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PAPER CHROMATOGRAPHY OF STEROIDS

David Kritchevsky and M.Calvin

July 28, 1950

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PAPER CHROMATOGRAPHY OF STEROIDS

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ABSTRACT

July 28, 1950

A method for the paper chromatography of sterols involving impregnated paper has been developed.

For publication in The Journal of the American Chemical Society

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(*) The work described in this paper was sponsored by the Atomic Energy Commission.

PAPER CHROMATOGRAPHY OF STEROIDS[1]

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The separation of cholesterol and cholestenone has been achieved by using paper impregnated with "Quilon"[2] as the stationary phase and simple primary alcohols as solvents. In effect, the stationary phase consists of the stearic acid residues.

Use of paper impregnated with rubber latex[3], silicic acid[4] and alumina[5] in paper chromatography has been reported. Of these, alumina paper was tried and found to give erratic results. The method of Zaffaroni and co-workers[6] for the paper chromatography of steroids using paper saturated with formamide

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(1) The work described in this paper was sponsored by the Atomic Energy Commission.

(2) Stearato chromic chloride, generously supplied by E. I. duPont de Nemours and Company, Inc.

(3) Boldingh, Experientia, 4, 270 (1948).

(4) Kirchner and Keller, J. Am. Chem. Soc., 72, 1867 (1950).

(5) Datta and Overell, Biochem. J., 44, xliii (1949).

(6) Zaffaroni, Burton and Keutmann, Science, 111, 6 (1950).

or propylene glycol as the stationary phase and a hydrocarbon solvent was also tried. In these experiments the steroids were found to move with the front. Using ordinary paper (Whatman No. 1), cholesterol was found either to move with the solvent front or remain at the origin.

For ease of location, tritiated cholesterol was used and the material located by scanning the paper with a windowless counting tube designed to locate weakly radiating substances on paper[7].

The presence of cholesterol at the points of high activity was confirmed by the red color developed after papers treated with a solution of silicotungstic acid were dried[8]. Cholestenone gave an olive green color with this reagent, but only when the steroid was present in relatively large amounts. Cholestenone was most easily detected by the yellow color obtained with a reagent consisting of a solution of iodine and potassium iodide in water[9].

The most satisfactory solvents, to date, have been methanol, ethanol, and ethanol-water 8:2. The latter solvent gives the best separation of cholesterol and cholestenone. The results are tabulated below.

Table 1

Solvent	Cholesterol (R_f)	Cholestenone (R_f)
Methanol	0.56	0.77
Ethanol	0.92	0.97
80% Ethanol	0.52	0.86

(7) Gray, Ikeda, Benson and Kritchevsky, Rev. Sci. Inst., in press.

(8) Montignie, Bull. soc. chim., 51, 690 (1932).

(9) Munier and Macheboeuf, Bull. soc. chim. Biol., 31, 1144 (1949).

All experiments were carried out as descending chromatograms using 1-1/2" x 15" strips of the impregnated paper. The paper was usually wet to a distance of about 25 cm. from the origin. R_f values were measured from the farthest point of the origin and the foremost point of the colored or active zone.

Projected work includes widening the range of usable solvents, development of supplementary color reactions and extension of this method to other steroids.

SUMMARY

A method for the paper chromatography of sterols involving impregnated paper has been developed.