X-RAY STRUCTURAL INVESTIGATION OF METHYL MERISTROTROPATE

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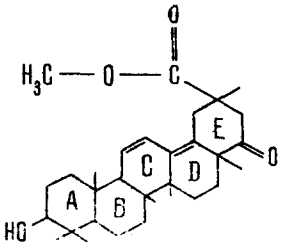
We have previously reported the results of a preliminary x-radiographic investigation of methyl meristrotropate (MM) $C_{31}H_{46}O_4$, isolated from the roots of Glycyrrhiza tryphylla Fisch. et Mey. [1, 2]. We have now completed a full x-ray structural analysis of MM; the crystal are rhombic, space group $P2_12_12$, a=12.492(3) Å, b=29.73(1) Å, c=7.356(1) Å, d_{meas} =1.18 g/cm^3, Z = 4.

The experiment was performed on a Hilger–Watts four-circle automatic x-ray difractometer with cooper radiation. The structure was interpreted by the direct method using a published program [3] and was refined by the method of least squares in the isotropic approximation to R = 0.14 for 1958 reflections. A molecule of water of crystallization was found by the Fourier difference synthesis, so that the actual composition of the substance studied was $C_{31}H_{46}O_4 \cdot 0.5H_2O$.

The structural formula of MM found has the form shown below, and differs from that proposed on the basis of the NMR spectra [4] by the position of the keto group at C_{22}, and not at C_6 (the results of mass-spectroscopic and chemical investigations [5] agree with our conclusions).



The bond lengths and valence angles are the usual ones. The conformations of rings A, B, and E are chair, and of C and D half-chair. In the crystal, the MM molecules are combined into infinite chains parallel to the b axis by strong hydrogen bonds (length 2.78 Å) between the hydroxy group of one molecule and the keto group of a neighboring molecule. The chains are linked into a layer parallel to the ab plane by hydrogen bonds from the hydroxy groups to the water molecules.

LITERATURE CITED

1. A. N. Shnulin, G. S. Amirova, and N. M. Mustafaev, Khim. Prirodn. Soedin., 661 (1972).
2. N. P. Kir'yalov and T. N. Naugol'naya, Zh. Obsch. Khim., No. 2, 33 (1963).
3. B. L. Tarnopol'skii, V. I. Andrianov, and Z. Sh. Safina, Roentgen-70. Automated System of Programs for X-Ray Structural Calculations [in Russian], Moscow (1972).
4. N. P. Kir'yalov and G. S. Amirova, Khim. Prirodn. Soedin., 60 (1968).
5. A. D. Zorina, Author's Abstract of Candidate's Dissertation, Leningrad (1974).

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