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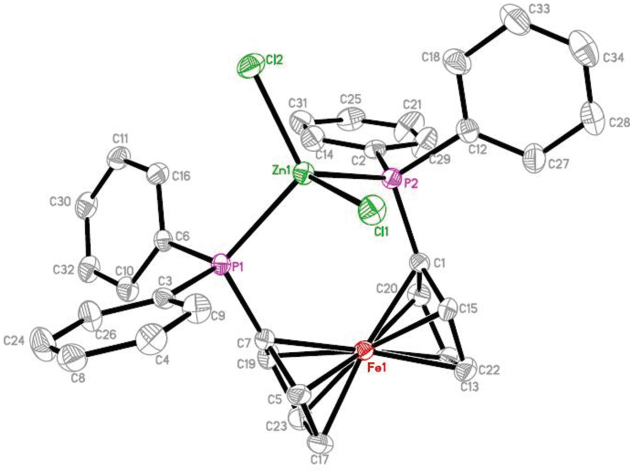
Crystal structure of dichlorido[1,1′-bis(diphenylphosphanyl)ferrocene-κ²*P*,*P*′]zinc(II), $C_{34}H_{28}FeCl_2P_2Zn$



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Abstract

$C_{34}H_{28}FeCl_2P_2Zn$, triclinic, $P\bar{1}$ (no. 2), $a = 9.6794(11)$ Å, $b = 9.7989(10)$ Å, $c = 18.2141(19)$ Å, $\alpha = 95.8740(10)°$, $\beta = 99.9350(10)°$, $\gamma = 115.596(2)°$, $V = 1503.9(3)$ Å^3, $Z = 2$, $R_{gt}(F) = 0.0362$, $wR_{ref}(F^2) = 0.0964$, $T = 298$ K.

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The crystal structure is shown in the figure. Tables 1 and 2 contain details on crystal structure and measurement conditions and a list of the atoms including atomic coordinates and displacement parameters.

Table 1: Data collection and handling.

Crystal:	Red block
Size:	0.50 × 0.46 × 0.21 mm
Wavelength:	Mo $K\alpha$ radiation (0.71073 Å)
μ:	1.59 cm^{-1}
Diffractometer, scan mode:	CCD area detector, φ and ω-scans
θ_{max}, completeness:	25°, >98%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	7846, 5214, 0.024
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 3878
$N(param)_{refined}$:	361
Programs:	Bruker programs [1], SHELX [2, 3]

Source of material

The reaction was carried out under an atomsphere of dry dinitrogen. Dichloromethane and *n*-hexane were dried with appropriate drying agents and distilled prior to use. The title compound was synthesized according to the literature [4]. [$ZnCl_2$(dppf)] was prepared by mixing $ZnCl_2$ (1 mmol) and dppf (1 mmol) in 10 mL dichloromethane under N_2. The mixture was refluxed for 4 h, then the residue was filtered off and the solution was concentrated to 5 mL. Crystals were obtained by slow evaporation of a hexane solution.

Experimental details

All H atoms were placed on geometrically idealized positions (C–H = 0.93–0.97 Å) and treated as riding on their parent atoms, with $U_{iso}(H) = 1.2U_{eq}(C)$ [1–3].

Discussion

The metalloligand 1,1′-bis(diphenylphosphanyl)ferrocene (dppf) occupies a special place in organometallic and catalysis research. Reasons for this are manifold: the presence of the two strong P donors, a redox-active ferrocene backbone, and an unusual bite angle of the two P donors [5–7]. The ligand dppf provides an ideal starting point for the synthesis of heterodinuclear complexes, because of the ability of the P donors to bind to a variety of metals [8]. Several efforts have been made to prepare and study metal complexes of dppf not only because of its versatile bonding modes, but also for the chemical reactivities.

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

Atom	*x*	*y*	*z*	U_{iso}*/U_{eq}
Zn1	0.84907(4)	0.58543(5)	0.74651(2)	0.02807(13)
P1	0.77899(10)	0.32364(10)	0.68975(5)	0.0254(2)
P2	1.13058(10)	0.71277(10)	0.79838(5)	0.0278(2)
Fe1	1.15413(5)	0.47942(6)	0.64939(3)	0.02907(15)
Cl1	0.83926(11)	0.71825(11)	0.65338(5)	0.0403(2)
Cl2	0.71055(12)	0.59425(11)	0.83123(6)	0.0446(3)
C1	1.2303(4)	0.6776(4)	0.73006(19)	0.0285(8)
C2	1.3417(4)	0.6173(4)	0.7396(2)	0.0357(9)
H2	1.3740	0.5855	0.7828	0.043*
C3	1.3938(4)	0.6146(5)	0.6724(2)	0.0440(10)
H3	1.4677	0.5818	0.6637	0.053*
C4	1.3150(4)	0.6700(4)	0.6202(2)	0.0418(10)
H4	1.3282	0.6802	0.5713	0.050*
C5	1.2129(4)	0.7073(4)	0.6550(2)	0.0355(9)
H5	1.1459	0.7447	0.6327	0.043*
C6	0.9257(4)	0.3138(4)	0.6424(2)	0.0295(8)
C7	0.9454(4)	0.3506(4)	0.5701(2)	0.0367(9)
H7	0.8898	0.3905	0.5401	0.044*
C8	1.0643(5)	0.3161(5)	0.5518(2)	0.0483(11)
H8	1.0996	0.3282	0.5074	0.058*
C9	1.1199(5)	0.2608(5)	0.6118(3)	0.0482(11)
H9	1.1984	0.2296	0.6140	0.058*
C10	1.0381(4)	0.2601(4)	0.6683(2)	0.0396(9)
H10	1.0539	0.2301	0.7145	0.048*
C11	0.5946(4)	0.2352(4)	0.61539(19)	0.0279(8)
C12	0.5664(4)	0.3273(4)	0.5679(2)	0.0380(9)
H12	0.6398	0.4304	0.5749	0.046*
C13	0.4292(4)	0.2663(5)	0.5102(2)	0.0438(10)
H13	0.4118	0.3285	0.4783	0.053*
C14	0.3201(4)	0.1157(5)	0.5000(2)	0.0434(10)
H14	0.2286	0.0750	0.4612	0.052*
C15	0.3458(5)	0.0249(5)	0.5472(2)	0.0525(12)
H15	0.2704	−0.0774	0.5409	0.063*
C16	0.4831(4)	0.0836(4)	0.6044(2)	0.0425(10)
H16	0.5000	0.0201	0.6355	0.051*
C17	0.7581(4)	0.1911(4)	0.75466(19)	0.0268(8)
C18	0.7492(4)	0.2336(4)	0.8278(2)	0.0335(9)
H18	0.7505	0.3279	0.8426	0.040*
C19	0.7384(5)	0.1362(4)	0.8793(2)	0.0414(10)
H19	0.7351	0.1663	0.9288	0.050*
C20	0.7327(4)	−0.0049(5)	0.8567(2)	0.0432(10)
H20	0.7263	−0.0697	0.8913	0.052*
C21	0.7364(4)	−0.0507(4)	0.7837(2)	0.0412(10)
H21	0.7295	−0.1475	0.7685	0.049*
C22	0.7504(4)	0.0471(4)	0.7324(2)	0.0355(9)
H22	0.7548	0.0166	0.6832	0.043*
C23	1.1973(4)	0.9205(4)	0.8162(2)	0.0324(8)
C24	1.3144(5)	1.0235(5)	0.7881(2)	0.0477(11)
H24	1.3682	0.9886	0.7598	0.057*
C25	1.3533(5)	1.1784(5)	0.8014(3)	0.0560(12)
H25	1.4336	1.2475	0.7826	0.067*
C26	1.2734(5)	1.2301(5)	0.8424(3)	0.0579(13)
H26	1.2983	1.3342	0.8506	0.069*
C27	1.1573(5)	1.1294(5)	0.8714(3)	0.0614(13)
H27	1.1050	1.1656	0.9001	0.074*
C28	1.1174(5)	0.9742(5)	0.8580(2)	0.0497(11)
H28	1.0370	0.9056	0.8769	0.060*
C29	1.3781(5)	0.7684(6)	0.9208(2)	0.0581(13)
H29	1.4372	0.8558	0.9030	0.070*
C30	1.4468(5)	0.7313(6)	0.9833(2)	0.0666(15)
H30	1.5523	0.7950	1.0076	0.080*
C31	1.3627(5)	0.6026(6)	1.0099(2)	0.0526(12)
H31	1.4115	0.5771	1.0510	0.063*
C32	1.2064(6)	0.5114(5)	0.9758(2)	0.0534(12)
H32	1.1476	0.4251	0.9945	0.064*
C33	1.1355(5)	0.5482(5)	0.9131(2)	0.0455(10)
H33	1.0290	0.4861	0.8900	0.055*
C34	1.2209(4)	0.6751(4)	0.88487(19)	0.0300(8)

In the crystal structure of the title compound, the zinc(II) atom displays a distorted tetrahedral coordination geometry provided by two chlorido ligands and the dppf ligand (*cf.* the figure). The average Zn-P bond distance of 2.4113(10) Å is similar to those observed in related complexes [9]. The Zn—Cl bond lengths (mean value 2.2423(10) Å) are not unexpceptional.

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