

The formation of double spots by Po(IV) stored in nitric acid solution

A recent paper by MATSUURA *et al.*¹ on the incomplete extraction of Po(IV) from certain aged solutions into hexone prompts us to describe similar results obtained in a paper chromatographic study of the purity of ²¹⁰Po tracer.

An aged solution of ²¹⁰Po stored in 1 *N* HNO₃ (as obtained from Harwell and left for 14 months) was chromatographed with butanol-*N* HCl. The fast moving spot obtained by other authors² in this solvent is due to a chloro-complex (probably PoCl₆²⁻), and it was assumed that this would form instantaneously during development as is the case with similar ions such as Bi(III) and Hg(II), which move with the same *R_F* value irrespective of whether they are applied as nitrates or chlorides, providing there is no undue excess of nitrate or HNO₃ in the solution to be chromatographed.

However, the ²¹⁰Po yielded two adjacent peaks as shown in Fig. 1. Chromato-

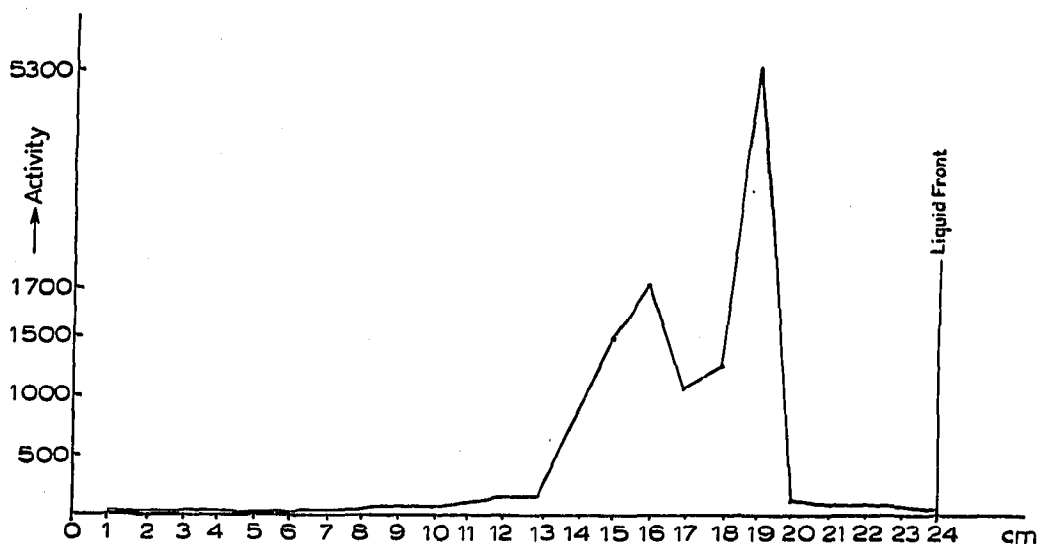


Fig. 1. Chromatogram of a ²¹⁰Po source kept in *N* HNO₃ (14 months) and developed with butanol-*N* HCl.

grams of mixtures of Bi(III), Hg(II), Au(III), Tl(III) and the ²¹⁰Po in amyl alcohol-2 *N* HCl and butanol-*N* HCl clearly showed that the second spot could not be due to any of these metals, which, however, are the only ones moving in the vicinity of the two Po peaks. The presence of higher or lower valencies of Po was then suspected and chromatograms were run of Po mixed with either FeCl₂ or K₂Cr₂O₇ and developed with butanol-HCl. While FeCl₂ failed to remove the slower spot, it disappeared after being mixed with dichromate. However, it also disappeared when evaporated to dryness and then taken up in about 6 *N* HCl or when both bands were eluted with 2 *N* HCl from a chromatogram and rechromatographed.

It thus seems that the second activity peak of the chromatogram (Fig. 1) is most likely the hydrolysis product observed by MATSUURA *et al.*¹. It is interesting to

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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¹ N. MATSUURA, A. OUCHI AND M. KOJIMA, *Bull. Chem. Soc. Japan*, 34 (1961) 411.

² E. E. DICKEY, *J. Chem. Educ.*, 30 (1953) 525.

³ F. W. LIMA, *J. Chem. Educ.*, 31 (1954) 153.

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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.* MEIGH¹, 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 SLAVIK⁵, using acetylated paper, and by MEIGH⁶ 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 WINTER⁸ have described the separation of derivatives of aromatic carbonyl compounds on paper treated with *N,N*-dimethylformamide using cyclohexane-cyclohexene as the solvent. BREUER⁹ used a similar type of paper, for separa-

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