



A catalytic three-component synthesis of isoxazol-5(4*H*)-ones under green conditions

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The three-component cyclocondensation of various aryl/heteroaryl aldehydes, hydroxylamine hydrochloride, and ethyl acetoacetate/ethyl 4-chloro-3-oxobutanoate have been established for the synthesis of isoxazol-5(4*H*)-ones using sodium malonate as an efficient catalyst. This reaction has been performed in water as a green reaction medium at 25°C. Optimization of the reaction conditions show that the reaction performs better in aqueous medium, at room temperature, and in the presence of 10 mol% of the catalyst. This procedure has several unique features, including shorter reaction times, easy separation of pure products, avoiding the hazardous organic solvents, simplicity of experimental procedures, operationally simple, and eco-friendly.

Keywords: Green synthesis, isoxazol-5(4*H*)-ones, sodium malonate, β -ketoester, three-component cyclocondensation

Heterocycles are obtaining more importance in recent years due to their pharmacological activities. The isoxazole core is a significant synthetic line of attack in drug discovery, and plays a significant role in arena of natural products and medicinal chemistry¹. Several marketed drugs, including sulfamethoxazole, sulfisoxazole, sulfafurazole, flucloxacillin, dicloxacillin, acetyl sulfisoxazole, valdecocix, parecoxib, danazol, zonisamide, leflunomide, risperidone, isocarboxazid, cloxacillin, and oxacillin containing isoxazole nucleus²⁻⁵. Isoxazol-5(4*H*)-ones, on the other hand, have widely been showed antibacterial, antifungal, androgen receptor antagonist, tyrosinase inhibitory, SIRT1 inhibitory, anti-obesity, anti-HIV, antioxidant, and anticancer activities (Figure 1)⁶⁻¹⁰. These five-membered heterocyclic compounds are regarded as active agrochemical compounds used as fungicides and insecticides^{11,12}. They have also been investigated for use in photovoltaic cells^{13,14}, monochromatic terahertz difference frequency^{15,16}, and laser dyes¹⁷. Isoxazol-5(4*H*)-ones have also been used as versatile building blocks in synthesis of other organic compounds¹⁷⁻²⁶. Conventional methods for the synthesis of isoxazol-5(4*H*)-ones are the cyclization of *O*-propioloyl/propargylic oximes^{27,28}, the reaction of ethyl acetoacetate and hydroxylamine hydrochloride followed condensation with aromatic aldehydes, and condensation of 1,3-dicarbonyls with benzaldoximes²⁹. Significant development has been made in reviewing

different routes for the construction of an isoxazol-5(4*H*)-one skeleton³⁰⁻⁶³.

Water as a universal solvent in which the vast majority of interactions occur in living systems have other unique properties, including abundance, cost-effectiveness, non-flammability, availability, non-hazardous, non-toxic, uniquely redox-stable, clean, and recyclable medium. Thus, water is preferred compared to organic solvents. For this reasons, the implementation of synthetically useful chemical reactions in the aquatic medium has attracted the attention of many chemists from the point of view of green chemistry as well as economically⁶⁴⁻⁷². Sodium malonate (SM) is a commercially available dibasic organic compound that we have recently used from this organic base as a catalyst for the synthesis of pyran annulated compounds^{73,74}. In this study, we have investigated the catalytic potential of SM for the green

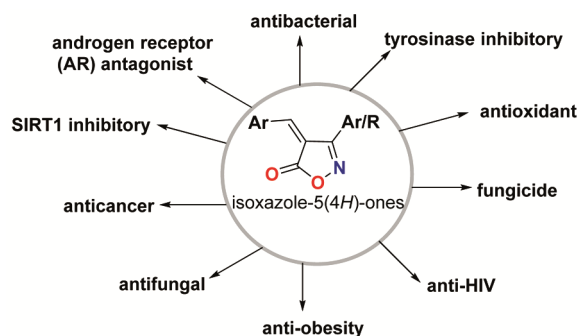


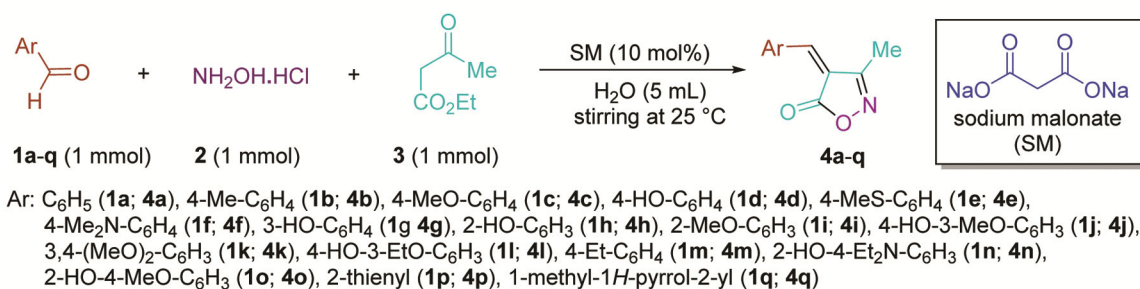
Figure 1 — Various biological effects of isoxazole-5(4*H*)-ones

synthesis of isoxazol-5(4*H*)-ones (**4a-q**) by treating aryl/heteroarylaldehydes (**1a-q**) with hydroxylamine hydrochloride (**2**) and ethyl acetoacetate (**3**) in a one-vessel method (Scheme I).

Results and Discussion

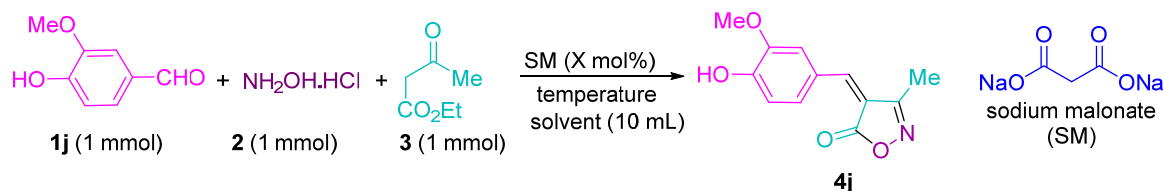
Initially, the cyclocondensation of vanillin (**1j**), hydroxylamine hydrochloride (**2**), and ethyl acetoacetate (**3a**) in water in the presence catalytic amounts of SM at room temperature (RT) was selected as the model reaction (Table I).

The different amounts of SM catalyst were explored. When the reaction performed in water using 2 mol% of SM, the isoxazol-5(4*H*)-one product (**4j**) was obtained in 55% isolated yield after 65 min. (Table I, entry 1). The yield of product was increased with the increase of the amount of catalyst (Table I, entries 2-5). The highest isolated yield of **4j** was obtained using 10 mol% of catalyst (Table I, entry 5). There was no improvement in the yield and reaction time when the catalyst amount increased from 10 mol% to 12 mol% (Table I, entry 6). The model



Scheme I — Cyclocondensation between aryl/heteroarylaldehydes **1a-q**, hydroxylamine hydrochloride **2**, and ethyl acetoacetate **3** to synthesis of isoxazol-5(4*H*)-ones **4a-q** in the presence of sodium malonate (SM)

Table I — Screening the reaction conditions for the synthesis of 4-(4-hydroxy-3-methoxybenzylidene)-3-methylisoxazol-5(4*H*)-one **4j**



Entry	SM (X mol%)	Solvent	Temp. (°C)	Time (min)	Isolated yields (%)
1	2	H ₂ O	25	65	55
2	4	H ₂ O	25	60	60
3	6	H ₂ O	25	50	65
4	8	H ₂ O	25	45	75
5 ^a	10	H ₂ O	25	35	94
6	12	H ₂ O	25	50	88
7	10	EtOH	25	45	60
8	10	EtOH:H ₂ O (1:1)	25	40	70
9	10	<i>n</i> -Hexane	25	70	45
10	10	Acetone	25	80	15
11	10	DCM	25	80	30
12	10	DMF	25	80	10
13	10	CHCl ₃	25	80	15
14	10	EtOAc	25	80	20
15	10	H ₂ O	50	60	75
16	10	H ₂ O	65	65	70
17	10	H ₂ O	Reflux	50	65
18	10	Solvent-free	25	95	30

^a Optimized reaction conditions.

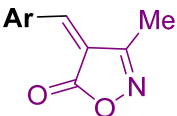
reaction was next investigated in solvents, including EtOH, a mixture of H₂O: EtOH (v/v; 1:1), *n*-hexane, acetone, dichloromethane (DCM), dimethylformamide (DMF), chloroform (CHCl₃), and ethyl acetate (EtOAc) to explore the effect of the solvent (Table I, entries 7-14). The use of any of these solvents, did not obtain better results than H₂O solvent. Next, the effect of temperature on the yield of reaction and reaction time was investigated using 10 mol% the catalyst and H₂O solvent (Table I, entries 15-17). Observations indicated that no particular improvement was achieved. It was also found that the low yield of the corresponding product (**4j**) was obtained under solventless conditions (Table I, entry 18).

After optimization of the reaction conditions, the substrate scope was explored using benzaldehyde (**1a**), substituted benzaldehydes (**1b-i** and **1k-o**) and heterocyclic aldehydes (**1p-q**). As shown in Table II, the reaction of the benzaldehyde (**1a**) as well as substituted benzaldehydes (**1b-i** and **1k-o**) bearing electron-donating groups at the different positions on the phenyl ring, given that the corresponding isoxazol-5(*4H*)-one products (**4a-i** and **4k-o**) in good to excellent isolated yields (Table II, entries 1-9 and 11-15). In addition, thiophene-2-carbaldehyde (**1p**) and 1-methyl-1*H*-pyrrole-2-carbaldehyde (**1q**) were also tested in this three-component reaction, and the

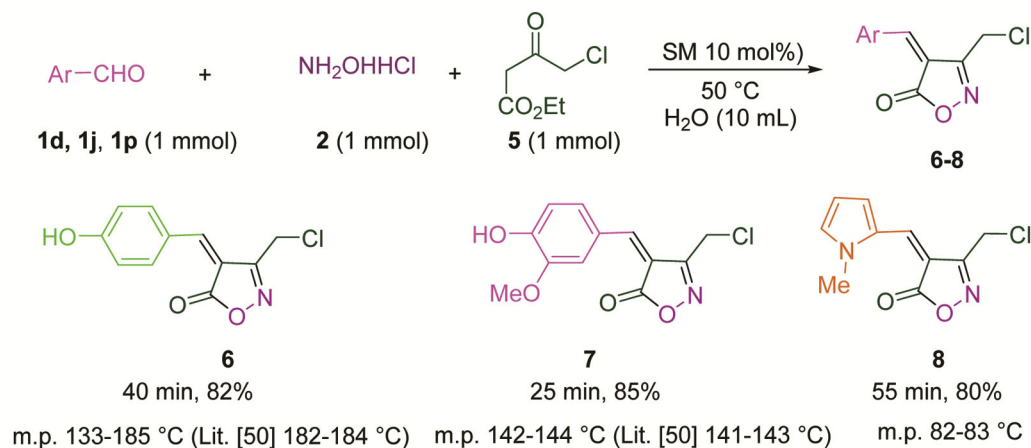
target isoxazol-5(*4H*)-ones (**4p** and **4q**) were synthesized in 85% and 80% isolated yields, respectively. The use of substituted benzaldehydes containing electron-withdrawing groups and aliphatic aldehydes in this experiment was not successful. In order to illustrate the generalization of the method to the β -ketoester precursor, the 3-chloromethylisoxazol-5(*4H*)-ones **6-8** were successfully synthesized by treating aldehydes (**1d**, **1j** and **1q**) with **2** and ethyl 4-chloro-3-oxobutanoate **5** (Scheme II).

The structure of the synthesized isoxazol-5(*4H*)-ones were confirmed using spectroscopic spectral data analysis. For example, in the ¹H NMR spectrum of 3-methyl-4-((1-methyl-1*H*-pyrrol-2-yl)methylene)-isoxazol-5(*4H*)-one (**4q**) characteristic signals were observed. The ¹H NMR of compound **4q** show three singlet signals at δ 2.25 and 3.85 ppm for two CH₃ groups of isoxazol moiety and *N*-methyl of pyrrole ring, respectively. A singlet peak at δ 7.16 ppm is attributed to CH vinyl between two heterocyclic rings. The three aromatic protons of pyrrole moiety were observed as the two doublets of doublets signals at δ 6.41 and 8.56 ppm as well as a triplet signal at δ 7.13 ppm. The ¹³C NMR spectrum of **4q** exhibits two signals at 11.1 and 34.2 ppm for CH₃ groups and also showed two signals at 160.9 and 169.2 ppm for C=N and C=O groups of isoxazol ring, respectively. Other expected signals have appeared in the appropriate

Table II — Synthesis of **4a-q** in the presence of sodium malonate (SM) according to Scheme I

Entry	Ar	Compd	Time (min)	Isolated yield (%)	m.p. (°C)	
					Found	Reported (Ref. 47-49)
						
1	C ₆ H ₅	4a	40	83	141-143	142-143
2	4-Me-C ₆ H ₄	4b	45	86	136-137	135-137
3	4-MeO-C ₆ H ₄	4c	30	88	173-175	173-174
4	4-HO-C ₆ H ₄	4d	35	85	215-217	211-213
5	4-MeS-C ₆ H ₄	4e	50	86	232-233	New
6	4-Me ₂ N-C ₆ H ₄	4f	45	85	227-229	225-226
7	3-HO-C ₆ H ₄	4g	30	84	198-200	200-201
8	2-HO-C ₆ H ₃	4h	25	80	197-199	198-200
9	2-MeO-C ₆ H ₃	4i	30	90	158-160	159-160
10	4-HO-3-MeO-C ₆ H ₃	4j	35 (35, 40, 55) ^a	94 (92, 86, 75) ^a	212-214	215-217
11	3,4-(MeO) ₂ -C ₆ H ₃	4k	30	89	236-238	238-240
12	4-HO-3-EtO-C ₆ H ₃	4l	30	87	138-140	New
13	4-Et-C ₆ H ₄	4m	60	75	182-185	201-203
14	2-HO-4-Et ₂ N-C ₆ H ₃	4n	80	77	251-253	253-257
15	2-HO-4-MeO-C ₆ H ₃	4o	35	93	211-213	213-215
16	2-Thienyl	4p	45	82	144-146	144-145
17	1-Methyl-1 <i>H</i> -pyrrol-2-yl	4q	45	90	213-215	New

^a The reaction is performed after the catalyst recovery.

Scheme II — Synthesis of 3-chloromethylisoxazol-5(4*H*)-ones **6-8**

regions. The structure of heterocyclic compound **8** is similar to **4q**, except that it has chloromethyl group instead of CH_3 group of the isoxazol-5(4*H*)-one ring. In the ^1H NMR of spectrum of **8**, all the expected signals have been observed, but there is no trace of the methyl signal of the isoxazol-5(4*H*)-one ring, and instead the chloromethyl signal has appeared as a singlet at δ 4.94 ppm.

In conclusion, 3-methyl/chloromethylisoxazol-5(4*H*)-ones as interesting five-membered heterocyclic compounds have been synthesized successfully *via* one-pot, multicomponent cyclocondensation of aryl/heteroaryl aldehydes, hydroxylamine hydrochloride, and β -ketoesters. The reactions were catalyzed by treating with SM as a marketable available dibasic organocatalyst at RT. The products were obtained under benign conditions in good to high yields. The eye-catching properties of this work are eco-friendly, the reusability of catalyst, relatively shorter reaction times, simple workup procedure, no use of hazardous solvents, and purification without chromatographic methods.

Experimental section

All chemicals, unless otherwise specified, were purchased from commercial sources and were used without further purification, with the exception of liquid aldehydes, which were distilled before using. The well-known products were characterized by comparison of their physical data with those of known samples or by their spectral data. Melting points were measured on a Buchi 510 melting point apparatus and are uncorrected. ^1H and ^{13}C NMR spectra were recorded on a Varian-INOVA 500 MHz instrument at central laboratory of the University of Tehran, Tehran, Iran. FT-IR spectra were recorded on a Perkin Elmer

RXI spectrometer. The development of reactions was monitored by thin layer chromatography (TLC) analysis on Merck pre-coated silica gel 60 F_{254} aluminum sheets, visualized by UV light.

General procedure for the synthesis of isoxazol-5(4*H*)-ones (**4a-q** and **6-8**) catalyzed by SM

The appropriate aldehyde (**1a-q**, 1 mmol), hydroxylamine hydrochloride (**2**, 1 mmol), β -ketoester (**3** or **5**, 1 mmol), and SM (10 mol%) as a catalyst was stirred in H_2O (10 mL) at RT. After completion of the reaction as indicated by TLC analysis, the precipitated products were filtered off, washed with cold water and dried. The crude products were purified by recrystallization from EtOH. The filtrate contains a catalyst. After evaporation of the solvent from the filtrate, the catalyst was remained and was used for consecutive runs. Spectral data for representative compounds were as follows:

3-Methyl-4-(4-(methylthio)benzylidene)isoxazol-5(4*H*)-one (4e): ^1H NMR (CDCl_3 , 500 MHz): δ 2.29 (s, 3H), 2.55 (s, 3H), 7.31 (d, $J = 8.6$ Hz, 2H), 7.33 (s, 1H), 8.32 (d, $J = 8.6$ Hz, 2H); ^{13}C NMR (125 MHz, CDCl_3): δ 11.6, 14.5, 125.1, 125.3, 128.8, 134.2, 148.6, 148.9, 161.1, 167.9.

4-(4-Hydroxy-3-methoxybenzylidene)-3-methylisoxazol-5(4*H*)-one (4j): ^1H NMR ($\text{DMSO}-d_6$, 500 MHz): δ 2.28 (s, 3H), 3.89 (s, 3H), 6.98 (d, $J = 8.4$ Hz, 1H), 7.90 (s, 1H), 7.93 (dd, $J = 1.8, 8.4$ Hz, 1H), 8.56 (d, $J = 1.8$ Hz, 1H), 10.81 (s, 1H); ^{13}C NMR ($\text{DMSO}-d_6$, 125 MHz): δ 11.9, 55.8, 114.1, 116.2, 117.3, 125.6, 132.2, 148.1, 152.3, 154.4, 162.9, 169.6.

4-(3-Ethoxy-4-hydroxybenzylidene)-3-methylisoxazol-5(4*H*)-one (4l): ^1H NMR ($\text{DMSO}-d_6$, 500 MHz): δ 1.41 (t, $J = 6.9$ Hz, 3H), 2.27 (s, 3H),

4.14 (q, $J = 6.9$ Hz, 2H), 6.99 (d, $J = 8.4$ Hz, 1H), 7.80 (s, 1H), 7.90 (dd, $J = 2.1, 8.4$ Hz, 1H), 8.54 (d, $J = 2.1$ Hz, 1H), 10.72 (s, 1H, OH); ^{13}C NMR (DMSO- d_6 , 125 MHz): δ 11.8, 15.1, 64.3, 114.1, 116.3, 118.1, 125.5, 132.0, 147.1, 152.4, 154.5, 162.8, 169.5.

4-(2-Hydroxy-3-methoxybenzylidene)-3-methylisoxazol-5(4H)-one (4o): ^1H NMR (500 MHz, DMSO- d_6): δ 2.24 (s, 3H), 3.86 (s, 3H), 6.89 (t, $J = 8.1$ Hz, 1H), 7.26 (dd, $J = 1.2, 8.1$ Hz, 1H), 8.11 (s, 1H), 8.32 (dd, $J = 1.2, 8.4$ Hz), 10.34 (s, 1H); ^{13}C NMR (75 MHz, DMSO- d_6): δ 12.5, 58.4, 116.8, 118.6, 119.8, 139.3, 145.0, 147.6, 149.5, 152.9, 162.2, 176.1.

3-Methyl-4-((1-methyl-1H-pyrrol-2-yl)methylene)isoxazol-5(4H)-one (4q): ^1H NMR (500 MHz, CDCl_3): δ 2.25 (s, 3H), 3.85 (s, 3H), 6.41 (dd, $J = 2.3, 4.5$ Hz, 1H), 7.13 (t, $J = 2.0$ Hz, 1H), 7.16 (s, 1H), 8.56 (dd, $J = 1.5, 4.5$ Hz, 1H); ^{13}C NMR (125 MHz, $\text{CDCl}_3 + \text{DMSO-}d_6$): δ 11.1, 34.2, 112.2, 126.0, 129.2, 131.6, 134.9, 160.9, 169.2.

3-(Chloromethyl)-4-((1-methyl-1H-pyrrol-2-yl)methylene)isoxazol-5(4H)-one (8): ^1H NMR (500 MHz, DMSO- d_6): δ 3.92 (s, 3H), 4.94 (s, 2H), 6.53-6.54 (m, 1H), 7.72 (t, $J = 1.6$ Hz, 1H), 7.78 (s, 1H), 8.44 (dd, $J = 1.6, 4.5$ Hz, 1H); ^{13}C NMR (125 MHz, DMSO- d_6): δ 34.8, 35.9, 103.2, 113.7, 127.0, 130.1, 134.8, 138.5, 161.8, 169.6.

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