difference map 0·27 and −0·46 e Å^{-3}. Atomic scattering factors from *International Tables for X-ray Crystallography* (1983). Local unpublished programs on NEC PC-9801 personal computers for calculations.

Discussion. The atomic parameters are given in Table 1.* Bond distances and angles in Table 2. The molecular structure with atom-numbering scheme is shown in Fig. 1. A stereoscopic view of the packing in Fig. 2.

Without the fused five- or six-membered ring found in the penicillin or cephalosporin derivatives, the β-lactam ring in the title compound has a different conformation, though the general features are similar to those of penicillins or cephalosporins; the bond lengths of the ring decrease from 1·550 (2) to 1·366 (2) Å in the order C(3)—C(4), C(2)—C(3), N(1)—C(4), N(1)—C(2). The lengths are in good agreement with those of penicillin derivatives and cephalosporins.

Differences from penicillins and cephalosporins are observed in the angles around C(4) and N(1). The angle S(6)—C(4)—N(1) = 117·9 (1)° is significantly greater than the corresponding averaged angle 104·5 (16)° from 21 penicillins or 110·5° from two cephalosporins with *R* factors less than 10% obtained through a search of the Cambridge Structural Database (Allen, Bellard, Brice, Cartwright, Doubleday, Higgs, Hummelink, Hummelink-Peters, Kennard, Motherwell, Rodgers & Watson, 1979). The angle C(4)—N(1)—H(1) = 132 (1)° is also greater than the averaged angle 117 (1)° of the penicillins and 127° of the cephalosporins.

Unlike the atoms in the β-lactam rings of the penicillins or the cephalosporins, the atoms in the isolated β-lactam ring lie within only 0·006 Å from a mean plane through the ring atoms, which is as planar as the phenyl rings in the compound, whose maximum atom deviation is 0·009$_5$ Å. The mean-plane displacements of the β-lactam ring atoms of the penicillins and the cephalosporins are greater by a factor of ten.

* Lists of structure factors, anisotropic temperature factors, H-atom parameters, geometrical data concerning H atoms and best planes have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 52035 (35 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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The Structure of 2,2′-[1,2-Ethanediylbis(oxy)]bis(benzenemethanol)

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Abstract. $C_{16}H_{18}O_4$, $M_r = 274{\cdot}31$, orthorhombic, $a = 13{\cdot}760\ (9)$, $b = 11{\cdot}732\ (7)$, $c = 9{\cdot}084\ (5)$ Å, $U = 1466{\cdot}4\ (15)$ Å^3, $D_x = 1{\cdot}243$ g cm^{-3}, $Z = 4$, space group *Pbcn* (D_{2h}^{14}) [from systematic absences $0kl$, $k = 2n + 1$; $h0l$, $l = 2n + 1$; $hk0$, $h + k = 2n + 1$], Mo $K\alpha$ radiation ($\bar{\lambda} = 0{\cdot}71069$ Å), μ(Mo $K\alpha$) = 0·83 cm^{-1}, $F(000) = 584$, final $R = 0{\cdot}0400$ for 897 observed reflections, 91 parameters. Molecules possess C_2 symmetry and are hydrogen bonded in pairs of infinite chains through the alcoholic functions.

Introduction. One strategy for the synthesis of reduced oxaazamacrocycles involves the generation of

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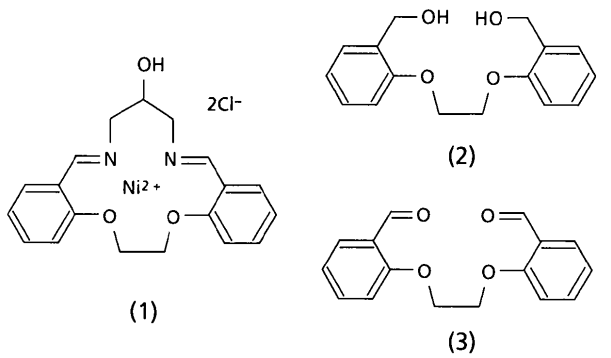
the precursor Schiff-base macrocycle as a metal complex *via* a metal-template cyclocondensation reaction followed by the reduction of the imine bonds to amines (Fenton, Murphy, Leong, Lindoy, Bashall & McPartlin, 1987). This may or may not be accompanied by demetallation depending on the strength of the metal–macrocycle interaction.

Experimental. During the attempted reduction of the nickel(II) Schiff-base macrocyclic complex (1) using $NaBH_4$ in methanol a crystalline organic compound was recovered together with a small amount of the Ni–amine complex (Winter, 1987). This compound was shown by X-ray crystal structure determination to be the title dialcohol (2). This product arises from hydrolysis of (1) under the conditions of the reduction to give the dialdehyde (3) which is then reduced to the dialcohol. No evidence was found for the anticipated reduced macrocycle or its nickel complex.



The crystals used for this study were obtained by leaving the reaction solution described to stand for three days. A crystal having dimensions 0·35 × 0·40 × 0·55 mm was used to collect X-ray data at room temperature in the range $3·5 < 2\theta < 50°$ on a Nicolet $R3$ diffractometer by the ω-scan method ($h\,0 \rightarrow 16$, $k\,0 \rightarrow 13$, $l\,0 \rightarrow 10$). Of the 1516 reflections measured, the 897 independent reflections for which $|F|/\sigma(|F|) > 6·0$ were corrected for Lorentz and polarization effects but not for absorption or secondary extinction. The structure was solved by direct methods. H atoms were detected and placed in calculated positions and refined in riding mode with isotropic thermal vibrational parameters related to those of the supporting atom: the alcoholic H atom was found to be disordered between two sites with necessarily equal populations. These hydrogens were constrained with, initially, O—H 0·92 Å, C—O—H 112° and refined in riding mode. Refinement on F by blocked-cascade least-squares methods converged to a final R of 0·0400 for 91 parameters, with allowance for thermal anisotropy of all non-hydrogen atoms. Scattering factors were taken from the *SHELXTL* program package (Sheldrick, 1983), which was used for the final refinement on a Data General Nova 3 computer; other programs form part of the Sheffield X-ray system. Unit weights were used throughout the refinement, and produced satisfactory convergence. The maximum value of Δ/σ in the final cycle was 0·004 (mean value 0·001). The final difference electron density function showed maxima and minima of +0·14 and −0·18 e Å^{-3}. Atomic positional parameters with e.s.d's are listed in Table 1.*

Table 1. *Atom coordinates* ($\times 10^4$) *and equivalent isotropic temperature factors* (Å$^2 \times 10^3$)

Equivalent isotropic U defined as one third of the trace of the orthogonalized U_{ij} tensor.

	x	y	z	U_{eq}
O(1)	4784 (2)	4187 (1)	1033 (2)	56 (1)
O(2)	4605 (1)	1140 (1)	−1098 (2)	48 (1)
C(1)	4826 (2)	3102 (2)	341 (3)	52 (1)
C(2)	3921 (2)	2407 (2)	582 (3)	41 (1)
C(3)	3185 (2)	2718 (3)	1534 (3)	57 (1)
C(4)	2364 (2)	2050 (3)	1691 (4)	70 (1)
C(5)	2276 (2)	1061 (3)	891 (4)	64 (1)
C(6)	3005 (2)	718 (2)	−80 (3)	50 (1)
C(7)	3825 (2)	1388 (2)	−212 (3)	39 (1)
C(8)	4545 (2)	183 (2)	−2055 (3)	45 (1)

Discussion. The molecular structure is shown in Fig. 1 and bond lengths and angles are given in Table 2. The molecule possesses crystallographically imposed C_2 symmetry across (0·5, y, −0·25): the torsion angle about the saturated C(8)—C(8^i) bond is 69·2 (5)°, so the molecule is twisted. The phenyl ring is planar (r.m.s deviation 0·005 Å). Bond lengths and angles are normal.

Molecules are hydrogen bonded in pairs of infinite chains parallel to the c axis of the unit cell through the alcohol fragment, the H atom of which is disordered between the two symmetry equivalent sites in each bond. Within one chain, the hydrogen bonds span translationally related molecules with the hydrogen-bonded interaction linking unique fragments related by C_2 symmetry [at (0·5, y, 0·25)]: chains are cross-linked through hydrogen bonds across inversion centres [at (0·5, 0·5, 0) *etc.*], as illustrated in Fig. 2. Thus, all such hydrogen sites must have 50% occupancy. The two distinct O···O contacts are essentially identical in length (2·73 and 2·74 Å) and are mutually inclined at 128°: the hydrogen bonds are each approximately linear.

We thank the SERC for an Award (to DJW) and the SERC and Royal Society for funds towards the purchase of the diffractometer.

* 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 52033 (9 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

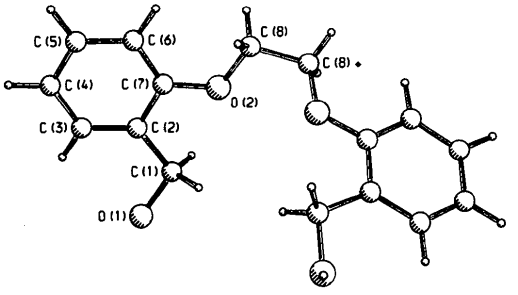


Fig. 1. View of the title compound.

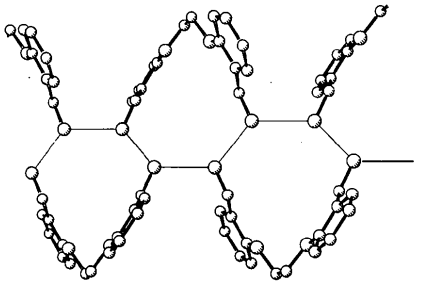


Fig. 2. Hydrogen-bond network across inversion centres.

Table 2. *Bond lengths* (Å) *and angles* (°)

O(1)—C(1)	1·422 (3)	O(2)—C(7)	1·372 (3)
O(2)—C(8)	1·423 (3)	C(1)—C(2)	1·505 (4)
C(2)—C(3)	1·381 (4)	C(2)—C(7)	1·402 (4)
C(3)—C(4)	1·382 (4)	C(4)—C(5)	1·374 (5)
C(5)—C(6)	1·394 (4)	C(6)—C(7)	1·381 (4)
C(8)—C(8^{i})	1·489 (5)	O(1)···O(1^{ii})	2·731 (4)
O(1)···O(1^{iii})	2·741 (4)		

C(7)—O(2)—C(8)	118·7 (2)	O(1)—C(1)—C(2)	112·7 (2)
C(1)—C(2)—C(3)	123·7 (2)	C(1)—C(2)—C(7)	117·7 (2)
C(3)—C(2)—C(7)	118·6 (2)	C(2)—C(3)—C(4)	121·0 (3)
C(3)—C(4)—C(5)	119·7 (3)	C(4)—C(5)—C(6)	121·0 (3)
C(5)—C(6)—C(7)	118·6 (3)	O(2)—C(7)—C(2)	114·1 (2)
O(2)—C(7)—C(6)	124·7 (2)	C(2)—C(7)—C(6)	121·1 (2)
O(2)—C(8)—C(8^{i})	106·5 (2)	O(1^{ii})··O(1)··O(1^{iii})	128·4 (3)

Symmetry operations: (i) $1-x$, y, $-0{\cdot}5-z$; (ii) $1-x$, y, $0{\cdot}5-z$; (iii) $1-x$, $1-y$, $-z$.

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Structural Studies of Mitomycins. II. Structure of Mitomycin A Hemihydrate

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Abstract. $C_{16}H_{20}N_3O_6.\frac{1}{2}H_2O$, $M_r = 359{\cdot}36$, monoclinic, $P2_1$, $a = 17{\cdot}893$ (2), $b = 10{\cdot}358$ (3), $c = 9{\cdot}029$ (1) Å, $\beta = 95{\cdot}16$ (1)°, $V = 1666{\cdot}6$ (5) Å^3, $Z = 4$, $D_x = 1{\cdot}40$ g cm^{-3}, Cu $K\alpha$, $\lambda = 1{\cdot}54184$ Å, $\mu = 8{\cdot}7$ cm^{-1}, $F(000) = 390$, $T = 293$ K, $wR = 0{\cdot}069$ for 3143 observed reflections with $F > 3\sigma(F)$. The benzoquinone ring deviates significantly from planarity. Its two O atoms are located on opposite sides of the least-squares plane through the benzene ring. Conformational differences between the two crystallographically independent molecules are observed around the substituent groups at C(7), C(9) and C(9*a*).

Introduction. Mitomycins are very effective antitumor antibiotics. To design more effective and less toxic mitomycins it is desirable to investigate the three-dimensional structures of mitomycins in detail. Mitomycin A is a member of the mitomycin family and its structure has been determined by X-ray analyses using heavy-atom derivatives, *i.e. N*-brosylmitomycin A (Tulinsky & van den Hende, 1967) and *N*-(*p*-bromobenzoyl)mitomycin A (I) (Hirayama & Shirahata, 1987). The aziridine ring is believed to play an important role in the antitumor activities of mitomycins. The aziridine rings in both heavy-atom derivatives are modified by large substituents and it is probable that these influence the inherent structure around the ring. To disclose the intrinsic structure of mitomycin A we have undertaken an X-ray analysis of native mitomycin A (II).

Experimental. Deep-violet crystals from chloroform, dimensions $0{\cdot}30 \times 0{\cdot}20 \times 0{\cdot}20$ mm. Enraf–Nonius CAD-4 diffractometer, graphite-monochromated Cu $K\alpha$ radiation. Cell dimensions from setting angles

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