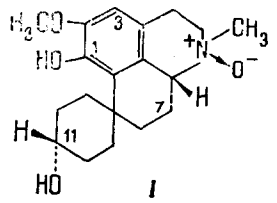

*I*

Trigamine N-oxide is the first representative of the homoproaporphine alkaloids containing a N-oxide group.

Furthermore, we have determined the configuration of the tertiary hydroxy group ($C_{11}$–H) of the base from the results of a study of the photon spectra of (II) and of its diacetyl derivative (III). In the spectra of compounds (I) and (II), the signals of this proton appeared at 3.95 ppm, and in (III), as the result of a downfield shift (5.02 ppm) it was possible to determine its half-width unambiguously, which amounted to 8.8 Hz. This showed that the $H_{11}$ proton was equatorial and, therefore, the hydroxy group at $C_{11}$ had the axial orientation. We confirmed the structure and configuration of trigamine by showing its identity with one of the products of the reductive decomposition of regelamine – 1-hydroxy-12-deoxy-1,12-de-epoxyregelamine.

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# THE STRUCTURE OF REGILININE


M. K. Yusupov, B. Chommadov, and Kh. A. Aslanov



UDC 547.944.6


We have previously [1, 2] reported on the isolation from the epigeal parts of Colchisum kesselringii Rgl. of, together with other alkaloids, regelinine, which was characterized by its melting point (253-254°C) and the $R_f$ value of its methiodide. The base in the free form is unstable and is fairly easily oxidized.

The UV spectrum of the base has absorption maxima at 243 and 293 nm, and the IR spectrum has absorption bands of active hydrogen (3370 $cm^{-1}$), of C=C bonds of a benzene ring (1600 $cm^{-1}$), and of methylene groups (1470 $cm^{-1}$). Its PMR spectrum shows the signals of the protons of a N-methyl group (2.36 ppm), of O-methyl groups present in a benzene ring (3.74 ppm), and of an aliphatic part of the molecule (3.32 ppm). In the weak field region of the spectrum there is the signal of one benzene-ring proton (6.44 ppm). The mass spectrum contains the peaks of ions with m/z 345 ($M^+$), 344 $(M-1)^+$ (100%), 302, 298, 284, 244, 242, 229.

On the basis of the spectral characteristics given above, regelinine was assigned to the group of homoproaporphine alkaloids with a spirocyclohexane ring, and according to its developed formula, $C_{17}H_{17}$ (OH) $(OCH_3)_2$ (–O–) $(NCH_3)$, it was analogous to regeline, isolated from the same plant [1-3]. We therefore assumed that this base might be an epimer of regeline differing by the configuration of the hydroxy group at $C_{11}$. A similar example was observed previously in relation to kesselringine and luteine in Colchicum luteum Baker [4-6]. To answer this question, regelinine methiodide was acetylated with acetyl chloride, giving an acetyl derivative. The IR spectrum of the latter contained the absorption band of an O-acetyl group (1750 $cm^{-1}$), and its PMR spectrum (in $CDCl_3$) contained the signals of the protons of two O-methyl groups (3.80 and 3.28 ppm), of two N-methyl groups (3.62 and 3.10 ppm), and of an acetyl group (2.08 ppm). The signal of an aromatic proton appeared at 6.50 ppm and that

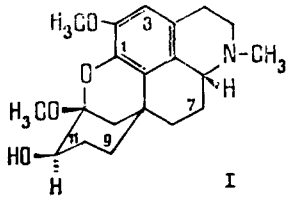

V. I. Lenin Tashkent State University. M. I. Kalinin Turkmen Agricultural Institute, Ashkhabad.


Translated from Khimiya Prirodnykh Soedinenii, No. 3, pp. 419-420, May-June, 1985. Original article submitted January 8, 1985.




0009-3130/85/2103-0396$09.50

of a third proton geminal to the $C_{11}$ acetoxy group at 5.0 ppm. The width of the signal of the $H_{11}$ proton was $\Delta\omega$ 25-27 Hz, which showed its axial nature. Consequently, the hydroxy group of regelinine at $C_{11}$ has the equatorial orientation and the base has the structure (I).




I

The structure of regelinine was confirmed by its identification with the methyl ether of luteine, and by analogy with the latter it was assigned the $C_{6a}$ R-configuration.

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