containing the 5-*O*-sulfamoylribose moiety. Conformational parameters follow the conventions of Altona & Sundaralingam (1972).

References

Altona, C. & Sundaralingam, M. (1972). *J. Am. Chem. Soc.* **94**, 8205–8212.

Cambridge Structural Database (1989). Univ. Chemical Laboratory, Lensfield Road, Cambridge, England.

Cordes, A. W. (1983). Personal communication.

Frenz, B. A. (1985). *Enraf–Nonius SDP-Plus Structure Determination Package*. Version 3.0. Enraf–Nonius, Delft, The Netherlands.

Haines, D. R., Leonard, N. J. & Wiemer, D. F. (1982). *J. Org. Chem.* **47**, 474–482.

Hanna, N. B., Dimitrijevich, S. D., Larson, S. B., Robins, R. K. & Revankar, G. R. (1988). *J. Heterocycl. Chem.* **25**, 1857–1868.

International Tables for X-ray Crystallography (1974). Vol. IV. Birmingham: Kynoch Press. (Present distributor Kluwer Academic Publishers, Dordrecht.)

Johnson, C. K. (1976). *ORTEPII*. Report ORNL-5138. Oak Ridge National Laboratory, Tennessee, USA.

Kini, G. D., Henry, E. M., Robins, R. K., Larson, S. B., Marr, J. J., Berens, R. L., Bacchi, C. J., Nathan, H. C. & Keithly, J. S. (1990). *J. Med. Chem.* **33**, 121–128.

Larson, S. B. (1980). PhD Dissertation, Brigham Young Univ., Provo, Utah, USA.

Larson, S. B., Henry, E. M., Kini, G. D. & Robins, R. K. (1990). *Acta Cryst.* **C46**, 1138–1141.

Larson, S. B., Kini, G. D. & Robins, R. K. (1990). *Acta Cryst.* **C46**, 1141–1143.

Prusiner, P. & Sundaralingam, M. (1976). *Acta Cryst.* **B32**, 419–426.

Sanghvi, Y. S., Hanna, N. B., Larson, S. B., Fujitaki, J. M., Willis, R. C., Smith, R. A., Robins, R. K. & Revankar, G. R. (1988). *J. Med. Chem.* **31**, 330–335.

Sheldrick, G. M. (1976). *SHELX76*. Program for crystal structure determination. Univ. of Cambridge, England.

Sheldrick, G. M. (1986). *SHELXS86*. Program for crystal structure solution. Univ. of Göttingen, Federal Republic of Germany.

Stewart, R. F., Davidson, E. R. & Simpson, W. T. (1965). *J. Chem. Phys.* **42**, 3175–3187.

Acta Cryst. (1990). **C46**, 1138–1141

Structure of 1-(5-*O*-Sulfamoyl-β-D-ribofuranosyl)-1*H*-1,2,4-triazole-3-thiocarboxamide

By Steven B. Larson,* Elizabeth M. Henry, Ganesh D. Kini and Roland K. Robins

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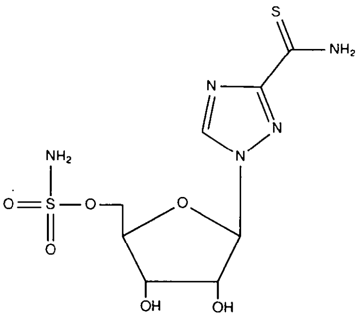
(*Received* 11 *September* 1989; *accepted* 14 *November* 1989)

Abstract. $C_8H_{13}N_5O_6S_2$, $M_r = 339{\cdot}34$, orthorhombic, $P2_12_12_1$, $a = 8{\cdot}5583\,(10)$, $b = 9{\cdot}0779\,(11)$, $c = 17{\cdot}360\,(3)$ Å, $V = 1348{\cdot}7\,(3)$ Å^3, $Z = 4$, $D_x = 1{\cdot}671$ g cm^{-3}, Cu $K\alpha$ ($\lambda = 1{\cdot}54178$ Å), $\mu = 38{\cdot}925$ cm^{-1}, $F(000) = 704$, $T = 295$ K, $R = 0{\cdot}0243$ for 2701 reflections ($F \geq 4\sigma_F$). The nucleoside has a *syn* conformation [χ_{CN}(N2—N1—C1′—O4′) = 50·6 (2)°]. The C5′—O5′ orientation is *gauche*$^-$–*gauche*$^+$ [$\varphi_{OO} = -72{\cdot}4\,(2)$; $\varphi_{CO} = 47{\cdot}4\,(2)$°] which places the sulfamoyl group (especially NH_2) over the ribofuranose ring, resulting in the formation of an intramolecular hydrogen bond to N2 of the triazole ring [d(H···N) = 2·39 (4) Å; ∠N—H···N = 166 (3)°]. The sugar conformation and puckering parameters are C2′-*endo* ($^2T_1$), $P = 155{\cdot}9$° and $\tau_m = 36{\cdot}4$°. All atoms in the triazole ring are within 0·0035 Å of their mean plane and the thiocarboxamide group is twisted out of this plane by 21·88 (6)°. The thiocarboxamide nitrogen is *cis* to N4 of the triazole. All possible hydrogen donors participate in hydrogen bonding [range for H···A distances is 2·07 (4)–2·65 (4) Å]. The sulfonamide amino group is nonplanar; the N atom is displaced from the plane of the three adjoining atoms by 0·230 (2) Å. There is no base stacking in the crystal structure.



(1)

Experimental. The title compound (1) was synthesized from 1-β-D-ribofuranosyl-1*H*-1,2,4-triazole-3-thiocarboxamide as described elsewhere (Kini, Henry, Robins, Larson, Marr, Berens, Bacchi,

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Table 1. *Summary of data collection and refinement for* (1)

(*a*) Data collection (295 K)	
Mode	ω–2θ scan
Scan range (°)	0·80 + 0·15tanθ
Background	Scan 0·25 times scan range before and after scan
Scan rate (° min^{-1})	1·4–16·49
Exposure time (h)	51·3
Stability correction range on *I*	1·000–1·014
Check reflections	139, 25$\bar{2}$, 5$\bar{1}$3
2θ range (°)	3·0–152·0
Range in *hkl*, min.	0, −11, −21
max.	10,11,21
Total reflections, measured, unique	5984, 2814
R_{int}	0·0212
Crystal dimensions (mm)	0·305 × 0·225 × 0·13
Crystal volume (mm^3)	0·00550
Crystal faces	{001}; (02$\bar{3}$); (0$\bar{2}$3); (0$\bar{2}\bar{3}$); (103); ($\bar{1}$0$\bar{3}$); (20$\bar{3}$); ($\bar{2}$03)
Transmission-factor range	0·427–0·706
(*b*) Structure refinement	
Reflections used ($F \geq 4\sigma_F$)	2701
No. of variables	243
Extinction parameter	1·17 (10) × 10^{-6}
Goodness of fit, *S*	1·409
R, *wR*	0·0243, 0·0355
R for all data	0·0264
Max., av. Δ/σ	0·009, 0·0007
Max., min. $\Delta\rho$ in ΔF map (e Å^{-3})	0·36, −0·43

Table 2. *Positional and isotropic thermal parameters for all atoms in* (1)

For non-H atoms, $U_{\text{eq}} = \frac{1}{3}\sum_i\sum_j U_{ij}a_i^*a_j^*\mathbf{A}_{ij}$, where $\mathbf{A}_{ij}$ is the dot product of the *i*th and *j*th direct-space unit-cell vectors.

	x	*y*	*z*	$U_{\text{eq}}/U_{\text{iso}}$(Å^2)
N1	0·7241 (2)	0·58371 (15)	0·65346 (8)	0·0246 (3)
N2	0·5841 (2)	0·58156 (14)	0·61677 (8)	0·0253 (3)
C3	0·5781 (2)	0·4440 (2)	0·59055 (9)	0·0259 (4)
N4	0·7054 (2)	0·3602 (2)	0·60738 (9)	0·0335 (4)
C5	0·7936 (2)	0·4518 (2)	0·64712 (10)	0·0317 (5)
C6	0·4406 (2)	0·3867 (2)	0·54762 (10)	0·0268 (4)
S7	0·26517 (5)	0·46342 (5)	0·55483 (3)	0·03412 (12)
N8	0·4697 (2)	0·2689 (2)	0·50507 (11)	0·0368 (5)
C1′	0·7913 (2)	0·7197 (2)	0·68424 (9)	0·0250 (4)
C2′	0·8365 (2)	0·8291 (2)	0·62103 (9)	0·0239 (4)
C3′	0·8320 (2)	0·9751 (2)	0·66499 (9)	0·0263 (4)
C4′	0·7005 (2)	0·9492 (2)	0·72296 (9)	0·0266 (4)
C5′	0·5459 (2)	1·0172 (2)	0·70113 (10)	0·0325 (5)
O2′	0·9846 (2)	0·7995 (2)	0·58903 (9)	0·0369 (4)
O3′	0·9725 (2)	0·9982 (2)	0·70698 (9)	0·0380 (4)
O4′	0·6790 (2)	0·79137 (13)	0·72960 (7)	0·0319 (3)
O5′	0·51687 (14)	0·9786 (2)	0·62085 (7)	0·0332 (4)
S6′	0·34748 (5)	0·94144 (4)	0·59452 (2)	0·02894 (11)
O7′	0·2422 (2)	1·05620 (15)	0·61576 (9)	0·0413 (4)
O8′	0·3658 (2)	0·9123 (2)	0·51417 (8)	0·0464 (5)
N9′	0·3059 (2)	0·7995 (2)	0·64561 (11)	0·0368 (5)
H5	0·878 (3)	0·427 (3)	0·6674 (13)	0·038 (6)
H8*A*	0·555 (5)	0·215 (4)	0·508 (2)	0·084 (12)
H8*B*	0·386 (3)	0·220 (3)	0·4801 (14)	0·041 (6)
H1′	0·884 (3)	0·696 (2)	0·7126 (12)	0·027 (5)
H2′	0·756 (2)	0·830 (2)	0·5819 (11)	0·022 (4)
H3′	0·808 (2)	1·058 (2)	0·6320 (11)	0·018 (4)
H4′	0·728 (3)	0·985 (3)	0·7759 (13)	0·036 (6)
H5′*A*	0·466 (3)	0·981 (3)	0·724 (2)	0·048 (7)
H5′*B*	0·552 (4)	1·135 (3)	0·709 (2)	0·060 (8)
HO2′	0·968 (4)	0·762 (4)	0·553 (2)	0·071 (10)
HO3′	1·035 (5)	1·028 (5)	0·676 (2)	0·11 (2)
H9′*A*	0·369 (4)	0·741 (4)	0·646 (2)	0·064 (9)
H9′*B*	0·215 (4)	0·771 (3)	0·638 (2)	0·055 (8)

Table 3. *Bond lengths* (Å) *and bond angles* (°) *in* (1)

1	2	3	1—2	1—2—3
N2	N1	C5	1·357 (2)	109·89 (13)
C5	N1	C1′	1·342 (2)	127·49 (15)
C1′	N1	N2	1·463 (2)	122·05 (13)
C3	N2	N1	1·330 (2)	102·04 (13)
N4	C3	C6	1·361 (2)	123·07 (14)
N4	C3	N2		114·83 (15)
C6	C3	N2	1·486 (2)	122·09 (15)
C5	N4	C3	1·318 (2)	102·63 (14)
N1	C5	N4		110·6 (2)
S7	C6	N8	1·660 (2)	123·47 (14)
S7	C6	C3		122·07 (12)
N8	C6	C3	1·323 (2)	114·45 (15)
C2′	C1′	O4′	1·530 (2)	105·95 (13)
C2′	C1′	N1		112·66 (13)
O4′	C1′	N1	1·402 (2)	109·10 (14)
C3′	C2′	O2′	1·530 (2)	112·58 (14)
C3′	C2′	C1′		101·44 (12)
O2′	C2′	C1′	1·410 (2)	112·73 (14)
C4′	C3′	O3′	1·528 (2)	107·94 (13)
C4′	C3′	C2′		102·30 (13)
O3′	C3′	C2′	1·421 (2)	111·24 (14)
C5′	C4′	O4′	1·508 (3)	108·29 (14)
C5′	C4′	C3′		114·68 (14)
O4′	C4′	C3′	1·449 (2)	107·35 (13)
O5′	C5′	C4′	1·458 (2)	106·93 (14)
C1′	O4′	C4′		109·11 (12)
S6′	O5′	C5′	1·5569 (13)	119·43 (11)
O7′	S6′	O8′	1·4258 (14)	117·20 (10)
O7′	S6′	N9′		107·69 (9)
O7′	S6′	O5′		110·75 (8)
O8′	S6′	N9′	1·4284 (14)	114·55 (10)
O8′	S6′	O5′		102·98 (8)
N9′	S6′	O5′	1·604 (2)	102·60 (8)

Nathan & Keithly, 1990). Crystals formed as yellowish, transparent, square-pyramidal prisms by slowly cooling a hot methanol solution of (1). Data collection and refinement are summarized in Table 1. Unit-cell parameters were obtained by least-squares refinement of the setting angles of 25 reflections with $52{\cdot}5 < 2\theta < 59{\cdot}3°$. An Enraf–Nonius CAD-4 diffractometer with a graphite monochromator was used for all measurements. Data reduction, which included Lorentz, polarization, decay and absorption corrections, was accomplished with the *SDP-Plus* software (Frenz, 1985). Crystal and instrument stability were monitored by remeasurement of three check reflections every hour. A linear fit of the intensities of these reflections was used to correct the data. Absorption corrections, based on crystal-face measurements, were applied. The function minimized was $\sum w(|F_o| - |F_c|)^2$, where $w^{-1} = (\sigma_F^2 + 0{\cdot}0004F^2)$, $\sigma_F = F\sigma_I/2I$, and $\sigma_I = (N_{\text{pk}} + N_{\text{bg1}} + N_{\text{bg2}})^{1/2}$.

Crystallographic coordinates for the two S atoms were deduced from a sharpened Patterson map. The initial positional parameters for the remaining 19 non-H atoms were determined from two successive electron density maps (six atoms from the first and 13 atoms from the second). All H atoms were located in a difference map as peaks of density 0·50–1·00 e Å^{-3} at $R = 0{\cdot}039$. All positional parameters, anisotropic thermal parameters for non-H atoms, isotropic thermal parameters for H atoms and an extinction parameter were refined by full-matrix least squares with *SHELX76* (Sheldrick, 1976). Scattering factors and anomalous-dispersion corrections were taken from *International Tables for X-ray Crystallography* (1974) except those of H which were taken

Table 4. *Hydrogen bonding in* (1)

D—	H···	*A*	Symmetry of *A* relative to *D*	*d*(*D*···*A*) (Å)	*d*(H···*A*) (Å)	∠(*D*—H···*A*) (°)
N8	H8*A*	S7	0·5+*x*, 0·5−*y*, 1·0−*z*	3·453 (2)	2·65 (4)	151· (3)
N8	H8*B*	N4	*x*−0·5, 0·5−*y*, 1·0−*z*	3·210 (2)	2·28 (3)	165· (2)
O2′	HO2′	O8′	1·0+*x*, 1·5−*y*, 1·0−*z*	2·819 (2)	2·16 (4)	153· (4)
O3′	HO3′	O7′	*x*+1·0, *y*, *z*	2·849 (2)	2·07 (4)	161· (4)
N9′	H9′*A*	N2	*x*, *y*, *z*	3·136 (2)	2·39 (4)	166· (3)
N9′	H9′*B*	O2′	*x*−1·0, *y*, *z*	2·920 (2)	2·16 (3)	151· (3)

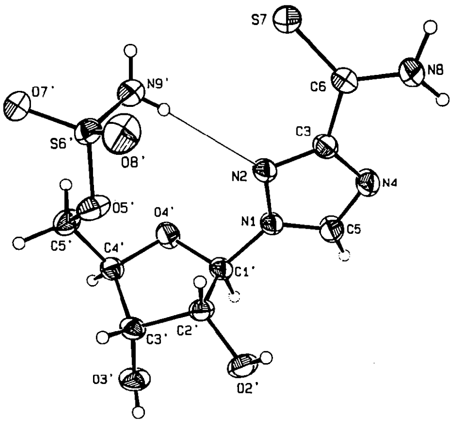

Fig. 1. Thermal-ellipsoid plot of (1) illustrating atom labeling, molecular conformation and intramolecular hydrogen bonding. The S7···N2 distance is 3·124 (2) Å and may account for the 22° twist of the carboxamide group out of the mean plane of the triazole ring. The ellipsoids are drawn at the 50% probability level.

from Stewart, Davidson & Simpson (1965). Least-squares-planes program from Cordes (1983); figures were drawn with *ORTEP*II (Johnson, 1976); parameter and geometry tables were produced with *FUER* and lists of structure-factor amplitudes were produced with *LISTFC* (Larson, 1980). The atomic coordinates are listed in Table 2. Bond lengths and bond angles are given in Table 3. Hydrogen-bonding geometries are detailed in Table 4. Fig. 1 illustrates the atom labeling and molecular conformation; Fig. 2 illustrates the unit-cell packing and hydrogen bonding.*

Related literature. The title compound (1) has shown some antiparasitic activity (Kini *et al.*, 1990). The preceding and following papers report the structures of the 3-carboxamide (Larson, Kini & Robins, 1990*a*) and 3-carbonitrile (Larson, Kini & Robins, 1990*b*) derivatives of (1). The thiocarboxamide group orientation (NH_2 *cis* to N4) is opposite to that observed in both ribavirin (Prusiner & Sundaralingam, 1976) and 5′-*O*-sulfamoylribavirin (Larson *et al.*, 1990*a*). Several other triazole nucleosides have been reported (Haines, Leonard & Wiemer, 1982; Sanghvi, Hanna, Larson, Fujitaki, Willis, Smith, Robins & Revankar, 1988; Hanna, Dimitrijevich, Larson, Robins & Revankar, 1988). No structures containing the 5-*O*-sulfamoylribofuranose moiety were found in the Cambridge Structural Database (1989). Conformational parameters follow the conventions of Altona & Sundaralingam (1972).

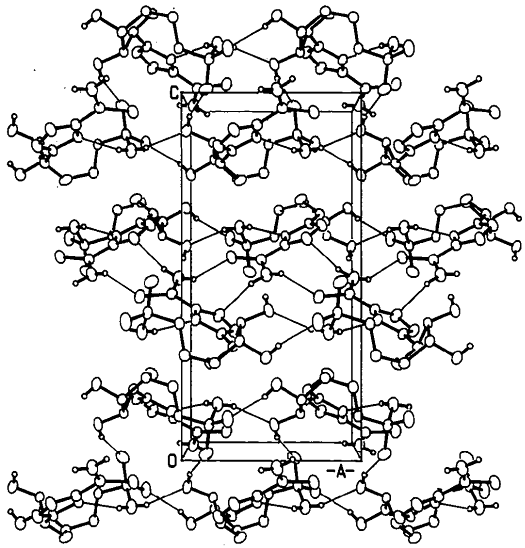

Fig. 2. Crystal packing diagram of (1) viewed along the *b* axis. C—H H atoms are omitted and hydrogen bonds are drawn as thin lines. There is no base stacking. Most of the hydrogen bonding occurs between molecules along the twofold screw axis parallel to the *a* axis which are linked with other 2_1 axes translated along the **b** direction forming sheets parallel to the *ab* plane.

References

Altona, C. & Sundaralingam, M. (1972). *J. Am. Chem. Soc.* **94**, 8205–8212.

Cambridge Structural Database (1989). Univ. Chemical Laboratory, Lensfield Road, Cambridge, England.

Cordes, A. W. (1983). Personal communication.

Frenz, B. A. (1985). *Enraf–Nonius SDP-Plus Structure Determination Package*. Version 3.0. Enraf–Nonius, Delft, The Netherlands.

Haines, D. R., Leonard, N. J. & Wiemer, D. F. (1982). *J. Org. Chem.* **47**, 474–482.

Hanna, N. B., Dimitrijevich, S. D., Larson, S. B., Robins, R. K. & Revankar, G. R. (1988). *J. Heterocycl. Chem.* **25**, 1857–1868.

International Tables for X-ray Crystallography (1974). Vol. IV. Birmingham: Kynoch Press. (Present distributor Kluwer Academic Publishers, Dordrecht.)

Johnson, C. K. (1976). *ORTEP*II. Report ORNL-5138. Oak Ridge National Laboratory, Tennessee, USA.

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* Tables of anisotropic thermal parameters, bond lengths and angles involving H atoms, torsion angles, least-squares planes and structure-factor amplitudes have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 52436 (14 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Kini, G. D., Henry, E. M., Robins, R. K., Larson, S. B., Marr, J. J., Berens, R. L., Bacchi, C. J., Nathan, H. C. & Keithly, J. S. (1990). *J. Med. Chem.* **33**, 121–128.
Larson, S. B. (1980). PhD Dissertation, Brigham Young Univ., Provo, Utah, USA.
Larson, S. B., Kini, G. D. & Robins, R. K. (1990*a*). *Acta Cryst.* C**46**, 1135–1138.
Larson, S. B., Kini, G. D. & Robins, R. K. (1990*b*). *Acta Cryst.* C**46**, 1141–1143.
Prusiner, P. & Sundaralingam, M. (1976). *Acta Cryst.* B**32**, 419–426.
Sanghvi, Y. S., Hanna, N. B., Larson, S. B., Fujitaki, J. M., Willis, R. C., Smith, R. A., Robins, R. K. & Revankar, G. R. (1988). *J. Med. Chem.* **31**, 330–335.
Sheldrick, G. M. (1976). *SHELX76*. Program for crystal structure determination. Univ. of Cambridge, England.
Stewart, R. F., Davidson, E. R. & Simpson, W. T. (1965). *J. Chem. Phys.* **42**, 3175–3187.

Acta Cryst. (1990). C**46**, 1141–1143

Structure of 1-(5-*O*-Sulfamoyl-*β*-D-ribofuranosyl)-1*H*-1,2,4-triazole-3-carbonitrile

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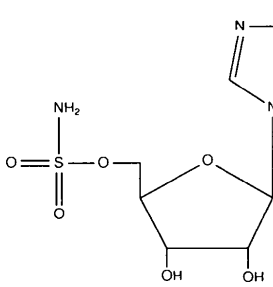
(*Received* 11 *September* 1989; *accepted* 14 *November* 1989)

Abstract. $C_8H_{11}N_5O_6S$, M_r = 305·26, orthorhombic, $P2_12_12_1$, a = 6·2993 (13), b = 7·8161 (13), c = 25·420 (5) Å, V = 1251·6 (4) Å^3, Z = 4, D_x = 1·620 g cm^{-3}, Cu $K\alpha$ (λ = 1·54178 Å), μ = 26·195 cm^{-1}, $F(000)$ = 632, T = 295 K, R = 0·0282 for 2394 reflections ($F \geq 4\sigma_F$). The sugar conformation and puckering parameters are $_3T^2$ (C3′*exo*/C2′-*endo*), P = 192·2° and τ_m = 38·0°. The C5′—O5′ side-chain orientation is *gauche–trans* [φ_{OO} = 59·8 (2); φ_{CO} = 176·96 (15)°]. The glycosylic torsion angle (N2—N1—C1′—O4′) is 64·2 (2)°. The triazole ring is planar [r.m.s. deviation: 0·001 (2) Å]. The C3—C6—N7 group is linear and is 1·4° out of the triazole plane. All possible hydrogen donors (four) participate in hydrogen bonding [range of H···A distances is 2·14 (3)–2·40 (3) Å]. The sulfonamide amino group, which is above the furanose ring, is slightly nonplanar. The base moieties are perpendicular to the a axis and base stacking occurs *via* sandwiching of the carbonitrile groups between triazole rings of adjacent molecules along the 2_1 axes parallel to the a axis.

Experimental. The title compound (1) was prepared from 1-*β*-D-ribofuranosyl-1*H*-1,2,4-triazole-3-carbonitrile as previously described (Kini, Henry, Robins, Larson, Marr, Berens, Bacchi, Nathan & Keithly, 1990). Crystals grew from a warm methanol/ether solution as colorless, transparent needles. Table 1 summarizes data collection and refinement. Unit-cell parameters were obtained by least-squares refinement of the setting angles of 25 reflections with 55·0 < 2θ < 59·5°. An Enraf–Nonius CAD-4 diffractometer with a graphite monochromator was used for all measurements. Data reduction, which included Lorentz, polarization, decay and absorption corrections, was accomplished with the *SDP-Plus* software (Frenz, 1985). Crystal and instrument stability were monitored by remeasurement of three check reflections every hour. A linear fit of the intensities of these reflections was used to correct the data. Absorption corrections, based on crystal-face measurements, were applied. The function minimized was $\sum w(|F_o| - |F_c|)^2$, where $w^{-1} = (\sigma_F{}^2 + 0{\cdot}0004F^2)$, $\sigma_F = F\sigma_I/2I$, and $\sigma_I = (N_{pk} + N_{bg1} + N_{bg2})^{1/2}$.



(1)

Atomic coordinates for all 20 non-H atoms were obtained by direct methods [*SHELXS86* (Sheldrick, 1986)]. All H atoms were located in a difference map as peaks of density 0·44–0·91 e Å^{-3} at R = 0·046. All positional parameters, anisotropic thermal parameters for non-H atoms, isotropic thermal parameters for H atoms and an extinction parameter were refined with *SHELX76* (Sheldrick, 1976). Scattering factors and anomalous-dispersion corrections were taken from *International Tables for X-ray Crystallography* (1974) except those of H which were taken from Stewart, Davidson & Simpson (1965). Least-squares-planes program from Cordes (1983); figures were drawn with *ORTEP*II (Johnson, 1976); parameter and geometry tables were produced with *FUER*

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