gauche conformation, forming an intramolecular hydrogen bond between N(3) of the 1-methyladenine base and O(5′) of the sugar moiety with N···O 2·899 (4) Å. This type of hydrogen bond is found in many crystal structures of purine nucleosides with the *syn* conformation. On the other hand, the conformation about the C(4′)—C(5′) bond of molecule (*b*) is unusually *trans-gauche*. Both molecules have the sugar conformation as C(2′)-*endo* type. The pseudorotation parameters (Altona & Sundaralingam, 1972) are $P = 158{\cdot}4°$ (^2E), $\tau_m = 37{\cdot}4°$ for molecule (*a*) and $P = 182{\cdot}3°$ ($_3T^2$), $\tau_m = 36{\cdot}8°$ for molecule (*b*).

Fig. 2 shows the crystal structure projected down the *a* axis. The most pronounced feature of the crystal structure is the alternating parallel stacking of two independent purine bases to form columns parallel to the *a* axis. The characteristic of the stacking mode is the prominent overlapping of the pyrimidine moieties of 1-methyladenine rings. Such an extent of base overlap has not yet been observed in the unsubstituted adenine derivatives (Saenger, 1984). The base separation is about 3·4 Å and the dihedral angle 2·2 (1)°. Six crystallographically independent water molecules occupy the spaces between the columns and participate in many hydrogen bonds as shown in Fig. 2.

This work was partly supported by a Grant-in-Aid for Scientific Research from the Ministry of Education, Science and Culture, Japan.

References

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

ARNOTT, S. & HUKINS, D. W. L. (1972). *Biochem. J.* **130**, 453–465.

HAKOSHIMA, T., OMORI, H., TOMITA, K., MIKI, H. & IKEHARA, M. (1981). *Nucleic Acids Res.* **9**, 711–729.

International Tables for X-ray Crystallography (1974). Vol. IV, p. 99. Birmingham: Kynoch Press. (Present distributor D. Reidel, Dordrecht, The Netherlands.)

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

JONES, J. W. & ROBINS, R. K. (1963). *J. Am. Chem. Soc.* **85**, 193–201.

LANDNER, J. E., JACK, A., ROBERTUS, J. D., BROWN, R. S., RHODES, D., CLARK, B. F. C. & KLUG, A. (1975). *Proc. Natl Acad. Sci. USA*, **72**, 4414–4418.

NISHIMURA, S. (1978). *Transfer RNA*, edited by S. ALTMAN, pp. 168–195. Cambridge: MIT Press.

QUIGLEY, G. J., WANG, A. H. J., SEEMEN, N. C., SUDDATH, F. L., RICH, A., SUSSMAN, J. L. & KIM, S. H. (1975). *Proc. Natl Acad. Sci. USA*, **72**, 4866–4870.

SAENGER, W. (1984). *Principles of Nucleic Acid Structure*. New York: Springer-Verlag.

STOUT, C. D., MIZUNO, H., RAO, S. T., SWAMINATHAN, P., RUBIN, J., BRENNAN, T. & SUNDARALINGAM, M. (1978). *Acta Cryst.* B**34**, 1529–1544.

TAYLOR, R. & KENNARD, O. (1982). *J. Am. Chem. Soc.* **104**, 3209–3212.

The Universal Crystallographic Computation Program System–Osaka (1979). Library of Programs, Computing Center, Osaka Univ., Japan.

WATENPAUGH, K., DOW, J., JENSEN, L. H. & FURBERG, S. (1968). *Science*, **159**, 206–207.

YAMAGATA, Y., FUKUMOTO, S., HAMADA, K., FUJIWARA, T. & TOMITA, K. (1983). *Nucleic Acids Res.* **11**, 6475–6486.

Acta Cryst. (1987). C**43**, 2120–2123

Structure of Bis(thiobenzamidoethyl) Disulfide

BY N. GALEŠIĆ

'Ruđer Bošković' Institute, PO Box 1016, 41001 *Zagreb, Yugoslavia*

AND A. VLAHOV

Department of Organic Chemistry, Faculty of Technology, University of Zagreb, Marulićev trg 20, 41000 *Zagreb, Yugoslavia*

(*Received* 24 *February* 1987; *accepted* 1 *June* 1987)

Abstract. $C_{18}H_{20}N_2S_4$, $M_r = 392{\cdot}61$, triclinic, $P\bar{1}$, $a = 10{\cdot}370$ (4), $b = 12{\cdot}125$ (6), $c = 8{\cdot}765$ (4) Å, $\alpha = 99{\cdot}90$ (4), $\beta = 98{\cdot}98$ (4), $\gamma = 108{\cdot}23$ (5)°, $V = 1004{\cdot}9$ (8) Å^3, $Z = 2$, $D_m = 1{\cdot}30$, $D_x = 1{\cdot}297$ g cm^{-3}, λ(Mo $K\alpha$) = 0·7107 Å, $\mu = 4{\cdot}56$ cm^{-1}, $F(000) = 412$, $T = 293$ K, final $R = 0{\cdot}061$ for 2291 reflections. The molecule is the dimer PhC(S)NHCH$_2$CH$_2$S—SCH$_2$CH$_2$NHC(S)Ph bridged by an S—S bond of 2·042 Å. The torsion angle C—S—S—C is 81·5 (3)°, and the C—S—S angles are 104·3 (2) and 104·0 (2)°. The C(sp^3)—S bond lengths are 1·819 (6) and 1·813 (6) Å. The C(sp^2)—N bonds in the thioamide groups are 1·333 (6) and 1·342 (7) Å. The C=S bond lengths are 1·659 (7) and 1·681 (7) Å and related S atoms are involved in intermolecular N—H···S hydrogen bonds of 3·432 (6) and 3·531 (6) Å, respectively.

Introduction. For many years our attention has been directed towards the chemistry and biology of sulfur-containing compounds, especially amides and corresponding thioamides. These compounds are interesting because of their pharmacological activities. Among other applications, they are used as bactericides (Gardner, Wenis & Lee, 1954; Meltzer, Lewis & King, 1955), fungicides (Weuffen, Wagner, Singer & Petermann, 1966) as well as radiosensitizers in cancer chemotherapy (Millar, 1982).

We have extended our studies on these compounds using the X-ray diffraction method in order to obtain more information about this type of chemical structure, and the crystal structure of a compound of this series is presented.

Experimental. Crystals suitable for X-ray examination were obtained by recrystallization from petroleum ether (b.p. 353–373 K). D_m by flotation in a mixture of petroleum ether and CCl_4. Intensity data collected from a crystal defined by the following planes/distances (between opposite faces in mm): $\pm 010/0{\cdot}075$, $\pm 001/0{\cdot}083$, $\pm 0\bar{1}1/0{\cdot}068$, $\pm 5\bar{1}\bar{1}/0{\cdot}660$. Philips PW1100 four-circle diffractometer, θ–2θ scanning technique, scan width 1·20°, scan rate 0·04° s^{-1}. Unit-cell parameters obtained from least-squares analysis of 18 reflections with 2θ values ranging from 10 to 18°. No systematically absent reflections; structure determination confirmed $P\bar{1}$ space group. Out of 2897 reflections scanned within a hemisphere $\pm h$, $\pm k$, l ($h \pm 14$, $k \pm 16$, l 12) up to $(\sin\theta)/\lambda = 0{\cdot}70$ Å^{-1}, 2704 unique reflections classified as observed. Two standard reflections ($\bar{4}20$, 031) measured every 2 h showed an average variation of 1%. Corrections applied for Lorentz and polarization effects and for absorption (Harkema, 1978), although the absorption correction did not affect refinement (transmission factors from 0·95 to 0·97). Structure solved by direct methods (*MULTAN*80; Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1980). The E map calculated with 346 largest E values ($E \geq 1{\cdot}40$) revealed the positions of all non-H atoms except two C atoms belonging to different benzene rings. Subsequent calculations performed mainly with *XRAY*76 (Stewart, Machin, Dickinson, Ammon, Heck & Flack, 1976). A difference map revealed the two remaining C atoms. All H atoms located from difference maps and theoretical calculations. In final calculations seven relatively strong low-order reflections ($\bar{1}\bar{5}1$, $\bar{1}41$, $\bar{2}\bar{3}1$, $\bar{1}\bar{3}1$, $\bar{2}\bar{2}1$, $\bar{1}\bar{5}2$, $\bar{1}\bar{3}3$) affected by extinction were rejected.

Full-matrix least-squares refinement on F for 2291 remaining reflections with $I > 3\sigma(I)$. Parameters of non-H atoms varied anisotropically and those of H atoms isotropically and separately in the subsequent cycles (217 and 81 parameters refined, respectively); R and wR 0·061 and 0·069, respectively; function minimized $\sum w(|F_o| - |F_c|)^2$ with $w = 1{\cdot}00/\sigma^2(F_o)$ giving $S = 1{\cdot}82$. Max. and mean parameter shifts were <0·2 and 0·1, respectively. Final difference map revealed residuals between −0·36 and 0·54 e Å^{-3}. Scattering factors of Cromer & Mann (1968) for non-H and those of Stewart, Davidson & Simpson (1965) for H atoms; anomalous-dispersion factors from Cromer & Liberman (1970). All calculations performed on a Univac 1110 computer at the University Computing Centre in Zagreb.

Discussion. The final atomic parameters are listed in Table 1* and bond lengths and angles with several nonbonded intramolecular distances in Table 2. Fig. 1 shows an *ORTEP* diagram (Johnson, 1965) of the structure and the atomic numbering where corresponding atoms in the two halves of the molecule are denoted as $A(i)$ and $A(i + 10)$.

The molecule is a dimer $PhC(S)NHCH_2CH_2S$–$SCH_2CH_2NHC(S)Ph$ bridged by the S–S bond. Its length of 2·042 (2) Å, the torsion angles C–S–S–C of 81·5 (3) and C–C–S–S of 58·8 (4) and 60·5 (5)° as well as related $C(sp^3)$–S bond lengths of 1·819 (6) and 1·813 (6) Å and C–S–S angles of 104·3 (2) and 104·0 (2)° (Table 2) are very similar to the related values of DL-homocystine (Bigoli, Lanfranchi, Leporati, Nardelli & Pellinghelli, 1981).

The phenyl rings are planar within ±0·004 (7) and ±0·002 (7) Å. They are widely apart from one another (Fig. 1) and the angle between their mean planes is 15·4 (2)°. In the markedly folded conformation of bis(*o*-chlorophenylaminocarbonylmethyl) disulfide the two phenyl rings are close to each other making a dihedral angle of 151 (1)° (Pierrot, Baldy, Maire, Mehrotra, Kapoor & Bachlas, 1984). Both atom groups consisting of the thioamide C atoms and the atoms bonded to them show slight pyramidal arrangements with the thiamide C atoms in the apical positions. These pyramidal arrangements are of a minor significance as can be shown by the atom displacements from the corresponding mean planes: C(7) −0·010 (6), C(1) 0·003 (6), N(1) 0·004 (5), S(1) 0·003 (3) and C(17) 0·010 (6), C(11) −0·003 (7), N(11) −0·004 (6), S(11) −0·003 (3) Å. These groups are not coplanar with the related phenyl rings with which they share the C(1) and C(11) atoms, respectively. The angles between their mean planes and the corresponding phenyl mean planes are 41·1 (2) and 30·6 (2)°, respectively.

While the differences between corresponding bond lengths and angles in the two halves of the molecule are relatively small and insignificant (Table 2) the differences between corresponding torsion angles are greater. The greatest differences are between the torsion

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* Lists of structure factors, anisotropic thermal parameters, mean planes, selected torsion angles, H-atom parameters and bond lengths and angles involving H atoms have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 44136 (33 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 1. *Fractional positional parameters* (×10⁴) *and equivalent isotropic temperature factors* (×10²) *for non*-H *atoms*

$U_{eq} = \frac{1}{3}\sum_i\sum_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$

	x	y	z	U_{eq}(Å²)
S(1)	2477 (2)	−1777 (2)	600 (2)	5·24 (7)
S(2)	6612 (1)	398 (2)	4700 (2)	4·76 (7)
N(1)	4911 (4)	−1903 (4)	1844 (5)	4·0 (2)
C(1)	2971 (5)	−3459 (5)	2148 (6)	3·7 (2)
C(2)	1789 (6)	−4420 (6)	1256 (7)	4·7 (3)
C(3)	1258 (6)	−5395 (6)	1852 (9)	5·8 (3)
C(4)	1852 (7)	−5461 (6)	3328 (9)	6·0 (3)
C(5)	3022 (7)	−4514 (7)	4212 (8)	6·2 (3)
C(6)	3575 (6)	−3535 (6)	3631 (7)	5·0 (3)
C(7)	3526 (5)	−2379 (5)	1545 (6)	3·8 (3)
C(8)	5732 (5)	−812 (5)	1483 (7)	4·4 (3)
C(9)	5838 (5)	308 (5)	2651 (7)	4·2 (3)
S(11)	12883 (2)	3350 (2)	6975 (2)	5·84 (8)
S(12)	8542 (1)	322 (1)	4628 (2)	4·96 (7)
N(11)	10370 (4)	2596 (4)	7569 (6)	4·8 (2)
C(11)	12181 (6)	2543 (5)	9615 (7)	4·4 (3)
C(12)	13514 (6)	3210 (6)	10534 (8)	6·0 (3)
C(13)	13931 (8)	2920 (8)	11955 (9)	7·6 (4)
C(14)	13092 (10)	2029 (9)	12460 (9)	8·2 (5)
C(15)	11771 (8)	1369 (7)	11561 (9)	6·9 (4)
C(16)	11326 (6)	1640 (6)	10130 (8)	5·7 (3)
C(17)	11743 (5)	2829 (5)	8042 (7)	4·2 (3)
C(18)	9708 (5)	2781 (6)	6092 (7)	4·9 (3)
C(19)	9584 (5)	1839 (5)	4653 (7)	4·3 (2)

Table 2. *Pairs of analogous values* (X_1, X_2) *for bond lengths* (Å) *and angles* (°) *of non*-H *atoms and several nonbonded intramolecular distances* (Å) *in the two halves of the molecule with related* Δ/σ*'s*

$\Delta/\sigma = |X_1 - X_2|/[\sigma^2(X_1) + \sigma^2(X_2)]^{1/2}$ (Bigoli, Lanfranchi, Leporati, Nardelli & Pellinghelli, 1982).

C(1)—C(2)	1·403 (7)	C(11)—C(12)	1·394 (7)	0·9
C(1)—C(6)	1·383 (9)	C(11)—C(16)	1·370 (9)	1·0
C(2)—C(3)	1·367 (10)	C(12)—C(13)	1·393 (12)	1·7
C(3)—C(4)	1·370 (11)	C(13)—C(14)	1·348 (13)	1·3
C(4)—C(5)	1·385 (8)	C(14)—C(15)	1·378 (11)	0·5
C(5)—C(6)	1·368 (10)	C(15)—C(16)	1·396 (11)	1·9
C(1)—C(7)	1·479 (9)	C(11)—C(17)	1·515 (9)	2·8
C(7)—S(1)	1·681 (7)	C(17)—S(11)	1·659 (7)	2·2
C(7)—N(1)	1·333 (6)	C(17)—N(11)	1·342 (7)	1·0
N(1)—C(8)	1·448 (8)	N(11)—C(18)	1·455 (9)	0·6
C(8)—C(9)	1·516 (9)	C(18)—C(19)	1·509 (9)	0·6
C(9)—S(2)	1·819 (6)	C(19)—S(12)	1·813 (6)	0·7
S(2)—S(12)	2·042 (2)			
C(2)—C(1)—C(6)	118·0 (6)	C(12)—C(11)—C(16)	119·7 (6)	2·0
C(2)—C(1)—C(7)	120·9 (5)	C(12)—C(11)—C(17)	118·1 (6)	3·6
C(6)—C(1)—C(7)	121·0 (4)	C(16)—C(11)—C(17)	122·1 (5)	1·7
C(1)—C(2)—C(3)	120·3 (6)	C(11)—C(12)—C(13)	118·0 (6)	2·7
C(2)—C(3)—C(4)	121·4 (5)	C(12)—C(13)—C(14)	122·3 (6)	1·2
C(3)—C(4)—C(5)	118·4 (7)	C(13)—C(14)—C(15)	120·0 (8)	1·5
C(4)—C(5)—C(6)	121·0 (6)	C(14)—C(15)—C(16)	118·9 (7)	2·3
C(1)—C(6)—C(5)	120·8 (5)	C(11)—C(16)—C(15)	121·1 (5)	0·4
C(1)—C(7)—S(1)	122·0 (4)	C(11)—C(17)—S(11)	121·9 (4)	0·2
C(1)—C(7)—N(1)	115·2 (5)	C(11)—C(17)—N(11)	114·9 (5)	0·4
S(1)—C(7)—N(1)	122·7 (5)	S(11)—C(17)—N(11)	123·1 (5)	0·6
C(7)—N(1)—C(8)	127·0 (5)	C(17)—N(11)—C(18)	124·7 (5)	3·3
N(1)—C(8)—C(9)	113·3 (5)	N(11)—C(18)—C(19)	113·1 (6)	0·3
C(8)—C(9)—S(2)	114·3 (5)	C(18)—C(19)—S(12)	115·8 (4)	2·3
C(9)—S(2)—S(12)	104·3 (2)	C(19)—S(12)—S(2)	104·0 (2)	1·1
S(1)···C(1)	2·766 (7)	S(11)···C(11)	2·775 (7)	0·9
S(1)···C(2)	3·231 (7)	S(11)···C(12)	3·130 (8)	9·5
S(1)···C(6)	3·927 (8)	S(11)···C(16)	3·978 (8)	4·5

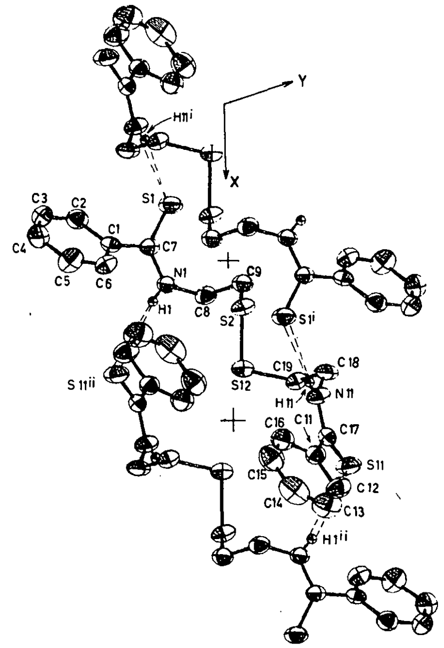

Fig. 1. Diagram of the structure viewed along **b** showing the hydrogen-bonding scheme and the atom numbering [symmetry codes: (i) $1-x, -y, 1-z$; (ii) $2-x, -y, 1-z$]. For clarity only the H atoms attached to N atoms are represented. The symmetry centres at $\frac{1}{2}, 0, \frac{1}{2}$ and $1, 0, \frac{1}{2}$ are denoted by +.

angles about the C(1)—C(7) and C(11)—C(17) bonds with Δ/σ's ranging from 8·5 to 12·5.*

It seems that two different N—H···S contacts could be weak hydrogen bonds (Fig. 1) by which the molecules are connected in the chains stretching parallel to (100): N(1)—H(1)···S(11ⁱⁱ) of 3·432 (6) and N(11)—H(11)···S(1ⁱ) of 3·531 (6) Å with H···S separations of 2·68 (5) and 2·74 (4) Å and N—H···S angles of 164 (4) and 161 (4)°, respectively.

The authors thank M. Bruvo MSc for collecting the diffractometric data. This investigation was supported by the Research Council of Croatia.

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* See Table 5 of the deposited material.

References

Bigoli, F., Lanfranchi, M., Leporati, E., Nardelli, M. & Pellinghelli, M. A. (1981). *Acta Cryst.* B**37**, 1258–1265.

Bigoli, F., Lanfranchi, M., Leporati, E., Nardelli, M. & Pellinghelli, M. A. (1982). *Acta Cryst.* B**38**, 498–502.

Cromer, D. T. & Liberman, D. (1970). *J. Chem. Phys.* **53**, 1891–1898.

Cromer, D. T. & Mann, J. B. (1968). *Acta Cryst.* A**24**, 321–324.

Gardner, T. S., Wenis, E. & Lee, J. (1954). *J. Org. Chem.* **19**, 753–757.

Harkema, S. (1978). *ACXR*. Absorption program for use in connection with a PW 1100 diffractometer. Proceedings of an International Summer School on Crystallographic Computing, Twente, The Netherlands – Working Sessions and Program Market, p. 28.

Johnson, C. K. (1965). *ORTEP*. Report ORNL-3794. Oak Ridge National Laboratory, Tennessee, USA.
Main, P., Fiske, S. J., Hull, S. E., Lessinger, L., Germain, G., Declercq, J.-P. & Woolfson, M. M. (1980). *MULTAN80. A System of Computer Programs for the Automatic Solution of Crystal Structures from X-ray Diffraction Data*. Univs. of York, England, and Louvain, Belgium.
Meltzer, R. I., Lewis, A. D. & King, J. A. (1955). *J. Am. Chem. Soc.* **77**, 4062–4066.
Millar, B. G. (1982). *Biochem. Pharmacol.* **31**, 2439–2445.
Pierrot, M., Baldy, A., Maire, C., Mehrotra, R. C., Kapoor, T. S. & Bachlas, B. P. (1984). *Acta Cryst.* C**40**, 1931–1934.
Stewart, J. M., Machin, P. A., Dickinson, C. W., Ammon, H. L., Heck, H. & Flack, H. (1976). The *XRAY76* system. Tech. Rep. TR-446. Computer Science Center, Univ. of Maryland, College Park, Maryland, USA.
Stewart, R. F., Davidson, E. R. & Simpson, W. T. (1965). *J. Chem. Phys.* **42**, 3175–3187.
Weuffen, W., Wagner, G., Singer, D. & Petermann, M. (1966). *Pharmazie*, **21**, 613–619.

Acta Cryst. (1987). C**43**, 2123–2125

Structure of Thiamin Disulfide Dinitrate

By Whanchul Shin* and Kwon Soo Chun

Department of Chemistry, College of Natural Sciences, Seoul National University, Seoul 151, *Korea*

(*Received* 16 *January* 1987; *accepted* 23 *June* 1987)

Abstract. 4,4′-Diamino-2,2′-dimethyl-5,5′-(dithiobis-{[2-(2-hydroxyethyl)-1-methylvinylene](formylimino)-methylene})dipyrimidinium dinitrate, $C_{24}H_{36}N_8O_4S_2^{2+}.2NO_3^-$, M_r = 688·76, monoclinic, $C2/c$, a = 11·064 (2), b = 12·715 (3), c = 22·858 (4) Å, β = 96·03 (1)°, V = 3198 (1) Å^3, Z = 4, D_x = 1·431 g cm^{-3}, λ(Cu $K\alpha$) = 1·5418 Å, μ = 19·87 cm^{-1}, $F(000)$ = 1448, T = 295 K, R = 0·065 (wR = 0·068) for 1366 reflections with $I > 3\sigma(I)$. Each half of the thiamin disulfide cation is related by a crystallographic twofold axis bisecting the disulfide bond. The two pyrimidine rings face each other with a dihedral angle of 4·1° so that the cation assumes a folded conformation. This conformation is stabilized in part by an intramolecular N(4′α)—H···O(2α) hydrogen bond [2·995 (6) Å]. The salt is stabilized by a hydrogen bond between O(5γ) and the nitrate O [2·801 (7) Å]. Hydrogen-bonding interactions form two-dimensional molecular layers and there are only C—H···O interactions between these layers. As observed in other ring-opened disulfide derivatives of thiamin, the *N*-formyl and ethylenic planes are perpendicular to each other so that the N(3)—C(4) bond remains an easily rotatable single bond.

Introduction. Thiamin (vitamin B_1) is labile against acid, alkali and heat (Dwidevi & Arnold, 1973). The thiazolium ring in thiamin is easily hydrolyzed in mildly alkaline solution to give various thiol or disulfide derivatives which, in turn, can easily be converted to thiamin upon acidification (Hopmann, 1982). Some thiamin disulfide derivatives such as thiamin tetrahydrofurfuryl disulfide (TTFD) and thiamin propyl disulfide (TPD) are used as therapeutic provitamins due to high absorptivity in the gastrointestinal tract (Suzuoki, Nishikawa & Numata, 1965). Thiamin disulfide is one of the open-ring derivatives which is frequently used as a starting material for syntheses of other thiamin derivatives. Among the many thiamin disulfide derivatives, only the crystal structures of TTFD (Shin & Kim, 1986) and TPD (Nishikawa, Kamiya, Asahi & Matsumura, 1969) have been reported to date. In an effort to obtain information on the structural characteristics of this class of compounds, the crystal structure of thiamin disulfide dinitrate (TDD) has been determined.

Experimental. Colorless tabular crystals obtained from an acetone–ethanol solution (1:1 v/v) of TDD (Sigma) by slow evaporation at room temperature; crystal *ca* 0·2 × 0·5 × 0·6 mm, Rigaku AFC diffractometer, graphite-monochromated Cu $K\alpha$ radiation, $2\theta < 122°$, ω–2θ scan, scan speed 4° min^{-1} in 2θ, ω-scan width $(1·3 + 0·4\tan\theta)°$, background measured for 12 s on either side of the peak; cell parameters by least-squares fit to observed 2θ values for 24 centred reflections with $22 < 2\theta < 50°$; intensity checks for three standard reflections showed little (±0·6%) variation; 2002 independent reflections (h −11 to 11, k 0 to 13, l 0 to 24), 1366 (68·2%) observed with $I > 3\sigma(I)$ and used in refinement; Lp corrections, no absorption or extinction corrections. Structure solved by direct methods with *MITHRIL* (Gilmore, 1984) and refined with *SHELX76* (Sheldrick, 1976) by full-matrix least squares on F with anisotropic thermal parameters; H atoms identified on a difference map and refined

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* To whom correspondence should be addressed.

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