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We have previously reported the isolation of sesquiterpene lactones from an ethanolic extract of the epigeal part of *Saussurea elegans* Ledeb. [1]. When the benzene fractions obtained in the separation of the ethereal sum of the ethanolic extract were rechromatographed on type KSK silica gel [with the eluent hexane-ethyl acetate (12:1)], two crystalline substances were isolated: (I) - $C_{32}H_{52}O_2$, mp 239-241°C (ethanol); and (II) - $C_{29}H_{50}O$, mp 137-139°C (acetone).

The mass spectrum of (I) had characteristic peaks (M^+ 468, m/z 408 (M^+ - CH_3COOH), 249, 218, 203) indicating that the compound belonged to the pentacyclic triterpenes of the β -amyryn series. The nature of the retrodiene decomposition indicates the presence of a double bond in the 12,13 position. Peaks with m/z 218 and 203 are characteristic for the ion α [2], including rings D and E of a pentacyclic triterpene. A fragment with m/z 249 relates to rings A and B, including an acetyl group. On the basis of the facts given above, the compound isolated was identified as β -amyryn acetate [3].

Substance (II) was identified as β -sitosterol (M^+ 414) from its physical constants and mass spectrum [4].

In the sample that we have isolated we also detected small amounts of stigmasterol (M^+ 412) and of campesterol (M^+ 400).

When the ethereal sum of the ethanolic extract was chromatographed on a column of type KSK silica gel, in addition to sesquiterpene lactones, from the fractions eluted by the benzene-ethyl acetate (1:1 and 1:2) systems we isolated two substances of flavonoid nature.

One of them, with the composition $C_{16}H_{12}O_6$, mp 288-290°C (ethanol), M^+ 300, according to its UV spectrum ($\lambda_{max}^{ethanol}$ 217, 277, 339 nm; $\log \epsilon$ 4.58, 4.30, 4.44) was a flavone derivative. The PMR spectrum of (I) showed the resonance signals of the protons of one methoxy group. Acetylation of the compound isolated with acetic anhydride in the presence of pyridine led to a triacetate with mp 165-166°C.

The UV spectra taken in the presence of $AlCl_3$, CH_3COONa , and CH_3ONa show the presence of phenolic hydroxy groups at C-5, C-7, and C-4'. By a comparison of PMR, mass, and IR spectra, this flavonoid was identified as hispidulin [5, 6].

The second substance, with the composition $C_{17}H_{14}O_6$, mp 217-219°C (ethanol), M^+ 314, was also shown by its UV spectrum ($\lambda_{max}^{ethanol}$ 217, 278, 334 nm) to be flavone derivative. Acetylation gave a diacetyl derivative with mp 153-154°C, and according to its PMR spectra it contained two methoxy groups.

The results of a study of the UV spectra taken in the presence of ionizing and complex-forming additives showed the presence of free phenolic hydroxy groups at C-5 and C-7 of the flavone nucleus.

An analysis of PMR, mass, and IR spectra and a comparison with literature information enabled this compound to be identified as pectolarigenin [7].

This is the first time that these compounds have been isolated from plants of the genus *Saussurea*.

LITERATURE CITED

1. I. D. Sham'yanov, A. Mallabaev, and G. P. Sidiyakin, *Khim. Prir. Soedin.*, 819 (1976); 442 (1978); 865 (1979); 258 (1980).

Institute of the Chemistry of Plant Substances, Academy of Sciences of the Uzbek SSR, Tashkent. Translated from *Khimiya Prirodnikh Soedinenii*, No. 6, pp. 796-797, November-December, 1983. Original article submitted May 31, 1983.

2. B. H. Budzihiewiz, J. M. Wilson, and C. Djerassi, *J. Am. Chem. Soc.*, **85**, 2688 (1963).
3. K.-H. Lee, T. Ibuka, H. Furukova, M. Huzuka, R.-Y. Wu, H. Halliris, and H.-C. Huang, *J. Pharm. Sci.*, **69**, No. 9, 1050 (1980).
4. R. Razakov, A. K. Kasimov, Kh. A. Aslanov, and A. S. Sadykov, *Khim. Prir. Soedin.*, **81** (1981).
5. L. Farkas, M. Nogradi, V. Sudarsanem, and W. Herz, *J. Org. Chem.*, 3228 (1966).
6. Y.-L. Liu and T. T. Mabry, *Phytochemistry*, **20**, No. 6, 1368 (1981).
7. L. P. Smirnova, G. G. Zapesochnaya, V. I. Sheichenko, and A. I. Ban'kovskii, *Khim. Prir. Soedin.*, 313 (1974).

COMPONENTS OF *Tanacetopsis mucronata*

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We have continued the separation of the combined nonpolar sesquiterpene lactones from the epigeal part of *Tanacetopsis mucronata* (Regel et Schmalh.) S. Kovalevsk. [1, 2].

On the chromatographic separation of the nonpolar fraction of the chloroform extract on a column of silica gel (1:30) with elution by hexane-ethyl acetate (93:7), from fractions 30-40 we isolated acicular crystals with mp 108°C (from hexane-ethyl acetate), with the composition $C_8H_8O_2$ (I), M^+ 136, R_f 0.75 [Silufol UV-254 plates, ethyl acetate-hexane (3:2) system].

The IR spectrum of (I) (ν_{max} , KBr) had absorption bands of a conjugated carbonyl (1672 cm^{-1}), of a hydroxy group (3400 cm^{-1}), and of an aromatic ring (1610, 1590 cm^{-1}).

The PMR spectrum of (I) (δ scale, JNM-4-H-100 MHz, 0 - HMDS, deutero-pyridine) showed the signals of the protons of a methyl group (singlet, 2.36 ppm) and of aromatic protons (doublets at 6.69 and 7.87 ppm).

On the basis of the results obtained and also of its mass-spectrometric fragmentation, substance (I) was identified as p-hydroxyacetophenone [3].

The mother solutions from mucrin [2] were chromatographed on a column of silica gel (1:30). On elution with the hexane-methyl ethyl ketone (9:1) system, fractions 35-37 deposited crystals with mp 204°C (from a mixture of hexane and ethyl acetate); composition $C_{10}H_8O_4$ (II), M^+ 192, R_f 49. IR spectrum (cm^{-1}): 3350 (OH), 1715 (C=O), 1640 (C=C), 1620 and 1575 (aromatic ring).

Substance (II) proved to be identical with the coumarin scopoletin [4].

Fractions 39-54 yielded mucronin, and fractions 58-67 a substance (III) with mp 167°C (from a mixture of hexane and ethyl acetate) with the composition $C_{15}H_{18}O_4$, M^+ 262, R_f 0.40.

The IR spectra of (III) showed the absorption bands of a hydroxy group (3550 cm^{-1}), of the carbonyl of a γ -lactone ring (1760 cm^{-1}), and of a ketone carbonyl (1680 cm^{-1}).

The properties and analysis of the spectral characteristics of substance (III) showed its identity with the sesquiterpene lactone tamirin [5].

LITERATURE CITED

1. S. K. Cherepanov, *Vascular Plants of the USSR* [in Russian], Leningrad (1981), p. 99.
2. B. Kh. Abduazimov, A. I. Yunusov, S. A. Khamidkhodzhaev, and G. P. Sidyakin, *Khim. Prir. Soedin.*, 398 (1981).
3. P. Karrer, *Organic Chemistry*, 4th edn., Elsevier, Amsterdam (1950).
4. G. A. Kuznetsova, *Natural Coumarins and Furocoumarins* [in Russian], Leningrad (1967), p. 74.
5. V. A. Mnatsakanyan and L. V. Revazova, *Khim. Prir. Soedin.*, 396 (1974).

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