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THIN-LAYER CHROMATOGRAPHY OF ELEMENTAL SULPHUR

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SUMMARY

Elemental sulphur may be chromatographed on silica gel G using hexane as solvent and may be detected by a silver nitrate reagent down to levels of 0·02 µg. Hexachlorobenzene has the same R value under these conditions but confusion with sulphur in hexachlorobenzene identification may be reduced by ultraviolet irradiation of the plate prior to spraying of the reagent.

I. INTRODUCTION

During identification of a pesticide formulation by thin-layer chromatography it was found that, in addition to the spots from technical DDT, a strong spot with high R_f showed up. This was subsequently identified as elemental sulphur. Murphy and Nagy (1965) have reported the identification of elemental sulphur by thin-layer chromatography in amounts of 20–25 µg. The present paper reports detection of sulphur to levels of 0·02 µg and warns against its confusion with hexachlorobenzene (HCB) under certain circumstances.

Pearson, Aldrich and Stone (1967) have reported that elemental sulphur may be confused with aldrin during gas-liquid chromatography of pesticide residues using an electron-capture detector.

II. MATERIALS AND METHODS

Thin-layer chromatography plates.—Glass plates (20 cm x 20 cm and 20 cm x 10 cm) were spread with Merck silica gel G to a thickness of 0·25 mm. The plates were allowed to air-dry and were then washed using aqueous acetone (50% v/v). After air-drying, the plates were stored in an atmosphere of 40% relative humidity at 25°C.

Chromogenic reagent.—Silver nitrate (1·7 g) was dissolved in water (5 ml), and 2-phenoxyethanol (20 ml) and acetone (200 ml) were added to form the reagent as described by Mitchell (1958).

Sulphur.—Sulphur was precipitated from sodium thiosulphate solution by the addition of dilute hydrochloric acid. It was then filtered and washed with water and ethanol and dried in a vacuum desiccator over solid sodium hydroxide. Solutions of sulphur in carbon disulphide were used for application to the thin-layer chromatography plate.

Hexachlorobenzene.—Crystals m.p. 230°C.

Thin-layer chromatography.—The sample, dissolved in carbon disulphide or acetone, was applied 3.5 cm from the base of a plate using a capillary pipette (1, 2 or 5 μ l). A portion of the silica gel G 12 cm from the sample origin was scraped off and the developing solvent was run to this line.

The developing solvent was hexane and the atmosphere of the chamber was kept saturated by lining the walls with filter paper. Development time was approximately 15–20 min.

Detection methods.—Two procedures, which differed in only one step, were used.

The first procedure was based on that of Mitchell (1958). After development, the plate was allowed to dry in an air draught. After an even spraying with chromogenic reagent, the plate was exposed to the unfiltered ultraviolet light from a medium-pressure mercury arc for 2–5 min.

The second procedure was similar to that of Hamilton (1968). After development, the plate was allowed to dry in an air draught. It was then exposed to the ultraviolet radiation for 20 min. After an even spraying with chromogenic reagent, the plate was further exposed to the ultraviolet light for approximately 2 min.

III. RESULTS AND DISCUSSION

As can be seen from Figures 1 and 2, HCB and sulphur have the same R_f values on the silica gel G/hexane system. However, sulphur differs widely from the chlorinated compounds in its response to the two detection procedures. Without irradiation of the plate prior to spraying, HCB can be seen at the 2–5 μ g level and the sulphur spot can be observed at 0.02 μ g. Using the second procedure (20 min irradiation before spraying), the relative sensitivities are reversed. It now requires about 1 μ g of sulphur to form a visible spot, but the chlorinated pesticides can be seen at levels below 0.1 μ g.

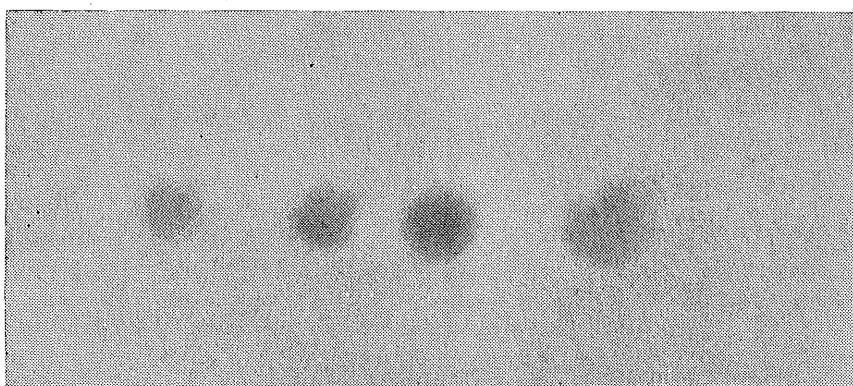


Fig. 1.—Spots, from left to right, sulphur 0.05 μ g, 0.1 μ g, 0.2 μ g and hexachlorobenzene 5 μ g. This plate was not irradiated before spraying with the chromogenic reagent.

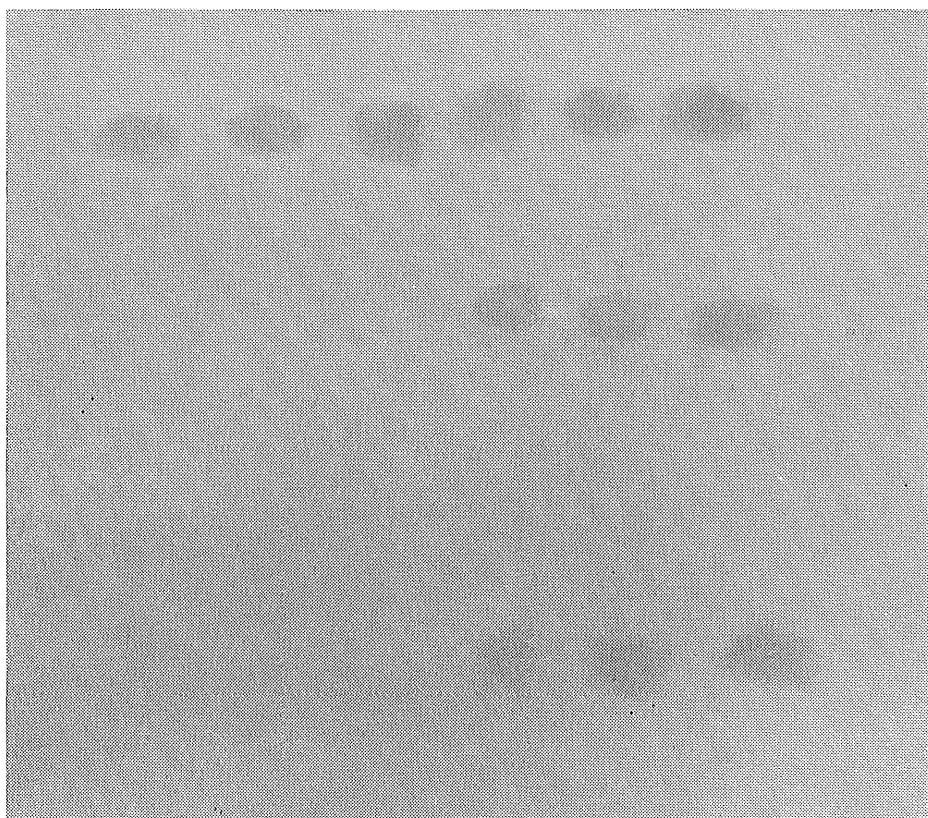


Fig. 2.—Spots, from left to right, sulphur 1 µg, 2 µg, 5 µg and a mixture of hexachlorobenzene, aldrin and p,p' DDT, 0.1 µg, 0.2 µg and 0.5 µg of each. This plate was irradiated for 20 min before spraying with the chromogenic reagent.

Another point is that the sulphur spots have a different appearance from those of HCB and this is most readily noticeable when the two sets of spots are on the same plate.

Sulphur comes through simple clean-up systems designed for chlorinated pesticides (Pearson, Aldrich and Stone 1967). If thin-layer chromatography is used as the identification step and the chemist is unaware that sulphur is present, then there is a possibility that the presence of HCB may be wrongly reported. H. E. Munro (personal communication, 1972) has found that sulphur comes through a long and complex clean-up procedure designed for 2,4-D residue analysis (Munro 1972). From this it appears that unless specific steps are taken to remove sulphur from a pesticide extract, there is a good chance it will come through the clean-up process.

If the plate is irradiated with ultraviolet light prior to spraying in the detection process, the sensitivity to sulphur is much reduced, thus decreasing the chance that sulphur will interfere with the thin-layer chromatographic identification of HCB.

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