# Crystal structure of hexaaqua-bis(1,1′-biphenyl-2,2′,6,6′-tetracarboxylic acid)cadmium(II) tetrahydrate, $[Cd(H_2O)_6](C_{16}H_9O_8)_2 \cdot 4H_2O$

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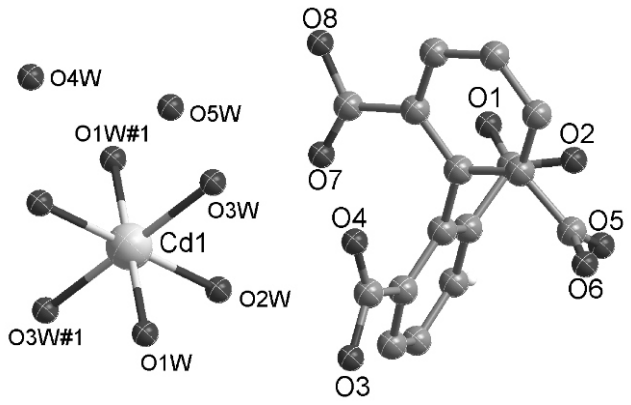


**Abstract**

$C_{32}H_{38}CdO_{26}$, triclinic, $P\bar{1}$ (no. 2), $a$ = 8.061(1) Å, $b$ = 10.857(1) Å, $c$ = 12.289(2) Å, $\alpha$ = 65.374(2)°, $\beta$ = 87.066(3)°, $\gamma$ = 76.325(2)°, $V$ = 948.6 Å$^3$, $Z$ = 1, $R_{gt}(F)$ = 0.038, $wR_{ref}(F^2)$ = 0.111, $T$ = 296 K.

**Source of material**

All chemicals and solvents used in the synthesis were of reagent grade and were used without further purification. $H_4$BPTC ($H_4$BPTC =1,1′-biphenyl-2,2′,6,6′-tetracarboxylic acid) was prepared according to the methods reported previously [1]. A mixture of $Cd(NO_3)_2 \cdot 4H_2O$ (15.42 mg, 0.05 mmol), $H_4$BPTC (33 mg, 0.1 mmol) and NaOH (4 mg, 0.1 mmol) in 6 mL water was sealed in a 25-mL Teflon-lined stainless-steel autoclave. The autoclave was heated at 393 K for 72 h and subsequently cooled slowly to room temperature. Colorless block-shaped crystals were collected, washed with deionized water and dried in air. Elemental analysis — found: C, 40.15 %; H, 4.12 %; calculated for $CdC_{32}H_{38}O_{26}$: C, 40.40 %; H, 4.03 %.

**Experimental details**

Hydrogen atoms on carbon atoms were generated geometrically and refined as riding atoms with $d$(C—H) = 0.93 Å and $U_{iso}$(H) = 1.2$U_{eq}$(C). The approximate positions of the water H atoms, obtained from a difference Fourier map, were restrained to ideal configuration of the water molecule and fixed in the final stages of refinement with $d$(O—H) = 0.85 Å.

**Discussion**

Recently, a series of multidimensional metal-organic coordination supramolecular complexes constructed from 1,1′-biphenyl-2,2′,3,3′-tetracarboxylic acid ($H_4$bptc) [2,3] and 1,1′-biphenyl-2,3′,3,4′-tetracarboxylic acid (*m*-$H_4$bptc) ligands have been reported [4,5]. $H_4$bptc and *m*-$H_4$bptc has proved to be a good candidate for the construction of distinct coordination polymers with high-dimensional networks and interesting properties because of its diverse coordination modes and high structural stability. However, to the best of our knowledge, only a few metal-organic compounds have been obtained with a similar ligand 1,1′-biphenyl-2,2′,6,6′-tetracarboxylic acid ($H_4$BPTC) [6,7]. With the aim of further understanding the coordination chemistry of the $H_4$BPTC ligand with various metal ions, we have focussed on research on coordination polymers based on $H_4$BPTC ligand.

The title compound is a ion-pair complex. The crystal structure contains one Cd(II) cation, two $H_3BPTC^-$ anions, six coordinated and four solvent water molecules in the asymmetric unit. The Cd(II) ion displays a centrosymmetrical $O_6$ octahedral coordination with six oxygen atoms from six coordinated water molecules with $d$(Cd—O) from 2.254(2) to 2.293(3) Å. The related bond distances and bond angles are all symmetrically equivalent. As to the $H_3BPTC^-$ anion, the two phenyl rings are twisted and almost perpendicular to each other (dihedral angle 87.9°). The four carboxylate groups (2-, 6-, 2′- and 6′-$COO^-$) form dihedral angles of 69.8°, 23.0°, 61.3°, and 11.3° with the planes of corresponding phenyl rings. The four carboxylate groups of $H_3BPTC^-$ anion are all free and uncoordinated to Cd(II) center, but they are connected with each other via substantial O–H···O interactions to form an $H_3BPTC^-$ chain anion. The $[Cd(H_2O)_6]^{2+}$ cations and the solvent water molecules fill in the space between adjacent anion chains with hydrogen bonding interactions to give a layer structure, in which the rich hydrogen bonds exist not only between coordinated water and solvent water molecules but also between coordinated or solvent water molecules and the carboxylate oxygen atoms of $H_3BPTC^-$ anions to further form a 3D metal-organic framework.

**Table 1.** Data collection and handling.

| | |
|---|---|
| Crystal: | colorless block, size 0.15 × 0.16 × 0.18 mm |
| Wavelength: | Mo $K_\alpha$ radiation (0.71073 Å) |
| $\mu$: | 6.76 cm$^{-1}$ |
| Diffractometer, scan mode: | Bruker Smart APEX II CCD, $\omega$ |
| $2\theta_{max}$: | 49.98° |
| $N(hkl)_{measured}$, $N(hkl)_{unique}$: | 5229, 3336 |
| Criterion for $I_{obs}$, $N(hkl)_{gt}$: | $I_{obs} > 2\,\sigma(I_{obs})$, 2910 |
| $N(param)_{refined}$: | 270 |
| Programs: | SHELXS-97, SHELXL-97 [8], DIAMOND [9] |

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

| Atom | Site | $x$ | $y$ | $z$ | $U_{iso}$ |
|---|---|---|---|---|---|
| H(2) | 2*i* | 1.0970 | 0.0806 | 0.0668 | 0.053 |
| H(3) | 2*i* | 0.5169 | 0.6227 | 0.0974 | 0.071 |
| H(8) | 2*i* | 0.7612 | –0.0020 | 0.5633 | 0.088 |
| H(3A) | 2*i* | 1.3122 | 0.2387 | 0.1981 | 0.035 |
| H(4) | 2*i* | 1.2073 | 0.4724 | 0.1546 | 0.038 |
| H(5) | 2*i* | 0.9182 | 0.5682 | 0.1249 | 0.033 |
| H(12) | 2*i* | 0.6137 | 0.0501 | 0.0193 | 0.040 |
| H(13) | 2*i* | 0.5274 | –0.0880 | 0.2013 | 0.045 |
| H(14) | 2*i* | 0.5992 | –0.0732 | 0.3736 | 0.040 |
| H(1WA) | 2*i* | 0.3186 | 0.7000 | 0.2769 | 0.089 |
| H(1WB) | 2*i* | 0.3327 | 0.7702 | 0.3453 | 0.089 |
| H(2WA) | 2*i* | 0.7776 | 0.4186 | 0.3798 | 0.095 |
| H(2WB) | 2*i* | 0.7964 | 0.5488 | 0.3591 | 0.095 |
| H(3WA) | 2*i* | 0.3227 | 0.3349 | 0.4400 | 0.089 |
| H(3WB) | 2*i* | 0.4630 | 0.3347 | 0.3730 | 0.089 |
| H(4WA) | 2*i* | –0.0377 | 0.3318 | 0.7369 | 0.142 |
| H(4WB) | 2*i* | 0.1244 | 0.2532 | 0.7405 | 0.142 |
| H(5WA) | 2*i* | 0.0575 | 0.2857 | 0.5438 | 0.093 |
| H(5WB) | 2*i* | 0.0959 | 0.2516 | 0.4482 | 0.093 |

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

| Atom | Site | $x$ | $y$ | $z$ | $U_{11}$ | $U_{22}$ | $U_{33}$ | $U_{12}$ | $U_{13}$ | $U_{23}$ |
|---|---|---|---|---|---|---|---|---|---|---|
| O(1) | 2*i* | 1.2250(3) | –0.0388(2) | 0.3097(2) | 0.047(1) | 0.028(1) | 0.028(1) | 0.0001(9) | –0.0065(9) | –0.0093(9) |
| O(2) | 2*i* | 1.1440(2) | 0.0118(2) | 0.1260(2) | 0.041(1) | 0.032(1) | 0.033(1) | 0.0037(8) | –0.0078(9) | –0.0197(9) |
| O(3) | 2*i* | 0.6195(3) | 0.5927(2) | 0.0939(2) | 0.033(1) | 0.032(1) | 0.063(2) | –0.0003(9) | 0.007(1) | –0.011(1) |
| O(4) | 2*i* | 0.5523(3) | 0.3885(2) | 0.1922(2) | 0.028(1) | 0.038(1) | 0.079(2) | –0.0088(9) | 0.011(1) | –0.026(1) |
| O(5) | 2*i* | 0.9711(2) | 0.2002(2) | –0.0526(2) | 0.028(1) | 0.040(1) | 0.028(1) | –0.0094(8) | 0.0034(8) | –0.0158(9) |
| O(6) | 2*i* | 0.7044(2) | 0.3028(2) | –0.1229(2) | 0.036(1) | 0.032(1) | 0.026(1) | –0.0013(8) | –0.0020(8) | –0.0099(9) |
| O(7) | 2*i* | 0.8371(4) | 0.1732(3) | 0.4122(2) | 0.123(2) | 0.083(2) | 0.030(1) | –0.071(2) | 0.014(1) | –0.024(1) |
| O(8) | 2*i* | 0.7382(4) | –0.0134(3) | 0.5045(2) | 0.106(2) | 0.056(2) | 0.025(1) | –0.042(1) | 0.007(1) | –0.015(1) |
| C(1) | 2*i* | 1.1544(3) | 0.0465(3) | 0.2126(3) | 0.023(1) | 0.032(1) | 0.029(2) | –0.007(1) | 0.003(1) | –0.016(1) |
| C(2) | 2*i* | 1.0832(3) | 0.1967(3) | 0.1907(2) | 0.024(1) | 0.027(1) | 0.020(1) | –0.003(1) | 0.002(1) | –0.013(1) |
| C(3) | 2*i* | 1.1952(3) | 0.2778(3) | 0.1851(3) | 0.022(1) | 0.036(1) | 0.032(2) | –0.007(1) | 0.000(1) | –0.018(1) |
| C(4) | 2*i* | 1.1324(3) | 0.4169(3) | 0.1600(3) | 0.032(1) | 0.032(1) | 0.035(2) | –0.015(1) | 0.001(1) | –0.013(1) |
| C(5) | 2*i* | 0.9591(3) | 0.4738(3) | 0.1431(2) | 0.033(1) | 0.024(1) | 0.028(2) | –0.009(1) | 0.005(1) | –0.013(1) |
| C(6) | 2*i* | 0.8442(3) | 0.3932(3) | 0.1525(2) | 0.025(1) | 0.025(1) | 0.021(1) | –0.004(1) | 0.003(1) | –0.011(1) |
| C(7) | 2*i* | 0.6575(3) | 0.4554(3) | 0.1473(3) | 0.029(1) | 0.031(1) | 0.031(2) | –0.007(1) | 0.003(1) | –0.017(1) |
| C(8) | 2*i* | 0.9061(3) | 0.2515(2) | 0.1759(2) | 0.027(1) | 0.024(1) | 0.016(1) | –0.008(1) | 0.002(1) | –0.010(1) |
| C(9) | 2*i* | 0.7922(3) | 0.1568(2) | 0.1848(2) | 0.018(1) | 0.021(1) | 0.028(1) | –0.0012(9) | 0.001(1) | –0.012(1) |
| C(10) | 2*i* | 0.7481(3) | 0.1450(3) | 0.0819(2) | 0.024(1) | 0.029(1) | 0.025(1) | –0.003(1) | –0.002(1) | –0.015(1) |
| C(11) | 2*i* | 0.8112(3) | 0.2225(3) | –0.0392(2) | 0.030(1) | 0.024(1) | 0.027(2) | –0.007(1) | 0.003(1) | –0.017(1) |
| C(12) | 2*i* | 0.6460(3) | 0.0550(3) | 0.0887(3) | 0.034(2) | 0.043(2) | 0.033(2) | –0.015(1) | 0.004(1) | –0.024(1) |
| C(13) | 2*i* | 0.5929(4) | –0.0265(3) | 0.1973(3) | 0.038(2) | 0.042(2) | 0.044(2) | –0.022(1) | 0.009(1) | –0.022(2) |
| C(14) | 2*i* | 0.6359(4) | –0.0178(3) | 0.3004(3) | 0.035(2) | 0.034(1) | 0.032(2) | –0.014(1) | 0.009(1) | –0.012(1) |
| C(15) | 2*i* | 0.7351(3) | 0.0745(3) | 0.2952(2) | 0.022(1) | 0.028(1) | 0.025(1) | –0.005(1) | 0.003(1) | –0.013(1) |
| C(16) | 2*i* | 0.7751(4) | 0.0852(3) | 0.4075(3) | 0.033(1) | 0.033(1) | 0.030(2) | –0.011(1) | 0.006(1) | –0.014(1) |
| Cd(1) | 1*h* | ½ | ½ | ½ | 0.0644(3) | 0.0367(2) | 0.0278(2) | 0.0031(2) | 0.0006(2) | –0.0094(2) |
| O(1W) | 2*i* | 0.3555(4) | 0.6901(2) | 0.3443(2) | 0.137(3) | 0.034(1) | 0.031(1) | 0.020(1) | –0.020(2) | –0.012(1) |
| O(2W) | 2*i* | 0.7323(4) | 0.4923(3) | 0.3894(3) | 0.078(2) | 0.078(2) | 0.056(2) | 0.010(2) | 0.024(2) | –0.021(2) |
| O(3W) | 2*i* | 0.4123(4) | 0.3661(3) | 0.4218(3) | 0.104(2) | 0.077(2) | 0.062(2) | –0.036(2) | 0.032(2) | –0.044(2) |
| O(4W) | 2*i* | 0.0495(5) | 0.3246(5) | 0.6961(3) | 0.094(3) | 0.225(5) | 0.081(3) | –0.069(3) | 0.040(2) | –0.094(3) |
| O(5W) | 2*i* | 0.0845(4) | 0.3168(3) | 0.4712(3) | 0.107(2) | 0.068(2) | 0.058(2) | –0.002(2) | –0.006(2) | –0.036(2) |

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