Z. Kristallogr. NCS 214 (1999) 565–566

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Crystal structure of bis(1,3-diisopropyl-4,5-dimethylimidazolium) tetrachloropalladate dihydrate, $(C_{11}H_{21}N_2)_2PdCl_4 \cdot 2H_2O$

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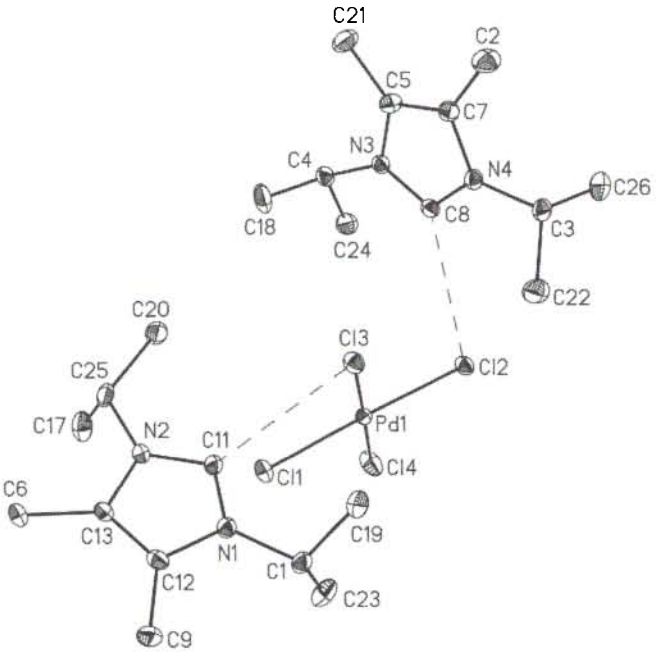
Received April 14, 1999, CCDC-No. 1267/165



Abstract

$C_{22}H_{46}Cl_4N_4O_2Pd$, monoclinic, $P12_1/n1$ (No. 14), $a = 13.176(5)$ Å, $b = 14.043(2)$ Å, $c = 17.289(6)$ Å, $\beta = 108.75(3)°$, $V = 3029.3$ Å^3, $Z = 4$, $R_{gt(F)} = 0.048$, $wR(F^2) = 0.112$, $T = 173$ K.

Source of material

Synthesis was done according to [1].

Discussion

Selected bond lengths [Å] and angles [°] are as follows: Pd(1)–Cl(1) 2.306(2), Pd(1)–Cl(2) 2.333(2), Pd(1)–Cl(3) 2.314(1), Pd(1)–Cl(4) 2.284(1), Cl(2)–C(8) 3.613(7), Cl(3)–C(11) 3.611(7), C(8)–N(3) 1.331(6), N(3)–C(5) 1.387(6), C(5)–C(7) 1.353(7), C(7)–N(4) 1.398(6), N(4)–C(8) 1.330(6), Cl(4)–Pd(1)–Cl(1) 89.68(5), Cl(4)–Pd(1)–Cl(3) 179.17(6), Cl(1)–Pd(1)–Cl(3) 89.84(5), Cl(4)–Pd(1)–Cl(2) 88.96(5), Cl(1)–Pd(1)–Cl(2) 177.97(5), Cl(3)–Pd(1)–Cl(2) 91.54(5).

The hydrogen atoms of the H_2O molecules have not been refined.

Table 1. Data collection and handling.

Crystal:	orange prism, size 0.30 × 0.35 × 0.35 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	9.90 cm^{-1}
Diffractometer, scan mode:	Siemens P4, ω
$2\theta_{max}$:	55.02°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	27183, 6956
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 4835
$N(param)_{refined}$:	308
Program:	SHELXL-97 [2]

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

Atom	Site	x	y	z	U_{iso}
H(11A)	4a	0.3337	0.2056	0.8880	0.041
H(9A)	4a	–0.0113	0.0997	0.9450	0.056
H(9B)	4a	0.0919	0.1001	1.0252	0.056
H(9C)	4a	0.0764	0.0170	0.9590	0.056
H(8A)	4a	0.7435	0.2973	0.8915	0.041
H(6A)	4a	–0.0736	0.2098	0.8447	0.055
H(6B)	4a	–0.0452	0.2316	0.7631	0.055
H(6C)	4a	–0.0257	0.3113	0.8325	0.055
H(4A)	4a	0.6765	0.4811	0.7353	0.049
H(3A)	4a	0.8584	0.3821	1.1020	0.050
H(2A)	4a	0.8376	0.6258	1.0311	0.073
H(2B)	4a	0.9245	0.5452	1.0710	0.073
H(2C)	4a	0.8117	0.5452	1.0874	0.073
H(1A)	4a	0.2696	0.0472	1.0371	0.047
H(25A)	4a	0.1164	0.3571	0.7717	0.051
H(24A)	4a	0.7583	0.3321	0.7373	0.060
H(24B)	4a	0.6414	0.3325	0.6705	0.060
H(24C)	4a	0.6606	0.2808	0.7566	0.060
H(23A)	4a	0.2753	–0.0427	0.9257	0.068
H(23B)	4a	0.3873	–0.0512	0.9977	0.068
H(23C)	4a	0.3771	0.0161	0.9209	0.068
H(22A)	4a	0.7313	0.2606	1.0525	0.073
H(22B)	4a	0.8432	0.2155	1.1064	0.073
H(22C)	4a	0.8076	0.2103	1.0089	0.073
H(21A)	4a	0.7527	0.6648	0.8954	0.069
H(21B)	4a	0.6475	0.6223	0.8295	0.069
H(21C)	4a	0.7599	0.6195	0.8124	0.069
H(20A)	4a	0.2998	0.3872	0.8379	0.063
H(20B)	4a	0.2729	0.3965	0.7411	0.063
H(20C)	4a	0.3313	0.3031	0.7875	0.063
H(19A)	4a	0.3795	0.1806	1.0814	0.067
H(19B)	4a	0.4435	0.1561	1.0193	0.067
H(19C)	4a	0.4516	0.0865	1.0944	0.067
H(18A)	4a	0.5258	0.4988	0.7789	0.076
H(18B)	4a	0.5141	0.3856	0.7823	0.076
H(18C)	4a	0.4952	0.4377	0.6965	0.076

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Table 2. Continued.

Atom	Site	x	y	z	U_{iso}
H(17A)	4a	0.0625	0.2219	0.6877	0.068
H(17B)	4a	0.1844	0.2009	0.6944	0.068
H(17C)	4a	0.1256	0.2942	0.648	0.068

Table 2. Continued.

Atom	Site	x	y	z	U_{iso}
H(26A)	4a	1.0085	0.3959	1.0585	0.082
H(26B)	4a	0.9819	0.2952	1.0132	0.082
H(26C)	4a	1.0161	0.3006	1.1107	0.082

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

Atom	Site	Occ.	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
Pd(1)	4a		0.55945(3)	0.09942(2)	0.81035(2)	0.0251(2)	0.0282(2)	0.0339(2)	–0.0002(1)	0.0105(1)	0.0021(1)
Cl(3)	4a		0.53828(9)	0.21952(8)	0.89550(8)	0.0323(5)	0.0414(5)	0.0481(6)	0.0005(4)	0.0159(5)	–0.0073(5)
Cl(2)	4a		0.74572(8)	0.11043(8)	0.86368(7)	0.0270(4)	0.0393(5)	0.0442(6)	–0.0018(4)	0.0128(4)	–0.0034(4)
Cl(4)	4a		0.5788(1)	–0.0180(1)	0.7247(1)	0.0406(6)	0.0582(8)	0.078(1)	–0.0029(6)	0.0172(6)	–0.0332(7)
Cl(1)	4a		0.37588(9)	0.08274(9)	0.75816(8)	0.0283(5)	0.0568(7)	0.0429(6)	–0.0017(4)	0.0098(4)	–0.0031(5)
N(4)	4a		0.8039(3)	0.3981(3)	0.9786(2)	0.032(2)	0.038(2)	0.029(2)	0.001(2)	0.006(1)	0.002(2)
N(1)	4a		0.2344(3)	0.1380(3)	0.9403(2)	0.027(2)	0.038(2)	0.036(2)	0.003(1)	0.012(2)	0.008(2)
N(2)	4a		0.1751(3)	0.2397(3)	0.8416(2)	0.024(2)	0.033(2)	0.040(2)	0.001(1)	0.009(1)	0.003(2)
N(3)	4a		0.7188(3)	0.4347(3)	0.8525(2)	0.028(2)	0.038(2)	0.033(2)	–0.003(1)	0.009(2)	0.003(2)
C(13)	4a		0.0870(3)	0.2061(3)	0.8619(3)	0.024(2)	0.032(2)	0.043(2)	–0.004(2)	0.010(2)	–0.001(2)
C(12)	4a		0.1244(3)	0.1423(3)	0.9231(3)	0.028(2)	0.034(2)	0.041(2)	–0.004(2)	0.015(2)	–0.004(2)
C(11)	4a		0.2629(3)	0.1965(3)	0.8895(3)	0.026(2)	0.035(2)	0.041(2)	0.003(2)	0.012(2)	0.007(2)
C(9)	4a		0.0653(4)	0.0849(3)	0.9668(4)	0.040(2)	0.043(3)	0.064(3)	–0.003(2)	0.024(2)	0.013(2)
C(8)	4a		0.7534(4)	0.3631(3)	0.9048(3)	0.034(2)	0.033(2)	0.033(2)	–0.005(2)	0.008(2)	–0.003(2)
C(7)	4a		0.8013(4)	0.4975(3)	0.9738(3)	0.040(2)	0.034(2)	0.040(3)	0.000(2)	0.007(2)	–0.005(2)
C(6)	4a		–0.0239(4)	0.2429(4)	0.8221(4)	0.022(2)	0.047(3)	0.064(3)	–0.002(2)	0.009(2)	0.004(2)
C(5)	4a		0.7485(4)	0.5195(3)	0.8948(3)	0.048(3)	0.032(2)	0.042(3)	–0.001(2)	0.016(2)	–0.001(2)
C(4)	4a		0.6554(4)	0.4271(4)	0.7646(3)	0.036(2)	0.046(2)	0.033(2)	–0.007(2)	0.003(2)	0.008(2)
C(3)	4a		0.8612(4)	0.3439(3)	1.0539(3)	0.046(3)	0.042(2)	0.028(2)	0.001(2)	0.001(2)	0.001(2)
C(2)	4a		0.8478(6)	0.5587(4)	1.0471(4)	0.085(5)	0.041(3)	0.050(3)	–0.004(3)	0.014(3)	–0.011(2)
C(1)	4a		0.3087(4)	0.0735(3)	1.0009(3)	0.035(2)	0.044(2)	0.041(3)	0.005(2)	0.016(2)	0.017(2)
C(25)	4a		0.1700(4)	0.3061(4)	0.7732(3)	0.032(2)	0.046(2)	0.048(3)	0.012(2)	0.010(2)	0.016(2)
C(24)	4a		0.6812(5)	0.3350(4)	0.7291(3)	0.058(3)	0.050(3)	0.034(3)	–0.007(2)	0.003(2)	–0.002(2)
C(23)	4a		0.3398(6)	–0.0081(4)	0.9575(4)	0.071(4)	0.045(3)	0.058(3)	0.023(3)	0.026(3)	0.013(3)
C(22)	4a		0.8059(6)	0.2491(5)	1.0556(4)	0.079(4)	0.061(3)	0.037(3)	–0.015(3)	0.011(3)	0.009(2)
C(21)	4a		0.7251(6)	0.6147(4)	0.8546(4)	0.073(4)	0.038(3)	0.055(3)	0.006(2)	0.013(3)	0.009(2)
C(20)	4a		0.2780(4)	0.3523(4)	0.7861(4)	0.039(3)	0.054(3)	0.067(4)	0.004(2)	0.022(2)	0.024(3)
C(19)	4a		0.4042(5)	0.1291(4)	1.0536(4)	0.049(3)	0.058(3)	0.049(3)	0.003(2)	–0.002(2)	0.014(2)
C(18)	4a		0.5371(4)	0.4383(6)	0.7547(4)	0.028(2)	0.098(5)	0.058(4)	–0.001(3)	0.005(2)	0.022(3)
C(17)	4a		0.1323(5)	0.2509(5)	0.6938(4)	0.042(3)	0.085(4)	0.041(3)	0.015(3)	0.010(2)	0.012(3)
C(26)	4a		0.9770(5)	0.3329(5)	1.0596(5)	0.043(3)	0.068(4)	0.082(5)	0.013(3)	0.004(3)	0.014(4)
O(1)	4a		0.5456(5)	0.5891(5)	0.9707(4)	0.076(4)	0.101(4)	0.060(3)	0.030(3)	0.028(3)	0.007(3)
O(2)	4a	0.50	0.446(2)	0.5599(8)	0.935(1)	0.23(2)	0.042(5)	0.09(1)	0.023(8)	0.07(1)	0.020(6)
O(3)	4a	0.50	0.480(2)	0.561(1)	0.999(2)	0.21(2)	0.10(1)	0.14(2)	0.07(1)	0.03(2)	–0.04(1)

Acknowledgment. Crystal mounting oil (type RS3000) was donated by Riedel de Haën.

References

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