

# Isolation and Identification of 3-Octanone in the Essential Oil of *Rosmarinus officinalis* L.

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The essential oil of freshly distilled leaves of *Rosmarinus officinalis* L. was found to contain 3-octanone, a compound hitherto not reported to be present in this oil. Identification was established by comparing the gas chromatographic retention time and by mass spectrometry.

In the course of our investigations dealing with the improvement of the analysis of naturally occurring monoterpenes from essential oils [1–6] we recently carried out a routine analysis on the freshly distilled oil of *Rosmarinus officinalis*. In the monoterpene hydrocarbon part of the gas chromatogram at 60 °C (Fig. 1) an unknown compound (peak 17) was detected. The compound was present in an amount of 2.2% (calc. on the basis of the total

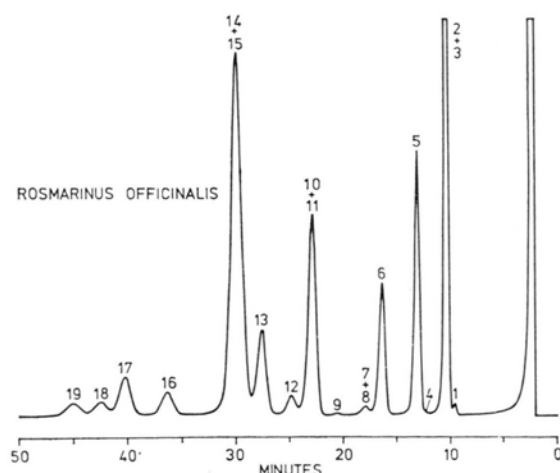


Fig. 1. Monoterpene hydrocarbon part of the gas chromatogram of the essential oil from *Rosmarinus officinalis* on a PEG 20M column at 60 °C: 1 = tricyclene; 2 =  $\alpha$ -pinene; 3 =  $\alpha$ -thujene; 4 =  $\alpha$ -fenchene; 5 = camphene; 6 =  $\beta$ -pinene; 7 = sabinene; 8 =  $\Delta_4$ -carene; 9 =  $\Delta_3$ -carene; 10 = myrcene; 11 =  $\alpha$ -phellandrene; 12 =  $\alpha$ -terpinene; 13 = limonene; 14 = 1.8-cineole; 15 =  $\beta$ -phellandrene; 16 =  $\gamma$ -terpinene; 17 = 3-octanone; 18 = p-cymene; 19 = terpinolene.

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oil). The first evidence that this compound was not a hydrocarbon but an oxygen-containing constituent, was obtained after liquid-solid column chromatography on silica gel. Elution with pentane gave the hydrocarbon fraction in which the new component could not be detected, whereas by elution with diethyl ether the oxygen-containing compounds were trapped. A gas chromatogram of this fraction showed two substances with relative retention times as short as those of the monoterpene hydrocarbons, viz. 1.8-cineole and the unknown compound. Comparing the relative retention time this compound was tentatively identified as 3-octanone. The compound was isolated from the diethyl ether fraction by means of preparative gas chromatography and transferred to a mass spectrometer. The MS, revealing a molecular weight of 128, was identical with that of a sample of pure 3-octanone and also in agreement with the MS of this compound given in literature [7–9].

That this compound was not an artefact formed during the distillation procedure of the oil could be demonstrated by the solid injection technique [10]. Also in the oil obtained by maceration of small portions of leaves with a mixture of pentane/diethyl ether (50/50) 3-octanone was present.

Previously 3-octanone has been reported in several species of the Labiatae, e.g. *Agastache formosana* [11], *A. rugosa* [12], *Glechoma hederacea* [13], *Lavandula angustifolia* [14], *L. hybrida* [15, 16], *L. spica* [17], *Mentha arvensis* [18–20], *M. gattefossei* [21], *M. japonica* [22], *M. piperita* [23, 24], *M. pulegium* [25, 26], *Mosla methylchavicolifera* [27], *Ocimum basilicum* [28–30], *Origanum vulgare* [31], *Schizonepeta tenuifolia* [32], *Thymus quinquecostatus* [33], but so far not in the essential oil of *Rosmarinus officinalis*.

## Experimental

**Isolation of the essential oil.** Fresh leaves (1 kg) of *Rosmarinus officinalis* L. were steam-distilled in a Clevenger type apparatus for ca. 12 h. The distillate was dried ( $\text{Na}_2\text{SO}_4$ ) to give 4 g of oil. Yield = 0.4%.

**Column chromatography (LSC).** 0.25 ml of the essential oil was subjected to LSC on 40 g "Silica gel 60, particle size 0.063–0.200 mm (70–230 mesh ASTM) for column chromatography" (E. Merck, Darmstadt, G.F.R.) with an established water content of 5% in order to prevent isomerisation processes during LSC [3]. A chromatographic tube



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(50cm  $\times$  18 mm I.D.) with a cooling jacket to keep the temperature at ca. 10 °C was used. The column was eluted with 300 ml pentane to give the monoterpene hydrocarbon fraction. Elution with diethyl ether (300 ml) gave a fraction with the oxygen-containing compounds. Both fractions were concentrated under reduced pressure in a rotary evaporator at ca. 0 °C to ca. 10 ml.

*Analytical gas chromatography* was achieved on a Becker Gas Chromatograph Model 409 (Packard-Becker, Delft, The Netherlands) equipped with F.I.D., using a 8 m  $\times$  1.5 mm I.D. copper column with 10% polyethyleneglycol (PEG) 20 M (= Carbowax 20 M) on Chromosorb W (60–80 mesh), acid washed. Carrier gas: N<sub>2</sub>. Temperature: 60 °C (for monoterpene hydrocarbon part) and 140 °C (for total oil). Sample size: 1  $\mu$ l. Quantitative data were obtained by means of an Infotronics CRS 208 electronic integrator (Infotronics Corporation, Shannon, Ireland).

*Isolation of 3-octanone* from the diethyl ether fraction obtained by LSC was carried out by means of *preparative gas chromatography* using a Becker Research Gas Chromatograph Type 3810 (Packard-Becker, Delft, The Netherlands) with F.I.D., an 8 m  $\times$  7 mm I.D. aluminium column with 20% PEG 20 M on Chromosorb W (60–80 mesh), acid washed. Carrier gas: N<sub>2</sub>. Temperature: 130 °C. Sample size: 100  $\mu$ l. After splitting at the end of the column the compound was trapped by forcing the effluent to run through a vessel filled with Chromosorb G (100–120 mesh) coated with 20% Silicone SF 96. The vessel was cooled to ca. –20 °C by a mixture of ice-NaCl.

*Mass spectrometry.* The coated Chromosorb sample with the trapped component was transferred into a MS apparatus. The MS was recorded at 70 eV on a AEI MS 902 (AEI Scientific Apparatus Ltd., Manchester, England). Temperature of ion source 130 °C, inlet system 150 °C above ambient.

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