

spreader* is used in all experiments. 10 mm³ solution of the appropriate concentration is applied to provide spots containing from 0.5 to 50 μg of material to the plate. (The plates were previously activated for 1 h at 110° and stored in a desiccator over P₂O₅.)

The plates are then exposed to an atmosphere of ozone (2-4 % ozone in air) for 30 min at the required temperature. A rectangular developing tank (30 × 26 × 6 cm) with a ground glass top is provided with an aluminum cover-plate. This metal plate is provided with an ozone inlet tube extending to the tank bottom and an exit tube leading to a water bubbler. Ozone is generated from a commercial instrument (Welshbach, Model T-23). All lines are made of Tygon. The unreacted ozone is led away from the bubbler into the hood exhaust chimney. Another convenient container is available commercially*.

When a higher reaction temperature is desired the tank is immersed in a thermostated oil bath.

After the ozone exposure the plate is placed under a gentle air stream, then sprayed with a 0.4 % of 2,4-dinitrophenylhydrazine (0.4 % aqueous solution in 2 N HCl) or a 5 % ethanolic solution of phosphomolybdic acid. The first reagent gives yellow or orange spots on a white or light yellow background while the second reagent gives blue-black spots on a light yellow background.

The sensitivities thus determined are somewhat better than those obtained after actual development but will serve as a very good guide in the choice of concentrations required for practical separations.

*Allied Chemical Corporation, Central Research Laboratory,
Morristown, N.J. (U.S.A.)*

R. FUHRMANN
D. JERALOMON

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Received October 22nd, 1965

* Desaga standard developing tank with controlled atmosphere cover. Brinkmann Instruments Catalogues Nos. 25-10-20 and 25-10-29, respectively. Brinkmann Instruments, Inc., 115 Cutter Mill Road, Great Neck, Long Island, N.Y.

J. Chromatog., 22 (1966) 468-471

An apparatus for quantitative application of samples as streaks in thin-layer chromatography

The use of thin-layer chromatography for quantitative analysis of chemical mixtures has been described recently and the merits of various techniques in sample application discussed¹. One problem has been the transfer of enough material to the chromatoplate to allow analysis of the separated components. This has been overcome

J. Chromatog., 22 (1966) 471-473

by applying the material as a series of closely spaced spots from a row of capillaries² or from a mechanically operated micrometer syringe³. The main disadvantage of these methods is the volume of liquid required to fill the various devices, as the amount available for analysis is often small.

The device described below overcomes this problem; it is simple to construct and small volumes can be quantitatively transferred to a silica-gel plate as a narrow uniform line without damaging the silica-gel surface. It is illustrated in Figs. 1 and 2, while Fig. 3 shows the separation of an insecticide mixture applied by this method.

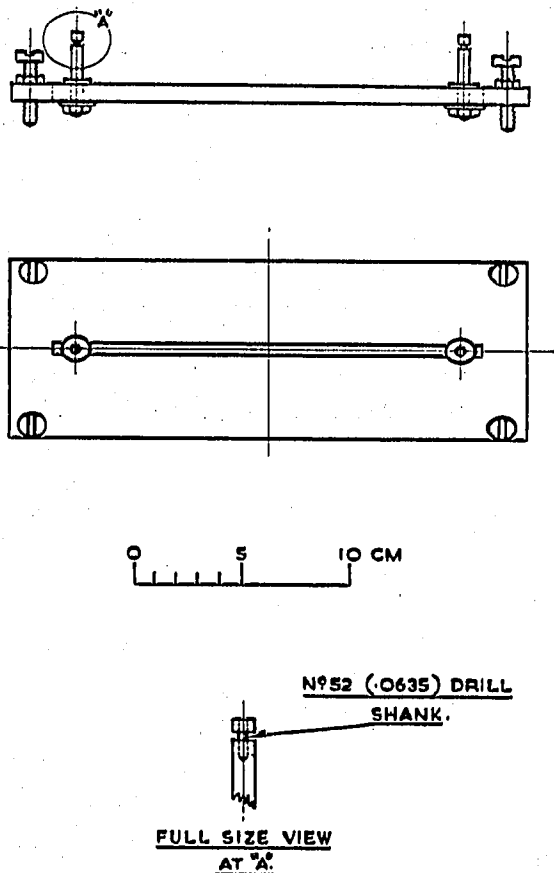


Fig. 1. General arrangement of the applicator.

The parallel horizontal strands of terylene thread are held between two vertical movable rods which can be adjusted to ensure the lines are taut and the length of thread chosen to suit the chromatoplate in use. The sample is applied from a 0.1 ml pipette by laying the pipette tip across the line and moving along the length. The liquid is held by surface tension as an even film between the two threads; a distance of 70 mm and a gap of 1.5 mm will easily support 100 μ l of most common solvents. The surface of the chromatoplate is brought in contact with the film and a narrow even streak is produced. The line is washed by repeating the procedure with pure solvent and quantitative transfer can be achieved. The line will support the film more easily if it has been lightly rubbed between fine emery paper. This produces small fibres which increase the contact points between the liquid and the line.

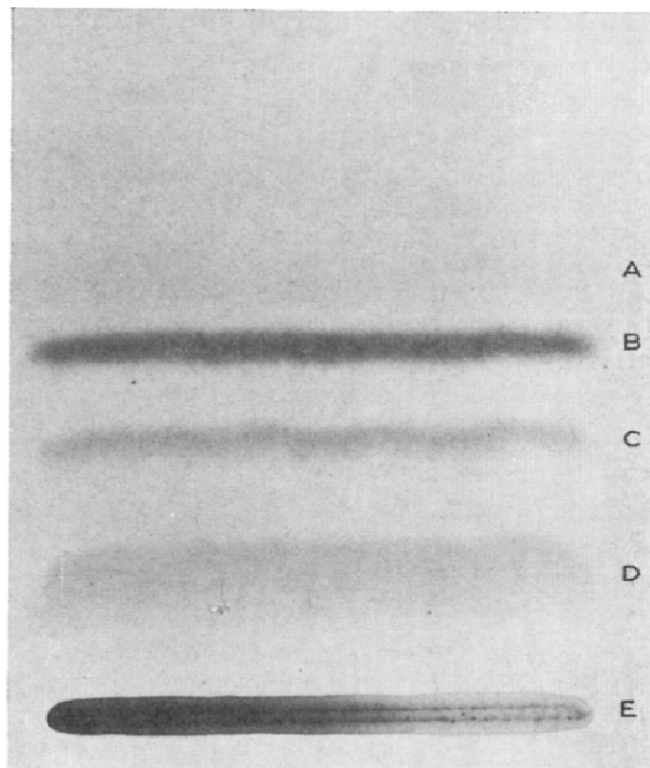
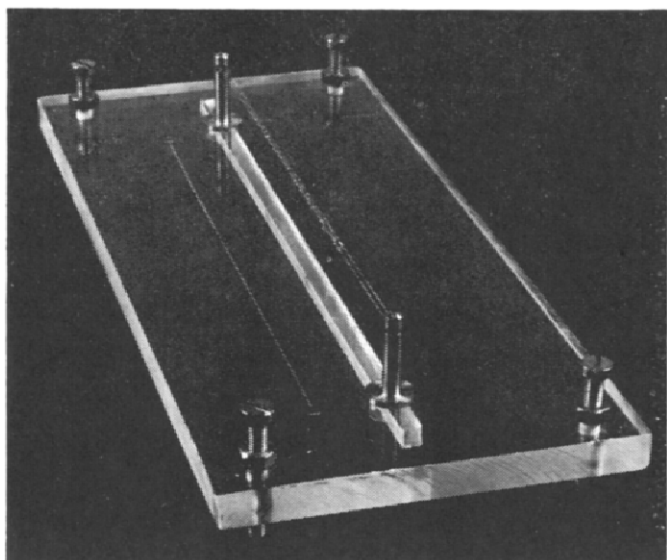


Fig. 2. The applicator with 100 μ l of solution applied to the threads.

Fig. 3. Application and separation of an insecticide mixture from a soil extract on a 300 μ layer silica gel plate. Solvent: redistilled chloroform. A = soil material; B = 3,4-dimethyl-6-chlorophenyl N-acetyl N-methylcarbamate; C = 3,4-dimethyl-6-chlorophenyl N-methylcarbamate; D = soil material; E = origin and soil material.

The method has been used in this laboratory for the application of solutions of insecticide residues to thin-layer plates. As an example, 3,4-dimethyl-6-chlorophenyl-N-methylcarbamate and 3,4-dimethyl-6-chlorophenyl N-acetyl N-methylcarbamate were analysed in soil extracts. Control experiments with known amounts of the two compounds applied at a concentration of 800 μ g in 200 μ l could be recovered almost quantitatively. The recoveries were 99% with a range of $\pm 3\%$ for 3,4-dimethyl-6-chlorophenyl N-methylcarbamate and 99% with a range of $\pm 1\%$ for 3,4-dimethyl-6-chlorophenyl N-acetyl N-methylcarbamate on six and four determinations respectively.

*Tropical Pesticides Research Unit,
Porton Down, nr. Salisbury (Great Britain)*

C. R. TURNER

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Received October 25th, 1965