

# METAL-ORGANIC COMPOUNDS

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## [PPN]<sub>2</sub>[Cr<sub>2</sub>(CO)<sub>10</sub>], a Salt of the Dimeric Cr<sup>1+</sup> Carbonyl Complex Without a Coordinating Counterion

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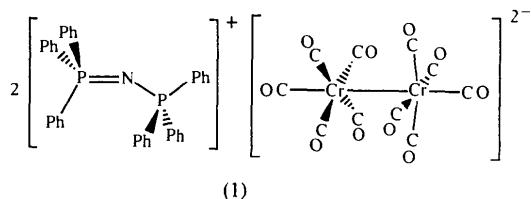
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### Abstract

The Cr<sup>1+</sup> carbonyl complex bis[bis(triphenylphosphine)iminium] decacarbonyldichromate(2–), [PPN]<sub>2</sub>[Cr<sub>2</sub>(CO)<sub>10</sub>], (1), where [PPN]<sup>+</sup> is [(Ph<sub>3</sub>P)<sub>2</sub>N]<sup>+</sup>, has been prepared by naphthalenide reduction of a Cr<sup>0</sup> precursor followed by counterion metathesis to introduce a non-coordinating cation. The [Cr<sub>2</sub>(CO)<sub>10</sub>]<sup>2-</sup> dianion in (1) is structurally similar to that in a previously characterized salt with a [K(DME)<sub>2</sub>]<sup>+</sup> counterion (DME = dimethoxyethane), despite the loss of the solid-state ion pairing present in the [K(DME)<sub>2</sub>]<sup>+</sup> salt.

### Comment

The chromium carbonyl anions [Cr(CO)<sub>5</sub>]<sup>2-</sup> and [Cr<sub>2</sub>(CO)<sub>10</sub>]<sup>2-</sup> are used extensively in the preparation of substituted chromium carbonyl complexes. They have been variously produced by the reduction of [Cr(CO)<sub>6</sub>] using NaBH<sub>4</sub>/NH<sub>3</sub>(liq) (Behrens & Hagg, 1961), sodium amalgam (Hayter, 1966), Na-bipy (bipy = 2,2'-bipyridine) (Linder, Behrens & Birkle, 1968), Na/K (Ellis, 1975; Ellis, Hentges, Kalina & Hagen, 1975), KSi (Hey-Hawkins & von Schnerring, 1991), or C<sub>8</sub>K (Ungureanu & Palie, 1975) as reducing agents. Several years ago, the preparation of [Cr(CO)<sub>5</sub>]<sup>2-</sup> by alkali-metal naphthalenide reduction was reported by our group (Maher, Beatty & Cooper, 1985; Maher, Beatty, Lee & Cooper, 1986) and we now wish to report the use of this approach, followed by counterion metathesis, to prepare the binuclear complex, and also the X-ray diffraction study of the unsolvated salt [PPN]<sub>2</sub>[Cr<sub>2</sub>(CO)<sub>10</sub>], (1) {[PPN]<sup>+</sup> = [(Ph<sub>3</sub>P)<sub>2</sub>N]<sup>+</sup>}.



(1) crystallizes in *P*ī with no crystallographically imposed symmetry. The asymmetric unit consists of one [Cr<sub>2</sub>(CO)<sub>10</sub>]<sup>2-</sup> dianion and two bis(triphenylphosphine)iminium, [PPN]<sup>+</sup>, monocations. The structures of the cations are unexceptional and will not be discussed here further. The molecular structure and labeling scheme for the anion are shown in Fig. 1. It has been reported previously (Hey-Hawkins & von Schnerring, 1991) that the [K(DME)<sub>2</sub>]<sup>+</sup> cation in [K(DME)<sub>2</sub>]<sub>2</sub>[Cr<sub>2</sub>(CO)<sub>10</sub>] bridges laterally between [Cr<sub>2</sub>(CO)<sub>10</sub>]<sup>2-</sup> anions to give polymeric one-dimensional chains of [K<sub>2</sub>Cr<sub>2</sub>(CO)<sub>10</sub>] parallel to [001]. These chains must involve strong electrostatic interactions between the anions and the cations, but comparison of the reported dimensions of the [Cr<sub>2</sub>(CO)<sub>10</sub>]<sup>2-</sup> unit in the polymer with those of the discrete dianions in (1) establishes, surprisingly, that the electrostatic interactions have little effect on the structural parameters. Thus, the Cr—Cr bond in (1) has a length of 2.995 (1) Å, while that in the ion-paired form is 2.999 (2) Å. The mean carbonyl C—O bond lengths in (1) [axial = 1.177 (4), equatorial = 1.154 (5) Å] are statistically indistinguishable from those in the ion-paired form [axial = 1.180 (8), equatorial = 1.157 (5) Å]. The differences between the Cr—C distances in (1) [axial = 1.792 (4), equatorial = 1.861 (5) Å] and those in the ion-paired form [axial = 1.802 (6), equatorial = 1.879 (5) Å] are more marked, but are only significant at the 2–3σ level. We conclude that while the ion-pairing interactions in the [K(DME)<sub>2</sub>]<sup>+</sup> salt impose unusual and obvious one-dimensional order in the solid state, they do little to change the effective electron density on the metal centers and hence do not perturb the observed back donation to the carbonyl ligands.

The significant differences between the parameters of the axial and equatorial carbonyl groups in (1) are anticipated on the basis of greater back donation of

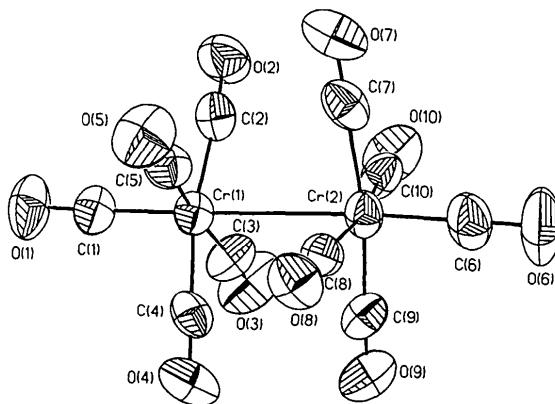


Fig. 1. View of the [Cr<sub>2</sub>(CO)<sub>10</sub>]<sup>2-</sup> dianion with displacement ellipsoids shown at the 50% probability level.

electron density from the metal center to the CO ligand *trans* to the Cr—Cr bond, which shortens the Cr—C bond and lengthens the C—O bond. In both structures, the equatorial carbonyls of Cr1 are staggered relative to those of Cr2.

The structure of  $[\text{Cr}_2(\text{CO})_{10}]^{2-}$  as the dichloromethane-solvated  $[\text{PPN}]^+$  salt was reported previously in space group  $C2/c$  with the solvated complex residing on crystallographic twofold axes (Handy, Ruff & Dahl, 1970). This structure suffered from a poorly resolved  $\text{CH}_2\text{Cl}_2$  solvate molecule, 19% decay in crystal intensity, and a lack of anisotropic displacement refinement, and a detailed comparison of bond lengths and angles with those of (1) will not, therefore, be presented here. Additionally, the structure of a molybdenum analogue of (1) has been reported previously (Bachman & Whitmire, 1993).

## Experimental

Reactions were carried out under an atmosphere of  $\text{N}_2$  by means of standard Schlenk and cannula techniques. A solution of  $[\text{Cr}(\text{CO})_5(\text{NMe}_3)]$  (Maher, Beatty, Lee & Cooper, 1986) in THF was reduced with 1 equivalent of alkali-metal naphthalenides ( $\text{Li}^+$ ,  $\text{Na}^+$ ) (Maher, Beatty & Cooper, 1985) at 195 K to give a (1:1) mixture of  $[\text{Cr}(\text{CO})_5]^{2-}$  and  $[\text{Cr}(\text{CO})_5(\text{NMe}_3)]$ , as determined by IR spectroscopy. Prolonged stirring (>12 h) of the resulting solution gave the binuclear carbonyl complex  $[\text{Cr}_2(\text{CO})_{10}]^{2-}$ . Metathesis with  $[\text{PPN}] \text{Cl}$  gave the  $[\text{PPN}]^+$  salt as a yellow-orange powder which was recrystallized from acetonitrile-diethyl ether at 203 K (79% yield).  $[\text{PPN}]_2[\text{Cr}_2(\text{CO})_{10}]$  was characterized by  $^1\text{H}$ ,  $^{13}\text{C}$  NMR and IR spectroscopy, and by microanalysis.

### Crystal data

$(\text{C}_{18}\text{H}_{15}\text{P})_2\text{N}]_2[\text{Cr}_2(\text{CO})_{10}]$	Mo $K\alpha$ radiation
$M_r = 1461.3$	$\lambda = 0.71073 \text{ \AA}$
Triclinic	Cell parameters from 25
$P\bar{1}$	reflections
$a = 12.847 (2) \text{ \AA}$	$\theta = 10-13^\circ$
$b = 15.196 (3) \text{ \AA}$	$\mu = 0.45 \text{ mm}^{-1}$
$c = 20.570 (4) \text{ \AA}$	$T = 293 (2) \text{ K}$
$\alpha = 94.61 (2)^\circ$	Block
$\beta = 90.07 (2)^\circ$	$0.40 \times 0.36 \times 0.36 \text{ mm}$
$\gamma = 114.53 (2)^\circ$	Orange
$V = 3638.8 (12) \text{ \AA}^3$	
$Z = 2$	
$D_x = 1.33 \text{ Mg m}^{-3}$	

### Data collection

Siemens P3 diffractometer	$R_{\text{int}} = 0.0736$
Wyckoff scans	$\theta_{\text{max}} = 22.55^\circ$
Absorption correction:	$h = 0 \rightarrow 13$
$\psi$ scans	$k = -16 \rightarrow 16$
$T_{\text{min}} = 0.922$ , $T_{\text{max}} = 0.951$	$l = -22 \rightarrow 22$
9993 measured reflections	3 standard reflections
9502 independent reflections	monitored every 197
5119 observed reflections	reflections
[ $I > 2\sigma(I)$ ]	intensity decay: <1%

### Refinement

Refinement on $F^2$	$\Delta\rho_{\text{max}} = 0.24 \text{ e \AA}^{-3}$
$R[F^2 > 2\sigma(F^2)] = 0.0390$	$\Delta\rho_{\text{min}} = -0.24 \text{ e \AA}^{-3}$
$wR(F^2) = 0.0783$	Extinction correction:
$S = 1.001$	<i>SHELXL93</i> (Sheldrick, 1993)
9477 reflections	Extinction coefficient:
758 parameters	0.00164 (14)
H atoms: riding on parent C atom with $U_{\text{iso}} = 0.08 \text{ \AA}^2$	Atomic scattering factors from <i>International Tables for Crystallography</i> (1992, Vol. C, Tables 4.2.6.8 and 6.1.1.4)
w = $1/[\sigma^2(F_o^2) + (0.0372P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$	
$(\Delta/\sigma)_{\text{max}} = -0.146$	

Table 1. Fractional atomic coordinates and equivalent isotropic displacement parameters ( $\text{\AA}^2$ )

	$x$	$y$	$z$	$U_{\text{eq}}$
Cr1	0.02808 (5)	0.12175 (4)	0.74413 (3)	0.0485 (2)
Cr2	-0.07579 (5)	-0.09477 (4)	0.74480 (3)	0.0524 (2)
P1	0.31262 (8)	0.58486 (7)	0.57906 (5)	0.0402 (3)
P2	0.41604 (8)	0.75646 (7)	0.50157 (5)	0.0392 (3)
P3	0.27800 (8)	0.56946 (7)	0.07307 (5)	0.0422 (3)
P4	0.33883 (8)	0.73305 (7)	-0.00873 (5)	0.0417 (3)
N1	0.3606 (2)	0.6490 (2)	0.52138 (14)	0.0423 (8)
N2	0.2776 (2)	0.6272 (2)	0.01286 (14)	0.0485 (9)
O1	0.1507 (3)	0.3347 (2)	0.7386 (2)	0.0912 (11)
O2	-0.0954 (3)	0.0951 (2)	0.6142 (2)	0.0973 (12)
O3	-0.1798 (3)	0.1179 (3)	0.8132 (2)	0.1164 (14)
O4	0.1306 (3)	0.1138 (2)	0.8741 (2)	0.0808 (10)
O5	0.2299 (3)	0.1024 (2)	0.6816 (2)	0.0843 (10)
O6	-0.1560 (3)	-0.3070 (2)	0.7532 (2)	0.1113 (14)
O7	-0.0022 (3)	-0.0968 (2)	0.6051 (2)	0.0931 (12)
O8	0.1640 (2)	-0.0596 (2)	0.78714 (14)	0.0652 (9)
O9	-0.1394 (3)	-0.0623 (2)	0.8826 (2)	0.0923 (12)
O10	-0.2983 (3)	-0.0943 (3)	0.6981 (2)	0.1064 (13)
C1	0.0996 (4)	0.2505 (3)	0.7416 (2)	0.0594 (12)
C2	-0.0500 (4)	0.1014 (3)	0.6637 (2)	0.0615 (13)
C3	-0.1009 (4)	0.1182 (3)	0.7863 (2)	0.0672 (13)
C4	0.0904 (3)	0.1165 (3)	0.8241 (2)	0.0530 (12)
C5	0.1490 (4)	0.1059 (3)	0.7039 (2)	0.0558 (12)
C6	-0.1264 (4)	-0.2233 (3)	0.7498 (2)	0.0735 (15)
C7	-0.0307 (4)	-0.0952 (3)	0.6585 (2)	0.0603 (13)
C8	0.0726 (4)	-0.0711 (3)	0.7715 (2)	0.0479 (11)
C9	-0.1137 (4)	-0.0725 (3)	0.8305 (3)	0.0630 (13)
C10	-0.2122 (4)	-0.0928 (3)	0.7158 (2)	0.0667 (13)
C11	0.2086 (2)	0.4366 (2)	0.48327 (12)	0.0601 (12)
C12	0.1266 (3)	0.3467 (2)	0.45820 (12)	0.0757 (15)
C13	0.0395 (2)	0.2914 (2)	0.4972 (2)	0.079 (2)
C14	0.0344 (2)	0.3259 (2)	0.5612 (2)	0.0760 (15)
C15	0.1165 (2)	0.4158 (2)	0.58628 (11)	0.0623 (12)
C16	0.2036 (2)	0.47112 (14)	0.54731 (13)	0.0449 (10)
C21	0.5350 (2)	0.6284 (2)	0.61534 (13)	0.0588 (12)
C22	0.6209 (2)	0.6147 (2)	0.6484 (2)	0.0821 (15)
C23	0.5934 (3)	0.5347 (3)	0.68414 (15)	0.084 (2)
C24	0.4800 (4)	0.4684 (2)	0.68692 (14)	0.091 (2)
C25	0.3941 (2)	0.4820 (2)	0.65391 (14)	0.0691 (13)
C26	0.4216 (2)	0.5620 (2)	0.61812 (13)	0.0430 (10)
C31	0.2902 (2)	0.6516 (2)	0.70571 (14)	0.0557 (11)
C32	0.2451 (3)	0.6963 (2)	0.75208 (10)	0.0664 (13)
C33	0.1600 (3)	0.7245 (2)	0.73382 (15)	0.0663 (14)
C34	0.1200 (2)	0.7079 (2)	0.6692 (2)	0.0614 (12)
C35	0.1651 (2)	0.6632 (2)	0.62281 (11)	0.0540 (11)
C36	0.2502 (2)	0.6350 (2)	0.64107 (12)	0.0394 (10)
C41	0.3952 (2)	0.8419 (2)	0.3921 (2)	0.080 (2)
C42	0.3425 (3)	0.8439 (2)	0.33347 (15)	0.103 (2)
C43	0.2465 (3)	0.7630 (3)	0.30850 (12)	0.086 (2)
C44	0.2033 (2)	0.6802 (2)	0.3421 (2)	0.087 (2)
C45	0.2559 (2)	0.6781 (2)	0.40068 (15)	0.0665 (13)
C46	0.3519 (2)	0.7590 (2)	0.42566 (11)	0.0441 (10)
C51	0.4484 (2)	0.8562 (2)	0.62124 (13)	0.0464 (10)

[(C<sub>18</sub>H<sub>15</sub>P)<sub>2</sub>N]<sub>2</sub>[Cr<sub>2</sub>(CO)<sub>10</sub>]

C52	0.4248 (2)	0.9123 (2)	0.67075 (10)	0.0606 (12)	C3—Cr1—C2	89.7 (2)	C56—P2—C66	108.97 (13)
C53	0.3485 (2)	0.9530 (2)	0.65877 (13)	0.0643 (13)	C5—Cr1—C2	91.1 (2)	N2—P3—C76	111.4 (2)
C54	0.2957 (2)	0.9375 (2)	0.5973 (2)	0.0615 (12)	C1—Cr1—Cr2	175.76 (13)	N2—P3—C86	108.90 (15)
C55	0.3193 (2)	0.8814 (2)	0.54778 (11)	0.0528 (11)	C4—Cr1—Cr2	82.68 (12)	C76—P3—C86	105.98 (12)
C56	0.3956 (2)	0.8407 (2)	0.55975 (11)	0.0388 (9)	C3—Cr1—Cr2	86.51 (14)	N2—P3—C96	115.05 (14)
C61	0.6477 (2)	0.8829 (2)	0.51810 (12)	0.0587 (12)	C5—Cr1—Cr2	85.06 (12)	C76—P3—C96	106.71 (14)
C62	0.7632 (2)	0.9077 (2)	0.50838 (14)	0.0713 (14)	C2—Cr1—Cr2	86.38 (13)	C86—P3—C96	108.33 (14)
C63	0.7967 (2)	0.8448 (2)	0.4703 (2)	0.0750 (14)	C6—Cr2—C8	92.8 (2)	N2—P4—C126	108.8 (2)
C64	0.7147 (3)	0.7572 (2)	0.44193 (14)	0.085 (2)	C6—Cr2—C10	98.4 (2)	N2—P4—C106	114.55 (14)
C65	0.5992 (2)	0.7324 (2)	0.45164 (13)	0.0703 (14)	C8—Cr2—C10	168.8 (2)	C126—P4—C106	108.88 (14)
C66	0.5657 (2)	0.7952 (2)	0.48973 (13)	0.0399 (9)	C6—Cr2—C7	96.1 (2)	N2—P4—C116	109.6 (2)
C71	0.0995 (2)	0.6117 (2)	0.11647 (12)	0.0572 (12)	C8—Cr2—C7	88.3 (2)	C126—P4—C116	106.45 (13)
C72	0.0100 (2)	0.6021 (2)	0.1573 (2)	0.0686 (13)	C10—Cr2—C7	89.5 (2)	C106—P4—C116	108.27 (13)
C73	-0.0136 (2)	0.5413 (2)	0.20745 (14)	0.0697 (14)	C6—Cr2—C9	93.3 (2)	P1—N1—P2	144.5 (2)
C74	0.0523 (3)	0.4901 (2)	0.21681 (11)	0.0647 (13)	C8—Cr2—C9	91.7 (2)	P4—N2—P3	138.9 (2)
C75	0.1419 (2)	0.4997 (2)	0.17599 (13)	0.0532 (11)	C10—Cr2—C9	88.7 (2)	O1—C1—Cr1	176.9 (4)
C76	0.1654 (2)	0.5605 (2)	0.12582 (11)	0.0403 (10)	C7—Cr2—C9	170.6 (2)	O2—C2—Cr1	175.6 (4)
C81	0.2865 (2)	0.3915 (2)	0.08107 (11)	0.0554 (11)	C6—Cr2—Cr1	174.59 (14)	O3—C3—Cr1	178.6 (4)
C82	0.2556 (2)	0.2943 (2)	0.06049 (15)	0.0688 (13)	C8—Cr2—Cr1	82.04 (12)	O4—C4—Cr1	179.3 (3)
C83	0.1898 (3)	0.25353 (14)	0.0032 (2)	0.0699 (14)	C10—Cr2—Cr1	86.86 (13)	O5—C5—Cr1	174.8 (4)
C84	0.1548 (2)	0.3100 (2)	-0.03347 (11)	0.0693 (13)	C7—Cr2—Cr1	85.28 (13)	O6—C6—Cr2	177.8 (4)
C85	0.1857 (2)	0.4073 (2)	-0.01289 (12)	0.0585 (12)	C9—Cr2—Cr1	85.41 (14)	O7—C7—Cr2	178.8 (4)
C86	0.2516 (2)	0.44801 (14)	0.04438 (13)	0.0436 (10)	N1—P1—C26	111.2 (2)	O8—C8—Cr2	177.6 (4)
C91	0.4146 (2)	0.6756 (2)	0.18045 (14)	0.0534 (11)	N1—P1—C16	108.43 (15)	O9—C9—Cr2	177.6 (4)
C92	0.5181 (3)	0.7205 (2)	0.21603 (12)	0.0733 (14)	C26—P1—C16	108.11 (13)	O10—C10—Cr2	178.0 (4)
C93	0.6143 (2)	0.7095 (2)	0.1935 (2)	0.093 (2)				
C94	0.6070 (2)	0.6535 (2)	0.1354 (2)	0.091 (2)				
C95	0.5036 (3)	0.6086 (2)	0.09978 (13)	0.0712 (14)				
C96	0.4074 (2)	0.6197 (2)	0.12233 (13)	0.0444 (10)				
C101	0.5451 (2)	0.8452 (2)	0.05723 (12)	0.0496 (11)				
C102	0.6124 (2)	0.9091 (2)	0.10836 (14)	0.0581 (12)				
C103	0.5629 (2)	0.9486 (2)	0.15553 (11)	0.0619 (13)				
C104	0.4460 (2)	0.9242 (2)	0.15159 (11)	0.0608 (12)				
C105	0.3787 (2)	0.8604 (2)	0.10047 (13)	0.0513 (11)				
C106	0.4282 (2)	0.8209 (2)	0.05329 (11)	0.0386 (9)				
C111	0.4911 (2)	0.8193 (2)	-0.10363 (14)	0.0615 (12)				
C112	0.5540 (2)	0.8178 (2)	-0.15830 (14)	0.0759 (14)				
C113	0.5498 (2)	0.7301 (3)	-0.18658 (11)	0.0780 (15)				
C114	0.4826 (3)	0.6439 (2)	-0.16020 (15)	0.0720 (14)				
C115	0.4197 (2)	0.6454 (2)	-0.10553 (14)	0.0576 (12)				
C116	0.4239 (2)	0.7330 (2)	-0.07725 (11)	0.0452 (10)				
C121	0.2485 (2)	0.8666 (2)	-0.02806 (14)	0.0758 (15)				
C122	0.1645 (3)	0.8927 (2)	-0.0513 (2)	0.098 (2)				
C123	0.0651 (2)	0.8224 (3)	-0.08210 (15)	0.083 (2)				
C124	0.0496 (2)	0.7260 (2)	-0.08973 (14)	0.0781 (15)				
C125	0.1335 (2)	0.6999 (2)	-0.06653 (14)	0.0636 (12)				
C126	0.2330 (2)	0.7702 (2)	-0.03570 (13)	0.0450 (10)				

Table 2. Selected geometric parameters (Å, °)

Cr1—C1	1.789 (4)	P3—N2	1.575 (3)
Cr1—C4	1.850 (5)	P3—C76	1.775 (2)
Cr1—C3	1.854 (5)	P3—C86	1.782 (2)
Cr1—C5	1.855 (5)	P3—C96	1.786 (2)
Cr1—C2	1.869 (5)	P4—N2	1.571 (3)
Cr1—Cr2	2.9947 (11)	P4—C126	1.778 (2)
Cr2—C6	1.796 (5)	P4—C106	1.783 (2)
Cr2—C8	1.860 (5)	P4—C116	1.785 (2)
Cr2—C10	1.863 (5)	O1—C1	1.178 (4)
Cr2—C7	1.868 (5)	O2—C2	1.151 (5)
Cr2—C9	1.871 (5)	O3—C3	1.154 (5)
P1—N1	1.558 (3)	O4—C4	1.162 (4)
P1—C26	1.781 (2)	O5—C5	1.155 (4)
P1—C16	1.784 (2)	O6—C6	1.176 (4)
P1—C36	1.790 (2)	O7—C7	1.160 (5)
P2—N1	1.575 (3)	O8—C8	1.155 (4)
P2—C46	1.775 (2)	O9—C9	1.139 (5)
P2—C56	1.782 (2)	O10—C10	1.155 (5)
P2—C66	1.785 (2)		
C1—Cr1—C4	96.7 (2)	N1—P1—C36	114.68 (14)
C1—Cr1—C3	97.7 (2)	C26—P1—C36	107.07 (14)
C4—Cr1—C3	88.6 (2)	C16—P1—C36	107.12 (13)
C1—Cr1—C5	90.7 (2)	N1—P2—C46	108.0 (2)
C4—Cr1—C5	89.0 (2)	N1—P2—C56	113.05 (14)
C3—Cr1—C5	171.5 (2)	C46—P2—C56	108.49 (14)
C1—Cr1—C2	94.2 (2)	N1—P2—C66	110.97 (15)
C4—Cr1—C2	169.0 (2)	C46—P2—C66	107.12 (13)

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