A new saturation chamber for thin-layer chromatography

It has been widely demonstrated that the use of saturation chambers is one of the best methods to accomplish a successful thin-layer chromatogram. STAHL[1] described a system, called "Sättigungskammer", where the distance between the plates is diminished by a glass rim fused to the anterior wall of the chamber in such a way that the volume is considerably reduced. For this reason, saturation is obtained shortly after the immersion of the complex in the container of the solvent system.

Because of the laboriousness of preparation, this device has not been very widely employed. The customary chambers of larger volume are more frequently used, even though they have several disadvantages like variability of the saturation state, waste of solvents and size.

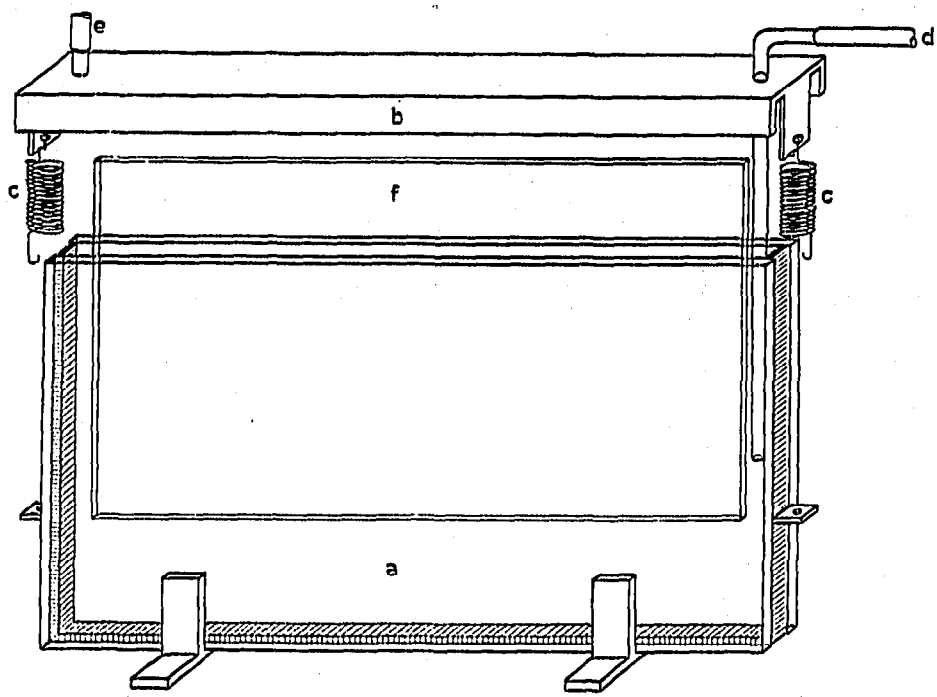

Fig. 1. Saturation chamber—schematic drawing. For an explanation of the letters, see the text.

Subsequently, other authors proposed variations of STAHL's S-Kammer by making use of spacers in different fashions (HONNEGER[2], WASICKY[3], DAVIES[4], JÄNCHEN[5]).

It is obvious that with the chambers described above, it is possible to obtain a better saturation of the atmosphere, chromatographic migration and a better reproducibility of the R_F of the substances analyzed, than with the customary chambers. On the other hand, however, there is a considerable loss of time, due to the adjustment of the walls and the immersion of the complex in the container. In addition, the container has to be adapted, as far as possible, to the section of the chamber to be immersed. Therefore, it is impossible to avoid differential evaporation especially of organic components of the solvent system due to different vapour pressures.

To overcome such drawbacks, we have developed a chamber which is easy to handle and has versatility of use.

The chamber (a) has external dimensions of 200 × 130 × 13 mm and internal

dimensions of 192 × 125 × 5.5. mm. It is constructed from two crystal plates held opposite each other by lead alloy on the sides and on the bottom. Airtight closure is ensured by a metal lid (b) having a rim of a few millimeters and lined internally with foam rubber covered with polyethylene. The lid is secured by two springs (c) anchored to the sides of the chamber. Fastened to the lid are also two tubes, one of them (d) reaches to the bottom of the chamber and serves for the introduction of the solvent system, while the other (e) enters only a few millimeters and prevents pressure inside the chamber.

There are two spacers on the bottom of the chamber lifting up the plate by a few mm. The plates (f) measure 186 × 120 mm. For a 10 cm height of front the ratio chamber volume: evaporating surface is 0.5–0.7 depending on the thickness of the plates used.

Before introducing the plate, prepared according to the standard procedure, into the chamber it is convenient to mark the height of the front desired.

If the experiment requires an atmosphere of a certain gas[6], this is introduced —after fastening the lid with the springs—through the longer tube. The gas is circulated for a few seconds in the chamber and the outflow tube is kept open until the air has been expelled completely.

Afterwards, a small amount of the solvent mixture is introduced with a syringe through the longer tube, making sure that the level does not reach the lower edge of the plate. Only after the saturation of the atmosphere—depending on the temperature of the chamber—is the solvent mixture introduced in the required amount (usually 15 ml) and the tube closed. When the front reaches the desired level, the migration can be stopped immediately by removing the mixture with a syringe through the longer tube.

With the chamber described above, it is possible to obtain chromatograms under saturation conditions or in a particular gaseous atmosphere better than with the customary devices.

Moreover, it is possible to carry out chromatograms at a given temperature without resorting to thermostatic chambers or cold rooms, by simply placing the apparatus in a thermostatic bath.

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