Dahl, L. F., Martell, C. & Wampler, D. L. (1961). *J. Am. Chem. Soc.* **83**, 1761–1762.
Dulebohn, J. I., Ward, D. L. & Nocera, D. G. (1988). *J. Am. Chem. Soc.* **110**, 4054–4056.
Frenz, B. A. (1985). *Structure Determination Package*. Enraf–Nonius, Delft, The Netherlands.
Germain, G., Main, P. & Woolfson, M. M. (1971). *Acta Cryst.* A**27**, 368–376.
Ibers, J. A. & Snyder, R. G. (1962). *Acta Cryst.* **15**, 923–930.
King, R. B., Shimura, M. & Brown, G. M. (1984). *Inorg. Chem.* **23**, 1398–1404.
Kubiak, C. P. & Eisenberg, R. (1977). *J. Am. Chem. Soc.* **99**, 6129–6131.
Mague, J. T., Johnson, M. P. & Lloyd, C. L. (1989). *J. Am. Chem. Soc.* **111**, 5012–5013.
Newton, M. G., King, R. B., Chang, M., Pantaleo, N. S. & Gimeno, J. (1977). *J. Chem. Soc. Chem. Commun.* pp. 531–532.
Newton, M. G., King, R. B., Lee, T.-W., Nørskov-Lauritsen, L. & Kumar, V. (1982). *J. Chem. Soc. Chem. Commun.* pp. 201-203.
North, A. C. T., Phillips, D. C. & Mathews, F. S. (1968). *Acta Cryst.* A**24**, 351–359.

Acta Cryst. (1990). C**46**, 131–132

Structure of $(CO)_5Cr[\overline{P(Me_3SiNH)NSNSNH}]$: the Chromium Pentacarbonyl Complex of a $P^{III}N_3S_2$ Ring

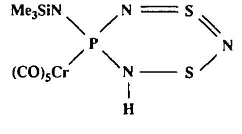
By Tristram Chivers, James F. Fait and Cees Lensink

Department of Chemistry, The University of Calgary, Calgary, Alberta, Canada T2N 1N4

(*Received* 16 *May* 1989; *accepted* 14 *July* 1989)

Abstract. Pentacarbonyl(5-trimethylsilylamino-4,5-dihydro-1λ^4,3-dithia-2,4,6-triaza-5-phosphorine)chromium, $[Cr(CO)_5(C_3H_{11}N_4PS_2Si)]$, $M_r = 418{\cdot}38$, monoclinic, $P2_1/n$, $a = 6{\cdot}266\,(2)$, $b = 13{\cdot}739\,(4)$, $c = 19{\cdot}959\,(5)$ Å, $\beta = 101{\cdot}97\,(3)°$, $V = 1680{\cdot}9\,(9)$ Å^3, $Z = 4$, $D_x = 1{\cdot}65$ g cm^{-3}, λ(Mo $K\alpha$) = 0·71069 Å, $\mu = 10{\cdot}47$ cm^{-1}, $F(000) = 848$, $T = 293$ K, $R = 0{\cdot}065$ for 1810 unique observed reflections. The molecule is an octahedral chromium complex in which the $Cr(CO)_5$ group occupies the axial position at the P atom of a six-membered $P^{III}N_3S_2$ ligand. The ring adopts a half-boat conformation with the N(H) nitrogen out of the plane of the other five ring atoms. The ring contains two short [1·560 (6) and 1·571 (7) Å] and two long [1·654 (7) and 1·684 (6) Å] S—N bonds. The corresponding bond angles at sulfur are 119·4 (4) (N=S=N) and 107·6 (3)° (N—S—N).

Experimental. The title compound (I) was obtained from the reaction of $Cr(CO)_5[P(NSiMe_3)N(SiMe_3)_2]$ with S_4N_4 followed by treatment of the product with 2-propanol (Chivers, Dhathathreyan, Lensink, Meetsma, van de Grampel & de Boer, 1989). A dark orange crystal (0·32 × 0·24 × 0·19 mm) obtained from a pentane solution was mounted on a glass fiber. Enraf–Nonius CAD-4 diffractometer, Mo radiation, graphite monochromator, ω–2θ scans. Unit-cell dimensions from 25 reflections $6 < \theta < 16°$. No indication of any higher metric symmetry or crystal decomposition. Data collected to $(\sin\theta)/\lambda$ of 0·59 Å^{-1}, $-7 < h < 7$, $0 < k < 16$, $0 < l < 23$. 3420 reflections measured, 2952 unique, 1810 reflections with $F > 4\sigma(F)$ considered observed. Two standard reflections, 3·5% intensity variation.



(I)

Solved by a combination of heavy-atom techniques and direct methods to give a partial structure of the $Cr\overline{PNSNSN}$ system. Remaining non-H atoms located in difference maps after refining the scale and thermal parameters. All H atoms bonded to carbon were located in difference Fourier maps; the hydrogens on N(1) and N(4) were not found. Corrected for absorption using *DIFABS* (Walker & Stuart, 1983). All non-H atoms refined anisotropically, with the H atoms riding on the C atom to which they are bonded with a common isotropic factor for all H atoms. Final cycles of least squares were performed with weights of $1/\sigma(F)^2$ with a maximum Δ/σ of 0·003, and $(\Delta\rho)_{\max} = 0{\cdot}534$, $(\Delta\rho)_{\min} = -0{\cdot}516$ e Å^{-3}, $R = 0{\cdot}065$, $wR = 0{\cdot}055$, $S = 2{\cdot}502$.

Neutral-atom scattering factors used with anomalous-dispersion corrections applied from *International Tables for X-ray Crystallography* (1974). No extinction corrections. Programs used were *XTAL* (Hall & Stewart, 1988) and *SHELX* (Sheldrick, 1976). Table 1 gives the atom coordinates for non-H atoms and Table 2 lists bond distances and angles.

0108-2701/90/010131-02$03.00

Table 1. *Atomic coordinates and isotropic thermal parameters* (Å^2 × 10^3) *for non*-H *atoms*

$U_{eq} = \frac{1}{3}$ of the trace of the orthogonalized U_{ij} matrix.

	x	*y*	*z*	U_{eq}
Cr	0·0229 (2)	0·35625 (9)	0·12221 (6)	26
S(1)	0·1315 (4)	0·6444 (2)	0·2066 (1)	40
S(2)	−0·3091 (4)	0·6008 (2)	0·1465 (1)	43
P(1)	−0·0234 (3)	0·4491 (1)	0·2148 (1)	27
Si	0·1480 (4)	0·4293 (2)	0·3731 (1)	30
N(1)	−0·002 (1)	0·3961 (4)	0·2907 (3)	32
N(2)	0·165 (1)	0·5385 (4)	0·2351 (3)	29
N(3)	−0·096 (1)	0·6748 (5)	0·1622 (3)	44
N(4)	−0·267 (1)	0·5132 (4)	0·2068 (3)	34
O(1)	−0·4454 (9)	0·3859 (4)	0·0442 (3)	44
O(2)	0·4857 (9)	0·3144 (4)	0·1975 (3)	46
O(3)	0·118 (1)	0·2412 (4)	0·0031 (3)	55
O(4)	0·1798 (9)	0·5413 (4)	0·0642 (3)	42
O(5)	−0·1193 (9)	0·1771 (4)	0·1901 (3)	46
C(1)	−0·269 (1)	0·3764 (6)	0·0740 (4)	33
C(2)	0·313 (2)	0·3320 (5)	0·1701 (4)	33
C(3)	0·081 (1)	0·2843 (6)	0·0483 (4)	33
C(4)	0·118 (1)	0·4711 (6)	0·0848 (4)	29
C(5)	−0·069 (1)	0·2461 (6)	0·1632 (4)	33
C(6)	0·085 (1)	0·3309 (5)	0·4287 (4)	41
C(7)	0·052 (2)	0·5480 (6)	0·3991 (4)	52
C(8)	0·443 (1)	0·4342 (6)	0·3748 (4)	46

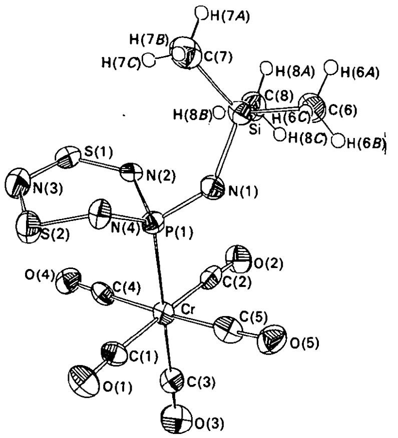


Fig. 1. *ORTEP* diagram and atomic numbering scheme for Cr(CO)$_5$[$\overline{\text{P(Me}_3\text{SiNH)NSNS}}$NH]. H atoms on N(1) and N(4) were not found.

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

P(1)—Cr	2·313 (2)	C(1)—Cr—P(1)	93.3 (2)
C(1)—Cr	1·903 (9)	C(2)—Cr—P(1)	87·7 (2)
C(2)—Cr	1·899 (9)	C(2)—Cr—C(1)	178·2 (3)
C(3)—Cr	1·873 (8)	C(3)—Cr—P(1)	176·0 (2)
C(4)—Cr	1·893 (8)	C(3)—Cr—C(1)	90·1 (3)
C(5)—Cr	1·866 (8)	C(3)—Cr—C(2)	89·0 (3)
N(2)—S(1)	1·560 (6)	C(4)—Cr—P(1)	87·5 (2)
N(3)—S(1)	1·571 (7)	C(4)—Cr—C(1)	91·6 (3)
N(3)—S(2)	1·654 (7)	C(4)—Cr—C(2)	89·9 (3)
N(4)—S(2)	1·684 (6)	C(4)—Cr—C(3)	90·1 (3)
N(1)—P(1)	1·661 (6)	C(5)—Cr—P(1)	89·8 (2)
N(2)—P(1)	1·693 (6)	C(5)—Cr—C(1)	89·4 (3)
N(4)—P(1)	1·738 (6)	C(5)—Cr—C(2)	89·2 (3)
N(1)—Si	1·777 (6)	C(5)—Cr—C(3)	92·6 (3)
C(6)—Si	1·842 (7)	C(5)—Cr—C(4)	177·2 (3)
C(7)—Si	1·849 (8)	N(3)—S(1)—N(2)	119·4 (4)
C(8)—Si	1·846 (8)	N(4)—S(2)—N(3)	107·6 (3)
C(1)—O(1)	1·147 (8)	N(1)—P(1)—Cr	119·1 (2)
C(2)—O(2)	1·134 (9)	N(2)—P(1)—Cr	113·3 (2)
C(3)—O(3)	1·143 (8)	N(2)—P(1)—N(1)	100·4 (3)
C(4)—O(4)	1·147 (8)	N(4)—P(1)—Cr	117·3 (2)
C(5)—O(5)	1·166 (8)	N(4)—P(1)—N(1)	102·0 (3)
		N(4)—P(1)—N(2)	102·1 (3)
		C(6)—Si—N(1)	103·3 (3)
		C(7)—Si—N(1)	110·2 (3)
		C(7)—Si—C(6)	110·9 (4)
		C(8)—Si—N(1)	111·2 (4)
		C(8)—Si—C(6)	110·8 (4)
		C(8)—Si—C(7)	110·2 (4)
		Si—N(1)—P(1)	131·2 (4)
		P(1)—N(2)—S(1)	123·7 (4)
		S(2)—N(3)—S(1)	123·1 (4)
		P(1)—N(4)—S(2)	115·7 (4)
		O(1)—C(1)—Cr	178·0 (7)
		O(2)—C(2)—Cr	177·6 (7)
		O(3)—C(3)—Cr	179·0 (7)
		O(4)—C(4)—Cr	177·7 (7)
		O(5)—C(5)—Cr	177·9 (7)

Fig. 1 displays an *ORTEP* drawing (Johnson, 1976) with the atomic numbering scheme.* The hydrogens on N(1) and N(4) have been unambiguously characterized by ^{1}H NMR and IR spectroscopy (Chivers, Dhathathreyan, Lensink, Meetsma, van de Grampel & de Boer, 1989).

Related literature. The structural parameters and half-boat conformation of the $P^{III}N_3S_2$ ring are similar to those of Cr(CO)$_5$[$\overline{\text{P(}^t\text{Bu)NSNS}}$NH] (Gieren, Ruiz-Peréz, Hübner, Herberhold, Schamel & Guldner, 1989) and $PNP^+S_3N_3O_2^-$ (Chivers, Cordes, Oakley & Pennington, 1983).

We thank the Natural Sciences and Engineering Research Council of Canada for financial support.

References

Chivers, T., Cordes, A. W., Oakley, R. T. & Pennington, W. T. (1983). *Inorg. Chem.* **22**, 2499–2435.

Chivers, T., Dhathathreyan, K. S., Lensink, C., Meetsma, A., van de Grampel, J. C. & de Boer, J. L. (1989). *Inorg. Chem.* In the press.

Gieren, A., Ruiz-Peréz, C., Hübner, T., Herberhold, M., Schamel, K. & Guldner, K. (1989). *J. Organomet. Chem.* **366**, 105–120.

Hall, S. R. & Stewart, J. M. (1988). *XTAl* 2.4 *User's Manual.* Univs. of Western Australia and Maryland, USA.

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.

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

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

---

* 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 52129 (32 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Acta Cryst. (1990). C46, 133–135

Structure of [1,2-Bis(diphenylphosphino)ethane](η^5-cyclopentadienyl)(iodo)iron

By Dale H. Hill, Masood Parvez and Ayusman Sen*

Department of Chemistry, The Pennsylvania State University, University Park, PA 16802, *USA*

(*Received* 28 *April* 1988; *accepted* 11 *July* 1989)

Abstract. [FeI(C_5H_5)($C_{26}H_{24}P_2$)], M_r = 646·28, triclinic, $P\bar{1}$, a = 12·607 (2), b = 13·218 (1), c = 9·253 (2) Å, α = 105·80 (2), β = 101·48 (1), γ = 67·55 (1)°, V = 1363·4 Å^3, Z = 2, D_x = 1·574 g cm^{-3}, λ(Mo $K\alpha$) = 0·71073 Å, μ = 18·06 cm^{-1}, F(000) = 648, T = 293 (1) K, R = 0·024 for 3837 observed data with $I > 3\sigma(I)$. (The standard reduced cell obtained by the transformation matrix [00$\bar{1}$/100/010] is a = 9·253, b = 12·607, c = 13·218 Å, α = 67·55, β = 74·20, γ = 78·52°.) The Fe atom is coordinated to an iodine [Fe—I 2·643 (1) Å], two P atoms of the ligand (Ph_2PCH_2)$_2$ [Fe—P 2·188 (1) and 2·189 (1) Å], and a cyclopentadiene ring [Fe—C in the range 2·054 (3)–2·106 (3) Å] with important angles: I—Fe—P 93·12 (2) and 89·61 (2)° and P—Fe—P 86·22 (3)°.

Experimental. Crystals were obtained by slowly evaporating solvent from a solution of [Fe(Cp)-(dppe)I] (Cp = C_5H_5, dppe = $Ph_2PCH_2CH_2PPh_2$) (Treichel & Molzahn, 1979; Green & Whitely, 1971) in benzene. A dark-red crystal of approximate size 0·35 × 0·31 × 0·27 mm was mounted on a glass fiber for data collection. Unit-cell dimensions were determined from a least-squares fit of the setting angles of 25 reflections with $10 < \theta < 15°$ on an Enraf–Nonius CAD-4 diffractometer equipped with a graphite monochromator. Intensity data were collected by a conventional $\omega/2\theta$ scan method using variable scan speed (1·2–3·3° min^{-1}) in the range $2 < \theta < 25°$ with h −14→14, k 4→14, and l 0→10. The intensities of 4443 unique reflections were measured of which 3837 had $I > 3\sigma(I)$ and were used for structure solution and refinement. The intensities of three reflections chosen as standard and measured at 2 h exposure-time intervals did not show significant variation. Data were corrected for Lorentz and polarization factors and for empirical absorption (North, Phillips & Mathews, 1968); maximum and minimum correction factors were 0·9994 and 0·9476, respectively.

The structure was solved by the heavy-atom method and refined by full-matrix least-squares calculations on F's. Anisotropic temperature factors were allowed for non-H atoms. A difference map revealed all the H atoms which were included in the subsequent refinements and allowed to refine with an overall isotropic temperature factor, B = 4·0 Å^2. The refinement converged with R = 0·024 and wR = 0·033, where $w = [\sigma^2(F_o) + (0{\cdot}050\,F_o)^2]^{-1}$. At the conclusion of the refinement, maximum shift/e.s.d was < 0·02 for the non-H atoms, the final difference map showed peaks between −0·30 and 0·23 e Å^{-3}, and S = 1·040. Atomic scattering factors for non-H atoms were taken from Cromer & Mann (1968) and for H from Stewart, Davidson & Simpson (1965); allowance was made for anomalous dispersion (Cromer & Liberman, 1970). The computer programs used in this study were from the Enraf–Nonius *Structure Determination Package* (B.A. Frenz & Associates Inc., 1985) and *ORTEP* (Johnson, 1976).

Table 1. *Final fractional coordinates and equivalent isotropic thermal parameters* (Å^2), *with e.s.d.'s in parentheses*

$$B_{eq} = \tfrac{4}{3}[a^2a^{*2}B_{11} + b^2b^{*2}B_{22} + c^2c^{*2}B_{33} + ab(\cos\gamma)a^*b^*B_{12} + ac(\cos\beta)a^*c^*B_{13} + bc(\cos\alpha)b^*c^*B_{23}].$$

	x	y	z	B_{eq}
I	0·75669 (2)	0·12565 (2)	0·51181 (2)	3·950 (4)
Fe	0·76118 (3)	0·13433 (3)	0·23121 (4)	2·728 (8)
P1	0·57825 (5)	0·23764 (5)	0·20719 (7)	2·91 (1)
P2	0·79394 (5)	0·29247 (5)	0·31546 (7)	2·79 (1)
C1	0·7899 (3)	−0·0359 (2)	0·1373 (4)	4·09 (7)
C2	0·8965 (2)	−0·0218 (2)	0·2014 (3)	3·87 (7)
C3	0·9142 (2)	0·0483 (3)	0·1305 (4)	4·35 (7)
C4	0·8201 (3)	0·0811 (3)	0·0222 (3)	4·86 (8)
C5	0·7448 (3)	0·0272 (3)	0·0261 (4)	4·94 (9)
C6	0·5552 (2)	0·3865 (2)	0·3067 (3)	3·66 (7)
C7	0·6653 (2)	0·3986 (2)	0·4010 (3)	3·36 (6)
C11	0·5190 (2)	0·2501 (2)	0·0117 (3)	3·15 (6)
C12	0·5629 (2)	0·3011 (2)	−0·0616 (3)	3·99 (7)
C13	0·5283 (3)	0·3044 (3)	−0·2119 (3)	4·44 (8)
C14	0·4508 (3)	0·2547 (3)	−0·2927 (3)	4·55 (8)
C15	0·4075 (3)	0·2028 (3)	−0·2225 (4)	4·48 (8)
C16	0·4408 (2)	0·1990 (2)	−0·0716 (3)	3·73 (7)
C21	0·4644 (2)	0·2075 (2)	0·2661 (3)	3·34 (6)
C22	0·4864 (2)	0·1040 (2)	0·2933 (3)	3·83 (7)
C23	0·3992 (2)	0·0772 (3)	0·3280 (4)	4·51 (7)
C24	0·2907 (3)	0·1527 (3)	0·3332 (4)	5·17 (8)
C25	0·2662 (3)	0·2577 (3)	0·3066 (4)	5·29 (9)
C26	0·3527 (2)	0·2853 (3)	0·2729 (3)	4·28 (7)
C31	0·8211 (2)	0·3559 (2)	0·1787 (3)	3·30 (6)
C32	0·7449 (3)	0·4531 (2)	0·1381 (4)	4·11 (7)
C33	0·7659 (3)	0·4939 (3)	0·0295 (4)	5·59 (9)
C34	0·8640 (4)	0·4386 (3)	−0·0411 (4)	6·7 (1)
C35	0·9429 (3)	0·3440 (3)	0·0002 (4)	6·7 (1)
C36	0·9222 (3)	0·3025 (3)	0·1090 (4)	5·10 (8)
C41	0·9115 (2)	0·3067 (2)	0·4617 (3)	3·18 (6)
C42	0·9167 (2)	0·4118 (2)	0·5339 (3)	4·07 (7)
C43	1·0088 (3)	0·4223 (3)	0·6392 (4)	4·79 (8)
C44	1·0963 (2)	0·3291 (3)	0·6707 (4)	4·73 (8)
C45	1·0930 (2)	0·2256 (3)	0·5993 (4)	4·43 (7)
C46	1·0012 (2)	0·2131 (2)	0·4957 (3)	3·56 (6)

---

* To whom all correspondence should be addressed.

0108-2701/90/010133-03$03.00