Fig. 1. A *PLUTO* diagram of the title compound showing the atom-numbering scheme.

eters $\Delta C_2(1,2) = 15.5$, $\Delta C_s(1) = 10.6°$ with an average internal absolute torsion angle of 25.6°. The B and C rings adopt normal chair conformations with average endocyclic absolute torsion angles of 52.8 and 54.4°, respectively. The D ring adopts an approximate 14α-envelope conformation with the asymmetry parameters $\Delta C_s(13) = 23.5$ (32.5), $\Delta C_s(14) = 5.2$ (1.4) and $\Delta C_2(13,14) = 12.7°$ (22.7), the values in parentheses being the ideal values for a 14α-envelope conformation (Duax & Norton, 1975). The X-ray structure of the title compound shows that the BC and CD ring junctions are *trans* [H(8)—C(8)—C(9)—H(9) = 167.0 (2) and C(18)—C(13)—C(14)—O(14) = −175.0 (2)°]. The progesterone side-chain orientation is normally restricted with respect to the D ring; the torsion angle C(16)—C(17)—C(20)—O(20) in 84 structures having a 20-one substitution is observed to be between 0 and −41°, *i.e.* synperiplanar to the C(16)—C(17) bond (Duax, Griffin, Rohrer & Weeks, 1980). In this structure, the carbonyl at C(20) is antiperiplanar to the unsaturated C(16)—C(17) bond [C(16)—C(17)—C(20)—O(20) = 167.1 (3)°] as has been observed in five other 16-en-20-one structures (Khazheeva, Simonov, Kamernitskii, Pavlova-Grishina & Skorova, 1982; Duax, Langs, Strong & Osawa, 1979; Surcouf, 1979; Duax, Weeks & Strong, 1979; Dastidar, Joseph, Madyastha & Guru Row, 1991). The molecules are stabilized in the crystal lattice by hydrogen bonding [O(3)$\cdots$O(7) = 2.767, O(3)$\cdots$H(O7) = 1.895 Å, O(3)$\cdots$H(O7)—O(7) = 173.2°].

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Structure of 1,8-Bis(hydroxymethyl)naphthalene

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**Abstract.** 1,8-Naphthalenedimethanol, $C_{12}H_{12}O_2$, M_r = 188.24, monoclinic, $P2_1/n$, $a = 8.549$ (4), $b = 4.856$ (3), $c = 22.604$ (4) Å, $\beta = 94.24$ (3)°, $V = 935.8$ (7) Å^3, $Z = 4$, $D_x = 1.34$ g cm^{-3}, $\lambda(\text{Mo } K\alpha) = 0.71073$ Å, $\mu = 0.84$ cm^{-1}, $F(000) = 400$, $T = 295$ K, $R = 0.042$ for 1455 unique reflections having $I > 3\sigma_I$. In the title structure the exterior angles at ring atoms C(1), C(8) and C(9) deviate significantly from 120° as a result of the steric interactions of the hydroxymethyl substituents. The ten C atoms com-

prising the naphthalene core have an average deviation of 0.012 (7) Å from the best least-squares plane describing the core. The conformation of the title compound is compared to those of other 1,8-disubstituted naphthalenes. Each hydroxy group is involved in hydrogen bonding as both acceptor and donor, creating infinite chains that propagate in the [010] directions. The neutron-adjusted parameters for the two H⋯O(acceptor) distances are 1.78 (3) and 1.80 (3) Å and for the two O—H⋯O(acceptor) angles are 165 (2) and 170 (2)°. The packing arrangement can be described as a modified herringbone structure with the shortest crystallographic axis coinciding with the b axis of the standard monoclinic setting.

Introduction. The molecular conformation and crystal packing of 1,8-disubstituted naphthalenes (*peri* substituted) have received considerable interest from crystallographers. A review by Balasubramaniyan (1966) formulated the basic structural consequences of *peri* substitution, but compounds with significant exceptions to these observations have been studied as recently as 1990 (Cosmo, Hambley & Sternhell, 1990). As part of our investigation of hydrogen bonding in the organic solid state, the structure of 1,8-naphthalenedicarboxylic acid has been reported (Fitzgerald, Gallucci & Gerkin, 1991) and compared to that of other *peri*-substituted naphthalenes. The present study of 1,8-bis(hydroxymethyl)naphthalene (I) permits further investigation into the effects of *peri* substitution as well as the opportunity to observe the consequences of changing the hydrogen-bonding moiety from a carboxylic acid group to a hydroxyl group.

(I)
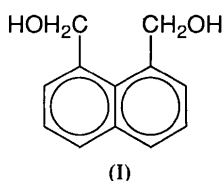
Experimental. 1,8-Bis(hydroxymethyl)naphthalene (1,8-BHMN) (I) was prepared by reduction of naphthalic anhydride (Aldrich Chemical Co.) with lithium aluminium hydride in tetrahydrofuran under reflux followed by quenching with hydrochloric acid (Beyler & Sarrett, 1952). The reaction mixture was subsequently saturated with sodium sulfate decahydrate to effect phase separation; the inorganic phase was extracted with ether and the combined organic solvents evaporated. Recrystallization from acetone afforded white acicular crystals of m.p. 428–429 K. These crystals were then dissolved in ethanol, which was slowly evaporated at 313–318 K to produce the experimental sample, a clear rectangular chunk with

approximate dimensions 0.31 × 0.27 × 0.23 mm. The sample was mounted with epoxy cement on a glass fiber and analyzed at room temperature with a Rigaku AFC-5S diffractometer utilizing monochromated Mo $K\overline{\alpha}$ radiation. Unit-cell parameters were obtained from a symmetry-restricted least-squares fit of the setting angles of 25 centered reflections with 22 $\leq 2\theta \leq 30°$. Intensity data were measured for 2559 reflections (exclusive of standards) with $+h$, $+k$, $\pm l$ indices ($h_{max} = 11$; $k_{max} = 6$; $-29 \leq l \leq 29$) and 2θ values in the range $4 \leq 2\theta \leq 55°$. The ω-scan technique was employed with scan widths $(1.8 + 0.35\tan\theta)°$ in ω and a background/scan time-ratio of 0.5. A variance was assigned to each reflection by means of the formula $\sigma_I^2 = \sigma_{cs}^2(I) + (0.03I)^2$ in which σ_{cs} is based on counting statistics and I is the integrated intensity. Six standard reflections ($0\overline{1}4$, $1\overline{1}3$, $00\overline{4}$, $\overline{3}22$, 602, $\overline{1},\overline{2},12$) were measured after every 150 reflections. Over the course of the intensity data collection they showed, on average, a maximum non-systematic relative intensity variation of ± 1.2%. No correction was made for decay or absorption; the data were corrected for Lorentz and polarization effects. Averaging equivalent reflections gave 2404 independent reflections with $R_{int} = 0.019$.

Among the primitive monoclinic space groups allowed by the data, the observed systematic absences ($0k0$, $k = $ odd; $h0l$, $h + l = $ odd) uniquely determine the space group as $P2_1/n$ (No. 14). The direct-methods program *SHELXS*86 (Sheldrick, 1985) was used to generate an E map from which the initial positions of all non-H atoms were identified. Fourier difference methods were used to locate the positions of the H atoms. Full-matrix least-squares refinement (Molecular Structure Corporation, 1989) was performed to minimize the function $\Sigma \sigma_F^{-2}(|F_o| - |F_c|)^2$ in which $\sigma_F = \sigma_I/2FLp$. Neutral-atom scattering factors and anomalous-dispersion factors were taken from Cromer & Waber (1974) for C and O; the scattering factor for H was taken from Stewart, Davidson & Simpson (1965). The non-H atoms were refined to the anisotropic stage using the data having $I > 3\sigma_I$; the H-atom positions were then identified and subsequently refined isotropically. The results for the final refinement cycle were: 1455 independent observations having $I > 3\sigma_I$; 175 variables; $R = 0.042$; $wR = 0.050$; $w = \sigma_F^{-2}$; $S = 1.79$; $(\Delta/\sigma)_{max} < 0.01$. Maximum and minimum peaks on the final electron-density difference map had values 0.20 and -0.16 e Å^{-3}, respectively. The maximum peak was located at (0.81, 0.61, 0.41), approximately 0.8 Å from C(8) and 0.9 Å from C(7); the minimum peak was located at (0.74, 0.35, 0.45), approximately 1.2 Å from O(2). [For the final refinement cycle for 1585 observations having $I > \sigma_I$: $R = 0.046$, $wR = 0.052$, $S = 1.76$, $(\Delta/\sigma)_{max} < 0.01$. For the final refinement cycle for 1916 observations having $I > 0$: $R = 0.060$,

$wR = 0.054$, $S = 1.64$, $(\Delta/\sigma)_{max} < 0.01$. These latter results are for comparison purposes only.]

The final atomic coordinates and equivalent isotropic and isotropic displacement parameters with their e.s.d.'s are given in Table 1.* Since the asymmetric unit in this structure is an entire 1,8-BHMN molecule, there are no symmetry constraints linking any sets of distances or angles in the molecule.

Discussion. Figs. 1 and 2 show two views of 1,8-BHMN; bond angles and selected interatomic distances are given. Table 2 lists bond lengths. The average C—C bond length for the naphthalene core is 1.395 (33) Å and the average interior angle is 120.0 (18)°, comparable to the respective values for other 1,8-disubstituted naphthalenes as shown in Table 3. The angles around C(1), C(8) and C(9) show deviations typical for *peri*-substituted naphthalenes; angles C(1)—C(9)—C(8), C(9)—C(1)—C(11) and C(9)—C(8)—C(12) are all significantly greater than 120°. However, the angles at C(1), 123.5 (2)°, and C(8), 125.3 (2)°, are considerably different from each other, a situation not typical of *peri*-substituted naphthalenes having identical substituents (Table 3). The asymmetry of these bond angles is a consequence of the arrangement of the hydroxymethyl substituents with respect to the naphthalene ring. The C(11) hydroxymethyl group has its O atom close to the plane of the naphthalene core and its H atoms above and below the plane; the C(12) hydroxymethyl group has one of its H atoms close to the plane of the naphthalene core with the O atom and the other H atom above and below the plane, respectively.

The ten C atoms comprising the naphthalene core of 1,8-BHMN have an average deviation of 0.012 Å from the best least-squares plane describing these atoms. In this respect, the results of the present study are similar to results for 1,8-dimethylnaphthalene and 1,8-dimethoxynaphthalene (Table 3), despite the occurrence of non-bonded interatomic distances within the 1,8-BHMN molecule that are significantly less than the sum of the van der Waals radii, as shown in Figs. 1 and 2. However, for 1,8-dimethylnaphthalene and 1,8-dimethoxynaphthalene, all of the atoms of the respective carbon skeletons are within 0.02 Å of the plane describing the naphthalene core. For 1,8-BHMN, however, C(11) and C(12) are −0.068 and +0.104 Å, respectively, from the core plane. Thus, the degree of planarity for the

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* Lists of structure factors, anisotropic displacement parameters, positional parameters and isotropic displacement parameters of ring and methyl H atoms, and least-squares best-fit planes data have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 55501 (19 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 1. *Final positional parameters and equivalent isotropic and isotropic displacement parameters* (Å²) *for* 1,8-*bis*(*hydroxymethyl*)*naphthalene with e.s.d.'s in parentheses*

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

|	x	y	z	B_{iso}/B_{eq}
O(1)	0.2898 (2)	0.1604 (3)	0.43870 (6)	3.79 (6)
O(2)	0.7582 (2)	0.3385 (3)	0.50861 (5)	3.74 (6)
C(1)	0.4796 (2)	0.4082 (4)	0.38165 (7)	2.85 (7)
C(2)	0.3512 (2)	0.4902 (4)	0.34556 (8)	3.61 (8)
C(3)	0.3610 (3)	0.6877 (4)	0.30089 (9)	4.06 (9)
C(4)	0.5004 (3)	0.8040 (4)	0.29183 (8)	3.88 (9)
C(5)	0.7814 (3)	0.8585 (4)	0.31628 (9)	3.90 (9)
C(6)	0.9162 (3)	0.7915 (5)	0.3482 (1)	4.3 (1)
C(7)	0.9130 (2)	0.5892 (5)	0.39207 (9)	3.82 (9)
C(8)	0.7783 (2)	0.4556 (4)	0.40511 (7)	3.02 (7)
C(9)	0.6315 (2)	0.5245 (3)	0.37273 (7)	2.77 (7)
C(10)	0.6378 (2)	0.7291 (4)	0.32704 (7)	3.20 (7)
C(11)	0.4522 (2)	0.1975 (4)	0.42913 (8)	3.18 (8)
C(12)	0.7999 (2)	0.2368 (4)	0.45213 (8)	3.48 (8)
H(11)	0.260 (3)	0.307 (5)	0.457 (1)	5.7 (6)
H(12)	0.741 (3)	0.184 (5)	0.530 (1)	5.5 (6)
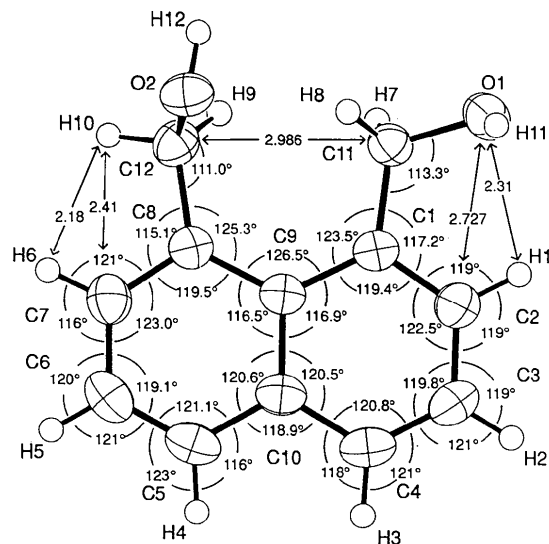
Fig. 1. An *ORTEP* (Johnson, 1976) drawing of 1,8-bis(hydroxymethyl)naphthalene showing the atomic numbering scheme. Displacement ellipsoids have been drawn at 50% probability for all atoms except H, for which they have been set artificially small. Bond angles are shown along with certain interatomic distances. Maximum e.s.d.'s for angles involving C and O are 0.2°, for angles involving H, 2°. Maximum e.s.d.'s for distances involving C and O are 0.003 Å, for distances involving H, 0.03 Å.
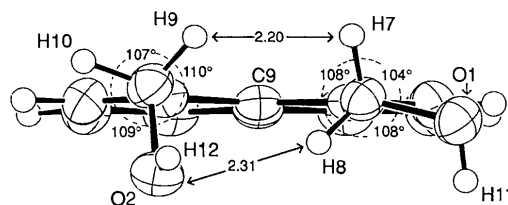

Fig. 2. An *ORTEP* view of the title compound along the C(9)—C(10) bond. Angles around C(11) and C(12) are shown along with certain interatomic distances. E.s.d.'s are described in Fig. 1.

Table 2. *Selected interatomic distances* (Å), C—O—H *angles* (°) *and hydrogen-bond parameters* (Å, °) *for* 1,8-*bis*(*hydroxymethyl*)*naphthalene with e.s.d.'s in parentheses*

C(1)—C(2)	1.378 (3)	C(11)—O(1)	1.432 (2)
C(1)—C(9)	1.444 (3)	C(11)—H(7)	0.98 (2)
C(2)—C(3)	1.400 (3)	C(11)—H(8)	0.99 (2)
C(3)—C(4)	1.348 (3)	C(12)—O(2)	1.438 (2)
C(4)—C(10)	1.417 (3)	C(12)—H(9)	1.04 (2)
C(5)—C(6)	1.353 (3)	C(12)—H(10)	1.00 (2)
C(5)—C(10)	1.416 (3)	O(1)—H(11)	0.87 (3)
C(6)—C(7)	1.398 (3)	O(2)—H(12)	0.91 (2)
C(7)—C(8)	1.373 (3)		
C(8)—C(9)	1.444 (2)	C(11)—O(1)—H(11)	107 (2)
C(9)—C(10)	1.437 (2)		
C(1)—C(11)	1.514 (3)	C(12)—O(2)—H(12)	104 (2)
C(8)—C(12)	1.505 (3)		

D—H···A	D···A	H···A	H···A*	D—H···A	D—H···A*
O(1)—H(11)···O(2')	2.753 (3)	1.90 (3)	1.80 (3)	166 (2)	165 (2)
O(2)—H(12)···O(1")	2.744 (2)	1.84 (3)	1.78 (3)	170 (2)	170 (2)

Symmetry code: (i) $-x + 1$, $-y + 1$, $-z + 1$; (ii) $-x + 1$, $-y$, $-z + 1$.

* Neutron-adjusted hydrogen-bond values from Ceccarelli, Jeffrey & Taylor (1981).

Table 3. *Selected experimental values* (Å, °) *from X-ray studies of five* 1,8-*disubstituted naphthalenes*

The mean values in columns 1, 2 and 3 are derived using naphthalene C atoms and the values in columns 4 and 5 are from single determinations.

	Mean interior angle	Mean ring bond length	Mean deviation from l.s. plane	C(9)—C(1)—R angle	C(9)—C(8)—R angle
This work	120.0 (18)	1.395 (33)	0.012 (7)	123.5 (2)	125.3 (2)
1,8-naphthalene-dicarboxylic acid (a)	119.9 (18)	1.396 (30)	0.055 (30)	123.4 (3)	123.5 (3)
1,8-bis(dimethyl-amino)naphthalene (b)	119.9 (14)	1.396 (36)	0.071 (51)	120.1 (3)	120.8 (3)
1,8-dimethoxy-naphthalene (c)	120.0 (17)	1.399 (32)	0.012 (6)	117.0 (1)*	117.0 (1)*
1,8-dimethylnaph-thalene (d)	120.0 (17)	1.392 (34)	0.006 (4)	124.7 (1)	124.8 (1)

References: (a) Fitzgerald, Gallucci & Gerkin (1991); (b) Einspahr, Robert, Marsh & Roberts (1973); (c) Cosmo, Hambley & Sternhell (1990); (d) Bright, Maxwell & deBoer (1973).

* Values equal by symmetry.

entire molecule in the present case is not as high as in either of the two cases just described.

Each hydroxy group in 1,8-BHMN is involved in hydrogen bonding as both an acceptor and donor. The parameters describing the two inequivalent hydrogen bonds are listed in Table 2. To obtain the neutron-adjusted values, the positions of H(11) and H(12) were adjusted to be 0.97 Å from their respective O atoms, this value being the mean O—H bond length from neutron diffraction studies reviewed by Ceccarelli, Jeffrey & Taylor (1981). The values for the H···O(acceptor) distances, O···O(acceptor) distances and O—H···O(acceptor)

angles are all similar to the corresponding mean values given in this review. The hydrogen-bond arrangement is such that the hydroxy groups are part of infinite chains which are propagated in opposite directions on two sides of the molecule (Fig. 3).

As shown in Fig. 3, the packing arrangement is such that the naphthalene rings are inclined approximately 44° with respect to the *ac* face. Hydrogen-bonded molecules are related by a center of inversion with infinite chains of hydrogen bonds propagating in the [010] directions. When two inversion-related molecules are viewed as one unit, the packing arrangement can be described as a flattened herringbone motif, defined as γ by Desiraju & Gavezzotti (1989). In agreement with their observations, the present crystal system is monoclinic with the shortest crystallographic axis coinciding with the *b* axis of the standard monoclinic setting. While this modified herringbone motif is common for aromatic hydrocarbons, it has not been observed for any of the dicarboxylic acids of naphthalene studied by this group: 1,8-naphthalenedicarboxylic acid (Fitzgerald, Gallucci & Gerkin, 1991), 1,2-naphthalenedicarboxylic acid (Fitzgerald, Gallucci & Gerkin, 1992) and 2,3-naphthalenedicarboxylic acid (Fitzgerald & Gerkin, 1992).
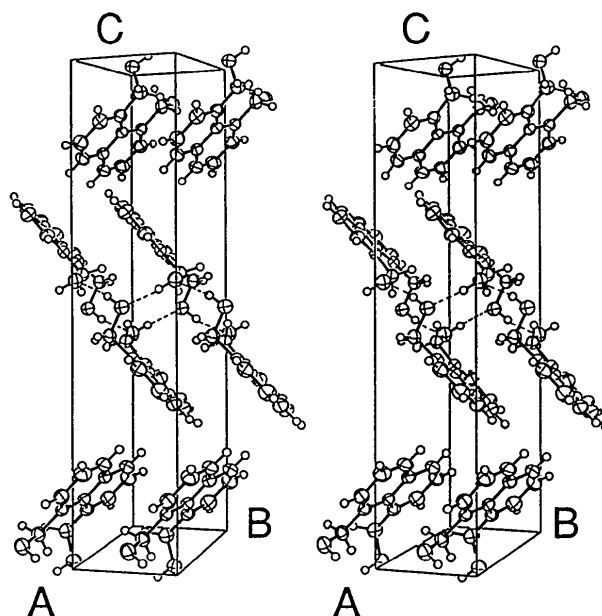
Fig. 3. An *ORTEP* stereoview of 1,8-bis(hydroxymethyl)naphthalene looking down the *a* axis. Four molecules in addition to the unit-cell contents are shown to illustrate packing arrangements.

research through purchase of the diffractometer system by an NIH grant is also gratefully acknowledged.

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Structure of 3-Benzyl-5-(2-hydroxyethyl)-4-methyl-1,3-thiazolium Chloride

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Abstract. $C_{13}H_{16}NOS^+.Cl^-$, $M_r = 269.79$, triclinic, $P\bar{1}$, $a = 6.954$ (1), $b = 9.049$ (1), $c = 11.541$ (2) Å, $\alpha = 73.87$ (1), $\beta = 74.47$ (1), $\gamma = 87.79$ (1)°, $V = 671.7$ (1) Å^3, $Z = 2$, $D_x = 1.334$ g cm^{-3}, $\lambda(\text{Cu } K\alpha) = 1.5418$ Å, $\mu = 38.5$ cm^{-1}, $F(000) = 284$, $T = 295$ K, $R = 0.046$ for 1742 reflections with $I \geq 2\sigma(I)$. The title compound, which is an antagonist of thiamin, assumes a conformation similar to the S form of thiamin. There are close S⋯Cl$^-$ electrostatic interactions [3.197 (1) Å] and a weak C(2)—H⋯Cl$^-$ hydrogen bond [3.421 (3) Å] as frequently observed in thiamin.

Introduction. The title compound (benzylthiamin) is an antagonist of thiamin (vitamin B$_1$) in which the 4-amino-2-methylpyrimidine ring is replaced by a phenyl ring. Benzylthiamin does not show any activity owing to the absence of the pyrimidine moiety which is essential for the activity of thiamin, although its function inside the protein is not clearly understood (Schellenberger, 1982). The crystal structure of N-benzyl-4-methylthiazolium bromide, which is homologous to benzylthiamin but devoid of the 5-(2-hydroxyethyl) side chain, has been determined

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(Power, Pletcher & Sax, 1970). Its conformation is similar to the S form of thiamin which is less frequently observed than the F form. In a continuing study of the conformational characteristics of thiamin analogs, we have determined the crystal structure of benzylthiamin.

Experimental. Transparent plate-shaped crystals of benzylthiamin (Aldrich Chemical Co.) obtained from a CHCl$_3$/toluene (1:1 v/v) solution by slow evaporation; crystal size ca 0.2 × 0.3 × 0.4 mm; Rigaku AFC diffractometer; graphite-monochromated Cu $K\alpha$ radiation; $2\theta \leq 120°$; ω–2θ scan, scan speed 2° min^{-1} in 2θ, ω-scan width $(1.7 + 0.1\tan\theta)°$; background measured for 10 s on either side of the peak; cell parameters by least-squares fit to observed 2θ values for 25 centered reflections with $25 \leq 2\theta \leq 50°$; intensity checks for three standard reflections showed little random variation ($\pm 1\%$); 1997 independent reflections (h 0 to 7, k −10 to 10, l −11 to 12), 1742 (87%) observed with $I \geq 2\sigma(I)$ and used in refinement; Lp corrections, no absorption or extinction correction. Structure solved by direct methods and refined by full-matrix least squares on F with anisotropic thermal parameters using SHELX76 (Sheldrick, 1976); H atoms identified in the

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