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cis-μ-1,4-Diphenyl-1,4-diphosphorinane-κ^2P:P'-bis[pentacarbonylchromium(0)] deuterochloroform solvate

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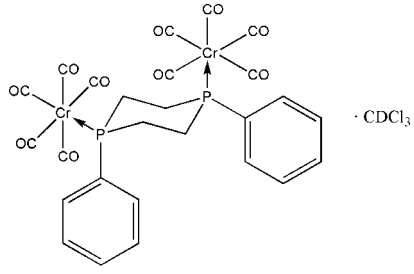
Key indicators: single-crystal X-ray study; T = 133 K; mean σ(C–C) = 0.006 Å; R factor = 0.052; wR factor = 0.115; data-to-parameter ratio = 23.6.

The title compound, $[Cr_2(C_{16}H_{18}P_2)(CO)_{10}]\cdot CDCl_3$, crystallizes with one $CDCl_3$ solvent molecule per formula unit, with the solvent molecule residing in a niche between the two phenyl rings. The crystal studied was an inversion twin. The crystal packing shows secondary C—H···O interactions.

Related literature

For related literature, see: Brooks *et al.* (1989); Clemente (2005); Cucciolito *et al.* (2003); Gallagher & Rae (1979); Gallagher, Peterson & Rae (1979); Hinton & Mann (1959); Issleib & Standtke (1963); Lee & Brown (1992); Plastas *et al.* (1973).



Experimental

Crystal data

$[Cr_2(C_{16}H_{18}P_2)(CO)_{10}]\cdot CDCl_3$
M_r = 776.72
Orthorhombic, $P2_12_12_1$
a = 10.7040 (12) Å
b = 11.6806 (12) Å
c = 25.7328 (16) Å
V = 3217.3 (5) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 1.08 mm^{-1}
T = 133 (2) K
0.18 × 0.11 × 0.09 mm

Data collection

Bruker SMART 1000 CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.742, T_{max} = 0.909
53237 measured reflections
9399 independent reflections
7487 reflections with $I > 2\sigma(I)$
R_{int} = 0.084

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.052
$wR(F^2)$ = 0.115
S = 1.06
9399 reflections
398 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.69 e Å^{-3}
$\Delta\rho_{min}$ = −0.44 e Å^{-3}
Absolute structure: (Flack, 1983), 4160 Friedel pairs
Flack parameter: 0.48 (2)

Table 1
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
C36—H36···Cl3^{i}	0.95	2.97	3.814 (4)	148
C23—H23···O12^{ii}	0.95	2.66	3.470 (5)	144
C3—H3*A*···O11^{iii}	0.99	2.43	3.258 (5)	141
C33—H33···O7^{iv}	0.95	2.53	3.458 (5)	167

Symmetry codes: (i) $x-\frac{1}{2}, -y+\frac{1}{2}, -z$; (ii) $-x+1, y+\frac{1}{2}, -z+\frac{1}{2}$; (iii) $-x, y-\frac{1}{2}, -z+\frac{1}{2}$; (iv) $-x+1, y-\frac{1}{2}, -z+\frac{1}{2}$.

Data collection: *SMART* (Bruker, 2001); cell refinement: *SAINT* (Bruker, 2001); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *XP* (Siemens, 1994); software used to prepare material for publication: *SHELXL97*.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: GG3122).

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supplementary materials

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cis-μ-1,4-Diphenyl-1,4-diphosphorinane-κ^2P:P'-bis[pentacarbonylchromium(0)] deuterochloroform solvate

M. Fild, O. N. Krüger, I. Silaghi-Dumitrescu, C. Thöne and A. Weinkauf

Comment

We are interested in configuration and conformation of various heterocyclic phosphorus ring systems and report herein the first structure of a transition metal carbonyl complex of the liugand 1,4-diphenyl-1,4-diphosphorinane (II). For the parent compound (II), prepared according to Hinton and Mann (Hinton & Mann, 1959), the structure of the *trans*-isomer has been reported (Brooks *et al.*, 1989). There are only two structures of cationic Ni complexes of (II) known, both of them are chelates (Clemente, 2005; Cucciolito *et al.*, 2003). The disulfide (Issleib & Standtke, 1963) and the dioxide (Gallagher & Rae, 1979; Gallagher *et al.*, 1979) derivatives of (II) have been obtained as *cis/trans* mixture. In the case of the dioxide, both *cis*- and *trans*-isomers, have been analysed by X-ray diffraction (Gallagher & Rae, 1979; Gallagher *et al.*, 1979). 1,4-Dipheny-1,4-diphosphorinane (II) reacts with acetonitrilepentacarbonylchromium(0) to give a mixture of *cis*- and *trans*-isomers in solution. The ^{31}P NMR spectrum shows two singlets very close to one another (30.8 p.p.m., 86%, and 31.7 p.p.m., 14%). Suitable single crystals were obtained only for the *cis*-isomer (I) from $CDCl_3$ by slow evaporation at room temperature. The crystal is a twinned as indicated by the Flack parameter of 0.48 (2) and was treated using a BASF value of 0.482 and TWIN command in the refinement.

The molecular structure of (I) is shown in Fig. 1. The six-membered diphosphorinane ring adopts a chair conformation with corresponding torsion angles 52.2 (3)° (C3—P4—C5—C6), −59.4 (3)° (C6—P1—C2—C3) and 66.6 (3)° (P1—C2—C3—P4). The substituents Ph and $Cr(CO)_5$ at P atoms are in a *cis*-arrangement. The geometry around phosphorus is distorted tetrahedral with angles ranging from 100.69 (16)° (C21—P1—C6) to 117.82 (12)° (C6—P1—Cr1) (Table 1). The P1—Cr1 and P4—Cr2 bond distances of 2,3786 (11) Å and 2.3695 (11) Å are similar to the P—Cr bond length of 2.3664 (5) Å in $Me_3PCr(CO)_5$ (Lee & Brown, 1992) and slightly shorter than the P—Cr bond length of 2.422 (1) Å in $Ph_3PCr(CO)_5$ (Plastas *et al.*, 1973). The *trans*-Cr1—C11 and *trans*-Cr2—C16 bond distances are correspondingly longer [1.860 (4) Å and 1.876 (4) Å *cf.* 1.850 (2) Å in $Me_3PCr(CO)_5$ and 1.845 (4) Å in $Ph_3PCr(CO)_5$].

Experimental

A mixture of 40 ml pentane, 0.63 g (2.314 mmol) of (II) and 1.58 g (6.778 mmol) $Cr(CO)_5(CH_3CN)$ was stirred at room temperature under exclusion of light. The reaction mixture was monitored by ^{31}P NMR spectroscopy (C_6D_6 capillary). The reaction was complete after 2 days of stirring. The precipitate was filtered off and washed with 20 ml pentane and 20 ml hexane. After drying *in vacuo*, 1.08 g of a yellowish solid was obtained. Crystallization from $CDCl_3$ gave (I). Yield: 71%; Mp = 165–168°C. Elemental analysis calculated: C 47.58, H 2.76%; found: C 47.88, H 2.95%. IR (KBr, ν, cm-1): 2065 (*versus*), 1920 (*versus*, br), 1437 (*m*), 1410 (*m*), 1131 (*m*), 845 (*m*), 743 (*m*), 695 (*s*), 673 (*versus*), 552 (*s*), 494 (*m*), 465 (*m*); ^{1}H NMR (400 MHz, $CDCl_3$): δ 2.67 (br, 8H, CH_2) 7,42 (br, 10H, Ph); ^{13}C NMR (100.61 MHz, $CDCl_3$): δ 23.7 (*pseudo*-d,

N = $^1J_{CP}$ + $^2J_{CP}$ = 17.7 Hz, CH_2) 127.9–135.9 (m, Ph) 216.1 (*pseudo*-d, N = $^2J_{CP}$ + $^5J_{CP}$= 13.2 Hz, CO–*cis*) 220.5 (s, CO–*trans*); ^{31}P NMR (81 MHz, $CDCl_3$): δ 30.8.

Figures

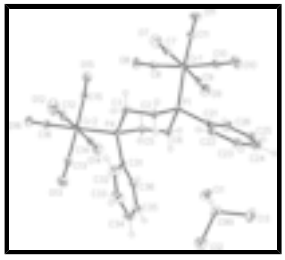


Fig. 1. The independent components of I, showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 50% probability level.

cis-μ-1,4-Diphenyl-1,4-diphosphorinane-κ^2*P*:*P*'- bis[pentacarbonylchromium(0)] deuterochloroform solvate

Crystal data

$[Cr_2(C_{16}H_{18}P_2)(CO)_{10}]\cdot CDCl_3$	D_x = 1.603 Mg m^{-3}
M_r = 776.72	Melting point: 440 K
Orthorhombic, $P2_12_12_1$	Mo $K\alpha$ radiation λ = 0.71073 Å
Hall symbol: P 2ac 2ab	Cell parameters from 9963 reflections
a = 10.7040 (12) Å	θ = 2.4–30.5º
b = 11.6806 (12) Å	μ = 1.08 mm^{-1}
c = 25.7328 (16) Å	T = 133 (2) K
V = 3217.3 (5) Å^3	Tablet, colourless
Z = 4	0.18 × 0.11 × 0.09 mm
F_{000} = 1560	

Data collection

Bruker SMART 1000CCD diffractometer	9399 independent reflections
Radiation source: fine-focus sealed tube	7487 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.084
Detector resolution: 8.192 pixels mm^{-1}	θ_{max} = 30.0º
T = 133(2) K	θ_{min} = 1.6º
ω– and φ–scans	h = −15→14
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)	k = −16→16
T_{min} = 0.742, T_{max} = 0.909	l = −34→36
53237 measured reflections	

Refinement

Refinement on F^2	Hydrogen site location: inferred from neighbouring sites
Least-squares matrix: full	H-atom parameters constrained
$R[F^2 > 2\sigma(F^2)] = 0.052$	$w = 1/[\sigma^2(F_o^2) + (0.0495P)^2 + 3.2419P]$ where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.115$	$(\Delta/\sigma)_{max} < 0.001$
$S = 1.06$	$\Delta\rho_{max} = 0.69$ e Å^{-3}
9399 reflections	$\Delta\rho_{min} = -0.44$ e Å^{-3}
398 parameters	Extinction correction: none
Primary atom site location: structure-invariant direct methods	Absolute structure: (Flack, 1983), 4161 Friedel pairs
Secondary atom site location: difference Fourier map	Flack parameter: 0.48 (2)

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted *R*-factor *wR* and goodness of fit *S* are based on F^2, conventional *R*-factors *R* are based on *F*, with *F* set to zero for negative F^2. The threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating *R*-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. *R*-factors based on F^2 are statistically about twice as large as those based on *F*, and *R*- factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
Cr1	0.16969 (5)	0.33718 (5)	0.19889 (2)	0.01353 (12)
Cr2	0.11283 (6)	−0.19314 (5)	0.10201 (2)	0.01561 (12)
P1	0.33788 (9)	0.23062 (7)	0.16269 (3)	0.01228 (16)
C2	0.3798 (4)	0.0946 (3)	0.19392 (13)	0.0139 (6)
H2A	0.4606	0.0675	0.1798	0.017*
H2B	0.3903	0.1071	0.2317	0.017*
C3	0.2795 (4)	0.0023 (3)	0.18494 (14)	0.0181 (7)
H3A	0.1982	0.0327	0.1971	0.022*
H3B	0.3000	−0.0649	0.2068	0.022*
P4	0.26110 (9)	−0.04632 (7)	0.11728 (3)	0.01291 (18)
C5	0.2301 (4)	0.0904 (3)	0.08382 (14)	0.0161 (7)
H5A	0.2274	0.0758	0.0459	0.019*
H5B	0.1465	0.1180	0.0944	0.019*
C6	0.3251 (3)	0.1858 (3)	0.09415 (12)	0.0161 (7)
H6A	0.3019	0.2533	0.0730	0.019*
H6B	0.4082	0.1596	0.0822	0.019*
C7	0.1770 (4)	0.2498 (3)	0.26106 (14)	0.0211 (8)

C8	0.0434 (4)	0.2358 (3)	0.17539 (13)	0.0164 (7)
C9	0.1597 (4)	0.4160 (3)	0.13461 (15)	0.0193 (8)
C10	0.2871 (4)	0.4425 (3)	0.22772 (17)	0.0228 (8)
C11	0.0446 (4)	0.4309 (3)	0.22544 (15)	0.0201 (8)
C12	0.1400 (4)	−0.2533 (3)	0.16923 (15)	0.0237 (9)
C13	0.2427 (4)	−0.2886 (3)	0.07611 (17)	0.0255 (9)
C14	0.0897 (4)	−0.1326 (3)	0.03402 (16)	0.0212 (8)
C15	−0.0192 (4)	−0.1020 (3)	0.12905 (16)	0.0207 (8)
C16	−0.0069 (4)	−0.3069 (3)	0.08905 (14)	0.0207 (8)
O7	0.1768 (3)	0.1983 (2)	0.29885 (11)	0.0316 (7)
O8	−0.0389 (3)	0.1801 (2)	0.16324 (12)	0.0277 (6)
O9	0.1520 (3)	0.4601 (3)	0.09523 (12)	0.0334 (7)
O10	0.3554 (3)	0.5059 (3)	0.24578 (14)	0.0439 (9)
O11	−0.0331 (3)	0.4897 (3)	0.24092 (13)	0.0330 (7)
O12	0.1572 (4)	−0.2922 (3)	0.20938 (12)	0.0444 (9)
O13	0.3198 (3)	−0.3462 (3)	0.06057 (14)	0.0388 (8)
O14	0.0782 (3)	−0.0974 (3)	−0.00664 (12)	0.0358 (8)
O15	−0.1029 (3)	−0.0537 (3)	0.14483 (12)	0.0318 (7)
O16	−0.0825 (3)	−0.3745 (3)	0.08212 (13)	0.0331 (7)
C21	0.4838 (3)	0.3107 (3)	0.16091 (13)	0.0136 (6)
C22	0.5888 (3)	0.2797 (3)	0.18981 (14)	0.0186 (7)
H22	0.5865	0.2131	0.2110	0.022*
C23	0.6971 (4)	0.3460 (3)	0.18781 (14)	0.0214 (8)
H23	0.7686	0.3235	0.2072	0.026*
C24	0.7013 (4)	0.4445 (4)	0.15782 (17)	0.0263 (9)
H24	0.7749	0.4900	0.1569	0.032*
C25	0.5973 (4)	0.4760 (3)	0.12927 (16)	0.0255 (9)
H25	0.5997	0.5430	0.1083	0.031*
C26	0.4891 (4)	0.4101 (3)	0.13107 (15)	0.0219 (8)
H26	0.4179	0.4331	0.1117	0.026*
C31	0.4194 (3)	−0.0828 (3)	0.09818 (15)	0.0156 (7)
C32	0.4960 (4)	−0.1476 (4)	0.13037 (15)	0.0254 (9)
H32	0.4669	−0.1688	0.1639	0.030*
C33	0.6136 (4)	−0.1820 (4)	0.11458 (19)	0.0350 (10)
H33	0.6642	−0.2268	0.1370	0.042*
C34	0.6580 (4)	−0.1504 (4)	0.06531 (17)	0.0306 (10)
H34	0.7392	−0.1727	0.0545	0.037*
C35	0.5841 (4)	−0.0878 (3)	0.03317 (18)	0.0288 (10)
H35	0.6141	−0.0658	−0.0001	0.035*
C36	0.4645 (4)	−0.0556 (3)	0.04871 (15)	0.0210 (8)
H36	0.4127	−0.0144	0.0253	0.025*
C99	0.8007 (4)	0.2053 (4)	0.05647 (15)	0.0260 (9)
H99	0.7908	0.1806	0.0935	0.031*
Cl1	0.65207 (10)	0.22884 (10)	0.02999 (4)	0.0332 (2)
Cl2	0.87929 (12)	0.09637 (10)	0.02228 (5)	0.0383 (3)
Cl3	0.88993 (12)	0.33268 (10)	0.05508 (4)	0.0375 (3)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cr1	0.0121 (3)	0.0124 (2)	0.0161 (3)	0.0000 (2)	−0.0004 (2)	−0.0022 (2)
Cr2	0.0157 (3)	0.0123 (3)	0.0188 (3)	−0.0028 (2)	−0.0007 (2)	−0.0002 (2)
P1	0.0126 (4)	0.0114 (4)	0.0128 (4)	0.0005 (3)	0.0001 (3)	−0.0002 (3)
C2	0.0185 (18)	0.0106 (14)	0.0125 (15)	−0.0001 (13)	−0.0010 (13)	0.0002 (12)
C3	0.020 (2)	0.0190 (18)	0.0158 (17)	−0.0020 (14)	0.0029 (14)	0.0035 (14)
P4	0.0138 (5)	0.0112 (4)	0.0137 (4)	−0.0013 (3)	0.0005 (3)	0.0004 (3)
C5	0.0169 (19)	0.0137 (16)	0.0176 (17)	−0.0029 (14)	−0.0027 (14)	0.0003 (13)
C6	0.0176 (18)	0.0165 (16)	0.0141 (15)	−0.0007 (14)	−0.0018 (13)	−0.0004 (13)
C7	0.023 (2)	0.0169 (18)	0.0236 (19)	0.0048 (15)	−0.0017 (15)	−0.0067 (15)
C8	0.0155 (19)	0.0156 (16)	0.0181 (17)	0.0013 (14)	−0.0002 (13)	−0.0032 (13)
C9	0.019 (2)	0.0151 (17)	0.0238 (19)	0.0000 (15)	−0.0041 (15)	−0.0046 (14)
C10	0.017 (2)	0.0193 (19)	0.032 (2)	0.0049 (15)	−0.0028 (16)	−0.0102 (17)
C11	0.020 (2)	0.0235 (19)	0.0171 (17)	0.0023 (15)	0.0003 (15)	−0.0019 (15)
C12	0.032 (2)	0.0119 (17)	0.027 (2)	−0.0042 (14)	−0.0012 (16)	0.0034 (15)
C13	0.023 (2)	0.0172 (19)	0.036 (2)	−0.0084 (16)	0.0004 (17)	−0.0076 (16)
C14	0.021 (2)	0.0172 (18)	0.025 (2)	−0.0036 (14)	−0.0013 (15)	−0.0076 (15)
C15	0.018 (2)	0.0156 (17)	0.028 (2)	−0.0061 (15)	−0.0019 (15)	0.0003 (15)
C16	0.021 (2)	0.0200 (18)	0.0208 (18)	−0.0047 (15)	0.0015 (14)	−0.0011 (15)
O7	0.046 (2)	0.0285 (15)	0.0199 (13)	0.0021 (14)	−0.0069 (13)	0.0045 (12)
O8	0.0219 (16)	0.0235 (15)	0.0378 (16)	−0.0063 (12)	−0.0058 (12)	−0.0014 (13)
O9	0.0368 (19)	0.0271 (15)	0.0362 (17)	0.0004 (13)	−0.0040 (14)	0.0111 (13)
O10	0.035 (2)	0.0392 (19)	0.058 (2)	−0.0085 (15)	−0.0069 (16)	−0.0282 (17)
O11	0.0267 (19)	0.0329 (17)	0.0395 (18)	0.0107 (14)	0.0113 (14)	−0.0043 (14)
O12	0.065 (3)	0.0329 (17)	0.0349 (17)	−0.0079 (17)	−0.0119 (17)	0.0115 (14)
O13	0.0254 (18)	0.0287 (16)	0.062 (2)	0.0015 (14)	0.0034 (16)	−0.0145 (16)
O14	0.050 (2)	0.0360 (17)	0.0212 (15)	−0.0045 (15)	−0.0064 (14)	0.0005 (13)
O15	0.0254 (17)	0.0264 (15)	0.0435 (18)	−0.0017 (13)	0.0053 (14)	−0.0047 (13)
O16	0.0289 (18)	0.0267 (16)	0.0436 (18)	−0.0133 (13)	0.0064 (14)	−0.0090 (13)
C21	0.0102 (17)	0.0135 (15)	0.0172 (16)	0.0029 (12)	−0.0014 (12)	−0.0019 (13)
C22	0.016 (2)	0.0180 (17)	0.0217 (18)	0.0005 (13)	−0.0013 (14)	0.0008 (14)
C23	0.019 (2)	0.0244 (19)	0.0211 (18)	−0.0030 (15)	−0.0017 (13)	−0.0027 (16)
C24	0.016 (2)	0.031 (2)	0.032 (2)	−0.0107 (16)	0.0026 (16)	−0.0065 (18)
C25	0.032 (3)	0.0193 (19)	0.026 (2)	−0.0077 (16)	0.0026 (17)	0.0029 (15)
C26	0.018 (2)	0.025 (2)	0.0230 (19)	−0.0028 (15)	−0.0003 (15)	0.0087 (16)
C31	0.0120 (18)	0.0103 (15)	0.0245 (18)	−0.0039 (12)	−0.0037 (14)	0.0012 (14)
C32	0.023 (2)	0.031 (2)	0.0223 (19)	0.0051 (17)	−0.0070 (15)	0.0078 (16)
C33	0.018 (2)	0.038 (2)	0.049 (3)	0.0115 (19)	−0.016 (2)	−0.009 (2)
C34	0.014 (2)	0.037 (2)	0.040 (2)	0.0004 (18)	0.0027 (17)	−0.019 (2)
C35	0.029 (3)	0.024 (2)	0.033 (2)	−0.0049 (17)	0.0140 (18)	−0.0107 (18)
C36	0.027 (2)	0.0147 (17)	0.0218 (19)	0.0029 (15)	0.0038 (15)	−0.0021 (15)
C99	0.019 (2)	0.036 (2)	0.0230 (19)	−0.0050 (16)	0.0010 (15)	0.0022 (17)
Cl1	0.0193 (5)	0.0412 (6)	0.0391 (6)	−0.0010 (4)	0.0013 (4)	0.0050 (5)
Cl2	0.0322 (6)	0.0342 (6)	0.0485 (7)	0.0081 (5)	0.0022 (5)	0.0083 (5)
Cl3	0.0374 (6)	0.0414 (6)	0.0338 (5)	−0.0186 (5)	0.0011 (5)	0.0006 (5)

Geometric parameters (Å, °)

Cr1—C7	1.899 (4)	C22—C23	1.396 (5)
Cr1—C8	1.896 (4)	C23—C24	1.386 (6)
Cr1—C9	1.896 (4)	C24—C25	1.384 (6)
Cr1—C10	1.909 (4)	C25—C26	1.392 (6)
Cr1—C11	1.860 (4)	C31—C32	1.390 (5)
Cr1—P1	2.3786 (11)	C31—C36	1.398 (5)
Cr2—C12	1.890 (4)	C32—C33	1.382 (6)
Cr2—C13	1.903 (4)	C33—C34	1.403 (7)
Cr2—C14	1.903 (4)	C34—C35	1.358 (7)
Cr2—C15	1.901 (4)	C35—C36	1.393 (6)
Cr2—C16	1.876 (4)	C99—Cl1	1.753 (4)
Cr2—P4	2.3695 (11)	C99—Cl2	1.761 (4)
P1—C2	1.836 (3)	C99—Cl3	1.769 (4)
P1—C6	1.845 (3)	C2—H2A	0.9900
P1—C21	1.821 (4)	C2—H2B	0.9900
P4—C31	1.815 (4)	C3—H3A	0.9900
P4—C5	1.844 (4)	C3—H3B	0.9900
C2—C3	1.539 (5)	C5—H5A	0.9900
C3—P4	1.842 (4)	C5—H5B	0.9900
C5—C6	1.531 (5)	C6—H6A	0.9900
C7—O7	1.144 (5)	C6—H6B	0.9900
C8—O8	1.139 (4)	C22—H22	0.9500
C9—O9	1.140 (5)	C23—H23	0.9500
C10—O10	1.140 (5)	C24—H24	0.9500
C11—O11	1.150 (5)	C25—H25	0.9500
C12—O12	1.144 (5)	C26—H26	0.9500
C13—O13	1.137 (5)	C32—H32	0.9500
C14—O14	1.131 (5)	C33—H33	0.9500
C15—O15	1.135 (5)	C34—H34	0.9500
C16—O16	1.144 (5)	C35—H35	0.9500
C21—C26	1.393 (5)	C36—H36	0.9500
C21—C22	1.396 (5)	C99—H99	1.0000
C11—Cr1—C8	88.37 (17)	C21—C22—C23	120.4 (3)
C11—Cr1—C9	89.64 (17)	C24—C23—C22	120.5 (4)
C8—Cr1—C9	89.13 (16)	C25—C24—C23	119.4 (4)
C11—Cr1—C7	92.11 (17)	C24—C25—C26	120.3 (4)
C8—Cr1—C7	87.85 (16)	C25—C26—C21	120.9 (4)
C9—Cr1—C7	176.46 (15)	C32—C31—C36	117.6 (4)
C11—Cr1—C10	87.21 (16)	C32—C31—P4	121.2 (3)
C8—Cr1—C10	174.78 (17)	C36—C31—P4	121.0 (3)
C9—Cr1—C10	93.62 (17)	C33—C32—C31	121.3 (4)
C7—Cr1—C10	89.54 (18)	C32—C33—C34	119.9 (4)
C11—Cr1—P1	175.46 (13)	C35—C34—C33	119.6 (4)
C8—Cr1—P1	95.06 (11)	C34—C35—C36	120.4 (4)
C9—Cr1—P1	87.44 (12)	C35—C36—C31	121.2 (4)
C7—Cr1—P1	91.00 (12)	Cl1—C99—Cl2	110.7 (2)

C10—Cr1—P1	89.50 (12)	Cl1—C99—Cl3	110.5 (2)
C16—Cr2—C12	90.26 (16)	Cl2—C99—Cl3	109.9 (2)
C16—Cr2—C15	87.37 (17)	C3—C2—H2A	109.3
C12—Cr2—C15	89.28 (18)	P1—C2—H2A	109.3
C16—Cr2—C13	91.25 (17)	C3—C2—H2B	109.3
C12—Cr2—C13	89.44 (19)	P1—C2—H2B	109.3
C15—Cr2—C13	178.12 (17)	H2A—C2—H2B	107.9
C16—Cr2—C14	90.63 (16)	C2—C3—H3A	108.4
C12—Cr2—C14	178.62 (19)	P4—C3—H3A	108.4
C15—Cr2—C14	91.82 (17)	C2—C3—H3B	108.4
C13—Cr2—C14	89.47 (18)	P4—C3—H3B	108.4
C16—Cr2—P4	178.66 (13)	H3A—C3—H3B	107.4
C12—Cr2—P4	90.81 (12)	C6—C5—H5A	108.4
C15—Cr2—P4	91.83 (11)	P4—C5—H5A	108.4
C13—Cr2—P4	89.58 (12)	C6—C5—H5B	108.4
C14—Cr2—P4	88.33 (12)	P4—C5—H5B	108.4
C21—P1—C2	104.25 (16)	H5A—C5—H5B	107.5
C21—P1—C6	100.69 (16)	C5—C6—H6A	108.5
C2—P1—C6	101.02 (15)	P1—C6—H6A	108.5
C21—P1—Cr1	112.96 (11)	C5—C6—H6B	108.5
C2—P1—Cr1	117.78 (12)	P1—C6—H6B	108.5
C6—P1—Cr1	117.82 (12)	H6A—C6—H6B	107.5
C3—C2—P1	111.7 (3)	C21—C22—H22	119.8
C2—C3—P4	115.6 (2)	C23—C22—H22	119.8
C31—P4—C3	103.24 (18)	C24—C23—H23	119.7
C31—P4—C5	104.17 (17)	C22—C23—H23	119.7
C3—P4—C5	101.13 (17)	C25—C24—H24	120.3
C31—P4—Cr2	114.21 (11)	C23—C24—H24	120.3
C3—P4—Cr2	116.85 (12)	C24—C25—H25	119.8
C5—P4—Cr2	115.40 (12)	C26—C25—H25	119.8
C6—C5—P4	115.5 (3)	C25—C26—H26	119.5
C5—C6—P1	114.9 (2)	C21—C26—H26	119.5
O7—C7—Cr1	177.4 (4)	C33—C32—H32	119.3
O8—C8—Cr1	174.8 (3)	C31—C32—H32	119.3
O9—C9—Cr1	177.6 (3)	C32—C33—H33	120.1
O10—C10—Cr1	178.5 (4)	C34—C33—H33	120.1
O11—C11—Cr1	178.7 (4)	C35—C34—H34	120.2
O12—C12—Cr2	178.3 (4)	C33—C34—H34	120.2
O13—C13—Cr2	179.5 (4)	C34—C35—H35	119.8
O14—C14—Cr2	178.7 (4)	C36—C35—H35	119.8
O15—C15—Cr2	175.5 (3)	C35—C36—H36	119.4
O16—C16—Cr2	177.8 (4)	C31—C36—H36	119.4
C26—C21—C22	118.5 (3)	Cl1—C99—H99	108.6
C26—C21—P1	118.5 (3)	Cl2—C99—H99	108.6
C22—C21—P1	123.0 (3)	Cl3—C99—H99	108.6
C8—Cr1—P1—C21	165.99 (16)	Cr2—P4—C5—C6	179.3 (2)
C9—Cr1—P1—C21	77.09 (17)	P4—C5—C6—P1	−61.1 (3)
C7—Cr1—P1—C21	−106.08 (17)	C21—P1—C6—C5	164.9 (3)
C10—Cr1—P1—C21	−16.55 (18)	C2—P1—C6—C5	58.0 (3)

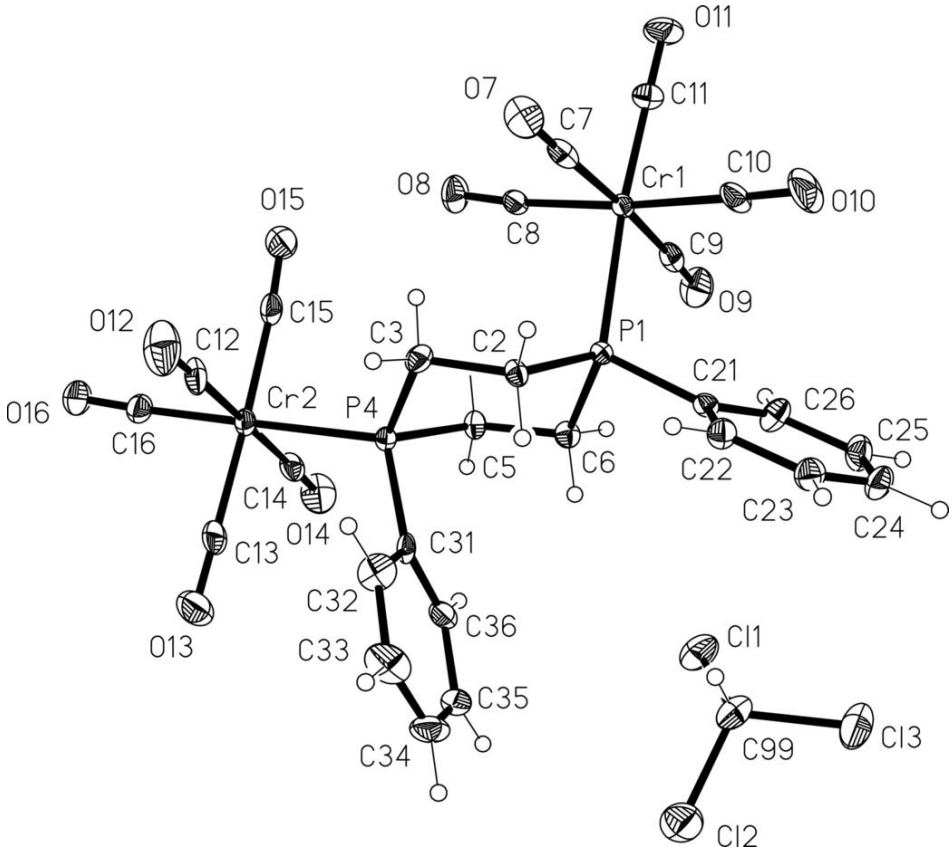
C8—Cr1—P1—C2	−72.33 (17)	Cr1—P1—C6—C5	−71.8 (3)
C9—Cr1—P1—C2	−161.22 (17)	C2—P1—C21—C26	168.6 (3)
C7—Cr1—P1—C2	15.60 (17)	C6—P1—C21—C26	64.2 (3)
C10—Cr1—P1—C2	105.13 (19)	Cr1—P1—C21—C26	−62.4 (3)
C8—Cr1—P1—C6	49.16 (17)	C2—P1—C21—C22	−14.2 (3)
C9—Cr1—P1—C6	−39.73 (17)	C6—P1—C21—C22	−118.6 (3)
C7—Cr1—P1—C6	137.09 (17)	Cr1—P1—C21—C22	114.8 (3)
C10—Cr1—P1—C6	−133.38 (18)	C26—C21—C22—C23	−1.3 (5)
C21—P1—C2—C3	−163.5 (2)	P1—C21—C22—C23	−178.5 (3)
C6—P1—C2—C3	−59.4 (3)	C21—C22—C23—C24	1.1 (6)
Cr1—P1—C2—C3	70.4 (3)	C22—C23—C24—C25	−0.8 (6)
P1—C2—C3—P4	66.6 (3)	C23—C24—C25—C26	0.7 (6)
C2—C3—P4—C31	51.6 (3)	C24—C25—C26—C21	−0.9 (6)
C2—C3—P4—C5	−56.0 (3)	C22—C21—C26—C25	1.2 (6)
C2—C3—P4—Cr2	177.9 (2)	P1—C21—C26—C25	178.5 (3)
C12—Cr2—P4—C31	90.69 (19)	C3—P4—C31—C32	46.2 (3)
C15—Cr2—P4—C31	180.00 (18)	C5—P4—C31—C32	151.5 (3)
C13—Cr2—P4—C31	1.25 (19)	Cr2—P4—C31—C32	−81.7 (3)
C14—Cr2—P4—C31	−88.23 (18)	C3—P4—C31—C36	−139.6 (3)
C12—Cr2—P4—C3	−29.91 (19)	C5—P4—C31—C36	−34.3 (3)
C15—Cr2—P4—C3	59.40 (19)	Cr2—P4—C31—C36	92.5 (3)
C13—Cr2—P4—C3	−119.3 (2)	C36—C31—C32—C33	1.3 (6)
C14—Cr2—P4—C3	151.17 (19)	P4—C31—C32—C33	175.7 (3)
C12—Cr2—P4—C5	−148.59 (19)	C31—C32—C33—C34	0.6 (6)
C15—Cr2—P4—C5	−59.29 (18)	C32—C33—C34—C35	−1.0 (6)
C13—Cr2—P4—C5	121.97 (19)	C33—C34—C35—C36	−0.5 (6)
C14—Cr2—P4—C5	32.48 (18)	C34—C35—C36—C31	2.5 (6)
C31—P4—C5—C6	−54.6 (3)	C32—C31—C36—C35	−2.8 (6)
C3—P4—C5—C6	52.2 (3)	P4—C31—C36—C35	−177.3 (3)

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C36—H36···Cl3^{i}	0.95	2.97	3.814 (4)	148
C23—H23···O12^{ii}	0.95	2.66	3.470 (5)	144
C3—H3A···O11^{iii}	0.99	2.43	3.258 (5)	141
C33—H33···O7^{iv}	0.95	2.53	3.458 (5)	167

Symmetry codes: (i) *x*−1/2, −*y*+1/2, −*z*; (ii) −*x*+1, *y*+1/2, −*z*+1/2; (iii) −*x*, *y*−1/2, −*z*+1/2; (iv) −*x*+1, *y*−1/2, −*z*+1/2.

Fig. 1


O11
C11
O7
C7
Cr1
C10
O10
O8
C8
C9
O9
O15
C15
O12
C12
C3
C2
P1
C21
C26
C25
O16
Cr2
P4
C16
C5
C6
C22
C23
C24
C14
O14
C31
C13
C32
C36
O13
C33
C35
C34
Cl1
C99
Cl3
Cl2