

A Modified Procedure for the Synthesis of *tert*-Butylacetic Acid

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In order to investigate steric effects in enamines we needed a series of neopentyl ketones and to prepare these ketones substantial amounts of 3,3-dimethylbutanoic acid (*tert*-butylacetic acid) were needed. 3,3-Dimethylbutanoic acid is accessible by several routes, *e.g.*, haloform cleavage of neopentyl methyl ketone,¹ *via* conjugate addition of methyl Grignard reagents to ethyl isopropylideneacyanoacetate^{2,3} and by the Willgerodt reaction of pinacolone.⁴ Recently a method using the boron trifluoride catalyzed addition of *tert*-butyl chloride to 1,1-dichloroethene appeared.⁵

We now report that this method can be modified by replacing gaseous boron trifluoride by the more easily handled and less expensive boron trifluoride dihydrate. The yields of 3,3-dimethylbutanoic acid by the modified procedure were in the range of 79–81% of isolated, distilled product in 1 mol runs to compare with the reported yield, 79%, by the original procedure.⁵

Experimental. Synthesis of 3,3-dimethylbutanoic acid. A typical procedure was: To a stirred solution of 33 ml of boron trifluoride dihydrate (*pract.* Fluka AG) in 200 ml of concentrated sulfuric acid was added dropwise over a period of 3 h a mixture of 92.5 g (1 mol) *tert*-butyl chloride (*puriss.* KEBO) and 146 g (1.5 mol) of 1,1-dichloroethene (*p.a.* KEBO). The temperature of the reaction mixture was maintained between 5–15 °C during addition. The mixture was then stirred at 10 °C for an additional 0.5 h, whereafter the mixture was poured onto crushed ice. The aqueous solution was saturated with sodium chloride and extracted several times with ether. Drying (MgSO₄), removal of solvent and distilling the crude product under reduced pressure afforded 94.0 g (81%) of pure (>98%, ¹H NMR) 3,3-dimethylbutanoic acid, b.p. 91–93 °C/20 mmHg (*litt.*⁶ 96 °C/26 mmHg).

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