

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

Rh—C(1)	2.198 (10)	C(1)—C(8)	1.524 (29)
Rh—C(2)	2.251 (13)	C(1)—C(2)	1.302 (22)
Rh—C(3)	3.005 (20)	C(2)—C(3)	1.393 (29)
Rh—C(4)	3.074 (15)	C(3)—C(4)	1.410 (40)
Rh—C(5)	2.210 (11)	C(4)—C(5)	1.528 (33)
Rh—C(6)	2.254 (14)	C(5)—C(6)	1.303 (21)
Rh—C(7)	3.037 (15)	C(6)—C(7)	1.354 (26)
Rh—C(8)	3.047 (12)	C(7)—C(8)	1.514 (31)
Rh—M(1,2)	2.128 (13)	C(n)—H(n)	0.950
Rh—M(5,6)	2.133 (14)		
Re—Cl(1)	2.524 (3)	Re—C(11)	1.867 (12)
Re—Cl(2)	2.504 (2)	C(9)—O(9)	1.158 (15)
Re—Cl(2)'	2.506 (3)	C(10)—O(10)	1.155 (12)
Re—C(9)	1.914 (13)	C(11)—O(11)	1.181 (14)
Re—C(10)	1.896 (11)	Re—Re'	3.380 (1)
M(1,2)—Rh—M(5,6)	82.5	Cl(1)—Re—Cl(2)	78.87 (8)
M(1,2)—Rh—M(5,6)'	97.5	Cl(1)—Re—Cl(2)'	78.82 (8)
M(1,2)—Rh—M(1,2)'	180	Cl(2)—Re—Cl(2)'	81.28 (7)
M(5,6)—Rh—M(5,6)'	180	Re—C(9)—O(9)	175 (1)
Re—Cl(1)—Re'	84.07 (11)	Re—C(10)—O(10)	177 (1)
Re—Cl(2)—Re'	84.83 (6)	Re—C(11)—O(11)	177 (1)

anion has been reported (Davis & Baenziger, 1977); the same D_{3h} structure has also been predicted for the manganese anion, $[\text{Mn}_2(\text{CO})_6\text{Cl}_3]^-$ (Cihonski, Walker & Levenson, 1975). The Re—Re distance is 3.380 (1) Å, considerably longer than the distance 3.08 Å found for the metal—metal bonded dirhenium decarbonyl (Dahl, Ishishi & Rundle, 1957; Gapotchenko, Alekseev, Kolobova, Anisimov, Ronova & Johansson, 1972). The Re atoms are bridged by three Cl atoms; however, only two of the Cl atoms are symmetry equivalent. The symmetry unique Cl atom has a Re—Cl bond length of

2.524 (3) Å which is statistically longer than the two nearly identical distances found for the symmetry related pair, 2.504 (2) and 2.506 (3) Å. The variation of 2.524 vs 2.505 Å may be due to solid-state packing forces since the Re—Cl distances reported previously (Davis & Baenziger, 1977) indicated equivalent chlorine distances equal to 2.49 Å. The rhenium to carbonyl carbon length average 1.89 (1) Å and the C—O bond distances average 1.16 (1) Å, and within each set the bond lengths are statistically equivalent.

References

- CIHONSKI, J. L., WALKER, M. L. & LEVENSON, R. A. (1975). *J. Organomet. Chem.* **102**, 335–337.
- DAHL, L. F., ISHISHI, E. & RUNDLE, R. E. (1957). *J. Chem. Phys.* **26**, 1750–1751.
- DAVIS, R. L. & BAENZIGER, N. C. (1977). *Inorg. Nucl. Chem. Lett.* **13**, 475–477.
- GAPOTCHENKO, N. I., ALEKSEEV, N. V., KOLOBOVA, N. E., ANISIMOV, K. N., RONOVA, I. A. & JOHANSSON, A. A. (1972). *J. Organomet. Chem.* **35**, 319–320.
- GREEN, M., KUC, T. A. & TAYLOR, S. H. (1970). *J. Chem. Soc. Chem. Commun.* pp. 1553–1554.
- GREEN, M., KUC, T. A. & TAYLOR, S. H. (1971). *J. Chem. Soc. A*, pp. 2334–2337.
- GREEN, M. & PARKER, G. J. (1974). *J. Chem. Soc. Dalton Trans.* pp. 333–343.
- JOHNSON, C. K. (1965). *ORTEP*. Report ONRL-3794. Oak Ridge National Laboratory, Tennessee, USA.
- ROSCH, N. & HOFFMANN, R. (1974). *Inorg. Chem.* **13**, 2656–2666.
- SCHROCK, R. R. & OSBORN, J. A. (1971). *J. Am. Chem. Soc.* **93**, 3089–3091.
- TEMPLETON, L. & TEMPLETON, D. (1973). *Am. Crystallogr. Assoc. Meet.*, Storrs, Connecticut, Abstract E10.
- USON, R., ORO, L. A., CLAVER, C. & GARRALDA, M. A. (1976). *J. Organomet. Chem.* **105**, 365–370.

Acta Cryst. (1991). **C47**, 541–544

Simple Oxygen Bridged Ti^{IV} Dimer: Structure of $[\text{Ti}(\mu\text{-O})(\text{OC}_6\text{H}_3\text{-2,6Pr}_2)_2(\text{NC}_5\text{H}_4\text{-4NC}_4\text{H}_8)]_2$

BY JOHN E. HILL, PHILIP E. FANWICK AND IAN P. ROTHWELL

Department of Chemistry, Purdue University, West Lafayette, IN 47907, USA

(Received 19 March 1990; accepted 17 August 1990)

Abstract. Di- μ -oxo-bis[bis(2-6-diisopropylphenolato)-4-pyrrolindylpyridinetitanium], $[\text{Ti}_2(\text{C}_{24}\text{H}_{30}\text{O}_2)_2(\text{O})_2(\text{C}_9\text{H}_{12}\text{N}_2)_2]$, $M_r = 1133.3$, triclinic, $P\bar{1}$, $a = 10.245$ (2), $b = 12.160$ (2), $c = 14.030$ (3) Å, $\alpha = 105.35$ (1), $\beta = 96.50$ (1), $\gamma = 110.08$ (1)°, $V = 1542$ (1) Å³, $Z = 1$, $D_x = 1.22$ g cm⁻³, $\lambda(\text{Mo K}\alpha) = 0.71073$ Å, $\mu = 3.06$ cm⁻¹, $F(000) = 608.0$, $T = 135$ K, $R = 0.061$ for 3092 reflections with $I > 3\sigma(I)$. There is pseudo trigonal bipyramidal geometry

around each Ti atom with two aryl oxide O atoms and one bridging O atom forming the trigonal plane. Ti—O(bridge) = 1.847 (av.); Ti—O(aryl oxide) = 1.825 Å (av.); O(bridge)—Ti—O(bridge) = 81.6 (1); Ti—O(bridge)—Ti = 98.4 (1)°; Ti—Ti(non-bonding interaction) = 2.796 (1) Å.

Introduction. We recently reported the isolation and structural characterization of a mononuclear aryl

oxide complex of titanium(IV) containing a rare, terminal oxo group, Ti(O)(OC₆H₃-2,6Prⁱ)₂(NC₅H₄-4NC₄H₈)₂ (I) (OC₆H₃-2,6Prⁱ = 2,6-diisopropyl phenoxide; NC₅H₄-4NC₄H₈ = 4-pyrrolidinylpyridine) (Hill, Fanwick & Rothwell, 1989). The bridged oxygen dimer complex being described here, [Ti(μ-O)(OC₆H₃-2,6Prⁱ)₂(NC₅H₄-4NC₄H₈)₂] (II), provides an excellent structural comparison between terminal and bridging oxo complexes with analogous ligand environments. Complex (II) also provides a comparison with the only other simple titanium(IV) oxide bridged dimer complex to be structurally characterized, [Ti(μ-O)(acac)₂]₂ (Smith, Caughlan & Campbell, 1972).

Experimental. Complex (II) was synthesized in nearly quantitative yield through the slow addition of a stoichiometric amount of H₂O to the η²-imine complex Ti(OC₆H₃-2,6Prⁱ)₂[η²-BuⁱNC(CH₂Ph)₂](NC₅H₄-4NC₄H₈) (III) in benzene solution. The amine BuⁱNHCH(CH₂Ph)₂ is generated *via* protonation of the η²-imine ligand. Bright yellow crystals of (II) suitable for crystallographic analysis are obtained by slow evaporation of a saturated benzene solution.

A needle-like crystal of (II) having approximate dimensions of 0.40 × 0.26 × 0.15 mm was mounted on a glass fiber in a random orientation. Preliminary examination and data collection were performed with Mo Kα radiation (λ = 0.71073 Å) on an Enraf-Nonius CAD-4 computer controlled κ-axis diffractometer equipped with a graphite crystal, incident beam monochromator.

Cell constants and an orientation matrix for data collection were obtained from least-squares refinement, using the setting angles of 25 reflections in the range 17 < θ < 21°, measured by the computer controlled diagonal slit method of centering.

The data were collected at a temperature of 135 K using the ω-2θ scan technique. The scan rate varied from 2 to 20° min⁻¹ (in ω). The variable scan rate allows rapid data collection for intense reflections (where a fast scan rate is used), and assures good counting statistics for weak reflections (where a slow scan rate is used). Data were collected to a 2θ range of 4.00–45.0°: h 0 to 11, k -13 to 12, l -15 to 14. The scan range (°) was determined as a function of θ to correct for the separation of the Kα doublet (CAD-4 Operations Manual, 1977); the scan width was calculated as ω-scan width = (0.79 + 0.350tanθ)°. Moving-crystal moving-counter background counts were made by scanning an additional 25% above and below this range. Thus the ratio of peak counting time to background counting time was 2:1. The counter aperture was also adjusted as a function of θ. The horizontal aperture width ranged from 1.6 to 2.0 mm; the vertical aperture was set at

4.0 mm. The diameter of the incident beam collimator was 0.7 mm and the crystal to detector distance was 21 cm. For intense reflections, an attenuator was automatically inserted in front of the detector; the attenuator factor was 25.7.

A total of 4317 reflections were collected, of which 4005 were unique. As a check on crystal and electronic stability, three representative reflections were measured every 83 min. The slope of the least-squares line through a plot of intensity *versus* time was -60 ± 6 counts h⁻¹ which corresponds to a total loss in intensity of 15.6%. An anisotropic decay correction was applied. The correction factors on I ranged from 0.971 to 1.185 with an average value of 1.074.

Lorentz and polarization corrections were applied to the data. The linear absorption coefficient is 3.1 cm⁻¹ for Mo Kα radiation. No absorption correction was made. Intensities of equivalent reflections were averaged, R_{int} = 0.035. Three reflections were rejected from the averaging process because their intensities differed significantly from the average. The agreement factors for the averaging of the 510 observed and accepted reflections was 3.5% based on intensity and 2.1% based on F_o.

The structure was solved using the Patterson heavy-atom method which revealed the position of one Ti atom. The remaining atoms were located and added to the structure factor calculations, but their positions were not refined. The structure was refined in full-matrix least squares on F where the function minimized was Σw(|F_o| - |F_c|)² and the weight w is defined by the Killean and Lawrence method with terms of 0.020 and 1.0 (Killean & Lawrence, 1969). The weights were further modified according to the method of Dunitz and Seiler using a sharpening factor of 4.0 (Dunitz & Seiler, 1973).

Scattering factors were taken from Cromer & Waber (1974). Anomalous-dispersion effects were included in F_c (Ibers & Hamilton, 1964); the values of f' and f'' were those of Cromer (1974). Only the 3092 reflections having intensities greater than 3.0 times their standard deviation were used in the refinements. The final cycle of refinement included 352 variable parameters and converged (largest parameter shift/e.s.d. was 0.38) with unweighted and weighted agreement factors of: R = 0.061, wR = 0.078. The standard deviation of an observation of unit weight was 1.80. There were 51 correlation coefficients greater than 0.50. The highest correlation coefficient was 0.77 between parameters 304 and 306. The highest peak in the final difference Fourier map had a height of 0.74 e Å⁻³ with an estimated error based on δF (Cruickshank, 1949) of 0.07. Plots of Σw(|F_o| - |F_c|)² *versus* |F_o|, reflection order in data collection, sinθ/λ, and various classes of indices showed no unusual trends.

All calculations were performed on a VAX computer using *SDP* (Frenz, 1978).

Discussion. Fig. 1 presents an *ORTEP* (Johnson, 1965) view of complex (II) emphasizing the central coordination sphere. It can be seen that each Ti atom has two aryl oxide ligands, one pyridine ligand, and two (bridging) O atoms arranged about it in a trigonal bipyramidal geometry. Fractional coordinates and isotropic thermal parameters are collected in Table 1,* while a listing of important bond distances and angles is given in Table 2.

The structural parameters of (II) are very similar to another simple oxygen bridged titanium dimer complex $[\text{Ti}(\mu\text{-O})(\text{acac})_2]_2$ (Smith, Caughlan & Campbell, 1972) (Table 3). The distances and angles around the $\text{Ti}(\mu\text{-O})_2\text{-Ti}$ core indicate that complex (II) is fairly typical for a Ti^{IV} oxo-bridged dimer. With this established, it is interesting to compare the structure of (II) with its monomeric analogue (I). Both complexes (I) and (II) have five-coordinate Ti atoms with a trigonal bipyramidal arrangement of ligands around the metal centers. Both complexes have two 2,6-diisopropylphenoxide ligands per Ti atom, with the two aryl oxides and an O atom forming the trigonal plane. The terminal-oxo complex (I) has two 4-pyrrolidinylpyridine ligands in the axial positions of the *tbp*, while in the bridged-oxo structure of (II), one of the pyrrolidinylpyridine ligands has been replaced by the other bridging O atom. A comparison of the bond distances and angles in complexes (I) and (II) is given in Table 3.

Evidence for a *trans* effect in complex (I) can be seen by examining the slightly longer Ti-O (aryl oxide) distances (0.046 Å av.) and significantly larger $\text{O(aryl oxide)-Ti-O(aryl oxide)}$ angle [$132.4(3)$ vs $109.3(1)^\circ$] distorting the triangle of O atoms by

* Lists of structure factors, anisotropic thermal parameters, full bond lengths and angles and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 53489 (45 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

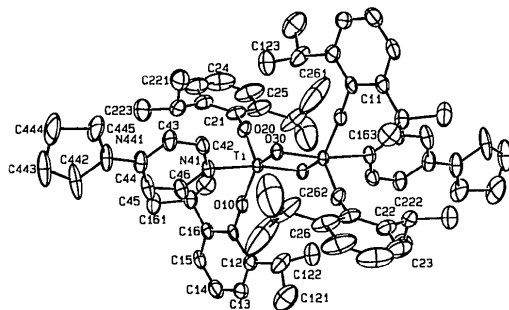


Fig. 1. *ORTEP* view of (II) with thermal ellipsoids drawn at 50% probability level.

Table 1. *Fractional coordinates and isotropic thermal parameters for (II)*

$$B_{\text{eq}} = (4/3)[a^2 B(1,1) + b^2 B(2,2) + c^2 B(3,3) + ab(\cos\gamma)B(1,2) + ac(\cos\beta)B(1,3) + bc(\cos\alpha)B(2,3)].$$

	x	y	z	$B_{\text{eq}}(\text{\AA}^2)$
Ti	0.08311 (7)	0.0521 (6)	0.09909 (5)	1.92 (1)
O(10)	0.1659 (3)	-0.0156 (2)	0.1723 (2)	2.69 (7)
O(20)	0.0173 (3)	0.1575 (3)	0.1826 (2)	2.83 (7)
O(30)	0.0752 (3)	0.0814 (2)	-0.0269 (2)	2.21 (6)
N(41)	0.3078 (4)	0.2014 (3)	0.1300 (3)	2.54 (8)
N(441)	0.6929 (4)	0.5109 (4)	0.2186 (3)	4.4 (1)
C(11)	0.2217 (4)	-0.0774 (4)	0.2212 (3)	2.27 (9)
C(12)	0.2299 (5)	-0.1871 (4)	0.1650 (3)	2.9 (1)
C(13)	0.2853 (5)	-0.2494 (4)	0.2168 (4)	3.6 (1)
C(14)	0.3303 (6)	-0.2053 (4)	0.3215 (4)	4.1 (1)
C(15)	0.3221 (5)	-0.0957 (4)	0.3752 (4)	3.4 (1)
C(16)	0.2682 (4)	-0.0287 (4)	0.3269 (3)	2.6 (1)
C(21)	-0.0352 (5)	0.1963 (4)	0.2642 (4)	3.4 (1)
C(22)	0.0523 (5)	0.3067 (4)	0.3420 (3)	3.5 (1)
C(23)	-0.0050 (7)	0.3444 (5)	0.4248 (4)	5.3 (1)
C(24)	-0.1409 (7)	0.2762 (6)	0.4297 (5)	6.9 (2)
C(25)	-0.2224 (7)	0.1682 (6)	0.3544 (5)	6.9 (2)
C(26)	-0.1723 (5)	0.1262 (5)	0.2699 (4)	4.9 (1)
C(42)	0.3316 (5)	0.2916 (4)	0.0871 (3)	3.0 (1)
C(43)	0.4559 (5)	0.3941 (4)	0.1131 (4)	3.4 (1)
C(44)	0.5698 (5)	0.4095 (4)	0.1885 (4)	3.3 (1)
C(45)	0.5476 (5)	0.3140 (5)	0.2300 (3)	3.8 (1)
C(46)	0.4192 (5)	0.2150 (4)	0.1996 (3)	3.0 (1)
C(121)	0.2516 (9)	-0.3097 (7)	-0.0065 (6)	6.9 (2)
C(122)	0.1719 (7)	-0.2399 (4)	0.0508 (4)	4.4 (1)
C(123)	0.0140 (8)	-0.3221 (6)	0.0279 (5)	6.4 (2)
C(161)	0.3833 (7)	0.1719 (6)	0.4727 (5)	5.1 (2)
C(162)	0.2530 (5)	0.0869 (4)	0.3868 (3)	2.9 (1)
C(163)	0.1179 (6)	0.0544 (5)	0.4270 (4)	4.8 (1)
C(221)	0.1929 (6)	0.4597 (5)	0.2688 (5)	4.9 (2)
C(222)	0.1997 (6)	0.3791 (4)	0.3326 (4)	3.8 (1)
C(223)	0.3044 (8)	0.4581 (6)	0.4344 (5)	6.6 (2)
C(261)	-0.353 (2)	0.0247 (8)	0.1117 (7)	15.1 (6)
C(262)	-0.2654 (6)	0.0080 (5)	0.1835 (6)	8.5 (2)
C(263)	-0.345 (1)	-0.0907 (8)	0.219 (1)	17.2 (4)
C(442)	0.8171 (6)	0.5257 (7)	0.2933 (4)	6.2 (2)
C(443)	0.9307 (7)	0.6446 (7)	0.2884 (5)	7.0 (2)
C(444)	0.8522 (7)	0.7172 (7)	0.2583 (7)	7.8 (2)
C(445)	0.7165 (6)	0.6165 (5)	0.1825 (6)	5.6 (2)

Table 2. *Selected bond distances (Å) and angles (°) for (II)*

Ti—Ti	2.796 (1)	Ti—O(30)	1.865 (2)
Ti—O(10)	1.833 (2)	Ti—O(30)	1.829 (2)
Ti—O(20)	1.817 (2)	Ti—N(41)	2.271 (3)
O(10)—Ti—O(20)	109.3 (1)	O(20)—Ti—N(41)	95.8 (1)
O(10)—Ti—O(30)	138.8 (1)	O(30)—Ti—N(41)	81.43 (9)
O(20)—Ti—O(30)	110.7 (1)	Ti—O(30)—Ti	98.4 (1)
O(10)—Ti—N(41)	85.2 (1)	O(30)—Ti—O(30)	81.6 (1)

Table 3. *Comparison of selected structural parameters between (I), (II) and $[\text{Ti}(\text{O})(\text{acac})_2]_2$*

	(II)	$[\text{Ti}(\text{O})(\text{acac})_2]_2$
Ti—Ti	2.796 (1)	2.729 (1)
Ti—O(bridge)	1.847 (av.)	1.828 (av.)
Ti—O—Ti	98.4 (1)	96.6 (1)
O—Ti—O	81.6 (1)	83.4 (2)
<hr/>		
	(II)	(I)
Ti—O(Ar)	1.825 (av.)	1.871 (av.)
Ti—N	2.271 (3)	2.204 (av.)
(Ar)O—Ti—O(Ar)	109.3 (1)	132.4 (3)
(Ar)O—Ti—O	124.8 (av.)	113.8 (av.)

bending the aryl oxides toward the terminal oxygen (away from the *trans* position).

We thank the National Science Foundation for support of this research.

References

- CAD-4 Operations Manual (1977). Enraf-Nonius, Delft, The Netherlands.
- CROMER, D. T. (1974). *International Tables for X-ray Crystallography*, Vol. IV, Table 2.3.2, p. 149. Birmingham: Kynoch Press. (Present distributor Kluwer Academic Publishers, Dordrecht.)
- CROMER, D. T. & WABER, J. T. (1974). *International Tables for X-ray Crystallography*, Vol. IV, Table 2.2B, p. 99. Birmingham: Kynoch Press. (Present distributor Kluwer Academic Publishers, Dordrecht.)
- CRUICKSHANK, D. W. T. (1949). *Acta Cryst.* **2**, 154–157.
- DUNITZ, J. D. & SEILER, P. (1973). *Acta Cryst.* **B29**, 589–595.
- FRENZ, B. A. (1978). *The Enraf-Nonius CAD-4 SDP – A Real Time System for Concurrent X-ray Data Collection and Crystal Structure Solution*. In *Computing in Crystallography*, edited by H. SCHENK, R. OLTJOF-HAZEKAMP, H. VAN KONINGSVELD & G. C. BASSI. Delft. Univ. Press.
- HILL, J. E., FANWICK, P. E. & ROTHWELL, I. P. (1989). *Inorg. Chem.* **28**, 3602–3604.
- IBERS, J. A. & HAMILTON, W. C. (1964). *Acta Cryst.* **17**, 781–782.
- JOHNSON, C. K. (1965). *ORTEP*. Report ORNL-3794. Oak Ridge National Laboratory, Tennessee, USA.
- KILLEAN, R. C. G. & LAWRENCE, J. L. (1969). *Acta Cryst.* **B25**, 1750–1752.
- SMITH, G. D., CAUGHLAN, C. N. & CAMPBELL, J. A. (1972). *Inorg. Chem.* **11**, 2989–2993.

Acta Cryst. (1991). **C47**, 544–547

Structure of [μ -Hexamethylenebis(diphenylphosphine)-*P,P'*]bis[di- μ -carbonyl-nonacarbonyl-triangulo-triiron(3 Fe–Fe)] {[Fe₃(CO)₁₁]Ph₂P(CH₂)₃]₂

BY GEORGE FERGUSON*

Department of Chemistry and Biochemistry, University of Guelph, Guelph, Ontario, Canada N1G 2W1

AND ROSAMUND HOURIHANE AND TREVOR R. SPALDING*

Department of Chemistry, University College, Cork, Ireland

(Received 26 June 1990; accepted 28 August 1990)

Abstract. (I), [Fe₆(C₅₂H₃₂O₂₂P₂)], $M_r = 1405.9$, triclinic, $P\bar{1}$, $a = 12.190$ (15), $b = 14.908$ (8), $c = 9.209$ (6) Å, $\alpha = 95.56$ (5), $\beta = 112.29$ (7), $\gamma = 108.37$ (8)°, $V = 1424$ (5) Å³, $Z = 1$, $D_x = 1.65$ g cm⁻³, $\lambda(\text{Mo } K\alpha) = 0.71073$ Å, $\mu = 16.3$ cm⁻¹, $F(000) = 706$, $T = 294$ K, $R = 0.042$ for 2219 observed reflections. The midpoint of the C₆ chain in the bis(diphenylphosphino)hexane ligand lies on a crystallographic inversion centre and each of the P atoms of bis-1,6-(diphenylphosphino)hexane is coordinated to one Fe atom of a Fe₃(CO)₁₁ cluster. In each cluster one Fe–Fe bond is bridged by two CO ligands; the remaining CO ligands are all terminal. The P atoms are each bonded to one of the bridged Fe atoms. Both bridging carbonyl ligands are asymmetrically bonded but the asymmetry in one bridge is more marked than in the other. The less symmetric system has one remarkably short Fe–C distance, 1.856 (7) Å, and one remarkably long one, 2.348 (6) Å. The unbridged Fe–Fe bond *trans* to the P atom is significantly longer, 2.702 (1) Å, than the other unbridged Fe–Fe bond, 2.681 (2) Å.

Introduction. Whereas the substitution of carbonyl ligands on [Fe₃(CO)₁₂] by monodentate phosphines has afforded many [Fe₃(CO)_{12-n}(PR)_n] derivatives ($n = 1, 2$ or 3) (see *e.g.* Grant & Manning, 1978), reaction with (Ph₂PCH₂)₂ (diphos) disrupts the Fe₃-core and produces Fe- and Fe₂-based compounds. We have observed that the reaction between [Fe₃(CO)₁₂] and bis-1,6-(diphenylphosphino)hexane (dpph) in tetrahydrofuran at 298 K for 4 h afforded three products which contained the Fe₃-core intact. One of these products analyzed as [Fe₃(CO)₁₁]₂(dpph) (I) and could be recrystallized from CH₂Cl₂ solution to give dark-green crystals. The only [Fe₃(CO)₁₁PR₃] compound to have been characterised previously (R = Ph) had a crystal structure in which the asymmetric unit contained two molecules which were structural isomers (Dahm & Jacobson, 1968). It was decided to characterize (I) in the anticipation that it would have a less complicated structure than [Fe₃(CO)₁₁PPh₃].

Experimental. Dark-green crystal, 0.35 × 0.41 × 0.48 mm, mounted on glass fibre in random orientation. Accurate cell dimensions and crystal orientation matrix determined on CAD-4 diffractometer

* E-mail addresses: GF CHMFERG@VM.UOGUELPH.CA
TRS STCH8006@IRUCCVAX