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**RESEARCH ARTICLE**

# An Efficient Three Component Synthesis of 1*H*-Indazolo[2, 1-*b*]phthalazine-triones Catalyzed by Orthophosphoric Acid in Water-Phenol System

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**Abstract**: Simple method for the synthesis of 1*H*-Indazolo [2,1-*b*]phthalazine-triones via one pot three component condensation of phthalhydrazide, aldehyde and dimedone catalyzed by orthophosphoric acid in water–phenol system is reported. This synthetic procedure proves with extremely lower reaction timings, high yields, recyclable of catalyst and green solvent system.

**Keywords:** 1*H*-Indazolo[2,1-*b*]phthalazinetrione, Multi component reactions, Orthophosphoric acid, Water-phenol system

## Introduction

Multi-component reactions are attractive and interesting transformations in organic synthesis due to atom-economy, convergent character, operational simplicity, structural diversity and complexicity[1-7] these reactions are emerging as a dominating tool for the synthesis of biologically significant compounds[8,9].

In recent years, the synthesis of new heterocyclic compounds has been a subject of unlimited interest due to their vast applicability. Amongst, nitrogen heterocyclic containing a phthalazine moiety was reported to possess anticonvulsant[10], cardiotonic[11], vasorelaxant[12] antimicrobial[13], anti-inflammatory[14], antifungal[15] and anticancer[16,17] activities. Newly, three-component reactions of dimedone (5,5-dimethylcyclohexane-1,3-dione), an aldehyde and phthalhydrazide to give 1*H*-indazolo[2,1-*b*]phthalazine-triones have fascinated the interest of the synthetic organic chemists.

Many methods had been reported with different catalytic systems includingp-TSA[18], $Me_3SiCl$[19], silica sulfuric acid[20],[21] $H_2SO_4$,cyanuric chloride[22], heteropolyacids[23], *N*-halo sulphonamides[24], silica supported poly phosphoric acid[25],[26] $Mg(HSO_4)_2$, phosphor sulfonic acids[27], nano silica sulphuric acid[28], sulfonated polyethylene glycol[29] and boron sulfonic acid[30].

However, some of these methodologies have some experimental problems and drawbacks due to longer reaction times, requires high stoichiometric expensive reagents, hazardous solvents and struggles with a lot of workup procedures. To overcome these drawbacks, we developed an efficient and new eco–friendly methodology with better reagent orthophosphoric acid in water–phenol system for one pot three-component synthesis of 1*H*-indazolo[2,1-*b*]phthalazine-triones (Scheme 1).

## Experimental

A mixture of phthalhydrazide (1.1 mmol), aromatic aldehyde (1.2 mmol), 5,5-dimethyl 1,3-cyclohexane dimedone (1.0 mmol), orthophosphoric acid (0.2 mmol) and 15 mL of water-phenol [1:1 (in volume)] was agitated magnetically under reflux at 70 °C for a certain time (Table 1). The reaction was monitored by TLC using ethyl acetate methylene dichloride (9:1). After completion of the reaction the precipitate of reaction mixture was collected and filtered. Thereafter, it was washed with water and dried. Finally, the crude product was recrystallized from hot ethanol to yield pure products.

### Spectral data

*2,3,4,13-Tetrahydro-3,3-dimethyl-13-(3-methylphenyl)-1H-indazolo[1,2-b]phthalazine-1,6,11-trione (4a,$C_{24}H_{22}N_2O_3$)*

Yellow powder, m.p.: 229-230 °C ; IR (KBr): 1639,1616; 1419, 1356, 1,120, 993, 864 $cm^{-1}$; $^1H$ NMR (400 MHz, $CDCl_3$): 0.95 (s,6H, $2xCH_3$), 1.86 (s, 2H, $CH_2$ near double bond), 2.36 (m, 5H, $CH_2$ near keto group and was for Ar-$CH_3$ ), 5.1 (s, 1H, -NCH-Ar), 7.21-7.60 (m, 8H, Ar-H). $^{13}C$ NMR (125 MHz, CDCl3): d = 21.5, 28.5, 28.6, 34.9, 38.1, 51.0,65.0, 118.8, 124.1, 127.7, 27.9, 128.0, 128.6, 129.0,129.2, 129.6, 133.4, 134.4, 136.3, 138.3, 150.7, 154.2,156.1, 192.1 ppm; MS (EI): *m/z* = 387.50 $(M+H)^+$.

*13-(3,4-Dimethylphenyl)-2,3,4,13-tetrahydro-3,3-dimethyl-1H-indazolo[1,2-b]phthalazine-1,6,11-trione (4b,$C_{25}H_{24}N_2O_3$)*

Yellow powder, m.p.: 251-250 °C; IR (KBr): 1,635, 1,616, 1,419, 1,357, 1,315, 1,263, 1,120, 1,066, 993, 952, 864 $cm^{-1}$; 1.00 (s,6H, $2xCH_3$), 1.89 (s, 2H, $CH_2$ near double bond), 2.39 (m, 8H, $CH_2$ for near keto group and was for 2xAr-$CH_3$), 5.25 (s, 1H, -NCH-Ar), 7.42-7.62 (m, 7H, Ar-H). $^{13}C$ NMR (125 MHz, CDCl3): d = 19.6, 19.9, 28.5, 28.7, 34.7,38.1, 51.0, 64.9, 118.9, 124.5, 127.8, 127.9, 128.5, 129.0,129.2, 130.0, 133.4, 133.7, 134.4, 136.9, 137.2, 150.7,154.2, 156.1, 192.1 ppm; MS (EI): *m/z* = 401.16 $(M+H)^+$.

*2,3,4,13-Tetrahydro-3,3-dimethyl-13-(3-phenoxyphenyl)-1H-indazolo[1,2-b]phthalazine-1,6,11-trione (4c,$C_{29}H_{24}N_2O_4$)*

Yellow powder, m.p.: 188-191 °C; IR (KBr): 1,635,1,616, 1,452, 1,365, 1,311, 1,272, 1,240, 1,070, 985,864 $cm^{-1}$; $^1H$ NMR (500 MHz, CDCl3): =1.16 (s, 3H), 1.19 (s, 3H), 2.33 (s, 2H), 3.21 and 3.36 (AB system, J = 19.0 Hz, 2H), 6.41 (s, 1H), 6.88 (dd, J = 8.0, 2.0 Hz,1H), 6.98 (d, J = 8.0 Hz, 2H), 7.01 (t, J = 2.0 Hz, 1H),7.08 (t, J = 7.5 Hz, 1H), 7.22 (d, J = 8.0 Hz, 1H), 7.27-7.31 (m, 3H), 7.83-7.88 (m, 2H), 8.27–8.30 (m, 1H), 8.33–8.36 (m, 1H) ppm; $^{13}$C NMR (125 MHz, $CDCl_3$):d = 28.4, 28.7, 34.6, 38.0, 51.0, 64.6, 117.1, 118.3, 118.6, 119.1, 122.4, 123.4, 127.8, 128.0, 129.0, 129.1,129.7, 129.9, 133.6, 134.6, 138.4, 151.0, 154.4, 156.0,156.7, 157.6, 192.1 ppm; MS (ESI): *m/z* = 465.11 $(M+H)^+$.

### *13-(3-Bromophenyl)-2,3,4,13-tetrahydro-3,3-dimethyl-1H-indazolo[1,2-b]phthalazine-1,6,11-trione (4d,$C_{23}H_{19}BrN_2O_3$)*

Yellow powder, m.p.: 223-225 °C ; IR (KBr):1,635,1,618, 1,458, 1,419, 1,363, 1,309, 1,265, 1,120, 1,068, 952,864 cm$_{-1}$; $^1$H NMR (500 MHz, CDCl3): d = 1.20 (s, 3H),1.21 (s, 3H), 2.34 (s, 2H), 3.24 and 3.41 (AB system, J = 19.0 Hz, 2H), 6.39 (s, 1H), 7.22 (t, J = 8.0 Hz, 1H), 7.41 (d, J = 8.0 Hz, 2H), 7.47 (t, J = 2.0 Hz, 1H), 7.85-7.89 (m, 2H), 8.26–8.29 (m, 1H), 8.35–8.38 (m, 1H) ppm; $^{13}$C NMR (125 MHz, $CDCl_3$): d = 28.5, 28.6, 34.7, 38.0,50.9, 64.3, 117.1, 122.8, 126.4, 127.8, 128.1, 129.0, 129.8,130.2, 131.9, 133.7, 134.6, 138.7, 151.2, 154.4, 156.0,192.0 ppm; MS (ESI): *m/z* = 452.02 $(M+H)^+$.

### *2,3,4,13-Tetrahydro-3,3-dimethyl-13-[3-(trifluoro-methyl)phenyl]-1H-indazolo[1,2-b]phtha lazine-1,6,11-trione (4e,$C_{24}H_{19}F_3N_2O_3$)*

Yellow powder, m.p.: 216-217 °C; IR (KBr): 1,635,1,616, 1,359, 1,311, 1,263, 1,078, 983, 864 cm$^{-1}$; $^1$H NMR(500 MHz, $CDCl_3$): d = 1.21 (s, 3H), 1.22 (s, 3H), 2.34 (s, 2H), 3.24 and 3.43 (AB system, J = 19.0 Hz, 2H), 6.49 (s, 1H), 7.48(t, J = 8.0 Hz, 1H), 7.55 (d, J = 8.0 Hz, 1H), 7.60 (s, 1H),7.68 (d, J = 8.0 Hz, 1H), 7.85–7.89 (m, 2H), 8.25–8.28 (m,1H), 8.36–8.39 (m, 1H) ppm; $^{13}$C NMR (100 MHz, CDCl3):d = 28.3, 28.8, 34.7, 38.1, 50.9, 64.3, 117.1, 123.5, 123.6 (q, J = 3.7 Hz), 125.6 (q, J = 3.6 Hz), 126.5 (q, J = 270.8 Hz),127.8, 128.2, 128.9, 129.0, 129.2, 131.1, 131.2 (q, J =37.5 Hz), 133.8, 134.7, 137.5, 151.4, 154.6, 156.1,192.1 ppm; MS (EI): *m/z* = 441.01 $(M+H)^+$.

### *13-(4-Hydroxy-3-methoxyphenyl)-3,3-dimethyl-3,4-dihydro-2H-indazolo[1,2-b] phthalazine-1,6,11(13H)-trione (**4f**)*

Yellow powder, m.p.: 250-253 °C. IR (KBr, cm$^{-1}$): 3408,2958, 1660, 1600, 1493, 1359, 1270, 1234, 1135, 1030, 790, 627; 0.98 (s,6H, 2x$CH_3$), 1.90 (s, 2H, $CH_2$near double bond), 2.38 (s, 8H, $CH_2$ for near keto group), 3.89 (s, 3H, $OCH_3$), 5.21 (s, 1H, -NCH-Ar), 7.40-7.65 (m, 7H, Ar-H), 9.20 (s,1H, Ar-OH); $^{13}$C NMR (75MHz, $CDCl_3$): δ = 28.4, 28.8, 34.6, 38.1, 51.0, 56.0, 64.8, 111.0, 114.6, 118.6, 119.2, 127.7, 128.0,128.2, 129.0, 129.2, 133.5, 134.5, 146.0, 146.4, 150.7, 156.1, 192.3 ppm; MS(ESI): *m/z* = 419.30 $(M+H)^+$.

### *3,3-Dimethyl-13-(3,4,5-trimethoxyphenyl)-3,4-dihydro-2H-indazolo[1,2-b] phthalazine-1, 6, 11 (13H)-trione(**4g**)*

Yellow powder, m.p.: 231-233 °C. IR (KBr, cm$^{-1}$): 2960, 1656, 1628, 1597, 1507,1466, 1425, 1361, 1313, 1266, 1125, 1000, 701; $^1$H NMR (300 MHz, $CDCl_3$): δ = 1.23 (s, 3H), 1.24 (s, 3H), 2.36 (s, 2H), 3.21 (d, J = 19.1 Hz, 1H), 3.46 (d, J = 19.4 Hz, 1H), 3.80 (s, 3H), 3.83 (s, 6H), 6.40 (s, 1H), 6.63 (s, 2H), 7.83-8.37 (m, 4H) ppm; $^{13}$C NMR (75 MHz, CDCl3): δ = 28.1, 29.0, 34.6, 38.1, 51.0, 56.2, 60.7, 65.0, 104.6, 118.3, 127.7, 128.0, 128.9, 129.0, 131.8, 133.6, 134.6, 138.3, 150.9, 153.4, 154.5, 156.1, 192.2 ppm; MS(ESI): *m/z* = 462.04 $(M+H)^+$.

### *13-(3-Chlorophenyl)-3,3-dimethyl-3,4-dihydro-2H-indazolo[1,2-b]phthalazine-1, 6, 11(13H)-trione (**4h**)*

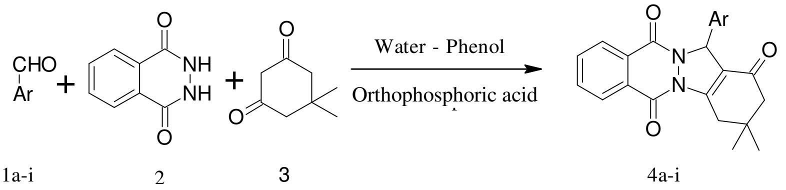
Yellow powder, m.p.: 206-207 $^{O}C$. IR (KBr, $cm^{-1}$): 3069, 2957, 2872, 1657, 1626, 1578, 1464, 1360, 1310, 1268, 1145, 788, 701, 677; $^{1}H$ NMR (300 MHz, $CDCl_3$): $\delta = 1.21$ (s, 6H), 2.34 (s, 2H), 3.21 (d, J = 19.1 Hz, 1H), 3. 41 (d, J = 19.1 Hz, 1H), 6.40 (s, 1H), 7.24-7.86 (m, 6H), 8.24- 8.37 (m, 2H) ppm; $^{13}C$ NMR (75 MHz, $CDCl_3$): $\delta = 28.5$, 28.6, 34.7, 38.0, 50.9, 64.3, 117.9, 125.8,127.0, 127.7, 128.1, 128.9, 129.0, 130.0, 133.7, 134.6, 138.5, 151.2, 154.4, 156.0, 192.1 ppm; MS (ESI): *m/z* = 407.10 $(M+H)^+$.

### *3,4-Dihydro-3,3-dimethyl-13-(2-chlorophenyl)-2H-indazolo[2,1-b]phthalazine-1,6, 11(13H)-trione(**4i**)*

Yellow powder, m.p.: 265-267 °C; IR (KBr, $cm^{-1}$): 2956, 1663, 1625; $^{1}H$ NMR (300 MHz, $CDCl_3$):1.22 (3H,s, Me), 1.23 (3H, s, Me), 2.33 (2H, s, $CH_2CO$), 3.24 and 3.42 (2H, AB system, J= 19.1 Hz, $CH_aH_bCO$), 6.69 (1H, s, CHN), 7.25-8.40 (8H, m, Ph); $^{13}C$ NMR (75 MHz, $CDCl_3$): 28.4, 28.8, 34.6, 38.0, 50.9, 64.0, 116.7, 127.2, 127.6, 128.0, 128.7, 129.0, 129.9, 130.5, 132.6, 133.0, 133.6, 134.5, 151.9, 154.2, 156.2, 192.1; MS (ESI):*m/z* = 407.08 $(M+H)^+$.

## Results and Discussion

Here, we report one pot, three component condensation of phthalhydrazide **2**, aromatic aldehydes (**1a-1i**) and dimedone **3** for the synthesis of aromatic 1*H*-indazolo[2,1-*b*] phthalazine-triones **4a-4i** using orthophosphoric acid as an important catalyst under water – phenol system in good to excellent yields and short reaction times (Scheme 1).



**Scheme 1**

In order to optimize the reaction conditions, phthalhydrazide (1.11 mmol), methoxy benzaldehyde (1.20 mmol) and dimedone (1.00 mmol) were used as model substrates (Table 2, entry **4a**). To evaluate the catalyst effect, first of all, the reaction was performed in water system. But no product was formed. Further, to optimize the reaction conditions, the reaction was carried out in presence of acidic catalysts such as hydrochloric acid, fuming nitric acid, acetic acid and ortho phosphoric acid in different solvent systems. Whereas the reaction had failed in the presence of strong acids like HCl, $HNO_3$as well as weak acetic acid. Now we had chosen water–phenol system (1:1) in the presence of orthophosphoric acid catalyst gave excellent yield 95% in only 15 min.time (Table 1).

**Table 1.** Optimization of the reaction conditions for the synthesis of aromatic 1*H*-indazolo [2,1-b] phthalazine-triones

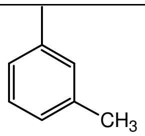
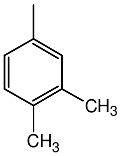
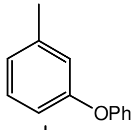
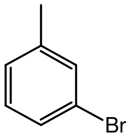
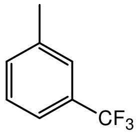
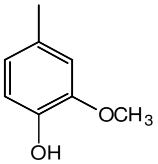
| Entry | Catalyst, mol% | Time, min | Yield, % |
|---|---|---|---|
| 1 | Water | 60 | 0 |
| 2 | Water - HCl (0.21 mmol) | 60 | 0 |
| 3 | Water-MeOH (1:1) /HCl (0.2 mmol) | 60 | 15 |
| 4 | Water – Phenol (1:1) /AcOH (0.21 mmol) | 60 | 5 |

*Contd...*

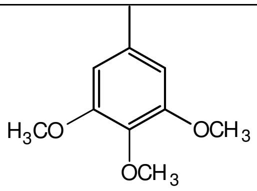
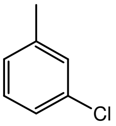
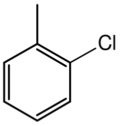
| | | | |
|---|---|---|---|
| 5 | Water-Phenol (1:1) /$HNO_3$ | 90 | 10 |
| 6 | Water-Phenol (1:1) / $H_2SO_4$(0.2 mmol) | 90 | 30 |
| 7 | Water / $H_3PO_4$ (0.2 mmol) | 30 | 0 |
| 8 | Water-DCM (1:1) / $H_3PO_4$ (0.2 mmol) | 30 | 0 |
| 9 | Water-THF (1:1) / $H_3PO_4$ (0.2 mmol) | 30 | 0 |
| 10 | Water-DMF (1:1) / $H_3PO_4$ (0.2 mmol) | 30 | 0 |
| 11 | Water-benzene (1:1) / $H_3PO_4$ (0.2 mmol) | 30 | 0 |
| 12 | Water-toluene (1:1) / $H_3PO_4$ (0.2 mmol) | 30 | 0 |
| 13 | Water – MeOH (1:1) /$H_3PO_4$ (0.2 mmol) | 25 | 60 |
| 14 | Water-Phenol (1:1) / $H_3PO_4$ (0.1 mmol) | 20 | 80 |
| 15 | Water-Phenol (1:1) / $H_3PO_4$ (0.2 mmol) | 15 | 95 |

There was no particular effect of substituents on aromatic aldehydes to pull back the reaction or to stop the reaction. All the reactions were found to be completed irrespective of electron with drawing or electron donating groups present in the aromatic aldehydes and afforded corresponding products at higher yields with less reaction time. The results were summarized below (Table 2).

**Table 2.** Synthesis of 1*H*-indazolo-[2,1-b]phthalazinetrione derivatives using orthophosphoric acid as a catalyst (Scheme 1)

| Entry | Ar | Product | Time, min | Isolated Yield, % |
|---|---|---|---|---|
| 1a |  | 4a | 15 | 91 |
| 1b |  | 4b | 20 | 92 |
| 1c |  | 4c | 20 | 92 |
| 1d |  | 4d | 13 | 94 |
| 1e |  | 4e | 12 | 87 |
| 1f |  | 4f | 10 | 93 |

*Contd...*

| | | | | |
|---|---|---|---|---|
| 1g |  | 4g | 4 | 91 |
| 1h |  | 4h | 15 | 84 |
| 1i |  | 4i | 14 | 90 |

## Conclusion

An efficient, one pot and three-component condensation was developed by simple, short experimental procedures to attained the 1*H*-Indazolo[2,1-*b*]phthalazine-triones. The reaction had been catalysed by orthophosphoric acid in a semi-miscible water phenol solvent system (1:1) was environmental benign, eco friendly, inexpensive practical procedure for the synthesis of 1*H*-Indazolo[2, 1-b]phthalazine-triones compounds.

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