glucose. The IR spectrum (KBr) had absorption bands at 1670, 1630, and 1585 cm^{-1}, and the UV spectrum (methanol) had λ_{max} 257 and 410 mμ (log ε 4.01, 3.60). A pentaacetate with mp 187° C was obtained.

From its physical constants and its behavior on chromatograms in systems 1–8, the substance was identified as a monoglucoside of chrysophanic acid (chrysophanein) [1, 3].

The substance with mp 190° C was hydrolyzed, giving emodin and glucose (1:1). The IR spectrum (KBr) showed absorption bands at 1672, 1631 and 1589 cm^{-1}, and the UV spectrum (methanol) had λ_{max} 284 and 423 mμ (log ε 3.98, 3.76). It gave a hexaacetate with mp 205° C.

The constants given and the chromatograms in systems 1–8 agree well with the data for glucoemodin [1, 2, 4].

The substances with mp 258° and 198° C were identified by mixed melting points with authentic emodin and chrysophanic acid and also from their R_f values in solvent systems 1–8.

Thus, the seeds of Rheum tataricum contain chrysophanein (1.01%), glucoemodin (1.07%), chrysophanic acid (0.20%), and emodin (0.34%) and the leaves the same anthraquinone derivatives in amounts of 0.05%, 0.2, 0.10, and 0.03%, respectively.

REFERENCES

1. H. Wagner and L. Hörhammer, Z. Naturf., 18b, 89, 1963.
2. O. E. Schultz and G. Mayer, Arzneimittel Forsch., 6, 334–342, 1956.
3. A. S. Romanova and A. I. Ban'kovskii, Med. prom. SSSR, no. 10, 18, 1964.
4. A. S. Romanova, et al., KhPS [Chemistry of Natural Compounds], 83–85, 1966.

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A STUDY OF THE CHEMICAL COMPOSITION OF BUPLEURUM AUREUM

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We have studied the epigeal part of the plant Bupleurum aureum Fisch collected in the environs of Novosibirsk.

The air-dry material was ground and subjected to systematic extraction with petroleum ether, benzene, and methanol. By chromatography on alumina, the petroleum ether extract was separated into three fractions with mp 67°–71°, 76°–79°, and 126°–135° C.

The first fraction, after repeated recrystallization from petroleum ether, was obtained in the form of lamellar crystals with mp 73°–74° C. From its melting point, a mixed melting point, and a mixed thin-layer chromatogram the substance was identified as 10-nonacosan-one. Literature data: mp 74°–74.5° C [1]. The plant contains 0.1% of this substance.

After repeated recrystallization from methanol the second fraction was obtained in the form of colorless acicular crystals with mp 78.5°–80° C. On the basis of its IR spectrum, it was established that the compound is a long-chain alcohol; its acetate had mp 63°–63.5° C. Literature data for hexacosyl acetate: mp 65° C [2]. A direct comparison of these alcohols and their acetates showed the identity of both compounds. The content of 1-hexacosan-ol in the plant is 0.04%.

The third fraction was rechromatographed on alumina and recrystallized from methanol. Colorless lustrous crystals with mp 168°–170° C deposited. Literature data for α-spinasterol: mp 172° C [3]. The IR spectra showed that the substance was an alcohol of the sterol series. It gave an acetate with mp 183°–183.5° C (literature data: mp 185° C [4] and 187° C [5]), a benzoate with mp 197°–198° C (literature data: mp 196°–197°C [6] and 201° C [7]), and a nitrobenzoate with mp 210°–211° C (literature data: mp 211°–212° C). Consequently, the alcohol was identified as α-spinasterol.

The chromatography of the benzene extract on alumina gave very small amounts of 10-nonocosan-one and 1-hexacosan ol.

When the methanolic extract was left to stand, a yellow granular precipitate deposited which gave a positive reaction for flavones. The chromatography of this precipitate on kapron powder gave a pure flavone glycoside with mp 188°–200° C (decomp.). An authentic sample of rutin was used as a reference sample for thin-layer chromatography on a fixed layer of silica gel. The chromatography showed that the substance was rutin. This was confirmed by hydrolysis, yielding quercetin, rhamnose, and glucose.

The methanolic extract, after the elimination of the solvent, gave colorless crystals with mp 102.5°–103° C (from methanol). A mixture with an authentic sample of ribitol gave no depression of the melting point.

The methanolic extract was treated on a column of EDE-10P ion exchanger: water eluted ribitol, and 5% acetic acid yielded an eluate giving a positive reaction for alkaloids with silicotungstic acid.

A separate sample of the plant material was steeped in ammonia and extracted with chloroform and the extracted material was purified via the hydrochloride. The yield of total alkaloids in the form of a glassy mass was about 0.012% of the weight of the air-dry herb.

From literature data [8] on the high content of phytosterols in the leaves and flowers of this plant it is known that the bulk of the precipitate obtained with digitonin is 10-nonocosan-one containing about 8% of α-spinasterol digitonide. This was responsible for the positive Lieberman-Burchard reaction. Our experiments showed that 10-nonocosan one does not form a digitonide, but the coprecipitation of α-spinosterol digitonide and 10-nonocosan-one takes place.

We were unable to confirm literature information [8] on the high content of saponins in the plant. When procedures for isolating saponins were repeated, it was found that the precipitation of an alcoholic solution of the "saponins" with a large amount of ether gave crystalline ribitol.

REFERENCES

1. S. Furukawa, Scientific Papers of the Institute of Physical and Chemical Research, 19, 27, 1932.
2. L. Marion, Canad. J. Research, 12, 554, 1935.
3. M. C. Hort and F. W. Heyl, J. Biol. Chem., 95, 311, 1932.
4. D. Larsen and F. W. Heyl, J. Am. Chem. Soc., 56, 2665, 1934.
5. C. W. Choppee, Chemistry of the Steroids 61–62, 1958.
6. L. C. King and C. D. Ball, J. Am. Chem. Soc., 61, 2910, 1939.
7. D. H. R. Barton and J. D. Cox, J. Chem. Soc., 1354, 1948.
8. L. N. D'yakonova, New Medicinal Plants in Siberia [in Russian], no. 4, 116, 1953.

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