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ABSTRACT

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by

David Kritchevsky and Martha R. Kirk

Radiation Laboratory and Department of Chemistry, University of California, Berkeley, California (*)

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March 26, 1951

The methods available for detection of steroids in paper chromatography have been summarized.

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

DETECTION OF STEROIDS IN PAPER CHROMATOGRAPHY

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The usual color tests for detection of steroids, many of which involve the use of corrosive acids, are not applicable to the detection of these compounds in paper chromatography. This report is an effort to systematize the current knowledge concerning the easy detection of steroids in paper chromatography. The methods listed below have all been applied in paper chromatography using "Quilon"-treated paper (1) with varying degrees of success. Several of these tests, which are negative or faint when applied to "Quilon"-treated paper, can, presumably, be used with untreated paper with good results.

The detection of many of these compounds through their ultraviolet absorption is difficult, if not impossible, when applied to "Quilon"-treated paper. It should work well with ordinary filter paper and probably can serve as a method of detection in papers treated with other impregnating materials.

A. Iodine - Various iodine reagents give the most satisfactory results. These are:

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1. Suspending the paper above iodine vapor. The spot is visible as the iodine color fades from the paper. This may be followed by a spray of saturated KI in methanol, then by a starch solution. The spot may show up darker blue than background, but generally is more clear on the reverse side of the paper where there is no interfering background color.

2. Dip paper in a saturated solution of I_2 in petroleum ether. (Can follow with KI and starch as above.)

3. Spray paper with saturated solution of I_2 in petroleum ether. (Can follow with KI and starch as above)

4. Suspend paper above solution of I_2 in chloroform heated to ca. 40°, (Can follow with KI and starch as above.)

5. Spray with solution of 0.2 g. I_2 and 2 g. KI in 200 cc. water. May follow with starch. (2).

B. Bromine - Suspend paper above 2% solution of Br_2 in chloroform at 40°. (3). May follow with KI and starch spray.

C. Trinitrobenzene in alcohol followed with 1 N sodium hydroxide.

D. Picric acid in alcohol followed with 0.1 N sodium hydroxide.

E. Ammoniacal silver reagent (for cortical steroids). (4)

F. 30% Zinc chloride in anhydrous methanol. Follow by heating to 130° in oven. (5).

G. Spray with 15-25% of $SbCl_5$ in chloroform.

H. Spray with a 15-25% solution of silicotungstic acid in alcohol. Follow by drying in oven for one or two minutes. (6). Longer time may destroy sprayed portion of the paper.

SUMMARY

The methods available for detection of steroids in paper chromatography have been summarized.

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