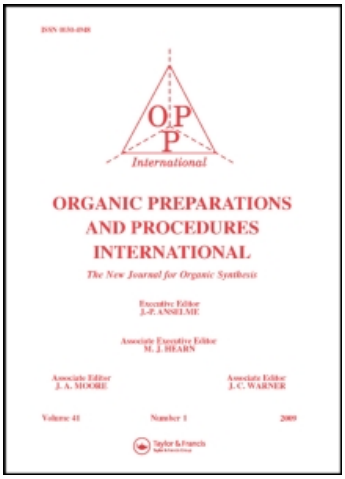

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METHYL γ-RESORCYLATE

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METHYL γ-RESORCYLATE

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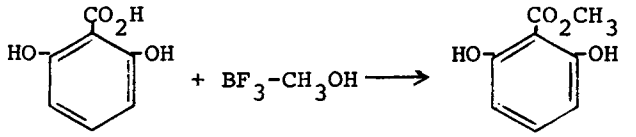

I II

Methyl γ-resorcylate (methyl 2,6-dihydroxybenzoate, II), an intermediate for the synthesis of biologically interesting heterocycles,[2-4] was needed as a starting material. Ester II has been prepared by the Fischer esterification of I (36% yield)[2] and by a two-step process involving the more expensive methyl iodide methylation of the silver salt of I (78% yield).[5] In this report, the less expensive one-step esterification of I with boron trifluoride-methanol led to chromatographically pure II (determined by thin-layer chromatography) in 55% yield.

EXPERIMENTAL[6]

A moisture-protected solution of 10 g. (0.065 mole) of γ-resorcylic acid (Aldrich Chemical Company; Karl Fischer titration showed the acid to contain 2.1% water) with 100 ml. of 12.5% boron trifluoride-methanol (Matheson Coleman and Bell) was refluxed for 4 days. A reflux period of 2 hrs. afforded the ester in only 18% yield while the final yield could not be improved beyond the 4 day reflux.[7] The product

was concentrated (rotary evaporator) and the residue was taken up in benzene. It was washed successively with water, aq. sodium bicarbonate, and water. The dried ($MgSO_4$) benzene solution was concentrated to about 50 ml. and chromatographed on a column (240 x 23 mm.) of 50 g. of silica gel (Davison grade 923, 100-200 mesh). Elution of the column with benzene (subsequent elution with dichloromethane afforded an additional small amount of ester) left on evaporation of solvent 6 g. (55%) of crystalline white ester, m.p. 68-70°, lit.[2] m.p. 69-71.

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1. Contribution No. 609 from Minnesota Mining and Manufacturing Company, Saint Paul, Minn. 55101

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6. Melting points were determined by the open capillary method on a Thomas Hoover unimelt apparatus and are uncorrected.

7. For a discussion of boron trifluoride in organic chemistry see A. V. Topchiev, S. V. Zavgorodnii, and Ya. M. Paushkin, "Boron Fluoride and its Compounds as Catalysts in Organic Chemistry," Pergamon Press, New York, N. Y., 1959.

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