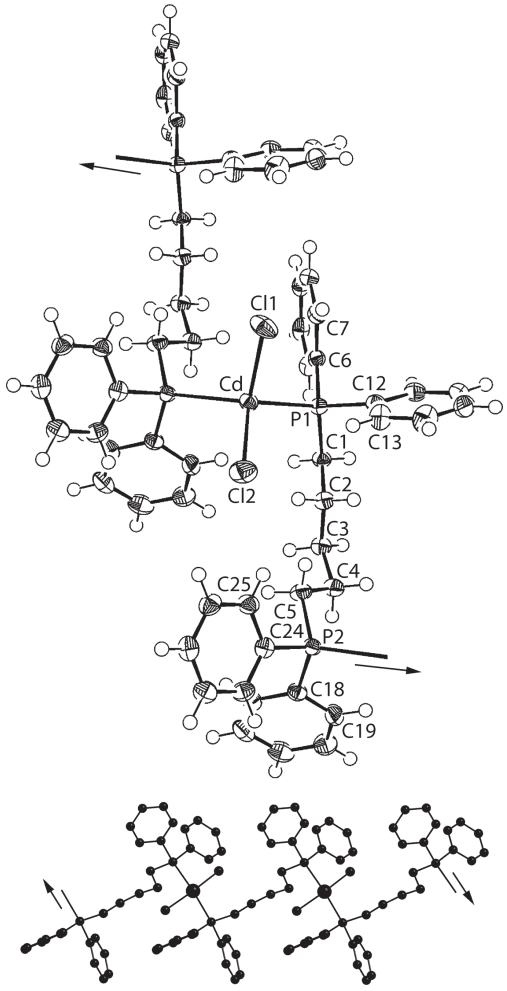
Yee Seng Tan and Edward R.T. Tiekink*

# Crystal structure of *catena*-poly[{$\mu_2$-1,5-bis(diphenylphosphanyl)pentane-$\kappa^2$*P*:*P*′}dichloridocadmium(II)], $C_{29}H_{30}CdCl_2P_2$

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***Corresponding author: Edward R.T. Tiekink,** Research Centre for Crystalline Materials, School of Science and Technology, Sunway University, 47500 Bandar Sunway, Selangor Darul Ehsan, Malaysia, e-mail: edwardt@sunway.edu.my. https://orcid.org/0000-0003-1401-1520

**Yee Seng Tan:** Research Centre for Crystalline Materials, School of Science and Technology, Sunway University, 47500 Bandar Sunway, Selangor Darul Ehsan, Malaysia

## Abstract

$C_{29}H_{30}CdCl_2P_2$, orthorhombic, $Pna2_1$ (no. 33), $a = 15.84368(4)$ Å, $b = 8.46281(2)$ Å, $c = 20.88054(8)$ Å, $V = 2799.705(14)$ Å$^3$, $Z = 4$, $R_{gt}(F) = 0.0275$, $wR_{ref}(F^2) = 0.0719$, $T = 100(2)$ K.

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Table 1 contains crystallographic data and Table 2 contains the list of the atoms including atomic coordinates and displacement parameters.

**Table 1:** Data collection and handling.

| | |
|---|---|
| Crystal: | Colourless block |
| Size: | 0.19 × 0.17 × 0.13 mm |
| Wavelength: | Cu *Kα* radiation (1.54184 Å) |
| $\mu$: | 9.20 mm$^{-1}$ |
| Diffractometer, scan mode: | XtaLAB Synergy, $\omega$ |
| $\theta_{max}$, completeness: | 67.0°, >99% |
| $N(hkl)_{measured}$, $N(hkl)_{unique}$, $R_{int}$: | 125730, 4962, 0.036 |
| Criterion for $I_{obs}$, $N(hkl)_{gt}$: | $I_{obs} > 2\ \sigma(I_{obs})$, 4959 |
| $N(param)_{refined}$: | 307 |
| Programs: | CrysAlis$^{PRO}$ [1], SHELX [2, 3], WinGX/ORTEP [4] |

## Source of material

The title compound was prepared by using a layering method. 1,5-Bis(diphenylphosphino)pentane (Sigma-Aldrich; 0.0660 g, 0.015 mmol) was dissolved in chloroform (Merck; 5 mL) and transferred to a 14 mL test tube. This was followed by careful layering of a buffer solution (Merck; 2 mL 1:1 *v/v* mixture of chloroform and ethanol). An ethanol solution (5 mL) of cadmium (II) chloride (Acros Organic; 0.0275 g, 0.015 mmol) was prepared and carefully layered upon the buffer solution. The test tube was screwed with a cap and allowed to stand under ambient conditions. Colourless block crystals were formed after one week. Yield: 0.074 g (78.6%). **M.pt** (Stuart SMP30 Melting Point apparatus): 515.0–516.5 K. **IR** (Bruker Vertex 70 V equipped with Platinum ATR from 400 to 80 cm$^{-1}$): 1433 (m) ν(P—$CH_2$); 1101 (w) ν(P—$C_{aromatic}$); 149 (w) ν(Cd—Cl); 203 (w) ν(Cd—P).

**Table 2:** Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å$^2$).

| Atom | x | y | z | $U_{iso}$*/$U_{eq}$ |
|---|---|---|---|---|
| Cd | 0.43651(2) | 0.14468(3) | 0.49853(2) | 0.01744(11) |
| Cl1 | 0.35253(6) | −0.00992(14) | 0.42354(6) | 0.0336(3) |
| Cl2 | 0.36351(7) | 0.36939(12) | 0.54656(6) | 0.0327(3) |
| P1 | 0.56331(5) | 0.23049(12) | 0.42723(5) | 0.0160(2) |
| P2 | 0.47101(6) | 0.95645(11) | 0.59335(5) | 0.0168(2) |
| C1 | 0.6389(3) | 0.3642(4) | 0.4655(2) | 0.0176(8) |
| H1A | 0.6663 | 0.3108 | 0.5023 | 0.021* |
| H1B | 0.6833 | 0.3949 | 0.4346 | 0.021* |
| C2 | 0.5913(2) | 0.5108(4) | 0.4886(2) | 0.0205(8) |
| H2A | 0.5424 | 0.4771 | 0.5148 | 0.025* |
| H2B | 0.5693 | 0.5688 | 0.4509 | 0.025* |
| C3 | 0.6465(3) | 0.6218(5) | 0.5282(2) | 0.0209(8) |
| H3A | 0.7009 | 0.6386 | 0.5058 | 0.025* |
| H3B | 0.6586 | 0.5727 | 0.5702 | 0.025* |
| C4 | 0.6025(2) | 0.7818(4) | 0.53841(19) | 0.0211(8) |
| H4A | 0.5927 | 0.8324 | 0.4963 | 0.025* |
| H4B | 0.6401 | 0.8518 | 0.5635 | 0.025* |
| C5 | 0.5177(2) | 0.7647(5) | 0.5737(2) | 0.0215(8) |
| H5A | 0.5265 | 0.7039 | 0.6136 | 0.026* |
| H5B | 0.4779 | 0.7045 | 0.5464 | 0.026* |
| C6 | 0.6214(2) | 0.0589(4) | 0.39951(19) | 0.0171(7) |
| C7 | 0.5763(3) | −0.0547(5) | 0.3653(2) | 0.0195(8) |
| H7 | 0.5199 | −0.0339 | 0.3524 | 0.023* |
| C8 | 0.6139(3) | −0.1987(5) | 0.3501(2) | 0.0226(8) |
| H8 | 0.5837 | −0.2756 | 0.3262 | 0.027* |
| C9 | 0.6964(3) | −0.2290(5) | 0.3702(2) | 0.0229(8) |
| H9 | 0.7219 | −0.3279 | 0.3607 | 0.027* |
| C10 | 0.7411(3) | −0.1157(5) | 0.4040(2) | 0.0229(8) |
| H10 | 0.7972 | −0.1370 | 0.4173 | 0.028* |
| C11 | 0.7042(3) | 0.0299(5) | 0.4186(2) | 0.0216(8) |
| H11 | 0.7352 | 0.1080 | 0.4412 | 0.026* |
| C12 | 0.5308(3) | 0.3377(4) | 0.3556(2) | 0.0190(8) |
| C13 | 0.4531(3) | 0.4168(6) | 0.3557(2) | 0.0261(9) |
| H13 | 0.4161 | 0.4069 | 0.3913 | 0.031* |
| C14 | 0.4303(3) | 0.5092(7) | 0.3039(3) | 0.0324(11) |
| H14 | 0.3780 | 0.5641 | 0.3042 | 0.039* |
| C15 | 0.4832(3) | 0.5218(5) | 0.2518(2) | 0.0300(10) |
| H15 | 0.4667 | 0.5840 | 0.2160 | 0.036* |
| C16 | 0.5602(3) | 0.4448(6) | 0.2510(3) | 0.0307(11) |
| H16 | 0.5962 | 0.4541 | 0.2149 | 0.037* |
| C17 | 0.5849(4) | 0.3536(4) | 0.3032(2) | 0.0251(9) |
| H17 | 0.6382 | 0.3025 | 0.3032 | 0.030* |
| C18 | 0.5473(3) | 1.0401(5) | 0.6490(2) | 0.0184(7) |
| C19 | 0.6010(3) | 1.1573(5) | 0.6257(2) | 0.0248(9) |
| H19 | 0.5920 | 1.2002 | 0.5842 | 0.030* |
| C20 | 0.6679(3) | 1.2116(5) | 0.6631(2) | 0.0304(9) |
| H20 | 0.7057 | 1.2889 | 0.6469 | 0.036* |
| C21 | 0.6785(3) | 1.1523(5) | 0.7238(3) | 0.0294(10) |
| H21 | 0.7241 | 1.1892 | 0.7493 | 0.035* |
| C22 | 0.6238(3) | 1.0392(5) | 0.7486(2) | 0.0268(9) |
| H22 | 0.6308 | 1.0018 | 0.7911 | 0.032* |
| C23 | 0.5590(2) | 0.9816(6) | 0.7106(2) | 0.0218(9) |
| H23 | 0.5225 | 0.9019 | 0.7267 | 0.026* |
| C24 | 0.3785(2) | 0.9049(5) | 0.64060(19) | 0.0191(8) |
| C25 | 0.3324(3) | 0.7688(5) | 0.6260(2) | 0.0222(8) |
| H25 | 0.3487 | 0.7040 | 0.5909 | 0.027* |
| C26 | 0.2624(3) | 0.7283(5) | 0.6628(2) | 0.0241(8) |
| H26 | 0.2314 | 0.6352 | 0.6530 | 0.029* |
| C27 | 0.2377(3) | 0.8230(5) | 0.7136(2) | 0.0238(8) |
| H27 | 0.1899 | 0.7950 | 0.7386 | 0.029* |
| C28 | 0.2828(3) | 0.9580(5) | 0.7278(2) | 0.0247(9) |
| H28 | 0.2660 | 1.0225 | 0.7627 | 0.030* |
| C29 | 0.3527(3) | 1.0004(5) | 0.6913(2) | 0.0217(8) |
| H29 | 0.3828 | 1.0946 | 0.7009 | 0.026* |

## Experimental details

The C-bound H atoms were geometrically placed (C—H = 0.95–0.99 Å) and refined as riding with $U_{iso}(H) = 1.2U_{eq}(C)$. The absolute structure was determined based on Friedel pairs included in the whole data set (classical method: Flack parameter: 0.004(7)).

## Comment

Recently, the crystal structure determination of $[CdCl_2(dppe)]_n$, where dppe is 1,2-bis(diphenylphosphino)ethane, was described [5]. The dppe ligands were found to be $\mu_2$-bridging with the outcome being a one-dimensional coordination polymer with a zig-zag topology. A survey of the literature showed that there were in fact several structural motifs for related phosphane adducts of cadmium (II) chloride, all of which featured tetrahedrally coordinated cadmium within $Cl_2P_2$ donor sets. There are mononuclear structures with mono- and bi-functional phosphane ligands, as exemplified by $CdCl_2(PPh_3)_2$ [6] and $CdCl_2$(dppf) [7], respectively; dppf is 1,1′-bis(diphenylphosphanyl) ferrocene. Prior to the most recent study [5], the only other polymeric structure was that formed between $CdCl_2$ and (*R*,*R*)-*trans*-4,5-bis(diphenylphosphinomethyl)-2,2-dimethyl-1,3-dioxalane [8]. The title one-dimensional coordination polymer $[CdCl_2(dppp)]_n$, (I), where dppp is 1,5-bis (diphenylphosphino)propane, was determined in continuation of on-going studies of cadmium coordination polymers [5, 9–11].

The asymmetric unit of (I) is shown in the top view of the figure (70% probability displacement ellipsoids; the unlabelled dppp molecule is related by the symmetry operation (i) $x, -1+y, z$) and comprises a cadmium(II), two chlorido ligands and a full dppp ligand. As anticipated, the cadmium atom is tetrahedrally coordinated within a $Cl_2P_2$ donor set. The Cd—Cl1, Cl2 bond lengths of 2.4359(10) and 2.4412(10) Å, respectively, are experimentally equivalent to each other as are the Cd—P1, P2$^i$ bond lengths of 2.6038(10) and 2.5994(10) Å, respectively. The tetrahedral

angles around the cadmium atom span a relatively narrow range, i.e. 101.76(4)°, for Cl1—Cd—P1, to 116.37(3)°, for P1—Cd—P2$^{i}$. The *n*-propanyl link has a curved shape as seen in the sequence of torsion angles (*cf*. upper view of the figure): P1—C1—C2—C3 [173.3(3)°], C1—C2—C3—C4 [168.2(3)°], C2—C3—C4—C5 [59.8(5)°] and C3—C4—C5—P2 [173.9(3)°], i.e. there is a + syn-clinal conformation about the C3—C4 bond. As indicated in the figure, the dppp ligands are $\mu_2$-bridging so that a one-dimensional coordination polymer results. The chain is propagated by translational symmetry along the *b*-axis so it has a linear topology, as shown in the lower view of the figure (all hydrogen atoms have been omitted). When viewed down the *b*-axis, the chain maybe described as having the $CdCl_2$ residues residing in a bay defined by the dppp molecules, indicating the chloride atoms are available to form intermolecular interactions.

In the crystal, the connections between the chains leading to a three-dimensional architecture are of the type phenyl-C—H$\cdots$Cl, involving the same chloride atom [C9—H9$\cdots$Cl1$^{ii}$: H9$\cdots$Cl1$^{ii}$ = 2.81 Å, C9$\cdots$Cl1$^{ii}$ = 3.499(5) Å with angle at H9 = 130° and C22—H22$\cdots$Cl1$^{iii}$: H22$\cdots$Cl1$^{iii}$ = 2.78 Å, C22—H22$\cdots$Cl1$^{iii}$ = 3.680(4) Å with angle at H22 = 159° for symmetry operations (ii) $1/2+x$, $-1/2-y$, $z$ and (iii) $1-x$, $1-y$, $1/2+z$] and phenyl-C—H$\cdots\pi$(phenyl) interactions [C26—H26$\cdots$Cg(18—C23)$^{iv}$: H26$\cdots$Cg(18—C23)$^{iv}$ = 2.81 Å, C26$\cdots$Cg(18—C23)$^{iv}$ = 3.666(5) Å with angle at H26 = 151° and C28—H28$\cdots$Cg(C6—C11)$^{v}$: H28$\cdots$Cg(C6—C11)$^{v}$ = 2.86 Å, C28$\cdots$Cg(C6—C11)$^{v}$ = 3.568(5) Å with angle at H28 = 132° for (iv) $-1/2+x$, $3/2-y$, $z$ and (v) $1-x$, $1-y$, $1/2+z$].

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