Electronic supplementary materials *Mendeleev Commun*., 2021, **31**, 347–349

Selective and efficient electrocatalytic way to spirobarbituric dihydrofurans

Michail N. Elinson, Anatoly N. Vereshchagin, Yuliya E. Ryzhkova and Mikhail P. Egorov

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1. Experimental section

All melting points were measured with a Gallenkamp melting point apparatus and are uncorrected. ^{1}H and ^{13C} NMR spectra were recorded with a Bruker AM-300 (300 and 75 MHz, respectively) at ambient temperature in $CDCl_3$ and DMSO-d_6 solutions. Chemical shift values are given in δ scale relative to Me_4Si. 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.

X-ray diffraction data were collected at 100K on a Bruker Quest D8 diffractometer equipped with a Photon-III area-detector (graphite monochromator, shutterless φ- and ω-scan technique), using Mo K_α-radiation. The intensity data were integrated by the SAINT program[1] and corrected for absorption and decay using SADABS.[2] The structure was solved by direct methods using SHELXT[3] and refined on F^2 using SHELXL-2018.[4] All non-hydrogen atoms were refined with anisotropic displacement parameters. Hydrogen atoms were placed in ideal calculated positions as riding atoms with relative isotropic displacement parameters; bond distances to H-atoms were refined. A rotating group model was applied for methyl groups. The SHELXTL program suite[1] was used for molecular graphics.

General procedure

A solution of 6-hydroxy-5-[(2-hydroxy-6-oxocyclohex-1-en-1-yl)(aryl)methyl]-1,3-dimethyl-pyrimidine-2,4-(1*H*,3*H*)-diones **1** (5 mmol) and sodium bromide (0.3 g, 3 mmol) in methanol (20 mL) was electrolyzed in an undivided cell equipped with a magnetic stirrer, a graphite anode and an iron cathode at 20 ºC under a constant current density of 100 mA/cm^2 (*I* = 500 mA, electrodes square 5 cm^2) until the catalytic quantity of 2.2 F/mol of electricity was passed. After the electrolysis was finished, the reaction mixture was concentrated to one fifth of its initial volume

(ca. 4 mL) and cold to 0° C to crystallize the solid compound **2**, which was then filtered, twice rinsed with an ice-cold ethanol/water solution (1:1 4 mL), and dried under reduced pressure.

1',3'-Dimethyl-3-phenyl-3,5,6,7-tetrahydro-2'*H*,4*H*-spiro[benzofuran-2,5'-pyrimidine]-2',4,4',6'(1'*H*,3'*H*)-tetraone (2a).

Yield 1.54 g (87%), mp: 245-247 °C. ^{1}H NMR (300 MHz, $CDCl_3$): δ 2.17-2.30 (m, 2H, CH_2), 2.38-2.51 (m, 2H, CH_2), 2.57 (s, 3H, CH_3), 2.74-2.96 (m, 2H, CH_2), 3.42 (s, 3H, CH_3), 4.70 (s, 1H, CH), 6.96-7.07 (m, 2 CH Ar), 7.25-7.35 (m, 3H, 3 CH Ar) ppm. ^{13}C NMR (75 MHz, $CDCl_3$): δ 21.6, 23.9, 28.3, 29.4, 37.0, 59.7, 90.5, 112.6, 128.3 (2C), 128.7 (2C), 129.0, 133.9, 150.0, 164.0, 166.6, 178.8, 193.6 ppm. MS (*m/z* relative intensity %): 354 [M^+] (14), 297 (21), 270 (77), 243 (10), 199 (10), 128 (45), 102 (48), 58 (100), 42 (79), 15 (73). IR (KBr) ν = 3447, 2957, 2899, 1763, 1696, 1650, 1441, 1380, 1284, 1038 cm^{-1}. Anal. calcd for $C_{19}H_{18}N_2O_5$: C, 64.40; H, 5.12; N, 7.91%. Found: C, 64.25; H, 5.06; N, 7.80%.

3-(4-Methoxyphenyl)-1',3'-dimethyl-3,5,6,7-tetrahydro-2'*H*,4*H*-spiro[benzofuran-2,5'-pyrimidine]-2',4,4',6'(1'*H*,3'*H*)-tetraone (2b).

Yield 1.61 g (84%), mp: 199-201 °C. ^{1}H NMR (300 MHz, $CDCl_3$): δ 2.16-2.30 (m, 2H, CH_2), 2.35-2.53 (m, 2H, CH_2), 2.65 (s, 3H, CH_3), 2.75-2.95 (m, 2H, CH_2), 3.42 (s, 3H, CH_3), 3.78 (s, 3H, OCH_3), 4.66 (s, 1H, CH), 6.82 (d, ^{3}J = 8.6 Hz, 2H, 2 CH Ar), 6.93 (d, ^{3}J = 8.6 Hz, 2H, 2 CH Ar) ppm. ^{13}C NMR (75 MHz, $CDCl_3$): δ 21.6, 23.9, 28.4, 29.4, 37.0, 55.4, 59.2, 90.5, 112.7, 114.1 (2C), 125.8, 129.4 (2C), 150.1, 160.1, 164.1, 166.7, 178.6, 193.7 ppm. MS (*m/z* relative intensity %): 384 [M^+] (100), 356 (11), 328 (32), 300 (64), 269 (8), 201 (18), 158 (9), 115 (11), 89 (3), 55 (6). IR (KBr) ν = 3463, 2956, 2913, 1713, 1693, 1641, 1441, 1384, 1249, 1040 cm^{-1}. Anal. calcd for $C_{20}H_{20}N_2O_6$: C, 62.49; H, 5.24; N, 7.29%. Found: C, 62.35; H, 5.18; N, 7.21%.

3-(4-Fluorophenyl)-1',3'-dimethyl-3,5,6,7-tetrahydro-2'*H*,4*H*-spiro[benzofuran-2,5'-pyrimidine]-2',4,4',6'(1'*H*,3'*H*)-tetraone (2c).

Yield 1.60 g (86%), mp: 236-238 °C. ^{1}H NMR (300 MHz, $CDCl_3$): δ 2.17-2.30 (m, 2H, CH_2), 2.38-2.50 (m, 2H, CH_2), 2.66 (s, 3H, CH_3), 2.74-2.95 (m, 2H, CH_2), 3.42 (s, 3H, CH_3), 4.68 (s, 1H, CH), 6.96-7.04 (m, 4H, 4 CH Ar) ppm. ^{13}C NMR (75 MHz, $CDCl_3$): δ 21.6, 23.9, 28.4, 29.5, 37.0, 58.8, 90.2, 112.6, 115.8 (d, $^{2}J_{C\text{-}F}$ = 22 Hz, 2C), 129.8, 130.1 (d, $^{3}J_{C\text{-}F}$ = 8 Hz, 2C), 150.0, 163.0 (d, $^{1}J_{C\text{-}F}$ = 249 Hz), 163.9, 166.5, 179.0, 193.7 ppm. MS (*m/z* relative intensity %): 372 [M^+] (51), 344 (17), 315 (31), 288 (100), 217 (5), 202 (5), 146 (23), 120 (13), 58 (13), 42 (13). IR (KBr) ν = 3435, 2952, 2910, 1766, 1694, 1641, 1441, 1385, 1235, 1040 cm^{-1}. Anal. calcd for $C_{19}H_{17}FN_2O_5$: C, 61.29; H, 4.60; F, 5.10; N, 7.52%. Found: C, 61.14; H, 4.52; F, 5.04; N, 7.38%.

3-(3-Chlorophenyl)-1',3'-dimethyl-3,5,6,7-tetrahydro-2'*H*,4*H*-spiro[benzofuran-2,5'-pyrimidine]-2',4,4',6'(1'*H*,3'*H*)-tetraone (2d).

Yield 1.61 g (83%), mp: 246-248 °C. ^{1}H NMR (300 MHz, $CDCl_3$): δ 2.18-2.32 (m, 2H, CH_2), 2.34-2.56 (m, 2H, CH_2), 2.67 (s, 3H, CH_3), 2.72-2.97 (m, 2H, CH_2), 3.42 (s, 3H, CH_3), 4.64 (s, 1H, CH), 6.90 (d, ^{3}J = 7.3 Hz, 1H, CH Ar), 6.99 (s, 1H, CH Ar), 7.18-7.35 (m, 2H, 2 CH Ar) ppm. ^{13}C NMR (75 MHz, DMSO-d_6): δ 21.3, 23.3, 27.7, 29.1, 36.5, 55.9, 89.9, 111.7, 127.4, 128.1,

128.3, 129.8, 132.8, 137.8, 150.2, 163.9, 166.1, 179.2, 193.3 ppm. MS (*m/z* relative intensity %): 390 [^{37}Cl, M^+] (8), 388 [^{35}Cl, M^+] (26), 333 (^{37}Cl, 7), 331 (^{35}Cl, 22), 306 (^{37}Cl, 33), 304 (^{35}Cl, 100), 269 (3), 233 (5), 176 (11), 136 (11), 127 (15), 58 (21), 18 (22). IR (KBr) ν = 3063, 2956, 2892, 1761, 1697, 1652, 1431, 1380, 1239, 1038 cm^{-1}. Anal. calcd for $C_{19}H_{17}ClN_2O_5$: C, 58.70; H, 4.41; Cl, 9.12; N, 7.21%. Found: C, 58.59; H, 4.48; Cl, 9.05; N, 7.13%.

1',3',6,6-Tetramethyl-3-phenyl-3,5,6,7-tetrahydro-2'*H*,4*H*-spiro[benzofuran-2,5'-pyrimidine]-2',4,4',6'(1'*H*,3'*H*)-tetraone (2e).

Yield 1.57 g (82%), mp: 174-176 °C. ^{1}H NMR (300 MHz, $CDCl_3$): δ 1.23 (s, 3H, CH_3), 1.31 (s, 3H, CH_3), 2.30 (d, ^2J = 16.4 Hz, 1H, CH_2), 2.37 (d, ^2J = 16.4 Hz, 1H, CH_2), 2.58 (s, 3H, CH_3), 2.65 (d, ^2J = 17.7 Hz, 1H, CH_2), 2.75 (d, ^2J = 17.7 Hz, 1H, CH_2), 3.42 (s, 3H, CH_3), 4.71 (s, 1H, CH), 6.94-7.06 (m, 2H, 2 CH Ar), 7.25-7.39 (m, 3H, 3 CH Ar) ppm. ^{13}C NMR (75 MHz, $CDCl_3$): δ 28.3, 28.7 (2C), 29.4, 34.4, 37.6, 51.5, 59.6, 90.8, 111.3, 128.3 (2C), 128.7 (2C), 129.0, 134.0, 150.1, 164.0, 166.6, 177.9, 193.0 ppm. MS (*m/z* relative intensity %): 382 [M^+] (75), 354 (5), 298 (67), 270 (100), 227 (11), 184 (8), 142 (14), 128 (22), 55 (22), 41 (22). IR (KBr) ν = 3411, 2959, 2874, 1714, 1693, 1654, 1432, 1380, 1285, 1044 cm^{-1}. Anal. calcd for $C_{21}H_{22}N_2O_5$: C, 65.96; H, 5.80; N, 7.33%. Found: C, 65.82; H, 5.86; N, 7.21%.

1',3',6,6-Tetramethyl-3-(*m*-tolyl)-3,5,6,7-tetrahydro-2'*H*,4*H*-spiro[benzofuran-2,5'-pyrimidine]-2',4,4',6'(1'*H*,3'*H*)-tetraone (2f).

Yield 1.68 g (85%), mp: 183-185 °C. ^{1}H NMR (300 MHz, $CDCl_3$): δ 1.22 (s, 3H, CH_3), 1.30 (s, 3H, CH_3), 2.30 (d, ^2J = 16.2 Hz, 1H, CH_2), 2.31 (s, 3H, CH_3), 2.37 (d, ^2J = 16.2 Hz, 1H, CH_2), 2.58 (s, 3H, CH_3), 2.64 (d, ^2J = 17.5 Hz, 1H, CH_2), 2.75 (d, ^2J = 17.5 Hz, 1H, CH_2), 3.42 (s, 3H, CH_3), 4.66 (s, 1H, CH), 6.74-6.84 (m, 2H, 2 CH Ar), 7.11 (d, ^3J = 7.6 Hz, 1H, CH Ar) 7.19 (t, ^3J = 7.6 Hz, 1H, CH Ar) ppm. ^{13}C NMR (75 MHz, $CDCl_3$): δ 21.4, 28.3, 28.7, 28.8, 29.4, 34.4, 37.7, 51.5, 59.6, 90.9, 111.3, 125.4, 128.6, 129.0, 129.7, 133.8, 138.5, 150.1, 164.0, 166.6, 177.8, 193.1 ppm. MS (*m/z* relative intensity %): 396 [M^+] (10), 325 (2), 284 (100), 271 (5), 241 (35), 198 (9), 141 (39), 115 (44), 58 (51), 41 (58). IR (KBr) ν = 3462, 2965, 2879, 1715, 1691, 1647, 1433, 1379, 1282, 1034 cm^{-1}. Anal. calcd for $C_{22}H_{24}N_2O_5$: C, 66.65; H, 6.10; N, 7.07%. Found: C, 66.46; H, 6.03; N, 7.00%.

3-(4-Chlorophenyl)-1',3',6,6-tetramethyl-3,5,6,7-tetrahydro-2'*H*,4*H*-spiro[benzofuran-2,5'-pyrimidine]-2',4,4',6'(1'*H*,3'*H*)-tetraone (2g).

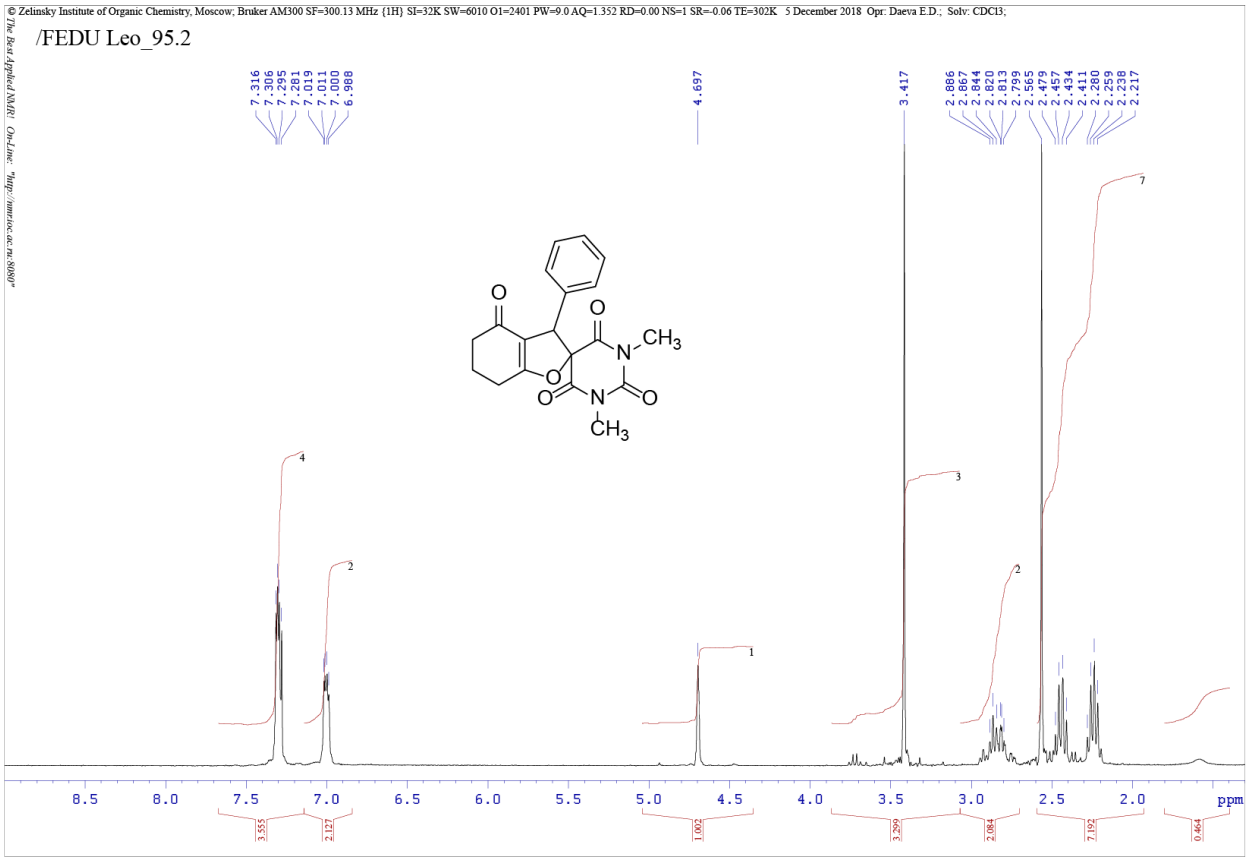
Yield 1.88 g (90%), mp: 265-267 °C. ^{1}H NMR (300 MHz, $CDCl_3$): δ 1.22 (s, 3H, CH_3), 1.29 (s, 3H, CH_3), 2.29 (d, ^2J = 16.6 Hz, 1H, CH_2), 2.35 (d, ^2J = 16.6 Hz, 1H, CH_2), 2.64 (d, ^2J = 17.8 Hz, 1H, CH_2), 2.67 (s, 3H, CH_3), 2.74 (d, ^2J = 17.8 Hz, 1H, CH_2), 3.41 (s, 3H, CH_3), 4.66 (s, 1H, CH), 6.95 (d, ^3J = 7.7 Hz, 2H, 2 CH Ar), 7.29 (d, ^3J = 7.7 Hz, 2H, 2 CH Ar) ppm. ^{13}C NMR (75 MHz, $CDCl_3$): δ 28.5, 28.7, 28.8, 29.5, 34.4, 37.7, 51.4, 58.8, 90.4, 111.2, 129.0 (2C), 129.7 (2C), 132.6, 135.1, 150.0, 163.8, 166.4, 178.1, 193.0 ppm. MS (*m/z* relative intensity %): 418 [^{37}Cl, M^+] (32), 416 [^{35}Cl, M^+] (92), 373 (7), 334 (^{37}Cl, 21), 332 (^{35}Cl, 64), 306 (^{37}Cl, 33), 304 (^{35}Cl, 100), 297 (28), 218 (8), 178 (^{37}Cl, 6), 176 (^{35}Cl, 15), 127 (19), 56 (35), 41 (36). IR (KBr) ν = 3443, 2964, 2875, 1705, 1694, 1645, 1439, 1382, 1223, 1029 cm^{-1}. Anal. calcd for $C_{21}H_{21}ClN_2O_5$: C, 60.51; H, 5.08; Cl, 8.50; N, 6.72%. Found: C, 60.46; H, 5.12; Cl, 8.37; N, 6.63%.

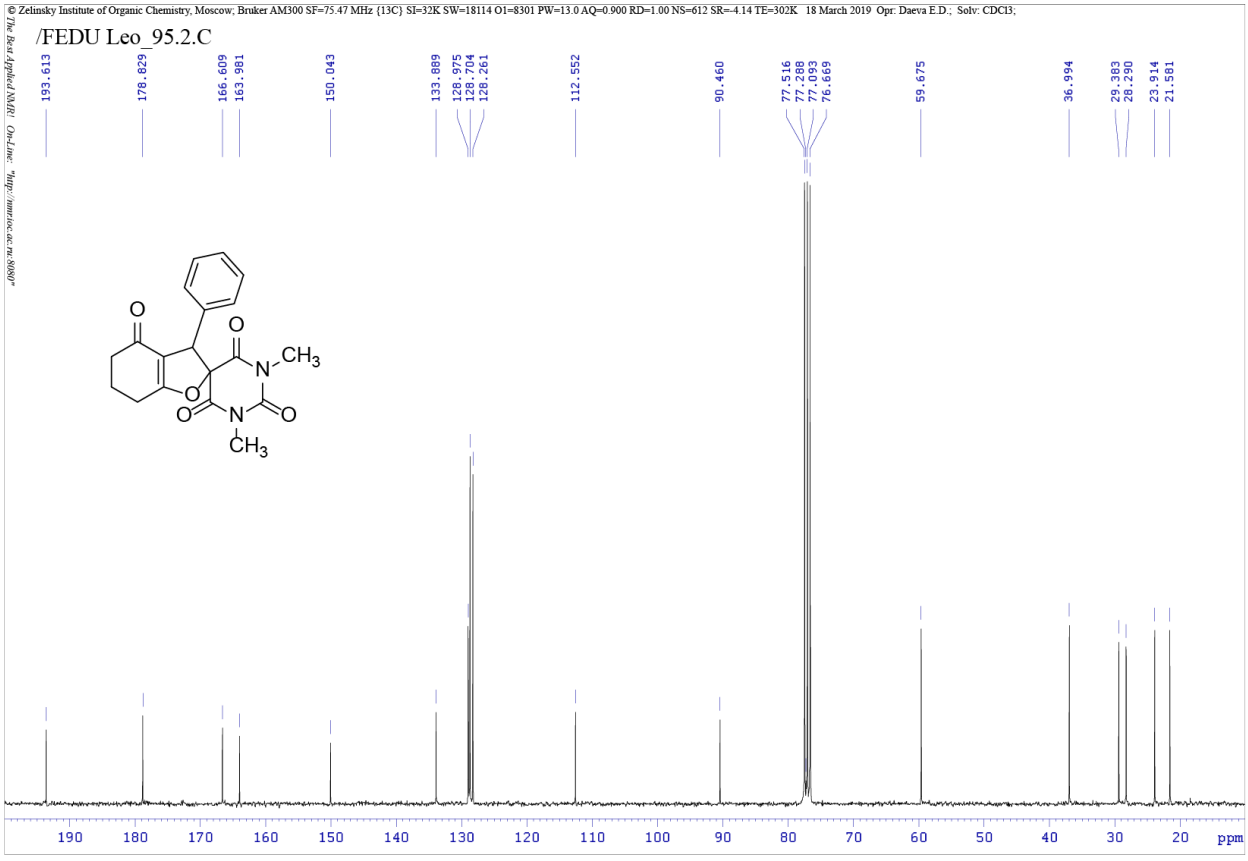
3-(3-Bromophenyl)-1',3',6,6-tetramethyl-3,5,6,7-tetrahydro-2'*H*,4*H*-spiro[benzofuran-2,5'-pyrimidine]-2',4,4',6'(1'*H*,3'*H*)-tetraone (2h).

Yield 2.15 g (93%), mp: 227-229 °C. ^{1}H NMR (300 MHz, $CDCl_3$): δ 1.23 (s, 3H, CH_3), 1.30 (s, 3H, CH_3), 2.30 (d, ^{2}J = 16.3 Hz, 1H, CH_2), 2.37 (d, ^{2}J = 16.3 Hz, 1H, CH_2), 2.65 (d, ^{2}J = 17.9 Hz, 1H, CH_2), 2.69 (s, 3H, CH_3), 2.77 (d, ^{2}J = 17.9 Hz, 1H, CH_2), 3.42 (s, 3H, CH_3), 4.63 (s, 1H, CH), 6.95 (d, ^{3}J = 7.8 Hz, 1H, CH Ar), 7.14 (s, 1H, CH Ar), 7.19 (t, ^{3}J = 7.8 Hz, 1H, CH Ar), 7.46 (d, ^{3}J = 7.8 Hz, 1H, CH Ar) ppm. ^{13}C NMR (75 MHz, $CDCl_3$): δ 28.5, 28.6, 28.9, 29.5, 34.5, 37.6, 51.4, 58.8, 90.5, 111.0, 122.9, 127.1, 130.2, 131.4, 132.2, 136.4, 149.9, 163.7, 166.3, 178.4, 193.0 ppm. MS (*m/z* relative intensity %): 462 [^{81}Br, M^+] (90), 460 [^{79}Br, M^+] (100), 378 (^{81}Br, 57), 376 (^{79}Br, 61), 350 (^{81}Br, 88), 348 (^{79}Br, 92), 279 (51), 269 (11), 220 (21), 182 (13), 127 (19), 83 (33), 55 (23). IR (KBr) ν = 3448, 2960, 2876, 1715, 1692, 1645, 1426, 1384, 1230, 1033 cm^{-1}. Anal. calcd for $C_{21}H_{21}BrN_2O_5$: C, 54.68; H, 4.59; Br, 17.32; N, 6.07%. Found: C, 54.53; H, 4.46; Br, 17.23; N, 5.96%.

2. ^{1}H and ^{13}C NMR spectra for compounds 2a-h

1',3'-Dimethyl-3-phenyl-3,5,6,7-tetrahydro-2'*H*,4*H*-spiro[benzofuran-2,5'-pyrimidine]-2',4,4',6'(1'*H*,3'*H*)-tetraone (2a).





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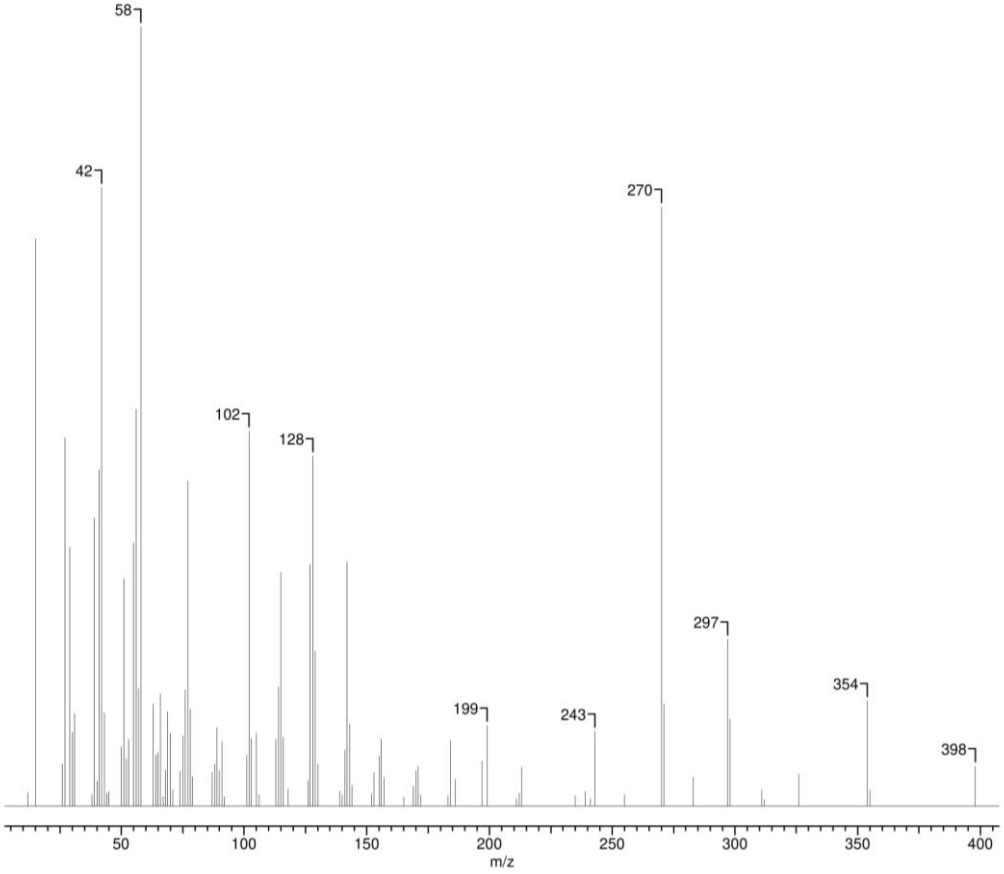

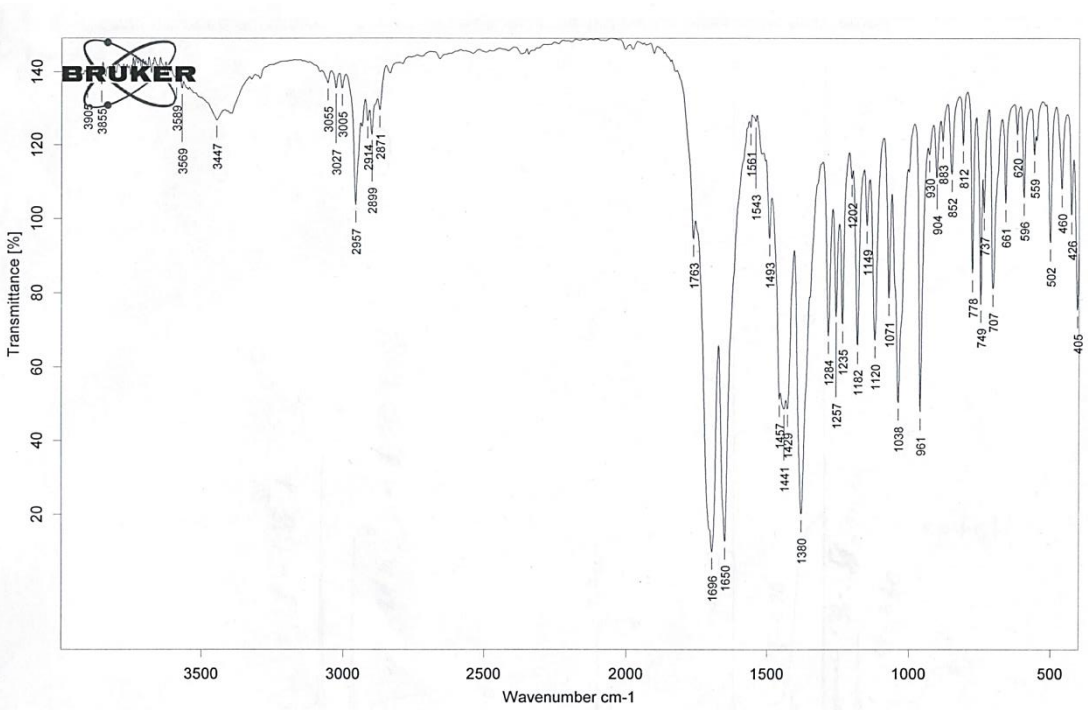

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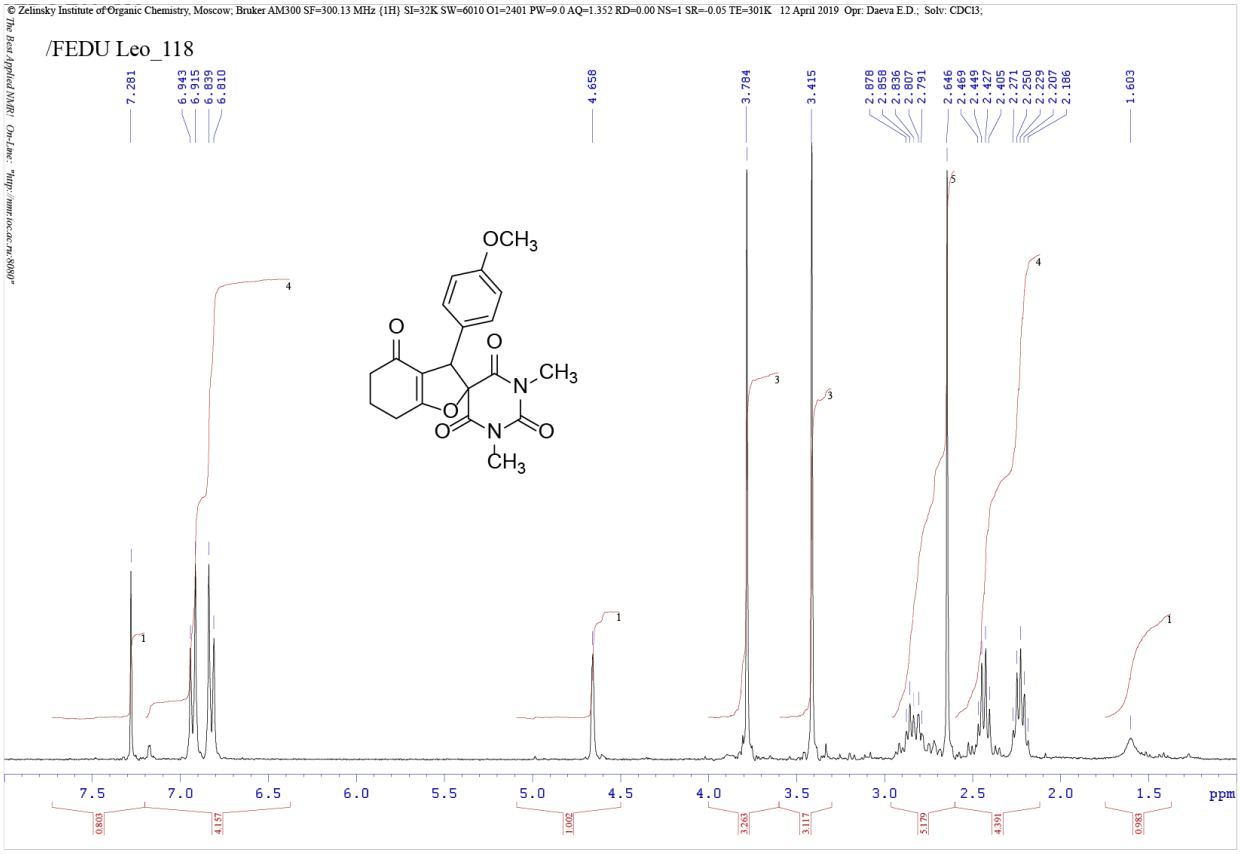
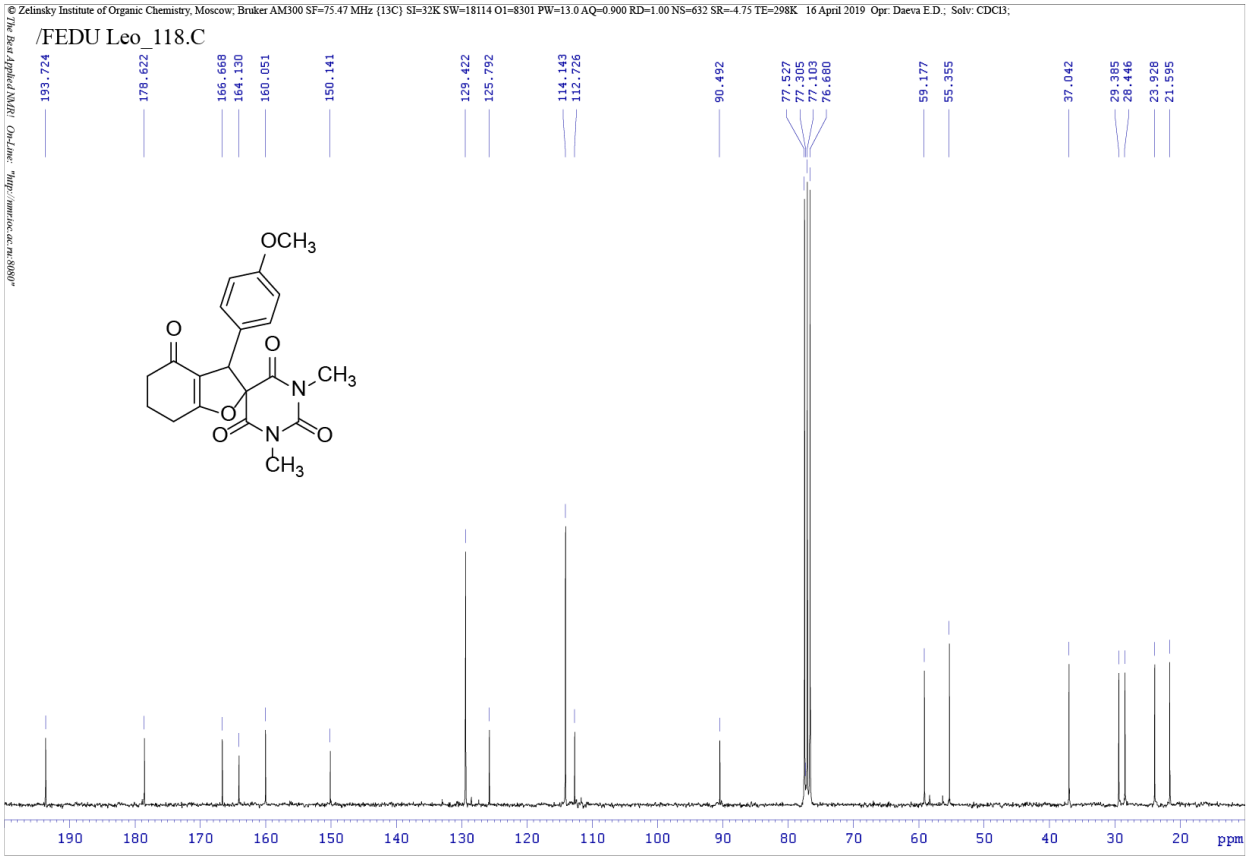
3-(4-Methoxyphenyl)-1',3'-dimethyl-3,5,6,7-tetrahydro-2'*H*,4*H*-spiro[benzofuran-2,5'-pyrimidine]-2',4,4',6'(1'*H*,3'*H*)-tetraone (2b).





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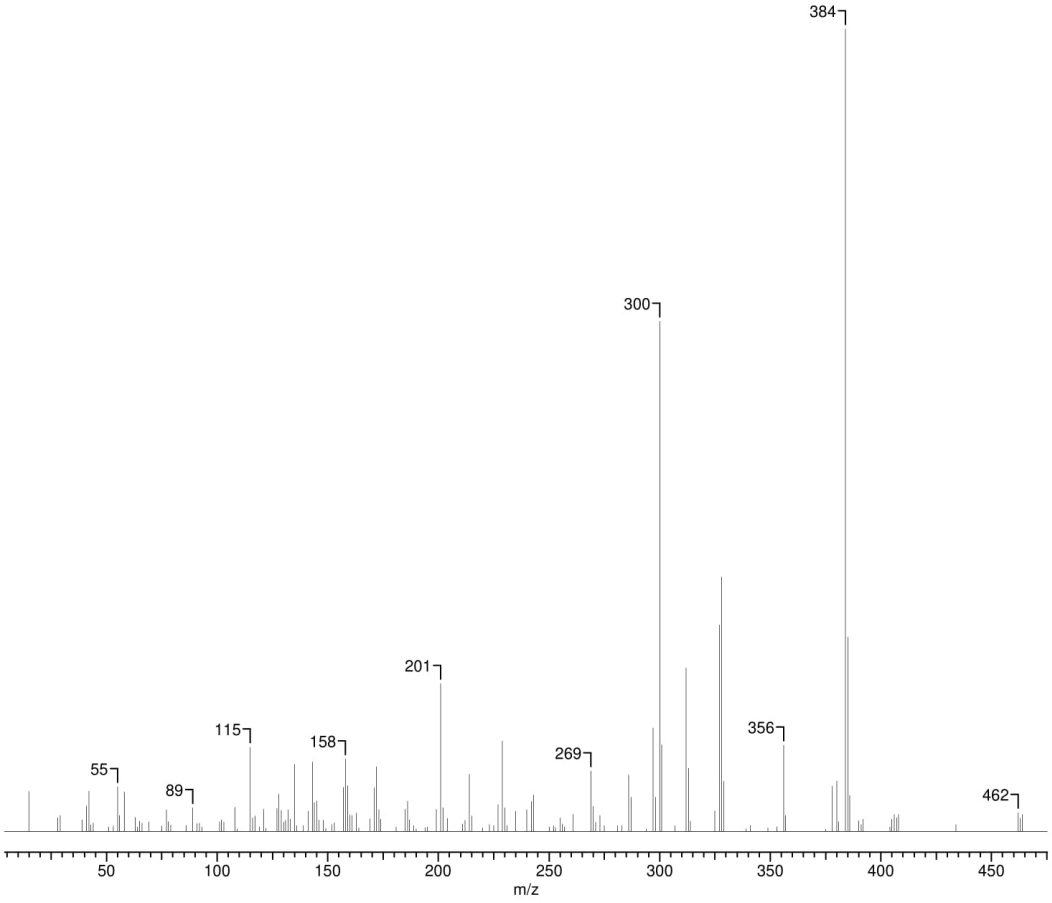

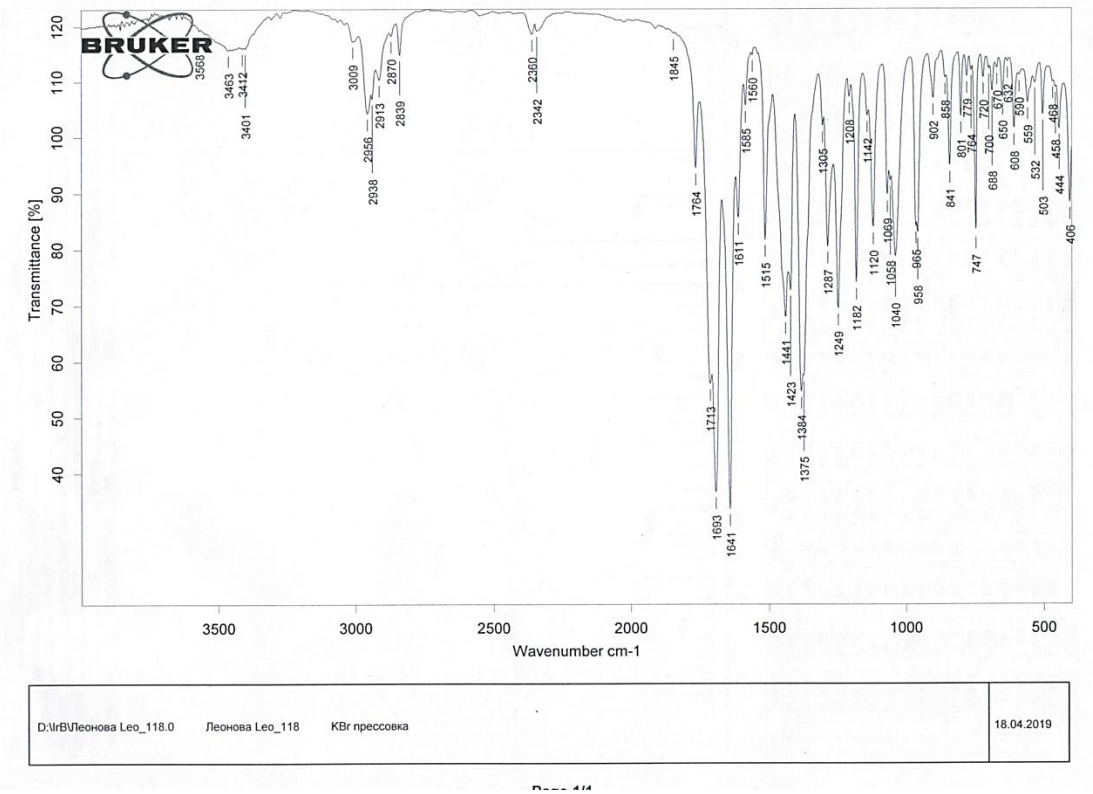

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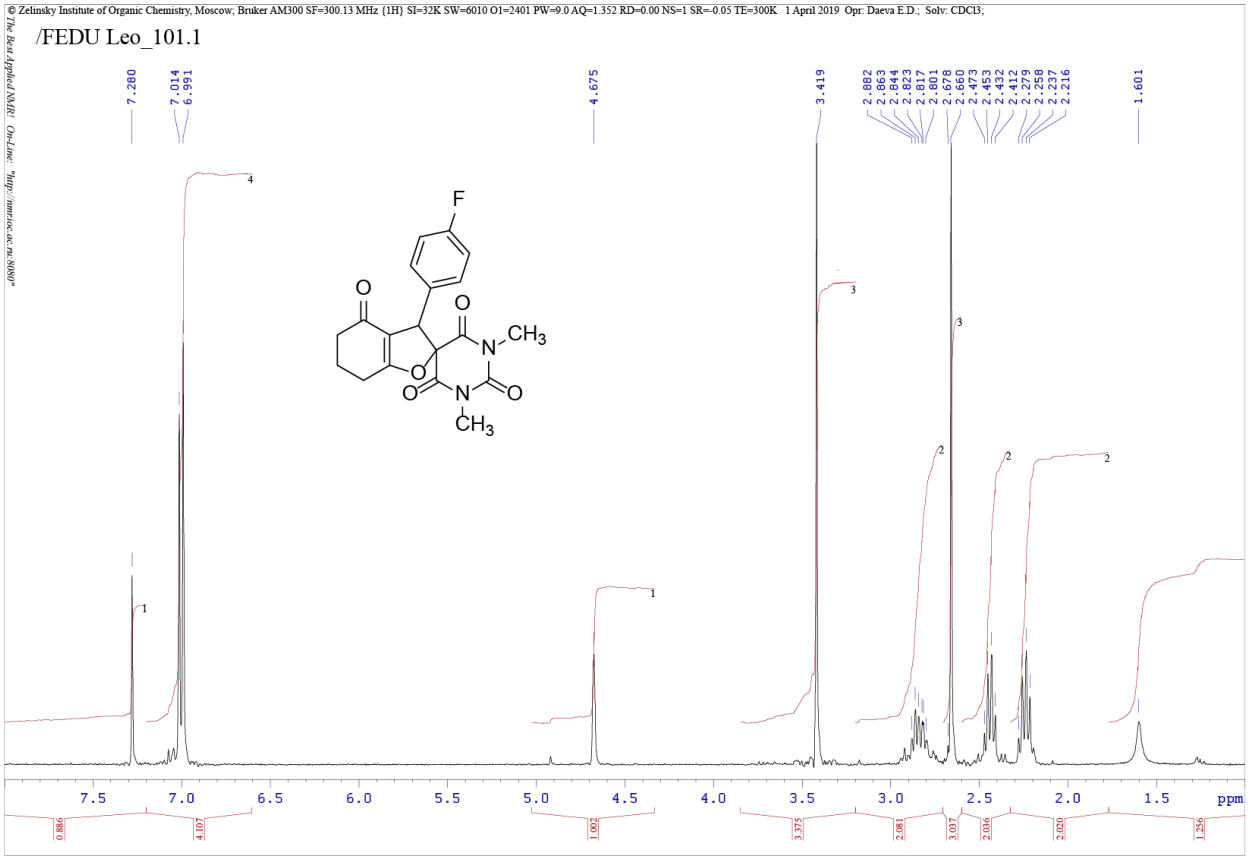
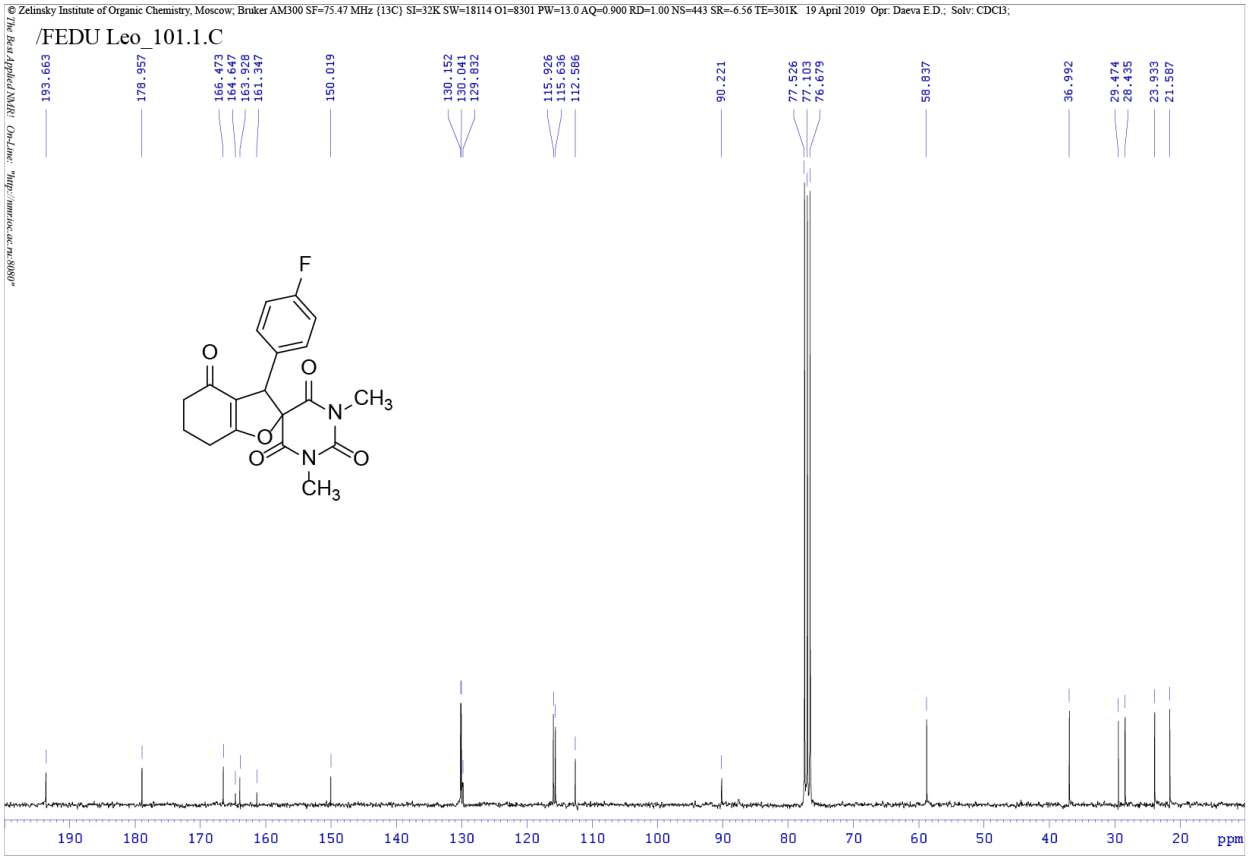
3-(4-Fluorophenyl)-1',3'-dimethyl-3,5,6,7-tetrahydro-2'*H*,4*H*-spiro[benzofuran-2,5'-pyrimidine]-2',4,4',6'(1'*H*,3'*H*)-tetraone (2c).





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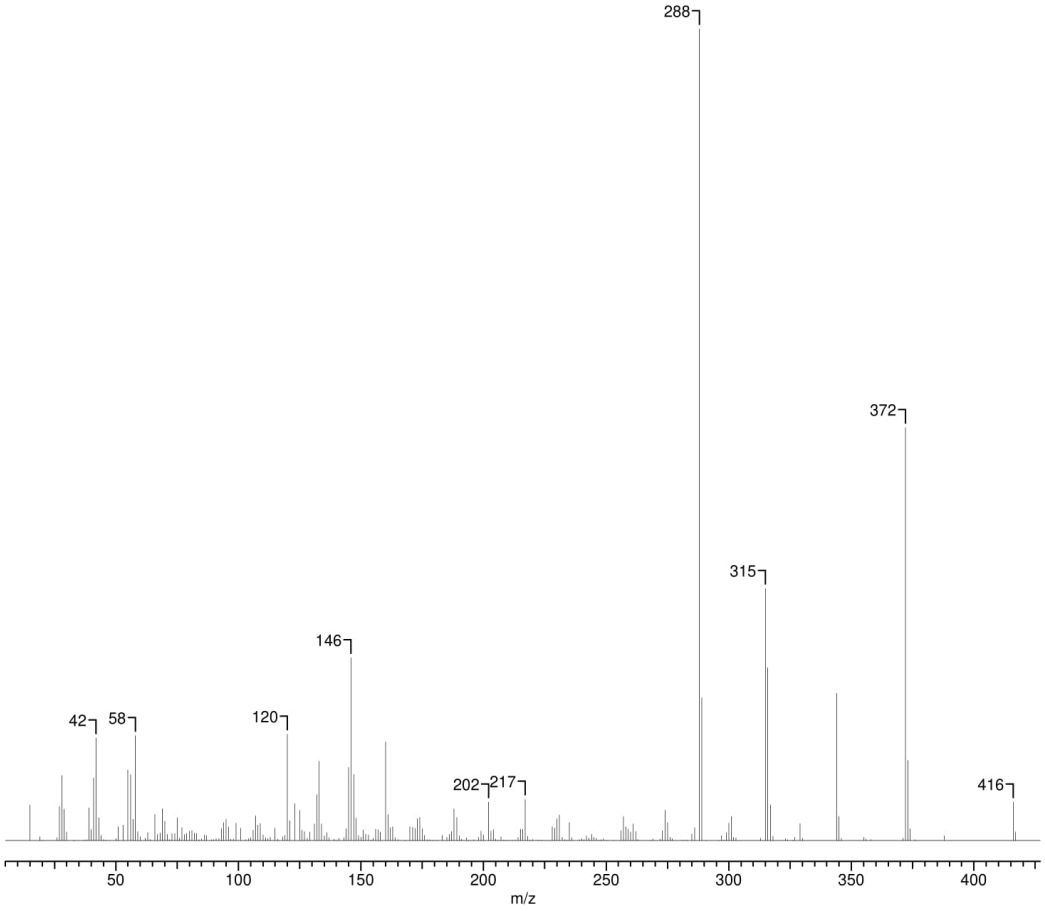

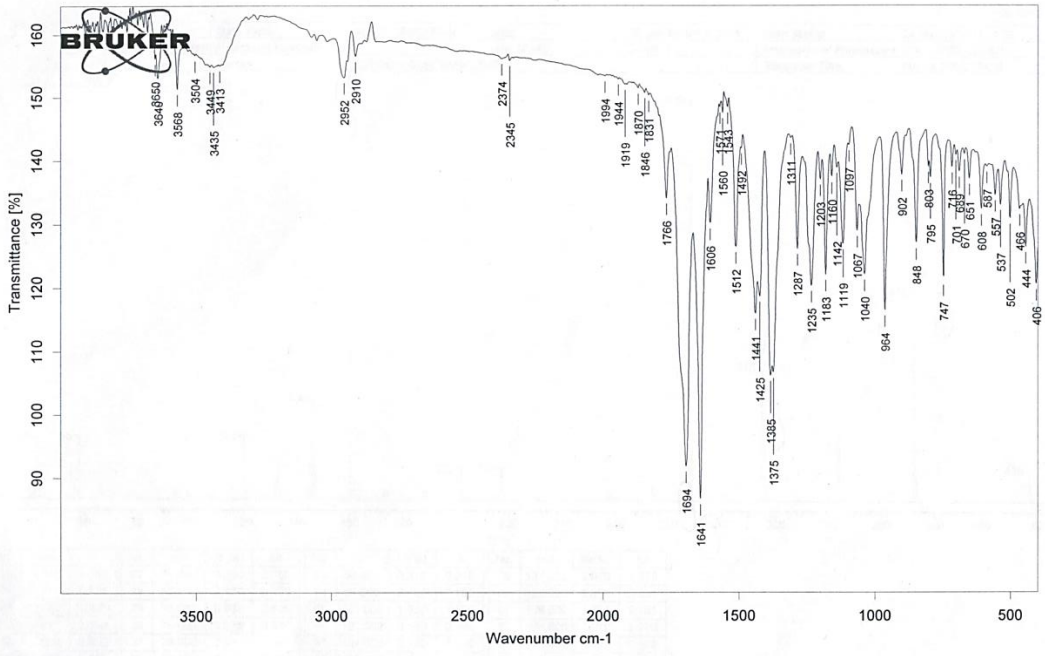

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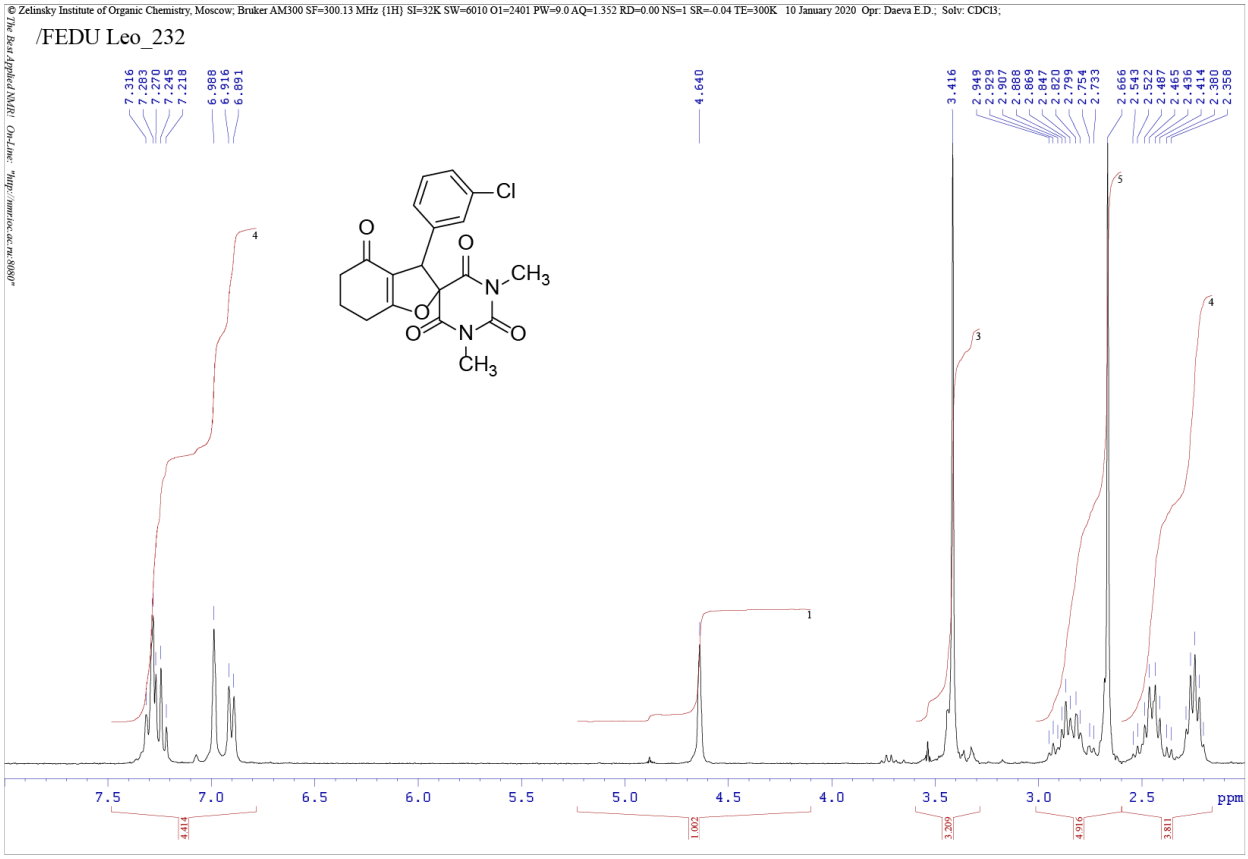
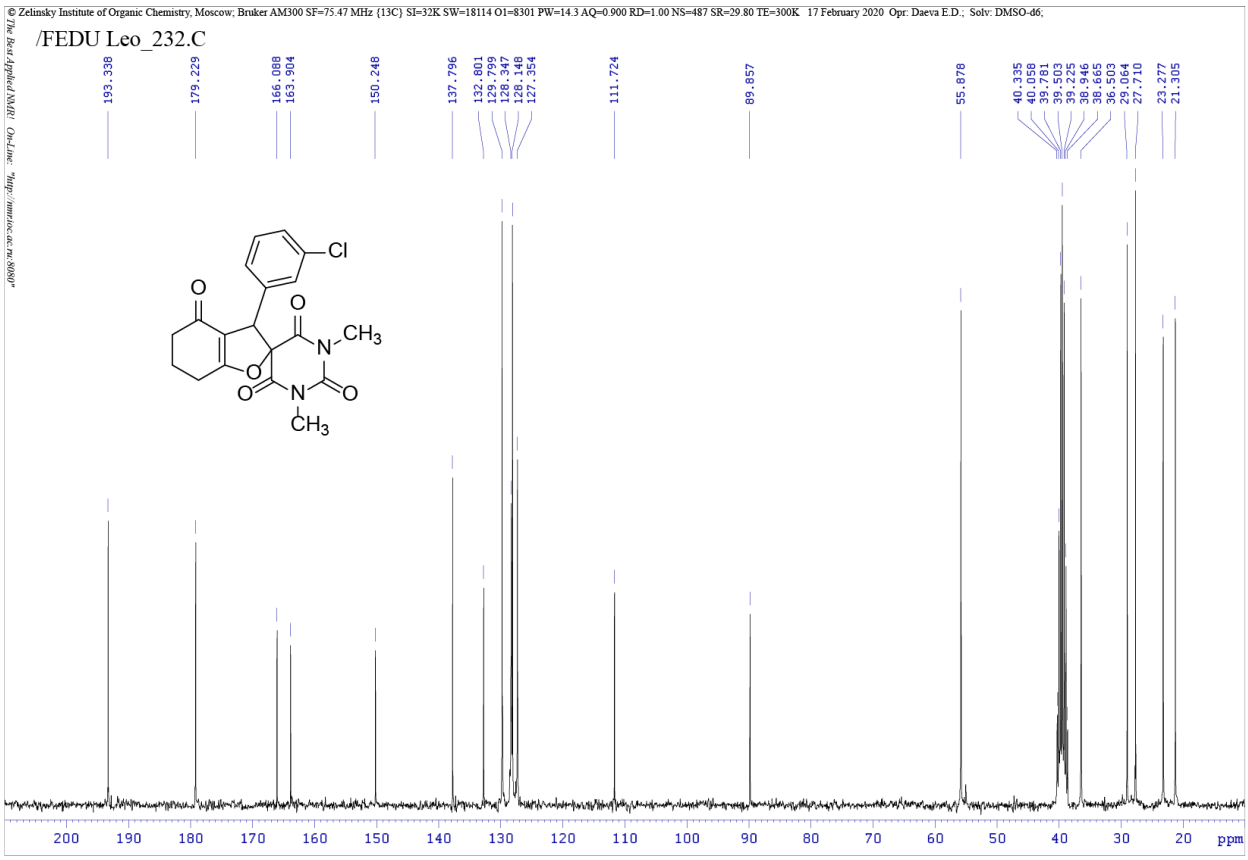
3-(3-Chlorophenyl)-1',3'-dimethyl-3,5,6,7-tetrahydro-2'*H*,4*H*-spiro[benzofuran-2,5'-pyrimidine]-2',4,4',6'(1'*H*,3'*H*)-tetraone (2d).





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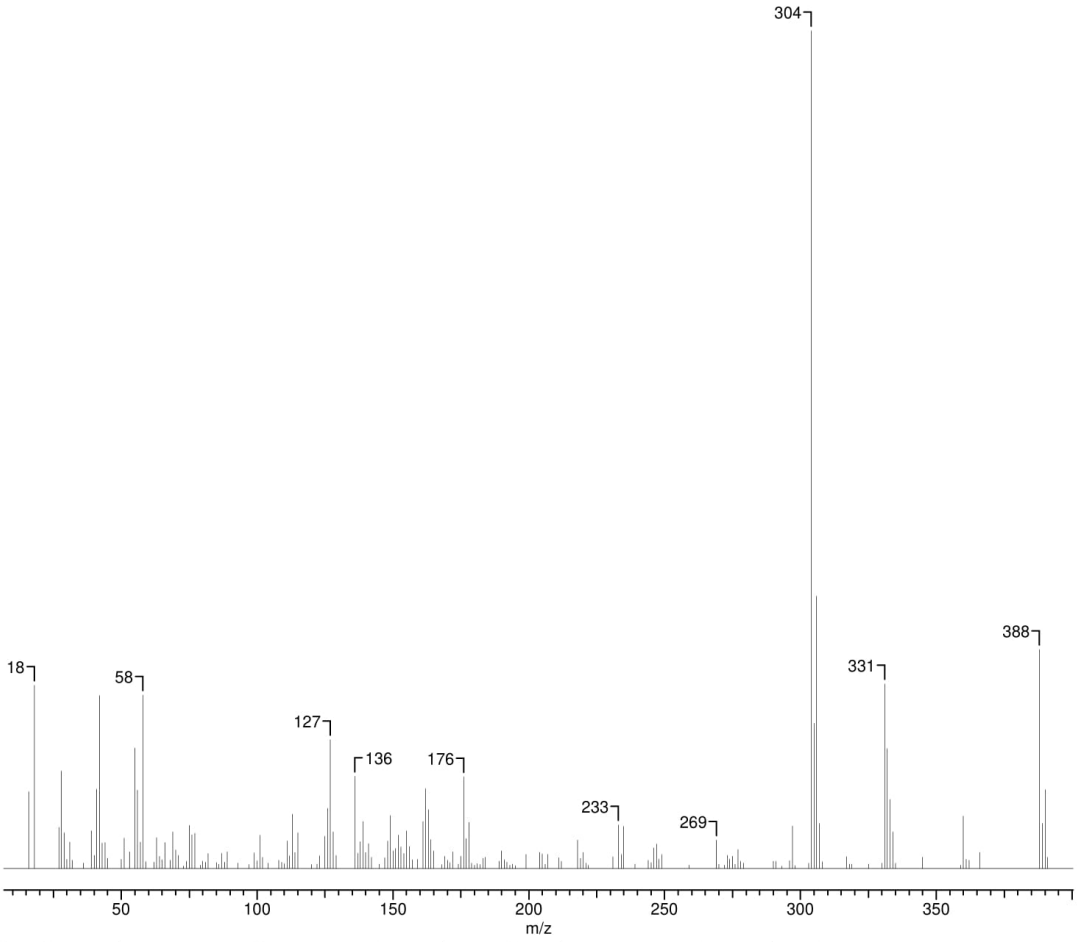
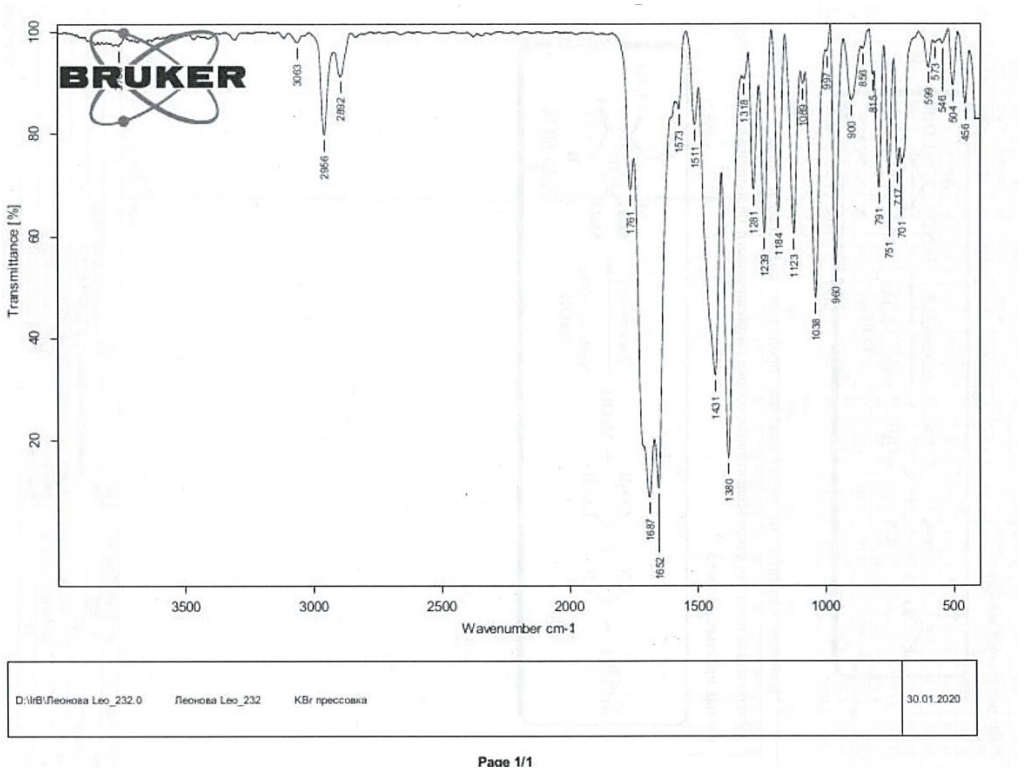
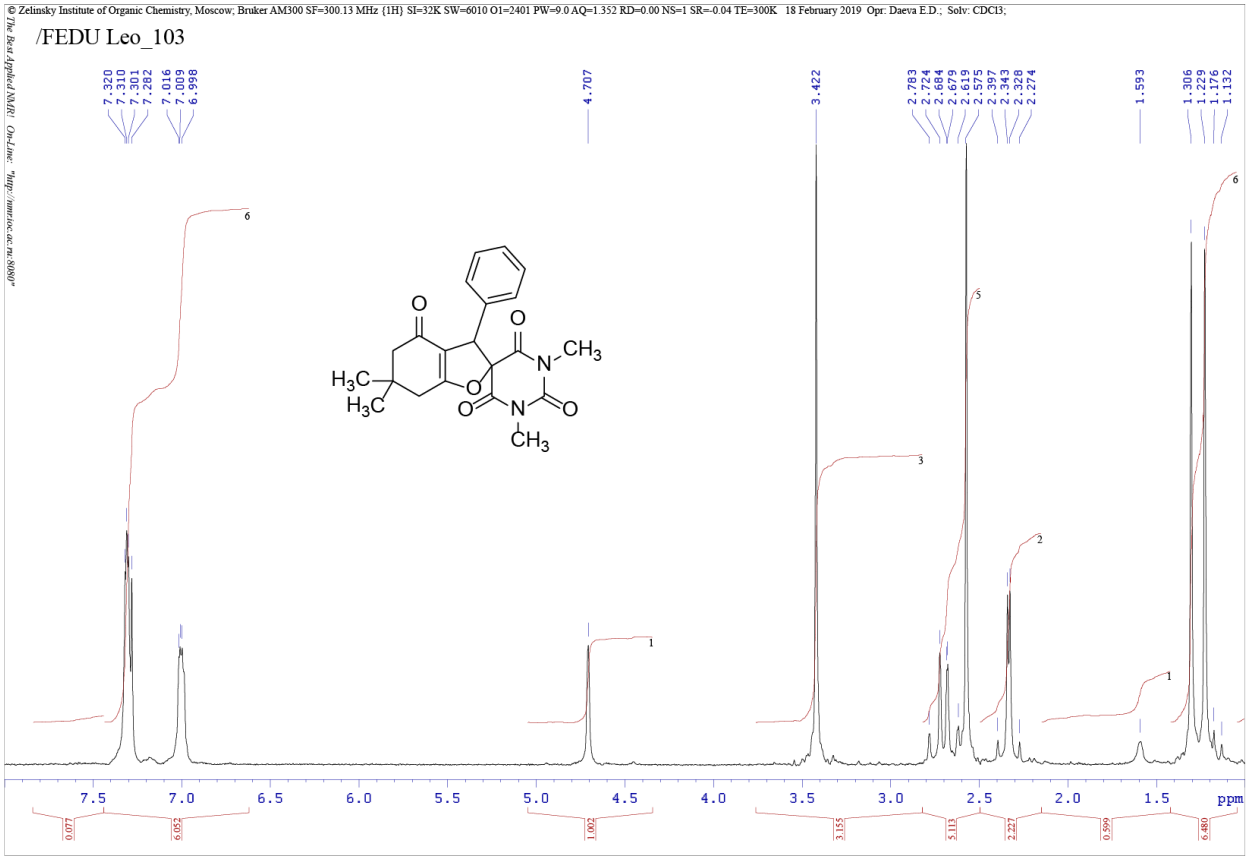
Spectrum Title	MASS SPECTRUM	***TIC***	721.86

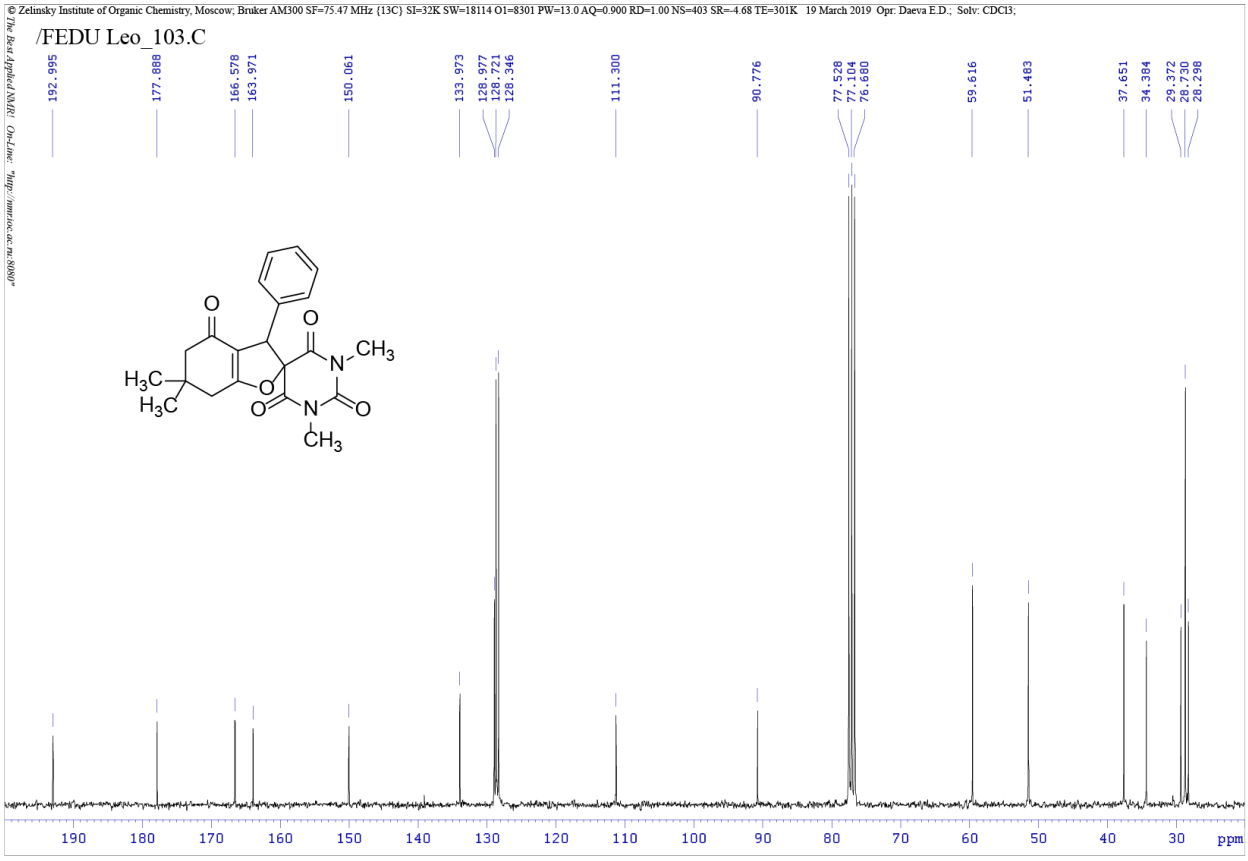




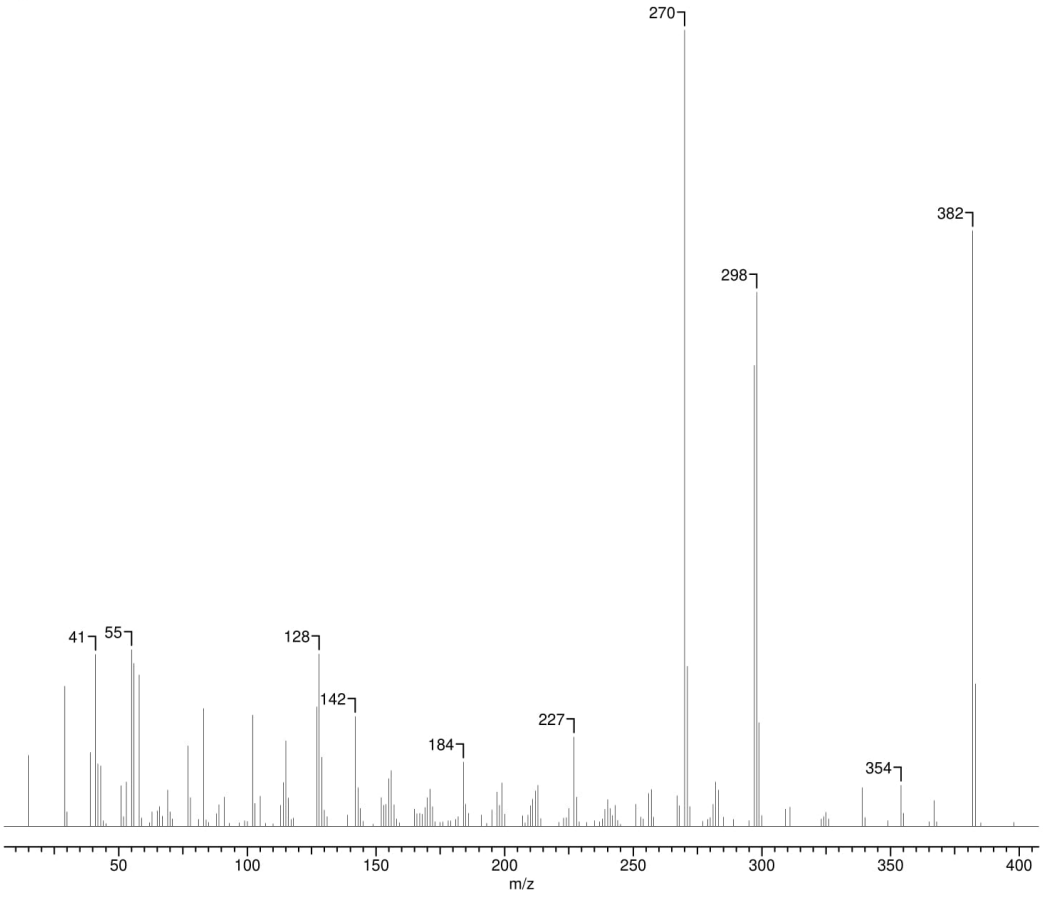
D:\IrB\Леонова Leo_232.0	Леонова Leo_232	KBr прессовка	30.01.2020

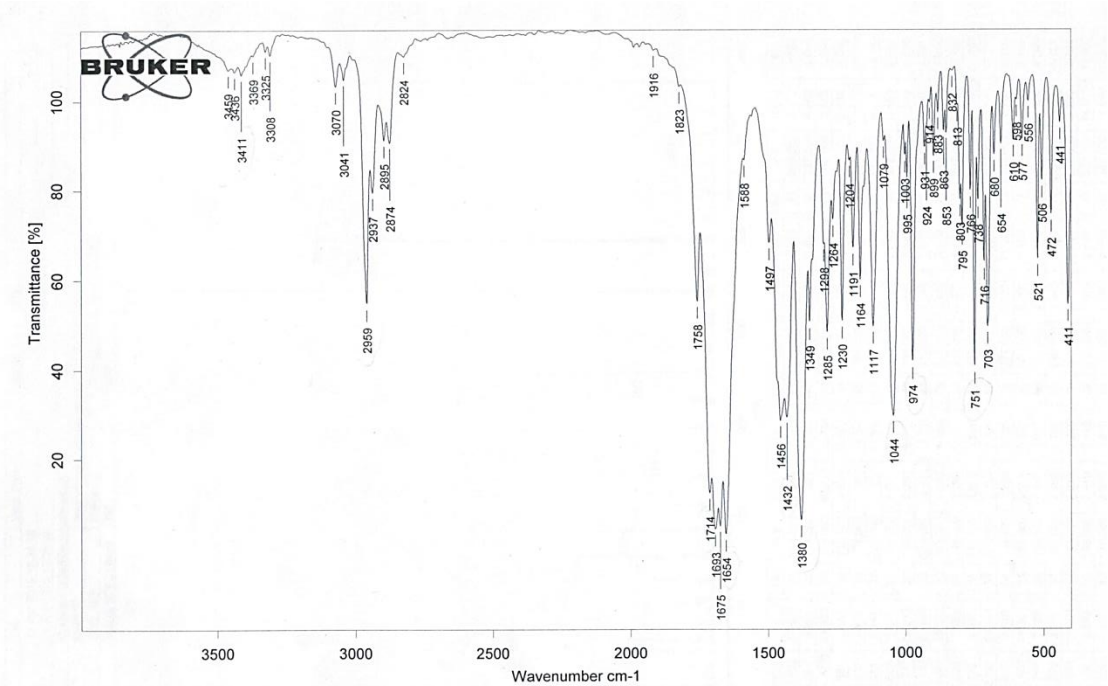
1',3',6,6-Tetramethyl-3-phenyl-3,5,6,7-tetrahydro-2'*H*,4*H*-spiro[benzofuran-2,5'-pyrimidine]-2',4,4',6'(1'*H*,3'*H*)-tetraone (2e).





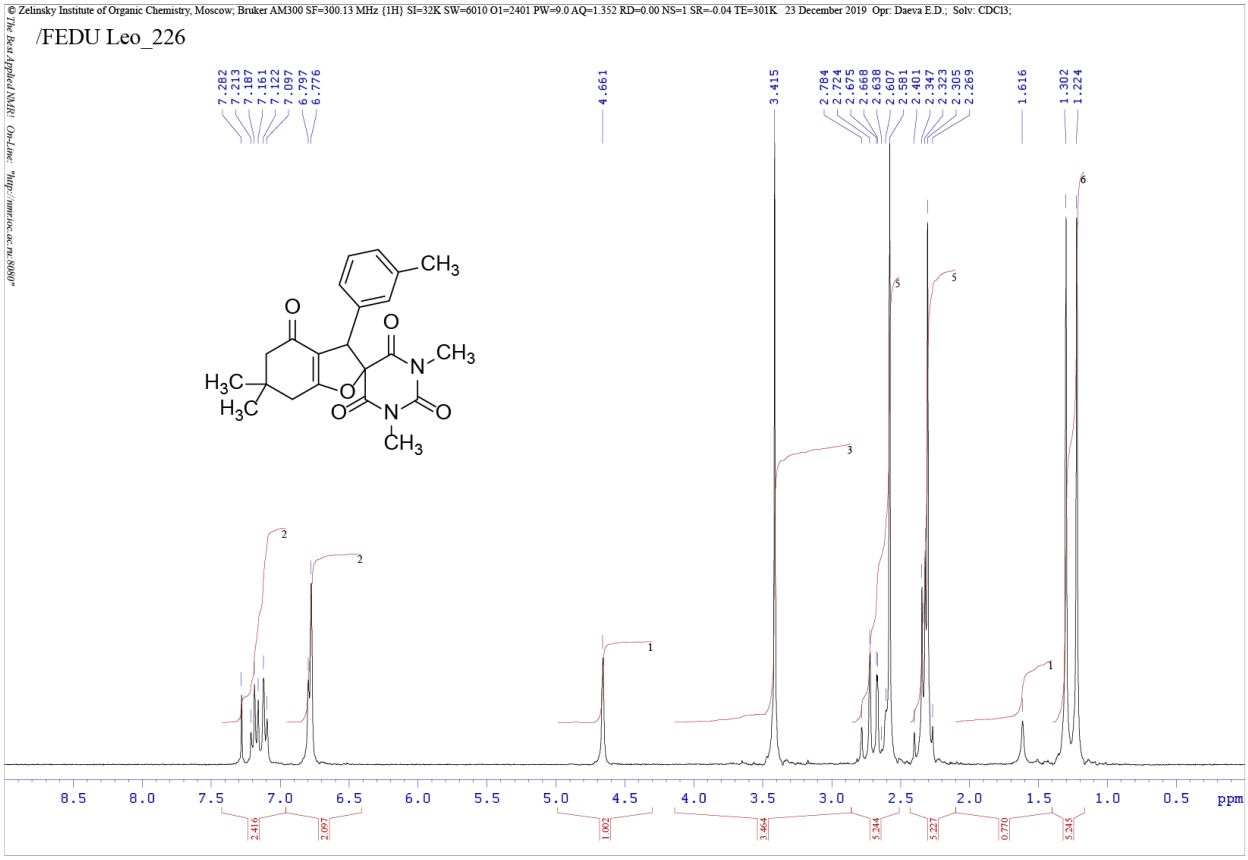
Count	160	***Data Type***	MASS SPECTRUM
Date	19 Apr 2019 15:35:48		
Date Stamp	02 Apr 1919 10:28:02		
File Name	C:\Users\12345\Desktop\ms4.JC		
Inlet Model	DIRECT	***Instrumental Parameters***	LOW RESOLUTION
Origin	LEO_103	***Owner***	Copyright(C) by Victor (2019)
Spectrum Title	MASS SPECTRUM	***TIC***	887.25

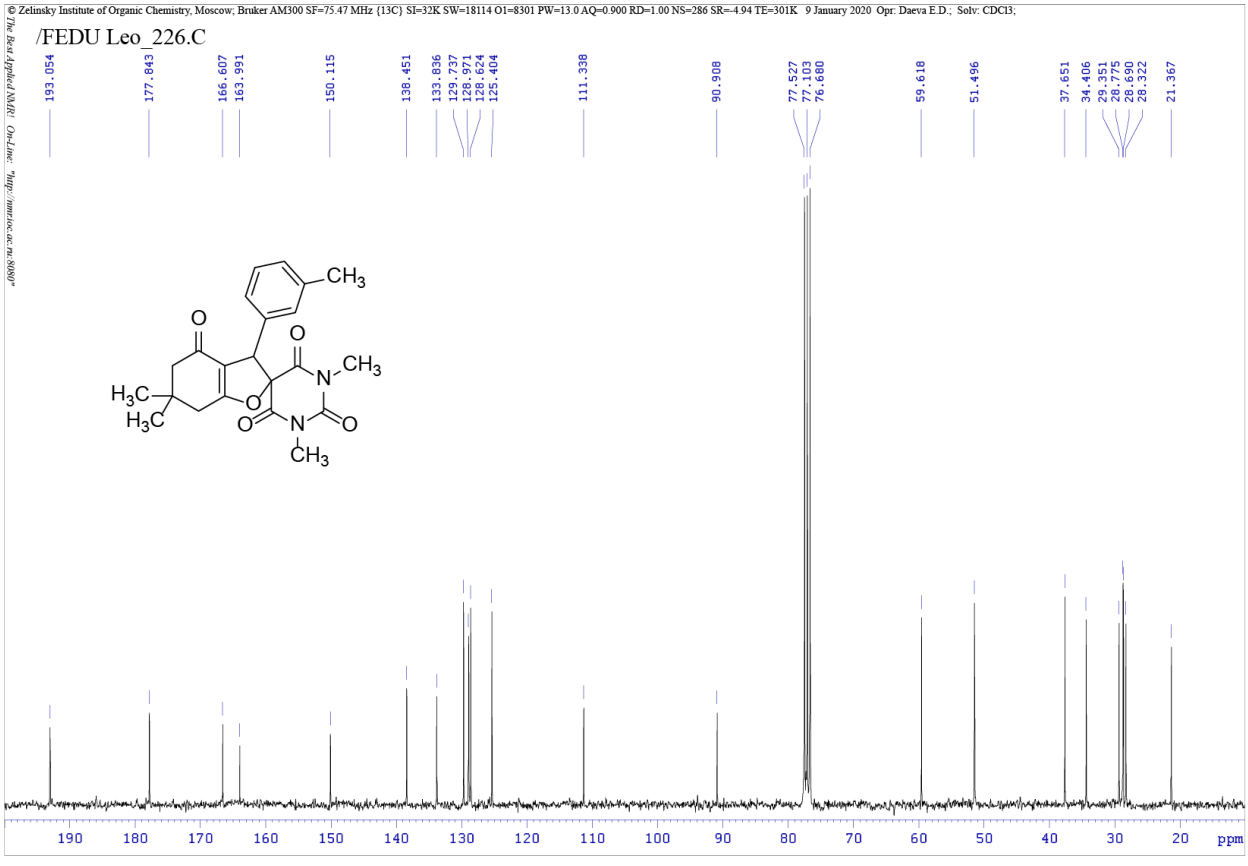




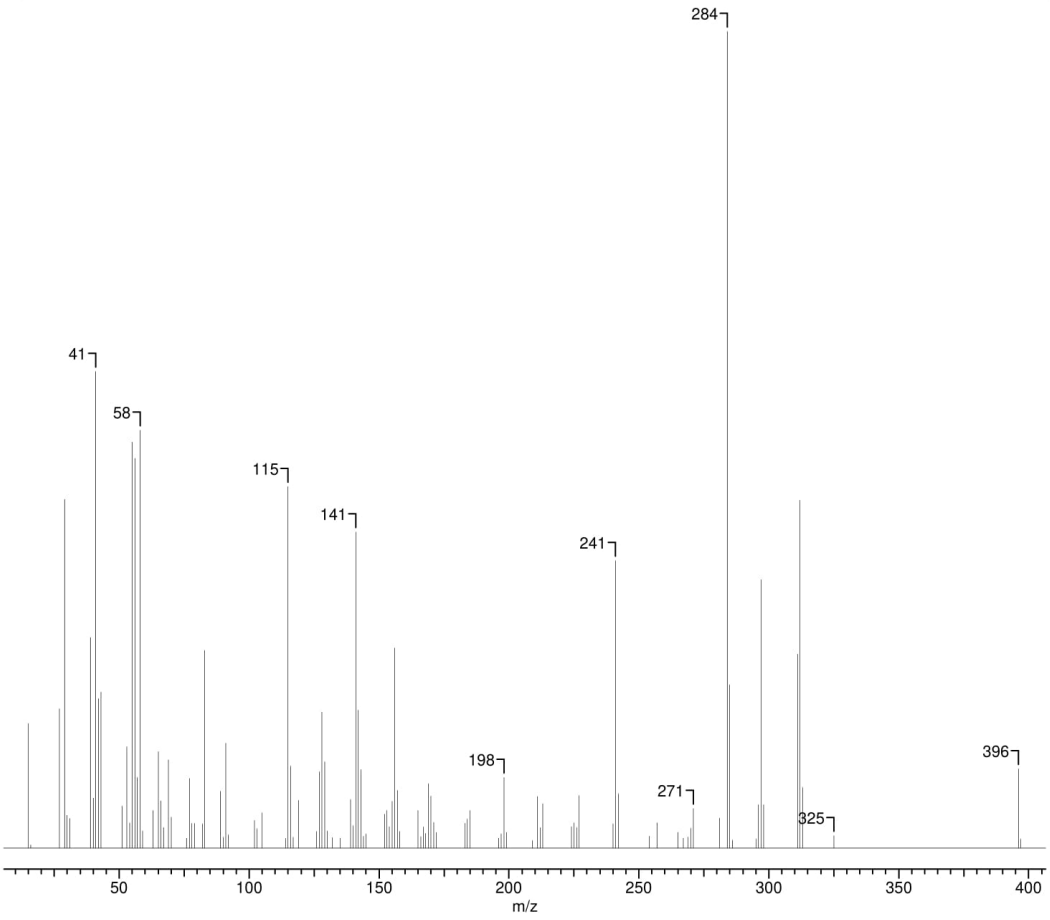
D:\IrB\Леонова Leo_103.0 Леонова Leo_103 KBr тонкий слой пленочка 18.04.2019

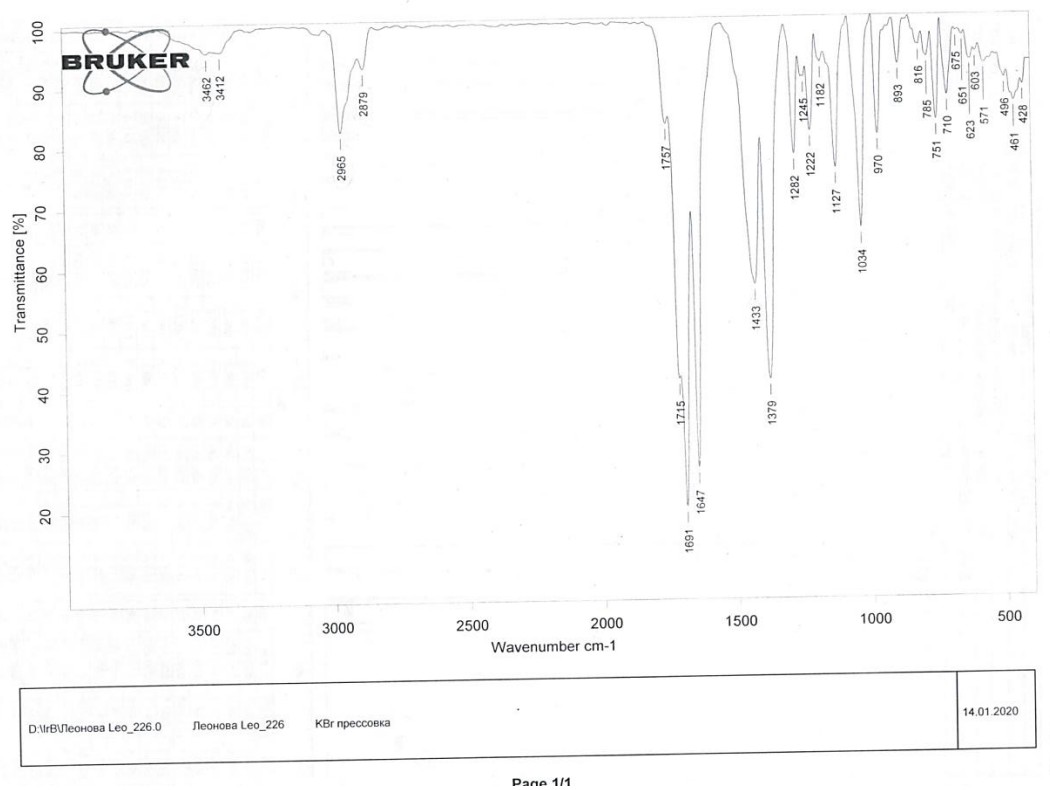
1',3',6,6-Tetramethyl-3-(*m*-tolyl)-3,5,6,7-tetrahydro-2'*H*,4*H*-spiro[benzofuran-2,5'-pyrimidine]-2',4,4',6'(1'*H*,3'*H*)-tetraone (2f).





Count	111	***Data Type***	MASS SPECTRUM
Date	10 Jan 2020 11:56:46	***Date Stamp***	09 Jan 1920 17:55:25
File Name	C:\Users\Natasha\Desktop\˝ ł \mass\225 226.JC		
Inlet Model	DIRECT	***Instrumental Parameters***	LOW RESOLUTION
Origin	LEO-226	***Owner***	Copyright(C) by Victor (2020)
Spectrum Title	MASS SPECTRUM	***TIC***	1142.46





D:\IrB\Леонова Leo_226.0	Леонова Leo_226	KBr прессовка	14.01.2020

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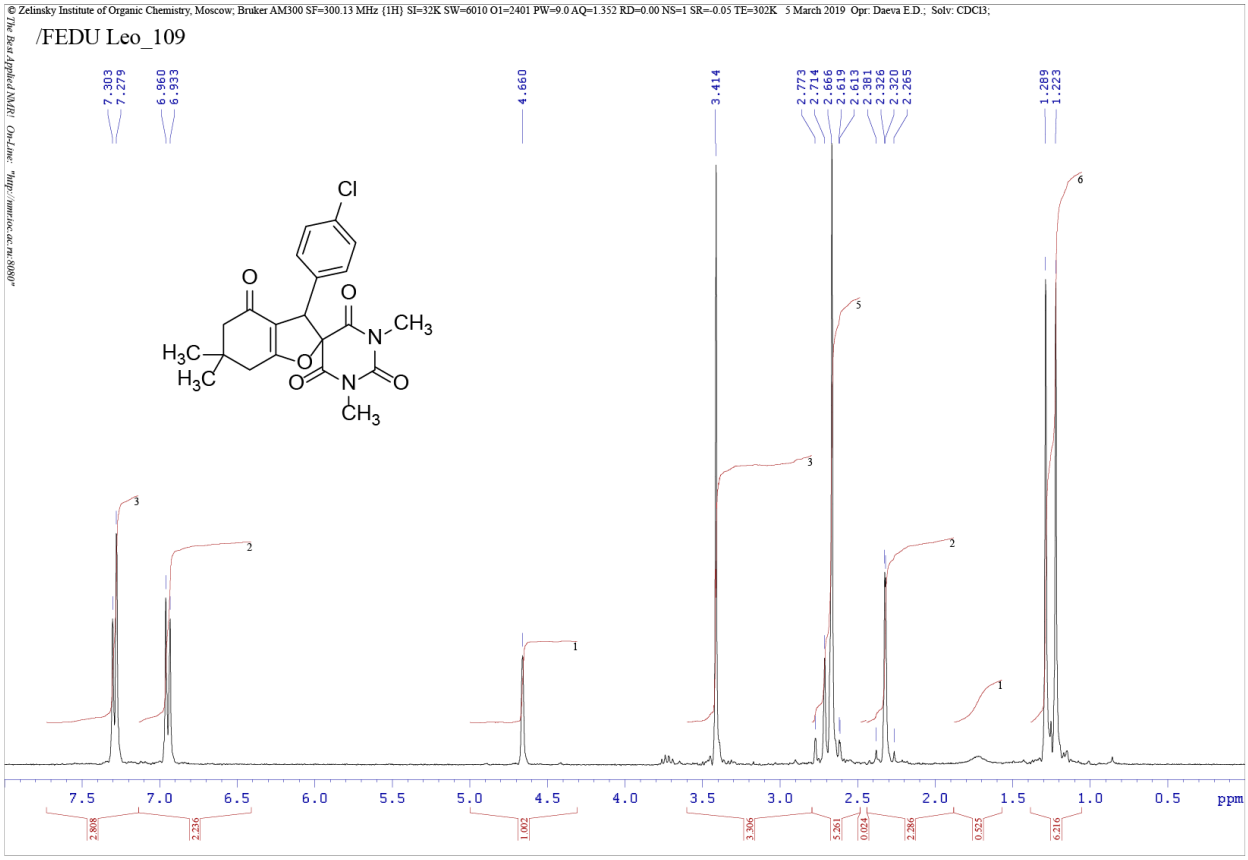
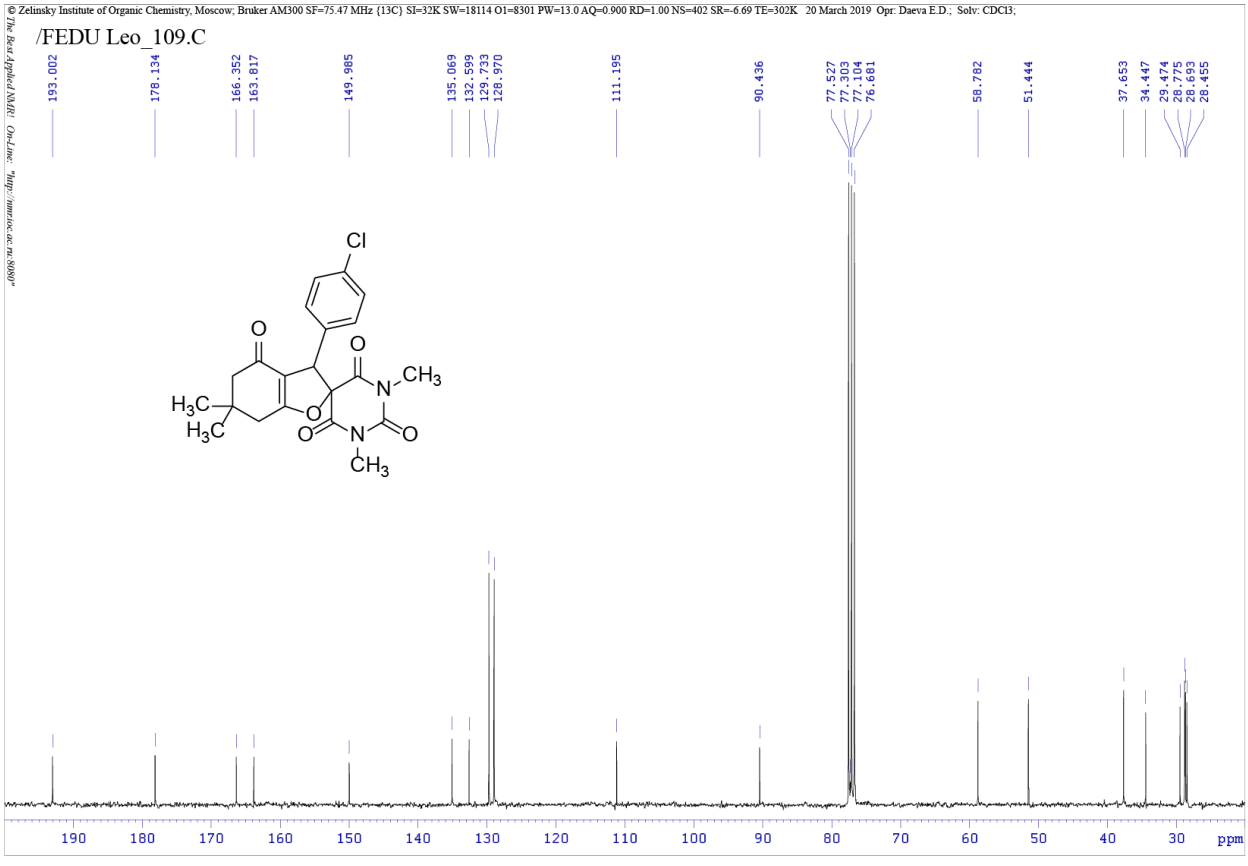
3-(4-Chlorophenyl)-1',3',6,6-tetramethyl-3,5,6,7-tetrahydro-2'*H*,4*H*-spiro[benzofuran-2,5'-pyrimidine]-2',4,4',6'(1'*H*,3'*H*)-tetraone (2g).





Count	142	***Data Type***	MASS SPECTRUM
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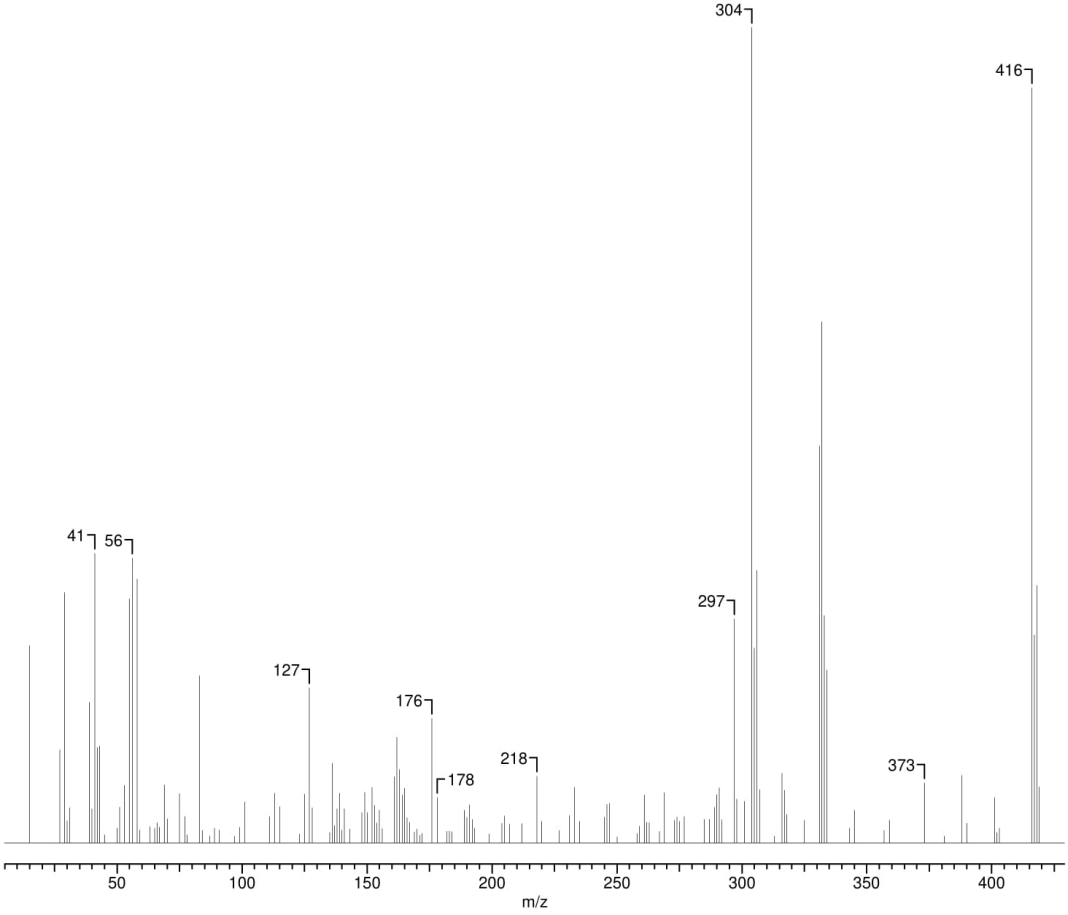
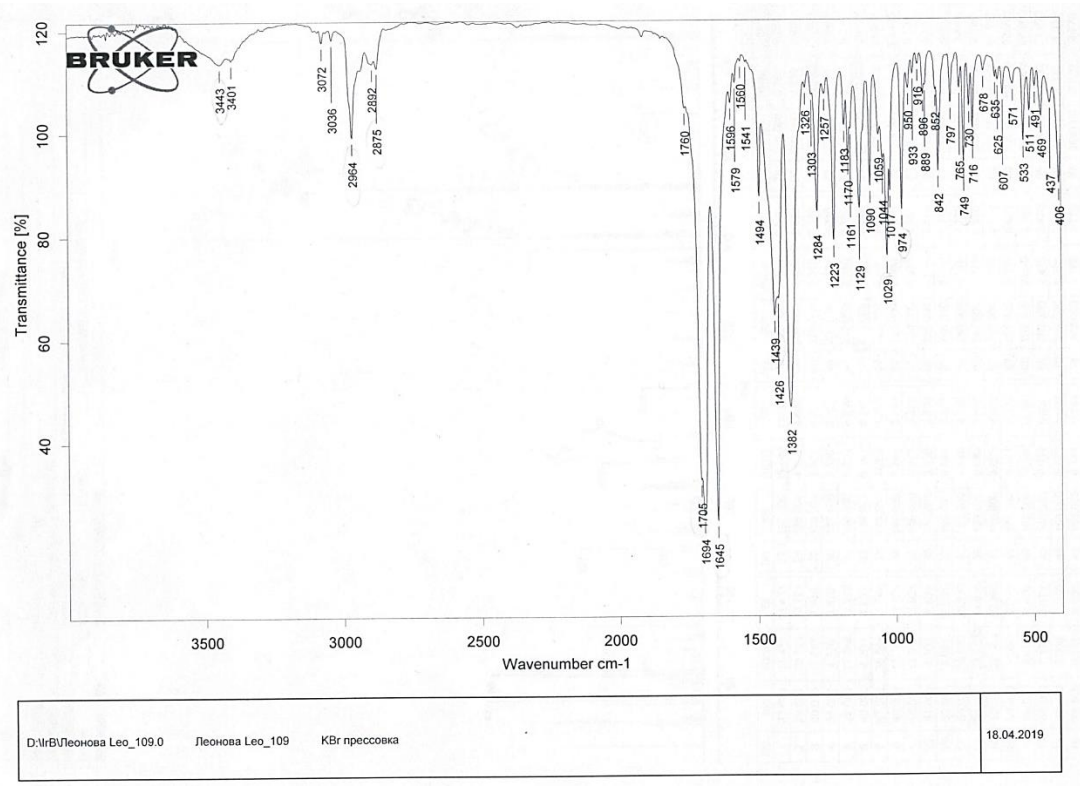
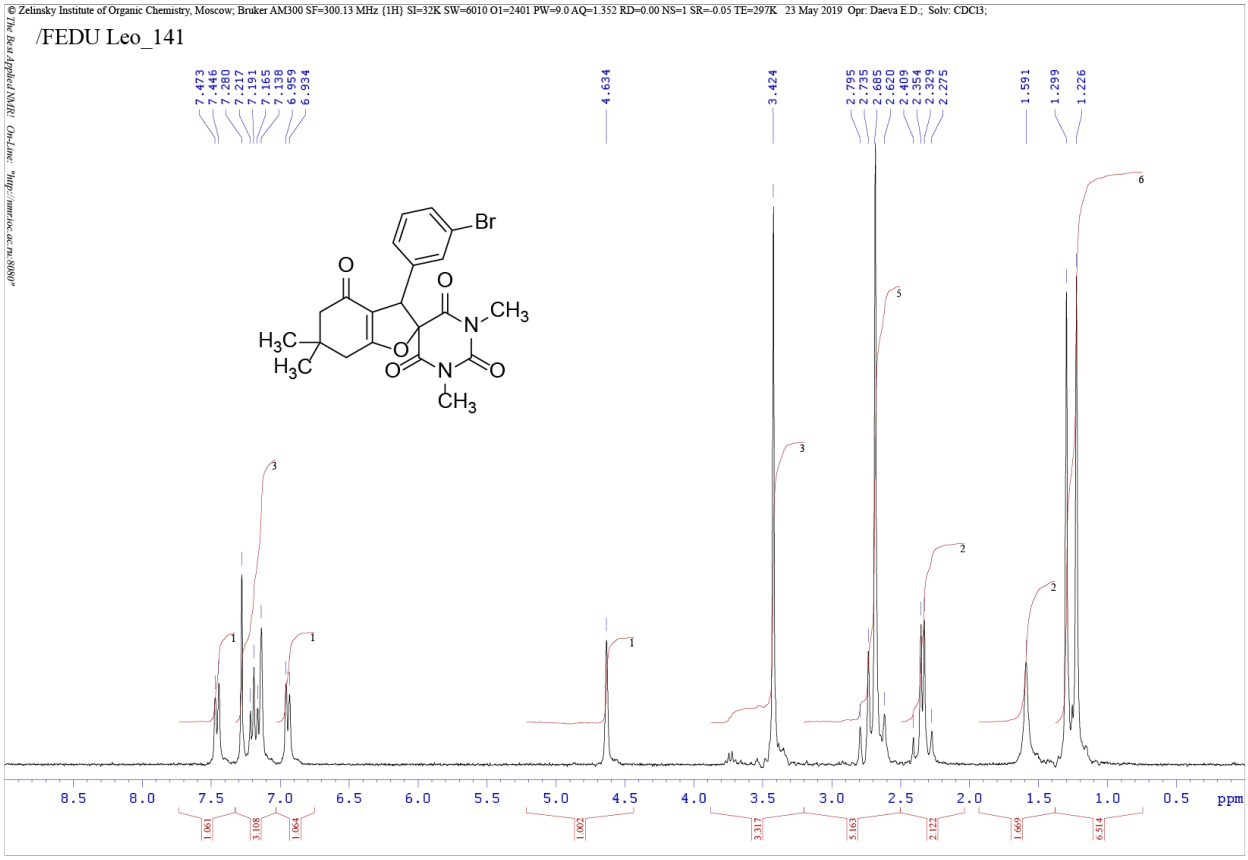
Date Stamp	02 Apr 1919 10:28:02		
File Name	C:\Users\12345\Desktop\ms4.JC		
Inlet Model	DIRECT	***Instrumental Parameters***	LOW RESOLUTION
Origin	LEO_109	***Owner***	Copyright(C) by Victor (2019)
Spectrum Title	MASS SPECTRUM	***TIC***	1225.67

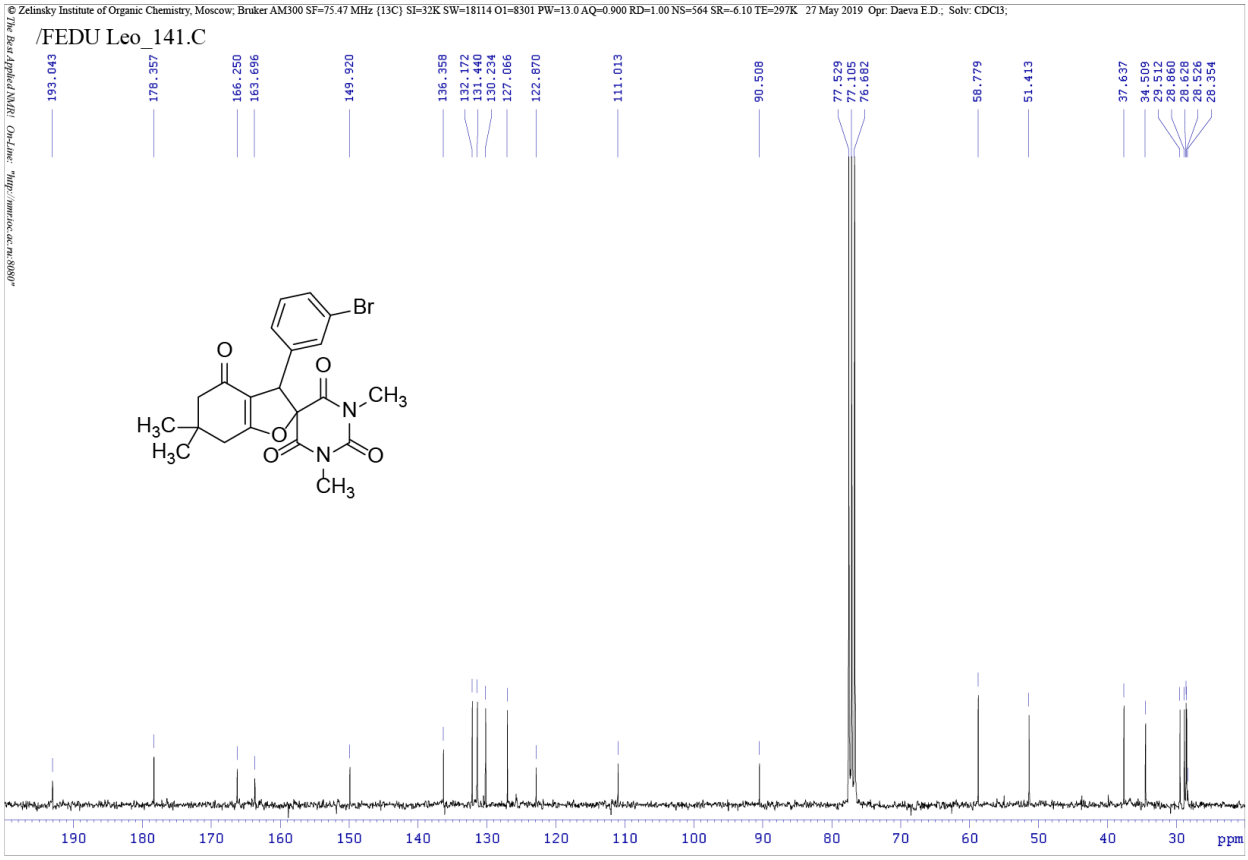




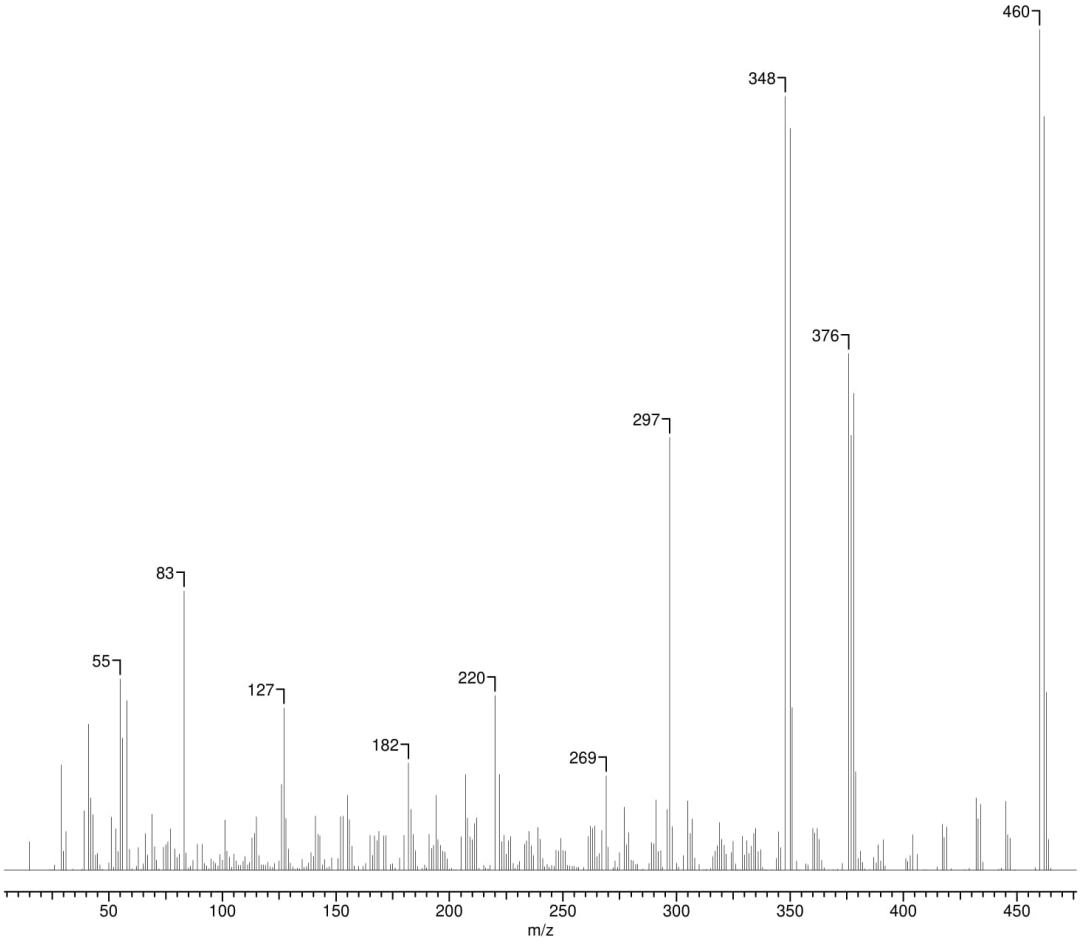
D:\IrB\Леонова Leo_109.0	Леонова Leo_109	KBr прессовка	18.04.2019

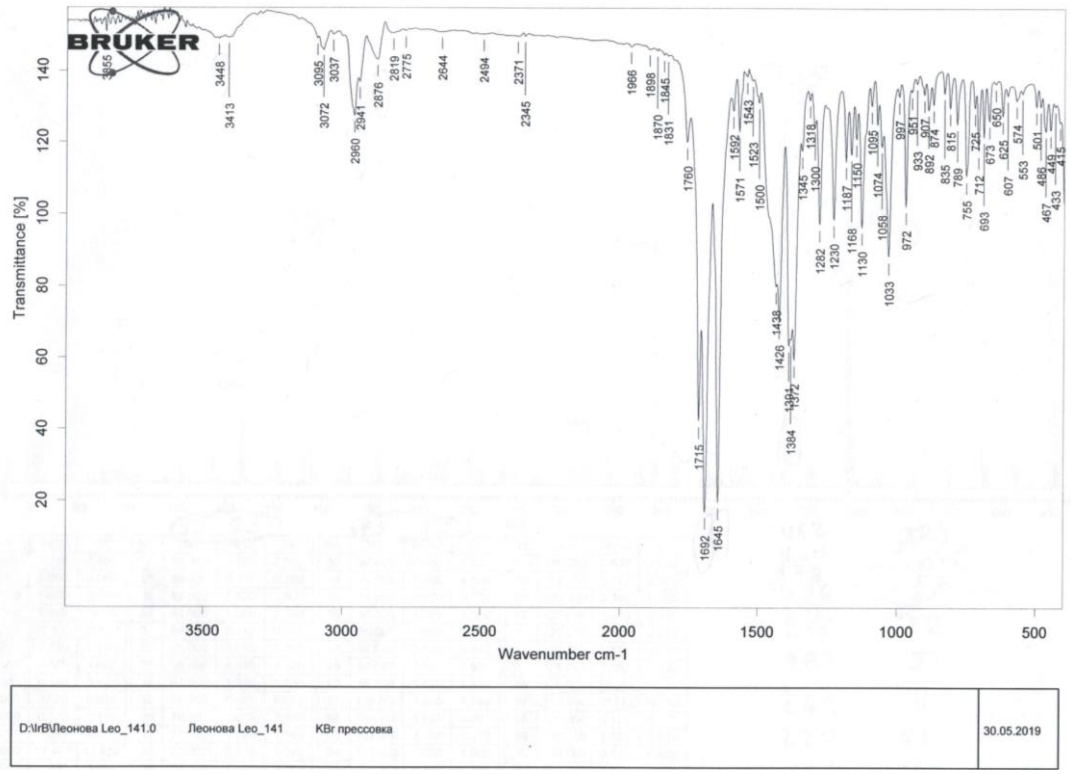
3-(3-Bromophenyl)-1',3',6,6-tetramethyl-3,5,6,7-tetrahydro-2'*H*,4*H*-spiro[benzofuran-2,5'-pyrimidine]-2',4,4',6'(1'*H*,3'*H*)-tetraone (2h).





Count	343	***Data Type***	MASS SPECTRUM
Date	26 Jun 2019 11:12:22	***Date Stamp***	24 May 1919 17:54:38
File Name	C:\Users\Natasha\Desktop\˜ Ł \mass\140 141.JC		
Inlet Model	DIRECT	***Instrumental Parameters***	LOW RESOLUTION
Origin	LEO_141	***Owner***	Copyright(C) by Victor (2019)
Spectrum Title	MASS SPECTRUM	***TIC***	1553.54





3. X-Ray data for compound 2h

Table 1. Crystal data and structure refinement for **2h**.

Identification code	**2h**	
Empirical formula	C21 H21 Br N2 O5	
Formula weight	461.31	
Temperature	100(2) K	
Wavelength	0.71073 Å	
Crystal system	Orthorhombic	
Space group	$Pna2_1$	
Unit cell dimensions	a = 19.5349(8) Å	α= 90°.
	b = 18.2072(7) Å	β= 90°.
	c = 5.5716(2) Å	γ = 90°.
Volume	1981.68(13) $Å^3$	
Z	4	
Density (calculated)	1.546 g/cm^3	
Absorption coefficient	2.111 mm^{-1}	
F(000)	944	
Crystal size	0.36 x 0.01 x 0.01 mm^3	
Theta range for data collection	2.085 to 34.649°.	
Index ranges	-31<=h<=31, -29<=k<=29, -8<=l<=8	
Reflections collected	72362	
Independent reflections	8361 [R(int) = 0.0618]	
Observed reflections	6508	
Completeness to theta = 25.242°	99.9 %	
Absorption correction	Semi-empirical from equivalents	
Max. and min. transmission	0.4967 and 0.4228	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	8361 / 1 / 277	
Goodness-of-fit on F^2	1.034	
Final R indices [I>2sigma(I)]	R1 = 0.0366, wR2 = 0.0715	
R indices (all data)	R1 = 0.0587, wR2 = 0.0783	
Absolute structure parameter	0.001(8)	
Largest diff. peak and hole	0.458 and -0.914 $e.Å^{-3}$	

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for **2h**. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
Br(1)	6561(1)	3784(1)	1805(1)	30(1)
O(1)	6878(1)	7306(1)	7697(3)	17(1)
O(2)	4991(1)	7139(1)	2749(3)	20(1)
O(3)	7430(1)	7846(1)	3601(4)	22(1)
O(4)	8630(1)	5734(1)	2532(4)	27(1)
O(5)	7252(1)	5941(1)	9014(3)	20(1)
N(1)	8022(1)	6790(1)	2961(4)	16(1)
N(2)	7862(1)	5771(1)	5584(4)	17(1)
C(1)	6233(1)	7539(1)	7199(4)	14(1)
C(2)	5952(1)	8159(1)	8614(4)	16(1)
C(3)	5392(1)	8554(1)	7163(4)	17(1)
C(4)	4904(1)	7970(1)	6099(5)	19(1)
C(5)	5257(1)	7394(1)	4569(4)	15(1)
C(6)	5926(1)	7177(1)	5410(4)	13(1)
C(7)	6385(1)	6580(1)	4438(4)	12(1)
C(8)	7070(1)	6801(1)	5838(4)	13(1)
C(9)	7526(1)	7209(1)	4082(4)	15(1)
C(10)	8193(1)	6072(1)	3600(5)	17(1)
C(11)	7404(1)	6146(1)	7010(5)	15(1)
C(12)	4986(1)	9067(2)	8805(5)	24(1)
C(13)	5719(2)	9008(1)	5157(5)	23(1)
C(14)	8403(1)	7128(2)	972(5)	21(1)
C(15)	8067(2)	5027(1)	6354(6)	30(1)
C(16)	6145(1)	5801(1)	4919(4)	12(1)
C(17)	6371(1)	5248(1)	3371(4)	14(1)
C(18)	6210(1)	4520(1)	3875(4)	18(1)
C(19)	5817(1)	4331(1)	5858(5)	19(1)
C(20)	5591(1)	4886(1)	7365(4)	19(1)
C(21)	5756(1)	5619(1)	6922(5)	16(1)

Table 3. Bond lengths [Å] and angles [°] for **2h**.

Br(1)-C(18)	1.896(2)
O(1)-C(1)	1.358(3)
O(1)-C(8)	1.435(3)
O(2)-C(5)	1.230(3)
O(3)-C(9)	1.205(3)
O(4)-C(10)	1.209(3)
O(5)-C(11)	1.215(3)
N(1)-C(9)	1.383(3)
N(1)-C(10)	1.395(3)
N(1)-C(14)	1.470(3)
N(2)-C(11)	1.379(3)
N(2)-C(10)	1.393(3)
N(2)-C(15)	1.476(3)
C(1)-C(6)	1.337(3)
C(1)-C(2)	1.482(3)
C(2)-C(3)	1.538(3)
C(2)-H(2A)	0.99(2)
C(2)-H(2B)	0.99(2)
C(3)-C(13)	1.530(3)
C(3)-C(12)	1.530(3)
C(3)-C(4)	1.546(3)
C(4)-C(5)	1.517(3)
C(4)-H(4A)	1.00(2)
C(4)-H(4B)	1.00(2)
C(5)-C(6)	1.443(3)
C(6)-C(7)	1.509(3)
C(7)-C(16)	1.518(3)
C(7)-C(8)	1.601(3)
C(7)-H(7)	0.98(3)
C(8)-C(11)	1.508(3)
C(8)-C(9)	1.517(3)
C(12)-H(12A)	1.03(2)
C(12)-H(12B)	1.03(2)
C(12)-H(12C)	1.03(2)
C(13)-H(13A)	0.99(2)
C(13)-H(13B)	0.99(2)
C(13)-H(13C)	0.99(2)
C(14)-H(14A)	0.92(2)

C(14)-H(14B)	0.92(2)
C(14)-H(14C)	0.92(2)
C(15)-H(15A)	0.91(2)
C(15)-H(15B)	0.91(2)
C(15)-H(15C)	0.91(2)
C(16)-C(21)	1.390(3)
C(16)-C(17)	1.398(3)
C(17)-C(18)	1.391(3)
C(17)-H(17)	0.97(4)
C(18)-C(19)	1.390(4)
C(19)-C(20)	1.386(4)
C(19)-H(19)	0.96(3)
C(20)-C(21)	1.395(3)
C(20)-H(20)	0.98(3)
C(21)-H(21)	0.95(3)

C(1)-O(1)-C(8)	107.21(17)
C(9)-N(1)-C(10)	124.7(2)
C(9)-N(1)-C(14)	117.6(2)
C(10)-N(1)-C(14)	117.6(2)
C(11)-N(2)-C(10)	124.3(2)
C(11)-N(2)-C(15)	117.6(2)
C(10)-N(2)-C(15)	117.8(2)
C(6)-C(1)-O(1)	114.47(19)
C(6)-C(1)-C(2)	127.3(2)
O(1)-C(1)-C(2)	118.18(19)
C(1)-C(2)-C(3)	109.83(19)
C(1)-C(2)-H(2A)	109.7
C(3)-C(2)-H(2A)	109.7
C(1)-C(2)-H(2B)	109.7
C(3)-C(2)-H(2B)	109.7
H(2A)-C(2)-H(2B)	108.2
C(13)-C(3)-C(12)	108.8(2)
C(13)-C(3)-C(2)	109.9(2)
C(12)-C(3)-C(2)	109.9(2)
C(13)-C(3)-C(4)	110.4(2)
C(12)-C(3)-C(4)	109.2(2)
C(2)-C(3)-C(4)	108.59(19)
C(5)-C(4)-C(3)	114.3(2)
C(5)-C(4)-H(4A)	108.7

C(3)-C(4)-H(4A)	108.7
C(5)-C(4)-H(4B)	108.7
C(3)-C(4)-H(4B)	108.7
H(4A)-C(4)-H(4B)	107.6
O(2)-C(5)-C(6)	123.2(2)
O(2)-C(5)-C(4)	122.1(2)
C(6)-C(5)-C(4)	114.7(2)
C(1)-C(6)-C(5)	120.9(2)
C(1)-C(6)-C(7)	110.88(19)
C(5)-C(6)-C(7)	128.2(2)
C(6)-C(7)-C(16)	115.27(18)
C(6)-C(7)-C(8)	98.10(17)
C(16)-C(7)-C(8)	114.02(18)
C(6)-C(7)-H(7)	109.6
C(16)-C(7)-H(7)	109.6
C(8)-C(7)-H(7)	109.6
O(1)-C(8)-C(11)	107.90(19)
O(1)-C(8)-C(9)	107.82(18)
C(11)-C(8)-C(9)	114.39(19)
O(1)-C(8)-C(7)	107.05(17)
C(11)-C(8)-C(7)	111.91(18)
C(9)-C(8)-C(7)	107.46(18)
O(3)-C(9)-N(1)	122.7(2)
O(3)-C(9)-C(8)	121.5(2)
N(1)-C(9)-C(8)	115.62(19)
O(4)-C(10)-N(2)	121.2(2)
O(4)-C(10)-N(1)	121.3(2)
N(2)-C(10)-N(1)	117.4(2)
O(5)-C(11)-N(2)	122.4(2)
O(5)-C(11)-C(8)	122.4(2)
N(2)-C(11)-C(8)	115.0(2)
C(3)-C(12)-H(12A)	109.5
C(3)-C(12)-H(12B)	109.5
H(12A)-C(12)-H(12B)	109.5
C(3)-C(12)-H(12C)	109.5
H(12A)-C(12)-H(12C)	109.5
H(12B)-C(12)-H(12C)	109.5
C(3)-C(13)-H(13A)	109.5
C(3)-C(13)-H(13B)	109.5
H(13A)-C(13)-H(13B)	109.5

C(3)-C(13)-H(13C)	109.5
H(13A)-C(13)-H(13C)	109.5
H(13B)-C(13)-H(13C)	109.5
N(1)-C(14)-H(14A)	109.5
N(1)-C(14)-H(14B)	109.5
H(14A)-C(14)-H(14B)	109.5
N(1)-C(14)-H(14C)	109.5
H(14A)-C(14)-H(14C)	109.5
H(14B)-C(14)-H(14C)	109.5
N(2)-C(15)-H(15A)	109.5
N(2)-C(15)-H(15B)	109.5
H(15A)-C(15)-H(15B)	109.5
N(2)-C(15)-H(15C)	109.5
H(15A)-C(15)-H(15C)	109.5
H(15B)-C(15)-H(15C)	109.5
C(21)-C(16)-C(17)	119.8(2)
C(21)-C(16)-C(7)	122.2(2)
C(17)-C(16)-C(7)	117.8(2)
C(18)-C(17)-C(16)	119.4(2)
C(18)-C(17)-H(17)	120.3
C(16)-C(17)-H(17)	120.3
C(19)-C(18)-C(17)	121.5(2)
C(19)-C(18)-Br(1)	120.55(17)
C(17)-C(18)-Br(1)	117.98(18)
C(20)-C(19)-C(18)	118.5(2)
C(20)-C(19)-H(19)	120.8
C(18)-C(19)-H(19)	120.8
C(19)-C(20)-C(21)	121.1(2)
C(19)-C(20)-H(20)	119.4
C(21)-C(20)-H(20)	119.4
C(16)-C(21)-C(20)	119.8(2)
C(16)-C(21)-H(21)	120.1
C(20)-C(21)-H(21)	120.1

Table 4. Anisotropic displacement parameters ($Å^2 x\ 10^3$) for **2h**. The anisotropic displacement factor exponent takes the form: $-2\pi^2[\ h^2\ a^{*2}U^{11} + ...\ + 2\ h\ k\ a^*\ b^*\ U^{12}\ ]$

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}

Br(1)	58(1)	13(1)	20(1)	-1(1)	3(1)	9(1)
O(1)	13(1)	20(1)	16(1)	-6(1)	-2(1)	3(1)
O(2)	18(1)	19(1)	23(1)	-3(1)	-6(1)	0(1)
O(3)	22(1)	14(1)	29(1)	2(1)	1(1)	-3(1)
O(4)	20(1)	29(1)	32(1)	0(1)	10(1)	6(1)
O(5)	18(1)	28(1)	15(1)	5(1)	-1(1)	-4(1)
N(1)	12(1)	17(1)	17(1)	3(1)	2(1)	-2(1)
N(2)	15(1)	16(1)	21(1)	3(1)	2(1)	3(1)
C(1)	15(1)	13(1)	15(1)	-1(1)	1(1)	1(1)
C(2)	18(1)	17(1)	14(1)	-4(1)	1(1)	1(1)
C(3)	18(1)	14(1)	18(1)	-2(1)	1(1)	3(1)
C(4)	14(1)	18(1)	26(1)	-3(1)	-2(1)	3(1)
C(5)	12(1)	13(1)	19(1)	1(1)	-1(1)	-2(1)
C(6)	12(1)	10(1)	15(1)	-1(1)	1(1)	-1(1)
C(7)	10(1)	13(1)	12(1)	0(1)	-1(1)	-1(1)
C(8)	12(1)	15(1)	13(1)	-1(1)	0(1)	0(1)
C(9)	13(1)	15(1)	17(1)	-1(1)	-1(1)	-3(1)
C(10)	12(1)	19(1)	19(1)	0(1)	1(1)	2(1)
C(11)	12(1)	17(1)	15(1)	1(1)	-1(1)	-2(1)
C(12)	22(1)	22(1)	28(1)	-8(1)	1(1)	5(1)
C(13)	35(2)	14(1)	20(1)	2(1)	0(1)	-4(1)
C(14)	18(1)	28(1)	17(1)	5(1)	2(1)	-4(1)
C(15)	25(1)	21(1)	44(2)	12(1)	5(1)	10(1)
C(16)	12(1)	12(1)	12(1)	1(1)	-1(1)	-1(1)
C(17)	17(1)	13(1)	13(1)	2(1)	0(1)	0(1)
C(18)	25(1)	13(1)	15(1)	-1(1)	-2(1)	2(1)
C(19)	22(1)	14(1)	22(1)	6(1)	-1(1)	-2(1)
C(20)	19(1)	21(1)	16(1)	5(1)	2(1)	-2(1)
C(21)	14(1)	16(1)	16(1)	1(1)	2(1)	-1(1)

Table 5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters ($Å^2$x 10^3) for **2h**.

	x	y	z	U(eq)
H(2A)	6323(8)	8508(8)	9006(10)	20

H(2B)	5758(4)	7973(4)	10130(30)	20
H(4A)	4660(6)	7717(6)	7450(30)	23
H(4B)	4550(8)	8225(6)	5100(20)	23
H(7)	6450(2)	6651(2)	2700(50)	14
H(12A)	4595(10)	9306(11)	7850(20)	36
H(12B)	5304(7)	9469(11)	9460(40)	36
H(12C)	4784(11)	8770(6)	10210(30)	36
H(13A)	5353(7)	9217(11)	4120(30)	35
H(13B)	6023(10)	8689(7)	4180(30)	35
H(13C)	5992(11)	9414(11)	5871(15)	35
H(14A)	8538(9)	6769(8)	-90(30)	32
H(14B)	8783(10)	7366(10)	1567(14)	32
H(14C)	8126(7)	7461(11)	190(30)	32
H(15A)	7727(9)	4825(7)	7260(40)	45
H(15B)	8458(11)	5055(2)	7240(40)	45
H(15C)	8143(11)	4742(7)	5040(30)	45
H(17)	6637(10)	5371(5)	1960(50)	17
H(19)	5704(4)	3829(16)	6177(11)	23
H(20)	5309(10)	4761(4)	8770(50)	22
H(21)	5602(5)	5994(13)	7990(40)	19

Table 6. Torsion angles [°] for **2h**.

C(8)-O(1)-C(1)-C(6)	8.9(3)
C(8)-O(1)-C(1)-C(2)	-169.9(2)
C(6)-C(1)-C(2)-C(3)	-23.4(3)
O(1)-C(1)-C(2)-C(3)	155.2(2)
C(1)-C(2)-C(3)-C(13)	-73.9(2)
C(1)-C(2)-C(3)-C(12)	166.4(2)
C(1)-C(2)-C(3)-C(4)	47.0(3)
C(13)-C(3)-C(4)-C(5)	64.4(3)
C(12)-C(3)-C(4)-C(5)	-175.9(2)
C(2)-C(3)-C(4)-C(5)	-56.1(3)
C(3)-C(4)-C(5)-O(2)	-143.0(2)
C(3)-C(4)-C(5)-C(6)	36.4(3)
O(1)-C(1)-C(6)-C(5)	-175.9(2)
C(2)-C(1)-C(6)-C(5)	2.8(4)
O(1)-C(1)-C(6)-C(7)	1.4(3)
C(2)-C(1)-C(6)-C(7)	-179.9(2)
O(2)-C(5)-C(6)-C(1)	170.7(2)
C(4)-C(5)-C(6)-C(1)	-8.7(3)
O(2)-C(5)-C(6)-C(7)	-6.0(4)
C(4)-C(5)-C(6)-C(7)	174.5(2)
C(1)-C(6)-C(7)-C(16)	111.9(2)
C(5)-C(6)-C(7)-C(16)	-71.1(3)
C(1)-C(6)-C(7)-C(8)	-9.6(2)
C(5)-C(6)-C(7)-C(8)	167.5(2)
C(1)-O(1)-C(8)-C(11)	-135.22(19)
C(1)-O(1)-C(8)-C(9)	100.8(2)
C(1)-O(1)-C(8)-C(7)	-14.6(2)
C(6)-C(7)-C(8)-O(1)	14.3(2)
C(16)-C(7)-C(8)-O(1)	-108.1(2)
C(6)-C(7)-C(8)-C(11)	132.3(2)
C(16)-C(7)-C(8)-C(11)	9.9(3)
C(6)-C(7)-C(8)-C(9)	-101.30(19)
C(16)-C(7)-C(8)-C(9)	136.32(19)
C(10)-N(1)-C(9)-O(3)	174.1(2)
C(14)-N(1)-C(9)-O(3)	-5.2(3)
C(10)-N(1)-C(9)-C(8)	-10.3(3)
C(14)-N(1)-C(9)-C(8)	170.4(2)
O(1)-C(8)-C(9)-O(3)	-36.0(3)

C(11)-C(8)-C(9)-O(3)	-156.0(2)
C(7)-C(8)-C(9)-O(3)	79.1(3)
O(1)-C(8)-C(9)-N(1)	148.38(19)
C(11)-C(8)-C(9)-N(1)	28.4(3)
C(7)-C(8)-C(9)-N(1)	-96.5(2)
C(11)-N(2)-C(10)-O(4)	174.1(2)
C(15)-N(2)-C(10)-O(4)	0.5(4)
C(11)-N(2)-C(10)-N(1)	-3.0(4)
C(15)-N(2)-C(10)-N(1)	-176.7(2)
C(9)-N(1)-C(10)-O(4)	179.5(2)
C(14)-N(1)-C(10)-O(4)	-1.3(4)
C(9)-N(1)-C(10)-N(2)	-3.3(3)
C(14)-N(1)-C(10)-N(2)	175.9(2)
C(10)-N(2)-C(11)-O(5)	-161.7(2)
C(15)-N(2)-C(11)-O(5)	11.9(4)
C(10)-N(2)-C(11)-C(8)	22.1(3)
C(15)-N(2)-C(11)-C(8)	-164.3(2)
O(1)-C(8)-C(11)-O(5)	30.0(3)
C(9)-C(8)-C(11)-O(5)	150.0(2)
C(7)-C(8)-C(11)-O(5)	-87.5(3)
O(1)-C(8)-C(11)-N(2)	-153.79(19)
C(9)-C(8)-C(11)-N(2)	-33.8(3)
C(7)-C(8)-C(11)-N(2)	88.7(2)
C(6)-C(7)-C(16)-C(21)	-29.1(3)
C(8)-C(7)-C(16)-C(21)	83.3(3)
C(6)-C(7)-C(16)-C(17)	155.7(2)
C(8)-C(7)-C(16)-C(17)	-91.9(2)
C(21)-C(16)-C(17)-C(18)	-0.8(3)
C(7)-C(16)-C(17)-C(18)	174.5(2)
C(16)-C(17)-C(18)-C(19)	1.4(4)
C(16)-C(17)-C(18)-Br(1)	-177.33(18)
C(17)-C(18)-C(19)-C(20)	-0.8(4)
Br(1)-C(18)-C(19)-C(20)	177.90(19)
C(18)-C(19)-C(20)-C(21)	-0.4(4)
C(17)-C(16)-C(21)-C(20)	-0.3(4)
C(7)-C(16)-C(21)-C(20)	-175.4(2)
C(19)-C(20)-C(21)-C(16)	0.9(4)

4. References

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