atoms as found in some cyclonucleosides (Brennan & Sundaralingham, 1973; Yamagata, Koshibe, Tokuoka, Fujii, Fujiwara, Kanai & Tomita, 1979; Yamagata, Suzuki, Fujii, Fujiwara & Tomita, 1979).

The bond distances and angles in the uracil moiety are in good agreement with standard values (Taylor & Kennard, 1982). However, in the furanose ring, the C(1′)—C(2′)—C(3′) angle [98·5 (4)°] is significantly smaller than the average value (101·3°) of the normal furanose rings (Sundaralingam, 1973), and it is probably due to the E_2 sugar puckering strained by cyclization. Indeed, this value agrees well with the suggested one from the correlation between the endocyclic bond angles and the pseudorotational parameters (Westhof & Sundaralingam, 1980).

In the present crystal no base stacking is observed. One hydrogen bond, *i.e.* N(3)—H···O(4) [2·834 (5) Å], is found between the adjacent molecules related by twofold screw symmetry to form an infinite hydrogen-bonded column along the *b* axis.

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X-ray Structure and Molecular-Packing Analysis of 1′-(*p*-Bromophenyl)-3′-ethyl-1′,3′,4′,5′-tetrahydro-1,2-dideoxy-D-*glycero*-α-D-*galacto*-heptofuranoso[2,1-*d*]imidazole-2′-thione Monohydrate

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(*Received* 25 *February* 1985; *accepted* 10 *June* 1985)

Abstract. 6-(*p*-Bromophenyl)-4-ethyl-3-hydroxy-2-(1,2,3-trihydroxypropyl)-2,3,3a,5,6,6a-hexahydrofuro-[2,3-*d*]imidazole-5(4*H*)-thione monohydrate, $C_{16}H_{21}BrN_2O_5S.H_2O$, $M_r = 451·3$, orthorhombic, $P2_12_12_1$, $a = 6·073$ (3), $b = 15·977$ (8), $c = 19·213$ (9) Å, $V = 1864$ (2) Å^3, $Z = 4$, $D_m = 1·62$ (1), $D_x = 1·61$ Mg m^{-3}, λ(Mo $K\alpha$) = 0·7107 Å, $\mu = 2·32$ mm^{-1}, $F(000) = 928$, room temperature, final $R = 0·080$ for 2147 observed reflexions. The sugar ring adopts the ^4E conformation and the dihedral angle in the bicyclic system is 115·0 (3)°. A three-dimensional network of hydrogen bonds links the molecules to stabilize the crystal structure. The lattice energy was computed in the atom–atom approach using van der Waals potential functions. These calculations account satisfactorily for all the features of the crystal packing, including rotation about selected bonds in the molecule.

Introduction. The crystal structure determination of the title compound (I) was undertaken as part of a continuing research project in this laboratory involving glucimidazoles and imidazole *C*-nucleosides. Some of these compounds, prepared in the Organic Chemistry Department of the University of Extremadura, have been studied (*e.g.* Estrada, Conde & Márquez, 1983, 1984; Conde, Millan, Conde & Márquez, 1985) in order to establish the conformational details of the molecule in the solid state. The application of aminonitrile synthesis to the preparation of the new 2-ethylamino-2-deoxyheptose having the D-*glycero*-D-

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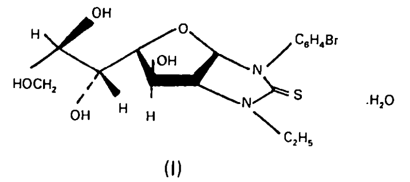
galacto configuration and its reaction with 4-bromophenyl isothiocyanate to afford the title compound has been recently reported (Galbis Pérez, Palacios Albarrán, Jiménez Requejo & Avalos Gonzáles, 1984). Its chemical nature was established from elemental analysis and spectroscopic IR and ^{1}H NMR data.



(I)

Experimental. Single crystals in the form of colourless needles elongated along [100]. D_m by flotation method. Crystal 0·37 × 0·17 × 0·08 mm. Unit-cell parameters by least squares from 25 reflexions, $6 < \theta < 20°$. Enraf–Nonius CAD-4 diffractometer, graphite monochromator, $2\theta < 60°$ $(0 \leq h \leq 8,\ 0 \leq k \leq 22,\ 0 \leq l \leq 27)$, ω–2θ scan mode. Two standard reflexions $(12\bar{1},\ 1\bar{1}\bar{3})$, variation in intensity less than 3% of the mean value. 3093 independent reflexions measured, 946 considered unobserved $[I < 2\sigma(I)]$. Lorentz and polarization correction; no correction for absorption $(\mu R \sim 0{\cdot}15)$ or extinction. Patterson function and heavy-atom method with the initial set of phases based on Br- and S-atom positions. Full-matrix least-squares refinement on F, anisotropic; difference Fourier synthesis revelated the H-atom positions; isotropic temperature factor for each H atom equal to that of the atom bonded to it; further least-squares refinement including the positional parameters of the H atoms and anomalous-dispersion correction for Br and S atomic scattering factors (*International Tables for X-ray Crystallography,* 1974) reduced wR to 0·082 (R = 0·080); weighting scheme based on the statistical count criterion $(w = 1/\sigma_F^2)$. $(\Delta/\sigma)_{\max} = 0{\cdot}03$, $S = 0{\cdot}80$ for 305 refined parameters. No. of reflexions/No. of parameters = 7·04. Final difference synthesis showed $0{\cdot}35 > \Delta\rho > -0{\cdot}40$ e Å^{-3}. The enantiomorphic form of the molecule was refined separately and converged to a final wR value of 0·085 (R = 0·083). The application of the $\mathscr{R}$ test (Hamilton, 1965) indicated that the first enantiomorph is correct at a significance level much lower than 0·005 $[wR(2)/wR(1) = 1{\cdot}036;\ \mathscr{R}_{1,1842,0.005} \simeq 1{\cdot}002]$ and, therefore, can be retained as the structural chirality. Moreover, this configuration is consistent with that defined by reference to the sugar moiety (Galbis Pérez *et al.,* 1984). Crystallographic programs of the *XRAY70* system (Stewart, Kundell & Baldwin, 1970) were used throughout.

Discussion. Fractional atomic coordinates and equivalent isotropic temperature factors (Hamilton, 1959) for non-hydrogen atoms are listed in Table 1.* Atom numbering and bond lengths and angles involving non-hydrogen atoms are given in Fig. 1.

Table 1. *Atomic fractional coordinates and equivalent isotropic temperature factors* (Å^2 × 10^3)

$$U_{\rm eq} = \tfrac{1}{3}\textstyle\sum_i\sum_j U_{ij}a^*_ia^*_ja_i.a_j\cos(\mathbf{a}_i.\mathbf{a}_j).$$

	x	y	z	$U_{\rm eq}$
Br	0·8503 (2)	0·61690 (8)	0·46738 (6)	636 (4)
S	0·7612 (3)	0·72967 (12)	0·81323 (11)	351 (5)
O(1)	1·2747 (9)	0·9150 (3)	0·6973 (3)	292 (13)
O(2)	1·4766 (10)	1·0111 (4)	0·8260 (3)	380 (16)
O(3)	0·8305 (10)	0·9509 (4)	0·7481 (3)	368 (16)
O(4)	1·1017 (17)	1·0615 (4)	0·6047 (3)	364 (16)
O(5)	0·6331 (15)	1·0684 (7)	0·5642 (4)	67 (3)
O(6)	0·0442 (17)	0·5290 (8)	0·0372 (4)	73 (3)
N(1)	1·1076 (10)	0·7883 (4)	0·7380 (3)	317 (17)
N(2)	1·0924 (10)	0·8360 (4)	0·8450 (3)	278 (16)
C(1)	0·9915 (11)	0·7865 (4)	0·7992 (4)	274 (17)
C(2)	1·2935 (11)	0·8455 (5)	0·7412 (4)	282 (18)
C(3)	1·2864 (11)	0·8767 (5)	0·8176 (3)	293 (17)
C(4)	1·2677 (11)	0·9731 (5)	0·8116 (3)	282 (17)
C(5)	1·2120 (11)	0·9895 (4)	0·7350 (3)	253 (17)
C(6)	0·9766 (12)	1·0127 (5)	0·7203 (4)	290 (18)
C(7)	0·9287 (13)	1·0225 (4)	0·6427 (4)	282 (18)
C(8)	0·7121 (14)	1·0681 (9)	0·6327 (5)	52 (3)
C(9)	1·0459 (14)	0·7464 (4)	0·6754 (3)	299 (19)
C(10)	0·8519 (15)	0·7669 (6)	0·6425 (5)	42 (2)
C(11)	0·7917 (15)	0·7272 (7)	0·5799 (5)	43 (5)
C(12)	0·9286 (16)	0·6668 (5)	0·5538 (4)	38 (2)
C(13)	1·1336 (18)	0·6482 (6)	0·5854 (5)	45 (3)
C(14)	1·1881 (14)	0·6872 (5)	0·6472 (5)	38 (2)
C(15)	1·0334 (15)	0·8439 (6)	0·9193 (4)	37 (2)
C(16)	1·1796 (18)	0·7924 (6)	0·9641 (5)	47 (3)

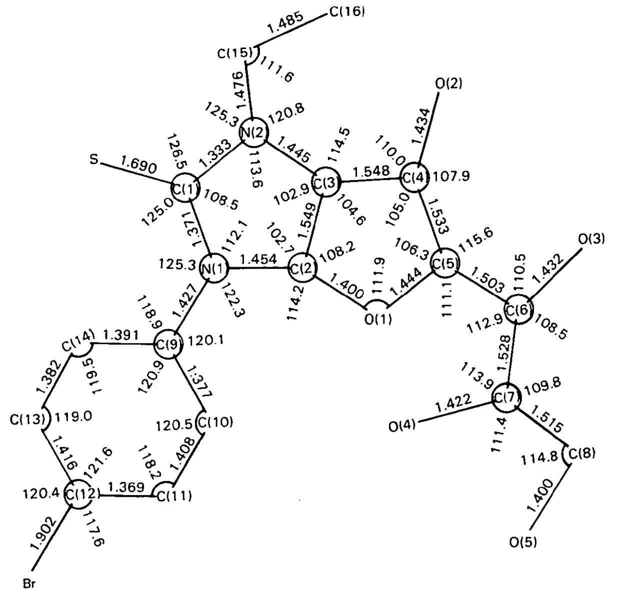


Fig. 1. Bond lengths (Å) and angles (°). (Standard deviations are in the ranges 0·02–0·05 Å and 1·7–3·1°, respectively.)

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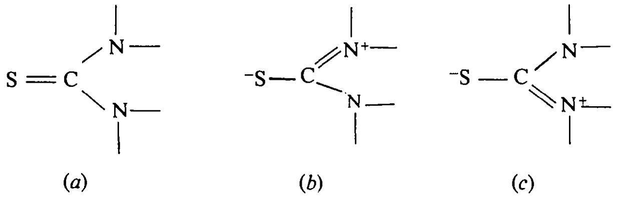
* Lists of structure factors, anisotropic thermal parameters and H-atom parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 42295 (16 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Molecular geometry

Bond distances and angles in the imidazolidine ring agree with the mean values reported for analogous glycofuranoimidazolidine-2-thione compounds (Estrada, Conde & Márquez, 1983, 1984; Conde, Millan, Conde & Márquez, 1985). The S—C bond length of 1·690 (7) Å and those observed for N(1)—C(1) = 1·371 (9) and N(2)—C(1) = 1·333 (9) Å should indicate the contribution of the canonical resonance forms of the thiourea system:



The observed asymmetry of the endocyclic N—C bonds ($\Delta/\sigma = 4{\cdot}2$) is due to the different substituents at the two N atoms. The imidazolidine ring is not planar: the ring atoms deviate significantly from the least-squares plane [$\sum(\Delta/\sigma)^2 = 19{\cdot}06$; χ^2 at 95% = 5·99] and the puckering amplitude (Cremer & Pople, 1975) is $Q = 0{\cdot}032\ (8)$ Å. This feature was also found in analogous structures (Estrada, Conde & Márquez, 1983; Conde, Millan, Conde & Márquez, 1985).

The phenyl ring shows a statistically significant deviation from planarity [$\sum(\Delta/\sigma)^2 = 18{\cdot}98$; χ^2 at 95% = 7·81] and forms a dihedral angle of 64·4 (2)° with the imidazolidine ring. Values of this dihedral angle in the range 45–80° were found for analogous compounds, and should indicate a significant contribution of intramolecular potential energy (phenyl–imidazolidine) over the intermolecular one.

The ethyl group is also twisted with respect to the imidazolidine ring as indicated by the torsion angle C(1)—N(2)—C(15)—C(16) = 98·9 (9)°.

Bond lengths and angles in the furanosyl ring are close to the mean values observed for analogous compounds. The observed asymmetry of the endocyclic bonds O(1)—C(2) = 1·400 (9) and O(1)—C(5) = 1·444 (8) Å ($\Delta/\sigma = 3{\cdot}65$) is a typical feature of these compounds due to anomeric effects. The furanose ring is not planar as expected. In terms of the ring-puckering coordinates (Cremer & Pople, 1975) the amplitude and phase magnitudes are $Q = 0{\cdot}188\ (6)$ Å and $\varphi = -42\ (2)°$ for the sequence O(1)—C(2)—C(3)—C(4)—C(5) and the resulting conformation corresponds to the puckering mode ^4E, similar to that found for other analogous compounds (Conde, López-Castro & Márquez, 1978; Estrada, Conde & Márquez, 1984) but it is different from that found in another recently reported structure (Conde, Millan, Conde & Márquez, 1985). The asymmetry parameter of Nardelli (1983*a*), ΔC_s[C(5)] = 0·014 (3), indicates approximate mirror-plane symmetry in the ring. The small value of the torsion angle O(1)—C(2)—C(3)—C(4) [−1·5 (7)°], a typical feature of these compounds, may be attributed to the distortion of the furanose group on its fusion with the imidazolidine ring.

The two fused rings in the bicyclic system show a *cis* form of coupling and the bonds at C(2) and C(3) atoms are nearly eclipsed, these features being similar to those observed for analogous compounds. The dihedral angle between the least-squares planes through the imidazolidine and furanose rings is 115·0 (3)°.

Crystal-packing analysis

Fig. 2 shows the contents of the unit cell viewed down [100]. The crystal structure is stabilized by a three-dimensional hydrogen-bonding network. Molecules are linked to form chains parallel to [100]. In these chains each molecule is linked to that related by a cell translation by O(3)—H(O3)···O(2)($x-1$, y, z). Also, each molecule is linked to the neighbour related by a twofold screw axis parallel to [100] by O(5)—H(O5)···Br($x-\frac{1}{2}$, $-y+\frac{3}{2}$, $-z+1$). Also, molecules are linked to form chains parallel to [001] by two O—H···O hydrogen bonds involving the hydration water molecule: O(6)—H(O61)···O(4)($-x+1$, $y-\frac{1}{2}$, $-z+\frac{1}{2}$). Finally, the contact O(4)—H(O4)···S($-x+2$, $y+\frac{1}{2}$, $-z+\frac{3}{2}$) between molecules related by a screw axis along [010] could be considered a hydrogen bond. Details of the geometry of these hydrogen bonds are given in Table 2.

For the O—H···O contacts listed in Table 2 the values of the parameter d, defined as the difference between the sum of the van der Waals radii of H and O (1·20 and 1·50 Å) and the interatomic distance (Taylor & Kennard, 1982), are 0·89 and 0·61 Å respectively, satisfying the rule $d > 0{\cdot}3$ Å, and the angles O—H···O

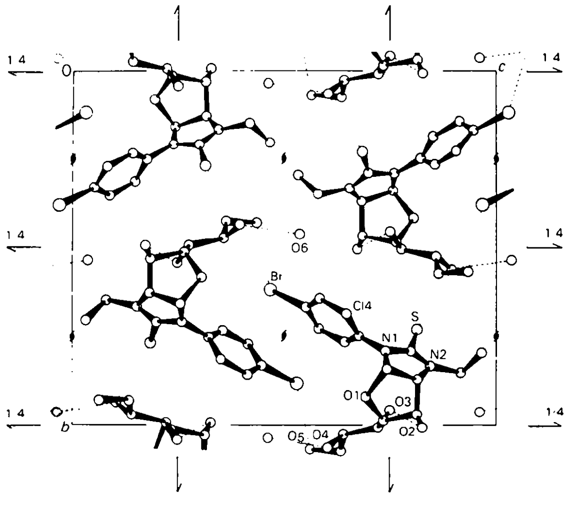


Fig. 2. A view of the unit-cell contents along [100].

Table 2. *Geometry of the possible hydrogen bonds*

X—H$\cdots Y$	$X\cdots Y$ (Å)	X—H (Å)	H$\cdots Y$ (Å)	X—H$\cdots Y$ (°)
O(5)—H(O5)$\cdots$Br^{i}	3·476 (11)	0·84 (23)	2·68 (23)	160 (22)
O(3)—H(O3)$\cdots$O(2^{ii})	2·790 (9)	1·00 (16)	1·82 (16)	163 (14)
O(6)—H(O61)$\cdots$O(4^{iii})	2·913 (10)	0·87 (25)	2·09 (25)	159 (23)
O(4)—H(O4)$\cdots$S^{iv}	3·225 (7)	0·67 (14)	2·64 (15)	148 (16)

Symmetry code: (i) $x-\frac{1}{2}, -y+\frac{3}{2}, -z+1$; (ii) $x-1, y, z$; (iii) $-x+1, y-\frac{1}{2}, -z+\frac{1}{2}$; (iv) $-x+2, y+\frac{1}{2}, -z+\frac{3}{2}$.

Table 3. *Molecular-packing analysis*

	(*a*)	(*b*)	(*c*)	(*d*)
Cell parameters				
a (Å)	6·073 (3)*	6·229	6·073*	6·237
b (Å)	15·977 (8)*	15·897	15·977*	15·932
c (Å)	19·213 (9)*	19·284	19·213*	19·301
Glucofurano-imidazolidine molecule				
Translation (Å)	0·15	0·10	0·13	0·09
Rotation (°)	7·0	6·8	5·9	6·0
Subrotations (°)				
C(2)—N(1)—C(9)—C(10)	−111·4 (9)*	−111·4*	−113·6	−113·6
C(1)—N(2)—C(15)—C(16)	98·9 (9)*	98·9*	95·6	95·0
O(1)—C(5)—C(6)—C(7)	−54·9 (8)*	−54·9*	−64·4	−63·5
Hydration water molecule				
Translation (Å)	0·09	0·06	0·07	0·05
Rotation (°)	2·2	1·9	1·9	2·0

(*a*) With cell parameters and torques fixed at experimental values (*); (*b*) with optimized cell parameters and torques fixed; (*c*) with cell parameters fixed and torques relaxed; (*d*) with optimized cell parameters and torques.

can be compared with the mean value of 165·8 (12)° for bonds with $O\cdots H > 1·81$ Å (Allen, Kennard & Taylor, 1983). The short O—H$\cdots$Br contact has $d > 0·3$ Å and angle O—H$\cdots$Br = 160 (22)° and so can be considered as a hydrogen bond. Finally, the presence of short contacts involving the S atom and having hydrogen-bond characteristics is a feature observed in other analogous compounds. No other intermolecular contact significantly shorter than the sum of the van der Waals radii has been detected. The molecular geometry and crystal packing were computed by *PARST* (Nardelli, 1983*b*).

A minimization of the crystal lattice energy with respect to lattice constants, molecular translation and rotation and also subrotation of molecular fragments about selected bonds as axes was performed by means of the program *PCK*6 (Williams, 1972*b*, 1974) starting from the experimental structure. Potential functions in the form $U(r) = -Ar^{-6} + B\exp(-Cr)$ were used to represent the non-bonded interatomic potential energy. The set of potential parameters included parameters fitted by Williams (1972*a*) for the C$\cdots$C and H$\cdots$H interactions, by Govers (1975) for N$\cdots$N, by Mason & Kreevoy (1955) for O$\cdots$O and S$\cdots$S and parameters of Burgos & Bonadeo (1977) for Br$\cdots$Br. For mixed interactions the combination rules of Mirskaya (1973) were used.

Four runs of energy minimization were carried out from two rigid bodies: the glucofuranoimidazolidine and the hydration water molecules, respectively; in the first, the X-ray molecular structure was retained and the lattice constants were fixed; in the second, cell parameters were included as variables; in the third, the phenyl ring, the ethyl group and the sugar chain rotations were relaxed; cell parameters were also included as variables in the last run.

Representative results of energy-minimization runs are given in Table 3. The calculated values for lattice energy, not included in Table 3, are higher than the real ones because the intermolecular hydrogen bonds were treated by van der Waals potential functions.

As shown in Table 3, the inclusion of cell parameters as variables leads to a significant (~3%) expansion of the cell along **a** and a minor expansion (~0·4%) along **c**, while a contraction (~5%) results along **b**. This unexpected expansion of the cell may be attributed to the approach used in which the hydrogen-bond contacts are represented by van der Waals potential functions. So, a higher contribution of the repulsive term for these contacts results and the contacts are expanded. As shown in Table 2, hydrogen-bond chains occur mainly along [100] and also along [001]. Apart from this, the theoretical configuration agrees well with the experimental structure, in spite of the existence of a hydration water molecule which links the neighbouring glucofuranoimidazolidine molecules *via* O—H$\cdots$O hydrogen bonds. All the optimized structures remain very similar with regard to the molecular parameters and the torques of the selected subrotations. As shown in Table 3, shifts of positional and orientational molecular parameters are lower than 0·15 Å and 7° respectively. Torsional angles of selected molecular fragments are reproduced within 2° for the ethyl and bromophenyl groups. The higher distortion which is observed for the sugar chain may be attributed to the presence of the intermolecular hydrogen bonds involving atoms in the chain.

The authors thank Professor J. A. Galbis Pérez for supplying the crystals and for helpful discussions on chemical aspects and Professor A. López-Castro for collecting the diffractometer data. The present work is part of a research project supported by a grant from the CAICYT of the Spanish Government.

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Structure of 1,2,4,5,6,7,8,9-Octachloro-10,10-dimethoxytricyclo[5.2.1.$0^{2,6}$]deca-4,8-dien-3-one

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(*Received* 29 *April* 1985; *accepted* 11 *June* 1985)

Abstract. $C_{12}H_6Cl_8O_3$, M_r=481·80, monoclinic, $P2_1/c$, $a = 11·028\ (9)$, $b = 11·871\ (3)$, $c = 13·231\ (9)$ Å, $\beta = 93·14\ (3)°$, $V = 1730\ (2)$ Å^3, $Z = 4$, $D_x = 1·850$ Mg m^{-3}, λ(Mo $K\alpha$) = 0·7107 Å, $\mu = 1·32$ mm^{-1}, $F(000) = 952$, $T = 293$ K, final $R = 0·035$ for 3518 observed reflections. The C(sp^3)—Cl and C(sp^2)—Cl bond-length ranges are 1·744 (2)–1·767 (2) and 1·688 (2)–1·696 (2) Å, respectively. The C(sp^3)—C(sp^3) bond lengths are somewhat extended and range from 1·572 (3) to 1·585 (3) Å. The bridgehead C—C—C and O—C—O angles are 91·6 (1) and 114·6 (2)°, respectively, while the O—C—O—C torsion angles are −53·5 (2) and −47·6 (3)°. The five-membered ring adjacent to the norbornene ring system is in the *endo* configuration with respect to it.

Introduction. In the cycloaddition reaction between 1,2,3,4-tetrachloro-5,5-dimethoxycyclopentadiene with dimethyl dibromomaleic ester the product was not dimethyl 5,6-dibromo-1,2,3,4-tetrachloro-7,7-dimethoxybicyclo[2.2.1]hept-2-ene-5,6-dicarboxylate (Vančik, Sunko & Lovrić, 1985) as expected. The X-ray structure determination showed that the result of the reaction was a partially hydrolyzed dimer of the cyclopentadiene derivative. The structure determination was undertaken to give additional information on this interesting strained molecule.

Experimental. Intensity data collected from a crystal 0·25 × 0·34 × 0·42 mm with crystal faces ±(1$\bar{1}$0), ±(001), ±($\bar{1}$11), ±(1$\bar{1}$1). Philips PW 1100 four-circle diffractometer, θ–2θ scanning technique, scan width 1·80°, scan rate 0·06 s^{-1}. Unit-cell parameters obtained from least-squares analysis of 16 reflections with 2θ values ranging from 12 to 20°. Absent reflections $h0l$, $l \neq 2n$ and $0k0$, $k \neq 2n$ confirmed space group $P2_1/c$. Out of 4362 reflections scanned within a quadrant ±h,k,l (h ±15, k 16, l 18), up to $\sin\theta/\lambda \leq 0·70$ Å^{-1}, 4166 unique reflections classified as observed. Three standard reflections ($\bar{3}1\bar{2}$, $10\bar{6}$, $0\bar{6}0$) measured every 2 h showed an averaged variation of 1·0 (1)%. Corrections applied for Lorentz and polarization effects. No correction for absorption or extinction. Structure solved by direct methods

0108-2701/85/111662-03$01.50