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Short Communication

The thin-layer chromatography of some organomercurial antiseptics on cellulose thin layers with aqueous solvents

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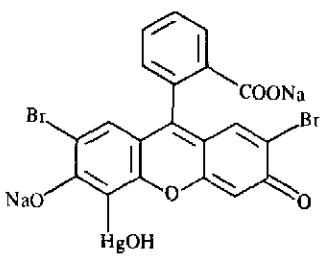
Introduction

In their book entitled *Plant Drug Analysis* Wagner *et al.* [1] presented an excellent collection of thin-layer chromatograms for the pharmacognostic identification of most drugs of natural origin. Synthetic drugs were not included in their collection.

In the authors' work on adsorption chromatography on cellulose (both by PC and TLC) some results were obtained for mercurial surface antiseptics that are of interest in pharmacognosy and these are reported here.

In Switzerland only two mercurials are extensively used, mercurochrome (synonyme merbromin) (I) and Merfen-Orange (Zyma, Nyon), the latter being phenylmercuric borate to which an orange dye (E 110) is added.

According to Schuster [2], Mercurochrome usually comprises of 13–40% acetylated and unacetylated dibromo(hydroxymercuri)fluorescein, 28–40% acetylated or unacetylated dibromobis(hydroxymercuri)fluorescein, 10–20% sodium dibromofluorescein and 0–7% sodium acetate. It is freshly prepared in the pharmacy, usually every 3 months, by boiling dibromofluorescein with mercuric acetate.



(I)

Hopes [3] described a polarographic method for determining mercurochrome and checked the purity of his standards by chromatography on silica gel layers with either ethanol–ether or acetic acid–chloroform as the developing solvent, to yield four or five spots.

A simple system is described here based on cellulose layers developed with 1 M NaCl, which seems to be satisfactory for distinguishing between different lots of mercurochrome obtained from a number of different pharmacies.

Experimental

Drops of the drug solutions are spotted directly on Macherey–Nagel Polygram Cel 300 precoated plastic sheets (40 × 80 mm), in order to yield spots 2–3 mm dia. The thin layer is then immediately developed with 1 M NaCl in small glass jars (11 cm high and 6 cm dia) closed with a rubber stopper. The solvent rises to the upper edge of the layer in about 10 min. Other aqueous solutions were examined as eluents; none gave better results than 1 M NaCl.

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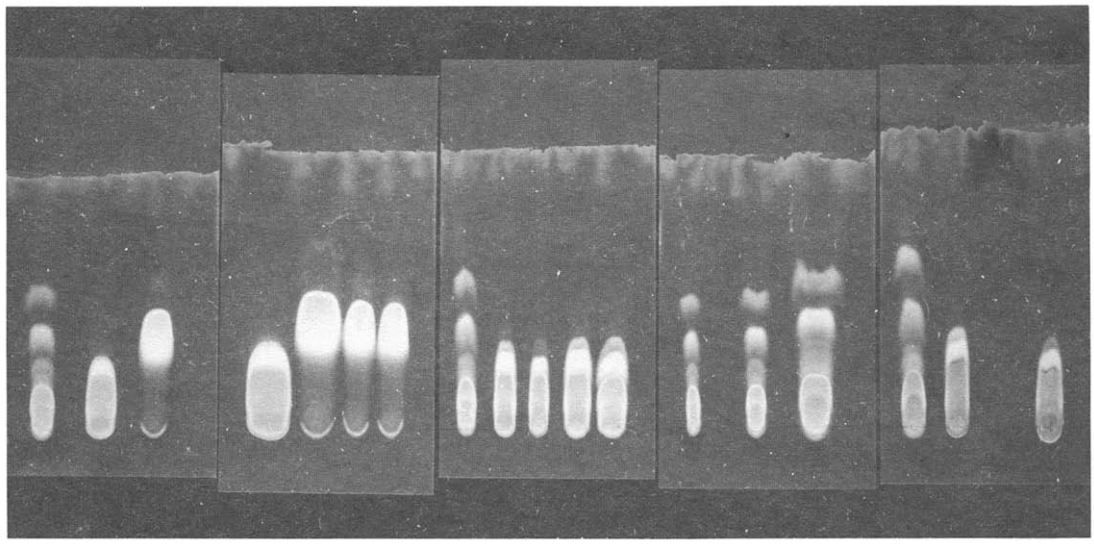

Figure 1
Chromatograms on Polygram Cel 300 precoated plastic sheets developed with 1 M NaCl and photographed under UV light (250–350 nm) with a Kodak 2A filter (absorbing up to 405 ml) with an Ektachrom daylight film. Description of tracks from left to right: layer 1, two samples of mercurochrome adjacent to one of the fluorescein; layer 2, various dilutions of fluorescein adjacent to a sample of mercurochrome; layer 3, five samples of mercurochrome collected from different pharmacies; layer 4, various loadings of the first sample of mercurochrome used for layer 3; layer 5, three samples of mercurochrome and one of $HgCl_2$. The chromatogram was exposed to H_2S fumes to detect $HgCl_2$.

Results

Samples of mercurochrome were obtained from five different pharmacies in central Lausanne over a period of 2 years and analysed by the proposed method. The results of interest are illustrated in Fig. 1. The first thin layer or track (from the left) shows the chromatograms of two samples of mercurochrome and next to them one of commercial fluorescein. A comparison with dibromofluorescein may have been advantageous, but none was available. The main constituents of mercurochrome are rather strongly adsorbed and seem to separate by displacement. The four faster zones presumably represent hydrolysed species. They were not found in all preparations examined. Six different UV fluorescing zones can be readily discerned in the first chromatogram using the CAMAG UV lamp for TLC. The second thin layer shows some heavily loaded chromatograms of mercurochrome and fluorescein. Here it was of interest to see whether further trace zones would become visible if larger amounts were applied. This was found not to be the case.

The third layer shows chromatograms of samples from five different pharmacies. All except the last two can be readily distinguished from each other by visual inspection. The fourth layer shows the same sample at three different loadings. The amount placed on the chromatogram does not affect the general appearance of the chromatographic pattern.

On the fifth layer are three samples of mercurochrome, run together with a spot of $HgCl_2$, and the entire chromatogram was exposed to H_2S fumes. Free mercury(II) moved with the liquid front and gave a black spot. None of three mercurochrome samples showed the presence of free mercury(II).

Some samples of mercurochrome were chromatographed after 1 and 2 years' storage, respectively, in the laboratory. There was no visible change with time. The developed chromatograms change colour on drying but are then quite stable for several weeks.

Merfen-Orange separates into a spot of phenylmercuric ion on the point of application and a spot of Orange E at $R_f = 0.145$. The phenylmercuric spot can be revealed by exposing to H_2S fumes or by dipping into a 1% chloroform solution of dithizone, the latter yielding a pink spot stable for some hours. No free mercury was detected, i.e. no black spot was observed at the solvent front.

References

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