Crystal structure of *cis*-bis(*N*,*N*-dimethyl-*N′*-4-chlorobenzoylthioureato)palladium(II), $Pd(C_{10}H_{10}ClN_2OS)_2$

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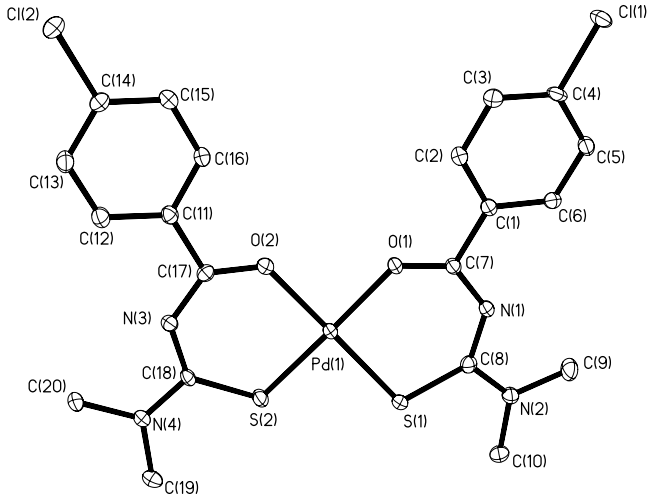

Abstract

$C_{20}H_{20}Cl_2N_4O_2PdS_2$, monoclinic, $P12_1/c1$ (No. 14), $a = 12.323(4)$ Å, $b = 8.629(3)$ Å, $c = 21.212(7)$ Å, $\beta = 97.170(6)°$, $V = 2238.0$ Å^3, $Z = 4$, $R_{gt}(F) = 0.045$, $wR_{ref}(F^2) = 0.133$, $T = 193$ K.

Source of material

The title compound was prepared according to the method described in the literature [1]. The metallic solution was added dropwise to the ligand in a 1:2 ratio with a small excess of ligand. The solid complex was filtered and recrystallized from a ethanol/dichloromethane mixture (1:1).

Discussion

We have worked on the preparation, structure characterization and properties of new thiourea derivatives. In this study, we have crystallized the *cis*-bis(*N*,*N*-dimethyl-*N′*-4-chlorobenzoylthioureato)palladium(II) complex and its structural analysis has been undertaken.

The title compound is a *cis*-complex with slightly distorted square planar coordination of the central Pd atom by 2 O and 2 S atoms. The Pd—S (2.240(1) Å & 2.243(1) Å) bond distances are equal within experimental error as are the Pd—O bonds. The distance of Pd from the best plane through the coordination sphere is 0.008(1) Å. The chelate ring systems, Pd–O–C–N–C–S, are nearly planar as well with the largest deviations from the best plane being 0.139(2) Å for S1. Accordingly; the dihedral angle between these chelate planes is 3.4(1)°. The molecular structure is very close to the related 1,1-diethyl Ni complex [2] and shows similar short C—N and C—S bonds indicating the known π-bonding character in the chelate rings (d(C7—O1) = 1.269(4) Å, d(C8—S1) = 1.734(4) Å). The bond lengths and angles in the thiourea moiety are typical for compound.

Table 1. Data collection and handling.

Crystal:	brown prism, size 0.24 × 0.41 × 0.48 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	12.81 cm^{-1}
Diffractometer, scan mode:	Bruker CCD, ω
$2\theta_{max}$:	50.2°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	13547, 3891
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 3532
$N(param)_{refined}$:	288
Program:	SHELXTL [3]

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

Atom	Site	x	y	z	U_{iso}
H(2)	4*e*	0.3852	–0.3451	–0.3723	0.032
H(3)	4*e*	0.4900	–0.3751	–0.2728	0.035
H(5)	4*e*	0.6134	–0.7789	–0.3336	0.031
H(6)	4*e*	0.5077	–0.7507	–0.4314	0.029
H(9A)	4*e*	0.4899	–0.8357	–0.5540	0.060
H(9B)	4*e*	0.4521	–0.9601	–0.6060	0.060
H(9C)	4*e*	0.3988	–0.9555	–0.5427	0.060
H(10A)	4*e*	0.2175	–0.8528	–0.6652	0.053
H(10B)	4*e*	0.3298	–0.9132	–0.6829	0.053
H(10C)	4*e*	0.2967	–0.7393	–0.6948	0.053
H(12)	4*e*	0.0427	0.2666	–0.5166	0.035
H(13)	4*e*	0.0524	0.4522	–0.4355	0.035
H(15)	4*e*	0.2985	0.2083	–0.3339	0.032
H(16)	4*e*	0.2864	0.0198	–0.4146	0.030
H(19A)	4*e*	–0.0122	–0.0452	–0.7601	0.045
H(19B)	4*e*	–0.1318	–0.0417	–0.7422	0.045
H(19C)	4*e*	–0.0555	–0.1834	–0.7223	0.045
H(20A)	4*e*	–0.1119	0.1489	–0.6249	0.044
H(20B)	4*e*	–0.1078	0.1914	–0.6964	0.044
H(20C)	4*e*	–0.0077	0.2290	–0.6456	0.044

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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(1)	4*e*	0.21837(2)	–0.31933(3)	–0.55932(1)	0.0223(2)	0.0200(2)	0.0207(2)	0.0033(1)	–0.0026(2)	0.0009(1)
S(1)	4*e*	0.21450(8)	–0.5463(1)	–0.61080(5)	0.0290(5)	0.0227(5)	0.0240(5)	0.0025(4)	–0.0081(4)	–0.0013(4)
Cl(2)	4*e*	0.18157(9)	0.4814(1)	–0.31719(5)	0.0404(6)	0.0320(5)	0.0347(6)	0.0051(4)	0.0040(5)	–0.0103(4)
S(2)	4*e*	0.12227(8)	–0.2245(1)	–0.64767(5)	0.0315(5)	0.0283(5)	0.0219(5)	0.0091(4)	–0.0035(4)	–0.0005(4)
Cl(1)	4*e*	0.64213(9)	–0.5978(1)	–0.22207(5)	0.0399(6)	0.0380(6)	0.0282(6)	–0.0059(4)	–0.0156(4)	0.0047(4)
O(1)	4*e*	0.3144(2)	–0.3991(3)	–0.4817(1)	0.033(2)	0.022(1)	0.025(1)	0.009(1)	–0.009(1)	–0.002(1)
O(2)	4*e*	0.2150(2)	–0.1172(3)	–0.5104(1)	0.037(2)	0.027(2)	0.028(2)	0.009(1)	–0.011(1)	–0.006(1)
N(1)	4*e*	0.3762(3)	–0.6404(4)	–0.5149(2)	0.024(2)	0.021(2)	0.020(2)	0.003(1)	–0.001(1)	0.002(1)
N(3)	4*e*	0.0810(3)	0.0300(4)	–0.5730(2)	0.021(2)	0.023(2)	0.024(2)	–0.000(1)	–0.001(1)	0.001(1)
N(4)	4*e*	–0.0186(3)	0.0040(4)	–0.6686(2)	0.024(2)	0.026(2)	0.024(2)	0.003(1)	–0.003(1)	0.001(1)
N(2)	4*e*	0.3447(3)	–0.7867(4)	–0.6034(2)	0.036(2)	0.026(2)	0.023(2)	0.006(2)	–0.004(2)	–0.004(1)
C(1)	4*e*	0.4357(3)	–0.5437(4)	–0.4117(2)	0.017(2)	0.022(2)	0.024(2)	–0.002(1)	0.002(1)	0.004(2)
C(2)	4*e*	0.4316(3)	–0.4341(4)	–0.3643(2)	0.029(2)	0.022(2)	0.026(2)	0.002(2)	–0.002(2)	0.003(2)
C(3)	4*e*	0.4934(3)	–0.4510(5)	–0.3056(2)	0.034(2)	0.028(2)	0.024(2)	0.000(2)	0.001(2)	–0.001(2)
C(4)	4*e*	0.5598(3)	–0.5787(5)	–0.2952(2)	0.024(2)	0.031(2)	0.019(2)	–0.008(2)	–0.007(2)	0.003(2)
C(5)	4*e*	0.5662(3)	–0.6908(4)	–0.3418(2)	0.027(2)	0.025(2)	0.027(2)	0.003(1)	0.000(2)	0.005(2)
C(6)	4*e*	0.5044(3)	–0.6735(4)	–0.3991(2)	0.023(2)	0.023(2)	0.025(2)	0.001(1)	–0.000(2)	–0.003(1)
C(7)	4*e*	0.3683(3)	–0.5246(4)	–0.4749(2)	0.020(2)	0.021(2)	0.021(2)	0.000(1)	0.000(1)	0.002(1)
C(8)	4*e*	0.3192(3)	–0.6586(4)	–0.5723(2)	0.027(2)	0.022(2)	0.024(2)	–0.002(2)	0.004(2)	0.003(2)
C(9)	4*e*	0.4286(4)	–0.8937(5)	–0.5740(2)	0.057(3)	0.029(2)	0.034(2)	0.015(2)	0.002(2)	–0.004(2)
C(10)	4*e*	0.2927(4)	–0.8264(5)	–0.6670(2)	0.047(3)	0.029(2)	0.028(2)	0.001(2)	–0.006(2)	–0.008(2)
C(11)	4*e*	0.1633(3)	0.1223(4)	–0.4727(2)	0.024(2)	0.022(2)	0.023(2)	0.000(2)	0.002(2)	0.005(2)
C(12)	4*e*	0.0941(3)	0.2539(5)	–0.4790(2)	0.029(2)	0.027(2)	0.030(2)	0.003(2)	0.001(2)	0.004(2)
C(13)	4*e*	0.1001(3)	0.3638(5)	–0.4315(2)	0.030(2)	0.026(2)	0.032(2)	0.007(2)	0.004(2)	0.001(2)
C(14)	4*e*	0.1760(3)	0.3452(4)	–0.3780(2)	0.025(2)	0.022(2)	0.026(2)	–0.004(2)	0.007(2)	–0.002(2)
C(15)	4*e*	0.2459(3)	0.2190(5)	–0.3711(2)	0.025(2)	0.029(2)	0.025(2)	0.002(2)	–0.001(2)	0.003(2)
C(16)	4*e*	0.2385(3)	0.1079(4)	–0.4190(2)	0.026(2)	0.022(2)	0.026(2)	0.003(2)	0.001(2)	0.003(2)
C(17)	4*e*	0.1539(3)	0.0002(4)	–0.5230(2)	0.023(2)	0.021(2)	0.024(2)	–0.003(2)	0.005(2)	–0.001(2)
C(18)	4*e*	0.0594(3)	–0.0551(4)	–0.6258(2)	0.019(2)	0.022(2)	0.021(2)	0.001(1)	0.000(1)	0.008(1)
C(19)	4*e*	–0.0579(3)	–0.0732(5)	–0.7284(2)	0.027(2)	0.037(2)	0.024(2)	0.000(2)	–0.004(2)	0.001(2)
C(20)	4*e*	–0.0655(3)	0.1565(5)	–0.6580(2)	0.028(2)	0.023(2)	0.034(2)	0.005(2)	–0.005(2)	0.004(2)

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