



RAPID ONE-POT MICROWAVE ASSISTED SYNTHESIS OF 4-ARYLIDENE-2-PHENYL-5(4H)-OXAZOLONES OR AZLACTONES

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Abstract:

Rapid, environmentally benign one pot synthesis series of 4-arylidene-2-phenyl-5(4H)-oxazolones or azlactone (**3a-q**) catalyzed by sodium acetate starting from easily available reactant molecules under microwave irradiation method. The reaction performed in combination of water and polyethylene glycol (PEG-400) as green solvent under the simple non-conventional technique with good to excellent yields (96-99 %). The cyclisation followed by condensation of hippuric acid **1** with various aldehydes **2 (a-q)** catalyzed by sodium acetate and catalytic amount of acetic anhydride. The final products were characterized by FTIR, ¹HNMR, Mass and compared there reported method.

Keywords: Hippuric acid, Aldehyde, Oxazolone or Azlactones, Water-PEG, Microwave irradiation technique.

Introduction:

4-Arylidene-2-phenyl-5(4H)oxazolones, which are also known as azlactones, are important intermediates of drug and or medicine from the several small molecules, such as amino acidsⁱ, peptides^{v, vi}, 2,2 di-substituted- 2H-oxazol-5-ones with region and stereo control^{vii}, precursors for other heterocyclic systems^{viii}, Furthermore, oxazolones have been reported to exhibit a wide range of pharmaceutical properties^{ix}, including anticancer^x, antitumor, antimicrobial^{xi}, anti-inflammatory^{xii}, antiviral^{xiii} and anti-HIV^{xiv} activities. These compounds can also be used as molecular photo switches^{xv} and optical sensors for the measurements of pH^{xvi}, as well as biosensor-coupling and photosensitive composition devices for protein analysis^{xvii}. Based on these importance, the development of new methods for the facile and environmental friendly synthesis of azlactones catalyzed by sodium bi-carbonate and PEG-Water as green catalyst and solvent.

Previously, several methods have been reported for the synthesis of oxazolone, for example, synthesis of a series of azlactones by the condensation of hippuric acid with various aromatic

aldehydes in the presence of acetic anhydride under ultrasonic irradiation conditions^{xviii}. Azlactones may also be synthesized under solvent-free conditions using Nano silica-supported tungstophosphoric acid^{xix} or using calcium acetate^{xx}, aluminum oxide^{xxi}, and neutral alumina^{xxii} under microwave irradiation conditions or organic inorganic hybrid polyoxometalates as a catalyst^{xxiii}, ytterbium (III) triflate as a catalyst^{xxiv}, under solvent free condition. By the important route for the synthesis of Azlactones-Erlenmeyer method^{xxv}, which involves the condensation of aldehydes with hippuric acid in the presence of sodium acetate and acetic anhydride and starting from hippuric acid^{xviii-xxv}. All these synthetic methods have been used hazardous catalyst, solvent and cost effective method etc. In earlier our research works for the synthesis of some heterocycles in combination of water-PEG as green solvent^{xxvi}.

It was envisaged that a totally green approach one-pot, one-stage method for the series of 4-arylidene-2-phenyl-5(4H)-oxazolones or azlactones in PEG-Water mediated catalyze by sodium acetate and acetic anhydride directly from hippuric acid and available various types aldehyde (Figure 1).

Experimental Method:

Materials

The starting chemicals were purchased from Sigma Aldrich. All of the melting points were determined in open head capillary tubes a simple melting apparatus. These data have been presented as the uncorrected values. IR spectra were recorded as KBr disks on a PerkinElmer RXIFTIR spectrometer. ¹H NMR spectra were measured on a Varian Gemini 300 MHz spectrometer (Palo Alto, CA, USA). Chemical shifts (δ) have been expressed in ppm downfield from TMS, which was used as an internal standard. H NMR spectra were recorded in DMSO-d₆ and the coupling constants (J) reported in Hz. Mass spectra were recorded QUART-MASS JEOL-Accu TOF JMS-T 100LC Mass spectrometer 70 eV. All of the reactions were monitored by thin-layer chromatography (TLC) using aluminum TLC sheets coated with silica gel F254 (Merck, Darmstadt, Germany). TLC was also used to assess the purity of the synthesized

General procedure for the preparation of azlactones 3a–q:

A mixture of hippuric acid (1.0 mmol), aromatic aldehyde (1.0 mmol), sodium acetate (0.5 mmol) was mixed in combination of Water-PEG (2:1) in the presence of catalytic amount of acetic anhydride stirred for a few minutes and were reflux and or microwave irradiate (Table 1). Upon completion of the reaction, as determined by TLC, the reaction mixture turned to a yellow solid, which was washed with cold water and recrystallized from ethanol to give the desired azlactone. The structures of the azlactones were confirmed based on a comparison of their melting point, IR, HNMR and MS data with those from the literature.

Results and Discussion:

We synthesized, reported series of 4-arylidene-2-phenyl-5(4H)-oxazolones starting from the model reaction of hippuric acid (1.0 mmol) **1**, aromatic aldehyde (1.0 mmol) **2 (a-q)**, sodium acetate (0.5 mmol) in solvent free and using solvent like methanol, ethanol, IPA, toluene, xylene, DCM, TCM, water, PEG-400 and combination of water-PEG and catalytic amount of acetic anhydride at reflux and microwave irradiation condition (Table 1). Herein, we observed good yield was obtained in combination of Water-PEG-400 (1:1) (Table 1, entry **11**). As we increases the quantity of water in PEG-400 as Water-PEG-400 (2:1) then yield of the product were increases as yield 98 % (Table 1, entry **13**) in less time of reaction compared to other optimizing of solvent (Table 1). The same reactions were performed under the microwave energy power at 350 watt, 90 °C. Herein we observed that good to excellent

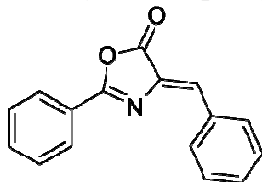
yield was obtained in very less time of reaction (Table 1, entry 14), as we increased the amount of water even also increasing time of reaction the yield of product were decreased (Table 1, entry 15).

Thus, all the derivatives of 4-arylidene-2-phenyl-5(4*H*)-oxazolones / azlactones were synthesized in combination of Water-PEG-400 (2:1) catalyzed by sodium acetate under microwave irradiation condition with better to excellent yields of the product 96-99 % (Table 2). The unsubstituted and electron withdrawing group (NO₂, Cl) to aromatic aldehyde gave excellent yield (Table 2, entry 1, 5) compared to other electron withdrawing and donating groups (Table 2).

Characterization data of some synthesized products:

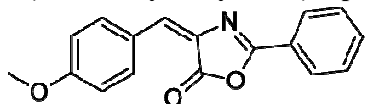
Spectral Characterization data 3a-q:

4-Benzylidene-2-phenyl-5(4*H*)-oxazolone (3a):



Mp. 171; IR (KBr): 1791, 1769 (C=O), 1654 (C=N), 1594 (C=C).; ¹H NMR (300 MHz, DMSO-d₆): δ 7.35 (s, 1H, CH=C), 7.33–7.75 (m, 6H, Ar-H), 8.13 (d, 2H, J = 7.5 Hz), 8.30 (d, 2H, J = 7.8 Hz).; MS (ESI) m/z (%): 249 (M⁺, 100).

4-(4-Methoxybenzylidene)-2 phenyl-5(4*H*)-oxazolone (3b):

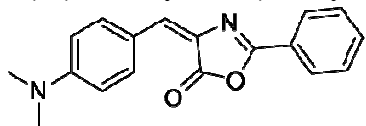


Mp. 158; IR (KBr): 1791, 1768 (C=O), 1655 (C=N), 1605 (C=C).; ¹H NMR (300 MHz, DMSO-d₆): δ 3.88 (s, 3H, CH₃), 7.11 (d, 2H, J = 9.0 Hz), 7.64 (d, 2H, J = 7.5 Hz), 7.69 (d, 1H, J = 6.9 Hz), 8.11 (d, 2H, J = 6.9 Hz), 8.30 (d, 2H, J = 9.0 Hz). For the E-isomer (71 %): 7.33 (s, 1H, CH=C), for the Z-isomer (29 %): 7.60 (s, 1H, CH=C).; MS (ESI) m/z (%): 279 (M⁺, 88), 105 (100).

4-(4-Chlorobenzylidene)-2-phenyl-5(4*H*)-oxazolone (3c):

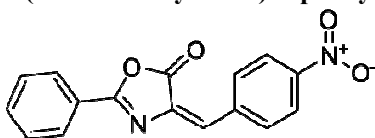
Mp. 192; IR (KBr): 1798, 1769 (C=O), 1654 (C=N), 1589 (C=C).; ¹H NMR (300 MHz, DMSO-d₆): δ 7.50 (d, 1H, J = 7.5 Hz), 7.61 (d, 1H, J = 8.7 Hz), 7.66 (d, 1H, J = 7.5 Hz), 7.73 (d, 1H, J = 7.5 Hz), 7.94 (d, 1H, J = 7.5 Hz), 8.14 (d, 2H, J = 7.5 Hz), 8.33 (d, 2H, J = 8.7 Hz).; For the E-isomer (86 %): 7.37 (s, 1H, CH=C), for the Z-isomer (14 %): 7.47 (s, 1H, CH=C). MS (ESI) m/z (%): 285 (M⁺⁺ 2, 30), 283 (M⁺, 90), 105 (100).

4-(4-(Di-methylamino)benzylidene)-2-phenyl-5(4*H*)-oxazolone (3d):



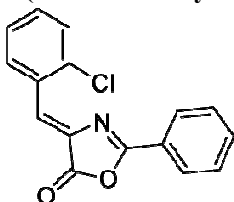
Mp. 206; IR (KBr): 1759, 1765 (C=O), 1649 (C=N), 1589 (C=C).; ¹H NMR (300 MHz, DMSO-d₆): δ 3.07 (s, 6H, 2CH₃), 6.83 (d, 2H, J = 9.0 Hz), 7.33 (s, 1H, CH=C), 7.58–7.66 (m, 3H), 8.06 (d, 2H, J = 6.6 Hz), 8.17 (d, 2H, J = 8.7 Hz).; MS (ESI): m/z (%): 292 (M⁺, 91), 105 (100).

4-(4-Nitrobenzylidene)-2-phenyl-5(4H)-oxazolone (3e):



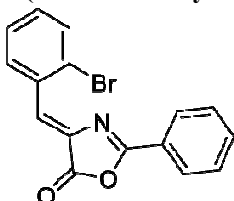
Mp. 241; IR (KBr): 1755, 1688 (C=O), 1625 (C=N), 1589 (C=C). ¹H NMR (300 MHz, DMSO-d₆): δ 7.26–7.58 [m, 6H, (5Ar-H + 1CH=C)], 7.74 (d, 2H, J = 7.5 Hz), 7.88 (d, 2H, J = 7.2 Hz).; MS (ESI) m/z (%): 294.15 (M⁺, 0.5), 105 (100).

4-(2-Chlorobenzylidene)-2-phenyl-5(4H) oxazolone (3f):



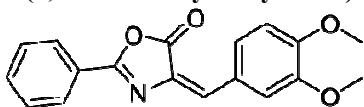
Mp. 154; IR (KBr): 1795 (C=O), 1689, 1655 (C=N), 1602 (C=C).; ¹H NMR (300 MHz, DMSO-d₆): δ 7.46 (s, 1H, CH=C), 7.50 (d, 2H, J = 7.8 Hz), 7.57–7.67 (m, 3H), 7.94 (d, 2H, J = 7.2 Hz), 8.15 (d, 1H, J = 6.9 Hz), 8.88 (d, 1H, J = 8.1 Hz).; MS (ESI) m/z (%): 285 (M⁺⁺², 7), 283 (M⁺, 21), 105 (100).

4-(2-Bromobenzylidene)-2-phenyl-5(4H)-oxazolone (3g):



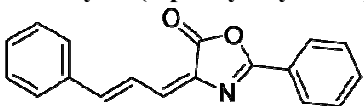
Mp. 146; IR (KBr): 1792 (C=O), 1653 (C=N), 1580 (C=C).; ¹H NMR (300 MHz, DMSO-d₆): δ 7.40–7.51(m, 2H), 7.57–7.67 (m, 3H, (2Ar-H + 1CH=C)), 7.74 (d, 1H, J = 7.5 Hz), 7.80 (d, 1H, J = 8.1 Hz), 7.94 (d, 1H, J = 7.2 Hz), 8.14 (d, 1H, J = 7.2 Hz), 8.86 (d, 1H, J = 8.1 Hz).; MS (ESI) m/z (%): 328 (M⁺, 5.6), 330 (M⁺⁺², 4.8), 327 (27.3), 329 (26.9), 248 (59), 105 (100).

4-(3,4-Dimethoxybenzylidene)-2-phenyl-5(4H)-oxazolone (3h):



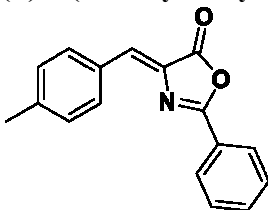
Mp. 153; IR (KBr): 1778 (C=O), 1653 (C=N), 1589 (C=C).; ¹H NMR (300 MHz, DMSO-d₆): δ 3.86 (s, 3H, OMe), 3.88 (s, 3H, OCH₃), 7.13 (d, 1H, J = 8.7 Hz), 7.32 (s, 1H, CH=C), 7.60–7.73 (m, 3H), 7.81 (d, 1H, J = 9.0 Hz), 8.08–8.14 (m, 3H).; MS (ESI) m/z (%): 309.15 (M⁺, 6.0), 105 (100).

2-Phenyl-4-(3-phenylallylidene)-5(4H)-oxazolone (3i):



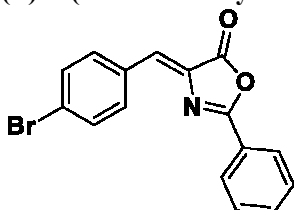
Mp. 131; IR (KBr): 1779 (C=O), 1646 (C=N), 1589 (C=C).; ¹H NMR (300 MHz, DMSO-d₆): δ 7.27 (d, 1H, CH=C, J = 11.4 Hz), 7.36–7.42 (m, 4H, Ar-H), 7.57–7.68 (m, 7H, (6 Ar-H + 1 CH=C)), 8.08 (d, 1H, CH=C, J = 12.0 Hz).; MS (ESI) m/z (%): 275.10 (M⁺, 12.57), 105 (100).

(Z)-4-(4-methylbenzylidene)-2-phenyloxazol-5(4H)-one (4j):



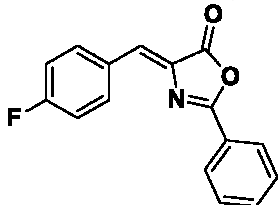
Mp.142-143°C; IR (KBr) $\nu_{\max}/\text{cm}^{-1}$: 1509 (C=C-Ar), 1790 (C=O), 1652 (C=N), 2763 (ArC-H).; ^1H NMR (300 MHz, CDCl_3): δ (ppm) 7.89(s, 1H, H-C=C), 7.58(d, 2H, Ar-H), 7.16(d, 2H, Ar-H), 2.30(s, 3H, CH_3 -Ar) 7.52-7.95(m, 5H, Ar-H).; Molecular Formula $\text{C}_{16}\text{H}_{11}\text{NO}_2$.; MS (ESI): m/z (%) 264.09 [M+H].

(Z)-4-(4-bromobenzylidene)-2-phenyloxazol-5(4H)-one (4k):



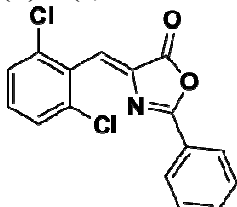
Mp. 197-198°C IR (KBr) $\nu_{\max}/\text{cm}^{-1}$: 1508 (C=C-Ar), 1791 (C=O), 1652 (C=N), 2763 (ArC-H), 689 (Ar-Br).; ^1H NMR (300 MHz, CDCl_3): δ (ppm) 7.89(s, 1H, H-C=C), 7.63(d, 2H, Ar-H), 7.53(d, 2H, Ar-H), 7.53-7.96 (m, 5H, Ar-H).; MS (ESI): m/z (%) 328 [M+H].

(Z)-4-(4-fluorobenzylidene)-2-phenyloxazol-5(4H)-one (4l):



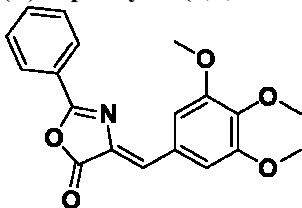
Mp.183-184°C; IR (KBr) $\nu_{\max}/\text{cm}^{-1}$: 1508 (C=C-Ar), 1792 (C=O), 1653 (C=N), 2762 (ArC-H), 1089 (Ar-F).; ^1H NMR (300 MHz, CDCl_3): δ (ppm) 7.89(s, 1H, H-C=C), 7.18(d, 2H, Ar-H), 7.73(d, 2H, Ar-H), 7.53-7.90 (m, 5H, Ar-H).; MS (ESI): m/z (%) 268.07 [M+H].

(Z)-4-(2,6-dichlorobenzylidene)-2-phenyloxazol-5(4H)-one (4m):



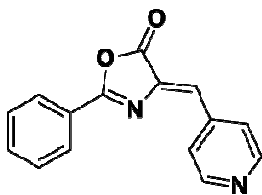
Mp.160-162°C; IR (KBr) $\nu_{\max}/\text{cm}^{-1}$: 1508 (C=C-Ar), 1793 (C=O), 1650 (C=N), 2762 (ArC-H), 789 (Ar-Cl).; ^1H NMR (300 MHz, CDCl_3): δ (ppm) 7.63(s, 1H, H-C=C), 7.50-7.96(m, 5H, Ar-H), 7.30(d, 2H, Ar-H), 7.49(dd, 1H, Ar-H).; MS (ESI): m/z (%) 318.01 [M+H].

(Z)-2-phenyl-4-(3,4,5-trimethoxybenzylidene)oxazol-5(4H)-one (4n):



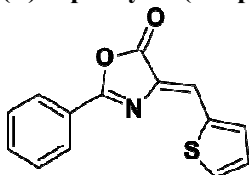
Mp.200-204°C; IR (KBr) $\nu_{\max}/\text{cm}^{-1}$:1465(Ar-OMe), 1510 (C=C-Ar), 1792 (C=O), 1652 (C=N), 2763 (ArC-H).; ^1H NMR (300 MHz, CDCl_3): δ (ppm) 7.38(s, 1H, H-C=C), 6.72(s,2H, Ar-H), 3.80(s, 9H, CH_3 -O-Ar), 7.51-7.94(m, 5H, Ar-H).; MS (ESI): m/z (%) 340.34 [M+H].

(Z)-2-phenyl-4-(pyridin-4-ylmethylene)oxazol-5(4H)-one (4o):



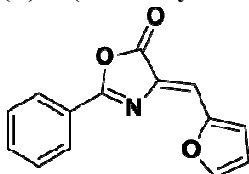
Mp.168-170°C IR (KBr) $\nu_{\max}/\text{cm}^{-1}$: 1509 (C=C-Ar), 1792 (C=O), 1465(C=N),1652 (C=N), 2763 (ArC-H); ($^{\circ}\text{C}$); ^1H NMR (300 MHz, CDCl_3): δ (ppm) 7.38(s, 1H, H-C=C), 8.52(d, 2H, Ar-H), 7.43(d, 2H,Ar-H), 7.51-7.95(m, 5H, Ar-H).; MS (ESI): m/z (%) 251.07 [M+H].

(Z)-2-phenyl-4-(thiophen-2-ylmethylene)oxazol-5(4H)-one (4p):



Mp.170-171°C; IR (KBr) $\nu_{\max}/\text{cm}^{-1}$: 1510 (C=C-Ar), 1792 (C=O), 1652 (C=N), 2762 (ArC-H), 837(C-S-C).; ^1H NMR (300 MHz, CDCl_3): δ (ppm) 7.38(s, 1H, H-C=C), 8.08(d, 1H, S-CH=CH), 7.51(dd, 1H, Ar-H), 7.70(d,1HAr-H), 7.51-7.95(m, 5H, Ar-H).; MS (ESI): m/z (%) 256.04 [M+H].

(Z)-4-(furan-2-ylmethylene)-2-phenyloxazol-5(4H)-one (4q):

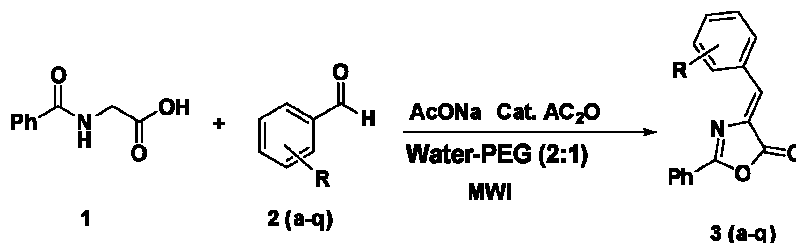


Mp.171-173°C; IR (KBr) $\nu_{\max}/\text{cm}^{-1}$: 1510 (C=C-Ar), 1793 (C=O), 1652 (C=N), 2763 (ArC-H), 1085(C-O-C).; ^1H NMR (300 MHz, CDCl_3): δ (ppm) 7.13(s, 1H, H-C=C), 8.14(d, 1H, -O-CH=CH), 6.83(dd, 1H, Ar-H), 7.60(d,1HAr-H), 7.51-7.96(m, 5H, Ar-H).; MS (ESI): m/z (%) 240.05 [M+H].

Conclusion:

In summary, we have developed a simple, rapid, efficient and environmental benign one-pot method for synthesis of azlactones or oxazolones using a combination of solvent as Water-PEG catalyzed by sodium acetate under microwave irradiation technique. The key advantages of this strategy over other conventional methods include its simple, non-hazardous catalyst, solvent as well as its facile work-up, short reaction time, high yield thus environmental friendly.

Figures and Scheme:



Reaction Scheme: Synthesis of 4-arylidene-2-phenyl-5(4H)-oxazolones or azlactones catalyzed by sodium acetate and acetic anhydride.

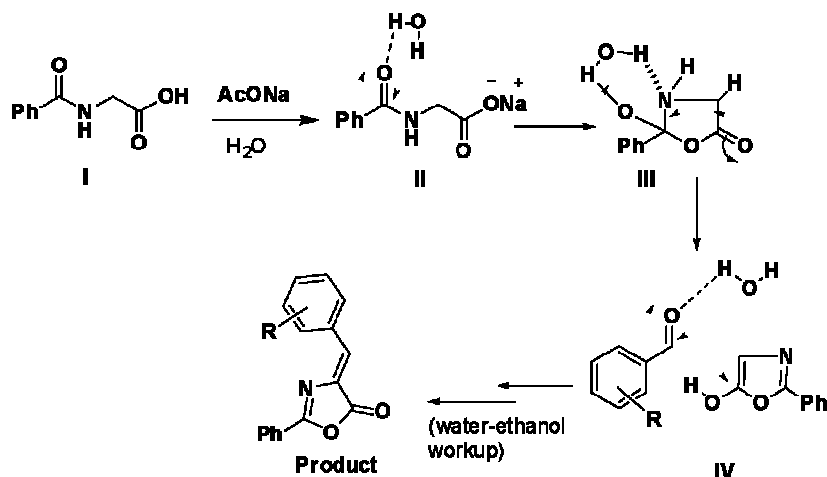


Figure 2. Plausible mechanistic path for the synthesis of 4-arylidene-2-phenyl-5(4H)-oxazolones or azlactones catalyzed by sodium acetate and acetic anhydride.

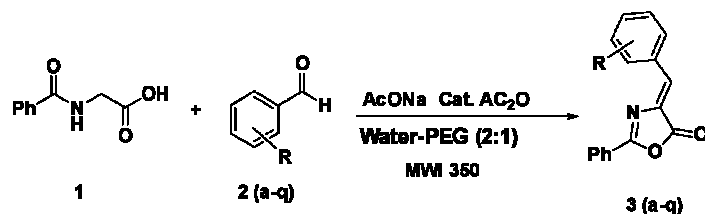
7. Tables:

Table 1. Optimization of solvent for the synthesis of 4-arylidene-2-phenyl-5(4H)-oxazolones / azlactones.

Sr. no.	Solvent	Reaction condition	Time (hr& min)	Yield ^a (%)
1	Without	Reflux	3	00
2	Ethanol	Reflux	3	42
3	Methanol	Reflux	3	46
4	Iso.pr.alcohol	Reflux	3	36
5	Toluene	Reflux	3	32
6	Xylene	Reflux	3	36
7	DCM	Reflux	3	30
8	TCM	Reflux	3	36
9	Water	Reflux	2.5	50
10	PEG	Reflux	2.5	56
11	Water-PEG (1:1)	Reflux	2.5	62
12	Water-PEG (1:2)	Reflux	2.5	49
13	Water-PEG (2:1)	Reflux	2	98
14	Water-PEG (2:1)	MWI	3 min	99
15	Water-PEG (3:1)	MWI	4	79

^a**Reaction Condition:** hippuric acid (1.0 mmol), aromatic aldehyde (1.0 mmol), sodium acetate (0.5 mmol) was mixed in solvent in the presence of catalytic amount of acetic anhydride stirred and were irradiation of microwave energy power at 350 watt.

Table 2. Synthesis of compound 3(a-i) with physical data:



Sr. no.	R	MWI	Yield ^b (%)	Melting point (°C) Reported [Lit.]	Melting point (°C) Found
1	H	3	99	166-168[xxi]	171
2	4-OMe	3	96	155-156[ix, xxvii]	158
3	4-Cl	4	98	189-190[ix, xxvii]	192
4	4-NMe ₂	3	96	205-206[ix]	206
5	4-NO ₂	3	98	238-240[iv]	241
6	2-Cl	4	98	150-152[iv]	154
7	2-Br	4	96	144-145[viii]	146
8	3,4-(OMe) ₂	3	96	148-150[xxi]	153
9	4-CH=CH-	4	96	130-131[xxi]	131
10	4-Me	3	98	142-143[xxx, xxxi]	141-142
11	4-Br	3	96	197-198[xxxii]	197-198
12	4-F	3	96	183-184[xxxii]	183-184
13	2,5-(Cl) ₂	4	96	160-162[xxxii]	160-162
14	3,4,5-(OMe) ₃	3	96	200-201[xxxii]	200-204
15	Pyridyl	4	98	141-142[xxx]	168-170
16	thiophen-2-yl	4	96	171-172[xxxii]	170-171
17	furan-2-yl	4	96	168-169[xxviii]	171-173

^b**Reaction Condition:** hippuric acid (1.0 mmol), aromatic aldehyde (1.0 mmol), sodium acetate (0.5 mmol) was mixed in combination of Water-PEG (2:1) in the presence of catalytic amount of acetic anhydride stirred and were microwave irradiated.

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