Three-component reaction of cyclohexyl isocyanide, barbituric acid and aryl aldehydes; synthesis of *N*-cyclohexyl-*N*-[aryl-(2,4,6-trioxohexahydropyrimidin-5-yl)methyl]formamides

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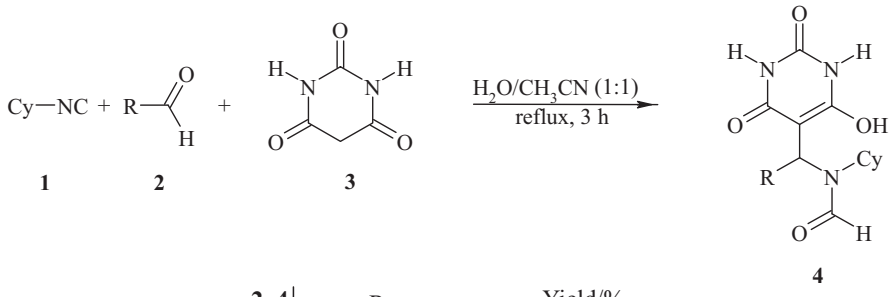
Three-component reaction of cyclohexyl isocyanide, barbituric acid and different aldehydes, in a mixture of water: acetonitrile (1:1) as solvent, produces the corresponding *N*-cyclohexyl-*N*-[aryl-(2,4,6-trioxohexahydropyrimidin-5-yl)methyl]formamides in excellent yields.

Keywords: cyclohexyl isocyanide, barbituric acid, aldehydes, *N*-cyclohexyl-*N*-[aryl-(2,4,6-trioxohexahydropyrimidin-5-yl) methyl]formamides, three-component reaction

A multi-component reaction (MCR) is defined as a reaction in which three or more compounds connect together by covalent bonds. MCRs are powerful tools in the modern drug discovery processes and allow fast, automated and high throughput generation of organic compounds.[1,2] Isocyanides, regarded for many years as compounds with unpleasant odour and very few chemical and pharmaceutical applications, are now looked upon as useful synthons with interesting chemical properties. The change in attitude can be attributed primarily to the renaissance of the isocyanide based multi component reactions (IMCRs).[3-5] Three-component reaction of isocyanides, aldehydes and carboxylic acids known as passerine reaction is one of the most important of IMCRs. Three-component condensation reaction between 4-nitrobenzaldehyde, *N*,*N*'-dimethylbarbituric acid, as a strong organic CH-acid, and alkyl or aryl isocyanides was reported to afford the corresponding furo[2,3-*d*]pyrimidine-2,4(1*H*, 3*H*)-diones.[6] Here we report that the reaction between cyclohexyl isocyanide, barbituric acid and aldehydes, in a mixture of water: acetonitrile (1 : 1) as solvent, produces *N*-cyclohexyl-*N*-[aryl-(2,4,6-trioxohexahydropyrimidin-5-yl) methyl]formamide derivatives in excellent yields (Scheme 1).

The structure of compounds **4a–i** was proved on the basis of the mass spectra and ^{1}H and ^{13}C NMR spectra. The mass spectra of these compounds displayed molecular ion peaks at appropriate *m/z* values. The obtained compounds **4a–i** could exist in solutions as ketone or enole tautomers (Scheme 2). The tautomerism of barbituric acid and substituted barbituric acids have been extensively investigated.[7-9] The ^{1}H NMR spectrum of *N*-cyclohexyl-*N*-[3-methoxyphenyl-(2,4,6-trioxo-hexahydropyrimidin-5-yl)methyl]formamide **4c** in d_6-DMSO showed that this compound existed entirely in the enol form. There were two sharp singlets at δ = 3.76 and 5.27 ppm corresponded to the protons of methoxy and methine protons respectively. A singlet was observed at δ = 8.72, related to the formyl proton. Two NH protons of barbituric acid moiety resonated as two very broad singlets at δ = 9.64 and 9.95 ppm witch disappeared after addition of a few drops of D_2O to d_6-DMSO solution of compound **4c**. A broad signal was observed at δ = 15.50 ppm for OH proton. Cyclohexyl



2, 4	R	Yield/%
a	3-NO_2-C_6H_4	98
b	4-CH_3O-C_6H_4	95
c	3-CH_3O-C_6H_4	95
d	4-Cl-C_6H_4	98
e	C_6H_5	98
f	2-Cl-C_6H_4	96
g	4-CH_3-C_6H_4	95
h	2-CH_3O-C_6H_4	95
i	$Ph(CH_3)CH$	95

Scheme 1

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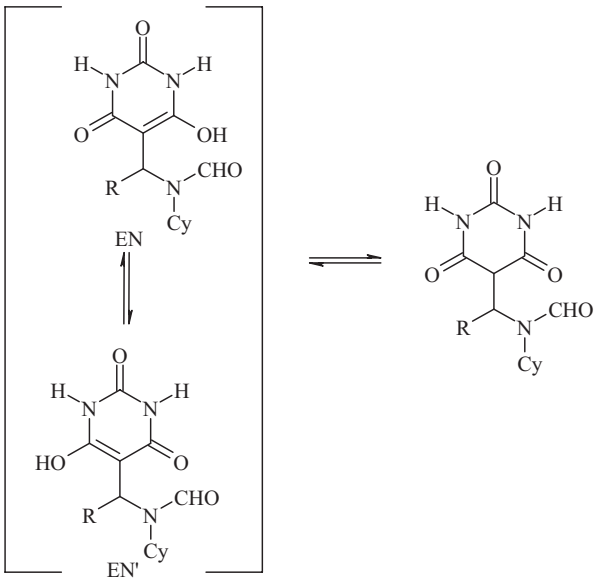


Scheme 2

fragment protons resonated as multiplets at δ = 1.23–1.93 and a multiplet at δ = 3.87 ppm and aromatic protons resonated at δ = 6.81–7.24 ppm. In the ^{13}C NMR spectrum of compound **4c** there was a signal at δ = 86.92 ppm, typical for the sp^2-hybridised C-5 atom of enolised barbituric acids.[9] Notably, in the ^{13}C NMR spectrum of compound **4c** the signals related to C-4 and C-6 atoms of barbituric acid moiety coincided. This is explained by the rapid, probably, intermolecular exchange between the two enolic forms EN–EN'. Similar behaviour has been previously reported for 5-substituted barbituric acids.[9] In the ^{13}C NMR spectrum of compound **4c** there were also peaks at δ = 25.05, 25.22, 25.65, 32.26 and 32.40 ppm for methylenes of cyclohexyl fragment, at 49.86 ppm for (N–CH), at 45.69 ppm for methine carbon, at 55.77 for methoxy group, at 112.71, 113.65, 119.50, 130.69, 141.24 and 160.12 for aromatic carbons, at δ = 150.94 for C-2 of barbituric acid moiety and at 164.54 ppm for formyl carbon.

The NMR spectra of compound **4h** in d_6-DMSO exhibited the signals related to both of the two enol forms EN and EN' of this compound. This showed that the exchange rate between the two enolic forms EN–EN' was so slow that NMR could distinguish the existence of two isomers.

NMR spectra of compounds **4a–i** showed that most of these compounds existed in d_6-DMSO solutions only in the enol form. However, the NMR spectra of *N*-cyclohexyl-*N*-[2-chlorophenyl-(2,4,6-trioxohexahydropyrimidin-5-yl)methyl] formamide **4f** showed that this compound existed in d_6-DMSO solution as a mixture of ketone and enol forms. For example, the ^{1}H NMR spectrum of compound **4f** exhibited, among with the signals related to the enol form, two doublets ($^3J_{HH}$ = 3 H_Z) for vicinal CH protons of ketone form. In the ^{13}C NMR spectrum of compound **4f**, there was a signal at δ = 49.08 ppm, typical for the sp^3-hybridised C-5 atom of barbituric acids.[9] The quantitative content of the enolic form of compound **4f** could be obtained from the ^{1}H NMR spectrum to be 63%.

Compound **4i** possesses two stereogenic centres and so could exist as two diastereomers. The NMR spectra of this compound showed the existence of two diastereomers in 60 : 40 ratio, both of them in the enolic form.

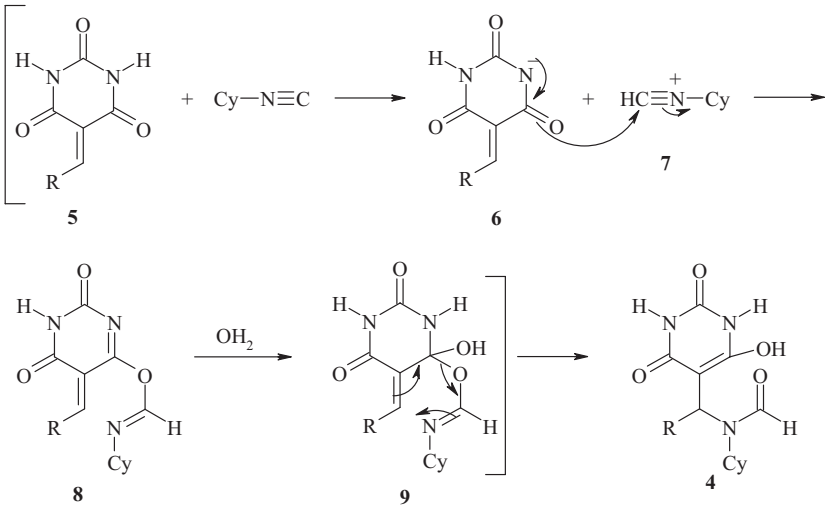
The proposed mechanism for the formation of compounds **4a–i** includes several stages; at the first 5-arylmethylidene barbituric acid **5** is formed from the Knovenagel condensation of aldehyde and barbituric acid (Scheme 3). Then, the proton transfer between intermediate **5** and cyclohexyl isocyanide, followed by the attack of the anion **6** from the harder atom oxygen on the isonitrilium cation **7** affords the adduct **8**. Addition of water to the intermediate **8** and subsequent rearrangement leads to the formamide **4.**

To prove that 5-arylmethylidenebarbituric acid **5** could be an intermediate in the reaction pathway, we prepared and isolated 5-(2-methoxyphenylmethylidene)barbituric acid from the reaction between 2-methoxybenzaldehyde and barbituric acid by a known procedure.[10] When this compound was reacted with cyclohexyl isocysnid in water: acetonitrile mixture (1 : 1), compound **4h** was obtained as the only product in nearly quantitative yield.

In conclusion, we report an effective, simple, one-pot, three-component reaction for preparation of *N*-cyclohexyl-*N*-[aryl-(2,4,6-trioxohexahydropyrimidin-5-yl)methyl]formamides in excellent yields. The present method carries the advantage that the reaction performed under neutral conditions and the substances can be mixed without any activation or modification.

Experimental

Melting points were determined with an electrothermal 9100 apparatus. Elemental analyses were performed using a Heraeus



Scheme 2

CHN-O-Rapid analyser. Mass spectra were recorded on a Finnigan-Mat 8430 mass spectrometer operating at an ionisation potential of 70 eV. IR spectra were recorded on a Shimadzu IR-470 spectrometer. ^{1}H and ^{13}C, NMR spectra were recorded on Bruker DRX-500 Avance spectrometer at solution in d_6-DMSO using TMS as internal standard. The chemicals used in this work purchased from Fluka (Buchs, Switzerland) and were used without further purification.

General procedure

To a magnetically stirred solution of barbituric acid (1 mmol) and aldehyde (1 mmol) in water: acetonitrile (1:1)(10 ml) was added a mixture of cyclohexyl isocyanide (1 mmol) in acetonitrile (0.5 ml) at room temperature. The reaction mixture was then stirred for 3 h at reflux temperature. Water (50 ml) was added and the precipitate was filtered. All products could be crystallised from ethyl acetate: hexane mixture.

N-Cyclohexyl-N-[3-nitrophenyl-(2,4,6-trioxohexahydropyrimidin-5-yl)methyl]formamide (**4a**): Colourless crystals, m.p. 240°C, yield 98%, IR (KBr) (ν_{max}, cm^{-1}): 3285 (NH), 1707, 1603 (C=O). Analyses: Calcd. for $C_{18}H_{20}N_4O_6$: C, 55.67; H, 5.19; N, 14.43%. Found: C, 55.69; H, 5.53; N, 14.59%. MS: *m/z* (%): 388 (M^+, 11). ^{1}H NMR (500 MHz, d_6-DMSO): δ 1.33–1.98 (10 H, m, 5 CH_2), 3.82 (1 H, m, CH of cyclohexyl), 5.63 (1 H, NCH), 7.61–8.34 (4 H, m, aromatic), 8.90 (O=CH), 9.73 and 10.02 (2 H, 2 broad s, 2 NH), 15.25 (1 H, broad s, OH). ^{13}C NMR (125.8 MH_Z, d_6-DMSO): δ 24.70, 24.92, 25.69, 32.15, 32.26 and 49.83 (5 CH_2 and CH of cyclohexyl), 45.60 (NCH), 86.45 (C-5 of barbituric acid), 122.00, 123.12, 131.25, 134.14, 142.31 and 148.73 (aromatic), 150.87 (C-2 of barbituric acid), 164.25 (CH= O), 174.36 (C-4,6 of barbituric acid).

N-Cyclohexyl-N-[4-methylphenyl-(2,4,6-trioxohexahydropyrimidin-5-yl)methyl]formamide (**4b**): Colourless crystals, m.p. 242°C, yield 95%, IR (KBr) (ν_{max}, cm^{-1}): 3300 (NH), 1715, 1599 (C=O). Analyses: Calcd. for $C_{19}H_{23}N_3O_5$: C, 61.11; H, 6.21; N, 11.25. Found: C, 61.46; H, 6.38; N, 11.13%. MS (*m/z*,%): 373 (3). ^{1}H NMR (500 MH_Z, d_6-DMSO): δ 1.22–1.91 (10 H, m, 5 CH_2), 3.76 (3 H, s, CH_3), 3.95 (1 H, m, CH of cyclohexyl), 5.63 (1 H, s, NCH), 6.86–7.39 (4 H, m, aromatic), 8.71 (O=CH), 9.72 and 9.98 (2 H, 2 broad s, 2 NH), 15.62 (1 H, broad s, OH). ^{13}C NMR (125.8 MH_Z, d_6-DMSO): δ 24.64, 24.88, 25.57, 32.25,32.34 and 49.84 (5 CH_2 and CH of cyclohexyl), 54.97 (OCH_3), 45.42 (NCH), 87.34 (C-5 of barbituric acid), 114.26, 128.32, 131.67, and 159.29 aromatic), 149.93 (C-2 of barbituric acid), 164.40 (CH= O), 176.04 (C-4,6 of barbituric acid).

N-Cyclohexyl-N-[3-methoxyphenyl-(2,4,6-trioxohexahydropyrimidin-5-yl)methyl]formamide (**4c**): Colourless crystals, m.p. 253°C, yield 95%, IR (KBr) (ν_{max}, cm^{-1}): 3295 (NH), 1709, 1602 (C=O). Analyses: Calcd. for $C_{19}H_{23}N_3O_5$: C, 61.11; H, 6.21; N, 11.25. Found: C, 61.22; H, 6.30; N, 11.39%. MS (*m/z*,%): 373 (5). ^{1}H NMR (500 MH_Z, d_6-DMSO): δ 1.23–1.93 (10 H, m, 5 CH_2), 3.76 (3 H, s, OCH_3), 3.87 (1 H, m, CH of cyclohexyl), 5.27 (1 H, s, NCH), 6.81–7.24 (4 H, m, aromatic), 8.72 (O=CH), 9.64 and 9.95 (2 H, 2 broad s, 2 NH), 15.50 (1 H, broad s, OH). ^{13}C NMR (125.8 MH_Z, d_6-DMSO): δ 25.05, 25.22, 25.65, 32.26, 32.40 and 49.86 (5 CH_2 and CH of cyclohexyl), 45.69 (NCH), 55.77 (OCH_3), 86.92 (C-5 of barbituric acid), 112.71, 113.65, 119.50, 130.69, 141.24 and 160.12 (aromatic), 150.94 (C-2 of barbituric acid), 164.93 (CH= O), 175.26 (C-4,6 of barbituric acid).

N-Cyclohexyl-N-[4-chlorophenyl-(2,4,6-trioxohexahydro-pyrimidin-5-yl)methyl]formamide (**4d**): Colourless crystals, m.p. 249°C, yield 98%, IR (KBr) (ν_{max}, cm^{-1}): 3310 (NH), 1709, 1603 (C=O). Analyses: Calcd. for $C_{18}H_{20}ClN_3O_4$: C, 57.22; H, 5.34; ; N, 11.12. Found: C, 57.34; H, 5.59; N, 11.50%. MS (*m/z*,%): 377 (7). ^{1}H NMR (500 MH_Z, d_6-DMSO): δ 1.21–1.92 (10 H, m, 5 CH_2), 3.80 (1 H, m, CH of cyclohexyl), 5.30 (1 H, s, NCH), 7.38–7.46 (4 H, m, aromatic), 8.78 (O=CH), 9.70 and 9.99 (2 H, 2 broad s, 2 NH), 15.20 (1 H, broad s, OH). ^{13}C NMR (125.8 MH_Z, d_6-DMSO): δ 24.99, 25.06, 25.71, 32.31, 32.40 and 49.80 (5 CH_2 and CH of cyclohexyl), 45.25 (NCH), 86.76 (C-5 of barbituric acid), 129.25, 129.47, 132.60, 138.99 (aromatic), 150.88 (C-2 of barbituric acid), 164.71 (CH= O), 175.01 (C-4,6 of barbituric acid).

N-Cyclohexyl-N-[phenyl-(2,4,6-trioxohexahydropyrimidin-5-yl)methyl]formamide (**4e**): Colourless crystals, m.p. 250°C, yield 98%, IR (KBr) (ν_{max}, cm^{-1}): 3300 (NH), 1706, 1601 (C=O). Analyses: Calcd. for $C_{18}H_{21}N_3O_4$: C, 62.96; H, 6.16; N, 12.24. Found: C, 62.70; H, 6.24; N, 12.19%. MS (*m/z*,%): 343 (7). ^{1}H NMR (500 MH_Z, d_6-DMSO): δ 1.22–1.95 (10 H, m, 5 CH_2), 3.80 (1 H, m, CH of cyclohexyl), 5.63 (1 H, s, NCH), 7.23–7.34 (4 H, m, aromatic), 8.72 (O=CH), 9.59 and 9.93 (2 H, 2 broad s, 2 NH), 15.63 (1 H, broad s, OH). ^{13}C NMR (125.8 MH_Z, d_6-DMSO): δ 25.00, 25.07, 25.65, 32.30, 32.39 and 49.87 (5 CH_2 and CH of cyclohexyl), 45.73 (NCH), 87.08 (C-5 of barbituric acid), 127.27, 128.03, 129.52, and 139.71 (aromatic), 150.97 (C-2 of barbituric acid), 164.34 (CH= O), 175.40 (C-4,6 of barbituric acid).

N-Cyclohexyl-N-[2-chlorophenyl-(2,4,6-trioxohexahydro-pyrimidin-5-yl)methyl]formamide (**4f**): Colourless crystals, m.p. 230°C, yield 96%, IR (KBr) (ν_{max}, cm^{-1}): 3300 (NH), 1717, 1601 C=O). Analyses: Calcd. for $C_{18}H_{20}ClN_3O_4$: C, 57.22; H, 5.34; N, 11.12. Found: C, 57.17; H, 5.43; N, 11.06%. MS (*m/z*,%): 377 (4). NMR data for enol form: ^{1}H NMR (500 MH_Z, d_6-DMSO): δ 1.12–1.89 (10 H, m, 5 CH_2), 3.79 (1 H, m, CH of cyclohexyl), 5.51 (1 H, s, NCH), 7.27–7.50 (4 H, m, aromatic), 8.62 (O=CH), 9.90 and 10.11 (2 H, 2 broad s, 2 NH), 15.50 (1 H, broad s, OH). ^{13}C NMR (125.8 MH_Z, d_6-DMSO): δ 25.10, 25.21, 25.83, 32.71, 32.84 and 49.08 (5 CH_2 and CH of cyclohexyl), 47.86 (NCH), 127.06, 129.43, 129.54, 132.89, 133.81 and 136.82 (aromatic), 151.79 (C-2 of barbituric acid), 164.77 (CH= O), 170.83 (C-4,6 of barbituric acid). NMR data for keto form: ^{1}H NMR (500 MH_Z, d_6-DMSO): δ 1.12–1.89 (10 H, m, 5 CH_2), 3.79 (1 H, m, CH of cyclohexyl), 3.76 (1 H, d $^3J_{HH}$ = 3 H_Z, CH-5 of barbituric acid), 5.18 (1 H, d $^3J_{HH}$ = 3 H_Z, NCH), 7.27–7.50 (4 H, m, aromatic), 8.62 (O=CH), 9.90 and 10.11 (2 H, 2 broad s, 2 NH). ^{13}C NMR (125.8 MH_Z, d_6-DMSO): δ 25.10, 25.21, 25.83, 32.71, 32.84 and 49.08 (5 CH_2 and CH of cyclohexyl), 47.86 (NCH), 50.51 (C-5 of barbituric acid), 126.03, 130.05, 130.78, 132.89, 133.81 and 136.02 (aromatic), 151.79 (C-2 of barbituric acid), 165.74 (CH= O), 169.76 (C-4,6 of barbituric acid).

N-Cyclohexyl-N-[4-methylphenyl-(2,4,6-trioxohexahydro-pyrimidin-5-yl)methyl]formamide (**4g**): Colourless crystals, m.p. 242°C, yield 95%, IR (KBr) (ν_{max}, cm^{-1}): 3295 (NH), 1706, 1599 (C=O). Analyses: Calcd. for $C_{19}H_{23}N_3O_4$: C, 63.85; H, 6.49; N, 11.76. Found: C, 63.68; H, 6.31; N, 11.71%. MS (*m/z*,%): 357 (8). ^{1}H NMR (500 MH_Z, d_6-DMSO): δ 1.23–1.97 (10 H, m, 5 CH_2), 2.28 (3 H, s, CH_3), 3.77 (1 H, m, CH of cyclohexyl), 5.25 (1 H, s, NCH), 7.12–7.26 (4 H, m, aromatic), 8.69 (O=CH), 9.62 and 9.93 (2 H, 2 broad s, 2 NH), 15.52 (1 H, broad s, OH). ^{13}C NMR (125.8 MH_Z, d_6-DMSO): δ 21.28 (CH_3), 24.80, 25.08, 25.67, 32.31, 32.40 and 49.83 (5 CH_2 and CH of cyclohexyl), 45.38 (NCH), 87.19 (C-5 of barbituric acid), 127.20, 130.02, 136.76 and 137.26 (aromatic), 150.95 (C-2 of barbituric acid), 165.95 (CH= O), 175.58 (C-4,6 of barbituric acid).

N-Cyclohexyl-N-[2-methoxyphenyl-(2,4,6-trioxohexahydro-pyrimidin-5-yl)methyl]formamide (**4h**): Colourless crystals, m.p. 252°C, yield 95%, IR (KBr) (ν_{max}, cm^{-1}): 3185 (NH), 1698, 1665 (C=O,). Analyses: Calcd. for $C_{19}H_{23}N_3O_5$: C, 61.11; H, 6.21; N, 11.25. Found: C, 61.22; H, 6.21; N, 11.46%. MS (*m/z*,%): 373 (8). NMR data for an enol form (NMR data for other enol form): ^{1}H NMR (500 MH_Z, d_6-DMSO): δ 1.13–1.86 (10 H, m, 5 CH_2), 3.51 (1 H, m, CH of cyclohexyl), 3.75 (3.79) (3 H, s, CH_3), 4.81 (5.25) (1 H, s, NCH), 6.81–7.26 (4 H, m, aromatic), 7.83 (9.23) (O=CH), 11.06 and 10.82 (10.63 and 11.12) (2 H, 2 broad s, 2 NH), 15.39 (1 H, broad s, OH). ^{13}C NMR(125.8 MHz, d_6-DMSO): δ 25.21, 25.41, 25.51, 25.94, 32.55, 33.00 and 48.89 (25.32, 25.40, 25.81, 32.36, 32.83, 49.66 (5 CH_2 and CH of cyclohexyl), 55.78 (56.20) (OCH_3), 47.71 (48.09) (NCH), 85.52 (C-5 of barbituric acid), 111.62, 121.05, 127.31, 128.71, 131.14, and 157.19 (110.87, 120.41, 126.35, 128.27, 129.45, 157.98 (aromatic), 150.90 (151.87) (C-2 of barbituric acid), 164.78 (O=CH), 169.90 and 171.21 (170.54 and 175.02) (C-4,6 enol form of barbituric acid).

N-Cyclohexyl-N-[1-phenylethyl-(2,4,6-trioxohexahydropyrimidin-5-yl)methyl]formamide (**4i**): Colourless crystals, m.p. 254°C, yield 95%, IR (KBr) (ν_{max}, cm^{-1}): 3330 (NH), 1710, 1599 (C=O). Analyses: Calcd. for $C_{20}H_{25}N_3O_4$: C, 64.67; H, 6.78; N, 11.31. Found: C, 64.48; H, 6.50; N, 11.28%. MS (*m/z*,%): 371 (11). NMR data for major isomer (for minor isomer) ^{1}H NMR (500 MHz, d_6-DMSO): δ 1.02–1.83 (10 H, m, 5 CH_2), 1.10 (1.20) (3 H, d $^3J_{HH}$ = 6 H_Z, CH_3), 3.62 (1 H, m, CH of cyclohexyl), 3.22 (3.15) (1 CH, m, CH), 4.06 (4.03) (1 H, d $^3J_{HH}$ = 6 H_Z, CH), 7.08–7.40 (5 H, m, aromatic), 8.79 (9.38) (H, broad s, O=CH), 10.70,11.11 (10.23, 10.91) (2 H, broad s, 2 NH) 15.65 (15.19) (1 H, broad s, OH). ^{13}C NMR (125.8 MH_Z, d_6-DMSO): δ 24.62, 25.10, 25.70, 31.68, 32.34, and 48.89 (24.91, 25.18, 25.85, 32.05, 32.72 and 49.61) (5 CH_2 and CH of cyclohexyl), 20.67 (20.72) (CH_3), 42.63 (41.31 (CH), 47.54 (47.00) (NCH), 86.11 (86.93) (C-5 of barbituric acid), 128.12, 128.59, 128.87, 144.34 (127.11, 127.25, 128.54, 144.55) (aromatic), 150.89 (150.64), (C-2 of barbituric acid), 165.91 (162.35) (CH= O), 175.79 (176.86) (C-4,6 enol form of barbituric acid).

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