note that about 60% of the total activity moves with the R_F value of $PoCl_6^{2-}$ and thus consists of either reversible hydrolysis products or unhydrolysed Po; only about 40% of the activity moves slower.

Since MATSUURA *et al.*[1] have also observed that the hydrolysis products can only be destroyed in HCl that is much more concentrated than 1 *N*, it seems that chromatography with butanol–*N* HCl can serve for the detection of hydrolysis of Po in HNO_3 solutions.

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Separation of 2,4-dinitrophenylhydrazones of aldehydes and ketones on paraffin-treated paper

Several methods have been described for paper chromatographic separation of 2,4-dinitrophenylhydrazones (DNP-hydrazones) of aldehydes and ketones. Methods employing conventional techniques, *e.g.* MEIGH[1], or procedures in which silicic acid-treated paper is used[2,3] are unsatisfactory. BUYSKE *et al.*[4] suggested separation of DNP-hydrazones on paper treated with N,N-dimethylformamide, but emphasised that a right control of temperature was necessary at 15–18°.

Reversed-phase chromatography seems to be the most satisfactory technique for the separation of the sparingly soluble DNP-hydrazones. Procedures employing this principle have been described by KOSTIR AND SLAVIK[5], using acetylated paper, and by MEIGH[6] who treated paper with dichlorodimethylsilane. The details given for the preparation of both types of paper are too laborious and time-consuming. More suitable ways of separating DNP-hydrazones have been reported by ELLIS *et al.*[7] who used filter paper impregnated with (a) propylene glycol or (b) vaseline, and developed the chromatograms with (a) Skelly Solve C–methanol and (b) aqueous methanol.

Chromatography of DNP-hydrazones of particular groups has also been reported. Thus SUNDT AND WINTER[8] have described the separation of derivatives of aromatic carbonyl compounds on paper treated with N,N-dimethylformamide using cyclohexane–cyclohexene as the solvent. BREUER[9] used a similar type of paper, for separa-

ting hydrazones of aromatic ketones, but developed the paper with cyclohexane–carbon tetrachloride–dimethylformamide. DUDEK AND STUCHLIK[10] treated paper with ligroin and used it for the separation of DNP-hydrazones of cyclic ketones.

The purpose of this communication is to suggest that the method described previously (ASATOOR[11]) for the separation of 2,4-dinitrophenyl derivatives of amines (DNP-amines) is also applicable to DNP-hydrazones of aldehydes and ketones. The procedure involves reversed-phase chromatography on Whatman No. 3 MM paper, impregnated with liquid paraffin, using the upper phase of a mixture of chloroform–methanol–water–liquid paraffin (10:10:6:4) as the solvent. Fig. 1 shows that good separations of DNP-hydrazones of some aliphatic aldehydes and ketones can be achieved in this manner.

DNP-hydrazones of aldehydes and ketones (Table I) were prepared by the method described by MANN AND SAUNDERS[12] and melting points were compared with figures given in the literature (BEILSTEIN[13]; HEILBRON AND BUNBURY[14]). Treatment of the paper with liquid paraffin and the preparation of the solvent were carried out as described before[11]. Chromatograms were run by the descending technique for 16 h. The

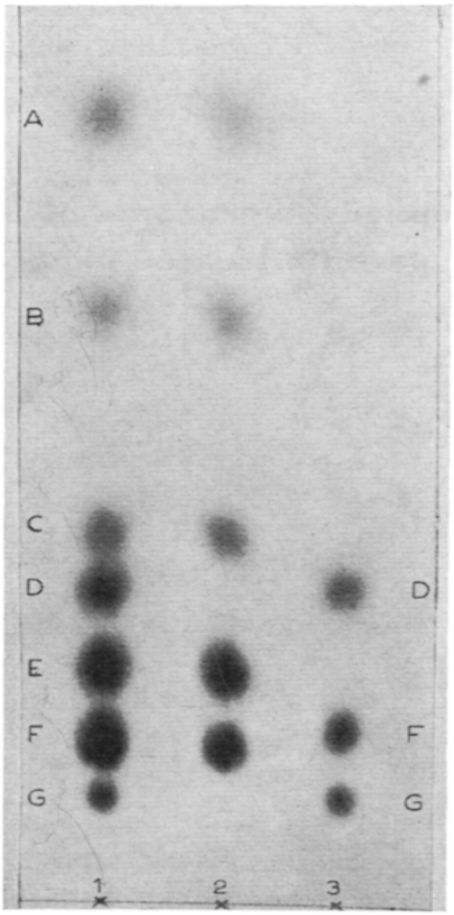

Fig. 1. 2,4-Dinitrophenylhydrazones of aldehydes and ketones. (1) DNP-hydrazone of: A. formaldehyde; B. acetaldehyde; C. propionaldehyde; D. acetone; E. butyraldehyde; F. isovaleraldehyde and methyl ethyl ketone; G. methyl *n*-propyl ketone. (2) A, B, C, E, F (derivatives of aldehydes). (3) D, F, G (derivatives of ketones).

spots were conveniently located by their characteristic appearance under u.v. light (Hanovia lamp, fitted with a Wood's glass filter). A permanent record was made by direct photography of u.v. illuminated chromatograms (Fig. 1).

There is considerable variation in R_F values owing to differences in the amount of paraffin incorporated into the paper, hence it is preferable to express the rate of

TABLE I

CHROMATOGRAPHY ON PARAFFIN PAPER

DNP-hydrazone of	R_{HCHO} *(mean of 5 experiments)*
Formaldehyde	1
Acetaldehyde	0.76
Propionaldehyde	0.47
n-Butyraldehyde	0.29
Isovaleraldehyde	0.19
Acetone	0.39
Methyl ethyl ketone	0.19
Methyl *n*-propyl ketone	0.13

movement of each spot relative to that of DNP-hydrazone of formaldehyde. This is illustrated in Table I where:

$$R_{HCHO} = \frac{\text{Distance travelled by DNP-hydrazone}}{\text{Distance travelled by DNP-hydrazone of formaldehyde}}$$

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