Melamine Trisulfonic Acid as a Highly Efficient and Reusable Catalyst for the Synthesis of β -Acetamido Ketones

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Abstract: A highly efficient protocol for the one-pot multi-component condensation of acetophenones with aromatic aldehydes, acetonitrile and acetyl chloride in the presence of melamine trisulfonic acid (MTSA) as a highly efficient and recyclable sulfonic acid-containing catalyst at room temperature is described. In this method, β -acetamido ketone derivatives are obtained in high to excellent yields and in relatively short reaction times.

Keywords: β -Acetamido ketone, Melamine trisulfonic acid (MTSA), Sulfonic acid-containing catalyst, One-pot multi-component reaction, Acetophenone, Aldehyde.

Introduction

β -Acetamido ketones are useful building blocks for a number of biologically and pharmaceutically valuable compounds¹⁻⁷. For example, they are precursors of molecules such as 1,3-amino alcohols¹⁻³ and γ -lactams⁴, as well as biologically attractive compounds such as nikkomycins or neopolyoxins^{5,6}. Moreover, it is reported that β -acetamido ketones can act as aglucosidase inhibitors⁷. The one-pot multi-component condensation of acetophenones with aromatic aldehydes, acetonitrile and acetyl chloride has been used as a most common synthetic route towards β -acetamido ketones. Some catalysts have been utilized to perform this transformations, e.g. La(OTf)₃⁷, CoCl₂⁸, heteropolyacids⁹, Zr(HSO₄)₄¹⁰, K₅CoW₁₂O₄₀.3H₂O¹¹, BiOCl¹², 1-chloromethyl-4-fluoro-1,4-diazoniabicyclo[2.2.2]octane-bis(tetrafluoroborate) {SelectfluorTM}¹³, ZnO¹⁴, ZrOCl₂.8H₂O¹⁵, polyaniline-supported salts¹⁶, and CeCl₃.7H₂O¹⁷. β -Acetamido ketones have been also prepared from acetophenones, aldehydes and amides¹⁸. However, the reported methods for the synthesis of these compounds are associated with one or more of the following drawbacks: (i) low yields, (ii) long reaction times, (iii) the use of large amount of catalyst, (iv) the use of toxic or expensive catalysts, (v) tedious work-up procedure, (vi) harsh reaction conditions, and (vii) performance the reaction under certain special conditions. So, search for finding a protocol for the preparation of β -acetamido ketones which are not associated with the above-mentioned disadvantages is still relevant.

Multi-component reactions (MCRs) play an important role in combinatorial chemistry because of the ability to synthesize target compounds with greater efficiency and atom economy by generating structural complexity in a single step from three or more reactants¹⁹⁻²³. Moreover, MCRs have many advantages over conventional synthetic methodologies, such as higher productivity, simple procedures and facile execution¹⁹⁻²³.

In recent years, there has been a rapid growth in the development of novel heterogeneous and homogeneous sulfonic acid-containing catalysts and reagents in organic synthesis, because of their unique properties such as efficiency, high reactivity, easy availability of their starting materials and ability to promote a wide range of reactions^{10,23-35}. They are also green, non-toxic, non-corrosive and inexpensive. Melamine trisulfonic acid (MTSA) is certainly one of the most interesting sulfonic acid-containing catalysts which has been recently synthesized, and used in some organic transformations including synthesis of coumarins³², synthesis of 3,4-dihydropyrimidin-2(1*H*)-ones/thiones³³, acetylation of alcohols, phenols, and amines³⁴ and chemoselective oxathioacetalization of aldehydes³⁵.

In this paper, we report a new, highly efficient and simple method for the synthesis of β -acetamido ketones *via* the one-pot multi-component condensation between acetophenones, arylaldehydes, acetonitrile and acetyl chloride using melamine trisulfonic acid (MTSA) as an inexpensive and reusable SO₃H-containing catalyst at room temperature (Scheme 1). It is worth noting that this method has none of the above-mentioned drawbacks at all.

Scheme 1

Experimental

All chemicals were purchased from Merck or Fluka Chemical Companies. All known compounds were identified by comparison of their melting points and spectral data with those reported in the literature. Progress of the reactions was monitored by TLC using silica gel SIL G/UV 254 plates. The ¹H NMR (300 MHz) and ¹³C NMR (75 MHz) were run on a Bruker Avance DPX-250, FT-NMR spectrometer (δ in ppm). Melting points were recorded on a Büchi B-545 apparatus in open capillary tubes.

Procedure for the preparation of melamine trisulfonic acid (MTSA)

A 250 mL suction flask charged with chlorosulfonic acid (5 mL, 75.2 mmol) was equipped with a gas inlet tube for conducting HCl gas over an adsorbing solution, i.e. water. Melamine (3.16 g, 25.07 mmol) was added in small portions over a period of 30 min at room temperature. HCl gas evolved from the reaction vessel immediately. After completion of the addition of melamine, the mixture was shaken for 30 min; meanwhile, the residual HCl was

exhausted by suction. The mixture was triturated with *n*-hexane (10 mL) and then filtered. The solid residue was washed with *n*-hexane (10 mL) and dried under vacuum. MTSA (7.9 g, 87%) was obtained as a white solid, which was stored in a capped bottle³².

General procedure for the synthesis of β -acetamido ketones

To a mixture of acetophenone (1 mmol), aldehyde (1 mmol), acetonitrile (3 mL) and acetyl chloride (0.3 mL) in a 10 mL round-bottomed flask, was added MTSA (0.018 g, 0.05 mmol), and the resulting mixture was stirred at room temperature. After completion of the reaction, as monitored by TLC, the solvent (acetonitrile) was evaporated, EtOAc (5 mL) was added, and the catalyst was recovered by filtration, washed with EtOAc (5 mL), dried and reused (MTSA was recovered three times without significant loss of its activity). After evaporation of the EtOAc from the filtrate, crushed ice (10 mL) was added to the mixture and stirred thoroughly. On solidification, the crude product was filtered, dried, and purified by short column chromatography on silica gel eluted with EtOAc/*n*-hexane (1/4).

Some selected spectral data of the products

Note: Compounds **1f**, **1k**, **1n**, **1p**, **1q**, **1r** and **2a** are new.

N-(3-(4-Nitrophenyl)-3-oxo-1-phenylpropyl)acetamide (**1c**)

¹H NMR (CDCl₃): δ 1.959 (s, 3H), 3.43 (q, J = 3.5, 1H), 3.78 (q, J = 3.5, 1H), 5.53 (q, J = 3.9, 1H), 6.95 (d, J = 6.3, 1H), 7.31 (s, 5H), 8.03 (d, J = 6.0, 2H), 8.23 (d, J = 5.8, 2H); ¹³C NMR (CDCl₃): δ 23.1, 44.3, 50.1, 123.8, 126.5, 127.8, 129.2, 140.9, 150.3, 169.9, 196.6.

N-(3-(4-Bromophenyl)-3-oxo-1-phenylpropyl)acetamide (**1d**)

¹H NMR (CDCl₃): δ 2.04 (s, 3H), 3.371 (q, J = 5.4, 1H), 3.66 (q, J = 5.4, 1H), 5.54 (q, J = 6.6, 1H), 6.99-7.75 (m, 9H); ¹³C NMR (CDCl₃): δ 23.2, 29.6, 43.5, 50.0, 126.5, 127.5, 128.6, 129.6, 131.9, 135.3, 140.8, 169.7, 197.2.

N-(1,3-Bis(4-methoxyphenyl)-3-oxopropyl)acetamide (**1f**)

¹H NMR (CDCl₃): δ 2.02 (s, 3H), 3.67 (s, 1H), 3.76 (s, 3H), 3.86 (s, 3H), 5.49 (s, 1H), 6.88 (q, J = 7.8, 5H), 7.72 (d, J = 8.3, 2H), 7.90 (d, J = 8.0, 2H); ¹³C NMR (CDCl₃): δ 23.4, 42.8, 49.7, 55.2, 55.5, 113.8, 113.9, 127.7, 129.6, 130.5, 133.1, 158.7, 163.7, 169.6, 197.2; MS (m/z): 327 (M⁺).

N-(3-Oxo-1,3-dip-tolylpropyl)acetamide (**1g**)

¹H NMR (CDCl₃): δ 1.93 (s, 3H), 2.27 (s, 3H), 2.37 (s, 3H), 3.36 (q, J = 6.2, 1H), 3.64 (q, J = 5.9, 1H), 5.55 (q, J = 7.1, 1H), 7.09 (d, J = 7.7, 2H), 7.22 (m, 2H), 7.41 (d, J = 7.7, 1H), 7.80 (d, J = 7.7, 2H); ¹³C NMR (CDCl₃): δ 21.1, 21.7, 23.1, 43.6, 49.8, 126.5, 128.3, 129.2, 129.3, 134.1, 136.9, 138.4, 144.2, 169.8, 197.8; MS (m/z): 295 (M⁺).

N-(1,3-Bis(4-nitrophenyl)-3-oxopropyl)acetamide (**1i**)

¹H NMR (CDCl₃): δ 2.10 (3H, s, 3H), 3.61 (q, J = 3.5, 1H), 3.88 (q, J = 3.8, 1H), 5.69 (s, 1H), 6.73 (s, 1H), 7.27 (s, 1H), 7.54 (s, 1H), 8.09-8.32 (m, 5H); ¹³C NMR (CDCl₃): δ 29.7, 49.1, 59.5, 124.1, 124.1, 124.4, 129.2, 140.3, 147.3, 147.7, 150.7, 169.8, 196.3.

N-(3-(4-Bromophenyl)-1-(4-nitrophenyl)-3-oxopropyl)acetamide (**1j**)

¹H-NMR (CDCl₃): δ 2.05 (s, 3H), 3.47 (q, J = 5.6, 1H), 3.79 (q, J = 5.2, 1H), 5.65 (q, J = 5.5, 1H), 7.02 (d, J = 7.9, 1H), 7.58 (d, J = 8.7, 2H), 7.61 (d, J = 8.4, 2H), 7.76 (d, J = 8.4, 2H), 8.15 (d, J = 8.4, 2H); ¹³C NMR (CDCl₃): δ 23.3, 42.7, 49.1, 113.8, 123.8, 127.4, 129.3, 129.5, 132.2, 134.8, 147.1, 148.4, 169.8, 196.8; MS (m/z): 390 (M⁺).

Compound (1k)

^1H NMR (CDCl_3): δ 2.07 (s, 3H), 3.46 (d, J = 6.8, 1H), 3.81 (d, J = 7.8, 1H), 5.63 (s, 1H), 7.09 (s, 1H), 7.2–7.6 (m, 3H), 7.7–8.2 (m, 4H), 9.96 (s, 1H); ^{13}C NMR (CDCl_3): δ 23.3, 42.8, 49.5, 127.1, 129.2, 129.6, 130.1, 132.1, 134.9, 135.5, 147.6, 170.01, 191.8, 197.1; MS (m/z): 373 (M^+).

***N*-(1-(4-Chlorophenyl)-3-oxo-3-*p*-tolylpropyl)acetamide (1l)**

^1H NMR (CDCl_3): δ 2.02 (s, 3H), 2.40 (s, 3H), 3.35 (q, J = 3.9, 1H), 3.9 (q, J = 5.3, 1H), 5.5 (q, J = 5.5, 1H), 7.26 (m, 5H), 7.37 (s, 1H), 7.77 (d, J = 8.0, 3H); ^{13}C NMR (CDCl_3): δ 21.7, 23.1, 42.8, 49.3, 127.9, 128.2, 128.7, 129.4, 133.1, 133.8, 139.6, 144.7, 170.1, 197.8; MS (m/z): 312 (M^+).

***N*-(3-(4-Methoxyphenyl)-1-(naphthalen-3-yl)-3-oxopropyl)acetamide (1n)**

^1H NMR (CDCl_3): δ 2.03 (s, 3H), 3.43 (d, J = 6.6, 1H), 3.79 (s, 4H), 5.72 (s, 1H), 6.90 (q, J = 6.8, 2H), 7.42–7.53 (m, 4H), 7.75–8.10 (m, 5H), 8.23 (s, 1H); ^{13}C NMR (CDCl_3): δ 22.6, 43.1, 50.6, 55.5, 113.8, 124.8, 125.4, 126.0, 127.6, 127.9, 128.5, 129.5, 130.5, 132.6, 133.2, 138.4, 163.7, 170.9, 196.4; MS (m/z): 347 (M^+).

***N*-(1-(Naphthalen-3-yl)-3-(4-nitrophenyl)-3-oxopropyl)acetamide (1p)**

^1H NMR (CDCl_3): δ 2.08 (s, 3H), 2.85–3.05 (2H, m), 5.5 (s, 1H), 7.47–8.5 (m, 12H); ^{13}C NMR (CDCl_3): δ 29.7, 49.8, 59.5, 124.7, 125.5, 126.4, 126.6, 126.8, 127.1, 127.5, 127.7, 128.1, 129.1, 129.4, 129.5, 130.5, 173.9, 194.1. MS (m/z): 362 (M^+).

***N*-(3-(4-Bromophenyl)-1-(naphthalen-3-yl)-3-oxopropyl)acetamide (1q)**

^1H NMR (CDCl_3): δ 1.98 (s, 3H), 3.41 (d, J = 6.4, 1H), 3.76 (d, J = 6.6, 1H), 5.70 (s, 1H), 7.27–7.59 (m, 6H), 7.69–7.92 (m, 6H); ^{13}C NMR (CDCl_3): δ 23.2, 43.5, 50.1, 124.7, 125.3, 126.1, 126.3, 127.6, 128.6, 129.6, 131.9, 132.7, 133.1, 135.1, 138.2, 170.1, 197.1; MS (m/z): 395 (M^+).

***N*-(1-(Anthracen-10-yl)-3-(4-methoxyphenyl)-3-oxopropyl)acetamide (1r)**

^1H NMR (CDCl_3): δ 2.06 (s, 3H), 3.84 (s, 1H), 4.14 (q, J = 7.0, 1H), 5.27 (s, 1H), 6.86 (d, J = 8.7, 2H), 7.13 (d, J = 6.3, 1H), 7.46 (t, J = 6.9, 2H), 7.56 (t, J = 8.4, 2H), 7.91 (d, J = 8.7, 2H), 8.05 (d, J = 5.4, 2H), 8.42 (s, 1H), 8.52 (s, 2H); ^{13}C NMR (CDCl_3): δ 23.2, 29.7, 46.1, 55.4, 59.5, 113.7, 124.2, 124.9, 125.4, 126.5, 128.8, 129.1, 129.3, 129.7, 130.5, 131.9, 142.6, 163.5, 195.2; MS (m/z): 397 (M^+).

Compound (2a)

^1H NMR (CDCl_3): δ 1.95 (s, 3H), 3.81 (s, 3H), 3.89 (s, 1H), 4.13 (q, J = 5.13, 1H), 5.27 (s, 1H), 5.83 (s, 3H), 6.9–7.02 (m, 6H), 7.15–7.38 (m, 10H), 7.51–7.64 (m, 9H), 7.67–7.83 (m, 4H); ^{13}C NMR (CDCl_3): δ 23.0, 43.1, 46.8, 69.8, 112.3, 121.3, 126.2, 127.9, 128.4, 128.8, 129.7, 129.8, 131.8, 131.9, 135.2, 138.1, 155.4, 168.2, 197.4; Anal. calcd. for $\text{C}_{60}\text{H}_{54}\text{Br}_3\text{N}_3\text{O}_9$: C, 60.01; H, 4.53; N, 3.50, found: C, 60.27; H, 4.67; N, 3.39.

Results and Discussion

Initially, melamine trisulfonic acid was prepared from the reaction of melamine with chlorosulfonic acid according to the reported procedure³². Considering the high efficacy of this SO_3H -containing compound to catalyze some organic transformation^{32–35}, and also the high importance of β -acetamido ketones, we decided to examine the applicability of this catalyst for the synthesis of β -acetamido ketones. For this purpose, as a model, the reaction of acetophenone (1 mmol) with benzaldehyde (1 mmol), acetonitrile (3 mL) and acetyl

chloride (0.3 ml) was examined in the presence of different molar ratios of MTSA at room temperature. The results are summarized in Table 1. As it can be seen in Table 1, higher yields of the product and shorter reaction times were obtained when the reaction was performed using 5 mol% of the catalyst. Moreover, increasing the amount of MTSA didn't affect significantly on the reaction results.

Table 1. The reaction of acetophenone with benzaldehyde, acetonitrile and acetyl chloride using different molar ratios of MTSA at room temperature.

Entry	Catalyst Amount, mol%	Time, min	Yield ^a , %
1	-	12 h	No reaction
2	1	200	51
3	3	60	85
4	5	30	92
5	7	30	92

^aIsolated yield.

To assess efficiency and scope of the method, different acetophenones (processing electron-releasing substituents, electron-withdrawing substituents and halogens on their aromatic ring) were condensed with structurally and electronically diverse aromatic aldehydes (bearing electron-releasing substituents, electron-withdrawing substituents and halogens on their aromatic ring), acetonitrile and acetyl chloride under the optimized reaction conditions. The respective results are displayed in Table 2. As Table 2 indicates, all reactions proceeded efficiently and the desired products were obtained in good to excellent yields in relatively short reaction times; thus, the protocol was general and efficient. Furthermore, β -acetamido ketones were obtained in high yields and in relatively short reaction times when 2-naphthaldehyde and anthracene-10-carbaldehyde were used in the reaction instead of benzaldehydes (Table 2, compounds **1n-r**).

Table 2. The preparation of β -acetamido ketones from acetophenones, arylaldehydes, acetonitrile and acetyl chloride using MTSA.

Product	Time, min	Yield ^a , %	M.p. °C (Lit.)
(1a)	30	92	100-102 (104-106) ¹⁰
(1b)	45	87	127-129 (130-132) ¹⁵
(1c)	100	90	77-79 (74-76) ¹⁵

<p>(1d)</p>	50	91	100-102 (98-101) ¹⁶
<p>(1e)</p>	30	93	109-111 (111-112) ⁹
<p>(1f)</p>	20	93	124-127 ^b
<p>(1g)</p>	30	94	118-119 (-) ¹⁴
<p>(1h)</p>	60	91	150-152 (153) ⁹
<p>(1i)</p>	130	86	185-188 (187-188) ¹⁰
<p>(1j)</p>	90	85	162-163 (-) ¹⁰
<p>(1k)</p>	90	83	104-105 ^b

(1l)	90	93	130-132 (-) ¹⁴
(1m)	100	88	114-116 (116-118) ³⁶
(1n)	75	91	108-110 ^b
(1o)	45	93	115-117 (112-114) ³⁶
(1p)	120	87	165-166 ^b
(1q)	75	85	137-139 ^b
(1r)	120	86	140-142 ^b

^aIsolated yield. ^bThis compound is new.

Interestingly, the condensation of 4-bromoacetophenone (3.2 eq.) with a tris-aldehyde (1 eq.), acetonitrile (9 mL) and acetyl chloride (0.9 mL) using MTSA (10 mol%) at room temperature afforded complex compound **2a** in 83% yield within 120 min (Scheme 2). This is the first report of the synthesis of this class of β -acetamido ketones.

Scheme 2

Table 3. Comparison of the results of the reaction of acetophenone with benzaldehyde, acetonitrile and acetyl chloride catalyzed by MTSA, with those obtained by the reported catalysts.

Catalyst, Reaction Conditions	Time, min	Yield ^a , %	Ref.
MTSA, r.t.	30	92	- ^a
La(OTf) ₃ , Reflux in MeCN ^b	180	80	7
H ₃ [PW ₁₂ O ₄₀], Reflux in MeCN	45	81	9
Zr(HSO ₄) ₄ , r.t.	30	90	10
K ₅ CoW ₁₂ O ₄₀ .3H ₂ O, r.t.	60	86	11
Selectfluor TM , r.t.	240	74	13
ZnO, Reflux in MeCN	360	90	14
ZrOCl ₂ .8H ₂ O, r.t.	300	90	15
Polyaniline-supported salt, 50 °C	60	90	16
CeCl ₃ .7H ₂ O, r.t.	420	96	17

^aOur catalyst. ^bIn this case, the reaction time and yield is related to the condensation of acetophenone with 4-nitrobenzaldehyde, acetonitrile and acetyl chloride.

To compare the efficiency of MTSA with the reported catalysts for the synthesis of β -acetamido ketones, we have tabulated the results of these catalysts to perform the condensation of acetophenone with benzaldehyde, acetonitrile and acetyl chloride, in Table

3. As it is clear from Table 3, MTSA is superior to the previously reported catalysts in term of reaction times, yields and reaction conditions.

Conclusions

In summary, we have developed a new method for the one-pot multi-component reaction of acetophenones with arylaldehydes, acetonitrile and acetyl chloride. The promising points for the presented methodology are efficiency, generality, high yields, short reaction times, cleaner reaction profile, simplicity, and ease of preparation of catalyst which makes it an attractive procedure for the preparation of β -acetamido ketones as biologically interesting compounds.

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