

On water noncatalytic tandem Knoevenagel–Michael reaction of aldehydes, *N,N'*-dimethylbarbituric acid and cyclohexane-1,3-diones

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All melting points were measured with a Gallenkamp melting point apparatus and are uncorrected. ¹H and ¹³C NMR spectra were recorded with a Bruker AM-300 (300 and 75 MHz, respectively) at ambient temperature in DMSO-*d*₆ solutions. Chemical shift values are given in δ scale relative to Me₄Si. IR spectra were registered with a Bruker ALPHA-T FT-IR spectrometer in KBr pellets. Mass-spectra (EI = 70 eV) were obtained directly with a Finningan MAT INCOS 50 spectrometer.

On-water procedure

The mixture of benzaldehyde (5 mmol), *N,N'*-dimethylbarbiturate (5 mmol, 0,78 g), and cyclohexane-1,3-dione **2a** or **2b** (3 mmol) in 20 mL of water was stirred at 60 C for 15 min. When the reaction was completed, the solid was filtrated, washed by cold ethanol (2 x 3 mL) and in some cases additionally was purified by recrystallization from EtOH.

6-Hydroxy-5-[(2-hydroxy-6-oxocyclohex-1-en-1-yl)(phenyl)methyl]-1,3-dimethylpyrimidine-2,4(1*H*,3*H*)-dione (3a)

Yield 1.71 g (96%), mp: 190-192 °C; ¹H NMR (300 MHz, CDCl₃): δ 2.04-2.10 (m, 2H, CH₂), 2.33-2.52 (m, 2H, CH₂), 2.62-2.73 (m, 2H, CH₂), 3.38 (s, 3H, CH₃), 3.47 (s, 3H, CH₃), 5.57 (s, 1H, CH), 7.11-7.34 (m, 5H, Ar), 8.10 (br s, 1H, OH), 13.15 (br s, 1H, OH) ppm. ¹³C NMR (75 MHz, CDCl₃): δ 19.9, 29.0, 29.2, 32.4, 33.6, 33.7, 92.3, 117.5, 126.3, 126.5 (2C), 128.3 (2C), 137.2, 150.8, 162.3, 164.7, 191.3, 193.6 ppm. MS (m/z, relative intensity %): 356 [M⁺] (11), 312 (1), 257 (3), 243 (14), 199 (39), 156 (21), 129 (18), 102 (35), 56 (27), 42 (100). IR (KBr): 3469, 2936, 2882, 1698, 1609, 1490, 1476, 1356, 1200, 882 cm⁻¹. Anal. calcd for C₁₉H₂₀N₂O₅: C, 64.04; H, 5.66; N, 7.86. Found C, 63.95; H, 5.57; N, 7.75%.

6-Hydroxy-5-[(2-hydroxy-6-oxocyclohex-1-en-1-yl)(4-methylphenyl)methyl]-1,3-dimethylpyrimidine-2,4(1*H*,3*H*)-dione (3b)

Yield 1.69 g (91%), mp: 155-157 °C; ¹H NMR (300 MHz, CDCl₃): δ 2.02-2.09 (m, 2H, CH₂), 2.33 (s, 3H, CH₃), 2.36-2.50 (m, 2H, CH₂), 2.60-2.72 (m, 2H, CH₂), 3.37 (s, 3H, CH₃), 3.45 (s, 3H, CH₃), 5.52 (s, 1H, CH), 7.02 (d *J* = 8.1 Hz, 2H, Ar), 7.10 (d *J* = 8.1 Hz, 2H, Ar), 10.06 (br s, 1H, OH), 13.13 (br s, 1H, OH) ppm. ¹³C NMR (75 MHz, CDCl₃): δ 19.9, 21.0, 29.0, 29.2, 32.4, 33.4, 33.7, 92.4, 117.7, 126.4 (2C), 129.0 (2C), 134.0, 135.7, 150.8, 162.3, 164.7, 191.3, 193.5 ppm. MS (m/z, relative intensity %): 370 [M⁺] (9), 313 (1), 271 (3), 257 (17), 199 (91), 159 (49), 129 (22), 115 (80), 58 (40), 28 (100). IR (KBr): 3449, 2938, 2887, 1707, 1603, 1509, 1475, 1354, 1194, 843, cm⁻¹. Anal. calcd for C₂₀H₂₂N₂O₅: C, 64.85; H, 5.99; N, 7.56. Found C, 64.73; H, 5.83; N, 7.39%.

6-Hydroxy-5-[(2-hydroxy-6-oxocyclohex-1-en-1-yl)(4-methoxyphenyl)methyl]-1,3-dimethylpyrimidine-2,4(1*H*,3*H*)-dione (3c)

Yield 1.58 g (82%), mp: 148-150 °C. ¹H NMR (300 MHz, DMSO-*d*₆): δ 2.04-2.09 (m, 2H, CH₂), 2.38-2.49 (m, 2H, CH₂), 2.61-2.73 (m, 2H, CH₂), 3.38 (s, 3H, CH₃), 3.46 (s, 3H, CH₃), 3.80 (s, 3H, OCH₃), 5.51 (s, 1H, CH), 6.84 (d, *J* = 8.6 Hz, 2H, Ar), 7.05 (d, *J* = 8.6 Hz, 2H, Ar), 10.80 (br s, 1H), 13.14 (br s, 1H) ppm. ¹³C NMR (DMSO-*d*₆): δ 19.8, 28.9, 29.1, 32.4, 33.0, 33.7, 55.2, 92.4, 113.7 (2C), 117.7, 127.5 (2C), 128.9, 150.8, 157.9, 162.3, 164.6, 191.3, 193.3 ppm. MS (m/z, relative intensity %): 386 [M⁺] (33), 287 (3), 274 (22), 229 (51), 199 (27), 156 (16), 117 (12), 89 (23), 42 (100). IR (KBr): 3435, 2941, 2834, 1700, 1604, 1509, 1246, 1039, 839, 756 cm⁻¹. Anal. calcd for C₂₀H₂₂N₂O₆: C, 62.17; H, 5.74; N, 7.25. Found C, 62.08; H, 5.71; N, 7.11%.

5-[(4-Fluorophenyl)(2-hydroxy-6-oxocyclohex-1-en-1-yl)methyl]-6-hydroxy-1,3-dimethylpyrimidine-2,4(1*H*,3*H*)-dione (3d)

Yield 1.69 g (90%), mp: 170-172 °C (Lit. mp¹: 154 °C). ¹H NMR (300 MHz, DMSO-*d*₆): δ 2.01-2.12 (m, 2H, CH₂), 2.32-2.53 (m, 2H, CH₂), 2.58-2.76 (m, 2H, CH₂), 3.38 (s, 3H, CH₃), 3.46 (s, 3H, CH₃), 5.50 (s, 1H, CH), 6.96-7.03 (m, 2H, Ar), 7.06-7.14 (m, 2H, Ar), 11.11 (br s, 1H), 13.12 (br s, 1H) ppm. ¹³C NMR (75 MHz, CDCl₃): δ 19.8, 29.0, 29.2, 32.5, 33.3, 33.7, 92.2, 115.2, (d, *J* = 21.5 Hz, 2C), 117.5, 128.1 (d, *J* = 7.5 Hz, 2C), 132.8 (d, *J* = 3.2 Hz), 150.7, 161.4 (d, *J* = 243.8 Hz), 162.3, 164.6, 191.7, 193.35 ppm. MS (m/z relative intensity %): 374 [M⁺] (43), 330 (7), 275 (7), 261 (17), 217 (35), 162 (11), 156 (22), 120 (41), 56 (32), 42 (100). IR (KBr): 3400, 2964, 2877, 1704, 1627, 1505, 1169, 1226, 838, 765 cm⁻¹. Anal. calcd for C₁₉H₁₉FN₂O₅: C, 60.96; H, 5.12; F 5.07; N, 7.48. Found C, 60.84; H, 5.07; F 4.98; N, 7.31%.

5-[(4-Chlorophenyl)(2-hydroxy-6-oxocyclohex-1-en-1-yl)methyl]-6-hydroxy-1,3-dimethylpyrimidine-2,4(1H,3H)-dione (3e)

Yield 1.72 g (88%), mp: 180-182 °C. ¹H NMR (300 MHz, DMSO-*d*₆): δ 2.02-2.11 (m, 2H, CH₂), 2.33-2.52 (m, 2H, CH₂), 2.60-2.74 (m, 2H, CH₂), 3.37 (s, 3H, CH₃), 3.46 (s, 3H, CH₃), 5.49 (s, 1H, CH), 7.08 (d, *J* = 7.8 Hz, 2H, Ar), 7.27 (d, *J* = 7.8 Hz, 2H, Ar), 10.15 (br s, 1H), 13.11 (br s, 1H) ppm. ¹³C NMR (75 MHz, CDCl₃): δ 19.8, 29.0, 29.3, 32.5, 33.4, 33.6, 92.0, 117.3, 128.0 (2C), 128.5 (2C), 132.0, 135.8, 150.7, 162.3, 164.6, 191.9, 193.3 ppm. MS (m/z relative intensity %): 392 [M⁺, 9, ³⁷Cl], 390 [M⁺, 24, ³⁵Cl], 336 [1, ³⁷Cl], 334 [2, ³⁵Cl], 308 [2, ³⁷Cl], 306 [4, ³⁵Cl], 293 [3, ³⁷Cl], 291 [6, ³⁵Cl], 279 [9, ³⁷Cl], 277 [11, ³⁵Cl], 235 (18), 199 (39), 136 (27), 115 (15), 58 (31), 42 (100). IR (KBr) ν = 3434, 2982, 2887, 1698, 1607, 1508, 1488, 1352, 1200, 882 cm⁻¹. Anal. calcd for C₁₉H₁₉ClN₂O₅: C, 58.39; H, 4.90; Cl, 9.07; N, 7.17%. Found C, 58.23; H, 4.84; Cl, 8.95; N, 7.03%.

5-[(3-Bromophenyl)(2-hydroxy-6-oxocyclohex-1-en-1-yl)methyl]-6-hydroxy-1,3-dimethylpyrimidine-2,4(1H,3H)-dione (3f)

Yield 1.81 g (83%), mp: 177-179 °C. ¹H NMR (300 MHz, DMSO-*d*₆): δ 2.07-2.10 (m, 2H, CH₂), 2.33-2.52 (m, 2H, CH₂), 2.63-2.73 (m, 2H, CH₂), 3.38 (s, 3H, CH₃), 3.46 (s, 3H, CH₃), 5.51 (s, 1H, CH), 7.07 (d, *J* = 7.8 Hz, 1H, Ar), 7.16 (t, *J* = 7.8 Hz, 1H, Ar), 7.26 (s, 1H, Ar), 7.36 (d, *J* = 7.8 Hz, 1H, Ar), 10.78 (br s, 1H), 13.13 (br s, 1H) ppm. ¹³C NMR (75 MHz, CDCl₃): δ 19.8, 29.0, 29.3, 32.4, 33.6, 33.7, 91.8, 117.1, 122.7, 125.3, 129.4, 129.6, 129.8, 139.9, 150.7, 162.3, 164.6, 191.8, 193.4 ppm. MS (m/z relative intensity %): 436 (M⁺, 14, ⁸¹Br), 434 (M⁺, 14, ⁷⁹Br), 323 (11), 279 (17), 251 (7), 199 (81), 156 (52), 101 (33), 58 (27), 42 (100). IR (KBr): 3480, 3071, 2948, 1694, 1593, 1475, 1395, 1292, 1241, 778 cm⁻¹. Anal. calcd for C₁₉H₁₉BrN₂O₅: C, 52.43; H, 4.40; Br, 18.36; N, 6.44. Found C, 52.31; H, 4.34; Br, 18.27; N, 6.35%.

6-Hydroxy-5-[(2-hydroxy-6-oxocyclohex-1-en-1-yl)(4-nitrophenyl)methyl]-1,3-dimethylpyrimidine-2,4(1H,3H)-dione (3g)

Yield 1.71 g (85%), mp: 180-182 °C. ¹H NMR (300 MHz, CDCl₃): δ 2.04-2.12 (m, 2H, CH₂), 2.36-2.54 (m, 2H, CH₂), 2.63-2.76 (m, 2H, CH₂), 3.37 (s, 3H, CH₃), 3.47 (s, 3H, CH₃), 5.56 (s, 1H, CH), 7.29 (d, *J* = 8.8 Hz, 2H, Ar), 8.16 (d, 2H, *J* = 8.8 Hz, Ar), 10.25 (br s, 1H), 13.05 (br s, 1H) ppm. ¹³C NMR (75 MHz, CDCl₃): δ 19.7, 29.3, 29.5, 32.5, 33.5, 34.2, 91.6, 116.9, 123.7(2C), 127.5(2C), 145.5, 146.4, 150.6, 162.5, 164.5, 192.6, 193.1. MS (m/z relative intensity %): 401 [M⁺] (100), 339 (5), 288 (27), 272 (18), 228 (47), 198 (33), 156 (49), 128 (20), 56 (29),

42 (96). IR (KBr) ν = 3443, 2946, 2884, 1702, 1597, 1514, 1491, 1345, 864, 853, cm^{-1} . Anal. calcd for $\text{C}_{19}\text{H}_{19}\text{N}_3\text{O}_7$: C, 56.86; H, 4.77; N, 10.47. Found C, 56.71; H, 4.64; N, 10.35%.

6-Hydroxy-5-[(2-hydroxy-4,4-dimethyl-6-oxocyclohex-1-en-1-yl)(phenyl)methyl]-1,3-dimethylpyrimidine-2,4(1*H*,3*H*)-dione (3h)

Yield 1.83 g (95%), mp: 185-187 °C (Lit. mp²: 186-188 °C). ¹H NMR (300 MHz, CDCl_3): δ 1.16 (s, 3H, CH_3), 1.30 (s, 3H, CH_3), 2.34-2.55 (m, 4H, 2 CH_2), 3.37 (s, 3H, CH_3), 3.46 (s, 3H, CH_3), 5.59 (s, 1H, CH), 7.14-7.33 (m, 5H, Ar), 10.60 (br s, 1H), 12.85 (br s, 1H) ppm. MS (m/z relative intensity %): 384 [M^+] (100), 320 (1), 283 (2), 243 (19), 227 (75), 171 (11), 156 (22), 102 (50), 56 (44), 42 (81).

6-Hydroxy-5-[(2-hydroxy-4,4-dimethyl-6-oxocyclohex-1-en-1-yl)(4-methylphenyl)methyl]-1,3-dimethylpyrimidine-2,4(1*H*,3*H*)-dione (3i)

Yield 1.79 g (90%), mp: 178-180 °C (Lit. mp²: 176-189 °C). ¹H NMR (300 MHz, CDCl_3): δ 1.15 (s, 3H, CH_3), 1.29 (s, 3H, CH_3), 2.34 (s, 3H, CH_3), 2.43-2.50 (m, 4H, 2 CH_2), 3.37 (s, 3H, CH_3), 3.46 (s, 3H, CH_3), 5.55 (s, 1H, CH), 7.01 (d, J = 8.1 Hz, 2H, Ar), 7.11 (d, J = 8.1 Hz, 2H, Ar), 11.0 (br s, 1H), 12.85 (br s, 1H) ppm. MS (m/z relative intensity %): 398 [M^+] (100), 297 (2), 257 (17), 227 (90), 171 (15), 156 (22), 115 (68), 55 (47), 42 (87).

6-Hydroxy-5-[(2-hydroxy-4,4-dimethyl-6-oxocyclohex-1-en-1-yl)(4-methoxyphenyl)methyl]-1,3-dimethylpyrimidine-2,4(1*H*,3*H*)-dione (3j)

Yield 1.78 g (86%), mp: 140-142 °C. ¹H NMR (300 MHz, CDCl_3): δ 1.15 (s, 3H, CH_3), 1.28 (s, 3H, CH_3), 2.30-2.54 (m, 4H, 2 CH_2), 3.37 (s, 3H, CH_3), 3.45 (s, 3H, CH_3), 3.80 (s, 3H, OCH_3), 5.53 (s, 1H, CH), 7.84 (d, J = 8.6 Hz, 2H, Ar), 7.04 (d, J = 8.6 Hz, 2H, Ar), 10.82 (br s, 1H), 12.83 (br s, 1H) ppm. ¹³C NMR (75 MHz, CDCl_3): δ 27.2, 28.9, 29.2, 30.0, 31.2, 33.0, 46.1, 47.1, 55.2, 92.7, 113.8, 127.7 (2C), 129.0 (2C), 138.0, 150.8, 157.9, 162.4, 164.1, 190.8, 191.3 ppm. MS (m/z relative intensity %): 414 [M^+] (16), 299 (1), 257 (88), 227 (58), 174 (21), 156 (82), 117 (48), 55 (86), 29 (100). IR (KBr) ν = 3437, 2961, 2868, 1701, 1606, 1509, 1389, 1264, 1173, 821 cm^{-1} . Anal. calcd for $\text{C}_{22}\text{H}_{26}\text{N}_2\text{O}_5$: C, 63.76; H, 6.32; N, 6.76. Found C, 63.68; H, 6.26; N, 6.63%.

5-[(4-Fluorophenyl)(2-hydroxy-4,4-dimethyl-6-oxocyclohex-1-en-1-yl)methyl]-6-hydroxy-1,3-dimethylpyrimidine-2,4(1*H*,3*H*)-dione (3k)

Yield 1.75 g (87%), mp: 150-152 °C. ¹H NMR (300 MHz, CDCl₃) δ 1.25 (s, 3H, CH₃), 1.28 (s, 3H, CH₃), 2.31-2.55 (m, 4H, 2CH₂), 3.37 (s, 3H, CH₃), 3.45 (s, 3H, CH₃), 5.53 (s, 1H, CH), 6.96-7.12 (m, 4H, Ar), 10.31 (br s, 1H), 12.82 (br s, 1H) ppm. ¹³C NMR (75 MHz, CDCl₃): δ 27.2, 28.9, 29.2, 30.0, 31.3, 33.2, 46.2, 47.1, 115.7, (d, *J*=21.2 Hz, 2C), 116.4, 128.2 (d, *J* = 7.8 Hz, 2C), 132.9 (d, *J* = 3.2 Hz), 150.8, 161.3 (d, *J* = 243.0 Hz), 162.5, 164.2 (2C), 191.2 (2C). MS (m/z relative intensity %): 402 [M⁺] (11), 261 (10), 245 (39), 191(11), 134 (43), 120(68), 56 (74), 42 (100). IR (KBr) ν = 3400, 2964, 2877, 1704, 1627, 1506, 1369, 1226, 838, 765 cm⁻¹. Anal. calcd for C₂₁H₂₃FN₂O₅: C, 62.68; H, 5.76; F, 4.72; N, 6.96. Found C, 62.53; H, 5.72; F, 4.57; N, 6.79%.

5-[(4-Chlorophenyl)(2-hydroxy-4,4-dimethyl-6-oxocyclohex-1-en-1-yl)methyl]-6-hydroxy-1,3-dimethylpyrimidine-2,4(1*H*,3*H*)-dione (3l)

Yield 1.86 g (89%), mp: 173-175 °C (Lit. mp²: 176 °C). ¹H NMR (300 MHz, CDCl₃): δ 1.15 (s, 3H, CH₃), 1.28 (s, 3H, CH₃), 2.33-2.52 (m, 4H, 2CH₂), 3.37 (s, 3H, CH₃), 3.46 (s, 3H, CH₃), 5.52 (s, 1H, CH), 7.07 (d, *J* = 8.1 Hz, 2H, Ar), 7.27 (d, *J* = 8.1 Hz, 2H, Ar), 10.80 (br s, 1H), 12.81 (br s, 1H) ppm. MS (m/z relative intensity %): 420 [M⁺, 20, ³⁷Cl], 418 [M⁺, 58, ³⁵Cl], 404 [3, ³⁷Cl], 402 [7, ³⁵Cl], 305 [1, ³⁷Cl], 303 [2, ³⁵Cl], 264 [28, ³⁷Cl], 262 [75, ³⁵Cl], 227 (100), 178 (16), 156 (44), 136 (52), 56 (50), 15 (98).

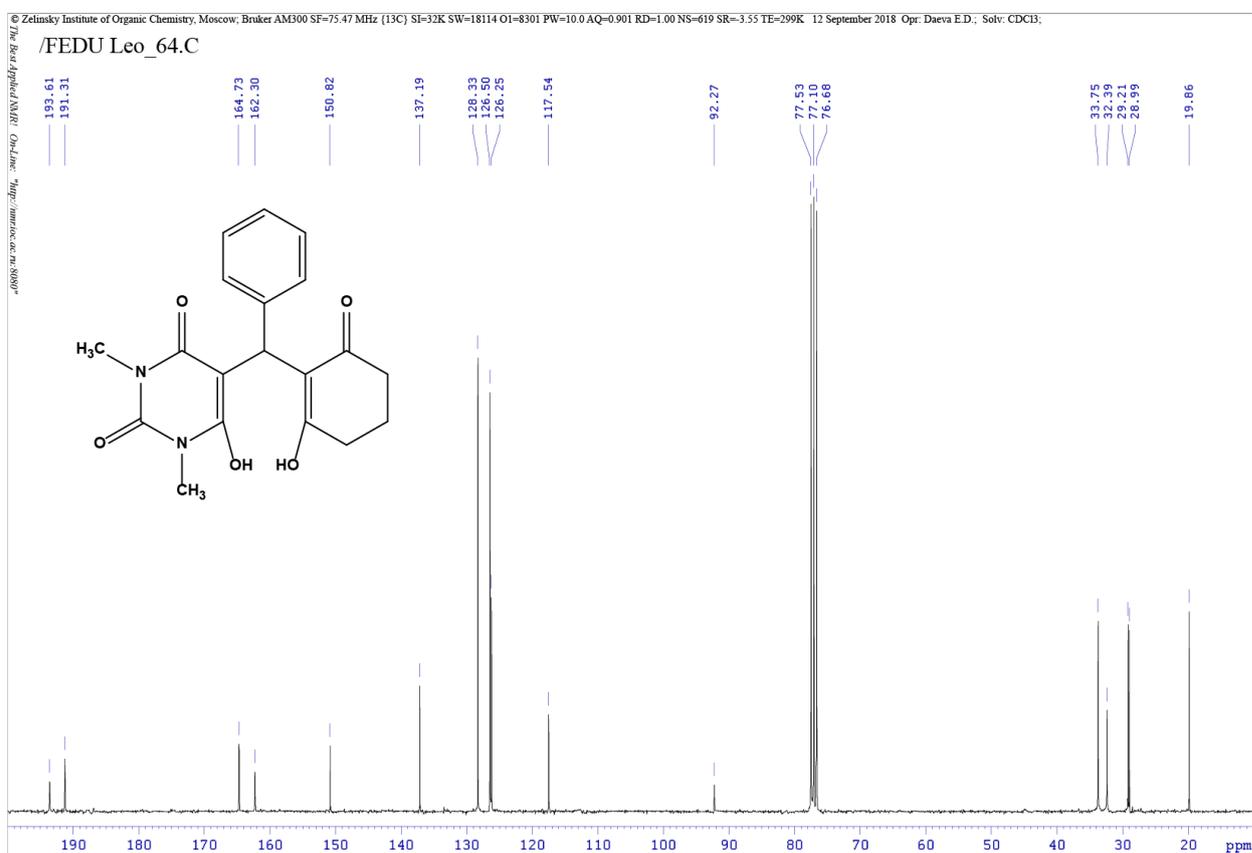
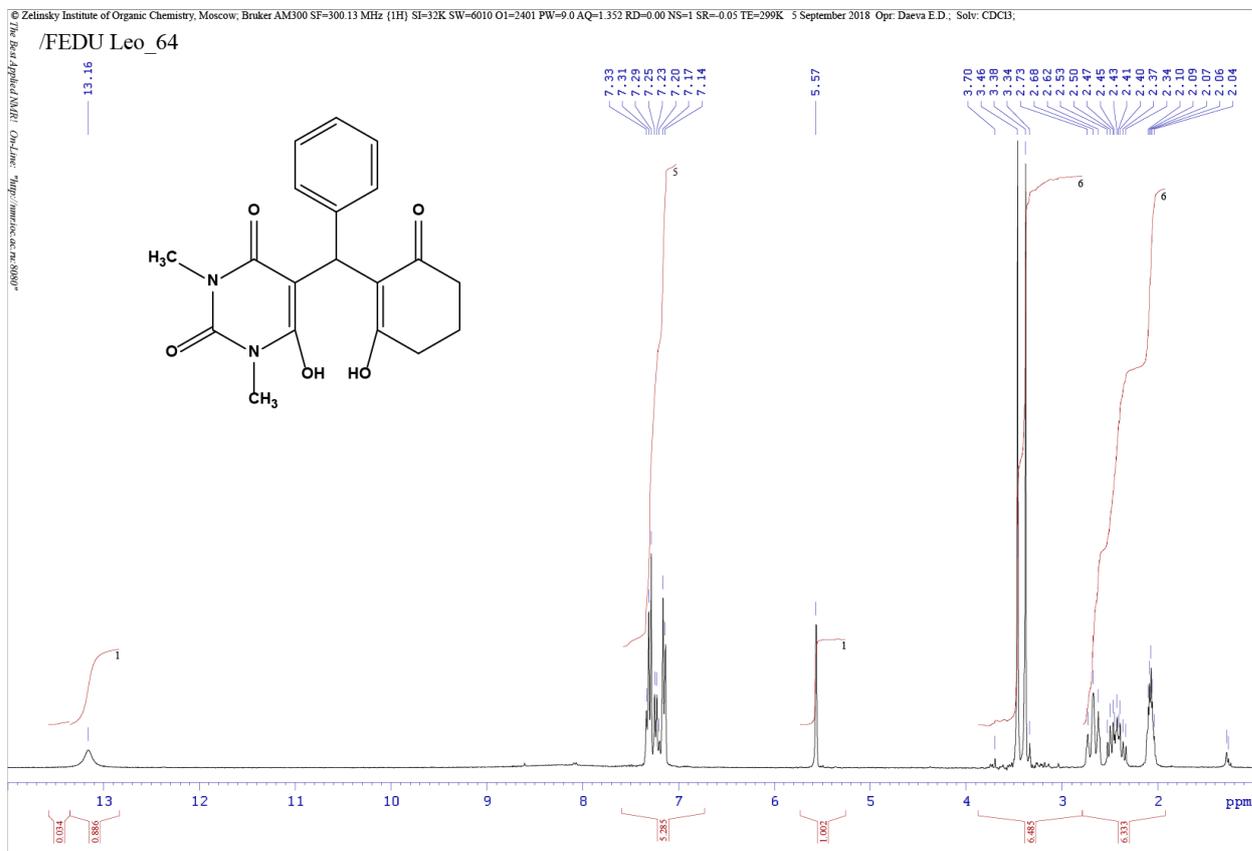
5-[(3-Bromophenyl)(2-hydroxy-4,4-dimethyl-6-oxocyclohex-1-en-1-yl)methyl]-6-hydroxy-1,3-dimethylpyrimidine-2,4(1*H*,3*H*)-dione (3m)

Yield 2.06 g (89%), mp: 170-172 °C. ¹H NMR (300 MHz, CDCl₃): δ 1.15 (s, 3H, CH₃), 1.29 (s, 3H, CH₃), 2.31-2.56 (m, 4H, 2CH₂), 3.38 (s, 3H, CH₃), 3.46 (s, 3H, CH₃), 5.53 (s, 1H, CH), 7.07 (d, *J* = 7.8 Hz, 1H, Ar), 7.17 (t, *J* = 7.8 Hz, 1H, Ar), 7.27 (s, 1H, Ar), 7.36 (d, *J*=7.8 Hz, 1H, Ar), 10.65 (br s, 1H), 12.85 (br s, 1H) ppm. ¹³C NMR (75 MHz, CDCl₃): δ 27.2, 29.0, 29.3, 30.0, 31.3, 33.6, 46.1, 47.1, 92.0, 116.1, 122.7, 125.4, 129.4, 129.9 (2C), 140.0, 150.7, 162.4, 164.3, 191.0, 191.6. MS (m/z relative intensity %): 464 (M⁺, 34, ⁸¹Br), 462 (M⁺, 34, ⁷⁹Br), 376 (1), 337 (4), 307 (27), 251 (8), 227 (59), 156 (30), 115 (30), 101 (30), 42 (100). IR (KBr) ν = 3436, 2958, 2881, 2345, 1709, 1609, 1471, 1368, 776, 753. Anal. calcd for C₂₁H₂₃BrN₂O₅: C, 54.44; H, 5.00; Br, 17.25; N, 6.05. Found C, 54.31; H, 4.85; Br, 17.09; N, 5.93%.

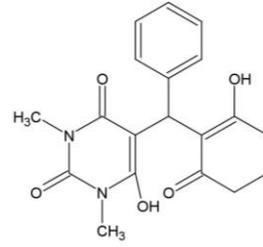
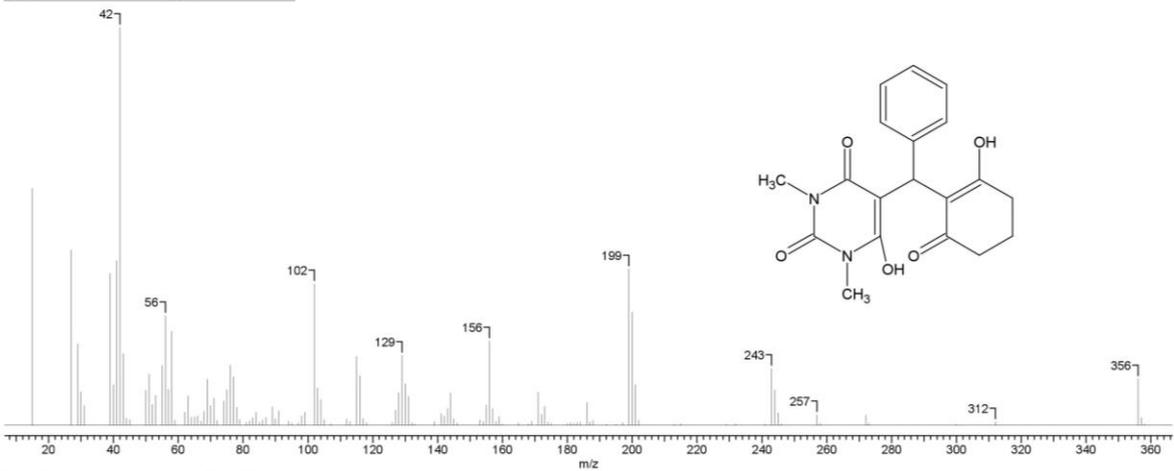
6-Hydroxy-5-[(2-hydroxy-4,4-dimethyl-6-oxocyclohex-1-en-1-yl)(4-nitrophenyl)methyl]-1,3-dimethylpyrimidine-2,4(1H,3H)-dione (3n)

Yield 2.10 g (98%), mp: 155-158 °C. ¹H NMR (300 MHz, CDCl₃): δ 1.17 (s, 3H, CH₃), 1.29 (s, 3H, CH₃), 2.34-2.58 (m, 4H, 2CH₂), 3.37 (s, 3H, CH₃), 3.47s, 3H, CH₃), 5.59 (s, 1H, CH), 7.31 (d, *J* = 8.8 Hz, 2H, Ar), 8.17 (d, *J* = 8.8 Hz, 2H, Ar), 10.85 (br s, 1H), 12.77 (br s, 1H) ppm. ¹³C NMR (75 MHz, CDCl₃): δ 27.3, 29.0, 29.4, 30.0, 31.3, 34.2, 46.3, 46.9, 91.9, 115.9, 123.7 (2C), 127.7 (2C), 145.7, 146.5, 150.6, 162.6, 164.2, 191.0, 192.1. MS (m/z relative intensity %): 429 [M⁺] (8), 317 (1), 288 (14), 256 (36), 189 (4), 156 (27), 101 (21), 75 (20), 42 (100). IR (KBr) ν = 3430, 2963, 2881, 1703, 1609, 1392, 1345, 1265, 853, 750 cm⁻¹.

1. A. Barakat, S. M. Soliman, Y. A. M. M. Elshaier, M. Ali, A. M. Al-Majid and J. A. Ghabbour, *J. Mol. Struct.* 2017, **1134**, 99.
2. M. A. Bigdeli, E. Sheikhhosseini, A. Habibi and S. Balalaieb, *Heterocycles*, 2011, **83**, 107.

^1H and ^{13}C NMR, MS and IR spectra for new compounds**6-Hydroxy-5-[(2-hydroxy-6-oxocyclohex-1-en-1-yl)(phenyl)methyl]-1,3-dimethylpyrimidine-2,4(1*H*,3*H*)-dione (3a)**

| | | | | | | | |
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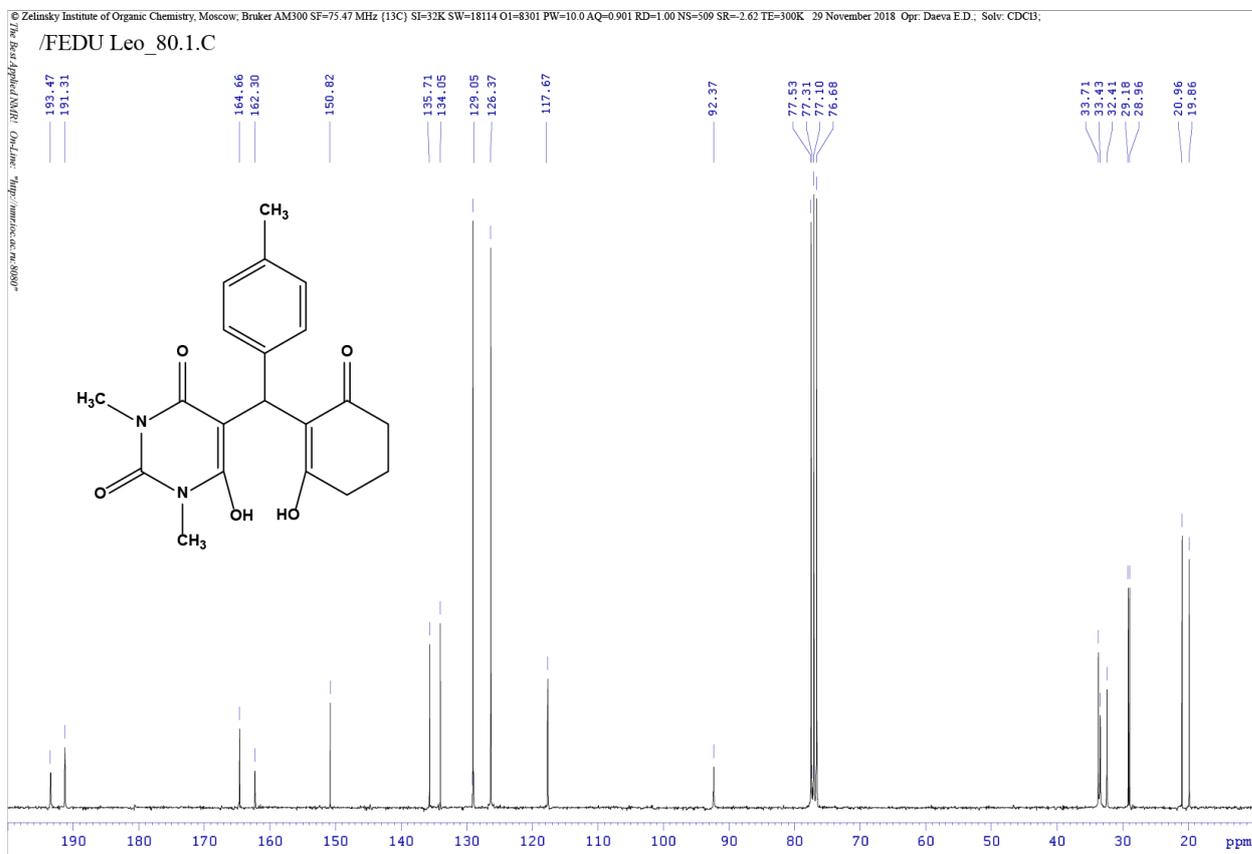
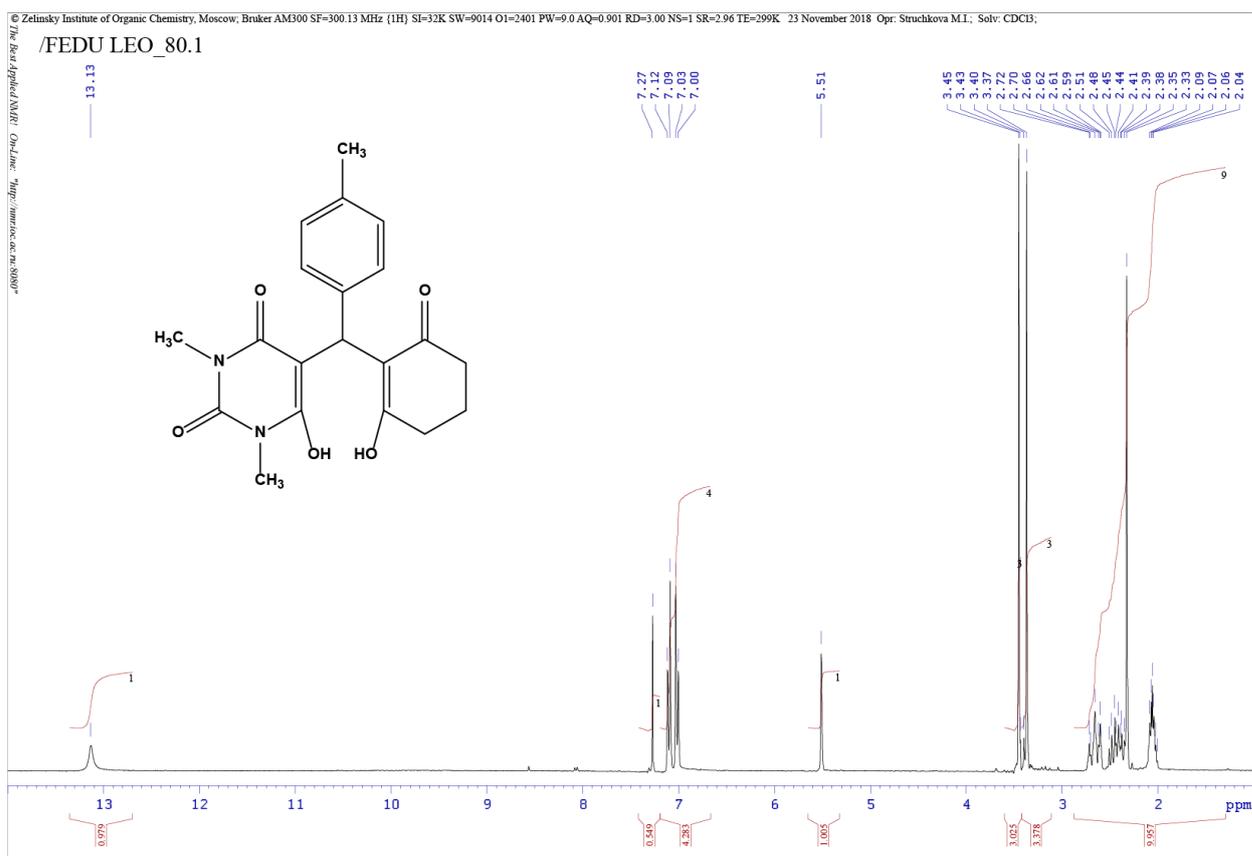
| No. | m/z | RI(%) | DI | No. | m/z | TIC(%) |
|-----|---------|---------|---------|-----|---------|--------|
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| 2 | 56.000 | 27.543 | 27.543 | 2 | 56.000 | 2.932 |
| 3 | 102.000 | 35.534 | 35.534 | 3 | 102.000 | 3.783 |
| 4 | 129.000 | 17.612 | 17.612 | 4 | 129.000 | 1.875 |
| 5 | 156.000 | 21.222 | 21.222 | 5 | 156.000 | 2.259 |
| 6 | 199.000 | 39.274 | 39.274 | 6 | 199.000 | 4.181 |
| 7 | 243.000 | 14.311 | 14.311 | 7 | 243.000 | 1.523 |
| 8 | 257.000 | 2.640 | 2.640 | 8 | 257.000 | 0.281 |
| 9 | 312.000 | 0.870 | 0.870 | 9 | 312.000 | 0.093 |
| 10 | 356.000 | 11.651 | 11.651 | 10 | 356.000 | 1.240 |



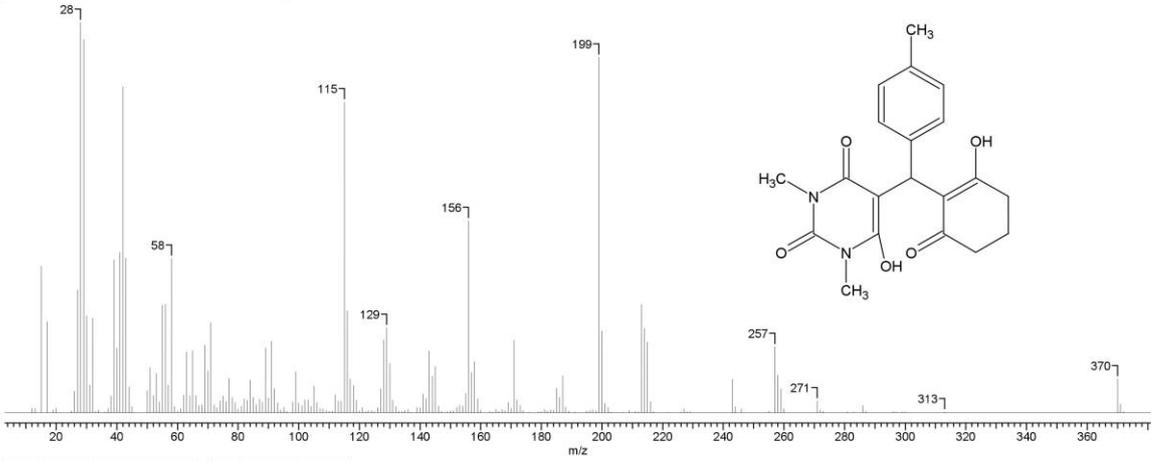
D:\ir\Анисина Leo-64 0 Анисина Leo-64 KBr прессовка

28.09.2018

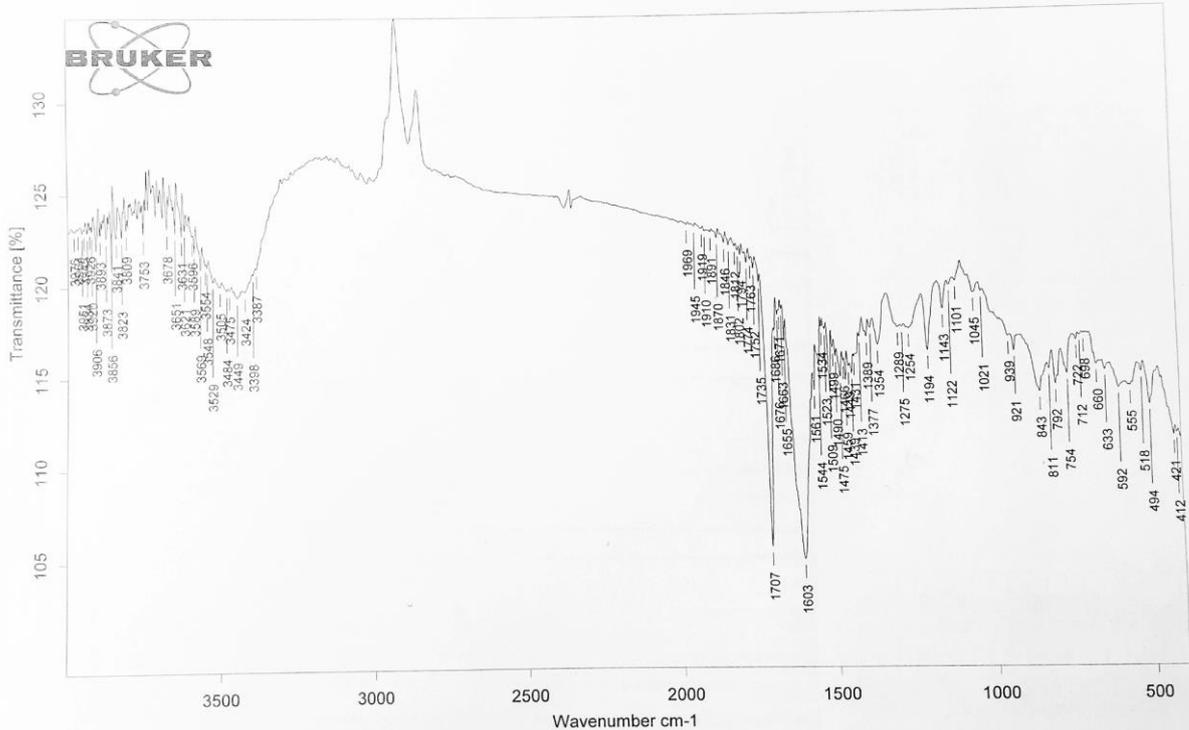
6-Hydroxy-5-[(2-hydroxy-6-oxocyclohex-1-en-1-yl)(4-methylphenyl)methyl]-1,3-dimethylpyrimidine-2,4(1*H*,3*H*)-dione (3b)



| | | | | | | | |
|-----------|---|-------------|-------------------------------|-------------------------|----------------------|----------------|----------------------|
| Count | 205 | Data Type | MASS SPECTRUM | Date | 24 Oct 2019 14:07:28 | Date Stamp | 03 Dec 1918 18:09:00 |
| File Name | C:\Users\Yuliya\AppData\Local\Temp\RaRa0.115\89 801 95.JC | Inlet Model | DIRECT | Instrumental Parameters | LOW RESOLUTION | Spectrum Title | MASS SPECTRUM |
| Origin | LEO-80.1 | Owner | Copyright(C) by Victor (2018) | | | | |
| TIC | 1639.13 | | | | | | |

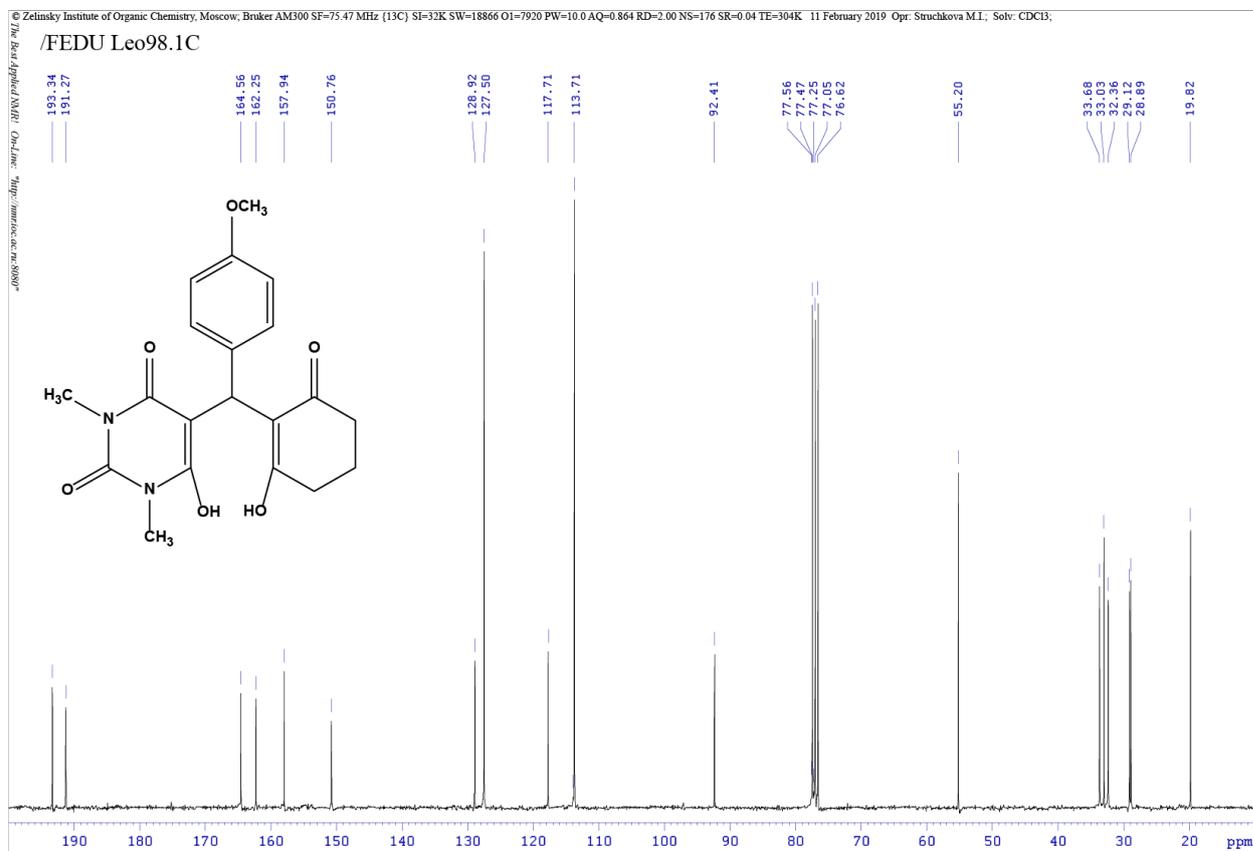
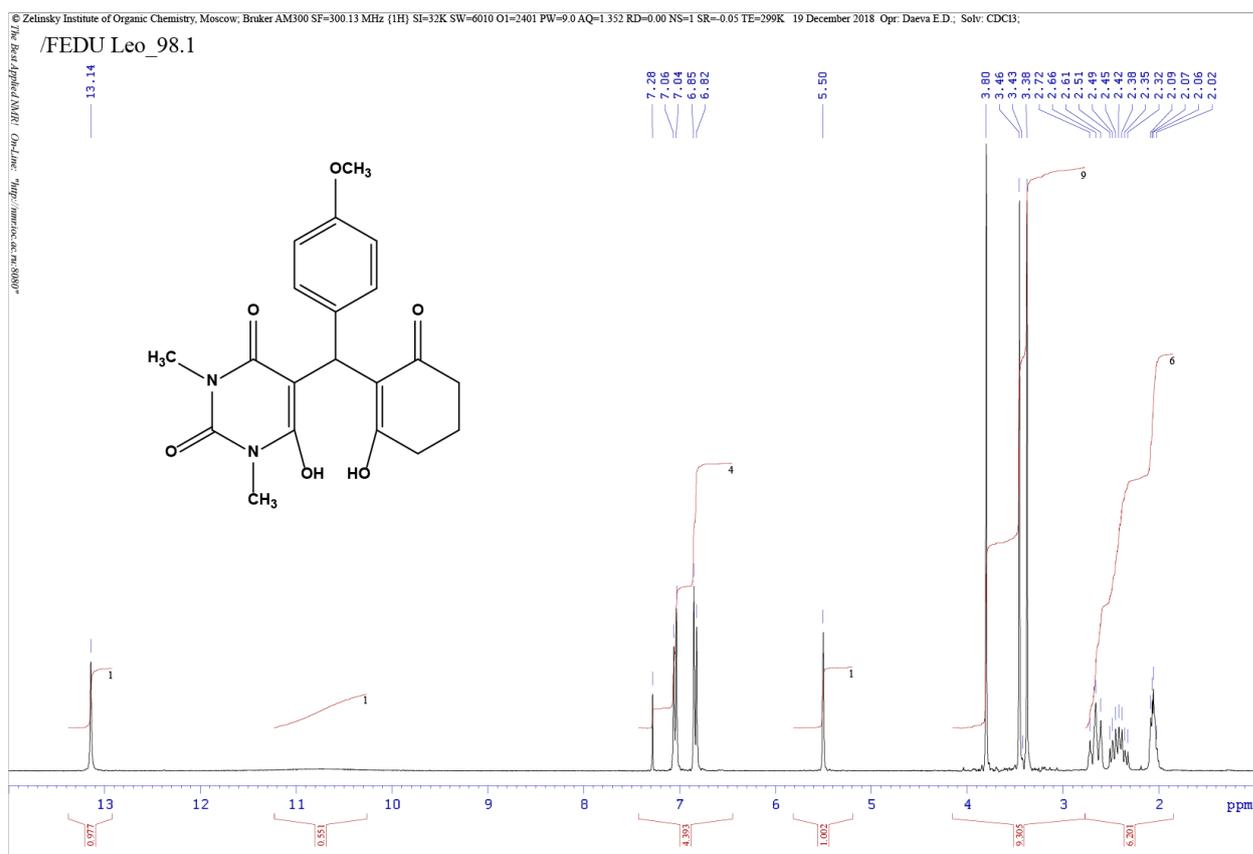


| No. | m/z | RI(%) | DI | No. | m/z | TIC(%) |
|-----|---------|---------|---------|-----|---------|--------|
| 1 | 28.000 | 100.000 | 100.000 | 1 | 28.000 | 6.101 |
| 2 | 58.000 | 39.594 | 39.594 | 2 | 58.000 | 2.416 |
| 3 | 115.000 | 79.618 | 79.618 | 3 | 115.000 | 4.857 |
| 4 | 129.000 | 21.812 | 21.812 | 4 | 129.000 | 1.331 |
| 5 | 156.000 | 49.225 | 49.225 | 5 | 156.000 | 3.003 |
| 6 | 199.000 | 91.039 | 91.039 | 6 | 199.000 | 5.554 |
| 7 | 257.000 | 16.932 | 16.932 | 7 | 257.000 | 1.033 |
| 8 | 271.000 | 3.030 | 3.030 | 8 | 271.000 | 0.185 |
| 9 | 313.000 | 0.190 | 0.190 | 9 | 313.000 | 0.012 |
| 10 | 370.000 | 8.711 | 8.711 | 10 | 370.000 | 0.531 |

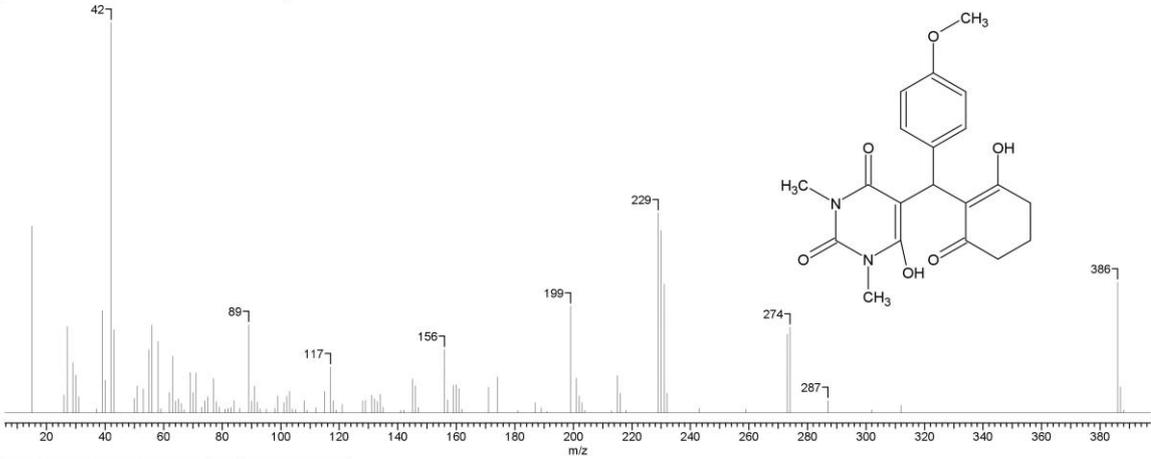


| | | | |
|-------------------------|------------------|---------------|------------|
| D:\r\В\Леонаво Лео_80.1 | Леонаво Лео_80.1 | КBr прессовка | 18.02.2019 |
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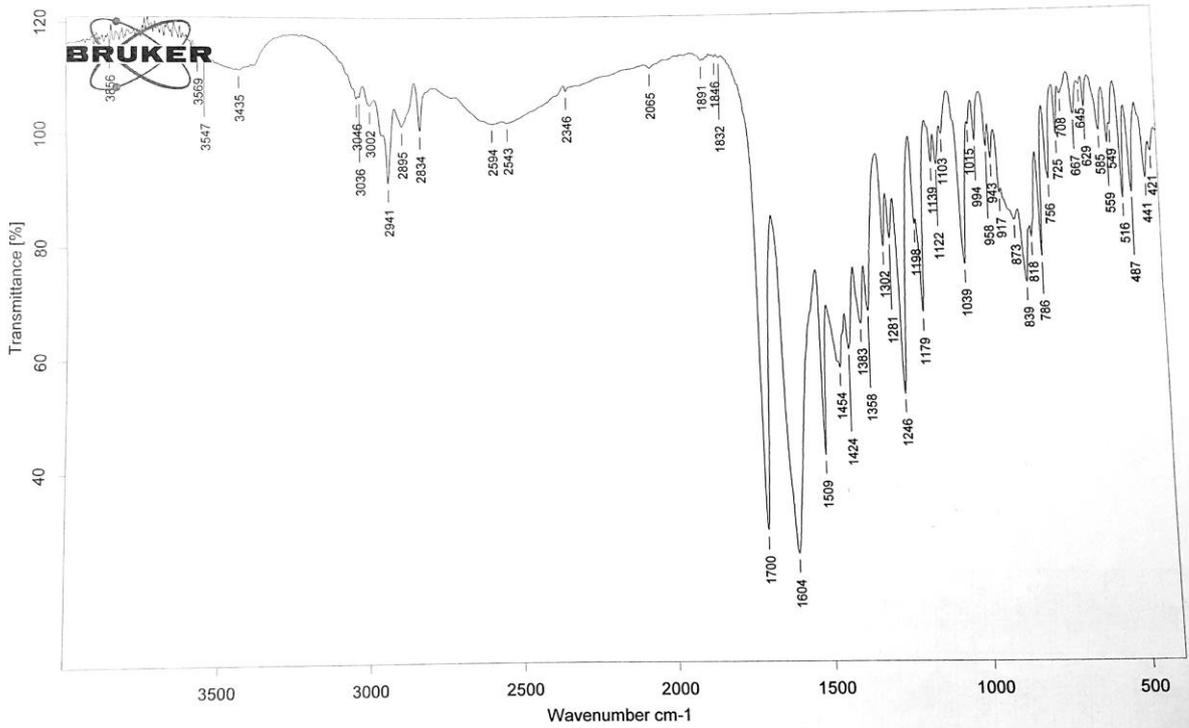
6-Hydroxy-5-[(2-hydroxy-6-oxocyclohex-1-en-1-yl)(4-methoxyphenyl)methyl]-1,3-dimethylpyrimidine-2,4(1H,3H)-dione (3c)



| | | | | | | | |
|-----------|---|-------------|-------------------------------|-------------------------|----------------------|----------------|----------------------|
| Count | 106 | Data Type | MASS SPECTRUM | Date | 24 Oct 2019 14:07:28 | Date Stamp | 04 Feb 1919 17:46:38 |
| File Name | C:\Users\Yulya\AppData\Local\Temp\Ra0.115\98.1.JC | Inlet Model | DIRECT | Instrumental Parameters | LOW RESOLUTION | Spectrum Title | MASS SPECTRUM |
| Origin | LEO-98-1 | Owner | Copyright(C) by Victor (2019) | | | | |
| TIC | 893.53 | | | | | | |

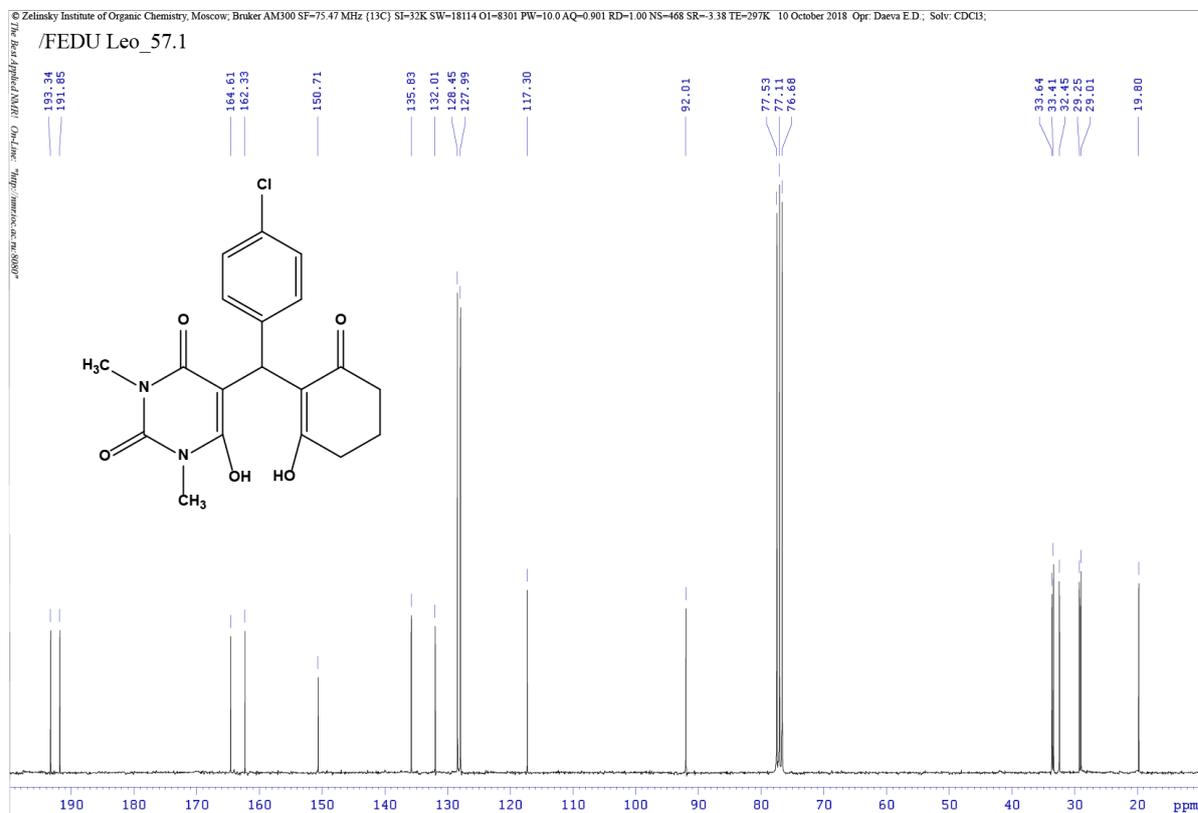
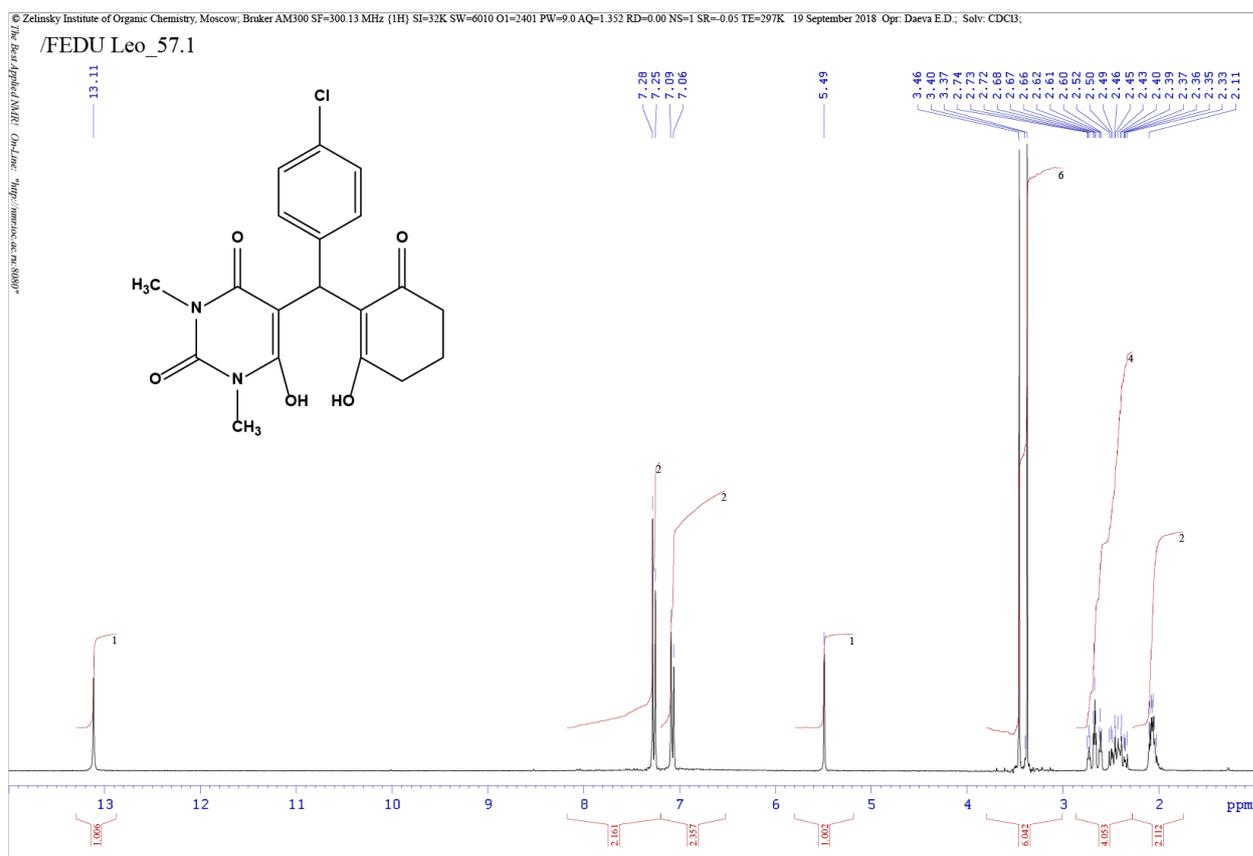


| No. | m/z | RI(%) | DI | No. | m/z | TIC(%) |
|-----|---------|---------|---------|-----|---------|--------|
| 1 | 42.000 | 100.000 | 100.000 | 1 | 42.000 | 11.192 |
| 2 | 89.000 | 22.602 | 22.602 | 2 | 89.000 | 2.530 |
| 3 | 117.000 | 11.781 | 11.781 | 3 | 117.000 | 1.318 |
| 4 | 156.000 | 16.212 | 16.212 | 4 | 156.000 | 1.814 |
| 5 | 199.000 | 27.293 | 27.293 | 5 | 199.000 | 3.054 |
| 6 | 229.000 | 51.195 | 51.195 | 6 | 229.000 | 5.730 |
| 7 | 274.000 | 21.932 | 21.932 | 7 | 274.000 | 2.455 |
| 8 | 287.000 | 3.090 | 3.090 | 8 | 287.000 | 0.346 |
| 9 | 386.000 | 33.453 | 33.453 | 9 | 386.000 | 3.744 |

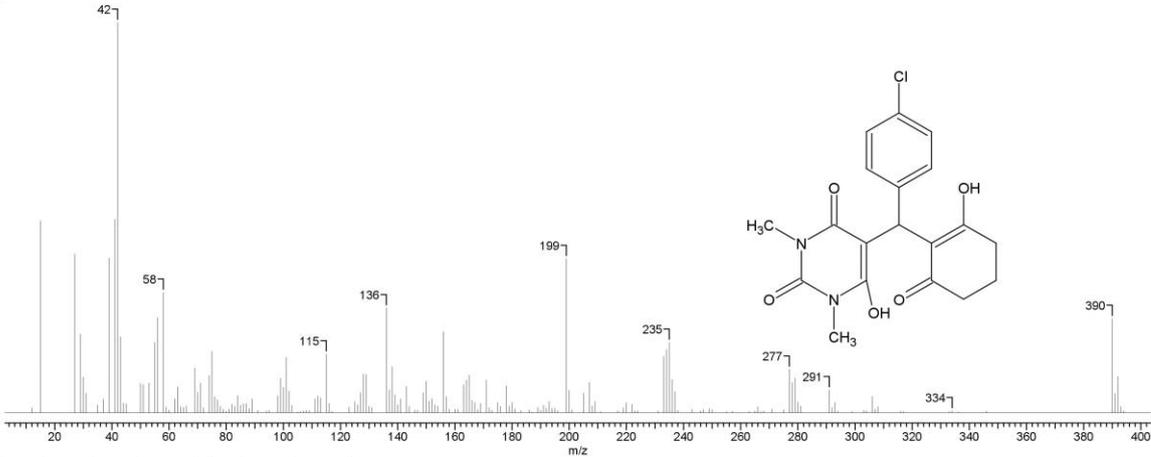


| | | | |
|------------------------|------------------|---------------|------------|
| D:\rB\Уеонова_Leo_98.1 | Уеонова_Leo_98.1 | KBr прессовка | 18.02.2019 |
|------------------------|------------------|---------------|------------|

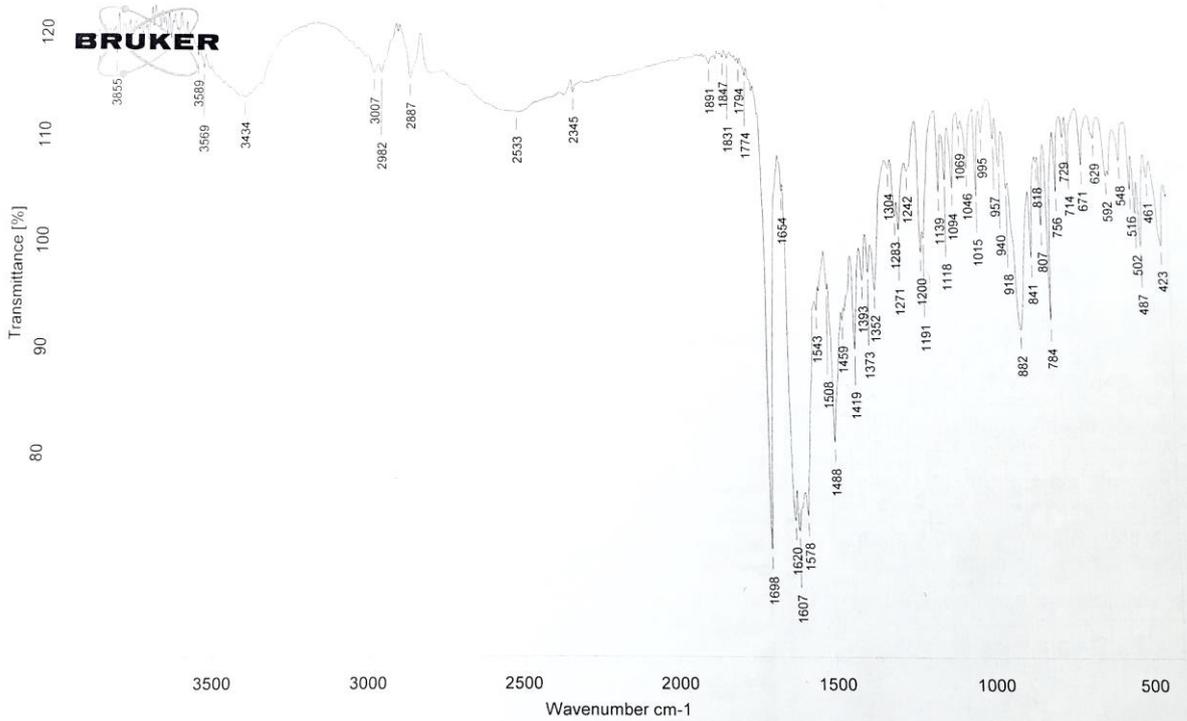
5-[(4-Chlorophenyl)(2-hydroxy-6-oxocyclohex-1-en-1-yl)methyl]-6-hydroxy-1,3-dimethylpyrimidine-2,4(1*H*,3*H*)-dione (3e)



| | | | | | | | |
|-----------|--|-------------|-------------------------------|-------------------------|----------------------|----------------|----------------------|
| Count | 184 | Data Type | MASS SPECTRUM | Date | 24 Oct 2019 14:07:28 | Date Stamp | 26 Sep 1918 18:16:27 |
| File Name | C:\Users\Yuliya\AppData\Local\Temp\Ra0.115\571 63 64 66.JC | Inlet Model | DIRECT | Instrumental Parameters | LOW RESOLUTION | Spectrum Title | MASS SPECTRUM |
| Origin | LEO_57.1 | Owner | Copyright(C) by Victor (2018) | | | | |
| TIC | 1079.33 | | | | | | |

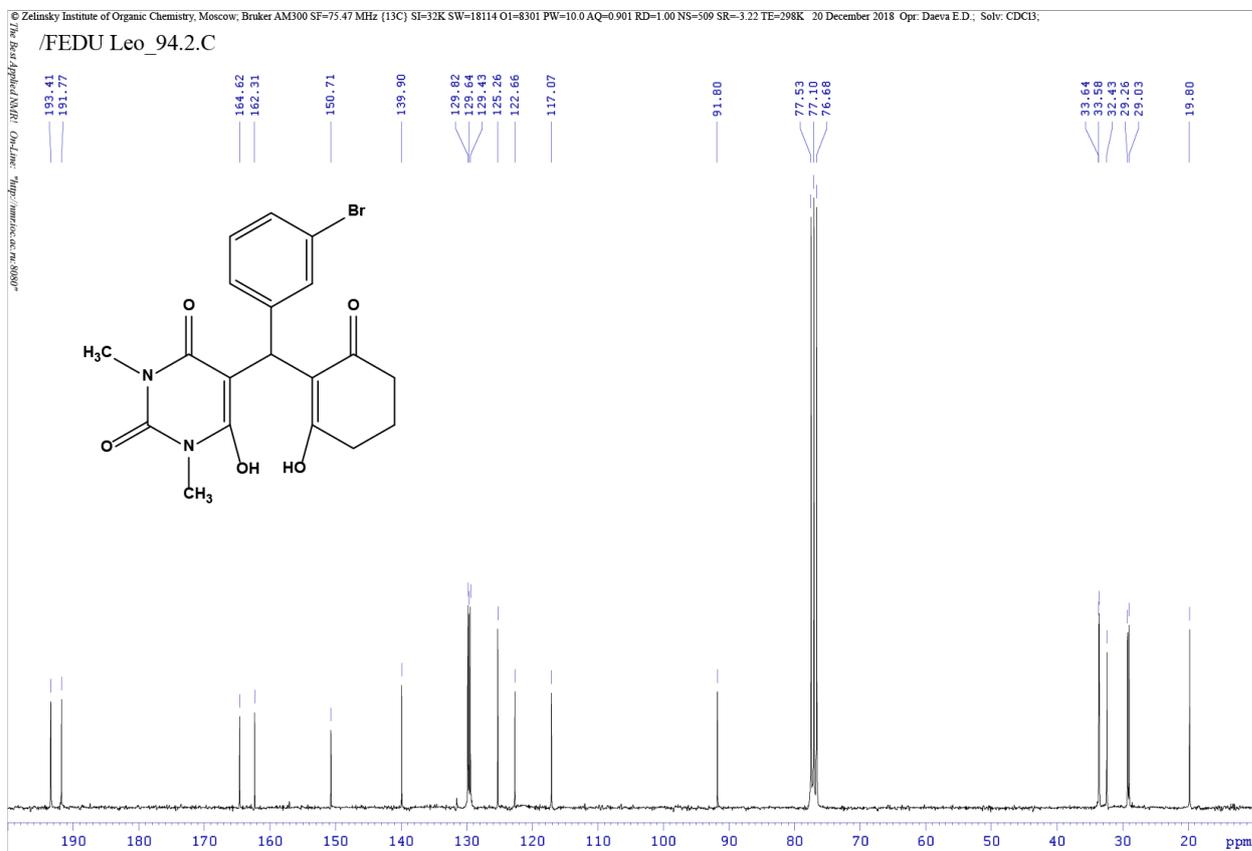
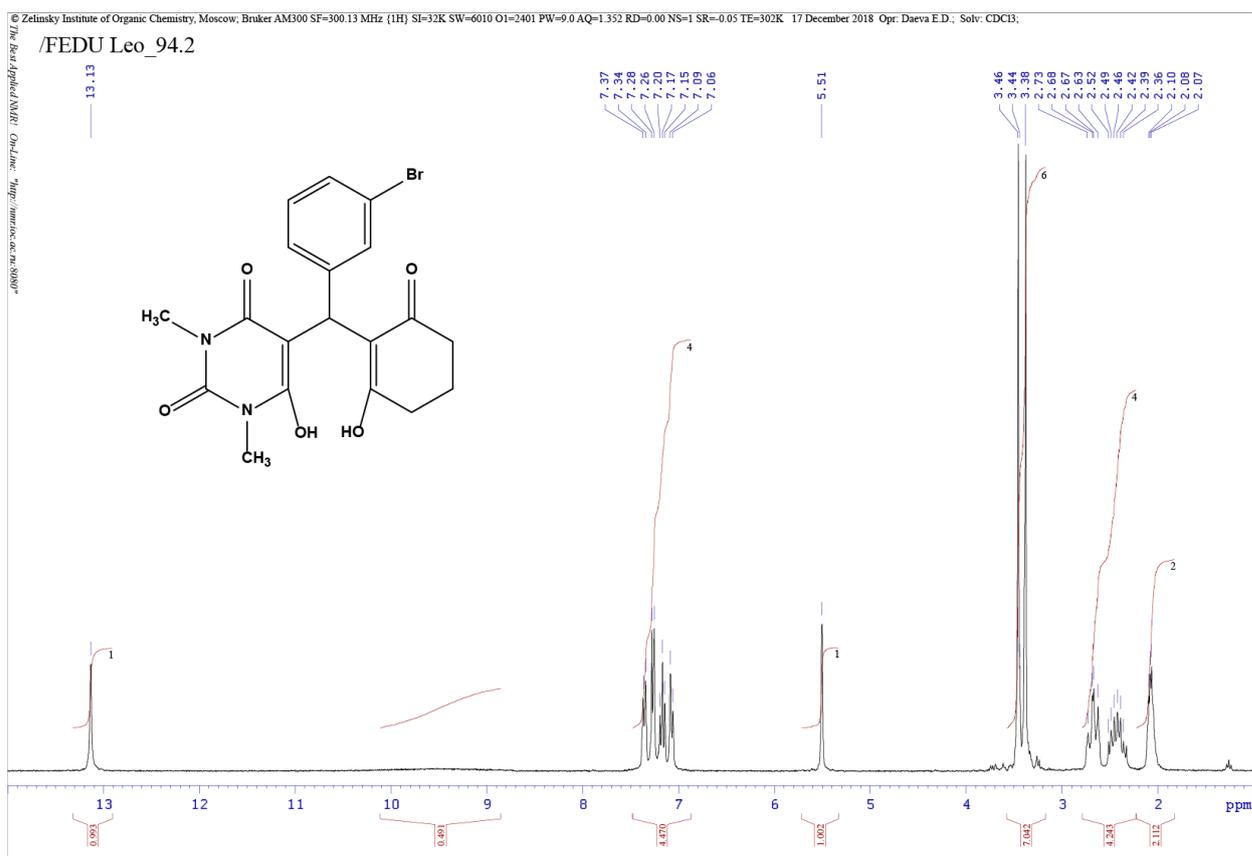


| No. | m/z | RI(%) | DI | No. | m/z | TIC(%) |
|-----|---------|---------|---------|-----|---------|--------|
| 1 | 42.000 | 100.000 | 100.000 | 1 | 42.000 | 9.265 |
| 2 | 58.000 | 30.803 | 30.803 | 2 | 58.000 | 2.854 |
| 3 | 115.000 | 15.032 | 15.032 | 3 | 115.000 | 1.393 |
| 4 | 136.000 | 26.873 | 26.873 | 4 | 136.000 | 2.490 |
| 5 | 199.000 | 39.494 | 39.494 | 5 | 199.000 | 3.659 |
| 6 | 235.000 | 18.062 | 18.062 | 6 | 235.000 | 1.673 |
| 7 | 277.000 | 11.101 | 11.101 | 7 | 277.000 | 1.029 |
| 8 | 291.000 | 5.851 | 5.851 | 8 | 291.000 | 0.542 |
| 9 | 334.000 | 0.540 | 0.540 | 9 | 334.000 | 0.050 |
| 10 | 390.000 | 24.082 | 24.082 | 10 | 390.000 | 2.231 |

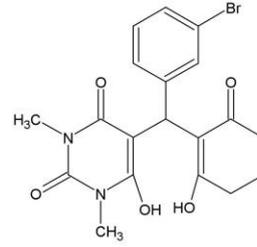
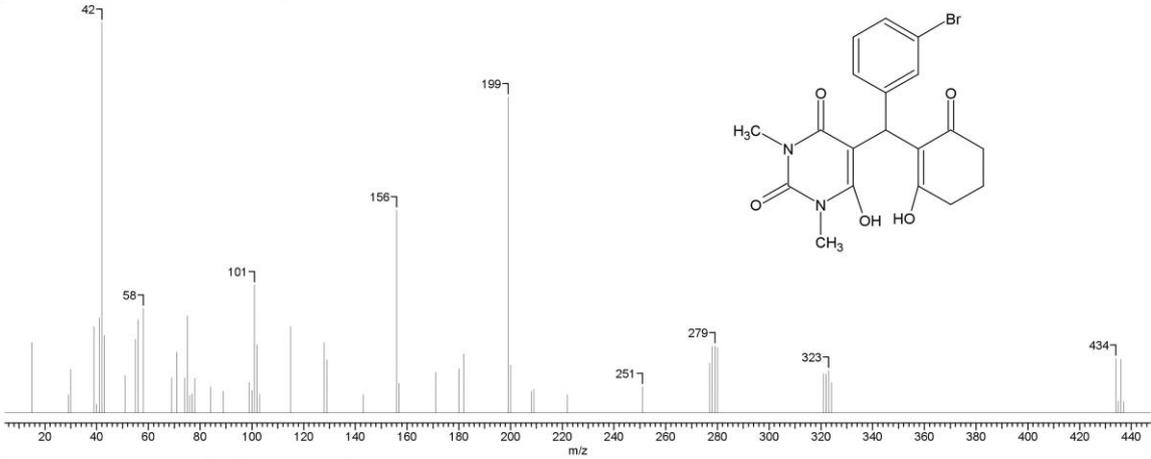


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|----------------------------|------------------|---------------|------------|
| D:\ir\В\Анисина Leo-57.1.0 | Анисина Leo-57,1 | KBr прессовка | 28.09.2018 |
|----------------------------|------------------|---------------|------------|

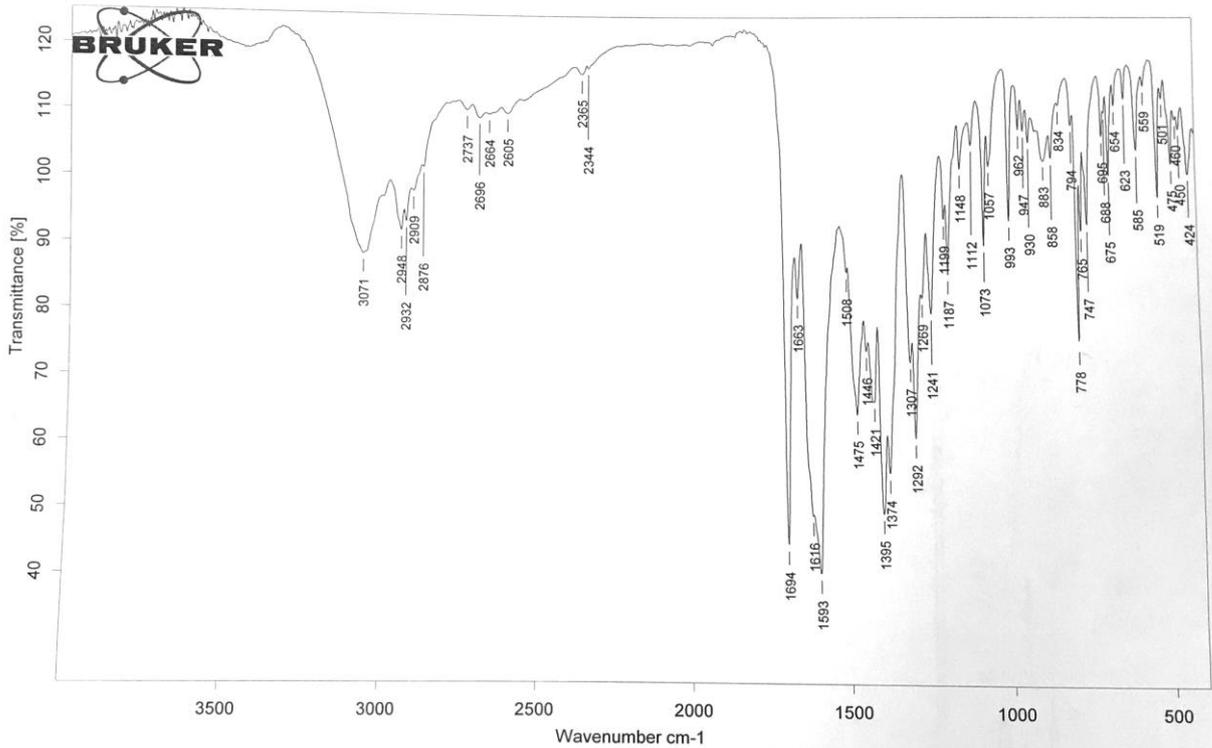
5-[(3-Bromophenyl)(2-hydroxy-6-oxocyclohex-1-en-1-yl)methyl]-6-hydroxy-1,3-dimethylpyrimidine-2,4(1H,3H)-dione (3f)



| | | | | | | | |
|-----------|--|-------------|-------------------------------|-------------------------|----------------------|----------------|----------------------|
| Count | 53 | Data Type | MASS SPECTRUM | Date | 24 Oct 2019 14:07:28 | Date Stamp | 27 Dec 1918 16:27:05 |
| File Name | C:\Users\Yuliya\AppData\Local\Temp\Ra0.278\94.JC | Inlet Model | DIRECT | Instrumental Parameters | LOW RESOLUTION | Spectrum Title | MASS SPECTRUM |
| Origin | LEO 94.2 | Owner | Copyright(C) by Victor (2018) | | | | |
| TIC | 845,27 | | | | | | |

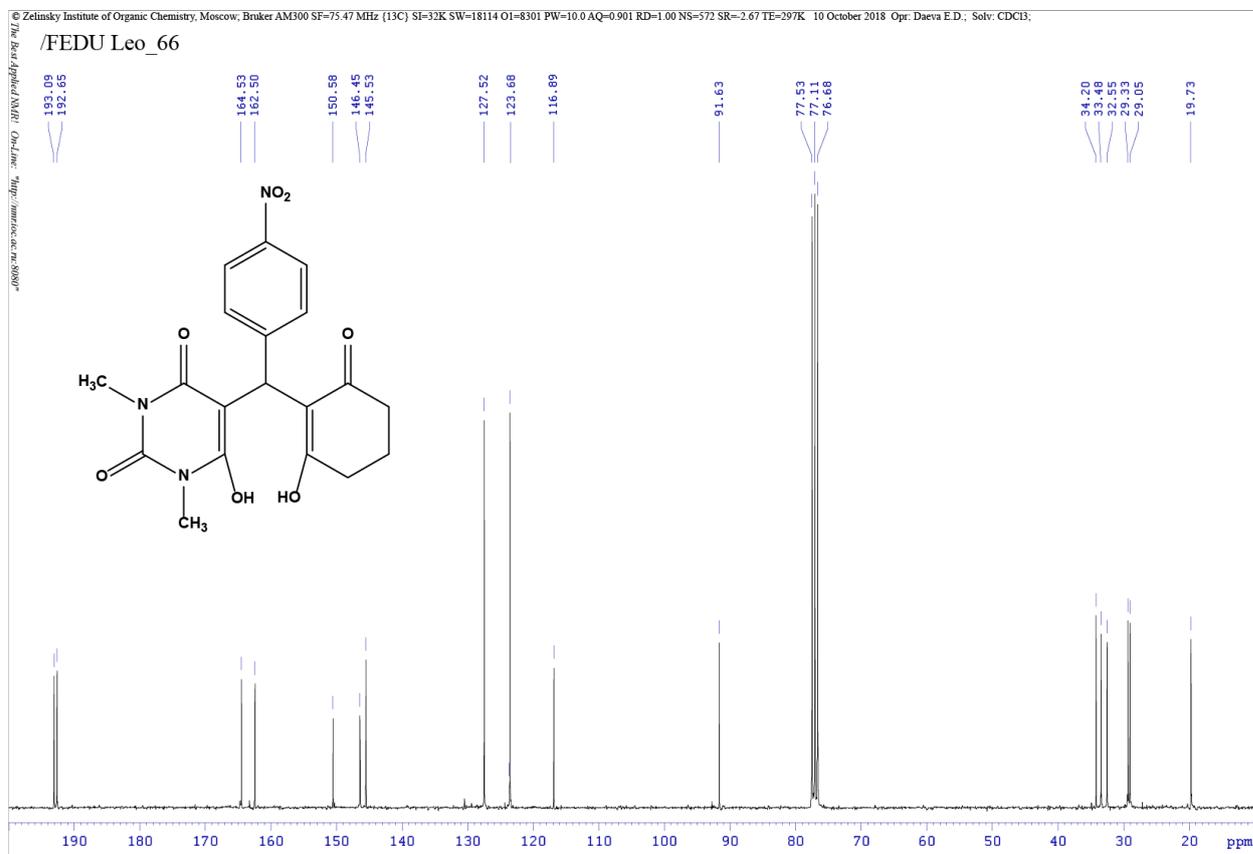
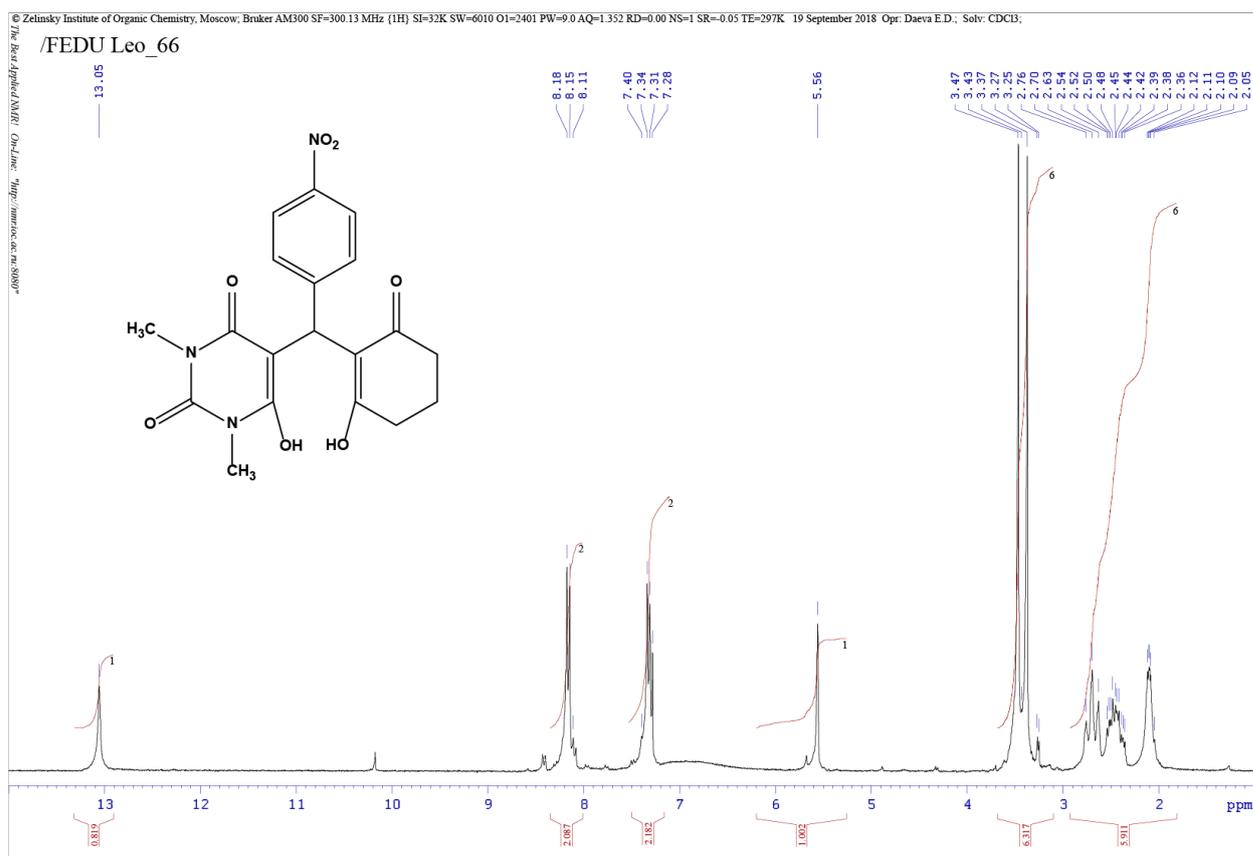


| No. | m/z | RI(%) | DI | No. | m/z | TIC(%) |
|-----|---------|---------|---------|-----|---------|--------|
| 1 | 42.000 | 100.000 | 100.000 | 1 | 42.000 | 11.830 |
| 2 | 58.000 | 26.753 | 26.753 | 2 | 58.000 | 3.165 |
| 3 | 101.000 | 32.753 | 32.753 | 3 | 101.000 | 3.875 |
| 4 | 156.000 | 51.925 | 51.925 | 4 | 156.000 | 6.143 |
| 5 | 199.000 | 80.918 | 80.918 | 5 | 199.000 | 9.573 |
| 6 | 251.000 | 6.681 | 6.681 | 6 | 251.000 | 0.790 |
| 7 | 279.000 | 17.102 | 17.102 | 7 | 279.000 | 2.023 |
| 8 | 323.000 | 10.831 | 10.831 | 8 | 323.000 | 1.281 |
| 9 | 434.000 | 13.901 | 13.901 | 9 | 434.000 | 1.645 |

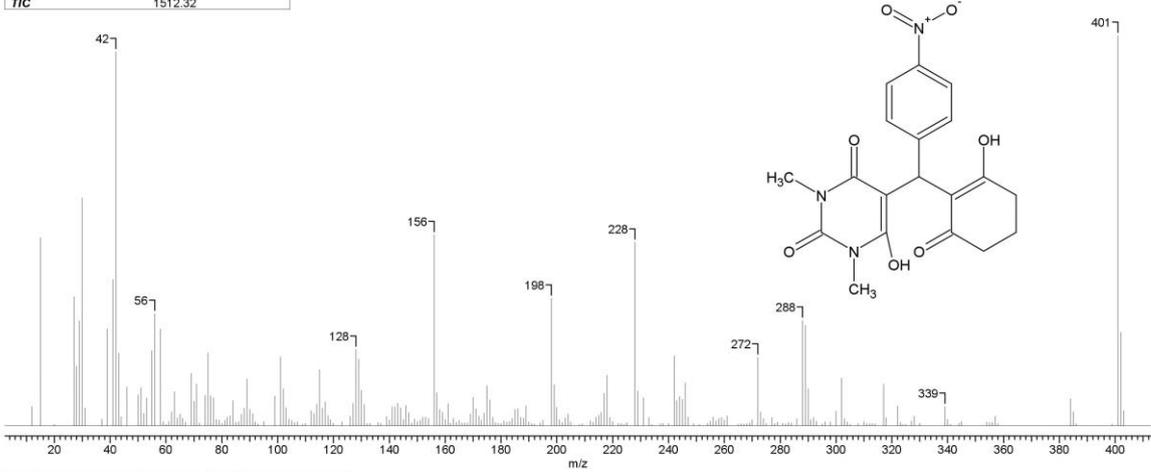


| | | | |
|------------------------|------------------|---------------|------------|
| D:\rB\Леонава Leo_94.2 | Леонава Leo_94.2 | КВr прессовка | 23.05.2019 |
|------------------------|------------------|---------------|------------|

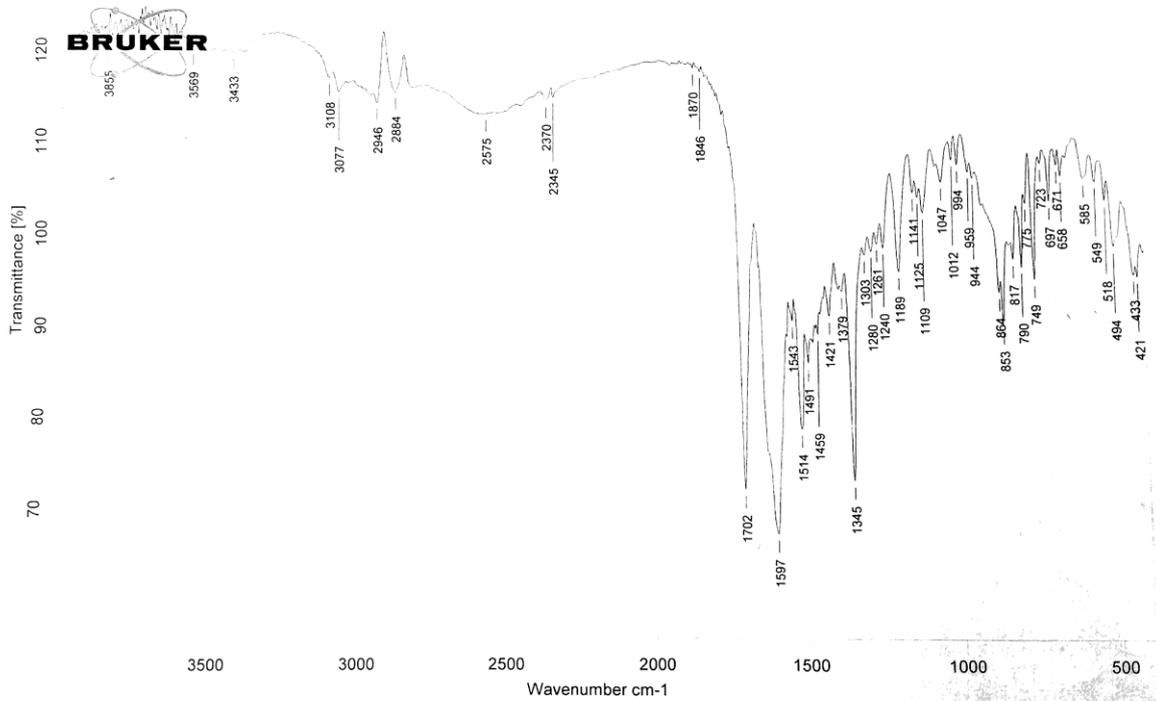
6-Hydroxy-5-[(2-hydroxy-6-oxocyclohex-1-en-1-yl)(4-nitrophenyl)methyl]-1,3-dimethylpyrimidine-2,4(1*H*,3*H*)-dione (3g)



| | | | | | | | |
|-----------|--|-------------|-------------------------------|-------------------------|----------------------|----------------|----------------------|
| Count | 252 | Data Type | MASS SPECTRUM | Date | 24 Oct 2019 14:07:28 | Date Stamp | 26 Sep 1918 18:16:27 |
| File Name | C:\Users\Yuliya\AppData\Local\Temp\RaRa0.115\571 63 64 66.JC | Inlet Model | DIRECT | Instrumental Parameters | LOW RESOLUTION | Spectrum Title | MASS SPECTRUM |
| Origin | LEO_66 | Owner | Copyright(C) by Victor (2018) | | | | |
| TIC | 1512.32 | | | | | | |

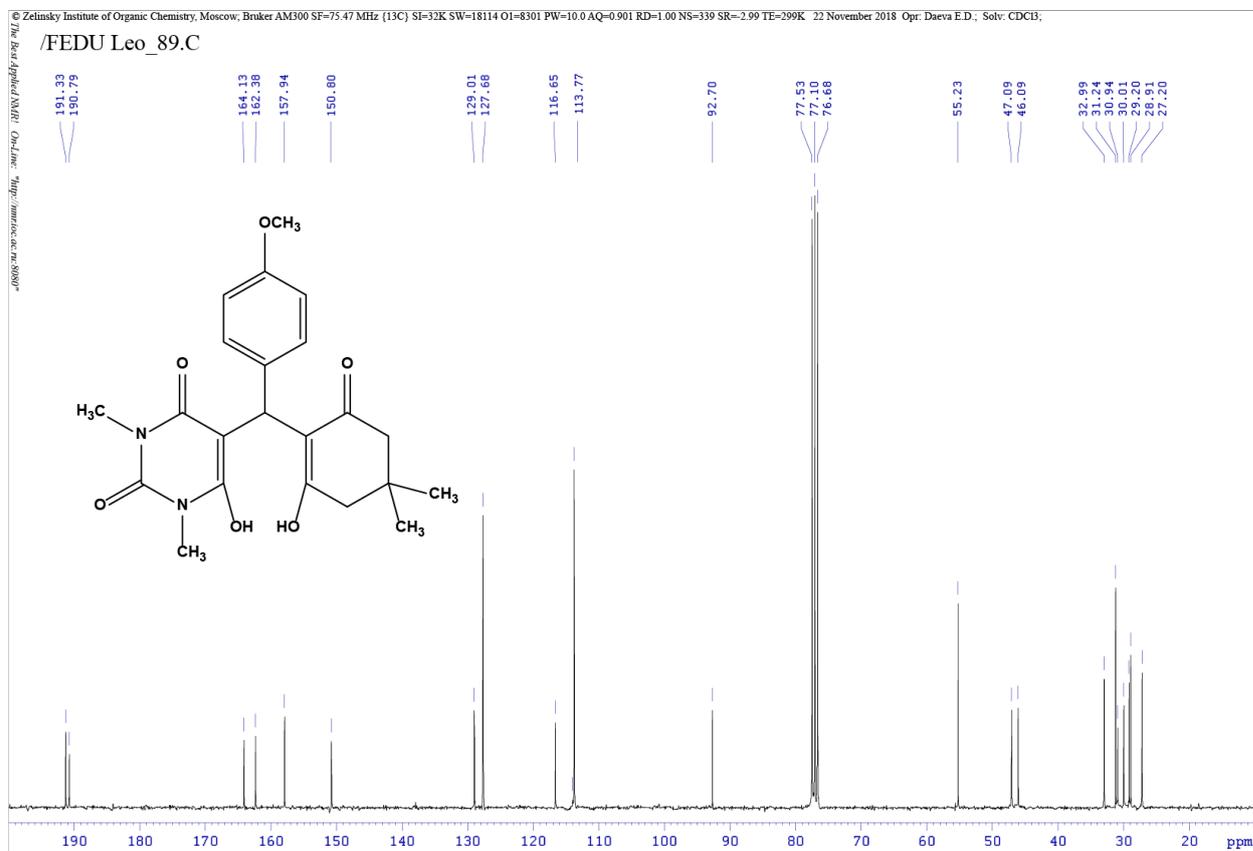
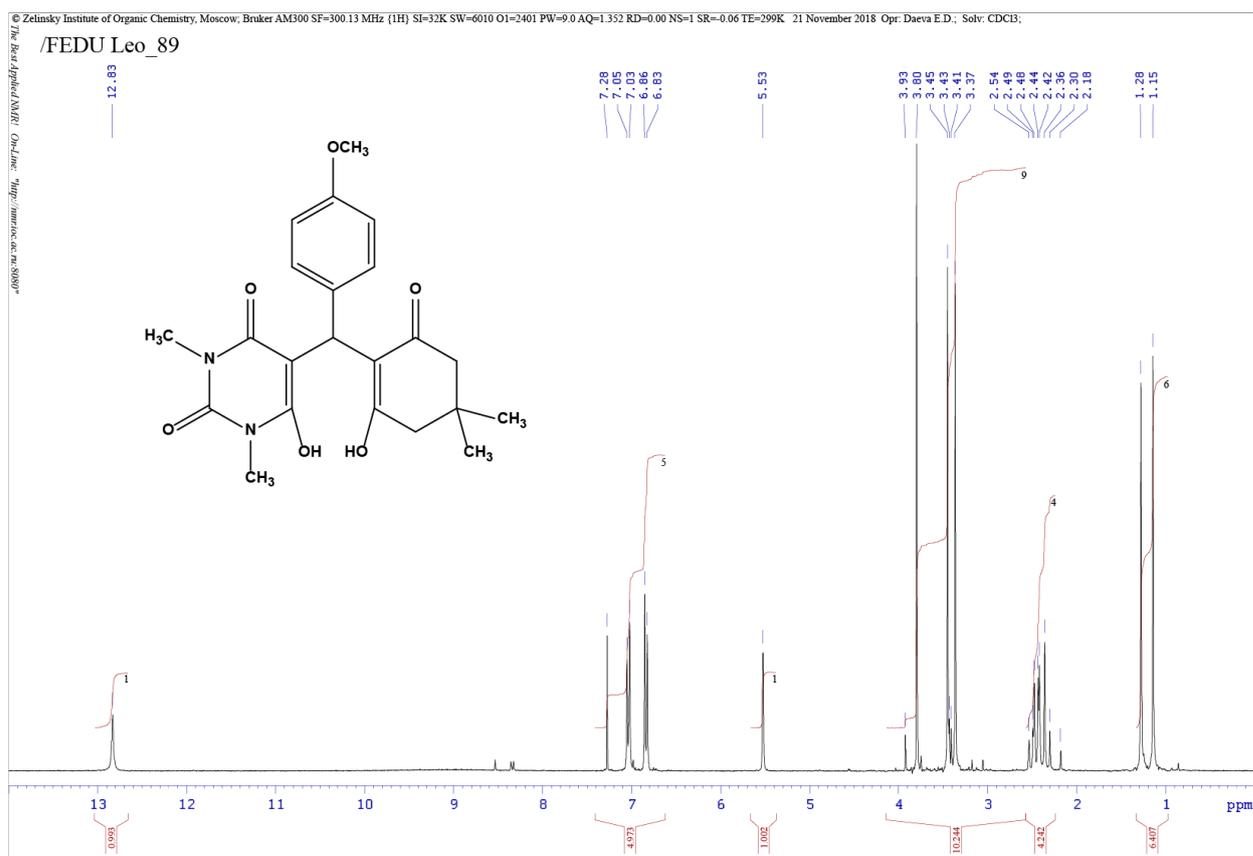


| No. | m/z | RI(%) | DI | No. | m/z | TIC(%) |
|-----|---------|---------|---------|-----|---------|--------|
| 1 | 42.000 | 95.810 | 95.810 | 1 | 42.000 | 6.335 |
| 2 | 56.000 | 28.723 | 28.723 | 2 | 56.000 | 1.899 |
| 3 | 128.000 | 19.552 | 19.552 | 3 | 128.000 | 1.293 |
| 4 | 156.000 | 48.865 | 48.865 | 4 | 156.000 | 3.231 |
| 5 | 198.000 | 32.633 | 32.633 | 5 | 198.000 | 2.158 |
| 6 | 228.000 | 47.035 | 47.035 | 6 | 228.000 | 3.110 |
| 7 | 272.000 | 17.572 | 17.572 | 7 | 272.000 | 1.162 |
| 8 | 288.000 | 26.993 | 26.993 | 8 | 288.000 | 1.785 |
| 9 | 339.000 | 4.980 | 4.980 | 9 | 339.000 | 0.329 |
| 10 | 401.000 | 100.000 | 100.000 | 10 | 401.000 | 6.612 |

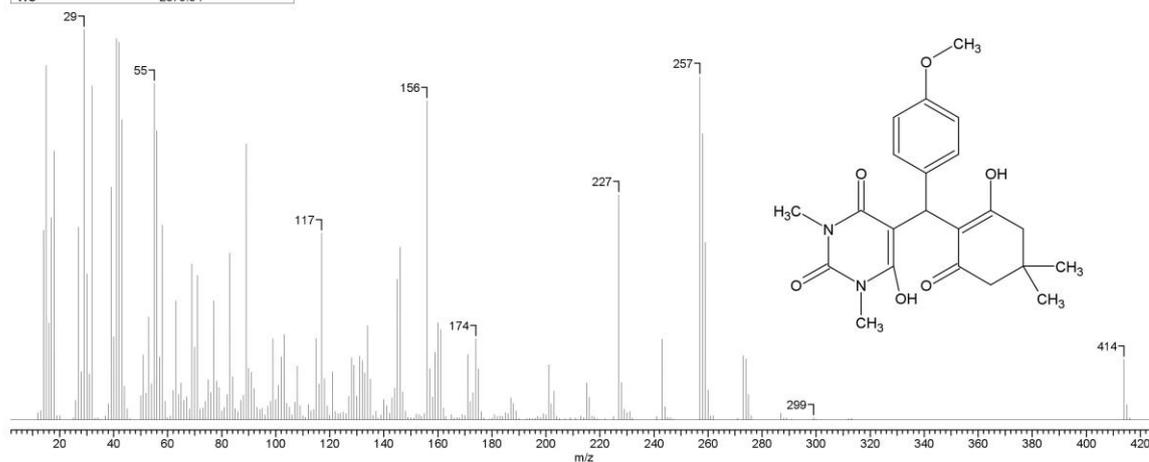


| | | | |
|------------------------|----------------|---------------|------------|
| D ЧРВ\Анисина Leo-66.0 | Анисина Leo-66 | КВг прессовка | 28.09.2018 |
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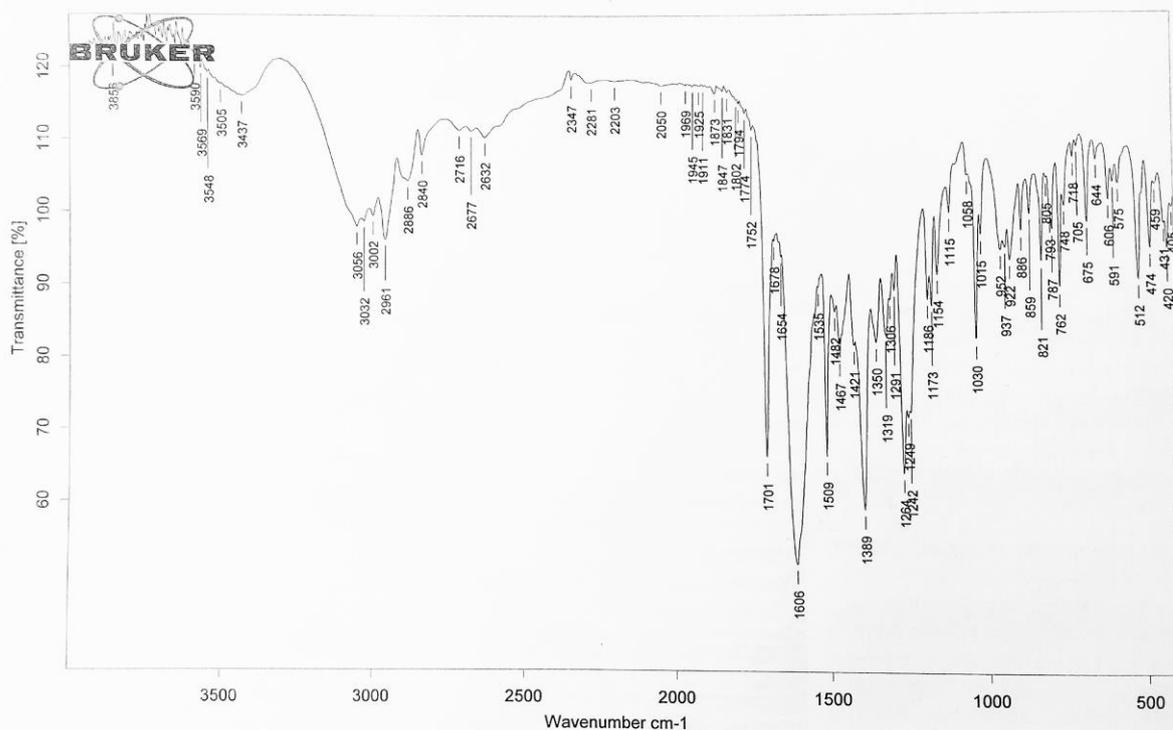
6-Hydroxy-5-[(2-hydroxy-4,4-dimethyl-6-oxocyclohex-1-en-1-yl)(4-methoxyphenyl)methyl]-1,3-dimethylpyrimidine-2,4(1*H*,3*H*)-dione (3j)



| | | | | | | | |
|-----------|--|-------------|-------------------------------|-------------------------|----------------------|----------------|----------------------|
| Count | 228 | Data Type | MASS SPECTRUM | Date | 24 Oct 2019 14:07:28 | Date Stamp | 03 Dec 1918 18:09:00 |
| File Name | C:\Users\Yuliya\AppData\Local\Temp\RafRa0.115\89.801.95.JC | Inlet Model | DIRECT | Instrumental Parameters | LOW RESOLUTION | Spectrum Title | MASS SPECTRUM |
| Origin | LEO-89 | Owner | Copyright(C) by Victor (2018) | | | | |
| TIC | 2879.84 | | | | | | |



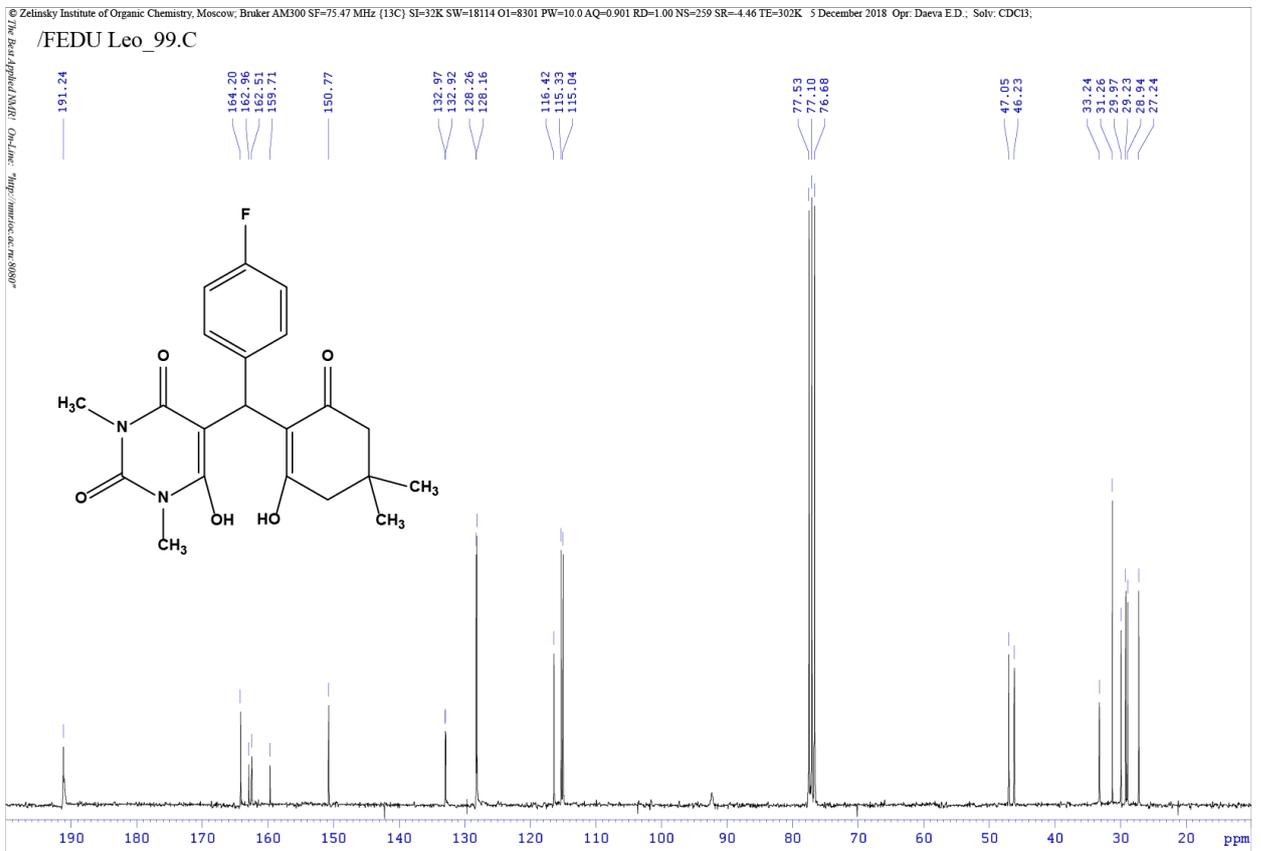
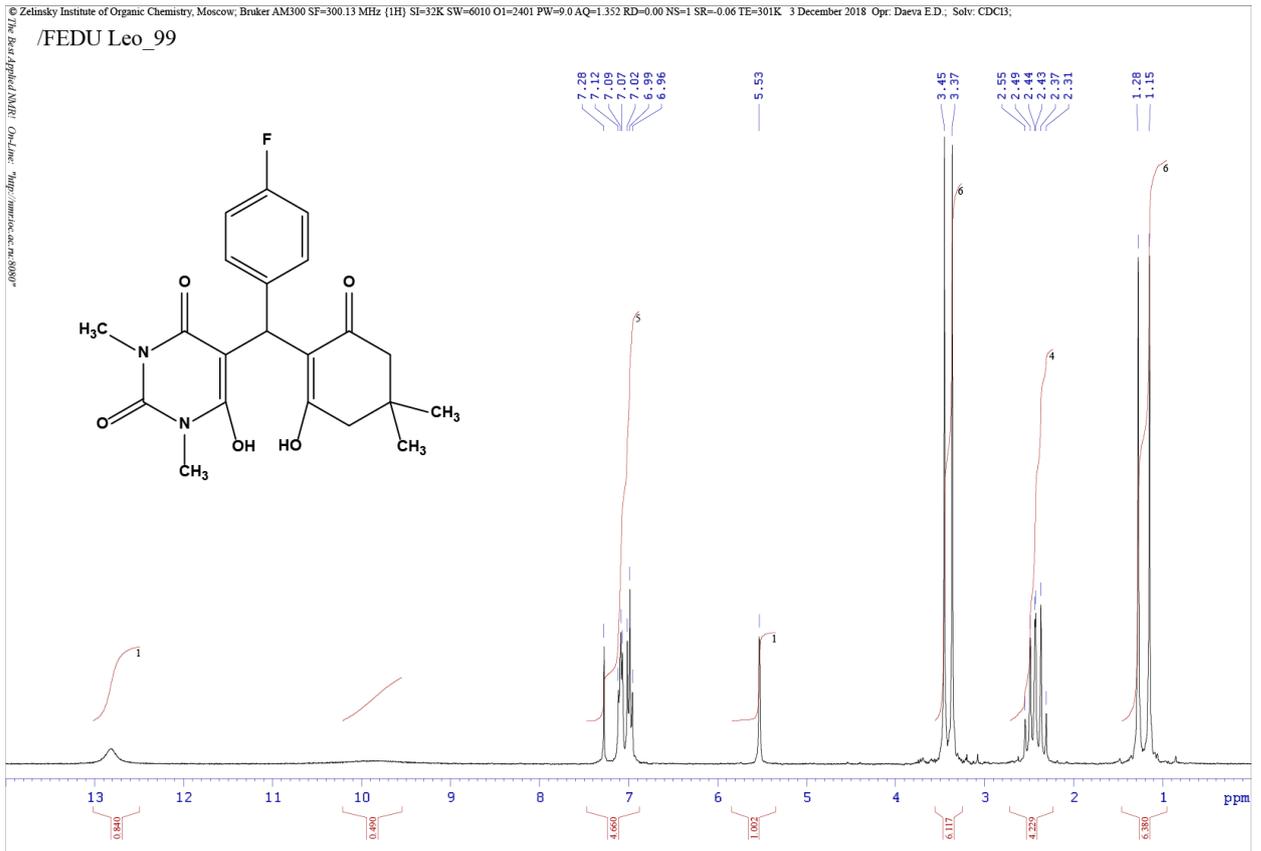
| No. | m/z | RI(%) | DI | No. | m/z | TIC(%) |
|-----|---------|---------|---------|-----|---------|--------|
| 1 | 29.000 | 100.000 | 100.000 | 1 | 29.000 | 3.472 |
| 2 | 55.000 | 86.149 | 86.149 | 2 | 55.000 | 2.991 |
| 3 | 117.000 | 47.795 | 47.795 | 3 | 117.000 | 1.660 |
| 4 | 156.000 | 81.708 | 81.708 | 4 | 156.000 | 2.837 |
| 5 | 174.000 | 20.762 | 20.762 | 5 | 174.000 | 0.721 |
| 6 | 227.000 | 57.596 | 57.596 | 6 | 227.000 | 2.000 |
| 7 | 257.000 | 87.869 | 87.869 | 7 | 257.000 | 3.051 |
| 8 | 299.000 | 0.470 | 0.470 | 8 | 299.000 | 0.016 |
| 9 | 414.000 | 15.522 | 15.522 | 9 | 414.000 | 0.539 |



D:\ИВ\Леонаова_Leo_89.0 Леонаова Leo_89 KBr прессовка

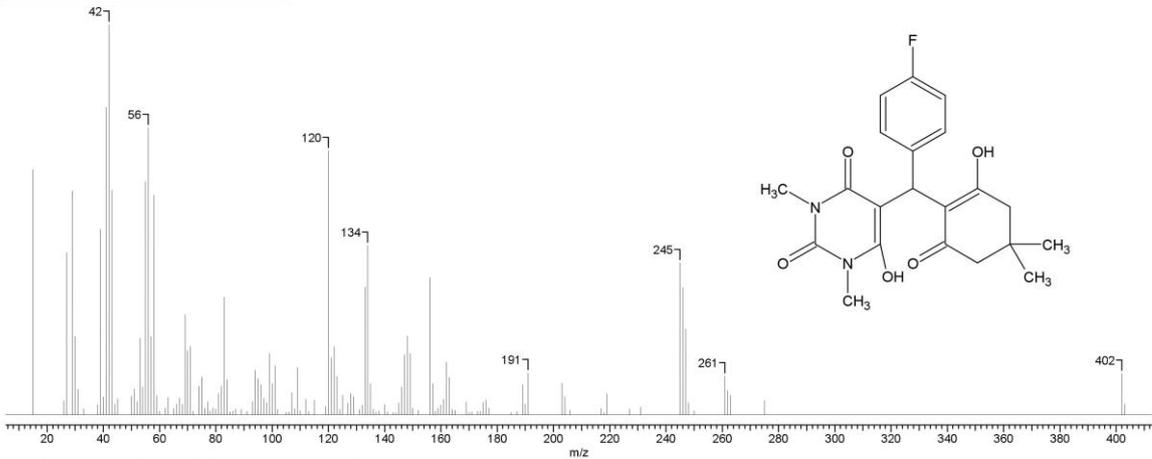
18.02.2019

5-[(4-Fluorophenyl)(2-hydroxy-4,4-dimethyl-6-oxocyclohex-1-en-1-yl)methyl]-6-hydroxy-1,3-dimethylpyrimidine-2,4(1H,3H)-dione (3k)

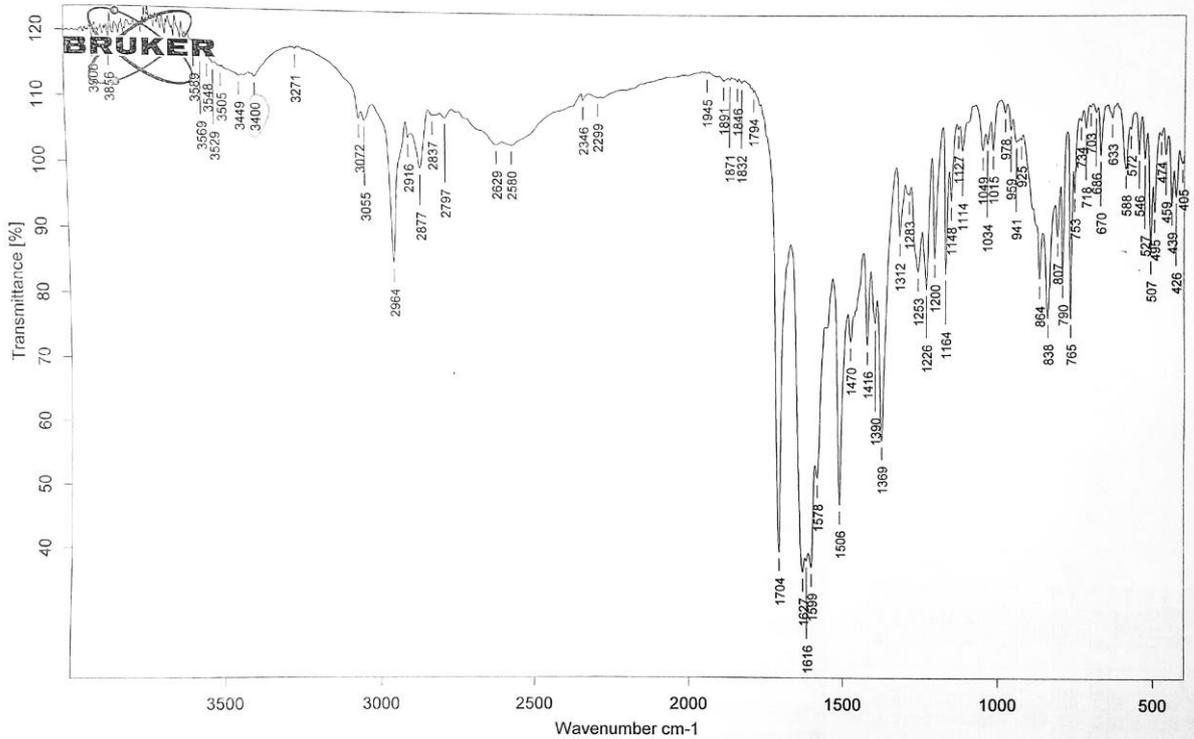


24 Oct 2019

| | | | | | | | |
|-----------|--|-------------|-------------------------------|-------------------------|----------------------|----------------|----------------------|
| Count | 142 | Data Type | MASS SPECTRUM | Date | 24 Oct 2019 14:07:28 | Date Stamp | 05 Dec 1918 17:46:41 |
| File Name | C:\Users\Yuliya\AppData\Local\Temp\Ra0.439\99.JC | Inlet Model | DIRECT | Instrumental Parameters | LOW RESOLUTION | Spectrum Title | MASS SPECTRUM |
| Origin | LEO-99 | Owner | Copyright(C) by Victor (2018) | | | | |
| TIC | 1587.12 | | | | | | |



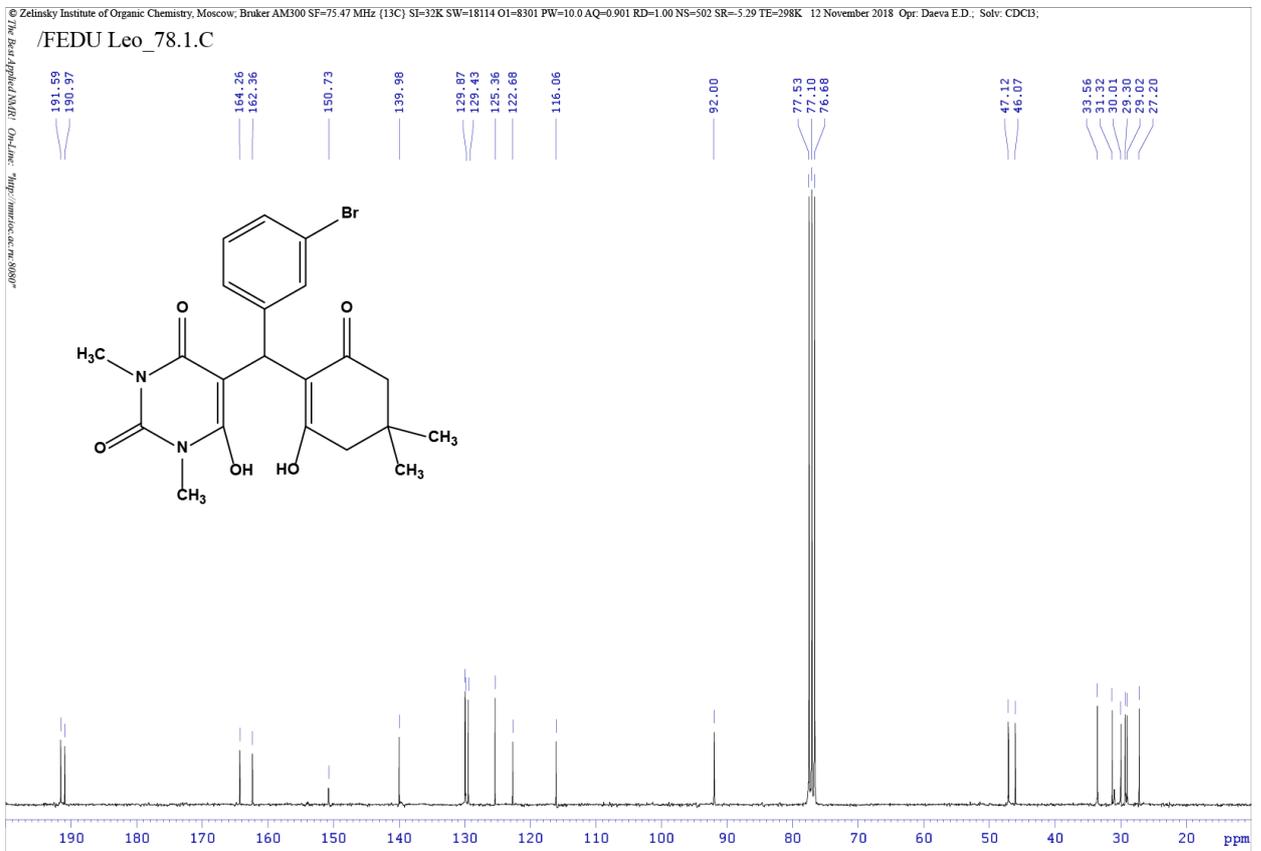
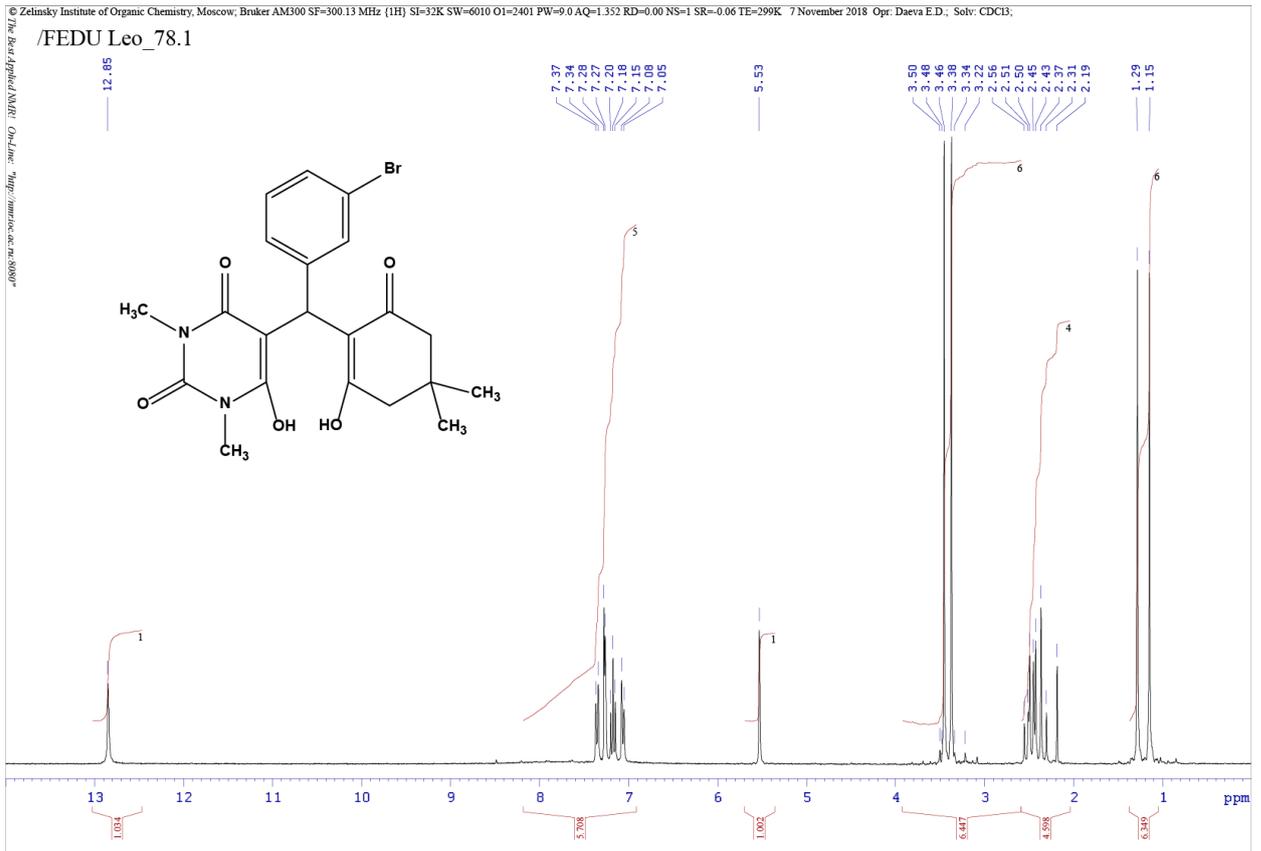
| No. | m/z | RI(%) | DI | No. | m/z | TIC(%) |
|-----|---------|---------|---------|-----|---------|--------|
| 1 | 42.000 | 100.000 | 100.000 | 1 | 42.000 | 6.301 |
| 2 | 56.000 | 73.507 | 73.507 | 2 | 56.000 | 4.631 |
| 3 | 120.000 | 67.617 | 67.617 | 3 | 120.000 | 4.260 |
| 4 | 134.000 | 43.384 | 43.384 | 4 | 134.000 | 2.734 |
| 5 | 191.000 | 10.761 | 10.761 | 5 | 191.000 | 0.678 |
| 6 | 245.000 | 38.904 | 38.904 | 6 | 245.000 | 2.451 |
| 7 | 261.000 | 9.981 | 9.981 | 7 | 261.000 | 0.629 |
| 8 | 402.000 | 10.621 | 10.621 | 8 | 402.000 | 0.669 |



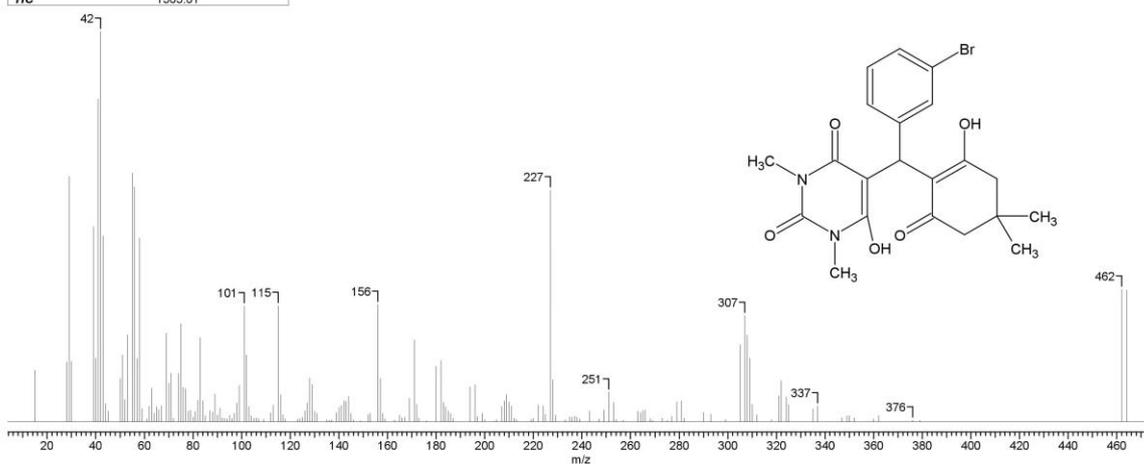
D:\rB\Леонова Leo_99.0 Леонова Leo_99 KBr прессовка

18.02.2019

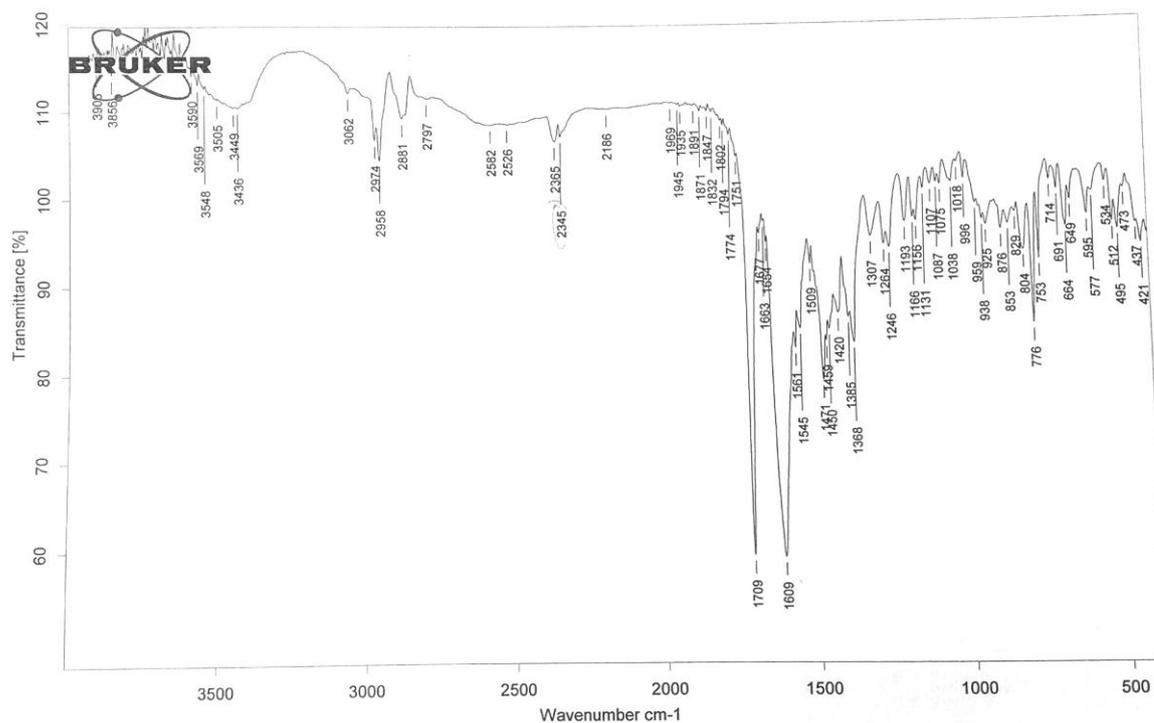
5-[(3-Bromophenyl)(2-hydroxy-4,4-dimethyl-6-oxocyclohex-1-en-1-yl)methyl]-6-hydroxy-1,3-dimethylpyrimidine-2,4(1H,3H)-dione (3m)



| | | | | | | | |
|-----------|---|-------------|-------------------------------|-------------------------|----------------------|------------|----------------------|
| Count | 186 | Data Type | MASS SPECTRUM | Date | 24 Oct 2019 14:07:28 | Date Stamp | 12 Nov 1918 17:35:41 |
| File Name | C:\Users\Yuliya\AppData\Local\Temp\Ra0.4391781.JC | Inlet Model | DIRECT | Instrumental Parameters | LOW RESOLUTION | | |
| Origin | LEO_78.1 | Owner | Copyright(C) by Victor (2018) | Spectrum Title | MASS SPECTRUM | | |
| TIC | 1565.61 | | | | | | |



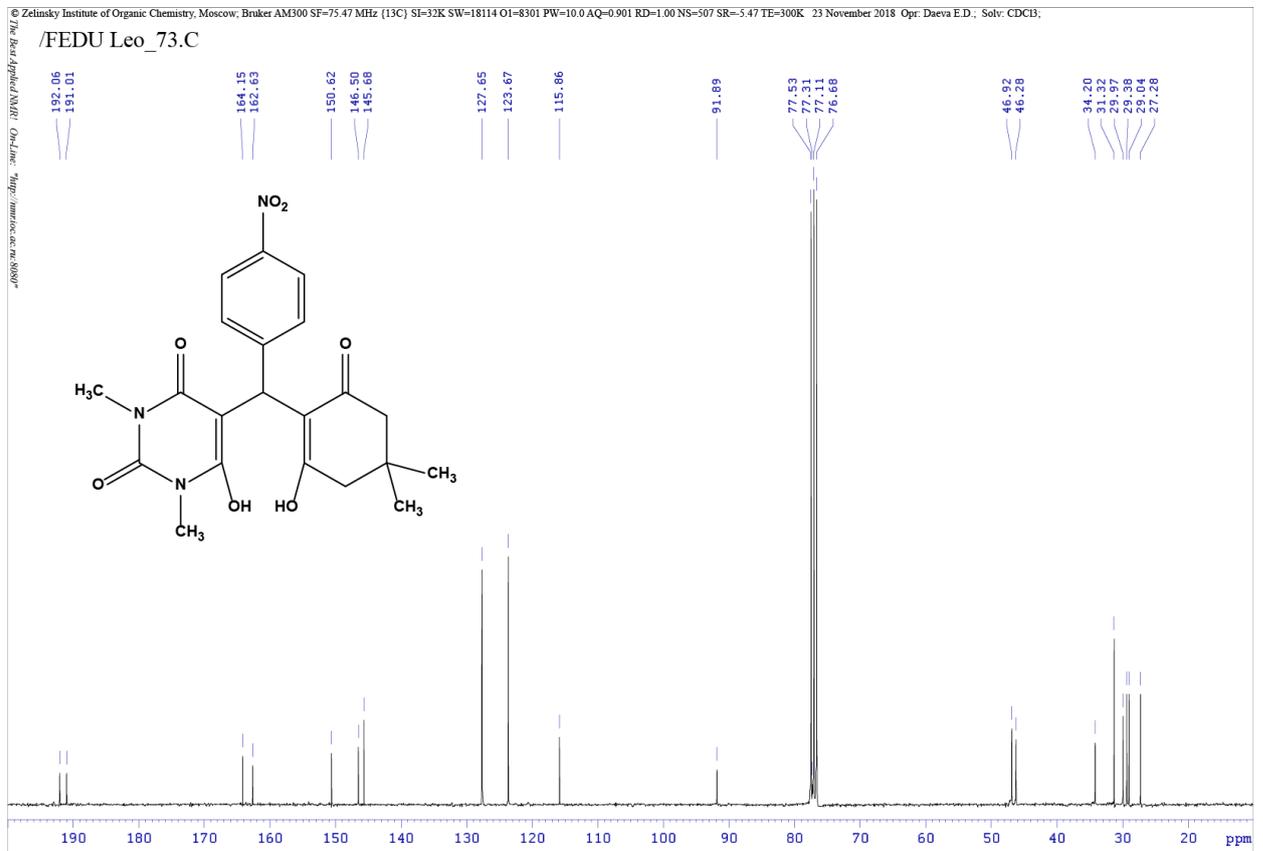
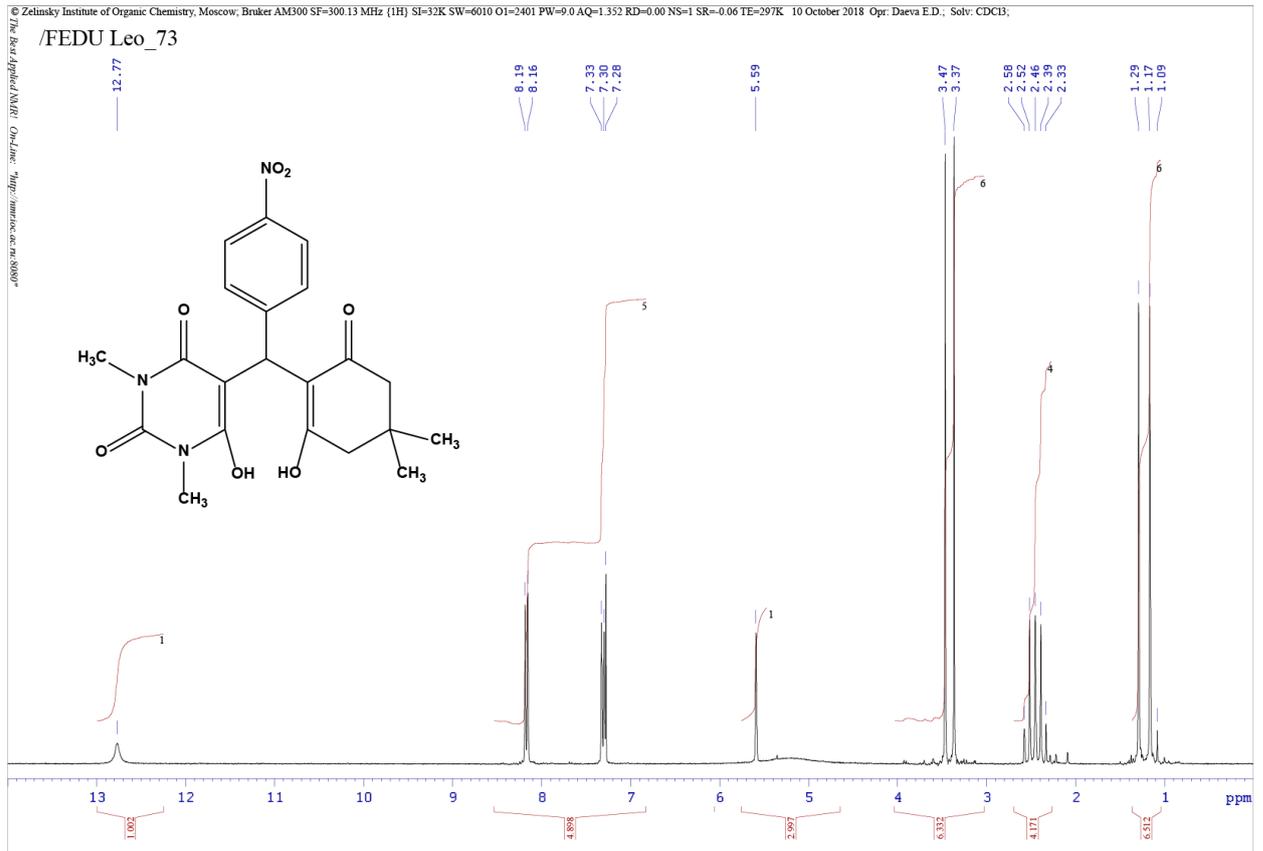
| No. | m/z | RI(%) | DI | No. | m/z | TIC(%) |
|-----|---------|---------|---------|-----|---------|--------|
| 1 | 42.000 | 100.000 | 100.000 | 1 | 42.000 | 6.387 |
| 2 | 101.000 | 29.673 | 29.673 | 2 | 101.000 | 1.895 |
| 3 | 115.000 | 29.743 | 29.743 | 3 | 115.000 | 1.900 |
| 4 | 156.000 | 30.053 | 30.053 | 4 | 156.000 | 1.920 |
| 5 | 227.000 | 59.386 | 59.386 | 5 | 227.000 | 3.793 |
| 6 | 251.000 | 7.621 | 7.621 | 6 | 251.000 | 0.487 |
| 7 | 307.000 | 27.203 | 27.203 | 7 | 307.000 | 1.738 |
| 8 | 337.000 | 4.010 | 4.010 | 8 | 337.000 | 0.256 |
| 9 | 376.000 | 0.380 | 0.380 | 9 | 376.000 | 0.024 |
| 10 | 462.000 | 33.903 | 33.903 | 10 | 462.000 | 2.166 |



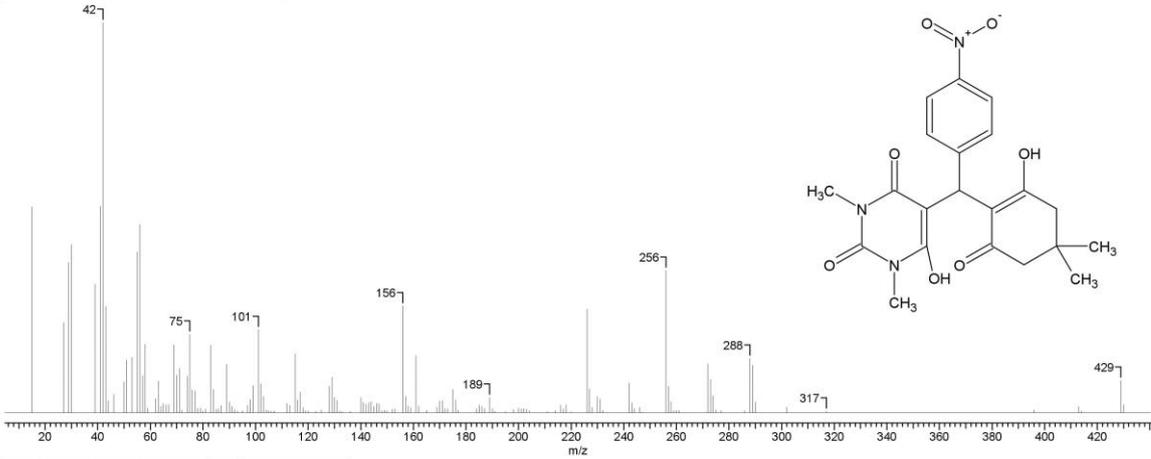
D:\lrB\Леонаво Leo_78.1 Леонаво Leo_78.1 KBr прессовка

18.02.2019

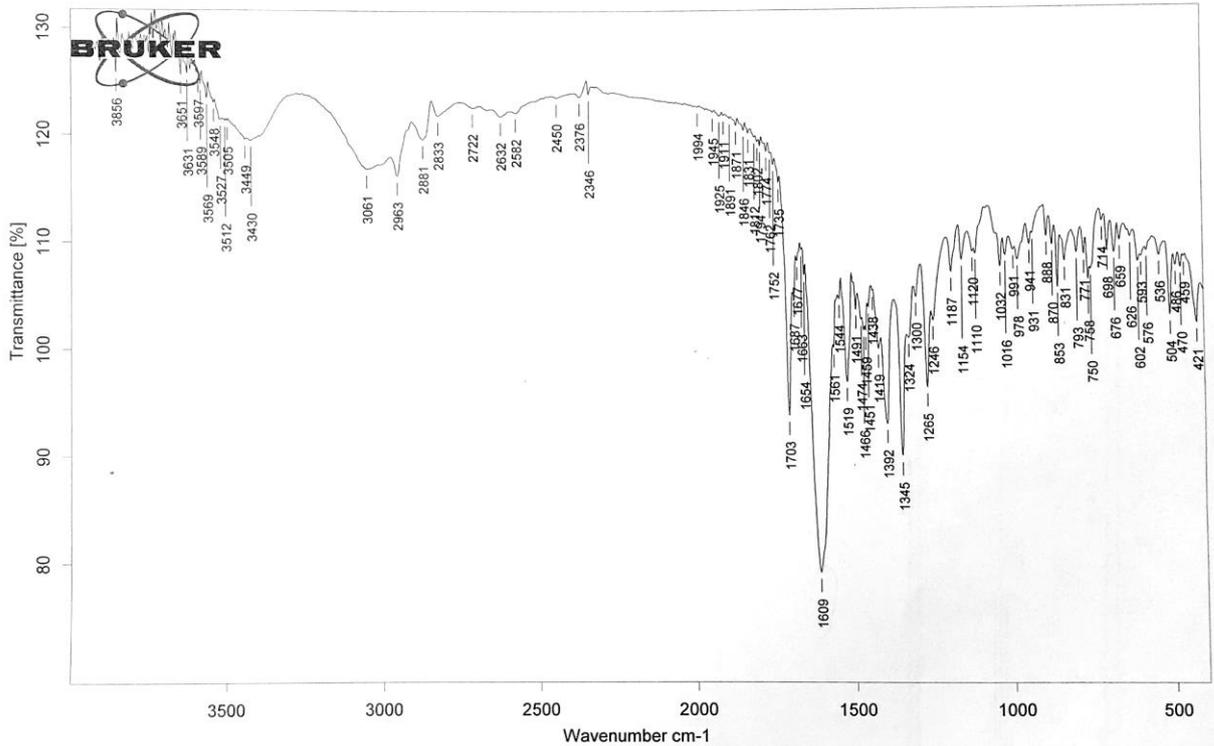
6-Hydroxy-5-[(2-hydroxy-4,4-dimethyl-6-oxocyclohex-1-en-1-yl)(4-nitrophenyl)methyl]-1,3-dimethylpyrimidine-2,4(1*H*,3*H*)-dione (3n)



| | | | | | | | |
|-----------|---|-------------|-------------------------------|-------------------------|----------------------|----------------|----------------------|
| Count | 155 | Data Type | MASS SPECTRUM | Date | 24 Oct 2019 14:07:28 | Date Stamp | 07 Nov 1918 14:24:03 |
| File Name | C:\Users\Yuliya\AppData\Local\Temp\Ra0.115\52.77.70.73.JC | Inlet Model | DIRECT | Instrumental Parameters | LOW RESOLUTION | Spectrum Title | MASS SPECTRUM |
| Origin | LEO-73 | Owner | Copyright(C) by Victor (2018) | | | | |
| TIC | 1080.70 | | | | | | |



| No. | m/z | RI(%) | DI | No. | m/z | TIC(%) |
|-----|---------|---------|---------|-----|---------|--------|
| 1 | 42.000 | 100.000 | 100.000 | 1 | 42.000 | 9.253 |
| 2 | 75.000 | 20.162 | 20.162 | 2 | 75.000 | 1.866 |
| 3 | 101.000 | 21.312 | 21.312 | 3 | 101.000 | 1.972 |
| 4 | 156.000 | 27.423 | 27.423 | 4 | 156.000 | 2.538 |
| 5 | 189.000 | 3.960 | 3.960 | 5 | 189.000 | 0.366 |
| 6 | 256.000 | 36.434 | 36.434 | 6 | 256.000 | 3.371 |
| 7 | 288.000 | 13.971 | 13.971 | 7 | 288.000 | 1.293 |
| 8 | 317.000 | 0.390 | 0.390 | 8 | 317.000 | 0.036 |
| 9 | 429.000 | 8.331 | 8.331 | 9 | 429.000 | 0.771 |



| | | | |
|-------------------------|----------------|---------------|------------|
| D:\r\B\Леонава Leo_73.0 | Леонава Leo_73 | КВr прессовка | 18.02.2019 |
|-------------------------|----------------|---------------|------------|