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Zinc dust catalysed efficient synthesis of 4-arylidene-2-phenyl-5(4*H*)-oxazolones

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A straight forward synthesis of 4-arylidene-2-phenyl-5(4*H*)-oxazolones (also called azlactones) by the condensation of arylaldehyde with hippuric acid in the presence of acetic anhydride and zinc dust as aheterogeneous catalyst is described. The reaction proceeds rapidly, does not require any additives and has a significantly shorter reaction rate. In the present method, aromatic aldehydes and hippuric acid have been successfully applied to synthesize various substituted azlactones in good to excellent yields. The final product has been characterised by FT-IR, ^{1}H and ^{13}C NMR, and mass spectrometry.

Keywords: Oxazolones, hippuric acid, aldehyde, zinc dust, mild condition

Heterocyclic molecules found in nature and synthetic proven breakthrough in biological activities and are essential in human life. In a broad classification of heterocyclic compounds available, azlactone heterocyclic compound exhibited vast range of pharmacological applications[1] (Figure 1). The molecule has five membered heterocyclic nucleus containing nitrogen and oxygen as heteroatom in their structural skeleton, which is one of the important classes of heterocycles. The literature data revealed that, position C-2 and C-4 of the azlactones are important and played important role in biological activities. Azlactone nucleus containing heterocyclic molecules found important pharmacological applications such as analgesic[2], anti-microbial, anti tumour[3], anti-bacterial[4], anti-fungal[5], anti-cancer[6], anti-inflammation[7], anti-HIV[8], anti-diabetic[9], anti-obesit[10], biosensors, photosensitive composition devices for proteins[11], tyrosinase inhibitory[12], in treatment of skin diseases[13], neuroleptic applications[14], photochemical and photophysical and as *p*H sensors[15]. Further, azlactones are found important intermediates in the synthesis of various organic motifs, including amino acids[16], peptides[17], α-acylamino alcohols, thiamine, N-substituted pyrroles, amides and other hetero aromatic molecules[18], and used in various alkaloid skeleton construction. Azlactone synthesis also called Erlenmeyer reaction first discovered in 1893 by Emil Erlenmeyer who demonstrated the reaction of benzaldehyde with hippuric acid in presence of acetic anhydride (Ac_2O) and sodium acetate as dehydrating and base catalyst respectively[19]. Subsequently, Erlenmeyer established the structure of azlactone using spectroscopic techniques and named it as an azlactone.

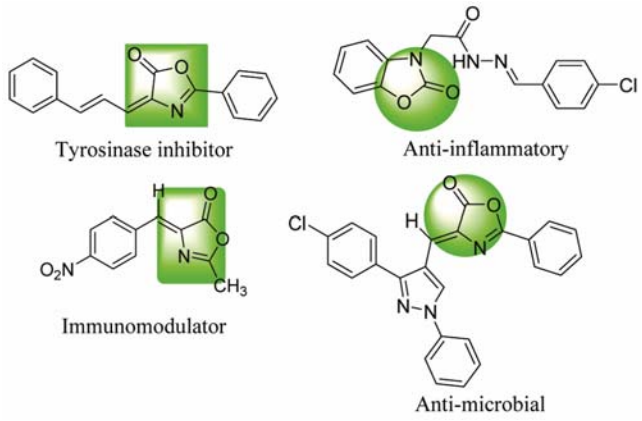


Figure 1 — Selected example of azlactone moiety containing bioactive molecule

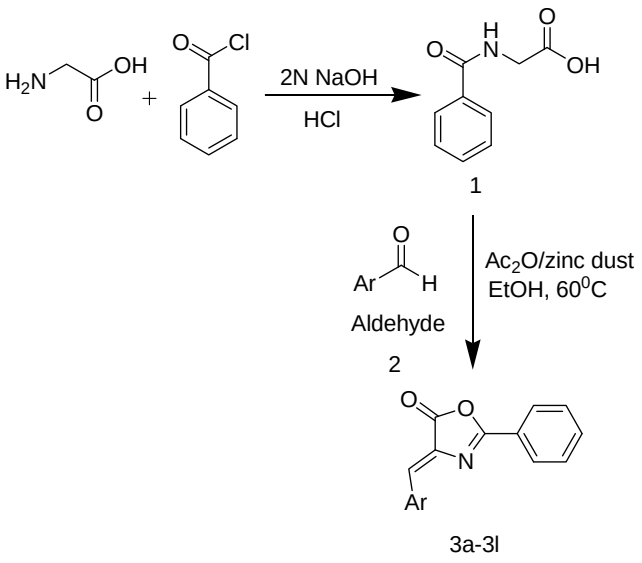
The reaction proceeds *via* a Perkin condensation through initial cyclization of hippuric acid gave the product Erlenmeyer azlactones. The organic synthesis promoted by the catalyst is one of the hot areas of research in the present days. In literature vast number of catalyst have been reported for the synthesis of azlactones are polyphosphoric acid[20], perchloric acid[21], $POCl_3$[22], carbodiimides[23], SO_3 in DMF[24], $Bi(OAc)_3$[25], $Bi(OTf)_3$[26], Al_2O_3[27], Silica-supported heteropolyacids[28], $Ca(OAc)_2$[29], Supported-KF[30], $ZnCl_2$[31], Fe_2O_3[32], Al_2O_3-H_3BO_3[33], CDMT[34] and ZnO[35]. Although all of these catalysed reactions

demonstrated certain advantages, still some of the catalysed reaction suffered disadvantages including use of the expensive catalyst, hazardous reagent, more reaction step involving longer time, tedious workup and recovery of the catalyst. Continuing our efforts to develop simple and efficient synthetic protocol in bioactive molecule synthesis. Here, we wish to report simple practical procedure for the synthesis of azlactone by the condensation of aromatic aldehyde and hippuric acid in presence of acetic anhydride and heterogeneous catalyst zinc dust.

Over the past few decades, the application of zinc dust in organic transformation is well exploited. Zinc dust has been utilized in various organic synthesis includes Williamson reaction[36], Barbier reaction[37], Diels-alder reaction[38], Friedel-crafts sulfonylation[39], Fries rearrangement[40], acylation of phenols, thiophenols, amines and alcohols[41], N-sulfonylation of amines[42] and many more important chemical transformations[43,44]. Further, Zinc dust also been employed as an acid neutralizer in the synthesis of Z-amino acids[45] and peptide synthesis employing Fmoc-amino acid chlorides[46].

Results and Discussion

Azlactones are versatile and promising heterocyclic molecules, which are used in vast area of research fields. The molecule contains many reactive sites permit to synthesize wide range of interesting molecule and showed promising applications in chemical biology and material science. Present work, chosen zinc dust as heterogeneous catalyst which is inexpensive and non-toxic, and not been reported for the synthesis of azlactone as per our knowledge of literature. The synthetic method demonstrated simple, greener and efficient reaction protocol developed for the synthesis of 4-arylidine-2-phenyl-5(4*H*)-oxazolones. One of the key building blocks for the azlactone synthesis is hippuric acid, which is synthesized by the reaction of benzoyl chloride and glycine in presence of base as depicted in Scheme I. After completion of the reaction, product isolated and recrystallized using hot water. To evaluate reaction conditions, we selected a model reaction of benzaldehyde (1mmol) with hippuric acid (1mmol) in the presence of Ac_2O (3.3mmol) and zinc dust at RT (Scheme I). After stirring for several hours', the product isolation found lesser yield of azlactone. This model experiment reaction gave us path to think of using organic solvent, and we have systematically optimized the reaction by employing several solvent available at our laboratory in a model reaction and the same is tabulated in Table I. To optimize suitable solvent the reaction carried out in a model reaction using different solvent system such as Chloroform ($CHCl_3$), Dichloromethane (CH_2Cl_2), Acetonitrile (CH_3CN), *N*,*N*-dimethylformamide (DMF), Methanol (MeOH), 1,4-dioxane, Ethanol (EtOH) and tetrahydrofuran (THF) at RT. The final isolated product revealed that, the reaction carried out in presence of ethanol emerged as a suitable solvent condition for this reaction and isolated product 78% yield (Table I, entry 9). To check the role of the



Scheme I — Synthesis of hippuric acid and azlactones

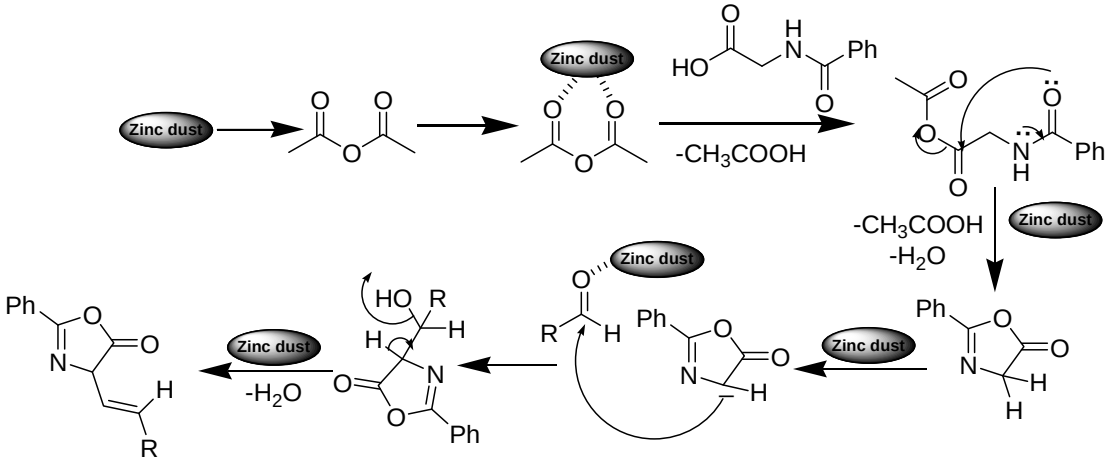
Table I — Optimization of the solvent and catalyst

Entry	Solvent*	Catalyst (mmol)	Time (min)	Yield[a] (%)
1	$CHCl_3$	1	45	38
2	CH_2Cl_2	1	40	40
3	CH_3CN	1	45	44
4	THF	1	40	48
5	MeOH	1	55	43
6	DMF	1	60	32
7	1,4-dioxane	1	70	30
8	Neat	1	25	58[d]
9	EtOH	1	35	78
10	EtOH	2	15	91[b,d]
11	EtOH	1	15	91[d]
12	Neat	1	35	48
13	EtOH	1.5	15	91[c]

* Solvent employed (3mL).
[a] Yields isolated
[b] 2mmol of zinc dust employed for the reaction
[c] 1.5mmol of zinc dust employed for the reaction
[d] Reaction carried out at 60°C

temperature on the rate of the reaction, the reaction is carried out by external heating near to boiling point of the solvent (60 °C), surprisingly the reaction at 60 °C completed within 15min as monitored by the TLC and isolated excellent yield up to 91% of the product (Table I).

To validate the importance of the solvent required in the present protocol, reaction carried out in model reaction compared with solvent-free (Table I, entry 8) the reaction carried out in presence of ethanol gave high yield (Table I, entry 11) compare to solvent-free condition. Over all the above reactions revealed that reaction required presence of a catalyst and Ac_2O with suitable solvent resulted efficient synthesis of azlactone. To check the optimal amount of zinc dust required for the catalysis, we examined series of reaction in a model reaction. The amount of zinc dust started 1mmol (66mg), 1.5 mmol and 2mmol of zinc dust (Table I). After reaction completion, the final product isolated revealed that, the reaction carried out in zinc dust, 1.5 and 2 mmol scale reaction not observed higher yield compare to 1mmolzinc dust used. Thus increased catalyst amount has no effect on the isolation of the higher product yield. From this experiment, we determined minimal amount of zinc dust required for the 1mmol scale reaction is 66mg (1mmol). To check the reaction compatibility and tolerance, various substituted benzaldehyde derivatives are subjected for the reaction. The method found to be tolerable to various substituted aromatic aldehyde gave moderate to excellent yields. To examine present protocol to alkyl aldehyde, the reaction is designed for the condensation of acetaldehyde with hippuric acid in presence of zinc dust at optimized condition at 1hr reflux resulted only 24% yield of product isolation. A plausible mechanism has been depicted for the zinc dust catalysed synthesis of 4-arylidene-2-phenyl-5-(4*H*)-oxazolones in Scheme II. The mechanism suggests that, initially the reaction proceeds *via* activating carbonyl group of acetic anhydride followed by nucleophilic addition of hippuric acid and cyclization takes place at oxygen centre eliminating acetic acid and water by forming 2-phenyl-5-oxazolone intermediate. Further, zinc dust is utilized in the deprotonating of formed intermediate, which produces oxazolone anion to which activated carbonyl compounds have been added to form the corresponding product. It seems that heating the reaction mixture accelerates the intermediate formation and therefore, accelerates the overall reaction accordingly. Pure product is obtained on recrystallizing the crude product using absolute alcohol, which is pure enough to collect spectral data without further purification like column chromatography (Table II). The homogeneity of the product 3a was confirmed by FT-IR, ^{1}H-, ^{13}C NMR spectroscopy and mass spectrometry analysis. The data shows characteristic peaks for FT-IR spectrum of product 3a at 1795.86 (CO), 1650.13 (C=N), 1563.43, 1443.11, 1325.62, 1160.36, 762.75, 652.12, ^{1}H NMR spectrum of product 3a exhibited characteristic singlet at 7.26 for 1 vinylic H, multiplet from 7.46 to 7.64 for 5 aromatic hydrogens present on aldehydic ring, multiplet from 8.18 to 8.23 for 5 aromatic hydrogens present on oxazolone ring. Characteristic peaks for ^{13}C NMR are as follows 125.63, 128.40, 128.92, 131.20, 131.80, 132.47, 133.36 and 133.54. HR-MS spectra shows m/z value at 250.0868 Da for $(M+H)^+$peak. By continuing our work, which is



Scheme II — Plausible mechanism for the zinc dust catalysed synthesis of 4-arylidene-2-phenyl-5-(*4H)*-oxazolones

Table II — Physical data of 4-arylidene-2-phenyl-5(4*H*)-oxazolones (**3a-l**)

Entry	Ar	Time (min)	Yield [a] (%)	Product	m.p. (°C)	
					Found	Reported
1	C_6H_5	15	91	**3a**	167-169	169[27]
2	4-ClC_6H_4	15	92	**3b**	188-190	190[47]
3	4-$OMeC_6H_4$	20	91	**3c**	155-157	154[47]
4	4-BrC_6H_4	20	88	**3d**	195-197	194-195[48]
5	4-$CH_3C_6H_4$	15	89	**3e**	168-169	167-168[27]
6	4-FC_6H_4	20	87	**3f**	183-184	183-185[27]
7	4-$N(Me)_2C_6H_4$	20	88	**3g**	212-214	212-214[49]
8	4-$NO_2C_6H_4$	15	89	**3h**	238-240	241[47]
9	2-BrC_6H_4	22	87	**3i**	144-145	144[49]
10	2-ClC_6H_4	20	89	**3j**	151-153	153[47]
11	2-$NO_2C_6H_4$	18	88	**3k**	163-164	164-165[60]
12	3,4-$(OMe)_2$	20	89	**3l**	151-153	152-154[60]

[a] Yields refers to isolated yields

Table III — Comparison of present method with different catalytic approaches reported to azlactone (3a) synthesis.

Sl. No	Catalyst	Condition	Time	Yield (%)	Ref.
1	Iodine	MW	65 sec	94	51
2	Mg/Al_2O_3	MW	5 min	90	52
3	K_3PO_4	80°C	30 min	93	53
4	$[C_6CmIm)_2]_2W_{10}O_{32}2H_2O$	US, RT	24 min	85	54
5	$Ca(OAc)_2$	MW	5 min	97	55
6	ZnO	RT	10 min	90	37
7	Fe_2O_3 nanoparticles	US, RT	10 min	95	56
8	$[Bmim]_3\ PW_{12}O_{40}$	80°C	75 min	89	57
9	[Bmim]OH	RT	90 min	87	58
10	[Bmim]OH@agar	RT	20 min	90	59
11	Zinc dust	60°C	15 min	91	This work

mainly focused on the establishment of simple, convenient and eco-friendly protocols to pharmacologically potent skeleton synthesis[61-67]. In the present study we have developed an efficient protocol for the synthesis of 4-arylidene-2-phenyl-5(4*H*)-oxazolones in mild conditions. In order to show merits of the present approach, we have summarized some of the previous literature reports of 4-benzylidene-2-phenyloxazol-5(4*H*)-one synthesis in Table III, which describes the merits of the present method like inexpensive catalyst, no hazardous solvent usage, shorter reaction time, easier workup, product isolation, good to excellent. Further, to check the compatibility of present method in multi gram scale synthesis, 4-benzylidene-2-phenyloxazol-5(4*H*)-one chosen, synthesis by taking benzaldehyde (1.02mL, 10 mmol), hippuric acid (1.79gm, 10 mmol), Ac_2O (3.3mL, 33mmol), zinc dust (1gm) EtOH (15 mL). The reaction mixture refluxed at 60°C for 40 min, and monitored by TLC, after reaction completion, filtered to separate the catalyst cooled to RT, and washed with hot ethanol and recrystallized by absolute alcohol gave, product isolation of 84% yield. The separated catalyst washed in a suitable solvent, dried, and reused for at least two cycles of gram scale synthesis. The reaction found to be faster reaction, easier work up and resulted high yield of product isolation.

Material and Methods

Laboratory grade chemicals were purchased from commercial sources and used as received without further purification. Melting points were determined in open capillaries and are uncorrected. FT-IR spectra were recorded in KBr pellets on a Shimadzu spectrometer. ^{1}H-, ^{13}C NMR spectra were recorded on a Bruker400 MHz spectrometer using TMS as an internal standard. LC-MS spectra were recorded in Waters; Synapt G2 High detection Mass spectrometry and HR-MS spectra were recorded in Waters; XEVOG2 XS Q-TOF Mass spectrometry. The progress of the reaction monitored by TLC, yields referred to isolated pure products.

Experimental Section

General procedure for the synthesis of azlactone derivatives

In a dry 50 mL RB flask taken aldehyde (1 mmol), hippuric acid (1mmol), acetic anhydride (3.3 mmol) in EtOH (3mL) followed by Zinc dust (1 mmol). The mixture was stirred at 60°C temperature for appropriate time (Table I). TLC monitored the progress of the reaction. After completion of the reaction, hot ethanol was added, the catalyst separated by filtration, and final product obtained in pure form by recrystallization using absolute ethanol without using chromatography technique.

Spectral data of representative compounds

4-Benzylidene-2-phenyloxazol-5(4*H*)-one (Table II, **3a**): Yellow solid. Yield 92%. m.p. 167-169°C. FT-IR (KBr): 1795.86, 1650.13, 1563.43, 1443.11, 1325.62, 1160.36, 762.75, 652.13 cm^{-1}; ^1H NMR ($CDCl_3$): δ 7.26 (s, 1H, -CH=), 7.46–7.64 (m, 5H, ArH), 8.18–8.23 (m, 5H, ArH); ^{13}C NMR ($CDCl_3$): δ 125.63, 128.40, 128.92, 131.20, 131.80, 132.47, 133.36, 133.54; HR-MS: Calcd $[M]^+$ 249.2650. Obsd *m/z* 250.0992 $(M+H)^+$

4-(4-Chlorobenzylidene)-2-phenyloxazol-5(4*H*)-one (Table II, **3b**): Yellow solid. Yield 94%. m.p. 188-190°C; FT-IR (KBr): 2917.74, 1795.06, 1767.50, 1652.82, 1582.56, 1484.27, 1327.73, 1300.07, 1161.15, 1070.72, 835.91 cm^{-1}; ^1H NMR ($CDCl_3$): δ 7.18 (1H, s), 7.45 (d, 2H), 7.51-7.62 (m, 3H), 8.14 (d, 2H), 8.14 – 8.17 (m, 2H); ^{13}C NMR ($CDCl_3$): δ 125.44, 128.44, 128.98, 129.23, 130.01, 131.77, 132.01, 133.50, 137.27, 163.90, 167.35; LC-MS Calcd $[M]^+$ 283.7091. Obsd *m/z* 284.0388$(M+H)^+$.

4-(4-Methoxybenzylidene)-2-phenyloxazol-5(4*H*)-one (Table II, **3c**): Orange solid. Yield 93%. m.p. 155-157°C; FT-IR (KBr): 2843.99, 1788.70, 1653.50, 1599.46, 1512.08, 1487.83, 1111.72, 924.55, 861.95 cm^{-1}; ^1H NMR ($CDCl_3$): δ 3.87 (s, 3H), 6.95-6.99 (d, 2H, *J* = 8.8 Hz), 7.26 (1H, s), 7.43 – 7.57 (m, 3H), 8.18 – 8.19 (m, 2H), 8.21 (d, 2H, *J* = 8.8 Hz); ^{13}C NMR ($CDCl_3$): δ 55.56, 113.74, 114.53, 127.06, 128.1, 128.8, 131.8, 132.31, 134.58, 162.2, 167.9; LC-MS: Calcd $[M]^+$ 279.2900. Obsd *m/z* 280.0536 $[M+H]^+$.

4-(4-Bromobenzylidene)-2-phenyloxazol-5(4*H*)-one, (Table II, **3d**): Yellow solid. Yield 90%. m.p. 195-197°C; FT-IR (KBr): 3418.49, 2389.63, 1767.50, 1652.82, 1582.56, 1484.39, 1326.25, 1237.73, 1161.15, 1108.49, 835.91 cm^{-1}; ^1H NMR ($CDCl_3$): δ 7.17 (1H, s), 7.25 (d, 2H), 7.52 - 7.65 (m, 3H), 8.06 - 8.08 (d, 2H), 8.17 – 8.19 (m, 2H); ^{13}C NMR ($CDCl_3$): δ 125.45, 125.90, 128.47, 129.09, 130.08, 131.77, 132.22, 133.64, 133.79, 163.97, 167.35 ; LC-MS: Calcd $[M]^+$ 328.1601. Obsd *m/z* 328.9163 [M+H]+.

Conclusion

We have demonstrated a simple, convenient and efficient approach for the synthesis of 4-arylidene-2-phenyl-5(4*H*)-oxazolones from the condensation of arylaldehydes, and hippuric acid catalysed by heterogeneous zinc dust catalyst. The method is tolerable to various substituted aromatic aldehydes. The present approach has several merits over reported protocols like in-expensive catalyst, eco-friendly approach, easy workup, good to excellent yield isolation and shorter reaction times. The crude final product is easily recrystallized using absolute alcohol and this protocol not required further chromatographic purification.

Supplementary Information

Supplementary information is available in the website http://nopr.niscair.res.in/handle/123456789/60.

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