A thin layer chromatographic method for estimation of geraniol in oils of palmarosa

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Routine methods for estimation of geraniol in the essential oils as described by Guenther prescribe a larger quantity of oil (10 ml) for estimation.[1] These methods may not be suitable for carrying out estimations in very small quantities, as in the case of oils obtained from plant breeding or cytogenetic and other types of experiments in developmental work where the oil is derived from a single or few plants only. Gas chromatographic examination requires considerably less oil (1-2 microliters) and gives accurate results. However, in view of the non-availability of the sophisticated instruments such as the gas chromatograph in most of the research laboratories, it was considered worthwhile to develop and present a method for estimation of geraniol in palmarosa oil by thin layer chromatography, so that the oil distilled in very small quantities might be analyzed immediately and not get polymerized waiting to be analyzed by gas chromatography.

The method developed involves thin layer chromatography of a sample of palmarosa oil and the same quantity of pure geraniol (isolated by the calcium chloride adduct method), spraying with vanillin-sulfuric acid, scratching out the developed spots of geraniol from both the oil and the reference sample of geraniol, and weighing in analytical balance.[3,4]

Experimental

The analytical sample was prepared by isolating geraniol by the calcium chloride adduct method.[2] The sample was checked by thin layer chromatography to give a single spot of geraniol only.

Thin layer chromatography of the pure geraniol specimen was carried out with a benzene and ethyl acetate mixture (85:15) in a Toshniwal TLC kit, by spotting samples of geraniol (1 μl, 2μl, 3μl, 4μl, 5μl, and 6μl) from a micro-liter pipette. After the plate was developed, it was sprayed with a vanillin-sulfuric acid (1:20) mixture, and the visible spots of geraniol were observed to be of the sizes proportional to the quantity of material spotted. After one hour of spraying the spots were scratched out separately and weighed. The weight of silica gel corresponding to the spots of geraniol was proportional to the weight of samples spotted, since the weights were proportional to the spot area.

The results are given in Table I.

Table I

Vol. of geraniol spotted (µl)	Wt. of silica gel from scratched spot (mg)
1	0.032
2	0.064
3	0.095
4	0.125
5	0.160
6	1.195

Samples of oils marked 1, 2, 3, 4, and 5 along with a pure specimen of oil (2.0 μl each) were spotted over a thin layer plate, developed with a mixture of benzene and ethyl acetate (85:15), and sprayed with a mixture of vanillin in sulfuric acid (1:20). The spots of geraniol in analytical samples of palmarosa oils and in pure geraniol were scratched out and weighed. The percentage of geraniol was determined by the following formula:

$$\% \text{ geraniol} = \frac{W' \times 100}{W}$$

where W' = weight of geraniol spot in analytical samples of palmarosa oil; and
W = weight of geraniol spot in pure specimen.

The results thus obtained were compared with those obtained by gas chromatographic analysis in a Perkin-Elmer gas chromatograph with carbowax column. The results are presented in Table II.

Table II

Sample No.	Spot weight (g)	% Geraniol by T.L.C.	% Geraniol by G.L.C.
1	0.026	81.3	80.3
2	0.022	68.7	67.9
3	0.022	68.7	67.0
4	0.025	78.1	78.4
5	0.022	68.7	69.4
6	0.032	---	100.0

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Results and Discussions

The experimental data presented above show that geraniol can be estimated quickly by thin layer chromatography and the results conform with those obtained by gas chromatography. The variation of results from the two methods is within ±1% experimental error range. However, as the thickness of the thin layer plate always varies with the adjustment over the applicator and the consistency of the silica gel slurry, it is always advisable to run a pure sample of geraniol of the same size as that of the oil. The geraniol sample develops certain impurities on long keeping; therefore before spotting a geraniol sample as the reference standard, its purity should be determined on a thin layer plate.

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