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Structure and Absolute Configuration of Bulbocapnine Methiodide, $C_{20}H_{22}NO_4^+.I^-$*

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Abstract. $M_r = 467\cdot307$, monoclinic, $P2_1$, $a = 10\cdot446\,(1)$, $b = 8\cdot711\,(1)$, $c = 10\cdot317\,(1)$ Å, $\beta = 97\cdot778\,(9)^\circ$, $V = 930\cdot2$ Å^3, $Z = 2$, $D_m = 1\cdot66\,(2)$, $D_x = 1\cdot668$ g cm^{-3}, $\lambda(\text{Mo }K\alpha_1) = 0\cdot7093$ Å, $\mu = 82\cdot12$ cm^{-1}, $F(000) = 468$, $T = 296\,(1)$ K. Final $R(F) = 0\cdot028$ for 2823 counter data. All H atoms were located and their parameters refined. Of the five fused rings, two six-membered aromatic rings form a biphenyl system with an angle of 30·2 (1)° between their plane normals. Bond distances and angles for non-H atoms are all within the ranges of expected values with individual e.s.d.'s in the range 0·004–0·007 Å for distances and 0·2–0·4° for angles. The H atom of the hydroxyl group is hydrogen-bonded to the I atom. The correct absolute configuration of the molecule was ascertained by a comparison of $R_w(F^2)$ values for the determined structure and its inverted structure, and further verified by a refinement in which the imaginary anomalous-scattering term for I^- shifted from a starting value of 0 toward a positive value.

Introduction. Bulbocapnine ($C_{19}H_{19}NO_4$) is an aporphine alkaloid extracted from roots of *Corydalis cava,* and possesses some pharmaceutical values (for Ménière's disease and other muscular tremors) in humans (for review of chemistry, see Shamma & Slusarchyk, 1964; Shamma, 1967). The crystal structure of its methyl iodide salt has previously been determined from film data and refined to an $R(F)$ value of 0·125 by Ashida, Pepinsky & Okaya (1963). Although the absolute configuration of the molecule has been established, detailed molecular parameters including atomic coordinates have not so far appeared in the literature. The data of the previous study presumably had precision substantially lower than what can now be obtained using a diffractometer. In order that a more reliable comparison might be made with two other aporphine alkaloids, hydrobromides of leucoxine and isoboldine, the structures of which have been reported by Brown & Hall (1977), the present study was undertaken.

Experimental. Title compound prepared by treating bulbocapnine hydrochloride (Merck & Co., Inc.) with *N,N*-diisopropylethylamine, followed by reaction with methyl iodide. Colorless crystals grown at room temperature from an aqueous solution. Crystal density determined by flotation in mixtures of bromobenzene and bromoform. A crystal with approximate dimensions 0·65 × 0·26 × 0·25 mm mounted on a Picker four-circle diffractometer with **b** approximately parallel to the φ axis of the diffractometer; lattice parameters refined by least-squares method from angle measurements of 18 strong reflections in 2θ range 48–50° (Busing, Ellison, Levy, King & Roseberry, 1968); systematic absences $0k0$, $k = 2n + 1$, consistent with $P2_1$. Intensity data collected by θ–2θ step scans in 2θ range 1–60° ($-14 \le h \le 14$, $0 \le k \le 12$, $0 \le l \le 14$), divided into three separate subgroups based on 2θ. Within any subgroup variation for two standard reflections (600 and 106) <0·8%. Of the 2871 unique nonzero reflections, 48 reflections with $F_o^2 < \sigma(F_o^2)$ excluded from final least-squares refinement. Absorption corrections calculated analytically by the method of Busing & Levy (1957); transmission range 0·641 to 0·679. Isotropic extinction corrections also applied. Max. mosaic spread of crystal used estimated to be 0·6°.

Structure solved by heavy-atom method. By iterative least-squares refinements and difference-Fourier syntheses [with the program *ORFFP*3 (Levy, 1977)] all 22 H atoms eventually located. Preliminary refinement for 26 non-H (anisotropic) and 22 H atoms (isotropic) carried out using block-diagonal least-squares program of Shiono (1971). Final refinement on F^2 carried out with the full-matrix program *ORXFLS*4 [updated version of *ORFLS* (Busing, Martin & Levy, 1962)] in which slack constraints were applied to one methylene group [involving C(17)] by assigning C—H distances of 1·0 Å and O—C—H and H—C—H angles of 109·5°, with assigned e.s.d.'s of 0·07–0·09 Å for the former and 4–6° for the latter (for similar examples, see Wei,

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Table 1. *Positional and isotropic thermal parameters*

The e.s.d.'s are given in parentheses in all tables and in the text. The digits in parentheses correspond to the least-significant digits of the parameters. The equivalent isotropic temperature factors for C, N, O and I atoms were calculated from the corresponding anisotropic thermal parameters and unit-cell parameters by the relation $B_{eq} = \frac{4}{3}(\beta_{11}a^2 + \beta_{22}b^2 + \beta_{33}c^2 + 2\beta_{13}ac\cos\beta)$ (Hamilton, 1959).

	x	y	z	B_{eq} or B(Å^2)
C(1)	−0·0106 (4)	0·3532 (5)	0·4055 (3)	3·46 (8)
C(2)	−0·0997 (3)	0·2852 (4)	0·3078 (4)	2·86 (8)
C(3)	−0·2310 (4)	0·2408 (5)	0·3418 (4)	3·67 (9)
C(4)	−0·3009 (4)	0·1335 (5)	0·2424 (5)	3·76 (9)
C(5)	−0·1644 (3)	0·1975 (4)	0·0725 (3)	2·48 (6)
C(6)	−0·1536 (3)	0·2927 (5)	−0·0493 (3)	3·08 (8)
C(7)	−0·0203 (3)	0·2706 (4)	−0·0857 (3)	2·75 (7)
C(8)	−0·0016 (4)	0·2561 (6)	−0·2151 (3)	3·48 (9)
C(9)	0·1217 (4)	0·2364 (5)	−0·2488 (4)	3·34 (8)
C(10)	0·2262 (3)	0·2245 (4)	−0·1512 (3)	2·42 (6)
C(11)	0·2075 (3)	0·2321 (3)	−0·0180 (3)	2·16 (6)
C(12)	0·0854 (3)	0·2646 (3)	0·0155 (3)	2·11 (5)
C(13)	0·0564 (3)	0·2944 (4)	0·1498 (3)	2·19 (6)
C(14)	0·1400 (3)	0·3634 (4)	0·2479 (3)	2·62 (6)
C(15)	0·1071 (4)	0·3898 (5)	0·3720 (3)	3·17 (8)
C(16)	−0·0678 (3)	0·2592 (3)	0·1844 (3)	2·30 (6)
C(17)	0·3097 (4)	0·4675 (7)	0·3714 (4)	4·54 (13)
C(18)	0·3759 (5)	0·1962 (7)	−0·3044 (4)	4·26 (11)
C(19)	−0·3637 (4)	0·3511 (5)	0·0942 (5)	3·62 (10)
C(20)	−0·3842 (4)	0·0898 (5)	0·0129 (6)	4·28 (12)
N(21)	−0·3029 (3)	0·1948 (3)	0·1050 (3)	2·90 (6)
O(22)	0·2607 (2)	0·4244 (4)	0·2408 (2)	3·42 (6)
O(23)	0·2072 (3)	0·4589 (5)	0·4487 (3)	4·66 (9)
O(24)	0·3515 (2)	0·2050 (4)	−0·1718 (2)	3·00 (6)
O(25)	0·3157 (2)	0·2044 (3)	0·0678 (2)	2·86 (5)
I	0·34881 (2)	0	0·35494 (2)	4·08 (1)
H(1)*	−0·028 (4)	0·368 (6)	0·486 (5)	2·9 (9)
H(3*a*)	−0·271 (5)	0·327 (7)	0·371 (6)	3·6 (11)
H(3*b*)	−0·213 (5)	0·197 (6)	0·425 (5)	3·8 (10)
H(4*a*)	−0·263 (5)	0·040 (7)	0·242 (6)	4·5 (12)
H(4*b*)	−0·403 (5)	0·114 (7)	0·254 (5)	3·9 (10)
H(5)	−0·152 (4)	0·088 (5)	0·046 (4)	2·1 (7)
H(6*a*)	−0·159 (4)	0·409 (6)	−0·026 (5)	3·1 (9)
H(6*b*)	−0·211 (6)	0·253 (6)	−0·113 (5)	3·3 (9)
H(8)	−0·079 (6)	0·276 (7)	−0·279 (6)	4·4 (12)
H(9)	0·135 (4)	0·230 (5)	−0·333 (4)	1·5 (7)
H(17*a*)	0·329 (7)	0·588 (11)	0·362 (6)	11·9 (44)
H(17*b*)	0·374 (5)	0·404 (6)	0·397 (5)	4·3 (12)
H(18*a*)	0·326 (5)	0·121 (6)	−0·348 (5)	3·1 (9)
H(18*b*)	0·466 (8)	0·171 (10)	−0·295 (7)	7·2 (19)
H(18*c*)	0·351 (6)	0·274 (9)	−0·348 (7)	4·9 (14)
H(19*a*)	−0·444 (5)	0·333 (6)	0·121 (5)	3·3 (9)
H(19*b*)	−0·389 (6)	0·369 (7)	−0·010 (6)	4·9 (13)
H(19*c*)	−0·315 (5)	0·431 (7)	0·136 (5)	3·2 (9)
H(20*a*)	−0·356 (5)	−0·021 (9)	0·025 (5)	4·6 (12)
H(20*b*)	−0·391 (6)	0·142 (8)	−0·077 (7)	4·8 (13)
H(20*c*)	−0·479 (5)	0·106 (7)	0·045 (5)	3·8 (10)
H(25)	0·305 (8)	0·180 (11)	0·140 (9)	7·8 (21)

* H atoms are numbered according to the C atoms or O atom to which they are attached.

1982; Wei & Einstein, 1984). Weights (w) used in the refinement were reciprocals of $\sigma^2(F_o^2) + (0{\cdot}04F_o^2)^2$, where $(0{\cdot}04F_o^2)^2$ is an empirical-correction term (Peterson & Levy, 1957). Scattering factors from Cromer & Waber (1974); anomalous-scattering corrections for I^- ($f' = -0{\cdot}726$, $f'' = 1{\cdot}812$) from Cromer (1974). On final cycle Δ/σ for non-H atoms $< 0{\cdot}022$; max. Δ in H parameters $0{\cdot}096\sigma$. Final refinement for the correct absolute configuration resulted in $R(F)$, $R(F^2)$ and $R_w(F^2)$ values of 0·028, 0·041 and 0·0740, respectively, for 2823 reflections used (data-to-variable ratio 8·69). The standard deviation of an observation of unit weight, S, was 1·443. With all 2871 unique nonzero reflections included, $R(F) = 0{\cdot}029$.

The corresponding measures obtained by refinement of the inverted structure are: $R(F) = 0{\cdot}031$; $R(F^2) = 0{\cdot}046$; $R_w(F^2) = 0{\cdot}0812$; $S = 1{\cdot}584$. Applying the R-factor ratio test (Hamilton, 1965), we obtain $\mathscr{R} = 1{\cdot}097$, much greater than the value $\mathscr{R}_{1,2498,0\cdot005} = 1{\cdot}002$ interpolated from the Hamilton tables for the 0·005 significance level for a one-dimensional hypothesis and 2498 degrees of freedom. Hence the probability of the inverted model's being correct can be rejected at the 99·5% confidence level. Thus, the absolute configuration was established. A final difference Fourier map for the correct absolute configuration showed $\Delta\rho$ excursions from −0·39 to 0·56 e Å^{-3} in the neighborhood of I^-. Using parameters obtained for the correct configuration, two cycles of refinement were carried out in which the imaginary component of the anomalous-dispersion correction was set to zero at the beginning. The f'' value shifted significantly to 1·03 (9) after one cycle and 1·35 (8) after the second cycle; $R(F)$, $R(F^2)$ and $R_w(F^2)$ values stood at 0·028, 0·041 and 0·0743, respectively, thus confirming that a positive phase shift indeed occurred in the scattering of I^-. This further substantiates the correctness of the choice of absolute configuration.*

Discussion. Positional parameters and isotropic temperature factors (or their equivalents) are listed in Table 1.†

The crystal structure of this quaternary aporphine salt is composed of *N*-methylbulbocapninium and I^- ions linked together by hydrogen bonds. The methyl group from methyl iodide binds to the N atom, thus imparting the positive charge to it. Since the twisted biphenyl system involving its absolute configuration and the resulting molecular geometry are of prime interest in this group of alkaloids, the molecule is shown in a stereoscopic view in Fig. 1. All figures were prepared with the program *ORTEP*II (Johnson, 1976). As in the cases of hydrobromides of leucoxine and isoboldine (Brown & Hall, 1977), the asymmetric carbon atom C(5) of bulbocapnine methiodide belongs to the *S* series (Cahn, Ingold & Prelog, 1956, 1966). The two aromatic rings consisting of C(1), C(2), C(16), C(13), C(14), C(15) (designated ring *A*) and C(12), C(7), C(8), C(9), C(10), C(11) (designated ring *D*) are connected by the C(13)—C(12) bond, the complete specification of configuration being 12,13a*R*,5*S*, as derived by Brown & Hall (1977).

* If the inverted parameters had been used in the refinement of f'', this would have resulted in a negative phase shift.

† Lists of structure factors, anisotropic thermal parameters, least-squares planes, and a stereoscopic pair showing the [010] projection of the unit cell have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 39560 (10 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 2. *Comparison of some molecular parameters*

	Leucoxinium ion	Isoboldinium ion	This work
Angles (°)			
C(1)···C(13)–C(12)	174·4 (1)	178·2 (4)	177·2 (2)
C(9)···C(12)–C(13)	177·1 (2)	177·5 (4)	173·4 (2)
Angles (°) between vectors			
C(13)→C(1) and C(12)→C(9)	173·7 (1)	176·2 (2)	171·6 (1)
Torsion angles (°)			
C(16)–C(13)–C(12)–C(7)	13·6 (4)	21·2 (8)	28·4 (4)
C(14)–C(13)–C(12)–C(11)	15·0 (5)	20·5 (10)	31·8 (5)
	Av. 14·3 (3)	Av. 20·9 (6)	Av. 30·1 (3)
Angles (°) between plane normals of rings *A* and *D*	15·5	21·1	30·2 (1)

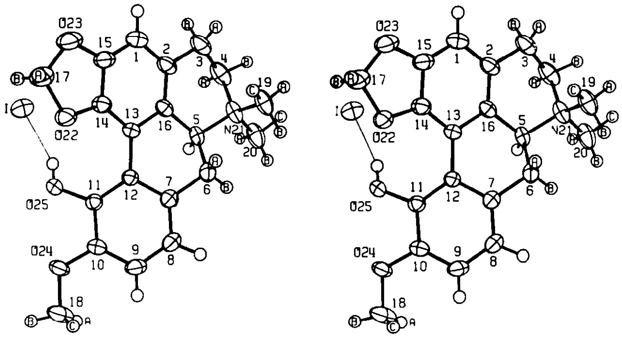

Fig. 1. Stereoscopic view of the bulbocapnine methiodide molecule. Each thermal ellipsoid for a non-H atom encloses 50% probability. The twisting of the biphenyl system around the C(13)–C(12) bond is clearly seen.

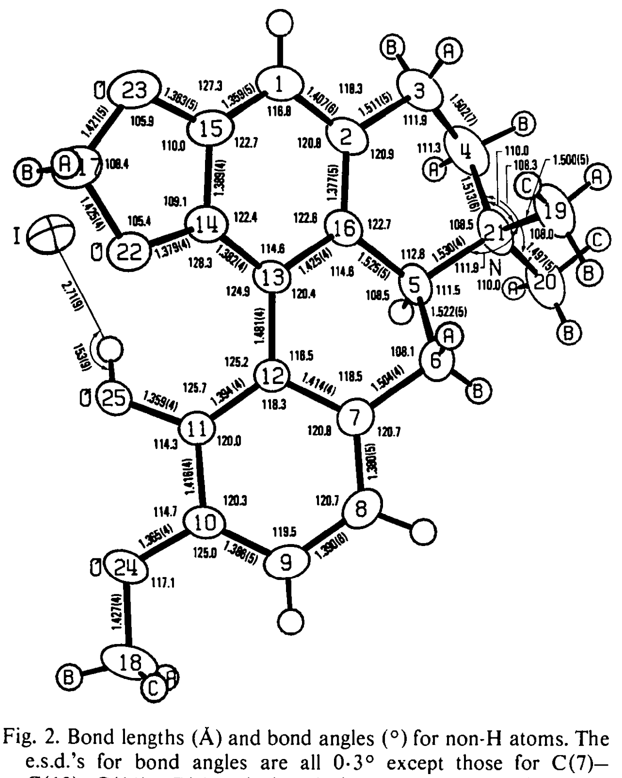

Fig. 2. Bond lengths (Å) and bond angles (°) for non-H atoms. The e.s.d.'s for bond angles are all 0·3° except those for C(7)–C(12)–C(11), C(7)–C(12)–C(13) and C(12)–C(11)–O(25), which are 0·2°, and that for C(3)–C(2)–C(16), which is 0·4°.

Bond lengths and angles and their e.s.d.'s, calculated by the program *ORFFE*4 [updated version of *ORFFE* (Busing, Martin & Levy, 1964)], are given in Fig. 2 which also shows the numbering scheme. All values involving non-H atoms of the fused-ring system appear normal. All 21 C–H distances and one O–H distance range from 0·79 (9) [for O(25)–H(25)] to 1·11 (5) Å [for C(4)–H(4*b*)], with an average of 0·96 Å. The six H–C–C angles involving the three H atoms attached to aromatic rings *A* and *D* are within two e.s.d.'s of 120° (e.s.d.'s are all 3°); the H(25)–O(25)–C(11) angle is 116 (6)°; and the remaining 41 tetrahedral angles for H–C–C, H–C–O, H–C–N and H–C–H are in the range of 94 (5) [for H(3*a*)–C(3)–H(3*b*)] to 122 (3)° [for H(3*a*)–C(3)–C(4)], the average value being 109°.

The C atoms of ring *A* are coplanar to within 0·015 (2) Å, with C(12) located 0·008 (5) Å above the plane (*i.e.* toward the viewer in Figs. 1 and 2), while those C atoms of ring *D* deviate from their best molecular plane by as much as 0·038 (2) Å with C(13) being 0·162 (5) Å above the best molecular plane. The non-collinearity of atoms C(1), C(13), C(12) and C(9) can be seen by the angles of 177·2 (2) and 173·4 (2)°, respectively, for C(1)···C(13)–C(12) and C(9)···C(12)–C(13), and the angle between vectors C(13)→C(1) and C(12)→C(9) is 171·6 (1)°. The bond C(13)–C(12) is twisted in such a way that the two torsion angles C(16)–C(13)–C(12)–C(7) and C(14)–C(13)–C(12)–C(11) (Klyne & Prelog, 1960) are 28·4 (4) and 31·8 (5)° respectively. The angle between the normals of the least-squares planes for rings *A* and *D* is 30·2 (1)°. A comparison of these molecular features with those determined by Brown & Hall (1977) for the analogous leucoxinium and isoboldinium ions is given in Table 2. It can be seen that the twist of rings *A* and *D* around the C(13)–C(12) bond is considerably greater in the present molecule than in the leucoxinium and isoboldinium ions. In addition, O(22) lies 0·126 (6) Å above the plane of ring *A*, while O(25) is 0·169 (5) Å below the plane of ring *D*. All exocyclic angles at C(14), C(13), C(12) and C(11) are significantly larger than 120° (Fig. 2). All these results indicate that the biphenyl system in this molecule is appreciably strained in order to minimize the interaction between O(22) and O(25). Although the O(22)···H(25) and O(22)···O(25) distances are 2·44 (10) and 2·733 (4) Å, the O(22)···H(25)–O(25) angle is only 103 (8)°. Hence, the possibility of hydrogen bonding between O(22) and O(25) is excluded in this case and the short intramolecular contacts quoted above are considered to be due to the geometrical strains imposed on the molecule.

As shown in Figs. 1 and 2, there is a hydrogen bond involving the O(25)–H(25) group as a donor and I^- as an acceptor. The O(25)···I^- separation is 3·434 (3) Å. The closest intermolecular contacts in this structure between non-H atoms involve O(23)···C(3) [3·259 (5) Å] and O(24)···C(19) [3·184 (5) Å]; the closest such contact between non-H and H atoms is 2·45 (6) Å for both O(23)···H(3*b*) and O(24)···H(19*c*).

A packing diagram viewed down **b** is available as supplementary material.

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2,3-Dihydro-1,4-diazepinium Picrate, $C_5H_9N_2^+.C_6H_2N_3O_7^-$

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Abstract. $M_r = 366·8$, monoclinic, $P2_1/c$, $a = 11·042$ (3), $b = 8·224$ (3), $c = 15·113$ (3) Å, $\beta = 92·40$ (2)°, $V = 1371·1$ Å^3, $Z = 4$, $D_x = 1·58$ g cm^{-3}, λ(Mo $K\alpha$) = 0·70926 Å, $\mu = 1·3$ cm^{-1}, $F(000) = 672$, $T = 293$ K, final $R = 0·042$ for 1336 observed data. The crystal structure contains discrete diazepinium cations and picrate anions linked in chains by N—H···O hydrogen bonds [2·898 (3), 2·777 (3) Å]. The cation contains a five-membered delocalized 1,5-diazapentadienium chain [N(4),C(5),C(6),C(7),N(1); mean C—C 1·382 (8), mean C—N 1·306 (9) Å] in a helical conformation with N(1) −0·059 (3) and N(4) 0·062 (3) Å from the five-atom plane; the methylene atoms C(2) and C(3) are −0·399 (3) and +0·444 (3) Å respectively from this plane. The picrate ring plane is planar but the nitro groups are inclined at 37·5 (3) and 25·6 (3)° (*ortho*) and 1·9 (3)° (*para*) to the ring. The picrate dimensions are consistent with significant contributions from a resonance form with an essentially normal C=O bond.

Introduction. The 2,3-dihydro-1,4-diazepinium cation, present in compound (1), is of chemical interest (Lloyd, Cleghorn & Marshall, 1974; Lloyd, 1975; Lloyd & McNab, 1978) because it possesses a delocalized

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