A NOTE ON THE ANALYSIS OF OIL OF PEPPERMINT BY AN ALUMINIUM OXIDE-SILICIC ACID DOUBLE COLUMN

By M. S. Karawya and S. K. Wahba

From the Pharmacognosy Department, Faculty of Pharmacy, Cairo University, Egypt, U.A.R.

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The separation and determination of the constituents of oil of peppermint have been effected using two columns in succession, one consisting of aluminium oxide and the other of silicic acid.

The separation of the constituents of oil of peppermint has been improved by using the behaviour of the constituents on aluminium oxide and silicic acid columns.

Experimental

Five g. of aluminium oxide is packed in column 1 (C_1) of the apparatus (Fig. 1), (0·7 cm. in internal diameter and 30 cm. in length) and 10 g. silicic acid in column 2 (C_2) (1·0 cm. in internal diameter and 30 cm. in length). Funnel (F_1) is filled with light petroleum (b.p. 30–50°) and Funnel (F_2) with a mixture of 5 per cent ether in light petroleum.

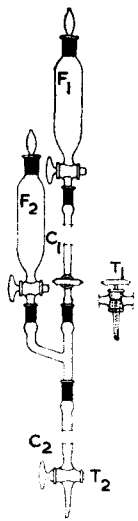

Fig. 1. Two Column System for the Evaluation of Oil of Peppermint.

About 100 mg. of oil of peppermint is accurately weighed and transferred quantitatively to the top of column 1 and light petroleum is allowed to percolate through. The three-way tap (T_1) is regulated so that the surface of the silicic acid in column 2 is always covered with about 1 cm. of the light petroleum effluent from column 1.

140 ml. of light petroleum is collected from the second column. At this stage the constituents of oil of peppermint are located as follows:

1. The hydrocarbons are present in the light petroleum effluent and are evaluated gravimetrically after allowing the solvent to evaporate at low temperature.
2. Menthone and menthyl acetate are on column 2.
3. Menthol is left on column 1.

The 5 per cent ether in light petroleum mixture is then allowed to pass through the silicic acid column immediately following on the light petroleum layer. Twenty ml. of this ether light petroleum mixture is collected, evaporated at 40° and assayed by the hydroxylamine hydrochloride method of the Egyptian Pharmacopoeia (1953) using 0·1N ethanolic potassium hydroxide to determine the eluted menthone.

Elution is continued with the same solvent mixture; 70 ml. of pure solvent is collected before the appearance of menthyl acetate. The following 20 ml. will elute all the menthyl acetate which is then determined by evaporating the solvents at 40° and using the Egyptian Pharmacopoeial method with 0·02N ethanolic potassium hydroxide and refluxing on a boiling water bath for 2 hr.

Menthol which is held by aluminium oxide column is then washed out by 15 ml. of chloroform and collected through the three-way tap (T_1).

The chloroformic effluent is then made up to 100 ml. by chloroform, and menthol is determined colorimetrically by the method of Masamune (1933) using an Ogal colorimeter. The results are shown in Table I.

TABLE I

ANALYSIS OF OIL OF PEPPERMINT ON ALUMINIUM OXIDE AND SILICIC ACID COLUMNS

Wt. of oil in mg.	Eluted constituents per cent			
	Hydrocarbons	Menthyl acetate	Menthone	Menthol
109	11·30	15·90	21·72	50·85
120	11·45	16·00	21·42	50·60
115	11·75	15·85	21·70	50·88
106	11·00	15·92	21·56	50·51
Mean	11·33	15·91	21·60	50·71

Small quantities of the oil (about 100 mg.) can be analysed by this technique, while about 15 g. of the oil is required for the pharmacopoeial methods.

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