$O(2^i)$—Na(1)—O(7)	179.74 (9)	O(1)—S(1)—O(2)	114.6 (1)
O(1)—Na(1)—O(8)	164.92 (9)	O(1)—S(1)—O(3)	112.8 (1)
$O(6^{ii})$—Na(1)—O(9)	158.41 (9)	O(1)—S(1)—C(1)	106.6 (1)
O(7)—Na(1)—O(9)	87.90 (9)	O(2)—S(1)—O(3)	110.8 (1)
O(8)—Na(1)—O(9)	77.14 (9)	O(2)—S(1)—C(1)	105.8 (1)
O(7)—Na(1)—O(8)	95.83 (9)	O(3)—S(1)—C(1)	105.4 (1)
O(1)—Na(1)—O(7)	86.28 (9)	O(4)—C(4)—C(3)	127.5 (3)
O(1)—Na(1)—O(9)	88.04 (9)	O(4)—C(4)—C(5)	114.7 (3)

Symmetry codes: (i) $x, y, z-1$; (ii) $2-x, -y, -z$.

Table 3. *Hydrogen-bonding geometry* (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*—H···*A*
O(9)—H(10)···$O(2^i)$	0.80 (3)	2.15 (3)	163 (3)
O(7)—H(5)···$O(3^{ii})$	0.81 (3)	2.04 (3)	162 (3)
O(9)—H(9)···$O(3^{iii})$	0.79 (3)	2.09 (3)	166 (3)
O(4)—H(4)···$O(7^{iv})$	0.75 (3)	2.18 (3)	134 (3)
O(8)—H(7)···$O(9^v)$	0.79 (3)	2.14 (3)	176 (3)
O(7)—H(6)···$O(8^{vi})$	0.79 (3)	2.09 (3)	165 (3)
O(4)—H(4)···O(6)	0.75 (3)	2.07 (3)	138 (3)

Symmetry codes: (i) $2-x, 1-y, 1-z$; (ii) $1+x, y, z$; (iii) $2-x, 1-y, -z$; (iv) $2-x, -y, 1-z$; (v) $3-x, 1-y, -z$; (vi) $x, y, 1+z$.

All H atoms were located on difference electron density maps and assigned isotropic displacement parameters equal to 1.2*B* of the attached atom at the time of their inclusion in the model.

Data collection and cell refinement: *MSC/AFC Diffractometer Control Software* (Molecular Structure Corporation, 1988). Data reduction: *TEXSAN* (Molecular Structure Corporation, 1991). Structure solution: *MITHRIL* (Gilmore, 1983), *DIRDIF* (Beurskens *et al.*, 1984). Structure refinement: *TEXSAN*. Molecular graphics: *ORTEP*II (Johnson, 1976).

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates, complete geometry, including H-atom geometry, intermolecular distances involving H atoms and least-squares-planes data have been deposited with the IUCr (Reference: CR1155). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Isothiocyanato Complexes of Rhenium. V. *trans*-Bis(isothiocyanato)(methoxy)(phenylimido)bis(triphenylphosphine)rhenium(V)†

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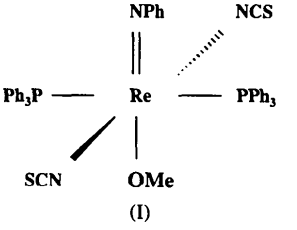
Abstract

The title compound consists of well separated molecules of $[Re(C_6H_5N)(CH_3O)(NCS)_2(C_{18}H_{15}P)_2]$. The Re atom is six-coordinate in a distorted octahedral environment. The two triphenylphosphine ligands are arranged *trans* to each other and there is a methoxy group in the position *trans* to the phenylimido group. The Re—NPh distance is 1.744 (4) Å and the NCS ligands are coordinated *via* the N atom.

Comment

The title compound, (I), consists of discrete monomeric molecules. An *ORTEP*II plot (Johnson, 1976) together with the atomic numbering scheme is shown in Fig. 1.



(I)

The Re atom is six-coordinate, the two *trans* triphenylphosphine ligands are bonded to the Re atom in an approximately linear fashion with a P—Re—P angle of 178.78 (4)°. The Re—P bond distances, 2.506 (1)

† Part IV: see Hübener, Abram & Strähle (1995).

and 2.507 (1) Å, fall within the normal range of Re—P distances observed in six-coordinate tertiary phosphine complexes [2.45–2.51 Å (Drew, Tisley & Walton, 1970; Hübener, Abram & Strähle, 1994*b*)]. The two NCS^- ligands are nitrogen bonded as has been found for all structurally characterized rhenium complexes with this ligand (Hahn, Nimry, Robinson, Salmon & Walton, 1978; de Carrondo, Shakir & Skapski, 1978; Conner & Walton, 1987; Hübener & Abram, 1993; Hübener, Abram & Strähle, 1994*a*,*b*, 1995). The isothiocyanate ligands are almost linear [N—C—S angles between 177.8 (6) and 178.2 (5)°] and do not show any unusual bond distances. The short Re—O bond distance [1.922 (3) Å] of the coordinated methoxy group is a result of the negligible *trans* influence of the phenylimido ligand.

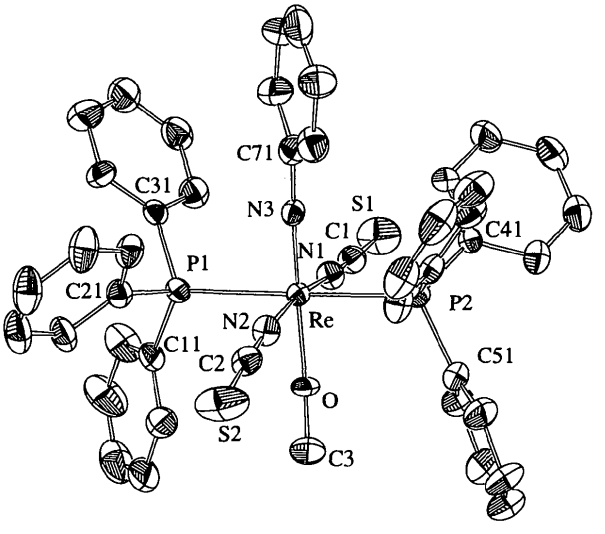


Fig. 1. *ORTEP*II (Johnson, 1976) plot of (I) with the atomic numbering scheme and 50% probability ellipsoids.

Experimental

$[Re(NPh)(OMe)(NCS)_2(Ph_3P)_2]$ can be prepared by the reaction of $[Re(NPh)Cl_3(Ph_3P)_2]$ (Chatt & Rowe, 1962) with KSCN or is formed in low yields by the reaction of the well known complex $[ReNCl_2(Ph_3P)_2]$ (Chatt, Garforth, Johnson & Rowe, 1964) with phenylhydrazine hydrochloride ($PhNH–NH_2.HCl$) and KSCN. The complex was crystallized from CH_2Cl_2/MeOH. The air-stable compound is only slightly soluble in organic solvents such as acetone or $CHCl_3$. IR studies carried out in KBr show the ν(NCS) stretching vibration for the isothiocyanate ligands to be at 2087 cm^{-1}; the Re=NPh absorption band is centred at 1077 cm^{-1}.

Crystal data

$[Re(C_6H_5N)(CH_3O)(NCS)_2(C_{18}H_{15}P)_2]$	Mo $K\alpha$ radiation
	λ = 0.71073 Å
M_r = 949.1	Cell parameters from 25 reflections
Triclinic	θ = 8–14°
$P\bar{1}$	μ = 3.2795 mm^{-1}
a = 10.216 (6) Å	T = 223 K
b = 13.236 (6) Å	Lozenge
c = 15.534 (7) Å	0.25 × 0.25 × 0.05 mm
α = 93.89 (3)°	Yellow–green
β = 99.92 (3)°	
γ = 102.28 (3)°	
V = 2009.7 (17) $Å^3$	
Z = 2	
D_x = 1.568 Mg m^{-3}	

Data collection

Enraf–Nonius CAD-4 diffractometer	R_{int} = 0.0149
ω scans	θ_{max} = 27°
Absorption correction: refined from ΔF (*DIFABS*; Walker & Stuart, 1983)	$h = -13 \rightarrow 13$
	$k = -17 \rightarrow 17$
	$l = -2 \rightarrow 20$
T_{min} = 0.64, T_{max} = 0.82	3 standard reflections monitored every 200 reflections
8863 measured reflections	frequency: 60 min
8096 independent reflections	intensity decay: 1.6%
6174 observed reflections [$I \geq 3\sigma(I)$]	

Refinement

Refinement on F^2	$\Delta\rho_{max}$ = 0.778 e $Å^{-3}$
$R(F)$ = 0.032	$\Delta\rho_{min}$ = −0.331 e $Å^{-3}$
$wR(F^2)$ = 0.036	Extinction correction: Zachariasen (1963)
S = 1.305	Extinction coefficient: 2.1928×10^{-9}
8096 reflections	Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV)
488 parameters	
H-atom parameters not refined	
$w = 1/\sigma^2(F)$	
$(\Delta/\sigma)_{max}$ = 0.003	

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters* ($Å^2$)

$U_{eq} = (1/3)\Sigma_i\Sigma_j U_{ij} a_i^* a_j^* \mathbf{a}_i.\mathbf{a}_j.$

	x	y	z	U_{eq}
Re	0.22662 (2)	0.23875 (2)	0.23529 (1)	0.02274 (7)
S1	0.0380 (2)	0.5292 (1)	0.3104 (1)	0.060 (1)
S2	0.3183 (3)	−0.0745 (1)	0.1112 (2)	0.079 (1)
P1	0.1251 (1)	0.1288 (1)	0.34300 (9)	0.0265 (6)
P2	0.3325 (1)	0.3504 (1)	0.13010 (8)	0.0271 (6)
O	0.0689 (4)	0.1860 (3)	0.1441 (2)	0.032 (2)
N1	0.1336 (5)	0.3565 (3)	0.2636 (3)	0.030 (2)
N2	0.2888 (5)	0.1104 (3)	0.1900 (3)	0.032 (2)
N3	0.3767 (4)	0.2834 (3)	0.3132 (3)	0.023 (2)
C1	0.0932 (6)	0.4295 (4)	0.2820 (4)	0.034 (3)
C2	0.3006 (6)	0.0325 (4)	0.1587 (4)	0.040 (3)
C3	0.0261 (8)	0.1153 (5)	0.0674 (4)	0.049 (4)
C11	−0.0591 (6)	0.0822 (5)	0.3162 (4)	0.036 (3)
C12	−0.1167 (8)	0.0255 (6)	0.2356 (5)	0.054 (4)
C13	−0.2600 (9)	−0.0082 (6)	0.2123 (5)	0.062 (5)
C14	−0.3406 (8)	0.0144 (6)	0.2691 (6)	0.070 (6)
C15	−0.2835 (7)	0.0692 (6)	0.3488 (6)	0.071 (6)
C16	−0.1429 (6)	0.1032 (5)	0.3735 (5)	0.050 (4)
C21	0.1900 (6)	0.0120 (4)	0.3601 (4)	0.034 (3)
C22	0.1042 (7)	−0.0834 (4)	0.3638 (4)	0.043 (3)
C23	0.1574 (8)	−0.1686 (5)	0.3821 (5)	0.066 (4)
C24	0.2951 (7)	−0.1593 (5)	0.3965 (5)	0.070 (4)

C25	0.3825 (6)	−0.0651 (5)	0.3934 (5)	0.062 (4)
C26	0.3304 (6)	0.0204 (5)	0.3745 (4)	0.043 (3)
C31	0.1624 (5)	0.1970 (4)	0.4543 (3)	0.029 (2)
C32	0.2179 (6)	0.1543 (5)	0.5274 (4)	0.039 (3)
C33	0.2440 (8)	0.2093 (6)	0.6103 (4)	0.053 (4)
C34	0.2133 (8)	0.3058 (5)	0.6207 (4)	0.054 (4)
C35	0.1578 (7)	0.3467 (5)	0.5495 (4)	0.050 (3)
C36	0.1311 (6)	0.2942 (5)	0.4652 (4)	0.043 (3)
C41	0.3655 (5)	0.4888 (4)	0.1634 (3)	0.027 (2)
C42	0.4055 (6)	0.5276 (4)	0.2516 (4)	0.034 (3)
C43	0.4351 (7)	0.6336 (5)	0.2772 (4)	0.044 (4)
C44	0.4250 (7)	0.7019 (5)	0.2137 (5)	0.049 (4)
C45	0.3879 (8)	0.6654 (5)	0.1275 (4)	0.053 (4)
C46	0.3586 (7)	0.5594 (5)	0.1007 (4)	0.045 (3)
C51	0.2238 (6)	0.3342 (4)	0.0226 (4)	0.036 (3)
C52	0.0982 (7)	0.3592 (5)	0.0187 (4)	0.048 (4)
C53	0.0061 (9)	0.3434 (7)	−0.0611 (5)	0.066 (5)
C54	0.039 (1)	0.3014 (7)	−0.1350 (5)	0.074 (7)
C55	0.163 (1)	0.2769 (7)	−0.1315 (5)	0.075 (7)
C56	0.2578 (8)	0.2943 (6)	−0.0529 (4)	0.053 (4)
C61	0.4985 (6)	0.3285 (4)	0.1156 (3)	0.034 (2)
C62	0.5075 (6)	0.2285 (5)	0.0891 (4)	0.049 (3)
C63	0.6359 (7)	0.2077 (5)	0.0882 (4)	0.060 (3)
C64	0.7508 (7)	0.2864 (7)	0.1111 (5)	0.073 (5)
C65	0.7424 (7)	0.3855 (7)	0.1347 (5)	0.060 (5)
C66	0.6150 (6)	0.4078 (5)	0.1367 (4)	0.042 (3)
C71	0.5033 (6)	0.3142 (5)	0.3657 (4)	0.037 (3)
C72	0.6174 (7)	0.2953 (5)	0.3324 (5)	0.050 (4)
C73	0.7459 (7)	0.3322 (6)	0.3821 (6)	0.056 (5)
C74	0.7649 (9)	0.3796 (6)	0.4644 (5)	0.061 (5)
C75	0.6493 (9)	0.4005 (6)	0.4971 (5)	0.053 (5)
C76	0.5254 (7)	0.3683 (6)	0.4515 (4)	0.049 (4)

Table 2. *Selected geometric parameters* (Å, °)

Re—P1	2.506 (1)	N1—C1	1.166 (8)
Re—P2	2.507 (1)	N2—C2	1.149 (8)
Re—N1	2.051 (5)	O—C3	1.407 (7)
Re—N2	2.057 (5)	N3—C71	1.368 (7)
Re—N3	1.744 (4)	C1—S1	1.606 (6)
Re—O	1.922 (3)	C2—S2	1.612 (6)
P1—Re—P2	178.78 (4)	N2—Re—N3	93.0 (2)
P1—Re—N1	93.1 (1)	N1—Re—O	86.0 (2)
P1—Re—N2	86.9 (1)	N2—Re—O	83.7 (2)
P1—Re—N3	89.9 (1)	N3—Re—O	175.9 (2)
P1—Re—O	92.2 (1)	Re—O—C3	140.2 (4)
P2—Re—N1	86.9 (1)	Re—N1—C1	173.5 (5)
P2—Re—N2	93.3 (1)	Re—N2—C2	168.4 (4)
P2—Re—N3	88.8 (1)	Re—N3—C71	172.2 (4)
P2—Re—O	89.1 (1)	N1—C1—S1	178.2 (5)
N1—Re—N2	169.8 (2)	N2—C2—S2	177.8 (6)
N1—Re—N3	97.3 (2)		

The positions of the H atoms were calculated and included in the structure-factor calculations. Cell refinement: *SET*4 (de Boer & Duisenberg, 1984). Data reduction: *SDP* (Enraf–Nonius, 1985). Program used to solve structure: *SHELXS*86 (Sheldrick, 1985). Programs used to refine structure: *SDP*. Refinement by full-matrix least-squares methods. Molecular graphics: *ORTEP*II (Johnson, 1976). Software used to prepare material for publication: *SDP*; *UTABLE* (Kretschmar, 1989). All computation was performed on a DEC VAX Station 3100.

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Lists of structure factors, anisotropic displacement parameters and H-atom coordinates have been deposited with the IUCr (Reference: JZ1024). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Tris[bis(ethylenediamine)copper(II)] Poly[hexakis(μ-cyano)hexakis(μ-selenocyanato)hexacopper(I)], $[Cu^{II}en_2]_3$-$[Cu^{I}_6(CN)_6(SeCN)_6]$

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Abstract

The structure of the title compound, $[Cu(C_2H_8N_2)_2]_3$-$[Cu_6(CN)_6(SeCN)_6]$, consists of a three-dimensional skeleton formed by the polymeric chains of anions, $[Cu^{I}_6(CN)_6(SeCN)_6]^{6-}$. In the cavities are located