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Preparation of 3,4,5-substituted furan-2(5H)-ones using aluminum hydrogen sulfate as an efficient catalyst

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ABSTRACT

Various derivatives of 3,4,5-substituted furan-2(5*H*)-ones have been readily prepared by using aluminum hydrogen sulfate [$Al(HSO_4)_3$] as an efficient catalyst in good yields and milder reaction conditions. The versatility of this protocol has been demonstrated with various substituted furan-2(5*H*)-ones.

1. Introduction

Multi-component reactions (MCRs) have gained much attention due to their ability in facile execution and productivity [1]. MCRs based on the use of acetylenic esters as starting material has gained much importance in organic synthesis, partly because of the diverse types of clinical and pharmacological activity associated with the products of this reaction [2–6]. Of all kinds of MCRs based on the use of acetylenic esters, methods to synthesize furan derivatives were considered as the most versatile ones for chemical construction of poly-substituted furans [7–11].

Substituted furan derivatives are fundamentally important heterocyclic molecules and are present in many natural and medicinal structures [12,13]. They can be used as valuable intermediates for the construction of heterocycles in organic synthesis. Thus, efforts for the synthesis of furan scaffolds are in demand by organic chemists. Among the various furan derivatives, butenolides have appeared in the literature as interesting components for the construction of natural and pharmacological compounds. These skeletons show a wide range of biological activities such as antimicrobial [14], antifungal [15], anti-inflammatory [16], anticancer [17] and anti-viral HIV-1 [18] activities. Due to this wide range of abundance and applicability, various approaches toward substituted butenolides have been developed, which involve the use of organo-lithium [19], boronic acids [20,21], transition-metal catalysts such as $Pd(OAc)_2$ [22], Ru [23], Cu(II) [24], AuCl [25], and secondary amines [26]. However, many of these methods involve the use of expensive catalysts and hazardous reagents in stoichiometric amounts.

A new route to the synthesis of furan skeletons was developed by Murthy et al. *via* the multi-component reaction of aromatic amines, aldehydes and acetylenic esters, which lead to the preparation of 3,4,5-substituted furan-2(5*H*)-one derivatives using β-cyclodextrin as a catalyst in water [27]. Recently, Nagarapu et al. reported that $SnCl_2$ can efficiently catalyze this reaction [28].

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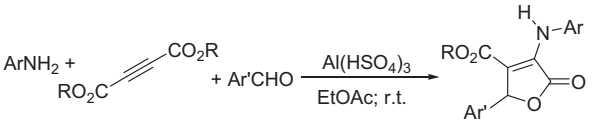

Scheme 1. Preparation of 3,4,5-substituted furan-2(5*H*)-one derivatives.

However, as the existing literature reports that the reaction performance is somewhat vitiated by its time-consuming aspects, the development of a new, efficient and green approach for the preparation of substituted furan-2(5*H*)-ones is highly desirable. In view of the above and as a part of our ongoing program on multi-component reactions [29], an efficient and convenient synthesis of 3,4,5-substituted furan-2(5*H*)-one derivatives has been accomplished by a multi-component reaction between aromatic amines, aldehydes and acetylenic esters, using $Al(HSO_4)_3$ as an efficient catalyst, with good yields (Scheme 1).

2. Experimental

2.1. Reagents and instrumentation

All reagents were purchased from Merck and Aldrich and used without further purification. All yields refer to isolated products after purification. The NMR spectra were recorded on a Bruker Avance DPX 400 MHz instrument. The spectra were measured in $CDCl_3$ relative to TMS (0.00 ppm). IR spectra were recorded on a PerkinElmer 781 spectrophotometer. Elemental analysis was performed on a Heraeus CHN-O-Rapid analyzer. Melting points were determined in open capillaries with a Buchi B-510 melting point apparatus. TLC was performed on Polygram SIL G/UV 254 silica gel plates.

2.2. General procedure

To a mixture of aldehyde (1 mmol), aromatic amine (1 mmol) and acetylenic esters (1 mmol) in ethyl acetate (5 mL), $Al(HSO_4)_3$ (0.05 g) was added as the catalyst, and the mixture was stirred for an appropriate time at room temperature. The progress of the reaction was monitored by TLC. Upon completion, the solvent was concentrated and the reaction mixture was diluted in CH_2Cl_2; the catalyst was isolated by simple filtration, and the crude product was washed with diethyl ether to afford the pure product.

2.3. Selected data

2.3.1. Methyl 4-(p-*tolylamino*)-2,5-dihydro-5-oxo-2-*phenylfuran*-3-carboxylate (**1a**)

^{1}H NMR (400 MHz, $CDCl_3$): 2.25 (s, 3H), 3.81 (s, 3H), 5.69 (s, 1H), 7.06 (d, J = 8.1 Hz, 2H), 7.14-7.38 (m, 7H), 8.93 (s, 1H, NH) ppm; IR (KBr): 3226, 2951, 1706, 1679, 1514, 1466, 1378, 1305, 1235, 1202, 1139, 998, 829, 811, 772 cm^{-1}; found: C, 70.69; H, 5.38; N, 4.39 $C_{19}H_{17}NO_4$; requires: C, 70.58; H, 5.30; N, 4.33%.

2.3.2. Methyl 4-(p-*tolylamino*)-2,5-dihydro-5-oxo-2-p-*tolylfuran*-3-carboxylate (**3a**)

^{1}H NMR (400 MHz, $CDCl_3$): 2.28 (s, 3H), 2.54 (s, 3H), 3.77 (s, 3H), 5.71 (s, 1H), 7.07 (d, J = 8.3 Hz, 2H), 7.15 (d, J = 8.3 Hz, 2H), 7.29 (d, J = 8.3 Hz, 2H), 7.39 (d, J = 8.3 Hz, 2H), 8.89 (s, 1H, NH) ppm; ^{13}C NMR (100 MHz, $CDCl_3$) δ: 20.7, 34.2, 51.8, 61.5, 112.9, 122.3, 125.6, 126.7, 129.7, 131.8, 133.5, 135.6, 151.3, 156.1, 162.8, 165.3 ppm IR (KBr): 3222, 2954, 1682, 1613, 1515, 1463, 1309, 1255, 1137, 1032, 994, 896, 848, 747 cm^{-1}; found: C, 71.27; H, 5.77; N, 4.21 $C_{20}H_{19}NO_4$; requires: C, 71.20; H, 5.68; N, 4.15%.

2.3.3. Ethyl 4-(p-*tolylamino*)-2,5-dihydro-5-oxo-2-p-*tolylfuran*-3-carboxylate (**4a**)

^{1}H NMR (400 MHz, $CDCl_3$): 1.26 (t, J = 6.8 Hz, 3H), 2.28 (s, 3H), 2.54 (s, 3H), 4.08 (q, J = 6.8 Hz, 2H), 5.72 (s, 1H), 7.08 (d, J = 8.2 Hz, 2H), 7.14 (d, J = 8.2 Hz, 2H), 7.30 (d, J = 8.2 Hz, 2H), 7.41 (d, J = 8.3 Hz, 2H), 8.89 (s, 1H, NH) ppm; ^{13}C NMR (100 MHz, $CDCl_3$) δ: 14.3, 20.6, 34.1, 51.6, 61.6, 112.9, 122.4, 125.6, 126.8, 129.6, 131.9, 133.5, 135.6, 151.2, 156.1, 162.6, 165.1 ppm IR (KBr): 3224, 3026, 2951, 1703, 1681, 1615, 1511, 1461, 1310, 1254, 1138, 1030, 994, 848, 745 cm^{-1}; found: C, 71.88; H, 6.09; N, 4.05 $C_{21}H_{21}NO_4$; requires: C, 71.78; H, 6.02; N, 3.99%.

2.3.4. Methyl 4-(p-*tolylamino*)-2-(4-*chlorophenyl*)-2,5-dihydro-5-oxofuran-3-carboxylate (**5a**)

^{1}H NMR (400 MHz, $CDCl_3$): 2.29 (s, 3H), 3.88 (s, 3H), 5.73 (s, 1H), 7.08 (d, J = 8.2 Hz, 2H), 7.16 (d, J = 8.2 Hz, 2H), 7.35 (d, J = 8.5 Hz, 2H), 7.51 (d, J = 8.5 Hz, 2H), 9.02 (s, 1H, NH) ppm; ^{13}C NMR (100 MHz, $CDCl_3$) δ: 20.3, 51.6, 60.8, 113.0, 122.9, 125.9, 126.8, 128.8, 129.2, 131.4, 134.9, 143.3, 156.1, 162.5, 165.4 IR (KBr): 3218, 2952, 1715, 1684, 1596, 1496, 1456, 1370, 1282, 1232, 1197, 1132, 1092, 1011, 928, 831, 7610 cm^{-1}; found: C, 63.89; H, 4.63; N, 4.01 $C_{19}H_{16}ClNO_4$; requires: C, 63.78; H, 4.51; N, 3.91%.

2.3.5. Methyl 4-(p-*tolylamino*)-2-(4-tert-*butylphenyl*)-2,5-dihydro-5-oxofuran-3-carboxylate (**6a**)

^{1}H NMR (400 MHz, $CDCl_3$): 1.27 (s, 9H), 2.27 (s, 3H), 3.76 (s, 3H), 5.70 (s, 1H), 7.09 (d, J = 8.4 Hz, 2H), 7.14 (d, J = 8.4 Hz, 2H), 7.27 (d, J = 8.4 Hz, 2H), 7.37 (d, J = 8.4 Hz, 2H), 8.92 (s, 1H, NH) ppm; ^{13}C NMR (100 MHz, $CDCl_3$) δ: 20.8, 31.1, 34.4, 51.9, 61.2, 112.7, 122.1, 125.4, 126.8, 129.4, 131.7, 133.6, 135.4, 151.2, 155.9, 162.7, 165.2 ppm IR (KBr): 3223, 2951, 1710, 1675, 1511, 1467, 1375, 1305, 1210, 1139, 825, 771 cm^{-1}; found: C, 72.89; H, 6.71; N, 3.75 $C_{23}H_{25}NO_4$; requires: C, 72.80; H, 6.64; N, 3.69%.

2.3.6. Ethyl 4-(p-tolylamino)-2-(4-tert-butylphenyl)-2,5-dihydro-5-oxofuran-3-carboxylate (**7a**)

^{1}H NMR (400 MHz, $CDCl_3$): 1.25-1.29 (m, 12H), 2.28 (s, 3H), 4.05 (q, J = 6.7 Hz, 2H), 5.70 (s, 1H), 7.08 (d, J = 8.3 Hz, 2H), 7.13 (d, J = 8.3 Hz, 2H), 7.27 (d, J = 8.3 Hz, 2H), 7.38 (d, J = 8.3 Hz, 2H), 8.91 (s, 1H, NH) ppm; ^{13}C NMR (100 MHz, $CDCl_3$) δ: 14.5, 20.8, 31.2, 34.5, 51.8, 61.5, 112.7, 122.3, 125.4, 126.7, 129.5, 131.9, 133.7, 135.4, 151.5, 156.2, 162.6, 165.4 ppm IR (KBr): 3221, 3024, 2950, 1709, 1675, 1512, 1375, 1212, 1139, 826, 771 cm^{-1}; found: C, 73.35; H, 6.99; N, 3.64 $C_{24}H_{27}NO_4$; requires: C, 73.26; H, 6.92; N, 3.56%.

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*2.3.7. Methyl 4-(4-chlorophenylamino)-2,5-dihydro-5-oxo-2-*p*-tolylfuran-3-carboxylate (**8a**)*

^{1}H NMR (400 MHz, $CDCl_3$): 2.55 (s, 3H), 3.83 (s, 3H), 5.75 (s, 1H), 7.07 (d, J = 8.0 Hz, 2H), 7.17 (d, J = 8.0 Hz, 2H), 7.32 (d, J = 8.2 Hz, 2H), 7.45 (d, J = 8.2 Hz, 2H), 8.98 (s, 1H, NH) ppm; ^{13}C NMR (100 MHz, $CDCl_3$) δ: 20.4, 61.5, 51.6, 112.9, 123.1, 126.8, 128.4, 128.8, 129.2, 131.4, 134.8, 134.9, 156.3, 162.6, 165.4 IR (KBr): 3219, 2952, 1714, 1684, 1595, 1496, 1456, 1426, 1371, 1338, 1283, 1232, 1197, 1132, 1092, 1011, 928, 831, 805, 760, 711, 699 cm^{-1}; found: C, 63.89; H, 4.60; N, 3.99 $C_{19}H_{16}ClNO_4$; requires: C, 63.78; H, 4.51; N, 3.91%.

*2.3.8. Methyl 4-(4-methoxyphenylamino)-2,5-dihydro-5-oxo-2-*p*-tolylfuran-3-carboxylate (**10a**)*

^{1}H NMR (400 MHz, $CDCl_3$): 2.53 (s, 3H), 3.80 (s, 3H), 5.73 (s, 1H), 6.88 (d, J = 8.8 Hz, 2H), 7.05-7.09 (m, 4H), 7.31 (d, J = 8.8 Hz, 2H), 8.85 (s, 1H, NH) ppm; ^{13}C NMR (100 MHz, $CDCl_3$) δ: 19.7, 51.8, 55.3, 61.4, 112.1, 124.6, 125.3, 126.4, 128.1, 128.5, 135.8, 136.4, 144.1, 157.2, 163.4, 165.1 ppm IR (KBr): 3221, 2950, 1707, 1677, 1513, 1466, 1375, 1304, 1207, 1141, 828, 771 cm^{-1}; found: C, 68.09; H, 5.51; N, 4.05 $C_{20}H_{19}NO_5$; requires: C, 67.98; H, 5.42; N, 3.96%.

3. Results and discussion

Our initial aim was to develop an efficient one-pot procedure for the synthesis of 3,4,5-substituted furan-2(5*H*)-one derivatives through the reaction of aromatic amines, aldehydes and acetylenic esters by employing $Al(HSO_4)_3$. Accordingly, the transformation of 4-methylaniline, dimethylacetylenedicarboxylate and benzaldehyde into methyl 4-(*p*-tolylamino)-2,5-dihydro-5-oxo-2-phenylfuran-3-carboxylate was investigated (Table 1). The reaction was carried out by using different solvents (Table 1, entries 1–6) or solvent-free conditions (Table 1, entry 7) at room temperature. Lower yield of the product was achieved under solvent-free conditions. It was found that the best results were obtained when 0.05 g of $Al(HSO_4)_3$ in EtOAc as solvent was employed (Table 1, entry 4).

Table 1
Optimization of the reaction conditions for the synthesis of methyl 4-(*p*-tolylamino)-2,5-dihydro-5-oxo-2-phenylfuran-3-carboxylate.

Entry	Catalyst (g)	T (°C)	Solvent (5 mL)	Time (h)	Yield (%)[a]
1	0.05	r.t.	*n*–Hexane	8	45
2	0.05	r.t.	CH_2Cl_2	8	50
3	0.05	r.t.	Et_2O	8	65
4	0.05	r.t.	EtOAc	8	78
5	0.05	r.t.	EtOH	8	51
6	0.05	r.t.	MeOH	8	55
7	0.05	r.t.	–	8	25
8	–	r.t.	EtOAc	10	–
9	0.025	r.t.	EtOAc	10	65
10	0.075	r.t.	EtOAc	6	77
11	0.1	r.t.	EtOAc	5	76

[a] Isolated yields.

To find out the optimized amount of $Al(HSO_4)_3$, the reaction was carried out by varying the quantity of catalyst (Table 1, entries 9–11). The maximum yield was obtained when 0.05 g of catalyst was used (Table 1, entry 4). Further increase in the amount of $Al(HSO_4)_3$ in the mentioned reaction did not have any significant effect on the product yield. The results are summarized in Table 1.

Next, the scope and efficiency of these procedures were explored for the synthesis of a wide variety of substituted 3,4,5-substituted furan-2(5*H*)-ones (Scheme 1, Table 2).

Generally, the results were excellent in terms of yield and product purity. A series of aromatic aldehydes and amines were investigated (Table 2, products **1a–17a**). In all cases, aromatic aldehydes containing electron-donating groups gave shorter times and higher yields than that with electron-withdrawing groups.

The work-up procedure is very clear-cut; this means that the products were isolated and purified by simple filtration and washing with diethyl ether. Our protocol making use of $Al(HSO_4)_3$ during the reaction process is better than those using hazardous liquid acidic catalysts.

Table 2
Synthesis of 3,4,5-substituted furan-2(5*H*)-one derivatives (Scheme 1).

Product	Aldehyde	Amine	R	Time (h)	Yield (%)[a]
1a	Benzaldehyde	4-Methylaniline	**Me**	8	78
2a	Benzaldehyde	4-Methylaniline	**Et**	8	80
3a	4-Methylbenzaldehyde	4-Methylaniline	**Me**	7	89
4a	4-Methylbenzaldehyde	4-Methylaniline	**Et**	7	90
5a	4-Chlorobenzaldehyde	4-Methylaniline	**Me**	10	71
6a	4-*tert*-Butylbenzaldehyde	4-Methylaniline	**Me**	8	81
7a	4-*tert*-Butylbenzaldehyde	4-Methylaniline	**Et**	8	80
8a	4-Methylbenzaldehyde	4-Chloroaniline	**Me**	10	85
9a	Benzaldehyde	4-Chloroaniline	**Me**	10	79
10a	4-Methylbenzaldehyde	4-Methoxyaniline	**Me**	7	86
11a	Benzaldehyde	Aniline	**Me**	8	84
12a	Benzaldehyde	Aniline	**Et**	9	77
13a	4-Methylbenzaldehyde	Aniline	**Me**	8	86
14a	4-Methylbenzaldehyde	Aniline	**Et**	8	84
15a	4-Chlorobenzaldehyde	Aniline	**Me**	10	75
16a	2-Chlorobenzaldehyde	Aniline	**Me**	12	70
17a	2,4-Dichlorobenzaldehyde	Aniline	**Me**	12	80

[a] Isolated yields. All known products had been reported previously in the literature and were characterized by comparison of their IR and NMR spectra with those of authentic samples [27,28].

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4. Conclusion

In summary, an efficient protocol for the preparation of 3,4,5-substituted furan-2(5*H*)-one derivatives was described. The reactions were carried out under ambient conditions with short reaction times and produce the corresponding products in good yields. The present methodology offers several advantages such as good yields, simple procedure, shorter reaction times and milder conditions; moreover, the products were purified *without* having resort to chromatography.

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