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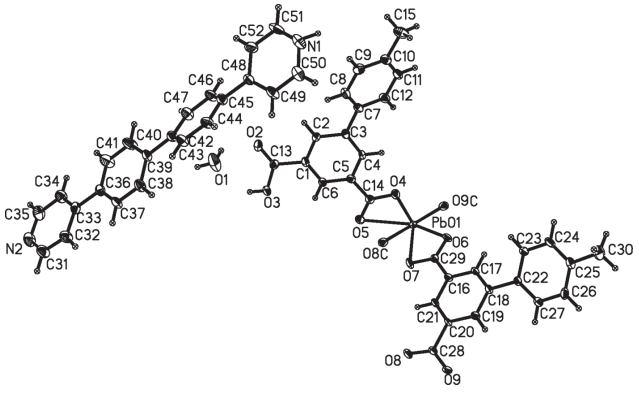
Crystal structure of 4-(4′-(pyridin-4-yl)-[1,1′-biphenyl]-4-yl)pyridin-1-ium *catena*-poly[{5-carboxy-4′-methyl-[1,1′-biphenyl]-3-carboxylato-κ^2*O*,*O*′}-(μ$_2$-4′-methyl-[1,1′-biphenyl]-3,5-dicarboxylato-κ^4*O*,*O*′:*O*″,*O*‴)lead(II)], $C_{52}H_{40}N_2O_9Pb$



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Abstract

$C_{52}H_{40}N_2O_9Pb$, triclinic, $P\bar{1}$ (no. 2), $a = 10.3025(4)$ Å, $b = 14.1740(9)$ Å, $c = 16.0428(9)$ Å, $\alpha = 115.835(6)°$, $\beta = 97.680(4)°$, $\gamma = 90.493(4)°$, $V = 2083.7(2)$ Å^3, $Z = 2$, $R_{gt}(F) = 0.0342$, $wR_{ref}(F^2) = 0.0612$, $T = 293(2)$ K.

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

Source of material

A mixture of $Pb(NO_3)_2{\cdot}2H_2O$ (0.1 mmol, 0.036 g), 3′,5′-dicarboxyl-4-methyl biphenyl (0.1 mmol, 0.026 g), 4,4′-bis(pyrid-4-yl)biphenyl (0.06 g, 0.2 mmol) and distilled water (10 mL) was heated in a 25 mL stainless steel reactor with a Teflon liner at 423 K for 37 h, followed by slow cooling to room temperature. Colourless crystals of the title compound formed.

Table 1: Data collection and handling.

Crystal:	Block, colourless
Size:	0.47 × 0.38 × 0.27 mm
Wavelength:	Mo $K\alpha$ radiation (0.71073 Å)
μ:	4.11 mm^{-1}
Diffractometer, scan mode:	Multiwire detector, φ and ω-scans
$2\theta_{max}$, completeness:	25.5°, >97%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	12074, 6798, 0.031
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 6185
$N(param)_{refined}$:	583
Programs:	SHELX [1]

Experimental details

C-bound hydrogen atoms were placed in calculated positions and were included in the refinement in the riding model approximation, with U_{iso}(H) set to 1.2U_{eq}(C). The hydrogen atom of the hydroxyl group was allowed to rotate fixed the C—O bond to best fit the experimental electron density, with U_{iso}(H) set to 1.5U_{eq}(O).

Discussion

The sytheses of coordination polymers (CPs) attract much attention because of their structural topologies and applications in gas storage, separation, sensing and heterogeneous catalysis Z. Kristallogr. NCS 2016; 231(3): 1003–1006 [2–7]. Multidentate organic ligands play a vital role since the structural integrity of these ligands in most cases remains unaltered throughout the formation of the CP. In particular, various conformations of the ligand system may play an important role in the self-assembly processes. The organic carboxylate ligands have been proven to be good candidates for the construction of CPs [8–10]. For example, polycarboxylates present versatile coordination modes and have been widely utilized to construct coordination polymers [11].

The asymmetric unit of the title structure contains one Pb(II) metal center, the organic cation 4-(4′-(pyridin-4-yl)-[1,1′-biphenyl]-4-yl)pyridin-1-ium, the mono anion 5-carboxy-4′-methyl-[1,1′-biphenyl]-3-carboxylate, the dianion

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

Atom	*x*	*y*	*z*	U_{iso}*/U_{eq}
Pb01	0.108278(14)	0.864722(13)	0.507989(11)	0.02419(6)
O1	0.2316(4)	0.2233(3)	0.7225(4)	0.0900(19)
H1A	0.1528	0.2395	0.7164	0.135*
H1B	0.2336	0.1568	0.6951	0.135*
O2	0.0274(4)	0.3456(3)	0.6748(2)	0.0501(10)
O3	0.1462(3)	0.3550(3)	0.5716(2)	0.0461(10)
H007	0.1508	0.2919	0.5560	0.069*
O4	0.1074(3)	0.8225(3)	0.6325(2)	0.0420(9)
O5	0.1486(3)	0.6728(3)	0.5179(2)	0.0403(9)
O6	0.2760(3)	1.0049(2)	0.6096(2)	0.0353(9)
O7	0.3637(3)	0.8570(2)	0.5301(2)	0.0393(10)
O8	0.8360(3)	0.8502(3)	0.5157(2)	0.0418(10)
O9	0.9667(3)	0.9967(2)	0.5929(2)	0.0318(8)
N1	0.4307(5)	0.6767(4)	1.3980(3)	0.0550(15)
H1	0.4205	0.7345	1.4453	0.066*
N2	0.6241(5)	−0.3930(4)	0.5609(3)	0.0564(15)
C1	0.0708(4)	0.5117(4)	0.6748(3)	0.0296(12)
C2	0.0315(4)	0.5686(4)	0.7627(3)	0.0296(11)
C3	0.0299(4)	0.6790(4)	0.7994(3)	0.0287(12)
C4	0.0605(4)	0.7258(4)	0.7448(3)	0.0281(11)
H00J	0.0576	0.7983	0.7677	0.034*
C5	0.0958(4)	0.6689(4)	0.6561(3)	0.0281(12)
C6	0.1024(4)	0.5612(4)	0.6226(3)	0.0293(12)
H00F	0.1283	0.5224	0.5645	0.035*
C7	−0.0035(4)	0.7425(4)	0.8958(3)	0.0301(12)
C8	0.0292(4)	0.7148(4)	0.9676(3)	0.0373(13)
H00M	0.0695	0.6528	0.9558	0.045*
C9	0.0031(5)	0.7778(4)	1.0574(3)	0.0419(14)
H00U	0.0252	0.7570	1.1047	0.050*
C10	−0.0555(4)	0.8715(4)	1.0772(3)	0.0363(13)
C11	−0.0901(5)	0.8981(4)	1.0058(3)	0.0404(14)
H015	−0.1325	0.9593	1.0173	0.048*
C12	−0.0634(5)	0.8357(4)	0.9159(3)	0.0411(14)
H00N	−0.0860	0.8567	0.8688	0.049*
C13	0.0763(4)	0.3955(4)	0.6418(3)	0.0333(12)
C14	0.1211(4)	0.7240(4)	0.5982(3)	0.0295(12)
C15	−0.0820(6)	0.9414(5)	1.1762(3)	0.064(2)
H01A	−0.1172	0.8987	1.2019	0.097*
H01B	−0.1439	0.9909	1.1742	0.097*
H01C	−0.0014	0.9787	1.2148	0.097*
C16	0.5104(4)	1.0093(4)	0.6203(3)	0.0253(11)
C17	0.5323(4)	1.1123(3)	0.6899(3)	0.0266(11)
H00H	0.4609	1.1484	0.7144	0.032*
C18	0.6569(4)	1.1628(3)	0.7238(3)	0.0252(11)
C19	0.7624(4)	1.1085(4)	0.6831(3)	0.0275(11)
H00K	0.8464	1.1420	0.7024	0.033*
C20	0.7428(4)	1.0046(3)	0.6139(3)	0.0230(11)
C21	0.6185(4)	0.9556(4)	0.5835(3)	0.0264(11)
H00B	0.6059	0.8861	0.5381	0.032*
C22	0.6808(4)	1.2705(4)	0.8039(3)	0.0263(11)
C23	0.5972(4)	1.3022(4)	0.8716(3)	0.0341(13)
H00Z	0.5230	1.2594	0.8633	0.041*
C24	0.6246(5)	1.3978(4)	0.9515(3)	0.0399(14)
H00E	0.5683	1.4175	0.9961	0.048*
C25	0.7323(5)	1.4634(4)	0.9660(3)	0.0381(14)
C26	0.8130(4)	1.4335(4)	0.8969(3)	0.0396(14)
H01D	0.8850	1.4779	0.9042	0.047*
C27	0.7869(4)	1.3384(4)	0.8173(3)	0.0339(13)
H01E	0.8421	1.3199	0.7720	0.041*
C28	0.8577(4)	0.9454(4)	0.5710(3)	0.0257(11)
C29	0.3761(4)	0.9547(4)	0.5849(3)	0.0276(11)
C30	0.7644(5)	1.5635(4)	1.0575(4)	0.0587(19)
H01F	0.6845	1.5949	1.0763	0.088*
H01G	0.8200	1.6119	1.0487	0.088*
H01H	0.8087	1.5466	1.1053	0.088*
C31	0.7237(6)	−0.3210(5)	0.6068(4)	0.0570(18)
H01R	0.8057	−0.3368	0.5882	0.068*
C32	0.7134(5)	−0.2238(4)	0.6805(3)	0.0491(16)
C33	0.5928(5)	−0.1973(4)	0.7096(3)	0.0356(13)
C34	0.4898(5)	−0.2715(4)	0.6613(4)	0.0490(17)
H014	0.4061	−0.2579	0.6774	0.059*
C35	0.5100(6)	−0.3668(5)	0.5886(4)	0.0563(18)
H01I	0.4380	−0.4152	0.5574	0.068*
C36	0.5753(4)	−0.0942(4)	0.7896(3)	0.0338(13)
C37	0.6776(5)	−0.0176(4)	0.8349(3)	0.0417(15)
H01V	0.7590	−0.0299	0.8147	0.050*
C38	0.6618(4)	0.0768(4)	0.9096(3)	0.0425(15)
H01M	0.7325	0.1270	0.9383	0.051*
C39	0.5429(4)	0.0980(4)	0.9426(3)	0.0345(13)
C40	0.4403(5)	0.0215(5)	0.8969(4)	0.061(2)
H01W	0.3587	0.0335	0.9168	0.073*
C41	0.4572(5)	−0.0731(5)	0.8217(4)	0.062(2)
H01T	0.3865	−0.1233	0.7924	0.075*
C42	0.5226(4)	0.1984(4)	1.0221(3)	0.0344(13)
C43	0.6262(4)	0.2588(4)	1.0915(3)	0.0410(15)
H012	0.7097	0.2343	1.0883	0.049*
C44	0.6088(4)	0.3533(4)	1.1645(3)	0.0391(14)
H01O	0.6804	0.3921	1.2088	0.047*
C45	0.4844(4)	0.3912(4)	1.1724(3)	0.0358(13)
H010	0.0068	0.5340	0.7965	0.035*
C46	0.3816(4)	0.3323(4)	1.1044(3)	0.0435(16)
H01J	0.2978	0.3561	1.1083	0.052*
C47	0.4005(4)	0.2383(4)	1.0303(4)	0.0441(16)
H01K	0.3293	0.2011	0.9847	0.053*
C48	0.4645(5)	0.4928(4)	1.2511(3)	0.0347(13)
C49	0.5572(5)	0.5776(4)	1.2854(3)	0.0474(17)
H01P	0.6324	0.5713	1.2577	0.057*
C50	0.5397(5)	0.6703(5)	1.3593(4)	0.0520(17)
H01X	0.6017	0.7273	1.3821	0.062*
C51	0.3384(6)	0.5996(5)	1.3675(4)	0.0554(19)
H01N	0.2635	0.6089	1.3960	0.066*
C52	0.3531(5)	0.5054(5)	1.2934(3)	0.0478(16)
H01L	0.2886	0.4503	1.2719	0.057*
H01S	0.7871	−0.1767	0.7102	0.059*

4′-methyl-[1,1′-biphenyl]-3,5-dicarboxylate and one water molecule. The lead atom is six-coordinated by six oxygen atoms from the two different carboxylato ligands. The Pb—O bond lengths range from 2.323(4) to 2.617(3) Å, respectively. The bond angles of O—Pb—O are in the range of 51.47(8)°

to 132.08(9)°. All other bond lengths and angles are in the expected ranges for such a compound [12]. All moieties are connected by hydrogen bonds d(O(1)···O(6)A) = 2.903(5) Å, d(O(1)···O(2)) = 2.967(6) Å, d(O(3)···O(8)B) = 2.642(4) Å. Symmetry codes: A x, $y-1$, z ; B $-x+1$, $-y+1$, $-z+1$.

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