Electronic supplementary materials *Mendeleev Commun.*, 2021, **31**, 698–700

Four component tandem Knoevenagel–Michael strategy for the assembly of arylaldehydes, *N*,*N′*-dimethylbarbituric acid, 4-hydroxy-6-methyl-2*H*-pyran-2-one and morpholine into unsymmetrical scaffold with three different heterocyclic rings

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1. Experimental section

All melting points were measured with a Stuart SMP 30 melting point apparatus and are uncorrected. ^{1}H and ^{13}C NMR spectra were recorded with a Bruker AM-300 (300 and 75 MHz, respectively) and Bruker DRX 500 (500 and 126 MHz, respectively) at ambient temperature in 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 Kα-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 arylaldehyde 1 (2 mmol), *N, N*-dimethylbarbituric acid (0.312 g, 2 mmol), 4-hydroxy-6-methyl-2*H*-pyran-2-one (0.252 g, 2 mmol) and morpholine (0.174, 2 mmol) was stirred in ethanol (4 ml) at room temperature within 30 min. Then, the reaction mixture was filtered, the solid was rinsed with an ice-cold ethanol/water solution (1:1, 2 mL), and dried under reduced pressure to isolate final compound **2**.

Morpholin-4-ium 5-((4-hydroxy-6-methyl-2-oxo-2*H*-pyran-3-yl)(phenyl)methyl)-1,3-dimethyl-2,4-dioxo-1,2,3,4-tetrahydropyrimidin-6-olate (2a).

Yield 0.90 g (98%), mp: 202-203 °C. ^{1}H NMR (300 MHz, DMSO-d_6): δ 2.13 (s, 3H, CH_3), 3.08-3.14 (m, 10H, 2 NCH_2 + 2 CH_3), 3.76 (t, ^{3}J = 4.8 Hz, 4H, 2 CH_2), 5.80 (s, 1H, CH), 6.07 (s, 1H, CH), 6.97-7.06 (m, 3H, 3 CH Ar), 7.11-7.18 (m, 2H, 2 CH Ar), 8.69 (br s, 2H, NH_2^+), 15.06 (s, 1H, OH) ppm. ^{13}C NMR (75 MHz, DMSO-d_6): δ 19.4, 28.0 (2C), 35.0, 43.5 (2C), 63.8 (2C), 89.8, 103.3, 103.6, 124.9, 127.0 (2C), 128.0 (2C), 144.0, 152.5, 159.3, 163.8 (2C), 166.6, 169.3 ppm. MS (*m/z* relative intensity %): 370 [M - C_4H_9NO]$^+$ (58), 285 (6), 243 (10), 215 (12), 187 (4), 156 (6), 102 (13), 58 (52), 29 (100). IR (KBr) ν = 3435, 3365, 2955, 2804, 2734, 2625, 2470, 1875, 1451, 1104 cm^{-1}. Anal. calcd for $C_{23}H_{27}N_3O_7$: C, 60.39; H, 5.95; N, 9.19%. Found: C, 60.21; H, 5.97; N, 9.05%.

Morpholin-4-ium 5-((4-hydroxy-6-methyl-2-oxo-2*H*-pyran-3-yl)(4-methylphenyl)methyl)-1,3-dimethyl-2,4-dioxo-1,2,3,4-tetrahydropyrimidin-6-olate (2b).

Yield 0.89 g (94%), mp: 197-198 °C. ^{1}H NMR (300 MHz, DMSO-d_6): δ 2.12 (s, 3H, CH_3), 2.21 (s, 3H, CH_3), 3.06-3.17 (m, 10H, 2 NCH_2 + 2 CH_3), 3.76 (t, ^{3}J = 4.9 Hz, 4H, 2 CH_2), 5.79 (s, 1H, CH), 6.01 (s, 1H, CH), 6.87 (d, ^{3}J = 8 Hz, 2H, 2 CH Ar), 6.94 (d, ^{3}J = 8 Hz, 2H, 2 CH Ar), 8.70 (br s, 2H, NH_2^+), 15.04 (s, 1H, OH) ppm. ^{13}C NMR (75 MHz, DMSO-d_6): δ 19.4, 21.0, 28.0 (2C), 34.6, 43.5 (2C), 63.8 (2C), 90.0, 103.3, 103.7, 126.9 (2C), 128.6 (2C), 133.5, 144.9, 152.5, 159.2, 163.8 (2C), 166.6, 169.2 ppm. MS (*m/z* relative intensity %): 384 [M - C_4H_9NO]$^+$ (67), 323 (2), 299 (14), 257 (35), 213 (77), 185 (15), 156 (39), 115 (44), 57 (57), 28 (100). IR (KBr) ν = 3435, 3025, 2966, 2867, 2728, 2456, 1878, 1682, 1452, 1108 cm^{-1}. Anal. calcd for $C_{24}H_{29}N_3O_7$: C, 61.14; H, 6.20; N, 8.91%. Found: C, 61.01; H, 6.23; N, 8.77%.

Morpholin-4-ium 5-((4-hydroxy-6-methyl-2-oxo-2*H*-pyran-3-yl)(4-methoxyphenyl)methyl)-1,3-dimethyl-2,4-dioxo-1,2,3,4-tetrahydropyrimidin-6-olate (2c).

Yield 0.95 g (97%), mp: 176-177 °C. ^{1}H NMR (300 MHz, DMSO-d_6): δ 2.11 (s, 3H, CH_3), 3.03-3.18 (m, 10H, 2 NCH_2 + 2 CH_3), 3.68 (s, 3H, OCH_3), 3.76 (t, ^{3}J = 4.9 Hz, 4H, 2 CH_2), 5.78 (s, 1H, CH), 5.99 (s, 1H, CH),

6.71 (d, ^{3}J = 8.5 Hz, 2H, 2 CH Ar), 6.89 (d, ^{3}J = 8.5 Hz, 2H, 2 CH Ar), 8.69 (br s, 2H, NH_2^+), 15.06 (s, 1H, OH) ppm. ^{13}C NMR (75 MHz, DMSO-d_6): δ 19.4, 28.0 (2C), 34.2, 43.4 (2C), 55.3, 63.8 (2C), 90.0, 103.3, 104.0, 113.4 (2C), 127.9 (2C), 135.7, 152.5, 157.1, 159.2, 163.8 (2C), 166.6, 169.2 ppm. MS (*m/z* relative intensity %): 400 [M - C_4H_9NO]$^+$ (10), 285 (1), 274 (51), 243 (76), 201 (38), 156 (72), 117 (28), 57 (67), 42 (100). IR (KBr) ν = 3085, 2958, 2805, 2733, 2468, 1682, 1587, 1450, 1298, 1107 cm^{-1}. Anal. calcd for $C_{24}H_{29}N_3O_8$: C, 59.13; H, 6.00; N, 8.62%. Found: C, 59.02; H, 6.02; N, 8.53%.

Morpholin-4-ium 5-((3-fluorophenyl)(4-hydroxy-6-methyl-2-oxo-2*H*-pyran-3-yl)methyl)-1,3-dimethyl-2,4-dioxo-1,2,3,4-tetrahydropyrimidin-6-olate (2d).

Yield 0.85 g (89%), mp: 192-193 °C. ^{1}H NMR (300 MHz, DMSO-d_6): δ 2.13 (s, 3H, CH_3), 3.09-3.18 (m, 10H, 2 NCH_2 + 2 CH_3), 3.76 (t, ^{3}J = 4.9 Hz, 4H, 2 CH_2), 5.81 (s, 1H, CH), 6.07 (s, 1H, CH), 6.71 (d, $^{3}J_{H\text{-}F}$ = 11.3 Hz, 1H, CH Ar), 6.81-6.90 (m, 2H, 2 CH Ar), 7.14-7.32 (m, 1H, CH Ar), 8.69 (br s, 2H, NH_2^+), 15.04 (s, 1H, OH) ppm. ^{13}C NMR (75 MHz, DMSO-d_6): δ 19.4, 28.0 (2C), 35.0, 43.5 (2C), 63.8 (2C), 89.5, 103.2, 103.3, 111.7 (d, $^{2}J_{C\text{-}F}$ = 21.5 Hz), 113.6 (d, $^{2}J_{C\text{-}F}$ = 21.5 Hz), 123.1(d, $^{4}J_{C\text{-}F}$ = 2.3 Hz), 129.7 (d, $^{3}J_{C\text{-}F}$ = 7.5 Hz), 147.6 (d, $^{3}J_{C\text{-}F}$ = 7.5 Hz), 152.4, 159.6, 162.8 (d, $^{1}J_{C\text{-}F}$ = 241.1 Hz), 163.8 (2C), 166.5, 169.3 ppm. IR (KBr) ν = 3435, 3013, 2817, 2745, 2498, 2198, 1682, 1579, 1443, 1298, 1108 cm^{-1}. MS (*m/z* relative intensity %): 388 [M - C_4H_9NO]$^+$ (88), 317 (2), 303 (23), 261 (47), 233 (41), 189 (13), 156 (35), 120 (43), 57 (66), 28 (100). Anal. calcd for $C_{23}H_{26}FN_3O_7$: C, 58.10; H, 5.51; F, 4.00; N, 8.84%. Found: C, 58.03; H, 5.53; F, 3.88; N, 8.70%.

Morpholin-4-ium 5-((4-hydroxy-6-methyl-2-oxo-2*H*-pyran-3-yl)(4-(trifluoromethyl)phenyl)methyl)-1,3-dimethyl-2,4-dioxo-1,2,3,4-tetrahydropyrimidin-6-olate (2e).

Yield 1.01 g (96%), mp: 143-145 °C. ^{1}H NMR (500 MHz, DMSO-d_6): δ 2.14 (s, 3H, CH_3), 3.03-3.18 (m, 10H, 2 NCH_2 + 2 CH_3), 3.76 (t, ^{3}J = 4.9 Hz, 4H, 2 CH_2), 5.85 (s, 1H, CH), 6.11 (s, 1H, CH), 7.19 (d, ^{3}J = 7.9 Hz, 2H, 2 CH Ar), 7.52 (d, ^{3}J = 7.9 Hz, 2H, 2 CH Ar), 8.75 (br s, 2H, NH_2^+), 14.65-15.19 (s, 1H, OH) ppm. ^{13}C NMR (126 MHz, DMSO-d_6): δ 19.6, 28.2 (2C), 35.5, 43.4 (2C), 63.8 (2C), 89.6, 101.5, 103.1, 125.1 (q, $^{3}J_{C\text{-}F}$ = 3.6 Hz, 2C), 125.3 (q, $^{1}J_{C\text{-}F}$ = 271.2 Hz), 126.0 (q, $^{2}J_{C\text{-}F}$ = 31.7 Hz), 127.8 (2C), 149.6, 152.7, 159.9, 164.0 (2C), 166.5, 168.9 ppm. MS (*m/z* relative intensity %): 438 [M - C_4H_9NO]$^+$ (7), 312 (62), 311 (82), 253 (32), 198 (31), 156 (66), 120 (16), 69 (42), 42 (100). IR (KBr) ν = 3435, 2979, 2868, 2750, 2503, 1923, 1684, 1586, 1327, 1109 cm^{-1}. Anal. calcd for $C_{24}H_{26}F_3N_3O_7$: C, 54.86; H, 4.99; F, 10.85; N, 8.00%. Found: C, 54.83; H, 5.01; F, 10.82; N, 7.98%.

Morpholin-4-ium 5-((2-chlorophenyl)(4-hydroxy-6-methyl-2-oxo-2*H*-pyran-3-yl)methyl)-1,3-dimethyl-2,4-dioxo-1,2,3,4-tetrahydropyrimidin-6-olate (2f).

Yield 0.80 g (81%), mp: 187-189 °C. ^{1}H NMR (300 MHz, DMSO-d_6): δ 2.10 (s, 3H, CH_3), 3.07 (s, 6H, 2 CH_3), 3.11 (t, ^{3}J = 4.8 Hz, 4H, 2 CH_2), 3.76 (t, ^{3}J = 4.8 Hz, 4H, 2 CH_2), 5.79 (s, 1H, CH), 5.94 (s, 1H, CH), 7.04-7.16 (m, 2H, 2 CH Ar), 7.17-7.24 (m, 2H, 2 CH Ar), 8.68 (br s, 2H, NH_2^+), 14.56 (s, 1H, OH) ppm. ^{13}C NMR (75 MHz, DMSO-d_6): δ 19.4, 27.9 (2C), 35.0, 43.4 (2C), 63.8 (2C), 88.3, 103.2, 103.8, 126.2, 127.1, 129.7, 130.6, 133.2, 142.0, 152.5, 159.2, 163.5 (2C), 165.9, 169.0 ppm. MS (*m/z* relative intensity %): 404 [M - C_4H_9NO]$^+$ (^{35}Cl, 3), 213 (26), 186 (7), 136 (12), 101 (12), 57 (31), 28 (100). IR (KBr) ν = 3434, 3155, 2957, 2921, 2691, 2613, 2465, 1871, 1681 cm^{-1}. Anal. calcd for $C_{23}H_{26}ClN_3O_7$: C, 56.16; H, 5.33; Cl, 7.21; N, 8.54%. Found: C, 56.05; H, 5.28; Cl, 7.09; N, 8.41%.

Morpholin-4-ium 5-((3-bromophenyl)(4-hydroxy-6-methyl-2-oxo-2*H*-pyran-3-yl)methyl)-1,3-dimethyl-2,4-dioxo-1,2,3,4-tetrahydropyrimidin-6-olate (2g).

Yield 0.91 g (85%), mp: 184-185 °C. ^{1}H NMR (300 MHz, DMSO-d_6): δ 2.12 (s, 3H, CH_3), 3.06-3.17 (m, 10H, 2 NCH_2 + 2 CH_3), 3.76 (t, ^{3}J = 4.8 Hz, 4H, 2 CH_2), 5.81 (s, 1H, CH), 6.06 (s, 1H, CH), 6.99 (d, ^{3}J = 7.8 Hz, 1H, CH Ar), 7.07 (s, 1H, CH Ar), 7.12 (t, ^{3}J = 7.8 Hz, 1H, CH Ar), 7.23 (d, ^{3}J = 7.8 Hz, 1H, CH Ar), 8.67 (br s, 2H, NH_2^+), 15.01 (s, 1H, OH) ppm. ^{13}C NMR (75 MHz, DMSO-d_6): δ 19.5, 28.1 (2C), 35.0, 43.4 (2C), 63.9 (2C), 89.5, 102.9, 103.2, 121.7, 126.3, 128.0, 129.5, 130.3, 147.5, 152.4, 159.6, 163.8 (2C), 166.4, 169.3 ppm. MS (*m/z* relative intensity %): 450 [M - C_4H_9NO]$^+$ (^{81}Br, 6), 448 [M - C_4H_9NO]$^+$ (^{79}Br, 7), 365 (^{81}Br, 1), 363 (^{79}Br, 1), 324 (^{81}Br, 19), 322 (^{79}Br, 19), 293 (10), 266 (^{81}Br, 5), 264 (^{79}Br, 5), 213 (23), 156 (25), 126 (8), 69 (32), 28 (100). IR (KBr) ν = 3435, 3040, 2955, 2915, 2747, 2651, 2508, 1982, 1684, 1453 cm^{-1}. Anal. calcd for $C_{23}H_{26}BrN_3O_7$: C, 51.50; H, 4.89; Br, 14.90; N, 7.83%. Found: C, 51.38; H, 4.93; Br, 14.78; N, 7.75%.

Morpholin-4-ium 5-((4-hydroxy-6-methyl-2-oxo-2*H*-pyran-3-yl)(4-nitrophenyl)methyl)-1,3-dimethyl-2,4-dioxo-1,2,3,4-tetrahydropyrimidin-6-olate (2h).

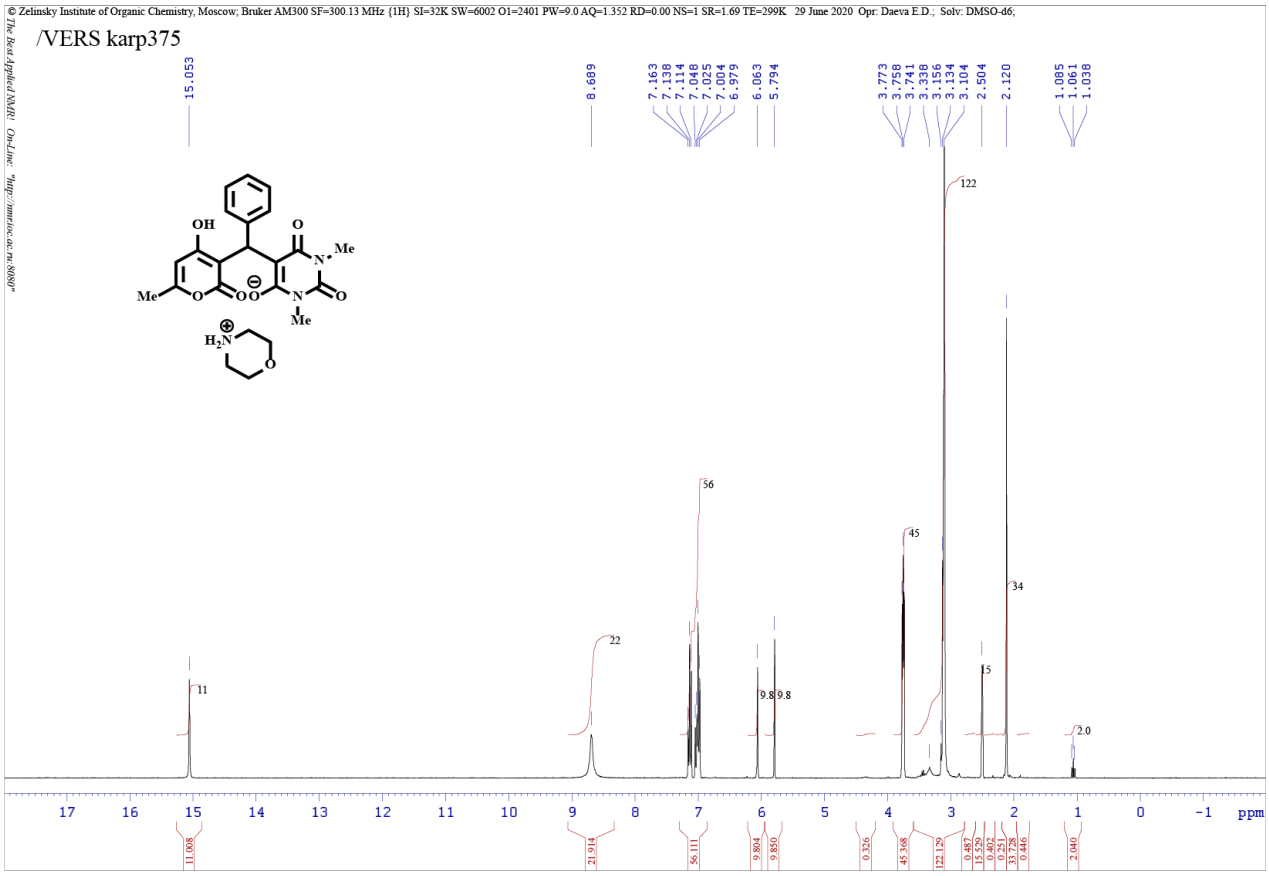
Yield 0.92 g (92%), mp: 201-203 °C. ^{1}H NMR (300 MHz, DMSO-d_6): δ 2.14 (s, 3H, CH_3), 3.05-3.17 (m, 10H, 2 NCH_2 + 2 CH_3), 3.76 (t, ^{3}J = 4.9 Hz, 4H, 2 CH_2), 5.85 (s, 1H, CH), 6.16 (s, 1H, CH), 7.24 (d, ^{3}J = 8.1 Hz, 2H, 2 CH Ar), 8.06 (d, ^{3}J = 8.1 Hz, 2H, 2 CH Ar), 8.68 (br s, 2H, NH_2^+), 14.90 (s, 1H, OH) ppm. ^{13}C NMR (75 MHz, DMSO-d_6): δ 19.5, 28.1 (2C), 35.8, 43.4 (2C), 63.8 (2C), 89.6, 102.7, 103.2, 123.4 (2C), 128.2 (2C), 145.5, 152.4, 153.5, 159.8, 163.8 (2C), 166.3, 169.4 ppm. MS (*m/z* relative intensity %): 415 [M - C_4H_9NO]$^+$ (1), 300 (3), 289 (69), 242 (53), 175 (21), 156 (59), 101 (18), 57 (61), 28 (100). IR (KBr) ν = 3435, 3085, 2955, 2812, 2743, 2474, 1879, 1684, 1575, 1348 cm^{-1}. Anal. calcd for $C_{23}H_{26}N_4O_9$: C, 54.98; H, 5.22; N, 11.15%. Found: C, 54.86; H, 5.19; N, 11.02%.

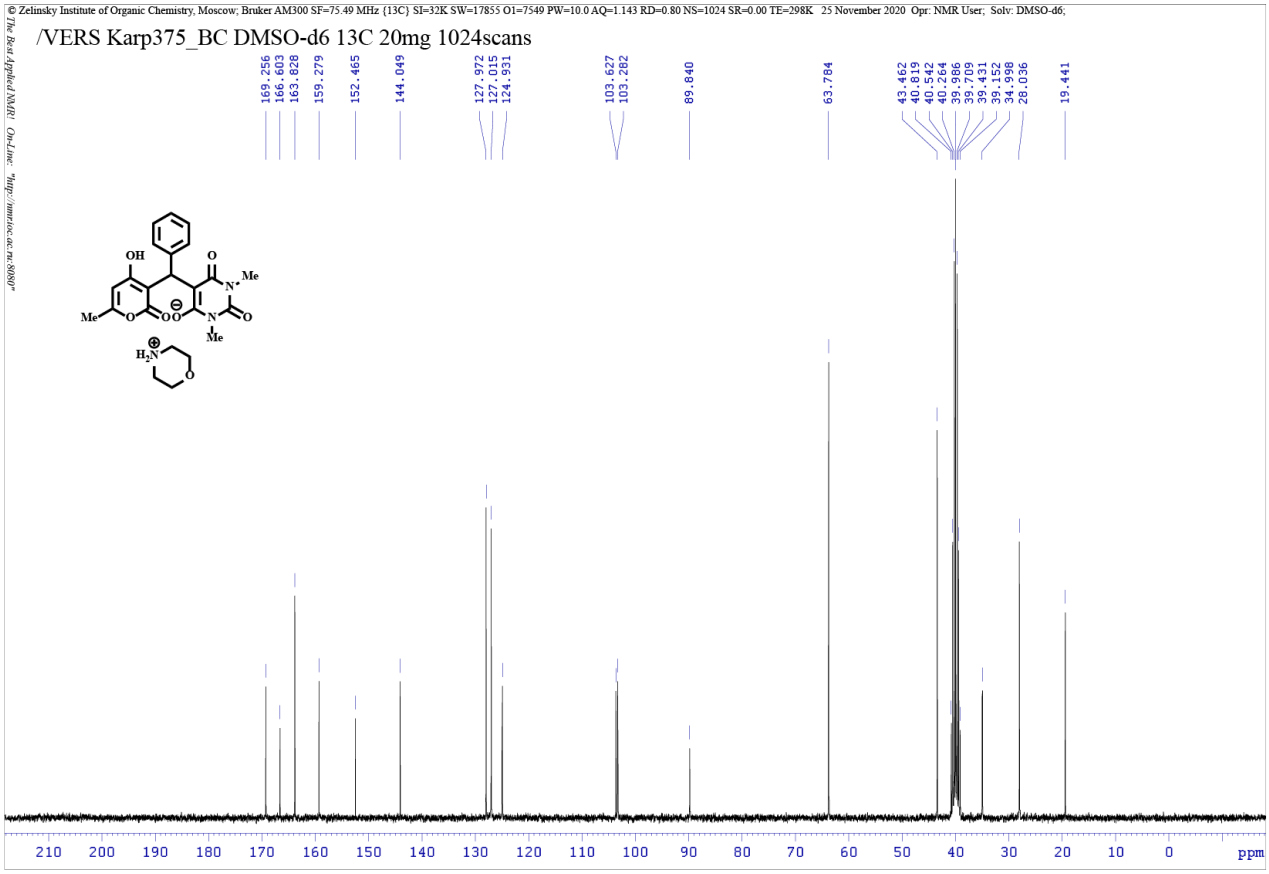
Morpholin-4-ium 5-((4-hydroxy-6-methyl-2-oxo-2*H*-pyran-3-yl)(4-(methoxycarbonyl)phenyl)methyl)-1,3-dimethyl-2,4-dioxo-1,2,3,4-tetrahydropyrimidin-6-olate (2i)

Yield 1.01 g (98%), mp: 133-134 °C. ^{1}H NMR (300 MHz, DMSO-d_6): δ 2.13 (s, 3H, CH_3), 3.06-3.18 (m, 10H, 2 NCH_2 + 2CH_3), 3.75 (t, ^{3}J = 4.9 Hz, 4H, 2 CH_2), 3.81 (s, 3H, CO_2Me), 5.82 (s, 1H, CH), 6.11 (s, 1H, CH), 7.12 (d, ^{3}J = 7.8 Hz, 2H, 2 CH Ar), 7.76 (d, ^{3}J = 7.8 Hz, 2H, 2 CH Ar), 8.61 (br s, 2H, NH_2^+), 14.94 (s, 1H, OH) ppm. ^{13}C NMR (75 MHz, DMSO-d_6): δ 19.4, 28.0 (2C), 35.5, 43.5 (2C), 52.3, 63.9 (2C), 89.8, 103.0, 103.2, 126.6, 127.3 (2C), 129.2 (2C), 150.7, 152.5, 159.5, 163.8 (2C), 166.4, 166.9, 169.3 ppm. MS (*m/z* relative intensity %): 398 $[M - C_4H_9NO – OCH_3 + H]^+$ (4), 287 (75), 271 (25), 243 (39), 186 (13), 156 (75), 129 (19), 57 (79), 42 (100). IR (KBr) ν = 3435, 2957, 2863, 2749, 2657, 2499, 2213, 1682, 1584 cm^{-1}. Anal. calcd for $C_{25}H_{29}N_3O_9$: C, 58.25; H, 5.67; N, 8.15%. Found: C, 58.12; H, 5.62; N, 8.02%.

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

Morpholin-4-ium 5-((4-hydroxy-6-methyl-2-oxo-2*H*-pyran-3-yl)(phenyl)methyl)-1,3-dimethyl-2,4-dioxo-1,2,3,4-tetrahydropyrimidin-6-olate (2a).





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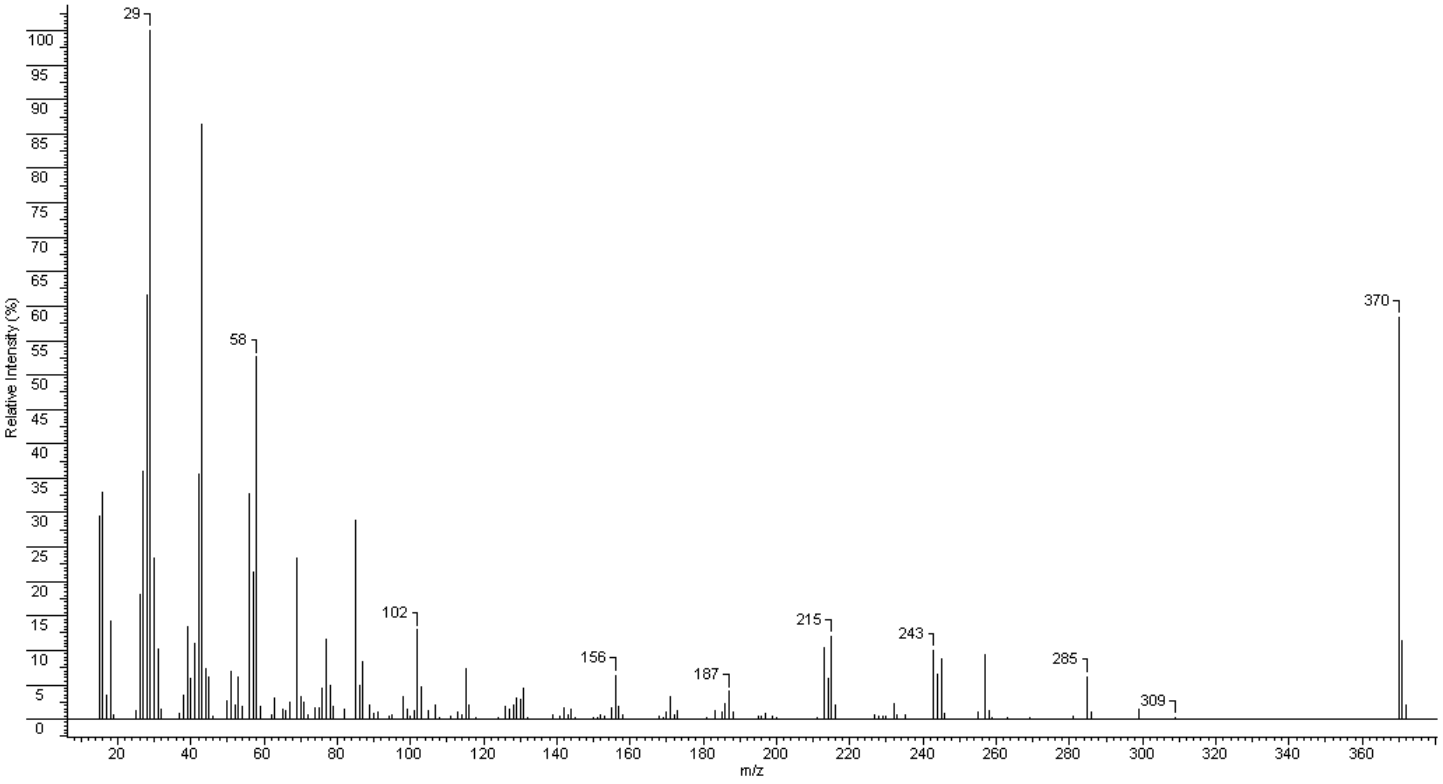

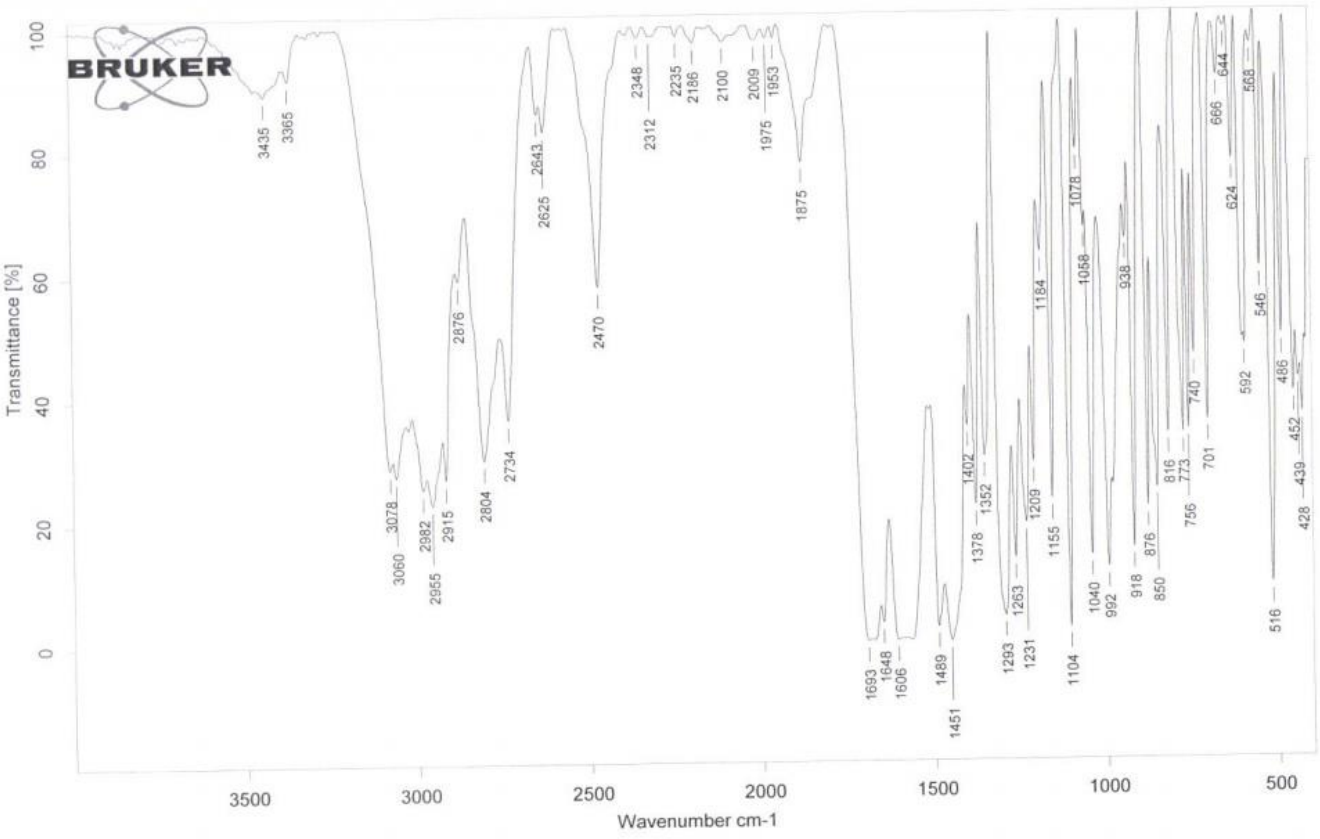

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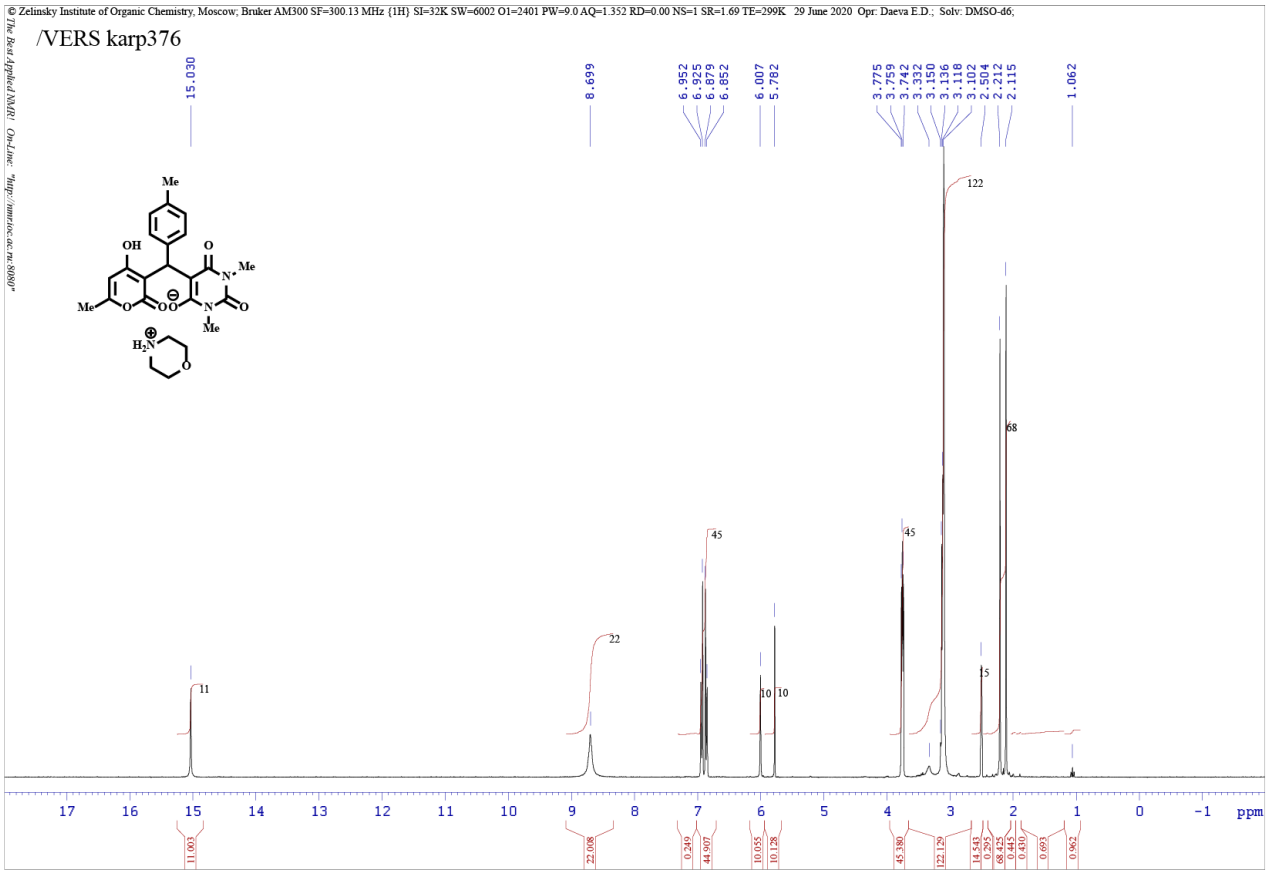
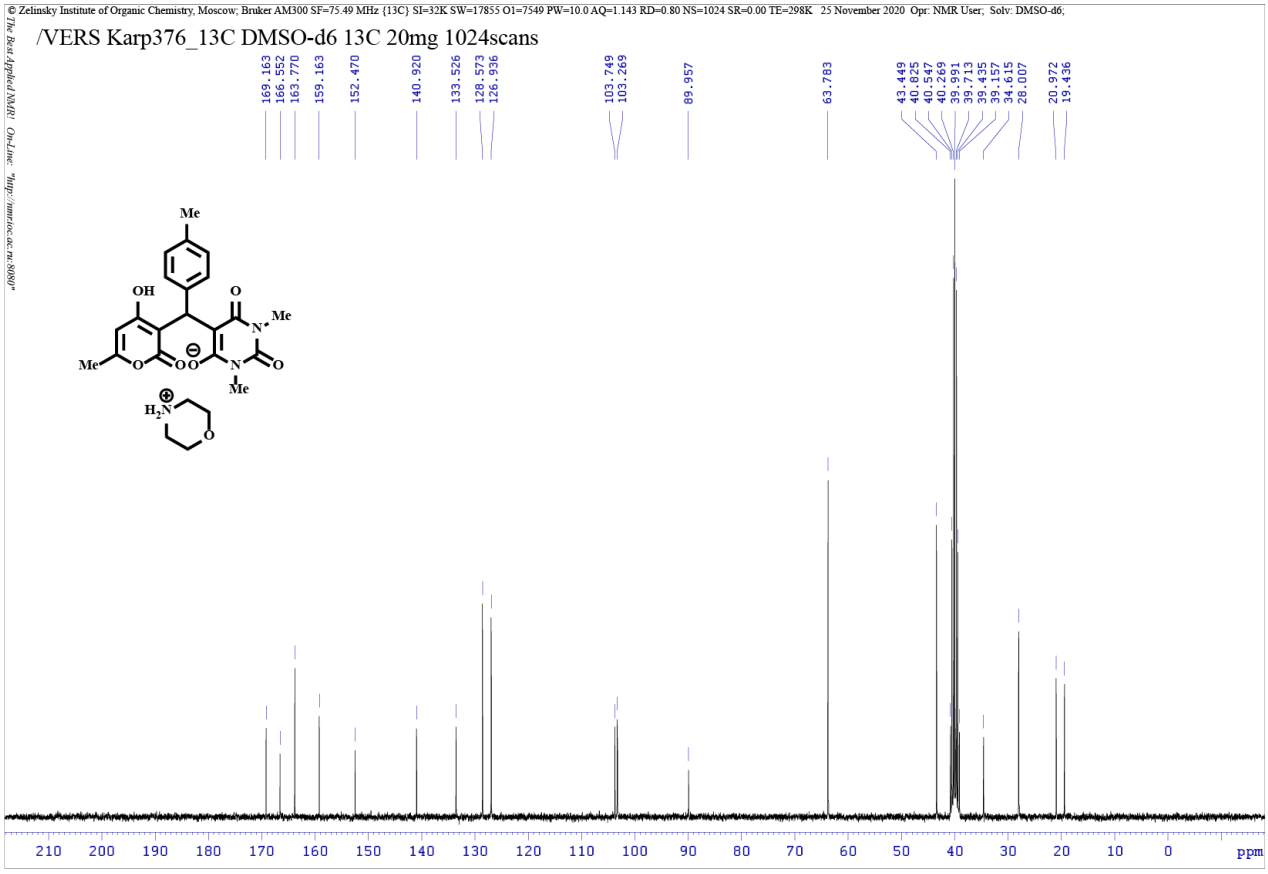
Morpholin-4-ium 5-((4-hydroxy-6-methyl-2-oxo-2H-pyran-3-yl)(4-methylphenyl)methyl)-1,3-dimethyl-2,4-dioxo-1,2,3,4-tetrahydropyrimidin-6-olate (2b).





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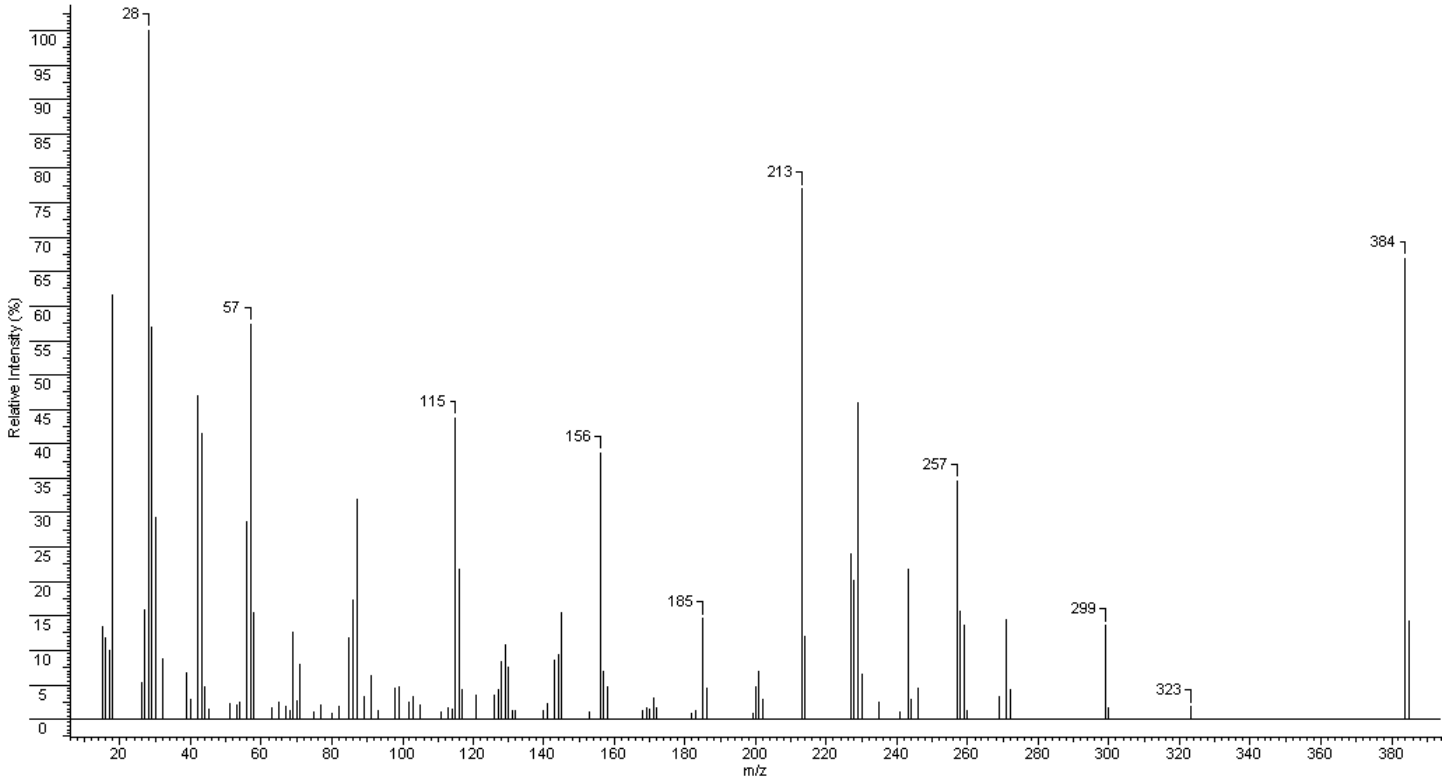

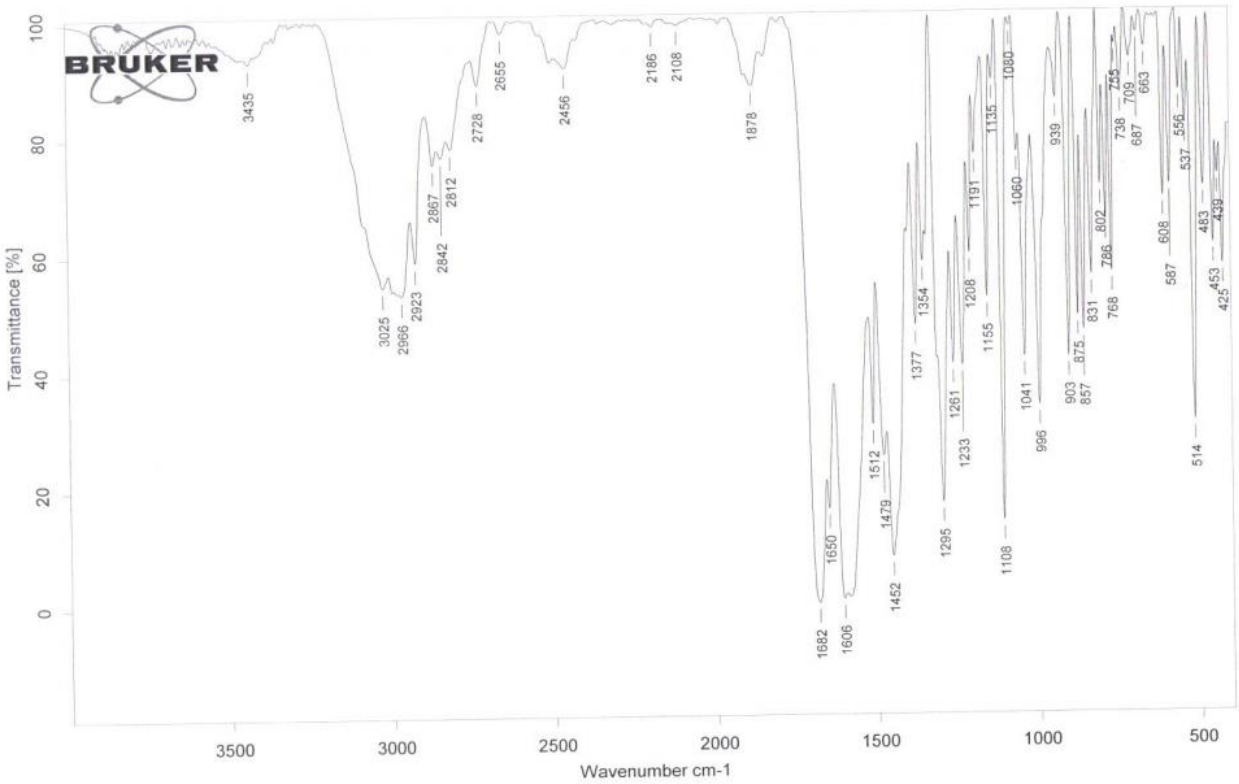

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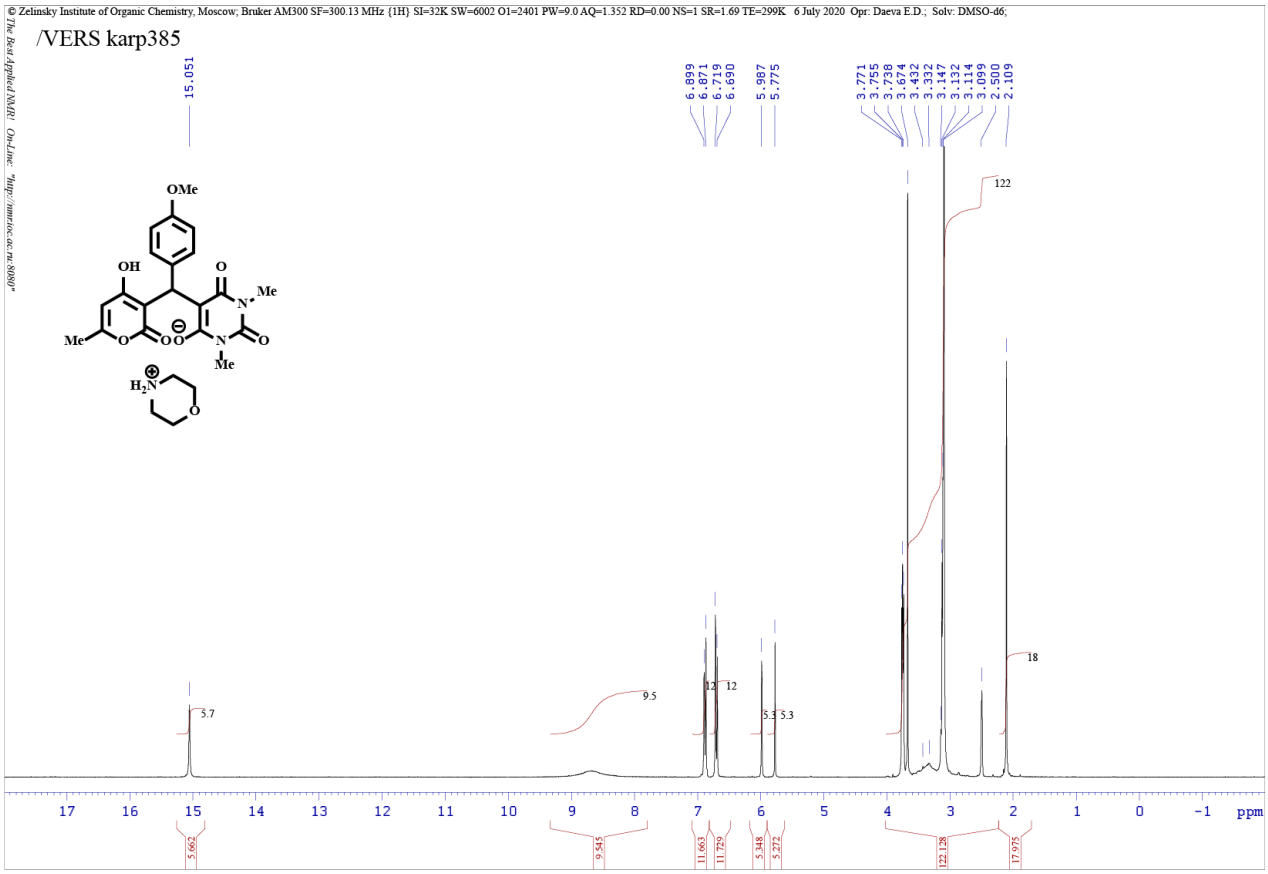
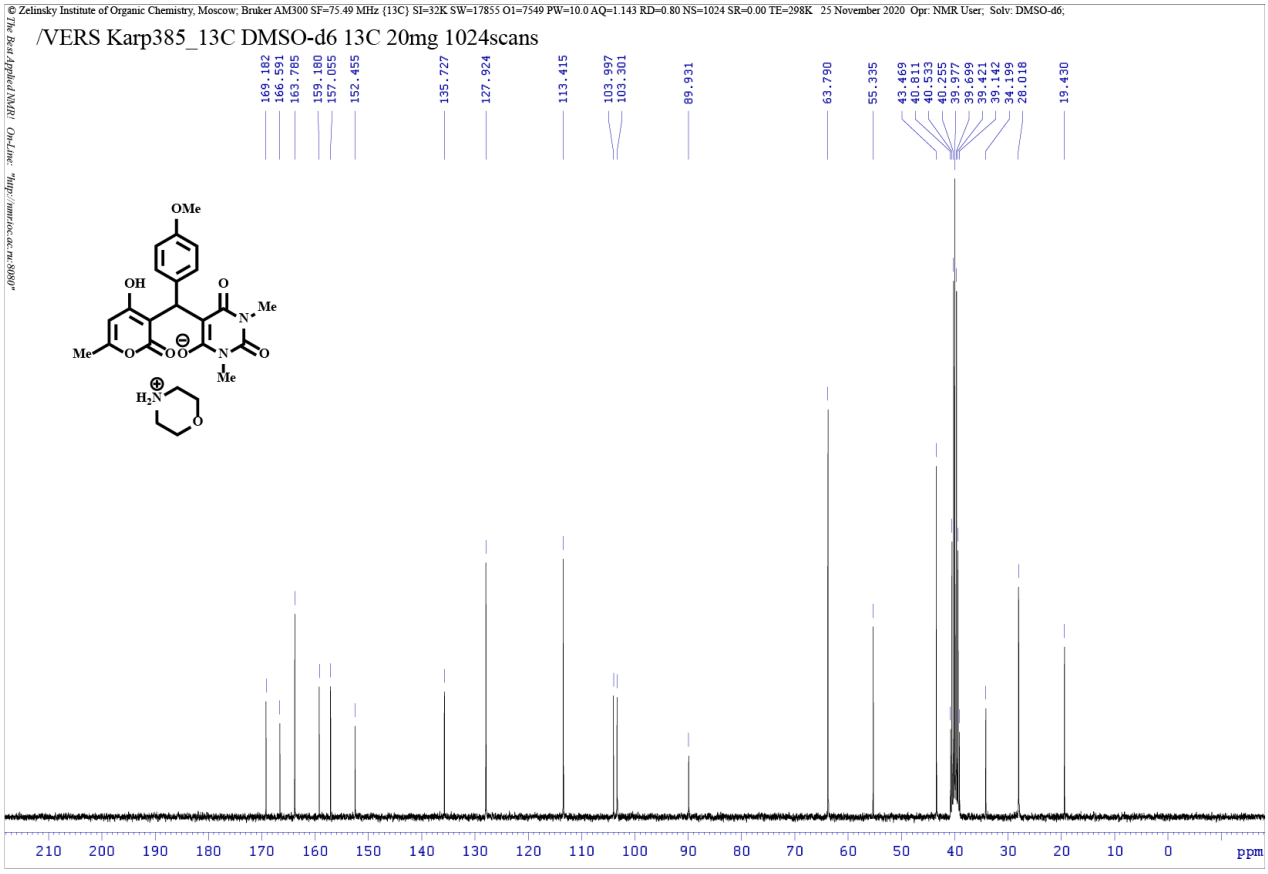
Morpholin-4-ium 5-((4-hydroxy-6-methyl-2-oxo-2*H*-pyran-3-yl)(4-methoxyphenyl)methyl)-1,3-dimethyl-2,4-dioxo-1,2,3,4-tetrahydropyrimidin-6-olate (2c).





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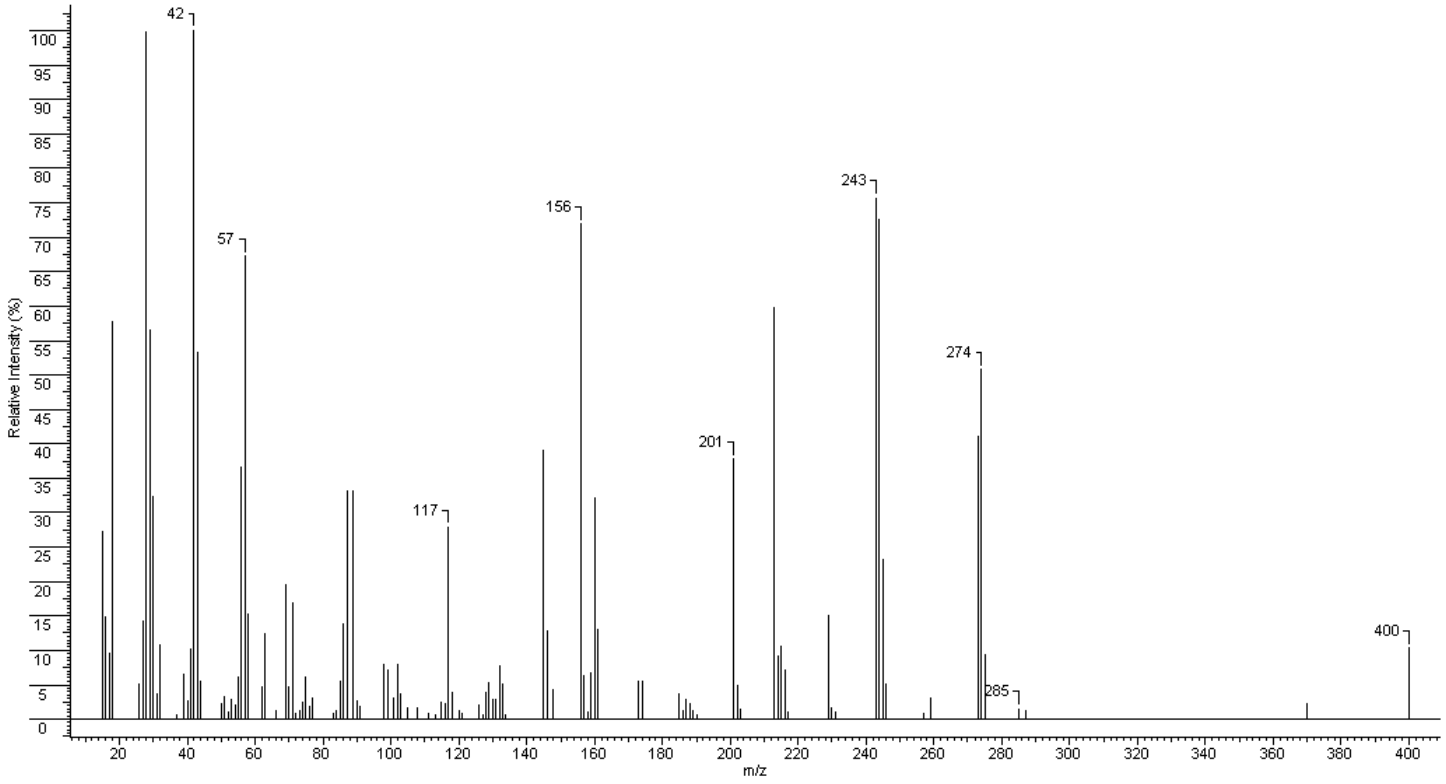

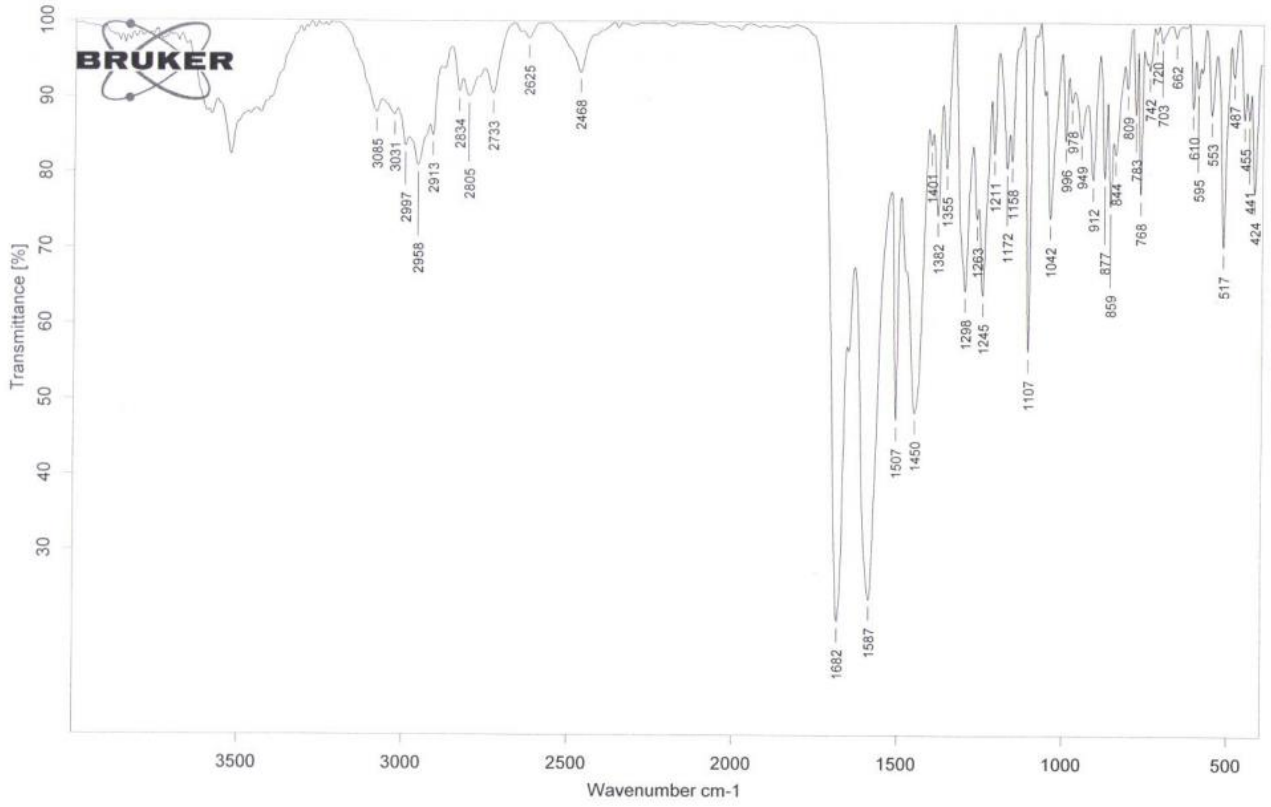

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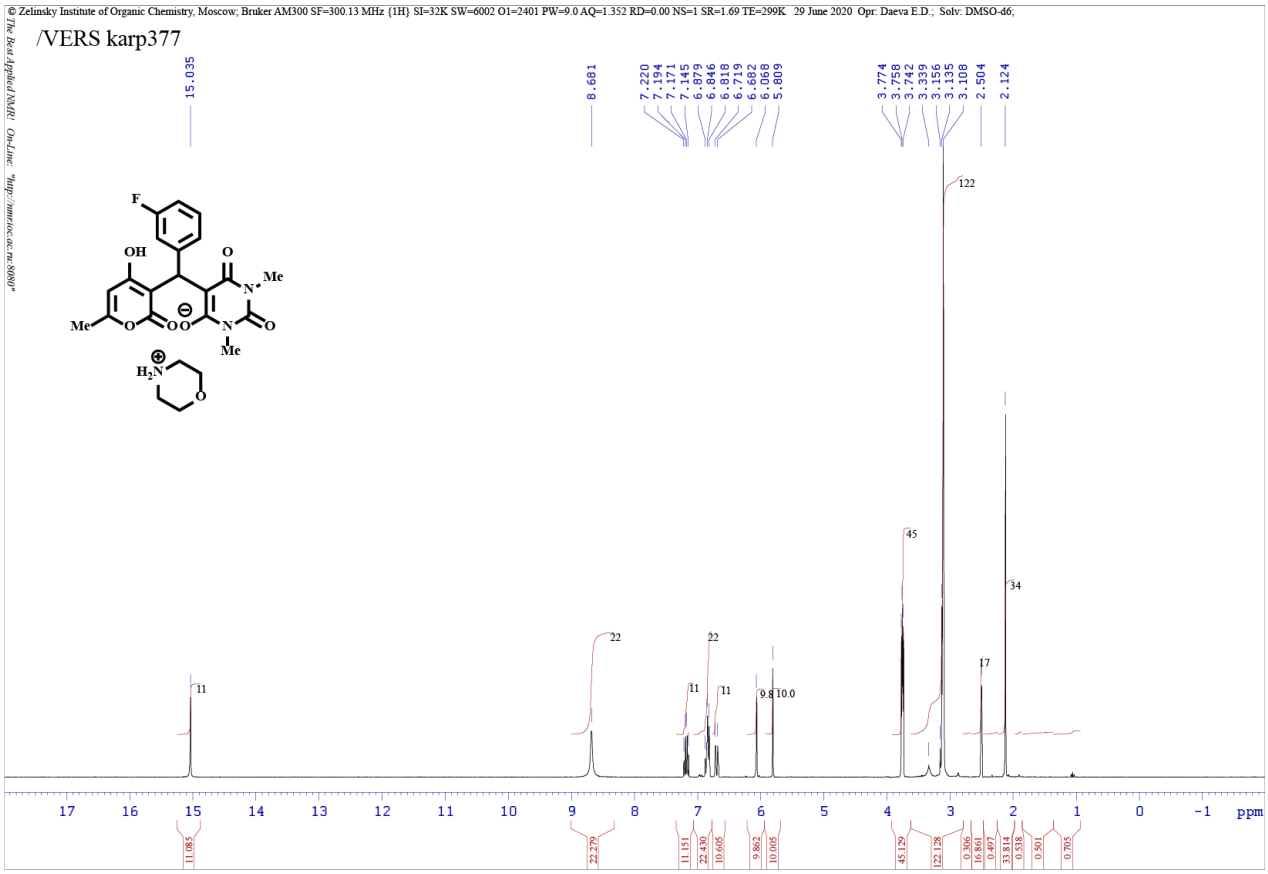
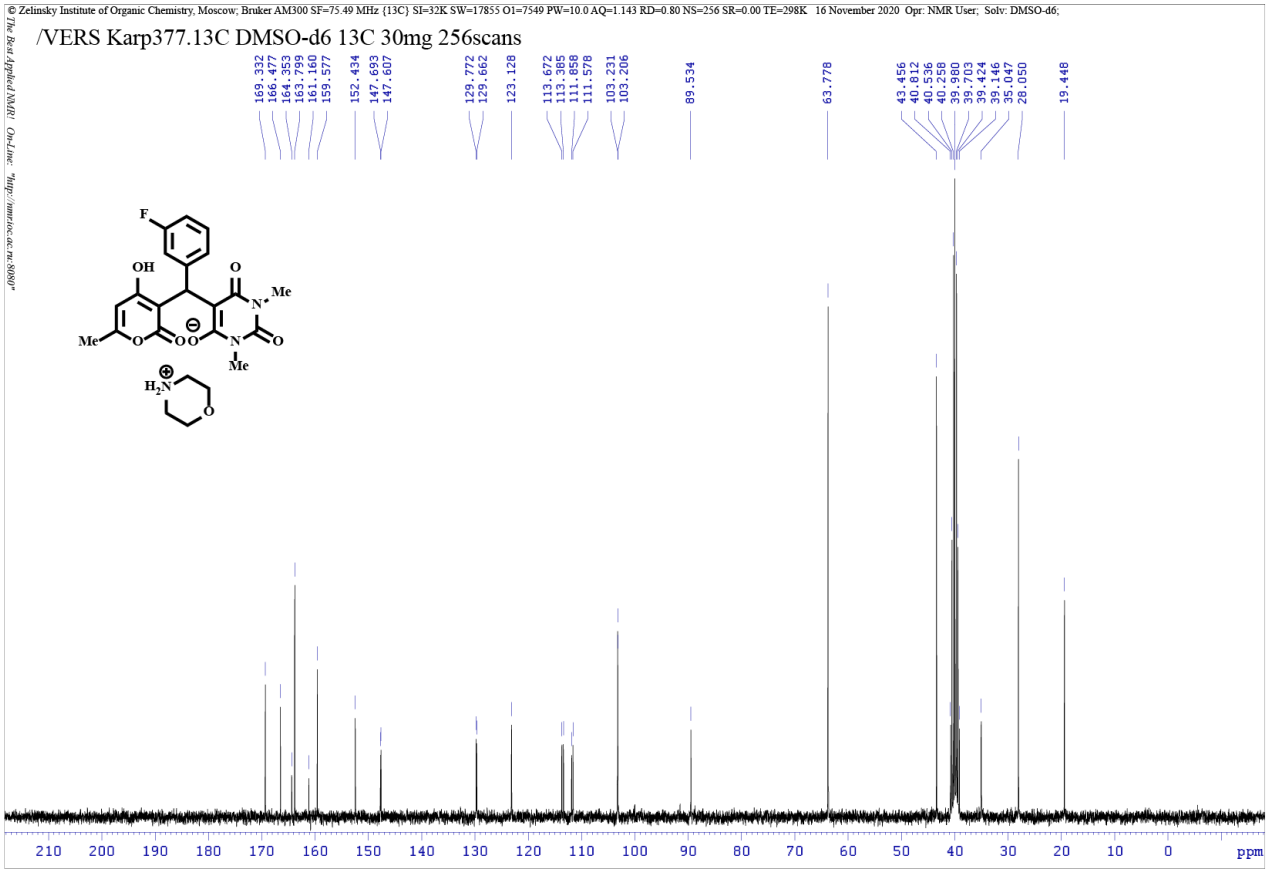
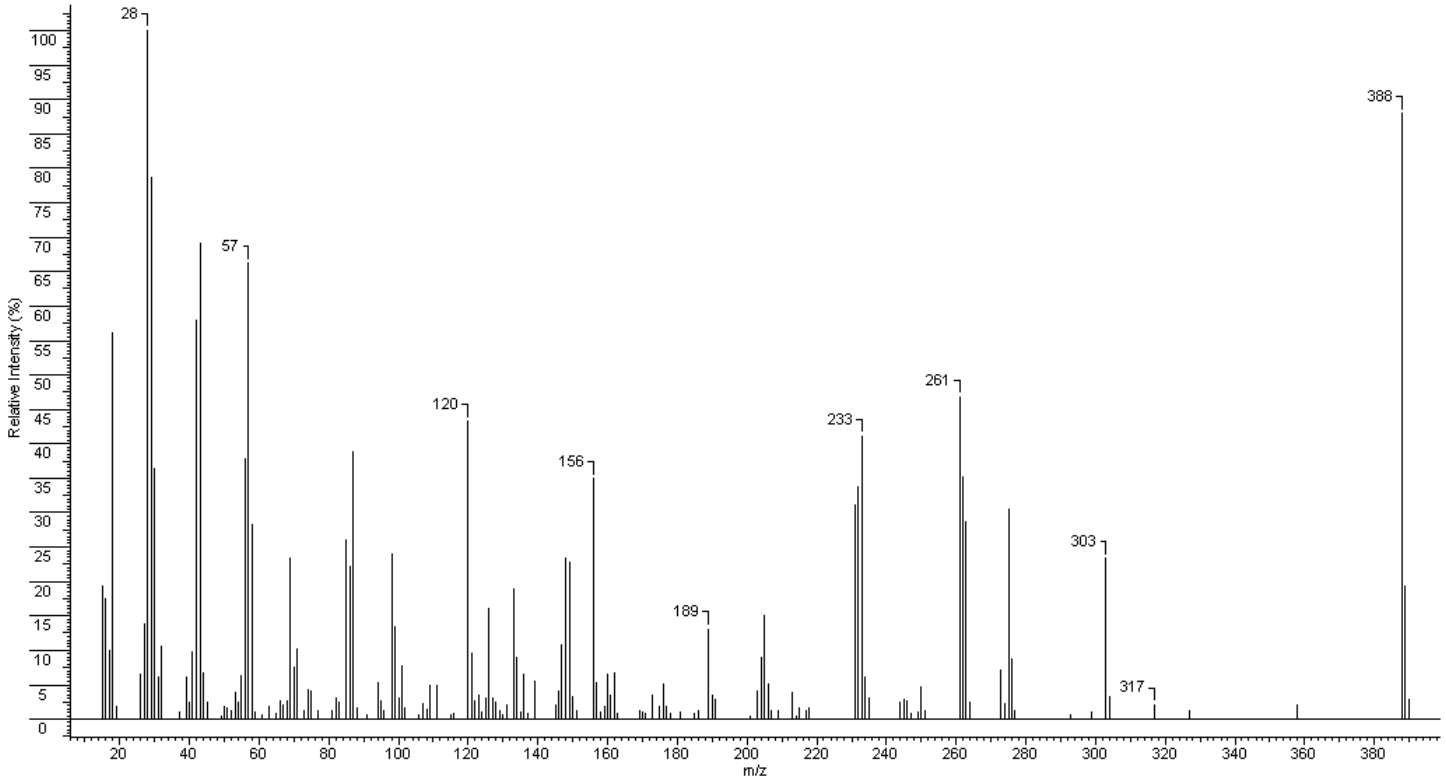
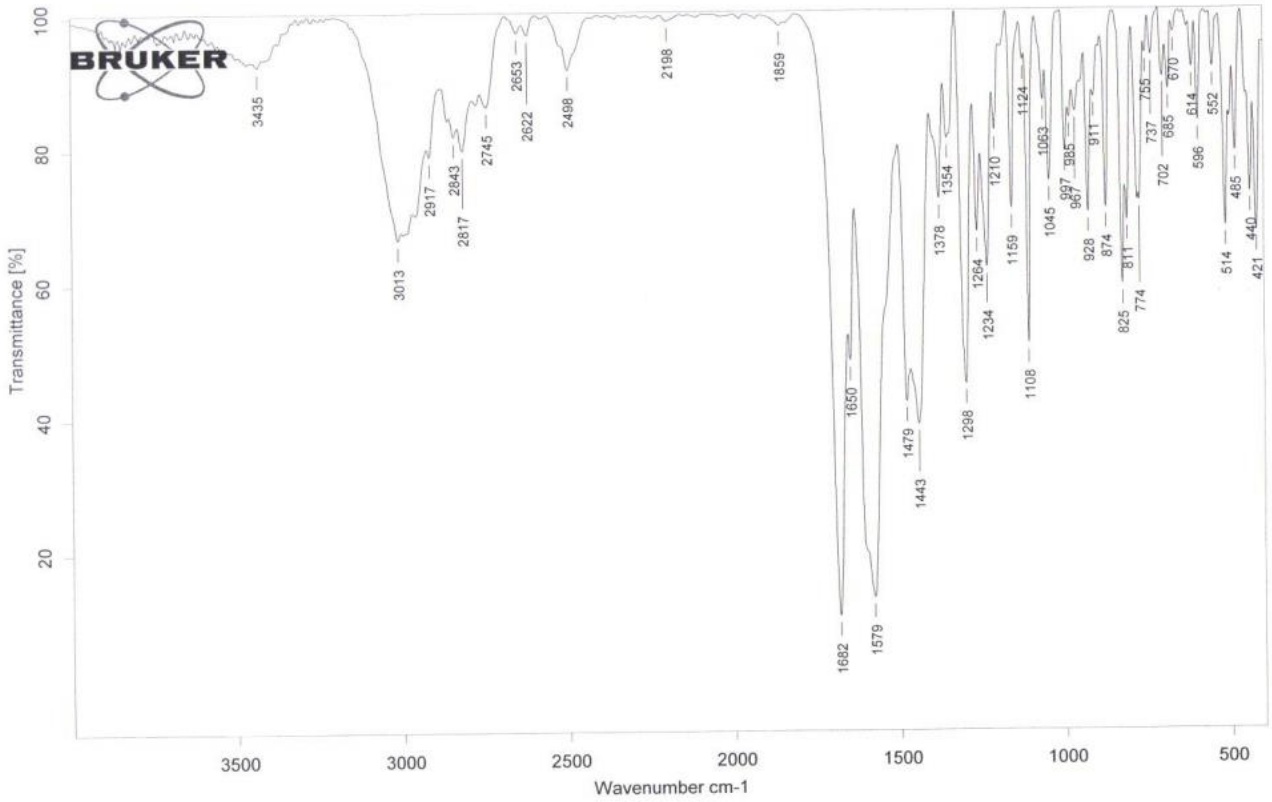
Morpholin-4-ium 5-((3-fluorophenyl)(4-hydroxy-6-methyl-2-oxo-2*H*-pyran-3-yl)methyl)-1,3-dimethyl-2,4-dioxo-1,2,3,4-tetrahydropyrimidin-6-olate (2d).





Count	157	***Data Type***	MASS SPECTRUM	***Date***	15 Dec 2020 17:21:46
Date Stamp	15 Dec 1920 16:13:10				
File Name	G:\09.04.21\ms11.JC				
Inlet Model	DIRECT	***Instrumental Parameters***	LOW RESOLUTION	***Origin***	KARP 377
Owner	Copyright(C) by Victor (2020)	***Spectrum Title***	MASS SPECTRUM	***TIC***	1677.61





D:\Ir\Карпенко Karp377.0	Карпенко Karp377	KBr прессовка	28.12.2020

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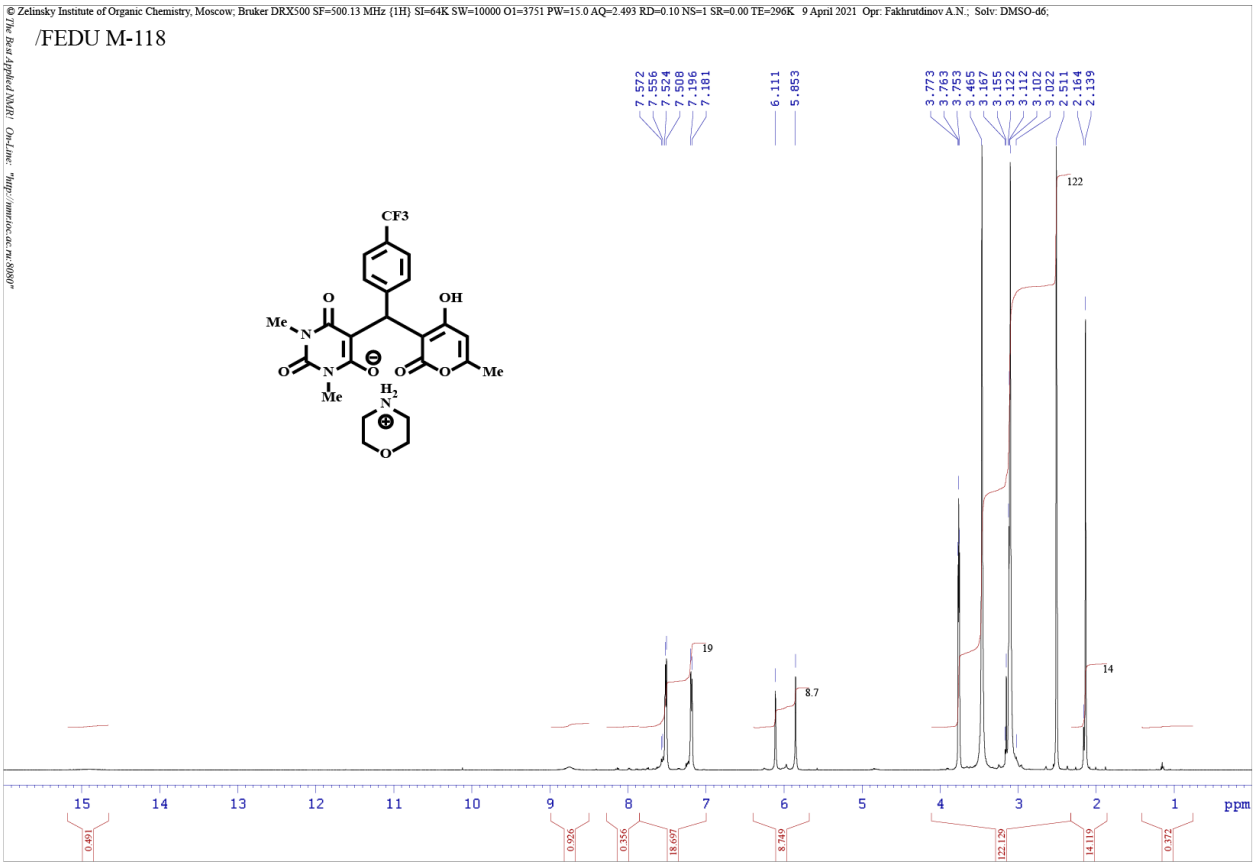
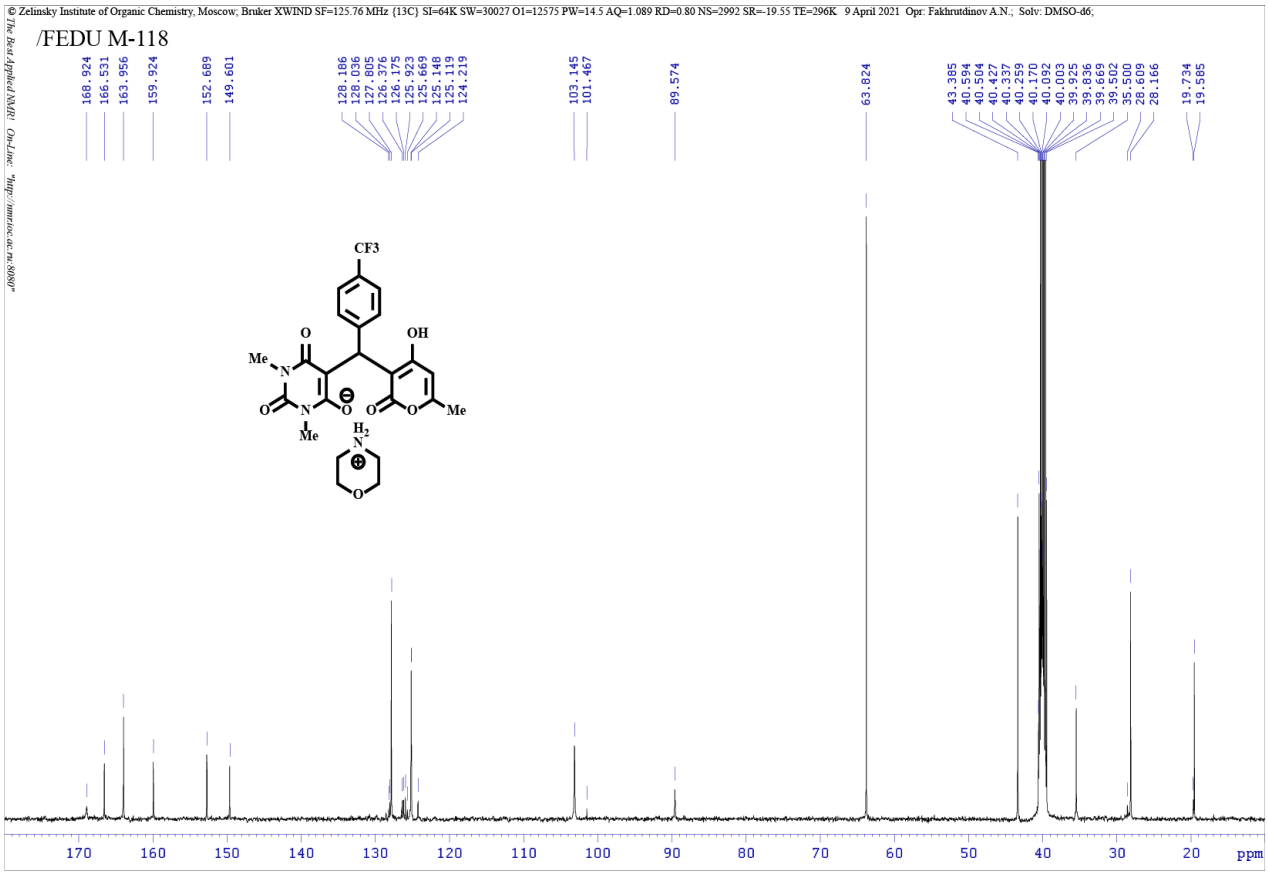
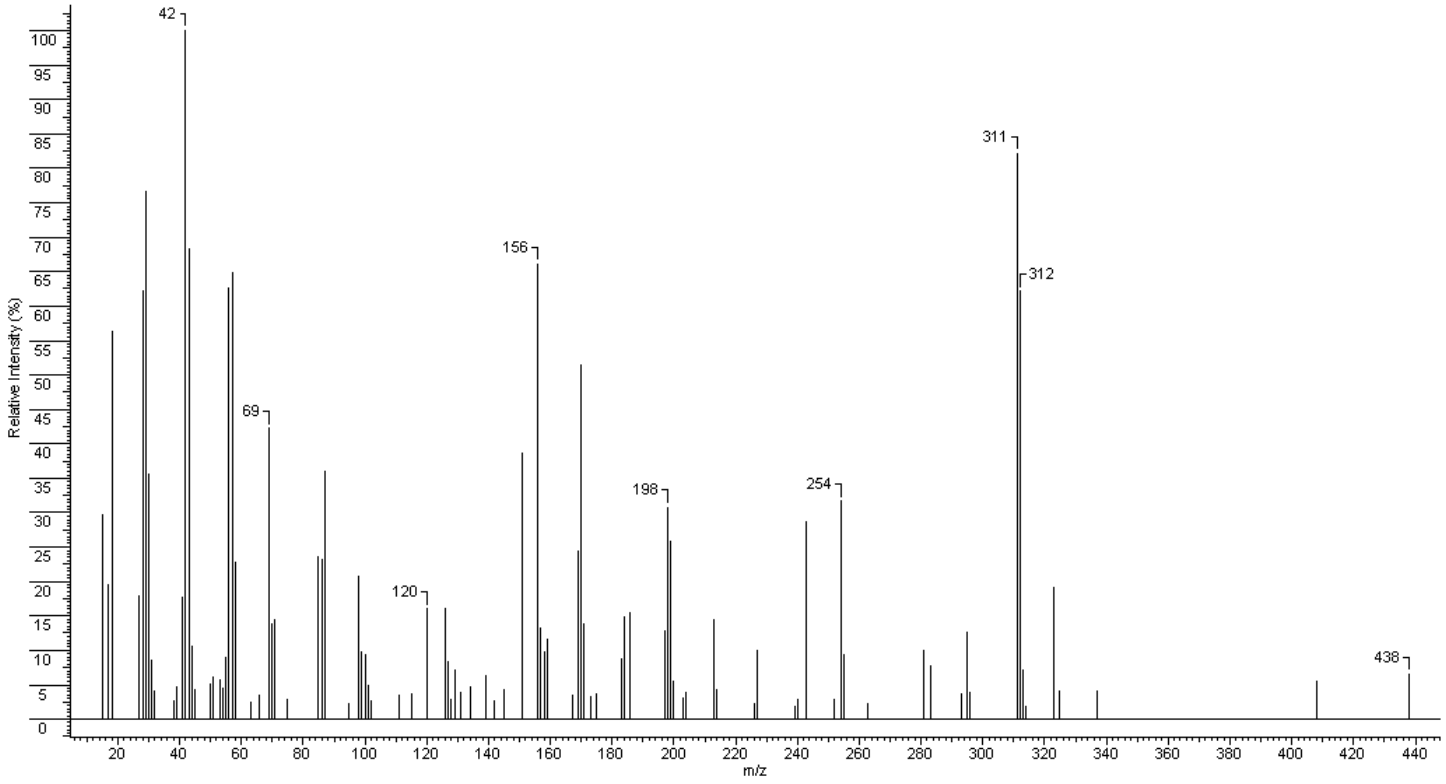
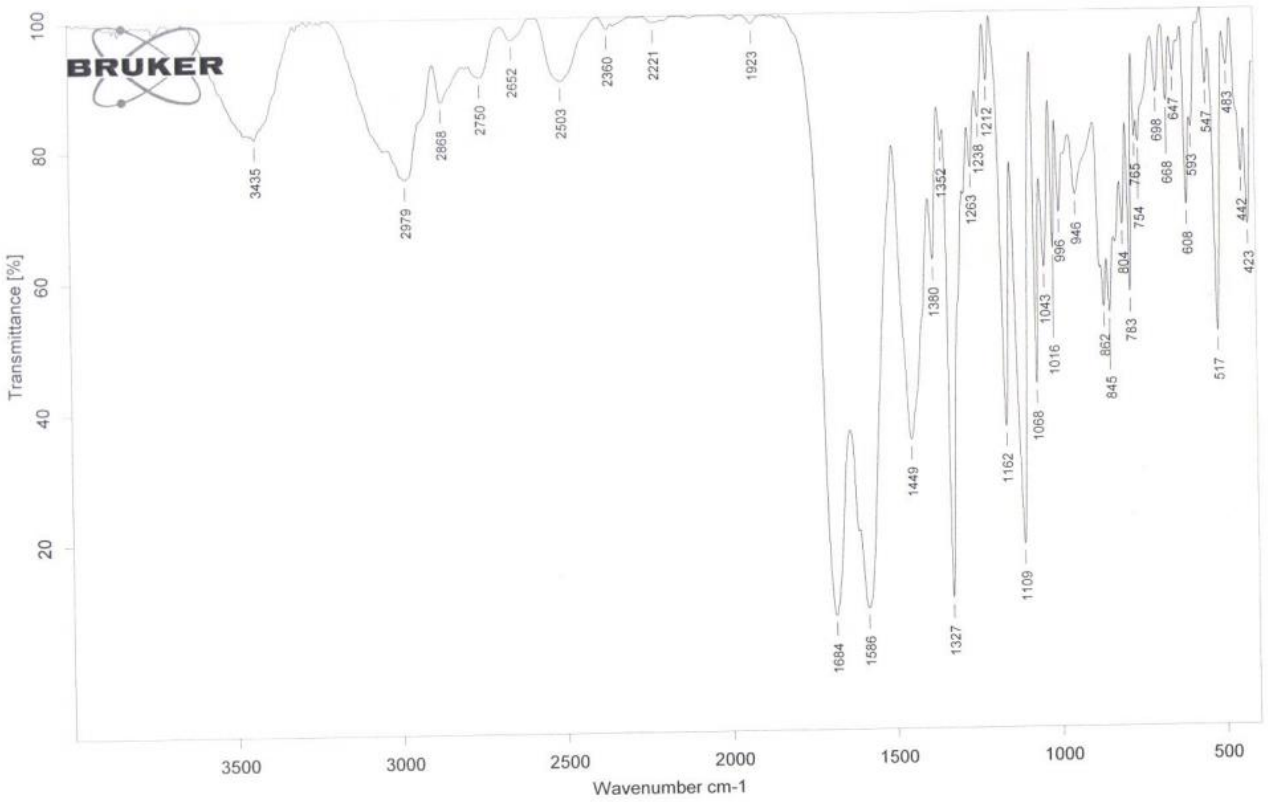
Morpholin-4-ium 5-((4-hydroxy-6-methyl-2-oxo-2*H*-pyran-3-yl)(4-(trifluoromethyl)phenyl)methyl)-1,3-dimethyl-2,4-dioxo-1,2,3,4-tetrahydropyrimidin-6-olate (2e).





Count	96	***Data Type***	MASS SPECTRUM	***Date***	15 Dec 2020 17:21:46
Date Stamp	15 Dec 1920 16:13:10				
File Name	G:\09.04.21\ms11.JC				
Inlet Model	DIRECT	***Instrumental Parameters***	LOW RESOLUTION	***Origin***	M 118
Owner	Copyright(C) by Victor (2020)	***Spectrum Title***	MASS SPECTRUM	***TIC***	1691.94





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12.01.2021

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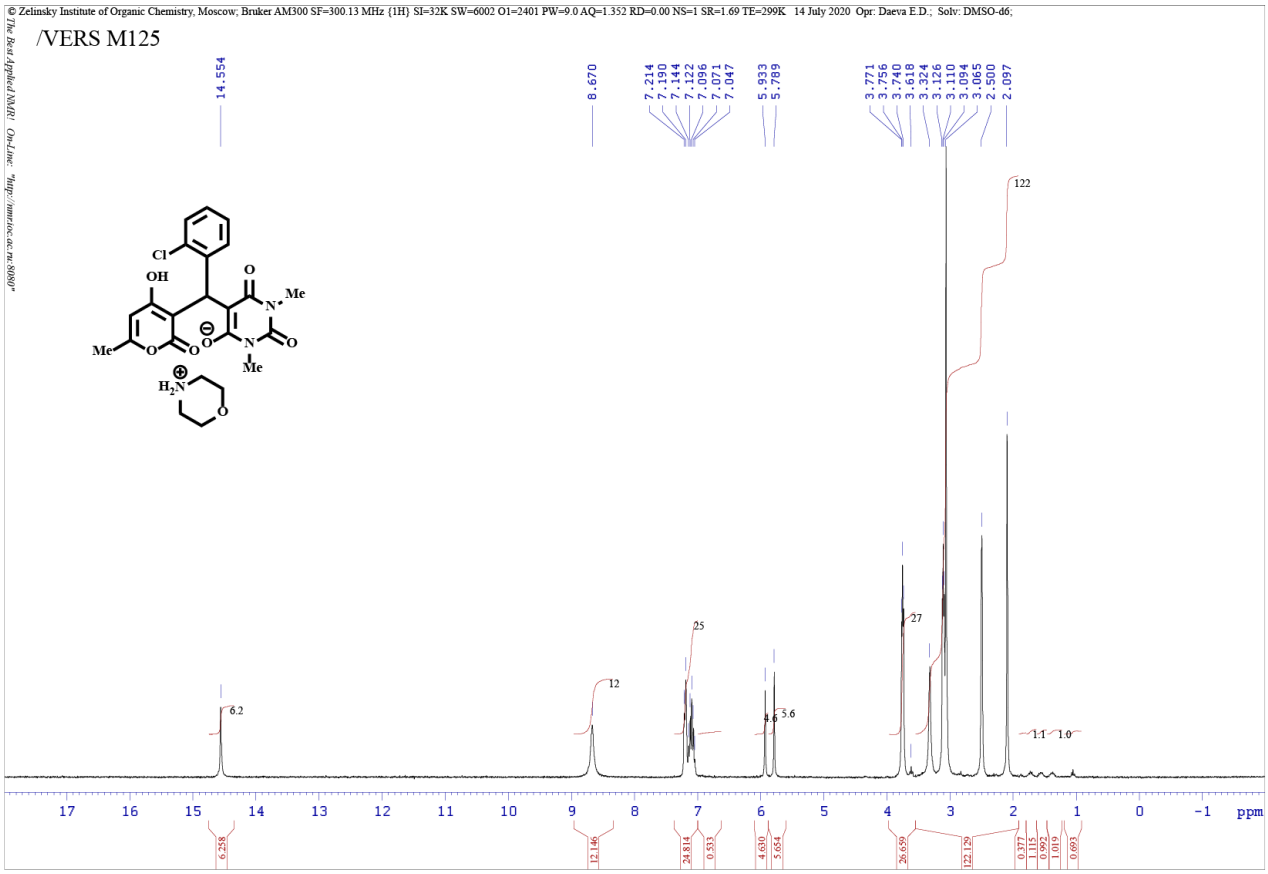
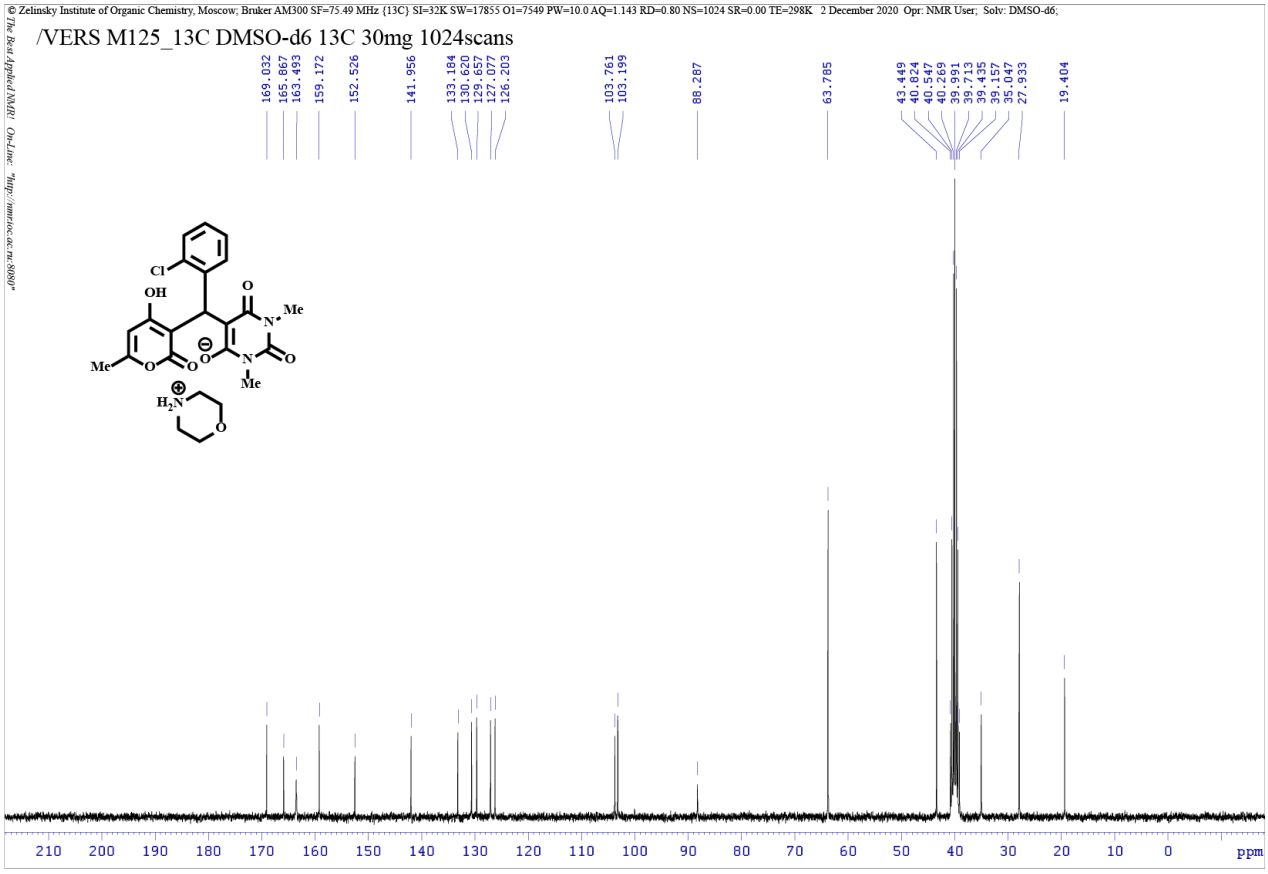
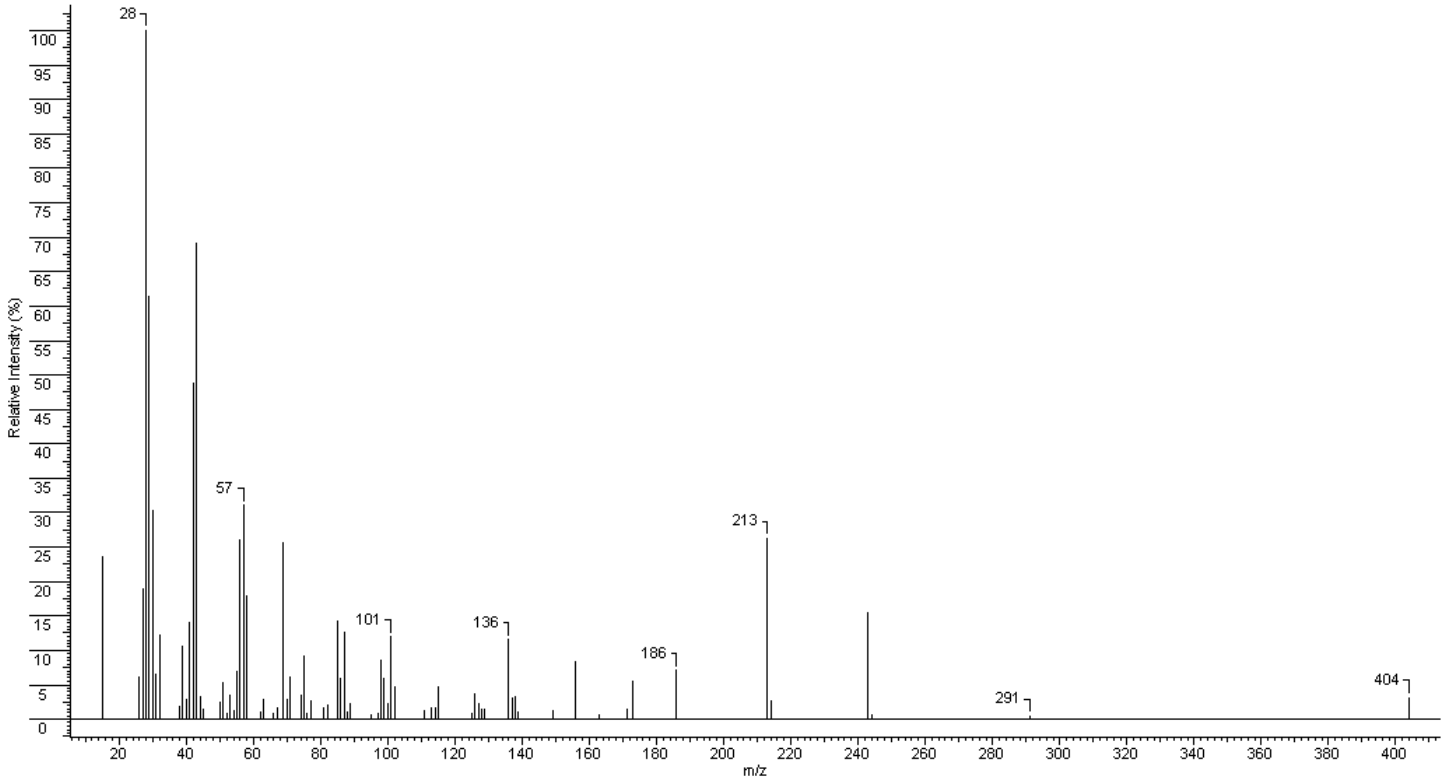
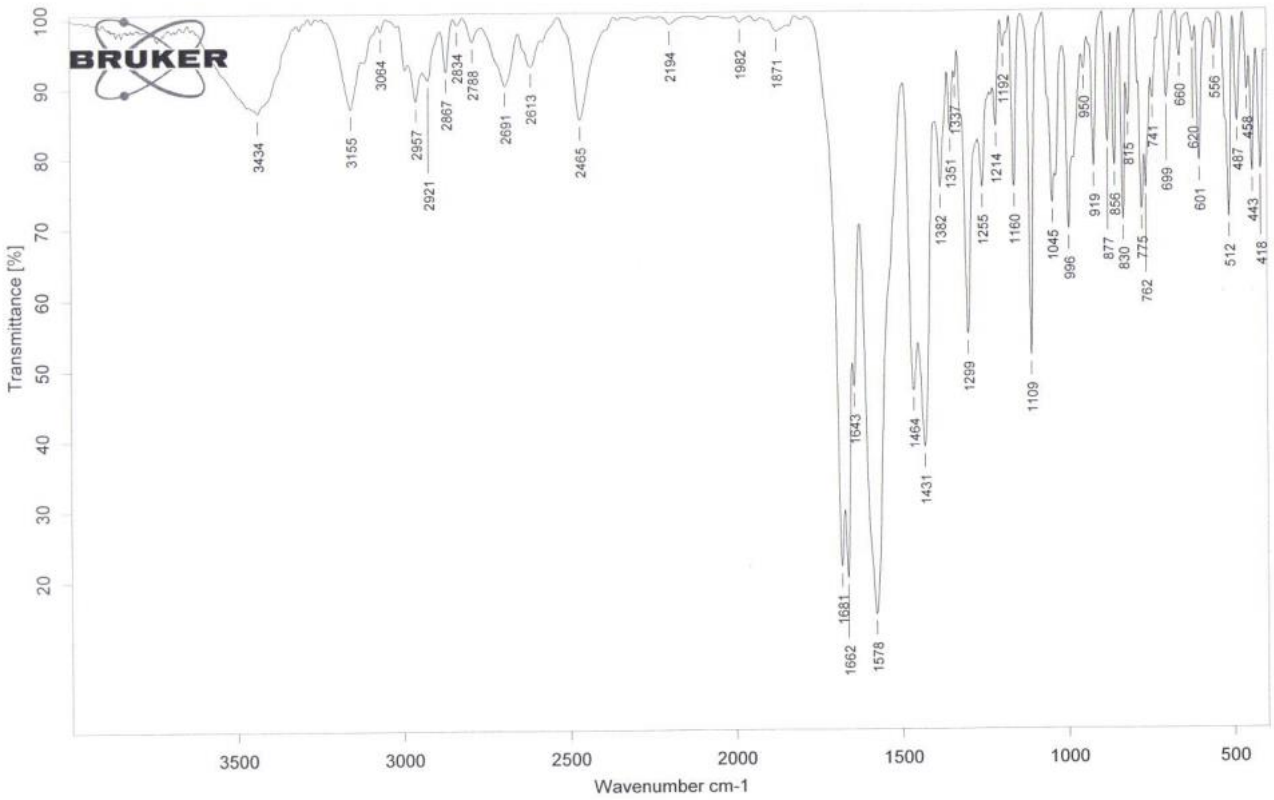
Morpholin-4-ium 5-((2-chlorophenyl)(4-hydroxy-6-methyl-2-oxo-2*H*-pyran-3-yl)methyl)-1,3-dimethyl-2,4-dioxo-1,2,3,4-tetrahydropyrimidin-6-olate (2f).





Count	75	***Data Type***	MASS SPECTRUM	***Date***	17 Dec 2020 16:01:42
Date Stamp	17 Dec 1920 14:58:51				
File Name	G:\09.04.21\ms6.JC				
Inlet Model	DIRECT	***Instrumental Parameters***	LOW RESOLUTION	***Origin***	M 125
Owner	Copyright(C) by Victor (2020)	***Spectrum Title***	MASS SPECTRUM	***TIC***	748.55





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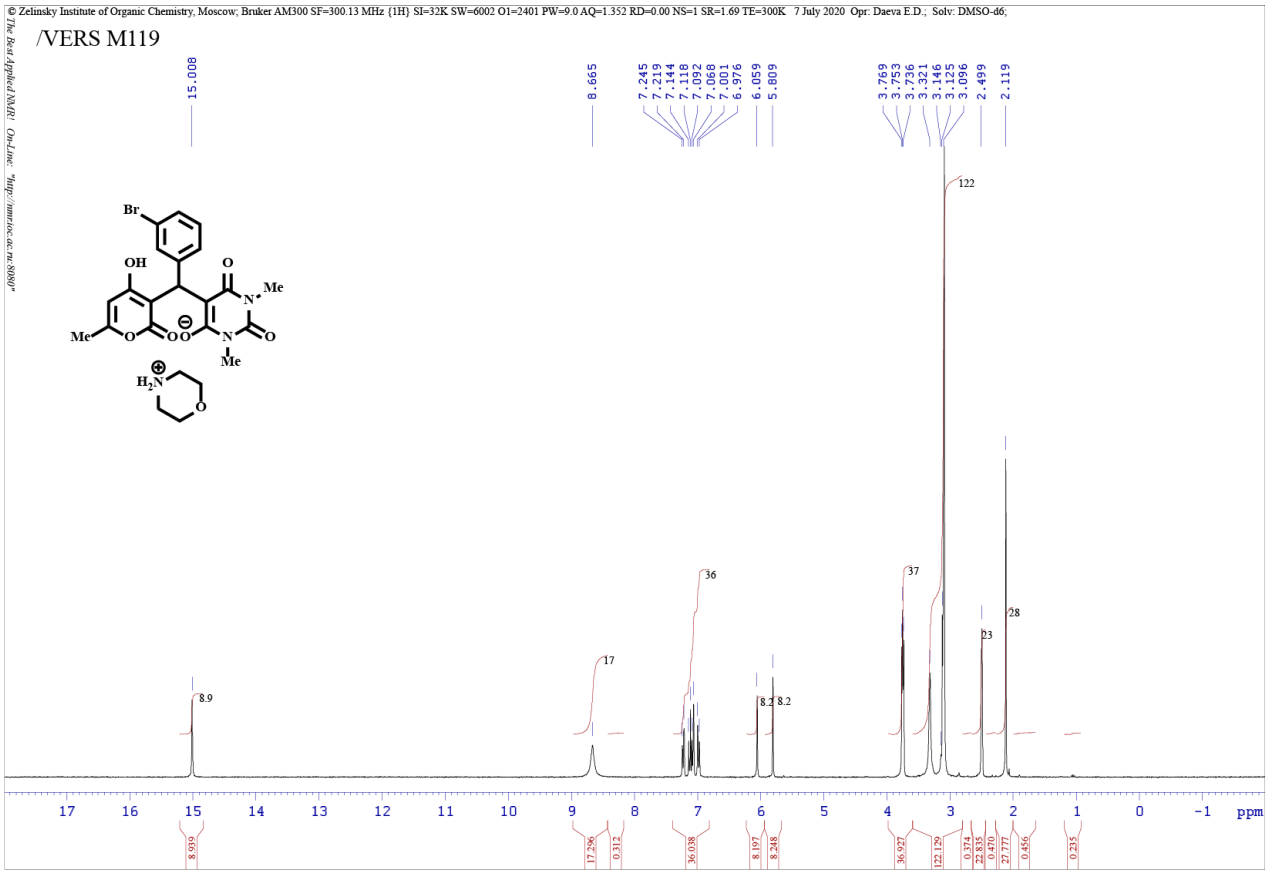
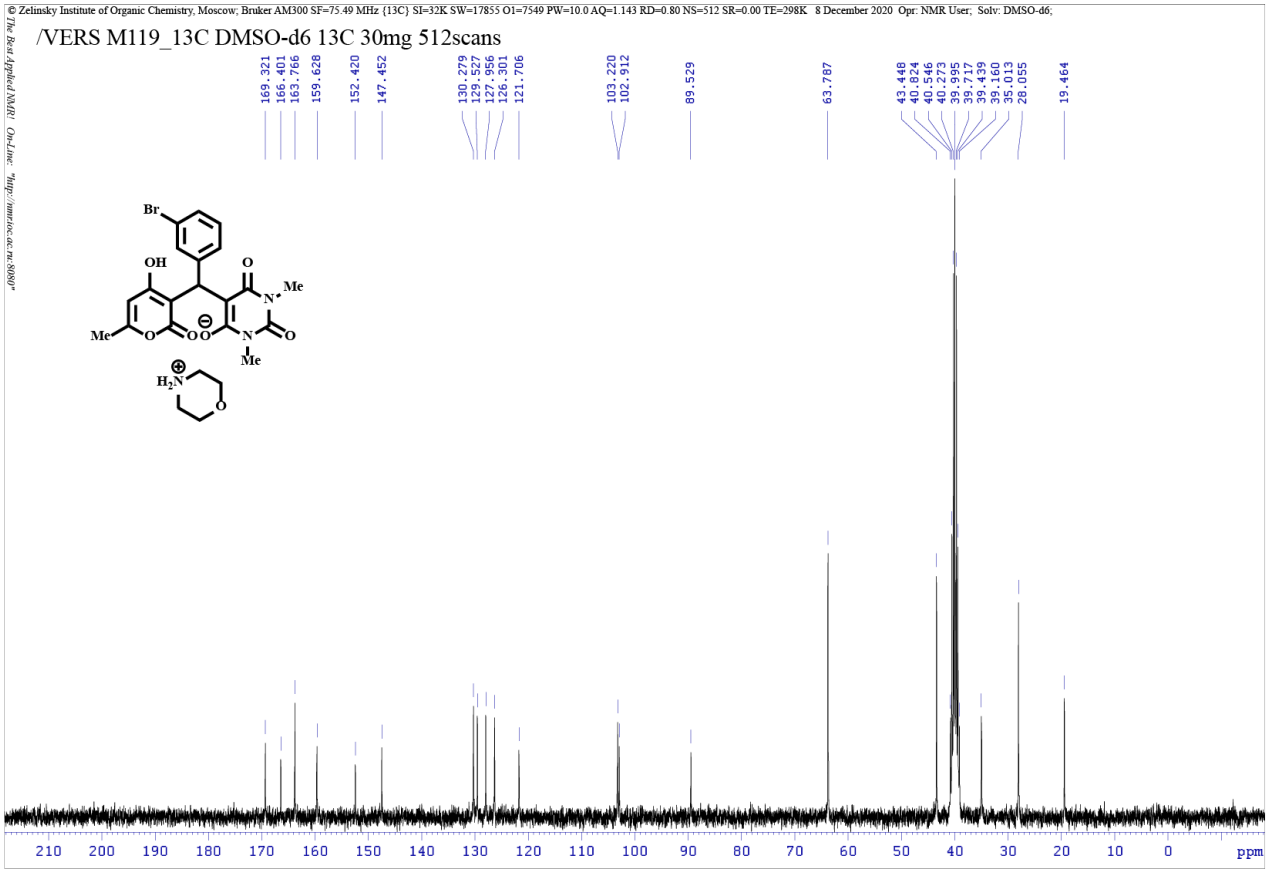
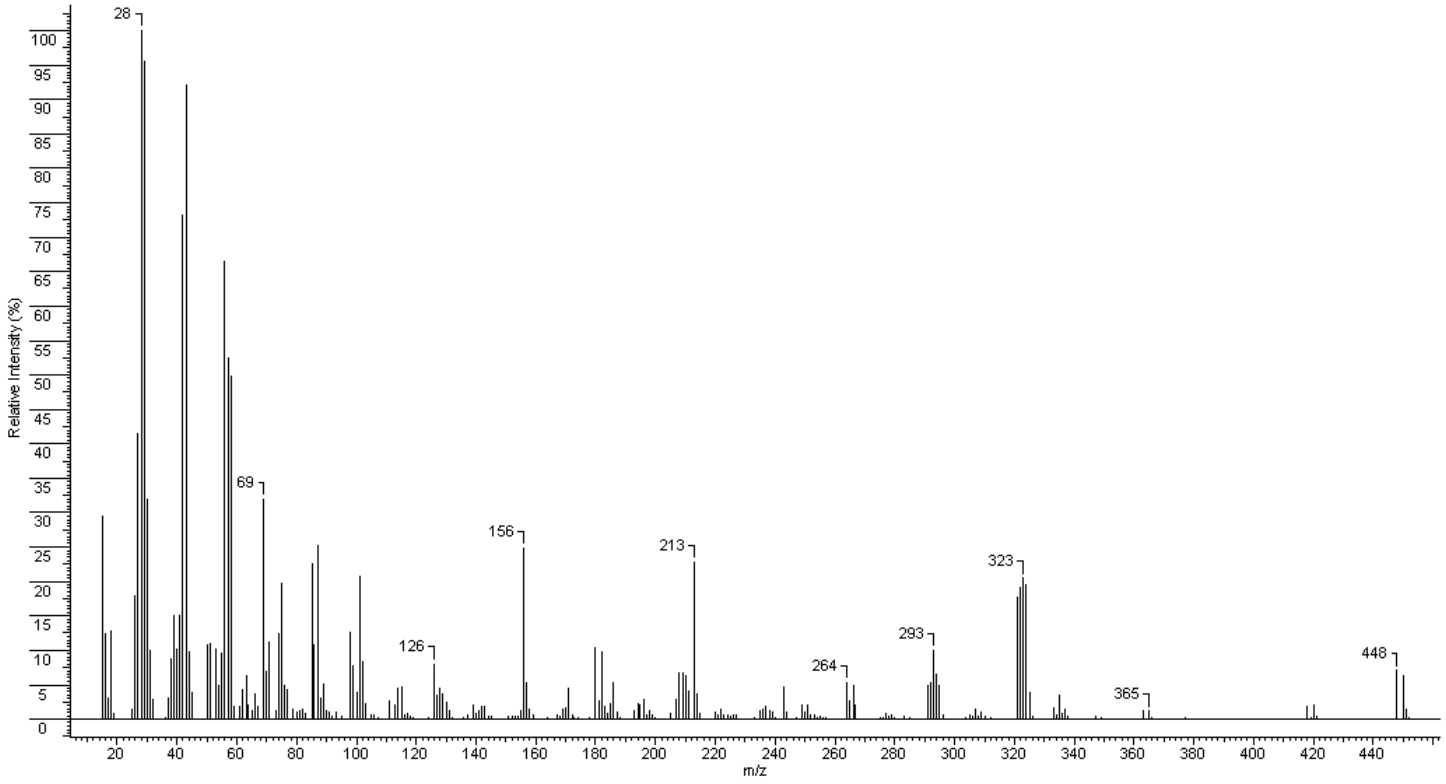
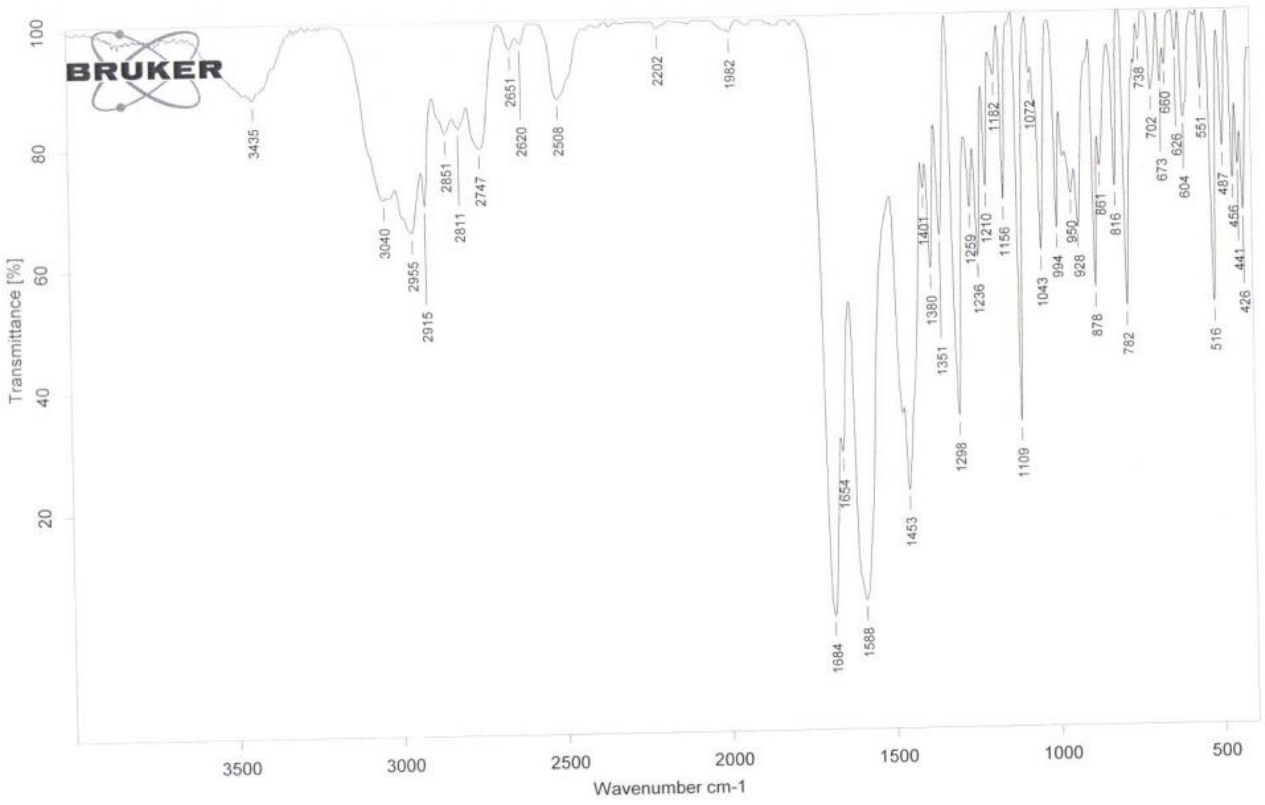
Morpholin-4-ium 5-((3-bromophenyl)(4-hydroxy-6-methyl-2-oxo-2*H*-pyran-3-yl)methyl)-1,3-dimethyl-2,4-dioxo-1,2,3,4-tetrahydropyrimidin-6-olate (2g).





Count	231	***Data Type***	MASS SPECTRUM	***Date***	15 Dec 2020 17:21:46
Date Stamp	15 Dec 1920 16:13:10				
File Name	G:\09.04.21\ms11.JC				
Inlet Model	DIRECT	***Instrumental Parameters***	LOW RESOLUTION	***Origin***	M 119
Owner	Copyright(C) by Victor (2020)	***Spectrum Title***	MASS SPECTRUM	***TIC***	1445.18





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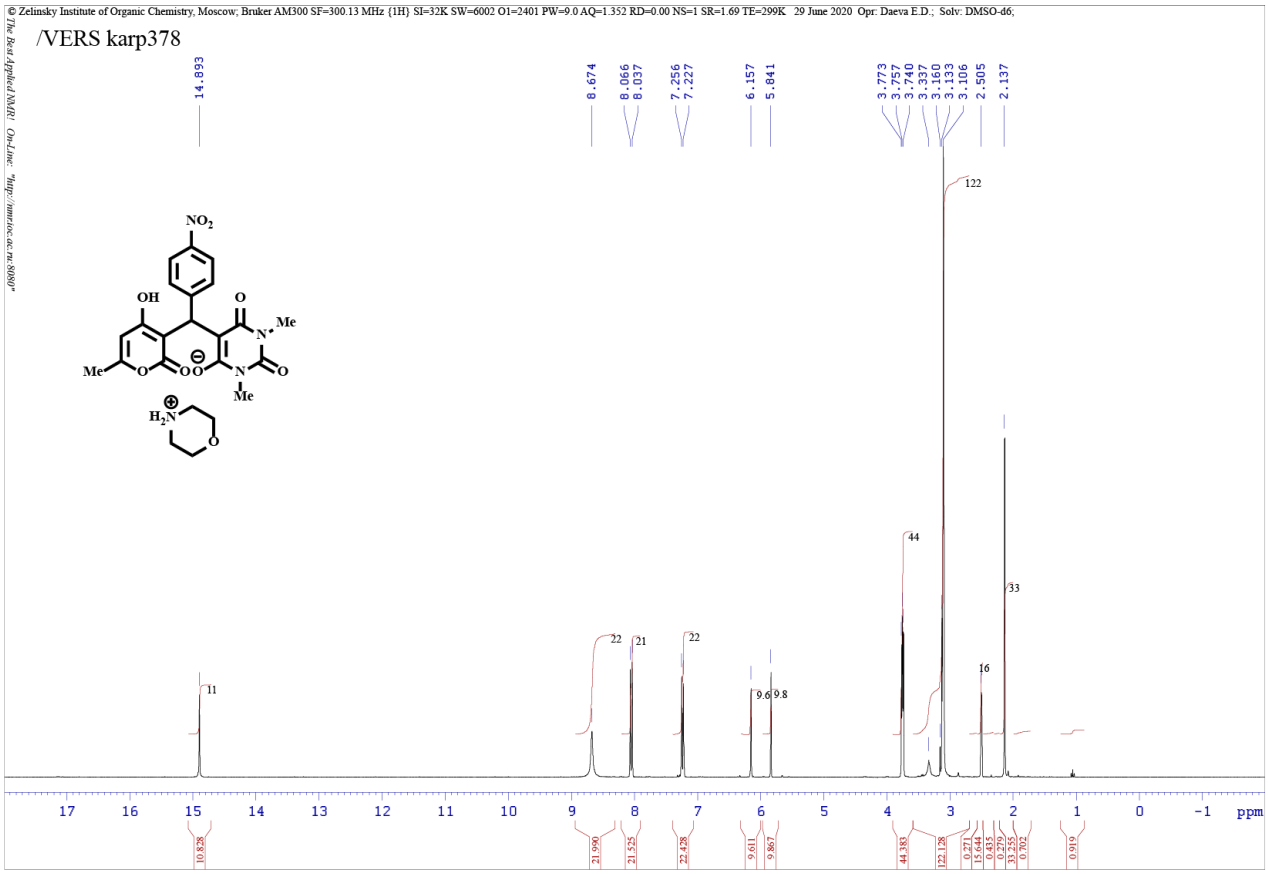
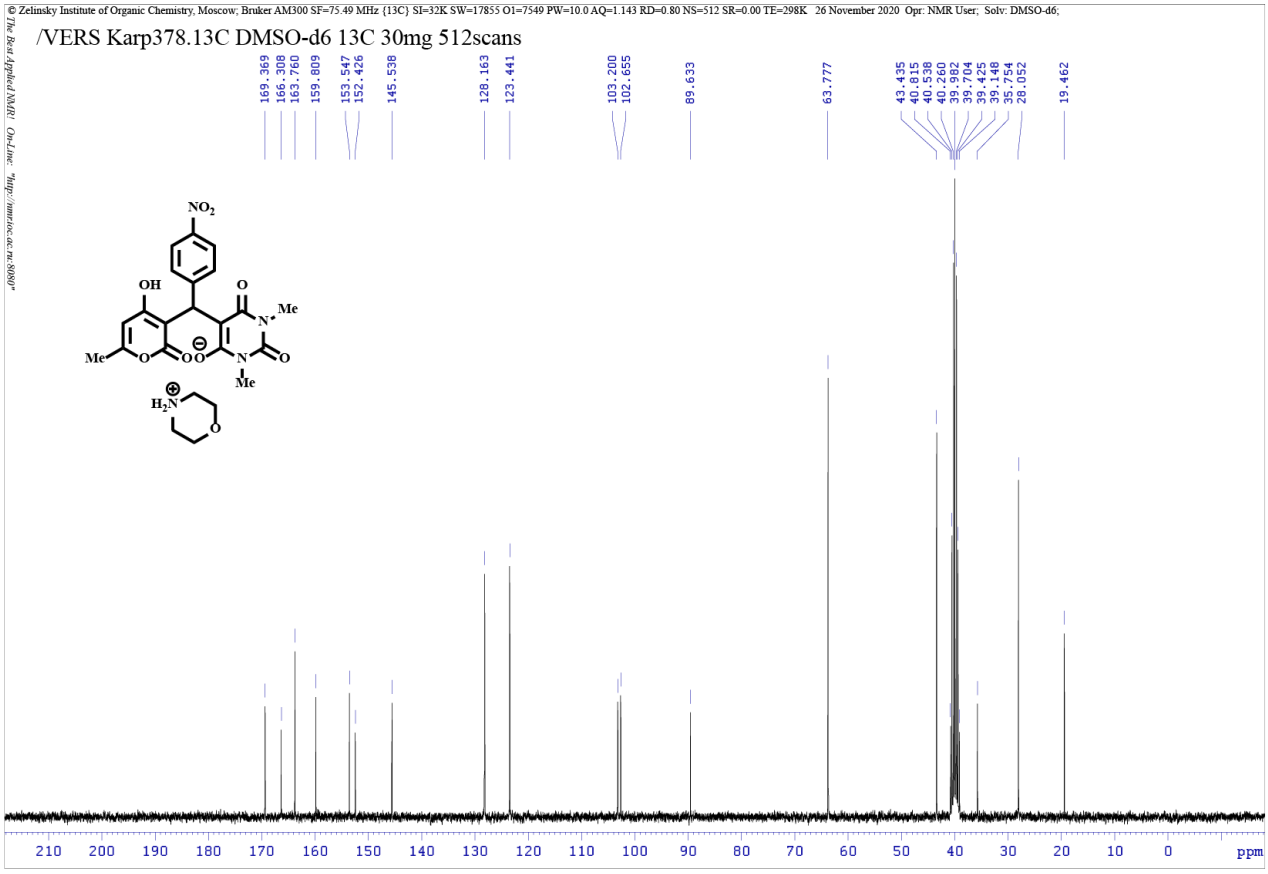
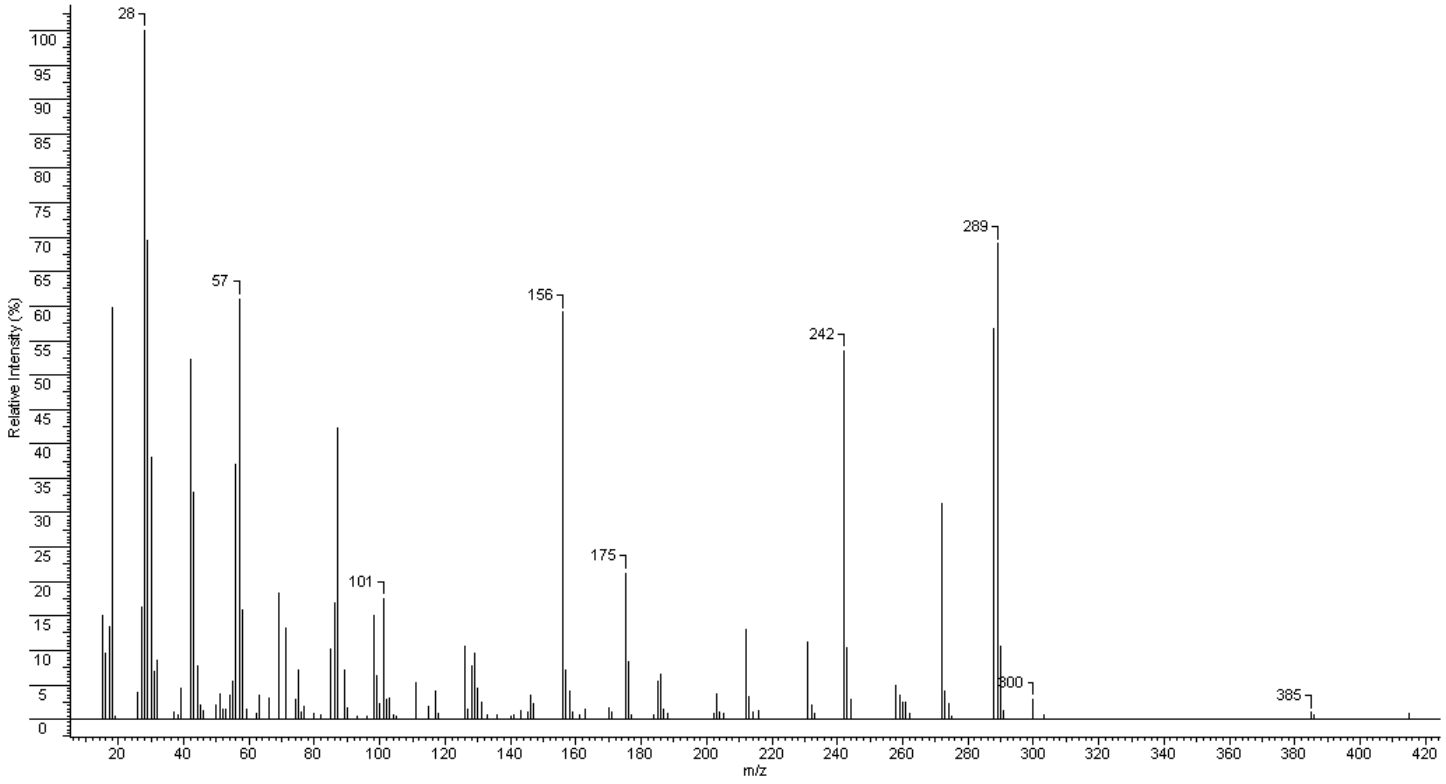
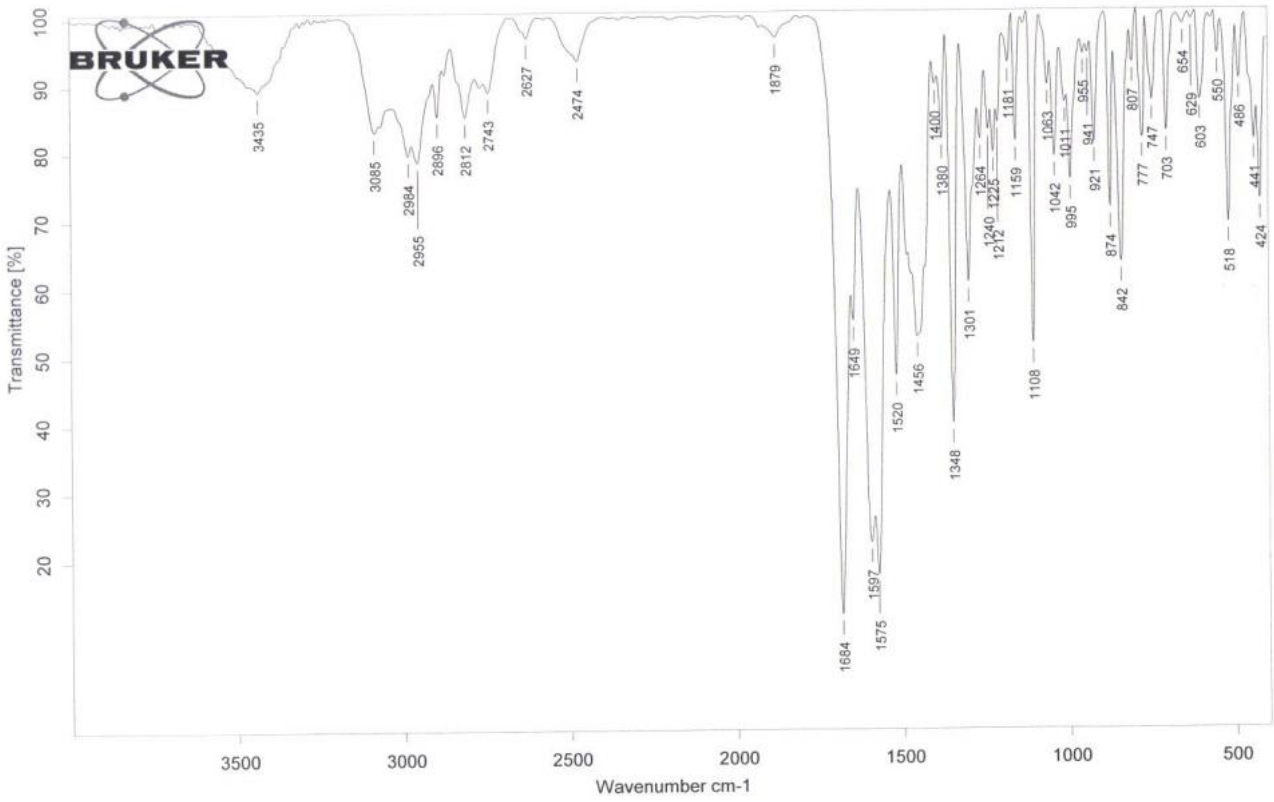
Morpholin-4-ium 5-((4-hydroxy-6-methyl-2-oxo-2*H*-pyran-3-yl)(4-nitrophenyl)methyl)-1,3-dimethyl-2,4-dioxo-1,2,3,4-tetrahydropyrimidin-6-olate (2h).





Count	122	***Data Type***	MASS SPECTRUM	***Date***	15 Dec 2020 17:21:46
Date Stamp	15 Dec 1920 16:13:10				
File Name	G:\09.04.21\ms11.JC				
Inlet Model	DIRECT	***Instrumental Parameters***	LOW RESOLUTION	***Origin***	KARP 378
Owner	Copyright(C) by Victor (2020)	***Spectrum Title***	MASS SPECTRUM	***TIC***	1237.98





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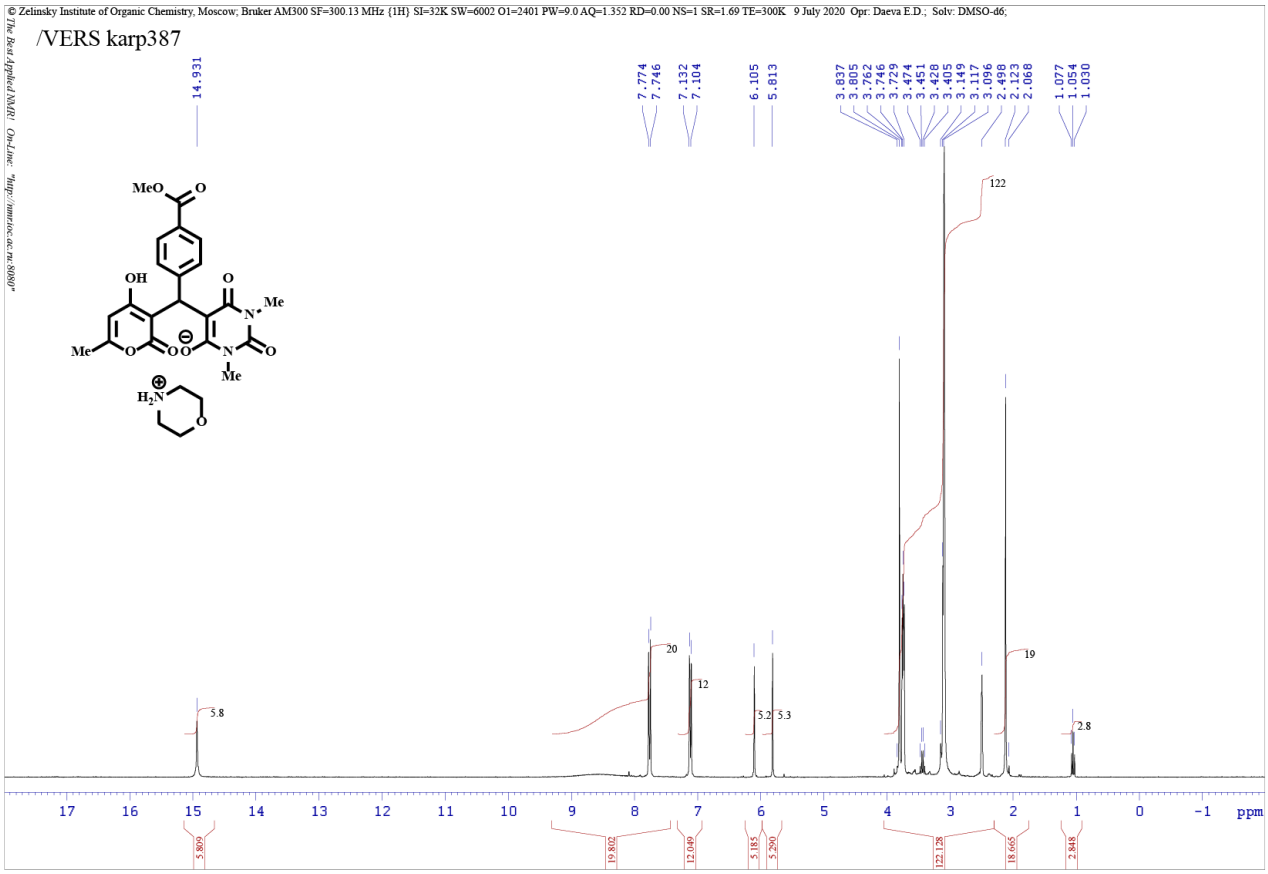
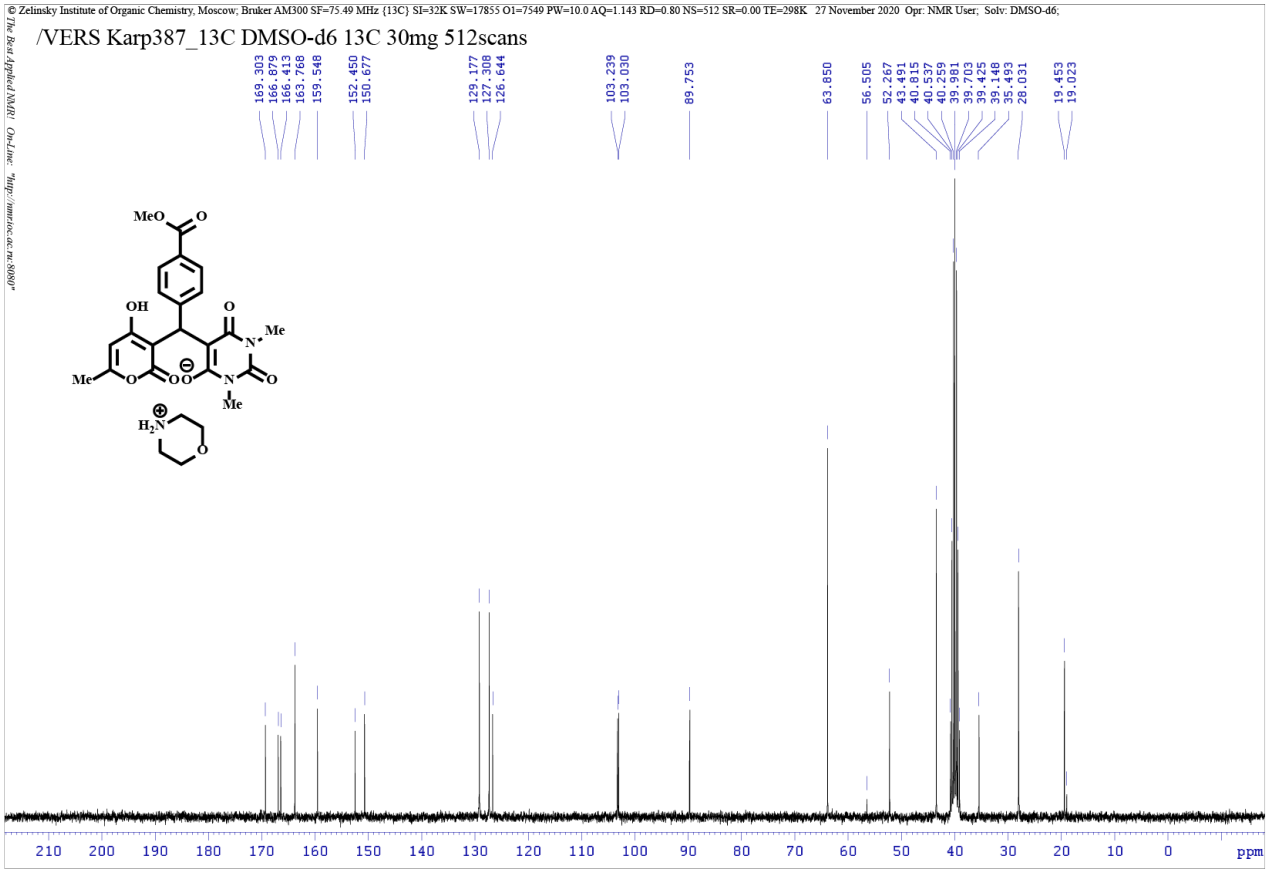
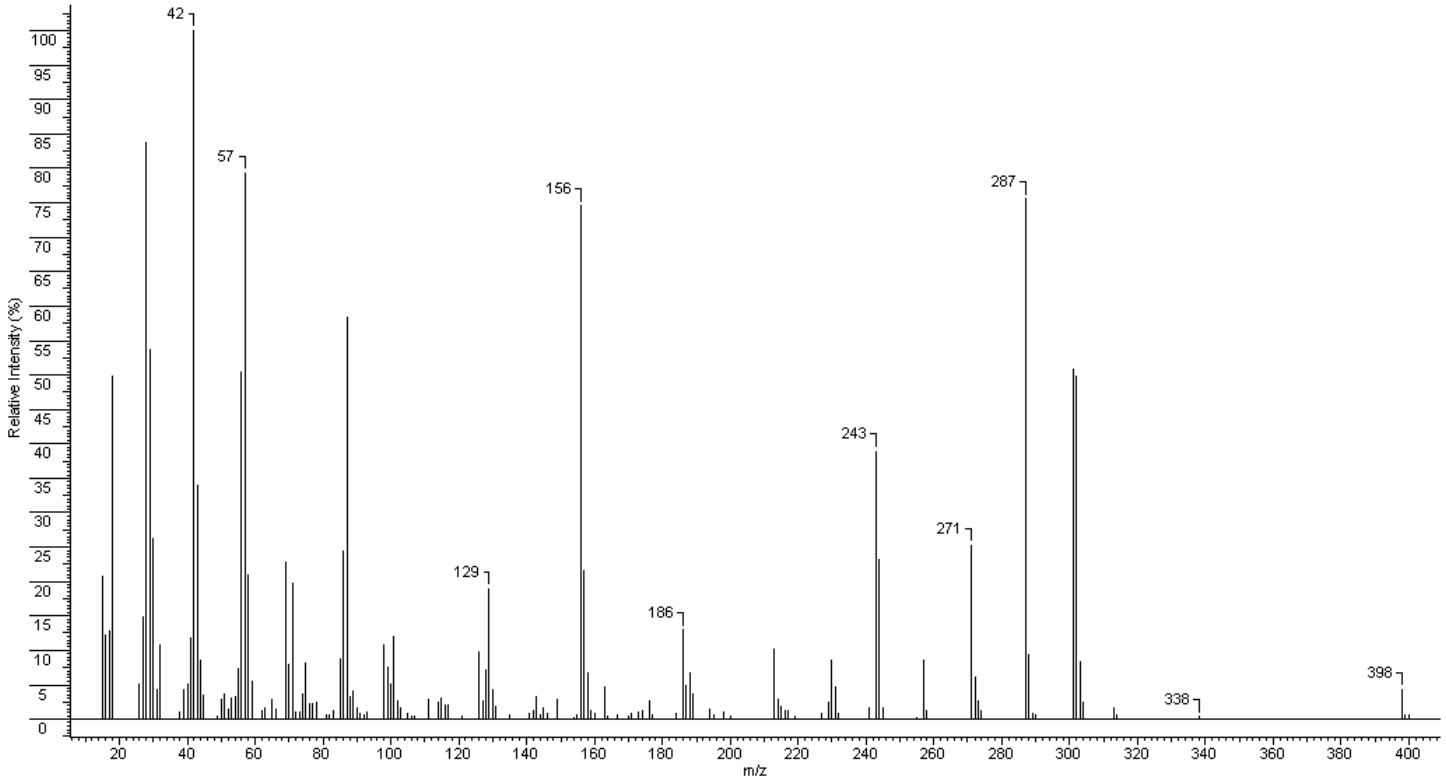
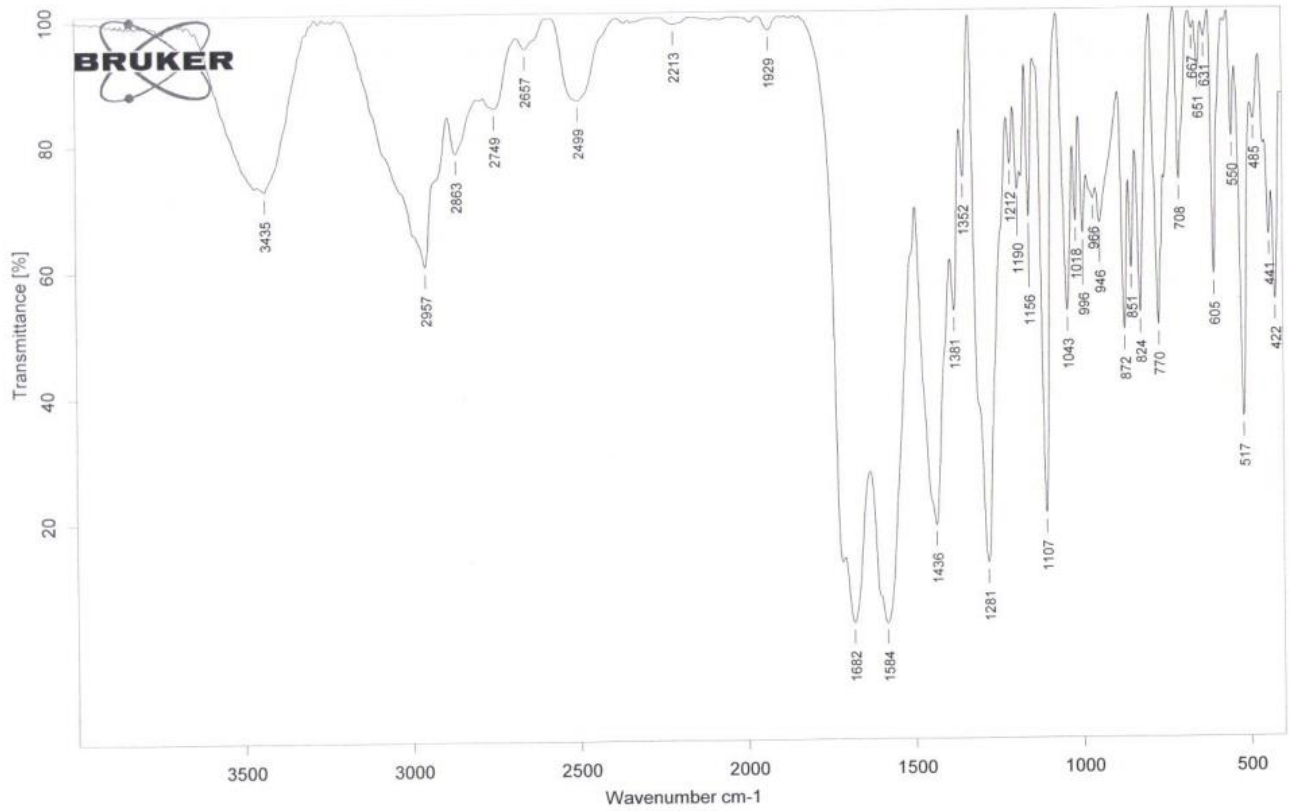
Morpholin-4-ium 5-((4-hydroxy-6-methyl-2-oxo-2*H*-pyran-3-yl)(4-(methoxycarbonyl)phenyl)methyl)-1,3-dimethyl-2,4-dioxo-1,2,3,4-tetrahydropyrimidin-6-olate (2i).





Count	146	***Data Type***	MASS SPECTRUM	***Date***	15 Dec 2020 17:21:46
Date Stamp	15 Dec 1920 16:13:10				
File Name	G:\09.04.21\ms11.JC				
Inlet Model	DIRECT	***Instrumental Parameters***	LOW RESOLUTION	***Origin***	KARP 387
Owner	Copyright(C) by Victor (2020)	***Spectrum Title***	MASS SPECTRUM	***TIC***	1438.17





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3. X-Ray data for compound 2h

Table 1. Crystal data and structure refinement

Identification code	**2h**
Empirical formula	$C_{23}H_{26}N_4O_9$
Formula weight	502.48
Temperature/K	296.15
Crystal system	monoclinic
Space group	$P2_1/c$
a/Å	10.5365(2)
b/Å	14.7314(2)
c/Å	15.4057(3)
α/°	90
β/°	98.4420(10)
γ/°	90
Volume/Å^3	2365.32(7)
Z	4
ρ_{calc}g/cm^3	1.411
μ/mm^{-1}	0.110
F(000)	1056.0
Crystal size/mm^3	0.2 × 0.2 × 0.2
Radiation	MoKα (λ = 0.71073)
2Θ range for data collection/°	3.846 to 61.09
Index ranges	$-15 \leq h \leq 14$, $-21 \leq k \leq 21$, $-22 \leq l \leq 12$
Reflections collected	26054
Independent reflections	7204 [R_{int} = 0.0353, R_{sigma} = 0.0358]
Data/restraints/parameters	7204/3/337
Goodness-of-fit on F^2	1.018
Final R indexes [I>=2σ (I)]	R_1 = 0.0522, wR_2 = 0.1290
Final R indexes [all data]	R_1 = 0.0821, wR_2 = 0.1481
Largest diff. peak/hole / e Å^{-3}	0.23/-0.21

Table 2. Fractional Atomic Coordinates ($\times 10^4$) and Equivalent Isotropic Displacement Parameters ($Å^2 \times 10^3$) for **2h**. U_{eq} is defined as 1/3 of of the trace of the orthogonalised U_{IJ} tensor.

Atom	***x***	***y***	***z***	**U(eq)**
O9	6599.7(17)	-44.2(9)	5604.6(11)	75.4(4)
O8	8489.5(16)	537.3(11)	5867.9(11)	80.1(5)
O3	963.5(15)	2858.9(11)	8893.7(13)	83.2(5)
O4	5237.5(11)	2570.1(7)	8825.5(8)	46.4(3)
O7	7144.0(10)	5895.6(7)	7429.6(8)	46.8(3)
O5	6971.5(13)	3731.4(8)	9031.4(9)	56.8(3)
O6	5652.9(12)	5367.7(8)	6411.2(8)	53.9(3)
O2	2674.8(11)	4508.8(9)	6877.3(10)	59.5(3)
N4	7334.7(18)	561.7(10)	5884.9(10)	57.9(4)
N3	3103.5(14)	2746.1(8)	8877.3(10)	46.4(3)
N2	1832.6(12)	3682.9(9)	7887.8(10)	48.5(3)
C12	4075.2(13)	3547.4(9)	7796.0(10)	37.1(3)
C9	6149.0(13)	4456.8(9)	7681.1(10)	35.5(3)
C13	4185.8(15)	2950.0(9)	8496.8(10)	38.7(3)
C11	5184.3(13)	3739.9(9)	7297.1(10)	35.6(3)
C19	5804.6(14)	2881.1(9)	6990.5(9)	36.7(3)
C5	6258.2(14)	5229.2(10)	7133.2(11)	39.0(3)
C8	6943.8(15)	4408.0(10)	8469.4(11)	41.4(3)
C15	2879.9(14)	3950.7(10)	7488.4(11)	41.7(3)
C22	6805.7(17)	1368.9(11)	6263.2(11)	46.4(4)
C6	7903.1(15)	5837.8(10)	8223.1(12)	45.2(4)
C7	7835.3(16)	5122.6(11)	8735.5(12)	47.5(4)
C20	7080.8(15)	2849.2(11)	6872.9(12)	48.7(4)
C14	1906.7(17)	3081.9(11)	8573.5(14)	52.8(4)
C21	7582.7(16)	2095.2(12)	6505.6(13)	54.7(4)
C18	5054.6(18)	2128.9(12)	6731.2(14)	57.3(5)
C10	8750.9(18)	6643.4(13)	8402.9(17)	66.5(6)

C23	5539(2)	1373.6(12)	6365.5(14)	61.7(5)
C16	3233(2)	2135.2(13)	9635.5(14)	65.6(5)
C17	566.3(17)	4053.9(16)	7551.0(17)	70.4(6)
O1	905.3(13)	5902.1(10)	4698.7(10)	69.5(4)
N1	3095.0(14)	6062.4(9)	5994.4(10)	46.6(3)
C2	770.8(18)	6137.0(15)	5569.0(16)	68.9(6)
C3	1946.2(19)	5294.2(16)	4718.3(15)	68.9(6)
C4	3186.6(18)	5732.7(16)	5100.7(13)	64.4(5)
C1	1939.4(19)	6628.8(13)	6009.7(16)	67.5(6)

Table 3. Anisotropic Displacement Parameters ($Å^2×10^3$) for **2h**. The Anisotropic displacement factor exponent takes the form: $-2\pi^2[h^2a^{*2}U_{11}+2hka^*b^*U_{12}+\ldots]$.

Atom	**U_{11}**	**U_{22}**	**U_{33}**	**U_{23}**	**U_{13}**	**U_{12}**
O9	108.3(12)	44.2(7)	78.1(11)	-9.9(7)	28.8(9)	-1.5(7)
O8	73.0(10)	82.7(10)	82.8(11)	-21.4(8)	5.6(8)	30.8(8)
O3	58.3(8)	85.1(10)	116.1(14)	12.5(9)	45.3(9)	-12.7(7)
O4	48.8(6)	36.6(5)	54.5(7)	9.9(5)	9.7(5)	1.8(4)
O7	40.7(6)	37.8(5)	61.2(7)	12.7(5)	5.6(5)	-3.9(4)
O5	66.8(8)	42.0(6)	56.1(7)	15.5(5)	-9.7(6)	-12.4(5)
O6	52.5(7)	52.6(7)	54.5(7)	18.1(5)	1.2(5)	-5.7(5)
O2	38.9(6)	58.0(7)	80.0(9)	22.8(7)	3.3(6)	3.2(5)
N4	80.0(12)	47.7(8)	46.8(9)	0.3(6)	12.6(8)	14.2(8)
N3	52.6(8)	37.1(6)	53.8(8)	0.7(6)	22.4(6)	-5.5(6)
N2	34.1(7)	47.5(7)	65.7(9)	-4.7(7)	13.0(6)	-2.7(5)
C12	34.4(7)	31.7(6)	46.9(8)	0.2(6)	11.7(6)	-0.8(5)
C9	33.3(7)	30.0(6)	44.2(8)	3.8(5)	8.8(6)	1.3(5)
C13	42.0(8)	29.8(6)	46.3(8)	-2.5(6)	13.1(6)	-1.9(5)
C11	33.4(7)	33.5(6)	40.3(7)	4.7(5)	7.1(5)	0.6(5)
C19	38.4(7)	35.8(7)	36.4(7)	3.6(5)	7.6(6)	0.4(5)
C5	34.0(7)	36.0(7)	48.5(9)	6.7(6)	11.0(6)	1.4(5)
C8	43.6(8)	32.5(7)	48.2(9)	5.4(6)	7.1(6)	0.7(6)

C15	35.2(7)	35.2(7)	55.1(9)	-0.8(6)	8.0(6)	-3.3(6)
C22	59.2(10)	40.5(7)	40.6(8)	1.0(6)	10.5(7)	8.5(7)
C6	34.8(7)	39.0(7)	61.6(10)	4.2(7)	6.4(7)	-1.1(6)
C7	45.1(9)	41.9(8)	52.8(10)	3.3(7)	-1.5(7)	-5.2(6)
C20	35.5(8)	45.3(8)	65.1(11)	-8.2(8)	6.6(7)	0.4(6)
C14	46.3(9)	45.6(8)	71.3(12)	-7.4(8)	24.4(8)	-9.3(7)
C21	39.3(8)	57.3(10)	66.8(12)	-10.3(8)	5.7(8)	9.3(7)
C18	49.9(10)	51.2(9)	76.1(13)	-14.6(9)	27.4(9)	-13.5(8)
C10	49.2(10)	49.9(10)	96.4(16)	10.9(10)	-2.9(10)	-13.8(8)
C23	67.8(12)	45.0(9)	78.9(14)	-14.6(9)	32.0(10)	-15.7(8)
C16	83.6(14)	55.8(10)	64.2(12)	10.3(9)	34.0(10)	-7.3(10)
C17	32.3(8)	82.5(14)	96.4(17)	-0.8(12)	9.2(9)	1.1(9)
O1	48.9(7)	77.9(9)	74.1(10)	-11.4(8)	-16.6(6)	7.5(6)
N1	42.6(7)	44.1(7)	50.0(8)	0.3(6)	-3.5(6)	-5.1(6)
C2	43.3(10)	64.2(11)	96.8(17)	-27.9(11)	2.0(10)	7.1(8)
C3	53.1(11)	89.2(15)	61.4(12)	-28.5(11)	-1.7(9)	7.2(10)
C4	47.8(10)	93.4(15)	50.3(11)	-3.7(10)	2.0(8)	2.7(10)
C1	57.5(11)	49.9(10)	90.0(15)	-26.0(10)	-6.3(10)	6.3(8)

Table 4. Bond Lengths for **2h**.

Atom	Atom	Length/Å	Atom	Atom	Length/Å
O9	N4	1.218(2)	C9	C11	1.5238(19)
O8	N4	1.221(2)	C9	C5	1.4314(19)
O3	C14	1.218(2)	C9	C8	1.372(2)
O4	C13	1.2778(18)	C11	C19	1.530(2)
O7	C5	1.3850(18)	C19	C20	1.384(2)
O7	C6	1.362(2)	C19	C18	1.385(2)
O5	C8	1.3177(18)	C8	C7	1.430(2)
O6	C5	1.2155(19)	C22	C21	1.366(2)
O2	C15	1.245(2)	C22	C23	1.366(3)
N4	C22	1.470(2)	C6	C7	1.325(2)

N3	C13	1.389(2)	C6	C10	1.487(2)
N3	C14	1.371(2)	C20	C21	1.386(2)
N3	C16	1.465(2)	C18	C23	1.379(3)
N2	C15	1.397(2)	O1	C2	1.412(3)
N2	C14	1.372(2)	O1	C3	1.413(2)
N2	C17	1.465(2)	N1	C4	1.476(2)
C12	C13	1.384(2)	N1	C1	1.479(2)
C12	C11	1.517(2)	C2	C1	1.502(3)
C12	C15	1.410(2)	C3	C4	1.499(3)

Table 5. Bond Angles for **2h**.

Atom	**Atom**	**Atom**	**Angle/°**	**Atom**	**Atom**	**Atom**	**Angle/°**
C6	O7	C5	122.02(12)	O6	C5	C9	126.61(14)
O9	N4	O8	123.65(17)	O5	C8	C9	124.95(14)
O9	N4	C22	118.36(17)	O5	C8	C7	114.72(14)
O8	N4	C22	118.00(16)	C9	C8	C7	120.33(13)
C13	N3	C16	118.90(15)	O2	C15	N2	117.33(14)
C14	N3	C13	123.27(14)	O2	C15	C12	125.44(15)
C14	N3	C16	117.83(15)	N2	C15	C12	117.21(14)
C15	N2	C17	118.43(16)	C21	C22	N4	119.69(16)
C14	N2	C15	124.21(14)	C21	C22	C23	121.39(16)
C14	N2	C17	117.36(15)	C23	C22	N4	118.91(16)
C13	C12	C11	122.08(13)	O7	C6	C10	112.03(15)
C13	C12	C15	119.91(14)	C7	C6	O7	120.58(14)
C15	C12	C11	117.87(13)	C7	C6	C10	127.39(17)
C5	C9	C11	115.39(13)	C6	C7	C8	120.34(15)
C8	C9	C11	126.37(12)	C19	C20	C21	121.13(15)
C8	C9	C5	118.17(13)	O3	C14	N3	121.92(19)
O4	C13	N3	117.12(14)	O3	C14	N2	121.90(18)
O4	C13	C12	123.80(14)	N3	C14	N2	116.18(14)
C12	C13	N3	119.08(14)	C22	C21	C20	119.35(16)

C12	C11	C9	116.90(12)	C23	C18	C19	122.24(17)
C12	C11	C19	113.42(11)	C22	C23	C18	118.54(16)
C9	C11	C19	113.71(11)	C2	O1	C3	108.72(15)
C20	C19	C11	122.40(13)	C4	N1	C1	111.53(15)
C20	C19	C18	117.34(15)	O1	C2	C1	110.95(19)
C18	C19	C11	119.93(14)	O1	C3	C4	111.38(17)
O7	C5	C9	118.53(13)	N1	C4	C3	109.62(17)
O6	C5	O7	114.86(13)	N1	C1	C2	110.05(14)

Table 6. Hydrogen Atom Coordinates (Å×10^4) and Isotropic Displacement Parameters (Å^2×10^3) for **2h**.

Atom	***x***	***y***	***z***	**U(eq)**
H11	4769	4016	6749	43
H7	8370	5085	9271	57
H20	7611	3342	7043	58
H21	8440	2084	6425	66
H18	4195	2134	6806	69
H10A	9278	6703	7948	100
H10B	9287	6569	8958	100
H10C	8235	7179	8418	100
H23	5016	878	6192	74
H16A	3194	1517	9436	98
H16B	2548	2246	9969	98
H16C	4041	2242	9998	98
H17A	616	4704	7528	106
H17B	-36	3880	7932	106
H17C	288	3821	6972	106
H2A	641	5591	5897	83
H2B	22	6521	5566	83
H3A	2004	5091	4127	83
H3B	1797	4766	5066	83
H4A	3881	5297	5122	77

H4B	3370	6237	4734	77
H1C	2035	7198	5709	81
H1D	1841	6765	6612	81
H5	6378(19)	3337(14)	8908(16)	81
H1A	3030(20)	5602(13)	6338(14)	81
H1B	3786(18)	6389(15)	6192(16)	81

4. References

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