

2. T. Kh. Khasanov, A. I. Saidkhodzhaev, and G. K. Nikonov, *Khim. Prirodn. Soedin.*, 807 (1972).
3. A. Sh. Kadyrov, T. Kh. Khasanov, A. I. Saidkhodzhaev, and G. K. Nikonov, 808 (1972).
4. Kh. M. Kamilov and G. K. Nikonov, *Khim. Prirodn. Soedin.*, 718 (1972).

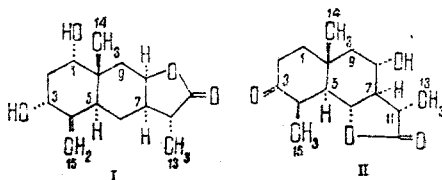
STRUCTURES OF ASHURBIN AND ARABSIN

S. Kh. Zakirov, Sh. Z. Kasymov, and G. P. Sidyakin

UDC 547.913

We have previously reported the isolation of the sesquiterpene lactones hanphyllin and granilin [1] from *Artemisia ashurbajevii* C. Winkl. Continuing the separation of the combined lactones, by means of a mixture of benzene and acetone (97:3) we have isolated a new sesquiterpene lactone with the composition $C_{15}H_{22}O_4$, mp 175-176°C, $[\alpha]_D^{18} +12.7^\circ$ (c 1.1; methanol), which we have called ashurbin. Ashurbin is readily soluble in acetone and pyridine and sparingly soluble in ether, benzene, chloroform, and water. Its IR spectrum had absorption bands at 3450-3150 cm^{-1} (OH group), 1780 cm^{-1} (γ -lactone carbonyl), and 1658 cm^{-1} (C=C). In the mass spectrum there are the peaks of ions with m/e 266 (M^+), $M-15$, $M-15-18$, $M-18-18$, and $M-15-18-18$, which shows the presence of two hydroxy groups in ashurbin. The NMR spectrum of ashurbin (deuteropyridine, δ scale) has the signals of tertiary and secondary methyls (singlet at 0.70 ppm and doublet at 1.05 ppm, $J = 8$ Hz). Signals at 3.39 and 4.43 ppm relate to hemihydroxyl protons. Consequently, both hydroxyls are secondary. The signal of the lactone proton is superposed on the signal of the hemihydroxyl proton at 4.43 ppm. Singlets at 4.56 and 4.91 ppm correspond to the protons of an exomethylene group, and the signals at 5.73 and 6.98 ppm to a hydroxylic proton. A comparison of the composition of granilin and ashurbin, and also a study of spectral characteristics, shows that ashurbin differs from granilin [2, 3] by the presence of a methyl group in place of an exomethylene group in the lactone ring. This group has the α orientation, since the signals of the proton at C_7 in granilin and ashurbin are in the form of multiplets in the 2.7 ppm-region and the sums of the spin-spin coupling constants differ by 3 Hz. Furthermore, the hydrogenation of granilin in the presence of 5% Pd/C gave dihydrogranilin with mp 174-175°C which was shown by its IR spectrum and by a mixed melting point to be identical with ashurbin. Consequently, ashurbin has the structure of 1,3 α -dihydroxy-5,7,8 α (H),11 β (H)-eudesm-4(15)-en-8,12-olide (I). Continuing a study of the lactones of the epigeal part of *Artemisia absinthium* L. [4], by the chromatographic separation of the combined lactones on silica gel (elution by benzene-acetone (9:1)) we isolated a mixture of two substances. By repeated recrystallization from benzene it was possible to separate them into two individual compounds. The first lactone was identified from its IR spectrum and melting point as absinthin [5], and the second ($C_{15}H_{22}O_4$, mp 193-194°C), as arabsin [4]. When artemisin was subjected to catalytic hydrogenation over 5% Pd/C, tetrahydroartemisin was obtained [6, 7], with mp 192-193°C, and according to its IR spectrum and a mixed melting point, this was identical with arabsin.

Thus, the structure and configuration of arabsin has been established as 8 α -hydroxy-3-oxo-4,5,7 α (H),6,11 β (H)-eudesman-6,12-olide (II):



Institute of the Chemistry of Plant Substances, Academy of Sciences of the Uzbek SSR, Tashkent. Translated from *Khimiya Prirodnikh Soedinenii*, No. 4, pp. 548-549, July-August, 1976. Original article submitted February 27, 1976.

This material is protected by copyright registered in the name of Plenum Publishing Corporation, 227 West 17th Street, New York, N. Y. 10011. No part of this publication may be reproduced, stored in a retrieval system, or transmitted, in any form or by any means, electronic, mechanical, photocopying, microfilming, recording or otherwise, without written permission of the publisher. A copy of this article is available from the publisher for \$7.50.

LITERATURE CITED

1. S. Kh. Zakirov, Sh. Z. Kasymov, U. Rakhmankulov, and G. P. Sidyakin, *Khim. Prirodn. Soedin.*, 397 (1976).
2. L. P. Nikonova and G. K. Nikonov, *Khim. Prirodn. Soedin.*, 289 (1972).
3. M. Maruyama and F. Shibata, *Phytochem.*, **10**, 2247 (1975).
4. I. S. Akhmedov, Sh. Z. Kasymov, and G. P. Sidyakin, *Khim. Prirodn. Soedin.*, 57 (1969), 691 (1970), 245 (1972).
5. L. Novotný, V. Herout, and F. Šorm, *Collect. Chem. Comm. Czech.*, **25**, 1492 (1960).
6. M. Sumi, *J. Am. Chem. Soc.*, **80**, 4869 (1958).
7. A. J. N. Bolt, W. Cocker, and T. B. U. McMurry, *J. Chem. Soc. Nov.*, 5235 (1963).

AN X-RAY DIFFRACTION INVESTIGATION OF A SINGLE CRYSTAL OF METHYL TRIPHYLLATE

A. N. Shnulin, Yu. T. Struchkov,
Kh. S. Mamedov, and G. S. Amirova

UDC 547.597

Methyl triphyllate (MT) has been isolated from the neutral products of the hydrolysis of an extract of the roots of *Glycyrrhiza triphylla* Fisch. et Mey [1]. Its composition has been established as $C_{31}H_{48}O_5$, mp 268–270°C. A single crystal of MT was obtained by recrystallization from ethanolic solution.

We have previously [2] reported x-ray structural investigations of another natural triterpene — methyl meristotropate. In the present paper we give preliminary x-ray diffraction results on MT. The parameters of the elementary cell were determined from rocking x-ray diffraction patterns and Weissenberg patterns with copper radiation.

The parameters were defined by the method of least squares for 12 reflections on a Hilger-Watts automatic four-circle x-ray diffractometer:

$$\begin{aligned}a &= 12.766(1) \text{ \AA}, \\b &= 51.882(4) \text{ \AA}, \\c &= 6.858(1) \text{ \AA}, \\\gamma &= 90.53^\circ(1)^\circ.\end{aligned}$$

Monoclinic System. Taking systematic extinctions into account leads to the space groups B2 and B2/m. The number of molecules per elementary cell found on the basis of the molecular weight and the density ($Z = 4$) unambiguously determines the space group B2.

A three-dimensional set of intensities consisting of 1804 independent reflections ($\lambda Cu_\alpha = 1.5418 \text{ \AA}$) has been obtained on the diffractometer mentioned.

LITERATURE CITED

1. N. P. Kir'yalov and G. S. Amirova, *Khim. Prirodn. Soedin.*, 311 (1965).
2. A. N. Shnulin et al., *Khim. Prirodn. Soedin.*, 517 (1975).

Institute of Inorganic and Physical Chemistry, Academy of Sciences of the Azerbaidzhan SSR, Baku. Institute of Heteroorganic Compounds, Academy of Sciences of the USSR, Moscow. Institute of Botany, Academy of Sciences of the Azerbaidzhan SSR, Baku. Translated from *Khimiya Prirodnikh Soedinenii*, No. 4, p. 549, July-August, 1976. Original article submitted December 22, 1975.

This material is protected by copyright registered in the name of Plenum Publishing Corporation, 227 West 17th Street, New York, N. Y. 10011. No part of this publication may be reproduced, stored in a retrieval system, or transmitted, in any form or by any means, electronic, mechanical, photocopying, microfilming, recording or otherwise, without written permission of the publisher. A copy of this article is available from the publisher for \$7.50.