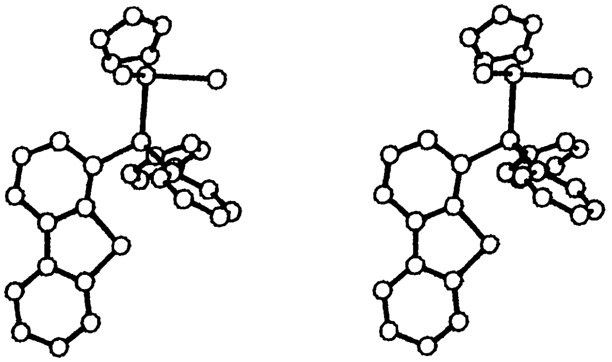

Fig. 5. Stereoview of (II) normal to the plane of the dibenzothiophene.

In ^{13}C NMR spectra of the compound it was observed that a few peaks were doubled due to coupling of ^{13}C to ^{31}P nuclei. The ^{31}P NMR spectrum of (II) exhibited a sextet, but when proton decoupled a sharp singlet was observed confirming that the coupling was between ^{1}H and ^{31}P nuclei. In (II), the Fe atom does not bind to the ring S of ligated (I). In this case, coordination of the metal center to the P atom of (I) maintains a close proximity of the Fe atom to the ring S. The possibility of Fe—S binding this affords may be important in the catalytic hydrodesulfurization of the dibenzothiophenic S.

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Studies in Werner Clathrates. II.* Structure and Thermal Analysis of Bis(isothiocyanato)tetrakis(4-phenylpyridine)nickel(II) Benzene Clathrate (1:4)

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Abstract. $C_{46}H_{36}N_6NiS_2.4C_6H_6$, $M_r = 1107\cdot7$, triclinic, $P\bar{1}$, $a = 9\cdot52\ (1)$, $b = 12\cdot19\ (3)$, $c = 13\cdot72\ (2)$ Å, $\alpha = 100\cdot3\ (1)$, $\beta = 90\cdot3\ (1)$, $\gamma = 105\cdot9\ (2)°$, $V = 1504\cdot0$ Å^3, $Z = 1$, host:guest = 1:4, $D_m = 1\cdot235$, $D_x = 1\cdot22$ g cm^{-3}, $\lambda(\text{Mo } K\alpha) = 0\cdot7107$ Å, $\mu = 3\cdot95$ cm^{-1}, $F(000) = 582$, $T = 294$ K, $R = 0\cdot097$ ($wR = 0\cdot078$) for 3782 observed reflections. The host molecule has a $-{+}{+}-$ configuration and guest molecules are contained in a large channel parallel to **a** which has been mapped by volume calculations. Thermal decomposition studies indicate release of guest molecules at *ca* 307 K.

Introduction. Werner clathrates are complexes of the type MX_2L_4 where M = divalent transition-metal cation, *e.g.* Mn^{2+}–Cu^{2+}, X = anionic ligand *e.g.* halide, NCS^-, NO_2^-, NCO^-, and L = electrically neutral substituted pyridine or α-arylalkylamine. Since the pioneering work in the 1950's (Schaeffer, Dorsey, Skinner & Christian, 1957) these compounds have been used to separate a wide variety of isomeric and other organic compounds. The 4-methylpyridine (4-Mepy) derivative of the bis(isothiocyanato)nickel(II) complex

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* Part 10: Nassimbeni, Niven & Taylor (1988*a*).

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entraps a wide variety of guest molecules in cavities of the channel, layer and cage types, and its physicochemical properties have recently been reviewed (Lipkowski, 1984).

We have studied the effect of varying the substituent on the base on the clathrating ability of the complex. The host complexes studied so far have been of the type $[Ni(NCS)_2(4\text{-}R\text{py})_4]$ where R = ethyl (Moore, Nassimbeni & Niven, 1987*a*), vinyl (Moore, Nassimbeni, Niven & Taylor, 1986) and phenyl (Nassimbeni, Papanicolaou & Moore, 1986; Nassimbeni, Niven & Taylor, 1987, 1988*a*, 1988*b*).

We report here the structure of another 4-phenylpyridine derivative with benzene as the guest molecule and describe the physical environment of the guest with relation to its thermal decomposition.

Experimental. The host powder of $[Ni(NCS)_2(4\text{-Phpy})_4]$ was prepared by treating an aqueous solution of nickel(II) isothiocyanate with a stoichiometric quantity of 4-phenylpyridine dissolved in a minimum of methanol. The clathrate was formed by dissolving the dried precipitate in benzene until saturated, then centrifuging to produce a clear blue solution. Slow evaporation at room temperature gave blue rectangular lath-shaped crystals after 72 h.

Crystal density was determined by flotation in water/saturated KI solution. Approximate cell parameters and space-group symmetry were determined photographically. A suitable crystal for data collection had to be mounted in a Lindemann-glass capillary with mother liquor, to prevent deterioration in the atmosphere by desorption of the guest solvent. Crystal mounting proved difficult because the crystals had the unusual ability to move in the capillary even after being firmly wedged. Mounting was eventually achieved by introducing glass fibres on both sides of the crystal (dimensions 0·46 × 0·59 × 0·46 mm), adding mother liquor, and sealing with a flame. Although this method of mounting prevented gross movement, there was continual recentring (10 times) throughout the data collection (total exposure time was 34·6 h).

Data collection: Nonius CAD-4 diffractometer, graphite-monochromated Mo $K\alpha$ radiation. 25 reflections ($16 < \theta < 17°$) for measurement of the lattice parameters at 294 K. ω–2θ scan technique, scan width $\Delta\omega = (1{\cdot}0 + 0{\cdot}35\tan\theta)°$, θ scan range 1–20°, final acceptance limit 20σ at 20° min^{-1} in ω and maximum recording time of 30 s. No absorption correction applied owing to severe 'decay'. Centring checked every 100 measured reflections whilst intensities of three standard reflections (229, 239, 1,1,10) monitored every hour indicated loss of intensity of 55% during the data collection. Intensities corrected for Lorentz and polarization factors but no decay correction applied owing to decay being non-isotropic. Range of indices *hkl*: h −9 to 9, k −11 to 11, l 0 to 13. 4716 unique reflections yielded 3782 observed using criterion $|F_o| \geq 2\sigma|F_o|$.

Table 1. *Fractional atomic coordinates* ($\times 10^4$) *and thermal parameters* (Å$^2 \times 10^3$) *with e.s.d.'s in parentheses for* $C_{46}H_{36}N_6NiS_2.4C_6H_6$

	x	y	z	$U_{\rm iso}/U_{\rm eq}$
Ni(1)	0	0	0	39 (1)*
N(1)	1849 (7)	−378 (6)	−537 (5)	44 (3)*
C(1)	2815 (9)	−701 (8)	−873 (6)	43 (4)*
S(1)	−4140 (3)	1200 (3)	1342 (2)	71 (1)*
N(11)	1201 (8)	1832 (7)	366 (5)	47 (3)*
C(12)	507 (10)	2628 (9)	543 (8)	60 (5)*
C(13)	1195 (11)	3839 (9)	783 (8)	67 (5)*
C(14)	2701 (10)	4197 (8)	851 (7)	47 (4)*
C(15)	3469 (10)	3361 (10)	654 (7)	61 (5)*
C(16)	2655 (10)	2182 (8)	414 (7)	51 (4)*
C(111)	3530 (10)	5491 (9)	1119 (7)	51 (4)*
C(112)	4756 (12)	5829 (10)	1762 (8)	70 (6)*
C(113)	5490 (13)	7048 (11)	2002 (9)	83 (6)*
C(114)	4965 (13)	7829 (10)	1633 (9)	70 (5)*
C(115)	3766 (14)	7481 (10)	1007 (9)	74 (6)*
C(116)	2989 (12)	6266 (10)	741 (8)	66 (5)*
N(21)	564 (8)	−282 (7)	1416 (5)	55 (4)*
C(22)	1944 (10)	−65 (9)	1758 (7)	57 (5)*
C(23)	2386 (11)	−163 (9)	2703 (7)	57 (5)*
C(24)	1298 (11)	−485 (9)	3370 (7)	55 (5)*
C(25)	−111 (10)	−721 (9)	3020 (7)	53 (4)*
C(26)	−446 (9)	−575 (10)	2062 (8)	61 (5)*
C(211)	1697 (12)	−597 (12)	4362 (8)	71 (6)*
C(212)	3009 (13)	157 (12)	4860 (8)	87 (7)*
C(213)	3423 (18)	8 (16)	5816 (11)	113 (9)*
C(214)	2501 (22)	−826 (21)	6256 (12)	137 (15)*
C(215)	1287 (20)	−1508 (14)	5768 (11)	112 (9)*
C(216)	832 (15)	−1457 (10)	4811 (9)	78 (6)*
C(11*G*)	3713 (20)	3649 (15)	5393 (14)	138 (3)
C(12*G*)	2694 (18)	3641 (15)	4673 (14)	138 (3)
C(13*G*)	3024 (19)	3262 (15)	3607 (14)	138 (3)
C(14*G*)	4255 (20)	2941 (15)	3462 (14)	138 (3)
C(15*G*)	5262 (18)	3000 (14)	4224 (14)	138 (3)
C(16*G*)	4974 (19)	3376 (15)	5289 (14)	138 (3)
C(21*G*)	−135 (19)	6339 (15)	2171 (13)	132 (3)
C(22*G*)	−1402 (20)	5593 (16)	1875 (13)	132 (3)
C(23*G*)	−1885 (18)	4576 (15)	2299 (13)	132 (3)
C(24*G*)	−999 (19)	4426 (15)	3081 (13)	132 (3)
C(25*G*)	402 (18)	5305 (15)	3370 (13)	132 (3)
C(26*G*)	800 (17)	6230 (15)	2864 (13)	132 (3)

* $U_{\rm eq}$ is $\frac{1}{3}$ the trace of the orthogonalized U_{ij} tensor.

Structure solution and refinement: heavy-atom method using *SHELXS*86 (Sheldrick, 1985) revealed Ni atom on centre of inversion at Wyckoff position (*a*). Full-matrix least-squares refinement using *SHELX*76 (Sheldrick, 1978) with anisotropic thermal parameters for all non-H host atoms and isotropic for all other atoms; all H atoms geometrically positioned with a single common temperature factor. Two independent guest molecules located in general positions, no H atoms on guest molecules and all guest C atoms were assigned a common isotropic temperature factor. 289 parameters refined, $R = 0{\cdot}097$, $wR = 0{\cdot}078$ with $w = [\sigma^2F_o]^{-1}$, $S = 8{\cdot}18$, max. $\Delta/\sigma = 0{\cdot}014$, $\Delta\rho$ within $+0{\cdot}62$ to $-0{\cdot}71$ e Å^{-3}. Complex neutral-atom scattering factors for non-H atoms from Cromer & Mann (1968) and for H atoms from Stewart, Davidson & Simpson (1965). Molecular parameters obtained from *PARST* (Nardelli, 1983), drawings from *PLUTO* (Motherwell, 1974), space-filling drawings from *ALCHEMY* (Tripos Associates, Inc., 1987) and volume calculations with *OPEC* (Gavezzotti, 1983).

Table 2. *Selected bond lengths* (Å) *and bond angles* (°) *with e.s.d.'s in parentheses for* $C_{46}H_{36}N_6NiS_2.4C_6H_6$

Ni(1)–N(1)	2·049 (9)	N(11)–Ni(1)–N(21)	91·8 (5)
Ni(1)–N(11)	2·176 (10)	N(1)–Ni(1)–N(21)	90·3 (5)
Ni(1)–N(21)	2·114 (9)	N(1)–Ni(1)–N(11)	89·6 (6)
N(1)–C(1)	1·168 (13)	Ni(1)–N(1)–C(1)	173·0 (11)
C(1)–S(1)	1·626 (11)	N(1)–C(1)–S(1)	177·8 (13)
C(14)–C(111)	1·536 (14)		
C(24)–C(211)	1·461 (17)		

Thermograms were carried out on a Stanton–Redcroft thermal analyser, operating at a uniform heating rate of 10 K min^{-1} (range 313 to 673 K) with nitrogen (60 ml min^{-1}) passed over samples. Crystals were removed from solution, dried by patting and weighed quickly before analysis. Platinum containers (alumina used as reference) located directly on the thermocouple so furnace temperature only measured. Lag between this and sample temperature calculated using 6 standards in the range 373 to 573 K. Enthalpy values calculated by comparison of DTA peak areas with a standard (naphthalene) of known ΔH value.

Discussion. Final atomic parameters are in Table 1* with selected bond distances and angles in Table 2. The bond lengths and angles are as expected for the pyridine and phenyl rings. Fig. 1 shows a perspective view of the host molecule with atomic nomenclature. The Ni atom has distorted octahedral coordination with the anionic moieties *trans* to each other.

For complexes of this type, one of the possible reasons for clathrate formation is the rotational freedom about the Ni–N bonds. This derivative has the additional pyridine–phenyl torsional parameters. Previously (Nassimbeni, Niven & Taylor, 1987; Moore, Nassimbeni & Niven, 1987*a*,*b*) we have defined these torsional parameters as follows:

$\tau_i = \min$ N(1)–Ni(1)–N(j1)–C(jx) where $x = 2$ or 6 and $i = 1$ for $j = 1$ and $i = 3$ for $j = 2$.

$\tau_k = \min$ C(my)–C(m4)–C(m11)–C(m1x) where $y = 3$ or 5 and $x = 2$ or 6 with $k = 2$ for $m = 1$ and $k = 4$ for $m = 2$.

Thus for this structure we have τ_1 = N(1)–Ni(1)–N(11)–C(16) = −17 (1)°; τ_2 = C(13)–C(14)–C(111)–C(116) = −38 (2)°; τ_3 = N(1)–Ni(1)–N(21)–C(22) = 30 (1)°; τ_4 = C(23)–C(24)–C(211)–C(212) = 35 (2)°.

For the inner torsion angles (τ_1 and τ_3) the centre of symmetry simply changes the sign of the symmetry-related angle. However, owing to the slight asymmetry

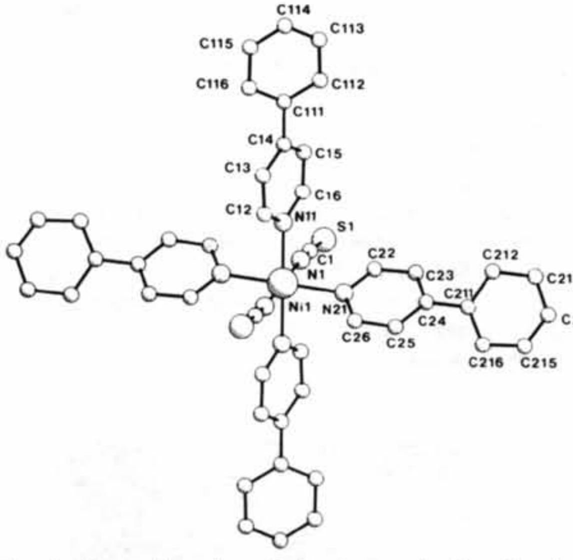

Fig. 1. Perspective view of the host molecule with atomic nomenclature.

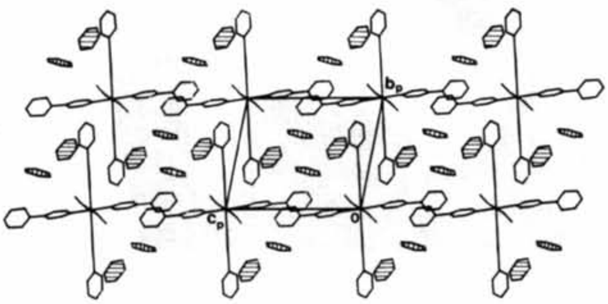

Fig. 2. Projection of the structure, viewed along [100], with guest molecules shaded for clarity.

Fig. 3. Space-filling diagram of the host molecules with the guest molecules omitted to display the shape of the cavity. The view is the same as in Fig. 2.

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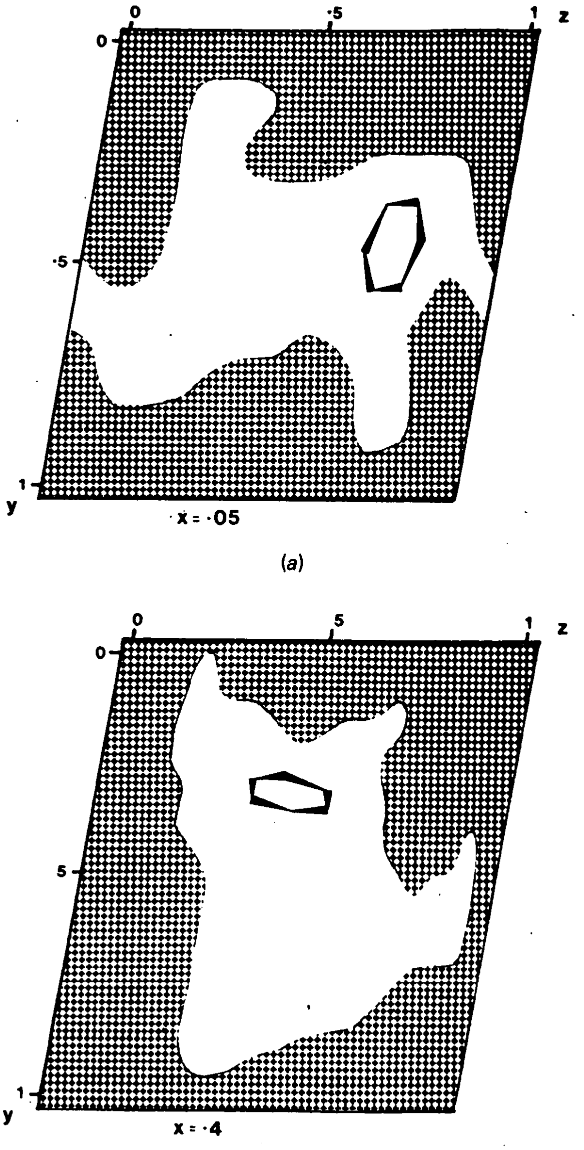
* Lists of structure factors, anisotropic thermal parameters, H-atom parameters, bond lengths and angles and torsion angles have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 51498 (30 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

of the pyridine ring it is more accurate to describe these angles as

τ_5(symmetry relation of τ_1) = –[N(1)—Ni(1)—N(11)—C(12)] + 180° = 17°;

τ_7(symmetry relation of τ_3) = –[N(1)—Ni(1)—N(21)—C(26)] – 180° = –25°.

We thus have a — + + — configuration of these ligands [following Lipkowski's (1984) definition]; this is the first time that this has been observed for the 4-Phpy derivative. Similarly the pyridine–phenyl torsional parameters give a — + + — configuration, indicating that the phenyl rings twist in the same direction as their parent pyridine moiety.



(a)

(b)

Fig. 4. Sections through the cavity, viewed in the *yz* plane and layered at (*a*) $x = 0{\cdot}05$ and (*b*) $x = 0{\cdot}40$, showing the extent of the cavity in the region of the guest molecules.

All four guest molecules are located in a large common channel running parallel to **a** and bounded by the host molecules at each corner of the unit cell. Passage between channels in the **c** direction is prevented by one set of 4-Phpy ligands which run 15° off parallel to the *b* axis and are tilted 30° to the *yz* plane, whilst the other pair of ligands are 7° off parallel to the *c* axis, tilted –15° to the *yz* plane and prevent interconnection in the **b** direction. Fig. 2 shows a packing diagram of the clathrate as viewed along [100] whilst Fig. 3 shows a space-filling diagram of the same view but with guest molecules removed. The extent of this channel, calculated from van der Waals radii, sectioned in the regions of two of the guest molecules, is displayed in Figs. 4(*a*) and (*b*).

The thermal analysis results are shown in Fig. 5 and summarized in Table 3. The mechanism of decomposition, as indicated by the TGA curve is as follows:

$$\mathrm{Ni(NCS)_2(4\text{-}Phpy)_4.4C_6H_6}$$
step 1 ↓
$$\mathrm{Ni(NCS)_2(4\text{-}Phpy)_4 + 4C_6H_6}$$
step 2 ↓
$$\mathrm{Ni(NCS)_2(4\text{-}Phpy)_2 + 2(4\text{-}Phpy)}$$
step 3 ↓
$$\mathrm{Ni(NCS)_2 + 2(4\text{-}Phpy).}$$

The agreement between observed and calculated weight loss is excellent. The release of the guest molecules at about 307 K is in agreement with our experience in handling these crystals for structural determination in that they decompose over a period of minutes once removed from their mother liquor. The decomposition pattern is typical of complexes of this type (Moore, Nassimbeni & Niven, 1987*b*).

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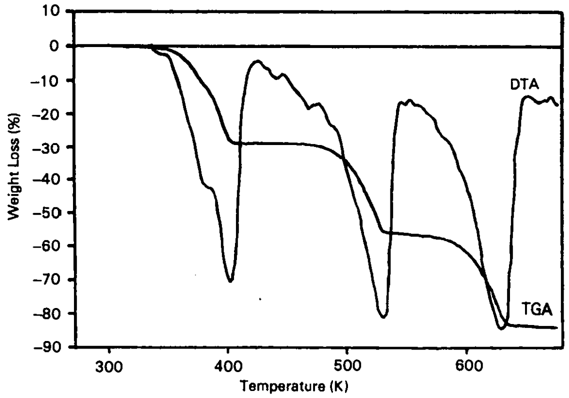

Fig. 5. Thermograms (DTA and TGA) of the clathrate.

Table 3. *Thermogravimetric and differential thermal analysis results*

Step	% weight loss Calc.	Meas.	Temp. (K) Sample	ΔH (kJ mol^{-1})
1	28·1	28	307	66
2	56·1	56	433	89
3	84·1	84	515	166
				ΔH_{tot} = 339 kJ mol^{-1}

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Structure of a Chiral Diphosphine Ligand in a Trinuclear Ruthenium Cluster: $Ru_3(CO)_{10}\{(Ph_2PCH_2)_2C_5H_8O_2\}.xCH_2Cl_2$*

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Abstract. Decacarbonyl-μ-[4,5-bis(diphenylphosphinomethyl)-2,2-dimethyl-1,3-dioxolane]-*triangulo*-triruthenium dichloromethane solvate, $[Ru_3(CO)_{10}(C_{31}H_{32}O_2P_2)].xCH_2Cl_2$ ($x = 0{\cdot}29$), $M_r = 1081{\cdot}95 + 84{\cdot}93x$, orthorhombic, $P2_12_12_1$, $a = 14{\cdot}344\,(5)$, $b = 14{\cdot}763\,(3)$, $c = 21{\cdot}830\,(4)$ Å, $V = 4622{\cdot}7$ Å^3, $Z = 4$, $D_m = 1{\cdot}62\,(2)$, $D_x = 1{\cdot}590$ ($x = 0{\cdot}29$) g cm^{-3}, λ(Mo $K\alpha$) = 0·7107 Å, $\mu = 10{\cdot}4 \rightarrow 11{\cdot}5$ ($x = 0 \rightarrow 1$) cm^{-1}, $F(000) = 2144 + 168x$, room temperature, final $R = 0{\cdot}060$ for 3533 reflections [$F > 3\sigma(F)$]. The structure consists of discrete molecules in which the chelating diphosphine ligand ($C_{31}H_{32}O_2P_2$ = diop) bridges two Ru atoms to form an eight-membered ring [Ru—P = 2·328 (3), 2·337 (4) Å]. The longest Ru—Ru distance [2·888 (1) Å] is associated with the diphosphine bridge.

Introduction. The use of chiral organometallic species as potential catalysts in stereoselective synthesis is now well established (Kagan, 1982). As part of our study of metal-cluster catalysts, we have examined the Ru_3—carbonyl cluster with the chiral diphosphine ligand 4,5-bis(diphenylphosphinomethyl)-2,2-dimethyl-1,3-dioxolane (diop) and we now report the structure of $Ru_3(CO)_{10}\{(Ph_2PCH_2)_2C_5H_8O_2\}.xCH_2Cl_2$.

Experimental. The compound was prepared from the reaction of $Ru_3(CO)_{12}$ with the phosphine ligand using the radical-ion catalysis method (Bruce, Kehoe, Matisons, Nicholson, Rieger & Williams, 1982) and purified by flash column chromatography. Dark-red crystals were obtained from methylene chloride/octane by cooling and the density was measured by flotation ($CCl_4/C_2H_4Br_2$). Preliminary data were from photographic examination and accurate cell dimensions were obtained from 25 accurately centred reflections ($9{\cdot}0 < \theta < 12{\cdot}8°$) using an Enraf–Nonius CAD-4 diffractometer fitted with a graphite monochromator and Mo radiation. The intensities of 4746 reflections were recorded (ω–2θ scan, $1{\cdot}5 < \theta < 25°$; h 0–17, k 0–17, l 0–26) using a crystal 0·3 × 0·2 × 0·15 mm positioned in a thin-walled glass capillary. The standard reflections (three) showed no change with time during

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* μ-[4,5-Bis(diphenylphosphinomethyl)-2,2-dimethyl-1,3-dioxolane]-decacarbonyl-*triangulo*-triruthenium methylene dichloride solvate.

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