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Note

Detection of adulteration in ginger galenicals by thin-layer chromatography

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Ginger (*Zingiber officinale* Roscoe) is a drug subject to extensive adulteration because of its importance in the drug, food and soft drink industries. Among the adulterants are capsicum and grains of paradise[1], both of which are added to give increased pungency, and curcuma[2], which is added to restore colour. In powdered ginger, most of the vegetable adulterants can be detected by simple microscopical examination, but in tinctures of ginger a chemical procedure followed by an organoleptic test is adopted[1]. This method takes advantage of the fact that the pungent principles capsaicin and paradol in capsicum and grains of paradise, respectively, are little affected in their pungency when heated with alkali, whereas gingerol, the pungent principle in ginger, is more readily decomposed with a resultant loss of pungency, as determined by tasting. As this method relies on the sensitivity of the taste-buds, the results are likely to vary among individuals, and a procedure that does not involve tasting is therefore desirable. In this investigation, a thin-layer chromatographic (TLC) procedure has been examined.

EXPERIMENTAL

Drugs

Ginger root (Halewood Chemicals Ltd., Staines, Great Britain), African ginger nat (Halewood Chemicals), grains of paradise, curcuma and *Capsicum annum* were used.

TLC plate

A Silica Gel F_{254}, (E. Merck, Darmstadt, G.F.R.) pre-coated plate, 10×20 cm layer thickness 0.25 mm was used.

Solvent systems

The two solvent systems used were (i) benzene–methanol (95:5)[3] and (ii) benzene.

Methods

A set of tinctures was prepared by macerating 1 g of each drug in a crushed or powdered form with 5 ml of 90% ethanol[4] for about 12 h. Another set was prepared by mixing 1 g of ginger root separately with equal amounts of capsicum and grains of paradise and macerating the mixture with 10 ml of 90% ethanol. A combined tincture of ginger and curcuma was prepared by mixing 1 g of ginger root with only

0.02 g of curcuma because of the strong colour of the latter, and the mixture was macerated with 10 ml of 90% ethanol. Finally, a combined tincture of all of the drugs was prepared by macerating a mixture of 1 g each of ginger root, capsicum, grains of paradise and 0.02 g of curcuma with 20 ml of 90% ethanol.

Sufficient of each preparation was applied to a plate until a fairly loaded spot was obtained. Chromatography was carried out at room temperature (33–34°) with the solvent system benzene–methanol (95:5), the solvent front being allowed to travel a distance of 10.2 cm during a period of about 48 min. After development the plate was dried by shaking it in the air and the coloured components as seen in ordinary light were marked. In the case of capsicum, although there were several spots, only those that were likely to be of diagnostic value to the work in hand were marked. The chromatogram is shown in Fig. 1.

A second chromatogram was prepared in order to study the pungent principles gingerol and paradol in ginger and grains of paradise, respectively. For this plate, mixed extracts of grains of paradise and ginger root, and of grains of paradise and African ginger, were prepared by using the same proportions of drugs and solvent as described for the equivalent preparations for the first chromatogram. African ginger was used at this stage because of its importance as a commercial variety of the drug, and also because it is recognised as being more pungent than ginger root (from Jamaica), which is the chief commercial source of the drug. The pure and mixed extracts were chromatographed using the same adsorbent as for the first method, but with benzene as the solvent. The solvent front was allowed to advance a distance of 10.5 cm during a period of about 60 min. After drying the plate, faint vertical lines were drawn by means of a pencil from the point of application of the spots to the solvent front. Starting from the solvent front and proceeding downwards, spots of a 3% solution of vanillin in concentrated sulphuric acid were applied so close together along each line as to form a vertical streak of the reagent. Then the plate was immediately sprayed with distilled water to develop the blue colour given by the pungent principles of the drugs. This technique is based on the fact that both gingerol and paradol give a blue colour on mixing with vanillin and sulphuric acid and diluting with a small volume of water[1]. The chromatogram obtained is shown in Fig. 2.

RESULTS AND DISCUSSION

From Fig. 1 and Table I, it can be seen that the components from capsicum and curcuma are so well separated from the components from ginger root that capsicum and curcuma can readily be detected in ginger extract. The presence of capsicum in a mixture can be discerned within a few seconds after the solvent front has passed the base line, because the red component a_3 separates almost immediately and travels along with the solvent front; the presence of curcuma soon becomes evident from the yellow component d_1, which moves slowly just above the baseline. The chromatograms given by ginger root and grains of paradise are identical, yielding the components c_1 and b_1, respectively, with the same R_F value. Examination of the plate under UV light, although revealing a few more minor components, did not show any differences between the two extracts. Thus, as can be seen from the mixed extract f, grains of paradise cannot be detected in the presence of ginger root under the conditions used for preparing the chromatogram shown in Fig. 1.

TABLE I

PROPERTIES OF COMPONENTS IN FIG. 1

Component	*R_F value*	*Colour*	*Source of component*
a_1	0.12–0.16	Red	Capsicum (a)
a_2	0.70–0.79	Red	Capsicum (a)
a_3	0.97–0.99	Red	Capsicum (a)
b_1	0.85–0.89	Yellowish	Grains of paradise (b)
c_1	0.85–0.89	Yellowish	Ginger root (c)
d_1	0.05–0.08	Yellow	Curcuma (d)
d_2	0.16–0.22	Yellow	Curcuma (d)
d_3	0.37–0.47	Yellow	Curcuma (d)

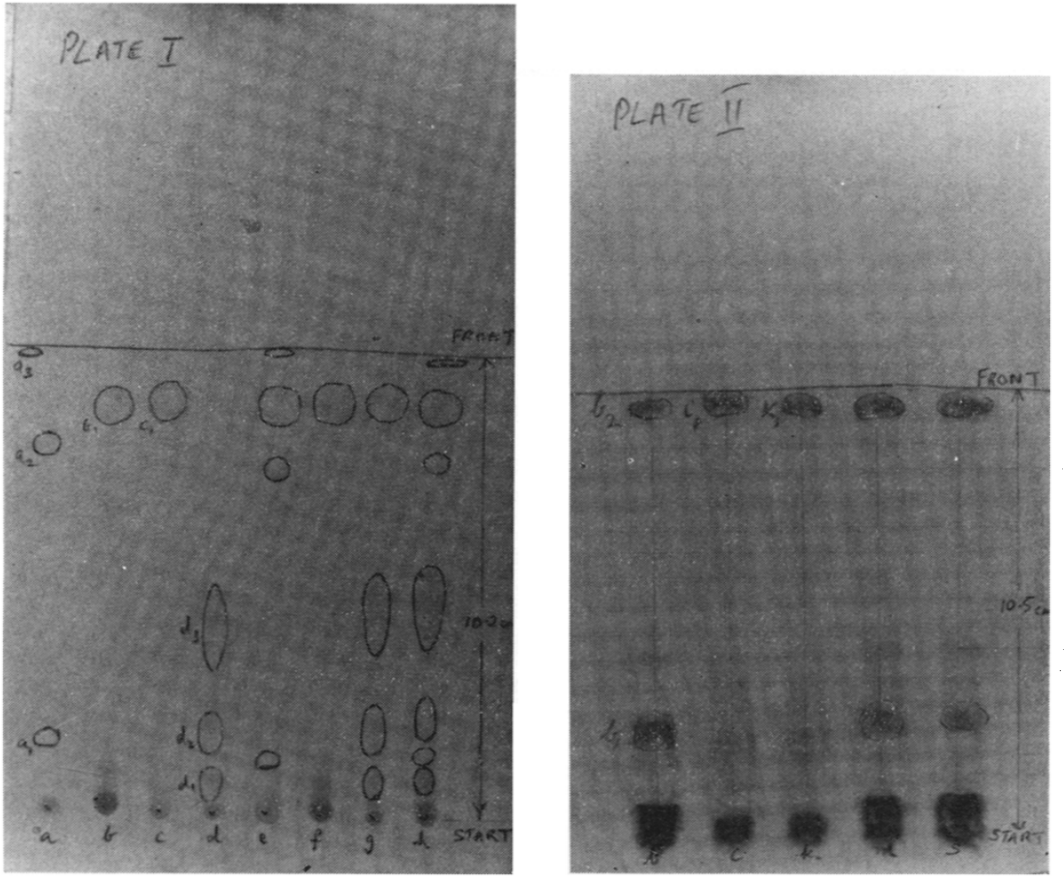

Fig. 1. First chromatogram (see text for method of preparation). (a) Capsicum; (b) grains of paradise; (c) ginger root; (d) curcuma; (e) ginger root+capsicum; (f) ginger root+grains of paradise; (g) ginger root+curcuma; (h) ginger root+capsicum+grains of paradise+curcuma. The components are described in detail in Table I.

Fig. 2. Second chromatogram (see text for method of preparation). (b) Grains of paradise; (c) ginger root; (k) African ginger; (m) grains of paradise+ginger root; (s) grains of paradise+African ginger. The components are described in detail in Table II.

TABLE II

PROPERTIES OF COMPONENTS IN FIG. 2

Component	*R_F value*	*Colour after treatment with vanillin–H_2SO_4*	*Source of component*
b_1	0.24–0.27	Blue	Grains of paradise (b)
b_2	0.96–0.97	Purple	Grains of paradise (b)
c_1	0.96–0.97	Purple	Ginger root (c)
k_1	0.96–0.97	Purple	African ginger (k)

The purpose of preparing the chromatogram shown in Fig. 2 (see also Table II) was to find out whether a study of the pungent principles would be of value in distinguishing grains of paradise from ginger. It can be seen from Fig. 2 that it is possible to detect grains of paradise because the component b_1 from grains of paradise has no equivalent from either ginger root or African ginger. Again, as can be seen from Fig. 2, each extract also yielded a component which gave a blue colour with the vanillin reagent, but which hardly moved off the baseline. The purple components common to the extracts, b_2, c_1, k_1, may not represent pungent principles. Two further observations on Fig. 2 need be emphasized. Firstly, although grains of paradise are known to contain the pungent principle paradol, the chromatogram shows that there could be more than one such component. Secondly, although African ginger is known to be more pungent than ginger root, the two drugs show no difference chromatographically as regards their pungent principles.

In conclusion, TLC affords a reliable and fast means of detecting adulteration in ginger galenicals, even when more than one adulterant is present. The method may also be of value in detecting exhausted ginger; for example, a certain sample of ginger powder sold as a food flavour was found to yield none of the components yielded by ginger on Figs. 1 and 2, thus indicating a possible previous exhaustion of the material followed by the addition of some flavour.

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