

adding anhydrous calcium oxide (~5 times the amount of acid by weight) and excess methyl iodide (8–10 equivalents). After a reaction time of 4–6 hr at room temperature, the solids are removed by filtration or centrifugation and the esters are isolated by standard work-up procedures. The results obtained with eight different acids are summarized in Table 1. The method is particularly useful for a single-step conversion of hydroxycarboxylic acids into the corresponding methoxyesters.

Table 1. Esterification of Carboxylic Acids with DMSO/CH<sub>3</sub>I/CaO Reagent at Room Temperature (24°)

Acids	Reaction time hr	Product	Yield %
Adipic acid	8	Dimethyl adipate	91
Benzoic acid	8	Methyl benzoate	90
Cinnamic acid	8	Methyl cinnamate	93
Aleuritic acid <sup>4</sup>	18	Methyl aleuritate	79
Butolic acid <sup>5</sup>	8	Methyl butolate	86
Shellolic acid <sup>6</sup>	12	Dimethyl shellolate	81
Isolongifolic acid <sup>7</sup>	8	Methyl isolongifolate	84
Longiforic acid <sup>8</sup>	12	Dimethyl longiforate	86

Dimethyl sulfoxide was thoroughly dried by distillation from calcium metal. The methyl esters obtained were identified by usual methods (comparison with authentic specimens by G.L.C., I.R., and m.p. or mixture m.p.).

#### Dimethyl Adipate:

Adipic acid (730 mg, 0.5 mmol) was dissolved in dry dimethyl sulfoxide (5 ml) and methyl iodide (6.7 g, 5 mol) was added, followed by the addition of anhydrous calcium oxide (2 g) and drierite (~1 g). The reaction mixture was stirred at room temperature for 8 hr. It was then filtered, the solid on the filter washed with petroleum ether (20 ml), and water (15 ml) added to the filtrate. The filtrate was extracted with petroleum ether (4 × 15 ml), the extracts washed with a 10% solution of sodium thiosulfate (2 × 15 ml), with aqueous sodium carbonate (10 ml), and with aqueous sodium chloride. The solution was dried, the solvent removed, and the residue distilled in vacuo; yield: 790 mg (91%); b.p. 102–103°/10 mm. The product was G.L.C. pure; I.R.:  $\nu_{C=O} = 1747 \text{ cm}^{-1}$ .

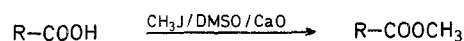
### A Convenient Preparation of Methyl Esters from Carboxylic Acids

Goverdhan MEHTA

Department of Chemistry, Indian Institute of Technology,  
Kanpur-16, India

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During an attempted etherification of 4-hydroxybenzoic acid by a modification<sup>1</sup> of the Kuhn methylation method<sup>2</sup>, we observed<sup>3</sup> that the product of the reaction was methyl 4-methoxybenzoate and not 4-methoxybenzoic acid (anisic acid) as was expected. The yield of ester was good and this prompted us to explore further the efficacy of the dimethyl sulfoxide/methyl iodide/base reagent in converting carboxylic acids into the corresponding methyl esters. The base employed was calcium oxide.



Our results were promising enough to encourage us to detail them in this report. The method consists of dissolving the acid in a minimum amount of dry dimethyl sulfoxide and

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