BRIEF COMMUNICATIONS

SHIKONIN FROM LITHOSPERMUM OFFICINALE

N. V. Tareeva, A. S. Romanova, A. I. Ban'kovskii, and P. N. Kibal'chich

Khimiya Prirodnykh Soedinenii, Vol. 2, No. 5, pp. 359–360, 1966

According to literature data [1] the roots of Lithospermum officinale L. (common gromwell) contain a red pigment which so far has not been studied.

The roots used for the investigation were collected in September 1965 in the Botanical Garden of the All-Union Scientific Research Institute for Medicinal and Aromatic Plants (Moscow Oblast). Qualitative reactions showed that they contained a pigment with similar properties to alkanin and shikonin [2, 3, 6, 7].

The pigment was isolated by Brockmann's method [4, 5]. 4.2 kg of the roots was extracted twice with petroleum ether (bp 40°–70° C) in a ratio of 1 : 6 for 18 hr. The red extracts were combined and evaporated in vacuum. This gave 7.95 g of a resinous substance. 2 grams of this substance was dissolved in 800 ml of petroleum ether and extracted with 1 *l* of 1 N aqueous potassium hydroxide, the pigment passing into the alkaline aqueous layer with a blue coloration. The alkaline solution was washed with petroleum ether (2 × 200 ml) and left to stand for 48 hr at room temperature. Then the solution was acidified with 50% acetic acid until a red color appeared (pH 6). This gave a precipitate of 1.1 g (yield on the raw material ~0.1%) of a dark red crystalline substance having the composition $C_{16}H_{16}O_5$, mp 143°–146° C (from benzene) which proved to be the known shikonin isolated from the roots of Lithospermum erythrorhizon Sieb. et Zucc.

The IR spectrum of the compound obtained had bands at 1622 cm^{-1} (C=O – OH of a carbonyl group linked by an intramolecular hydrogen bond to a neighbouring hydroxy group, 3150 (OH group), 1579 cm^{-1} (C=C double bond); and the IR spectrum had λ_{max} 215, 277, 515, 554 mμ (log ε 4.561, 3.960, 2.866, 3.661). The IR and UV spectra were identical with those of an authentic sample of shikonin. A 1% solution in benzene was dextrorotary.

REFERENCES

1. C. Wehmer, Die Pflanzenstoffe, **2**, 1018, 1931.
2. W. Karrer, Konstitution und Vorkommen der organischen Pflanzenstoffe, Basel und Stuttgart, 490, 1958.
3. H. Arakawa and M. Nakazaki, Chem. Ind., no. 25, 947, 1961.
4. H. Brockmann and H. Roth, Naturw., **23**, 246, 1935.
5. H. Brockmann, J. L. Annalen der Chemie, **521**, 1, 1936.
6. H. Raudnitz and E. Stein, Ber. Dtsch. Chem. Ges., **67**, 1955, 1934.
7. M. Nakazaki, Chem. and Industry., no. 25, 947, 1961.

31 March 1966

All-Union Scientific Research Institute for
Medicinal and Aromatic Plants

OSTRUTHOL FROM XANTHOGALLUM PURPURASCENS

G. K. Nikonov, Zh. A. Manaeva, and G. Yu. Pek

Khimiya Prirodnykh Soedinenii, Vol. 2, No. 5, pp. 360–361, 1966

The dihydropyranocoumarin xanthogallin has been isolated previously [1, 2] from the roots of X. purpurascens Lallem.

By chromatography on alumina with elution by benzene we have obtained from the mother liquor an additional amount of xanthogallin (0.82%) and a lactone of the composition $C_{21}H_{22}O_7$ with mp 141°–143° C (0.11%). The UV spectrum of the lactone has the following absorption maxima: λ_{max} 220, 250, 260, 267, 310 mμ (log ε 4.14, 4.16, 4.11, 4.14, 4.05); these are characteristic for furocoumarins substituted in position 5. Its IR spectrum (taken on a UR-10 spectrograph) exhibits absorption bands at 3505 cm^{-1} (hydroxyl), 3170, 3128 (C – H bond of a furan ring), 1697, 1630 (vibrations of furan and α-pyrone rings), and 1609, 1585 cm^{-1} (skeletal vibrations of a benzene ring) which also shows that the lactone belongs to the furocoumarin group.

The NMR spectrum (figure) shows that the lactone is a 5-monosubstituted furocoumarin (the spectrum was taken