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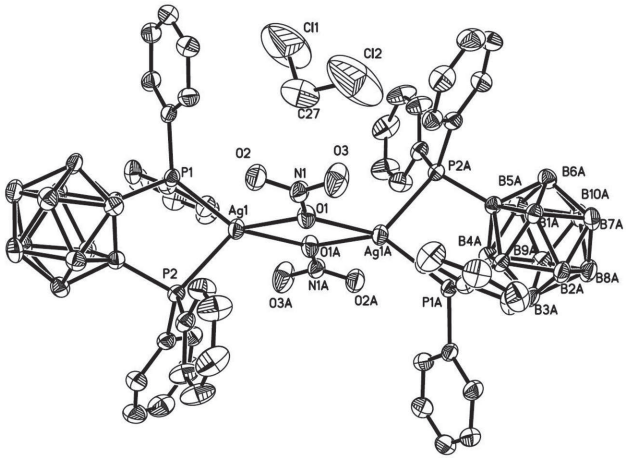
Crystal structure of bis(*μ*-nitrato-κ^2*O:O*)-bis[1,2-bis(diphenylphosphino)-1,2-dicarba-*closo*-dodecaborane-κ^2*P,P′*]disilver(I) dichloromethane monosolvate, $C_{54}H_{64}B_{20}Cl_4O_6P_4Ag_2$



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Abstract

$C_{54}H_{64}B_{20}Cl_4O_6P_4Ag_2$, triclinic, $P\bar{1}$ (no. 2), $a = 10.1200(10)$ Å, $b = 11.9101(11)$ Å, $c = 14.4999(15)$ Å, $\alpha = 86.432(2)°$, $\beta = 80.670(1)°$, $\gamma = 84.031(2)°$, $V = 1713.4(3)$Å^3, $Z = 1$, $R_{gt}(F) = 0.0535$, $wR_{ref}(F^2) = 0.1544$, $T = 298$ K.

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

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Table 1: Data collection and handling.

Crystal:	Yellow blocks
Size:	0.80 × 0.43 × 0.18 mm
Wavelength:	Mo *Kα* radiation (0.71073 Å)
μ:	8.7 cm^{-1}
Diffractometer, scan mode:	Bruker SMART, *φ* and *ω*
$2\theta_{max}$, completeness:	50°, >98%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	8733, 5920, 0.038
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 4341
$N(param)_{refined}$:	415
Programs:	SHELX [7], Bruker programs [8]

Source of material

The reaction was carried out under an atmosphere of dry nitrogen. Ethanol and dichloromethane were dried and distilled under nitrogen prior to use. 1,2-$(PPh_2)_2$-1,2-$C_2B_{10}H_{10}$, was synthesized according to the literature [1]. All the other chemicals were purchased and used as received. $AgNO_3$ (16.9 mg, 0.1 mmol) and 1,2-$(PPh_2)_2$-1,2-$C_2B_{10}H_{10}$ (51.2 mg, 0.1 mmol) were mixed in chloromethane (10 mL). The mixture was stirred for 6 h under a dry N_2 atmosphere, during which a yellow solution formed. After filtration, the solution was concentrated. The crystals were grown from a dichloromethane/n-hexane solution. (54.1%, m.p. 275–277 K). **FT-IR** (KBr) *ν* (cm^{-1}): 2989, 2966 2930, 2872 (C—H); 2614, 2602, 2585, 2556 (B—H); 1071 (C—P).

Experimental details

All H atoms were placed geometrically and treated as riding on their parent atoms, with B—H 1.10, C—H 0.96, with $U_{iso}(H) = 1.2U_{eq}(B)$, $U_{iso}(H) = 1.5U_{eq}(C)$.

Discussion

The title structure possesses a binuclear structure with two bidentate organic ligands and two bridiging nitrato ligands (*cf*. the figure). Both Ag atoms have the same coordination environment because the complex is located round an inversion

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

Atom	*x*	*y*	*z*	U_{iso}*/U_{eq}
Ag1	0.09951(4)	0.09046(3)	0.39690(3)	0.04984(17)
P1	0.32796(12)	0.10265(10)	0.31109(8)	0.0384(3)
P2	0.00759(12)	0.23383(10)	0.28739(8)	0.0395(3)
Cl1	0.3154(5)	0.2567(5)	0.7494(3)	0.265(3)
Cl2	0.0975(6)	0.3305(5)	0.8800(2)	0.268(3)
N1	0.0408(5)	0.1597(4)	0.6003(3)	0.0534(11)
O1	0.0043(4)	0.0854(3)	0.5541(2)	0.0534(9)
O2	0.1177(5)	0.2259(4)	0.5602(3)	0.0727(12)
O3	0.0018(6)	0.1602(5)	0.6841(3)	0.1086(19)
B1	0.2590(6)	0.2522(5)	0.1386(4)	0.0437(13)
H1	0.2346	0.1778	0.1055	0.052*
B2	0.4277(6)	0.2775(5)	0.1463(4)	0.0507(14)
H2	0.5148	0.2213	0.1157	0.061*
B3	0.4231(6)	0.3413(5)	0.2540(4)	0.0481(14)
H3	0.5065	0.3272	0.2947	0.058*
B4	0.2524(6)	0.3536(4)	0.3088(4)	0.0422(13)
H4	0.2235	0.3440	0.3852	0.051*
B5	0.1611(6)	0.4511(5)	0.2388(4)	0.0495(14)
H5	0.0749	0.5084	0.2692	0.059*
B6	0.1652(7)	0.3869(5)	0.1314(4)	0.0494(14)
H6	0.0815	0.4019	0.0910	0.059*
B7	0.3340(7)	0.3690(5)	0.0748(4)	0.0547(16)
H7	0.3593	0.3717	−0.0019	0.066*
B8	0.4349(7)	0.4253(5)	0.1480(5)	0.0609(17)
H8	0.5261	0.4661	0.1186	0.073*
B9	0.3280(7)	0.4726(5)	0.2506(5)	0.0545(15)
H9	0.3497	0.5434	0.2892	0.065*
B10	0.2748(7)	0.4916(5)	0.1392(5)	0.0579(16)
H10	0.2627	0.5757	0.1040	0.070*
C1	0.3193(5)	0.2406(4)	0.2423(3)	0.0395(11)
C2	0.1565(5)	0.3096(4)	0.2334(3)	0.0399(10)
C3	0.4704(5)	0.1114(4)	0.3718(3)	0.0436(11)
C4	0.4411(6)	0.1590(5)	0.4574(4)	0.0563(14)
H4A	0.3527	0.1841	0.4809	0.068*
C5	0.5429(7)	0.1697(6)	0.5093(4)	0.0718(17)
H5A	0.5234	0.2040	0.5664	0.086*
C6	0.6726(7)	0.1292(6)	0.4753(5)	0.078(2)
H6A	0.7417	0.1371	0.5089	0.094*
C7	0.6997(6)	0.0771(6)	0.3920(5)	0.0733(18)
H7A	0.7869	0.0468	0.3705	0.088*
C8	0.6001(5)	0.0691(5)	0.3398(4)	0.0579(14)
H8A	0.6202	0.0350	0.2826	0.069*
C9	0.3729(5)	−0.0076(4)	0.2272(3)	0.0440(11)
C10	0.4781(6)	−0.0159(5)	0.1540(4)	0.0621(15)
H10A	0.5349	0.0416	0.1409	0.074*
C11	0.4996(7)	−0.1096(5)	0.0997(4)	0.0727(18)
H11	0.5715	−0.1153	0.0508	0.087*
C12	0.4157(7)	−0.1942(5)	0.1175(5)	0.0709(17)
H12	0.4298	−0.2563	0.0801	0.085*
C13	0.3130(7)	−0.1875(5)	0.1891(5)	0.0758(18)
H13	0.2575	−0.2459	0.2017	0.091*
C14	0.2891(6)	−0.0954(4)	0.2436(4)	0.0564(14)
H14	0.2165	−0.0912	0.2920	0.068*
C15	−0.1099(5)	0.3413(4)	0.3477(4)	0.0469(12)
C16	−0.2313(6)	0.3816(5)	0.3217(5)	0.0676(17)
H16	−0.2544	0.3602	0.2663	0.081*
C17	−0.3193(6)	0.4546(6)	0.3792(6)	0.085(2)
H17	−0.4020	0.4815	0.3623	0.102*
C18	−0.2856(7)	0.4868(6)	0.4597(6)	0.088(2)
H18	−0.3444	0.5373	0.4967	0.105*
C19	−0.1694(7)	0.4471(7)	0.4861(5)	0.090(2)
H19	−0.1470	0.4696	0.5414	0.108*
C20	−0.0823(6)	0.3729(6)	0.4319(4)	0.0699(17)
H20	−0.0029	0.3432	0.4523	0.084*
C21	−0.0618(5)	0.1887(4)	0.1895(3)	0.0477(12)
C22	−0.0559(5)	0.0728(4)	0.1814(4)	0.0528(13)
H22	−0.0159	0.0240	0.2236	0.063*
C23	−0.1093(7)	0.0296(6)	0.1105(5)	0.0738(18)
H23	−0.1033	−0.0482	0.1047	0.089*
C24	−0.1695(7)	0.0988(8)	0.0504(5)	0.084(2)
H24	−0.2058	0.0683	0.0037	0.100*
C25	−0.1786(7)	0.2121(7)	0.0563(4)	0.0785(19)
H25	−0.2209	0.2587	0.0138	0.094*
C26	−0.1245(6)	0.2596(5)	0.1263(4)	0.0627(15)
H26	−0.1305	0.3376	0.1305	0.075*
C27	0.1641(10)	0.3281(8)	0.7698(6)	0.115(3)
H27A	0.1712	0.4053	0.7453	0.139*
H27B	0.1040	0.2948	0.7358	0.139*

center. The Ag atom is tetrahedrally surrounded by two P atoms and two oxygen atoms of two nitrato ligands. The Ag—O bond lengths are 2.326(3) and 2.455(3) Å, which is longer than the corresponding bond reported for the complex $[(Ag_2(\mu\text{-}ONO_2)_2)\{1,2\text{-}(PCy)_2\text{-}1,2\text{-}C_2B_{10}H_{10}\}_2]$ [2]. The Ag—P bond lengths are 2.4554(13) and 2.4726(13) Å, which are similar with the corresponding bond length of 2.49(3) in the complex $[(Ag_2(\mu\text{-}I)_2)\{1,2\text{-}(PCy)_2\text{-}1,2\text{-}C_2B_{10}H_{10}\}_2]$ [2]. The P—Ag—P angle is 90.80(4)°, which is smaller than the corresponding value of $[(Ag_2(\mu\text{-}ONO2)_2)\{1,2\text{-}(PCy)_2\text{-}1,2\text{-}C_2B_{10}H_{10}\}_2]$. A similar change of P—Ag—P angles has also been observed previously [3]. The P(1)—C(1)—C(2)—P(2) angle in the title structure is 0°, while the value of the free ligand is 10.9(4)° [4, 5], indicating that this fragment is planar. The average bond lengths of C—P in the complexes (1.872(3) Å) are in agreement with that of 1.894(3)Å in the free ligand, suggesting that the coordination with the metal atom has no obvious influence on this distance [6].

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