Chemistry of Natural Compounds, Vol. 41, No. 5, 2005

CRYSTAL AND MOLECULAR STRUCTURE OF 12-EPINAPELLINE

**Atta-ur-Rahman,[1] Atia-tul-Vakhab,[1] B. Tashkhodzhaev,[2]
K. K. Turgunov,[2] M. N. Sultankhodzhaev,[1,2] and M. I. Choudry[1]**

UDC 547.944/945+548.737

Previous investigations showed that the conformation of ring A in crystal structures of napelline-type alkaloids depended on the presence or absence of an intramolecular H-bond between an α-oriented C1 hydroxyl and the N atom. Thus, ring A in 12-epilucidusculin had the boat conformation [1]; in acophine it adopted the twist—chair conformation [2]; whereas in 12-acetyl-12-epinapelline it had the ideal chair conformation [3]. These facts generate interest in the structure of 12-epinapelline (**1**), which was isolated from *Aconitum karacolicum* Rap. and characterized by IR, PMR, and mass spectral methods [4].

Single crystals were grown from ethanol solution and were elongated transparent prisms. Table 1 lists the principal crystallographic data and the experimental conditions of the x-ray structure analysis. The structure was solved and refined by the usual methods [5].

Figure 1 shows the molecular structure and atomic numbering for **1**. **1** with the perhydrophenanthrene C skeleton consists of six main rings. Ring A (C1-C5,C10) adopts an almost ideal chair conformation (within ±0.011 Å). Rings B (C5-C7,C10,C20) and C (C7-C10,C20) have the 20α-envelope conformation (within ±0.068 and ±0.064 Å, respectively) where C20 deviates by 0.887 and 0.840 Å, respectively. Six-membered ring D (C8,C9,C11-C14) has an ideal boat conformation (±0.011 Å). Five-membered ring E adopts the 14α-envelope conformation (±0.031 Å) with C14 deviating (0.758 Å) from the plane of the other four atoms. Heterocycle F (C4,C5,C10,C19,C20,N) is a slightly distorted chair (±0.051 Å). Rings A and B are *cis*-fused; C and D, *cis*. The conformation and fusion of rings in **1** are consistent with those observed in 12-acetyl-12-epinapelline. Substituents in the C skeleton are hydroxyls on C1 with the α-equatorial orientation and on C12 and C15 with the β-orientation.

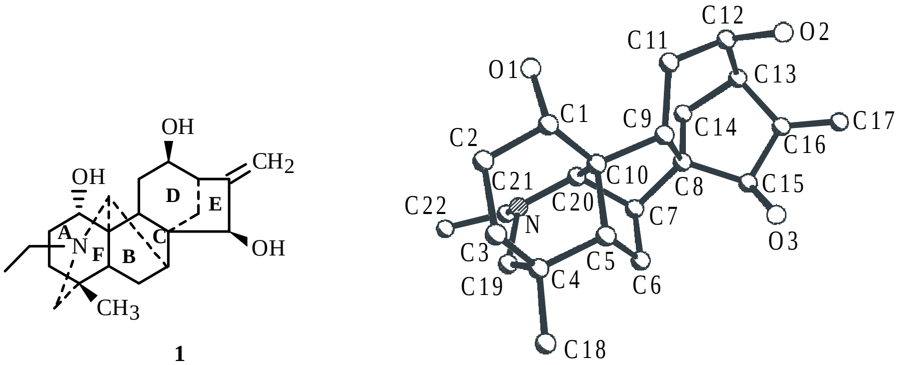
TABLE 1. Crystallographic Data, Experimental Conditions, and Refinement Parameters for **1**

Empirical formula	$C_{22}H_{34}NO_3$
Molecular weight	360.50
Temperature, K	293 (2)
Space group	$P2_12_12_1$, Z = 4
a, Å	9.952 (2)
b, Å	13.369 (3)
c, Å	14.718 (3)
V, Å^3	1958.2 (7)
ρ, g/cm^3	1.223
Absorption coefficient, μ, mm^{-1}	0.080
Crystal dimensions, mm	0.98×0.50×0.50
Range θ°	from 2.55 to 25.97
Total number of reflections	2099
Number of reflections [I > 2σ(I)]	1884
R-factor [I>2σ(I)]	R1 = 0.0554, wR2 = 0.1364
R-factor (whole dataset)	R1 = 0.0627, wR2 = 0.1435
GOOF	1.043
Difference ED peak	0.339 and -0.219 e Å^{-3}

---

1) Research Institute of Chemistry, International Center of Chemical Sciences of the University of Karachi, Karachi-75270, Pakistan; 2) S. Yu. Yunusov Institute of the Chemistry of Plant Substances, Academy of Sciences of the Republic of Uzbekistan, 700170, Tashkent, fax (99871) 120 64 75. Translated from Khimiya Prirodnykh Soedinenii, No. 5, pp. 498-499, September-October, 2005. Original article submitted March 22, 2005.

0009-3130/05/4105-0609



1

Fig. 1. Molecular structure and atomic numbering for 12-epinapelline.

The molecular packing in the crystal involves an intermolecular H-bond O–H...O along the *a* screw axis. The parameters of the H-bond are O2...O1, 2.83 Å, O2...H1 2.02 Å, and 169° angle around H1. The resulting infinite chain is weakly bonded through a H-bond to another chain formed by translation along the *c* axis (O3–H...O1) with parameters 2.96 and 2.29 Å and 169°. This interaction forms a two-dimensional network in the x0z plane.

Data from the x-ray structure analysis were deposited as a CIF-file in the Cambridge crystallographic Data Center (No. CCDC 266492).

REFERENCES

1. A. Yoshino and Y. Litaka, *Acta Crystallogr.*, **21**, 57 (1966).
2. B. Tashkhodzhaev, M. N. Sultankhodzhaev, and I. M. Yusupova, *Khim. Prir. Soedin.*, 267 (1993).
3. B. T. Salimov, K. K. Turgunov, B. Tashkhodzhaev, and F. N. Dzhakhangirov, *Khim. Prir. Soedin.*, 129 (2004).
4. M. N. Sultankhodzhaev and M. S. Yunusov, *Khim. Prir. Soedin.*, 386 (1987).
5. B. Tashkhodzhaev and A. I. Saidkhodzhaev, *Khim. Prir. Soedin.*, 125 (2005).