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Author
Ajuria-Garza, Sergio.

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Sergio Ajuria-Garza

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SIR: The use of centrifugal force to accelerate paper chromatography has been extensively developed in recent years (2). The objective of this investigation was to develop a method for the centrifugal acceleration of thin-layer chromatography to further enhance its inherent advantages (4).

Description of the apparatus. The apparatus consits essentially of two identical chromatographic chambers mounted on a horizontal rotor and driven by a universal electric motor (Figure 1). The rotor and the chambers are enclosed in a dry box to protect the operator against mechanical, chemical and radioactivity hazards.

Each chamber (Figure 2) contains a 5×30cm glass plate (the chromatogram), a solvent feeding tank and a solvent receiving tank, the latter for use in the continuous elution technique. When this technique is not used a lucite block is placed in the tank to reduce the free volume of the chamber. The solvent is transferred from the tank to the plate and viceversa through a strip of filter paper (Whatman No. 1) held against the adsorbent layer by a small lucite plate secured in a recess provided for that purpose.

The chambers are covered with a tight-fitting transparent lucite plate to which a scale can be affixed; this allows easy observation of the progress of the separation. The clearance between the surface of the chromatogram and the cover is about 3 mm. The chambers are firmly bolted by means of wing nuts to the aluminum rotor drived by a 1/3 hp universal electric motor controlled by a rheostat.

Method of operation. A wad of filter paper is placed in the feeding tank. The thin-layer plate, prepared in the usual manner, is placed in the chamber and fitted with the feeding filter paper strip. The sample solution is then applied from a micropipette at a line 1 cm from the end of the filter paper strip. An

appropriate solvent is added to the feeding tank and the chamber is tightly closed. It is convenient to start the motor only after the solvent has reached the initial spot by capillary action. In the early stages the process is almost as fast with the centrifugal field as without it and a better solvent front is obtained in the latter case.

Pre-saturation of the chamber is unnecessary because of the small free volume of the chamber. The optimum operating speed is a function of the medium and the solvent used, but it is generally between 300 and 500 rpm.

Testing of the apparatus. The performance of the apparatus was tested with a mixture of 2% methyl red and 2% methylene blue in aqueous solution. The stationary phase was Dowex-1 ion-exchange resin (minus 400 mesh and 8% cross-linkage) and the mobile phase was distilled water. The R_f values obtained were zero for methyl red and one for methylene blue, with the solvent front at 12 cm from the origin.

Good separations were obtained in 20 minutes at 300 rpm.

Other techniques. Centrifugal acceleration of paper chromatography is also possible in the apparatus described above without any modification. The paper strip is placed over the glass plate, secured with the lucite holders and one of the ends is immersed in the feeding tank.

The chromatographic chambers can also be used for horizontal, ascending or descending chromatography using either thin-layer chromatographic plates or paper strips, and for multiple development separations (3).

For horizontal chromatography the procedure is the same as described previously, but the centrifugal field is not applied. Some separations proceed so rapidly of their own accord that centrifugal acceleration is not necessary.

For ascending or descending chromatography special tanks can be placed inside the chamber to allow feeding the solvent in the required manner.

Separation of Zr^{95} and Nb^{95} and of Zr^{95} and Hf^{181}. A mixture of Zr^{95} and Nb^{95} has been separated by ascending thin-layer chromatography in 5x30cm glass plates using Dowex-1 ion-exchange resin (minus 400 mesh and 8% crosslinkage) as stationary phase and 10 M hydrochloric acid as mobile phase (1). The R_f values are zero for Zr and one for Nb. A development time of one hour is required for the solvent front to travel 14.5 cm from the origin.

A mixture of niobium-free Zr^{95} and Hf^{95} was resolved under the same conditions requiring the same development time (1). The R_f values are zero for Zr and one for Hf.

For comparison, these two separations were carried out by the centrifugal technique in the apparatus described here, using the same stationary and mobile phases. The development time was reduced to 10 minutes in both cases.

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LITERATURE CITED

(1) Ajuria-Garza, S., Unpublished data, University of California, Lawrence Radiation Laboratory, Berkeley, California.

(2) Pavlicek, M., Rosmus, J., and Deyl Z., J. Chromatog. 7, 19 (1962).

(3) Stahl, E., Arch. Pharmaz. 292/64, 411 (1959).

(4) Truter, E. V., "Thin Film Chromatography," p. ix, Interscience Publishers, New York, 1963.

Sergio Ajuria-Garza

University of California
Lawrence Radiation Laboratory
Berkeley, California 94720

Figure Captions

Fig. 1. Apparatus for centrifugal acceleration of thin-layer chromatography. The enclosing box is not shown for greater clarity.

a. Chromatographic chambers
b. Rotor
c. Electric motor

Fig. 2. Chromatographic chamber

a. Glass plate
b. Solvent feeding tank
c. Solvent receiving tank
d. Paper strip holder
e. Cover

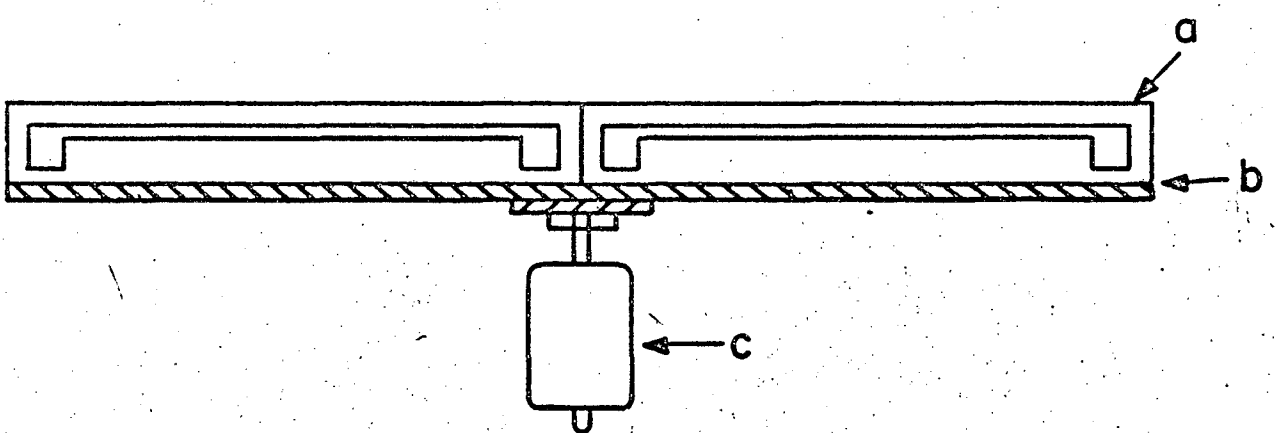

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Fig. 1.

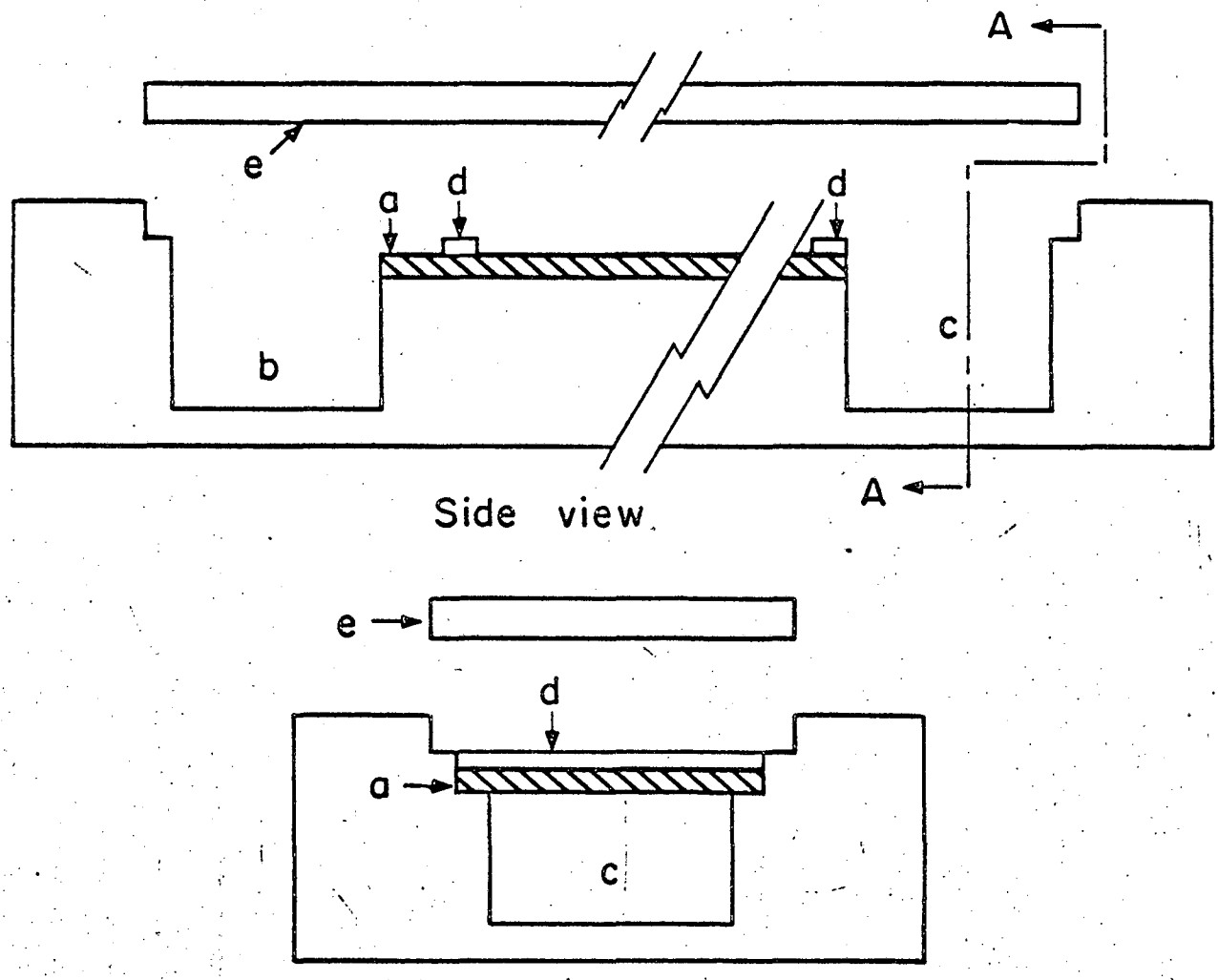

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Fig. 2.

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