Structural Studies of Nitrido-complexes: *X*-Ray Crystal Structure of Tetraphenylarsonium Pentakis(isothiocyanato)nitridorhenate(VI)

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The crystal structure of the title complex $[AsPh_4]_2[ReN(NCS)_5]$ has been determined from single-crystal *X*-ray data. The crystals are monoclinic, with unit-cell dimensions a = 12.330(1), b = 18.600(1), c = 13.385(1) Å, β = 118.74(1)°, space group *Pc*, and Z = 2. Least-squares refinement has reached R 0.029 for 4 074 independent reflections. The structure consists of tetrahedral tetraphenylarsonium cations, and octahedral $[ReN(NCS)_5]^{2-}$ anions which contain a terminal nitride ligand and NCS groups bonded through the nitrogen atoms. Approximate 4*mm* (C_{4v}) point symmetry is found for the anion, with the rhenium atom displaced 0.21 Å out of the plane of four equatorial nitrogens and N(nitride)–Re–N(isothiocyanate) angles of *ca.* 96°. The triply bonded Re–N(nitride) distance is 1.657 Å. The mean Re–N(equatorial) bond length is 2.021 Å, while the Re–N(axial) bond *trans* to the nitride ligand is appreciably longer at 2.307 Å. In the tetraphenylarsonium cations the mean As–C bond length is 1.910 Å.

MONONUCLEAR nitrido-complexes are formed by metals in their higher oxidation state. In general, these complexes are more common for second- and third-row transition metals than for first row. It has been suggested[1] that this is a reflection of the high-oxidation-state stabilisation brought about by nitride, a factor which will favour the formation of such complexes with heavier elements, and is consistent with the role of N^{3-} as a very strong π-donor ligand.

As part of a program of structural studies on transition-metal nitrido-complexes[2] we report here *X*-ray crystallographic data on a complex of stoicheiometry $[AsPh_4]_2[ReN(NCS)_5]$ which has recently been prepared by Gibson and Lack.[3] This is the first rhenium(VI) nitrido-complex to be characterised by *X*-ray methods. We have determined its crystal structure in order to decide whether the thiocyanate groups were *N*- or *S*-bonded and to provide structural detail for the e.s.r. study being carried out by Gibson and Lack on this and similar complexes.

EXPERIMENTAL

Tetraphenylarsonium pentakis(isothiocyanato)nitridorhenate(VI) was obtained[3] by refluxing potassium thiocyanate with $[AsPh_4][ReNCl_4]$ in methanol in the presence of excess of $[AsPh_4][NCS]$, and was recrystallised from chloroform as deep red plates. Preliminary Weissenberg photographs showed the crystal to be monoclinic with systematic absences $h0l$, $l = 2n + 1$, consistent with space groups $P2/c$ and Pc. Accurate unit-cell dimensions were obtained by measurement of 16 high-angle α_1 reflections on a diffractometer, and performing a least-squares calculation.

Crystal Data.—$C_{53}H_{40}As_2N_6ReS_5$, $M = 1\,257.3$, Monoclinic, $a = 12.330(1)$, $b = 18.600(1)$, $c = 13.385(1)$ Å, $\beta = 118.74(1)°$, $U = 2\,691.5$ Å^3, $D_m = 1.53$ g cm^{-3} (by flotation), $Z = 2$, $D_c = 1.53$, $F(000) = 1\,242$. Space group Pc (no. 7). Cu-K_α radiation, $\lambda = 1.541\,8$ Å, $\mu(\text{Cu-}K_\alpha) = 74.9$ cm^{-1}.

Intensity data were collected for a crystal of dimensions *ca.* $0.12 \times 0.15 \times 0.20$ mm mounted about its c axis on a Siemens off-line automatic four-circle diffractometer. A 'five-value' measuring procedure (one side of peak, background, full peak, background on other side of peak, other side of peak) was employed.[4] Cu-K_α radiation at a take-off angle of 3°, a nickel β filter, and a Na(Tl)I scintillation counter were used. A total of 4 075 independent reflections (to θ 61°) were measured by use of the θ—2θ technique, and 148 of these were judged to be 'unobserved' since their net count was less than 2.58σ. The 803 reflection was measured as a reference every 50 reflections and its net count did not alter significantly during the data collection (*ca.* 8 d). This reflection was used to scale the data, and Lorentz and polarisation corrections were applied.

Solution and Refinement of the Structure.—Given a Z value of 2 and the stoicheiometry of the title complex, the most likely assumption was that it crystallised in space group $P2/c$, with $[AsPh_4]^+$ cations in general positions and the rhenium atom on a special position on a two-fold axis. An analysis of the reflection intensity statistics, however, showed clearly that the structure was non-centrosymmetric, and calculations were based on space group Pc, with two independent $[AsPh_4]^+$ cations. A few cycles of least-squares refinement gave R 0.39 for the rhenium atom only, while R decreased to 0.25 when the two arsenic atoms were included. Even at this relatively low R factor considerable

TABLE 1

Fractional co-ordinates with estimated standard deviations in parentheses

Atom	*x*	*y*	*z*
Re(1)	0(—)	0.739 55(2)	0.750 00(—)
N(1)	−0.084 4(8)	0.834 9(4)	0.720 2(7)
N(2)	−0.169 0(7)	0.691 2(4)	0.692 2(7)
N(3)	0.081 4(7)	0.643 5(4)	0.808 1(6)
N(4)	0.165 0(7)	0.789 7(4)	0.839 1(7)
N(5)	−0.004 2(8)	0.745 2(5)	0.920 3(8)
N(6)	0.004 6(10)	0.735 8(5)	0.628 4(7)
C(1)	−0.144 1(8)	0.887 3(4)	0.687 8(8)
C(2)	−0.269 1(8)	0.672 2(5)	0.658 7(7)
C(3)	0.131 6(8)	0.587 4(5)	0.827 2(8)
C(4)	0.258 4(9)	0.819 5(5)	0.881 5(8)
C(5)	−0.013 0(9)	0.755 9(5)	1.001 2(8)
S(1)	−0.221 8(3)	0.959 2(2)	0.645 3(3)
S(2)	−0.406 4(3)	0.644 4(2)	0.610 8(2)
S(3)	0.197 4(3)	0.512 5(2)	0.852 8(4)
S(4)	0.387 6(3)	0.861 2(2)	0.936 4(3)
S(5)	−0.025 4(3)	0.770 1(2)	1.113 5(2)
As(1)	0.343 80(8)	0.856 22(4)	0.558 08(7)
C(11)	0.377 9(8)	0.858 7(4)	0.434 6(7)
C(12)	0.497 1(10)	0.852 7(6)	0.454 5(9)
C(13)	0.521 4(13)	0.850 8(6)	0.361 4(12)
C(14)	0.426 3(14)	0.854 1(6)	0.253 3(10)
C(15)	0.306 2(15)	0.860 0(6)	0.231 6(9)
C(16)	0.279 2(11)	0.862 6(6)	0.321 1(9)
C(21)	0.220 0(7)	0.925 7(4)	0.536 7(7)
C(22)	0.199 4(9)	0.984 0(5)	0.468 6(8)
C(23)	0.111 3(11)	1.035 0(6)	0.458 2(11)
C(24)	0.048 4(10)	1.026 0(7)	0.515 8(14)
C(25)	0.069 4(11)	0.967 5(7)	0.589 4(12)
C(26)	0.156 1(9)	0.916 8(5)	0.598 0(9)
C(31)	0.289 0(8)	0.762 7(4)	0.568 1(8)
C(32)	0.213 4(10)	0.724 4(5)	0.468 7(10)
C(33)	0.174 0(12)	0.655 6(6)	0.477 3(13)
C(34)	0.206 5(12)	0.626 5(6)	0.578 1(14)
C(35)	0.278 1(11)	0.664 2(6)	0.680 5(12)
C(36)	0.320 1(8)	0.732 8(5)	0.675 5(9)
C(41)	0.490 7(7)	0.877 5(4)	0.697 7(7)
C(42)	0.501 2(10)	0.943 3(4)	0.746 9(10)
C(43)	0.607 2(10)	0.957 7(6)	0.848 6(10)
C(44)	0.697 0(9)	0.906 0(7)	0.900 5(9)
C(45)	0.684 2(9)	0.840 2(6)	0.850 6(9)
C(46)	0.579 4(8)	0.825 0(5)	0.747 8(8)
As(2)	0.664 35(8)	0.356 22(4)	0.722 73(7)
C(51)	0.636 4(8)	0.387 1(4)	0.576 6(7)
C(52)	0.692 0(9)	0.350 8(5)	0.524 0(8)
C(53)	0.665 0(12)	0.371 6(6)	0.414 6(9)
C(54)	0.582 4(10)	0.425 9(6)	0.360 0(8)
C(55)	0.529 5(11)	0.461 9(6)	0.412 8(9)
C(56)	0.553 9(10)	0.441 8(5)	0.522 4(8)
C(61)	0.506 3(7)	0.350 8(4)	0.717 9(6)
C(62)	0.402 0(10)	0.332 3(8)	0.614 0(9)
C(63)	0.288 8(9)	0.326 0(9)	0.609 2(10)
C(64)	0.279 2(9)	0.337 5(7)	0.706 6(9)
C(65)	0.379 4(10)	0.347 9(7)	0.806 6(9)
C(66)	0.494 9(9)	0.353 9(6)	0.813 0(7)
C(71)	0.771 9(7)	0.422 0(4)	0.838 3(7)
C(72)	0.808 8(8)	0.408 2(5)	0.950 6(7)
C(73)	0.887 4(9)	0.456 3(5)	1.034 2(8)
C(74)	0.928 2(9)	0.515 7(6)	1.002 0(10)
C(75)	0.892 8(10)	0.530 1(6)	0.889 4(11)
C(76)	0.814 1(10)	0.482 2(5)	0.805 3(9)
C(81)	0.737 5(8)	0.262 3(4)	0.755 2(7)
C(82)	0.854 5(11)	0.251 2(5)	0.844 2(10)
C(83)	0.903 7(11)	0.183 2(7)	0.864 3(10)
C(84)	0.834 6(13)	0.127 3(6)	0.798 6(12)
C(85)	0.720 1(10)	0.137 5(5)	0.709 2(10)
C(86)	0.670 3(9)	0.206 8(5)	0.686 0(8)

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[1] W. P. Griffith, *Co-ordination Chem. Rev.*, 1972, **8**, 369.

[2] F. L. Phillips and A. C. Skapski, *Acta Cryst.*, 1975, **B31**, 2667; F. L. Phillips, A. C. Skapski, and M. J. Withers, *Transition Metal Chem.*, 1975/1976, **1**, 28; F. L. Phillips and A. C. Skapski, *J. Cryst. Mol. Structure*, 1975, **5**, 83 and refs. therein.

[3] J. F. Gibson and G. M. Lack, unpublished work.

[4] F. H. Allen, D. Rogers, and P. G. H. Troughton, *Acta Cryst.*, 1971, **B27**, 1325.

difficulty was encountered in locating the lighter atoms. With all the atoms of the anion R was 0.175, while isotropic refinement with all the phenyl rings included gave R 0.067.

At this point a correction for absorption was applied to the data according to the method of Busing and Levy [5] using a $12 \times 12 \times 12$ grid and crystal path lengths determined by the vector-analysis procedure of Coppens *et al.*[6] This reduced R to 0.061, while a few cycles of anisotropic refinement gave R 0.039. It was now possible to locate the hydrogen atoms, and these were included as a fixed atom contribution with temperature factors of the parent carbon atoms to give R 0.034.

The anomalous-dispersion correction was now included, and refinement was carried out with the two alternative sets of co-ordinates, xyz and $\bar{x}\bar{y}\bar{z}$, to give R factors of 0.041 and 0.032 respectively. The latter set was clearly the correct one, and these co-ordinates were used subsequently. Introduction of a weighting scheme and removal of the strongest reflection for extinction reduced R to its final value of 0.029.

TABLE 2

Fractional co-ordinates of the hydrogen atoms

Atom *	x	y	z
H(12)	0.567	0.849	0.535
H(13)	0.610	0.850	0.370
H(14)	0.437	0.861	0.175
H(15)	0.248	0.863	0.145
H(16)	0.193	0.864	0.305
H(22)	0.243	0.989	0.420
H(23)	0.088	1.073	0.400
H(24)	−0.017	1.061	0.513
H(25)	0.010	0.954	0.620
H(26)	0.170	0.877	0.645
H(32)	0.200	0.748	0.398
H(33)	0.123	0.627	0.403
H(34)	0.177	0.579	0.593
H(35)	0.293	0.641	0.745
H(36)	0.380	0.759	0.735
H(42)	0.433	0.983	0.710
H(43)	0.617	1.009	0.883
H(44)	0.773	0.918	0.970
H(45)	0.733	0.798	0.890
H(46)	0.570	0.773	0.705
H(52)	0.753	0.309	0.563
H(53)	0.707	0.346	0.373
H(54)	0.563	0.443	0.275
H(55)	0.470	0.505	0.370
H(56)	0.513	0.468	0.563
H(62)	0.407	0.325	0.538
H(63)	0.210	0.314	0.528
H(64)	0.193	0.333	0.698
H(65)	0.367	0.352	0.875
H(66)	0.577	0.364	0.888
H(72)	0.780	0.363	0.970
H(73)	0.917	0.445	1.118
H(74)	0.987	0.550	1.063
H(75)	0.927	0.575	0.863
H(76)	0.787	0.491	0.718
H(82)	0.897	0.293	0.893
H(83)	0.987	0.171	0.928
H(84)	0.873	0.073	0.808
H(85)	0.673	0.096	0.650
H(86)	0.587	0.216	0.615

* The atoms are labelled so that they have the same number as the carbon atom to which they are attached.

The weighting scheme used in the final stages of refinement was that of Hughes [7] where $w = 1$ for $F < F^*$, $w^{\frac{1}{2}} = F^*/F$ for $F \geqslant F^*$, and $F^* = 45$ is the optimum value. Atomic-scattering factors for rhenium and sulphur were those reported by Cromer and Waber,[8] for arsenic, nitrogen, and carbon by Doyle and Turner,[9] and for hydrogen by Stewart *et al.*[10] Values of the anomalous-dispersion correction for rhenium, arsenic, and sulphur were those given by Cromer and Liberman.[11] The July 1972 version [12] of the '*X*-Ray '63' system [13] was used for the solution and refinement (CRYLSQ program) of the structure. Calculations were carried out on the University of London CDC 7600 computer, while structural illustrations were drawn with the aid of the Imperial College CDC 6400. Table 1 lists the final atomic fractional co-ordinates, and Table 2 the unrefined hydrogen atom co-ordinates. The anisotropic thermal parameters and the observed and calculated structure amplitudes are listed in Supplementary Publication No. SUP 22198 (20 pp.).*

DISCUSSION

Of the two ionic species found in the structure, $[AsPh_4]^+$ and $[ReN(NCS)_5]^{2-}$, the anion is the main focus of interest. Its structure is shown in Figure 1, together with the thermal-vibration ellipsoids.[14] The rhenium atom is co-ordinated to a triply bonded terminal nitride ligand and to five NCS groups, to give a somewhat distorted octahedral geometry of approximate point symmetry $4mm$ (C_{4v}). An N-bonded mode of binding is found, which is an exception to the general rule [15] that transition-metal elements in the second half of the second series and those of the third series form S-bonded thio-

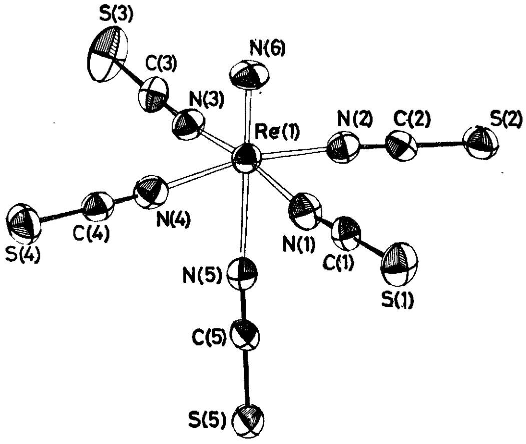

FIGURE 1 Structure of the $[ReN(NCS)_5]^{2-}$ anion. Thermal-vibration ellipsoids are scaled to include 26% probability

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* For details see Notices to Authors No. 7, *J.C.S. Dalton*, 1977, Index issue.

[5] W. R. Busing and H. A. Levy, *Acta Cryst.*, 1957, **10**, 180.

[6] P. Coppens, L. Leiserowitz, and D. Rabinovich, *Acta Cryst.*, 1965, **18**, 1035.

[7] E. W. Hughes, *J. Amer. Chem. Soc.*, 1941, **63**, 1737.

[8] D. T. Cromer and J. T. Waber, *Acta Cryst.*, 1965, **18**, 104.

[9] P. A. Doyle and P. S. Turner, *Acta Cryst.*, 1968, **A24**, 390.

[10] R. F. Stewart, E. R. Davidson, and W. T. Simpson, *J. Chem. Phys.*, 1965, **42**, 3175.

[11] D. T. Cromer and D. Liberman, *J. Chem. Phys.*, 1970, **53**, 1891.

[12] J. M. Stewart, G. J. Kruger, H. L. Ammon, C. Dickinson, and S. R. Hall, University of Maryland Technical Report, TR 192.

[13] '*X*-Ray '63' system of programs, ed. J. M. Stewart, University of Maryland Technical Report TR 646, July 1972 version.

[14] C. K. Johnson, ORTEP, Oak Ridge National Laboratory, Report ORNL 3794, 1965.

[15] A. F. Wells, 'Structural Inorganic Chemistry,' 4th edn., Clarendon Press, Oxford, 1975, p. 746.

cyanates or bridged compounds. The more important bond lengths and bond angles are listed in Tables 3 and 4 respectively.

TABLE 3

Selected bond lengths (Å) with standard deviations in parentheses

(*a*) Anion			
Re(1)–N(1)	1.997(8)	Re(1)–N(4)	2.024(8)
Re(1)–N(2)	2.050(8)	Re(1)–N(5)	2.307(12)
Re(1)–N(3)	2.013(7)	Re(1)–N(6)	1.657(12)
N(1)–C(1)	1.172(12)	C(1)–S(1)	1.583(9)
N(2)–C(2)	1.147(13)	C(2)–S(2)	1.582(10)
N(3)–C(3)	1.178(12)	C(3)–S(3)	1.565(10)
N(4)–C(4)	1.152(12)	C(4)–S(4)	1.598(10)
N(5)–C(5)	1.156(17)	C(5)–S(5)	1.603(13)
Mean N–C	1.161	Mean C–S	1.586
(*b*) Cations			
As(1)–C(11)	1.892(11)	As(2)–C(51)	1.904(9)
As(1)–C(21)	1.913(9)	As(2)–C(61)	1.921(10)
As(1)–C(31)	1.895(8)	As(2)–C(71)	1.917(7)
As(1)–C(41)	1.917(6)	As(2)–C(81)	1.917(8)
Mean As–C	1.910	Mean C–C (phenyl rings)	1.380

The Re≡N bond length is 1.657 Å. There do not appear to be any comparable rhenium(VI) nitrido-complexes which have been structurally characterised, although there are data available for rhenium(V) complexes. For instance, in $[ReN(S_2CNEt_2)_2]$ the Re-N distance is 1.656 Å,[16] while in $[ReNCl_2(PPh_3)_2]$ it is 1.603 Å.[17] Both these complexes are five-co-ordinate. Normally, a bond length in a five-co-ordinate complex will be slightly shorter than in an equivalent six-co-ordinate complex; on the other hand a rhenium(V) distance should be slightly longer than one of Re^{VI}. Therefore the distance found in the title complex agrees reasonably well with the other values.

Because N^{3-} is a very strong π-donor ligand, in all the terminal nitrido-complexes there is evidence for a pushing away of the adjacent groups from their normal positions. Both steric and π-electronic factors may be involved in this process. In the title complex the mean of the N(nitride)–Re–N(equatorial isothiocyanate) angles is 96°, such that the rhenium atom is 0.21 Å out of the plane of the equatorial nitrogen atoms in the direction of the nitrido-ligand. In $[AsPh_4]_2[NbO(NCS)_5]$ the O–Nb–N(equatorial) angles range from 93 to 97°,[18] and although the final standard deviations are high the situation is essentially comparable.

TABLE 4

Selected bond angles (°) with standard deviations in parentheses

(*a*) Anion			
N(1)–Re(1)–N(6)	95.9(4)	N(1)–Re(1)–N(5)	84.2(4)
N(2)–Re(1)–N(6)	96.8(5)	N(2)–Re(1)–N(5)	83.8(4)
N(3)–Re(1)–N(6)	95.5(4)	N(3)–Re(1)–N(5)	84.4(4)
N(4)–Re(1)–N(6)	95.7(5)	N(4)–Re(1)–N(5)	83.7(4)
N(1)–Re(1)–N(2)	88.7(3)	N(3)–Re(1)–N(4)	90.5(3)
N(2)–Re(1)–N(3)	89.1(3)	N(4)–Re(1)–N(1)	89.3(3)
N(1)–Re(1)–N(3)	168.6(4)	N(5)–Re(1)–N(6)	179.4(4)
N(2)–Re(1)–N(4)	167.5(4)		
Re(1)–N(1)–C(1)	170.8(7)	N(1)–C(1)–S(1)	178.7(9)
Re(1)–N(2)–C(2)	171.8(7)	N(2)–C(2)–S(2)	178.5(1.0)
Re(1)–N(3)–C(3)	169.5(1.0)	N(3)–C(3)–S(3)	179.5(1.1)
Re(1)–N(4)–C(4)	174.5(1.0)	N(4)–C(4)–S(4)	178.1(1.1)
Re(1)–N(5)–C(5)	171.9(8)	N(5)–C(5)–S(5)	179.6(1.0)
(*b*) Cations			
C(11)–As(1)–C(21)	110.4(4)	C(51)–As(2)–C(61)	107.5(4)
C(11)–As(1)–C(31)	108.7(4)	C(51)–As(2)–C(71)	110.5(4)
C(11)–As(1)–C(41)	110.1(4)	C(51)–As(2)–C(81)	109.9(4)
C(21)–As(1)–C(31)	110.2(4)	C(61)–As(2)–C(71)	110.9(4)
C(21)–As(1)–C(41)	108.6(3)	C(61)–As(2)–C(81)	108.6(4)
C(31)–As(1)–C(41)	108.8(3)	C(71)–As(2)–C(81)	109.4(3)

Of the five Re–N(isothiocyanate) distances, the four equatorial ones are similar (mean 2.021 Å), while the axial Re–N *trans* to the nitride is much longer (2.307 Å). Again there are no direct comparisons available for Re^{VI}; however, in $[ReCl_3(PPh_3)_2(NCMe)]$ the Re–N distance is 2.05 Å,[19] while in $[AsPh_4][ReOBr_4(NCMe)]$ the

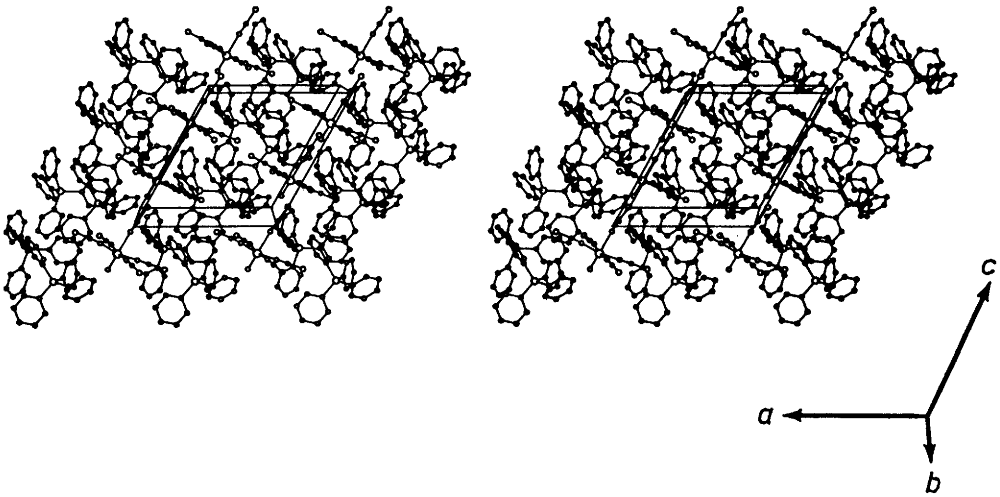

FIGURE 2 Stereoscopic view showing the packing of $[AsPh_4]^+$ and $[ReN(NCS)_5]^{2-}$ ions

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[16] S. R. Fletcher and A. C. Skapski, *J.C.S. Dalton*, 1972, 1079.

[17] R. J. Doedens and J. A. Ibers, *Inorg. Chem.*, 1967, **6**, 204.

[18] B. Kamenar and C. K. Prout, *J. Chem. Soc.* (*A*), 1970, 2379.

[19] M. G. B. Drew and D. G. Tisley, *Chem. Comm.*, 1970, 600.

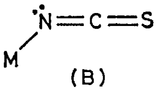
Re–N distance *trans* to oxygen is 2.31 Å.[20] In the title complex the long axial distance seems to be a result of the steric repulsion by the equatorial isothiocyanate groups, although one would also expect a *trans* influence due to the nitrido-ligand to be a contributing factor.

There are no statistically significant differences in the bond lengths of the five isothiocyanate groups, and mean values are 1.16 (N–C) and 1.586 Å (C–S). The groups themselves are linear, with N–C–S angles in the range 178.1—179.6°; however, there does seem to be some slight bending at the nitrogen atoms, where Re–N–C bond angles vary from 169.5 to 174.5°. This bending could be explained simply in terms of the packing requirements and steric interactions. Both N–C and C–S bonds have some multiple-bond character; therefore, if some bending of the long pole-like structure of the NCS group were necessary for packing it will come about at the nitrogen atom.

An alternative electronic argument has also been put forward.[21-23] From an examination of the two resonance structures (A) and (B) one would expect the distances

$$M-\overset{+}{N}\equiv C-S^{-}$$

(A)



(B)

N–C and C–S and the angle M–N–C to be interdependent in the nitrogen-bonded isothiocyanate complexes, due to the hybridisation requirements of forms (A) and (B). To detect correlation between these distances and the angle, Hazell[21] suggested plotting each one against the angle. From crystallographic data for 12 isothiocyanates he showed that the N–C distance generally decreases with increasing M–N–C angle, while the C–S distance increases. These experimental values were compared, in the same plot, with the curves representing the values calculated[24,25] by assuming resonance between forms (A) and (B). From this comparison he came to the conclusion that N–C distances were in good agreement with the theoretical curve but S–C bond lengths were generally shorter than predicted, and suggested that this could be attributed to d_{π}–p_{π} bonding between sulphur and carbon atoms. For the title complex we find that the mean Re–N–C angle (171.7°) and mean bond lengths (N–C 1.16 and C–S 1.586 Å), fall well within the ranges defined by the other data points, and are consistent with a dominant contribution from resonance form (A).

In the two independent tetraphenylarsonium cations the mean of the eight As–C bond distances (1.910 Å) is in excellent agreement with equivalent distances reported for other complexes.[26] The C–As–C angles (107.5—110.9°), suggest that there is little, if any, distortion from ideal tetrahedral geometry. The packing arrangement of the ions in the unit cell is shown in Figure 2 as a pair of stereoscopic drawings.[14]

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