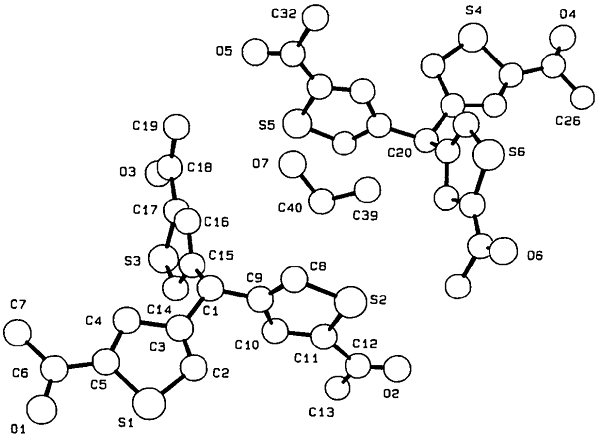

Fig. 1. Perspective drawing showing the numbering scheme.

drawn by *ORTEP* (Johnson, 1965).* The clathrate crystallizes in the common TATM–solvent ratio of 2/1.

Related literature. Related clathrates are inclusion compounds of TATM with ethyl acetate (van Rooyen & Roos, 1991*a*), benzene (van Rooyen & Roos, 1991*b*) and hexane (Roos & Dillen, 1992).

The authors wish to thank Dr Meth-Cohn for the title compound.

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* Lists of structure factors, bond lengths and angles involving H atoms, anisotropic thermal parameters and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 55295 (36 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: GE0310]

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Acta Cryst. (1992). **C48**, 2231–2233

2-Hydroxy-4,4-dimethyl-2-(4-tolyl)-morpholinium Bromide

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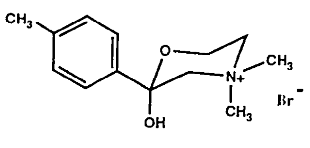
Abstract. $C_{13}H_{20}NO_2^+.Br^-$, $M_r = 302.2$, monoclinic, $P2_1/c$, $a = 8.697$ (2), $b = 12.741$ (3), $c = 12.940$ (2) Å, $\beta = 103.39$ (2)°, $V = 1394.9$ (8) Å^3, $Z = 4$, $D_x = 1.439$ g cm^{-3}, λ(Mo $K\alpha$) = 0.71073 Å, $\mu = 29.1$ cm^{-1}, $F(000) = 624$, $T = 296$ K, $R = 0.038$ for 1662 observations with $I > 2\sigma(I)$ (of 2460 unique data). The morpholinium ring adopts the chair conformation with endocyclic torsion angle magnitudes 49.1 (4)–61.9 (4)°. The hydroxyl group is in the axial position of the morpholinium ring, with C—OH bond distance 1.401 (4) Å. The hydroxy H atom points towards a Br ion; the interaction has O···Br distance 3.292 (2) Å, H···Br distance 2.61 (3) Å, and angle at H 160 (4)°.

Experimental. The title compound (I) was prepared as described by Garcia (1986). Crystals were recrystallized from methanol and had m.p. 492–493 K. A clear colorless crystal with dimensions 0.28 × 0.32 × 0.36 mm was used for data collection on an Enraf–Nonius CAD-4 diffractometer with Mo $K\alpha$ radiation and a graphite monochromator. Cell dimensions were determined from setting angles of 25 reflections



(I)

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Table 1. *Coordinates and equivalent isotropic thermal parameters* (Å²)

$$B_{eq} = (8\pi^2/3)\textstyle\sum_i\sum_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	B_{eq}
Br	0.34596 (4)	0.07420 (3)	0.19773 (3)	4.031 (8)
O1	0.1290 (3)	0.5187 (2)	0.2245 (2)	3.41 (6)
O2	0.0151 (3)	0.4782 (2)	0.3657 (2)	4.01 (6)
N	0.3085 (3)	0.3549 (2)	0.3554 (2)	3.06 (7)
C1	0.2923 (4)	0.4655 (3)	0.3957 (3)	2.70 (8)
C2	0.1446 (4)	0.5244 (3)	0.3360 (3)	3.07 (8)
C3	0.1305 (4)	0.4132 (3)	0.1885 (3)	4.13 (9)
C4	0.2845 (4)	0.3599 (3)	0.2375 (3)	3.60 (9)
C5	0.1980 (4)	0.2786 (3)	0.3890 (3)	4.3 (1)
C6	0.4740 (5)	0.3175 (3)	0.4025 (3)	4.5 (1)
C7	0.1691 (4)	0.6386 (3)	0.3678 (3)	2.94 (8)
C8	0.1257 (4)	0.6751 (3)	0.4574 (3)	3.45 (9)
C9	0.1610 (4)	0.7766 (3)	0.4923 (3)	3.70 (9)
C10	0.2400 (4)	0.8443 (3)	0.4389 (3)	3.52 (9)
C11	0.2824 (4)	0.8081 (3)	0.3497 (3)	3.83 (9)
C12	0.2491 (4)	0.7057 (3)	0.3140 (3)	3.35 (9)
C13	0.2766 (5)	0.9557 (3)	0.4752 (4)	5.5 (1)

Table 2. *Bond distances* (Å), *bond angles* (°) *and selected torsion angles* (°)

C2—O1	1.420 (4)	C7—C8	1.380 (5)
C2—O2	1.401 (4)	C7—C12	1.387 (5)
C1—C2	1.533 (4)	C8—C9	1.380 (5)
C2—C7	1.514 (5)	C9—C10	1.383 (6)
C1—N	1.520 (4)	C10—C11	1.371 (6)
C4—N	1.492 (5)	C11—C12	1.392 (5)
C3—C4	1.504 (5)	N—C5	1.500 (5)
C3—O1	1.424 (5)	N—C6	1.504 (4)
C2—O1—C3	112.0 (3)	C1—N—C4	108.0 (3)
O1—C2—O2	111.3 (3)	C4—N—C5	112.1 (3)
O1—C2—C1	111.0 (3)	C4—N—C6	108.8 (3)
O1—C2—C7	107.5 (3)	O2—C2—C1	106.8 (3)
O2—C2—C7	113.7 (3)	C1—C2—C7	106.6 (3)
C2—C1—N	114.5 (3)	O1—C3—C4	111.3 (3)
C3—C4—N	110.5 (3)	C1—N—C5	112.6 (3)
C1—N—C6	108.1 (3)		
O1—C2—C1—N	−49.6 (4)	O1—C3—C4—N	61.9 (4)
O2—C2—C7—C8	30.2 (4)	O2—C2—C1—N	71.9 (3)

having $12 > \theta > 10°$. The ω–2θ scans were designed for $I = 50\sigma(I)$, subject to maximum scan time of 60 s, with scan rates varied from 1.5 to 4.0° min^{-1}. A quadrant of data having $1 < \theta < 25°$, $0 \le h \le 10$, $0 \le k \le 15$, $-15 \le l \le 15$ was measured and corrected for background, Lorentz and polarization effects. ψ scans of four reflections exhibited no decrease in intensity with rotation about the diffraction vector, thus no absorption correction was applied. Three standard reflections (200, 020, 002) exhibited only random fluctuations in intensity, and no decay correction was made. A total of 2754 data was measured. Equivalent data were averaged, $R_{int} = 0.022$, yielding 2460 unique data. Systematic absences $h0l$ with l odd and $0k0$ with k odd indicated space group $P2_1/c$. The structure is isomorphous to the *p*-cyanophenyl analog (Altbach *et al.*, 1988), and coordinates from that structure were used as a beginning refinement model. Refinement was carried out by full-matrix least squares based on F, using 1662 data for which $I > 2\sigma(I)$ and weights $w = 4F_o^2[\sigma^2(I) + (0.02F_o^2)^2]^{-1}$, the *Enraf–Nonius Structure Determination Package* (Frenz & Okaya, 1980), scattering factors of Cromer & Waber (1974), and anomalous coefficients of Cromer (1974). Heavy-atom coordinates were refined with anisotropic thermal parameters; H-atom coordinates were located by ΔF and were refined with isotropic thermal parameters. Final $R = 0.038$ for 1662 observed data (0.081 for all 2460 data), $wR = 0.037$ and $S = 1.467$ for 234 variables. Maximum shift was 0.08σ in the final cycle, maximum residual density was 0.49, minimum 0.19 e Å^{-3}.

The fractional coordinates of the title compound are given in Table 1. Fig. 1 is a perspective drawing showing the atom numbering. Fig. 2 illustrates the unit cell. Bond distances, bond angles and selected torsion angles are presented in Table 2.*

Related literature. The bond lengths C2—O1 1.420 (4), C2—O2 1.401 (4) and C2—C7 1.514 (5) Å, and bond angles C2—O1—C3 112.0 (3), O1—

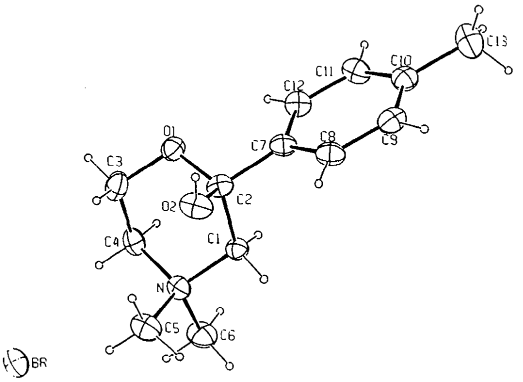

Fig. 1. *ORTEP* drawing (Johnson, 1965) of the asymmetric unit, representing heavy atoms as 30% probability ellipsoids and H atoms as circles of arbitrary radii.

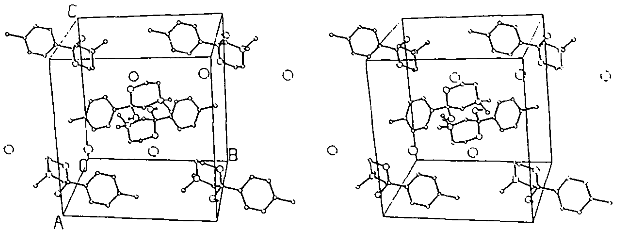

Fig. 2. Stereoview of the unit cell.

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* Lists of H-atom coordinates and thermal parameters, anisotropic thermal parameters, torsion angles, deviations from least-squares planes, and structure-factor amplitudes have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 55346 (19 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: HH0601]

C2—O2 111.3 (3) and O2—C2—C7 113.7 (3)°, of 2-hydroxy-4,4-dimethyl-2-(4-tolyl)morpholinium bromide are similar to those of 2-(4-cyanophenyl)-2-hydroxy-4,4-dimethylmorpholinium bromide [C2—O1 1.425 (5), C2—O2 1.391 (6), C2—C7 1.516 (6) Å; C2—O1—C3 112.3 (3), O1—C2—O2 111.7 (3), O2—C2—C7 114.0 (4)° (Altbach *et al.*, 1988)]. Bond lengths and bond angles are also in agreement with those of 4,4-dimethyl-2-oxomorpholinium bromide (Garcia-Guajardo, Fronczek & Gandour, 1986). The torsion angle O1—C2—C1—N −49.6 (4)° of the title molecule is in agreement with that of 2-(4-cyanophenyl)-2-hydroxy-4,4-dimethylmorpholinium bromide [−50.3 (5)° (Altbach *et al.*, 1988)]. Pharmacological activity of related compounds is reported by Anderson *et al.* (1966) and Lee *et al.* (1992).

We thank the NIH for support of this work through Grant # GM42016.

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Structure of 1,4-Bis(dibenzylamino)benzene

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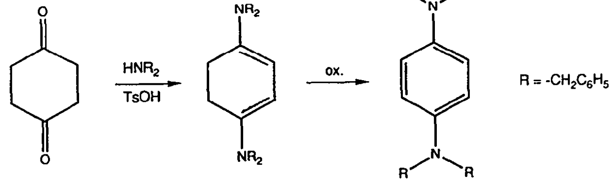
Abstract. *N*,*N*,*N*′,*N*′-Tetrabenzyl-*p*-phenylenediamine, $C_{34}H_{32}N_2$, $M_r = 468.68$, monoclinic, $P2_1/n$, $a = 14.766\,(3)$, $b = 5.604\,(2)$, $c = 15.897\,(3)$ Å, $\beta = 97.34\,(2)°$, $V = 1304.6\,(6)$ Å^3, $Z = 2$, $D_x = 1.19$ Mg m^{-3}, $\lambda(\text{Mo }K\alpha) = 0.71069$ Å, $\mu = 0.064$ mm^{-1}, $F(000) = 500$, room temperature, $R = 0.040$ for 1372 unique reflections having $|F| > 4\sigma(|F|)$. The N atoms of the title compound show sp^2 hybridization. Conjugation with the aromatic ring system is not complete. The torsion angle C(11)—N(1)—C(2)—C(3) is 156.78°.

Experimental. Condensation of 1,4-cyclohexanedione with two equivalents of dibenzylamine in the presence of a catalytic amount of toluene-4-sulfonic acid gave 1,4-bis(dibenzylamino)-1,3-cyclohexadiene (1). 1,4-Bis(dibenzylamino)benzene (2) was prepared by oxidation of compound (1). This system was selected as a model for the preparation of substituted *p*-phenylenediamine derivatives. The crystals of (2) were grown in acetone. A yellowish crystal of about 0.2 × 0.2 × 0.1 mm, cut from a needle, was chosen for structure analysis. Determination of structure and intensity measurements were carried out at room temperature with a Syntex $P2_1$ automated single-crystal diffractometer using graphite-monochromated Mo $K\alpha$ radiation.



(1) (2)