CRYSTAL STRUCTURE OF THE NEW DILACTONE ANABSIN

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We have previously reported the isolation and determination of the structure of the diguaianolide anabsin $C_{30}H_{40}O_7$ [1], which was obtained from *Artemisia absinthium* L. family Compositae.

In order to determine the linkage of the monomeric moieties of anabsin and its spatial structure unambiguously, we have performed an x-ray structural investigation.

The crystals are monoclinic, $a = 9.863(4)$ Å, $b = 14.240(6)$ Å, $c = 11.149(6)$ Å, $\gamma = 103.61°$, $V = 1521.90$ Å^3, $M = 512.65$, $\rho_{calc} = 1.12$ g/cm^3, $Z = 2$, space group $P2_1$.

A three-dimensional set of intensities (2035 reflections) was obtained on a Syntex $P2_1$ automatic diffractometer ($\theta/2\theta$ method, using CuK_α radiation and not taking absorption into account).

The structure was determined by the direct method using the tangent formula by the "Rentgen-75" system of programs [2]. To calculate the phase variants, from 300 normalized amplitudes ($E \geqslant 1.4$) as the coordinate and reference reflections we took the following:

Index of the reflection	Coordinate reflections			Reference reflections				
h	$\bar{1}$	0	$\bar{9}$	0	$\bar{3}$	$\bar{4}$	3	4
k	1	3	3	1	4	5	6	8
l	1	0	0	0	1	7	5	4

In the E-synthesis with the best R factor (0.24) we found 30 out of the 37 atoms of the molecule. Having assigned to these 30 atoms the scattering capacities of carbon atoms, we plotted a three-dimensional electron density series (R = 0.39) which showed up all the atoms of the anabsin molecule. After arranging atoms according to variety, the structure of the diguaianolide was refined by the method of successive approximations to R = 0.22.

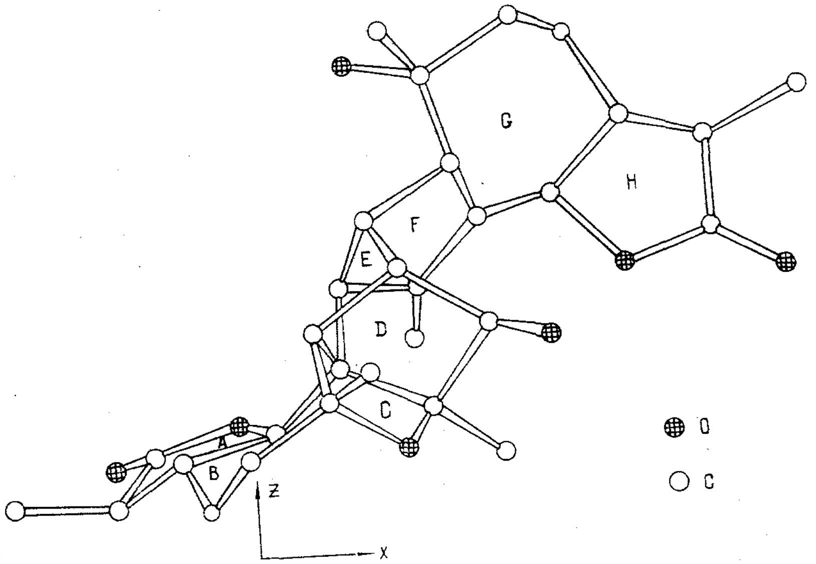

Fig. 1. Conformation of the anabsin molecule as projected on the XZ plane.

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The results obtained permitted the position of attachment of the two monomeric lactones to be established unambiguously and the conformation of anabsin to be determined. The conformation of the molecule as projected on the XZ plane is shown in Fig. 1.

The five-membered rings A, C, D, and H have the envelope conformation, while the five-membered ring F is plane. The seven-membered ring G has the chair conformation and ring B a distorted twisted chair. The ring linkages are: A/B and G/H, trans; E/F, cis. The bond lengths and valence angles are the usual ones.

LITERATURE CITED

1. Sh. Z. Kasymov, N. D. Abdullaev, G. P. Sidyakin, and M. R. Yagudaev, Abstracts of the Second International Symposium on the Chemistry of Natural Compounds, Bulgaria (1978). p. 421.
2. V. I. Andrianov, Z. Sh. Safina, and B. L. Tarnopol'skii, Zh. Strukt. Khim., 15, 911 (1974).

FEGVOLIDE — A LACTONE FROM THE ROOTS OF *Ferula gigantea*

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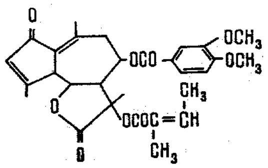
A series of sesquiterpene lactones has been isolated from *Ferula gigantea* B. Fedtsch. *F. latifolia* Korov has also been assigned to *F. gigantea* [2]. From an acetone extract of giant fennel roots collected at the classical find site of *F. latifolia* (environs of the village of Shuroabad, TadzhSSR), by chromatographing three times on a column of silica gel L40/100 μ we have isolated three sesquiterpene lactones: $C_{29}H_{32}O_9$ (I), mp 210-212°; $C_{26}H_{28}O_9$ (II), mp 213-215°C; and $C_{29}H_{32}O_9$ (III), mp 218-220°C.

On the basis of the agreement of their melting points and the identity of their IR and PMR spectra, compounds (I) and (II) were identified as malaphyll and malaphyllin, respectively. Substance (III) proved to be new not previously described in the literature, and we have called it fegvolide. The parameters of its IR spectrum are close to those of the IR spectra of malaphyll and malaphyllin and show that (III) is a diacyloxyguaianolide; 1790 cm^{-1} (γ-lactone $C{=}O$); 1710 cm^{-1} ($C{=}O$ of an acyloxy group); 1690 ($C{=}O$ of an α,β-unsaturated cyclopentanone); 1640, 1620, 1650 cm^{-1} ($C{=}C$). The PMR spectrum of compound (III) ($CDCl_3$, TMS, Varian HA-100D) is characteristic for 6,11-diacyloxy-1-oxoguai-2,8-dien-4,5-olides and differs from the PMR spectra of malaphyll and malaphyllin by the parameters of only one acyloxy group — an angelic acid residue: 6.21 us, $W_{1/2}$ = 4 Hz, 1H(H-2); 4.76, q, $J_{4,5}$ = 10 Hz, $J_{4,10}$ = 11.0 Hz, 1H(H-4); 3.73, q, $J_{4,5}$ = 10.0 Hz, $J_{5,6}$ = 11.0 Hz, 1H(H-5); 5.78 sx, $J_{6,7e}$ = 4.0 Hz, $J_{5,6} = J_{6,7a}$ = 11.0 Hz, 1H(H-6); 2.96, q, $J_{7e.7a}$ = 18 Hz; $J_{7e,6}$ = 4.0 Hz, 1H(H-7e); 2.56, q, $J_{7a,7e}$ = 18 Hz, $J_{7a,6}$ = 11.0 Hz; 3.66, d, $J_{4,10}$ = 10.2 Hz, 1H(H-10); 2.25, s, 6H(CH_3-14, CH_3-15); 1.64, s, 3H(CH_3-13); veratroyl: 7.64, q, J_1 = 8.0 Hz, J_2 = 2.5 Hz, 1H(H-6'); 7.49, d, J = 2.5 Hz, 1H(H-2'); 6.84, d, J = 8.0 Hz, 1H(H-5'); 3.93, s, (OCH_3); 3.89, s, (OCH_3); angeloyl: 6.24, q, 1H($CH{=}$); 1.96, d, 3H($CH_3{-}CH{=}$); 1.88 us, ($CH_3{-}C{=}C{-}\underline{CH}_3$).

Thus, fegvolide corresponds to the following structure:



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