

## Dichloridotris(trimethylphosphine)-nickel(II)

Ruixia Cao, Qibao Wang and Hongjian Sun\*

School of Chemistry and Chemical Engineering, Shandong University, Shanda Nanlu 27, Jinan 250100, People's Republic of China

Correspondence e-mail: hjsun@sdu.edu.cn

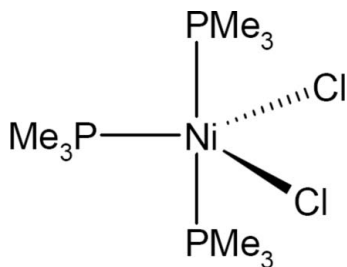
Received 21 November 2007; accepted 4 January 2008

Key indicators: single-crystal X-ray study;  $T = 293$  K; mean  $\sigma(\text{P}-\text{C}) = 0.003$  Å;  $R$  factor = 0.030;  $wR$  factor = 0.081; data-to-parameter ratio = 26.1.

The title compound,  $[\text{NiCl}_2(\text{C}_3\text{H}_9\text{P})_3]$ , was obtained as a product of the reaction of  $[\text{NiCl}_2(\text{PMe}_3)_2]$  with an equivalent trimethylphosphine in diethyl ether. It easily loses trimethylphosphine at room temperature to give  $\text{NiCl}_2(\text{PMe}_3)_2$ . There are two independent molecules in the asymmetric unit, and their bond lengths and angles are similar. The Ni environment is trigonal bipyramidal. One Ni, one P and two Cl atoms lie in the equatorial plane, with the remaining two P atoms occupying axial positions. The equatorial Ni—P bond length is shorter than the axial bond lengths.

### Related literature

The crystal structure of the related cobalt compound has been reported by Jiao *et al.* (2007). For related literature, see: Doriand & Gray (1966).



### Experimental

#### Crystal data

$[\text{NiCl}_2(\text{C}_3\text{H}_9\text{P})_3]$	$V = 3470.6$ (12) Å <sup>3</sup>
$M_r = 357.83$	$Z = 8$
Orthorhombic, $P2_12_12_1$	Mo $K\alpha$ radiation
$a = 10.481$ (2) Å	$\mu = 1.68$ mm <sup>-1</sup>
$b = 11.741$ (2) Å	$T = 293$ (2) K
$c = 28.203$ (6) Å	$0.30 \times 0.25 \times 0.22$ mm

#### Data collection

Bruker $P4$ diffractometer	7309 reflections with $I > 2\sigma(I)$
Absorption correction: multi-scan (SADABS; Sheldrick, 2004)	$R_{\text{int}} = 0.073$
$T_{\text{min}} = 0.633$ , $T_{\text{max}} = 0.709$	4 standard reflections every 50 reflections
26702 measured reflections	intensity decay: 0.02%
7557 independent reflections	

#### Refinement

$R[F^2 > 2\sigma(F^2)] = 0.030$	$\Delta\rho_{\text{max}} = 0.63$ e Å <sup>-3</sup>
$wR(F^2) = 0.081$	$\Delta\rho_{\text{min}} = -0.84$ e Å <sup>-3</sup>
$S = 1.06$	Absolute structure: Flack (1983), 3276 Friedel pairs
7557 reflections	Flack parameter: 0.006 (8)
289 parameters	
H-atom parameters constrained	

Table 1

Selected bond lengths (Å).

Ni1—P3	2.1754 (7)	Ni1—Cl1	2.2901 (7)
Ni1—P1	2.2051 (8)	Ni1—Cl2	2.4369 (8)
Ni1—P2	2.2088 (8)		

Data collection: XSCANS (Bruker, 2001); cell refinement: XSCANS; data reduction: SHELXTL (Sheldrick, 2001); program(s) used to solve structure: SHELXS97 (Sheldrick, 2008); program(s) used to refine structure: SHELXL97 (Sheldrick, 2008); molecular graphics: SHELXTL; software used to prepare material for publication: SHELXTL.

This work was supported by the NSFC (grant Nos. 20572062 and 20372042).

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BR2064).

### References

- Bruker (2001). XSCANS. Bruker AXS Inc., Madison, Wisconsin, USA.  
 Doriand, Z. & Gray, H. B. (1966). *J. Am. Chem. Soc.* **88**, 1394–1398.  
 Flack, H. D. (1983). *Acta Cryst.* **A39**, 876–881.  
 Jiao, G. L., Li, X. Y., Sun, H. J. & Xu, X. F. (2007). *J. Organomet. Chem.* **692**, 4251–4258.  
 Sheldrick, G. M. (2001). SHELXTL. Version 5.0. Bruker AXS Inc., Madison, Wisconsin, USA.  
 Sheldrick, G. M. (2004). SADABS. University of Göttingen, Germany.  
 Sheldrick, G. M. (2008). *Acta Cryst.* **A64**, 112–122.

**supplementary materials**

*Acta Cryst.* (2008). E64, m335 [ doi:10.1107/S1600536808000317 ]

## Dichloridotris(trimethylphosphine)nickel(II)

R. Cao, Q. Wang and H. Sun

### Comment

The title molecular (Fig.1) contains two molecules in an asymmetric unit. Bond lengths and angles in the two molecules are approximately the same. The nickel atom lies in the center of a trigonal bipyramid in which two chlorine atoms and one P atom form an equatorial plane, two other phosphorus are arranged in axial positions. The equatorial Ni—P bond length is shorter than the axial ones. The nickel atoms do not lie on a straight line joining the apical phosphorus atoms. This is due to the apical groups experiencing greater repulsion from the equatorial  $\text{PMe}_3$  groups. Similar crystal structures have been reported in the literature *e.g.* tris(trimethylphosphine)diiodocobalt(II) (Jiao *et al.*(2007)). The lengths of Co—I (2.6) is longer than the range of Ni—Cl bond lengths, angle in the axial position of this compound ( $169.8^\circ$ ) is a little bigger than that in the title compound ( $167.3$ – $168.0^\circ$ ).

### Experimental

Standard vacuum techniques were used in manipulations of volatile and air sensitive material. Literature procedure was followed in the preparation of dichlorodi(trimethylphosphine)nickel (Doriand *et al.*(1966)). Other chemicals were used by purchased. To the solution of  $\text{NiCl}_2(\text{PMe}_3)_2$  (1.0 g, 3.55 mmol) in 50 ml of diethyl ether was added trimethylphosphine (0.27 g, 3.55 mmol) at 193 K. This mixture was allowed to warm to 293 K and stirred for 6 h to form a dark blue solution, which was filtered. Crystallization from ether at 277 K afforded dark blue crystals suitable for X-ray diffraction analysis. (yield: 1.14 g, 89.8%, m. p: 397.15 K).

### Refinement

All H atoms were positioned geometrically. All the H atoms are refined using a riding model with C—H = 0.96 Å and with  $U_{\text{iso}}(\text{H}) = 1.5$  times  $U_{\text{eq}}(\text{C})$ .

### Figures

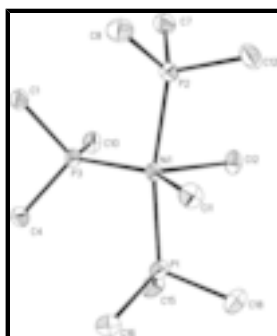


Fig. 1. The molecular structure of (I), with atom labels and 30% probability displacement ellipsoids for non-H atoms. (Only one of the two molecules is shown.)

## Dichloridotris(trimethylphosphine)nickel(II)

### Crystal data

$[\text{NiCl}_2(\text{C}_3\text{H}_9\text{P})_3]$	$F_{000} = 1504$
$M_r = 357.83$	$D_x = 1.370 \text{ Mg m}^{-3}$
Orthorhombic, $P2_12_12_1$	Mo $K\alpha$ radiation
Hall symbol: P 2ac 2ab	$\lambda = 0.71073 \text{ \AA}$
$a = 10.481 (2) \text{ \AA}$	Cell parameters from 6412 reflections
$b = 11.741 (2) \text{ \AA}$	$\theta = 1.6\text{--}24.2^\circ$
$c = 28.203 (6) \text{ \AA}$	$\mu = 1.68 \text{ mm}^{-1}$
$V = 3470.6 (12) \text{ \AA}^3$	$T = 293 (2) \text{ K}$
$Z = 8$	Block, blue
	$0.30 \times 0.25 \times 0.22 \text{ mm}$

### Data collection

Bruker P4 diffractometer	$R_{\text{int}} = 0.073$
Radiation source: fine-focus sealed tube	$\theta_{\text{max}} = 27.1^\circ$
Monochromator: graphite	$\theta_{\text{min}} = 1.4^\circ$
$T = 293(2) \text{ K}$	$h = -13 \rightarrow 13$
$\omega$ scans	$k = -15 \rightarrow 14$
Absorption correction: multi-scan (SADABS; Sheldrick, 2004)	$l = -35 \rightarrow 36$
$T_{\text{min}} = 0.633$ , $T_{\text{max}} = 0.709$	4 standard reflections
26702 measured reflections	every 50 reflections
7557 independent reflections	intensity decay: 0.02%
7309 reflections with $I > 2\sigma(I)$	

### Refinement

Refinement on $F^2$	Hydrogen site location: inferred from neighbouring sites
Least-squares matrix: full	H-atom parameters constrained
$R[F^2 > 2\sigma(F^2)] = 0.030$	$w = 1/[\sigma^2(F_o^2) + (0.0528P)^2]$
$wR(F^2) = 0.081$	where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.06$	$(\Delta/\sigma)_{\text{max}} = 0.002$
7557 reflections	$\Delta\rho_{\text{max}} = 0.63 \text{ e \AA}^{-3}$
289 parameters	$\Delta\rho_{\text{min}} = -0.84 \text{ e \AA}^{-3}$
Primary atom site location: structure-invariant direct methods	Extinction correction: none
Secondary atom site location: difference Fourier map	Absolute structure: Flack (1983)
	Flack parameter: 0.006 (8)

*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  $F^2 > 2\sigma(F^2)$  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 ( $\text{\AA}^2$ )*

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
Ni1	0.72334 (3)	0.45758 (2)	0.757236 (10)	0.02415 (8)
Ni2	0.89293 (3)	-0.01317 (2)	0.980401 (10)	0.02370 (7)
P3	0.78376 (6)	0.52513 (5)	0.68901 (2)	0.02503 (12)
Cl1	0.59423 (6)	0.50132 (6)	0.82016 (2)	0.03563 (14)
P6	0.82135 (6)	0.04616 (5)	1.04857 (2)	0.02697 (13)
Cl4	0.83645 (6)	-0.15585 (5)	0.92899 (2)	0.03198 (13)
P5	0.77420 (6)	0.10238 (5)	0.93714 (2)	0.02798 (13)
Cl3	1.07328 (6)	0.10289 (5)	0.95390 (2)	0.03036 (12)
P2	0.88341 (7)	0.53231 (6)	0.79757 (2)	0.03072 (13)
P4	1.03877 (6)	-0.12977 (5)	1.00949 (2)	0.02620 (13)
Cl2	0.82969 (7)	0.27500 (5)	0.76850 (2)	0.03829 (15)
P1	0.56145 (7)	0.35783 (5)	0.72950 (2)	0.03075 (14)
C1	0.8573 (3)	0.6661 (2)	0.68588 (9)	0.0321 (5)
H1A	0.9325	0.6674	0.7053	0.048*
H1B	0.7980	0.7223	0.6971	0.048*
H1C	0.8799	0.6827	0.6536	0.048*
C2	0.6552 (3)	0.0922 (3)	1.05323 (11)	0.0411 (6)
H2A	0.6000	0.0318	1.0430	0.062*
H2B	0.6420	0.1579	1.0336	0.062*
H2C	0.6363	0.1111	1.0856	0.062*
C3	0.9884 (3)	-0.2639 (2)	1.03651 (11)	0.0374 (6)
H3A	1.0623	-0.3070	1.0456	0.056*
H3B	0.9390	-0.3068	1.0141	0.056*
H3C	0.9375	-0.2482	1.0640	0.056*
C4	0.6659 (3)	0.5473 (2)	0.64191 (9)	0.0343 (5)
H4A	0.7085	0.5718	0.6135	0.051*
H4B	0.6059	0.6045	0.6516	0.051*
H4C	0.6216	0.4772	0.6358	0.051*
C5	1.1530 (3)	-0.0718 (2)	1.05139 (9)	0.0343 (5)
H5A	1.2195	-0.1264	1.0568	0.051*
H5B	1.1106	-0.0552	1.0808	0.051*
H5C	1.1891	-0.0031	1.0387	0.051*
C6	0.7796 (3)	0.2542 (2)	0.95028 (10)	0.0379 (6)

## supplementary materials

---

H6A	0.8669	0.2787	0.9520	0.057*
H6B	0.7383	0.2683	0.9801	0.057*
H6C	0.7366	0.2956	0.9257	0.057*
C7	1.0461 (3)	0.5101 (3)	0.77589 (11)	0.0422 (6)
H7A	1.1057	0.5418	0.7981	0.063*
H7B	1.0560	0.5470	0.7457	0.063*
H7C	1.0618	0.4300	0.7724	0.063*
C8	0.8737 (4)	0.6837 (2)	0.81107 (11)	0.0452 (7)
H8A	0.7882	0.7024	0.8206	0.068*
H8B	0.8960	0.7270	0.7834	0.068*
H8C	0.9316	0.7016	0.8364	0.068*
C9	0.6053 (3)	0.0687 (2)	0.93156 (12)	0.0410 (6)
H9A	0.5664	0.1200	0.9093	0.061*
H9B	0.5647	0.0766	0.9619	0.061*
H9C	0.5958	-0.0081	0.9205	0.061*
C10	0.8988 (3)	0.4322 (2)	0.66093 (9)	0.0348 (6)
H10A	0.8632	0.3573	0.6579	0.052*
H10B	0.9747	0.4288	0.6799	0.052*
H10C	0.9196	0.4614	0.6301	0.052*
C11	1.1422 (3)	-0.1830 (2)	0.96282 (10)	0.0374 (6)
H11A	1.1893	-0.1209	0.9493	0.056*
H11B	1.0914	-0.2185	0.9387	0.056*
H11C	1.2005	-0.2378	0.9758	0.056*
C12	0.8933 (3)	0.4679 (3)	0.85625 (10)	0.0460 (7)
H12A	0.9048	0.3871	0.8531	0.069*
H12B	0.8159	0.4829	0.8734	0.069*
H12C	0.9643	0.4998	0.8732	0.069*
C13	0.8233 (3)	-0.0500 (2)	1.09934 (9)	0.0375 (6)
H13A	0.7871	-0.0123	1.1264	0.056*
H13B	0.9097	-0.0715	1.1063	0.056*
H13C	0.7743	-0.1168	1.0921	0.056*
C14	0.8227 (3)	0.1000 (3)	0.87549 (10)	0.0397 (6)
H14A	0.8126	0.0243	0.8631	0.060*
H14B	0.9105	0.1224	0.8731	0.060*
H14C	0.7706	0.1518	0.8577	0.060*
C15	0.5937 (4)	0.2620 (3)	0.67975 (11)	0.0461 (7)
H15A	0.6638	0.2130	0.6876	0.069*
H15B	0.6148	0.3060	0.6522	0.069*
H15C	0.5193	0.2168	0.6734	0.069*
C16	0.4163 (3)	0.4338 (3)	0.71333 (12)	0.0464 (7)
H16A	0.3517	0.3801	0.7043	0.070*
H16B	0.4337	0.4839	0.6872	0.070*
H16C	0.3869	0.4775	0.7400	0.070*
C17	0.9083 (3)	0.1690 (3)	1.07128 (12)	0.0450 (7)
H17A	0.8740	0.1905	1.1015	0.067*
H17B	0.8998	0.2314	1.0495	0.067*
H17C	0.9968	0.1498	1.0747	0.067*
C18	0.5033 (3)	0.2572 (3)	0.77371 (11)	0.0445 (7)
H18A	0.4602	0.2979	0.7985	0.067*

H18B	0.5740	0.2159	0.7869	0.067*
H18C	0.4451	0.2048	0.7590	0.067*

*Atomic displacement parameters ( $\text{\AA}^2$ )*

	$U^{11}$	$U^{22}$	$U^{33}$	$U^{12}$	$U^{13}$	$U^{23}$
Ni1	0.02733 (15)	0.02569 (14)	0.01943 (13)	-0.00089 (11)	0.00117 (11)	0.00090 (10)
Ni2	0.02460 (15)	0.02604 (14)	0.02045 (13)	0.00344 (11)	-0.00030 (10)	0.00021 (10)
P3	0.0271 (3)	0.0272 (3)	0.0208 (2)	0.0007 (2)	0.0019 (2)	0.0027 (2)
Cl1	0.0369 (3)	0.0433 (3)	0.0267 (3)	0.0036 (3)	0.0070 (2)	-0.0058 (2)
P6	0.0296 (3)	0.0274 (3)	0.0238 (3)	0.0001 (2)	0.0045 (2)	-0.0017 (2)
Cl4	0.0346 (3)	0.0306 (3)	0.0308 (3)	-0.0030 (2)	-0.0036 (2)	-0.0047 (2)
P5	0.0269 (3)	0.0291 (3)	0.0279 (3)	0.0028 (3)	-0.0007 (2)	0.0050 (2)
Cl3	0.0298 (3)	0.0325 (3)	0.0288 (3)	-0.0044 (2)	0.0025 (2)	-0.0002 (2)
P2	0.0312 (3)	0.0346 (3)	0.0263 (3)	-0.0017 (3)	-0.0050 (2)	0.0017 (2)
P4	0.0255 (3)	0.0279 (3)	0.0252 (3)	0.0038 (2)	-0.0017 (2)	-0.0002 (2)
Cl2	0.0429 (4)	0.0313 (3)	0.0407 (3)	0.0099 (3)	0.0048 (3)	0.0083 (2)
P1	0.0335 (4)	0.0305 (3)	0.0283 (3)	-0.0073 (3)	0.0022 (3)	-0.0017 (2)
C1	0.0317 (14)	0.0327 (12)	0.0318 (12)	0.0001 (10)	0.0021 (10)	0.0063 (10)
C2	0.0389 (15)	0.0440 (15)	0.0403 (14)	0.0060 (13)	0.0135 (12)	0.0076 (12)
C3	0.0345 (14)	0.0347 (13)	0.0429 (15)	0.0016 (11)	-0.0011 (11)	0.0072 (11)
C4	0.0361 (14)	0.0391 (13)	0.0277 (11)	-0.0002 (11)	-0.0042 (10)	0.0042 (10)
C5	0.0309 (13)	0.0391 (13)	0.0330 (12)	0.0022 (11)	-0.0040 (10)	-0.0035 (10)
C6	0.0403 (15)	0.0314 (12)	0.0420 (14)	0.0032 (11)	0.0055 (12)	0.0074 (10)
C7	0.0335 (14)	0.0498 (16)	0.0433 (15)	-0.0002 (13)	-0.0032 (12)	0.0053 (13)
C8	0.0547 (19)	0.0398 (14)	0.0411 (15)	-0.0059 (13)	-0.0055 (14)	-0.0109 (12)
C9	0.0338 (15)	0.0399 (14)	0.0493 (16)	0.0014 (12)	-0.0068 (12)	0.0076 (12)
C10	0.0403 (15)	0.0352 (13)	0.0290 (12)	0.0045 (11)	0.0073 (11)	0.0004 (9)
C11	0.0325 (14)	0.0413 (14)	0.0383 (13)	0.0090 (11)	-0.0002 (11)	-0.0041 (11)
C12	0.0442 (17)	0.0624 (18)	0.0315 (13)	0.0027 (15)	-0.0078 (12)	0.0088 (13)
C13	0.0430 (16)	0.0403 (13)	0.0292 (12)	0.0020 (12)	0.0013 (11)	0.0028 (10)
C14	0.0417 (16)	0.0492 (15)	0.0282 (12)	0.0030 (13)	-0.0016 (11)	0.0059 (11)
C15	0.061 (2)	0.0377 (14)	0.0394 (15)	-0.0137 (14)	0.0029 (14)	-0.0092 (12)
C16	0.0386 (17)	0.0482 (16)	0.0525 (18)	-0.0079 (13)	-0.0049 (13)	0.0011 (13)
C17	0.0518 (18)	0.0394 (14)	0.0438 (15)	-0.0082 (13)	0.0086 (14)	-0.0130 (12)
C18	0.0526 (19)	0.0380 (14)	0.0429 (16)	-0.0113 (13)	0.0106 (14)	0.0021 (12)

*Geometric parameters ( $\text{\AA}$ ,  $^\circ$ )*

Ni1—P3	2.1754 (7)	C5—H5B	0.9600
Ni1—P1	2.2051 (8)	C5—H5C	0.9600
Ni1—P2	2.2088 (8)	C6—H6A	0.9600
Ni1—Cl1	2.2901 (7)	C6—H6B	0.9600
Ni1—Cl2	2.4369 (8)	C6—H6C	0.9600
Ni2—P6	2.1781 (7)	C7—H7A	0.9600
Ni2—P5	2.2086 (7)	C7—H7B	0.9600
Ni2—P4	2.2099 (7)	C7—H7C	0.9600
Ni2—Cl4	2.2932 (7)	C8—H8A	0.9600
Ni2—Cl3	2.4472 (7)	C8—H8B	0.9600

## supplementary materials

---

P3—C10	1.808 (3)	C8—H8C	0.9600
P3—C1	1.828 (3)	C9—H9A	0.9600
P3—C4	1.832 (3)	C9—H9B	0.9600
P6—C17	1.822 (3)	C9—H9C	0.9600
P6—C13	1.823 (3)	C10—H10A	0.9600
P6—C2	1.828 (3)	C10—H10B	0.9600
P5—C14	1.812 (3)	C10—H10C	0.9600
P5—C9	1.820 (3)	C11—H11A	0.9600
P5—C6	1.822 (3)	C11—H11B	0.9600
P2—C8	1.821 (3)	C11—H11C	0.9600
P2—C12	1.822 (3)	C12—H12A	0.9600
P2—C7	1.830 (3)	C12—H12B	0.9600
P4—C5	1.814 (3)	C12—H12C	0.9600
P4—C11	1.816 (3)	C13—H13A	0.9600
P4—C3	1.827 (3)	C13—H13B	0.9600
P1—C16	1.822 (4)	C13—H13C	0.9600
P1—C18	1.823 (3)	C14—H14A	0.9600
P1—C15	1.830 (3)	C14—H14B	0.9600
C1—H1A	0.9600	C14—H14C	0.9600
C1—H1B	0.9600	C15—H15A	0.9600
C1—H1C	0.9600	C15—H15B	0.9600
C2—H2A	0.9600	C15—H15C	0.9600
C2—H2B	0.9600	C16—H16A	0.9600
C2—H2C	0.9600	C16—H16B	0.9600
C3—H3A	0.9600	C16—H16C	0.9600
C3—H3B	0.9600	C17—H17A	0.9600
C3—H3C	0.9600	C17—H17B	0.9600
C4—H4A	0.9600	C17—H17C	0.9600
C4—H4B	0.9600	C18—H18A	0.9600
C4—H4C	0.9600	C18—H18B	0.9600
C5—H5A	0.9600	C18—H18C	0.9600
P3—Ni1—P1	95.96 (3)	H5A—C5—H5B	109.5
P3—Ni1—P2	95.14 (3)	P4—C5—H5C	109.5
P1—Ni1—P2	168.00 (3)	H5A—C5—H5C	109.5
P3—Ni1—Cl1	140.87 (3)	H5B—C5—H5C	109.5
P1—Ni1—Cl1	86.52 (3)	P5—C6—H6A	109.5
P2—Ni1—Cl1	87.75 (3)	P5—C6—H6B	109.5
P3—Ni1—Cl2	107.63 (3)	H6A—C6—H6B	109.5
P1—Ni1—Cl2	86.05 (3)	P5—C6—H6C	109.5
P2—Ni1—Cl2	86.26 (3)	H6A—C6—H6C	109.5
Cl1—Ni1—Cl2	111.50 (3)	H6B—C6—H6C	109.5
P6—Ni2—P5	95.57 (3)	P2—C7—H7A	109.5
P6—Ni2—P4	96.24 (3)	P2—C7—H7B	109.5
P5—Ni2—P4	167.25 (3)	H7A—C7—H7B	109.5
P6—Ni2—Cl4	134.65 (3)	P2—C7—H7C	109.5
P5—Ni2—Cl4	87.36 (3)	H7A—C7—H7C	109.5
P4—Ni2—Cl4	87.75 (3)	H7B—C7—H7C	109.5
P6—Ni2—Cl3	110.95 (3)	P2—C8—H8A	109.5
P5—Ni2—Cl3	85.67 (3)	P2—C8—H8B	109.5



P4—Ni2—C13	85.64 (3)	H8A—C8—H8B	109.5
C14—Ni2—C13	114.40 (3)	P2—C8—H8C	109.5
C10—P3—C1	104.12 (13)	H8A—C8—H8C	109.5
C10—P3—C4	102.56 (13)	H8B—C8—H8C	109.5
C1—P3—C4	96.91 (13)	P5—C9—H9A	109.5
C10—P3—Ni1	111.20 (9)	P5—C9—H9B	109.5
C1—P3—Ni1	119.71 (9)	H9A—C9—H9B	109.5
C4—P3—Ni1	119.79 (10)	P5—C9—H9C	109.5
C17—P6—C13	102.02 (15)	H9A—C9—H9C	109.5
C17—P6—C2	102.53 (15)	H9B—C9—H9C	109.5
C13—P6—C2	97.91 (14)	P3—C10—H10A	109.5
C17—P6—Ni2	113.03 (11)	P3—C10—H10B	109.5
C13—P6—Ni2	119.42 (10)	H10A—C10—H10B	109.5
C2—P6—Ni2	119.09 (11)	P3—C10—H10C	109.5
C14—P5—C9	100.74 (15)	H10A—C10—H10C	109.5
C14—P5—C6	101.65 (14)	H10B—C10—H10C	109.5
C9—P5—C6	105.08 (14)	P4—C11—H11A	109.5
C14—P5—Ni2	111.26 (10)	P4—C11—H11B	109.5
C9—P5—Ni2	117.54 (10)	H11A—C11—H11B	109.5
C6—P5—Ni2	118.11 (10)	P4—C11—H11C	109.5
C8—P2—C12	102.61 (16)	H11A—C11—H11C	109.5
C8—P2—C7	105.13 (16)	H11B—C11—H11C	109.5
C12—P2—C7	101.05 (15)	P2—C12—H12A	109.5
C8—P2—Ni1	116.93 (12)	P2—C12—H12B	109.5
C12—P2—Ni1	110.24 (12)	H12A—C12—H12B	109.5
C7—P2—Ni1	118.62 (10)	P2—C12—H12C	109.5
C5—P4—C11	101.98 (14)	H12A—C12—H12C	109.5
C5—P4—C3	104.01 (14)	H12B—C12—H12C	109.5
C11—P4—C3	100.25 (14)	P6—C13—H13A	109.5
C5—P4—Ni2	117.76 (9)	P6—C13—H13B	109.5
C11—P4—Ni2	110.91 (10)	H13A—C13—H13B	109.5
C3—P4—Ni2	119.26 (10)	P6—C13—H13C	109.5
C16—P1—C18	102.08 (17)	H13A—C13—H13C	109.5
C16—P1—C15	105.26 (17)	H13B—C13—H13C	109.5
C18—P1—C15	100.82 (14)	P5—C14—H14A	109.5
C16—P1—Ni1	118.12 (11)	P5—C14—H14B	109.5
C18—P1—Ni1	111.04 (12)	H14A—C14—H14B	109.5
C15—P1—Ni1	117.16 (12)	P5—C14—H14C	109.5
P3—C1—H1A	109.5	H14A—C14—H14C	109.5
P3—C1—H1B	109.5	H14B—C14—H14C	109.5
H1A—C1—H1B	109.5	P1—C15—H15A	109.5
P3—C1—H1C	109.5	P1—C15—H15B	109.5
H1A—C1—H1C	109.5	H15A—C15—H15B	109.5
H1B—C1—H1C	109.5	P1—C15—H15C	109.5
P6—C2—H2A	109.5	H15A—C15—H15C	109.5
P6—C2—H2B	109.5	H15B—C15—H15C	109.5
H2A—C2—H2B	109.5	P1—C16—H16A	109.5
P6—C2—H2C	109.5	P1—C16—H16B	109.5
H2A—C2—H2C	109.5	H16A—C16—H16B	109.5

## supplementary materials

---

H2B—C2—H2C	109.5	P1—C16—H16C	109.5
P4—C3—H3A	109.5	H16A—C16—H16C	109.5
P4—C3—H3B	109.5	H16B—C16—H16C	109.5
H3A—C3—H3B	109.5	P6—C17—H17A	109.5
P4—C3—H3C	109.5	P6—C17—H17B	109.5
H3A—C3—H3C	109.5	H17A—C17—H17B	109.5
H3B—C3—H3C	109.5	P6—C17—H17C	109.5
P3—C4—H4A	109.5	H17A—C17—H17C	109.5
P3—C4—H4B	109.5	H17B—C17—H17C	109.5
H4A—C4—H4B	109.5	P1—C18—H18A	109.5
P3—C4—H4C	109.5	P1—C18—H18B	109.5
H4A—C4—H4C	109.5	H18A—C18—H18B	109.5
H4B—C4—H4C	109.5	P1—C18—H18C	109.5
P4—C5—H5A	109.5	H18A—C18—H18C	109.5
P4—C5—H5B	109.5	H18B—C18—H18C	109.5

Fig. 1

