Electronic supplementary materials *Mendeleev Commun.*, 2026, **36**, 58–60

Assembly of aldehydes and dimethylbarbituric acid into spirotricyclic furopyrimidines under the action of *N*-bromosuccinimide in water

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Table of contents

1. Experimental section S1

2. ^{1}H and ^{13}C NMR spectra for new compounds **3c,d,f,g-k** S4

3. References S12

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) at ambient temperature in $CDCl_3$ 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. Elemental analysis was performed on elemental analyzer multi EA® 5000.

General procedure

Aldehyde **1** (1 mmol), *N*,*N*′-dimethylbarbituric acid **2** (2 mmol), and NBS (1.2 mmol) were stirred in water (5 ml) at ambient temperature. The reaction was completed (the color of the reaction mixture became pale), the formed solid was filtered, washed with an ice-cold ethanol/water solution (1:1, 3 ml), and dried to isolate pure substituted spiro[furo[2,3-*d*]pyrimidine-6,5'-pyrimidine]-2,2',4,4',6'(1'*H*,3*H*,3'*H*)-pentones **3**.

1,1′,3,3′-Tetramethyl-5-phenyl-1,5-dihydro-2*H*,2′*H*-spiro(furo[2,3-*d*]pyrimidine-6,5′-pyrimidine)-2,2′,4,4′,6′(1′*H*,3*H*,3′*H*)-pentone (3a).
Yield 0.39 g (98%), white powder, mp: 256–258 °C (lit.[S1] mp 256–258°C). Spectral data correspond to literary.

1,1′,3,3′-Tetramethyl-5-(4-methylphenyl)-1,5-dihydro-2*H*,2′*H*-spiro[furo[2,3-*d*]pyrimidine-6,5′-pyrimidine]-2,2′,4,4′,6′(1′*H*,3*H*,3′*H*)-pentone (3b).
Yield 0.40 g (97%), white powder, mp: 220–221 °C (lit.[S2] mp 219–221°C). Spectral data correspond to literary.

5-(3-Methoxyphenyl)-1,1',3,3'-tetramethyl-1,5-dihydro-2*H*,2'*H*-spiro[furo[2,3-*d*]pyrimidine-6,5'-pyrimidine]-2,2',4,4',6'(1'*H*,3*H*,3'*H*)-pentone (3c).

Yield 0.41 g (95%), white powder, mp 238–239°C. ^{1}H NMR spectrum, *δ*, ppm (*J*, Hz): 2.65 (3H, s, CH_3), 3.33 (3H, s, CH_3), 3.45 (3H, s, CH_3), 3.54 (3H, s, CH_3), 3.78 (3H, s, OCH_3), 4.90 (1H, s, CH), 6.59 (1H, s, H Ar), 6.65 (1H, d, ^3J = 7.6, H Ar), 6.89 (1H, d, ^3J = 7.6, H Ar), 7.26 (1H, t, ^3J = 7.6, H Ar). ^{13}C NMR spectrum, *δ*, ppm: 28.3, 28.6, 29.6, 30.1, 55.5, 56.3, 85.7, 90.4, 113.8, 115.2, 120.6, 130.1, 134.4, 149.7, 151.3, 158.5, 160.0, 162.8, 163.0, 165.6. IR spectrum, ν, cm–1: 2954, 1696, 1661, 1517, 1438, 1380, 1284, 1043, 750. Mass spectrum, m/z (I_{rel}, %): 428 [M]+ (43), 400 (8), 341 (14), 313 (12), 273 (100), 227 (20), 216 (19), 172 (24), 132 (7), 58 (7). Found, %: C, 55.93; H, 4.67; N, 13.01. $C_{20}H_{20}N_4O_7$. Calculated, %: C, 56.07; H, 4.71; N, 13.08.

5-(2-Bromophenyl)-1,1',3,3'-tetramethyl-1,5-dihydro-2*H*,2'*H*-spiro[furo[2,3-*d*]pyrimidine-6,5'-pyrimidine]-2,2',4,4',6'(1'*H*,3*H*,3'*H*)-pentone (3d).

Yield 0.46 g (96%), white powder, mp 247–248°C (decomp); (lit.[S3] mp 254.4–254.8°C). Spectral data correspond to literary.

1,1',3,3'-Tetramethyl-5-(3-nitrophenyl)-2*H*,2'*H*-spiro(furo[2,3-*d*]pyrimidine-6,5'-pyrimidine]-2,2',4,4',6'(1'*H*,3*H*,3'*H*)-pentone (3e).

Yield 0.39 g (97%), white powder, mp: 266–268 °C (lit.[S4] mp 265–267°C). Spectral data correspond to literary.

Methyl 4-(1,1',3,3'-tetramethyl-2,2',4,4',6'-pentaoxo-1,1',3,3',4,4',5,6'-octahydro-2*H*,2′*H*-spiro-[furo[2,3-*d*]pyrimidine-6,5'-pyrimidin]-5-yl)benzoate (3f).

Yield 0.43 g (95%), white solid, mp 232–233°C. ^{1}H NMR spectrum, *δ*, ppm (*J*, Hz): 2.61 (3H, s, CH_3), 3.32 (3H, s, CH_3), 3.47 (3H, s, CH_3), 3.55 (3H, s, CH_3), 3.93 (3H, s, OCH_3), 4.98 (1H, s, CH), 7.18 (2H, d, ^3J = 6.9, H Ar), 8.03 (2H, d, ^3J = 6.9, H Ar). ^{13}C NMR spectrum, *δ*, ppm: 28.4, 28.6, 29.9, 30.2, 52.5, 59.0, 85.4, 89.9, 128.6 (2C), 130.2 (2C), 131.3, 138.0, 149.5, 151.2, 158.7, 162.8, 162.9, 165.3, 166.3. IR spectrum, ν, cm^{-1}: 2957, 1698, 1672, 1520, 1431, 1386, 1282, 1183, 1041, 750. Mass spectrum, *m/z* (I_{rel}, %): 456 $[M]^+$ (61), 441 (14), 425 (8), 397 (9), 369 (32), 301 (100), 255 (20), 200 (39), 129 (24), 66 (14). Found, %: C, 55.19; H, 4.37; N, 12.14. $C_{21}H_{20}N_4O_8$. Calculated, %: C, 55.26; H, 4.42; N, 12.28.

5-(2,3-Dimethoxyphenyl)-1,1',3,3'-tetramethyl-1,5-dihydro-2*H*,2'*H*-spiro[furo[2,3-*d*]pyrimidine-6,5'-pyrimidine]-2,2',4,4',6'(1'*H*,3*H*,3'*H*)-pentone (3g).

Yield 0.41 g (90%), white powder, mp 256–257°C. ^{1}H NMR spectrum, *δ*, ppm (*J*, Hz): 2.73 (3H, s, CH_3), 3.35 (3H, s, CH_3), 3.43 (3H, s, CH_3), 3.51 (3H, s, CH_3), 3.84 (3H, s, OCH_3), 3.85 (3H, s, OCH_3), 5.34 (1H, s, CH), 6.67 (1H, d, ^3J = 8.0, H Ar), 6.89 (1H, d, ^3J = 8.0, H Ar), 7.03 (1H, t, ^3J = 8.0, H Ar). ^{13}C NMR spectrum, *δ*, ppm: 28.2, 28.4, 29.4, 30.0, 52.1, 56.1, 60.9, 85.4, 88.8, 113.5, 121.2, 124.3, 126.6, 146.6, 149.9, 151.3, 152.2, 158.9, 162.6, 163.4, 166.0. IR spectrum, ν, cm^{-1}: 2942, 2836, 1696, 1657, 1518, 1483, 1445, 1384, 1290, 750. Mass spectrum, *m/z* (I_{rel}, %): 458 $[M]^+$ (9), 427 (100), 370 (6), 313 (53), 303 (11), 273 (20), 216 (9), 172 (8), 77 (4), 58 (9). Found, %: C, 54.89; H, 4.77; N, 12.15. $C_{21}H_{22}N_4O_8$. Calculated, %: C, 55.02; H, 4.84; N, 12.22.

5-(2,5-Dimethoxyphenyl)-1,1',3,3'-tetramethyl-1,5-di-hydro-2*H*,2'*H*-spiro[furo[2,3-*d*]pyrimidine-6,5'-pyrimidine]-2,2',4,4',6'(1'*H*,3*H*,3'*H*)-pentone (3h).

Yield 0.42 g (92%), white powder, mp 245–247°C. ^{1}H NMR spectrum, *δ*, ppm (*J*, Hz): 2.70 (3H, s, CH_3), 3.33 (3H, s, CH_3), 3.42 (3H, s, CH_3), 3.51 (3H, s, CH_3), 3.69 (3H, s, OCH_3), 3.72 (3H, s, OCH_3), 5.44 (1H, s, CH), 6.60 (1H, d, ^4J = 2.2, H Ar), 6.75 (1H, d, ^3J = 8.9, H Ar), 6.80 (1H, dd, ^3J = 8.9, ^4J = 2.2, H Ar). ^{13}C NMR

spectrum, *δ*, ppm: 28.3, 28.4, 29.3, 30.0, 51.6, 55.8, 56.2, 85.1, 89.0, 110.5, 114.6, 116.2, 122.1, 150.1, 150.8, 151.4, 153.8, 158.7, 162.6, 163.3, 165.9. IR spectrum, ν, cm^{-1}: 2949, 2834, 1689, 1660, 1501, 1441, 1382, 1281, 1183, 1023. Mass spectrum, *m/z* (I_{rel}, %): 458 $[M]^+$ (49), 427 (100), 370 (15), 313 (70), 303 (14), 273 (18), 216 (10), 172 (17), 101 (4), 58 (11). Found, %: C, 54.93; H, 4.81; N, 12.11. $C_{21}H_{22}N_4O_8$. Calculated, %: C, 55.02; H, 4.84; N, 12.22.

5-(3,4-Dimethoxyphenyl)-1,1',3,3'-tetramethyl-1,5-di-hydro-2*H*,2'*H*-spiro[furo[2,3-*d*]pyrimidine-6,5'-pyrimidine]-2,2',4,4',6'(1'*H*,3*H*,3'*H*)-pentone (3i).

Yield 0.43 g (93%), white powder, mp 232–234°C. ^{1}H NMR spectrum, *δ*, ppm (*J*, Hz): 2.68 (3H, s, CH_3), 3.33 (3H, s, CH_3), 3.45 (3H, s, CH_3), 3.55 (3H, s, CH_3), 3.84 (3H, s, OCH_3), 3.88 (3H, s, OCH_3), 4.89 (1H, s, CH), 6.54 (1H, d, ^4J = 1.5, H Ar), 6.65 (1H, dd, ^3J = 8.2, ^4J = 1.5, H Ar), 6.83 (1H, d, ^3J = 8.2, H Ar). ^{13}C NMR spectrum, *δ*, ppm: 28.2, 28.7, 29.5, 30.0, 56.0 (2C), 59.4, 85.6, 90.4, 111.0, 111.3, 121.0, 125.0, 149.2, 149.8, 150.0, 151.3, 158.7, 162.6, 163.2, 165.6. IR spectrum, ν, cm^{-1}: 2965, 1693, 1657, 1522, 1422, 1259, 1146, 1025, 752. Mass spectrum, *m/z* (I_{rel}, %): 458 $[M]^+$ (65), 400 (14), 372 (7), 343 (9), 303 (100), 246 (13), 202 (10), 138 (10), 101 (6), 58 (10). Found, %: C, 54.88; H, 4.79; N, 12.08. $C_{21}H_{22}N_4O_8$. Calculated, %: C, 55.02; H, 4.84; N, 12.22.

5-(5-Bromo-2-methoxyphenyl)-1,1',3,3'-tetramethyl-1,5-dihydro-2*H*,2'*H*-spiro[furo[2,3-*d*]pyrimidine-6,5'-pyrimidine]-2,2',4,4',6'(1'*H*,3*H*,3'*H*)-pentone (3j).

Yield 0.49 g (96%), yellowish powder, mp 286–287°C. ^{1}H NMR spectrum, *δ*, ppm (*J*, Hz): 2.73 (3H, s, CH_3), 3.35 (3H, s, CH_3), 3.43 (3H, s, CH_3), 3.52 (3H, s, CH_3), 3.73 (3H, s, OCH_3), 5.41 (1H, s, CH), 6.73 (1H, d, ^3J = 8.7, H Ar), 7.15 (1H, d, ^4J = 1.9, H Ar), 7.41 (1H, dd, ^3J = 8.7, ^4J = 1.9, H Ar). ^{13}C NMR spectrum, *δ*, ppm: 28.3, 28.5, 29.3, 30.0, 51.0, 56.2, 84.7, 88.8, 111.6, 113.7, 123.6, 132.7, 133.1, 150.1, 151.3, 155.7, 158.6, 162.8, 163.1, 165.6. IR spectrum, ν, cm^{-1}: 2951, 2845, 1696, 1659, 1516, 1443, 1287, 1039, 752. Mass spectrum, *m/z* (I_{rel}, %): 508 $[M(^{81}Br)]^+$ (25), 506 $[M(^{79}Br)]^+$ (25), 477 (^{81}Br) (100), 475 (^{79}Br) (94), 363 (^{81}Br) (65), 361 (^{79}Br) (62), 323 (^{81}Br) (22), 321 (^{79}Br) (25), 211 (^{81}Br) (18), 209 (^{79}Br) (20), 58 (29). Found, %: C, 47.18; H, 3.73; Br, 15.63; N, 10.88. $C_{20}H_{19}BrN_4O_7$. Calculated, %: C, 47.35; H, 3.78; Br, 15.75; N, 11.04.

5-(2,3-Dichlorophenyl)-1,1',3,3'-tetramethyl-1,5-dihydro-2*H*,2'*H*-spiro[furo[2,3-*d*]pyrimidine-6,5'-pyrimidine]-2,2',4,4',6'(1'*H*,3*H*,3'*H*)-pentone (3k).

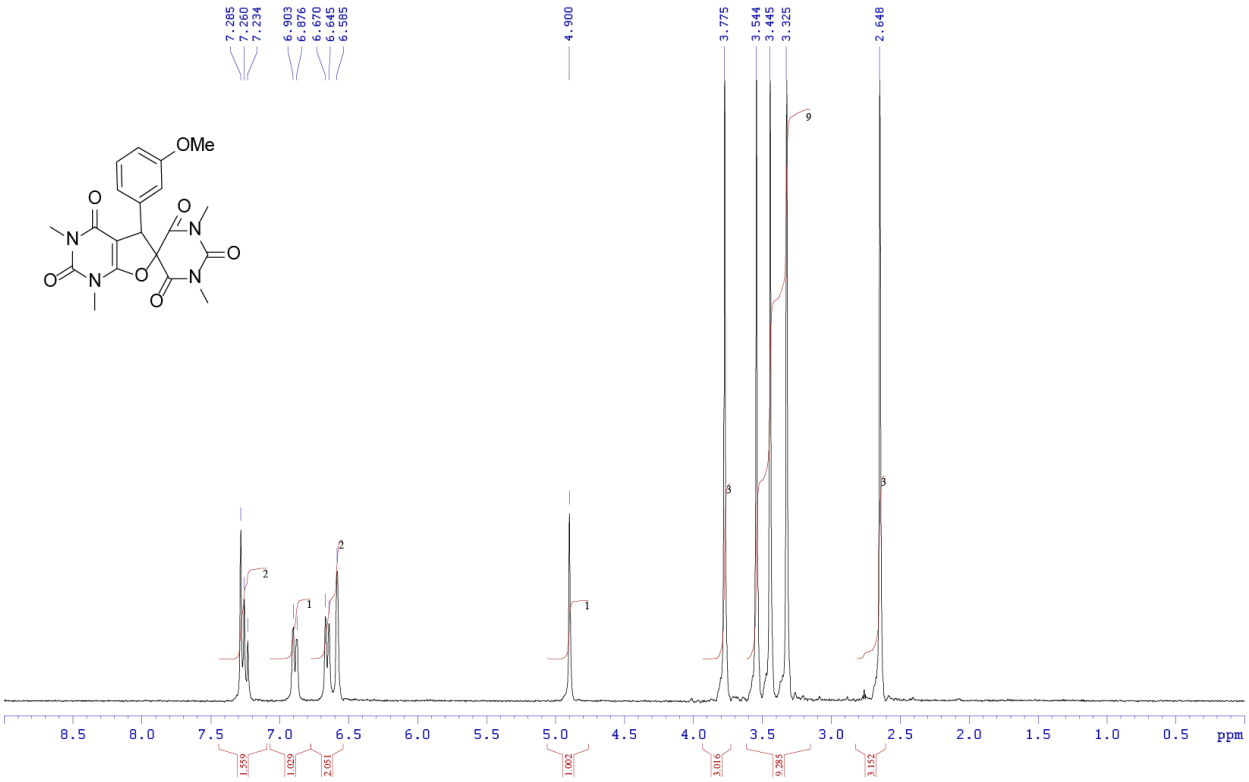
Yield 0.46 g (98%), white powder, mp 267–268°C (decomp). ^{1}H NMR spectrum, *δ*, ppm (*J*, Hz): 2.77 (3H, s, CH_3), 3.32 (3H, s, CH_3), 3.42 (3H, s, CH_3), 3.52 (3H, s, CH_3), 5.59 (1H, s, CH), 7.10 (1H, d, ^3J = 7.6, H Ar), 7.24 (1H, t, ^3J = 7.6, H Ar), 7.47 (1H, d, ^3J = 7.6, H Ar). ^{13}C NMR spectrum, *δ*, ppm: 28.3, 28.6, 29.5, 30.1, 54.9, 85.7, 88.4, 127.8, 129.2, 131.2, 132.1, 133.3, 133.5, 149.4, 151.2, 158.4, 162.8, 163.0, 165.2. IR spectrum, ν, cm^{-1}: 2956, 1698, 1672, 1519, 1431, 1387, 1188, 1042, 748. Mass spectrum, *m/z* (I_{rel}, %): 470 $[M(^{37}Cl, ^{37}Cl)]^+$ (2), 468 $[M(^{37}Cl, ^{35}Cl)]^+$ (5), 466 $[M(^{35}Cl, ^{35}Cl)]^+$ (8), 435 (^{37}Cl, ^{37}Cl) (11), 433 (^{37}Cl, ^{35}Cl) (31), 431 (^{35}Cl, ^{35}Cl) (100), 374 (15), 321 (^{37}Cl, ^{37}Cl) (3), 319 (^{37}Cl, ^{35}Cl) (41), 317 (^{35}Cl, ^{35}Cl) (99), 260 (14), 210 (19), 58 (16). Found, %: C, 48.68; H, 3.43; Cl, 15.09; N, 11.85. $C_{19}H_{16}Cl_2N_4O_6$. Calculated, %: C, 48.84; H, 3.45; Cl, 15.17; N, 11.99.

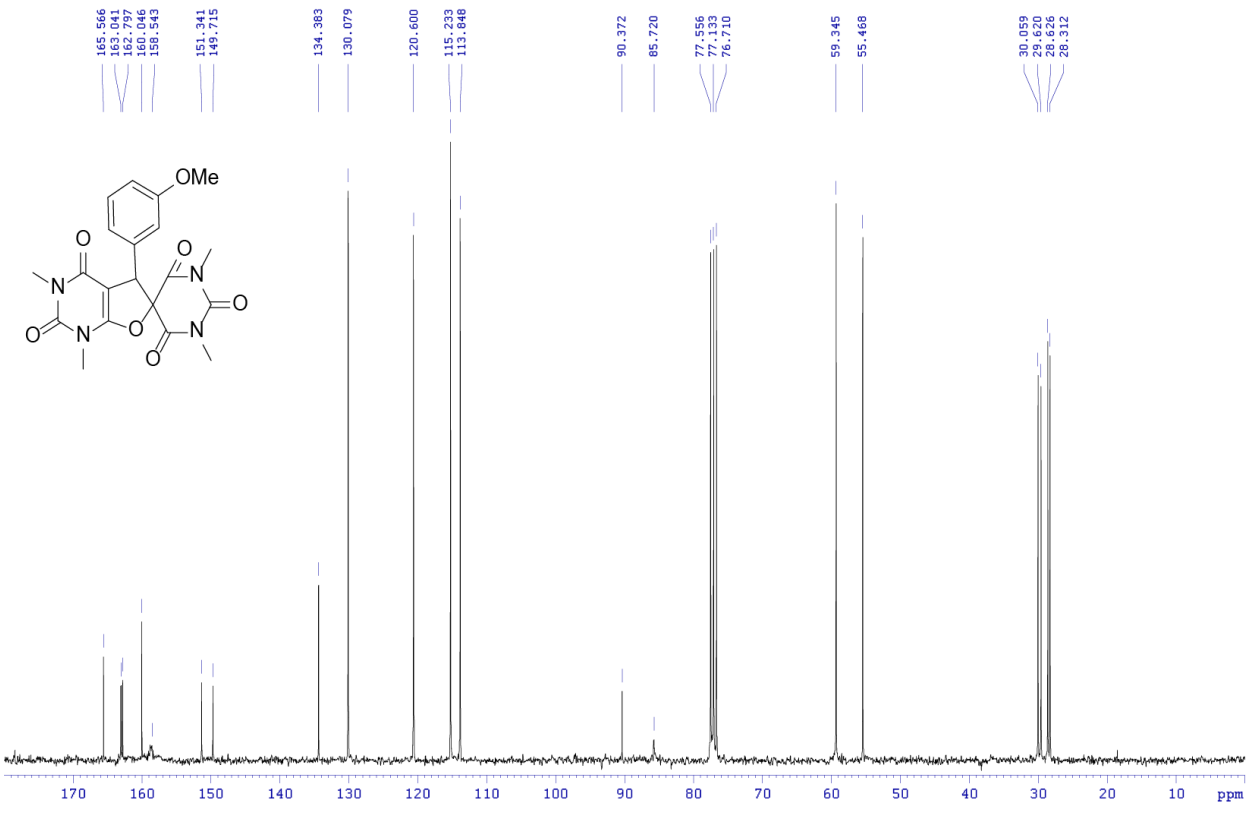
5-(2,4-Dichlorophenyl)-1,1',3,3'-tetramethyl-1,5-dihydro-2*H*,2'*H*-spiro[furo[2,3-*d*]pyrimidine-6,5'-pyrimidine]-2,2',4,4',6'(1'*H*,3*H*,3'*H*)-pentaone (3l).

Yield 0.43 g (92%), white powder, mp: 269–270 (lit.[S4] mp 268–270°C). Spectral data correspond to literary.

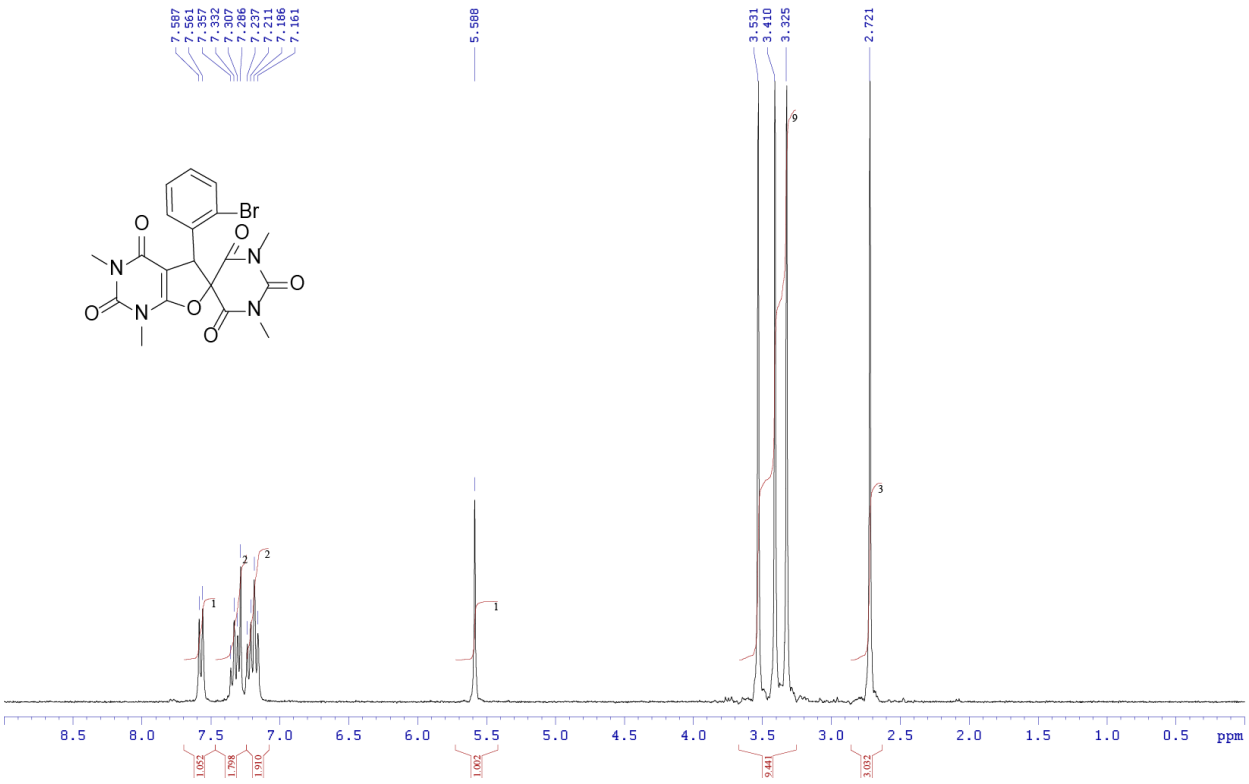
^{1}H and ^{13}C NMR spectra for compounds

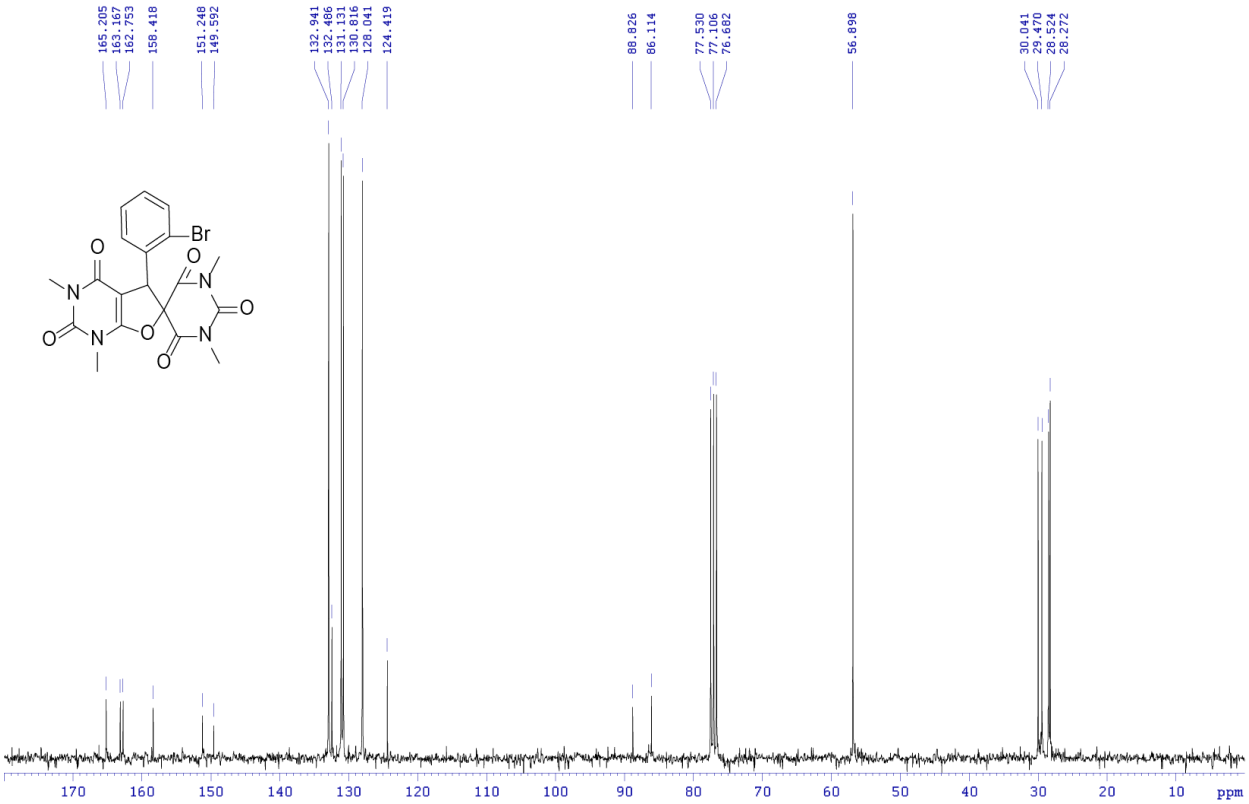
5-(3-Methoxyphenyl)-1,1',3,3'-tetramethyl-1,5-dihydro-2*H*,2'*H*-spiro[furo[2,3-*d*]pyrimidine-6,5'-pyrimidine]-2,2',4,4',6'(1'*H*,3*H*,3'*H*)-pentone (3c).



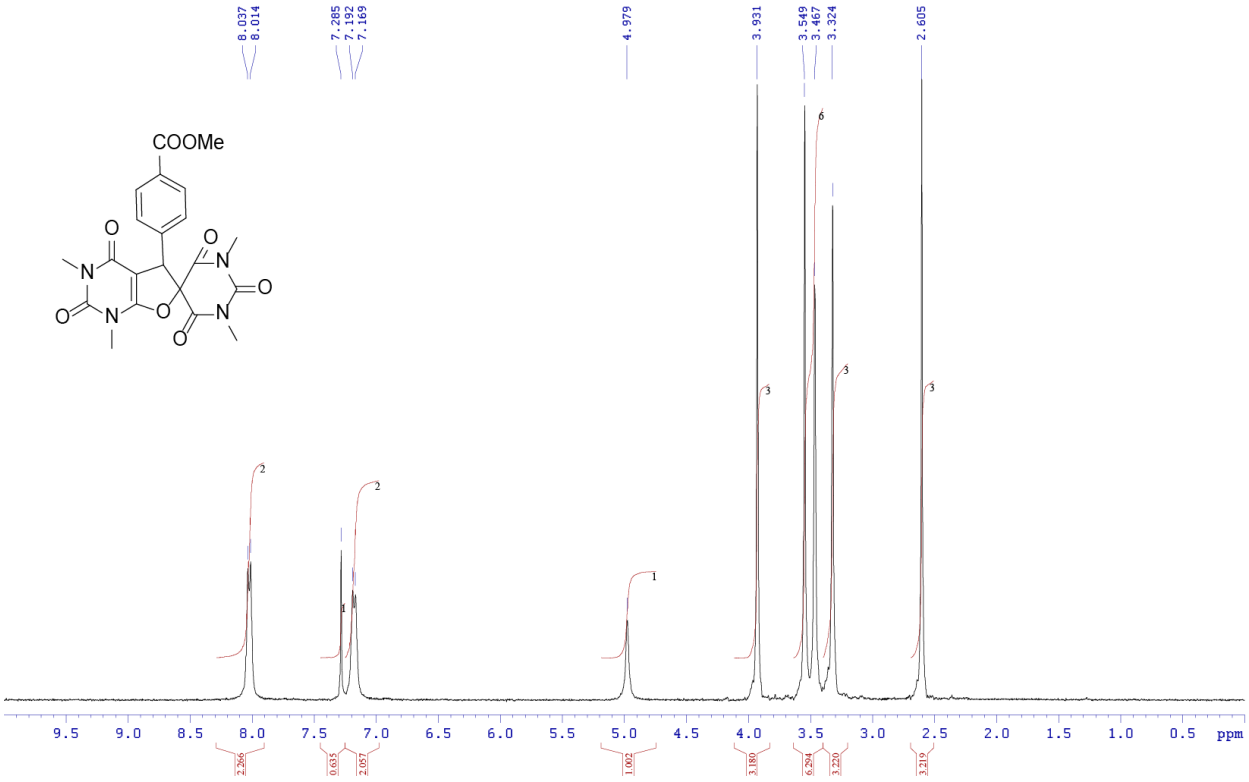


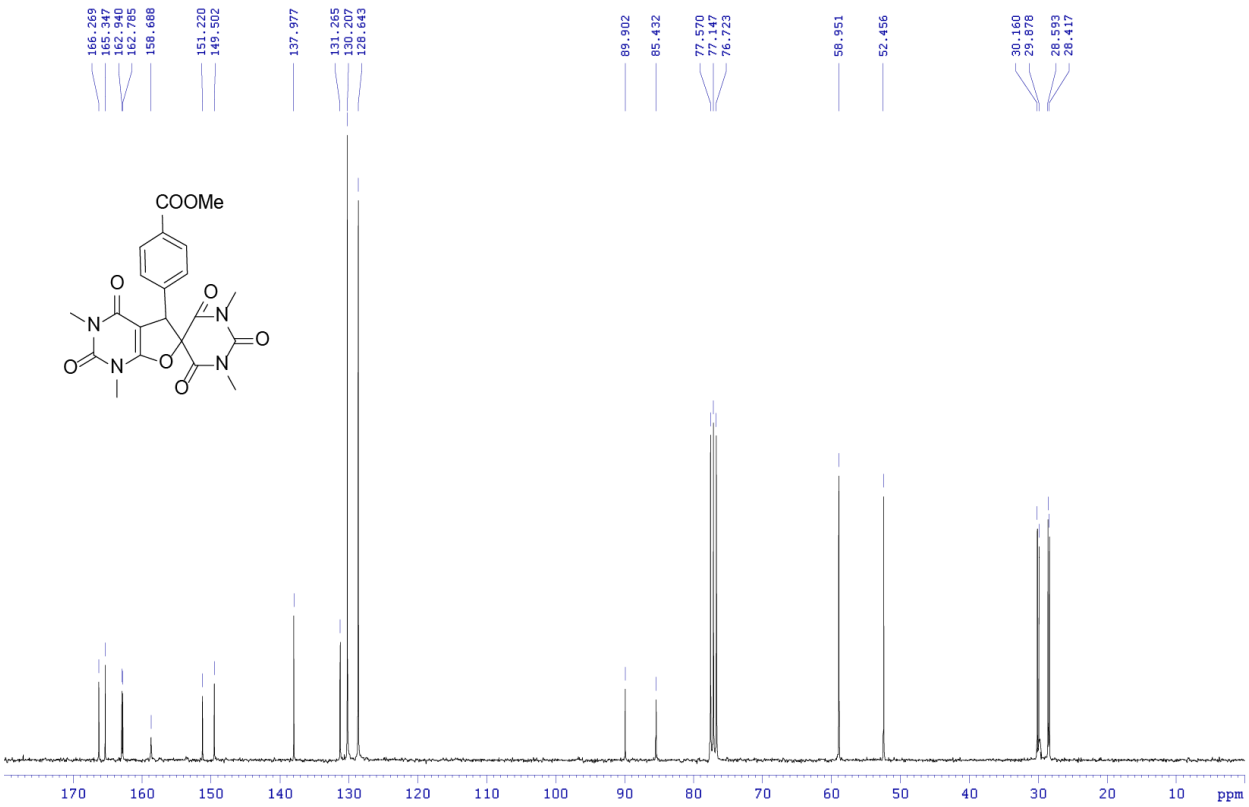
5-(2-Bromophenyl)-1,1',3,3'-tetramethyl-1,5-dihydro-2*H*,2'*H*-spiro[furo[2,3-*d*]pyrimidine-6,5'-pyrimidine]-2,2',4,4',6'(1'*H*,3*H*,3'*H*)-pentone (3d).



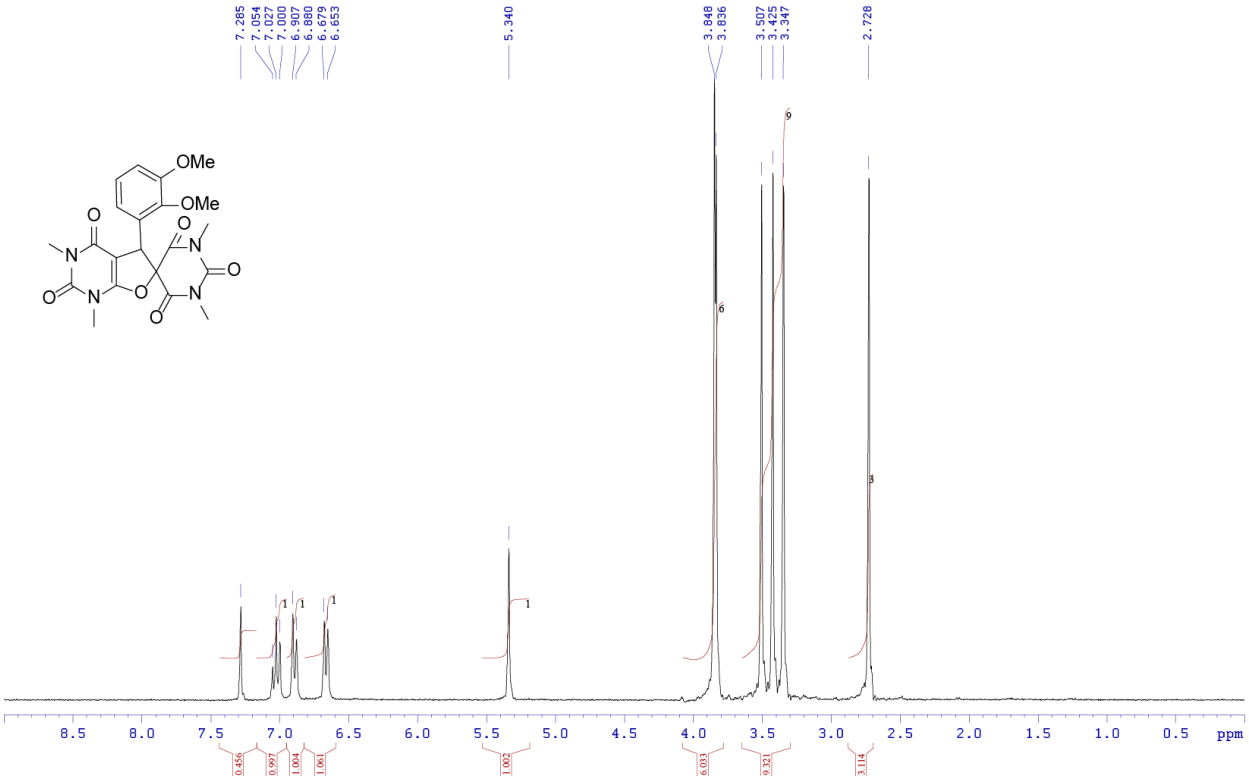


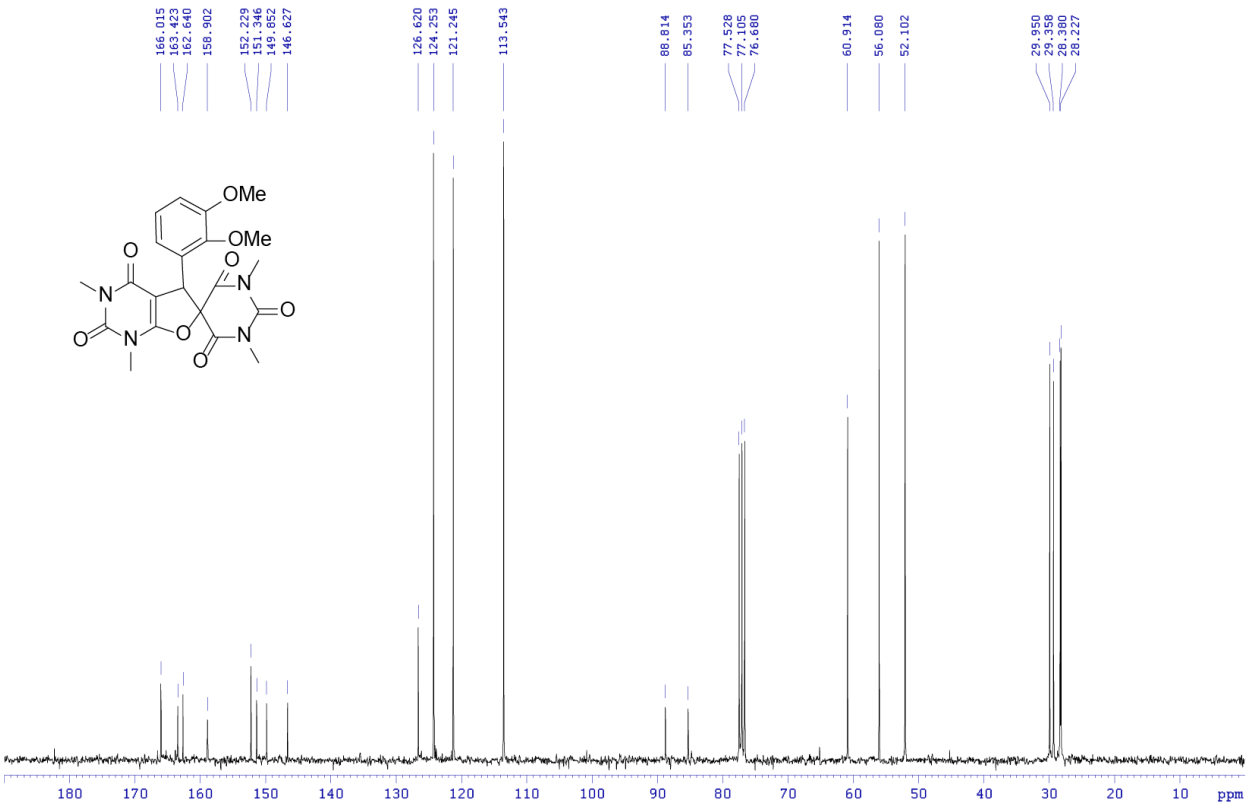
Methyl 4-(1,1',3,3'-tetramethyl-2,2',4,4',6'-pentaoxo-1,1',3,3',4,4',5,6'-octahydro-2*H*,2'*H*-spiro[furo[2,3-*d*]pyrimidine-6,5'-pyrimidin]-5-yl)benzoate (3f).



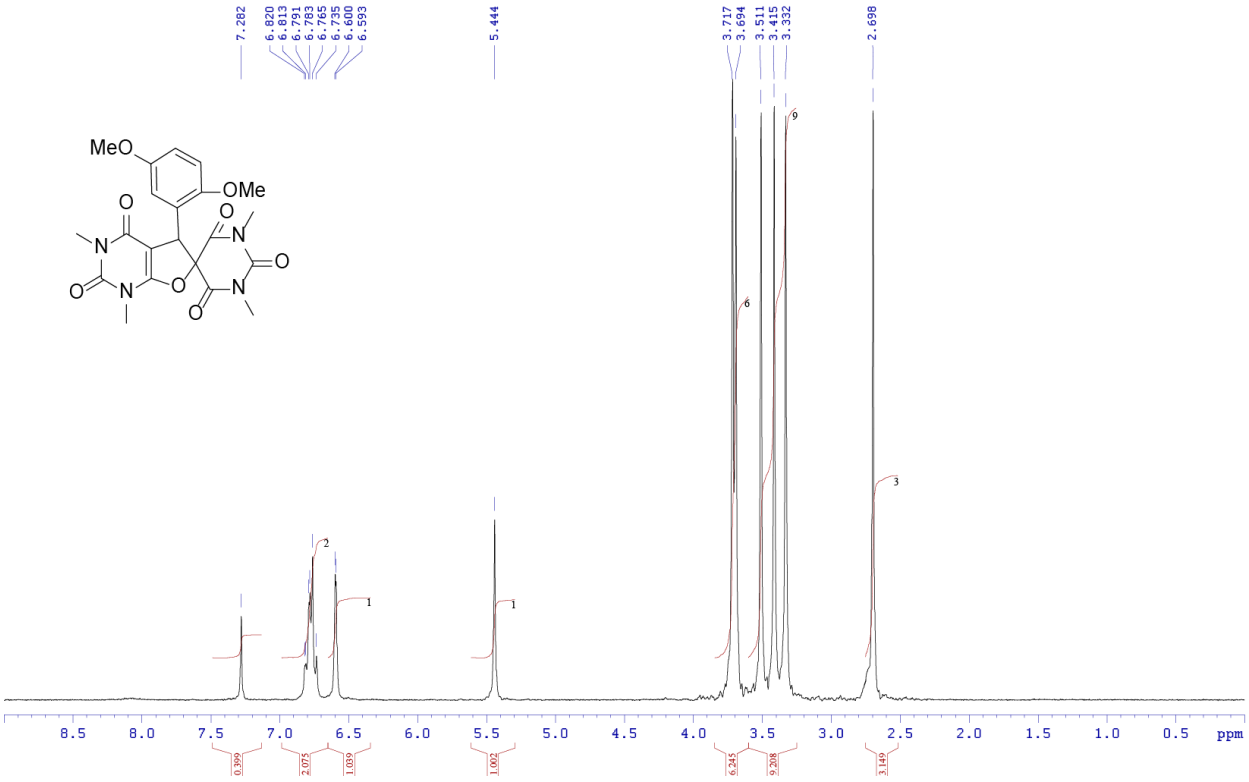


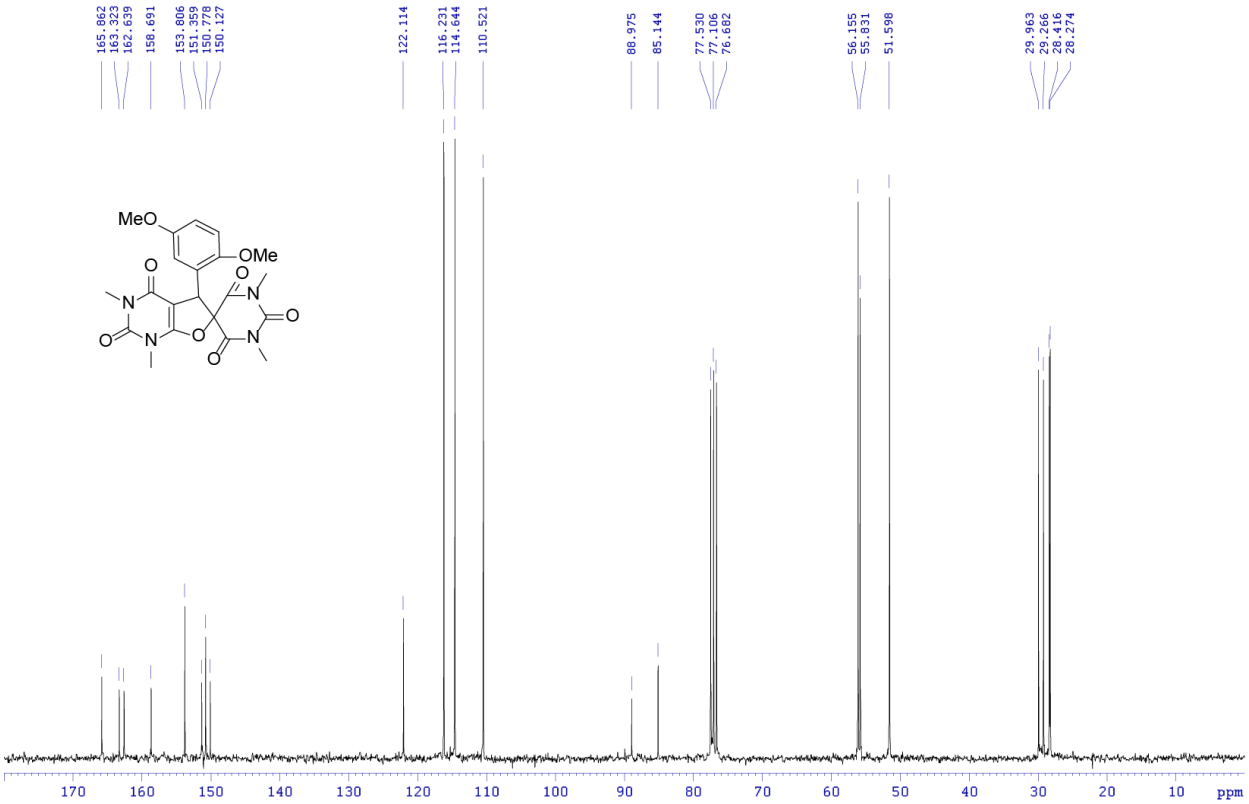
5-(2,3-Dimethoxyphenyl)-1,1',3,3'-tetramethyl-1,5-dihydro-2*H*,2'*H*-spiro[furo[2,3-*d*]pyrimidine-6,5'-pyrimidine]-2,2',4,4',6'(1'*H*,3*H*,3'*H*)-pentone (3g).



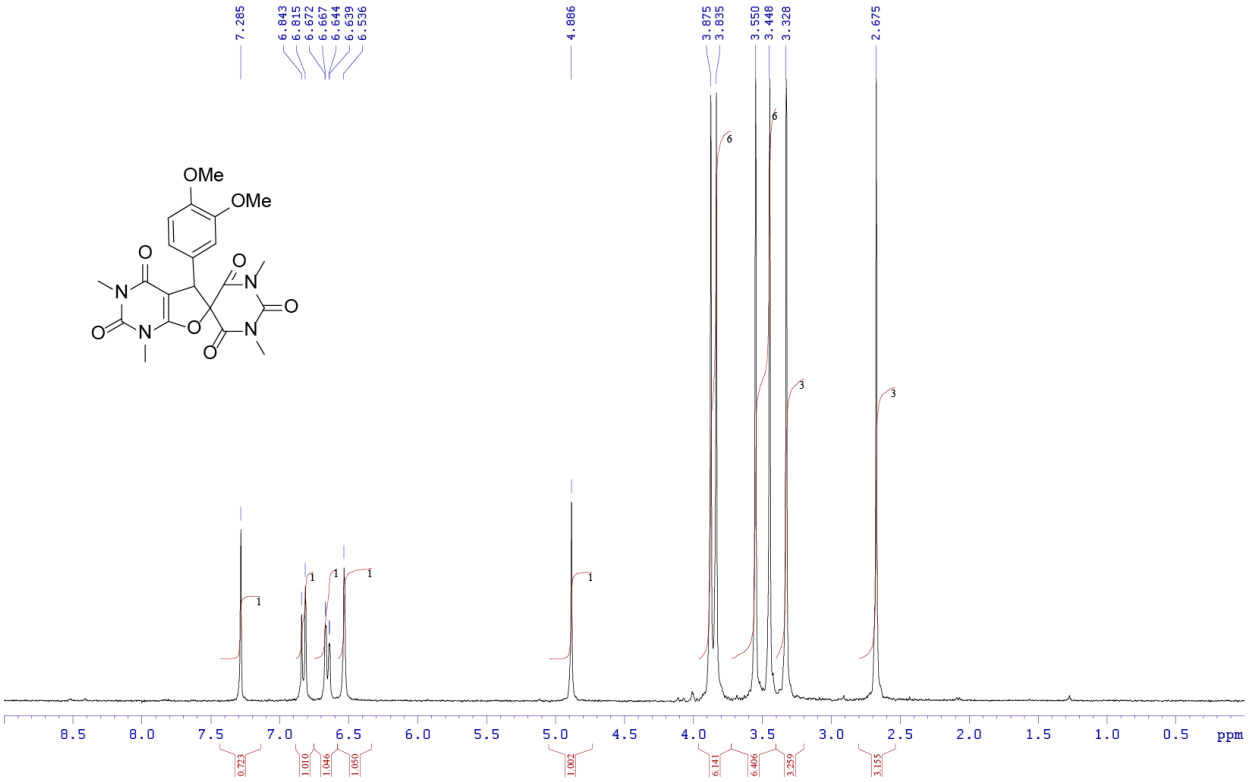


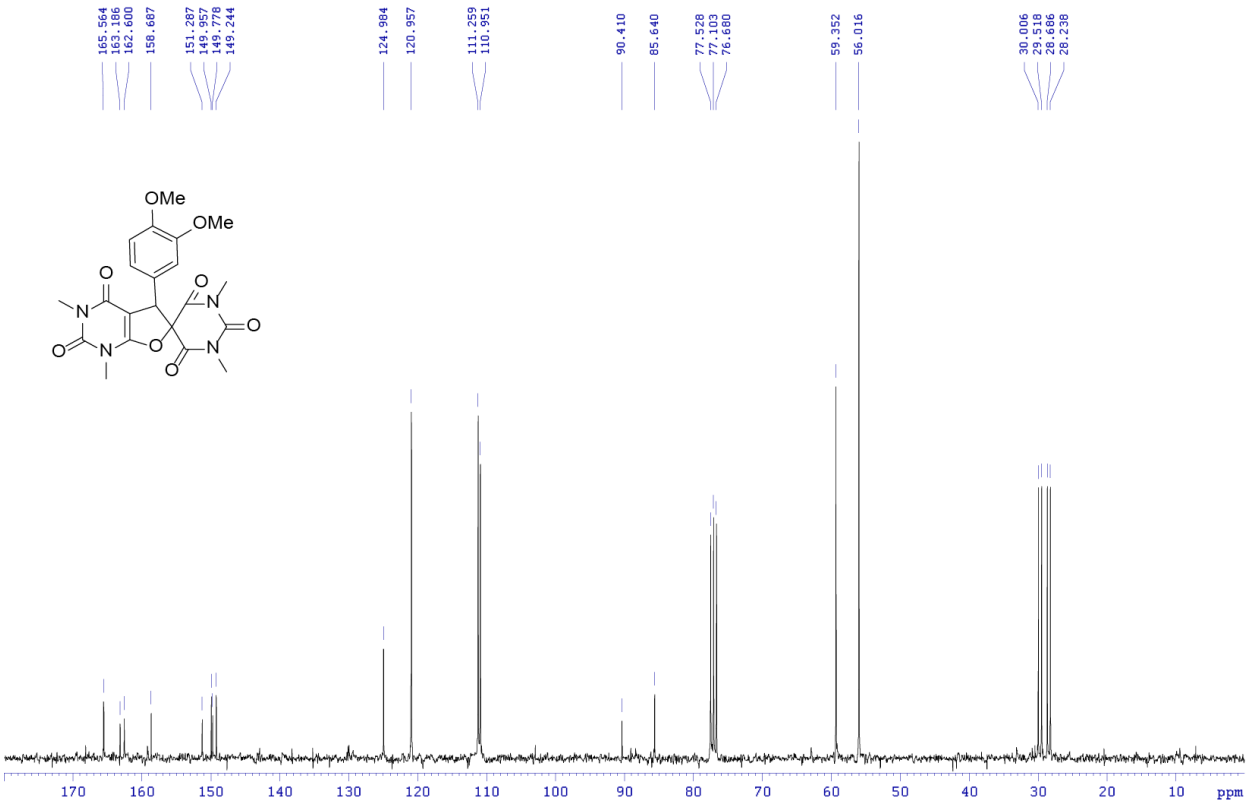
5-(2,5-Dimethoxyphenyl)-1,1',3,3'-tetramethyl-1,5-dihydro-2*H*,2'*H*-spiro[furo[2,3-*d*]pyrimidine-6,5'-pyrimidine]-2,2',4,4',6'(1'*H*,3*H*,3'*H*)-pentone (3h).



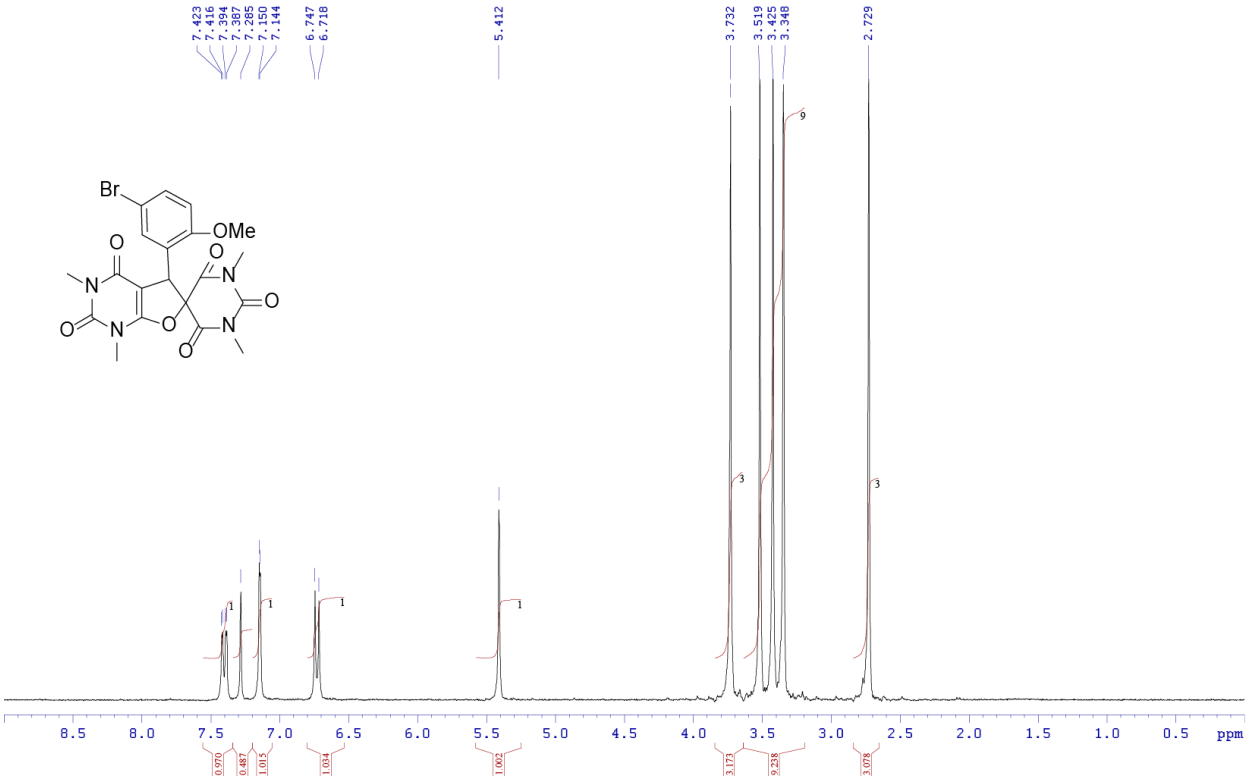


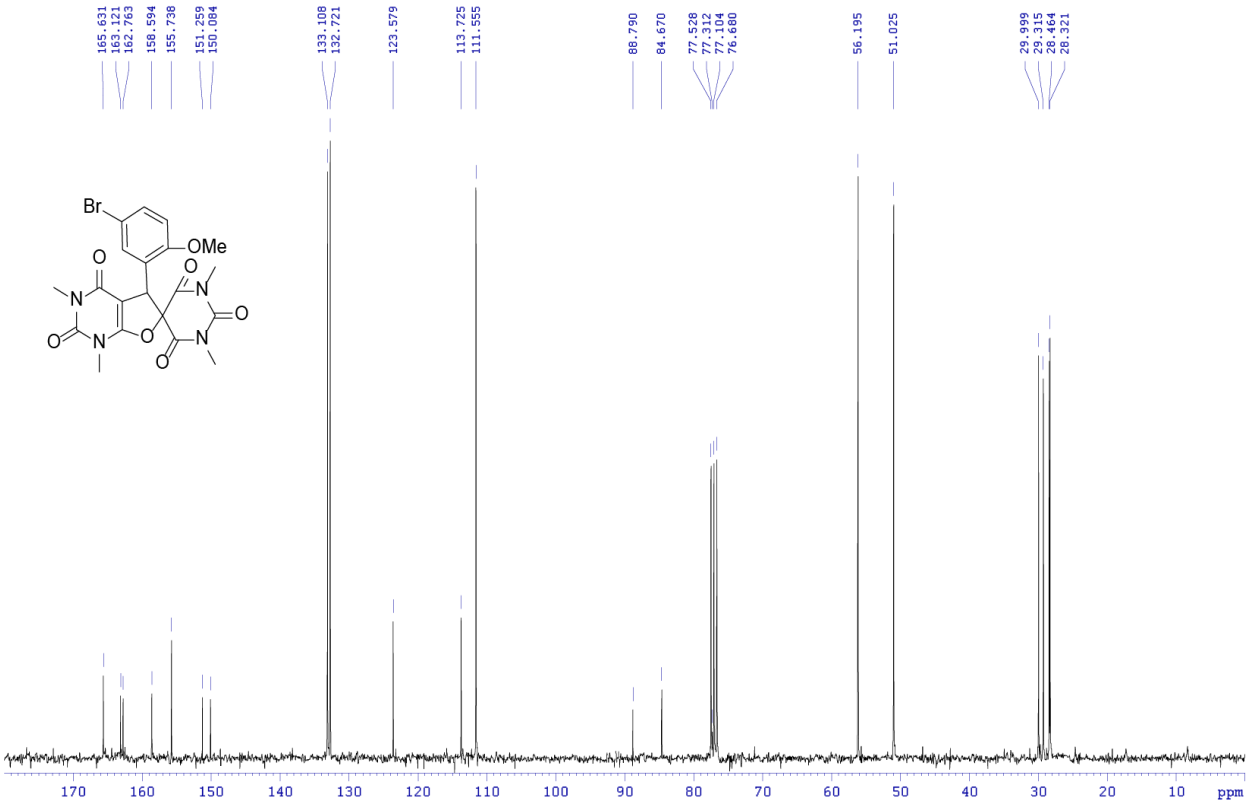
5-(3,4-Dimethoxyphenyl)-1,1',3,3'-tetramethyl-1,5-dihydro-2*H*,2'*H*-spiro[furo[2,3-*d*]pyrimidine-6,5'-pyrimidine]-2,2',4,4',6'(1'*H*,3*H*,3'*H*)-pentone (3i).



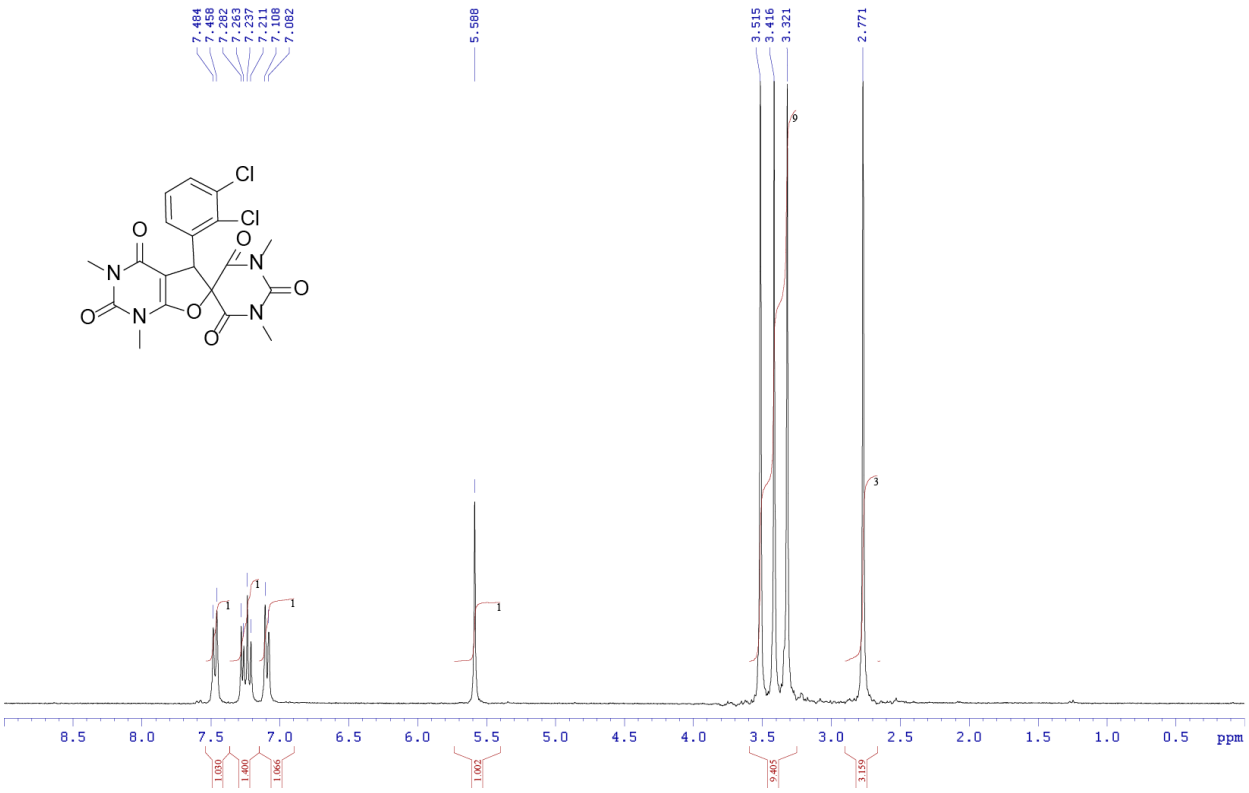


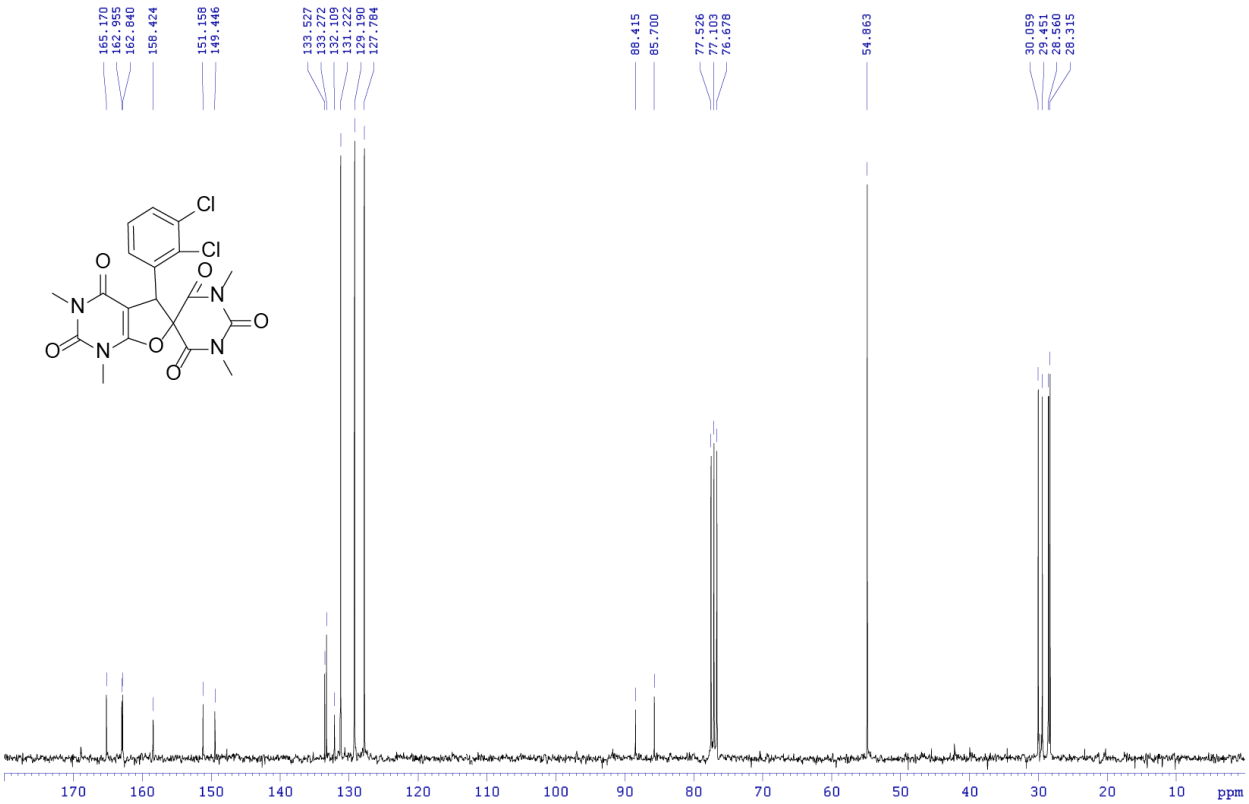
5-(5-Bromo-2-methoxyphenyl)-1,1',3,3'-tetramethyl-1,5-dihydro-2*H*,2'*H*-spiro[furo[2,3-*d*]pyrimidine-6,5'-pyrimidine]-2,2',4,4',6'(1'*H*,3*H*,3'*H*)-pentone (3j).





5-(2,3-Dichlorophenyl)-1,1',3,3'-tetramethyl-1,5-dihydro-2*H*,2'*H*-spiro[furo[2,3-*d*]pyrimidine-6,5'-pyrimidine]-2,2',4,4',6'(1'*H*,3*H*,3'*H*)-pentone (3k).





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