



Syntheses and Reactions of 4'-[(ω -Bromoalkyl)oxy]- and 4',4'''-(Polymethylenedioxy)-bis Substituted Chalcones

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Received 30 September 2009; Accepted 25 November 2009

Abstract: Base catalyzed condensation of 4-hydroxyacetophenone (**1**) with several aldehydes resulted 4'-hydroxychalcones (**2a-c**). Reaction of α,ω -dibromoalkanes (**3**, $n=2, 4$ & **6**) with (**2a-c**) in equimolar ratio resulted a mixture of 4'-[(ω -bromoalkyl)oxy]- substituted chalcones (**4a-i**) and 4',4'''-(polymethylenedioxy)-bis substituted chalcones (**5a-i**) separated by ethanol as soluble (**4a-i**) and insoluble (**5a-i**) products. Compounds (**5a-i**) were also synthesized by another route. Compound **1** on treatment with **3** in molar ratio 2:1 resulted 4',4'''-(polymethylenedioxy)-diacetophenone (**4[#]a-c**) which on base catalyzed condensation gave compounds (**5a-i**). Reaction of **4d** with substituted phenols and thiophenols gave corresponding phenoxy and thiophenoxy substituted chalcones (**6a-f**) where as compounds **5a, 5b, 5d, 5h** and **5e** on treatment with hydrazine hydrate furnished corresponding bis-2-pyrazolines (**7a-e**). The structures of all synthesized compounds were confirmed on the basis of analytical and spectral data.

Keywords: Chalcones, Bromoalkoxychalcones, Polymethylenedioxychalcones, Bis-2-pyrazolines.

Introduction

Chalcones are natural or synthetic compounds bearing the 1,3-diphenylpropenone framework. Depending on the substitution pattern on the two aromatic rings, a wide range of pharmacological activities have been identified for various chalcones. These include among others, cytotoxic, antitumor, antiviral and antiprotozoal activities^{1,2}. More recently, there has been strong interest in the potential antimalarial³ activity of chalcones and bis-chalcones. The antimalarial activity of chalcones was first noted when lipochalcone-A, a natural product isolated from Chinese liquorice roots, was reported to exhibit potent *in vivo* and *in vitro* antimalarial activity⁴. Subsequently, a synthetic analogue, 2,4-dimethoxy-4'-butoxychalcone

was reported to have outstanding antimalarial activity⁵. Various substituted pyrazolines and their derivatives are important biological agents and a significant amount of research activity has been directed towards this class⁶⁻¹⁰. Prompted by the varied biological activities of the chalcones, bis-chalcones and pyrazolines and in continuation to our work on 4'-alkoxy substituted chalcones¹¹ and other substituted analogues¹²⁻¹⁵ for biological evaluation and synthesis of variously sized heterocycles it was contemplated in this present work to synthesize variously substituted 4'-[(ω -bromoalkyl)oxy]chalcones, 4',4'''-polymethylenedioxybischalcones and 4',4'''-polymethylenedioxy-bis-2-pyrazolines.

Experimental

All melting points were determined in open capillaries in liquid paraffin bath and are uncorrected. The IR spectra were recorded on Perkin-Elmer-783 spectrophotometer (ν_{\max} in cm^{-1}) using KBr pellets. ¹H NMR spectra were recorded on a Bruker DRX-300 MHz spectrophotometer (δ , ppm) using CDCl_3 / DMSO-d_6 as a solvent and TMS as internal standard. The purity of the compounds was checked by TLC on silica gel coated glass plates. The elemental analyses were performed on Carlo-Erba-1108 analyzer and were within $\pm 0.5\%$ of the calculated values. Syntheses of 4'-hydroxychalcones and α, ω -dibromoalkanes ($\text{Br}(\text{CH}_2)_n\text{-Br}$; $n = 2, 4$ & 6) were carried out by literature methods¹⁶.

General procedure for the preparation of 4'-[(ω -bromoalkyl)oxy]-substituted chalcones (4a-i) and 4',4'''-(polymethylenedioxy)-bis substituted chalcones (5a-i)

An appropriate 4'-hydroxy chalcone **2** (0.01 mol) and α, ω -dibromoalkane **3** (0.01 mol) ($n = 2, 4$ or 6) in dry acetone (100 mL) and anhydrous potassium carbonate (0.02 mol) were refluxed (10-15 h). The completion of the reaction was judged by negative test with alcoholic ferric chloride. After completion of the reaction, acetone was distilled off and the residue in the flask was treated with ice cold water and stirred well. Separated solid was filtered and washed with 5% NaOH solution and finally with water (3×30 mL). The solid was dried, dissolved in boiling ethanol and filtered hot. Some solid remains insoluble which was washed again with hot ethanol. The filtrate after concentration afforded a solid which on crystallization from proper solvent gave compounds (**4a-i**). Whereas ethanol insoluble solid resulted bis chalcones (**5a-i**)

4a: Pale yellow (methanol): yield 65%; m.p. 96 °C: IR (KBr): 3050 (Ar-H), 2910, 2860 (C-H): 1660 (C=O), 1600, 1500 (C=C), 1250 (asym. C-O-C): 1030 (sym. C-O-C): ¹H NMR (δ , CDCl_3): 3.65, (t, 2H, $-\text{CH}_2\text{Br}$): 4.40 (t, 2H, $-\text{OCH}_2-$): 6.90-8.10 (m, 10H, Ar-H & olefinic protons): Anal. (%) Calcd. for $\text{C}_{17}\text{H}_{15}\text{BrO}_2$ (331.20): C, 61.65; H, 4.56. Found C, 61.35; H, 4.30.

4b: Pale yellow (methanol): yield 60%; m.p. 103 °C: IR (KBr): 3080 (Ar-H), 2910 (C-H): 1660 (C=O), 1600, 1520 (C=C), 1220 (asym. C-O-C): 1040 (sym. C-O-C): ¹H NMR (δ , CDCl_3): 3.65, (t, 2H, $-\text{CH}_2\text{Br}$): 3.85 (s, 3H, $-\text{OCH}_3$): 4.35 (t, 2H, $-\text{OCH}_2-$): 6.80-8.10 (m, 10H, Ar-H & olefinic protons): Anal. (%) Calcd. for $\text{C}_{18}\text{H}_{17}\text{BrO}_3$ (361.23): C, 59.85; H, 4.74. Found C, 59.57; H, 4.57.

4c: Colourless (ethanol): yield 70%; m.p. 145 °C: IR (KBr): 3060 (Ar-H), 2900 (C-H): 1660 (C=O), 1600, 1500 (C=C), 1230 (asym. C-O-C): 1020 (sym. C-O-C): Anal. (%) Calcd. for $\text{C}_{17}\text{H}_{14}\text{BrClO}_2$ (365.65): C, 55.84; H, 3.85. Found C, 56.93; H, 3.93.

4d: Pale yellow (methanol): yield 60%; m.p. 113 °C: IR (KBr): 3050 (Ar-H), 2900, 2850 (C-H): 1660 (C=O), 1600, 1510 (C=C), 1240 (asym. C-O-C): 1030 (sym. C-O-C): Anal. (%) Calcd. for $\text{C}_{19}\text{H}_{19}\text{BrO}_2$ (359.25): C, 63.50; H, 5.29. Found C, 63.32; H, 5.54.

4e: Pale yellow (methanol): yield 60%; m.p. 110 °C: IR (KBr): 3080 (Ar-H), 2900 (C-H): 1660 (C=O), 1600, 1570, 1510 (C=C), 1220 (asym. C-O-C): 1040 (sym. C-O-C): Anal. (%) Calcd. for $\text{C}_{20}\text{H}_{21}\text{O}_3\text{Br}$ (389.28): C, 61.71; H, 5.44: Found C, 61.32; H, 5.36.

4f: Colourless (ethanol): yield 70%; m.p. 116 °C: IR (KBr): 3060 (Ar-H), 2910 (C-H):

1660 (C=O), 1600, 1500 (C=C), 1230 (asym. C-O-C): 1030 (sym. C-O-C): Anal. (%) Calcd. for C₁₉H₁₈BrClO₂ (393.70): C, 57.96; H, 4.61. Found C, 57.70, H, 4.56.

4g: Colourless (methanol): yield 65%: m.p. 88 °C: IR (KBr): 3050 (Ar-H), 2900 (C-H): 1650 (C=O), 1600, 1570 (C=C), 1230 (asym. C-O-C): 1040 (sym. C-O-C): Anal. (%) Calcd. for C₂₁H₂₃BrO₂ (387.30): C, 65.12; H, 5.99. Found C, 65.42, H, 6.12.

4h: Colourless (ethanol): yield 70%: m.p. 90 °C: IR (KBr): 3070 (Ar-H), 2940,2880 (C-H): 1660 (C=O), 1600, 1570, 1510 (C=C), 1220 (asym. C-O-C): 1030 (sym. C-O-C): Anal. (%) Calcd. for C₂₂H₂₅O₃Br (417.35): C, 63.30; H, 6.03. Found C, 63.48, H, 5.97.

4i: Colourless (methanol): yield 60%: m.p. 117 °C: IR (KBr): 3040 (Ar-H), 2940,2860 (C-H): 1660 (C=O), 1600, 1580, 1500 (C=C), 1260 (asym. C-O-C): 1040 (sym. C-O-C): Anal. (%) Calcd. for C₂₁H₂₂BrClO₂ (421.75): C, 59.80; H, 5.26. Found C, 59.65, H, 5.20.

5a: Colourless (CHCl₃): yield 16%: m.p. 188 °C: IR (KBr): 3030 (Ar-H), 2960,2900 (C-H): 1660 (C=O), 1600, 1500 (C=C), 1230 (asym. C-O-C): 1040 (sym. C-O-C): Anal. (%) Calcd. for C₃₂H₂₆O₄ (474.56): C, 81.01; H, 5.48. Found C, 80.90, H, 5.5.

5b: Colourless (CHCl₃): yield 20%: m.p. 201 °C: IR (KBr): 3050 (Ar-H), 2940,2860 (C-H): 1660 (C=O), 1610, 1590 (C=C), 1220 (asym. C-O-C): 1030 (sym. C-O-C): Anal. (%) Calcd. for C₃₄H₃₀O₆ (534.59): C, 76.39; H, 5.66. Found C, 76.24, H, 5.55.

5c: Colourless (dioxan): yield 26%: m.p. 260 °C: IR (KBr): 3040 (Ar-H), 2950,2880 (C-H): 1650 (C=O), 1600, 1580 (C=C), 1240 (asym. C-O-C): 1040 (sym. C-O-C): Anal. (%) Calcd. for C₃₂H₃₄Cl₂O₄ (543.44): C, 70.72; H, 4.45. Found C, 70.82, H, 4.39.

5d: Colourless (CHCl₃): yield 16%: m.p. 180 °C: IR (KBr): 3060, 3020 (Ar-H), 2900,2840 (C-H): 1640 (C=O), 1600, 1570 (C=C), 1260 (asym. C-O-C): 1050 (sym. C-O-C): Anal. (%) Calcd. for C₃₄H₃₀O₄ (502.59): C, 81.25; H, 6.02. Found C, 81.02, H, 5.80.

5e: Colourless (dioxane-alcohol: 80:20 v/v): yield 18%: mp. 200 °C: IR (KBr): 3050 (Ar-H), 2950,2850 (C-H): 1660 (C=O), 1600, 1530 (C=C), 1280 (asym. C-O-C): 1040 (sym. C-O-C): Anal. (%) Calcd. for C₃₆H₃₄O₆ (562.62): C, 76.86; H, 6.09. Found C, 76.70, H, 6.16.

5f: Pale yellow (CHCl₃): yield 28%: m.p. 240 °C: IR (KBr): 3040 (Ar-H), 2900,2860 (C-H): 1660 (C=O), 1590, 1500 (C=C), 1250 (asym. C-O-C): 1030 (sym. C-O-C): Anal. (%) Calcd. for C₃₄H₂₈O₄Cl₂ (571.48): C, 71.46; H, 4.94. Found C, 71.20, H, 4.82.

5g: Colourless (CHCl₃): yield 20%: m.p. 182 °C: IR (KBr): 3050 (Ar-H), 2970,2880 (C-H): 1660 (C=O), 1600, 1510 (C=C), 1230 (asym. C-O-C): 1020 (sym. C-O-C): Anal. (%) Calcd. for C₃₆H₃₄O₄ (530.65): C, 81.48; H, 6.46. Found C, 81.20, H, 6.50.

5h: Colourless (CHCl₃): yield 21%: m.p. 195 °C: IR (KBr): 3040 (Ar-H), 2950,2860 (C-H): 1660 (C=O), 1600, 1550, 1500 (C=C), 1220 (asym. C-O-C): 1030 (sym. C-O-C): Anal. (%) Calcd. for C₃₈H₃₈O₆ (590.70): C, 77.26; H, 6.48. Found C, 77.12, H, 6.26.

5i: Colourless (dioxane-ethanol: 80:20 v/v): yield 27%: m.p. 218 °C: IR (KBr): 3050 (Ar-H), 2900,2860 (C-H): 1660 (C=O), 1600, 1500 (C=C), 1250 (asym. C-O-C): 1040 (sym. C-O-C): Anal. (%) Calcd. for C₃₆H₃₂O₄Cl₂ (599.54): C, 72.12; H, 5.38. Found C, 72.34, H, 5.28.

General procedure for the preparation of 4',4'''-(polymethlenedioxy) diacetophenones (4[#]a-c)

4-Hydroxyacetophenone (1) and α, ω -dibromoalkane (3 n= 2, 4 or 6) in the molar ratio 2:1 in acetone (100 mL) and anhydrous K₂CO₃ (0.025 mol) were refluxed (10 to 15 h). The completion of the reaction was judged by the negative alcoholic ferric chloride test. On completion of the reaction, acetone was distilled off and the residue was treated with cold water (3 x 30 mL). The solid obtained on recrystallization from ethanol afforded

the compounds **4[#]a-c**. Using this procedure following compounds were prepared.

4[#]a: Colourless: yield 67%: m.p. 161 °C: IR (KBr): 3060 (Ar-H), 2960, 2920, 2890 (C-H): 1680 (C=O), 1600, 1510 (C=C), 1250 (asym. C-O-C): 1040 (sym. C-O-C): Anal. (%) Calcd. for C₁₈H₁₈O₄ (298.34): C, 72.41; H, 6.04. Found C, 72.15, H, 6.21.

4[#]b: Colourless: yield 70%: m.p. 141 °C: IR (KBr): 3080, 3040 (Ar-H), 2960, 2870 (C-H): 1680 (C=O), 1600, 1580, 1510 (C=C), 1255 (asym. C-O-C): 1050 (sym. C-O-C): ¹H NMR (CDCl₃) 2.01(m,4H,-(CH₂)₂-): 2.59 (s,6H,2 x -COCH₃): 4.11 (m,4H, 2 x -OCH₂-): 6.93-7.90 (m,8H,Ar-H). Anal. (%) Calcd. for C₂₀H₂₂O₄ (326.40): C, 73.60; H, 6.79. Found C, 73.46, H, 6.50.

4[#]c: Colourless: yield 71%: m.p. 120 °C: IR (KBr): 3035 (Ar-H), 2950, 2880 (C-H): 1670 (C=O), 1600, 1580, 1510 (C=C), 1240 (asym. C-O-C): 1010 (sym. C-O-C): ¹H NMR (CDCl₃) 1.30-2.10 (m,8H,-(CH₂)₄-): 2.58 (s,6H,2 x -COCH₃): 4.04 (m, 4H, 2 x -OCH₂-): 6.90-7.88 (m,8H,Ar-H): Anal. (%) Calcd. for C₂₂H₂₆O₄ (354.39): C, 74.47; H, 6.08. Found C, 74.35, H, 7.15.

General procedure for the preparation of 4',4'''-(polymethylenedioxy) bis substituted chalcones (5a-i) from compounds 4[#]a-c

Diacetophenone **4[#]** (0.01 mol) and appropriate substituted aromatic aldehyde (0.025 mol) were dissolved in ethanol (100 mL). To this solution NaOH (20 mL, 15%) was added and the mixture was stirred constantly at room temperature (5 h), when a semi solid mass separated. It was acidified with dilute HCl and separated solid was filtered, washed with water (2 x 30 mL), dried and recrystallized from proper solvent to afford analytical samples of (**5a-i**) in 60 to 80% yield and were fully characterized by m.p., m.m.p., co-TLC and super imposable IR spectra with authentic samples prepared by another route.

General procedure for the preparation of 4'-{[4-(p-substituted phenoxy / thiophenoxy)butyl]oxy} chalcones (6a-f)

Equimolar amount of appropriate 4'-[(4-bromo butyl)oxy]-chalcone (**4d**) and phenol / thiophenol in dry acetone (100 mL) in the presence of anhydrous K₂CO₃ (0.25 mol) were refluxed (6-8 h). The solvent was distilled and the residue in the flask was treated with ice cold water. The solid separated was washed with 5% sodium hydroxide solution and finally with cold water (3 x 30 mL) and dried. Recrystallization from ethanol / methanol gave analytical samples of compounds **6a-f**.

6a: Colourless: yield 67%: m.p. 136 °C: IR (KBr): 3020 (Ar-H), 2980 (C-H): 1650 (C=O), 1600, 1590, 1510 (C=C), 1230 (asym. C-O-C): 1030 (sym. C-O-C): Anal. (%) Calcd. for C₂₅H₂₄O₃ (372.46): C, 80.62; H, 6.49. Found C, 80.20, H, 6.25.

6b: Colourless: yield 60%: m.p. 125 °C: IR (KBr): 3050 (Ar-H), 2910 (C-H): 1660 (C=O), 1600, 1580 (C=C), 1260 (asym. C-O-C): 1030 (sym. C-O-C): Anal. (%) Calcd. for C₂₅H₂₃ClO₃ (406.9): C, 73.79; H, 5.70. Found C, 73.51, H, 5.56.

6c: Colourless: yield 65%: m.p. 115 °C: IR (KBr): 3010 (Ar-H), 2960 (C-H): 1650 (C=O), 1610, 1570 (C=C), 1240 (asym. C-O-C): 1050 (sym. C-O-C): Anal. (%) Calcd. for C₂₅H₂₃BrO₃ (451.35): C, 66.53; H, 5.14. Found C, 66.60, H, 5.20.

6d: Pale yellow: yield 58%: m.p. 114 °C: IR (KBr): 3060 (Ar-H), 2960, 2880 (C-H): 1650 (C=O), 1590, 1570, 1500 (C=C), 1250 (asym. C-O-C): 1030 (sym. C-O-C): Anal. (%) Calcd. for C₂₅H₂₃NO₅ (417.46): C, 71.93; H, 5.55. Found C, 71.40, H, 5.50.

6e: Colourless: yield 70%: m.p. 96 °C: IR (KBr): 3050 (Ar-H), 2900 (C-H): 1650 (C=O), 1610, 1580 (C=C), 1260 (asym. C-O-C): 1040 (sym. C-O-C): Anal. (%) Calcd. for C₂₅H₂₄O₂S (388.52): C, 77.28; H, 6.2. Found C, 77.50, H, 6.17.

6f: Cream colourless: yield 80%: m.p. 60 °C: IR (KBr): 3010 (Ar-H), 2960 (C-H): 1660 (C=O), 1600, 1570, 1500 (C=C), 1260 (asym. C-O-C): 1020 (sym. C-O-C): Anal. (%) Calcd. for C₂₅H₂₃O₂ClS (423.97): C, 70.99; H, 5.48. Found C, 70.60, H, 5.40.

General procedure for the preparation of 3,3'-[poly methylene bis (oxy-o-phenylene)] bis[5-(p-substituted phenyl)-2-pyrazolines] (7a-e)

To a solution of bis chalcone **5** (0.01 mol) in DMF (40 mL), hydrazine hydrate (0.2 mol, 95%) was added drop wise. The reaction mixture was heated under reflux for 8-10 h. The contents were poured into ice water with stirring. The resulting solid product was filtered, washed with water and dried. Further purification was done by crystallization using suitable solvents.

7a: Colourless: (DMF-H₂O:80:20 v/v): yield 75%.: m.p. 195 °C: IR (KBr) 3307(N-H), 3060 (Ar-H), 2930,2820 (C-H), 1620 (C=N), 1603,1590 (C=C), 1420 (CH₂), 1260 (C-O-C asym.), 1030 (C-O-C sym).¹H NMR (DMSO-d₆, ppm) 3.00-3.20 and 3.40-3.60 (m, 4H, 2x-CH₂- of pyrazoline), 5.60-5.78 (m, 2H, 2x-CH- of pyrazoline), 8.4 (s, 1H, NH), 6.80-7.80 (m, 18H, Ar-H), 4.45 (s, 4H, 2 x -OCH₂-): Anal. (%) Calcd. for C₃₂H₃₀N₄O₂ (502.60): N, 11.15. Found N, 11.10.

7b: Colourless: DMF: yield 75%: m.p. 240 °C: IR (KBr) 3310(N-H), 3040 (Ar-H), 2900,2850 (C-H), 1630 (C=N), 1590, 1510 (C=C), 1450 (CH₂), 1250 (C-O-C asym.), 1080 (C-O-C sym).¹H NMR (DMSO-d₆, ppm) 3.18-3.24 and 3.60-3.89 (m, 4H, 2x-CH₂- of pyrazoline), 5.53-5.60 (m, 2H, 2x-CH- of pyrazoline), 8.9 (s, 1H, NH), 6.80-8.27 (m, 16H, Ar-H), 4.48 (s, 4H, 2 x -OCH₂-), 3.85 (s, 6H, 2x -OCH₃): Anal. (%) Calcd. for C₃₄H₃₄N₄O₄ (562.68): N, 9.95. Found N, 9.98.

Similarly other compounds **7c**, yield 64%, m.p. 192 °C, **7d** yield 66%, m.p. 180 °C and **7e**, yield 68%, m.p. 200 °C were prepared in colourless crystalline form after recrystallization of **7c** and **7d** with DMF-ethanol (80:20 v/v) mixture and **7e** with DMF-H₂O (80:20 v/v).

Results and Discussion

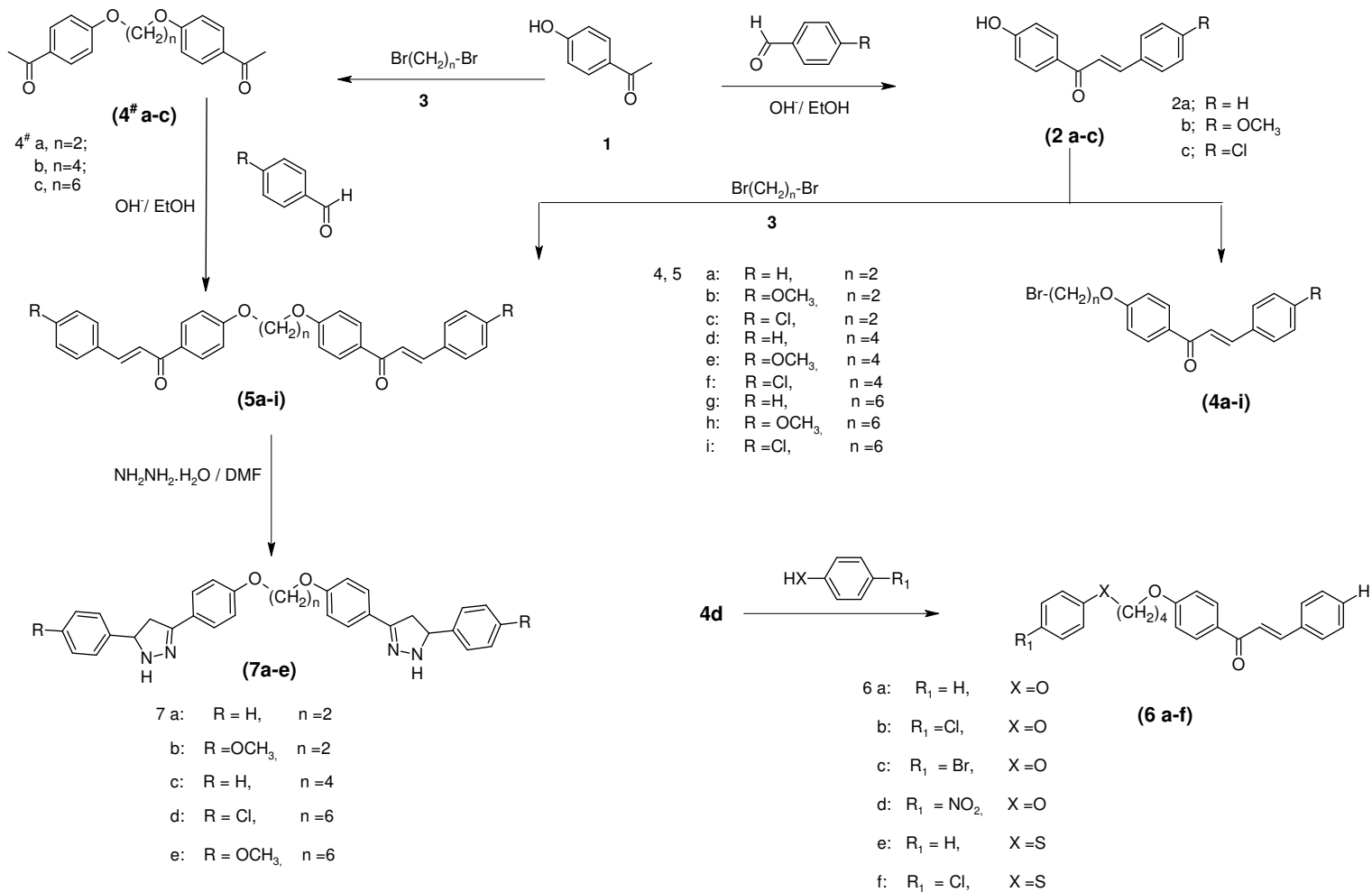
The synthesis of chalcones was effected via carbon-carbon bond formation using a Claisen-Schmidt condensation reaction. *p*-Hydroxyacetophenone (**1**) was condensed with appropriate aromatic aldehydes in equimolar ratio in presence of base at room temperature. The formation of chalcones (α,β -unsaturated ketones) **2**, was adjudged by the appearance of the carbonyl stretch at 1650-1670 cm⁻¹ and disappearance of carbonyl stretch at 1683.8 cm⁻¹ of *p*-hydroxyacetophenone. The trans alkene (*E*-form) nature of these chalcones was judged by ¹H NMR spectroscopy.

Compound **2** on refluxing with α,ω -dibromoalkanes (3, n= 2, 4 & 6) in equimolar ratio in presence of dry acetone and anhydrous potassium carbonate resulted two products which were separated by using ethanol. The ethanol soluble compound on purification was analyzed as 4'-[(ω -bromoalkyl)oxy]-substituted chalcones (**4a-i**). The ethanol insoluble compound was characterized as 4',4''-(polymethylenedioxy) bis substituted chalcone (**5a-i**) on the basis of its analysis and spectral results.

Synthesis of compounds (**5a-i**) was also carried out by another route. *p*-Hydroxyacetophenone (**1**) was treated with α,ω -dibromoalkane (3, n= 2, 4 & 6) in the molar ratio 2:1 in presence of acetone and anhydrous potassium carbonate. The reaction mixture after usual workup afforded 4',4''-(polymethylenedioxy)-diacetophenone (4[#]: n= 2, 4 & 6). Structure of these compounds was confirmed on the satisfactory elemental analysis and spectroscopic data. The details have been given in experimental section. Base catalyzed condensation of compounds (4[#] a, b & c) with different substituted aromatic aldehydes gave bis chalcones (**5a-i**). Identity of these compounds obtained by different routes was confirmed on basis of m.p., m.m.p., co-TLC and super imposable IR spectra.

Compound 4'-[(4-bromobutyl)oxy] chalcone (**4d**) on treatment with different *p*-substituted phenols and thiophenols resulted compounds (**6a-f**) characterized as 4'-{[(4-(*p*-substituted phenoxy / thiophenoxy)butyl)oxy] chalcones.

Finally compounds (**5a**, **5b**, **5d**, **5h** and **5i**) were treated with hydrazine hydrate in presence of DMF afforded the corresponding bis-2-pyrazolines (**7a-e**). The analytical and spectral data of synthesized compounds were in conformity with the proposed structure (Scheme 1).



Conclusion

A convenient synthetic methodology for a new class of chalcones, bis chalcones and bis-pyrazolines was described. It can be concluded that this class of compounds certainly holds great promise towards the pursuit for further synthetic and biological evaluation.

Acknowledgments

The authors are thankful to the Head department of Chemistry, University College of Science, M.L. Sukhadia University Udaipur-Rajasthan, for providing laboratory facilities. Thanks are also due to M/S Sadier Research Laboratories Inc, Philadelphia (USA) and Director CDRI Lucknow (UP) for elemental analysis and spectral data. One of us (RSS) is also thankful to UGC for financial assistance.

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