

to the reference state of infinite dilution. We have since discovered that our communication was based upon an interpretation of their procedure which is erroneous, namely, that a separate extrapolation was made for each pair of solutions in order to determine the heat content of the solution at each final concentration. Instead, only the value for the lowest final concentration depends entirely upon extrapolation; the values for the heat content of all other recorded concentrations being obtainable by *alternately adding* and *subtracting* the measured values for the intermediate heats of dilution. Our objections to their procedure are consequently without foundation.

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#### ROTENONE. XXIV. SYNTHESIS OF TETRAHYDROTUBANOL

*Sir:*

As has been shown recently [Haller and LaForge, *THIS JOURNAL*, **53**, 4460 (1931); **54**, 1988 (1932)], tubaic acid ( $C_{12}H_{12}O_4$ ), obtained from rotenone by the action of alcoholic potash, is converted by drastic catalytic hydrogenation into tetrahydrotubaic acid ( $C_{12}H_{16}O_4$ ). When heated to its melting point, tetrahydrotubaic acid loses carbon dioxide and yields a crystalline product, tetrahydrotubanol ( $C_{11}H_{16}O_2$ ), which has been identified as an alkyl resorcinol. Previous work on rotenone and its derivatives indicated that the side chain is an isoamyl group. It was suggested that tetrahydrotubanol is 2,6-dihydroxy-1-isoamylbenzene. This compound has now been synthesized and found to be identical with tetrahydrotubanol. The synthesis was accomplished by the following set of reactions. 2,6-Dimethoxybenzotrile [Lobry de Bruyn, *Rec. trav. chim.*, **2**, 210 (1883)] was allowed to react with isobutylmagnesium bromide and the resulting ketimine was hydrolyzed to 2,6-dimethoxyphenyl isobutyl ketone. The ketone was reduced to 2,6-dimethoxy-1-isoamylbenzene, which on demethylation yielded 2,6-dihydroxy-1-isoamylbenzene. A comparison of its physical and optical properties with those of tetrahydrotubanol showed them to be identical. Experimental details will be presented in an early paper.

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