

amounts made it possible to use a sensitivity of "50 μA for full-scale" (Radiometer PO 4). Thus, it would be possible to separate still lower amounts of anions by the method described.

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- 1 W. KEMULA, *Roczniki Chem.*, 26 (1952) 281.
- 2 W. KEMULA AND A. GÓRSKI, *Roczniki Chem.*, 26 (1952) 639.
- 3 W. KEMULA, D. SYBILSKA AND J. GEISLER, *Roczniki Chem.*, 29 (1955) 643.
- 4 W. KEMULA AND J. WITWICKA, *Roczniki Chem.*, 29 (1955) 1153.
- 5 W. KEMULA AND Z. STACHURSKI, *Roczniki Chem.*, 30 (1956) 1285.

Received April 25th, 1967

J. Chromatog., 31 (1967) 266-268

Separation of halide anions on hydrous zirconium oxide

The hydrous oxide and other compounds of zirconium have successfully been used for separating inorganic cations¹⁻³. It has also been stated that hydrous zirconium oxide exhibits anion exchange properties^{2,4,5} but we suppose that it has not yet been employed for the separation of anion mixtures.

The purpose of the work has been to study the applicability of hydrous zirconium oxide for separating chloride, bromide and iodide mixtures.

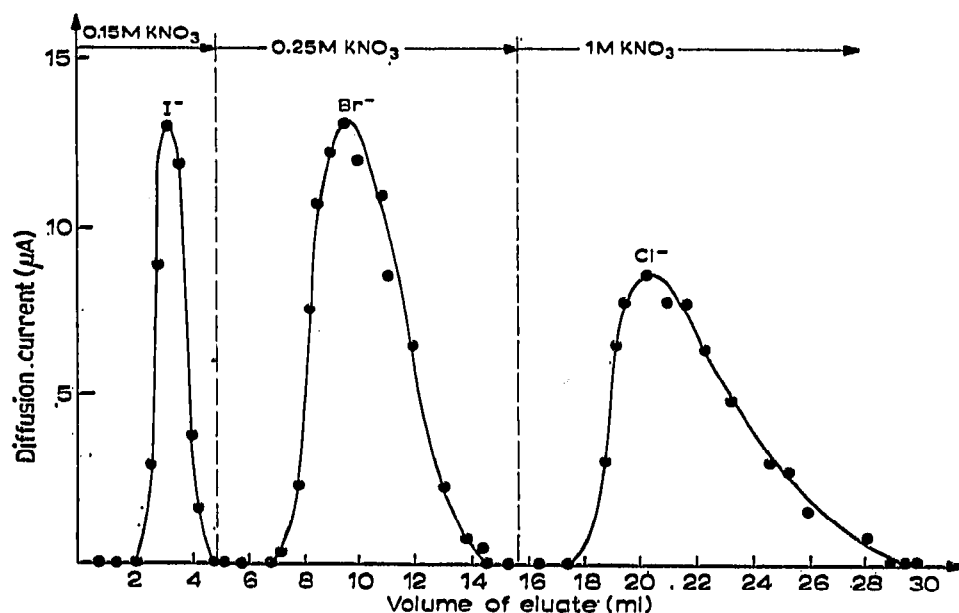


Fig. 1. Separation of I⁻, Br⁻ and Cl⁻ on a hydrous zirconium oxide, 0.075 cm² × 12 cm column.

Experimental

Preparation of hydrous zirconium oxide. 100 g of $ZrOCl_2 \cdot 8 H_2O$ was dissolved in 1500 ml of water. 1000 ml aqueous solution of 6 % NH_3 was added with continuous stirring. The precipitate was washed four times with water, by decantation; thereafter it was transferred onto the sintered glass funnel and washed till the disappearance of OH^- and Cl^- ions. The washed precipitate was dried in the air, ground and sized. Particles, the size of which was 0.12–0.15 mm, were dried at the temperature of $120 \pm 2^\circ$ till constant weight was established, (drying time approx. 15 h). The loss of weight after drying was found to be 18.5 %. The columns were prepared by introducing a suspension of the sorbent in 0.2 M KNO_3 into a glass tube with a sintered glass plate at the bottom. The column was washed with 0.2 M KNO_3 until all the Cl^- ions disappeared and then with 0.2 M HNO_3 till hydrogen ions appeared in the effluent. Bed volume dimensions were $0.075 \text{ cm}^2 \times 12 \text{ cm}$. A 50 μl sample containing 1.6 μmole KCl , 1.6 μmole KBr and 1.6 μmole KI was added to the column. The flow rate was 110 cm/h. I^- ion was eluted from the column with 0.15 M of KNO_3 solution, Br^- ion with 0.25 M KNO_3 , and Cl^- ion with 1 M KNO_3 . The effluent was analyzed by a chromatopolarographic method⁶ modified by the author for analyzing anions which produce anodic waves on the dropping mercury electrode⁷. The values of the diffusion current were read from the polarograms and registered at intervals of 2–5 min. A Radiometer PO₄ polariter was employed.

Results

As seen in Fig. 1 a complete separation of the three halide anions has been obtained. The order of appearance of the anions in the effluent is the same as that on aluminium oxide⁸ or ferric hydroxides⁹. The adsorbability of hydrous zirconium oxide and ortho-ferric hydroxide are very similar. Hydrous zirconium oxide, however, is less sensitive to attack by acids when compared with ortho-ferric hydroxide. But the two above-mentioned sorbents are superior to aluminium oxide with respect to capacity. The aluminium oxide column of $0.075 \text{ cm}^2 \times 29 \text{ cm}$ (treated with acid) does not permit a complete separation of halide anions⁸.

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- 1 K. A. KRAUS AND H. O. PHILLIPS, *J. Am. Chem. Soc.*, 78 (1956) 694.
- 2 K. A. KRAUS, H. O. PHILLIPS, T. A. CARLSON AND J. S. JOHNSON, *Proc. Intern. Conf. Peaceful Uses At. Energy, 2nd, Geneva, 1957*, published 1958, Vol. 28, paper 1832, p. 3.
- 3 C. B. AMPHLETT, *Proc. Intern. Conf. Peaceful Uses At. Energy, 2nd, Geneva, 1957*, published 1958, Vol. 28, Paper 271, p. 12.
- 4 K. A. KRAUS AND H. O. PHILLIPS, *J. Am. Chem. Soc.*, 78 (1956) 249.
- 5 C. B. AMPHLETT, L. A. McDONALD AND M. J. REDMAN, *J. Inorg. Nucl. Chem.*, 6 (1958) 236.
- 6 W. KEMULA, *Roczniki Chem.*, 26 (1952) 281.
- 7 S. TUSTANOWSKI, *J. Chromatog.*, 31 (1967) 266.
- 8 S. TUSTANOWSKI, *J. Chromatog.*, 31 (1967) 270.
- 9 A. LEWANDOWSKI AND S. TUSTANOWSKI, in preparation; S. TUSTANOWSKI, *Thesis*, Poznań, 1964.

Received April 25th, 1967