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Unexpected Knoevenagel self-condensation reaction of tetronic acid: synthesis of a new class of organic heterocyclic salts

Ahmad Shaabani *, Afshin Sarvary, Sajjad Keshipour, Ali Hossein Rezayan, Rahim Ghadari

Department of Chemistry, Shahid Beheshti University, PO Box 19396-4716, Tehran, Iran

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ABSTRACT

A new class of pyridinium, quinolinium, isoquinolinum, and *N*-methylimidazolium-3-(2,5-dihydro-5-oxofuran-3-yl)-4-hydroxyfuran-2(5*H*)-one salts have been prepared in high yields by reacting pyridine, quinoline, isoquinoline, *N*-methylimidazole, 1,4-diazabicyclo[2.2.2]octane, and their derivatives with tetronic acid in CH_2Cl_2.

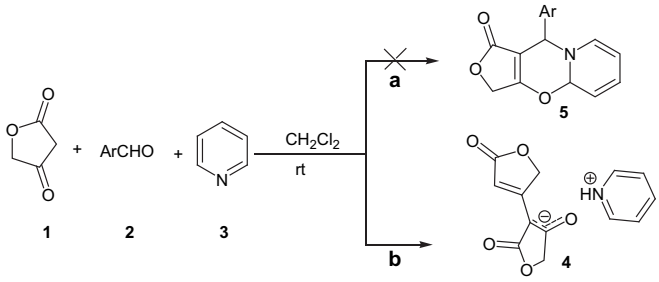
1. Introduction

Organic heterocyclic salts such as pyridinium, quinolinium, and *N*-methylimidazolium salts are an important class of new compounds, which were used as nonlinear optics, ionic liquids, and key intermediate in organic reactions.[1–3]

Multicomponent condensation reactions (MCRs), due to atom economy, simplicity, and amenability to the automated synthesis, have an advantageous position among other organic synthesis methods. The development of new MCRs is an interestingly research topic in applied sciences.[4–6]

As a part of our current studies on the multicomponent reactions,[7–9] and also chemistry of tetronic acid,[10] we have investigated the possibility of trapping the heterodiene generated from the reaction between tetronic acid **1** and aldehydes **2** with pyridine **3**. In the event, the expected MCR product **5** was not observed;[11,12] instead the reaction afforded the corresponding pyridinium-3-(2,5-dihydro-5-oxofuran-3-yl)-4-hydroxyfuran-2(5*H*)-one salt **4** (Scheme 1).

The reaction conditions are mild and the reactions are carried out in CH_2Cl_2 in high yield. A wide range of pyridines and its derivatives were reacted efficiently with tetronic acid under the same reaction conditions to give the corresponding salts (Fig. 1, **4a**–**k**). It is interesting to note that, when pyridine derivatives with electron-withdrawing groups such as –CHO **6** and –CN **7** were used, the reaction did not proceed (Scheme 2). Also, the condensation product between an aldehyde and tetronic acid did not observed.



Scheme 1. Proposed (a) and observed (b) reaction between reagents.

The reaction of tetronic acid with benzylamine **8** was also investigated. As indicated in Scheme 3, the product **9** was obtained; but in the case of 2-aminopridine **10**, product **4e** was obtained (Scheme 3). These results show that, pyridine moiety has an important role in the reaction pathway and presence of pyridine moiety is essential for reaction proceeding.

Furthermore, removal of pyridine by acidic work-up afforded 3-(2,5-dihydro-5-oxofuran-3-yl)-4-hydroxyfuran-2(5*H*)-one **11** as the product (Scheme 4). Although the product **11** (anhydrotetronic acid) is known in the older literature, there is very little characterization data.[13]

To explore the scope and limitations of this reaction, we extended our studies to the use of various amines such as quinoline, isoquinoline, *N*-methylimidazole, 1,4-diazabicyclo[2.2.2]octane (DABCO), and

* Corresponding author. Tel.: +98 2129902800; fax: +98 2122431663.
E-mail address: a-shaabani@cc.sbu.ac.ir (A. Shaabani).

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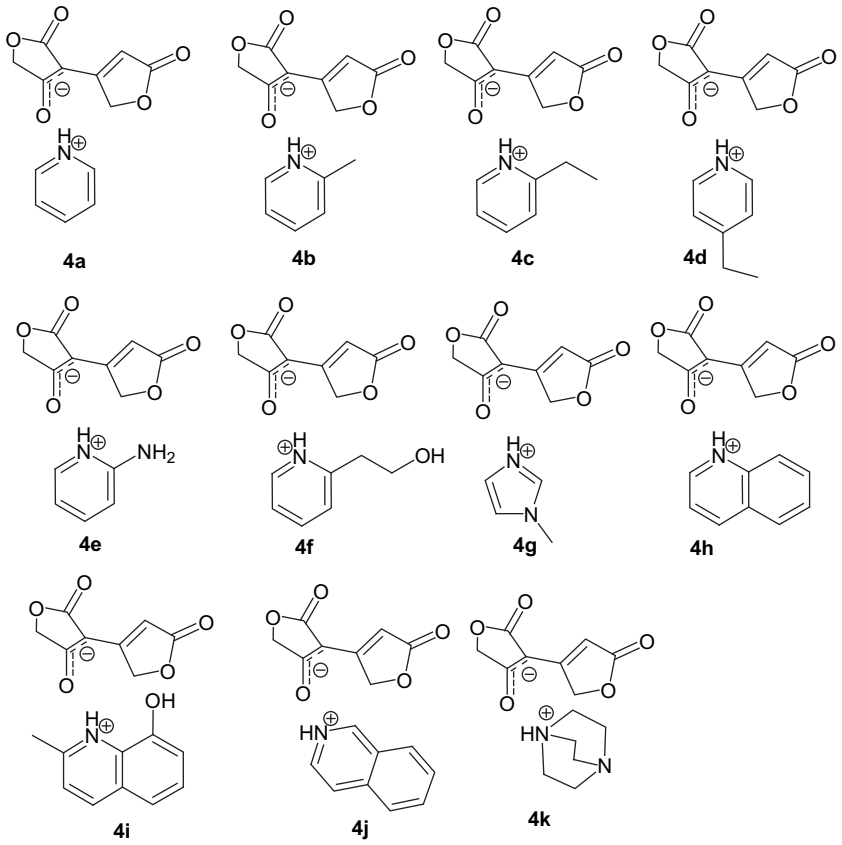
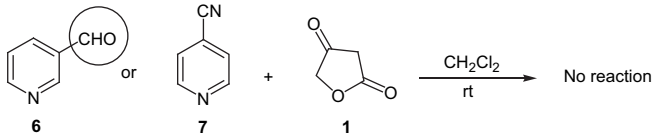
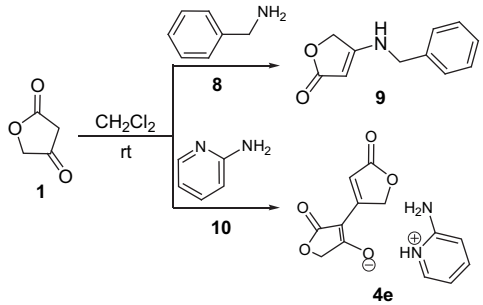


Product	4a	4b	4c	4d	4e	4f	4g	4h	4i	4j	4k
Yield(%)	80	65	62	65	96	72	56	58	70	61	62

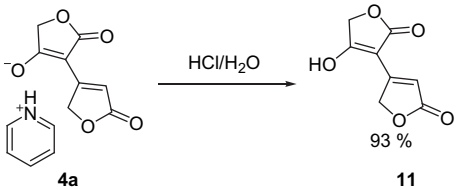
Figure 1. Synthesis of 3-(2,5-dihydro-5-oxofuran-3-yl)-4-hydroxyfuran-2(5*H*)-one salts **4a**–**k**.



Scheme 2. Effect of electron-withdrawing substitution on pyridine in the reaction.



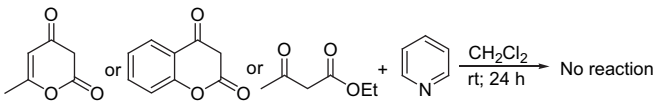
Scheme 3. Effect of the pyridine moiety in the reaction.



Scheme 4. Acidic removal of pyridine.

their derivatives. As indicated in Figure 1, the reaction was proceeded very efficiently and led to the formation of the corresponding 3-(2,5-dihydro-5-oxofuran-3-yl)-4-hydroxyfuran-2(5*H*)-one salts **4g**–**k** in excellent yields at room temperature, without any undesired byproducts.

In continue of our research, different CH-acids, which, their structures are similar to tetronic acid, have been reacted in the same reaction conditions. In these cases, no reaction has been occurred (Scheme 5).



Scheme 5. Examining different CH-acids instead of tetronic acid.

In order to obtain the best solvent, reaction between tetronic acid **1** with pyridine **3** has been investigated in water and various organic solvents (Table 1). As can be seen in Table 1, CH_2Cl_2 is the

Table 1
Effects of solvent on the reaction time and yield[a]

Entry	Solvent	Time (h)	Yield (%)
1	H_2O	12	64
2	MeOH	12	53
3	EtOH	12	30
4	CH_3CN	12	61
5	CH_2Cl_2	6	80
6	$C_6H_5CH_3$	12	15

[a] Pyridine (1.0 mmol) and tetronic acid (2.0 mmol) at room temperature.

best solvent respect to yield and short reaction time (Table 1, entry 5). In the cases of H_2O and acetonitrile the reaction times are long for the reasonable yield (Table 1, entries 1 and 4).

The structures of compounds **4a**–**k** were deduced from their IR, mass, ^{1}H NMR, and ^{13}C NMR spectra. For example, the ^{1}H NMR spectrum of **4a** exhibited two singlets identified as two methylene groups at δ=4.48 and 5.06 (2OCH_2), a singlet at δ=5.92 (CO–C*H*=C), three signals for the pyridine moiety at δ=8.08 (2H, br s), 8.60 (1H, br s), 8.92 (2H, br s) and a broad singlet at δ=9.94 for NH group. The ^{1}H-decoupled ^{13}C NMR spectrum of **4a** showed 11 distinct resonances in agreement with proposed structure. The mass spectra of these compounds displayed molecular ion peaks at the appropriate *m*/*z* values.

In conclusion, we have demonstrated a very simple and highly efficient approach to the synthesis of pyridinium, quinolinium, isoquinolinum, *N*-methylimidazolium, and DABCO-3-(2,5-dihydro-5-oxofuran-3-yl)-4-hydroxyfuran-2(5*H*)-one salts in CH_2Cl_2 at room temperature. The work-up procedure is very simple and the products do not require further purification.

2. Experimental

2.1. General

Melting points were measured on an Electrothermal 9100 apparatus and are uncorrected. Mass spectra were recorded on a FINNIGAN-MAT 8430 mass spectrometer operating at an ionization potential of 70 eV. IR spectra were recorded on a Shimadzu IR-470 spectrometer. ^{1}H and ^{13}C NMR spectra were recorded on a BRUKER DRX-300 AVANCE spectrometer at 300.13 and 75.47 MHz. NMR spectra were obtained in DMSO-d_6. The chemicals used, were purchased from Merck and Fluka Chemical Companies.

*2.1.1. Typical procedure for preparation of product (**4a**).* To a magnetically stirred solution of tetronic acid (0.20 g, 2.0 mmol) in CH_2Cl_2 (10 mL), pyridine (0.08 g, 1.0 mmol) was added. The reaction mixture was stirred for 6 h at room temperature. After completion of the reaction (monitored by TLC method), the solvent was removed under vacuum and the residue was crystallized from CH_2Cl_2/*n*-hexane (3:1) and the product **4a** was obtained as a white solid (0.21 g, 80%); mp 162–165 °C. IR (KBr) (ν_{max}, cm^{-1}): 1735, 1673, 1602. ^{1}H NMR (300 MHz, DMSO-d_6): δ_H=4.48 (2H, s, OCH_2), 5.06 (2H, s, OCH_2), 5.92 (1H, s, –CO–CH=C), 8.08 (2H, br s, pyridine protons), 8.60 (1H, br s, pyridine proton), 8.92 (2H, br s, pyridine protons), 9.94 (1H, br s, NH). ^{13}C NMR (75 MHz, DMSO-d_6): δ_C=69.2, 71.8, 89.9, 103.0, 127.7, 142.5, 146.9, 158.5, 173.2, 175.3, 187.1. MS (EI, 70 eV), *m*/*z*, (%): 182 (M^+−79, 50), 154 (50), 126 (30), 108 (40), 80 (20), 66 (100), 39 (80). Anal. Calcd for $C_{13}H_{11}NO_5$: C, 59.77; H, 4.24; N, 5.36. Found: C, 59.74; H, 4.28; N, 5.32.

*2.1.2. Product (**4b**).* White solid (0.18 g, 65%); mp 192–196 °C. IR (KBr) (ν_{max}, cm^{-1}): 2546, 2135, 2048, 1730, 1699. ^{1}H NMR (300 MHz, $CDCl_3$): δ_H=2.69 (3H, s, CH_3), 4.17 (2H, s, OCH_2), 5.00 (2H, s, OCH_2), 5.73 (1H, s, –CO–CH=C), 7.81–7.90 (2H, m, pyridine protons), 8.40–8.45 (1H, m, pyridine proton), 8.75 (1H, d, $^3J_{HH}$=5.2 Hz, pyridine proton), 9.63 (1H, br s, NH). ^{13}C NMR (75 MHz, $CDCl_3$): δ_C=20.1 (CH_3), 70.5, 71.8, 87.0, 97.8, 124.6, 127.9, 142.0, 145.9, 154.3, 160.8, 174.8, 176.3, 188.2. MS (EI, 70 eV), *m*/*z*, (%): 182 (M^+−93), 154 (80), 126 (40), 108 (50), 66 (100), 39 (60). Anal. Calcd for $C_{14}H_{13}NO_5$: C, 61.09; H, 4.76; N, 5.09. Found: C, 61.21; H, 4.58; N, 5.18.

*2.1.3. Product (**4c**).* White solid (0.18 g, 62 %); mp 163–165 °C. IR (KBr) (ν_{max}, cm^{-1}): 2540, 2150, 1727, 1698. ^{1}H NMR (300 MHz, DMSO-d_6): δ_H=1.29 (3H, t, $^3J_{HH}$=7.5 Hz, CH_2CH_3), 2.98 (2H, q, $^3J_{HH}$=7.5 Hz, CH_2CH_3), 4.18 (2H, s, OCH_2), 5.00 (2H, s, OCH_2), 5.74 (1H, s, –CO–CH=C), 7.84 (1H, t, $^3J_{HH}$=7.4 Hz, pyridine proton), 7.92 (1H, d, $^3J_{HH}$=8.0 Hz, pyridine proton), 8.45 (1H, t, $^3J_{HH}$=8.5 Hz, pyridine proton), 8.78 (1H, d, $^3J_{HH}$=5.4 Hz, pyridine proton), 9.73 (1H, br s, NH). ^{13}C NMR (75 MHz, DMSO-d_6): δ_C=13.4, 27.1, 70.5, 71.8, 87.2, 97.9, 124.7, 126.4, 142.5, 146.0, 159.0, 160.7, 174.7, 176.2, 190.9. MS (EI, 70 eV), *m*/*z*, (%): 182 (M^+−107, 30), 154 (30), 126 (20), 106 (100), 66 (50), 39 (40). Anal. Calcd for $C_{15}H_{15}NO_5$: C, 62.28; H, 5.23; N, 4.84. Found: C, 62.41; H, 5.34; N, 4.72.

*2.1.4. Product (**4d**).* White solid (0.19 g, 65%); mp 127–131 °C. IR (KBr) (ν_{max}, cm^{-1}): 2546, 2157, 1727, 1695. ^{1}H NMR (300 MHz, DMSO-d_6): δ_H=1.24 (3H, t, $^3J_{HH}$=7.5 Hz, CH_2CH_3), 2.90 (3H, q, $^3J_{HH}$=7.5 Hz, CH_2CH_3), 4.20 (2H, s, OCH_2), 5.01 (2H, s, OCH_2), 5.75 (1H, s, –CO–CH=C), 7.94 (2H, d, $^3J_{HH}$=5.1 Hz, pyridine protons), 8.80 (2H, d, $^3J_{HH}$=5.1 Hz, pyridine protons), 9.97 (1H, br s, NH). ^{13}C NMR (75 MHz, DMSO-d_6): δ_C=14.0, 28.7, 70.4, 71.8, 87.3 98.2, 126.8, 142.0, 160.6, 164.9, 174.7, 176.2, 190.8. MS (EI, 70 eV), *m*/*z*, (%):182 (M^+−107, 40), 154 (60), 126 (30), 106 (100), 66 (95), 39 (70). Anal. Calcd for $C_{15}H_{15}NO_5$: C, 62.28; H, 5.23; N, 4.84. Found: C, 62.15; H, 5.30; N, 4.69.

*2.1.5. Product (**4e**).* White solid (0.26 g, 96%); mp 167–171 °C. IR (KBr) (ν_{max}, cm^{-1}): 3342, 3188, 2613, 2048, 1976, 1712, 1664. ^{1}H NMR (300 MHz, DMSO-d_6): 4.13 (2H, s, OCH_2), 5.01 (2H, s, OCH_2), 5.72 (1H, s, –CO–CH=C), 6.85 (1H, d, $^3J_{HH}$=6.1 Hz, pyridine proton), 6.95 (1H, d, $^3J_{HH}$=8.8 Hz, pyridine proton), 7.88–7.93 (4H, m, pyridine and NH_2 protons), 9.37 (1H, br s, NH). ^{13}C NMR (75 MHz, DMSO-d_6): δ_C=70.7, 71.8, 86.8, 96.9, 112.6, 113.8, 136.6, 144.5, 154.4, 161.1, 175.0, 176.4, 191.7. MS (EI, 70 eV), *m*/*z*, (%):182 (M^+−94, 40), 154 (70), 126 (50), 108 (50), 94 (100), 66 (100), 39 (70). Anal. Calcd for $C_{13}H_{12}N_2O_5$: C, 56.52; H, 4.38; N, 10.14. Found: C, 56.64; H, 4.32; N, 10.23.

*2.1.6. Product (**4f**).* White solid (0.21 g, 72%); mp 130–134 °C. IR (KBr) (ν_{max}, cm^{-1}): 3404, 2515, 2104, 1729, 1677. ^{1}H NMR (300 MHz, DMSO-d_6): 2.44 (2H, br s, OH), 3.05 (2H, br s, CH_2CH_2OH), 3.74 (2H, br s, CH_2CH_2OH), 4.13 (2H, br s, OCH_2), 4.96 (2H, br s, OCH_2), 5.69 (1H, br s, –CO–CH=C), 7.88 (2H, br s, pyridine protons), 8.42 (1H, br s, pyridine proton), 8.74 (1H, br s, pyridine proton), 9.75 (1H, br s, NH). ^{13}C NMR (75 MHz, DMSO-d_6): δ_C=37.1, 59.9, 70.5, 71.8, 86.9, 97.8, 124.9, 127.8, 142.2, 145.8, 156.1, 160.7, 175.5, 178.2, 191.1. Anal. Calcd for $C_{15}H_{15}NO_6$: C, 59.01; H, 4.95; N, 4.59. Found: C, 58.82; H, 5.03; N, 4.68.

*2.1.7. Product (**4g**).* White solid (0.15 g, 56%); mp 163–168 °C. IR (KBr) (ν_{max}, cm^{-1}): 2449, 1976, 1730, 1709. ^{1}H NMR (300 MHz, DMSO-d_6): δ_H=3.84 (3H, s, CH_3), 4.11 (2H, s, OCH_2), 4.98 (2H, s, OCH_2), 5.70 (1H, s, –CO–CH=C), 7.64 (2H, s, imidazole protons), 9.01 (1H, s, imidazole proton). ^{13}C NMR (75 MHz, DMSO-d_6): δ_C=35.8, 70.7, 71.8, 86.7, 96.9, 120.2, 123.5, 136.2, 161.1, 175.0, 176.4, 191.7. MS (EI, 70 eV), *m*/*z*, (%):182 (M^+−82, 60), 154 (60), 126 (40), 108 (50), 82 (100), 66 (80), 39 (50). Anal. Calcd for $C_{12}H_{12}N_2O_5$: C, 54.55; H, 4.58; N, 10.60. Found: C, 54.50; H, 4.62; N, 10.76.

*2.1.8. Product (**4h**).* White solid (0.18 g, 58%); mp 192–196 °C. IR (KBr) (ν_{max}, cm^{-1}): 2438, 2140, 2033, 1735, 1709. ^{1}H NMR (300 MHz, DMSO-d_6): δ_H=4.06 (2H, s, OCH_2), 5.01 (2H, s, OCH_2), 5.80 (1H, s, –CO–CH=C), 7.88 (1H, t, $^3J_{HH}$=7.2 Hz, quinoline proton), 7.98 (1H, t, $^3J_{HH}$=4.8 Hz, quinoline proton), 8.07 (1H, t, $^3J_{HH}$=7.2 Hz, quinoline proton), 8.18 (1H, d, $^3J_{HH}$=8.0 Hz, quinoline proton), 8.28 (1H, d, $^3J_{HH}$=7.7 Hz, quinoline proton), 9.03 (1H, d, $^3J_{HH}$=8.0 Hz, quinoline proton), 9.23 (1H, d, $^3J_{HH}$=4.8 Hz, quinoline proton). ^{13}C NMR (75 MHz, DMSO-d_6): δ_C=70.1, 71.8, 88.2, 99.7, 122.4, 123.1, 128.8, 129.4, 130.3, 134.0, 140.3, 144.7, 147.1, 159.9, 174.3, 176.0, 189.8. MS (EI, 70 eV), *m*/*z*, (%): 183 (M^+−128, 20), 154 (50), 126 (30), 129 (10), 108 (50), 66 (100), 39 (100). Anal. Calcd for $C_{17}H_{13}NO_5$: C, 65.59; H, 4.21; N, 4.50. Found: C, 65.38; H, 4.34; N, 4.29.

*2.1.9. Product (**4i**).* White solid (0.24 g, 70%); mp 257–263 °C. IR (KBr) (ν_{max}, cm^{-1}): 3409, 2454, 2320, 1724, 1705. ^{1}H NMR

(300 MHz, DMSO-d_6): δ_H=2.92 (3H, s, CH_3), 4.18 (2H, s, OCH_2), 4.99 (2H, s, OCH_2), 5.73 (1H, s, –CO–CH=C), 7.37 (1H, br s, quinoline proton), 7.64 (2H, br s, quinoline protons), 7.86 (1H, d, $^3J_{HH}$=8.1 Hz, quinoline proton), 8.87 (1H, d, $^3J_{HH}$=8.2 Hz, quinoline proton), 9.59 (1H, br s, NH), 11.69 (1H, br s, OH). ^{13}C NMR (75 MHz, DMSO-d_6): δ_C=21.3, 70.5, 71.8, 87.7, 97.9, 116.1, 118.9, 124.6, 124.8, 128.3, 129.7, 144.9, 148.6, 158.1, 160.6, 174.7, 176.2, 190.9. Anal. Calcd for $C_{18}H_{15}NO_6$: C, 63.34; H, 4.43; N, 4.10. Found: C, 63.31; H, 4.40; N, 4.13.

*2.1.10. Product (**4j**).* White solid (0.19 g, 61%); mp 192–196 °C. IR (KBr) (ν_{max}, cm^{-1}): 2529, 2164, 1725, 1707. ^{1}H NMR (300 MHz, DMSO-d_6): δ_H=4.18 (2H, s, OCH_2), 4.97 (2H, s, OCH_2), 5.73 (1H, s, –CO–CH=C), 7.95–8.62 (6H, m, isoquinoline protons), 9.78 (1H, br s, isoquinoline proton), 12.45 (1H, br s, NH). ^{13}C NMR (75 MHz, DMSO-d_6): δ_C=70.3, 71.8, 87.5, 98.4, 124.9, 127.6, 127.7, 130.5, 130.7, 133.7, 136.7, 138.6, 148.6, 160.4, 174.6, 176.1, 190.6. Anal. Calcd for $C_{17}H_{13}NO_5$: C, 65.59; H, 4.21; N, 4.50. Found: C, 65.43; H, 4.15; N, 4.41.

*2.1.11. Product (**4k**).* White solid (0.18 g, 62%); mp 185–188 °C. IR (KBr) (ν_{max}, cm^{-1}): 2452, 1975, 1725, 1706. ^{1}H NMR (300 MHz, DMSO-d_6): δ_H=2.52 (6H, br s, $3CH_2N$), 2.80–2.82 (6H, m, $3CH_2N^+$), 4.45 (2H, s, OCH_2), 4.75 (2H, s, OCH_2), 5.16 (1H, s, –CO–CH=C), 10.08 (1H, br s, NH). ^{13}C NMR (75 MHz, DMSO-d_6): δ_C=44.8, 51.2, 70.7, 71.8, 86.7, 96.8, 161.2, 175.0, 176.5, 191.8. Anal. Calcd for $C_{14}H_{18}N_2O_5$: C, 57.13; H, 6.16; N, 9.52. Found: C, 56.92; H, 6.08; N, 9.67.

*2.1.12. Product (**10**).* White solid (0.18 g, 95%); mp 128–132 °C. IR (KBr) (ν_{max}, cm^{-1}): 3426, 1727, 1695. ^{1}H NMR (300 MHz, DMSO-d_6): δ_H=4.34 (2H, s, CH_2), 4.52 (2H, br s, OCH_2), 5.64 (1H, br s, –CO–CH=C), 7.72 (2H, br s, aromatic protons), 8.10 (1H, br s, aromatic proton), 8.52 (2H, br s, aromatic protons), 12.94 (1H, br s, NH). ^{13}C NMR (75 MHz, DMSO-d_6): δ_C=55.3, 68.3, 96.5, 126.6, 141.8, 142.2, 143.4, 176.5, 181.6. Anal. Calcd for $C_{11}H_{11}NO_2$: C, 69.83; H, 5.86; N, 7.40. Found: C, 69.80; H, 5.93; N, 7.31.

*2.1.13. Typical procedure for preparation of product (**11**).* Product **4a** was washed with 5 mL concentrated HCl; then it was washed with H_2O (2×5 mL). Desired product was obtained as a white solid (0.17 g, 93%). Mp 245–247 °C. IR (KBr) (ν_{max}, cm^{-1}): 3350, 1727, 1695. ^{1}H NMR (300 MHz, DMSO-d_6): δ_H=4.16 (2H, s, OCH_2), 4.55 (2H, s, OCH_2), 5.52 (1H, s, –CO–CH=C), 8.70 (1H, br s, OH) . ^{13}C NMR (75 MHz, DMSO-d_6): δ_C=68.4, 71.8, 91.8, 106.7, 156.9, 172.2, 174.8, 184.4. Anal. Calcd for $C_8H_6O_5$: C, 52.76; H, 3.32. Found: C, 52.60; H, 3.28.

Acknowledgements

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