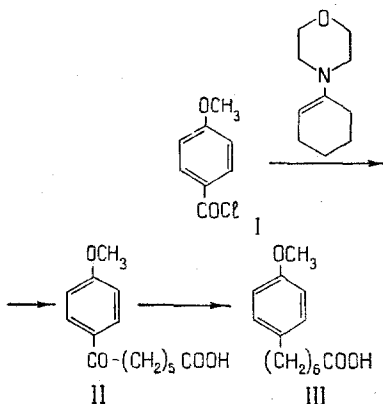


To build up the side chain we used the method of condensing an enamine with anisoyl chloride (I), which has been employed widely in recent years in the synthesis of acids [3]. In our case as the enamine we chose 1-morpholinocyclohexene, prepared from morpholine and cyclohexanone in the presence of p-toluenesulfonic acid by a known method [4].



The keto acid (II) with mp 132–133° C, and the composition C<sub>14</sub>H<sub>18</sub>O<sub>4</sub> was obtained. The IR spectrum of the acid contained, in addition to frequencies characteristic for a carboxy group (1718 cm<sup>-1</sup>) a frequency of 1680 cm<sup>-1</sup> due to a carbonyl group conjugated with an aromatic nucleus. In addition, the frequencies 2872 and 2935 cm<sup>-1</sup> show the presence of methylene groups in the molecule of the substance and the frequencies 1412 and 1430 cm<sup>-1</sup> relate to methylene groups in the α-position with respect to the carbonyl. The keto acid (II) forms a 2,4-dinitrophenylhydrazone with mp 115–117° C, C<sub>20</sub>H<sub>22</sub>O<sub>7</sub>N<sub>4</sub>.

The keto acid was reduced by a modification of the Kizhner-Wolff [Wolff-Kishner] method [5] to the acid (III). In the spectrum of this acid, the frequency of the carbonyl group had disappeared and the intensity of the frequency due to the methylene groups (2935 cm<sup>-1</sup>) had increased, and a methylene group had also remained in the α-position to a carbonyl group (1412 cm<sup>-1</sup>).

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7 July 1957

Institute of Chemical Sciences AS KazSSR

UDC 547.913.001.2

#### A NEW TERPENE HYDROCARBON — ACHILLENE

A. D. Dembitskii, R. A. Yurina, and M. I. Goryaev

Khimiya Prirodnikh Soedinenii, Vol. 4, No. 4, p. 251, 1968

In a study of the essential oil of *Achillea filipendulina* (fernleaf yarrow), from the fraction boiling at 60–65° C (40 mm Hg) by preparative gas-liquid chromatography, we isolated a substance possessing a strong peculiar odor and characterized by the following constants:  $n_D^{20}$  1.4526,  $d_4^{20}$  0.7836,  $[\alpha]_D^{20} + 64^\circ$ ,  $MR_D$  46.87. The molecular weight of the substance isolated, determined by mass spectrometry, was 136.

Strong bands at 890, 914, 998, 1636, and 3079 cm<sup>-1</sup> in the IR spectrum of the substance show the presence in its molecule of one vinyl -CH=CH<sub>2</sub> group and one methylene C=CH<sub>2</sub> group. The low intensity of the 1667 cm<sup>-1</sup> band and the moderate intensity of the 667 cm<sup>-1</sup> band are apparently due to the presence of a third multiple -CH=CH- bond. This is shown by the presence of a strong band at 1659 cm<sup>-1</sup> in the Raman spectrum of the substance.

The infrared and electronic spectra of the hydrocarbon confirm the absence of conjugation between its multiple bonds.

Thus, the data given show that we have isolated an aliphatic terpene hydrocarbon the structure of which obeys the isoprene rule. The most probable structure for it is 2,6-dimethyl-1,4,7-octatriene. To this terpene hydrocarbon we have given the name achillene.

27 February 1968

Institute of Chemical Sciences. AS KazSSR

UDC 543.852.3

#### BRITANNIN — A LACTONE FROM INULA BRITANNICA

K. S. Rybalko, V. I. Sheichenko, G. A. Maslova, E. Ya. Kiseleva, and I. A. Gubanov

*Khimiya Prirodnkh Soedinenii*, Vol. 4, No. 4, pp. 251-252, 1968

From the epigeal part of *Inula britannica* L. (British inula) family Compositae, collected in the flowering phase in the sinks of the northeastern outskirts of the Muyun-Kuma desert (close to Chu station, Dzhambul'skaya Oblast), we have isolated a colorless crystalline substance with the composition  $C_{19}H_{26}O_7$ , mp 189-191° C (from ethanol),  $[\alpha]_D^{18} -22.88^\circ$  (c 2.3; chloroform),  $1H_{labile}$ . On chromatography in a thin layer of neutral alumina (activity grade IV) in the petroleum ether-benzene-chloroform-methanol (5:4:2:1) system it had  $R_f$  0.2; and in the benzene-ethanol (9:1) system  $R_f$  0.32 (spots revealed with a 0.5% solution of potassium permanganate in 0.5% sulfuric acid).

The IR spectrum of the substance had absorption bands at  $3540\text{ cm}^{-1}$  (OH group) and  $1768\text{ cm}^{-1}$  (carbonyl of a  $\gamma$ -lactone), broad bands in the  $1710-1728\text{ cm}^{-1}$  region (C=O),  $1240-1270\text{ cm}^{-1}$  (OCO), and an absorption band in the  $1668\text{ cm}^{-1}$  region (C=C-C=O). The NMR spectrum shows signals of tertiary and quaternary methyl groups (doublet at 0.95 ppm and singlet at 1.00 ppm), of two acetoxy groups (singlets at 2.03 and 2.22 ppm), and of an exocyclic ethylene group conjugated with the lactone carbonyl (doublets at 5.41 and 6.14 ppm) [1]. When the substance was hydrogenated with a Pt catalyst (Adams), one mole of hydrogen was absorbed to form a dihydro derivative  $C_{19}H_{28}O_7$  with mp 222-224° C (from ethanol),  $1H_{labile}$ . The IR spectrum of the dihydro derivative had maxima at  $3500$  and  $3530\text{ cm}^{-1}$  (OH),  $1775$  ( $\gamma$ -lactone),  $1745$ ,  $1705$  (C=O), and  $1260\text{ cm}^{-1}$  (OCO), and the NMR spectrum lacked signals of an exocyclic methylene group.

The initial substance and its dihydro derivative dissolves in alkalis on heating, with the consumption of 3 moles of alkali, which confirms the spectral indication of the presence of a lactone ring and two ester groups. However, it has not yet been possible to isolate the hydrolysis products. Reduction of the dihydro derivative with lithium aluminum hydride led to a pentol  $C_{15}H_{28}O_5$  with mp 248-250.5° C,  $5H_{labile}$ . The IR spectrum of the latter had a maximum at  $3250-3450\text{ cm}^{-1}$  and absorption bands of a  $\gamma$ -lactone, of OCO, and of C=O were absent.

The substance isolated is a new sesquiterpene lactone not previously described in the literature. We have called it britannin.

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19 February 1968

All-Union Scientific-Research Institute for Medicinal Plants

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