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Crystal structure of 1,1'-bis(diphenylphosphino)ferrocene-(1,1'-bis(diphenylphosphino)ferrocene- κ^2P,P')-(O-isobutyl sulfurodithioite- κ^2S,S')copper(I), $C_{39}H_{37}CuFeOP_2S_2$

Table 1: Data collection and handling.

Crystal:	Brownish yellow block
Size:	0.25 × 0.20 × 0.11 mm
Wavelength:	Mo K α radiation (0.71073 Å)
μ :	1.21 mm ⁻¹
Diffractometer, scan mode:	Enraf-Nonius CAD-4, ω
$\theta_{\max}$, completeness:	25.5°, 99%
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$, R_{int} :	12016, 6745, 0.017
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2 \sigma(I_{\text{obs}})$, 5281
$N(\text{param})_{\text{refined}}$:	442
Programs:	CAD-4 [1, 2], Olex2 [3]

The molecular structure is shown in the figure. Table 1 contains crystallographic data and Table 2 contains the list of the atoms including atomic coordinates and displacement parameters.

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Abstract

$C_{39}H_{37}CuFeOP_2S_2$, triclinic, $P\bar{1}$ (no. 2), $a = 9.6132(10)$ Å, $b = 11.5667(12)$ Å, $c = 16.8739(18)$ Å, $\alpha = 99.732(2)^\circ$, $\beta = 96.342(2)^\circ$, $\gamma = 93.446(2)^\circ$, $V = 1832.0(3)$ Å³, $Z = 2$, $R_{\text{gt}}(F) = 0.0455$, $wR_{\text{ref}}(F^2) = 0.1212$, $T = 293.0$ K.

CCDC no.: 1877712

Source of material

Add O-isobutyl S-hydrogen sulfurodithioite (0.05 mol) and copper nitrate (0.05 mol) to 50 mL of distilled water, heat the mixture and stir to reflux for 4 h. Then add 50 mL DMF solution containing 1,1'-bis(diphenylphosphino)ferrocene (0.05 mol); heat and stir to reflux 2 h. The reaction mixture was left to stand for 20 days, and finally brown-yellow block crystals were obtained.

Experimental details

Hydrogen atoms were placed in their geometrically idealized positions and constrained to ride on their parent atoms. The isobutyl group suffers a disorder.

Comment

Ferrocene is a metal organic complex with aromatic sandwich structure. Ferrocene and its derivatives have been widely used in catalysis [4], electrochemistry [5], functional materials [6], medicine [7], additives [8] and other fields due to their special chemical structure.

The molecular structure of the title complex is shown in the figure. Bond lengths and angles are within normal ranges. In the complex, the Cu1–P1 bond length is 2.267 Å and the Cu1–P2 bond length is 2.237 Å. The Cu1–S1 bond length is 2.420 Å and the Cu1–S2 bond length is 2.376 Å. The

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Table 2: Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²).

Atom	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> _{iso} [*] / <i>U</i> _{eq}
Cu1	0.24820(4)	0.55680(4)	0.26054(3)	0.04362(13)
Fe1	0.59701(5)	0.72905(4)	0.39448(3)	0.04062(14)
S1	0.12243(12)	0.49275(12)	0.12634(7)	0.0725(3)
S2	0.08020(15)	0.40423(12)	0.27398(8)	0.0816(4)
P1	0.46810(9)	0.49958(7)	0.25089(5)	0.0380(2)
P2	0.24546(9)	0.73535(8)	0.33573(6)	0.0413(2)
O1	−0.0404(5)	0.3007(4)	0.1367(2)	0.1123(13)
C1	0.5718(6)	0.2881(4)	0.2865(3)	0.0835(12)
H1	0.6251(6)	0.3337(4)	0.3316(3)	0.1002(14)*
C2	0.5775(7)	0.1662(4)	0.2728(4)	0.1003(14)
H2	0.6348(7)	0.1313(4)	0.3086(4)	0.1204(17)*
C3	0.5012(7)	0.0994(4)	0.2087(4)	0.0977(14)
H3	0.5064(7)	0.0183(4)	0.1992(4)	0.1172(16)*
C4	0.4156(6)	0.1504(4)	0.1570(3)	0.0925(13)
H4	0.3619(6)	0.1038(4)	0.1125(3)	0.1110(16)*
C5	0.4082(5)	0.2712(4)	0.1702(3)	0.0745(11)
H5	0.3482(5)	0.3049(4)	0.1351(3)	0.0894(13)*
C6	0.4888(4)	0.3416(3)	0.2347(2)	0.0548(9)
C7	0.4847(4)	0.6247(4)	0.1260(3)	0.0654(10)
H7	0.3969(4)	0.6474(4)	0.1376(3)	0.0785(12)*
C8	0.5460(5)	0.6674(5)	0.0654(3)	0.0842(12)
H8	0.4994(5)	0.7182(5)	0.0364(3)	0.1010(14)*
C9	0.6748(5)	0.6351(6)	0.0482(3)	0.0891(13)
H9	0.7169(5)	0.6653(6)	0.0081(3)	0.1069(15)*
C10	0.7422(5)	0.5589(5)	0.0893(3)	0.0815(12)
H10	0.8296(5)	0.5365(5)	0.0768(3)	0.0978(14)*
C11	0.6813(4)	0.5150(4)	0.1493(3)	0.0654(10)
H11	0.7274(4)	0.4622(4)	0.1766(3)	0.0785(12)*
C12	0.5512(4)	0.5490(4)	0.1695(2)	0.0496(8)
C13	0.0146(5)	0.8072(5)	0.2510(3)	0.0788(12)
H13	−0.0180(5)	0.7284(5)	0.2439(3)	0.0946(14)*
C14	−0.0698(6)	0.8875(6)	0.2208(3)	0.0995(14)
H14	−0.1594(6)	0.8632(6)	0.1942(3)	0.1194(17)*
C15	−0.0189(7)	1.0026(6)	0.2308(3)	0.0996(14)
H15	−0.0763(7)	1.0562(6)	0.2115(3)	0.1195(17)*
C16	0.1127(7)	1.0419(5)	0.2680(3)	0.0916(13)
H16	0.1459(7)	1.1202(5)	0.2724(3)	0.1099(16)*
C17	0.1963(6)	0.9615(4)	0.2993(3)	0.0722(11)
H17	0.2859(6)	0.9868(4)	0.3256(3)	0.0866(13)*
C18	0.1474(4)	0.8446(4)	0.2917(2)	0.0590(9)
C19	0.1709(4)	0.6415(3)	0.4686(2)	0.0455(8)
H19	0.1988(4)	0.5712(3)	0.4418(2)	0.0546(9)*
C20	0.1266(4)	0.6466(4)	0.5442(2)	0.0534(9)
H20	0.1260(4)	0.5799(4)	0.5683(2)	0.0641(10)*
C21	0.0836(4)	0.7490(3)	0.5840(2)	0.0539(9)
H21	0.0544(4)	0.7518(3)	0.6350(2)	0.0647(10)*
C22	0.0834(4)	0.8479(3)	0.5486(2)	0.0510(8)
H22	0.0529(4)	0.9171(3)	0.5752(2)	0.0613(10)*
C23	0.1286(4)	0.8438(3)	0.4735(2)	0.0451(8)
H23	0.1288(4)	0.9109(3)	0.4499(2)	0.0541(9)*
C24	0.1740(3)	0.7406(3)	0.4323(2)	0.0393(7)
C25	0.4186(4)	0.8106(3)	0.3689(2)	0.0473(8)
C26	0.4822(4)	0.8543(3)	0.4490(3)	0.0583(9)
H26	0.4384(4)	0.8516(3)	0.4985(3)	0.0700(11)*

Table 2 (continued)

Atom	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> _{iso} [*] / <i>U</i> _{eq}
C27	0.6201(5)	0.9028(3)	0.4454(3)	0.0692(10)
H27	0.6880(5)	0.9393(3)	0.4918(3)	0.0831(12)*
C28	0.6423(5)	0.8892(3)	0.3634(3)	0.0681(10)
H28	0.7289(5)	0.9139(3)	0.3429(3)	0.0817(12)*
C29	0.5206(4)	0.8314(3)	0.3158(3)	0.0567(9)
H29	0.5074(4)	0.8099(3)	0.2566(3)	0.0680(11)*
C30	0.5497(4)	0.5678(3)	0.4200(2)	0.0452(8)
H30	0.4592(4)	0.5394(3)	0.4342(2)	0.0543(9)*
C31	0.6644(4)	0.6252(3)	0.4754(2)	0.0562(9)
H31	0.6665(4)	0.6444(3)	0.5344(2)	0.0675(11)*
C32	0.7739(4)	0.6521(3)	0.4310(3)	0.0554(9)
H32	0.8651(4)	0.6937(3)	0.4538(3)	0.0665(11)*
C33	0.7294(4)	0.6104(3)	0.3479(2)	0.0455(8)
H33	0.7845(4)	0.6178(3)	0.3032(2)	0.0546(9)*
C34	0.5890(3)	0.5569(3)	0.3400(2)	0.0374(7)
C35	0.0476(5)	0.3946(4)	0.1744(3)	0.0748(12)
C36A ^a	−0.0522(11)	0.2663(10)	0.0488(4)	0.115(2)
H36a ^a	−0.0244(11)	0.3329(10)	0.0245(4)	0.138(3)*
H36b ^a	0.0086(11)	0.2041(10)	0.0343(4)	0.138(3)*
C37A ^b	−0.2070(15)	0.2226(13)	0.0181(6)	0.123(2)
H37A ^b	−0.2722(15)	0.2846(13)	0.0236(6)	0.148(3)*
C38A ^a	−0.2475(12)	0.1169(9)	0.0568(6)	0.131(3)
H38a ^a	−0.2577(12)	0.1434(9)	0.1128(6)	0.197(4)*
H38b ^a	−0.3348(12)	0.0782(9)	0.0290(6)	0.197(4)*
H38c ^a	−0.1755(12)	0.0629(9)	0.0529(6)	0.197(4)*
C36B ^b	−0.107(3)	0.3120(12)	0.0565(9)	0.130(3)
H36c ^b	−0.185(3)	0.3607(12)	0.0597(9)	0.156(4)*
H36d ^b	−0.040(3)	0.3435(12)	0.0248(9)	0.156(4)*
C37B ^a	−0.1549(12)	0.1825(10)	0.0221(5)	0.130(2)
H37B ^a	−0.2283(12)	0.1504(10)	0.0502(5)	0.156(3)*
C38B ^b	−0.025(2)	0.1117(18)	0.0208(16)	0.136(3)
H38d ^b	0.016(2)	0.1115(18)	0.0754(16)	0.205(5)*
H38e ^b	−0.052(2)	0.0323(18)	−0.0061(16)	0.205(5)*
H38f ^b	0.042(2)	0.1470(18)	−0.0076(16)	0.205(5)*
C39	−0.1948(8)	0.1672(6)	−0.0699(4)	0.1304(19)
H39a	−0.1308(8)	0.2170(6)	−0.0915(4)	0.196(3)*
H39b	−0.2893(8)	0.1856(6)	−0.0843(4)	0.196(3)*
H39c	−0.1850(8)	0.0864(6)	−0.0919(4)	0.196(3)*

^aOccupancy: 0.703(7), ^bOccupancy: 0.297(7).

S1—Cu1—S2 bite angle is 74.6° and the P1—Cu1—P2 bond angle is 113.1°.

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