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# Structure of 1,2,3,5,6,10b$\alpha$-Hexahydro-6$\beta$-hydroxy-4$\alpha$-methyl-6$\alpha$-phenylpyrrolo[2,1-*a*]isoquinolinium Iodide

BY MASOOD PARVEZ

*Department of Chemistry, The Pennsylvania State University, University Park, PA* 16802, *USA*

BRUCE E. MARYANOFF AND DAVID F. MCCOMSEY

*Chemical Research Department, Janssen Research Foundation, Spring House, PA* 19477, *USA*

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**Abstract.** $C_{19}H_{22}NO^+.I^-$, $M_r = 407{\cdot}30$, orthorhombic, *Pbca*, $a = 13{\cdot}687\,(2)$, $b = 14{\cdot}056\,(2)$, $c = 18{\cdot}055\,(4)$ Å, $V = 3473{\cdot}3$ Å$^3$, $Z = 8$, $D_x = 1{\cdot}558$ Mg m$^{-3}$, $\lambda$(Cu $K\alpha$) = 1·5418 Å, $\mu = 14{\cdot}64$ mm$^{-1}$, $F(000) = 1632$, $T = 293\,(1)$ K, $R = 0{\cdot}0689$ for 1762 observed reflections with $I > 3\sigma(I)$. The six-membered central ring is in a distorted chair conformation and the five-membered ring is *cis*-fused to it. The planes of the phenyl rings are inclined at 111·9 (3)°. The I atom and the O atom of the hydroxy group are in van der Waals contact [I···H(O1) 2·57 Å, I···HO1—O(1) 163·0°].

**Experimental.** The preparation of the title compound has been described elsewhere (Sorgi, Maryanoff, McComsey, Graden & Maryanoff, 1989). Colorless crystals were obtained by slow crystallization at room temperature from a mixture of methanol:2-propanol. An elongated needle of approximate size 0·16 × 0·22 × 0·40 mm was used for data collection. Accurate cell dimensions and a crystal-orientation matrix were determined on an Enraf–Nonius CAD-4 diffractometer by a least-squares refinement of the setting angles of 25 reflections in the $\theta$ range 20–30°. Intensity data were collected by the $\omega$–$2\theta$ scan method with $\omega$-scan width $(0{\cdot}60 + 0{\cdot}14\tan\theta)°$ and variable scan speed of 1·3–5·5° min$^{-1}$ using graphite-monochromatized Cu $K\alpha$ radiation with indices $h$ 0 to 16, $k$ 0 to 16 and $l$ 0 to 21 in the $\theta$ range 5–65°. The intensities of three standard reflections measured every 2 h showed no evidence of crystal decay. Intensities of 3399 reflections were measured, 1762 had $I > 3\sigma(I)$, and were used in the structure solution and refinement. Data were corrected for Lorentz and polarization factors and for empirical absorption (North, Phillips & Mathews, 1968); maximum and minimum transmission coefficients were 1·00 and 0·33, respectively.

The structure was solved by the heavy-atom method. Refinement of the structure was by full-matrix least-squares calculations on $F$'s, initially with isotropic and finally with anisotropic temperature factors for the non-H atoms. At an intermediate stage in the refinement a difference map revealed all H atoms which were included in subsequent cycles at geometrically idealized positions (C—H 0·95 Å), with an overall isotropic temperature factor. Refinement converged with $R = 0{\cdot}069$ and $wR = [\sum w\Delta^2/\sum F_o^2]^{1/2} = 0{\cdot}093$. Max. shift/e.s.d. was less than 0·01, $S = 1{\cdot}596$, $w = 1/[\sigma^2(F_o) + 0{\cdot}10(F_o)^2]$, min. and max. electron density in the final difference map being $-0{\cdot}39$ and 0·43 e Å$^{-3}$, respectively. Scattering factors were those of Cromer & Mann (1968) and Stewart, Davidson & Simpson (1965) and allowance was made for anomalous dispersion (Cromer & Liberman, 1970). All computer programs used were part of the Enraf–Nonius *Structure Determination Package* (B. A. Frenz & Associates Inc., 1985), and the figures were plotted using *ORTEP* (Johnson, 1976).

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Table 1. *Final fractional coordinates and equivalent isotropic thermal parameters* (Å$^2$) *with e.s.d.'s in parentheses*

$$B_{eq} = (1/3)\sum_i\sum_j B_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

| | $x$ | $y$ | $z$ | $B_{eq}$ |
|---|---|---|---|---|
| I | 0·55828 (5) | 0·75100 (5) | 0·51746 (4) | 4·24 (1) |
| O | 0·5282 (4) | 0·6017 (4) | 0·3650 (4) | 3·3 (1) |
| N | 0·7005 (5) | 0·4250 (5) | 0·4460 (4) | 2·6 (1) |
| C1 | 0·7000 (8) | 0·6344 (7) | 0·2726 (5) | 3·8 (2) |
| C2 | 0·7845 (8) | 0·6716 (8) | 0·2462 (6) | 4·3 (2) |
| C3 | 0·8709 (8) | 0·6475 (7) | 0·2813 (6) | 4·7 (2) |
| C4 | 0·8723 (7) | 0·5871 (7) | 0·3419 (6) | 4·1 (2) |
| C5 | 0·7847 (6) | 0·5477 (7) | 0·3674 (5) | 2·9 (2) |
| C6 | 0·6962 (6) | 0·5721 (6) | 0·3317 (5) | 2·9 (2) |
| C7 | 0·6007 (6) | 0·5279 (6) | 0·3563 (5) | 2·6 (2) |
| C8 | 0·5564 (6) | 0·4623 (6) | 0·2982 (4) | 2·5 (2) |
| C9 | 0·6031 (8) | 0·4355 (7) | 0·2341 (4) | 3·5 (2) |
| C10 | 0·5574 (8) | 0·3766 (8) | 0·1831 (5) | 4·4 (2) |
| C11 | 0·4655 (8) | 0·3429 (8) | 0·1969 (6) | 4·5 (2) |
| C12 | 0·4193 (8) | 0·3697 (8) | 0·2609 (7) | 5·0 (3) |
| C13 | 0·4620 (7) | 0·4276 (7) | 0·3115 (6) | 3·7 (2) |
| C14 | 0·6091 (6) | 0·4828 (6) | 0·4335 (4) | 2·5 (2) |
| C15 | 0·7912 (6) | 0·4859 (7) | 0·4345 (5) | 3·2 (2) |
| C16 | 0·7978 (7) | 0·5417 (9) | 0·5065 (5) | 4·6 (2) |
| C17 | 0·7611 (7) | 0·4744 (8) | 0·5649 (6) | 4·7 (3) |
| C18 | 0·7061 (7) | 0·3970 (8) | 0·5276 (5) | 3·5 (2) |
| C19 | 0·7016 (6) | 0·3382 (7) | 0·3988 (5) | 3·2 (2) |

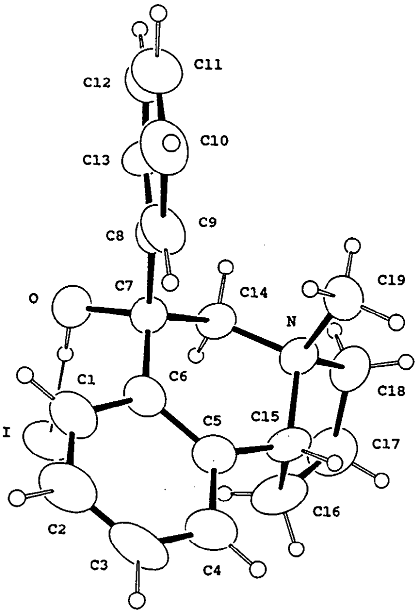

Fig. 1. *ORTEP* (Johnson, 1976) drawing of the title compound with crystallographic numbering scheme.

The final fractional coordinates are given in Table 1* and bond distances and angles for non-H atoms are in Table 2. The molecular structure with crystallographic numbering scheme is depicted in Fig. 1.

Table 2. *Bond distances* (Å) *and angles* (°) *with e.s.d.'s in parentheses*

| | | | |
|---|---|---|---|
| O—C7 | 1·44 (1) | C6—C7 | 1·51 (1) |
| N—C14 | 1·51 (1) | C7—C8 | 1·52 (1) |
| N—C15 | 1·52 (1) | C7—C14 | 1·54 (1) |
| N—C18 | 1·53 (1) | C8—C9 | 1·38 (1) |
| N—C19 | 1·49 (1) | C8—C13 | 1·40 (1) |
| C1—C2 | 1·36 (1) | C9—C10 | 1·39 (1) |
| C1—C6 | 1·38 (1) | C10—C11 | 1·37 (2) |
| C2—C3 | 1·38 (2) | C11—C12 | 1·37 (2) |
| C3—C4 | 1·38 (2) | C12—C13 | 1·36 (2) |
| C4—C5 | 1·40 (1) | C15—C16 | 1·52 (1) |
| C5—C6 | 1·41 (1) | C16—C17 | 1·50 (2) |
| C5—C15 | 1·49 (1) | C17—C18 | 1·48 (1) |

| | | | |
|---|---|---|---|
| C14—N—C15 | 110·7 (7) | C6—C7—C8 | 113·0 (7) |
| C14—N—C18 | 108·9 (6) | C6—C7—C14 | 111·8 (7) |
| C14—N—C19 | 111·3 (6) | C8—C7—C14 | 113·8 (7) |
| C15—N—C18 | 103·7 (6) | C7—C8—C9 | 124·1 (8) |
| C15—N—C19 | 111·9 (6) | C7—C8—C13 | 117·4 (7) |
| C18—N—C19 | 110·0 (7) | C9—C8—C13 | 118·5 (8) |
| C2—C1—C6 | 123·2 (9) | C8—C9—C10 | 120·8 (9) |
| C1—C2—C3 | 118·3 (10) | C9—C10—C11 | 120·1 (9) |
| C2—C3—C4 | 121·6 (11) | C10—C11—C12 | 118·9 (9) |
| C3—C4—C5 | 119·4 (9) | C11—C12—C13 | 122·3 (10) |
| C4—C5—C6 | 119·2 (8) | C8—C13—C12 | 119·4 (9) |
| C4—C5—C15 | 116·5 (8) | N—C14—C7 | 114·9 (6) |
| C6—C5—C15 | 124·2 (7) | N—C15—C5 | 112·9 (7) |
| C1—C6—C5 | 118·3 (8) | N—C15—C16 | 102·7 (7) |
| C1—C6—C7 | 121·2 (8) | C5—C15—C16 | 113·4 (8) |
| C5—C6—C7 | 120·4 (8) | C15—C16—C17 | 104·8 (9) |
| O—C7—C6 | 109·3 (7) | C16—C17—C18 | 108·2 (8) |
| O—C7—C8 | 103·7 (6) | N—C18—C17 | 105·9 (8) |
| O—C7—C14 | 104·4 (6) | | |

**Related literature.** Structures of related hexahydropyrrolo[2,1-*a*]isoquinolines have been reported by Maryanoff, McComsey, Inners, Mutter, Wooden, Mayo & Olofson (1989) and Maryanoff, McComsey, Gardocki, Shank, Costanzo, Nortey, Schneider & Setler (1987).

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* Lists of structure amplitudes, anisotropic temperature factors, H-atom parameters and torsion angles have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 52505 (25 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.