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Note

Gel chromatography of metal ions

The hydrolysis polymers of Cr(III) and Rh(III)

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Gel filtration has been used by several workers in order to detect the existence of hydrolysis polymers of metal ions. In a review of the literature[1], a number of metals that form zones excluded by various molecular sieves were listed. Work on the hydrolysis polymer of iron(III) also showed that irreversible adsorption and depolymerisation can occur on Sephadex gel, thus making the isolation of such polymers incomplete[2]. We have previously surveyed a number of metal ions by means of thin-layer gel chromatography[3] and found that not all metals form hydrolysis polymers that can be separated by gel filtration. For example, zirconium(IV) formed two zones while aluminium(III), lanthanum(III) and thorium(IV) did not, under the conditions that we used. We thought it of interest to extend the search for the existence of hydrolysis polymers and in this paper we report two further polymers that have not been described previously, those of chromium(III) and rhodium(III).

CHROMIUM(III)

About 5 g of $Cr(NO_3)_3 \cdot 9\ H_2O$ were dissolved in 2.5 ml of 0.1 *N* sodium nitrate solution, and 2 *N* sodium hydroxide solution was added in three 2-ml portions. An intensely green solution of pH 3.5 was obtained without any visible precipitate. Further additions of 2 *N* sodium hydroxide solution produced a precipitate.

Portions of 0.3 ml of this solution were chromatographed on Sephadex G-10, G-15, G-25 and LH-20 (column I.D., 1.2 cm; height of bed 25.5, 25.5, 29.9 and 24 cm, respectively) using 0.1 *N* sodium nitrate solution as eluent. Non-hydrolyzed chromium(III) yielded a single zone on all columns. Hydrolyzed solutions yielded three zones, of which one was eluted with the void volume of the column (as determined by the elution of Blue Dextran 2000) while the other two were only partially separated, the slower zone moving with the speed of the non-hydrolyzed ion. The results are given in Table I.

All zones were detected visually by their intense blue and green colours. It was also evident by visual examination that some irreversible adsorption coloured most of the column.

TABLE I

ELUTION VOLUMES OF Cr(III) SOLUTIONS ON SEPHADEX G-10, G-15 AND G-25 (DRY PARTICLE DIAMETER 20–80 μm) AND LH-20 (25–100 μm) COLUMNS, USING 0.1 N SODIUM NITRATE SOLUTION AS ELUENT

Sample	*Elution volume (ml)*			
	G-10	*G-15*	*G-25*	*LH-20*
Blue Dextran 2000	9.5	9.8	12.4	8.1
$Cr(NO_3)_3$ (as reference of low-molecular-weight species elution volume)	17.3	18.8	24.6	17.9
Hydrolyzed solution of Cr(III):				
First fraction	9.6	9.7	12.6	13.8
Last fraction	17.8	19.0	25.0	17.8

RHODIUM(III)

Rhodium(III) hydroxide was formed by precipitating Na_3RhCl_6 (6.4 g) solution with a slight excess of 1 N sodium hydroxide solution. Freshly precipitated rhodium(III) hydroxide was dissolved in the minimum volume of 0.5 N hydrochloric acid, to yield a yellow solution. A 0.3-ml volume of this solution was placed on a Sephadex LH-20 column and was separated into two well separated bands, of which the first was eluted with the same speed as Blue Dextran 2000 and was thus completely excluded from the gel. The results are given in Table II.

TABLE II

ELUTION VOLUMES OF Rh(III) SOLUTIONS ON A SEPHADEX LH-20 GEL COLUMN USING 0.5 N HYDROCHLORIC ACID AS ELUENT

Column 1.2 cm I.D.; bed length 28.5 cm.

Sample	*Elution volume (ml)*
Blue Dextran 2000	10.1
$Co(NH_3)_6^{3+}$ (low-molecular-weight ionic species)	15.2
$Rh(OH)_3$ dissolved in 0.5 N HCl:	
First fraction	10.1
Second fraction	18.8

REFERENCES

1 N. Yoza, *J. Chromatogr.*, 86 (1973) 325.
2 R. A. Henry and L. B. Rogers, *Separ. Sci.*, 3 (1968) 11.
3 M. Sinibaldi, *J. Chromatogr.*, 76 (1973) 280.