*Journal of Chromatography*, 190 (1980) 445–447


CHROM. 12,547

## Note

# Coating of glass capillary chromatographic columns: a new variant of the static method

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(Received November 14th, 1979)

Coating of the inside walls of glass capillary columns with a film of stationary phase is one of the most important and difficult problems in capillary chromatography. The coating technology defines the quality of the coating and, consequently, the efficiency and life of the column.

Methods of coating can be divided into two groups: those which utilize a dynamic method, in which a relatively concentrated (up to 10%) solution of the stationary phase in an appropriate solvent is passed through the column at a strictly controlled rate[1,2], and the static technique, in which the column is completely filled with a dilute (*ca.* 1%) solution of the stationary phase, with subsequent evaporation of the solvent[3,4].

The dynamic method is very simple, but yields good results only with non-polar stationary phases. Polar stationary phases, having a high surface tension, wet the glass capillary surface poorly and deposit unstable films that tend to be easily destroyed, with the formation of drops.

The static technique, consisting in vacuum evaporation of the solvent from the completely filled column at room temperature (18–20°)[3], is generally used to obtain high-quality films, but in practice its application involves great difficulties. The solution of stationary phase must be absolutely free of dissolved gas in order to prevent dispersion of the solution inside the column and there must be a complete absence of gas bubbles within the sealed end of the column and the stationary phase solution, because a decrease in pressure at the open end of the column can push the solution out of the column. Also, the static method of coating is time consuming (up to hundreds of hours).

Improved variants of the static method have been suggested by Ilkova and Mistryukov[4] and Jennings *et al.*[5]. They suggested gradually moving a completely filled column with an open end into an air thermostat, which heats the column to the temperature necessary for solvent evaporation. According to Jennings *et al.*, instanta-

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neous evaporation of the solvent and spraying of the column walls with an aerosol of the stationary phase take place at the moment the capillary enters the hot zone of the thermostat. High temperatures (up to 150°) and pressures (up to 20 atm) inside the column ensure good contact of the liquid phase with the hard surface (the column wall) and consequently high-quality coatings.

In this method, a coiled column is placed on a horizontal shaft driven by an electric motor and, as the shaft rotates, the column is screwed into the air thermostat. To achieve instantaneous evaporation of the solvent, a preliminary heater[5] is sometimes placed in front of the air thermostat in order to prevent undesirable condensation of solvent vapour.

The use of high pressures inside the capillary makes it essential for there to be no temperature gradient over the whole volume of the thermostat. In our opinion, a liquid thermostat meets this demand much better than an air thermostat.

It is suggested that gradual vertical insertion of the capillary column into a liquid heated to the required temperature (vacuum oil, glycerine, etc.) should be used for coating a stationary phase on glass capillary column walls. A device for column insertion may have various designs, and such a device allows the gradual, coil-by-coil insertion of the column into the liquid thermostat at any speed. The principle of operation is shown in Fig. 1. If a device of this kind is unavailable, one can use adhesive tape to stick the coils on both sides. When the tape enters the hot liquid it is easily removed, freeing the column.

The advantages of the suggested method are as follows:

(1) a considerable temperature increase at the air–liquid interface makes

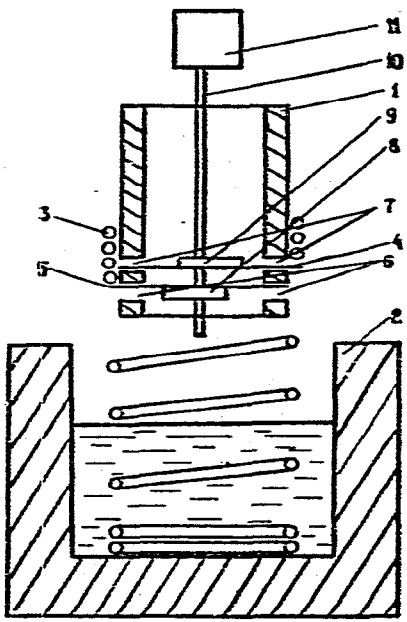


Fig. 1. Device for the insertion of the column into hot liquid. 1 = Thick-walled cylinder; 2 = liquid thermostat; 3 = capillary column; 4, 5 = pins; 6, 7 = holes in cylinder walls; 8, 9 = eccentrics working in antiphase; 10 = shaft; 11 = motor.

preliminary heating unnecessary and ensures instantaneous evaporation of the solvent when the column enters the liquid;

(2) as a result of the large thermal capacity and thermal inertia of the liquid, the column, when it enters the liquid, is in a homogeneous thermal field, which removes the risk of condensation of solvent vapour;

(3) vertical insertion of the column into the thermostat makes it unnecessary to use rubber rollers, which create the risk of column breakage, especially at high speeds, and gives the possibility of processing columns with different coil diameters;

(4) the absence of column rotation removes the risk of entangling of the coils and breakage, and helps to control the coating process by the rate of removal of solvent vapour from the column outlet, which in this instance remains motionless.

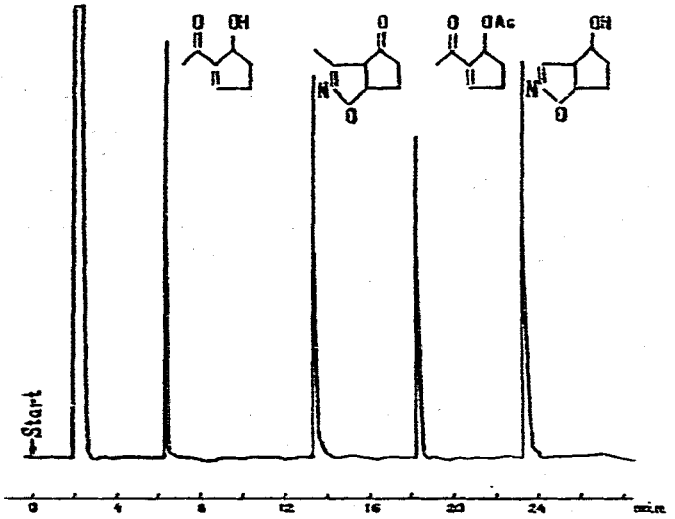


Fig. 2. Example of a gas chromatogram obtained using a glass capillary column coated with stationary phase according to the method described.

Fig. 2 shows a chromatogram obtained after the analysis of a mixture of some N-heterocyclics on a column coated according to the above method. Glass columns were drawn from borosilicate glass by a standard procedure. The column length was 50 m, I.D. 0.5 mm, and the stationary phase was Apiezon K + 10% Carbowax 20M. The column was filled under a pressure of nitrogen with a 1% solution of stationary phase in dichloromethane. Glycerine heated to 150° was used as the working liquid. The coating time was 15 min and the column efficiency was about 2000 plates per metre.

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