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Note

Large-volume spotting apparatus and its application to the semi-quantitative determination of organochlorine pesticides in tea

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A simple and inexpensive field method for checking the level of organochlorine pesticide residues in tea is needed by tea-purchasing stations in China. Thin-layer chromatography (TLC) is suitable and different methods have been reported[1,2]. However, the pesticides have to be extracted, and the extracts concentrated and purified prior to the TLC determination. The usual techniques cannot be applied by the workers in tea-purchasing stations, who have little experience in chemistry. To simplify the operation, a device that can apply a relatively large volume of sample to a TLC plate and can carry out clean-up and spotting in one step was developed.

EXPERIMENTAL

Apparatus

Figs. 1 and 2 show the apparatus, which consists of spotters made from medical syringes, hair dryers and a modified magnetic agitator. The tip of the spotter is made from a syringe needle by cutting off the long and thin part and filling the base part tightly with a bundle of cotton thread, and cotton. On spotting, the solution percolating through the tip is absorbed by the thin layer of a TLC plate. The flow of cool air from the hair dryer and the heat applied to the TLC plate by the magnetic agitator promote the evaporation of the solvents, and thus maintain the sample spot on the TLC plate at a certain size.

Procedure

Extraction and clean-up were based on the method described by Chen *et al.*[3]. The TLC determination was similar to that described by Laitem and Gaspar[1].

Ground tea (10 g) was soaked in 50 ml of hexane – acetone (9:1, v/v) and stirred for 30 min using the magnetic agitator. An aliquot of the extracts (5 ml) was washed with concentrated sulphuric acid (1 ml) in a stoppered test-tube. The upper phase was transferred to a spotter packed with alumina (2 cm^3) and with the tip removed. After the liquid had been drawn off the adsorbent, the tip was added to the spotter, and the spotter was placed in the spotting apparatus with the tip right on the spotting position on the TLC plate. The heating unit of the magnetic agitator and the hair dryer were switched on. Hexane (4 ml) was added to the spotter. During spotting, the temperature of the TLC plate was maintained at *ca.* 60°C. When spotting was completed, a

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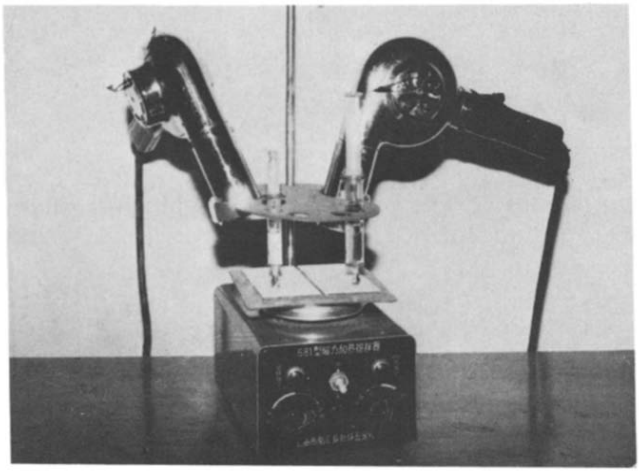

Fig. 1. Photograph of spotting apparatus.

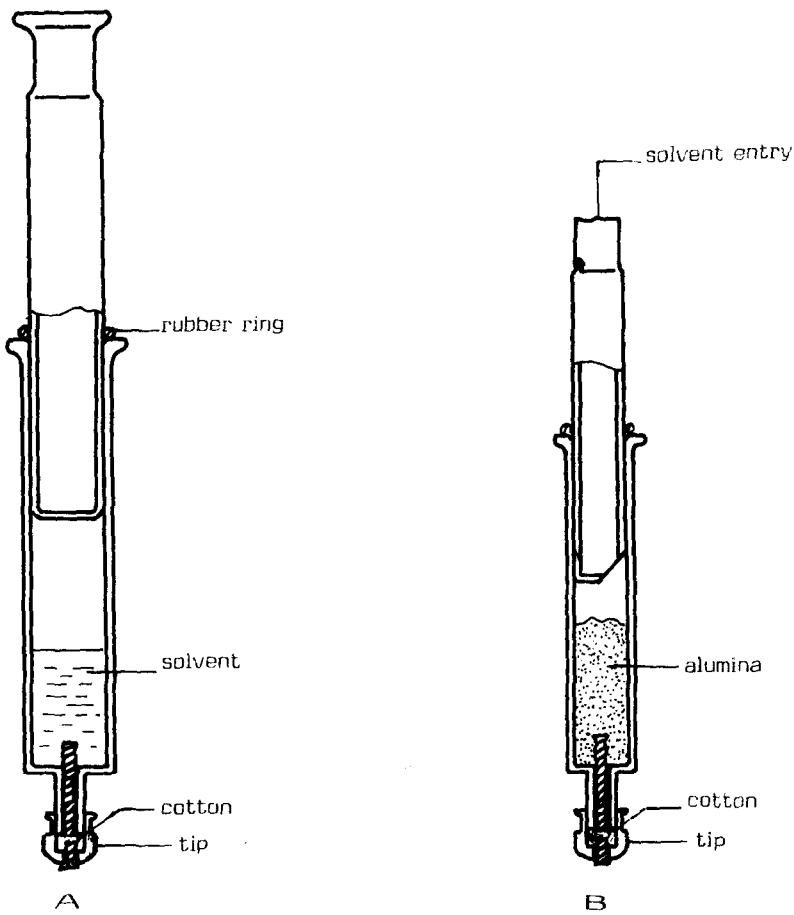

Fig. 2. Diagrams of spotters: (A) for liquid sample; (B) for clean-up.

standard solution (5 μl) was spotted beside the sample spot on the TLC plate. The TLC plate was then developed. The residue level of the pesticide was estimated by comparing the spot size with that of the standard.

RESULTS AND DISCUSSION

The working principle of the device is similar to that described by Moosmann[4], but the present device has little influence on the original spot size and the R_F value, and thus no extra modification of the TLC plates is needed. Also, the device can carry out column chromatography clean-up and spotting at the same time. Tea samples fortified with BHC (0.4 mg kg^{-1}) produced the same spot size as that of an equal amount of the standard. The time for analysing one sample is about 2 h.

To determine the influence of the relatively high temperature and prolonged flow of air on pesticides, several pesticides with different vapour pressures were spotted on a TLC plate using this device (0.4 μg in 5 ml of acetone). The spots on the plate were scraped into a Pasteur pipette and the pesticides were eluted to a test-tube with acetone (5 ml). The recoveries were tested by gas–liquid chromatography. BHC, malathion, dimethoate and quinalphos all gave recoveries higher than 90%, whereas dichlorvos, which is very volatile, gave a recovery of less than 30%.

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