Electronic Supplementary Material (ESI) for New Journal of Chemistry

Supporting Information (SI)

General: All the chemicals were procured from either Sigma Aldrich Chemicals Pvt. Ltd. or Spectrochem, India. Silica gel [(60-120 mesh) was used for chromatographic separation. Silica gel G [E-Merck (India)] was used for TLC. Petroleum ether refers to the fraction boiling between 60°C and 80°C. IR spectra were recorded on a Perkin-Elmer L 120-000A spectrometer (ν_{max} in cm^{-1}) on KBr disks. ^{1}H NMR and ^{13}C spectra were recorded on a Bruker DPX-300 and Bruker DPX-400 Bruker DPX-500 spectrometer in $CDCl_3$ (chemical shift in δ) with TMS as internal standard. MS were recorded on a Q-TOF microTm instrument at the Indian Institute of Chemical Biology. CHN was recorded on 2400 series II CHN analyzer Perkin Elmer. Melting points were determined in open capillaries and are uncorrected. HRMS were recorded on a Q-tof Micro YA263 instrument.

General procedure for the preparation of compound 3a-h and 1c:

A mixture of the compounds **1a** (500 mg, 2.74 mmol), *p*-chloroiodobenzene (786 mg, 3.29 mmol), $Pd(PPh_3)_2Cl_2$ (0.13 mmol) and CuI (0.27 mmol) in dry DMF (5 mL) and dry Et_3N (1.5 mL) was stirred at room temperature for 1h. After completion of the reaction (as monitored by TLC), the reaction mixture was poured in water. This was extracted with dichloromethane (3 x 15 mL). The combined organic extract was washed with brine (1 x 15) and dried over Na_2SO_4. The solvent was distilled off. The resulting crude product was purified by column chromatography over silica gel (60-120 mesh) using petroleum ether-ethyl acetate mixture (4:1) as an eluent to give the product **3a**. Similarly precursors **3b-h** were prepared from the corresponding iodobenzene derivatives. The compound **1c** was prepared by refluxing 1,3-diethyl-5-hydroxypyrimidine-2,4(1*H*,3*H*)-dione (1gm, 5.4 mmol) with 1-bromobut-2-yne (0.85 gm, 6.4 mmol) and anhydrous K_2CO_3 (1.5 gm, 10.8 mmol) in acetone (75 mL) for 5h and purified by column chromatography using petroleum ether-ethyl acetate mixture (3:2) as an eluent.

Compound 3a:

Yield: 85%, solid; m.p. 158-160 ^{o}C; IR (KBr): ν_{max} = 1642, 1657, 1716, 2232 cm^{-1};

^{1}H-NMR ($CDCl_3$, 500 MHz): δ_H = 3.38 (s, 6H, -NC**H**$_3$), 4.91 (s, 2H, -C**H**$_2$), 7.09 (s, 1H, Ar**H**), 7.29-7.30 (m, 2H, Ar**H**), 7.33-7.35 (m, 2H, Ar**H**).

MS: m/z = 305 [M^{+}+H].

Anal. Calcd. For: $C_{15}H_{13}ClN_2O_3$: C, 59.12; H, 4.30; N, 9.19; Found: C, 59.34; H, 4.26; N, 9.34.

Compound 3b:

Yield: 89%, solid; m.p. 118-120 ^{o}C; IR (KBr): ν_{max} = 1635, 1644, 1704, 2215 cm^{-1};

^{1}H-NMR ($CDCl_3$, 400 MHz): δ_H = 3.38 (s, 6H, -NC**H**$_3$), 4.91 (s, 2H, -C**H**$_2$), 7.12 (s, 1H, Ar**H**), 7.27-7.35 (m, 3H, Ar**H**), 7.40-7.42 (m, 2H, Ar**H**).

^{13}C-NMR ($CDCl_3$, 100 MHz): δ_C = 28.3, 37.0, 59.9, 83.3, 88.5, 122.0, 128.4, 128.9, 131.0, 131.7, 132.2, 150.7, 160.2.

MS: m/z = 271 [M^{+}+H].

Anal. Calcd. For: $C_{15}H_{14}N_2O_3$: C, 66.66; H, 5.22; N, 10.36; Found: C, 66.82; H, 5.24; N, 10.61.

Compound 3c:

Yield: 86%, solid; m.p. 124-126 ^{o}C; IR (KBr): ν_{max} = 1648, 1676, 1708, 2210 cm^{-1};

^{1}H-NMR ($CDCl_3$, 400 MHz): δ_H = 1.22-1.42 (m, 6H, -NCH$_2$C**H**$_3$), 3.76-3.81 (m, 2H, -NC**H**$_2$CH$_3$), 4.01-4.12 (m, 2H, -NC**H**$_2$CH$_3$), 4.91 (s, 2H, -C**H**$_2$), 7.11 (s, 1H, Ar**H**), 7.29-7.33 (m, 3H, Ar**H**), 7.34-7.40 (m, 2H, Ar**H**).

MS: m/z = 299 [M^{+}+H]

Anal. Calcd. For $C_{17}H_{18}N_2O_3$: C, 68.44; H, 6.08; N, 9.39; Found: C, 68.56; H, 6.04; N, 9.48.

Compound 3d:

Yield: 86%, solid; m.p. 122-124 ^{o}C; IR (KBr): ν_{max} = 1651, 1667, 1708, 2224 cm^{-1}.

^{1}H-NMR ($CDCl_3$, 500 MHz): δ_H = 1.22-1.29 (m, 6H, -$NCH_2C\mathbf{H}_3$), 3.76 (q, 2H, *J* = 7.2 Hz, -$NC\mathbf{H}_2CH_3$), 4.01 (q, 2H, *J* = 7.1 Hz, -$NC\mathbf{H}_2CH_3$), 4.90 (s, 2H, -CH_2), 7.08 (s, 1H, Ar**H**), 7.28-7.30 (m, 2H, Ar**H**), 7.32-7.34 (m, 2H, Ar**H**).

MS: m/z = 333 [M^++H].

Anal. Calcd. For: $C_{17}H_{17}ClN_2O_3$: C, 61.36; H, 5.15; N, 8.42; Found: C, 61.62; H, 5.18; N, 8.63.

Compound 3e:

Yield: 88%, solid; m.p. 143-145 ^{o}C; IR (KBr): ν_{max} = 1644, 1655, 1716, 2229 cm^{-1};

^{1}H-NMR ($CDCl_3$, 500 MHz): δ_H = 2.35 (s, 3H, -$C\mathbf{H}_3$), 3.38 (s, 6H, -$NC\mathbf{H}_3$), 4.90 (s, 2H, -CH_2), 7.10 (s, 1H, Ar**H**), 7.11 (d, 2H, *J* = 6.4 Hz, Ar**H**), 7.29 (d, 2H, *J* = 6.4 Hz, Ar**H**),

MS: m/z = 285 [M^++H].

Anal. Calcd. For: $C_{16}H_{16}N_2O_3$: C, 67.59; H, 5.67; N, 9.85; Found; C, 67.74; H, 5.71; N, 9.72.

Compound 3f:

Yield: 88%, solid; m.p. 92-94 ^{o}C; IR (KBr): ν_{max} = 1644, 1672, 1703, 2215 cm^{-1};

^{1}H-NMR ($CDCl_3$, 500 MHz): δ_H = 1.21-1.27 (m, 6H, -$NCH_2C\mathbf{H}_3$), 2.35 (s, 3H, -$C\mathbf{H}_3$), 3.77 (q, 2H, *J* = 7.2 Hz, -$NC\mathbf{H}_2CH_3$,), 4.05 (q, 2H, *J* = 7.1 Hz, -$NC\mathbf{H}_2CH_3$), 4.90 (s, 2H, -$C\mathbf{H}_2$), 7.06 (s, 1H, Ar**H**), 7.28-7.34 (m, 2H, Ar**H**), 7.31-7.33 (m, 2H, Ar**H**),

MS: m/z = 313 [M^++H].

Anal. Calcd. For $C_{18}H_{20}N_2O_3$: C, 69.21; H, 6.45; N, 8.97; Found: C, 69.01; H, 6.50; N, 9.11.

Compound 3g:

Yield: 80%, solid; m.p. 158-160 ^{o}C; IR (KBr): ν_{max} = 1714, 1672, 1652 cm^{-1};

^{1}H NMR ($CDCl_3$, 400 MHz): δ_H (ppm) = 2.61 (s, 3H, -$COC\mathbf{H}_3$), 3.39 (s, 3H, -$NC\mathbf{H}_3$), 3.40 (s, 3H, -$NC\mathbf{H}_3$), 4.95 (s, 2H, -$C\mathbf{H}_2$), 7.13 (s, 1H, Ar**H**), 7.50 (d, 2H, *J* = 8.0 Hz, Ar**H**), 7.91 (d, 2H, *J* = 8.4 Hz, Ar**H**).

MS: m/z = 341 [M^++H].

Anal. Calcd. For $C_{17}H_{16}N_2O_4$: C, 65.38; H, 5.16; N, 8.97; Found: C, 67.55; H, 5.20; N, 8.62.

Compound 3h:

Yield: 82%, solid; m.p. 116-118 ^oC; IR (KBr): ν_{max} = 1648, 1676, 2212 cm^{-1}; ^{1}H-NMR ($CDCl_3$, 400 MHz): δ_H = 1.22 (m, 6H, -$NCH_2C\mathbf{H}_3$), 2.60 (s, 3H, -$COC\mathbf{H}_3$), 3.77 (q, 2H, *J* = 7.2 Hz, -$NC\mathbf{H}_2CH_3$), 4.01 (q, 2H, *J* = 6.8 Hz, -$NC\mathbf{H}_2CH_3$), 4.94 (s, 2H, -$C\mathbf{H}_2$), 7.11 (s, 1H, Ar**H**), 7.48 (d, 2H, *J* = 8.0 Hz, Ar**H**), 7.90 (d, 2H, *J* = 8.0 Hz, Ar**H**),

MS: m/z = 341 $[M^+ + H]$.

Anal. Calcd. For $C_{19}H_{20}N_2O_4$: C, 67.05, H, 5.92, N, 8.23; Found: C, 67.31; H, 5.94; N, 8.39.

Compound 1c:

Yield: 85%, solid, m.p. 94 ^oC; IR (KBr): ν_{max} = 1646, 1698, 2242 cm^{-1};

^{1}H-NMR ($CDCl_3$, 400 MHz): δ_H = 1.23 (t, 3H, -$NCH_2C\mathbf{H}_3$ *J* = 7.2 Hz), 1.32 (t, 3H, -$NCH_2C\mathbf{H}_3$ *J* = 7.2 Hz), 1.86 (t, 3H, -$C\mathbf{H}_3$, *J* = 2.4 Hz), 3.81 (q, 2H, -$NC\mathbf{H}_2CH_3$ *J* = 7.2 Hz), 4.03 (q, 2H, -$NC\mathbf{H}_2CH_3$ *J* = 7.2 Hz), 4.64 (t, 2H, -$C\mathbf{H}_2$, *J* = 2.4 Hz), 7.01 (s, 1H, Ar**H**).

MS: m/z = 237 $[M^+ + H]$.

Anal. Calcd. For $C_{12}H_{16}N_2O_3$: C, 61.00; H, 6.83; N, 11.86; Found: C, 61.17; H, 6.80; N, 11.97.

General procedure for the preparation of compounds 4a-j:

To a stirred solution of $AgSbF_6$ (9 mg, 0.026 mmol) in HOAc (5 mL), 5-(3-(4-chlorophenyl) prop-2-ynyloxy)-1,3-dimethylpyrimidine-2,4(1*H*, 3*H*)-dione **3a**, (100 mg, 0.26 mmol) was added at room temperature and stirred at 80 ^oC for 4h. After completion of the reaction (as monitored by TLC), the reaction mixture was cooled and neutralized with saturated $NaHCO_3$ solution. This was extracted with dichloromethane (3 x 10 mL). The combined organic extract was washed with brine (1 x10) and dried over Na_2SO_4. The solvent was distilled off. The resulting crude product was purified by column chromatography over silica gel (60-120 mesh) using petroleum ether-ethyl acetate mixture (2:3) as eluent to give the product **4a**.

Similarly compounds **4b-j** were obtained from the corresponding precursors.

4-(4-chlorophenyl)-10-hydroxy-7,9-dimethyl-1-oxa-7,9-diazaspiro[4.5]dec-3-ene-6,8-dione (4a) :

Yield 87%, yellow, solid, m.p. 170-172 ^{o}C; IR (KBr): ν_{max} = 3345, 1709, 1673 cm^{-1}.

^{1}H NMR (CDCl$_3$, 400 MHz): δ_H (ppm) = 2.81 (s, 3H, -NC**H**$_3$), 3.20 (s, 4H, -NC**H**$_3$ & -O**H**) **[**But in D$_2$O exchange NMR, peak for –O**H** is vanished then it appears as 3.20(s, 3H, -NC**H**$_3$)], 4.76 (d, 1H, *J* = 3.6 Hz; -C**H**OH), 4.90 (dd, 1H, *J* = 1.6 Hz, 14 Hz, C**H**$_a$H$_b$), 5.11 (dd, 1H, *J* = 1.6 Hz, 13.6 Hz, CH$_a$**H**$_b$), 6.19 (d, 1H, *J* = 1.6 Hz, =C**H**), 6.95 (dd, 2H, *J* = 1.2 Hz, 8.4 Hz, Ar**H**), 7.36 (dd, 2H, *J* = 1.6 Hz, 8.4 Hz, Ar**H**),

^{13}C NMR (75 MHz) δ_C (ppm) = 27.9, 33.8, 76.1, 82.5, 89.8, 128.4, 128.7, 130.0, 130.7, 134.6, 137.9, 151.5, 168.6

DEPT (in 135 mode): 27.9, 33.8, 76.1(-ve), 82.5, 128.4, 128.7, 130.0

HRMS [M^{+}+Na] calculated: 345.0618, observed: 345.0623

10-hydroxy-7,9-dimethyl-4-phenyl-1-oxa-7,9-diazaspiro[4.5]dec-3-ene-6,8-dione(4b):

Yield 78%, solid, m.p. 146-148 ^{o}C IR (KBr): ν_{max} = 3427, 1716, 1674 cm^{-1}.

^{1}H NMR (CDCl$_3$, 400 MHz): δ_H (ppm) = 2.75 (s, 3H, -NC**H**$_3$), 3.19 (s, 3H, -NC**H**$_3$), 3.44 (s, 1H, -O**H**), 4.77 (s, 1H, -C**H**OH), 4.90 (d, 1H, *J* = 13.6 Hz, C**H**$_a$H$_b$), 5.12 (d, 1H, *J* = 13.6 Hz, CH$_a$**H**$_b$), 6.15 (s, 1H, =C**H**), 6.99 (m, 2H, Ar**H**), 7.31 (d, 3H, *J* = 2.4 Hz, Ar**H**),

^{13}C NMR (100MHz): δ_C (ppm) = 27.9, 33.9, 76.3, 82.9, 90.0, 127.2, 128.5, 128,7, 129.0, 132.3, 139.2, 151.5, 168.8

HRMS [M^{+}+Na] calculated: 311.1008, observed: 311.3597

7,9-diethyl-10-hydroxy-4-phenyl-1-oxa-7,9-diazaspiro[4.5]dec-3-ene-6,8-dione (4c):

Yield 75%, solid, m.p. 126-128 ^{o}C IR (KBr): ν_{max} = 3391, 1712, 1674: cm^{-1}

^{1}H NMR (CDCl$_3$, 400 MHz): δ_H (ppm) = 0.84 (t, 3H, *J* = 7.2 Hz, -NCH$_2$C**H**$_3$), 1.20 (t, 3H, *J* = 7.2 Hz, -NCH$_2$C**H**$_3$), 3.16 (q, 2H, *J* = 7.2 Hz, -NC**H**$_2$CH$_3$), 3.29 (s, 1H, -O**H**), 3.81-3.94

(m, 2H, -NC**H**$_2$CH$_3$), 4.78 (s, 1H, -C**H**OH), 4.89 (d, 1H, *J* = 13.6 Hz, -C**H**$_a$H$_b$), 5.13 (d, 1H, *J* = 13.2 Hz, -CH$_a$**H**$_b$), 6.15 (s, 1H, =C**H**), 7.04-7.05 (m, 2H, Ar**H**), 7.27-7.30 (m, 3H, Ar**H**) ;

^{13}C NMR (100 MHz): δ_C (ppm) = 13.0, 13.2, 36.7, 42.6, 76.0, 82.1, 89.4, 127.4, 128.3, 128.4, 128.6, 129.3, 132.4, 139.5, 151.0, 168.5.

HRMS [M$^+$+Na] calculated: 339.1321, observed: 339.1324

4-(4-chlorophenyl)-7,9-diethyl-10-hydroxy-1-oxa-7,9-diazaspiro[4.5]dec-3-ene-6,8-dione(4d):

Yield 84%, solid, m.p. 164-166 ^{o}C IR (KBr): ν_{max} = 3493, 1709, 1675 cm^{-1}

^{1}H NMR (CDCl$_3$, 400 MHz): δ_H (ppm) = 0.89 (t, 3H, *J* = 7.2 Hz, -NCH$_2$C**H**$_3$), 1.19 (t, 3H, *J* = 7.2 Hz, -NCH$_2$C**H**$_3$), 3.21 (q, 2H, *J* = 6.8 Hz, -NC**H**$_2$CH$_3$), 3.41 (s, 1H, -O**H**), 3.80-3.95 (m, 2H, -NC**H**$_2$CH$_3$), 4.78 (s, 1H, -C**H**OH), 4.87 (d, 1H, *J* = 13.6 Hz, C**H**$_a$H$_b$), 5.11 (d, 1H, *J* = 13.6 Hz, -CH$_a$**H**$_b$), 6.17 (s, 1H, =C**H**), 6.99 (d, 2H, *J* = 8.4 Hz, Ar**H**), 7.27 (d, 2H, *J* = 8.4 Hz, Ar**H**) ;

^{13}C NMR (100 MHz): δ_C (ppm) = 13.1, 13.3, 36.8, 42.5, 75.9, 81.8, 89.4, 128.7, 130.2, 130.9, 134.7, 138.3, 151.0, 168.3.

HRMS [M$^+$+Na] calculated: 373.0931, observed: 373.0931

10-hydroxy-7,9-dimethyl-4-p-tolyl-1-oxa-7,9-diazaspiro[4.5]dec-3-ene-6,8-dione (4e):

Yield 80%, solid, m.p. 174-176 ^{o}C IR (KBr): ν_{max} = 3433, 1717, 1673 cm^{-1}

^{1}H NMR (CDCl$_3$, 400 MHz): δ_H (ppm) = 2.32 (s, 3H, -C**H**$_3$), 2.78 (s, 3H, -NC**H**$_3$), 3.21 (s, 3H, -NC**H**$_3$), 3.22 (s, 1H, -O**H**), 4.76 (d, 1H, *J* = 2.8 Hz; C**H**OH), 4.89 (d, 1H, *J* = 13.2 Hz, C**H**$_a$H$_b$), 5.11 (d, 1H, *J* = 13.2 Hz, CH$_a$**H**$_b$), 6.12 (s, 1H, =C**H**), 6.88 (d, 2H, *J* = 7.8 Hz, Ar**H**), 7.11 (d, 2H, *J* = 8.0 Hz, Ar**H**),

^{13}C NMR (100MHz): δ_C (ppm) = 21.2, 28.0, 34.0, 76.2, 82.9, 89, 8, 127.0, 128.7, 129.2, 138.5, 139.1, 151.6, 168.9.

HRMS [M$^+$+Na] calculated: 325.1165, observed: 325.1186

7,9-diethyl-10-hydroxy-4-p-tolyl-1-oxa-7,9-diazaspiro[4.5]dec-3-ene-6,8-dione (4f):

Yield 82%, solid, m.p. 122-124 ^{o}C IR (KBr): ν_{max} = 3497, 1719, 1675; cm^{-1}

^{1}H NMR ($CDCl_3$, 400 MHz): δ_H (ppm) = 0.86 (t, 3H, *J* = 6.4 Hz, -NCH$_2$C**H**$_3$), 1.21 (t, 3H, *J* = 6.4 Hz, -NCH$_2$C**H**$_3$), 2.31 (s, 3H, -C**H**$_3$), 3.17-3.21 (m, 3H, -NC**H**$_2$CH$_3$ & -O**H**), 3.83-3.92 (m, 2H, -NC**H**$_2$CH$_3$), 4.77 (s, 1H, -C**H**OH), 4.87 (d, 1H, *J* = 13.2 Hz, C**H**$_a$H$_b$), 5.12 (d, 1H, *J* = 13.2 Hz, -CH$_a$**H**$_b$), 6.11 (s, 1H, =C**H**), 6.93 (d, 2H, *J* = 6.8 Hz, Ar**H**), 7.09 (d, 2H, *J* = 6.8 Hz, Ar**H**)

^{13}C NMR (100 MHz) δ_C (ppm) = 13.1, 13.2, 21.1, 36.7, 42.6, 75.9, 82.0, 89.3, 127.2, 128.8, 129.1, 129.4, 138.5, 139.5, 151.1, 168.5

HRMS [M$^+$+Na] calculated: 353.1478, observed: 353.1503

4-(4-acetylphenyl)-10-hydroxy-7,9-dimethyl-1-oxa-7,9-diazaspiro[4.5]dec-3-ene-6,8-dione (4g):

Yield 69%, solid, m.p. 170-172 ^{o}C IR (KBr): ν_{max} = 3345, 1715, 1670, 1603 cm^{-1}

^{1}H NMR ($CDCl_3$, 400 MHz): δ_H (ppm) = 2.59 (s, 3H, -COC**H**$_3$), 2.78 (s, 3H, -NC**H**$_3$), 3.22 (s, 3H, -NC**H**$_3$), 3.27 (s, 1H, -O**H**), 4.79 (s, 1H, - C**H**OH), 4.93 (dd, 1H, *J* = 1.4 Hz, 14.0 Hz, -C**H**$_a$H$_b$), 5.14 (dd, 1H, *J* = 0.8 Hz, 14.0 Hz, CH$_a$**H**$_b$), 6.26 (s, 1H, =C**H**), 7.12 (d, 2H, *J* = 8.0 Hz, Ar**H**), 7.91 (d, 2H, *J* = 8.4 Hz, Ar**H**),

^{13}C NMR (100MHz) δ_C (ppm) = 26.6, 28.1, 33.8, 76.4, 82.6, 90.0, 127.5, 128.5, 130.6, 136.8, 137.1, 138.2, 151.6, 168.5, 197.3

HRMS [M$^+$+Na] calculated: 353.1114, observed: 353.1136

4-(4-acetylphenyl)-7,9-diethyl-10-hydroxy-1-oxa-7,9-diazaspiro[4.5]dec-3-ene-6,8-dione (4h):

Yield 62%, solid, m.p. 144-146 ^{o}C IR (KBr) ν_{max} = 3308, 1704, 1680, 1668 cm^{-1}

^{1}H NMR ($CDCl_3$, 400 MHz): δ_H (ppm) = 0.84 (t, 3H, -NCH$_2$C**H**$_3$, *J* = 7.2 Hz), 1.23 (t, 3H, *J* = 7.2 Hz, -NCH$_2$C**H**$_3$), 2.59 (s, 3H, -COC**H**$_3$), 3.18 (q, 2H, *J* = 7.2 Hz, -NC**H**$_2$CH$_3$), 3.32 (s,

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1H, -O**H**), 3.83-3.95 (m, 2H, -NC**H**$_2$CH$_3$), 4.80 (s, 1H, -C**H**OH), 4.92 (d, 1H, *J* = 13.6 Hz, C**H**$_a$H$_b$), 5.16 (d, 1H, *J* = 13.6 Hz, -CH$_a$C**H**$_b$), 6.26 (s, 1H, =C**H**), 7.17 (d, 2H, *J* = 7.6 Hz, Ar**H**), 7.89 (d, 2H, *J* = 8.0 Hz, Ar**H**) ;

^{13}C NMR (100 MHz): δ_C (ppm) = 13.1, 13.3, 26.6, 36.8, 42.5, 76.1, 81.8, 89.5, 127.7, 128.5, 131.0, 136.8, 137.3, 138.5, 151.0, 168.2, 197.3

HRMS [M$^+$+Na] calculated: 381.1427, observed: 381.1465

7,9-diethyl-10-hydroxy-4-methyl-1-oxa-7,9-diazaspiro[4.5]dec-3-ene-6,8-dione (4i):

Yield 59%, solid, m.p. 118-120 ^{o}C IR (KBr): ν_{max} = 3443, 1713, 1675 cm^{-1}

^{1}H NMR (CDCl$_3$, 400 MHz): δ_H (ppm) = 1.17 (t, 3H, *J* = 7.2 Hz, -NCH$_2$C**H**$_3$), 1.28 (t, 3H, *J* = 7.2 Hz, -NCH$_2$C**H**$_3$), 1.72 (d, 3H, *J* = 2.0 Hz, -C**H**$_3$), 3.13 (s, 1H, -O**H**), 3.55-3.65 (m, 2H, -NC**H**$_2$CH$_3$), 3.78-3.86 (m, 1H, -NC**H**$_a$H$_b$CH$_3$), 3.89-3.98 (m, 1H, -NCH$_a$**H**$_b$CH$_3$), 4.70 (d, 1H, *J* = 10.8 Hz, -C**H**$_a$H$_b$) 4.73 (s, 1H,-C**H**OH), 4.94 (dd, 1H, *J* = 2.0 Hz, *J* = 10.8 Hz, -CH$_a$**H**$_b$), 5.84 (s, 1H, =C**H**)

^{13}C NMR (100 MHz): δ_C (ppm) = 12.5, 13.5, 13.7, 36.6, 43.1, 75.3, 82.0, 89.5, 126.7, 133.8, 151.7, 168.7

HRMS [M$^+$+Na] calculated: 277.1165, observed: 277.1174

1,3,7-trimethylfuro[3,2-d]pyrimidine-2,4(1H,3H)-dione (4j): Yield 85%, Reference No. 15