The crystal packing is illustrated in Fig. 2. The molecules are held together by normal van der Waals interactions. No intermolecular contact is shorter than the sum of the appropriate van der Waals radii.

References

ALTONA, C. & SUNDARALINGAM, M. (1970). *Tetrahedron*, **26**, 925–939.

BHATTACHARJEE, S. K. & CHACKO, K. K. (1979). *Tetrahedron*, **35**, 1999–2007.

BUCHANAN, G. L., KITSON, D. H., MALLINSON, P. R., SIM, G. A., WHITE, D. N. J. & COX, P. J. (1983). *J. Chem. Soc. Perkin Trans.* 2, pp. 1709–1712.

CHOI, C. S. & BULUSU, S. (1974). *Acta Cryst.* B**30**, 1576–1580.

CHOI, C. S., SANTARO, A. & ABEL, J. E. (1976). *Acta Cryst.* B**32**, 354–358.

HARTUNG, H., BAUMEISTER, U., RAUCH, H. & KRECH, F. (1988). *Acta Cryst.* C**44**, 1438–1440.

HARTUNG, H., HICKEL, S., KAISER, J. & RICHTER, R. (1979). *Z. Anorg. Allg. Chem.* **458**, 130–138.

JASKÓLSKI, M. (1980). *PRARA*. Program for data reduction from Syntex data tapes. Univ. of Poznań, Poland.

JASKÓLSKI, M. (1982). *EDIT*. Procedure to edit atomic information from atom data files. Univ. of Poznań, Poland.

KAISER, J., RICHTER, R. & HARTUNG, H. (1978). *Tetrahedron*, **34**, 1993–1997.

KRECH, F. & ISSLEIB, K. (1976). *Z. Anorg. Allg. Chem.* **425**, 209–216.

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.

MASTRYUKOV, V. S., OSINA, E. L., DOROFEEVA, O. V., POPIK, M. V., VILKOV, L. V. & BELIKOVA, N. A. (1979). *J. Mol. Struct.* **52**, 211–224.

MASTRYUKOV, V. S., POPIK, M. V., DOROFEEVA, O. V., GOLUBINSKII, A. V., VILKOV, L. V., BELIKOVA, N. A. & ALLINGER, N. L. (1981). *J. Am. Chem. Soc.* **103**, 1333–1337.

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

SIM, G. A. (1983). *Tetrahedron*, **39**, 1181–1185.

SKANCKE, P. N. (1987). *J. Mol. Struct.* **151**, 11–14.

ZEFIROV, N. S., ROGOZINA, S. V., KURKUTOVA, E. H., GONCHAROV, A. V. & BELOV, N. V. (1974). *J. Chem. Soc. Chem. Commun.* pp. 260–261.

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1,5-Diphosphabicyclo[3.3.1]nonane 1,5-Disulfide

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Abstract. $C_7H_{14}P_2S_2$, $M_r = 224{\cdot}2$, monoclinic, $P2_1/c$, $a = 11{\cdot}021\,(2)$, $b = 8{\cdot}055\,(1)$, $c = 12{\cdot}195\,(3)$ Å, $\beta = 90{\cdot}50\,(2)°$, $V = 1082{\cdot}6\,(4)$ Å^3, $Z = 4$, $D_m = 1{\cdot}36$ (flotation in aqueous KI solution), $D_x = 1{\cdot}38$ Mg m^{-3}, $\lambda(\text{Mo } K\alpha) = 0{\cdot}71069$ Å, $\mu = 0{\cdot}66$ mm^{-1}, $F(000) = 472$, $T = 293$ K, final $R = 0{\cdot}032$ ($wR = 0{\cdot}030$) for 1224 observed reflections. The 1,5-diphosphabicyclo[3.3.1]nonane 1,5-disulfide molecule has a chair–chair conformation and (in very good approximation) C_{2v} (*mm*) symmetry. The strong repulsion between the *endo*-methylene groups is reflected in the transannular C···C distance of 3·296 (4) Å and causes a marked flattening of the diphosphorinane rings.

Introduction. Recently the bicyclo[3.3.1]nonane system has been the subject of much theoretical and experimental work showing its relevance to chemical synthesis and stereochemistry. In the preceding paper (Baumeister, Hartung & Krech, 1988) we report the results of an X-ray analysis of 1-phosphabicyclo[3.3.1]nonane 1-sulfide. The main interest in the molecular structure of this compound was directed to the substitution of one bridgehead C atom by a P atom and its influence on the stereochemistry of the bicyclic skeleton. We now present the crystal structure of the closely related compound 1,5-diphosphabicyclo[3.3.1]nonane 1,5-disulfide in which both bridgehead positions are occupied by P atoms.

Experimental. 1,5-Diphosphabicyclo[3.3.1]nonane 1,5-disulfide was first synthesized by Krech (1985). A crystal with dimensions 0·22 × 0·31 × 0·35 mm was investigated on a Syntex $P2_1$ diffractometer using graphite-monochromatized Mo $K\alpha$ radiation. Lattice parameters were derived from least-squares refinement of the setting angles of 15 reflections. Intensities for 1595 unique reflections with $2 \leq 2\theta \leq 48°$ were measured in the *h,k,l* range $\overline{12}$,0,0 to 12,9,13, 1224 with $I \geq 1{\cdot}96\sigma(I)$ were considered to be observed. The check reflection intensities varied from 4·44 (5) to 4·57 (5) × 10^4 (=2·8%) for $\bar{1}35$ and from 10·9 (1) to 11·5 (1) × 10^4 (=4·9%) for $\bar{2}33$. The structure was solved by standard Patterson and Fourier techniques and refined (on *F*) by full-matrix least squares with anisotropic thermal parameters for the non-H atoms

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and isotropic ones for H atoms (all found in a difference Fourier map). The weighting scheme was $w = 1/\sigma^2(F_o)$, $\sigma(F_o)$ based on counting statistics. An empirical secondary-extinction correction was performed with $F_{\rm corr} = F_c\,(1 - 10^{-7}\chi F_c^2/\sin\theta)$, where χ refined to 8·8 (2). The refinement converged to $R = 0{\cdot}032$ ($wR = 0{\cdot}030$). Further details of refinement: 7·8 reflections/parameter; in final cycle $\Delta/\sigma \leq 0{\cdot}002$; max. and min. heights in final $\Delta\rho$ map +0·25 and −0·29 e Å^{-3}, respectively; atomic scattering factors those of *SHELX*76 (Sheldrick, 1976).

All calculations were performed on an ESER 1040 computer of the Halle University using programs *SHELX*76, *PRARA* (Jaskólski, 1980) and *EDIT* (Jaskólski, 1982).

Discussion. Final atomic parameters are listed in Table 1.* The essential structural and conformational features of the 1,5-diphosphabicyclo[3.3.1]nonane 1,5-disulfide molecule are given in Fig. 1 and Table 2. The molecule adopts a chair–chair conformation and has a very well approximated (but not crystallographically imposed) C_{2v} (*mm*) symmetry. The diphosphorinane rings are distorted from ideal cyclohexane geometry [characterized by a C—C—C angle of 111·0° and a torsion angle close to 56° (Altona & Sundaralingam, 1970)] by outward displacement of the C(2) and C(5) methylene groups. Atom C(2) is deviated from the best plane through P(1), C(1), C(3), P(2) (plane I) by −0·565 (5) Å and atom C(5) from the plane through P(1), C(4), C(6), P(2) (plane II) by −0·574 (5) Å, but the deviations of atom C(7) from these planes are 0·858 (4) and 0·847 (4) Å, respectively. The interplanar angle I/II is 111·2°. The flattening of the diphosphorinane rings can also be visualized in terms of the corresponding endocyclic torsion angles (Table 2). They have mean values of 54·4 (30)° for ring *A* [C(1)...C(7), P(1)] and 54·1 (29)° for ring *B* [C(4)... C(7), P(2)] and are significantly reduced for the C—C—C wings compared with the P—C—P wing of the bicyclic skeleton. This appreciable flattening is caused by the repulsion between the C(2) and C(5) methylene groups. In a hypothetical chair–chair conformer of bicyclo[3.3.1]nonane composed of ideal cyclohexane rings, the C(2)···C(5) separation would be *ca* 2·5 Å and the shortest transannular H···H contact distance would have the physically impossible value of *ca* 0·75 Å (Sim, 1983). The C(2)···C(5) and H(22)··· H(51) separations in 1,5-diphosphabicyclo[3.3.1]nonane 1,5-disulfide are 3·296 (6) and 2·01 (6) Å, respectively. These values are somewhat greater than

Table 1. *Positional parameters and equivalent isotropic temperature factors* (Å^2) *of non*-H *atoms with e.s.d.'s in parentheses*

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

	x	y	z	$U_{\rm eq}$
P(1)	0·33580 (8)	0·13381 (13)	0·16731 (8)	0·0329 (3)
P(2)	0·09320 (8)	−0·02740 (12)	0·18630 (8)	0·0328 (3)
S(1)	0·43411 (9)	0·31619 (14)	0·11015 (9)	0·0495 (4)
S(2)	−0·08263 (9)	−0·02134 (14)	0·15737 (9)	0·0447 (3)
C(1)	0·3342 (4)	0·1257 (6)	0·3155 (3)	0·038 (2)
C(2)	0·2585 (4)	−0·0102 (6)	0·3700 (3)	0·041 (2)
C(3)	0·1255 (4)	−0·0211 (6)	0·3327 (3)	0·039 (1)
C(4)	0·1683 (4)	−0·2034 (5)	0·1238 (4)	0·042 (2)
C(5)	0·3066 (4)	−0·2189 (5)	0·1420 (4)	0·046 (2)
C(6)	0·3801 (4)	−0·0661 (6)	0·1129 (4)	0·046 (2)
C(7)	0·1750 (3)	0·1488 (5)	0·1327 (3)	0·032 (1)

Table 2. *Bond lengths* (Å) *and angles* (°) *and torsion angles* (°)

P(1)—S(1)	1·957 (1)	P(1)—C(7)	1·823 (4)
P(2)—S(2)	1·967 (1)	P(2)—C(3)	1·819 (4)
P(1)—C(1)	1·808 (4)	P(2)—C(4)	1·813 (4)
P(1)—C(6)	1·810 (5)	P(2)—C(7)	1·807 (4)

S(1)—P(1)—C(1)	113·2 (2)	C(3)—P(2)—C(7)	103·8 (2)
S(1)—P(1)—C(6)	112·6 (2)	C(4)—P(2)—C(7)	103·4 (2)
S(1)—P(1)—C(7)	114·1 (1)	P(1)—C(1)—C(2)	118·0 (3)
S(2)—P(2)—C(3)	111·1 (1)	P(1)—C(6)—C(5)	119·3 (3)
S(2)—P(2)—C(4)	113·4 (1)	P(1)—C(7)—P(2)	110·6 (2)
S(2)—P(2)—C(7)	114·1 (1)	P(2)—C(3)—C(2)	118·1 (2)
C(1)—P(1)—C(6)	109·8 (2)	P(2)—C(4)—C(5)	117·2 (3)
C(1)—P(1)—C(7)	102·5 (2)	C(1)—C(2)—C(3)	115·8 (3)
C(6)—P(1)—C(7)	103·8 (2)	C(4)—C(5)—C(6)	115·4 (3)
C(3)—P(2)—C(4)	110·4 (2)		

S(1)—P(1)—C(1)—C(2)	179·0 (3)	S(2)—P(2)—C(3)—C(2)	−175·0 (3)
C(6)—P(1)—C(1)—C(2)	−54·1 (3)	C(4)—P(2)—C(3)—C(2)	58·3 (3)
C(7)—P(1)—C(1)—C(2)	55·7 (3)	C(7)—P(2)—C(3)—C(2)	−51·9 (3)
S(1)—P(1)—C(6)—C(5)	−175·2 (3)	S(2)—P(2)—C(4)—C(5)	−180·0 (3)
C(1)—P(1)—C(6)—C(5)	57·6 (4)	C(3)—P(2)—C(4)—C(5)	−54·6 (3)
C(7)—P(1)—C(6)—C(5)	−51·3 (4)	C(7)—P(2)—C(4)—C(5)	55·9 (3)
S(1)—P(1)—C(7)—P(2)	178·6 (2)	S(2)—P(2)—C(7)—P(1)	178·4 (2)
C(1)—P(1)—C(7)—P(2)	−58·7 (2)	C(3)—P(2)—C(7)—P(1)	57·3 (2)
C(6)—P(1)—C(7)—P(2)	55·7 (2)	C(4)—P(2)—C(7)—P(1)	−58·0 (2)
P(1)—C(1)—C(2)—C(3)	−52·9 (4)	P(2)—C(4)—C(5)—C(6)	−52·8 (4)
C(1)—C(2)—C(3)—P(2)	50·2 (4)	C(4)—C(5)—C(6)—P(1)	50·7 (4)

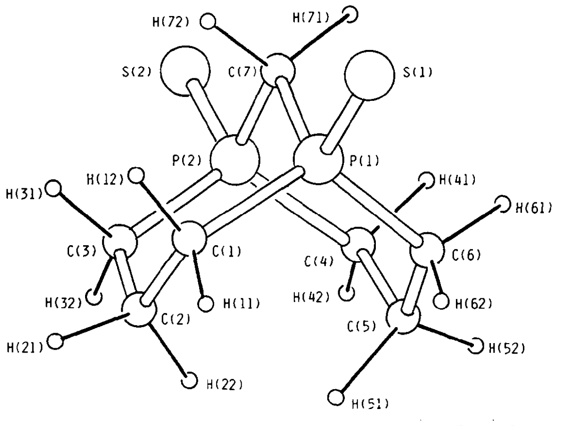

Fig. 1. Molecular structure of 1,5-diphosphabicyclo[3.3.1]nonane 1,5-disulfide.

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* Lists of structure factors, anisotropic thermal parameters and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 44851 (10 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

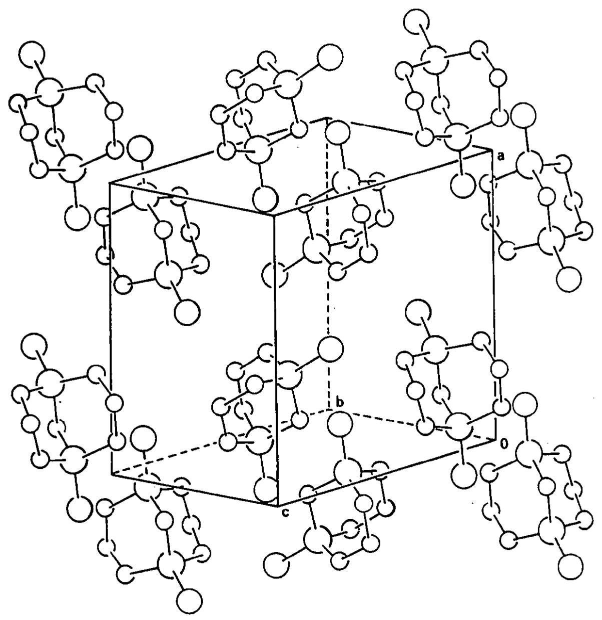

Fig. 2. Molecular packing of 1,5-diphosphabicyclo[3.3.1]nonane 1,5-disulfide.

those obtained for a series of carbabicyclo[3.3.1]-nonanes (*e.g.* Sim, 1983). Since a slight but significant lengthening of these distances has also been observed in 1-phosphabicyclo[3.3.1]nonane 1-sulfide (Baumeister, Hartung & Krech, 1988), the P atoms in the bridgehead positions of the heterobicyclic system seem to be responsible for it.

The coordination around the P atoms is that of a slightly distorted tetrahedron. The C—P—S bond angles are all increased from tetrahedral to 111–114° [mean 113·1 (10)°] whereas most of the C—P—C angles are decreased [mean 105·6 (32)°]. The excellent agreement of the observed bond lengths with the corresponding values in 1-phosphabicyclo[3.3.1]nonane 1-sulfide [given in square brackets] is illustrated by the following data: $\overline{\text{P—C}}$ = 1·813 (6) [1·807 (7)], $\overline{\text{P—S}}$ = 1·962 (5) [P—S = 1·958 (1)], $\overline{\text{C—C}}$ = 1·531 (9) Å [1·530 (5) Å]. The van der Waals radius for P is 1·80 Å (Bondi, 1964) so the nonbonded P(1)···P(2) distance of 2·983 (1) Å is remarkably short. In 1,5-diphosphabicyclo[3.3.0]octane 1,5-disulfide the directly bonded P atoms are separated by 2·200 (5) Å (Hartung, Hickel, Kaiser & Richter, 1979). The C—H distances are in the range 0·85 (3)–1·09 (4) Å [mean 0·95 (6) Å].

The crystal structure (Fig. 2) consists of discrete molecules with all intermolecular contacts between non-H atoms greater than the sums of the corresponding van der Waals radii. Two S···H contacts are slightly shorter than 3·0 Å.

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References

Altona, C. & Sundaralingam, M. (1970). *Tetrahedron*, **26**, 925–939.
Baumeister, U., Hartung, H. & Krech, F. (1988). *Acta Cryst.* C**44**, 1435–1438.
Bondi, A. (1964). *J. Phys. Chem.* **68**, 441–451.
Hartung, H., Hickel, S., Kaiser, J. & Richter, R. (1979). *Z. Anorg. Allg. Chem.* **458**, 130–138.
Jaskólski, M. (1980). *PRARA*. A program for data reduction from Syntex data tapes. Univ. of Poznań, Poland.
Jaskólski, M. (1982). *EDIT*. Procedure to edit atomic information from atom data files. Univ. of Poznań, Poland.
Krech, F. (1985). Unpublished work.
Sheldrick, G. M. (1976). *SHELX76*. Program for crystal structure determination. Univ. of Cambridge, England.
Sim, G. A. (1983). *Tetrahedron*, **39**, 1181–1185.

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Structure of 1,3-Diphenyl-4,5-dihydro-1*H*-1,2,4-triazin-6-one

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Abstract. $C_{15}H_{13}N_3O$, $M_r = 251·29$, orthorhombic, $P2_12_12_1$, $a = 11·511$ (3), $b = 17·96$ (1), $c = 6·118$ (1) Å, $V = 1264·8$ (1·2) Å^3, $Z = 4$, $D_x = 1·32$ g cm^{-3}, λ(Mo $K\alpha$) = 0·71069 Å, $\mu = 0·93$ cm^{-1}, $F(000) = 528$, $T = 293$ K, final $R(F) = 0·032$ for 960 observed reflections. The six-membered triazine ring is not planar. The phenyl ring in position 3 is nearly coplanar with the mean plane of the heterocycle; the

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