No difference in density between extreme fractions was found greater than 19 parts in 10,000,000.

HALIFAX, NOVA SCOTIA

NOTE

Monopotassium Phosphate.—The generally published value for the melting point of monopotassium phosphate is 96°. This value, unfortunately, appears in such standard reference books as the Handbook of Chemistry and Physics (published by The Chemical Rubber Company), Van Nostrand's Chemical Annual, the Smithsonian Physical Tables and the International Critical Tables. An investigation shows Tilden's determination to be the source of this datum. Gmelin-Kraut's Handbuch der anorganischen Chemie, Vol. 2, Part 1, p. 140, however, states "Thc crystals remain shiny at 204° (Graham). They melt upon heating to a clear, glassy potassium monometaphosphate which becomes opaque on cooling."

Reference to Tilden's paper, entitled "Note on the melting points and their relation to the solubility of hydrated salts"[1] reveals that he reports the melting points of 42 hydrated salts and the one anhydrous salt, KH_2PO_4. No explanation is given as to why this single anhydrous salt was used nor is it mentioned except in his list of compounds with their respective melting points. These melting points were determined in glass tubes, bent twice at right angles, into one leg of which moist filter paper had previously been inserted and that end sealed.

In connection with investigations carried on in this Laboratory, monopotassium phosphate was made by the neutralization of crystallized phosphoric acid of the highest purity[2] with pure potassium hydroxide. The salt was subjected to four recrystallizations and a 0.05 *M* solution had a *P*H value of 5.03 at 27.0°.

Pure monopotassium phosphate, prepared in the manner just stated, was not found to melt in contact with its boiling saturated aqueous solution Melting-point determinations were then made in melting-point capillaries. These were attached to a thermometer (Bureau of Standards No. 26387) and immersed in a bath of Crisco contained in a test-tube provided with a stirrer. This test-tube was in turn immersed in a bath of Crisco in a large tube also provided with a stirrer. Heating was carried on very slowly from 244° upward. No stem correction was necessary as the thermometer dipped into the bath beyond the points where the temperature readings were made. The melting point was not found to be sharp, fusion commencing at 252.6° and being complete at 254.3° (corr.) There is evidently no true melting point, fusion being accom-

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[1] Tilden, *J. Chem. Soc.*, **45**, 266 (1884).

[2] Ross, Jones and Durgin, *Ind. Eng. Chem.*, **17**, 1081 (1925).

panied by decomposition, as disclosed by an evolution of gas (water vapor) from the mass.

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THE REACTIVITY OF THE METHYLATED SUGARS. I. THE ACTION OF ALKALINE HYDROGEN PEROXIDE SOLUTION ON TETRAMETHYL-*d*-GLUCOSE

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The methylated sugars, because of the replacement of a labile hydrogen by a non-polar methyl group, would seem to offer an opportunity for direct experimental evidence of the validity of certain theories offered in explanation of the reactivity of the simple sugars.

Thus Nef[2] and his students extended the conceptions of Lobry de Bruyn,[3] Wohl and Neuberg[4] and Fischer[5] to a comprehensive explanation not only of the reciprocal interconversion of sugars under the influence of mild alkalies, but to the formation of isomeric ketoses, and various ene-diols whose subsequent dissociation led to the formation, in the presence of oxidizing agents, of the several acids containing from one to six carbon atoms, found experimentally. Nef believed that these intermediate ene-diols formed by a process of selective addition and loss of water.[2a,6] In the presence of alkaline oxidizing agents, dissociation took place at the point of unsaturation into active aldehydic forms, which could either oxidize to the corresponding acids or rearrange to form ordinary aldehydes. These latter, in turn, might give rise to similar ene-diols, thus repeating the process.

Illustrating with *d*-glucose and the 1,2-ene-diol common to *d*-glucose, *d*-mannose and *d*-fructose

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[1] Abstracted from a dissertation submitted by Edwin Lysle Gustus to the Graduate School of Northwestern University, in partial fulfilment of the requirements for the degree of Doctor of Philosophy.

[2] (a) Nef, *Ann.*, **357**, 214 (1907); (b) **376**, 1 (1910); (c) **403**, 204 (1913). (d) Anderson, *Am. Chem. J.*, **42**, 401 (1909). (e) Lewis, *ibid.*, **42**, 301 (1909). (f) Glattfeld, *ibid.*, **50**, 135 (1913). (g) Lewis and Buckborough, THIS JOURNAL, **36**, 2385 (1914). (h) Jensen and Upson, *ibid.*, **47**, 3023 (1925).

[3] Lobry de Bruyn and Van Ekenstein, *Rec. trav. chim.*, **14**, 156, 203, 213 (1895); **16**, 162, 259, 262, 278 (1897); **19**, 1 (1900).

[4] Wohl and Neuberg, *Ber.*, **33**, 3099 (1900).

[5] Fischer, *Ber.*, **28**, 1149 (1895).

[6] Spoehr, *Am. Chem. J.*, **43**, 228 (1910).