

STUDIES ON CONSTITUENTS OF *ANGELICA DAHURICA*, II.<sup>1</sup>  
IDENTIFICATION OF  $\gamma$ -NONALACTONE AND  $\gamma$ -DECALACTONE BY GC AND  
GC/MS AS A PART OF THE ODOR COMPONENTS

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*Angelica dahurica* Benth. et Hook is a perennial herb of the family Umbelliferae. Its roots, locally known as "byakushi (*Angelicae daburicae* Radix)", have been used as a Chinese drug for analgesic and sedative purposes. The furocoumarin constituents of this plant have been studied extensively (1-3), but there has been no previous research on the chemical constituents responsible for its peculiar odor. In continuation of our previous studies on the same species (4), we now report the separation and identification of two lactones,  $\gamma$ -nonalactone and  $\gamma$ -decalactone, as a part of the odor constituents.

These constituents were obtained from the  $\text{CH}_2\text{Cl}_2$  extract of the dried roots. The extract was purified by column chromatography followed by preparative tlc to give odor components. The two  $\gamma$ -lactones were separated and identified by comparison of the retention times (Rt) on glc and mass spectra on combined gc-ms with those of authentic samples. The other odor components are under investigation.

#### EXPERIMENTAL

**PLANT MATERIAL.**—Roots of *A. dahurica* were collected in Kobe, Japan. A voucher specimen is on deposit in the Faculty of Pharmaceutical Sciences, Kobe-Gakuin University, Nishi-ku, Kobe, Japan 673.

**EXTRACTION AND SEPARATION.**—Roots (1 kg) were ground and extracted with  $\text{CH}_2\text{Cl}_2$  for about 48 h at room temperature. The concentrated extract (50 g) was chromatographed on a silica gel (Mallinckrodt 100 mesh, 100 g) column (3 × 80 cm), and elution was started with *n*-pentane followed by *n*-pentane- $\text{CH}_2\text{Cl}_2$  mixtures. Elution with 30%  $\text{CH}_2\text{Cl}_2$  in *n*-pentane afforded odor fraction. Further purification by preparative tlc using a silica gel (Merck Kieselgel 60 G) with *n*-pentane- $\text{CH}_2\text{Cl}_2$  (5:2) gave oily odor substances (Rf 0.14, 100 mg).

**ANALYSIS BY GLC AND GC/MS.**—Hitachi GC series type 163 (FID), with a 3 mm × 1 m, 5% PEG-20M on Chromosorb W (HP) glass column, was used for the glc analysis of the odor substances. Optimal conditions for the separation of 10- $\mu\text{g}$  samples were as follows: column temperature 150°; injector temperature 200°;  $\text{N}_2$  carrier gas flow rate, 50 ml/min.

Combined gc/ms was performed by Hitachi GC-MS model RMU60 single focusing spectrometer with 10% OV-101 glass column (3 mm × 1 m). Measured conditions were as follows: oven temperature, 100-200° (program rate 10°/min); injection temperature, 200°; helium carrier gas flow rate, 45 ml/min; ionization voltage, 20 eV; accelerating voltage, 1.3 kV; ion source temperature, 160°.

**RESULT OF ANALYSIS.**—In glc,  $\gamma$ -nonalactone and  $\gamma$ -decalactone were detected at Rt 240 sec and 384 sec, in accordance with those of authentic samples, respectively. The following gc/ms data for the two lactones, as indicated only by prominent ions, were consistent with those recorded for authentic samples:  $\gamma$ -nonalactone, *m/z* 150 ( $\text{M}^+$ , rel. int. 5), 138(6), 128(17), 114(5), 100(5), 85(100), 57(10), 56(13), 55(11);  $\gamma$ -decalactone, *m/z* 170 ( $\text{M}^+$ , rel. int. 8), 158(7), 123(15), 100(6), 85(100), 57(11), 56(12), and 55(14).

#### LITERATURE CITED

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<sup>1</sup>For part I, see Fujiwara *et al.* (4).