BRIEF COMMUNICATIONS

DETERMINATION OF THE SOLUBILITY OF GOSSYPOL

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With the introduction of the method of direct extraction, it is proposed to isolate gossypol from cotton seeds into an oil. For this purpose, before extraction the material is moistened with a miscella the choice of the concentration of which depends on the degree of solubility of the gossypol in it. However, there are very few quantitative indices on the solubility of gossypol in the literature, and they are contradictory [1, 2].

We have studied the influence of the nature of the solvent and the concentration of the miscella on the degree of their saturation with gossypol. As the solvents we used petroleum ether (bp 40-60°C), hexane, and the extraction gasoline that is used in the enterprises of the oils and fats industry. Samples of model miscellas were prepared from salad-grade cottonseed oil containing no gossypol. The concentration of the miscella ranged from 0 to 50% (Table 1). Higher concentrations are not used under industrial conditions and they were not studied. To the miscella was added an accurately weighed sample of pure gossypol (mp 182°C) in an amount sufficient for the formation of a saturated solution, and it was then heated to a temperature close to the boiling point of the corresponding solvent with vigorous stirring for 10 min. The insoluble part of the gossypol was filtered off, washed with petroleum ether to defat it, dried, and weighed. The amount of gossypol that had dissolved was determined from the difference between the weight of the sample added and that which remained on the filter.

Phosphatides and free fatty acids were not added, since they have no appreciable effect on the degree of solubility of gossypol in a miscella [2].

The results obtained indicate that the solubility of gossypol in miscellas rises with an increase in the amount of oil in them, and this to a greater degree the higher the boiling point of the solvent used.

At concentrations of the miscellas of from 0 to 25% the solubility of the gossypol changed in proportion to the amount of oil in it, and a further rise in the concentration led to a sharp increase in its solubility, which reached 2.83% in the pure oil.

The results of the determinations of the degree of extraction of gossypol from defatted gossypol-rich cottonseed pulp [3] with hexane miscellas that we have performed have shown that here, as in the case of model miscellas, a direct proportionality is observed between the amount of gossypol extracted and the concentration of the miscellas in the range from 0 to 25%.

TABLE 1

Oil content of the miscella, %	Solubility of the gossypol, %		
	in petroleum ether	in hexane	in gasoline
50	1,14	1,20	1,51
35	0,68	0,74	1,10
30	0,45	0,50	0,71
25	0,32	0,35	0,51
15	0,22	0,25	0,47
13	0,21	0,22	0,43
10	0,17	0,20	0,42
5	0,14	0,15	0,39
0	0,11	0,12	0,36

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COMPONENTS OF *Artemisia leucodes*

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We have continued the study of the chemical composition of the epigeal part of *Artemisia leucodes* Schrenk collected in the budding stage in the Chemkent province, Kazakh SSR [1].

The chromatographic separation of the total lactone material on neutral alumina followed by rechromatography of the individual fractions in a silica gel column yielded two substances.

Substance I with mp 176-178°C was readily soluble in all organic solvents. A comparison of IR spectra and a mixed melting point showed that compound (I) was *l*-camphor, which has been isolated from the essential oil of *A. leucodes* previously [2].

Substance (II) had mp 190-191°C (from ethanol) and the composition $C_{17}H_{20}O_5$, with R_f 0.43 on Silufol in the chloroform—ethyl acetate (9:1) system. The IR spectrum of (II) contained absorption bands at (cm^{-1}) 1790 (carbonyl of a γ-lactone ring), 1745, 1240 (carbonyl of an ester group), 1685 (α,β-unsaturated cyclopentanone), and 1640 and 1625 (conjugated double bond). The facts given for (II) correspond to the sesquiterpene lactone matricarin [3]. A direct comparison of (II) with matricarin which we obtained by the acetylation of austricin, showed their identity.

This is the first time that matricarin has been isolated from *Artemisia leucodes*.

From *A. leucodes* in the flowering stage, together with sesquiterpene lactones [4], we isolated yellow needles with mp 227-228°C (benzene—chloroform), having the composition $C_{18}H_{16}O_7$ which were assigned, on the basis of their UV spectrum (λ_{max} 252, 277, 345 nm) and qualitative reactions to the flavone derivatives. It was established from the PMR spectrum that the flavonoid contained three OCH_3 groups (3.66, 3.70, and 3.82 ppm, 3 H each). The presence in the mass spectrum of the peaks of ions with m/z 165 and 162 showed that two of the OCH_3 groups are present in ring B [5].

Analysis of the PMR, mass, and UV spectra, and also a comparison of the physicochemical properties with literature figures enabled the flavonoid isolated to be identified as eupatilin [5, 6]. Eupatalin has been isolated previously from *Artemisia frigida* [7].

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