*Chemistry of Natural Compounds, Vol. 41, No. 4, 2005*

# COMPOSITION OF *Carlina acanthifolia* ROOT ESSENTIAL OIL

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UDC 547.913

*The root of* Carlina acanthifolia *All. (Asteraceae) contained 1.0% of essential oil (expressed in g per 100 g of dried plant material). Using GC and GC/MS, nine components were identified (100% of total oil). The structure of benzyl 2-furylacetylene (carlina oxide), which is the principal component of the oil (91.5%), was spectrometrically identified.*

**Key words**: *Carlina acanthifolia* All., essential oil composition, GC, GC/MS, benzyl 2-furylacetylene (carlina oxide), IR, MS, NMR.

The genus *Carlina* L. (Asteraceae) consists of about twenty species which are distributed from the Canaries through Europe to Asia. In the Flora of Serbia the genus is represented by three species: *C. vulgaris* L., *C. acaulis* L., and *C. acanthifolia* All. (syn. *C. utzka* Hacq.) [1].

*Carlinae radix*, the root of *C. acaulis*, is used in traditional medicine as a diuretic, diaphoretic, and stomachic and also as a gargle against catarrh. Externally, extracts of the root are used to wash herpetic eruptions, suppurating rashes (pyodermias) and other skin conditions, as well as against toothache. The drug contains 1–2% of essential oil, tannins, resins and inulin. The essential oil has antibacterial and antifungal activity. Examination of commercial samples of *Carlinae radix* has shown that most of them did not come from *C. acaulis*, as required by the Erg. B. 6, but consisted largely of the roots of *C. acanthifolia*. The root of *C. acanthifolia* is to be looked upon as an adulterant or a substitute for the root of *C. acaulis* [2, 3].

In this paper we investigated the composition of *C. acanthifolia* root essential oil.

*C. acanthifolia* is a perennial species with leaf rosette, without stem (rarely with stem up to a height of some centimeters) and with singular capitula, up to 10 cm in diameter. It is widespread in the hills and mountains of Eastern Serbia [1].

We established that the essential oil yield in the root of *C. acanthifolia* was 1.0% (expressed in g of oil per 100 g of dried plant material). Fresh isolated essential oil was a yellow liquid with an intense narcotic odor.

Using GC and GC/MS, nine components (100% of total oil) were identified (Table 1). The principal component of the oil (91.5%) was benzyl 2-furylacetylene (carlina oxide), an acetylene derivative with antimicrobial properties. Its structure was confirmed by IR, EI/MS, CI/MS, $^{1}$H NMR, and $^{13}$C NMR [4].

In the investigated essential oil the sesquiterpene hydrocarbons $\beta$-sesquiphellandrene, $\alpha$-zingiberene, *ar*-curcumene, and $\gamma$-curcumene were present in significantly lower quantities (2.8, 2.4, 1.6 and 1.1%, respectively). The rest of the components were detected in amounts less than 1%.

Results of the present study showed that *C. acanthifolia* root essential oil is very similar to the oil of *C. acaulis* root.

The most abundant constituent of *C. acaulis* root essential oil was also identified as benzyl 2-furylacetylene (carlina oxide) (80–97%) [3, 5, 6].

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 0009-3130/05/4104-0410 

TABLE 1. Composition of the *C. acanthifolia* Root Essential Oil

| Compound | RI* | RI** | Content, % |
|---|---|---|---|
| *trans*-$\beta$-Farnesene | 1457.2 | 1457 | 0.4 |
| $\gamma$-Curcumene | 1479.5 | 1483 | 1.1 |
| *ar*-Curcumene | 1483.1 | 1481 | 1.6 |
| $\alpha$-Zingiberene | 1484.4 | 1494 | 2.4 |
| $\beta$-Bisabolene | 1508.7 | 1506 | 0.1 |
| $\beta$-Sesquiphellandrene | 1524.6 | 1523 | 2.8 |
| *cis*, *trans*-Farnesal | 1534.4 | N.a. | 0.1 |
| *cis*-1-(2-Furyl)-2-phenylcyclopropane | 1546.1 | N.a. | Tr. |
| Benzyl 2-furylacetylene (Carlina oxide) | 1613.1 | N.a. | 91.5 |
| Total identified | | | 100.0 |

RI*: retention indices relative to C9-C24 alkanes on HP-5MS column.
RI**: retention indices [8].
N.a.: non available.
Tr.: trace (< 0.1%).

## EXPERIMENTAL

GC: Hewlett Packard 5890 II gas chromatograph, equipped with HP-5 fused silica capillary column (25 m × 0.32 mm, 0.52 μm film thickness) and FID. GC/MS: Hewlett Packard, GCD series II model G 1800 C, operating in the EI mode at 70 eV, equipped with fused silica 30 m × 0.25 mm, HP-5MS capillary column, with film thickness 0.25 μm. IR: Perkin Elmer FTIR 1725X spectrometer, neat liquid. MS: Finnigan-MAT 8230 BE geometry, resolution 1000, EI/CI source at 200°C. EI 70 eV, 0.5 mA; CI 1 mtorr of isobutane, 150 eV, 0.2 mA. NMR: Varian Gemini 2000.

**Plant Material.** The roots of *C. acanthifolia* were collected in August 2003 in Mt. Suva Planina (Eastern Serbia), during the period of full flowering. Once harvested, the plant material was dried at room temperature. Voucher specimens were deposited in the Herbarium of the Institute of Pharmacognosy, Faculty of Pharmacy, Belgrade.

**Isolation of Essential Oil.** The isolation of essential oil from dried powdered *C. acanthifolia* root was performed by steam distillation in a Clevenger-type apparatus, according to Procedure III of the Yugoslav Pharmacopoeia IV [7].

**Analysis of Essential Oil.** The composition of the oil and the relationship between the constituents were studied using GC and GC/MS. The operating conditions for GC analysis were: temperature program 40–280°C at a rate of 4°C/min, injector temperature 250°C, detector temperature 280°C, and carrier gas $H_2$ (1 mL/min). GC/MS analysis was performed using He as carrier gas (1 mL/min) with temperature program 40–280°C at a rate of 4°C/min, injector temperature 250°C, and detector temperature 280°C. The components of the oil were identified by comparison of their mass spectra with those from Adams, Wiley, NIST/NBS libraries.The results were correlated with their retention indices [8].

**Benzyl 2-Furylacetylene (Carlina Oxide)**. IR spectrum (neat liquid, ν, $cm^{-1}$): 2242 (C≡C), 3084 (C-H Ar), 1606, 1584, 1490 (C=C Ar), 984 (furyl), 739 (Ar).

EI/MS spectrum (EI, 70 eV), *m/z* ($I_{rel}$, %): 183 (12.7), 182 (96.9), 181 (36.6), 154 (16.1), 153 (99.9), 152 (68.8), 151 (17.5), 128 (11.2), 76 (23.2), 5 (10.4).

CI/MS spectrum (1 mtorr of isobutane, 150 eV), *m/z* ($I_{rel}$, %): 183 (100) $[M+H]^+$, 239 (50) $[M+isobutene+H]^+$, 365 (9) $[2M+H]^+$.

$^1H$ NMR spectrum (200 MHz, $CDCl_3$, δ, ppm, J/Hz, TMS=0): 3.82 (2H, s, $CH_2$), 6.34 (1H, dd, $J_1$ = 3.4, $J_2$ = 1.8, CH furano), 6.51 (1H, d, J = 3.4, CH furano), 7.2–7.4 (6H, m, CH phenyl + CH furano).

$^{13}C$ NMR spectrum (50 MHz, $CDCl_3$): 25.7 (1C, $CH_2$), 72.9 (1C, C≡C), 91.9 (1C), 110.7 (1C, CH), 114.2 (1C, CH), 126.7 (1C, CH), 127.9 (2C, phenyl), 128.6 (2C, phenyl), 135.9 (1C), 137.3 (1C), 142.9 (1C, CH).

**ACKNOWLEDGMENT**

This research was supported by the Ministry of Science, Technology and Development of the Republic of Serbia (Grant Nos. 1568 and 1755).

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