A New Biginelli Reaction Procedure using Potassium Hydrogen Sulfate as the Promoter for an Efficient Synthesis of 3,4-Dihydropyrimidin-2(1*H*)-one

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Simple and improved conditions have been found to carry out the Biginelli reaction for the synthesis of 3,4-dihydropyrimidin-2(1*H*)-one derivatives. This synthesis was performed using potassium hydrogen sulfate as the promoter in ethylene glycol solution. Compared with the classical Biginelli reaction conditions, this new method has the advantage of excellent yields (85-95%) and short reaction time (0.5-2 h).

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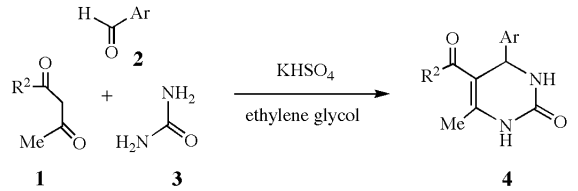
Dihydropyrimidinone derivatives have attracted considerable interest in recent years because this type of compounds exhibits attractive pharmacological profiles as calcium channel blockers, antihypertensive agents, alpha-la-antagonists and neuropeptide Y (NPY) antagonists [1]. In addition, several marine alkaloids containing the dihydropyrimidinone-5-carboxylate motifs also showed interesting biological properties [2].

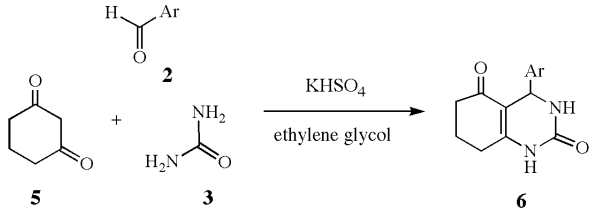
Biginelli synthesis involves reaction of ethyl acetoacetate, benzaldehyde and urea in alcohol solution in the presence of a catalytic amount of hydrogen chloride to give 3,4-dihydropyrimidin-2(1*H*)-ones [3]. A major drawback of the classical Biginelli reaction is the poor to moderate yields, particularly when substituted aromatic aldehydes were employed. Therefore, several improved procedures for the preparation of DHPMs ("Beginelli compounds") have been reported during the last two decades [4-11]. Among the improved synthetic methods is to use BF_3•OEt_2 as promoter as reported by Hu and Sidler [12]. Later on, Kappe and coworkers further improved this reaction by employing microwave irradiation in the presence of PPE to give higher chemical yields of dihydropyrimidinone products [13]. Recently, the use of lanthanide compounds [14-15], Lewis acids [16-20], silica sulfuric acid [21], and $InBr_3$ [22] also gave improved yield. In our previous communication [23], we reported that boric acid can catalyze Beginelli reaction. All these improved procedures can overcome the drawback of the classical Biginelli reaction to some extent.

However, attention has so far been mainly paid to using open-chained 1,3-dicarbonyl compounds to afford dihydropyrimidinone derivatives, while the use of cyclic 1,3-dicarbonyl compounds has been seldom reported. Herein, we would like to report a new economic approach to the Beginelli reaction products using $KHSO_4$ as the promoter in ethylene glycol. This catalyst is efficient not only for open-chained 1,3-dicarbonyl compounds, but also for cyclic 1,3-dicarbonyl compounds. The synthesis can be finished within 2 hours at 100 °C to give very high yields. (Scheme 1, Scheme 2).

Scheme 1



Scheme 2



The results (Table 1, Table 2) show that a wide range of aldehydes can take part in this reaction to give excellent yields (85-95%) of products. This new procedure is simple and the work-up consists of simple filtration. All the products were characterized by IR and ^{1}H NMR analysis, and their melting points are identical to those of the known compounds reported in the literature.

According to the mechanism suggested by Folkers, Johnson and Kappe, we think the reaction may proceed through imine formation from the aldehyde and urea, which is activated by protonation. Subsequent addition of the carbanion to the imine followed by cyclodehydration afford dihydropyrimidin-2(1*H*)-one (Scheme 3).

During the reaction process, the hydrogen ion, H^+, is donated by the potassium hydrogen sulfate. The hydrogen ion can not only help the dehydration but also benefit the enolization of 1,3-diketone or β-keto ester to form the enolate intermediate. The solvent ethylene glycol can particularly accelerate the dehydration reaction of the last step.

Table 1

$KHSO_4$ Catalyzed Cyclic 1,3-Dicarbonyl Compounds

Entry	ArCHO	Yield(%)	Mp(°C)
6a	4-ClC_6H_4CHO	93	259.9-261.3
6b	2,4-$(Cl)_2$-C_6H_3CHO	95	226.8-227.7
6c	3,4-$(Cl)_2$-C_6H_3CHO	95	>300
6d	3-$NO_2C_6H_4CHO$	92	268.1-268.4

In the search for a more facile method for the rapid assembly of a targeted library, we investigated the use of microwave irradiation to assist the three-component reaction. In the presence of $KHSO_4$, compare to the previous procedure, the reaction time could be dramatically shortened from 2 h to 5 min, and the amount of solvent decreased from 10 mL to 1 mL, with similar yields.

In conclusion, $KHSO_4$ can be applied as an efficient catalyst not only for the open-chained 1,3-dicarbonyl compounds, but also for cyclic 1,3-dicarbonyl compounds. In addition, the catalyst is suitable for aromatic, aliphatic and hetrocyclic aldehydes. The application of this novel catalyst resulted in decreased reaction time and increased yields of the potentially biologically active dihydropyrimidinone derivatives. This method also has the advantage of an easy work-up and being environmentally friendly because of the atomic economy.

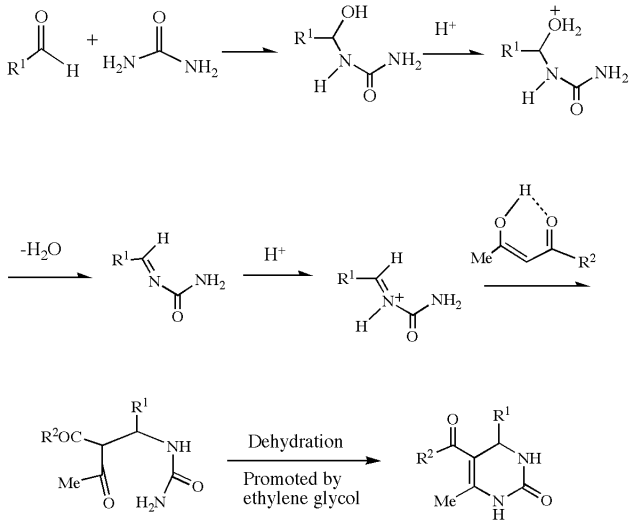
Table 2

$KHSO_4$ Catalyzed Open-Chained 1,3-Dicarbonyl Compounds

Entry	ArCHO	R^2	Yield(%)			Mp(°C)	
			A [a]	B [b]	C [c]	Found	Reported
4a	C_6H_5CHO	OEt	95	94	78	202-203	202-203 [20]
4b	2-ClC_6H_4CHO	OEt	91	-	51	215-217	215-218 [13]
4c	3,4-$OCH_2OC_6H_3CHO$	OEt	90	-	49	186-187	187-188 [20]
4d	4-$NO_2C_6H_4CHO$	OEt	92	91	58	207-208	207-208.5 [20]
4e	4-$NMe_2C_6H_4CHO$	OEt	86	-	-	257-258	256-257 [20]
4f	2-OHC_6H_4CHO	OEt	86	-	19	202-203	201-203 [20]
4g	2,4-$(Cl)_2$-C_6H_3CHO	OEt	91	-	69	248-250	249-250 [20]
4h	4-ClC_6H_4CHO	OEt	93	92	56	214-215	213-215 [20]
4i	4-OHC_6H_4CHO	OEt	87	-	67	228-230	227-229 [20]
4j	4-$NO_2C_6H_4CHO$	Me	91	-	-	227-229	230 [15]
4k	4-$OCH_3C_6H_4CHO$	Me	91	-	-	165-168	168-170 [15]
4l	4-$NO_2C_6H_4CHO$	OMe	93	92	41	237-238	235-237 [12]
4m	4-$OCH_3C_6H_4CHO$	OMe	90	87	28	193-196	192-194 [12]
4 n	4-ClC_6H_4CHO	OMe	95	95	56	206-208	204-207 [12]
4o	4-FC_6H_4CHO	OMe	87	88	-	193-195	192-194 [15]
4p	2-$NO_2C_6H_4CHO$	OMe	90	-	-	280-282	280-282 [23]
4q	2-NO_2-5-Cl-C_6H_3CHO	OMe	89	-	-	290-292	290-292 [23]
4r	$CH_3CH_2CH_2CHO$	OEt	85	-	15	152-153	152-154 [20]
4s	$(CH_3)_2CHCHO$	OEt	86	-	10	172-174	170-172 [20]
4t	Furfural	OEt	85	-	-	205	203-205 [18]

[a] Method A: cat. $KHSO_4$ in ethylene glycol at 100 °C for 0.5-2 h; [b]. Method B: 1.3 equiv of BF_3•OEt_2, 10 mol % CuCl, 10 mol % AcOH, in THF, reflux for 18 h [12]; [c] Method C: cat. HCl in EtOH, reflux for 18 h [14, 24].

Scheme 3



EXPERIMENTAL

Melting points were determined in open capillaries and are uncorrected. IR spectra were recorded on a Shimadzu spectrometer. ^1H NMR spectra were measured on a DPX 400 MHz spectrometer using TMS as internal standard, DMSO-d_6 as solvent.

General Procedure for 3,4-Dihydropyrimidin-2(1*H*)-ones **4**.

A solution of the appropriate aldehyde (3 mmol), 1,3-dicarbonyl compounds (3 mmol), urea (3.6 mmol), $KHSO_4$ (0.6 mmol), in ethylene glycol (10 mL) is heated at 100 °C, while stirring for 0.5-2 h. Then it is cooled down to room temperature, and

poured into 50 mL ice-water. The solid products are filtered, washed with ice-water and ethanol (95%), dried and recrystallized from hot ethanol to give the pure product.

4-Phenyl-5-ethoxycarbonyl-6-methyl-3,4-dihydropyrimidin-2(1*H*)-one (**4a**).

This compound was obtained as a solid: mp 202-203°C (Lit. [20] 202-204 °C). ^{1}H NMR (400 MHz, DMSO-d_6): δ 9.17 (s, 1H), 7.72 (s, 1H), 7.32-7.21 (m, 5H), 5.14 (s, 1H), 3.98 (q, *J*=7.2 Hz, 2H), 2.24 (s, 3H), 1.08 (t, *J*=7.2 Hz, 3H). IR (KBr): 3414, 3230, 3109, 2936, 1702, 1649 cm^{-1}.

4-(2-Chlorophenyl)-5-ethoxycarbonyl-6-methyl-3,4-dihydropyrimidin-2(1*H*)-one (**4b**).

This compound was obtained as a solid: mp 215-217 °C (Lit. [13] 214 °C). ^{1}H NMR (400 MHz, DMSO-d_6): δ 9.30 (s, 1H), 7.72 (s, 1H), 7.23-7.16 (m, 4H), 5.67 (d, *J*=2.5 Hz, 1H), 3.91 (q, *J*=7.5Hz, 2H), 2.32 (s, 3H), 1.08 (t, *J* = 7.5 Hz, 3H). IR (KBr): 3360, 3220, 3100, 1690, 1640 cm^{-1}.

4-(3,4-Methylenedioxylphenyl)-5-ethoxycarbonyl-6-methyl-3,4-dihydropyrimidin-2(1*H*)-one (**4c**).

This compound was obtained as a solid: mp 186-187 °C (Lit. [20] 187-188 °C). ^{1}H NMR (400 MHz, DMSO-d_6): δ 9.16 (s, 1H), 7.66 (s, 1H), 6.83 (d, *J*=8.0 Hz, 1H), 6.73 (s, 1H), 6.67 (d, *J*=8.0 Hz, 1H), 5.96 (s, 2H), 5.05 (d, *J*=3.2 Hz, 1H), 3.97 (q, *J*=7.2 Hz, 2H), 2.23 (s, 3H), 1.09 (t, *J*=7.2Hz, 3H). IR (KBr): 3414, 3233, 2975, 1700, 1696, 1638 cm^{-1}.

4-(4-Nitrophenyl)-5-ethoxycarbonyl-6-methyl-3,4-dihydropyrimidin-2(1*H*)-one (**4d**).

This compound was obtained as a solid: mp 207-208 °C (Lit. [20] 207-208.5 °C). ^{1}H NMR (400 MHz, DMSO-d_6): δ 9.32 (s, 1H), 8.28 (d, *J*=8.4 Hz, 2H), 7.89 (s, 1H), 7.63 (d, *J*=8.4 Hz, 2H), 5.26 (d, *J*=3.2 Hz, 1H), 3.94 (q, *J*=7.2 Hz, 2H), 2.28 (s, 3H), 1.07 (t, *J*=7.2 Hz, 3H). IR (KBr): 3416, 3235, 3109, 2975, 1701, 1645 cm^{-1}.

4-(4-Dimethylaminophenyl)-5-ethoxycarbonyl-6-methyl-3,4-dihydropyrimidin-2(1*H*)-one (**4e**).

This compound was obtained as a solid: mp 257-258 °C (Lit. [20] 256-257 °C). ^{1}H NMR (400 MHz, DMSO-d_6): δ 9.07 (s, 1H), 7.57 (s, 1H), 7.02 (d, *J*=8.4 Hz, 2H), 6.64 (d, *J*=8.4 Hz, 2H), 5.02 (d, *J*=3.2 Hz, 1H), 3.96 (q, *J*=7.2 Hz, 1H), 2.83 (s, 6H), 2.22 (s, 3H), 1.10 (t, *J*=7.2 Hz, 3H). IR (KBr): 3420, 3245, 3117, 2976, 1704, 1647 cm^{-1}.

4-(2-Hydroxylphenyl)-5-ethoxycarbonyl-6-methy-3,4-dihydropyrimidin-2(1*H*)-one (**4f**).

This compound was obtained as a solid: mp 202-203 °C (Lit. [20] 201-203°C). ^{1}H NMR (400 MHz, DMSO-d_6): δ 9.33 (s, 1H), 8.20 (d, *J*=8.4 Hz, 2H), 7.87 (s, 1H), 7.49 (d, *J*=8.4 Hz, 2H), 5.26 (d, *J*=3.2 Hz, 1H), 3.97 (q, *J*=7.2 Hz, 2H), 2.26 (s, 3H), 1.08 (t, *J*=7.2 Hz, 3H). IR (KBr): 3414, 3236, 3119, 2984, 1702, 1644 cm^{-1}.

4-(2,4-Dichlorophenyl)-5-ethoxycarbonyl-6-methyl-3,4-dihydropyrimidin-2(1*H*)-one (**4g**).

This compound was obtained as a solid: mp 248-250 °C (Lit. [20] 249-250 °C). ^{1}H NMR (400 MHz, DMSO-d_6): δ 9.33 (s, 1H), 7.77 (s, 1H), 7.31-7.57 (m, 3H), 5.59 (d, *J*=2.8 Hz, 1H), 3.90 (q, *J*=7.2 Hz, 2H), 2.29 (s, 3H), 1.00 (t, *J*=7.2 Hz, 3H). IR (KBr): 3415, 3219, 3104, 2969, 1699, 1641 cm^{-1}.

4-(4-Chlorophenyl)-5-ethoxycarbonyl-6-methyl-3,4-dihydropyrimidin-2(1*H*)-one (**4h**).

This compound was obtained as a solid: mp 214-215°C (Lit. [20] 213-215 °C). ^{1}H NMR (400 MHz, DMSO-d_6): δ 9.23 (s, 1H), 7.75 (s, 1H), 7.37 (d, *J*=8.4 Hz, 2H), 7.23 (d, *J*=8.4 Hz, 2H), 5.12 (d, *J*=2.8 Hz, 1H), 3.98 (q, *J*=7.1 Hz, 2H), 2.24 (s, 3H), 1.08 (t, *J*=7.1 Hz, 3H). IR (KBr): 3419, 3242, 3116, 2979, 1703, 1648 cm^{-1}.

4-(4-Hydroxylphenyl)-5-ethoxycarbonyl-6-methy-3,4-dihydropyrimidin-2(1*H*)-one (**4i**).

This compound was obtained as a solid: mp 228-230 °C (Lit. [20] 227-229 °C). ^{1}H NMR (400 MHz, DMSO-d_6): δ 9.32 (s, 1H), 9.11 (s, 1H), 7.61 (s, 1H), 7.02 (d, *J*=8.4 Hz, 2H), 6.69 (d, *J*=8.4 Hz, 2H), 5.04 (d, *J*=3.2 Hz, 1H), 3.97 (q, *J*=7.1 Hz, 2H), 2.23 (s, 3H), 1.08 (t, *J*=7.2 Hz, 3H). IR (KBr): 3417, 3241, 3120, 2984, 1687, 1649 cm^{-1}.

4-(4-Nitrophenyl)-5-aceto-6-methyl-3,4-dihydropyrimidin-2(1*H*)-one (**4j**).

This compound was obtained as a solid: mp 227-229°C (Lit.15 230°C). ^{1}H NMR (400MHz, DMSO-d_6): δ 1H), 7.51 (d, *J*=8.4 Hz, 2H), 5.39 (s, 1H), 2.32 (s, 3H), 2.19 (s, 3H). IR (KBr): 3242, 3116, 2979, 1703, 1699, 1648 cm^{-1}.

4-(4-Methoxyphenyl)-5-aceto-6-methyl-3,4-dihydropyrimidin-2(1*H*)-one (**4k**).

This compound was obtained as a solid: mp 165-168 °C (Lit. [15] 166-168 °C). ^{1}H NMR (400 MHz, DMSO-d_6): δ 9.16 (s, 1H), 7.78 (s, 1H), 7.16 (d, *J*=8.7 Hz, 2H), 6.88 (d, *J*=8.7 Hz, 2H), 5.20 (d, *J*=3.0 Hz, 1H), 3.72 (s, 3H), 2.28 (s, 3H), 2.07 (s, 3H). IR (KBr): 3242, 1714, 1624 cm^{-1}.

4-(4-Nitrophenyl)-5-methoxycarbonyl-6-methyl-3,4-dihydropyrimidin-2(1*H*)-one (**4l**).

This compound was obtained as a solid: mp 237-238 °C (Lit. [12] 236-238 °C). ^{1}H NMR (400 MHz, DMSO-d_6): δ 9.36 (s, 1H), 8.21 (d, *J*=8.8Hz, 2H), 7.93 (s, 1H), 7.51 (d, *J*=8.8 Hz, 2H), 5.29 (d, *J*=2.7Hz, 1H), 3.54 (s, 3H), 2.27(s, 3H). IR (KBr): 3362, 3221, 3113, 2949, 1711, 1635 cm^{-1}.

4-(4-Methoxyphenyl)-5-methoxycarbonyl-6-methyl-3,4-dihydropyrimidin-2(1*H*)-one (**4m**).

This compound was obtained as a solid: mp 193-196 °C(Lit. [12] 192-194 °C). ^{1}H NMR (400 MHz, DMSO-d_6): δ 9.19 (s, 1H), 7.69 (s, 1H), 7.14 (d, *J*=8.7 Hz, 2H), 6.88 (d, *J*=8.7 Hz, 2H), 5.09 (d, *J*=2.7 Hz, 1H), 3.72 (s, 3H), 3.52 (s, 3H), 2.24 (s, 3H). IR (KBr): 3415, 3247, 3111, 2953, 1719, 1682 cm^{-1}.

4-(4-Chlorophenyl)-5-methoxycarbonyl-6-methyl-3,4-dihydropyrimidin-2(1*H*)-one (**4n**).

This compound was obtained as a solid: mp 206-208 °C (Lit. [12] 204-207 °C). ^{1}H NMR (400 MHz, DMSO-d_6): δ 9.30 (s, 1H), 7.82 (s, 1H), 7.37 (d, *J*=8.4 Hz, 2H), 7.23 (d, *J*=8.4 Hz, 2H), 5.14 (d, *J*=2.8 Hz, 1H), 3.53 (s, 3H), 2.25 (s, 3H). IR (KBr): 3364, 3221, 3103, 2947, 1712, 1636 cm^{-1}.

4-(4-Fluorophenyl)-5-methoxycarbonyl-6-methyl-3,4-dihydropyrimidin-2(1*H*)-one (**4o**).

This compound was obtained as a solid: mp 193-195 °C (Lit. [15] 192-194 °C). ^{1}H NMR (400 MHz, DMSO-d_6): δ 9.27 (s,

1H), 7.79 (s, 1H), 7.29-7.23 (m, 4H), 5.16 (d, *J*=3.0 Hz, 1H), 3.54 (s, 3H), 2.26 (s, 3H). IR (KBr): 3326, 1682, 1603 cm^{-1}.

4-(2-Nitrophenyl)-5-methoxycarbonyl-6-methyl-3,4-dihydropyrimidin-2(1*H*)-one (**4p**).

This compound was obtained as a solid: mp 280-282 °C (Lit. [23] 280-282 °C). ^{1}H NMR (400 MHz, DMSO-d_6): δ 9.38 (s, 1H), 8.13-8.11(m, 2H), 7.91 (s, 1H), 7.66-7.61 (m, 2H), 5.29 (d, *J*=3.1Hz, 1H), 3.53 (s, 3H), 2.27 (s, 3H). IR(KBr): 3539, 3232, 3108, 2954, 1702, 1644 cm^{-1}.

4-(2-Nitro-5-chlorophenyl)-5-methoxy-carbonyl-6-methyl-3,4-dihydropyrimidin-2(1*H*)-one (**4q**).

This compound was obtained as a solid: mp 290-292 °C (Lit. [23] 290-292 °C). ^{1}H NMR (400MHz, DMSO-d_6): δ 9.45 (s, 1H), 7.95 (s, 1H), 7.92 (s, 1H), 7.61-7.45 (m, 2H), 5.80 (d, *J*=2.7 Hz, 1H), 3.39 (s, 3H), 2.26 (s, 3H). IR (KBr): 3359, 3233, 3119, 2953, 1703, 1644 cm^{-1}.

4-(*n*-Propyl)-5-ethoxycarbonyl-6-methyl-3,4-dihydropyrimidin-2(1*H*)-one (**4r**).

This compound was obtained as a solid: mp 152-153 °C (Lit. [20] 152-154 °C). ^{1}H NMR (400MHz, DMSO-d_6): δ 8.55 (s, 1H), 6.30 (s, 1H), 4.31 (s, 1H), 4.15 (q, *J*=7.2 Hz, 2H), 2.28 (s, 3H), 1.40-1.59(m, 4H), 1.28 (t, *J*=7.2 Hz, 3H), 0.90 (t, *J*=7.2 Hz, 3H). IR (KBr): 3418, 3245, 3117, 1705, 1674, 1645 cm^{-1}.

4-(*i*-Propyl)-5-ethoxycarbonyl-6-methyl-3,4-dihydropyrimidin-2(1*H*)-one (**4s**).

This compound was obtained as a solid: mp 172-174 °C (Lit. [20] 170-171 °C). ^{1}H NMR (400MHz, DMSO-d_6): δ 8.60 (s, 1H), 6.29 (s, 1H), 4.22 (s, 1H), 4.15 (q, *J*=7.2 Hz, 2H), 2.28 (s, 3H), 1.89 (m, 1H), 1.26 (t, *J*=7.2 Hz, 3H), 0.90 (d, *J*=6.4 Hz, 3H) 0.85(d, .*J*=6.4 Hz, 3H). IR (KBr): 3410, 3235, 3100, 1702, 1645 cm^{-1}.

4-(Furfural)-5-ethoxycarbonyl-6-methyl-3,4-dihydropyrimidin-2(1*H*)-one (**4t**).

This compound was obtained as a solid: mp 205 °C (Lit. [18] 203-205 °C). ^{1}H NMR (400 MHz, DMSO-d_6): δ 9.22 (s, 1H), 7.74 (s, 1H), 7.53 (s, 1H), 6.33 (d, *J*=2.8 Hz, 1H), 6.07 (d, *J*=2.8 Hz, 1H), 5.20 (s, 1H), 3.98 (q, *J*=7.2 Hz, 2H), 2.22 (s, 3H), 1.09 (t, *J*=7.2 Hz, 3H). IR (KBr): 3413, 3239, 3119, 2984, 1702, 1644, 1457 cm^{-1}.

4-(4-Chlorophenyl)-1,2,3,4,5,6,7,8-octahydro-2,5-oxo-quinoxalin (**6a**).

This compound was obtained as a solid: mp 259.9-261.3 °C. ^{1}H NMR (400 MHz, DMSO-d_6): δ 9.53 (s, 1H), 7.79 (s, 1H), 7.25 (d, *J*=8.4 Hz, 2H), 7.20 (d, *J*=8.4 Hz, 2H), 5.17(s, 1H), 1.81-1.92 (m, 2H), 2.20-2.21 (m, 2H) 2.35-2.49 (m, 2H). IR (KBr): 3222, 3094, 2952, 1699, 1609, 1483, 1375, 1242, 1184, 1128, 1093, 1013, 950, 819, 542 cm^{-1}.

4-(2,4-Dichlorophenyl)-1,2,3,4,5,6,7,8-octahydro-2,5-oxo-quinoxalin (**6b**).

This compound was obtained as a solid: mp 226.8-227.7 °C. ^{1}H NMR (400MHz, DMSO-d_6): δ 9.59 (s, 1H), 7.71 (s, 1H), 7.19-7.44 (m, 4H), 5.52(s, 1H), 1.82-1.93 (m, 2H), 2.17-2.28 (m, 2H) 2.35-2.48 (m, 2H). IR (KBr): 3244, 3103, 2950, 1701, 1648, 1456, 1368, 1240, 1184, 1133, 1095, 1042, 952, 902, 808, 773, 532cm^{-1}.

4-(3,4-Dichlorophenyl)-1,2,3,4,5,6,7,8-octa-hydro-2,5-oxo-quinoxalin (**6c**).

This compound was obtained as a solid: mp >300 °C. ^{1}H NMR (400MHz, DMSO-d_6): δ 9.59 (s, 1H), 7.83 (s, 1H), 7.59 (d, J=8.4Hz, 1H), 7.41(s, 1H), 7.11 (d, J=8.4Hz, 1H), 5.52 (s, 1H, CH), 1.82-1.93 (m, 2H), 2.17-2.28 (m, 2H) 2.35-2.48 (m, 2H). IR (KBr): 3283, 3258, 3065, 2962, 1706, 1676, 1617, 1442, 1371, 1243, 1189, 1132, 1021, 946, 758 cm^{-1}.

4-(3-Nitrophenyl)-1,2,3,4,5,6,7,8-octahydro-2,5-oxo-quinoxalin (**6d**).

This compound was obtained as a solid: mp 268.1-268.4 °C. ^{1}H NMR (400MHz, DMSO-d_6): δ 9.64 (s, 1H, NH), 7.92 (s, 1H, NH), 7.52-7.71 (m, 4H), 5.34 (s, 1H), 1.82-1.93 (m, 2H), 2.17-2.28 (m, 2H) 2.35-2.48 (m, 2H). IR (KBr): 3341, 3256, 3094, 2951, 2885, 1710, 1667, 1614, 1525, 1471, 1354, 1241, 1184, 1131, 954, 899, 811, 721, 679, 538, 487 cm^{-1}.

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REFERENCE AND NOTES

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