References

Bullen, G. J. & Dann, P. E. (1974). *J. Chem. Soc. Dalton Trans.* pp. 705–709.

Bullen, G. J. & Tucker, P. A. (1972). *J. Chem. Soc. Dalton Trans.* pp. 1651–1658, 2437–2442.

Burford, N., Chivers, T. C., Cordes, A. W., Laidlaw, W. G., Noble, M. C., Oakley, R. T. & Swepston, P. N. (1982). *J. Am. Chem. Soc.* **104**, 1282–1290.

Chivers, T., Cordes, A. W., Oakley, R. T. & Pennington, W. T. (1983). *Inorg. Chem.* **22**, 2429–2435.

Chivers, T., Rao, M. N. S. & Richardson, J. F. (1985). *Inorg. Chem.* **24**, 2237–2243.

Enraf–Nonius (1982). *Structure Determination Package.* Enraf–Nonius, Delft, The Netherlands.

Germain, G., Main, P. & Woolfson, M. M. (1971). *Acta Cryst.* A**27**, 368–376.

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.

Oakley, R. T. & Cordes, A. W. (1988). *Acta Cryst.* C**44**, 1840–1842.

Paddock, N. L. (1964). *Q. Rev. Chem. Soc.* **18**, 168–210.

Acta Cryst. (1988). C**44**, 1840–1842

Structures of Spirocyclic Phosphathiazenes: $(Ph_6P_3N_4)_2S$

By R. T. Oakley

Guelph-Waterloo Centre for Graduate Work in Chemistry, Guelph Campus,
Department of Chemistry and Biochemistry, University of Guelph, Guelph, Ontario, Canada N1G 2W1

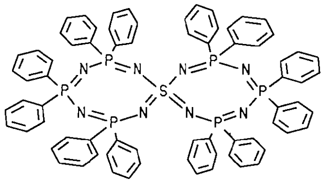
and A. W. Cordes

Department of Chemistry and Biochemistry, University of Arkansas, Fayetteville, AR 72701, USA

(*Received* 1 *April* 1988; *accepted* 9 *June* 1988)

Abstract. 3,3′,5,5′,7,7′-Hexaphenylspirobi($1\lambda^6$,2,4,6,8,$3\lambda^5$,$5\lambda^5$,$7\lambda^5$-thiatetraazatriphosphocine), $C_{72}H_{60}N_8P_6S$, $M_r = 1255{\cdot}2$, orthorhombic, $Pbca$, $a = 49{\cdot}76\,(1)$, $b = 13{\cdot}104\,(6)$, $c = 19{\cdot}59\,(1)$ Å, $V = 12774\,(15)$ Å^3, $Z = 8$, $D_x = 1{\cdot}31$ g cm^{-3}, $\lambda(\text{Mo }K\alpha) = 0{\cdot}71073$ Å, $\mu = 2{\cdot}7$ cm^{-1}, $F(000) = 5232$, $T = 293$ K, $R = 0{\cdot}068$ for 2704 unique observed reflections. Two SP_3N_4 rings share the S atom in the spiro molecule. One of the rings is roughly in the saddle (S_4N_4) conformation with the N atoms 0·55 (1) to 0·76 (1) Å above and below the S—P4—P5—P6 group which is planar within 0·130 (4) Å. The other ring is approximately in a tub conformation, with the S—N4 and P2—N2 tub ends displaced 0·68 to 1·09 Å from the P1—N1—P3—N3 boat bottom which is planar within 0·03 Å. The average S—N, N—P, P—N and N—P bond distances (moving out in the rings from the S atom) are 1·56 (2), 1·61 (2), 1·58 (2) and 1·58 (2) Å, respectively. The NSN, NPN, SNP and PNP bond angles average 109 (6), 121 (3), 127 (1) and 132 (3)°, respectively.

Experimental. Compound (I) prepared as a minor by-product of the reduction of $(PPh_2)_3N_4SCl$ with $SbPh_3$. The pale yellow crystal used for data collection was obtained from acetonitrile solution. Data crystal 0·2 × 0·4 × 0·5 mm mounted on a glass fiber. Intensities measured with an Enraf–Nonius CAD-4 diffractometer using ω–2θ scans of 4–16° min^{-1} in θ. Unit cell determined from least-squares analysis of angle data for 22 reflections with $18 < 2\theta < 22°$. No absorption correction was made. Data collected to $(\sin\theta)/\lambda$ of 0·47 Å^{-1}, $0 < h < 46$, $0 < k < 12$, $0 < l < 18$. Three standard reflections (383, 467, 5,1,12) decreased less than 0·5% over 46·3 h of data collection. 5361 reflections measured, 2657 reflections with $I < 3\sigma(I)$ considered unobserved.



(I)

Solved by direct methods using *MULTAN* (Germain, Main & Woolfson, 1971). Full-matrix least squares minimized $\sum w(\Delta F)^2$. H atoms were constrained to idealized positions (C—H = 0·95 Å) with isotropic thermal parameters $B = 5{\cdot}0$ Å^2; the S and P atoms were refined anisotropically and the C atoms isotropically for a total of 384 variables. $R = 0{\cdot}068$, $wR = 0{\cdot}078$, $S = 1{\cdot}79$, where non-Poisson $w^{-1} = [\sigma^2(I) + 0{\cdot}0025I^2]/4F^2$. $R = 0{\cdot}11$ for all reflections with $F_{\text{obs}} > 0$ and 0·19 for all reflections (1773 had $F_{\text{obs}} = 0$). Final $(\Delta/\sigma)_{\max} < 0{\cdot}4$, $\Delta\rho_{\max} = 0{\cdot}46\,(4)$ and $\Delta\rho_{\min} =$

0108-2701/88/101840-03$03.00

Table 1. *Fractional coordinates and isotropic or equivalent isotropic thermal parameters*

The thermal parameters are of the form $B = 8\pi^2U$ and $B_{\rm eq} = \frac{8}{3}\sum_i\sum_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j$.

Starred atoms were refined isotropically.

	x	y	z	$B/B_{\rm eq}$(Å^2)
S	0·61626 (6)	−0·0487 (2)	0·7407 (2)	2·46 (7)
P1	0·60544 (7)	0·0078 (3)	0·8784 (2)	2·77 (8)
P2	0·55404 (6)	0·0770 (3)	0·8295 (2)	2·78 (8)
P3	0·56070 (6)	−0·0934 (2)	0·7347 (2)	2·73 (8)
P4	0·63724 (7)	0·1144 (3)	0·6628 (2)	2·94 (8)
P5	0·69098 (7)	0·0304 (3)	0·6683 (2)	3·72 (9)
P6	0·66691 (7)	−0·1507 (3)	0·7239 (2)	3·37 (9)
N1	0·6134 (2)	−0·0708 (7)	0·8193 (4)	2·6 (2)*
N2	0·5819 (2)	0·0855 (7)	0·8705 (4)	2·6 (2)*
N3	0·5467 (2)	−0·0246 (7)	0·7908 (4)	3·0 (2)*
N4	0·5896 (2)	−0·0657 (7)	0·7015 (4)	2·6 (2)*
N5	0·6262 (2)	0·0624 (6)	0·7319 (4)	2·2 (2)*
N6	0·6652 (2)	0·0756 (7)	0·6336 (5)	3·3 (2)*
N7	0·6882 (2)	−0·0604 (7)	0·7217 (5)	3·5 (2)*
N8	0·6353 (2)	−0·1300 (6)	0·7076 (4)	2·4 (2)*
C1	0·6002 (2)	−0·0769 (9)	0·9506 (6)	2·7 (3)*
C2	0·5977 (3)	−0·1790 (9)	0·9411 (6)	3·8 (3)*
C3	0·5929 (3)	−0·244 (1)	0·9966 (7)	5·4 (4)*
C4	0·5917 (3)	−0·200 (1)	1·0613 (7)	4·8 (3)*
C5	0·5940 (3)	−0·102 (1)	1·0711 (6)	4·2 (3)*
C6	0·5984 (2)	−0·0366 (9)	1·0156 (6)	3·5 (3)*
C7	0·6339 (2)	0·0831 (8)	0·9020 (6)	2·9 (3)*
C8	0·6593 (3)	0·043 (1)	0·8952 (7)	5·2 (4)*
C9	0·6819 (3)	0·101 (1)	0·9142 (7)	6·2 (4)
C10	0·6794 (3)	0·196 (1)	0·9373 (7)	5·8 (4)*
C11	0·6543 (3)	0·236 (1)	0·9453 (7)	5·3 (4)*
C12	0·6317 (3)	0·182 (1)	0·9266 (6)	4·2 (3)*
C13	0·5262 (2)	0·0986 (9)	0·8868 (6)	3·1 (3)*
C14	0·5299 (3)	0·120 (1)	0·9555 (6)	4·4 (3)*
C15	0·5073 (3)	0·134 (1)	0·9986 (7)	5·9 (4)*
C16	0·4828 (3)	0·129 (1)	0·9718 (7)	5·9 (4)*
C17	0·4784 (3)	0·112 (1)	0·9047 (7)	6·3 (4)*
C18	0·5004 (3)	0·092 (1)	0·8627 (7)	5·2 (4)*
C19	0·5532 (2)	0·1838 (9)	0·7718 (6)	3·5 (3)*
C20	0·5335 (3)	0·186 (1)	0·7219 (7)	5·6 (4)*
C21	0·5323 (3)	0·273 (1)	0·6784 (8)	7·2 (4)*
C22	0·5501 (3)	0·346 (1)	0·6861 (8)	6·8 (4)*
C23	0·5701 (3)	0·345 (1)	0·7316 (8)	7·1 (4)*
C24	0·5716 (3)	0·262 (1)	0·7774 (7)	4·7 (3)*
C25	0·5386 (2)	−0·1004 (8)	0·6617 (5)	2·4 (3)*
C26	0·5474 (2)	−0·0823 (9)	0·5969 (6)	3·7 (3)*
C27	0·5291 (3)	−0·086 (1)	0·5434 (7)	5·1 (3)*
C28	0·5032 (3)	−0·109 (1)	0·5539 (7)	5·5 (4)*
C29	0·4937 (3)	−0·126 (1)	0·6180 (7)	5·3 (4)*
C30	0·5119 (3)	−0·1204 (9)	0·6732 (6)	4·0 (3)*
C31	0·5616 (2)	−0·2209 (8)	0·7696 (6)	2·6 (3)*
C32	0·5806 (3)	−0·2884 (9)	0·7506 (6)	4·1 (3)*
C33	0·5811 (3)	−0·387 (1)	0·7782 (7)	5·8 (4)*
C34	0·5614 (3)	−0·414 (1)	0·8222 (7)	6·5 (4)*
C35	0·5422 (3)	−0·348 (1)	0·8426 (7)	6·2 (4)*
C36	0·5417 (3)	−0·248 (1)	0·8180 (6)	4·5 (3)*
C37	0·6139 (2)	0·1072 (8)	0·5918 (5)	2·6 (3)*
C38	0·5907 (3)	0·162 (1)	0·5944 (6)	3·9 (3)*
C39	0·5730 (3)	0·160 (1)	0·5393 (7)	5·6 (4)*
C40	0·5794 (3)	0·107 (1)	0·4838 (7)	5·3 (4)*
C41	0·6019 (3)	0·050 (1)	0·4805 (7)	5·3 (4)*
C42	0·6201 (2)	0·049 (1)	0·5355 (6)	4·0 (3)*
C43	0·6397 (2)	0·2467 (9)	0·6844 (6)	3·1 (3)*
C44	0·6418 (3)	0·321 (1)	0·6347 (7)	4·8 (3)*
C45	0·6460 (3)	0·422 (1)	0·6521 (7)	6·1 (4)*
C46	0·6491 (3)	0·448 (1)	0·7182 (8)	6·5 (4)*
C47	0·6476 (3)	0·378 (1)	0·7693 (8)	6·7 (4)*
C48	0·6430 (3)	0·276 (1)	0·7507 (7)	4·8 (3)*
C49	0·7127 (2)	−0·004 (1)	0·5970 (6)	4·0 (3)*
C50	0·7105 (3)	0·048 (1)	0·5388 (7)	6·5 (4)*
C51	0·7287 (4)	0·021 (1)	0·4807 (9)	9·2 (5)*
C52	0·7450 (3)	−0·055 (1)	0·4939 (8)	7·5 (4)*
C53	0·7479 (4)	−0·109 (1)	0·5495 (8)	8·8 (5)*
C54	0·7302 (3)	−0·083 (1)	0·6047 (7)	6·1 (4)*
C55	0·7095 (3)	0·128 (1)	0·7118 (6)	4·5 (3)*
C56	0·7262 (3)	0·103 (1)	0·7639 (8)	7·2 (4)*
C57	0·7408 (4)	0·185 (1)	0·7990 (9)	9·9 (6)*
C58	0·7350 (3)	0·279 (1)	0·7813 (9)	8·6 (5)*
C59	0·7206 (4)	0·308 (1)	0·7285 (9)	9·1 (5)*
C60	0·7072 (3)	0·227 (1)	0·6925 (8)	7·1 (4)*
C61	0·6756 (2)	−0·2465 (9)	0·6631 (6)	3·1 (3)*
C62	0·6600 (3)	−0·267 (1)	0·6080 (7)	4·8 (3)*
C63	0·6683 (3)	−0·340 (1)	0·5598 (7)	5·2 (4)*
C64	0·6920 (3)	−0·394 (1)	0·5690 (7)	5·6 (4)*
C65	0·7074 (3)	−0·373 (1)	0·6235 (8)	6·3 (4)*
C66	0·6990 (3)	−0·303 (1)	0·6707 (7)	5·5 (4)*
C67	0·6718 (2)	−0·2130 (9)	0·8053 (6)	3·5 (3)*
C68	0·6545 (3)	−0·286 (1)	0·8258 (7)	6·1 (4)*
C69	0·6580 (3)	−0·340 (1)	0·8859 (8)	7·8 (5)*
C70	0·6800 (3)	−0·316 (1)	0·9234 (8)	7·5 (5)*
C71	0·6977 (3)	−0·245 (1)	0·9061 (8)	6·8 (4)*
C72	0·6932 (3)	−0·189 (1)	0·8458 (7)	5·2 (4)*

Table 2. *Selected bond distances* (Å) *and angles* (°) *with e.s.d.'s in parentheses*

S–N1	1·573 (7)	P3–C25	1·807 (9)
S–N4	1·551 (7)	P3–C31	1·806 (9)
S–N5	1·546 (7)	P4–N5	1·612 (7)
S–N8	1·566 (7)	P4–N6	1·588 (8)
P1–N1	1·600 (7)	P4–C37	1·814 (9)
P1–N2	1·561 (7)	P4–C43	1·789 (10)
P1–C1	1·816 (9)	P5–N6	1·567 (8)
P1–C7	1·788 (9)	P5–N7	1·590 (8)
P2–N2	1·605 (7)	P5–C49	1·822 (10)
P2–N3	1·574 (7)	P5–C55	1·794 (11)
P2–C13	1·804 (9)	P6–N7	1·587 (8)
P2–C19	1·800 (10)	P6–N8	1·627 (7)
P3–N3	1·584 (7)	P6–C61	1·783 (9)
P3–N4	1·618 (7)	P6–C67	1·807 (10)

N1–S–N4	112·3 (4)	N2–P2–N3	120·1 (4)
N1–S–N5	108·1 (4)	N2–P2–C13	109·8 (4)
N1–S–N8	109·5 (4)	N2–P2–C19	106·3 (4)
N4–S–N5	110·7 (4)	N3–P2–C13	104·7 (4)
N4–S–N8	102·5 (4)	N3–P2–C19	110·4 (4)
N5–S–N8	113·7 (4)	C13–P2–C19	104·5 (4)
N1–P1–N2	122·2 (4)	N3–P3–N4	122·9 (4)
N1–P1–C1	101·9 (4)	N3–P3–C25	108·1 (4)
N1–P1–C7	110·2 (4)	N3–P3–C31	106·0 (4)
N2–P1–C1	111·6 (4)	N4–P3–C25	103·4 (4)
N2–P1–C7	105·2 (4)	N4–P3–C31	109·7 (4)
C1–P1–C7	104·4 (4)	C25–P3–C31	105·5 (4)
N5–P4–N6	117·8 (4)	N7–P6–N8	121·0 (4)
N5–P4–C37	113·8 (4)	N7–P6–C61	110·1 (4)
N5–P4–C43	103·6 (4)	N7–P6–C67	105·7 (5)
N6–P4–C37	105·5 (4)	N8–P6–C61	102·7 (4)
N6–P4–C43	109·6 (4)	N8–P6–C67	112·3 (4)
C37–P4–C43	106·0 (4)	C61–P6–C67	103·8 (4)
N6–P5–N7	119·7 (4)	S–N1–P1	127·8 (5)
N6–P5–C49	104·3 (5)	P1–N2–P2	130·8 (5)
N6–P5–C55	110·9 (5)	P2–N3–P3	135·4 (5)
N7–P5–C49	111·8 (5)	S–N4–P3	126·4 (4)
N7–P5–C55	105·5 (5)	S–N5–P4	126·8 (5)
C49–P5–C55	103·6 (5)	P4–N6–P5	133·0 (5)
		P5–N7–P6	129·3 (5)
		S–N8–P6	128·1 (5)

−0·34 (4) e Å^{-3} on final difference map. Atomic scattering factors and anomalous-dispersion correction from *International Tables for X-ray Crystallography* (1974) and programs used were those of Enraf–Nonius (1982) *SDP*.* Table 1 gives the atom coordinates and Fig. 1 shows the ring atoms with the numbering scheme, and Table 2 gives the distances and angles involving the ring atoms.

---

* Lists of structure factors and anisotropic thermal parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 51075 (38 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

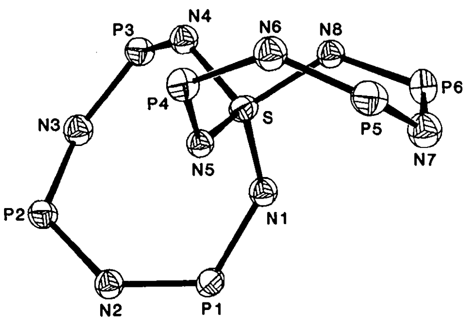


Fig. 1. *ORTEP*II diagram (Johnson, 1976) with 30% ellipsoids for the ring atoms (phenyl rings omitted for figure clarity).

Related literature. The spirocyclic nature of the structure is to be compared with that of $(Ph_4P_2N_3)_2S$ (Chivers, Rao & Richardson, 1985) and $(Ph_6P_3N_4)$-$S(S_2N_3)$ (Cordes, Oakley & Morito, 1988). The tub and saddle conformations of the two eight-membered rings are in keeping with the conformational motifs of sterically crowded cyclophosphazenes (Paddock, 1964; Bullen & Tucker, 1972; Bullen & Dann, 1974).

We thank the National Science Foundation, the State of Arkansas, the Research Corporation, and the Natural Sciences and Engineering Research Council of Canada for financial support.

References

Bullen, G. J. & Dann, P. E. (1974). *J. Chem. Soc. Dalton Trans.* pp. 705–709.

Bullen, G. J. & Tucker, P. A. (1972). *J. Chem. Soc. Dalton Trans.* pp. 1651–1658, 2437–2442.

Chivers, T., Rao, M. N. S. & Richardson, J. F. (1985). *Inorg. Chem.* **24**, 2237–2243.

Cordes, A. W., Oakley, R. T. & Morito, E. (1988). *Acta Cryst.* C**44**, 1838–1840.

Enraf–Nonius (1982). *Structure Determination Package.* Enraf–Nonius, Delft, The Netherlands.

Germain, G., Main, P. & Woolfson, M. M. (1971). *Acta Cryst.* A**27**, 368–376.

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.

Paddock, N. L. (1964). *Q. Rev. Chem. Soc.* **18**, 168–210.

Acta Cryst. (1988). C**44**, 1842–1844

Structure of 2-*tert*-Butyl-4-methyl-2,4-dihydropyrrolo[3,4-*b*]indole

By Hans Preut, Gerald Dyker and Richard P. Kreher

Fachbereich Chemie, Universität Dortmund, D-4600 *Dortmund* 50, *Postfach* 500500,
Federal Republic of Germany

(*Received* 25 *January* 1988; *accepted* 23 *May* 1988)

Abstract. $C_{15}H_{18}N_2$, $M_r = 226{\cdot}32$, orthorhombic, *Pbca*, $a = 9{\cdot}278\ (5)$, $b = 16{\cdot}334\ (7)$, $c = 17{\cdot}498\ (11)$ Å, $V = 2652\ (2)$ Å^3, $Z = 8$, $D_x = 1{\cdot}134$ Mg m^{-3}, $F(000) = 976$, $\lambda(\text{Mo}\ K\alpha) = 0{\cdot}71073$ Å, $\mu = 0{\cdot}06$ mm^{-1}, $T = 291\ (1)$ K, final $R = 0{\cdot}073$ for 1095 unique observed $[F \geq 3{\cdot}0\sigma(F)]$ diffractometer data. The three rings of the fused hetarene are nearly planar. The position of the methyl C atom at the N atom of the pyrrole ring does not deviate significantly from the least-squares plane through this central pyrrole ring. Thus the N atom may be considered sp^2-hybridized. According to the results of this analysis the molecule is to be regarded as a conjugated heteroaromatic system with 14 π electrons. There are no interactions between the molecules exceeding van der Waals forces.

Experimental. The tricyclic hetarene was prepared by the rational and efficient method of Kreher & Dyker (1987). In order to characterize the molecular geometry of the heterocyclic $(4n + 2)\pi$ system and to compare the experimental parameters with calculated values (CNDO) the crystal structure was determined. The planarity of the tricyclic hetarene is an essential feature for delocalization; distortion should be favourable for localized five- and six-membered π systems. The compound was crystallized from *n*-pentane at 248 K. Crystal size $0{\cdot}47 \times 0{\cdot}26 \times 0{\cdot}32$ mm, $\omega/2\theta$ scan, scan speed $1{\cdot}54$–$3{\cdot}33°\ \text{min}^{-1}$ in θ, Nonius CAD-4 diffractometer, graphite-monochromated Mo $K\alpha$; lattice parameters from least-squares fit with 25 reflections up to $2\theta = 24{\cdot}5°$; six standard reflections recorded every 2·5 h, only random deviations; 5190 reflections measured, $1{\cdot}5 \leq \theta \leq 25{\cdot}0°$, $-11 \leq h \leq 11$, $0 \leq k \leq 19$, $0 \leq l \leq 20$; after averaging ($R_{\text{int}} = 0{\cdot}043$) 2329 unique reflections, 1095 with $F \geq 3{\cdot}0\sigma(F)$; Lorentz–polarization correction, no absorption correction; systematic absences $(0kl)\ k = 2n + 1$, $(h0l)\ l = 2n + 1$, $(hk0)\ h = 2n + 1$ conform to space group *Pbca;* structure solution *via* direct methods, ΔF syntheses and full-matrix least-squares