

[2,2-Bis(diphenylphosphanyl)propane- κ^2P,P']tetracarbonylchromium(0) dichloromethane monosolvate

Normen Peulecke,* Stephan Peitz, Bernd H. Müller, Anke Spannberg and Uwe Rosenthal

Leibniz-Institut für Katalyse e. V. an der Universität Rostock, Albert-Einstein-Strasse 29a, 18059 Rostock, Germany

Correspondence e-mail: normen.peulecke@catalysis.de

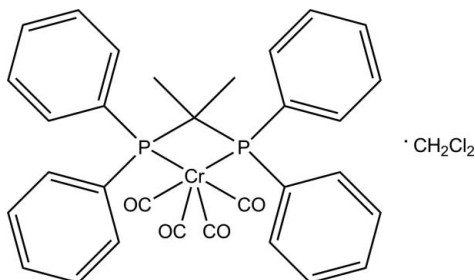
Received 21 October 2010; accepted 26 October 2010

Key indicators: single-crystal X-ray study; $T = 150$ K; mean $\sigma(\text{C}-\text{C}) = 0.003$ Å; R factor = 0.033; wR factor = 0.087; data-to-parameter ratio = 19.0.

The title compound, $[\text{Cr}(\text{C}_{27}\text{H}_{26}\text{P}_2)(\text{CO})_4]\cdot\text{CH}_2\text{Cl}_2$, was obtained by the reaction of $\text{Ph}_2\text{PCMe}_2\text{PPh}_2$ with $\text{Cr}(\text{CO})_6$ in refluxing toluene by substitution of two carbonyl ligands. The CrC_4P_2 coordination geometry at the Cr atom is distorted octahedral, with a $\text{P}-\text{Cr}-\text{P}$ bite angle of $70.27(2)^\circ$.

Related literature

For the original synthesis of $\text{Ph}_2\text{PCMe}_2\text{PPh}_2$, see: Hewertson & Watson (1962). For an alternative synthesis of the title compound, see: Al-Jibori & Shaw (1983). For the synthesis of $\text{Ph}_2\text{PCMe}_2\text{PPh}_2$ and Mo or W carbonyl complexes of related ligands with different substituents at the central carbon, see: Hogarth & Kilmartin (2007). For complexation of $\text{Ph}_2\text{PCMe}_2\text{PPh}_2$ and structural characterization of monomeric complexes of Pd or Ru, see: Barkley *et al.* (1995, 1998); Anandhi *et al.* (2003).



Experimental

Crystal data

| | |
|---|---|
| $[\text{Cr}(\text{C}_{27}\text{H}_{26}\text{P}_2)(\text{CO})_4]\cdot\text{CH}_2\text{Cl}_2$ | $\gamma = 93.020(4)^\circ$ |
| $M_r = 661.38$ | $V = 1532.40(14) \text{ \AA}^3$ |
| Triclinic, $P\bar{1}$ | $Z = 2$ |
| $a = 8.9998(5) \text{ \AA}$ | Mo $K\alpha$ radiation |
| $b = 9.4895(5) \text{ \AA}$ | $\mu = 0.69 \text{ mm}^{-1}$ |
| $c = 18.3178(9) \text{ \AA}$ | $T = 150 \text{ K}$ |
| $\alpha = 99.811(4)^\circ$ | $0.50 \times 0.50 \times 0.27 \text{ mm}$ |
| $\beta = 94.856(4)^\circ$ | |

Data collection

| | |
|---|--|
| Stoe IPDS II diffractometer | 25497 measured reflections |
| Absorption correction: numerical (<i>X-SHAPE</i> and <i>X-RED32</i> ; Stoe & Cie, 2005) | 7051 independent reflections |
| $T_{\min} = 0.700$, $T_{\max} = 0.834$ | 5824 reflections with $I > 2\sigma(I)$ |
| | $R_{\text{int}} = 0.038$ |

Refinement

| | |
|---------------------------------|--|
| $R[F^2 > 2\sigma(F^2)] = 0.033$ | 372 parameters |
| $wR(F^2) = 0.087$ | H-atom parameters constrained |
| $S = 1.06$ | $\Delta\rho_{\text{max}} = 0.71 \text{ e \AA}^{-3}$ |
| 7051 reflections | $\Delta\rho_{\text{min}} = -0.65 \text{ e \AA}^{-3}$ |

Data collection: *X-AREA* (Stoe & Cie, 2005); cell refinement: *X-AREA*; data reduction: *X-AREA*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *XP* in *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *SHELXL97*.

This work was supported by the Leibniz-Institut für Katalyse e. V. an der Universität Rostock.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: CV2782).

References

- Al-Jibori, S. & Shaw, B. L. (1983). *Inorg. Chim. Acta*, **74**, 235–239.
- Anandhi, U., Holbert, T., Lueng, D. & Sharp, P. R. (2003). *Inorg. Chem.* **42**, 1282–1295.
- Barkley, J., Ellis, M., Higgins, S. J. & McCart, M. K. (1998). *Organometallics*, **17**, 1725–1731.
- Barkley, J. V., Grimshaw, J. C., Higgins, S. J., Hoare, P. B., McCart, M. K. & Smith, A. K. (1995). *J. Chem. Soc. Dalton Trans.* pp. 2901–2908.
- Hewertson, W. & Watson, H. R. (1962). *J. Chem. Soc.* 1490–1494.
- Hogarth, G. & Kilmartin, J. (2007). *J. Organomet. Chem.* **692**, 5655–5670.
- Sheldrick, G. M. (2008). *Acta Cryst.* **A64**, 112–122.
- Stoe & Cie (2005). *X-SHAPE*, *X-RED32* and *X-AREA*. Stoe & Cie, Darmstadt, Germany.

supplementary materials

Acta Cryst. (2010). E66, m1495 [doi:10.1107/S1600536810043692]

[2,2-Bis(diphenylphosphanyl)propane- κ^2P,P']tetracarbonylchromium(0) dichloromethane monosolvate

N. Peulecke, S. Peitz, B. H. Müller, A. Spannenberg and U. Rosenthal

Comment

2,2-Bis(diphenylphosphino)propane was first prepared by cleavage of triphenylphosphine with sodium in liquid ammonia and following treatment with 2,2-dichloropropane (Hewertson & Watson, 1962). Most of the small bite-angle diphosphine complexes, of the type $[M(\text{CO})_4\{\text{Ph}_2\text{PC}(\text{R}1\text{R}2)\text{PPh}_2\}]$ ($M = \text{Mo}, \text{W}$; $\text{R}1 = \text{H}, \text{Me}, \text{Et}, \text{Pr}, \text{allyl}$, $\text{R}2 = \text{Me}, \text{allyl}$), have been prepared *via* elaboration of the methylene backbones in $[M(\text{CO})_4(\text{Ph}_2\text{PCH}_2\text{PPh}_2)]$ ($\text{Ph}_2\text{PCH}_2\text{PPh}_2 = \text{dppm}$) as a result of successive deprotonation and alkyl halide addition (Hogarth & Kilmartin, 2007). The above mentioned chromium complex $[\text{Cr}(\text{CO})_4(\text{Ph}_2\text{PCMe}_2\text{PPh}_2)]$ was prepared also by this way, but not structurally characterized yet (Al-Jibori & Shaw, 1983). Molecular structures of monomeric ruthenium (Barkley *et al.*, 1998; Anandhi *et al.*, 2003) and palladium (Barkley *et al.*, 1995) complexes of 2,2-bis(diphenylphosphino)propane are already known.

Here we describe the synthesis of the known chromium complex $\text{C}_{31}\text{H}_{26}\text{CrO}_4\text{P}_2$ by direct reaction of $\text{Ph}_2\text{PCMe}_2\text{PPh}_2$ with $\text{Cr}(\text{CO})_6$. Crystals suitable for X-ray analysis were obtained from dichloromethane/methanol solution. The asymmetric unit contains one complex molecule and additionally one solvent molecule dichloromethane. The chromium center is coordinated by the chelating diphosphine $\text{Ph}_2\text{PCMe}_2\text{PPh}_2$ and four carbonyl ligands in a distorted octahedral geometry. A bite-angle $\text{P}-\text{Cr}-\text{P}$ of $70.27(2)^\circ$ was observed. The $\text{P}-\text{C}-\text{P}$ angle of the complexed ligand is $92.07(7)^\circ$. In the crystal structure, short distance of $3.807(2) \text{ \AA}$ between the centroids of aromatic rings $\text{C}14-\text{C}19$ from the neighbouring molecules suggests an existence of weak $\pi-\pi$ interactions.

Experimental

$\text{Cr}(\text{CO})_6$ (175 mg, 0.8 mmol) was added to a solution of $\text{Ph}_2\text{PCMe}_2\text{PPh}_2$ (309 mg, 0.75 mmol) in 20 ml of toluene and the resulting solution was stirred at reflux temperature for 72 h. Subsequently, the formed yellow solution was cooled down to 0°C and filtered. Toluene was removed in vacuum and the product was extracted with dichloromethane. The major part of dichloromethane was removed and the remaining solution was over-layered with methanol to get crystals of the title compound at -40°C , which are suitable for X-ray crystal structure analysis. The analytical data of the yellow compound correspond with those in the literature.

Refinement

All H atoms were placed in idealized positions with $d(\text{C}-\text{H}) = 0.99$ (CH_2), 0.98 (CH_3) and 0.95 \AA (CH) and refined using a riding model with $U_{\text{iso}}(\text{H})$ fixed at $1.5U_{\text{eq}}(\text{C})$ for CH_3 and $1.2U_{\text{eq}}(\text{C})$ for CH_2 and CH .

Figures

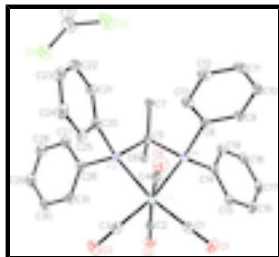


Fig. 1. The molecular structure of the title compound showing the atom-labelling scheme. H atoms are omitted for clarity. Thermal ellipsoids are drawn at the 30% probability level.

[2,2-Bis(diphenylphosphanyl)propane- κ^2P,P']tetracarbonylchromium dichloromethane monosolvate

Crystal data

$[\text{Cr}(\text{C}_{27}\text{H}_{26}\text{P}_2)(\text{CO})_4] \cdot \text{CH}_2\text{Cl}_2$

$M_r = 661.38$

Triclinic, $P\bar{1}$

Hall symbol: -P 1

$a = 8.9998$ (5) Å

$b = 9.4895$ (5) Å

$c = 18.3178$ (9) Å

$\alpha = 99.811$ (4)°

$\beta = 94.856$ (4)°

$\gamma = 93.020$ (4)°

$V = 1532.40$ (14) Å³

$Z = 2$

$F(000) = 680$

$D_x = 1.433$ Mg m⁻³

Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å

Cell parameters from 4481 reflections

$\theta = 2.2$ – 29.6 °

$\mu = 0.69$ mm⁻¹

$T = 150$ K

Prism, yellow

$0.50 \times 0.50 \times 0.27$ mm

Data collection

Stoe IPDS II
diffractometer

Radiation source: fine-focus sealed tube
graphite

ω scans

Absorption correction: numerical
(*X-SHAPE* and *X-RED32*; Stoe & Cie, 2005)

$T_{\min} = 0.700$, $T_{\max} = 0.834$

25497 measured reflections

7051 independent reflections

5824 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.038$

$\theta_{\max} = 27.5$ °, $\theta_{\min} = 2.2$ °

$h = -11 \rightarrow 11$

$k = -12 \rightarrow 12$

$l = -23 \rightarrow 23$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.033$

$wR(F^2) = 0.087$

$S = 1.06$

Primary atom site location: structure-invariant direct methods

Secondary atom site location: difference Fourier map

Hydrogen site location: inferred from neighbouring sites

H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0478P)^2 + 0.3172P]$

7051 reflections
372 parameters
0 restraints

$$\text{where } P = (F_o^2 + 2F_c^2)/3$$

$$(\Delta/\sigma)_{\max} = 0.001$$

$$\Delta\rho_{\max} = 0.71 \text{ e } \text{\AA}^{-3}$$

$$\Delta\rho_{\min} = -0.65 \text{ e } \text{\AA}^{-3}$$

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 (\AA^2)

| | <i>x</i> | <i>y</i> | <i>z</i> | $U_{\text{iso}}^*/U_{\text{eq}}$ |
|-----|--------------|--------------|---------------|----------------------------------|
| C1 | 1.01536 (18) | 0.20775 (19) | 0.18509 (10) | 0.0267 (4) |
| C2 | 0.76174 (18) | 0.13420 (18) | 0.22785 (10) | 0.0248 (3) |
| C3 | 0.96144 (18) | 0.26041 (19) | 0.33115 (10) | 0.0277 (4) |
| C4 | 1.02766 (18) | 0.46144 (19) | 0.25461 (10) | 0.0270 (4) |
| C5 | 0.57483 (17) | 0.44246 (17) | 0.20067 (9) | 0.0216 (3) |
| C6 | 0.45608 (17) | 0.31571 (19) | 0.19234 (10) | 0.0259 (3) |
| H6A | 0.3801 | 0.3405 | 0.2268 | 0.039* |
| H6B | 0.5041 | 0.2307 | 0.2038 | 0.039* |
| H6C | 0.4087 | 0.2955 | 0.1411 | 0.039* |
| C7 | 0.49660 (19) | 0.57332 (19) | 0.18302 (10) | 0.0277 (4) |
| H7A | 0.4490 | 0.5525 | 0.1319 | 0.041* |
| H7B | 0.5702 | 0.6552 | 0.1883 | 0.041* |
| H7C | 0.4204 | 0.5963 | 0.2176 | 0.041* |
| C8 | 0.80565 (17) | 0.54283 (18) | 0.10865 (10) | 0.0241 (3) |
| C9 | 0.81566 (19) | 0.5386 (2) | 0.03282 (11) | 0.0300 (4) |
| H9 | 0.7802 | 0.4547 | -0.0017 | 0.036* |
| C10 | 0.8776 (2) | 0.6569 (2) | 0.00739 (12) | 0.0369 (4) |
| H10 | 0.8841 | 0.6533 | -0.0444 | 0.044* |
| C11 | 0.9293 (2) | 0.7788 (2) | 0.05695 (13) | 0.0381 (5) |
| H11 | 0.9712 | 0.8592 | 0.0393 | 0.046* |
| C12 | 0.9204 (2) | 0.7847 (2) | 0.13238 (13) | 0.0356 (4) |
| H12 | 0.9558 | 0.8691 | 0.1665 | 0.043* |
| C13 | 0.85938 (18) | 0.66646 (19) | 0.15817 (11) | 0.0292 (4) |
| H13 | 0.8544 | 0.6703 | 0.2101 | 0.035* |
| C14 | 0.66313 (18) | 0.26334 (18) | 0.06079 (9) | 0.0240 (3) |
| C15 | 0.73759 (18) | 0.14068 (19) | 0.03805 (10) | 0.0264 (4) |
| H15 | 0.8206 | 0.1186 | 0.0687 | 0.032* |
| C16 | 0.6919 (2) | 0.0507 (2) | -0.02876 (11) | 0.0316 (4) |

supplementary materials

| | | | | |
|------|--------------|--------------|---------------|--------------|
| H16 | 0.7451 | -0.0313 | -0.0443 | 0.038* |
| C17 | 0.5691 (2) | 0.0798 (2) | -0.07279 (10) | 0.0325 (4) |
| H17 | 0.5380 | 0.0180 | -0.1186 | 0.039* |
| C18 | 0.4914 (2) | 0.1991 (2) | -0.05008 (10) | 0.0309 (4) |
| H18 | 0.4053 | 0.2176 | -0.0797 | 0.037* |
| C19 | 0.53874 (19) | 0.2914 (2) | 0.01568 (10) | 0.0282 (4) |
| H19 | 0.4864 | 0.3744 | 0.0303 | 0.034* |
| C20 | 0.74847 (18) | 0.63022 (18) | 0.34037 (9) | 0.0241 (3) |
| C21 | 0.6667 (2) | 0.74999 (19) | 0.33735 (11) | 0.0321 (4) |
| H21 | 0.5747 | 0.7410 | 0.3069 | 0.039* |
| C22 | 0.7189 (3) | 0.8825 (2) | 0.37862 (12) | 0.0403 (5) |
| H22 | 0.6632 | 0.9640 | 0.3755 | 0.048* |
| C23 | 0.8507 (2) | 0.8967 (2) | 0.42396 (12) | 0.0410 (5) |
| H23 | 0.8867 | 0.9880 | 0.4513 | 0.049* |
| C24 | 0.9306 (2) | 0.7781 (2) | 0.42962 (11) | 0.0367 (4) |
| H24 | 1.0197 | 0.7869 | 0.4623 | 0.044* |
| C25 | 0.88029 (19) | 0.6458 (2) | 0.38738 (10) | 0.0289 (4) |
| H25 | 0.9367 | 0.5648 | 0.3906 | 0.035* |
| C26 | 0.56456 (17) | 0.39289 (17) | 0.35684 (9) | 0.0232 (3) |
| C27 | 0.44294 (19) | 0.4714 (2) | 0.37624 (11) | 0.0309 (4) |
| H27 | 0.4247 | 0.5550 | 0.3557 | 0.037* |
| C28 | 0.3486 (2) | 0.4280 (2) | 0.42524 (12) | 0.0377 (4) |
| H28 | 0.2654 | 0.4816 | 0.4380 | 0.045* |
| C29 | 0.3749 (2) | 0.3072 (2) | 0.45557 (11) | 0.0381 (5) |
| H29 | 0.3098 | 0.2776 | 0.4892 | 0.046* |
| C30 | 0.4951 (2) | 0.2296 (2) | 0.43722 (11) | 0.0371 (4) |
| H30 | 0.5133 | 0.1469 | 0.4585 | 0.045* |
| C31 | 0.5902 (2) | 0.27148 (19) | 0.38763 (10) | 0.0293 (4) |
| H31 | 0.6728 | 0.2170 | 0.3748 | 0.035* |
| C32 | 0.2113 (3) | 0.9291 (3) | 0.33113 (16) | 0.0622 (7) |
| H32A | 0.1155 | 0.8720 | 0.3145 | 0.075* |
| H32B | 0.1881 | 1.0281 | 0.3515 | 0.075* |
| Cl1 | 0.30771 (6) | 0.85575 (7) | 0.40154 (4) | 0.05184 (15) |
| Cl2 | 0.31373 (9) | 0.93219 (10) | 0.25513 (5) | 0.0796 (2) |
| Cr1 | 0.88203 (3) | 0.30677 (3) | 0.242280 (15) | 0.02031 (8) |
| O1 | 1.09906 (15) | 0.14688 (15) | 0.14916 (8) | 0.0396 (3) |
| O2 | 0.69755 (15) | 0.02391 (13) | 0.21958 (8) | 0.0353 (3) |
| O3 | 1.00875 (16) | 0.22861 (17) | 0.38596 (8) | 0.0426 (3) |
| O4 | 1.12468 (14) | 0.54764 (15) | 0.26147 (9) | 0.0401 (3) |
| P1 | 0.73655 (4) | 0.38536 (4) | 0.14524 (2) | 0.02048 (10) |
| P2 | 0.69140 (4) | 0.44708 (4) | 0.29237 (2) | 0.01995 (10) |

Atomic displacement parameters (\AA^2)

| | U^{11} | U^{22} | U^{33} | U^{12} | U^{13} | U^{23} |
|----|------------|------------|-------------|-------------|------------|------------|
| C1 | 0.0248 (8) | 0.0285 (9) | 0.0269 (9) | -0.0012 (7) | 0.0000 (7) | 0.0071 (7) |
| C2 | 0.0244 (7) | 0.0281 (9) | 0.0225 (9) | 0.0037 (6) | 0.0024 (6) | 0.0059 (7) |
| C3 | 0.0239 (8) | 0.0286 (9) | 0.0303 (10) | 0.0001 (6) | 0.0025 (7) | 0.0050 (7) |

| | | | | | | |
|-----|--------------|--------------|--------------|---------------|--------------|--------------|
| C4 | 0.0233 (8) | 0.0305 (9) | 0.0274 (9) | 0.0040 (7) | 0.0026 (7) | 0.0052 (7) |
| C5 | 0.0195 (7) | 0.0232 (8) | 0.0223 (8) | -0.0007 (6) | 0.0019 (6) | 0.0055 (6) |
| C6 | 0.0208 (7) | 0.0305 (9) | 0.0255 (9) | -0.0047 (6) | 0.0011 (6) | 0.0045 (7) |
| C7 | 0.0262 (8) | 0.0297 (9) | 0.0282 (9) | 0.0041 (7) | 0.0018 (7) | 0.0083 (7) |
| C8 | 0.0197 (7) | 0.0257 (8) | 0.0293 (9) | 0.0016 (6) | 0.0050 (6) | 0.0107 (7) |
| C9 | 0.0283 (8) | 0.0338 (9) | 0.0312 (10) | 0.0033 (7) | 0.0074 (7) | 0.0120 (8) |
| C10 | 0.0367 (9) | 0.0418 (11) | 0.0396 (11) | 0.0070 (8) | 0.0164 (8) | 0.0207 (9) |
| C11 | 0.0294 (9) | 0.0347 (10) | 0.0577 (14) | 0.0022 (7) | 0.0145 (9) | 0.0242 (10) |
| C12 | 0.0270 (8) | 0.0296 (9) | 0.0516 (13) | -0.0030 (7) | 0.0042 (8) | 0.0128 (9) |
| C13 | 0.0252 (8) | 0.0287 (9) | 0.0347 (10) | -0.0008 (7) | 0.0035 (7) | 0.0095 (8) |
| C14 | 0.0235 (7) | 0.0274 (8) | 0.0216 (8) | -0.0035 (6) | 0.0033 (6) | 0.0067 (7) |
| C15 | 0.0245 (8) | 0.0299 (9) | 0.0248 (9) | -0.0010 (6) | 0.0042 (7) | 0.0046 (7) |
| C16 | 0.0311 (9) | 0.0329 (9) | 0.0296 (10) | -0.0017 (7) | 0.0090 (7) | 0.0004 (8) |
| C17 | 0.0352 (9) | 0.0396 (10) | 0.0202 (9) | -0.0110 (8) | 0.0033 (7) | 0.0017 (8) |
| C18 | 0.0307 (9) | 0.0385 (10) | 0.0234 (9) | -0.0066 (7) | -0.0021 (7) | 0.0101 (8) |
| C19 | 0.0276 (8) | 0.0322 (9) | 0.0255 (9) | -0.0007 (7) | 0.0013 (7) | 0.0084 (7) |
| C20 | 0.0255 (7) | 0.0247 (8) | 0.0226 (8) | -0.0036 (6) | 0.0068 (6) | 0.0049 (7) |
| C21 | 0.0390 (9) | 0.0258 (9) | 0.0310 (10) | 0.0009 (7) | 0.0028 (8) | 0.0038 (8) |
| C22 | 0.0588 (12) | 0.0235 (9) | 0.0390 (12) | 0.0011 (8) | 0.0100 (10) | 0.0046 (8) |
| C23 | 0.0545 (12) | 0.0281 (10) | 0.0363 (11) | -0.0162 (9) | 0.0128 (9) | -0.0043 (8) |
| C24 | 0.0323 (9) | 0.0430 (11) | 0.0294 (10) | -0.0122 (8) | 0.0051 (8) | -0.0055 (8) |
| C25 | 0.0268 (8) | 0.0319 (9) | 0.0259 (9) | -0.0030 (7) | 0.0049 (7) | -0.0002 (7) |
| C26 | 0.0240 (7) | 0.0244 (8) | 0.0204 (8) | -0.0043 (6) | 0.0026 (6) | 0.0037 (7) |
| C27 | 0.0286 (8) | 0.0334 (9) | 0.0323 (10) | 0.0017 (7) | 0.0072 (7) | 0.0086 (8) |
| C28 | 0.0301 (9) | 0.0443 (11) | 0.0390 (11) | -0.0010 (8) | 0.0127 (8) | 0.0047 (9) |
| C29 | 0.0386 (10) | 0.0449 (11) | 0.0314 (10) | -0.0105 (8) | 0.0124 (8) | 0.0083 (9) |
| C30 | 0.0467 (11) | 0.0348 (10) | 0.0326 (11) | -0.0041 (8) | 0.0093 (9) | 0.0136 (8) |
| C31 | 0.0338 (9) | 0.0273 (9) | 0.0281 (10) | -0.0001 (7) | 0.0063 (7) | 0.0075 (7) |
| C32 | 0.0465 (13) | 0.085 (2) | 0.0622 (17) | 0.0223 (13) | 0.0112 (12) | 0.0258 (15) |
| Cl1 | 0.0416 (3) | 0.0617 (4) | 0.0546 (4) | 0.0032 (2) | 0.0064 (2) | 0.0164 (3) |
| Cl2 | 0.0798 (5) | 0.1007 (6) | 0.0752 (5) | 0.0227 (4) | 0.0300 (4) | 0.0472 (5) |
| Cr1 | 0.01840 (12) | 0.02201 (14) | 0.02079 (15) | 0.00010 (9) | 0.00154 (10) | 0.00511 (10) |
| O1 | 0.0335 (7) | 0.0421 (8) | 0.0431 (8) | 0.0087 (6) | 0.0132 (6) | 0.0005 (7) |
| O2 | 0.0371 (7) | 0.0261 (7) | 0.0414 (8) | -0.0056 (5) | 0.0009 (6) | 0.0059 (6) |
| O3 | 0.0417 (8) | 0.0550 (9) | 0.0330 (8) | 0.0023 (6) | -0.0071 (6) | 0.0186 (7) |
| O4 | 0.0281 (6) | 0.0383 (7) | 0.0523 (9) | -0.0088 (6) | 0.0027 (6) | 0.0071 (7) |
| P1 | 0.01951 (18) | 0.0222 (2) | 0.0203 (2) | -0.00082 (15) | 0.00222 (15) | 0.00564 (16) |
| P2 | 0.01915 (18) | 0.0205 (2) | 0.0203 (2) | -0.00140 (14) | 0.00230 (15) | 0.00456 (16) |

Geometric parameters (Å, °)

| | | | |
|--------|-------------|---------|-----------|
| C1—O1 | 1.155 (2) | C16—H16 | 0.9500 |
| C1—Cr1 | 1.8484 (19) | C17—C18 | 1.384 (3) |
| C2—O2 | 1.149 (2) | C17—H17 | 0.9500 |
| C2—Cr1 | 1.8817 (17) | C18—C19 | 1.383 (3) |
| C3—O3 | 1.151 (2) | C18—H18 | 0.9500 |
| C3—Cr1 | 1.8533 (19) | C19—H19 | 0.9500 |
| C4—O4 | 1.148 (2) | C20—C25 | 1.391 (2) |
| C4—Cr1 | 1.8860 (17) | C20—C21 | 1.392 (3) |

supplementary materials

| | | | |
|------------|-------------|-------------|-------------|
| C5—C7 | 1.527 (2) | C20—P2 | 1.8347 (17) |
| C5—C6 | 1.545 (2) | C21—C22 | 1.388 (3) |
| C5—P1 | 1.8943 (16) | C21—H21 | 0.9500 |
| C5—P2 | 1.8963 (17) | C22—C23 | 1.376 (3) |
| C6—H6A | 0.9800 | C22—H22 | 0.9500 |
| C6—H6B | 0.9800 | C23—C24 | 1.381 (3) |
| C6—H6C | 0.9800 | C23—H23 | 0.9500 |
| C7—H7A | 0.9800 | C24—C25 | 1.389 (3) |
| C7—H7B | 0.9800 | C24—H24 | 0.9500 |
| C7—H7C | 0.9800 | C25—H25 | 0.9500 |
| C8—C13 | 1.392 (3) | C26—C31 | 1.388 (2) |
| C8—C9 | 1.393 (3) | C26—C27 | 1.393 (3) |
| C8—P1 | 1.8393 (16) | C26—P2 | 1.8263 (16) |
| C9—C10 | 1.394 (2) | C27—C28 | 1.384 (2) |
| C9—H9 | 0.9500 | C27—H27 | 0.9500 |
| C10—C11 | 1.376 (3) | C28—C29 | 1.380 (3) |
| C10—H10 | 0.9500 | C28—H28 | 0.9500 |
| C11—C12 | 1.383 (3) | C29—C30 | 1.373 (3) |
| C11—H11 | 0.9500 | C29—H29 | 0.9500 |
| C12—C13 | 1.394 (2) | C30—C31 | 1.391 (2) |
| C12—H12 | 0.9500 | C30—H30 | 0.9500 |
| C13—H13 | 0.9500 | C31—H31 | 0.9500 |
| C14—C15 | 1.393 (3) | C32—C12 | 1.737 (3) |
| C14—C19 | 1.399 (2) | C32—C11 | 1.755 (3) |
| C14—P1 | 1.8184 (18) | C32—H32A | 0.9900 |
| C15—C16 | 1.385 (3) | C32—H32B | 0.9900 |
| C15—H15 | 0.9500 | Cr1—P1 | 2.3644 (5) |
| C16—C17 | 1.380 (3) | Cr1—P2 | 2.3767 (5) |
| O1—C1—Cr1 | 179.44 (16) | C23—C22—C21 | 120.44 (19) |
| O2—C2—Cr1 | 175.10 (15) | C23—C22—H22 | 119.8 |
| O3—C3—Cr1 | 178.39 (16) | C21—C22—H22 | 119.8 |
| O4—C4—Cr1 | 174.55 (16) | C22—C23—C24 | 119.97 (18) |
| C7—C5—C6 | 108.25 (13) | C22—C23—H23 | 120.0 |
| C7—C5—P1 | 117.15 (11) | C24—C23—H23 | 120.0 |
| C6—C5—P1 | 109.61 (12) | C23—C24—C25 | 119.77 (19) |
| C7—C5—P2 | 121.62 (12) | C23—C24—H24 | 120.1 |
| C6—C5—P2 | 106.88 (11) | C25—C24—H24 | 120.1 |
| P1—C5—P2 | 92.07 (7) | C24—C25—C20 | 120.90 (18) |
| C5—C6—H6A | 109.5 | C24—C25—H25 | 119.6 |
| C5—C6—H6B | 109.5 | C20—C25—H25 | 119.6 |
| H6A—C6—H6B | 109.5 | C31—C26—C27 | 119.18 (15) |
| C5—C6—H6C | 109.5 | C31—C26—P2 | 119.40 (13) |
| H6A—C6—H6C | 109.5 | C27—C26—P2 | 121.43 (13) |
| H6B—C6—H6C | 109.5 | C28—C27—C26 | 120.27 (17) |
| C5—C7—H7A | 109.5 | C28—C27—H27 | 119.9 |
| C5—C7—H7B | 109.5 | C26—C27—H27 | 119.9 |
| H7A—C7—H7B | 109.5 | C29—C28—C27 | 120.18 (19) |
| C5—C7—H7C | 109.5 | C29—C28—H28 | 119.9 |
| H7A—C7—H7C | 109.5 | C27—C28—H28 | 119.9 |

| | | | |
|-------------|-------------|---------------|-------------|
| H7B—C7—H7C | 109.5 | C30—C29—C28 | 120.01 (17) |
| C13—C8—C9 | 118.82 (15) | C30—C29—H29 | 120.0 |
| C13—C8—P1 | 119.29 (13) | C28—C29—H29 | 120.0 |
| C9—C8—P1 | 121.74 (14) | C29—C30—C31 | 120.39 (18) |
| C8—C9—C10 | 120.26 (19) | C29—C30—H30 | 119.8 |
| C8—C9—H9 | 119.9 | C31—C30—H30 | 119.8 |
| C10—C9—H9 | 119.9 | C26—C31—C30 | 119.97 (18) |
| C11—C10—C9 | 120.31 (19) | C26—C31—H31 | 120.0 |
| C11—C10—H10 | 119.8 | C30—C31—H31 | 120.0 |
| C9—C10—H10 | 119.8 | Cl2—C32—Cl1 | 112.25 (14) |
| C10—C11—C12 | 120.14 (17) | Cl2—C32—H32A | 109.2 |
| C10—C11—H11 | 119.9 | Cl1—C32—H32A | 109.2 |
| C12—C11—H11 | 119.9 | Cl2—C32—H32B | 109.2 |
| C11—C12—C13 | 119.81 (19) | Cl1—C32—H32B | 109.2 |
| C11—C12—H12 | 120.1 | H32A—C32—H32B | 107.9 |
| C13—C12—H12 | 120.1 | C1—Cr1—C3 | 94.87 (8) |
| C8—C13—C12 | 120.66 (18) | C1—Cr1—C2 | 87.32 (7) |
| C8—C13—H13 | 119.7 | C3—Cr1—C2 | 87.49 (7) |
| C12—C13—H13 | 119.7 | C1—Cr1—C4 | 84.72 (7) |
| C15—C14—C19 | 118.54 (16) | C3—Cr1—C4 | 89.19 (7) |
| C15—C14—P1 | 118.92 (13) | C2—Cr1—C4 | 171.09 (7) |
| C19—C14—P1 | 122.46 (14) | C1—Cr1—P1 | 98.01 (6) |
| C16—C15—C14 | 120.68 (17) | C3—Cr1—P1 | 166.91 (6) |
| C16—C15—H15 | 119.7 | C2—Cr1—P1 | 90.76 (5) |
| C14—C15—H15 | 119.7 | C4—Cr1—P1 | 94.31 (5) |
| C17—C16—C15 | 120.13 (18) | C1—Cr1—P2 | 168.24 (6) |
| C17—C16—H16 | 119.9 | C3—Cr1—P2 | 96.88 (6) |
| C15—C16—H16 | 119.9 | C2—Cr1—P2 | 93.54 (5) |
| C16—C17—C18 | 119.94 (17) | C4—Cr1—P2 | 95.07 (5) |
| C16—C17—H17 | 120.0 | P1—Cr1—P2 | 70.265 (16) |
| C18—C17—H17 | 120.0 | C14—P1—C8 | 102.23 (8) |
| C19—C18—C17 | 120.19 (17) | C14—P1—C5 | 108.57 (7) |
| C19—C18—H18 | 119.9 | C8—P1—C5 | 106.88 (7) |
| C17—C18—H18 | 119.9 | C14—P1—Cr1 | 121.91 (6) |
| C18—C19—C14 | 120.47 (17) | C8—P1—Cr1 | 119.29 (6) |
| C18—C19—H19 | 119.8 | C5—P1—Cr1 | 96.77 (5) |
| C14—C19—H19 | 119.8 | C26—P2—C20 | 99.66 (7) |
| C25—C20—C21 | 118.50 (16) | C26—P2—C5 | 106.62 (7) |
| C25—C20—P2 | 115.71 (13) | C20—P2—C5 | 112.64 (7) |
| C21—C20—P2 | 125.64 (13) | C26—P2—Cr1 | 124.58 (6) |
| C22—C21—C20 | 120.37 (18) | C20—P2—Cr1 | 116.97 (5) |
| C22—C21—H21 | 119.8 | C5—P2—Cr1 | 96.31 (5) |
| C20—C21—H21 | 119.8 | | |

Fig. 1

