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2-(4-Cyanophenyl)-2-hydroxy-4,4-dimethylmorpholinium Bromide

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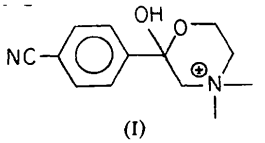
Abstract. $C_{13}H_{17}N_2O_2^+.Br^-$, $M_r = 313{\cdot}20$, monoclinic, $P2_1/c$, $a = 8{\cdot}687$ (1), $b = 12{\cdot}225$ (2), $c = 13{\cdot}419$ (2) Å, $\beta = 103{\cdot}90$ (1)°, $V = 1383{\cdot}3$ (6) Å^3, $Z = 4$, $D_x = 1{\cdot}504$ g cm^{-3}, λ(Mo $K\alpha$) = 0·71073 Å, $\mu = 29{\cdot}4$ cm^{-1}, $F(000) = 640$, $T = 295$ (1) K, $R = 0{\cdot}045$ for 1728 reflections with $I > 2{\cdot}0\sigma(I)$ (2427 unique observations). The hydroxyl group is in the axial position of the six-membered morpholine ring, and is oriented towards the anion by a strong ion–dipole interaction: O···Br = 3·215 (2), H···Br = 2·31 (4) Å, O–H···Br = 165 (4)°.

Experimental. The compound (I) was synthesized as described (Garcia, 1986). A colorless prismatic crystal was mounted in random orientation in a glass capillary for protection against water vapor. Details of data collection and structural refinement are given in Table 1.



(I)

The Br^- ion was located from a Patterson map, and the remaining non-hydrogen atoms were located in successive difference Fourier syntheses. Although some H-atom positions were visible in difference maps, ideal positions were computed for all H atoms and then refined in least squares; their isotropic thermal parameters were held fixed at 1·1 times the B_{eq} of the attached atom. The structure was refined in full-matrix least squares with Enraf–Nonius *SDP* (Frenz, 1978). The final cycle of refinement included 214 variable parameters and converged to $R = 0{\cdot}045$. Atomic scattering factors, including those for anomalous dispersion, were taken from *International Tables for X-ray Crystallography* (1974).

Final positional and equivalent isotropic thermal parameters are given in Table 2, and bond lengths, bond angles and torsion angles are shown in Table 3.*

Fig. 1 shows the molecular cation and the atomic numbering scheme.

Table 1. *Experimental details*

Crystal	Colorless prismatic 0·24 × 0·36 × 0·44 mm
Instrument	Enraf–Nonius CAD-4 diffractometer
Monochromator	Incident-beam, graphite
Unit cell	25 reflections, $22{\cdot}0 < 2\theta < 23{\cdot}8°$
Mode	ω–2θ
Standards	200, 020, 002
R_{int}	0·020
Corrections	Background, Lorentz, polarization. Empirical absorption [0·757–0·996 on (I)]. Linear decay [0·986–1·165 on (I)].
2θ range (°)	3·2–50·0
hkl ranges	$h = 0$ to 10 $k = 0$ to 14 $l = -15$ to 15
Reflections	2810 total 2427 unique 1728 with $I > 2{\cdot}0\sigma(I)$
Solution	Heavy-atom methods
Function minimized	$\sum w(\lvert F_o\rvert - \lvert F_c\rvert)^2$
Weights	$4F_o^2\mathrm{Lp}^2/[S^2(C + R^2B) + (0{\cdot}020F_o^2)^2]$ S = scan rate, C = integrated count, R = scan time/background time, B = background count
Parameters refined	214
R, wR, R(all)	0·045, 0·047, 0·084
Goodness of fit	2·31
Maximum shift/e.s.d.	0·14
$\Delta\rho$ (e Å^{-3})	1·2 (1), –0·6 (1) (near Br^-)

Table 2. *Positional parameters and their e.s.d.'s*

	x	y	z	B_{eq}(Å^2)
Br	0·34610 (6)	0·07699 (5)	0·18867 (4)	3·68 (1)
O1	0·1194 (3)	0·5402 (3)	0·2376 (2)	2·81 (7)
O2	0·0142 (3)	0·4915 (3)	0·3751 (3)	3·18 (8)
N1	0·2956 (4)	0·3588 (3)	0·3502 (3)	2·66 (9)
N2	0·3241 (6)	1·0628 (4)	0·5178 (4)	4·7 (1)
C1	0·2898 (5)	0·4713 (4)	0·3962 (3)	2·4 (1)
C2	0·1439 (5)	0·5378 (4)	0·3457 (3)	2·3 (1)
C3	0·1128 (5)	0·4312 (4)	0·1945 (4)	3·1 (1)
C4	0·2649 (5)	0·3717 (4)	0·2354 (4)	3·0 (1)
C5	0·1801 (6)	0·2794 (4)	0·3796 (4)	3·8 (1)
C6	0·4595 (6)	0·3131 (4)	0·3913 (4)	3·9 (1)
C7	0·1771 (5)	0·6556 (4)	0·3813 (4)	2·4 (1)
C8	0·1326 (5)	0·6928 (4)	0·4684 (4)	3·0 (1)
C9	0·1728 (6)	0·7974 (4)	0·5054 (4)	3·2 (1)
C10	0·2566 (5)	0·8658 (4)	0·4542 (4)	2·6 (1)
C11	0·3017 (5)	0·8288 (4)	0·3671 (4)	3·1 (1)
C12	0·2626 (5)	0·7242 (4)	0·3319 (4)	2·8 (1)
C13	0·2949 (6)	0·9757 (4)	0·4903 (4)	3·4 (1)

The equivalent isotropic thermal parameter, for atoms refined anisotropically, is defined by $\frac{4}{3}[a^2B_{11} + b^2B_{22} + c^2B_{33} + abB_{12}\cos\gamma + acB_{13}\cos\beta + bcB_{23}\cos\alpha]$.

* Lists of structure factors, anisotropic thermal parameters and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 51013 (25 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Table 3. *Bond lengths* (Å), *bond angles* (°) *and torsion angles* (°)

O1–C2	1·415 (4)	C2–C7	1·522 (5)
O1–C3	1·447 (4)	C3–C4	1·493 (5)
O2–C2	1·400 (4)	C7–C8	1·393 (5)
N1–C1	1·513 (4)	C7–C12	1·389 (4)
N1–C4	1·507 (4)	C8–C9	1·386 (5)
N1–C5	1·515 (4)	C9–C10	1·394 (5)
N1–C6	1·504 (5)	C10–C11	1·394 (5)
N2–C13	1·136 (5)	C10–C13	1·439 (5)
C1–C2	1·521 (5)	C11–C12	1·378 (5)
C2–O1–C3	111·8 (3)	O1–C3–C4	110·8 (3)
C1–N1–C4	107·9 (3)	N1–C4–C3	110·5 (3)
C1–N1–C5	112·7 (3)	C2–C7–C8	120·2 (3)
C1–N1–C6	108·0 (3)	C2–C7–C12	120·2 (3)
C4–N1–C5	111·5 (3)	C8–C7–C12	119·4 (3)
C4–N1–C6	109·5 (3)	C7–C8–C9	120·3 (3)
C5–N1–C6	107·2 (3)	C8–C9–C10	119·6 (3)
N1–C1–C2	114·2 (3)	C9–C10–C11	120·3 (3)
O1–C2–O2	111·3 (3)	C9–C10–C13	120·0 (3)
O1–C2–C1	111·8 (3)	C11–C10–C13	119·7 (3)
O1–C2–C7	105·8 (3)	C10–C11–C12	119·5 (3)
O2–C2–C1	107·4 (3)	C7–C12–C11	120·9 (3)
O2–C2–C7	113·5 (3)	N2–C13–C10	179·3 (4)
C1–C2–C7	107·0 (3)		
C3–O1–C2–O2	−65·9 (4)	O2–C2–C7–C8	26·6 (6)
C3–O1–C2–C1	54·2 (5)	O2–C2–C7–C12	−158·4 (4)
C3–O1–C2–C7	170·3 (3)	C1–C2–C7–C8	−91·6 (5)
C2–O1–C3–C4	−61·0 (5)	C1–C2–C7–C12	83·4 (5)
C4–N1–C1–C2	49·7 (5)	O1–C3–C4–N1	61·6 (5)
C5–N1–C1–C2	−73·9 (5)	C2–C7–C8–C9	175·2 (4)
C6–N1–C1–C2	167·9 (4)	C12–C7–C8–C9	0·2 (7)
C1–N1–C4–C3	−54·7 (5)	C2–C7–C12–C11	−176·1 (4)
C5–N1–C4–C3	69·6 (5)	C8–C7–C12–C11	−1·0 (7)
C6–N1–C4–C3	−172·0 (4)	C7–C8–C9–C10	0·8 (7)
N1–C1–C2–O1	−50·3 (5)	C8–C9–C10–C11	−0·9 (7)
N1–C1–C2–O2	72·1 (4)	C8–C9–C10–C13	178·2 (4)
N1–C1–C2–C7	−165·7 (4)	C9–C10–C11–C12	0·1 (7)
O1–C2–C7–C8	149·0 (4)	C13–C10–C11–C12	−179·0 (4)
O1–C2–C7–C12	−36·0 (5)	C10–C11–C12–C7	0·8 (7)

Related literature. Synthesis: Garcia (1986); structure of 4,4-dimethyl-2-oxomorpholinium bromide: Garcia-Guajardo, Fronczek & Gandour (1986); synthesis and pharmacological activity of related compounds: Anderson, Corey, Force, Jensen, Matz & Rivard (1966).

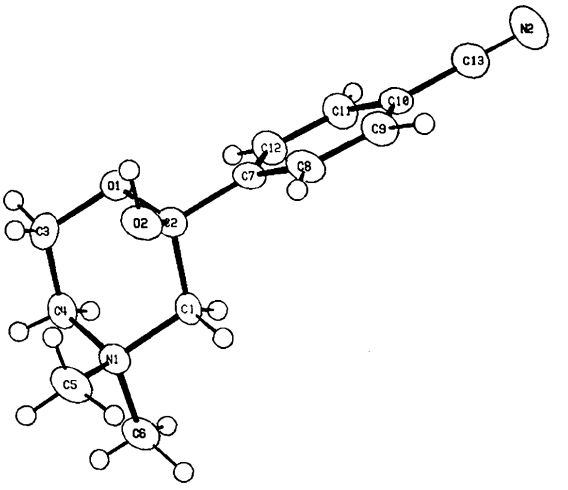

Fig. 1. $[C_{13}H_{17}N_2O_2]^+$, 40% ellipsoids (Johnson, 1965).

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Structure of a Hexahydrobenzo[*a*]quinolizine Derivative

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Abstract. Racemic (2*R*,3*R*,11b*S*)-1,2,3,4,6,7-Hexahydro-9,10-dimethoxy-3-methoxycarbonyl-11b*H*-benzo[*a*]quinolizine-2-carbonitrile, $C_{18}H_{22}N_2O_4$, M_r = 330·38, triclinic, $P\bar{1}$, a = 8·459 (2), b = 9·671 (2), c = 11·017 (2) Å, α = 106·64 (3), β = 99·86 (3), γ = 98·90 (2)°, V = 830·72 Å^3, Z = 2, D_x = 1·32 Mg m^{-3},

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