haben den Farbton mehr ins Rot verschoben, als bei Entwicklung mit reinem Isatinreagens. Es wurde die in Tabelle I folgende Reihe bei Butanol–Eisessig–Wasser (4:1:5) nachgewiesen.

Die Farben entsprechen den Konzentrationen von 20 μg/cm^2. Die entwickelten Bogen wurden mit Paraffin fixiert.

Gegenüber den mit reinem Isatin gefärbten Bogen wurde eine stärkere Intensität der mit Isatin–Cd-Komplex angefärbten Flecke beobachtet und eine grössere Farbendifferenz, besonders bei Mischungen von Aminosäuren mit naheliegenden R_F-Werten wie: Cys-SH, Cys-SS, Lys, Hist, Arg und Tyr, Met, Val, Try, Norval. Gewisse Schwierigkeiten bieten ähnliche Farben von Arginin, Glycin, Valin und Leucin, welche jedoch wegen ihrer grossen R_F-Unterschiede ziemlich leicht identifiziert werden können.

Die nebeneinander wandernden Aminosäuren weisen keinen gleichen Farbton in Konzentrationen von 0.8–4 μg/cm^2 auf, und die Empfindlichkeit (siehe Tabelle I) der Farbreaktion ist gegenüber dem Isatin höher, und der Empfindlichkeit der Ninhydrinreaktion ähnlich[5–7]. Ausser obengenannten Eigenschaften, die dem besprochenen Isatin-Reagens einen Vorteil gegenüber reiner Isatin-Färbung geben, konnte festgestellt werden, dass die mit Isatin–Cd-Komplex gefärbten Bogen eine grössere Haltbarkeit aufweisen. Während die mit reinem Isatin gefärbten Flecke schon nach dreiwochiger Aufbewahrung keine qualitative Auswertung gestatten, ist die mit Isatin–Cd-Komplex hervorgerufene Farbe noch nach derselben Zeit deutlich und die Flecke noch leicht erkennbar.

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[1] J. BARROLLIER, *Naturwiss.*, 42 (1955) 416.
[2] F. BODE, *Biochem. Z.*, 326 (1955) 423.
[3] J. NOWORYTKO UND M. SARNECKA-KELLER, *Acta Biochim. Polon.*, II, 1 (1955) 91.
[4] T. KNAUT, *16. Intern. Milchwirtschaftskongress*, II, 2 (1962) 828.
[5] J. BLAUTH-OPIENSKA, *et al.*, *Ann. Univ. Mariae Curie-Sklodowska, Lublin-Polonia*, D 11 (1956) 175.
[6] A. SAIFER UND J. ORESKES, *Anal. Chem.*, 28 (1956) 501.
[7] I. M. HAIS UND K. MAČEK, *Papirova chromatografie*, Praha, 1954.

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The exchange capacity of papers impregnated with zirconium phosphate

Papers impregnated with zirconium phosphate (ZP) have found extensive application in numerous problems[1]; however, so far only two methods of preparation have been used.

ALBERTI AND GRASSINI[2] dipped papers into a 30 % solution of zirconyl chloride in 4 *N* HCl, while CABRAL[3] used a 7 % solution; subsequent precipitation with excess H_3PO_4 and extensive washing with acid and water is practised in both methods.

No information was available so far concerning the relative exchange capacity of these papers. For such a study we prepared papers containing varying amounts of zirconium phosphate and studied the chromatographic behaviour of UO_2(II) and Pb(II). These two ions were selected because they have a suitable range of R_F values on all papers.

While carrying out this work we also wanted to obtain some data on the temperature variation of R_F values on zirconium phosphate papers. The chromatograms were thus performed at four different temperatures: 5°, 20°, 35° and 50°.

Strips (5 × 46 cm) of Whatman No. 1 filter paper were drawn as uniformly as possible through a solution of zirconyl chloride octahydrate (Merck) at different concentrations (5, 10, 15, 20, 25, 30%) in 4 *N* HCl and air dried for 12 h. The dry strips were then dipped into a 60 % solution of H_3PO_4 in 4 *N* HCl and after drying in air for 6 h, washed first in 2 *N* HCl for 30 min and then twice with distilled water for 30 min.

It is very important that the amounts of the various solutions for washing the paper and times of contact are strictly adhered to. R_F values were found to vary considerably with the number of washes of the paper. For example UO_2^{2+} developed with 1 *N* $HClO_4$ had an R_F value of 0.6 on a 5 % paper after three washes and of 0.8 after seven washes. On a 30 % paper the R_F value was 0.2 after three washes and 0.4 after seven washes.

All chromatograms were carried out with 1 % solutions of uranyl and lead nitrates in aqueous $HClO_4$ on paper strips 21 cm long (starting point 2 cm from one end). In order to obtain a convenient range of R_F values for measurements in the whole range of papers the uranyl ion was developed with 3 *N* $HClO_4$ and the lead ion with 0.4 *N* $HClO_4$.

Table I shows the R_F values obtained with a development of 1–2 h at the temperatures indicated.

A mathematical correlation between R_F values and the degree of impregnation may be obtained[4] using the equation:

$$\alpha = \left(\frac{1}{R_F} - 1\right) \frac{A_L}{A_S} \qquad (1)$$

where A_L/A_S is the ratio of the amounts of the mobile phase and the zirconium phosphate, the amount of cellulose remaining constant.

Here it can be assumed that the distribution coefficient α does not change with the degree of impregnation. Then $(1/R_F - 1)$ should vary linearly with an increase in A_S providing that A_L does not change appreciably due to the varying amounts of zirconium phosphate in the pores of the paper.

As shown in Fig. 1 this is indeed the case, good linear relationships between $(1/R_F - 1)$ and the degree of impregnation being obtained for the whole range of papers. Equation (1) may thus be used for calculating the separation effects which may be obtained with various degrees of impregnation if data on one kind of paper are available.

The temperature variation between 5° and 50° is not very considerable, somewhat higher R_F values being observed in all cases at higher temperatures. However there seems to be no definite advantage in working at any specific or at a rigidly controlled temperature.

TABLE I

R_F VALUES OF UO_2^{2+} AND Pb^{2+} ON PAPERS IMPREGNATED WITH VARIOUS AMOUNTS OF ZIRCONIUM PHOSPHATE AND DEVELOPED AT VARIOUS TEMPERATURES

	UO_2^{2+} ions developed with 3 N $HClO_4$						*Pb^{2+} ions developed with 0.4 N $HClO_4$*					
Paper impregnated with % Zr OCl_2	*5%*	*10%*	*15%*	*20%*	*25%*	*30%*	*Paper impregnated with % Zr OCl_2*	*5%*	*10%*	*15%*	*20%*	*25%*	*30%*
Temp. 5°	0.70	0.62	0.43	0.38	0.33	0.29	Temp. 5°	0.67	0.66	0.52	0.51	0.43	0.40
Temp. 20°	0.71	0.63	0.46	0.44	0.38	0.30	Temp. 20°	0.74	0.68	0.58	0.57	0.52	0.47
Temp. 35°	0.78	0.58	0.46	0.45	0.36	0.33	Temp. 35°	0.79	0.73	0.62	0.55	0.52	0.45
Temp. 50°	0.80	0.62	0.52	0.40	0.37	0.34	Temp. 50°	0.86	0.85	0.69	0.66	0.65	0.48

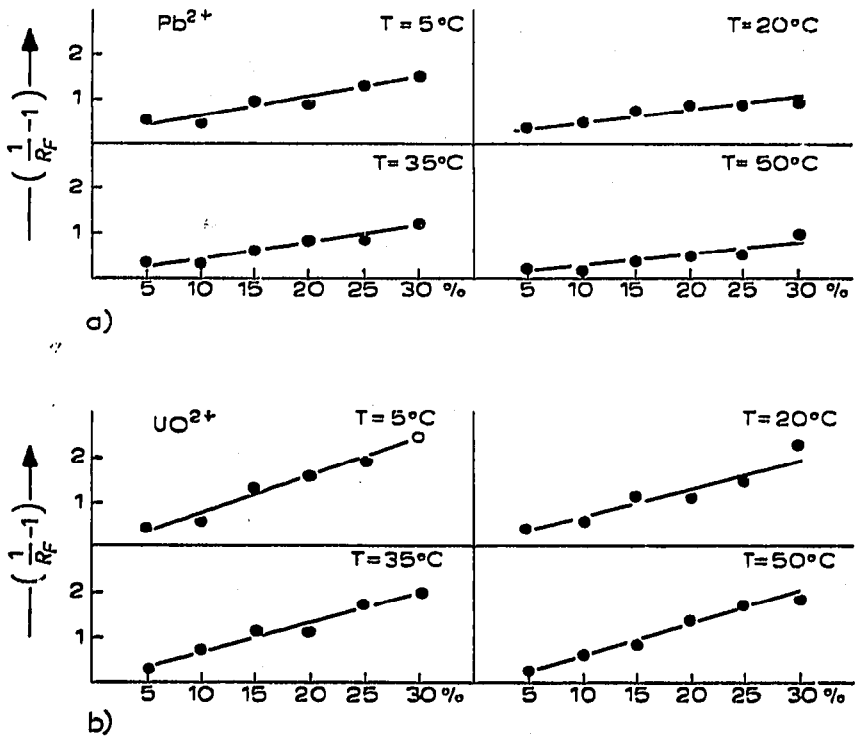

Fig. 1. $(1/R_F - 1)$ plotted against the amount of zirconium phosphate held on the paper. The zirconium phosphate content is here expressed in % $ZrOCl_2$ in the solution used to impregnate the papers. (a) Pb^{2+} ions developed at various temperatures with 0.4 N $HClO_4$. (b) UO_2^{2+} ions developed at various temperatures with 3 N $HClO_4$.

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1 J. P. ADLOFF, *J. Chromatog.*, 5 (1961) 366;
M. S. NUNES DA COSTA AND M. A. S. JERONIMO, *J. Chromatog.*, 5 (1961) 456;
G. ALBERTI AND G. GRASSINI, *J. Chromatog.*, 8 (1962) 103;
P. CATELLI, *J. Chromatog.*, 9 (1962) 534;
I. D. COUSSIO, G. B. MARINI-BETTÒLO AND V. MOSCATELLI, *J. Chromatog.*, 11 (1963) 238.
2 G. ALBERTI AND G. GRASSINI, *J. Chromatog.*, 4 (1960) 83.
3 J. M. PEIXOTO C. BRAL, *J. Chromatog.*, 4 (1960) 87.
4 M. LEDERER AND S. KERTES, *Anal. Chim. Acta*, 15 (1956) 226.

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