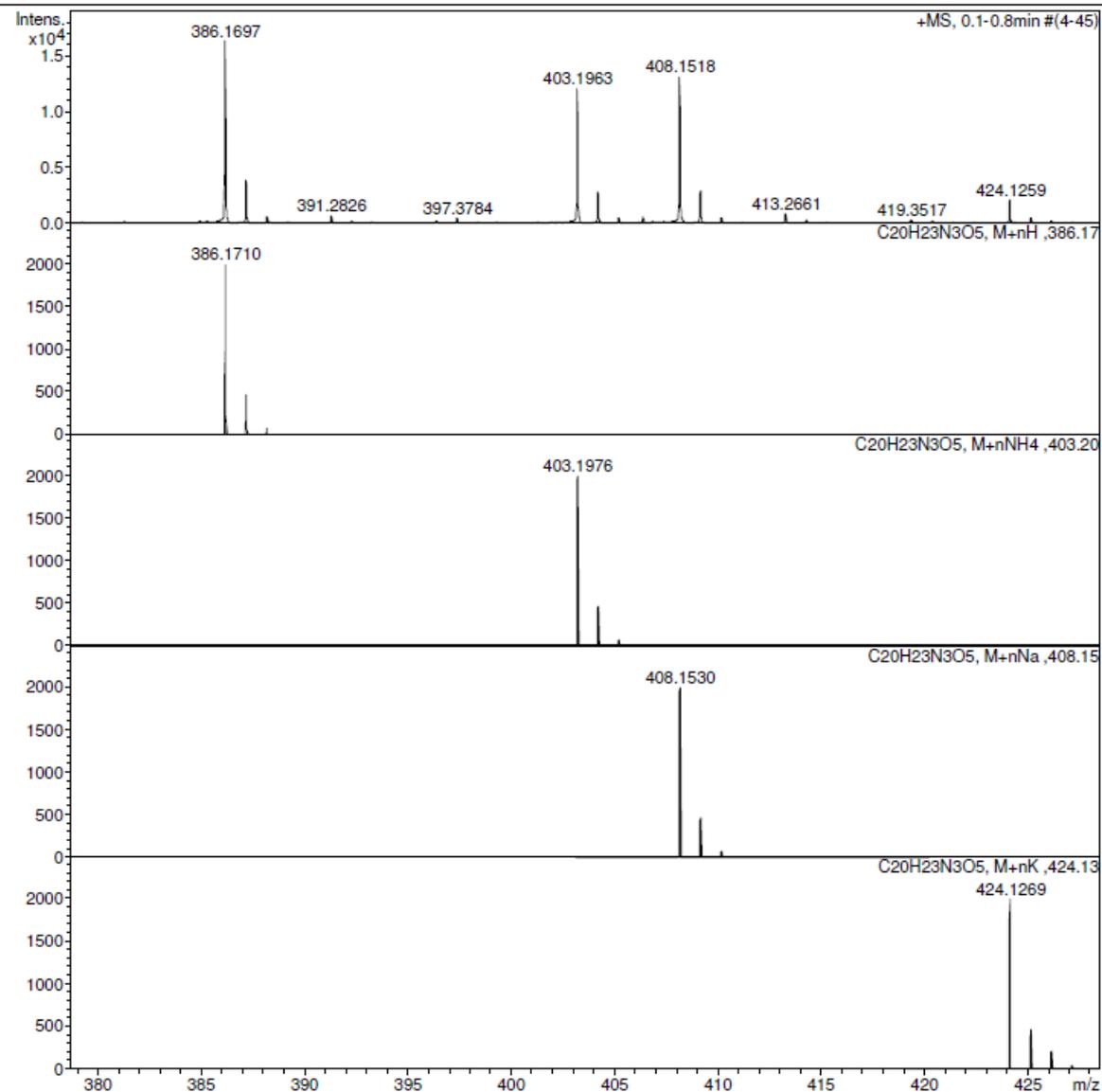
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HRMS of 4-benzyl-3-methyl-5-oxo-4,5-dihydro-1H-pyrazol-4-yl
1-[N-(quinolin-8-ylmethyl)carbamoyl]cyclopropane-1-carboxylate, **6e**

Display Report

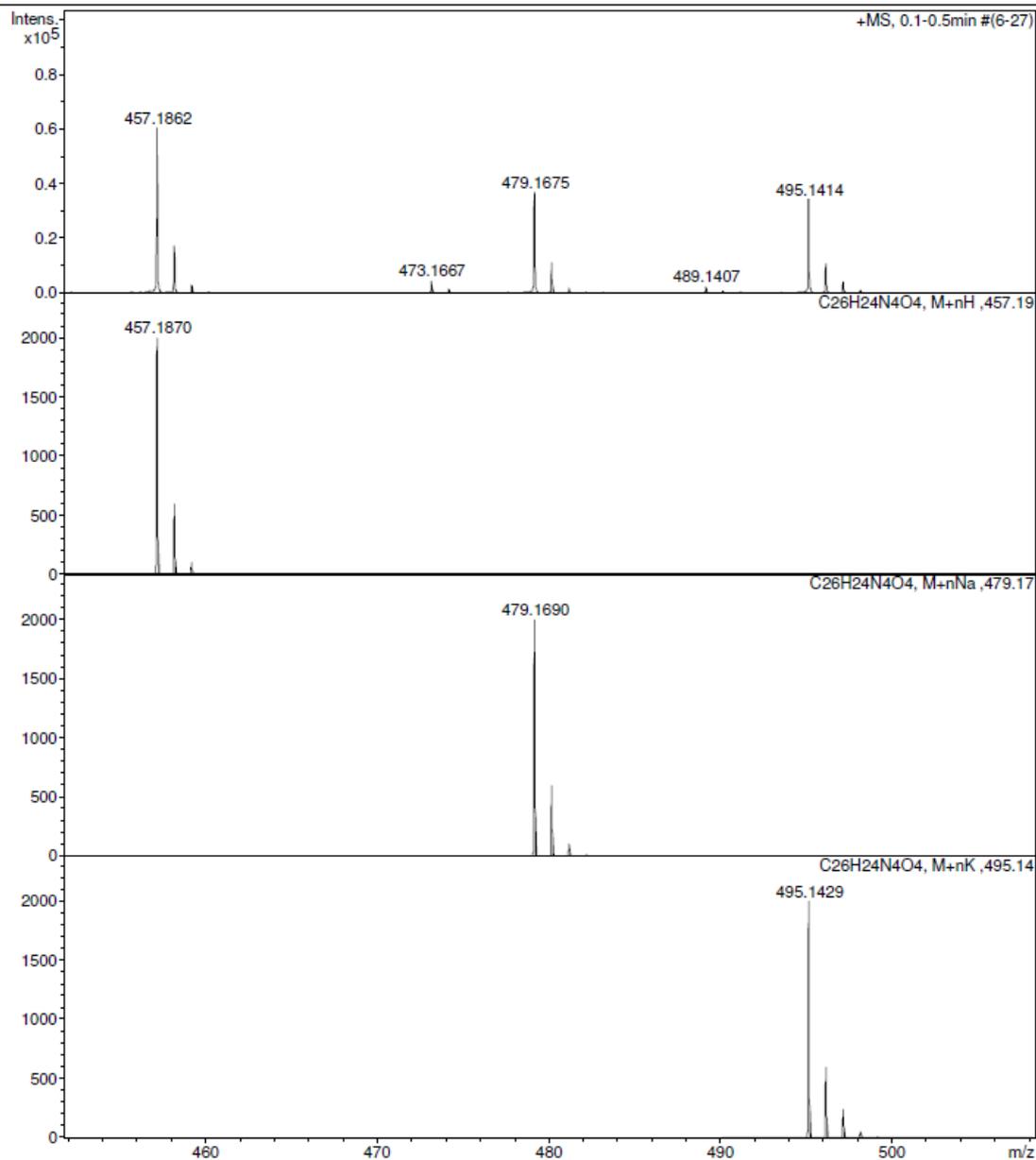
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HRMS of 4-benzyl-3-methyl-5-oxo-4,5-dihydro-1H-pyrazol-4-yl
(quinolin-8-yl)methyl cyclopropane-1,1-dicarboxylate, **7**

Display Report

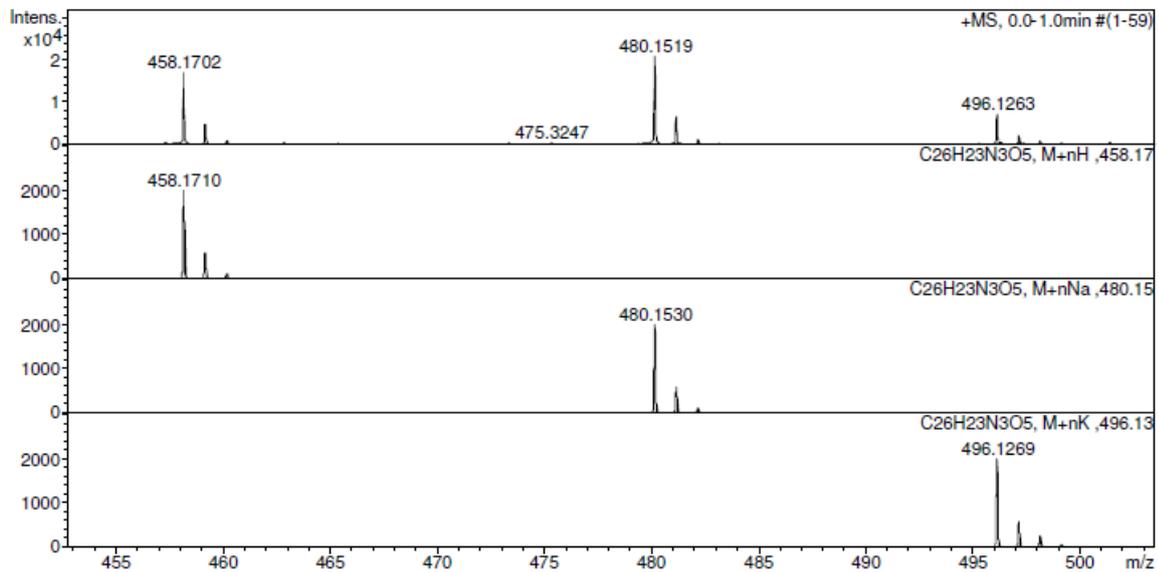
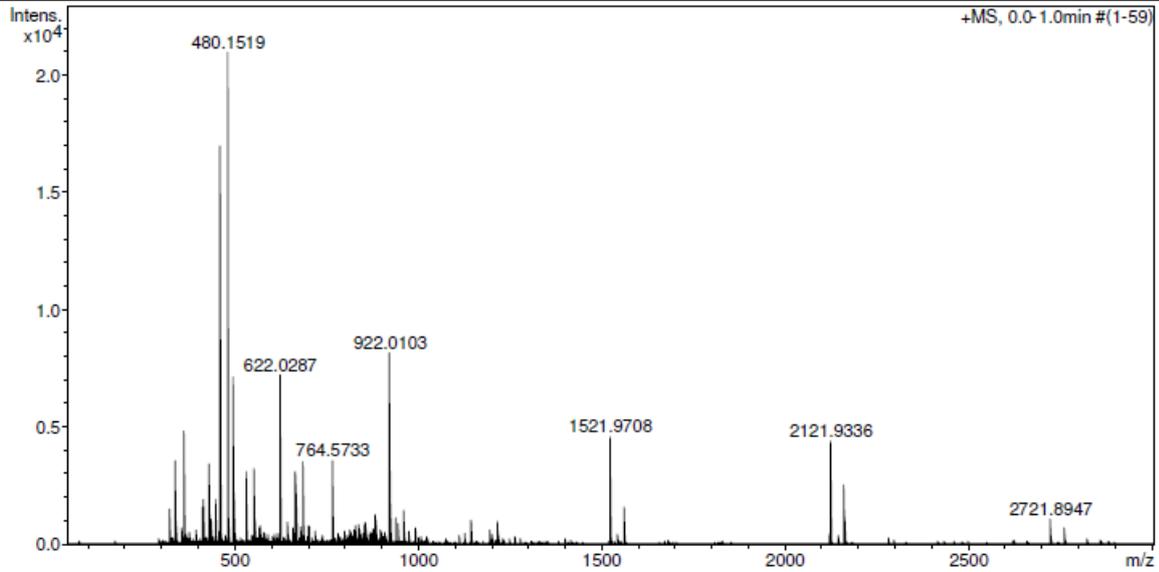
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HRMS of 4-benzyl-3-methyl-5-oxo-4,5-dihydroisoxazol-4-yl
1-[N-(quinolin-8-ylmethyl)carbamoyl]cyclopropane-1-carboxylate, **10**

Display Report

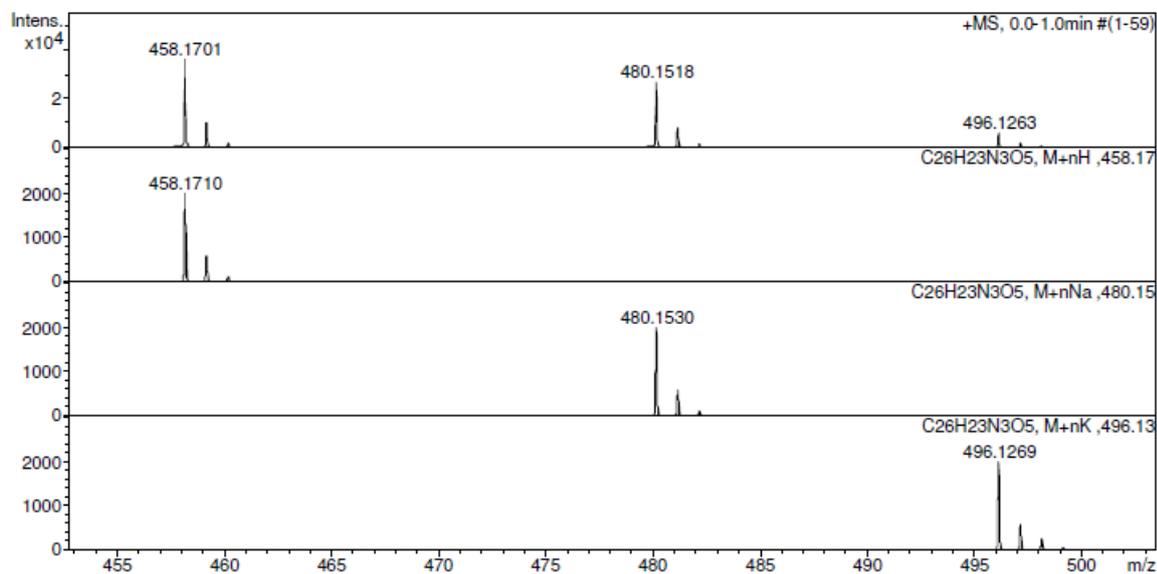
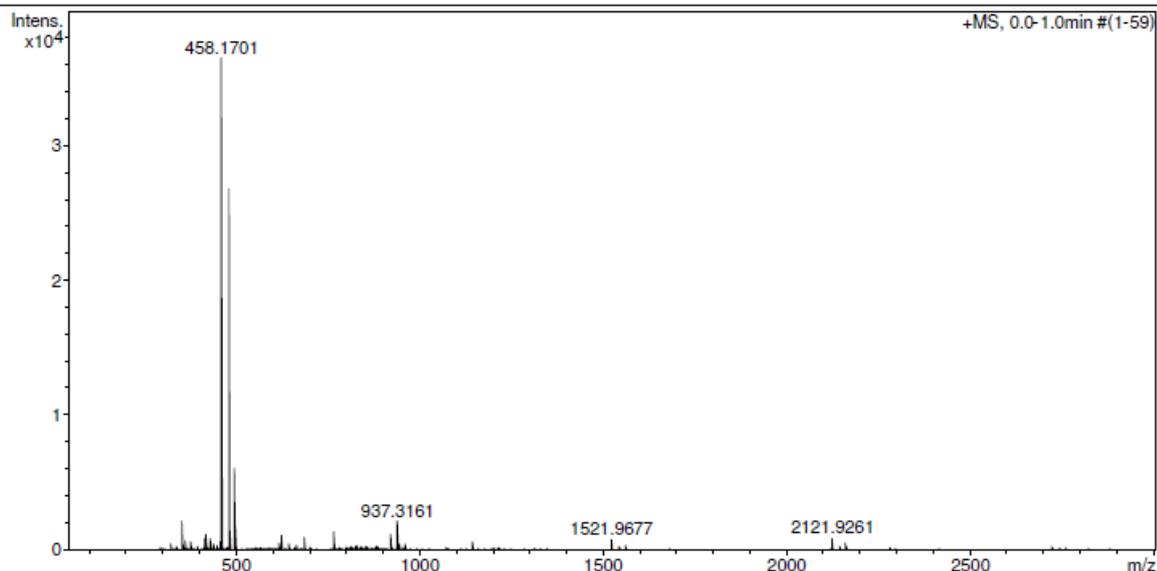
Analysis Info

Analysis Name D:\Data\Chizhov\Terentiev\Wilbb-1163_&clb.d
Method tune_wide.m
Sample Name /TERN BB-1163
Comment CH3CN 100 %, dil. 200, calibrant added

Acquisition Date 04.07.2016 15:53:23
Operator BDAL@DE
Instrument / Ser# micrOTOF 10248

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 of 4-butyl-3,5-dioxo-1,2-diphenylpyrazolidin-4-yl
1-[N-(1-phenyl-1H-pyrazol-5-ylmethyl)carbamoyl]cyclobutane-1-carboxylate, **13**

Display Report

Analysis Info

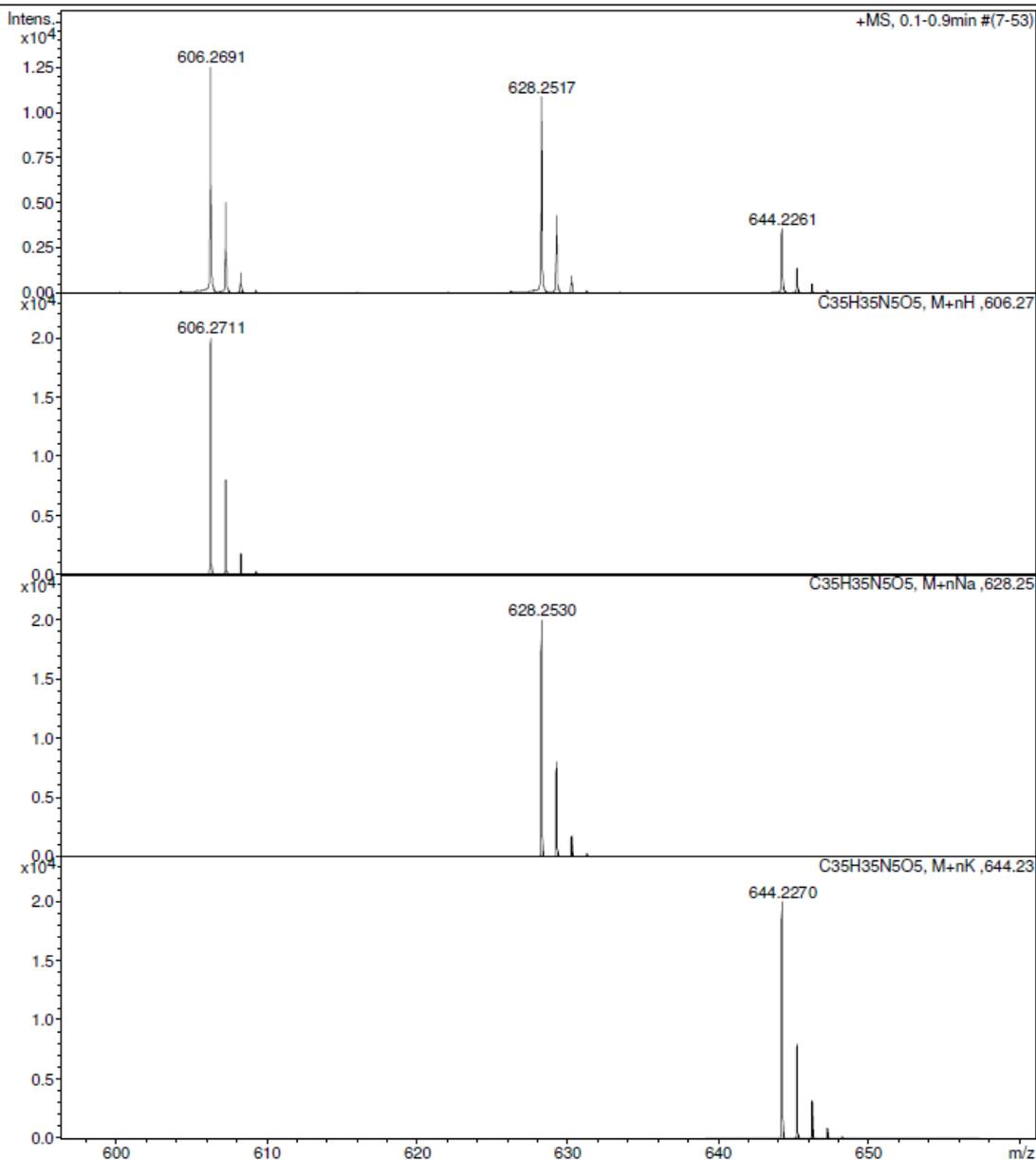
Analysis Name D:\Data\Kolotyrkina\2016\Viil\0607023.d
Method tune_wide.m
Sample Name /TERN BB-1118
Comment C35H35N5O5 mH 606.271 calibrant added

Acquisition Date 07.06.2016 15:03:55

Operator BDAL@DE
Instrument / Ser# micrOTOF 10248

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

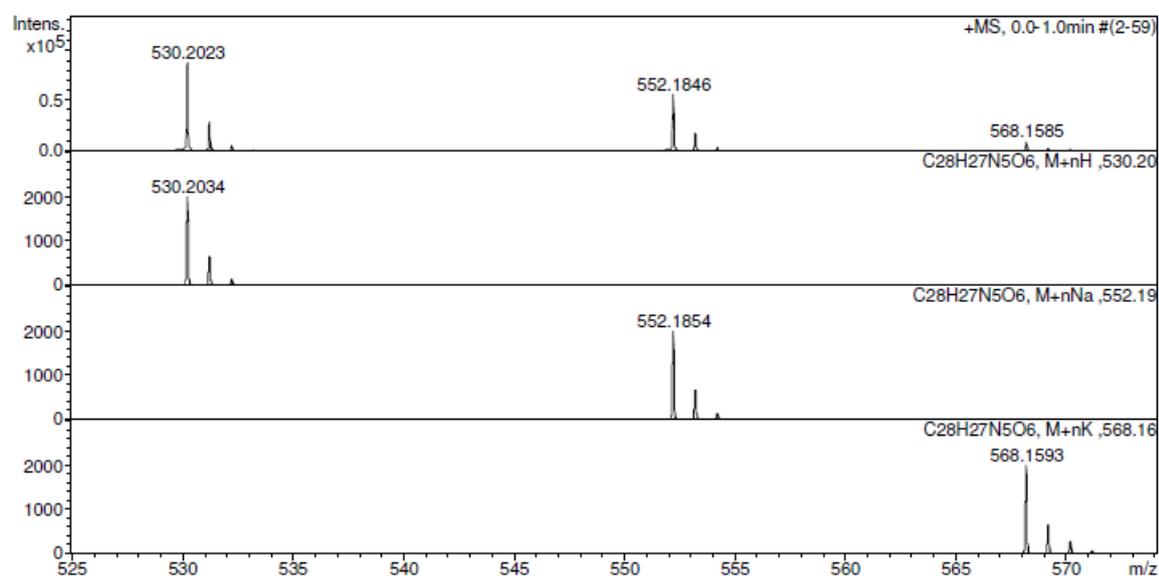
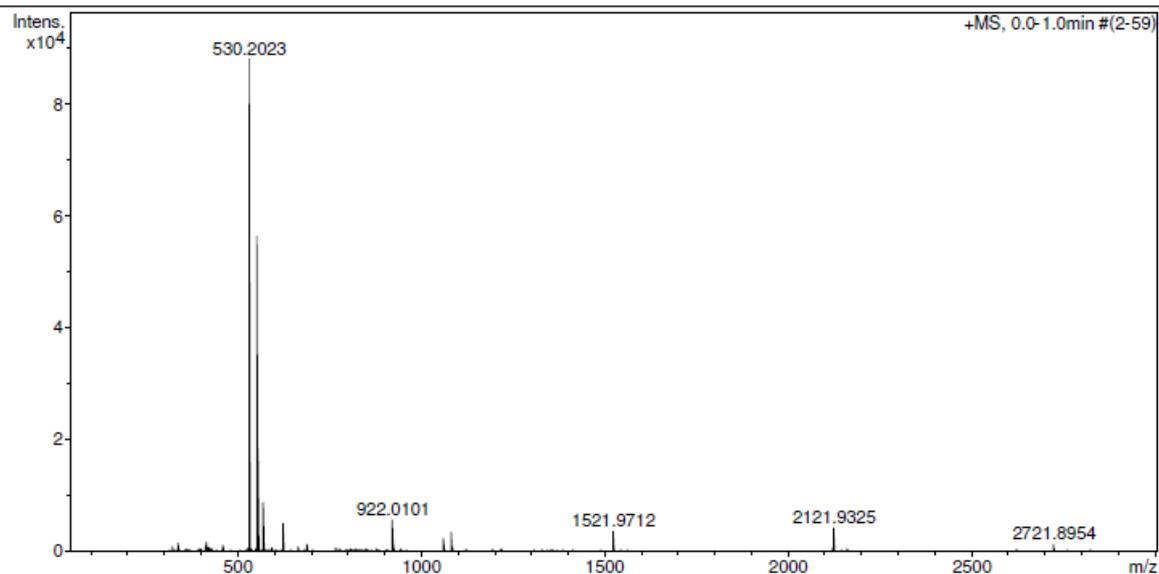


HRMS of 5-benzyl-1,3-dimethyl-2,4,6-trioxohexahydropyrimidin-5-yl
 1-[N-(1-phenyl-1H-pyrazol-5-ylmethyl)carbamoyl]cyclopropane-1-carboxylate, **16**

Display Report

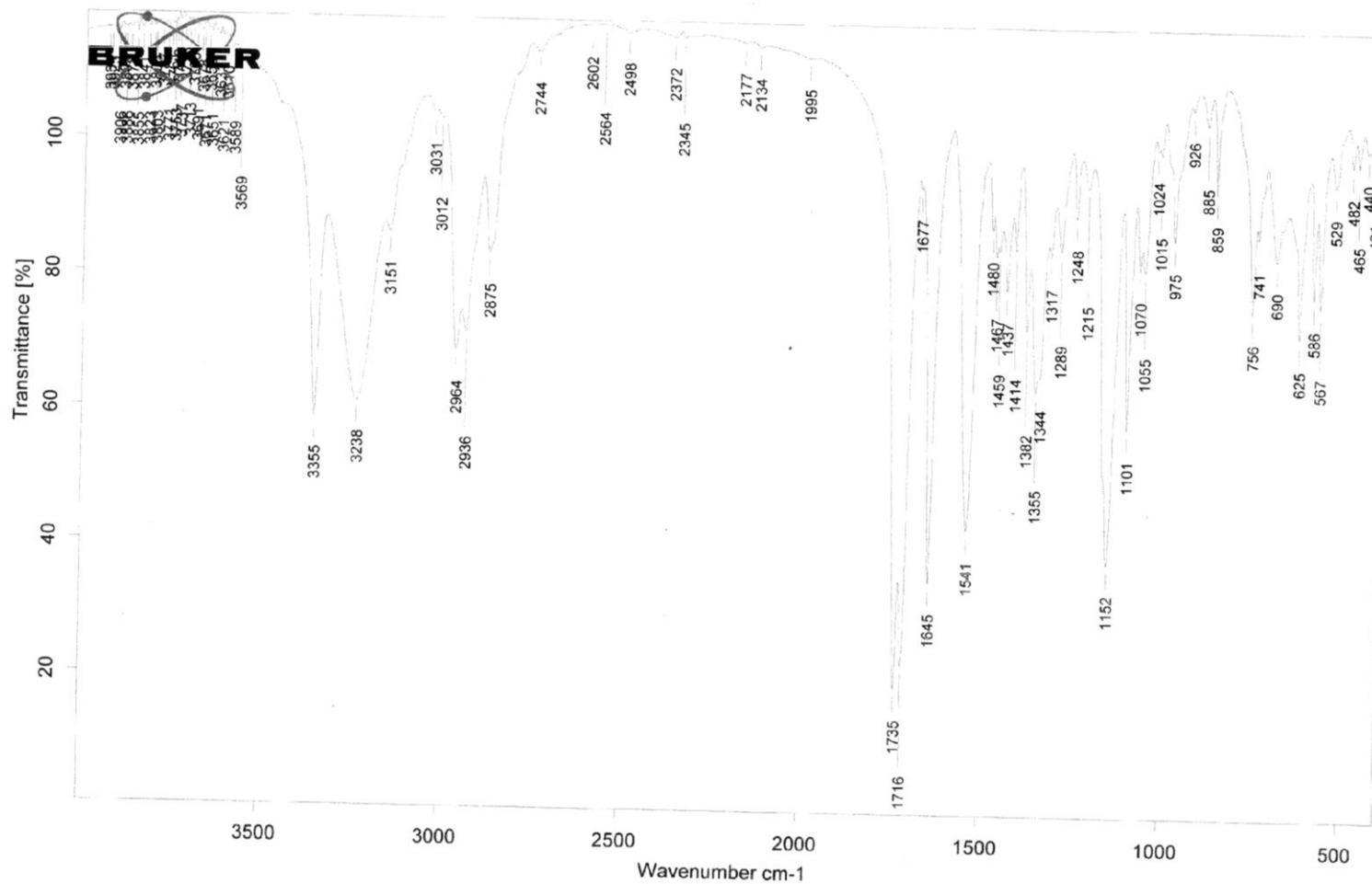
Analysis Info		Acquisition Date	04.07.2016 15:25:44	
Analysis Name	D:\Data\Chizhov\Terentiev\Wife-69_&clb.d	Operator	BDAL@DE	
Method	tune_wide.m	Instrument / Ser#	micrOTOF 10248	
Sample Name	/TERN E-69			
Comment	CH3CN 100 %, dil. 2000, calibrant added			

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



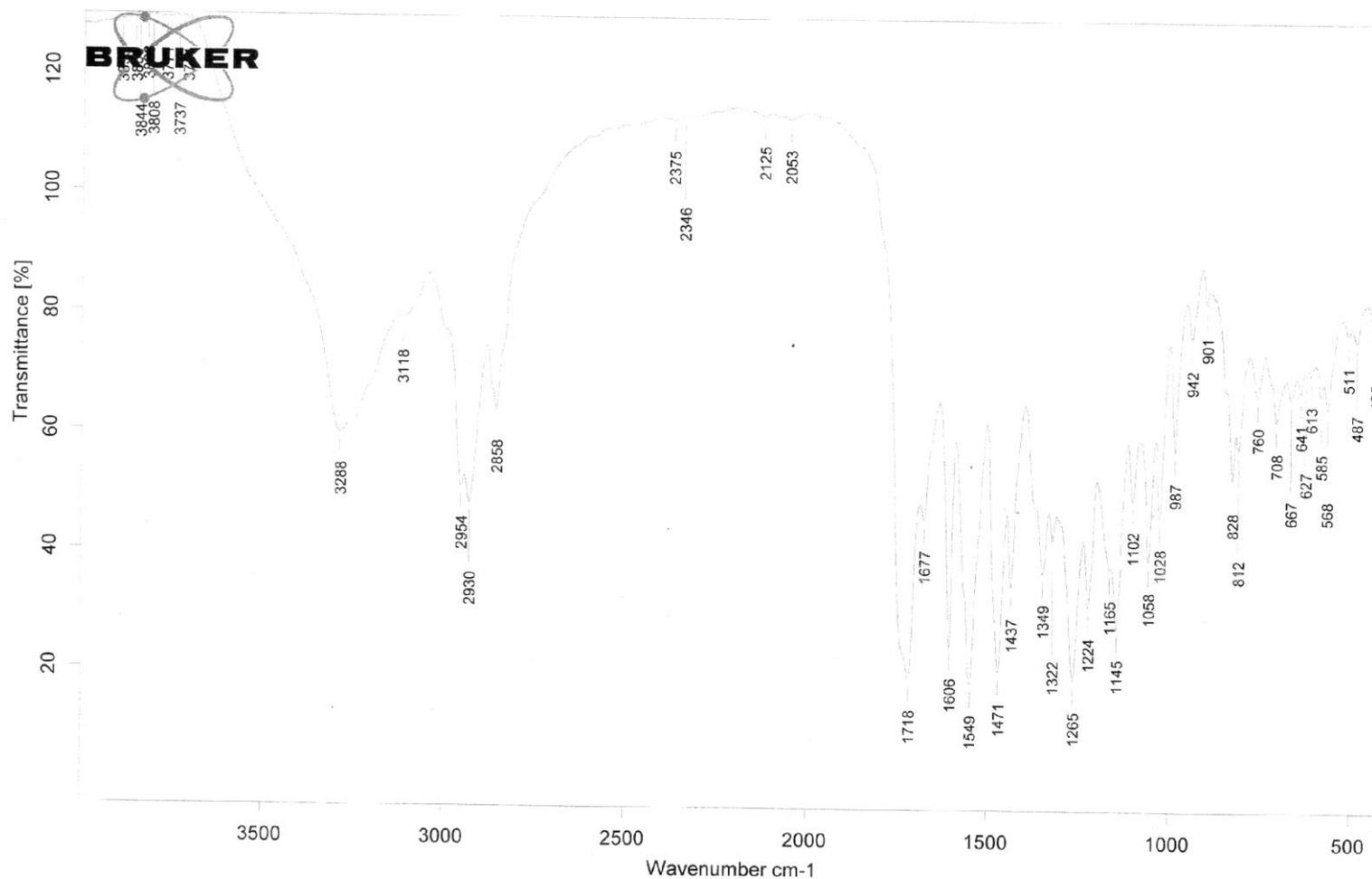
IR spectra of hybrid compounds 6a-e, 7, 10, 13 and 16

IR of 4-butyl-3-methyl-5-oxo-4,5-dihydro-1H-pyrazol-4-yl 1-(N-butylcarbamoyl)cyclopropane-1-carboxylate, **6a**



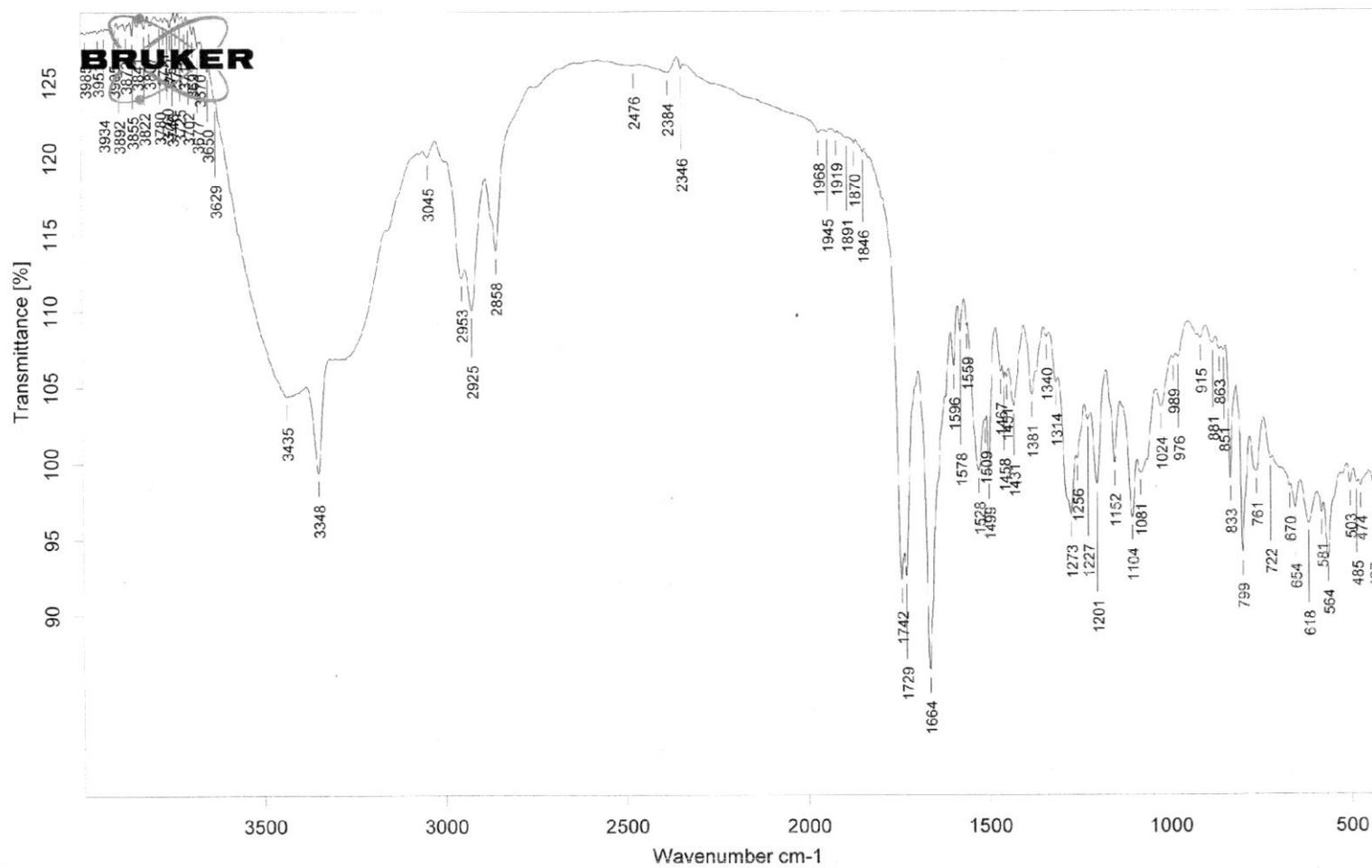
D:\EDL\BB-1068.0 Виль. ВВ-1068 , пресовка с KBr,0,5/200. 31.03.2016

IR of 4-hexyl-3-methyl-5-oxo-4,5-dihydro-1H-pyrazol-4-yl 1-[N-(6-methoxybenzo[d]thiazol-2-yl)carbamoyl]cyclopropane-1-carboxylate, **6b**



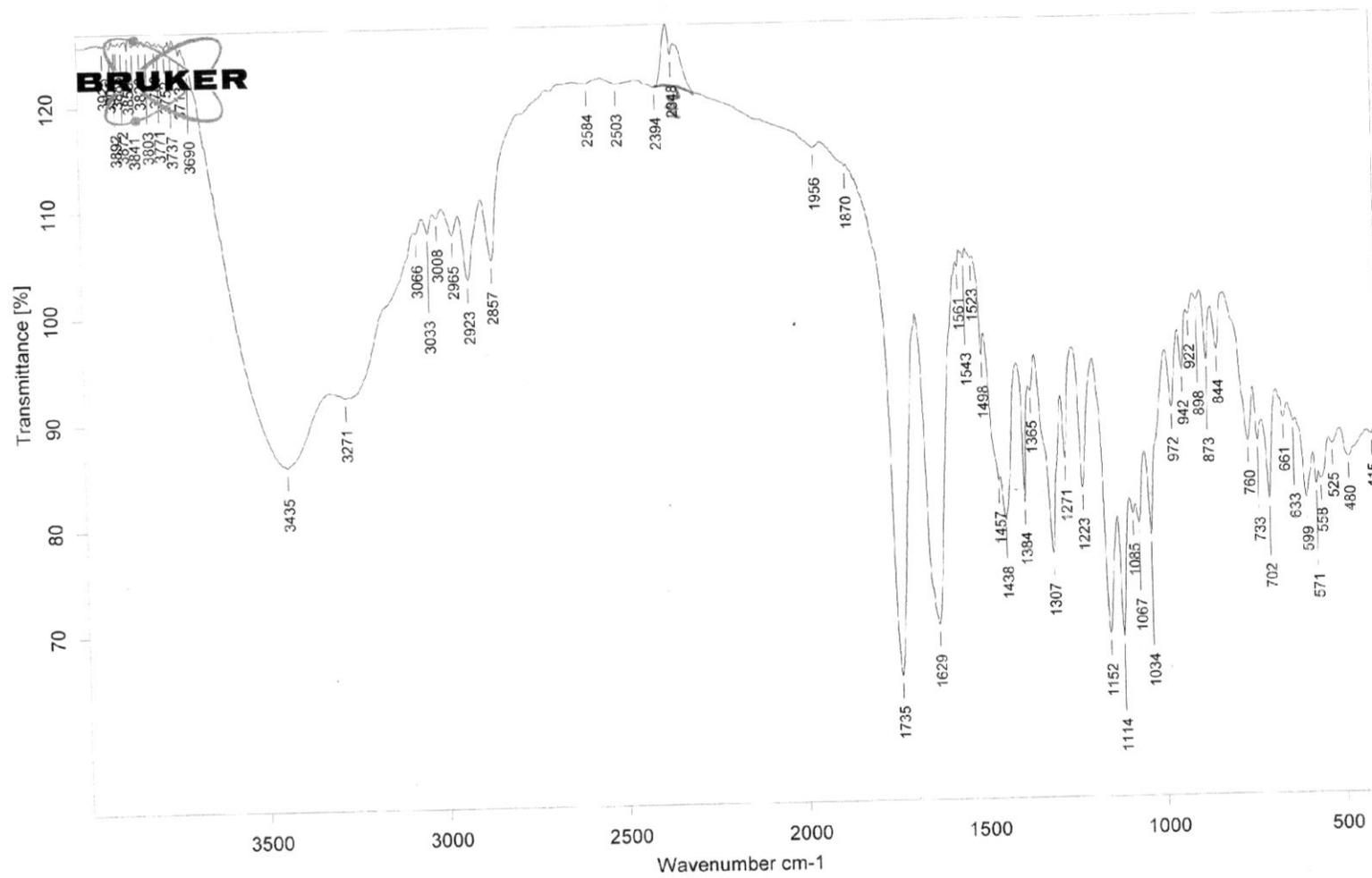
DATE: LIBB-1121.0 ВИЛЬ. ВВ-1121 , прессовка с KBr. 12.07.2016

IR of 4-hexyl-3-methyl-5-oxo-4,5-dihydro-1*H*-pyrazol-4-yl 1-[*N*-(quinolin-8-ylmethyl)carbamoyl]cyclobutane-1-carboxylate, **6c**



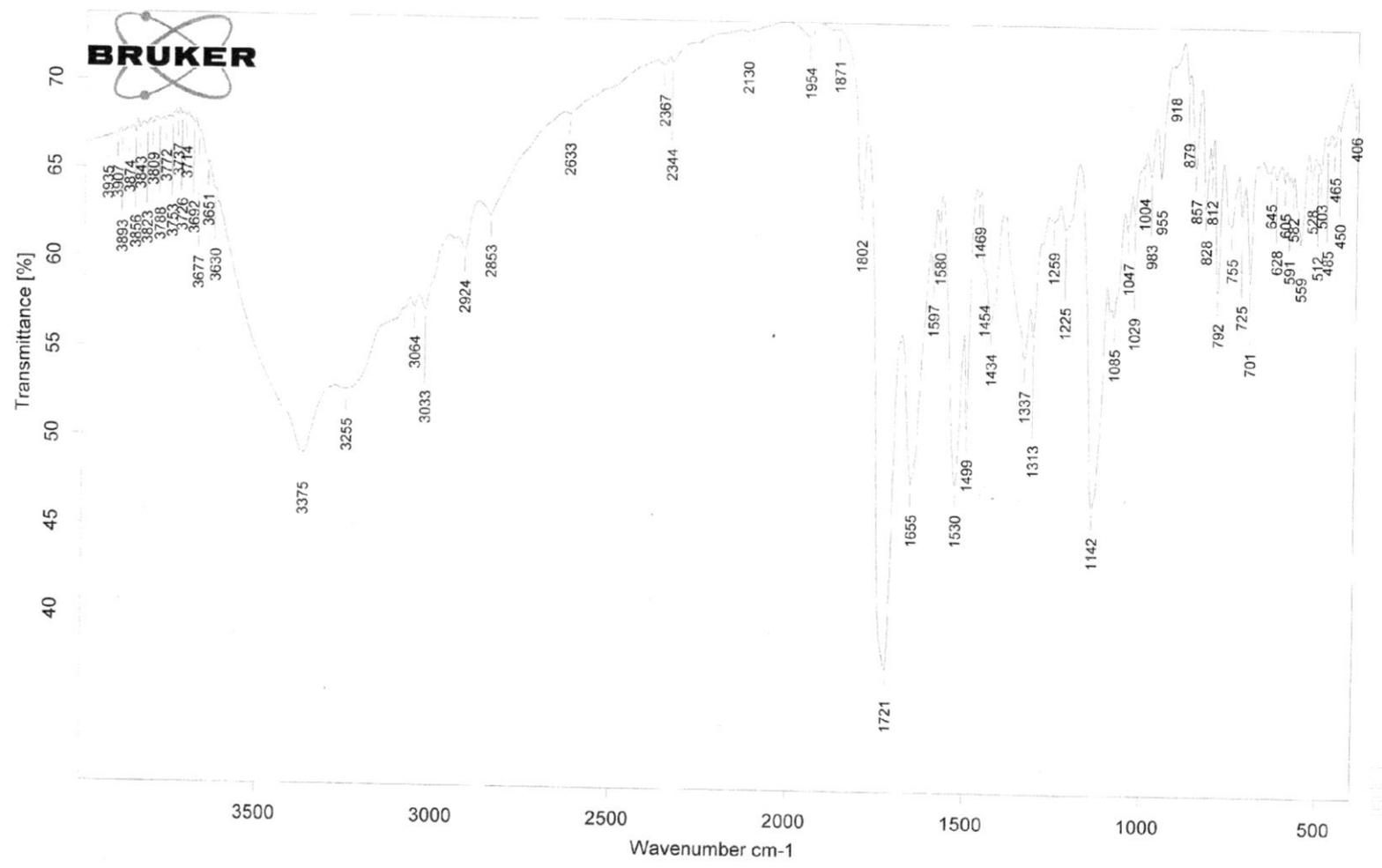
D:\EDL\BB-1107.0	ВИЛЬ. ВВ-1107 , прессовка с KBr.	12.07.2016
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IR of 4-benzyl-3-methyl-5-oxo-4,5-dihydro-1H-pyrazol-4-yl 1-(morpholin-4-ylcarbonyl)cyclopropane-1-carboxylate, **6d**



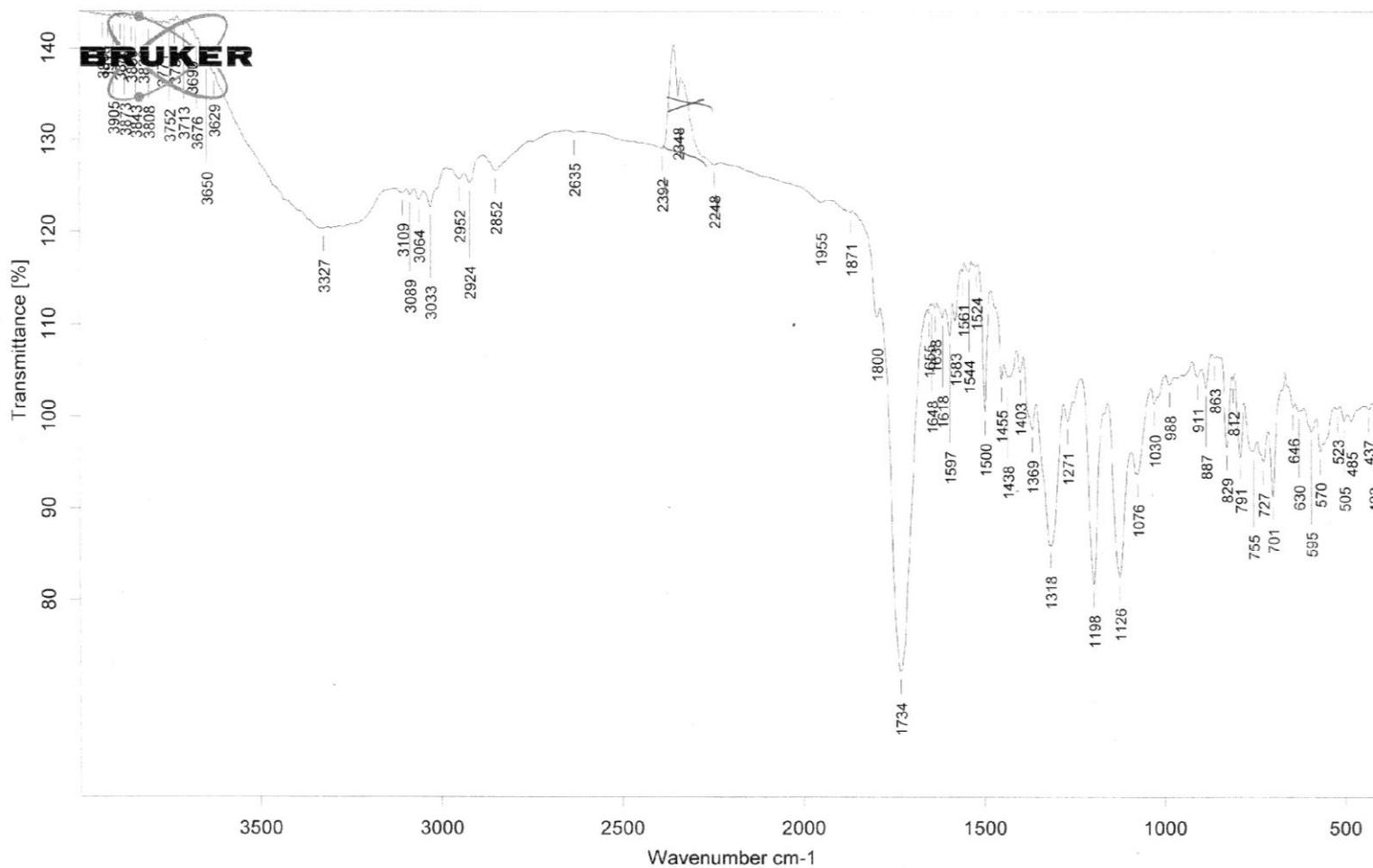
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IR of 4-benzyl-3-methyl-5-oxo-4,5-dihydro-1H-pyrazol-4-yl 1-[N-(quinolin-8-ylmethyl)carbamoyl]cyclopropane-1-carboxylate, **6e**



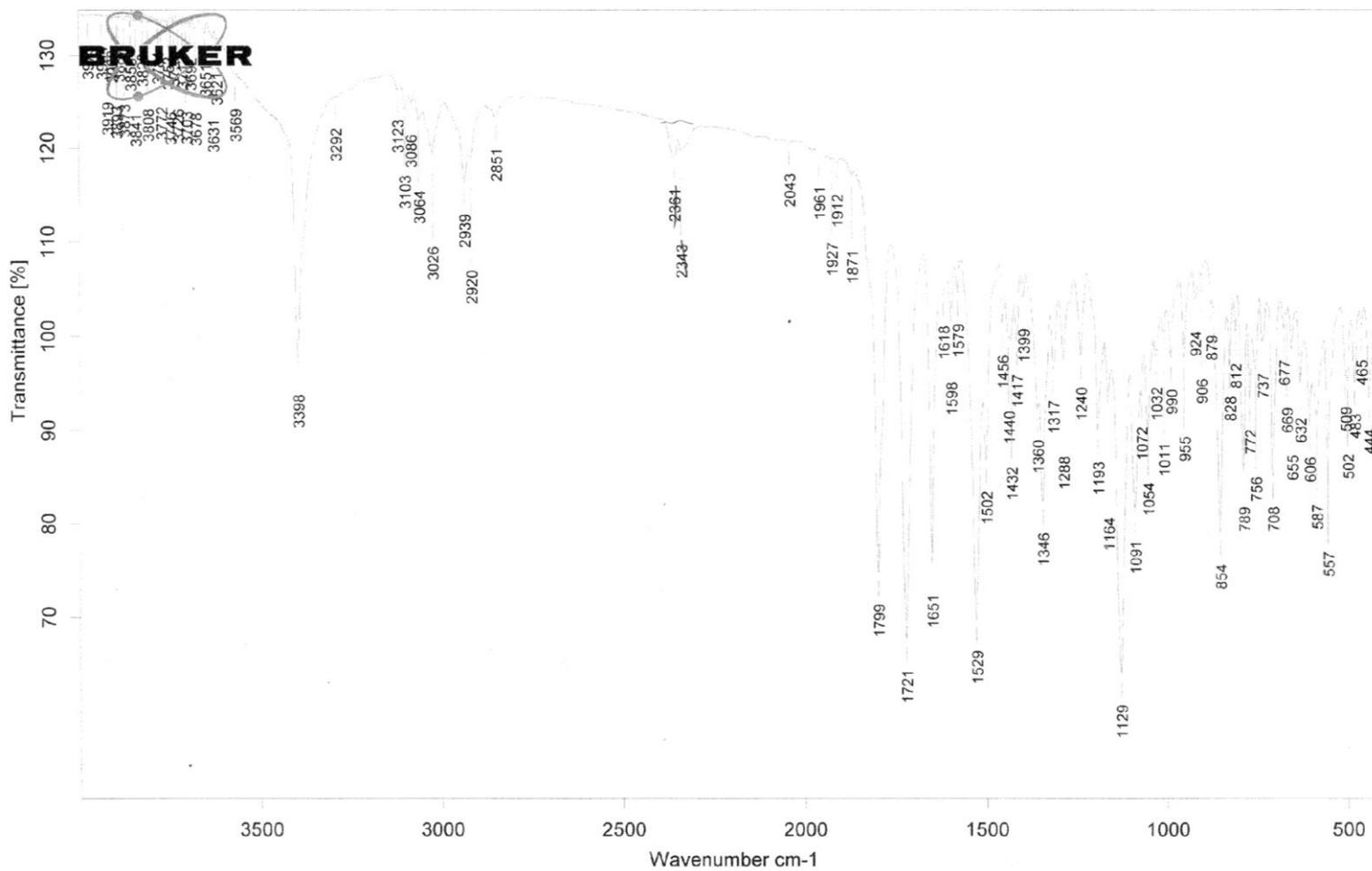
D:\EDL\BB-1119.0 Виль. ВВ-1119 , прессовка с KBr. 12.07.2016

IR of 4-benzyl-3-methyl-5-oxo-4,5-dihydro-1H-pyrazol-4-yl (quinolin-8-yl)methyl cyclopropane-1,1-dicarboxylate, **7**

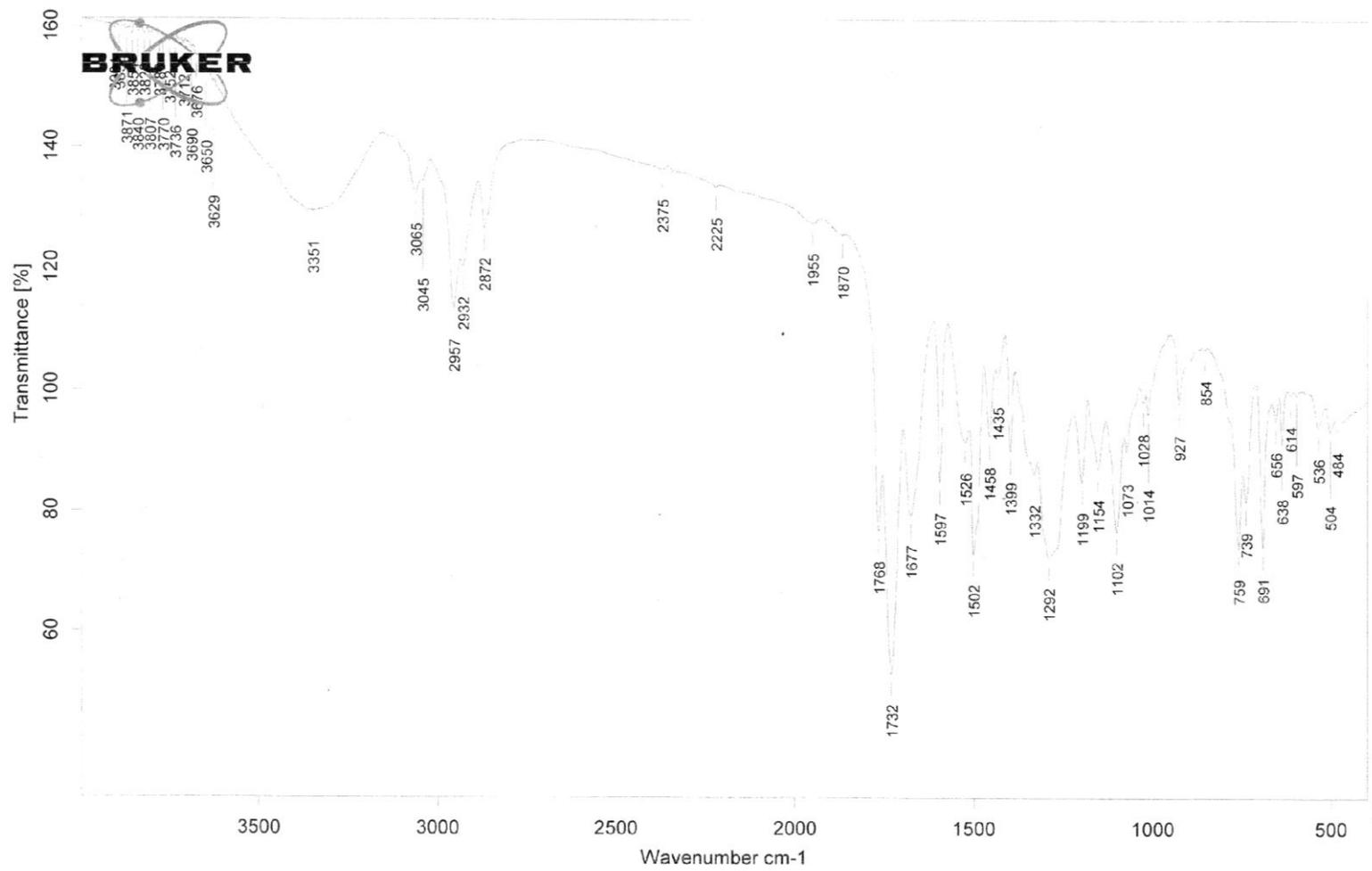


D:\EDL\BB-1148.0	ВИЛЬ. ВВ-1148 , прессовка с KBr.	12.07.2016
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IR of 4-benzyl-3-methyl-5-oxo-4,5-dihydroisoxazol-4-yl 1-[N-(quinolin-8-ylmethyl)carbamoyl]cyclopropane-1-carboxylate, **10**

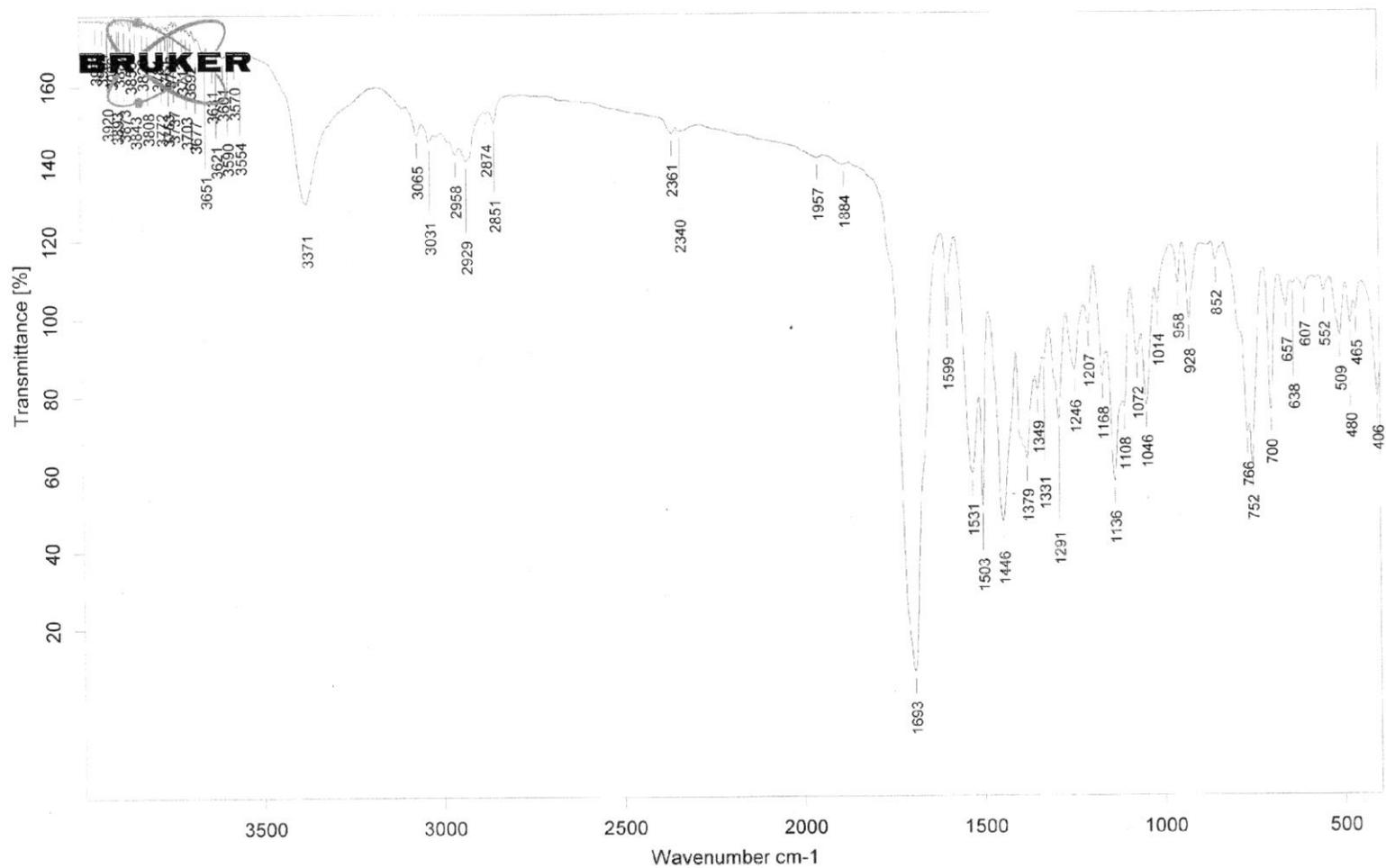


IR of 4-butyl-3,5-dioxo-1,2-diphenylpyrazolidin-4-yl 1-[N-(1-phenyl-1H-pyrazol-5-ylmethyl)carbamoyl]cyclobutane-1-carboxylate, **13**



D:\EДL\BB-1118.0	Виль. ВВ-1118 , прессовка с KBr.	13.07.2016
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IR of 5-benzyl-1,3-dimethyl-2,4,6-trioxohexahydropyrimidin-5-yl 1-[N-(1-phenyl-1H-pyrazol-5-ylmethyl)carbamoyl]cyclobutane-1-carboxylate, **16**



D:\EDL\BB-c-69.0	ВИЛЬ, ВВ-с-69+СНСЗ, тонкий слой, пл.КВг.	13.07.2016
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