

An apparatus for rapid circular electrophoresis on thin layers

Among the various TLC techniques, the circular method of HASHMI *et al.*¹ can be very useful for rapid evaluation of appropriate solvents and conditions. Therefore it seems important to apply this technique to TLE as well.

A simple perspex apparatus was devised (Fig. 1) consisting of an upper chamber of which the bottom plate is cooled by water flowing through box A. The thin-layer material (B, 6.5×7 cm) is pressed on top of A by a cover plate (C) which is fixed by screws (D). Voltage is applied between a central Pt electrode (F) and a circular Pt electrode (E) inserted into a circular groove in the rim of the cover plate.

The apparatus is especially suitable for thin layers on plastic sheets as they are better cooled and more resistant against the pressure of the central electrode (spring G) than layers on glass; however, thin glass plates can be used. Efficient cooling permits the use of high current densities. Before use the layer is briefly immersed in the selected electrolyte, and excess liquid is removed by blotting between filter paper. After insertion in the apparatus, a spot of the liquid to be analyzed (not more than $0.5 \mu\text{g}$) is applied through the hole for the central electrode. The electrode is carefully applied in order to prevent damage to the layer which will result in an irregular pattern of the ultimate electropherogram. After applying the voltage (500 V), the electric current strongly drops from 6 to 1.5 mA. Separations are carried out within a few minutes.

The method has been applied to the analysis of three groups of inorganic ions. In Fig. 2 is shown the result of an analysis of a mixture of several phosphorus containing acids (hypophosphorous P^1 , orthophosphorous P^3 , orthophosphoric P^5 and

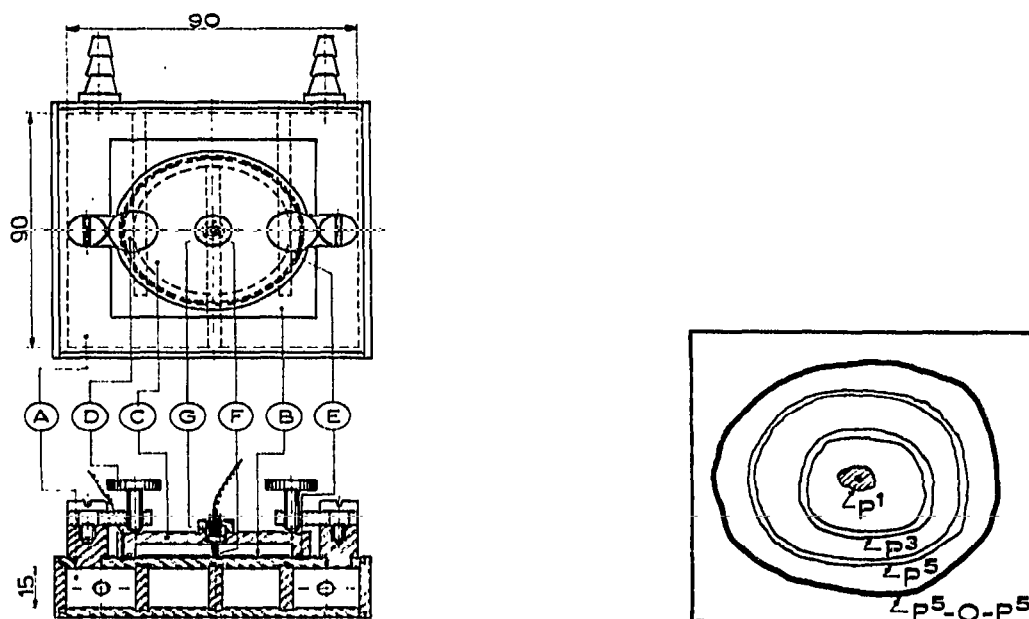


Fig. 1. Upper and side view of the apparatus for circular TLE. Dimensions in mm.

Fig. 2. Separation of the oxyphosphorous acids P^1 , P^3 , P^5 and $\text{P}^5\text{-O-P}^5$ in $0.005 M$ trichloroacetic acid at 500 V, 2 mA, 3 min. Eastman cellulose 6064 on plastic.

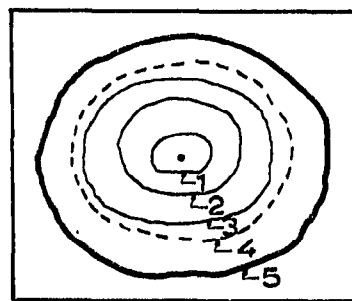
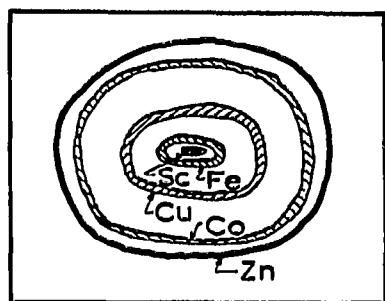


Fig. 3. Separation of the chlorides of Co, Cu, Zn, Fe(III) and Sc in 0.005 M α -hydroxyisobutyric acid; 500 V, 2 mA, 5 min. S & S cellulose, type 1440 on 1 mm thick glass.

Fig. 4. Separation of some iridium(III) aquahalo anions in 0.001 N HCl; 500 V, 1.5 mA, 5 min. SiO₂ MN N-HR on plastic. 1 = IrCl₃(H₂O)₃; 2 = Ir₂Cl₇(H₂O)₃⁻¹; 3 = IrCl₄(H₂O)₂⁻¹; 4 = Ir₂Cl₈(H₂O)₂⁻²; 5 = IrCl₅(H₂O)⁻².

pyrophosphoric P⁵-O-P⁵). The electrolyte was 0.005 M trichloroacetic acid. As can be seen, the four acids are well separated. Detection was done with the usual ammoniummolybdate-stannous chloride reagent.

Fig. 3 shows the separation of the metals Co, Cu, Zn, Fe and Sc in α -hydroxyisobutyric acid as electrolyte. Here, the detection was performed with a mixture of rubeanic acid, quercetin and PAN [1-(pyridyl-2'-azo)-naphthol-(2)] in aceton-ethanol (1:3).

In Fig. 4 can be seen the separation of aquahalocomplexes of iridium, with the general formula $[\text{}^{192}\text{Ir}_n(\text{III})\text{Cl}_{4n+2-x}(\text{H}_2\text{O})_x]^{x-n-2}$, using 0.001 N HCl as electrolyte. Detection was carried out by means of autoradiography with the Gevaert Structurix X-ray film².

As can be seen, the ultimate spots are not perfectly circular. Furthermore, the intensity along the rings is not constant, as it is difficult to place the tip of the central electrode exactly in the center of the sample. An objection to the method is the decreasing migration rate with time as a result of the changing current density. Thus no fixed value can be attributed to the transport rate of a specific species. Moreover, the circular spots tend to come together when they near the circular electrode so that an optimum time of analysis should be selected. However, the sequence in the transport rates is not different from those measured on electrophoresis strips.

In conclusion we can say that this simple apparatus can be used for a very rapid qualitative analysis. Moreover, it may be useful for a rapid selection of optimum separation conditions for a quantitative analysis of a specific mixture.

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