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CHEMICAL STUDY OF *Petasites albus*

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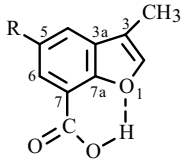
*The new benzofurans petalbin (**1**) and petalbone (**2**) were isolated from roots of* Petasites albus*. Their structures were established using physicochemical and spectral (IR, PMR, ^{13}C NMR, ^{13}C DEPT 135) data.*

Keywords: *Petasites albus*, benzofuran derivatives, petalbin, petalbone, spectroscopy, IR, PMR, ^{13}C NMR, DEPT 135.

In continuation of research on compounds extracted from roots of *Petasites albus* (L.) Gaertn., rechromatography of fractions obtained from a chromatography column [1] isolated two pure compounds of composition $C_{11}H_{10}O_3$, mp 133–134°C (**1**) and $C_{12}H_{10}O_4$, mp 138–140°C (**2**).



1, 2

1: R = CH_3
2: R = $COCH_3$

Analysis of IR, PMR, ^{13}C NMR, and ^{13}C DEPT 135 spectral data established that these compounds were new. The names petalbin (**1**) and petalbone (**2**) were proposed for them.

The IR spectrum of **1** had absorption bands at characteristic frequencies for carboxyl C=O (1670 cm^{-1}) and a benzofuran system (1660, 1620, 1580). The PMR spectrum of **1** exhibited resonances for methyls bonded to an aromatic ring at 2.56 (3H, s, CH_3–C= on C-5) and 2.70 ppm (3H, s, CH_3–C= on C-3); three resonances for aromatic protons at 7.06 (1H, s, CH=), 7.43 (1H, s, CH=), and 8.14 ppm (1H, s, CH=) that were assigned to H-4, H-2, and H-6, respectively; and a singlet for a carboxylic acid at 12.52 ppm (1H, s, COOH).

The ^{13}C DEPT 135 spectrum indicated that **1** contained only five protonated C atoms, two methyls (26.30 ppm, CH_3 on C-5; 26.75, CH_3 on C-3) and three aromatic (100.21, C-4; 113.04, C-2; 126.83, C-6).

A comparison of the PMR and ^{13}C DEPT 135 spectra of petalbusin, which was isolated earlier from this plant, with those of **1** led to the conclusion that the latter had a methyl (CH_3–Ar) instead of the isopropenyl group in petalbusin.

Therefore, **1** had the structure 3,5-dimethyl-7-carboxybenzofuran.

The IR spectrum of **2** had absorption bands at characteristic frequencies 1690 cm^{-1} (overall band of two conjugated carbonyls) and 1650, 1620, 1610, 1590 (aromatic system) [2, 3].

The PMR spectrum of **2** showed resonances for two methyls at 2.55 (3H, s, CH_3–C=) and 2.80 ppm (3H, s, CH_3–C=O). Singlets (1H) at 7.05, 7.70, and 8.50 ppm in the PMR spectrum belonged to three aromatic protons of furan (–CH=) and benzene (2–CH=) rings. A 1H singlet at 12.60 ppm was analogous to resonances for carboxylic acids in PMR spectra of petalbusin (12.70) [1] and petalbin (12.52).

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The ^{13}C NMR spectrum of **2** was recorded with full suppression of proton spin–spin coupling in order to determine the number of C atoms in it.

The spectrum showed 12 singlets belonging to 12 C atoms of **2**. These included two methyls on double bonds (20.0 and 21.0 ppm for C-5 and C-3, respectively) and three protonated (99.0, C-4; 114.5, C-2; 128.0, C-6) and seven unprotonated (118.0, 120.0, 148.5, 154.0, 160.0, 163.0, 187.5) aromatic C atoms.

Considering that the compounds contained in the plant material are components of the biosynthetic process occurring in the plant and, as a rule, are structurally similar to each other [4], we compared the PMR and ^{13}C NMR spectra of petalbone and petalbusin.

Thus, the comparison of PMR spectra of **2** and petalbusin showed that the spectrum of **2** was missing two singlets (5.20 and 5.70, CH_2=C<) that appeared in the PMR spectrum of petalbusin [1]. The ^{13}C NMR spectrum of **2** had a resonance for a carbonyl C atom (163.0 ppm), which was also present in the ^{13}C NMR spectrum of petalbusin (162.0), and an additional resonance (187.5) that belonged to a carbonyl C atom of a $H_3CC(O)$-5 group.

In summary, the comparison of PMR and ^{13}C NMR spectra of petalbusin and **2** allowed the latter to be assigned the structure 5-acetyl-3-methyl-7-carboxybenzofuran.

EXPERIMENTAL

General Comments. All solvents were freshly distilled.

The purity of the compounds was established by chromatography on UV-254 plates using benzene. IR spectra were recorded in mineral oil on a UR-20 spectrophotometer; PMR and ^{13}C NMR spectra, in acetone-d_6 with TMS internal standard on a Bruker spectrometer at 300 MHz (^1H) and 75 MHz (^{13}C). Chemical shifts are given on the δ-scale. Melting points were measured on a Boetius stage.

Extraction, chromatography, and preparation of pure compounds were performed as before [1].

Rechromatography. Fractions 16-18 that were obtained earlier [1] by elution with hexane were rechromatographed over a column of Al_2O_3 (neutral, act. III-IV) (h = 30, d = 1 cm) with elution by hexane. Fractions of 50 mL were collected.

Petalbin (1). $C_{11}H_{10}O_3$, mp 133–134°C (hexane), isolated from fractions 13–14. IR spectrum (ν_{max}, cm^{-1}): 1670, 1660, 1620, 1580, 1090, 1070, 950.

Petalbone (2). Fractions 16–20 afforded a crystalline compound of formula $C_{12}H_{10}O_4$, mp 138–140°C (hexane). IR spectrum (ν_{max}, cm^{-1}): 1690, 1650, 1620, 1610, 1590.

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