ADDITIONS AND CORRECTIONS

VOL. 6, 1941

R. C. Huston and A. H. Agett, "The Reaction of Ethylene Oxide with Grignard's Reagent", Page 132. Our attention has been called to the fact that the melting points of the amide (95.5°) and the anilide (67°) of 3,4-dimethylpentanoic acid as reported in this journal (1) in 1941 do not agree with the melting points of these derivatives when the acid was prepared by (a) the reduction of 3-isopropyl-3-butenoic acid (2), (b) the treatment of the Grignard reagent from 2,3-dimethyl-1-bromobutane with carbon dioxide (2), and (c) the oxidation of 3,4-dimethylpentanal (3).

The acid has been again prepared in this laboratory by three methods, two of which are repetitions of those already reported (1). Trimethylethylene was treated with hydrogen bromide in the presence of benzoyl peroxide and sunlight and the tertiary halide was removed by repeated washings with water. One portion of the 3-methyl-2-bromobutane was converted into the Grignard reagent and treated with ethylene oxide. The resulting 3,4-dimethyl-1-pentanol was then oxidized to 3,4-dimethylpentanoic acid.

A second portion of the 3-methyl-2-bromobutane was converted into 3,4-dimethylpentanoic acid through the malonic ester synthesis.

The third method consisted of the following series of reactions.

Ethyl 3-hydroxy-3,4-dimethylpentanoate was prepared from methyl isopropyl ketone and ethyl bromoacetate by the Reformatski reaction. The hydroxyester was dehydrated to a mixture of ethyl 3,4-dimethylpentanoates. This mixture of esters was hydrogenated at low pressure over a platinum oxide catalyst to ethyl 3,4-dimethylpentanoate and this was in turn hydrolyzed to the acid. Another portion of the mixture of unsaturated esters was hydrogenated under high pressure over a copper oxide catalyst to 3,4-dimethyl-1-pentanol which was identified by the melting point (51–52°) of its 3,5-dinitrobenzoate (4).

The 3,4-dimethylpentanoic acid, prepared by any of the three methods, gave an amide which melted at 137–138° and an anilide which melted at 110–111°. Mixture melting points showed no depression.

(1) HUSTON AND AGETT, *J. Org. Chem.*, **6**, 132 (1941).
(2) Private communication from Dr. Avery A. Morton of the Massachusetts Institute of Technology, Cambridge, Mass.
(3) Private communication from Dr. J. C. Lalicero of the Rohm and Haas Company, Philadelphia, Pa.
(4) SCHMERLING, *J. Am. Chem. Soc.*, **67**, 1438 (1945).

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Ng. Ph. Buu-Hoï, P. Cagniant, Ng. Hoán, and Ng. H. Khôi, "Potential Nitrogen-Heterocycle Carcinogens. VI." Page 952. A clerical error on our part made us state the m.p. of *8-methyl-5-methoxy-1-tetralone* as 108°, and that of its semicarbazone as 200°, at variance with the literature. These should read: for the ketone, m.p. 38°, and for the semicarbazone, m.p. 240°.

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July 25, 1951.