Crystal structure of *trans*-dichloro-bis(phenyl-2,4,6-trimethylbenzyl-selenido)palladium(II), $Pd(C_{16}H_{18}Se)_2Cl_2$

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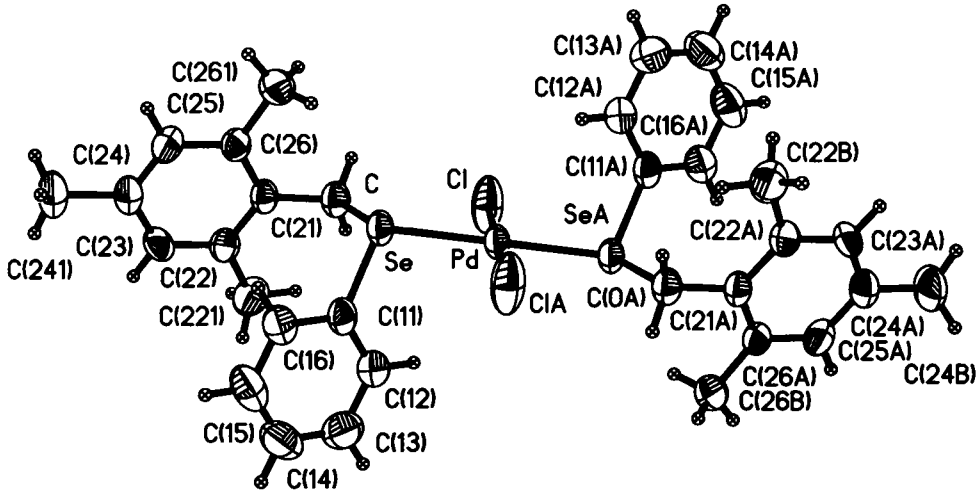

Abstract

$C_{32}H_{36}Cl_2PdSe_2$, monoclinic, $P12_1/n1$ (no. 14),
$a = 9.459(2)$ Å, $b = 12.519(3)$ Å, $c = 13.077(3)$ Å,
$\beta = 91.899(4)°$, $V = 1547.6$ Å^3, $Z = 2$,
$R_{gt}(F) = 0.041$, $wR_{ref}(F^2) = 0.093$, $T = 293$ K.

Source of material

The title compound was synthesized from diphenyl diselenide, 2,4,6-trimethylbenzyl chloride and $Pd(PhCN)_2Cl_2$ in two steps.

Experimental details

The hydrogen atoms were allowed to ride on idealized positions with $U_{iso} = 1.5\,U_{eq}$ (methyl) or $1.2\,U_{eq}$ (rest) of their carrier carbon atoms.

Discussion

Palladacycles have emerged as powerful catalysts for the Heck reactions [1-3]. During our study of this type of reactions, the title compound was synthesized. This compound proved to be an efficient catalyst. In this compound, Pd is coordinated to two Se and two Cl atoms in a distorted square planar geometry. The bond angle Cl–Pd–Se and Cl^i–Pd–Se are 85.63(4)° and 94.37(4)°, respectively, deviating from the ideal 90° (i: $-x,-y,-z$). The Cl^i–Pd–Se and Se–Pd–Se^i angles, however, are the ideal 180°. The bond distances Pd—Se and Pd—Cl are 2.4299(6) Å and 2.282(2) Å, respectively, within the normal range for this type of compounds. The compound showed excellent catalytic activity with the TON parameter in the order of 10^5.

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Table 1. Data collection and handling.

Crystal:	red fragment, size 0.10 × 0.10 × 0.16 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	31.44 cm^{-1}
Diffractometer, scan mode:	Siemens SMART CCD, ω
$2\theta_{max}$:	46.52°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	6849, 2230
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 2137
$N(param)_{refined}$:	170
Programs:	SADABS [4], SHELXTL [5]

Table 2. Atomic coordinates and displacement parameters (in Å^2).

Atom	Site	x	y	z	U_{iso}
H(0A)	4e	−0.0671	−0.0178	0.2831	0.071
H(0B)	4e	0.0734	−0.0824	0.2687	0.071
H(12)	4e	0.2982	−0.0600	0.0708	0.090
H(13)	4e	0.5414	−0.0795	0.0921	0.112
H(14)	4e	0.6679	0.0330	0.1996	0.114
H(15)	4e	0.5547	0.1624	0.2896	0.110
H(16)	4e	0.3122	0.1840	0.2694	0.084
H(23)	4e	0.3612	0.0166	0.5371	0.074
H(25)	4e	0.0548	0.2353	0.5291	0.077
H(22A)	4e	0.2320	−0.1360	0.3273	0.097
H(22B)	4e	0.2865	−0.1609	0.4393	0.097
H(22C)	4e	0.3809	−0.0947	0.3660	0.097
H(24A)	4e	0.3753	0.1556	0.6578	0.114
H(24B)	4e	0.2262	0.1925	0.6928	0.114
H(24C)	4e	0.3124	0.2649	0.6195	0.114
H(26A)	4e	−0.1274	0.2354	0.4099	0.091
H(26B)	4e	−0.1752	0.1214	0.3734	0.091
H(26C)	4e	−0.0879	0.1943	0.3013	0.091

Table 3. Atomic coordinates and displacement parameters (in $Å^2$).

Atom	Site	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
Pd	2*a*	0	0	0	0.0446(4)	0.0528(4)	0.0285(3)	–0.0068(3)	–0.0058(2)	0.0022(2)
Se	4*e*	0.08076(6)	0.08163(4)	0.16009(4)	0.0485(3)	0.0526(4)	0.0306(3)	–0.0027(2)	–0.0058(2)	0.0000(2)
Cl	4*e*	–0.1087(2)	–0.1399(2)	0.0770(1)	0.124(2)	0.087(1)	0.0459(9)	–0.058(1)	–0.0115(9)	0.0121(8)
C	4*e*	0.0347(6)	–0.0115(4)	0.2785(4)	0.052(3)	0.055(3)	0.035(3)	–0.007(3)	–0.003(2)	0.004(2)
C(11)	4*e*	0.2823(6)	0.0629(4)	0.1689(4)	0.053(3)	0.053(3)	0.033(3)	–0.006(3)	–0.004(2)	0.006(2)
C(12)	4*e*	0.3495(6)	–0.0153(5)	0.1150(5)	0.054(4)	0.077(4)	0.048(3)	0.007(3)	–0.002(3)	–0.004(3)
C(13)	4*e*	0.4948(7)	–0.0264(6)	0.1276(5)	0.072(5)	0.090(5)	0.064(4)	0.017(4)	0.009(4)	0.000(4)
C(14)	4*e*	0.5703(7)	0.0405(7)	0.1921(6)	0.051(4)	0.100(5)	0.077(5)	–0.001(4)	–0.007(4)	0.014(4)
C(15)	4*e*	0.5030(7)	0.1180(6)	0.2454(5)	0.061(4)	0.086(5)	0.072(5)	–0.018(4)	–0.019(3)	0.008(4)
C(16)	4*e*	0.3582(6)	0.1306(5)	0.2339(4)	0.054(3)	0.062(4)	0.051(3)	–0.010(3)	–0.007(3)	0.006(3)
C(21)	4*e*	0.0962(5)	0.0362(4)	0.3748(4)	0.048(3)	0.049(3)	0.029(3)	–0.005(2)	–0.002(2)	0.003(2)
C(22)	4*e*	0.2191(6)	–0.0073(4)	0.4217(4)	0.051(3)	0.047(3)	0.036(3)	0.000(3)	0.000(2)	0.005(2)
C(23)	4*e*	0.2794(6)	0.0446(5)	0.5061(4)	0.043(3)	0.064(4)	0.040(3)	0.006(3)	–0.010(2)	0.009(3)
C(24)	4*e*	0.2214(6)	0.1371(5)	0.5457(4)	0.057(3)	0.056(4)	0.040(3)	–0.008(3)	–0.007(3)	–0.005(3)
C(25)	4*e*	0.0973(6)	0.1755(5)	0.5011(4)	0.064(4)	0.052(3)	0.038(3)	0.004(3)	0.003(3)	–0.005(3)
C(26)	4*e*	0.0342(5)	0.1275(4)	0.4158(4)	0.046(3)	0.053(3)	0.032(3)	–0.002(2)	0.002(2)	0.003(2)
C(221)	4*e*	0.2857(7)	–0.1089(5)	0.3853(5)	0.078(4)	0.062(4)	0.053(4)	0.019(3)	–0.002(3)	0.001(3)
C(241)	4*e*	0.2901(7)	0.1926(6)	0.6373(5)	0.081(5)	0.088(5)	0.057(4)	–0.008(4)	–0.017(3)	–0.020(4)
C(261)	4*e*	–0.1014(6)	0.1739(5)	0.3710(4)	0.057(3)	0.076(4)	0.049(3)	0.015(3)	–0.004(3)	0.006(3)

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