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Detailed Procedure for Bromination

(i) Bromination of o-Cresol (3) to 4-Bromo-o-cresol

To a suspension of 0.091 g (0.5 mmol) of V_2O_5 in 24 mL of acetonitrile-water (1:1) was added 1.8 mL (15.9 mmol) of 30% hydrogen peroxide and stirred for ca.5 min in an ice bath (~5 °C). To the clear solution were added sequentially 0.97 g (3 mmol) of tetrabutylammonium bromide and 0.108 g (1 mmol) o-cresol (3), and the whole was stirred for 1.5 h (~5 °C, ice bath). The solvent was removed *in vacuo* and the residue extracted with ethylacetate (3 x 20 mL). The organic extract was first washed with 5% sodium metabisulphite solution (2 x 10 mL) then with water (2 x 10 mL) and finally dried over anhydrous Na_2SO_4 . The solvent was removed *in vacuo* and the residue was purified by column chromatography (silica gel, hexane: ethylacetate = 24:1) to afford the product, 4-bromo-o-cresol. The yield was 0.173g (92 %).

M.P. 62 °C (Lit1 64 °C).

Anal. calcd for C_7H_7BrO : C, 44.95; H, 3.77; Br, 42.72.

Found: C, 45.02; H, 3.73; Br, 42.65%.

¹H-NMR, $\delta_{\rm H}$ (CDCl₃): 2.21 (s, 3H, -CH₃), 4.92 (brs, 1H, -OH), 6.63 (d, 1H, J = 8.4 Hz, ArH), 7.15 (dd, 1H, J₁= 2.2 Hz, J₂ = 8.4 Hz, ArH), 7.24 (d, 1H, J = 2.3 Hz, ArH).

¹Buckingham, J and Macdonald, F. Dictionary of Organic Compounds, vol.2, Chapman and Hall, London, 1996, pp.1050.

(ii) <u>Bromination of 4-Benzyloxy-4',6'-dimethoxy-2'-hydroxychalcone</u> (13) to 4-Benzyloxy-3'-bromo-4',6'-dimethoxy-2'-hydroxychalcone (16)

To a stirred suspension of 0.025 g (0.14 mmol) of V_2O_5 in 7 mL acetonitrile-water (1:1) was added 0.5 mL (4.42 mmol) of 30% hydrogen peroxide in an ice-bath (~5 °C). After 5 min, 0.271 g (0.84 mmol) of tetrabutylammonium bromide was added to the above solution followed by the addition of 0.11 (0.28 mmol) of 4-benzyloxy-4',6'-dimethoxy -2'-hydroxychalcone (13) and the whole was stirred for 1h (at ~5 °C, ice bath). The solvent was removed *in vacuo* and the residue extracted with dichloromethane (3 x 10 mL). The organic extract was first washed with 5% sodium metabisulphite solution (2 x 5 ml), then with water (2 x 10 mL) and finally dried over anhydrous Na_2SO_4 . The solvent was removed *in vacuo* and the residue was purified by column chromatography (silica gel, petroleum ether : ethylacetate = 9 : 1) to afford the product, 4-benzyloxy-3'-bromo-4',6'-dimethoxy-2'-hydroxychalcone (16) as yellow crystals. The yield was 0.095 g (72%). M.P. 197-198°C.

Anal. calcd for C₂₄H₂₁BrO₅: C, 61.42; H, 4.52; Br, 17.02

Found: C, 61.35; H, 4.47; Br, 17.11%

¹H-NMR, $\delta_{\rm H}$ (CDCl₃): 3.97 (s, 3H, OCH₃), 3.98 (s, 3H, OCH₃), 5.10 (s, 2H, -OCH₂Ph), 6.03 (s, 1H, 5'H), 6.99 (d, 2H, J = 8.7 Hz, ArH), 7.34-7.45 (m, 5H, ArH), 7.54 (d, 2H, J = 8.7 Hz, ArH), 7.74 (d, 1H, J = 15.5 Hz, >CH=CH-), 7.82 (d, 1H, J = 15.5 Hz, >CH=CH-), 14.98 (s, 1H, OH, D₂O exchangeable)

¹³C-NMR, δ_C (CDCl₃): 56.05, 56.28, 70.08, 87.11, 90.20, 106.87, 115.24 (2C), 124.54, 127.45 (2C), 128.16, 128.20, 128.65 (2C), 130.26 (2C), 136.38, 143.42, 160.71, 161.71, 162.16, 163.17, 192.61.