TMSCl-catalysed condensation of α -diketone compounds with urea/thiourea derivatives under solvent-free conditions

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Abstract. An efficient, rapid and green synthesis of glycoluril, imidazolidine-2-one, imidazole-2-one and imidazole-2-thiol derivatives have been accomplished by the reaction of urea/thiourea derivatives and α -diketone at 100°C in the presence of trimethylsilyl chloride (TMSCl) as Lewis acid catalyst under solvent-free conditions. This approach offers many advantages such as good product yields, various products, short reaction time, easy isolation of products, facile purification and environmentally benign reaction conditions.

Keywords. TMSCl; solvent-free; green chemistry; glycoluril; imidazolidine-2-one; imidazole-2-thiol.

1. Introduction

The development of the chemistry of N-heterocycles based on urea or thiourea such as glycolurils, imidazolidine-2-ones, imidazole-2-ones, imidazolidine-2-thiones and imidazole-2-thiones is of great interest due to high physiological activity of these materials.^{1,2}

Glycoluril as the simplest member of this class of heterobicyclic compounds was synthesized for the first time in the 19th century.³ Glycoluril and its derivatives are the subject of many studies because of their synthetic accessibility, hydrogen-bond donating/accepting ureidyl functionality, curved and rigid structure.^{4,5} Mebicar (2,4,6,8-tetramethyl glycoluril) as one of representatives of this class is used in medical practice as a day tranquillizer.⁶ The glycolurils have been received a great deal of attention due to their practical applications such as polymer crosslinking, fertilizers, crystal engineering, explosives, stabilizer of organic compounds against photodegradation, radio iodination agents for biomolecules, combinatorial chemistry, catalysts, bleaching activators and as monomer in supramolecular chemistry.^{7,8} In this study, by condensation reaction of α -diketone with urea under acidic conditions, glycoluril was prepared as the only product, while under alkaline conditions hydantoin and glycoluril were obtained as the major and minor products, respectively.⁴

Imidazolidine-2-ones, imidazole-2-ones, imidazole-2-thiones and imidazolidine-2-thiones are important

heterocycles with an embedded cyclic urea/thiourea scaffold, which is a core element of many pharmaceutically relevant compounds.⁹ They can exhibit a wide range of biological activities including inhibitions of the respiratory syncytial virus (RSV) fusion and NNRT (the nonnucleoside reverse transcriptase).¹⁰ Furthermore, these materials play important roles as progesterone receptor antagonists in the selective inhibition of farnesyltransferase (FTase) and the activation of K⁺ channels.¹¹ Among imidazolidine-2-ones, diphenin is known as antiepileptic drug.¹²

A number of methods for the synthesis of these materials have been reported.^{13–23} However, some of these pathways suffer from various drawbacks such as tedious work-up, unsatisfactory yields, refluxing for long periods with high boiling solvents. The aim of this work is to synthesize some glycoluril derivatives, imidazolidine-2-ones, imidazole-2-ones and imidazole-2-thiol in the presence of TMSCl (trimethylsilyl chloride) under solvent-free conditions.

2. Experimental

IR spectra were prepared on a galaxy series FT-IR 5000 spectrophotometer using KBr discs. ¹H NMR and ¹³C NMR spectra were obtained in DMSO-*d*₆ using Bruker spectrophotometer (300 MHz). Chemical shifts were expressed in (ppm) downfield from TMS. Melting points were determined using an electrothermal digital apparatus and are uncorrected. All chemical including urea/thiourea derivatives, benzil, trimethylsilyl chloride

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(known as TMSCl), (2-hydroxyethyl) trimethylammonium chloride (known as choline chloride) and solvents were commercially available and purchased from Merck or Fluka company and used without further purification. Water and other solvents were distilled before use.

2.1 General procedure for the synthesis of glycoluril, imidazolidine-2-one, imidazole-2-one, and imidazole-2-thiol derivatives

A mixture of α -diketone (1 mmol) and one of the urea or thiourea derivatives (1.5 mmol) in TMSCl (10 mol %) was added into a test tube with a magnetic stirring bar. The test tube was heated in an oil bath at 100°C for 30 min and then was slowly cooled to room temperature and a mixture of hot EtOH-H₂O (5:5 mL) added slowly and filtered off to extract the product. For most of the reactions purification was not necessary and the products were analysed by ¹H-NMR spectroscopy and melting point. However, appropriate recrystallization in diethyl ether was used for further purification. All compounds were characterized on the basis of their spectroscopic data (IR, NMR) and melting point by comparison with those reported in the literature.^{11,12,17,19}

2.1a Tetrahydro-3a,6a-diphenylimidazo[4,5-d]imidazole-2,5-(1H,3H)-dione (table 2, entry 1): Yield 0.21 g (72%), m.p.>300°C, IR (KBr, cm⁻¹): 1687, 1710, 2847, 3070, 3230; ¹H NMR (300 MHz, DMSO-*d*₆): δ 7.05–7.07 (10H, m, Ar-H), 7.74 (4H, brs, NH, D₂O-exchangable); ¹³C NMR (75 MHz, DMSO-*d*₆): δ 82.2, 127.4, 127.8, 128.2, 138.7, 161.2 (C=O); *Anal.* Calcd for C₁₆H₁₄N₄O₂: C, 65.30; H, 4.79; N, 19.04. Found: C, 65.17; H, 4.86; N, 18.87.

2.1b 5-Hydroxy-1-methyl-4,5-diphenyl-1H-imidazole-2(5H)-one (table 2, entry 2): Yield 0.20 g (78%), m.p.>300°C, IR (KBr, cm⁻¹): 1691, 1726, 2883, 2953, 3092, 3203 (OH); ¹H NMR (300 MHz, DMSO-*d*₆): δ 2.52 (3H, s, CH₃), 6.76–7.12 (10H, m, Ar-H), 8.46 (1H, brs, OH, D₂O-Exchangable); ¹³C NMR (75 MHz, DMSO-*d*₆): δ 25.9, 83.6, 127.7, 128.2, 128.3, 128.6, 128.7, 135.5, 159.5, 160.1; *Anal.* Calcd for C₁₆H₁₄N₂O₂: C, 72.16; H, 5.30; N, 10.52. Found: C, 72.02; H, 5.41; N, 10.46.

2.1c 4,5-Dihydroxy-1,3-dimethyl-4,5-diphenylimidazolidine-2-one (table 2, entry 3): Yield 0.24 g (83%), m.p: 220–222°C, IR (KBr, cm⁻¹): 1664, 2793, 2928, 2989, 3171, 3420; ¹H NMR (300 MHz, DMSO-*d*₆):

δ 2.56 (6H, s, 2 $\times$ CH₃), 6.58 (2H, brs, OH, D₂O-exchangable), 6.89–7.01 (10H, m, Ar-H); ¹³C NMR (75 MHz, DMSO-*d*₆): δ 26.0, 92.8, 127.3, 127.7, 127.8, 138.0, 160.1; *Anal.* Calcd for C₁₇H₁₈N₂O₃: C, 68.44; H, 6.08; N, 9.39. Found: C, 68.56; H, 5.97; N, 9.47.

2.1d 5-Hydroxy-1,4,5-triphenyl-1H-imidazole-2(5H)-one (table 2, entry 4): Yield 0.24 g (75%), m.p.>300°C, IR (KBr, cm⁻¹): 1691, 1724, 3041, 3063, 3340; ¹H NMR (300 MHz, DMSO-*d*₆): δ 6.86–7.26 (15H, m, Ar-H), 8.81 (1H, brs, OH, D₂O-Exchangable); ¹³C NMR (75 MHz, DMSO-*d*₆): δ 104.1, 125.4, 127.0, 127.7, 127.9, 128.4, 129.0, 129.3, 132.3, 135.4, 137.2, 159.1, 159.8; *Anal.* Calcd for C₂₁H₁₆N₂O₂: C, 76.81; H, 4.91; N, 8.53. Found: C, 76.68; H, 4.80; N, 8.70.

2.1e 1-Benzyl-5-hydroxy-4,5-diphenyl-1H-imidazole-2(5H)-one (table 2, entry 5): Yield 0.26 g (76%), m.p: 283–285°C, IR (KBr, cm⁻¹): 1689, 1714, 2872, 2926, 3065, 3205; ¹H NMR (300 MHz, DMSO-*d*₆): δ 3.89 (1H, dd, *J* = 16.7 Hz, CH₂), 4.37 (1H, dd, *J* = 16.7 Hz, CH₂), 6.66–7.32 (15H, m, Ar-H), 8.42 (1H, brs, OH, D₂O-Exchangable); ¹³C NMR (75 MHz, DMSO-*d*₆): δ 45.1, 90.6, 126.9, 127.5, 128.0, 128.3, 128.4, 128.6, 129.1, 133.3, 137.5, 139.4, 159.4, 160.6; *Anal.* Calcd for C₂₂H₁₈N₂O₂: C, 77.17; H, 5.30; N, 8.18. Found: C, 77.30; H, 5.20; N, 8.10.

2.1f 4,5-Diphenyl-1H-imidazole-2-thiol (table 2, entry 6): Yield 0.17 g (68%), m.p.>300°C, IR (KBr, cm⁻¹): 1209, 1498, 1674, 2748, 3013, 3161; ¹H NMR (300 MHz, DMSO-*d*₆): δ 7.33–7.35 (10H, m, Ar-H), 12.54 (2H, brs, NH, SH (Tautomerization), D₂O-Exchangable); ¹³C NMR (75 MHz, DMSO-*d*₆): δ 125.2, 127.9, 128.6, 128.8, 129.2, 160.4; *Anal.* Calcd for C₁₅H₁₂N₂S: C, 71.40; H, 4.79; N, 11.10; S, 12.71. Found: C, 71.31; H, 4.85; N, 11.01; S, 12.80.

2.1g 1-Methyl-4,5-diphenyl-1H-imidazole-2-thiol (table 2, entry 7): Yield 0.16 g (63%), m.p:194–196°C, IR (KBr, cm⁻¹): 1273, 1493, 1599, 2752, 2910, 2937, 3049; ¹H NMR (300 MHz, DMSO-*d*₆): δ 3.29 (3H, s, CH₃), 7.23–7.48 (10H, m, Ar-H), 12.82 (1H, brs, SH, D₂O-Exchangable); ¹³C NMR (75 MHz, DMSO-*d*₆): δ 32.2, 124.6, 126.8, 127.3, 128.0, 128.6, 128.9, 129.2, 129.6, 129.8, 131.1, 161.9; *Anal.* Calcd for C₁₆H₁₄N₂S: C, 72.15; H, 5.30; N, 10.52; S, 12.04. Found: C, 72.01; H, 5.39; N, 10.48; S, 12.15; MS (*m/z*, %): 266 (M⁺), 165, 136, 118, 97, 83, 69, 57(100).

2.1h *Tetrahydroimidazo[4,5-d]imidazole-2,5(1H,3H)-dione* (table 2, entry 9): Yield 0.10 g (70%), m.p>300°C, IR (KBr, cm^{-1}): 1701, 1763, 2843, 3074, 3223; ^1H NMR (300 MHz, $\text{DMSO}-d_6$): δ 5.23 (2H, s, $2 \times \text{CH}$), 7.18 (4H, brs, NH, D_2O -Exchangable); ^{13}C NMR (75 MHz, $\text{DMSO}-d_6$): δ 65.0, 161.7; *Anal.* Calcd for $\text{C}_4\text{H}_6\text{N}_4\text{O}_2$: C, 33.81; H, 4.26; N, 39.42. Found: C, 33.94; H, 4.12; N, 39.52.

2.1i *5-Hydroxy-1-phenyl-1H-imidazole-2(5H)-one* (table 2, entry 10): Yield 0.12 g (72%), m.p>300°C, IR (KBr, cm^{-1}): 1705, 1730, 2874, 3103, 3211; ^1H NMR (300 MHz, $\text{DMSO}-d_6$): δ 6.05 (1H, s, CH), 7.08–7.58 (6H, m, Ar-H), 8.53 (1H, brs, OH, D_2O -Exchangable); ^{13}C NMR (75 MHz, $\text{DMSO}-d_6$): δ 65.6, 119.6, 123.5, 129.2, 138.4, 157.2; *Anal.* Calcd for $\text{C}_9\text{H}_8\text{N}_2\text{O}_2$: C, 61.36; H, 4.58; N, 15.90. Found: C, 61.47; H, 4.51; N, 16.01.

2.1j *1-Benzyl-5-hydroxy-1H-imidazole-2(5H)-one* (table 2, entry 11): Yield 0.14 g (77%), m.p>300°C, IR (KBr, cm^{-1}): 1695, 1700, 2856, 2941, 3032, 3115, 3221; ^1H NMR (300 MHz, $\text{DMSO}-d_6$): δ 3.99 (1H, dd, $J = 15.5$ Hz, CH_2), 4.55 (1H, dd, $J = 15.5$ Hz, CH_2), 5.04 (1H, s, CH), 7.25–7.36 (6H, m, Ar-H), 7.81 (1H, brs, OH, D_2O -Exchangable); ^{13}C NMR (75 MHz, $\text{DMSO}-d_6$): δ 43.6, 65.3, 127.6, 128.1, 128.9, 137.8, 159.3, 160.4; *Anal.* Calcd for $\text{C}_{10}\text{H}_{10}\text{N}_2\text{O}_2$: C, 63.15; H, 5.30; N, 14.73. Found: C, 63.28; H, 5.22; N, 14.86.

2.1k *Tetrahydro-3a-methyl-6a-propylimidazo[4,5-d]imidazole-2,5(1H,3H)-dione* (table 2, entry 12): Yield 0.12 g (68%), m.p>300°C, IR (KBr, cm^{-1}): 1680, 1724, 2887, 2980, 3250; ^1H NMR (300 MHz, $\text{DMSO}-d_6$): δ 0.93 (3H, t, $J = 7.1$ Hz, CH_3), 1.32 (3H, s, CH_3), 1.62 (2H, q, $J = 7.1$ Hz, CH_2), 7.09 (2H, brs, NH, D_2O -Exchangable), 7.20 (2H, brs, NH, D_2O -Exchangable); ^{13}C NMR (75 MHz, $\text{DMSO}-d_6$): δ 8.0, 21.9, 28.5, 75.9, 78.2, 160.1; *Anal.* Calcd for $\text{C}_8\text{H}_{14}\text{N}_4\text{O}_2$: C, 48.47; H, 7.12; N, 28.26. Found: C, 48.61; H, 7.00; N, 28.42.

2.1l *1H-phenanthro [9, 10-d] imidazole-2-thiol* (table 2, entry 13): Yield 0.16 g (64%), m.p>300°C, IR (KBr, cm^{-1}): 1195, 1502, 1620, 2759, 2946, 3165; *Anal.* Calcd for $\text{C}_{15}\text{H}_{10}\text{N}_2\text{S}$: C, 71.97; H, 4.03; N, 11.19; S, 12.81. Found: C, 72.24; H, 4.16; N, 10.98; S, 12.77.

2.1m *1-Methyl-1H-phenanthro [9, 10-d] imidazole-2-thiol* (table 2, entry 14): Yield 0.15 g (60%),

m.p>300°C, IR (KBr, cm^{-1}): 1116, 1487, 1537, 1612, 2752, 2945, 3078, 3138; *Anal.* Calcd for $\text{C}_{16}\text{H}_{12}\text{N}_2\text{S}$: C, 72.70; H, 4.58; N, 10.60; S, 12.13. Found: C, 72.93; H, 4.41; N, 10.46; S, 12.31.

3. Results and discussion

During the course of our study aiming at improving the ecocompatibility of certain organic processes,²⁴ we have been particularly interested in the development of organic transformations in the presence of TMSCl catalyst. Here, we report the reaction of benzil **1** and urea/thiourea derivatives for the formation of title compound at 100°C in the presence of catalytic amount of TMSCl under solvent-free conditions (scheme 1).

In order to determine the most appropriate reaction conditions, the condensation reaction of benzil **1** (1 mmol) with urea **2** (1 mmol) was carried out in different solvents and solvent-free in the presence of TMSCl. The results are shown in table 1. The best results were obtained with 10 mol% of catalyst under solvent-free conditions (SFC) at 100°C and gave 4,4'-diphenyl glycoluril **3** without formation of any by-products. When we examined this condensation under SFC between benzil and urea with molar ratio 1:1, the corresponding product yield was only 45%, again we repeated this reaction with molar ratio 1:1.5, but we observed a significant increase in the corresponding yield (72%) (table 1, entry 6). We also condensed benzil and urea with molar ratio 1:2, respectively but the yield of product was only 74%. In the case of the other α -diketone derivatives, this condensation was carried out between α -diketone compounds and urea/thiourea derivatives (table 2, entries 2–14) with molar ratio 1:2, 1:1.5 and 1:1, respectively but the yield of products was slightly differed from each other (for example, in entry 2, condensation of N-methyl urea and benzil was carried out with molar ratio 1:1, 1:1.5, 1:2 and the product of entry 2 was formed with 75%, 78% and 79% yields, respectively). So, we decided to do all reactions with molar ratio 1:1.5 between α -diketone compounds and urea/thiourea derivatives.

After optimization of reaction conditions, we examined the generality of this procedure for other substrates using α -diketone compounds and urea/thiourea derivatives (table 2). In the case of the urea derivatives such as N-methyl urea, N-benzyl urea, N,N'-dimethyl urea and N-phenyl urea with α -diketone, the major product was not a glycoluril derivative and the product was imidazolidine-2-one or imidazole-2-one (table 2, entries 2–5,10,11). So, the major product for reaction

Scheme 1. Condensation of benzil and urea with molar ratio 1:1.5.**Table 1.** Optimization of reaction conditions.

Entry	Conditions	Yield ^a (%)
1	EtOH, Reflux, TMSCl (10 mol %), 4h	48
2	THF, Reflux, TMSCl (10 mol %), 4h	0
3	CHCl ₃ , Reflux, TMSCl (10 mol %), 4h	26
4	CH ₃ CN, Reflux, TMSCl (10 mol %), 4h	0
5	SFC, 100°C, TMSCl (10 mol %), benzil:urea (1:1), 30 min	45
6	SFC, 100°C, TMSCl (10 mol %), benzil:urea (1:1.5), 30 min	72
7	SFC, 100°C, ChCl.2urea (0.3 mL), 30 min	32
8	SFC, 100°C, ChCl.2urea (0.3 mL), TMSCl, 30 min	70
9	SFC, 100°C, benzil:urea (1:1.5), Catalyst free, 3h	0

^aIsolated yields**Table 2.** Condensation of urea/thiourea derivatives and α -diketone compounds.

Entry	Urea derivatives	α -diketone	Product	Yield(%)
1	<chem>NC(=O)N</chem>	<chem>c1ccccc1C(=O)C(=O)c2ccccc2</chem>	<chem>c1ccccc1C2C(=O)NC(=O)N2c3ccccc3</chem>	72
2	<chem>NC(=O)NC</chem>	<chem>c1ccccc1C(=O)C(=O)c2ccccc2</chem>	<chem>CN1C(=O)N(C1C(=O)N2C(=O)N(C2)c3ccccc3)c4ccccc4</chem>	78
3	<chem>CN(C)C(=O)NC</chem>	<chem>c1ccccc1C(=O)C(=O)c2ccccc2</chem>	<chem>CN1C(=O)N(C1C(=O)N(C2C(=O)N(C2)c3ccccc3)c4ccccc4)C(O)C5C(=O)N(C5)c6ccccc6</chem>	83
4	<chem>NC(=O)Nc1ccccc1</chem>	<chem>c1ccccc1C(=O)C(=O)c2ccccc2</chem>	<chem>c1ccccc1C2C(=O)N(C2C(=O)N3C(=O)N(C3)c4ccccc4)c5ccccc5</chem>	75

Table 2. (continued).

Entry	Urea derivatives	α -diketone	Product	Yield(%)
5				76
6				68
7				63
8			No reaction	0
9				70
10				72
11				77
12				68

Table 2. (continued).

Entry	Urea derivatives	α -diketone	Product	Yield(%)
13				64
14				60

of thiourea and N-methyl thiourea with α -diketone was imidazole-2-thiol (table 2, entries 6,7,13,14), and in the case of the N,N'-diphenyl thiourea condensation with benzil did not observe any reaction (table 2, entry 8).

Also, we have carried out this reaction in the presence of ChCl₂urea ionic liquid as one of deep eutectic solvents (DESs) at 100°C with the same molar ratio but product yield was only 32% (table 1, entry 7), again, reaction was carried out in the presence of both TMSCl and ChCl₂urea (table 1, entry 8), we observed that product yield was increased to 70%. From this, we concluded that in addition to added urea to benzil, urea in ChCl₂urea ionic liquid in the presence of TMSCl reacts with benzil and this increases the efficiency of product. We carried out the condensation of benzil and other derivatives of urea such as N-methyl urea **4** and N,N'-dimethyl urea **5** under the same conditions in entry 8, but the only product was 4,4'-diphenyl glycoluril **3**. On the other hand, we found that reactivity of the urea in ChCl₂urea is more than urea derivatives (scheme 2).

On the basis of the experimental results, probable mechanism for the synthesis of title compounds is shown in scheme 3. Initially, TMSCl catalyst as

Lewis acid activates the carbonyl groups of benzil, then nucleophilic attack of the nitrogen urea or thiourea derivatives to the activated carbonyl groups of benzil produced intermediate **I**. When R, R' = H, the reaction can proceed through the *in situ* formation of unstable compound **II** by water elimination, which is further attacked by a second molecule of urea to give compound **III**. Comparing the structures of urea derivatives (when R = H, R' = Ph, PhCH₂, CH₃ or R, R' = CH₃) and of the corresponding products, it can be seen in all the cases that, these derivatives of compounds **IV**, **V** produced, which in this state due to existence steric hindrance of alkyl or aryl group in **IV**, **V**, the reaction pathway could not be further proceeded.²⁵

When thiourea was condensed with benzil, probably at first step, via water elimination, compound **VI** formed in this state, thiourea as reducing agent one of the C=N band, produced compound **VII**, finally by water elimination from **VII**, the corresponding product as thiol or thione was produced. Probably thiourea via hydrolysis converted to urea and hydrogen sulfide (H₂S), which H₂S, via reduction of C=N band produces intermediate **VII** and finally by water elimination from **VII**, aromatic stable compound **VIII** produced.²⁶

Scheme 2. Reactivity of urea in ChCl₂urea ionic liquid and its derivatives with benzil.

Scheme 3. Probable mechanism for condensation of benzil and urea or thiourea derivatives.

4. Conclusion

In summary, we have successfully developed a simple, green and highly efficient one-pot synthesis of glycoluril, imidazolidine-2-one, imidazole-2-one and imidazole-2-thiol compounds from easily available starting materials using TMSCl (trimethylsilyl chloride) as a Lewis acid catalyst under solvent-free conditions. For the first time, we have prepared each of these compounds under the same conditions. This protocol is attractive in terms of: atom economy, short reaction time, simple and clean reaction profiles, easy work-up and various products.

Supplementary information

Supplementary data (experimental details and spectroscopic characterization (^1H and ^{13}C -NMR) of the compounds) are available through the journal website (www.ias.ac.in/chemsci).

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