

Table 2. Selected bond lengths (Å) and angles (°) and ranges of bond lengths and angles

C(1)–N(2)	1.45 (1)	C(5)–N(3)	1.39 (1)
C(1)–C(2)	1.33 (2)	N(3)–C(6)	1.38 (1)
C(2)–C(3)	1.48 (1)	C(6)–N(2)	1.37 (1)
C(3)–C(4)	1.33 (1)	C(5)–O(1)	1.23 (1)
C(4)–Sn(1)	2.174 (10)	C(6)–O(2)	1.22 (1)
Sn(1)–N(1)	2.120 (8)	N(3)–C(51)	1.42 (1)
N(1)–N(2)	1.44 (1)	Sn(1)–C(7)	2.185 (7)
N(1)–C(5)	1.36 (1)	Sn(1)–C(8)	2.164 (11)
C(2)–C(1)–N(2)	116.3 (7)	N(2)–N(1)–C(5)	106.3 (8)
C(2)–C(1)–C(41)	127.5 (7)	N(1)–N(2)–C(1)	110.4 (7)
N(2)–C(1)–C(41)	115.9 (9)	N(1)–N(2)–C(6)	108.1 (8)
C(1)–C(2)–C(3)	125.3 (7)	C(1)–N(2)–C(6)	118.9 (6)
C(1)–C(2)–C(31)	122.3 (8)	N(1)–C(5)–N(3)	109.1 (8)
C(3)–C(2)–C(31)	112.6 (9)	N(1)–C(5)–O(1)	126 (1)
C(2)–C(3)–C(4)	125 (1)	N(3)–C(5)–O(1)	151 (1)
C(2)–C(3)–C(21)	109.9 (7)	C(5)–N(3)–C(6)	108.6 (9)
C(4)–C(3)–C(21)	125.6 (9)	C(5)–N(3)–C(51)	126.0 (8)
C(3)–C(4)–Sn(1)	126.0 (8)	C(6)–N(3)–C(51)	125.2 (8)
C(3)–C(4)–C(11)	121.2 (9)	N(3)–C(6)–N(2)	107.7 (8)
Sn(1)–C(4)–C(11)	112.1 (5)	N(3)–C(6)–O(2)	127 (1)
C(4)–Sn(1)–N(1)	98.4 (3)	N(2)–C(6)–O(2)	125.7 (9)
C(4)–Sn(1)–C(7)	110.0 (4)	Sn(1)–C(7)–C(71)	111.0 (7)
C(4)–Sn(1)–C(8)	119.3 (4)	Sn(1)–C(7)–C(72)	109.7 (6)
N(1)–Sn(1)–C(7)	103.7 (3)	Sn(1)–C(7)–C(73)	108.2 (5)
N(1)–Sn(1)–C(8)	104.8 (4)	Sn(1)–C(8)–C(81)	109.7 (8)
C(7)–Sn(1)–C(8)	117.0 (4)	Sn(1)–C(8)–C(82)	110.3 (9)
Sn(1)–N(1)–N(2)	112.3 (6)	Sn(1)–C(8)–C(83)	108 (1)
Sn(1)–N(1)–C(5)	127.5 (5)		

In the phenyl rings C–C bond distances and C–C–C angles are in the range 1.33 (2) to 1.42 (2) Å [mean 1.37 (2) Å] and 117 (1) to 123 (1)° [mean 120 (1)°]. The C–C bond distances to the phenyl rings and in the *tert*-butyl groups are in the range 1.46 (1) to 1.52 (2) Å [mean 1.49 (2) Å] and the C–C–C angles in the *tert*-butyl groups are in the range 107 (1) to 111 (1)° [mean 109.6 (1)°].

coplanarity of the two double bonds. The dihedral angles between the planes through these double bonds – least-squares planes through Sn(1), C(4), C(11), C(3), C(2), C(21) and through C(3), C(2), C(31), C(1), N(2), C(41) – and the planes through the corresponding phenyl groups bound to the *sp*<sup>2</sup>-hybridized C atoms are in the range 53.5 (5) to 84.2 (5)° and therefore a significant resonance between the  $\pi$ -systems does not exist. A least-squares plane through the atoms C(5), C(6), N(1), N(2), N(3), O(1), O(2) shows that no atom deviates significantly from planarity. The dihedral angle between this plane and the least-squares plane through the phenyl group bound to the urazole ring is 45.0 (5)°.

Intermolecular distances do not indicate interactions exceeding van der Waals forces.

#### References

- CROMER, D. T. (1974). *International Tables for X-ray Crystallography*, Vol. IV, Table 2.3.1. Birmingham: Kynoch Press.
- CROMER, D. T. & WABER, J. T. (1974). *International Tables for X-ray Crystallography*, Vol. IV, Table 2.2B. Birmingham: Kynoch Press.
- FRENZ, B. A. (1981). *Enraf–Nonius Structure Determination Package*, 4th ed., version 18. Enraf–Nonius, Delft.
- GRUGEL, CH., NEUMANN, W. P. & SCHRIEWER, M. (1979). *Angew. Chem. Int. Ed. Engl.* **18**, 543–544.
- JOHNSON, C. K. (1976). ORTEPII. Report ORNL-5138. Oak Ridge National Laboratory, Tennessee.
- WAAL, B. W. VAN DE (1976). POP1. *Plot of Packing Program*. Twente Univ. of Technology, The Netherlands.

*Acta Cryst.* (1984). **C40**, 1795–1797

## Trichloro(phenylimido)bis(triphenylphosphine)rhenium(V) Dichloromethane (1/1), [ReCl<sub>3</sub>(C<sub>6</sub>H<sub>5</sub>N){P(C<sub>6</sub>H<sub>5</sub>)<sub>3</sub>}<sub>2</sub>].CH<sub>2</sub>Cl<sub>2</sub>

BY E. FORSELLINI AND U. CASELLATO

*Istituto di Chimica e Tecnologia dei Radioelementi del CNR, Padova, Italy*

R. GRAZIANI

*Istituto di Chimica Generale, Università di Padova, Italy*

AND M. C. CARLETTI AND L. MAGON

*Istituto Chimico, Università di Ferrara, Italy*

(Received 10 April 1984; accepted 30 May 1984)

**Abstract.**  $M_r = 920.85$ , triclinic,  $P\bar{1}$ ,  $a = 12.296$  (4),  $b = 16.961$  (5),  $c = 11.022$  (4) Å,  $\alpha = 74.00$  (5),  $\beta = 99.72$  (5),  $\gamma = 108.46$  (5)°,  $V = 2088$  (1) Å<sup>3</sup>,  $D_m$ (floatation) = 1.61,  $D_x = 1.60$  g cm<sup>-3</sup> for  $Z = 2$ , Mo  $K\alpha$ ,  $\lambda = 0.7107$  Å,  $\mu = 34$  cm<sup>-1</sup>,  $F(000) = 996$ , room temperature.  $R = 0.045$  and  $R_w = 0.049$  for 5812 observed reflections. The coordination about Re is

distorted octahedral with two apical triphenylphosphines and with the phenylimido group (coordinated to Re *via* the N) and the three Cl atoms in the basal plane. Distances are: Re–P 2.503 (2) and 2.490 (2) Å; Re–Cl 2.415 (2), 2.411 (2), 2.402 (2) Å; Re–N 1.726 (6) Å. The Re–N–C angle is close to linearity at 172.6 (6)°.

Table 1. Fractional positional parameters ( $\times 10^4$ , for Re  $\times 10^5$ ) and thermal parameters ( $\times 10^3$ )

	$U_{eq} = \frac{1}{3} \sum U_{ii}$			$U_{eq}$ or $U_{iso}$ ( $\text{\AA}^2$ )
	x	y	z	
Re	-15570 (3)	75224 (2)	62440 (3)	32.3 (2)
Cl(1)	-625 (2)	8863 (1)	6840 (2)	47 (1)
Cl(2)	-2527 (2)	6113 (1)	5905 (3)	60 (1)
Cl(3)	-2091 (2)	6850 (2)	8365 (2)	56 (1)
N	-1279 (5)	7894 (4)	4671 (6)	40 (4)
P(1)	306 (2)	7154 (1)	6910 (2)	37 (1)
P(2)	-3430 (2)	7867 (1)	5815 (2)	39 (1)
C(1)	1239 (5)	7640 (3)	5608 (6)	42 (2)
C(2)	1711 (5)	7166 (3)	5102 (6)	55 (2)
C(3)	2431 (5)	7576 (3)	4126 (6)	65 (3)
C(4)	2680 (5)	8460 (3)	3656 (6)	65 (3)
C(5)	2208 (5)	8934 (3)	4162 (6)	56 (2)
C(6)	1487 (5)	8524 (3)	5138 (6)	49 (2)
C(7)	1235 (4)	7558 (4)	8215 (6)	43 (2)
C(8)	774 (4)	7668 (4)	9208 (6)	53 (2)
C(9)	1500 (4)	7933 (4)	10228 (6)	65 (3)
C(10)	2686 (4)	8088 (4)	10253 (6)	66 (3)
C(11)	3147 (4)	7977 (4)	9260 (6)	67 (3)
C(12)	2422 (4)	7712 (4)	8240 (6)	54 (2)
C(13)	253 (6)	6021 (4)	7333 (5)	45 (2)
C(14)	734 (6)	5646 (4)	8504 (5)	59 (2)
C(15)	716 (6)	4789 (4)	8777 (5)	85 (4)
C(16)	217 (1)	4307 (4)	7880 (5)	84 (3)
C(17)	-264 (6)	4682 (4)	6710 (5)	81 (3)
C(18)	-246 (6)	5539 (4)	6437 (5)	60 (2)
C(19)	-3494 (4)	8534 (3)	4190 (5)	40 (2)
C(20)	-4273 (4)	8285 (3)	3194 (5)	56 (2)
C(21)	-4208 (4)	8821 (3)	1979 (5)	60 (2)
C(22)	-3363 (4)	9607 (3)	1760 (5)	56 (2)
C(23)	-2583 (4)	9857 (3)	2755 (5)	54 (2)
C(24)	-2648 (4)	9320 (3)	3970 (5)	46 (2)
C(25)	-3719 (5)	8518 (4)	6722 (5)	43 (2)
C(26)	-3136 (5)	8593 (4)	7904 (5)	52 (2)
C(27)	-3419 (5)	9067 (4)	8592 (5)	63 (3)
C(28)	-4285 (5)	9466 (4)	8098 (5)	58 (2)
C(29)	-4868 (5)	9391 (4)	6916 (5)	60 (2)
C(30)	-4584 (5)	8917 (4)	6228 (5)	51 (2)
C(31)	-4717 (6)	6953 (4)	5953 (6)	48 (2)
C(32)	-4970 (6)	6437 (4)	5093 (6)	64 (3)
C(33)	-5951 (6)	5736 (4)	5179 (6)	87 (3)
C(34)	-6680 (6)	5552 (4)	6125 (6)	97 (4)
C(35)	-6427 (6)	6068 (4)	6985 (6)	84 (4)
C(36)	-5446 (6)	6768 (4)	6899 (6)	64 (3)
C(37)	-1172 (8)	8103 (8)	3376 (9)	47 (2)
C(38)	-555 (9)	8894 (9)	2804 (10)	60 (2)
C(39)	-449 (12)	9061 (9)	1514 (13)	89 (4)
C(40)	-931 (11)	8463 (8)	861 (13)	80 (3)
C(41)	-1563 (11)	7722 (9)	1389 (12)	86 (4)
C(42)	-1701 (10)	7484 (7)	2725 (11)	71 (3)
C(43)	3891 (15)	5530 (11)	1545 (17)	137 (5)
*Cl(4)	5233 (30)	6005 (20)	2038 (34)	240 (10)
Cl(5)	3930 (24)	5354 (17)	182 (28)	266 (9)
*Cl(4A)	2916 (31)	5947 (19)	1681 (32)	133 (5)

\* These atoms were introduced in statistical positions with population parameter 0.5.

**Introduction.** As part of a study on the preparation and the properties of a variety of nitrido and arylimido complexes of Re<sup>V</sup> (Forsellini, Casellato, Graziani & Magon, 1982; Rossi, Marchi, Duatti, Magon, Casellato, Graziani & Polizzotti, 1984), we report here the structure of [ReCl<sub>3</sub>(NC<sub>6</sub>H<sub>5</sub>)(PPh<sub>3</sub>)<sub>2</sub>].CH<sub>2</sub>Cl<sub>2</sub> in order to obtain better bonding parameters and to clarify the nature of the Re—N bond in these species.

**Experimental.** The title compound was prepared by reacting [ReOCl<sub>3</sub>(PPh<sub>3</sub>)<sub>2</sub>] with phenylformamidine (1:2, metal to ligand) in boiling tetrahydrofuran for 2 h. The resulting brown solution was filtered and then evaporated to one third of its volume and a green

Table 2. Bond distances ( $\text{\AA}$ )

Coordination			
Re—N	1.726 (6)	Re—Cl(1)	2.415 (2)
Re—P(1)	2.503 (2)	Re—Cl(2)	2.411 (2)
Re—P(2)	2.490 (2)	Re—Cl(3)	2.402 (2)
P—C			
P(1)—C(1)	1.83 (1)	P(2)—C(19)	1.84 (1)
P(1)—C(7)	1.83 (1)	P(2)—C(25)	1.82 (1)
P(1)—C(13)	1.83 (1)	P(2)—C(31)	1.83 (1)
PhN			
N—C(37)	1.39 (1)		
C(37)—C(38)	1.35 (1)	C(40)—C(41)	1.29 (2)
C(38)—C(39)	1.39 (2)	C(41)—C(42)	1.44 (2)
C(39)—C(40)	1.33 (2)	C(37)—C(42)	1.37 (1)
CH <sub>2</sub> Cl <sub>2</sub> *			
C(43)—Cl(4)	1.66 (3)		
C(43)—Cl(5)	1.62 (3)		
C(43)—Cl(4A)	1.62 (3)		

\* This molecule is disordered: Cl(4) and Cl(4A) have been introduced in statistical positions with a population parameter of 0.5.

Table 3. Bond angles ( $^\circ$ )

Coordination			
P(1)—Re—P(2)	174.1 (1)	P(2)—Re—Cl(1)	90.2 (1)
Cl(1)—Re—Cl(2)	173.3 (1)	P(2)—Re—Cl(2)	89.9 (1)
Cl(3)—Re—N	173.1 (2)	P(2)—Re—Cl(3)	86.0 (1)
P(1) Re—N	92.9 (2)	Cl(1)—Re—N	96.4 (2)
P(2) Re—N	92.8 (2)	Cl(2)—Re—N	90.3 (2)
P(1)—Re—Cl(1)	87.9 (1)	Cl(1)—Re—Cl(3)	90.5 (1)
P(1)—Re—Cl(2)	91.3 (1)	Cl(2)—Re—Cl(3)	82.9 (1)
P(1)—Re—Cl(3)	88.4 (1)		
Ligands			
Re—N—C(37)	172.6 (6)		
Re—P(1)—C(1)	109.3 (2)	Re—P(2)—C(19)	107.6 (2)
Re—P(1)—C(7)	118.3 (2)	Re—P(2)—C(25)	119.6 (2)
Re—P(1)—C(13)	117.7 (2)	Re—P(2)—C(31)	115.9 (2)
C(1)—P(1)—C(7)	101.7 (3)	C(19)—P(2)—C(25)	101.8 (3)
C(1)—P(1)—C(13)	104.7 (3)	C(19)—P(2)—C(31)	108.0 (3)
C(7)—P(1)—C(13)	103.1 (3)	C(25)—P(2)—C(31)	102.6 (3)
CH <sub>2</sub> Cl <sub>2</sub>			
Cl(4)—C(43)—Cl(5)	107 (2)		
Cl(4A)—C(43)—Cl(5)	118 (2)		

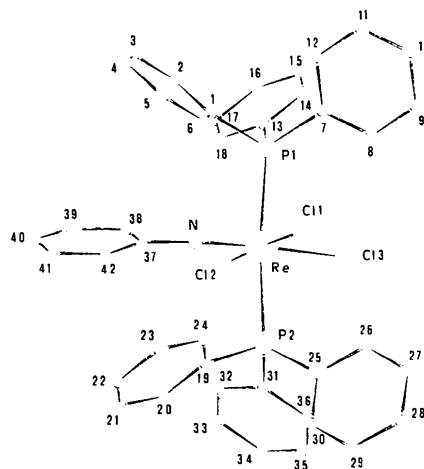


Fig. 1. The molecular structure of [ReCl<sub>3</sub>(NC<sub>6</sub>H<sub>5</sub>)(PPh<sub>3</sub>)<sub>2</sub>] showing atomic numbering.

product was obtained after addition of EtOH. The complex was crystallized from  $\text{CH}_2\text{Cl}_2/\text{EtOH}$ .

Prismatic fragment, maximum dimension 0.2 mm, cell constants by least squares from 25 reflections ( $6^\circ < \theta < 15^\circ$ ), Philips PW 1100 four-circle diffractometer, Mo  $K\alpha$  radiation, integrated intensities collected up to  $\theta = 25^\circ$ ,  $-14 \leq h \leq 14$ ,  $-18 \leq k \leq 18$ ,  $0 \leq l \leq 10$ ,  $\theta/2\theta$  scan mode, background-peak-background, scan rate  $2^\circ \text{ min}^{-1}$ , 7044 independent reflections measured, 5812 with  $I > 3\sigma(I)$  used, no variation in standard reflections  $\bar{6}44$  and  $\bar{1}23$ , corrections for Lp and absorption (North, Phillips & Mathews, 1968),  $0.40 < \mu R < 0.68$ . The diffraction effects are dominated by the heavy-atom contributions and the structure was solved by Patterson and Fourier methods. The phenyl rings of the phosphine groups were introduced as rigid bodies with C-C distances of 1.395 Å and C-H distances of 0.95 Å. All H atoms were assigned fixed thermal parameters of  $0.06 \text{ \AA}^2$ . A difference Fourier synthesis showed the presence of a disordered  $\text{CH}_2\text{Cl}_2$  molecule, statistically distributed in two differently oriented positions approximately symmetrical with respect to the C(43)-Cl(5) axis. Accordingly, the Cl(4) and Cl(4A) atoms were introduced and refined with a fixed population parameter of 0.5. Anisotropic refinement of Re, Cl, P and N atoms of the molecule and isotropic refinement of all other non-hydrogen atoms converged at  $R = 0.045$  and  $R_w = 0.049$ , when the ratio  $(\Delta/\sigma)_{\text{max}}$  was 0.5. Function minimized  $\sum w(\Delta F)^2$  with  $w = 2.9133/[\sigma^2(F) + 0.000579F^2]$ . Maximum and minimum heights in final difference Fourier map 1.6 and  $-0.9 \text{ e \AA}^{-3}$  near the solvent molecule, whose disorder was probably not completely represented by the model adopted. Calculations performed mainly with *SHELX* (Sheldrick, 1975). Atomic scattering factors for Re from *International Tables for X-ray Crystallography* (1974), with corrections for anomalous dispersion (Cromer, 1965), and for H from Stewart, Davidson & Simpson (1965). Those for Cl, P, C and N were supplied internally by *SHELX*.

**Discussion.** Final atomic parameters are given in Table 1. Bond lengths and angles are given in Tables 2 and 3.\*

The structure consists of discrete, monomeric molecules of the complex interspersed with solvent

\* Lists of structure factors, anisotropic thermal parameters and H-atom parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 39553 (38 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

molecules of dichloromethane. As shown in Fig. 1 the Re atom is in a slightly distorted octahedral coordination geometry with meridional N and Cl atoms and apical triphenylphosphines. Atoms of the base plane Re, N, Cl(1), Cl(2), Cl(3) are coplanar within  $\pm 0.01 \text{ \AA}$ , and the phenylimido phenyl ring is tilted by only  $5.2(3)^\circ$  with respect to the mean equatorial plane. The molecule is distorted from ideal octahedral geometry by a movement of the ligands *cis* to the phenylimido group away from the N atom, probably caused by steric repulsion. The structure markedly resembles that found in other arylimido and alkylimido compounds. The Re-N bond distance of  $1.726(6) \text{ \AA}$  is at the longer limit of the range of the values if compared with  $1.709(4)$  and  $1.690(5) \text{ \AA}$  in  $[\text{ReCl}_3(\text{NC}_6\text{H}_4\text{OCH}_3)(\text{PEt}_2\text{Ph})_2]$  (Bright & Ibers, 1968), and with  $1.685(11) \text{ \AA}$  in  $[\text{ReCl}_3(\text{NCH}_3)(\text{PEtPh}_2)_2]$  (Bright & Ibers, 1969). It was proposed that the formal double Re-N bond in these compounds has considerable triple-bond character (Chatt, Garforth, Johnson & Rowe, 1964). Slightly shorter distances were found in nitrido complexes:  $1.660(8) \text{ \AA}$  in  $[\text{ReNCl}_2(\text{PMe}_2\text{Ph})_3]$  (Forsellini, Casellato, Graziani & Magon, 1982) and  $1.602(9) \text{ \AA}$  in  $[\text{ReNCl}_2(\text{PPh}_3)_2]$  (Doedens & Ibers, 1967) (but in this latter compound the Re atom is five-coordinate). The Re-Cl bond lengths [mean  $2.409(4) \text{ \AA}$ ] compare very well with the values found in the above complexes [mean  $2.422(4) \text{ \AA}$ ], whereas the Re-P bond lengths [mean  $2.497(6) \text{ \AA}$ ] are significantly longer [mean  $2.470(4) \text{ \AA}$ ]. These distances seem to be sensitive to the presence of electronegative substituents in the arylimido group.

#### References

- BRIGHT, D. & IBERS, J. A. (1968). *Inorg. Chem.* **7**, 1099-1111.  
 BRIGHT, D. & IBERS, J. A. (1969). *Inorg. Chem.* **8**, 703-709.  
 CHATT, J., GARFORTH, J. D., JOHNSON, N. P. & ROWE, G. A. (1964). *J. Chem. Soc.* pp. 1012-1019.  
 CROMER, D. T. (1965). *Acta Cryst.* **18**, 17-23.  
 DOEDENS, R. J. & IBERS, J. A. (1967). *Inorg. Chem.* **6**, 204-210.  
 FORSELLINI, E., CASELLATO, U., GRAZIANI, R. & MAGON, L. (1982). *Acta Cryst.* **B38**, 3081-3083.  
*International Tables for X-ray Crystallography* (1974). Vol. IV. Birmingham: Kynoch Press.  
 NORTH, A. C. T., PHILLIPS, D. C. & MATHEWS, F. S. (1968). *Acta Cryst.* **A24**, 351-359.  
 ROSSI, R., MARCHI, A., DUATTI, A., MAGON, L., CASELLATO, U., GRAZIANI, R. & POLIZZOTTI, G. (1984). *Inorg. Chim. Acta.* In the press.  
 SHELDRICK, G. M. (1975). *SHELX*. Program for crystal structure determination. Univ. of Cambridge, England.  
 STEWART, R. F., DAVIDSON, E. R. & SIMPSON, W. T. (1965). *J. Chem. Phys.* **42**, 3175-3187.