

X-Ray Analysis of Nic-11 from *Nicandra physaloides*; a 17-Spirobicyclo-methylsteroid

By MICHAEL BEGLEY, LESLIE CROMBIE,* PETER J. HAM, and DONALD A. WHITING*
(Department of Chemistry, The University, Nottingham NG7 2RD)

Summary The X-ray structure of the steroid Nic-11 is reported.

Nicandra physaloides (Solanaceae), a plant with insect repellent properties, is rich in steroidal derivatives. We have so far elucidated the structures of six of these compounds¹ (Nic-1, 3, 7, 10, 12, and 17)[†] and now report on Nic-11. Nic-11, m.p. 270°, C₂₈H₄₆O₇,[‡] formed an ethyl acetal crystallising in space group *P*2₁2₁2₁ with *a* = 8.04, *b* = 16.34, *c* = 20.43 Å and *Z* = 4. Intensity data were collected with Cu-*K*_α radiation using an automatic four-circle diffractometer, and 2143 reflections were considered observed. The structure was determined by direct methods using the Multan programme,² and refined by block-diagonal least-squares to a current *R* index of 9.5%. The acetal has structure (1) and Nic-11 is thus revealed as an oxidised hexacyclic methylsteroid, carrying a dioxaspirobicyclic system at C-17.³

The compound is related to Nic-3 [(2) rings A—C as in (3)]¹ from which it is likely to be derived by 17-β-hydroxylation.³ Intramolecular opening of the 24,25-epoxide by the 17-β-hydroxy-group would then give rise to the dioxabicyclic nonane spiro-attachment of (3). The stereochemistry of Nic-11 accords with such a derivation.

(Received, 24th August 1973; Com. 1210.)

[†] See ref. 1 for terminology.

[‡] Elemental analysis and mass spectrometry.

¹ (a) M. J. Begley, L. Crombie, P. J. Ham, and D. A. Whiting, *J.C.S. Chem. Comm.*, 1972, 1108; (b) *ibid.*, p. 1250.

² G. Germain, P. Main, and M. M. Woolfson, *Acta Cryst.*, 1971, **A27**, 368.

³ R. B. Bates and S. R. Morehead, *Tetrahedron Letters*, 1973, in the press.

