achievable but remains larger than 3·0 Å, still far too large to form a stable hydrogen bond.

Using the method of Cremer & Pople (1975), we calculated almost equal phase angles $\varphi_2 = -61$ (7) (I) or -70 (13)° (II) and $\theta_2 = 177{\cdot}7$ (2) (I) or 175·5 (9)° (II), with total puckering amplitudes $Q =$ 0·563 (2) (I) or 0·56 (1) Å (II) for the sequences C(1′)—C(2′)—O(3′)—C(4′)—C(5′)—O(5′).

The packing in both crystals is partly determined by hydrogen bonds involving atoms of the dioxane and base rings (Table 4 summarizes all the intermolecular hydrogen bonds) and partly by parallel base-stacking forces. *PLUTO* plots of the crystal packings are shown in Fig. 2. In structure (I), two different stacking patterns are observable: (1) the closest stacking [$d_{\rm mean} = 3{\cdot}4$ (2) Å] is found between pairs of bases related to each other by the twofold axis along *i* [bases *A* and *B* in Fig. 2(*a*); dihedral angle between the two bases = 10·80 (1)°]; (2) the other pattern [$d_{\rm mean} = 3{\cdot}6$ (2) Å] is found between bases which are related by the twofold axis lying along *a* or *b* [bases *A* and *C* in Fig. 2(*a*); dihedral angle = 12·86 (1)°]. The stacking patterns for both observations are similar: the rings are only partially overlapped and the carbonyl groups and the ring N atoms often form close contacts with adjacent bases while the C atoms are less involved. In structure (II) only Br(5) is positioned in close contact with the adjacent base and is located above the center of the ring [$d_{\rm mean} = 3{\cdot}31$ (1) Å].

The authors thank J. P. Van Cuyck for his help in preparing the pictures.

References

Allen, F. H., Kennard, O., Watson, D. G., Brammer, L., Orpen, A. G. & Taylor, R. (1987). *J. Chem. Soc. Perkin Trans.* 2, pp. S1–S19.

Cremer, D. & Pople, J. A. (1975). *J. Am. Chem. Soc.* **97**, 1354–1358.

De Winter, H. L., Blaton, N. M., Peeters, O. M., De Ranter, C. J., Van Aerschot, A. & Herdewijn, P. (1991). *Acta Cryst.* C**47**, 2245–2247.

Frenz, B. A. (1985). *Enraf–Nonius Structure Determination Package.* College Station, Texas, USA, and Enraf–Nonius Delft, The Netherlands.

Ibers, J. A. & Hamilton, W. C. (1964). *Acta Cryst.* **17**, 781–782.

Main, P., Fiske, S., Hull, S. E., Lessinger, L., Germain, G., Declercq, J.-P. & Woolfson, M. M. (1982). *MULTAN82. A System of Computer Programs for the Automatic Solution of Crystal Structures from X-ray Diffraction Data.* Univs. of York, England, and Louvain, Belgium.

Motherwell, W. D. S. & Clegg, W. (1978). *PLUTO.* Program for plotting molecular and crystal structures. Univ. of Cambridge, England.

Nardelli, M. (1983). *Comput. Chem.* **7**, 95–98.

Nyburg, S. C. (1974). *Acta Cryst.* B**30**, 253–254.

Saenger, W. (1988). *Principles of Nucleic Acid Structure.* New York: Springer-Verlag.

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

Stoe & Co. (1985). *REDU4. Data Reduction Program.* Stoe & Co., Darmstadt, Germany.

Van Aerschot, A., Janssen, G. & Herdewijn, P. (1990). *Bull. Soc. Chim. Belg.* **99**, 769–777.

Acta Cryst. (1991). C**47**, 2423–2425

Structure of the *cis* Isomer of a Six-Membered Phosphorus Phenylhydrazine Ring

By B. Wallis, Ch. Donath and M. Meisel

Institut für Anorganische Chemie, Rudower Chaussee 5, O-1199 Berlin, Germany

and J. Fuchs

Institut für Anorganische und Analytische Chemie der Freien Universität Berlin, Fabeckstrasse 34-36, W-1000 Berlin 33, Germany

(*Received* 17 *April* 1991; *accepted* 29 *May* 1991)

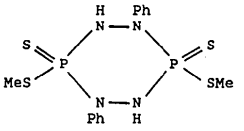
Abstract. 3,6-Bis(methylthio)-1,4-diphenyl-3,6-dithioxo-1,2,4,5-tetraaza-3λ^5,6λ^5-diphosphorinane crystallizes with two solvent molecules of acetonitrile, $C_{14}H_{18}N_4P_2S_4.2CH_3CN$, $M_r = 514{\cdot}64$, monoclinic, $C2/c$, $a = 12{\cdot}118$ (8), $b = 13{\cdot}452$ (10), $c =$ 15·608 (4) Å, $\beta = 98{\cdot}22$ (4)°, $V = 2518$ Å^3, $Z = 4$, D_x = 1·353 Mg m^{-3}, λ(Mo $K\alpha$) = 0·71069 Å, $\mu =$ 4·64 mm^{-1}, $F(000) = 1072$, $T = 293$ K, $R = 0{\cdot}038$ for 2513 unique observed reflections with $I \geq 2\sigma(I)$. The molecules of the title compound have point symmetry 2. In accordance with the *cis* stereochemistry of the methylthio substituents the six-membered ring adopts a twist conformation; torsion angles PNNP +67·87 (3), NNPN −45·81 (3) and NPNN −19·73 (1)°.

Introduction. To extend our knowledge of the preparative potential of py.PS_2Cl (1) (py = pyridine), the

0108-2701/91/112423-03$03.00 © 1991 International Union of Crystallography

reaction of this compound with substituted hydrazines has been studied (Donath, Meisel & Pauli, 1991). According to the general reaction behaviour of (1) with nucleophilic agents (Meisel, Donath & Grunze, 1981) and especially with primary amines (Donath & Meisel, 1987), the formation of phosphorus–nitrogen ring systems should also be expected with hydrazines.

By reaction of (1) with PhNH—NH_2 in the presence of NEt_3 and following alkylation of the reaction mixture by methyl iodide, the six-membered title compound (2) can be obtained as a minor product after separation by column chromatography.



(2)

Experimental. Crystals were obtained by recrystallization from MeCN; a prismatic specimen (0·5 × 0·4 × 0·3 mm) was mounted in a glass capillary. Stoe four-circle diffractometer (ω scan), 39 reflections for refining lattice parameters, empirical absorption correction (min. and max. factors 0·847 and 1·113, respectively) with *DIFABS* (Walker & Stuart, 1983), $(\sin\theta_{max})/\lambda = 0{\cdot}6386$ Å^{-1}, $-18 \le h \le 18$, $0 \le k \le 22$, $-25 \le l \le 25$. Two standard reflections measured every 90 min without any significant variation during data collection, 4617 reflections measured, 2752 unique ($R_{int} = 0{\cdot}0408$), 239 were considered as unobserved with $I < 2\sigma(I)$. Structure solved by direct methods (*SHELXS*86; Sheldrick, 1986), H atoms located by difference Fourier syntheses, full-matrix least-squares refinement (on F) with anisotropic temperature factors for non-H atoms, isotropic factors for H atoms. Final $R = 0{\cdot}038$, $wR = 0{\cdot}031$, weighting scheme $w = 1/\sigma(F_o^2)$, $\Delta/\sigma_{max} = 0.27$, max. electron density in the final difference Fourier map $\le$ 0·3 e Å^{-3}. Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV). Computer program used: *XTAL*2.4 (Hall & Stewart, 1988).

Discussion. Final atomic coordinates and equivalent isotropic thermal parameters are given in Table 1.* Selected interatomic distances and bond angles are reported in Table 2.

* Lists of structure factors, anisotropic thermal parameters, interatomic distances and bond angles and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 54290 (43 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 1. *Atomic coordinates* (× 10^4) *and equivalent isotropic thermal parameters* (Å^2 × 10^3)

$U_{eq} = (U_{11} + U_{22} + U_{33})/3.$

	x	y	z	U_{eq}
P(1)	1298·0 (4)	2894·5 (4)	2280·3 (3)	32·2
S(1)	1759·6 (5)	4359·6 (4)	2509·6 (4)	43·7
S(2)	2411·3 (5)	2004·3 (4)	1967·0 (4)	49·1
N(1)	0093 (1)	3036 (1)	1593·6 (9)	34·4
N(2)	0823 (1)	2512 (1)	3162 (1)	35·1
C(1)	3124 (3)	4186 (3)	3114 (3)	70
C(11)	−0016 (2)	3327 (1)	0704 (1)	33
C(12)	0878 (2)	3743 (2)	0374 (1)	41
C(13)	0763 (2)	4001 (2)	−0496 (1)	49
C(14)	−0217 (2)	3851 (2)	−1030 (1)	55
C(15)	−1108 (2)	3458 (2)	0696 (1)	54
C(16)	−1014 (2)	3195 (2)	0169 (1)	45
C(1)*A**	4084 (2)	4684 (2)	0810 (2)	61
C(2)*A**	4010 (4)	3687 (2)	0452 (3)	76
N(3)*A**	4148 (2)	5448 (2)	1082 (2)	96

* Acetonitrile molecule

Table 2. *Selected bond distances* (Å) *and angles* (°)

P(1)—S(1)	2·066 (2)	S(1)—C(1)	1·798 (3)
P(1)—S(2)	1·919 (1)	N(1)—N(2^{i})	1·412 (2)
P(1)—N(1)	1·694 (2)	N(1)—(C11)	1·431 (2)
P(1)—N(2)	1·648 (2)		
Phenyl group C—C (mean) 1·38			
S(1)—P(1)—S(2)	117·2 (1)	P(1)—N(1)—C(11)	126·6 (1)
S(1)—P(1)—N(1)	100·9 (1)	P(1)—N(1)—N(2^{i})	114·3 (1)
S(2)—P(1)—N(1)	118·9 (1)	C(11)—N(1)—N(2^{i})	115·6 (1)
S(2)—P(1)—N(2)	111·1 (1)	P(1)—N(2)—N(1^{i})	116·8 (1)
S(1)—P(1)—N(2)	105·8 (1)	P(1)—S(1)—C(1)	99·9 (1)
Acetonitrile molecule			
C(1)—C(2)	1·450 (4)	C(1)—N(3)	1·111 (4)
N(3)—C(1)—C(2)	179·5 (4)		

Symmetry code: (i) $-x, y, \frac{1}{2} - z$.

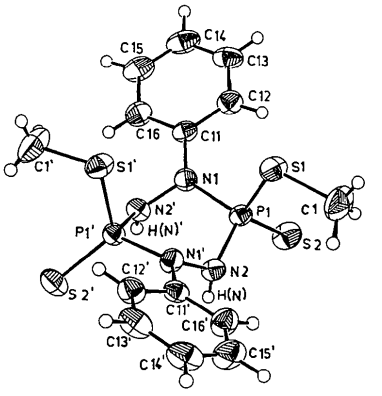


Fig. 1. Molecular geometry of the title compound. Thermal ellipsoids are drawn at 50% probability level. The twofold axis is perpendicular to the ring.

Fig. 1 shows the numbering of the atoms and the molecular structure of the title compound. The P_2N_4 ring has a distorted twist conformation as can be seen by comparison with the respective torsion angles of the ideal twist form of cyclohexane: −70·6

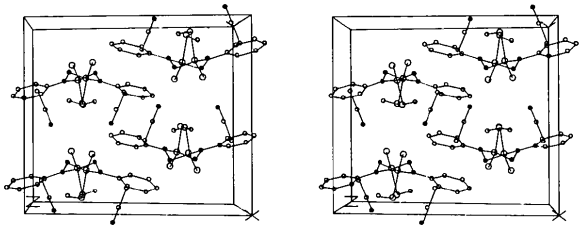


Fig. 2. Arrangement of the molecules (H atoms deleted) in the unit cell, viewed along x. Stereoplot drawn with *CELLGRAPH* (Reck & Kretschmar, 1989).

and two times +33·2° (Hendrickson, 1961). The pseudoaxial substituents at the P atoms are arranged in *cis* positions with respect to the P_2N_4 ring. The angle between the planar phenyl ring and the least-squares plane through the P_2N_4 ring is 23·6°.

The unit cell (Fig. 2) contains two pairs of molecules which are symmetry related by a glide plane. The shortest intermolecular distances exist between phenyl groups [C(12)···C(14^{ii}) 3·521 Å] and between a phenyl group and the methyl group of the acetonitrile molecule [C(12)···C(2^{iii}) 3·524 Å] [(ii) $-x$, $1-y$, $-z$; (iii) $\frac{1}{2}-x$, $\frac{1}{2}-y$, $-z$]. These distances do not indicate interactions stronger than van der Waals forces.

The comparatively short N—N bond length together with the sum of the bond angles around N(1) (356·5°) shows that the bonding state at the N(1) atom tends to sp^2 hybridization. The shortest N—N distance in a phosphorus hydrazine heterocycle (1.400 Å) has been found in *cis*-3,6-dithioxo-3,6-diphenoxy-1,2,4,5-tetraaza-3λ^5,6λ^5-diphosphacyclohexane (Engelhardt & Hartl, 1976), which has an angular sum of 359·3°.

References

Donath, Ch. & Meisel, M. (1987). *Phosphorus Sulfur*, **30**, 451–454.

Donath, Ch., Meisel, M. & Pauli, J. (1991). In preparation.

Engelhardt, U. & Hartl, H. (1976). *Acta Cryst.* **B32**, 1133–1138.

Hall, S. R. & Stewart, J. M. (1988). Editors. *XTAL2.4 Users Manual.* Univs. of Western Australia, Australia, and Maryland, USA.

Hendrickson, J. B. (1961). *J. Am. Chem. Soc.* **83**, 4537–4547.

Meisel, M., Donath, Ch. & Grunze, H. (1981). *ACS Symp. Ser.* **171**, 161–164.

Reck, G. & Kretschmar, R. G. (1989). *CELLGRAPH.* Computer program for illustration of organic and inorganic crystal structures. Analytisch Zentrum Berlin-Adlershof, Germany.

Sheldrick, G. M. (1986). *SHELXS86.* Program for the solution of crystal structures. Univ. of Göttingen, Germany.

Walker, N. & Stuart, D. (1983). *Acta Cryst.* **A39**, 158–166.

Acta Cryst. (1991). **C47**, 2425–2428

Structure of 1,2,3,4,5,6-Hexa-*O*-acetyl-D-glucitol (Sorbitol Hexaacetate)

By Jürgen Kopf,* Cornelia Topf, Martina Morf and Bärbel Zimmer

Institute of Inorganic and Applied Chemistry, University of Hamburg, Martin-Luther-King-Pl. 6, *D*-2000 *Hamburg* 13, *Germany*

and Peter Köll

Department of Chemistry, Organic Chemistry, University of Oldenburg, Carl-von-Ossietzky-Str. 9–11, *D*-2900 *Oldenburg, Germany*

(*Received* 12 *February* 1991; *accepted* 23 *April* 1991)

Dedicated to Professor Dr E. Weiß on the occasion of his 65th birthday

Abstract. $C_{18}H_{26}O_{12}$, M_r = 434·40, monoclinic, $P2_1$, a = 10·253 (1), b = 8·370 (1), c = 12·548 (1) Å, β = 95·98 (5)°, V = 1071·0 (2) Å^3, Z = 2, D_x = 1·347 g cm^{-3}, λ(Mo $K\alpha_1$) = 0·709261 Å, μ = 1·1 cm^{-1}, $F(000)$ = 460, T = 293 K, R = 0·053 for 1681 observed reflexions. The molecules of the title compound have a planar zigzag carbon-chain conformation which aligns O(2) 1,3-parallel to O(4). The primary O(1) is found in the same O//O relation to O(3).

Introduction. The X-ray structures of many alditols (sugar alcohols) up to a chain length of C_7 have been determined. Generally, planar (zigzag) conformations are expected but in most cases they are found in bent (sickle) carbon-chain conformations in order to avoid unfavorable 1,3-parallel interactions of C and O atoms (designated C//O and O//O). The

* To whom correspondence should be addressed.

0108-2701/91/112425-04$03.00 © 1991 International Union of Crystallography