

Polarographic determination of anions in the effluent from chromatographic columns

The chromato-polarographic method was introduced by KEMULA in 1952¹. This author and his collaborators used it to separate inorganic cations^{1,2}, isomers of the organic nitro-compounds³, amino acids⁴, alkaloids⁵, etc.

This paper describes a modification of the method so that it can be used for halide anions in the effluent from chromatographic columns of 2–4 mm diameter. Owing to the fact that columns of such small diameter were used, a polarographic cell of extremely low capacity was required. In contrast to the cited papers by KEMULA the dropping mercury electrode was polarised anodically. To avoid the influence of

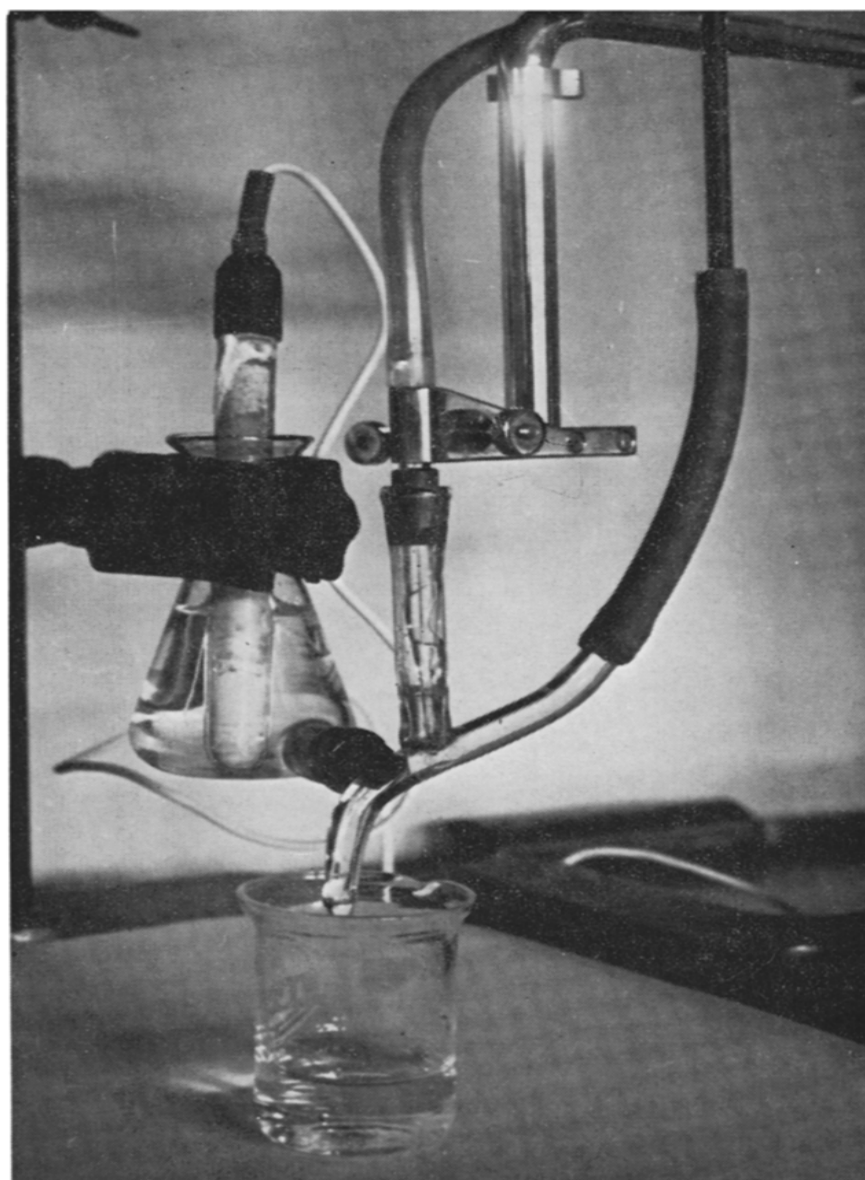


Fig. 1. General view of the polarographic cell.

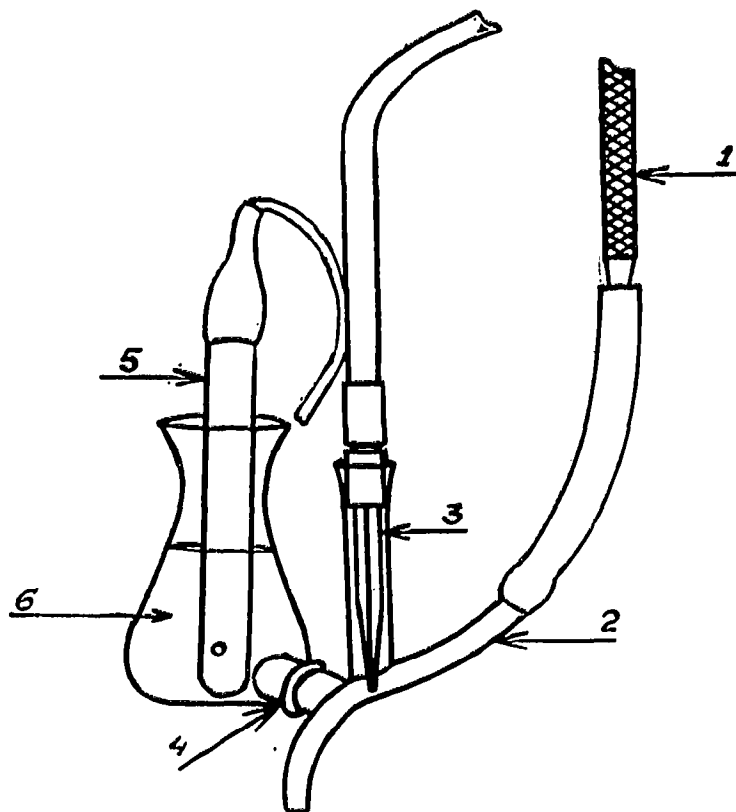


Fig. 2. Sketch of the polarographic cell. The description is given in the text.

analysed solution on the potential of the reference electrode, the internal mercury electrode was replaced by an external calomel electrode.

The general picture of the cell is shown in Fig. 1. A sketch of the cell is presented in Fig. 2. The main part of the cell is made up of a thick-walled capillary tube, its internal diameter being 1.5 mm. The effluent from the column (1) flows through the rubber tube to the capillary (2). Into its vertical arm is introduced a polarographic capillary (3), drawn so that the external diameter of the drawn end is 1 mm. This capillary is connected with the mercury reservoir. The part of the cell described above is connected by a capillary side-arm (4), which has an agar plug (agar in 0.2 M KNO_3), to the 25 ml flask (6) containing 0.2 M KNO_3 . The saturated calomel electrode (5) (Radiometer K 510) is submerged in this solution. While passing through the capillary (2), the effluent rinses the dropping electrode and leaves the cell jointly with the mercury drops.

The value of the diffusion current that corresponds to the concentration of the anion studied in the effluent was determined periodically at definite periods of time (2–5 min). The values of the current, expressed in microamperes, were plotted on a diagram as a function of the volume of eluant. It is also possible to record the elution curve continuously, as reported in the papers of KEMULA. Applying the present method, the following anions have been studied in an effluent: I^- , Br^- , Cl^- and SCN^- . Solutions of KNO_3 were used as eluant. The linear flow rate ranged from 20–210 cm/h. The amount of anions separated were from 0.5 to 3 μ mole. These

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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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.

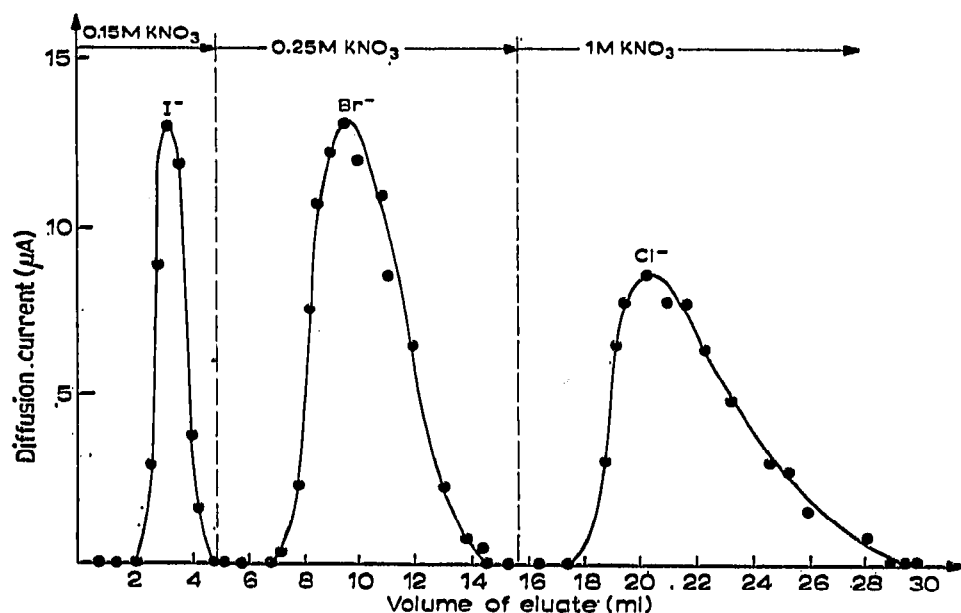


Fig. 1. Separation of I^- , Br^- and Cl^- on a hydrous zirconium oxide, $0.075 \text{ cm}^2 \times 12 \text{ cm}$ column.