coordinate or σ bonds to the N(1, 2 or 4) atoms; the square-planar or octahedral arrangement around the central metal can change simply with change of anion (Bowers & Popov, 1968). The square-planar complex reported here, coordinated through the N(3) atom, appears to be yet another variation similar to the σ complex formed with palladium bis(5-phenyltetrazolate) and triphenylphosphine (Kreutzer *et al.*, 1972).

PJP is indebted to the SERC for a research studentship.

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Structure of Bis(tetraphenylphosphonium) Oxothiotetra(thiocyanato-*N*)tungstate(VI) Methyl Cyanide Solvate

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(*Received* 27 *May* 1987; *accepted* 9 *October* 1987)

Abstract. $[P(C_6H_5)_4]_2[WOS(NCS)_4].CH_3CN$, $M_r = 1184{\cdot}1$, triclinic, $P\bar{1}$, $a = 13{\cdot}414\ (6)$, $b = 19{\cdot}84\ (2)$, $c = 11{\cdot}962\ (7)$ Å, $\alpha = 107{\cdot}34\ (5)$, $\beta = 117{\cdot}95\ (4)$, $\gamma = 75{\cdot}06\ (3)°$, $V = 2644\ (6)$ Å^3, $Z = 2$, $D_x = 1{\cdot}48$ Mg m^{-3}, $T = 293$ K, Mo $K\alpha$ ($\lambda = 0{\cdot}71069$ Å), $F(000) = 1187$, $\mu = 23{\cdot}5$ cm^{-1}, $R = 0{\cdot}050$ for 9320 reflections. The environment of W^{VI} is an approximate octahedron with S and O atoms *cis*-orientated and four N-bonded NCS groups. The W–N bonds to the NCS groups *trans* to the S or O atoms are significantly longer than the other two. The W–N–C and N–C–S angles are all linear within 10° except for the W–N(2)–C(2)–S(2) moiety. The only short intermolecular contact is the S(3)···S(3) distance [3·509 (4) Å].

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0108-2701/88/020369-02$03.00

Experimental. Triclinic prism (0·40 × 0·32 × 0·30 mm) obtained at 273 K from the reaction of $[P(C_6H_5)_4]_2WS_4$ and AgSCN in CH_3CN. Philips PW 1100 diffractometer, graphite monochromator, cell parameters from 25 reflections ($7° < \theta < 14°$). Intensities were collected by a flying step scan technique, scan width 1·4°, scan speed 0·02 s in steps of 0·02° (θ); 11803 independent reflections with $3° < \theta < 29°$ ($\pm h, \pm k, +l$; $h_{max} = 16$, $k_{max} = 26$, $l_{max} = 13$); 9320 with $F > 6\sigma(F)$. Three standard reflections were measured every hour, no significant intensity decay was observed; Lp correction; absorption ignored. Structure solution by Patterson and difference syntheses. Refinements on F for 359 parameters (*SHELX*76, Sheldrick 1976); anisotropic thermal parameters for W, S, P, O and N, C for the anion; isotropic for C and N atoms of the cation and solvate; phenyl H on external C–C–C

Table 1. *Final atomic coordinates* ($\times 10^4$, *for* W $\times 10^5$) *and isotropic thermal parameters* (Å^2)

	x	y	z	B_{eq} or B
W	19977 (2)	29551 (2)	16773 (3)	4·78 (1)*
S(1)	280 (2)	4010 (2)	4024 (2)	9·4 (1)*
S(2)	4586 (2)	2420 (1)	5958 (2)	7·9 (1)*
S(3)	488 (2)	791 (1)	1074 (2)	6·37 (9)*
S(4)	4772 (3)	1539 (2)	195 (4)	12·3 (2)*
S(5)	764 (2)	3170 (1)	−135 (2)	7·9 (1)*
N(1)	1000 (6)	3422 (4)	2648 (6)	6·5 (3)*
C(1)	474 (7)	3664 (4)	3235 (7)	5·5 (3)*
N(2)	3136 (5)	2551 (3)	3468 (6)	6·3 (3)*
C(2)	3732 (6)	2496 (3)	4502 (7)	5·1 (3)*
N(3)	1288 (5)	1998 (3)	1342 (6)	6·2 (3)*
C(3)	956 (5)	1495 (4)	1236 (6)	4·8 (2)*
N(4)	3098 (6)	2240 (4)	937 (7)	8·9 (3)*
C(4)	3808 (7)	1933 (4)	611 (9)	6·9 (4)*
O	2820 (4)	3625 (3)	2284 (5)	6·1 (2)*
P(1)	2656 (1)	−1 (1)	5874 (1)	3·91 (5)*
P(2)	3150 (1)	5396 (1)	1115 (1)	3·88 (5)*
C(111)	4138 (5)	18 (3)	6413 (5)	4·0 (1)
C(112)	4932 (6)	−586 (4)	6783 (6)	5·2 (1)
C(113)	6078 (6)	−569 (4)	7187 (7)	5·8 (1)
C(114)	6449 (6)	26 (4)	7279 (7)	5·8 (1)
C(115)	5693 (7)	619 (4)	6949 (8)	6·6 (2)
C(116)	4531 (6)	614 (4)	6516 (6)	5·3 (1)
C(121)	1883 (5)	880 (3)	5709 (6)	4·1 (1)
C(122)	1795 (6)	1173 (4)	4742 (7)	5·7 (1)
C(123)	1198 (6)	1853 (4)	4588 (7)	5·8 (1)
C(124)	694 (7)	2213 (4)	5398 (7)	6·3 (2)
C(125)	779 (7)	1938 (5)	6366 (8)	7·3 (2)
C(126)	1392 (6)	1257 (4)	6530 (7)	5·8 (1)
C(131)	2494 (5)	−282 (3)	7069 (6)	4·2 (1)
C(132)	1787 (5)	−773 (3)	6732 (6)	4·7 (1)
C(133)	1624 (6)	−936 (4)	7676 (7)	5·8 (1)
C(134)	2167 (6)	−611 (4)	8932 (8)	6·2 (2)
C(135)	2889 (7)	−128 (4)	9286 (8)	6·7 (2)
C(136)	3072 (6)	34 (4)	8353 (7)	5·9 (2)
C(141)	2068 (5)	−602 (3)	4346 (5)	3·9 (1)
C(142)	959 (6)	−434 (4)	3457 (7)	5·5 (1)
C(143)	478 (7)	−919 (4)	2306 (7)	6·1 (2)
C(144)	1083 (6)	−1561 (4)	2032 (7)	5·7 (1)
C(145)	2193 (6)	−1727 (4)	2897 (7)	5·9 (1)
C(146)	2692 (6)	−1249 (3)	4055 (6)	5·0 (1)
C(211)	3100 (5)	6196 (3)	667 (5)	4·0 (1)
C(212)	2028 (6)	6551 (3)	−42 (6)	4·9 (1)
C(213)	1976 (6)	7138 (4)	−483 (7)	5·9 (1)
C(214)	2965 (6)	7363 (4)	−220 (7)	6·0 (2)
C(215)	4019 (6)	7025 (4)	488 (7)	6·0 (2)
C(216)	4085 (6)	6428 (3)	924 (6)	5·0 (1)
C(221)	4553 (5)	5179 (3)	2361 (6)	4·6 (1)
C(222)	5330 (7)	4642 (4)	2076 (8)	6·3 (2)
C(223)	6421 (8)	4487 (5)	3070 (9)	7·9 (2)
C(224)	6688 (8)	4877 (5)	4261 (9)	7·5 (2)
C(225)	5976 (9)	5408 (5)	458 (1)	8·9 (2)
C(226)	4851 (8)	5567 (5)	3597 (9)	8·0 (2)
C(231)	2087 (5)	5541 (3)	1702 (6)	4·3 (1)
C(232)	2004 (6)	6190 (4)	2574 (7)	5·9 (2)
C(233)	1224 (7)	6279 (5)	3099 (8)	7·1 (2)
C(234)	561 (7)	5771 (4)	2765 (8)	6·6 (2)
C(235)	644 (6)	5153 (4)	1914 (7)	5·9 (2)
C(236)	1413 (5)	5043 (3)	1383 (6)	4·8 (1)
C(241)	2889 (5)	4673 (3)	−270 (6)	4·1 (1)
C(242)	3026 (6)	3974 (3)	−141 (7)	5·1 (1)
C(243)	2847 (6)	3419 (4)	−1224 (7)	6·0 (2)
C(244)	2551 (6)	3557 (4)	−2398 (8)	6·2 (2)
C(245)	2405 (6)	4241 (4)	−2539 (8)	6·2 (2)
C(246)	2563 (5)	4805 (4)	−1478 (6)	5·0 (1)
C(11)	669 (1)	2717 (7)	385 (1)	11·9 (4)
C(12)	554 (1)	2963 (8)	347 (1)	13·9 (4)
N(11)	759 (1)	2645 (8)	384 (1)	17·6 (5)

* Equivalent isotropic thermal parameters: $B_{eq} = \frac{8}{3}\pi^2 \sum_i \sum_j U_{ij} a_i^* a_j^* \mathbf{a}_i.\mathbf{a}_j$.

Table 2. *Bond lengths* (Å) *and angles* (°) *for the anion*

W–O	1·715 (6)	W–S(5)	2·108 (2)
W–N(1)	2·043 (8)	W–N(3)	2·189 (8)
W–N(2)	2·211 (6)	W–N(4)	2·091 (8)
N(1)–C(1)	1·14 (1)	C(1)–S(1)	1·59 (1)
N(2)–C(2)	1·136 (9)	C(2)–S(2)	1·601 (7)
N(3)–C(3)	1·15 (1)	C(3)–S(3)	1·600 (9)
N(4)–C(4)	1·16 (1)	C(4)–S(4)	1·54 (1)
N(1)–W–N(2)	83·2 (3)	O–W–N(1)	96·6 (3)
N(1)–W–N(3)	83·4 (3)	O–W–N(2)	87·5 (3)
N(1)–W–N(4)	163·7 (3)	O–W–N(3)	165·2 (2)
N(1)–W–S(5)	96·1 (2)	O–W–N(4)	93·0 (3)
N(2)–W–N(3)	77·9 (2)	O–W–S(5)	103·3 (2)
N(2)–W–N(4)	84·0 (3)	N(3)–W–N(4)	84·0 (3)
N(2)–W–S(5)	169·2 (2)	N(3)–W–S(5)	91·4 (2)
N(4)–W–S(5)	94·6 (2)		
W–N(1)–C(1)	176·6 (6)	N(1)–C(1)–S(1)	178·5 (7)
W–N(2)–C(2)	165·1 (6)	N(2)–C(2)–S(2)	179 (1)
W–N(3)–C(3)	176·2 (6)	N(3)–C(3)–S(3)	179 (1)
W–N(4)–C(4)	169·0 (8)	N(4)–C(4)–S(4)	179 (1)

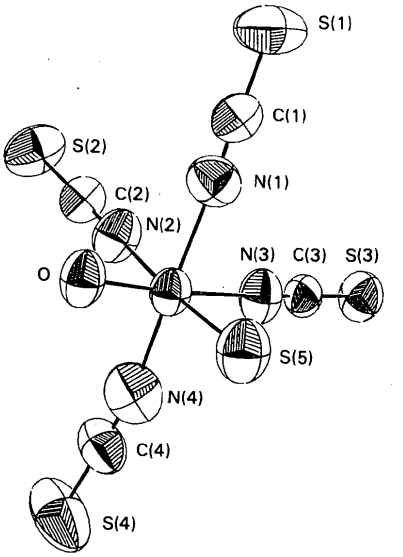

Fig. 1. Perspective view of the $[WOS(NCS)_4]$ dianion with the atomic labelling scheme.

bisector with C–H = 0·95 Å and $U(H) = 1{\cdot}1U(C)$. Final $R = 0{\cdot}050$, $w = 1$, $S = 2{\cdot}044$, $(\Delta/\sigma)_{max} = 0{\cdot}20$, largest peak in final difference map 1·03 e Å^{-3}, largest hole −0·85 e Å^{-3}, atomic scattering factors from *International Tables for X-ray Crystallography* (1974). Gould concept 32/87 computer. Atomic coordinates are given in Table 1, bond lengths and angles for the anion in Table 2.* Fig. 1 shows the structure of the anion and the atom-labelling scheme.

* Lists of structure factors, anisotropic thermal parameters, H-atom parameters and intramolecular distances and angles in the anion and the cation have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 44453 (60 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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