Fraction 3 was analysed. Combustion analyses gave values of 62·4, 62·5 per cent carbon, and 11·4 per cent hydrogen; calculated values for methyl-2-azoxy-2,5-dimethylhexane are 62·8 per cent C, 11·6 per cent H. Molecular weights were 168, 170 for fraction 3 compared with a calculated 172. The yield of methyl-2-azoxy-2,5-dimethylhexane, considering fraction 2 to be mainly this compound, was 11·0 gm. (0·064 mole), 57 per cent. The refractive index was N_D^{20} 1·4330, and boiling point at 738·5 mm. was 184° with some decomposition.

Reduction with sodium in alcohol yielded the olefin 2,5-dimethyl-2-hexene as the only product isolated.

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[1] Aston, Menard and Mayberry, *J. Amer. Chem. Soc.*, **54**, 1592 (1932).
[2] Aston and Parker, *J. Amer. Chem. Soc.*, **56**, 1387 (1934).
[3] Langley, Lythgoe and Riggs, [*Chem. and Indust.*, 75 (1951)].
[4] Aston and Ailman, *J. Amer. Chem. Soc.*, **60**, 1930 (1938).

Improved Spraying Reagents for the Detection of Sugars on Paper Chromatograms

PHENOLIC spraying reagents for the detection of sugars on paper chromatograms as introduced by Forsyth[1], while greatly improving the selectivity of the method, had the disadvantages that the mineral acid attacked the paper, making it brittle when dried, and giving rise to dark background colours, and that the solvent caused diffusion of the sugar, with consequent poor definition of the spot boundary. Partridge[2] found reduced attack on the paper by the use of trichloracetic acid as acidifying agent, and reported improved spot definition with aniline hydrogen phthalate dissolved in butanol[3]. Other agents proposed have included aniline hydrogen oxalate[4], aniline – trichloracetic acid[5], benzidine – acetic acid[6], and ethanolic solutions of phenols and amines acidified with small amounts of hydrochloric acid[7].

Experiments carried out in this laboratory have shown that good definition with only moderate attack on the paper can be obtained by using butanol solutions of phenols (0·2 per cent), acidified immediately before use with an equal volume of 0·25 *N* hydrochloric acid in butanol. Tests carried out using phthalic and acetic acids as acidifying agents for phenols showed that these acids were too weak to be effective; but consideration of the relative strengths of acids indicated that phosphoric acid might prove suitable. This acid has been used by de Whalley[8] for the detection and estimation of raffinose. Chromatograms were prepared carrying rhamnose, xylose, arabinose, dextrose, galactose, mannose, lævulose, sorbose, sucrose, maltose, lactose and raffinose, using upward development in *n*-butanol : ethanol : water (4 : 1 : 5). These were sprayed with 0·2 per cent ethanolic solutions of resorcinol; naphthoresorcinol; orcinol; alpha-naphthol and phloroglucinol, each reagent being acidified with (i) an equal volume of 0·25 *N* hydrochloric acid, and (ii) 0·1 volume of orthophosphoric acid (1·85 sp. gr.).

In each case acidification with phosphoric acid increased the number of sugars detectable by the reagent. For example, naphthoresorcinol – hydrochloric acid showed only lævulose, sorbose, sucrose and raffinose, whereas phosphoric acid brought up all the twelve sugars mentioned. Partridge[2] noted that pentoses could be detected by naphthoresorcinol on heating the sprayed chromatogram in a moist atmosphere, and it was observed when using phosphoric acid that the papers always felt damp after heating; the enhanced effects found may be due to the hygroscopic nature of this acid. The spot definition was good with phosphoric acid in ethanol, but in general no improvement resulted from preparing the reagents in butanol. With alpha-naphthol, butanol reduced both the range of the reagent and the clarity of the spots.

A reagent prepared by mixing butanol solutions of 2 *N* aniline (1 volume) and 2 *N* phosphoric acid (2 volumes) showed an increased range over aniline hydrogen phthalate, all twelve sugars again being detectable.

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[1] Forsyth, W. G. C., *Nature*, **161**, 239 (1946).
[2] Partridge, S. M., *Biochem. J.*, **42**, 238 (1948).
[3] Partridge, S. M., *Nature*, **164**, 443 (1949).
[4] Horrocks, R. H., and Manning, G. B., *Lancet*, **256**, 1042 (1949).
[5] Hirst, E. L., and Jones, J. K. N., *Discuss. Farad. Soc.*, No. 7, 268 (1949).
[6] Horrocks, R. H., *Nature*, **164**, 444 (1949).
[7] Novellie, L., *Nature*, **166**, 745 (1950).
[8] de Whalley, H. C. S., *Inter. Sugar J.*, **52**, 127, 151 (1950).

Inorganic Analysis by Paper Ionophoresis

SINCE the description of the electrophoresis of amino-acids by Wieland and Fischer[1] several workers[2,3] have made observations on the ionophoresis of inorganic ions inside filter paper. However, these studies were purely theoretical and did not indicate the possibility of separating inorganic ions for analytical purposes. Preliminary work in this laboratory[4] has shown that with the use of 1 *N* hydrochloric acid as electrolyte and 70 volts, a number of mixtures of metal ions can be separated into three fractions: anionic, cationic and isoelectric.

In this communication further experiments along these lines are described which indicate that separations analogous to those obtained by paper chromatography are possible by the use of ionophoretic techniques.

The technique employed was essentially that of Durrum[5]. A strip of paper 1 cm. × 20 cm. is hung over a T-shaped glass rod so as to hang with its ends in one limb of U-tubes filled with the electrolyte, and the whole is covered with an inverted gas jar. Carbon electrodes are placed in the other limb of the U-tubes and connected to a source of D.C. of 70–150 volts. The solution to be analysed is placed at the apex of the paper strip and the paper strip moistened carefully with electrolyte solution. Current is passed for two to three hours, the paper removed and held in hydrogen sulphide or other suitable reagent.

In the earlier communication[4], it was noticed that the copper group cations could be resolved into three bands, namely, copper–cadmium–lead, bis-