

NOTES

A Modified Technique for Filter Paper Disc Chromatography

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A modified disc desiccator technique is described, which proved to be simpler, more accurate and gave better results than any other paper disc technique. All conditions concerning the modified technique are discussed.

A previous communication¹ described a scheme for inorganic micro-qualitative analysis based on circular paper chromatography. In this scheme a chromatographic assembly was described consisting of octagonal glass plates (18-cm apothem), one has a 4-mm central hole and one ground face. A short wick of polyethylene sponge is used to transfer the solvent mixture from the petri dish to the centre of the filter paper disc. Better results and separations were obtained by modification of this technique into a simpler disc-desiccator technique.

A filter paper disc of the appropriate size (27 cm diameter) is prepared as shown in (Fig. 1a); each filter paper disc will be suitable for the analysis of single

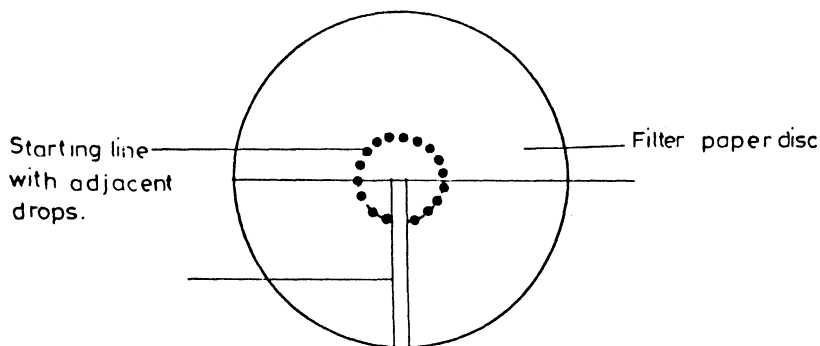


Fig. 1a

mixture by the previously described scheme¹. The paper wick pending exactly from the centre of the disc is used to feed the whole disc with the solvent mixture placed in a tall form beaker at the bottom of the desiccator.

A large desiccator (30 cm diameter) is suitable for good separations. At the bottom of the desiccator is placed some of the solvent mixture to effect equilibration, while the main part of the solvent mixture is placed in the tall form beaker placed on the bottom of the desiccator and immediately below the central hole of the desiccator-porcelain disc. The filter paper disc is placed on this

porcelain disc with the wick pending inside the solvent in the beaker. The whole modified assembly is shown in Fig. 1b.

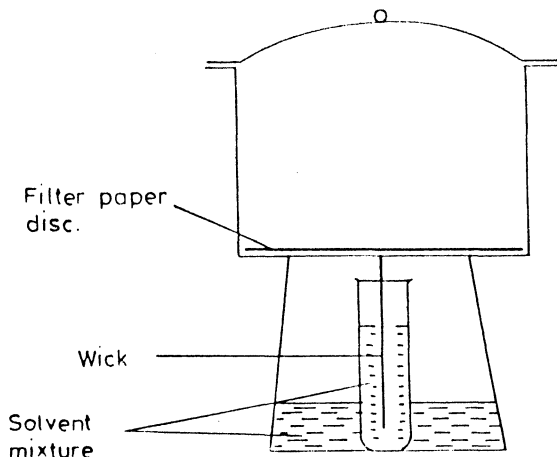


Fig. 1b

Procedure

1. The filter paper disc is prepared as shown in Fig. 1a, with a pencilled circular starting line and a wick about 10 mm wide.

2. The solvent mixture is placed at the bottom of the desiccator and in the tall form beaker as shown in Fig. 1b.

3. The test solution (of cations or anions) is placed in small adjacent drops on the circular starting line (about 20 drops) of the filter paper disc. The test solution is allowed to dry, then the filter paper disc is placed inside the desiccator with the paper wick pending in the solvent mixture placed in the beaker (Fig. 1b).

4. Allow the paper disc to stand until the solvent front reaches all points on the rim of the paper; within the next 15 minutes remove the paper from the desiccator and dry.

5. Using clean scissors cut the paper disc into the suitable number of sector-strips necessary for complete analysis of cations or anions. Store the strips in a glass vial.

The study of the different conditions for the modified technique revealed the following facts:

A. *Relation between width of the wick and time and sharpness of separation:* A series of experiments were undertaken to find the relation between the width of the wick on one side and the time and sharpness of separations on the other side. Table 1 shows the results of these experiments. It should be clear from Table 1 that the very slow motion of the solvent is against sharp separations. In case of wicks 1, 2 and 3 mm wide, after 48 hrs, the solvent front becomes invisible as the filter paper disc becomes entirely humid. As the width of the wick is increased, the movement of the solvent increases and the separation of the constituents of the test substance (*e.g.*, black ink) becomes

more sharp. A 10 mm wide wick is evidently the best as it leads to a reasonable time of running, as well as very good separations. Increase in width of the wick more than 10 mm was found impractical, as it takes a large part of the body of the paper and makes the starting line shorter than necessary, without any great advantage.

TABLE 1
RELATION BETWEEN THE WIDTH OF THE WICK AND TIME
AND SHARPNESS OF SEPARATION

| Wick | Time necessary to reach the rim | Sharpness of separation |
|-------|---------------------------------|--------------------------|
| 1 mm | 72 hours | no visible solvent front |
| 2 mm | 60 hours | no visible solvent front |
| 3 mm | 48 hours | no visible solvent front |
| 5 mm | 42 hours | moderately sharp |
| 6 mm | 39 hours | moderately sharp |
| 7 mm | 33 hours | moderately sharp |
| 8 mm | 29 hours | sharp |
| 9 mm | 24 hours | sharp |
| 10 mm | 20 hours | very sharp |

B. *Relation between type of paper and time and sharpness of separation:* Different types of filter papers were tried, using in all cases a 10 mm wick. It was found out that in all experiments the separations take place with the same sharpness and the same R_R values. When Whatmann No. 54 filter papers were used, the running time was reduced to about one-fifth. When Whatmann No. 31 ET filter papers were used, the running time was reduced much further. Table 2 shows the relation between the type of paper and the time necessary to reach the rim.

TABLE 2
RELATION BETWEEN THE TYPE OF PAPER AND THE
TIME NECESSARY TO REACH THE RIM

| Type of paper | Time necessary to reach the rim |
|--------------------|---------------------------------|
| Whatmann No. 1 | 20 hours |
| Whatmann No. 54 | 3.45 hours |
| Whatmann No. 31 ET | 2.2 hours |

C. *Relation between R_F and R_R values:* Spots of black Parker Quink ink were placed on descending paper sheets, as well as on a disc-desiccator paper, and both run with the same solvent mixture. After running for appropriate time both chromatograms were compared as to the sharpness of separation, as well as to R_F and R_R values. Excellent sharpness of separation was obtained on disc-desiccator chromatograms. In these particular experiments, the solvent front was not allowed to reach the filter paper rim in any case, to make certain of calculating extremely accurate R_F and R_R values. Table 3 shows the mean of the results obtained with six experiments.

TABLE 3
RELATION BETWEEN R_F and R_R

| Dye | R_F value | R_R value |
|--------|-------------|-------------|
| Yellow | 0.370 | 0.370 |
| Blue | 0.367 | 0.367 |
| Pink | 0.372 | 0.372 |

D. *The use of R_R ruler:* In the previous communication¹, the use of R_F ruler was mentioned. In this modified technique the use of R_R ruler was tested and found successful. The filter paper disc is left in the desiccator till the solvent front reaches all points on the rim of the filter paper; then the disc is removed, dried and sprayed. The length of R_R ruler should be equal to the distance from the starting line to the paper rim, and should be equivalent to the value $R_R = 0$ to $R_R = 1$

E. *Separations:* The practicability of this modified technique was proved with great success by application to mixtures of cations, anions, sugars, amino acids and ink pigments, employing several solvent mixtures². Excellent separations depending on sharp distinct lines were obtained in all cases and with all types of solvent mixtures.

It is quite clear from the experimental part that this modified technique is a real progress. Further potentialities of the technique are discussed in the following:

1. Filter paper disc could be pencilled each, for the analysis of four simple mixtures consisting each of two, three, or even four components. This is shown in Fig. 1c.

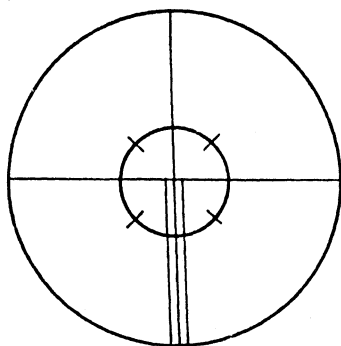


Fig. 1c

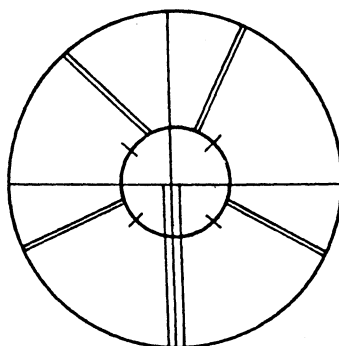


Fig. 1d

2. Filter paper discs could also be provided with slots to separate the sectors, as shown in Fig. 1d. In this case the number of sectors could be increased to five instead of four. In this method the mixtures under test never mix. By this method simple mixtures consisting of up to four components may be allowed.

3. It was found quite favourable and advantageous to modify a desiccator and make the upper part as low as possible, as shown in Fig. 1b. This modified

desiccator helps quick equilibration which is essential for rapid movement and good separations. It also has the advantage that the solvent front is better seen as it becomes nearer to vision.

REFERENCES

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