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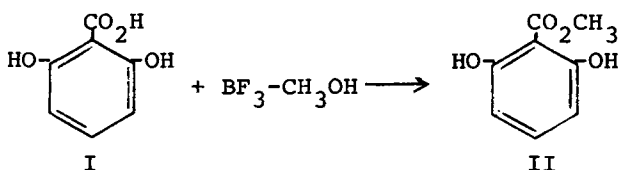
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METHYL γ -RESORCYLATE

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Methyl γ -resorcylicate (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.

EXPERIMENTAL⁶

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

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