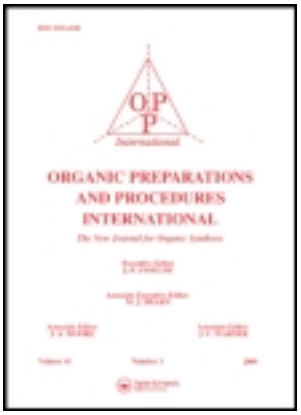

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Efficient Synthesis of 4,4′-(Arylmethylene)-*bis*(3-methyl-1-phenylpyrazol-5-ol) Derivatives in PEG-400 under Catalyst-free Conditions

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Pollution control demands are a source of inspiration to design and develop new reaction mediums to achieve high efficiency. Much attention has been directed toward the development of environmentally benign syntheses, including the use of solvent-free conditions,[1] and application of solvents such as ionic liquids[2] or water.[3] These protocols have been successfully used to achieve various chemical transformations, but there are some disadvantages that limit the use of these procedures. For example, in many organic reactions, solvents play a critical role to promote more efficient molecular interactions, but most organic compounds are not soluble in water. Moreover, due to the sensitivity of many catalysts to aqueous conditions, the use of water as solvent is limited to some special reactions. Ionic liquids also require tedious preparation and their environmental safety is still debatable.

Recently, liquid polymers or low melting point polymers have emerged as alternative green reaction media with unique properties such as thermal stability, commercial availability, non-volatility, immiscibility with a number of organic solvents, and recyclability.[4–14] Poly(ethylene glycol)s (PEGs) are preferred to other polymers because they are inexpensive, completely non-halogenated, easily degradable, and of low toxicity.[4,5] Among them, low molecular weight liquid PEGs can be regarded as protic solvents containing aprotic sites.[6] Several organic transformations such as Heck reaction,[7] asymmetric dihydroxylation,[8] Suzuki cross-coupling reaction,[9] oxydehydrogenation of alcohols and cyclic dienes,[10] metal mediated radical polymerization,[11] Michael addition of amines to conjugated alkenes,[12] Wacker reaction,[13] and partial reduction of alkynes[14] have been performed using PEGs as reaction media.

Pyrazolines are important five-membered heterocyclic compounds with different biological activities and other useful applications.[15,16] The conventional chemical approach

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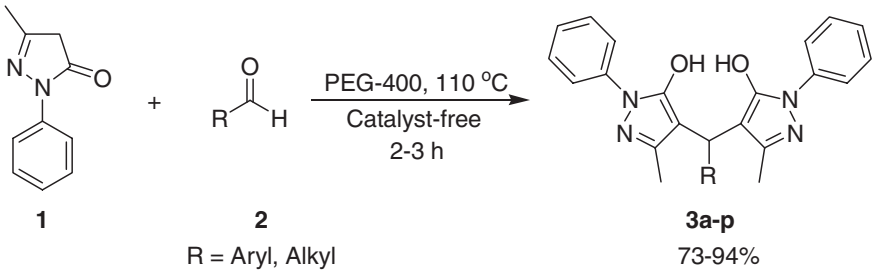
to 4,4′-(arylmethylene)-*bis*(3-methyl-1-phenylpyrazol-5-ols) involves Knoevenagel synthesis of the corresponding arylidenepyrazolones and its base-promoted Michael reaction, as well as the one-pot tandem Knoevenagel-Michael reaction of arylaldehydes with two equivalents of 5-methyl-2-phenyl-2,4-dihydro-3*H*-pyrazol-3-one.[17,18] Furthermore, recent methods such as application of sodium dodecylsulfate (SDS) in aqueous media,[19] and electrocatalytic synthesis[20] have been reported for the preparation of these compounds. However, most of the methods suffer from limitations such as low yields, long reaction times, application of hazardous solvents, tedious work-up procedures, and non-compliance with green chemistry protocols. Moreover, there is no catalyst-free protocol for the preparation of 4,4′-(arylmethylene)-*bis*(3-methyl-1-phenylpyrazol-5-ols). Considering the importance of pyrazoline derivatives, and in continuation of our studies to find green and efficient methodologies for preparation of organic compounds under catalyst-free conditions,[21,22] we have devised a general, efficient, rapid and catalyst-free protocol for the synthesis of 4,4′-(arylmethylene)-*bis*(3-methyl-1-phenylpyrazol-5-ols) derivatives *via* the condensation of 3-methyl-1-phenyl-5-pyrazolone (**1**) with aromatic and aliphatic aldehydes (**2**) in poly(ethylene glycol)-400 (PEG-400) at 110°C under neutral conditions (*Scheme 1*).



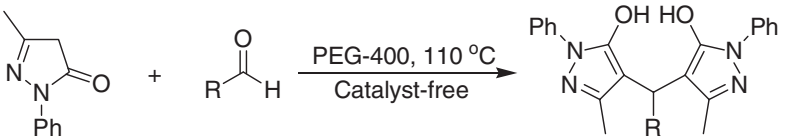
Scheme 1

To determine the best conditions, the condensation of 3-methyl-1-phenyl-5-pyrazolone (**1**) (2 mmol) with benzaldehyde (1 mmol) was examined in different amounts of PEG-400 at range of 80–120°C without catalyst. The best result was observed when 1 mL of PEG-400 was applied as reaction medium at 110°C; in this case, the desired product was produced in high yield (92%) within 2 h. Increasing the reaction time or the amount of PEG-400 did not improve the results. The reaction was also performed in the absence of PEG-400 at 110°C; however, in these conditions, the product was obtained in 23% after 6 h.

To assess the efficiency and the scope of the catalyst-free protocol, 3-methyl-1-phenyl-5-pyrazolone was treated with structurally diverse aromatic and aliphatic aldehydes under the optimized reaction conditions using PEG-400 as solvent. The corresponding results are displayed in *Table 1*.

Table 1 indicates that various aromatic aldehydes bearing halogens, electron-withdrawing or electron-releasing substituents on their aromatic rings as well as aliphatic aldehydes were efficiently condensed with 3-methyl-1-phenyl-5-pyrazolone to the corresponding 4,4′-(arylmethylene)-*bis*(3-methyl-1-phenylpyrazol-5-ol) derivatives in good to excellent yields and in relatively short reaction times. The results showed that the presence of halogens as well as electron-releasing substituents CH_3 and OCH_2CH_3 on the

Table 1
Catalyst-free Preparation of 4,4′-(Arylmethylene)-*bis*(3-methyl-1-phenylpyrazol-5-ol) Derivatives in PEG-400 at 110°C

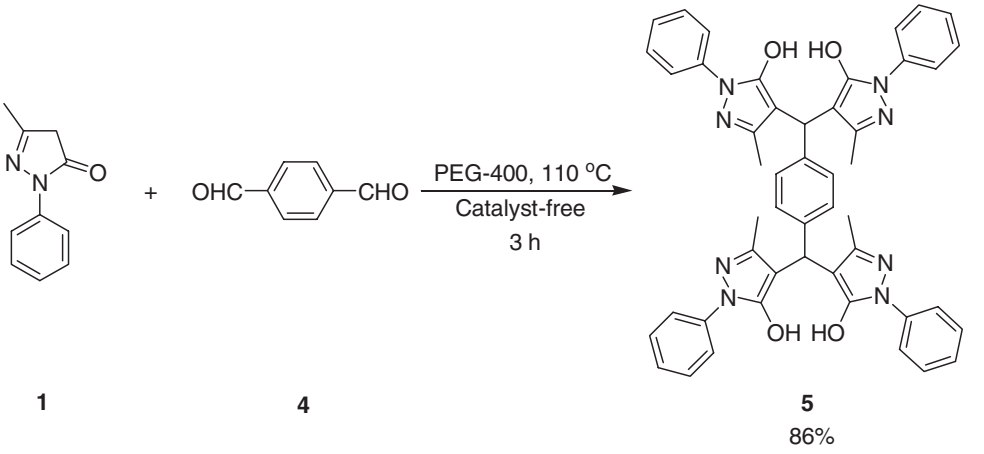


1 **2** **3a-p**

Entry	R	Product	Time (h)	Yield (%)	mp. °C (*lit.*)
1	C_6H_5	**3a**	2	92	168–170 (171–172)[19]
2	4-ClC_6H_4	**3b**	2.5	90	213–215 (207–209)[19]
3	3-ClC_6H_4	**3c**	2	89	150–152 (153–154)[19]
4	2-ClC_6H_4	**3d**	2	89	235–236 (236–237)[19]
5	4-HOC_6H_4	**3e**	3	83	149–150 (152–153)[19]
6	4-$EtOC_6H_4$	**3f**	3	90	185–188
7	4-$NO_2C_6H_4$	**3g**	1	94	229–231 (230–232)[19]
8	3-$NO_2C_6H_4$	**3h**	1.5	91	145–147 (149–150)[19]
9	2-$NO_2C_6H_4$	**3i**	1	91	221–223 (224–225)[19]
10	2-Thienyl	**3j**	2	89	190–192
11	2-Furyl	**3k**	1.5	89	189–191
12	2-Pyridyl	**3l**	1.5	84	230–232
13	3-BrC_6H_4	**3m**	2	90	172–175
14	4-MeC_6H_4	**3n**	3	91	201–203 (203–204)[19]
15	H	**3o**	2	73	225–227 (227–229)[19]
16	$(CH_3)_2CH$	**3p**	2	78	210–212 (213–214)[19]

aromatic ring of aldehydes had no significant effect on the reaction yields (*Table 1*, Entries 2–4, 6, 13 and 14); however, a hydroxy group decreased the yield slightly (*Table 1*, Entry 5). Moreover, electron-withdrawing substituents gave excellent yields in decreased times (*Table 1*, Entries 7–9). Interestingly, the catalyst-free method was successfully applied for the condensation of 3-methyl-1-phenyl-5-pyrazolone with heteroaromatic aldehydes as well as aliphatic aldehydes (*Table 1*, Entries 10–12, 15 and 16). Because of the neutral nature of PEG-400, acid-sensitive aldehydes (for example furfural) as well as basic aldehydes (for example 2-pyridinecarbaldehyde) were successfully applied in the reaction (*Table 1*, Entries 11 and 12).

Interestingly, the condensation of 3-methyl-1-phenyl-5-pyrazolone (2 equivalents) with terephthaldehyde (1 equivalent) in PEG-400 at 110°C afforded di-4,4′-(arylmethylene)-*bis*(3-methyl-1-phenylpyrazol-5-ol) **5** in 86% yield within 3 h (*Scheme 2*). It is worth noting that in nearly none of the reported methods for the preparation of these compounds, the synthesis of di-4,4′-(arylmethylene)-*bis*(3-methyl-1-phenylpyrazol-5-ols) has been studied.



1 **4** **5**
86%

Scheme 2

In summary, we have reported the first catalyst-free method for the synthesis of 4,4′-(arylmethylene)-*bis*(3-methyl-1-phenylpyrazol-5-ols) in a green recyclable media.[23] The promising points for the presented methodology are efficiency, generality, high yield, relatively short reaction time, low cost, cleaner reaction profile, ease of product isolation, simplicity, potential for recycling of the solvent, and finally compliance with the green chemistry protocols.

Experimental Section

All chemicals were purchased from Merck or Fluka Chemical Companies. The ^{1}H NMR (500 MHz) and ^{13}C NMR (125 MHz) were run on a Bruker Avance DPX-500, FT-NMR spectrometer, δ in ppm. Mass spectra were recorded on a Shimadzu GC MS-QP 1000 EX apparatus. Melting points were recorded on a Büchi B-545 apparatus in open capillary tubes.

General Procedure for the Synthesis of 4,4′-(Arylmethylene)-bis(3-methyl-1-phenylpyrazol-5-ols)

To a mixture of 3-methyl-1-phenyl-5-pyrazolone (0.35 g, 2 mmol) and aldehyde (1 mmol) in a 10 mL round-bottomed flask connected to a reflux condenser, was added PEG-400 (1 mL), and the resulting mixture was stirred in an oil-bath (110°C). The progress of the reaction was monitored by TLC using EtOAc/*n*-hexane (1/4) as eluent. After completion of the reaction, the reaction mixture was cooled to room temperature and water (20 mL) was added. PEG-400 dissolved in the water and the insoluble crude product was isolated by simple filtration. The crude product was dissolved in warm EtOH or aqueous EtOH (6 mL) and was allowed to stand at room temperature for 5–6 h. The crystalline solids were collected, washed with ethanol and dried. The filtrate containing PEG-400 was extracted with Et_2O (2 × 20 mL) to remove any organic compounds dissolved in the aqueous phase.

The aqueous layer was separated and the water was evaporated under reduced pressure to give pure PEG-400 which was used for the next run under similar reaction conditions.

Selected Spectral Data of the Products

Compound 3a: Pale yellow solid (from 3:1 EtOH/H_2O), mp. 168–170°C (*lit.*[27] 171–172°C); ^{1}H NMR (DMSO-d_6): δ 2.33 (s, 6H, $2CH_3$), 4.88 (s, 1H, CH), 7.07 (m, 1H), 7.18 (m, 6H), 7.44 (t, 4H), 7.58 (d, 4H); ^{13}C NMR DMSO-d_6): δ 12.0, 33.5, 121.4, 126.2, 126.8, 127.6, 128.2, 129.1, 137.3, 142.9, 146.1.

Compound 3b: White solid (from EtOH), mp. 213–215°C (*lit.*[27] 207–209°C); ^{1}H NMR (DMSO-d_6): δ 2.32 (s, 6H), 4.97 (s, 1H), 7.26 (d, $J = 8.2$ Hz, 4H), 7.34 (d, $J = 8.0$ Hz, 2H), 7.44 (t, $J = 7.1$ Hz, 4H), 7.71 (d, $J = 7.6$ Hz, 4H).

Compound 3c: White solid (from EtOH), mp. 150–152°C (*lit.*[27] 153–154°C).

Compound 3d: White solid (from 5:1 EtOH/H_2O), mp. 235–236°C (*lit.*[27] 236–237°C); ^{1}H NMR (DMSO-d_6): δ 2.26 (s, 6H, $2CH_3$), 5.08 (s, 1H, CH), 7.27–7.29 (m, 2H), 7.41–7.43 (m, 8H), 7.75–7.77 (m, 4H).

Compound 3e: White solid (from 3:1 EtOH/H_2O), mp. 149–150°C (*lit.*[27] 152–153°C); ^{1}H NMR (DMSO-d_6): δ 2.31 (s, 6H, $2CH_3$), 4.86 (s, 1H, CH), 6.66–6.68 (d, $J = 8.5$ Hz, 2H), 7.05–7.07 (d, $J = 8.5$ Hz, 2H), 7.23–7.26 (t, $J = 7$ Hz, 2H), 7.43–7.46 (t, $J = 8$ Hz, 4H), 7.71–7.73 (d, $J = 8$ Hz, 4H), 9.16 (s, 1H, OH), 12.40 (s, 1H, OH), 13.95 (s, 1H, OH); ^{13}C NMR (DMSO-d_6): δ 12.5, 33.2, 40.9, 115.7, 121.4, 126.4, 128.9, 129.8, 133.2, 143.6, 147.0, 156.4.

Compound 3f: White solid (from EtOH), mp. 185–188°C; ^{1}H NMR (DMSO-d_6): δ 1.06–1.08 (t, $J = 7$ Hz, 3H, CH_3), 2.34 (s, 6H, $2CH_3$), 3.44–3.48 (q, $J = 7$ Hz, 2H, CH_2), 5.01 (s, 1H, CH) 7.25–7.29 (m, 4H), 7.38–7.46 (m, 6H), 7.71–7.73 (d, $J = 8$ Hz, 4H), 13.94 (s, 1H, OH); ^{13}C NMR (DMSO-d_6): δ 12.4, 19.4, 33.7, 56.9, 121.5, 122.4, 126.5, 127.3, 129.8, 130.8, 131.2, 146.0, 147.2; MS (m/z): 480 (M^+).

Anal. Calcd. for $C_{29}H_{28}N_4O_3$: C, 72.48; H, 5.87; N, 11.66. Found: C, 72.23; H, 5.98; N, 11.52.

Compound 3g: Pale yellow solid (from EtOH), mp. 229–231°C (*lit.*[27] 230–232°C); ^{1}H NMR (DMSO-d_6): δ 2.35 (s, 6H, $2CH_3$), 5.13 (s, 1H, CH), 7.25–7.27 (m, 2H, Arm H), 7.43–7.46 (t, $J = 7$ Hz, 4H), 7.51–7.53 (d, $J = 8$ Hz, 2H), 7.70–7.72 (d, $J = 8$ Hz, 4H), 8.16–8.18 (d, $J = 8$ Hz, 2H), 12.64 (s, 1H, OH), 13.86 (s, 1H, OH); ^{13}C NMR (DMSO-d_6): δ 12.5, 19.4, 34.0, 56.9, 121.5, 124.2, 126.6, 129.5, 129.8, 146.8, 147.1, 151.2.

Compound 3h: Pale yellow solid (from EtOH), mp. 145–147°C (*lit.*[27] 149–150°C).

Compound 3i: White solid (from EtOH), mp. 221–223°C (*lit.*[27] 224–225°C); ^{1}H NMR (DMSO-d_6): δ 2.29 (s, 6H, $2CH_3$), 5.47 (s, 1H, CH), 7.20–7.26 (m, 2H), 7.40–7.49 (m, 5H), 7.61–7.75 (m, 7H).

Compound 3j: White solid (from 5:1 EtOH/H_2O), mp. 190–192°C; ^{1}H NMR (DMSO-d_6): δ 2.38 (s, 6H, $2CH_3$), 5.11 (s, 1H, CH), 6.89–6.92 (m, 2H), 7.09–7.48 (m, 7H,), 7.77–7.85 (m, 4H), 13.56 (s, 1H, OH); ^{13}C NMR (DMSO-d_6): δ 12.6, 33.3, 121.3, 125.1, 125.3, 128.3, 128.6, 131.4, 131.5, 131.9, 134.5, 139.7, 144.9, 154.8. MS (m/z): 442 (M^+). *Anal. Calcd.* for $C_{25}H_{22}N_4O_{32}S$: C, 67.85; H 5.01; N 12.66. Found: C, 67.56; H, 5.14; N, 12.57.

Compound 3k: White solid (from EtOH), mp. 189–191°C; ^{1}H NMR (DMSO-d_6): δ 2.21 (s, 6H, $2CH_3$), 4.87 (s, 1H, CH), 6.14 (s, 1H), 6.48 (s, 1H), 7.37 (t, 2H), 7.44 (t, 4H), 7.53 (s, 1H), 7.78 (d, 4H); ^{13}C NMR (DMSO-d_6): δ 12.2, 28.6, 106.4, 110.2, 121.6, 126.4, 129.9, 142.4, 146.9, 154.4; MS (m/z): 426 (M^+).

Anal. Calcd. for $C_{25}H_{22}N_4O_3$: C, 70.41; H, 5.20; N, 13.14. Found: C, 70.63; H, 5.29; N, 13.03.

Compound 3l: White solid (from EtOH), mp. 230–232°C; ^{1}H NMR (DMSO-d_6): δ 2.29 (s, 6H, $2CH_3$), 5.11 (s, 1H, CH), 7.21 (t, 2H), 7.41 (m, 1H), 7.48 (t, 4H), 7.66 (d, 5H), 8.47 (m, 2H); ^{13}C NMR (DMSO-d_6): δ 12.1, 31.6, 104.5, 121.0, 123.7, 126.5, 129.1, 136.3, 137.6, 138.1, 146.5, 147.8, 148.5; MS (m/z): 437 (M^+).

Anal. Calcd. for $C_{26}H_{23}N_5O_2$: C, 71.38; H, 5.30; N, 16.01. Found: C, 71.21; H, 5.42; N, 15.93.

Compound 3m: White solid (from 5:1 EtOH/H_2O), mp. 172–175°C; ^{1}H NMR (DMSO-d_6): δ 2.30 (s, 6H, $2CH_3$), 4.89 (s, 1H, CH), 6.82–7.71 (m, 14H), 12.40 (s, 1H, OH), 13.92 (s, 1H, OH); ^{13}C NMR (DMSO-d_6): δ 10.0, 14.3, 117.9, 123.6, 125.4, 127.2, 127.7, 128.3, 128.7, 129.7, 132.4, 133.6, 140.4, 145.9, 154.4; MS (m/z): 515 (M^+).

Anal. Calcd. for $C_{27}H_{23}BrN_4O_2$: C, 62.92; H, 4.50; N, 10.87. Found: C, 62.64; H, 4.61; N, 10.73.

Compound 3n: White solid (from 3:1 EtOH/H_2O), mp. 201–203°C (*lit.*[27] 203–204°C); ^{1}H NMR (DMSO-d_6): δ 2.24 (s, 3H, CH_3), 2.31 (s, 6H, $2CH_3$), 4.91 (s, 1H, CH), 7.08–7.71 (m, 14H), 12.40 (s, 1H, OH), 13.92 (s, 1H, OH); ^{13}C NMR (DMSO-d_6): δ 12.5, 21.4, 33.6, 40.9, 121.4, 126.4, 127.9, 129.6, 129.8, 135.7, 140.0, 147.1.

Compound 3o: White solid (from 2:1 EtOH/H_2O), mp. 225–227°C (*lit.*[27] 227–229°C); ^{1}H NMR (DMSO-d_6): δ 2.15 (s, 6H, $2CH_3$), 3.31 (s, 2H, CH), 7.28–7.30 (m, 2H), 7.46–7.48 (m, 4H), 7.73–7.75 (m, 4H).

Compound 3p: White solid (from 2:1 EtOH/H_2O), mp. 210–212°C (*lit.*[27] 213–214°C); ^{1}H NMR (DMSO-d_6): δ 0.84 (s, 6H, $2CH_3$), 2.21 (s, 6H, $2CH_3$), 2.55–2.69 (m, 1H, CH), 2.97–3.08 (d, 1H, CH), 7.25–7.28 (m, 2H), 7.40–7.48 (m, 4H), 7.71–7.73 (m, 4H).

Compound 5: Pale yellow solid (from EtOH), mp. 193–196°C; ^{1}H NMR (DMSO-d_6): δ 2.16 (s, 12H, $4CH_3$), 4.71 (s, 2H, CH), 7.00–7.03 (t, $J = 7.5$ Hz, 4H), 7.06 (s, 4H), 7.17–7.21 (t, $J = 8.0$ Hz, 8H), 7.55–7.57 (d, $J = 8.0$ Hz, 8H), 13.40 (s, 4H, OH); ^{13}C NMR (DMSO-d_6): δ 12.2, 33.8, 120.6, 121.5, 125.9, 127.5, 129.0, 131.1, 137.0, 139.2, 146.7; MS (m/z): 448 (M^+-$C_{20}H_{18}N_4O_2$), 431, 354.

Anal. Calcd. for $C_{48}H_{42}BrN_8O_4$: C, 72.53; H, 5.33; N, 14.10. Found: C, 72.77; H, 5.21; N, 14.19.

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