Crystal structure of bis(*p*-nitrobenzoato-κ^1O;κ^2O,O')[bis(2-benzimidazolylmethyl)amine-κ^3N,N',N'']lead (II) — methanol (1:1), $C_{31}H_{27}N_7O_9Pb$

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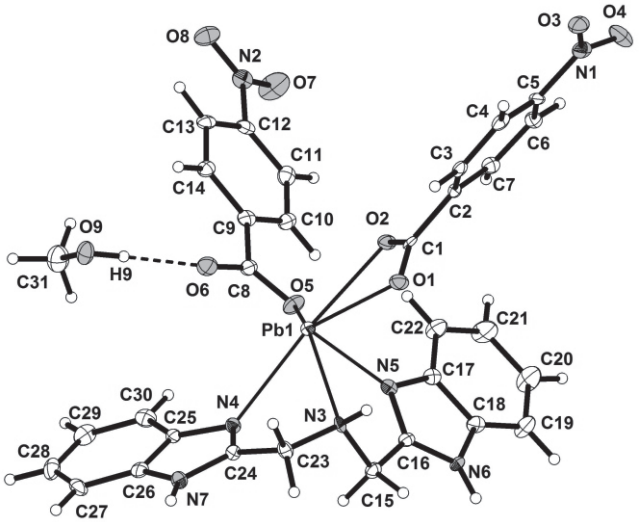

Abstract

$C_{31}H_{27}N_7O_9Pb$, triclinic, $P\bar{1}$ (no. 2), a = 7.5465(3) Å, b = 10.8333(4) Å, c = 19.9801(7) Å, α = 95.476(3)°, β = 94.404(3)°, γ = 108.206(3)°, V = 1534.8 Å^3, Z = 2, $R_{gt}(F)$ = 0.0271, $wR_{ref}(F^2)$ = 0.0480, T = 99 K.

Table 1. Data collection and handling.

Crystal:	colourless needles, size 0.07×0.09×0.25 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	55.65 cm^{-1}
Diffractometer, scan mode:	Xcalibur, Eos, Gemini, ω
$2\theta_{max}$:	52°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	12318, 6027
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 5519
$N(param)_{refined}$:	435
Programs:	SHELX [8]

Source of material

Bis(benzimidazol-2-yl-methyl)amine (*bbma*) was synthesized according to published procedure [1]. The title complex was synthesized by stiring stoichiometric quantities (1:1:1:1 molar ratio) of *bbma* (0.139 g, 0.5 mmol), $Pb(NO_3)_2$ (0.166 g, 0.5 mmol), *p*-nitrobenzoic acid (0.084 g, 0.5 mmol) and triethylamine (2 ml, 0.25 mmol/ml) in a methanol solution (30 mL). After stirring for 2 hours at room temperature, the solution was filtered and allowed to stand at room temperature for slow evaporation. Colourless crystals suitable for X-ray structure determination were formed.

Discussion

The lead(II) complexes with multi-functional organic ligands have attracted considerable attention owing to their unusual structures, and functional properties such as luminescence, adsorption and biological function [2–3]. Lead(II) tends toward the formation of complexes where the central atom bears a combination of O, N, and S-donor ligands complex and coordination numbers can range from two up to ten, and even twelve in rare cases [4–7]. The ligand bis(2-benzimidazolylmethyl)amine (*bbma*) is a tridentate ligand with two aromatic benzimidazole side arms and has widely been used to prepare metal complexes. Lead(II) complexes based on *bbma* haven't been reported until now. In this study, we report the synthesis and crystal structure of a new complex, $[Pb(p\text{-}NO_2C_6H_4COO)_2(C_{16}H_{15}N_5)]\cdot CH_3OH$. The X-ray diffraction analysis reveals that the title complex consists of one $[Pb(p\text{-}NO_2C_6H_4COO)_2(C_{16}H_{15}N_5)]$ unit and a lattice methanol molecule. In the mononuclear complex, the lead(II) is hexa-coordinated showing a N_3O_3 donor set with three N atoms from the tridentate ligand *bbma* and three O atoms from two *p*-nitrobenzoato ligands. One *p*-nitrobenzoato ligand offers two carboxylate O atoms in bidentate coordination mode, while the other is in mono-dentate coordination mode. The coordination geometry around Pb(II) ion is pentagonal pyramidal. The axial coordination site is occupied by the benzimidazolic-N5 atom of *bbma*, the Pb1–N5 bond length is 2.367(3) Å. The equatorial plane is defined by O1, O2, O5, N4 and N3, the O1–Pb1–O2, O2–Pb1–O5, O5–Pb1–N4, N4–Pb1–N3 and N3–Pb1–O1 bond angles are 49.55(7)°, 76.25(8)°, 95.30(8)°, 63.06(8)° and 76.12(7)°, respectively. All angles are 360.31° in sum, which shows that O1, O2, O5, N3 and N4 are nearly in the same plane. There are four kinds of hydrogen bonds in the crystal. The unit $[Pb(p\text{-}NO_2C_6H_4COO)_2(C_{16}H_{15}N_5)]$ associated with two adjacent units [N6–H6···O2, d(D···A) = 2.722(4) Å] to form a 1D chain. The methanol molecule acts as an hydrogen bond-donor and hydrogen bond-acceptor towards the adjacent carboxylate oxygen atom and the nitrogen atom in *bbma*, respectively, [O9–H9···O6, d(D···A) = 2.663(4) Å; N7–H7A···O9, d(D···A) = 2.724(4) Å] to form a 2D layer.

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

Atom	Site	x	y	z	U_{iso}
H(9)	2*i*	–0.2671	0.7706	0.0566	0.030
H(6)	2*i*	0.7968	0.7925	0.3330	0.014
H(3)	2*i*	0.3250	0.4684	0.2546	0.014
H(23A)	2*i*	0.2146	0.3666	0.1536	0.016
H(23B)	2*i*	0.4368	0.4197	0.1500	0.016
H(22)	2*i*	0.2448	0.9726	0.3342	0.025
H(19)	2*i*	0.8657	1.0214	0.4267	0.026

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Table 2. continued.

Atom	Site	*x*	*y*	*z*	U_{iso}
H(15A)	2*i*	0.5946	0.6444	0.1964	0.015
H(15B)	2*i*	0.6220	0.5737	0.2615	0.015
H(11)	2*i*	–0.0809	1.2627	0.2774	0.020
H(30)	2*i*	0.1742	0.8406	0.0515	0.021
H(14)	2*i*	–0.4067	0.8617	0.1449	0.017
H(29)	2*i*	0.1258	0.8429	–0.0654	0.025
H(27)	2*i*	0.2263	0.4964	–0.1036	0.021
H(13)	2*i*	–0.5634	1.0113	0.1739	0.017
H(10)	2*i*	0.0711	1.1075	0.2523	0.019
H(28)	2*i*	0.1511	0.6739	–0.1415	0.026

Table 2. continued.

Atom	Site	*x*	*y*	*z*	U_{iso}
H(21)	2*i*	0.4112	1.1477	0.4149	0.032
H(3A)	2*i*	–0.2052	0.3985	0.4075	0.016
H(20)	2*i*	0.7130	1.1706	0.4627	0.034
H(4)	2*i*	–0.3449	0.3695	0.5079	0.017
H(6A)	2*i*	–0.1513	0.7622	0.5639	0.017
H(7)	2*i*	–0.0298	0.7920	0.4598	0.017
H(31A)	2*i*	–0.3694	0.8417	–0.0597	0.044
H(31B)	2*i*	–0.1743	0.9127	–0.0135	0.044
H(31C)	2*i*	–0.3660	0.9222	0.0121	0.044
H(7A)	2*i*	0.3077	0.4054	0.0195	0.016

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}
Pb(1)	2*i*	0.10487(2)	0.65857(1)	0.221864(7)	0.01010(7)	0.01409(8)	0.01054(8)	0.00524(5)	0.00143(5)	0.00223(5)
O(2)	2*i*	–0.0064(3)	0.7236(2)	0.3348(1)	0.013(1)	0.017(2)	0.016(2)	0.007(1)	0.005(1)	0.007(1)
O(1)	2*i*	0.0483(3)	0.5330(2)	0.3343(1)	0.016(1)	0.020(2)	0.017(2)	0.010(1)	0.005(1)	0.005(1)
O(6)	2*i*	–0.1426(3)	0.7696(2)	0.1394(1)	0.019(1)	0.020(2)	0.021(2)	0.009(1)	0.002(1)	–0.002(1)
O(9)	2*i*	–0.3296(4)	0.7508(2)	0.0183(1)	0.023(2)	0.017(2)	0.018(2)	0.007(1)	–0.005(1)	–0.003(1)
N(4)	2*i*	0.2632(4)	0.6380(3)	0.1092(2)	0.013(2)	0.012(2)	0.011(2)	0.005(1)	0.002(1)	0.002(1)
O(5)	2*i*	0.1013(3)	0.8858(2)	0.2139(1)	0.015(1)	0.018(2)	0.026(2)	0.007(1)	–0.000(1)	0.007(1)
N(6)	2*i*	0.6888(4)	0.8072(3)	0.3264(2)	0.009(1)	0.016(2)	0.012(2)	0.007(1)	–0.001(1)	0.002(1)
O(4)	2*i*	–0.2969(4)	0.6408(3)	0.6524(1)	0.037(2)	0.027(2)	0.019(2)	0.017(1)	0.013(1)	0.005(1)
N(3)	2*i*	0.3499(4)	0.5246(3)	0.2216(1)	0.014(2)	0.016(2)	0.006(2)	0.004(1)	0.002(1)	0.003(1)
O(8)	2*i*	–0.5969(4)	1.2160(3)	0.2194(1)	0.019(2)	0.023(2)	0.034(2)	0.011(1)	0.004(1)	0.007(1)
N(5)	2*i*	0.3975(4)	0.7811(3)	0.2838(2)	0.015(2)	0.009(2)	0.012(2)	0.006(1)	0.004(1)	0.004(1)
N(2)	2*i*	–0.4304(4)	1.2461(3)	0.2401(2)	0.020(2)	0.017(2)	0.019(2)	0.008(2)	0.005(2)	0.004(2)
C(23)	2*i*	0.3246(5)	0.4467(3)	0.1559(2)	0.018(2)	0.012(2)	0.011(2)	0.006(2)	0.002(2)	–0.000(2)
C(26)	2*i*	0.2438(5)	0.5657(4)	–0.0030(2)	0.008(2)	0.016(2)	0.016(2)	–0.000(2)	0.002(2)	0.005(2)
C(2)	2*i*	–0.1037(5)	0.5981(3)	0.4248(2)	0.011(2)	0.018(2)	0.009(2)	0.008(2)	0.001(2)	0.003(2)
O(7)	2*i*	–0.3377(4)	1.3544(3)	0.2693(2)	0.032(2)	0.020(2)	0.054(2)	0.013(1)	–0.004(2)	–0.010(2)
C(24)	2*i*	0.2954(5)	0.5245(3)	0.1005(2)	0.011(2)	0.013(2)	0.011(2)	0.003(2)	0.001(2)	0.001(2)
C(22)	2*i*	0.3691(5)	0.9822(4)	0.3524(2)	0.016(2)	0.019(2)	0.026(2)	0.008(2)	–0.001(2)	0.001(2)
C(19)	2*i*	0.7417(5)	1.0118(4)	0.4081(2)	0.016(2)	0.021(2)	0.027(3)	0.007(2)	–0.006(2)	–0.001(2)
C(15)	2*i*	0.5415(5)	0.6172(3)	0.2385(2)	0.012(2)	0.014(2)	0.014(2)	0.006(2)	0.001(2)	0.003(2)
C(11)	2*i*	–0.1461(5)	1.1809(4)	0.2511(2)	0.018(2)	0.016(2)	0.016(2)	0.005(2)	0.002(2)	0.005(2)
C(17)	2*i*	0.4588(5)	0.8925(3)	0.3313(2)	0.013(2)	0.011(2)	0.015(2)	0.004(2)	0.003(2)	0.004(2)
C(30)	2*i*	0.1845(5)	0.7715(4)	0.0206(2)	0.015(2)	0.014(2)	0.021(2)	0.001(2)	0.005(2)	0.003(2)
C(14)	2*i*	–0.3414(5)	0.9422(4)	0.1724(2)	0.012(2)	0.014(2)	0.016(2)	0.002(2)	0.003(2)	0.004(2)
C(29)	2*i*	0.1562(5)	0.7721(4)	–0.0485(2)	0.017(2)	0.018(2)	0.027(2)	0.003(2)	0.003(2)	0.015(2)
C(27)	2*i*	0.2157(5)	0.5653(4)	–0.0727(2)	0.015(2)	0.020(2)	0.013(2)	–0.001(2)	0.006(2)	0.000(2)
C(25)	2*i*	0.2285(5)	0.6663(4)	0.0434(2)	0.011(2)	0.018(2)	0.011(2)	0.006(2)	0.002(2)	0.005(2)
C(16)	2*i*	0.5403(5)	0.7343(3)	0.2832(2)	0.009(2)	0.015(2)	0.013(2)	0.004(2)	0.003(2)	0.008(2)
C(13)	2*i*	–0.4337(5)	1.0311(3)	0.1886(2)	0.012(2)	0.018(2)	0.017(2)	0.006(2)	0.007(2)	0.010(2)
C(10)	2*i*	–0.0570(5)	1.0892(3)	0.2357(2)	0.013(2)	0.019(2)	0.017(2)	0.006(2)	0.003(2)	0.009(2)
C(12)	2*i*	–0.3341(5)	1.1487(4)	0.2266(2)	0.016(2)	0.017(2)	0.014(2)	0.010(2)	0.008(2)	0.007(2)
O(3)	2*i*	–0.4458(3)	0.4365(3)	0.6146(1)	0.014(1)	0.031(2)	0.022(2)	0.006(1)	0.006(1)	0.012(1)
C(28)	2*i*	0.1716(5)	0.6703(4)	–0.0943(2)	0.015(2)	0.027(2)	0.016(2)	–0.003(2)	–0.002(2)	0.007(2)
C(21)	2*i*	0.4681(5)	1.0845(4)	0.4003(2)	0.025(2)	0.021(2)	0.036(3)	0.014(2)	0.000(2)	–0.009(2)
C(9)	2*i*	–0.1535(5)	0.9703(3)	0.1960(2)	0.014(2)	0.014(2)	0.015(2)	0.004(2)	0.005(2)	0.009(2)
C(8)	2*i*	–0.0559(5)	0.8689(4)	0.1811(2)	0.012(2)	0.021(2)	0.014(2)	0.006(2)	0.003(2)	0.009(2)
C(3)	2*i*	–0.1970(5)	0.4723(3)	0.4388(2)	0.011(2)	0.014(2)	0.014(2)	0.008(2)	–0.002(2)	–0.000(2)
C(20)	2*i*	0.6508(5)	1.0993(4)	0.4287(2)	0.026(2)	0.019(2)	0.036(3)	0.009(2)	–0.008(2)	–0.013(2)
N(1)	2*i*	–0.3392(4)	0.5458(3)	0.6080(2)	0.016(2)	0.026(2)	0.019(2)	0.013(2)	0.004(2)	0.012(2)
C(4)	2*i*	–0.2775(5)	0.4548(4)	0.4984(2)	0.010(2)	0.014(2)	0.019(2)	0.004(2)	–0.001(2)	0.006(2)
C(6)	2*i*	–0.1644(5)	0.6892(4)	0.5313(2)	0.015(2)	0.014(2)	0.014(2)	0.007(2)	0.002(2)	0.002(2)
C(18)	2*i*	0.6423(5)	0.9100(4)	0.3591(2)	0.016(2)	0.014(2)	0.017(2)	0.008(2)	0.004(2)	0.006(2)
C(1)	2*i*	–0.0143(5)	0.6177(4)	0.3597(2)	0.004(2)	0.022(2)	0.013(2)	0.004(2)	–0.002(2)	0.003(2)
C(7)	2*i*	–0.0901(5)	0.7062(4)	0.4704(2)	0.013(2)	0.014(2)	0.016(2)	0.005(2)	0.002(2)	0.004(2)
C(5)	2*i*	–0.2583(5)	0.5629(4)	0.5436(2)	0.010(2)	0.024(2)	0.011(2)	0.010(2)	0.005(2)	0.007(2)
C(31)	2*i*	–0.3081(6)	0.8657(4)	–0.0131(2)	0.034(2)	0.028(3)	0.026(3)	0.013(2)	–0.001(2)	0.004(2)
N(7)	2*i*	0.2879(4)	0.4782(3)	0.0350(2)	0.015(2)	0.013(2)	0.013(2)	0.004(1)	0.001(1)	–0.000(1)

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