

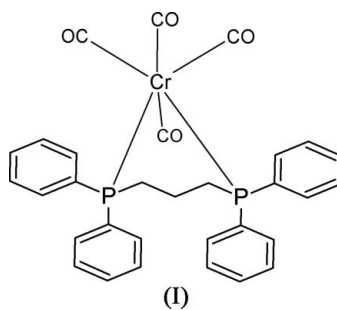
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Key indicators

Single-crystal X-ray study
 $T = 293$ K
Mean $\sigma(\text{C}-\text{C}) = 0.004$ Å
 R factor = 0.038
 wR factor = 0.106
Data-to-parameter ratio = 20.4For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.**[1,3-Bis(diphenylphosphino)propane]-tetracarbonylchromium(0)**In the title molecule, $[\text{Cr}(\text{C}_{27}\text{H}_{26}\text{P}_2)(\text{CO})_4]$, the Cr atom is octahedrally coordinated by the four carbonyl ligands and a bidentate phosphine ligand, which is bonded as a chelate in a *cis* position. The average Cr–P and Cr–C bond lengths are 2.3770 (5) and 1.865 (2) Å, respectively.Received 23 March 2006
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Comment

It is generally believed that the metal (M) to carbon monoxide bond involves both $\text{OC}-M$ σ -bonding and $M-\text{CO}$ π -back-bonding. In view of this phenomenon, the bonding characteristics of metal carbonyls with a phosphine ligand in phosphine-substituted metal carbonyls are of interest. A search of the Cambridge Structural Database (Version 5.27; Allen, 2002) revealed only 21 complexes of group VI metal carbonyls with a 3-carbon backbone bidentate phosphine. However, there were no examples of chromium carbonyl complexes. Previously, we reported several crystal structures of phosphine-substituted group VI metal carbonyls (Shawkataly *et al.*, 1996, 1997). We present here the crystal structure of the title compound, (I).The title molecule has an expected octahedral geometry (Fig. 1). The Cr–C bond lengths of the *cis* carbonyl ligands (with respect to the P atom) are slightly longer than those for the *trans* carbonyl group (Table 1). This trend was also observed in $\text{Cr}[\text{Ph}_2\text{P}(\text{CH}_2)_2\text{PPh}_2](\text{CO})_4$ (Bennett *et al.*, 1971) and $\text{Cr}[\text{Ph}_2\text{P}(\text{CH}_2)_4\text{PPh}_2](\text{CO})_4$ (Ueng & Shih, 1992). The bidentate phosphine bite angle [89.124 (17)°] is intermediate between that observed in $\text{Cr}[\text{Ph}_2\text{P}(\text{CH}_2)_2\text{PPh}_2](\text{CO})_4$ [83.41 (8)°] and that in $\text{Cr}[\text{Ph}_2\text{P}(\text{CH}_2)_4\text{PPh}_2](\text{CO})_4$ [93.29 (5)°]. Comparison of the mean Cr–C and C–O distances in (I) [1.865 (2) and 1.141 (6) Å, respectively] with those in $\text{Cr}(\text{CO})_6$ [1.909 (3) and 1.137 (4) Å, respectively (Whitaker & Jeffery, 1967); 1.918 (2) and 1.141 (2) Å, respectively (Jost *et al.*, 1975)], indicates stronger bonding owing to the back-bonding abilities of the bidentate phosphine. The Cr–P bond lengths, with an average value of

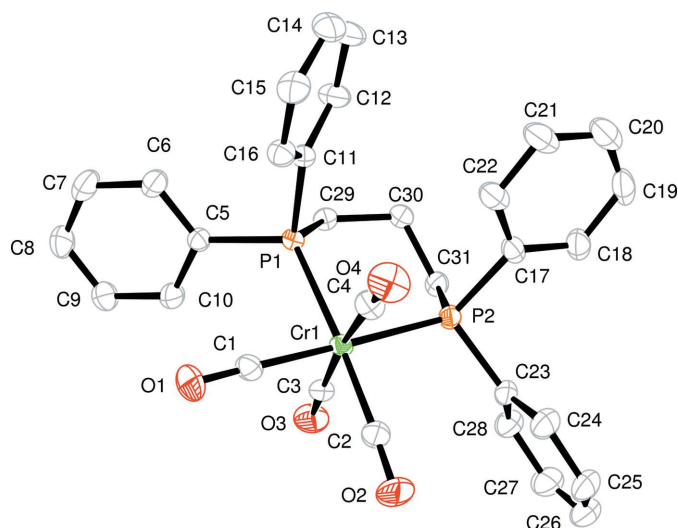


Figure 1
View of (I), with the atom numbering scheme and 25% probability displacement ellipsoids. H atoms have been omitted for clarity.

2.3770 (5) Å, are relatively short in spite of the presence of the bulky phosphine ligand.

Experimental

A mixture of $\text{Cr}(\text{CO})_6$ (234 mg, 1.064 mmol) and $\text{Ph}_2\text{P}(\text{CH}_2)_3\text{PPh}_2$ (439 mg, 1.065 mmol) was refluxed in a purified mixture of petroleum ether (373–403 K, 30 ml) and *n*-butanol (20 ml) for ca 12 h under dry deoxygenated nitrogen. The solvent was rotary evaporated and the reaction mixture was dissolved in acetone (3 ml) and filtered. Yellow crystals (454 mg, 74% yield) were obtained by slow evaporation of the acetone solution at room temperature. Analysis calculated for $\text{C}_{31}\text{H}_{26}\text{CrO}_4\text{P}_2$: C 64.58, H 4.42%; found C 64.59, H 4.15%.

Crystal data

$[\text{Cr}(\text{C}_{27}\text{H}_{26}\text{P}_2)(\text{CO})_4]$
 $M_r = 576.46$
Monoclinic, $P2_1/n$
 $a = 10.0562$ (1) Å
 $b = 20.8494$ (3) Å
 $c = 14.1616$ (2) Å
 $\beta = 106.4183$ (7)°
 $V = 2848.13$ (6) Å³

$Z = 4$
 $D_x = 1.344$ Mg m⁻³
Mo $K\alpha$ radiation
 $\mu = 0.55$ mm⁻¹
 $T = 293$ (2) K
Prism, yellow
 $0.4 \times 0.38 \times 0.28$ mm

Data collection

Siemens SMART CCD
diffractometer
 ω scans
Absorption correction: multi-scan
(SADABS; Sheldrick, 1996)
 $T_{\min} = 0.772$, $T_{\max} = 0.899$

22851 measured reflections
7010 independent reflections
5175 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.055$
 $\theta_{\text{max}} = 28.3^\circ$

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.038$
 $wR(F^2) = 0.106$
 $S = 1.01$
7010 reflections
343 parameters
H-atom parameters constrained

$$w = 1/[\sigma^2(F_o^2) + (0.0569P)^2 + 0.004P]$$

where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\text{max}} = 0.001$
 $\Delta\rho_{\text{max}} = 0.27 \text{ e \AA}^{-3}$
 $\Delta\rho_{\text{min}} = -0.44 \text{ e \AA}^{-3}$

Table 1

Selected geometric parameters (Å, °).

Cr1—C2	1.854 (2)	Cr1—C4	1.889 (2)
Cr1—C1	1.8547 (19)	Cr1—P2	2.3719 (5)
Cr1—C3	1.863 (2)	Cr1—P1	2.3821 (5)
C2—Cr1—C3	87.71 (10)	P2—Cr1—P1	89.124 (17)
C2—Cr1—C4	86.29 (10)		

All H atoms were placed at calculated positions and refined using a riding model, with C—H = 0.93 and 0.97 Å, respectively, and $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$.

Data collection: XSCANS (Siemens, 1994); cell refinement: XSCANS; data reduction: XSCANS; program(s) used to solve structure: SHELXS86 (Sheldrick, 1985); program(s) used to refine structure: SHELXL97 (Sheldrick, 1997); molecular graphics: ORTEP-3 for Windows (Farrugia, 1997); software used to prepare material for publication: WinGX (Farrugia, 1999).

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