

CHROM. 5517

Letter to the Editor

Improved separation in thin-layer chromatography using a single-component solvent in an unsaturated chamber. II.

Sir,

We read with interest GEISS's comments¹ on our paper entitled *Improved separation in thin-layer chromatography using a single-component solvent in an unsaturated chamber*².

Our only aim was to report an unexpected observation which might be of practical value. We did not wish to go into details and we assumed that your readers have a fair knowledge of basic principles of thin-layer chromatography (TLC).

In judging the resolution of two compounds, the band widths or spot diameters should be taken into account. In our paper, we therefore mentioned that in unsaturated chambers, no increase in spot diameters was observed. Exact diameters were not available owing to the initial set-up of our experiments³, in which spot diameters were not measured. It should be noted that the participants in these inter-laboratory experiments^{2,3} were free to choose either a saturated or an unsaturated chamber. Comparing separate ΔR_F values from saturated chambers with those from unsaturated ones (as was done in Table I, ref. 1) is therefore misleading: one can only compare the mean of the ΔR_F values from saturated chambers with the mean of the ΔR_F values from unsaturated chambers.

Admittedly, ΔR_F values increase with integral solvent flow and, therefore, the ratio $R_{F \text{ max.}}/R_{F \text{ min.}}$ would have been more appropriate than our initial criterion $R_{F \text{ max.}} - R_{F \text{ min.}}$. Improved separation would then show up as an increase in $R_{F \text{ max.}}/R_{F \text{ min.}}$. The mean values in Table I were derived from our paper².

TABLE I

 $R_{F \text{ max.}}/R_{F \text{ min.}}$ VALUES IN SATURATED AND UNSATURATED CHAMBERS

Ratio	Saturated chamber	Unsaturated chamber
$R_F(C_{12})/R_F(Y)$	1.29	1.32
$R_F(Y)/R_F(C_1)$	1.32	1.43
$R_F(C_1)/R_F(R)$	1.83	2.02
$R_F(R)/R_F(B)$	2.21	2.76
$R_{F \text{ max.}}(C_{12})/R_{F \text{ min.}}(B)$	7.19	9.71

From these values, it is obvious that both the resolution of adjacent pairs, as well as the overall resolution, are significantly improved in unsaturated chambers.

GEISS's remarks on the tenability of the theory of TLC with multi-component solvents seem to be hardly relevant to our article. However, improved separations by means of the vapour programming technique have proved their usefulness in practice^{4,5}.

In our opinion, the answer to the question whether or not a "pushing-up" effect occurs in unsaturated chambers is a clear-cut one. Even if complete saturation of the initially unsaturated chambers is attained within 15 min, the chambers will not be saturated before this time, and evaporation of solvent from the wetted layer will take place. Pushing-up will therefore occur and it cannot be ignored in the overall separation.

Finally, unsaturated chambers would always give lower a values in our system, irrespective of the choice of the standard chromatogram, as some simple algebra will show:

Reference standard = Expt. XXX, $R_F^0(Y) = 0.77$, $R_F^0(B) = 0.12$
(unsaturated)

Chromatogram to be corrected = Expt. XVII, $R_F(Y) = 0.47$, $R_F(B) = 0.09$
(saturated)

$$a = \frac{R_F^0(Y) - R_F^0(B)}{R_F(Y) - R_F(B)} = \frac{0.77 - 0.12}{0.47 - 0.09} = 1.71$$

If a chromatogram from an unsaturated chamber had been chosen as the reference standard, the only difference would have been that for unsaturated chambers, a values of about 1 would have been found, whereas saturated chambers would have shown a values in the range 1.2–2.5 (an exception is the case of Expt. XV, the results for which are an extreme).

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