Bin Xu*, Qing-Lin Yang, Meng Yun, Xiao-Yu Zhang, Yan-Fang Wu and Xiu-Yan Dong

Synthesis and crystal structure of poly[(μ₂-nitrito-κ⁴O₁,O':O',O'')-nitratokO-(μ₂-1,4-bis((1H-imidazol-1-yl)methyl)benzene-κ²N:N')cadmium(II)], C₁₆H₁₄N₆O₆Cd

Table 1: Data collection and handling.

<table>
<thead>
<tr>
<th>Crystal:</th>
<th>Colorless block</th>
</tr>
</thead>
<tbody>
<tr>
<td>Size:</td>
<td>0.19 × 0.18 × 0.13 mm</td>
</tr>
<tr>
<td>Wavelength:</td>
<td>Ga Kα radiation (1.34139 Å)</td>
</tr>
<tr>
<td>μ:</td>
<td>7.09 mm⁻¹</td>
</tr>
<tr>
<td>Diffractometer, scan mode:</td>
<td>Bruker D8 Venture, ϕ and ω-scans</td>
</tr>
<tr>
<td>R_max, completeness:</td>
<td>53.9°, &gt;98%</td>
</tr>
<tr>
<td>N(hkl)_measured, N(hkl)_unique, R_ref</td>
<td>13166, 3109, 0.062</td>
</tr>
<tr>
<td>Criterion for I_obs, R(I)_gt</td>
<td>I_obs &gt; 2σ(I_obs), 2989</td>
</tr>
<tr>
<td>N(param)_refined:</td>
<td>244</td>
</tr>
<tr>
<td>Programs:</td>
<td>Bruker programs [1], SHELX [2], OLEX2 [3]</td>
</tr>
</tbody>
</table>

Table 2: Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²).

<table>
<thead>
<tr>
<th>Atom</th>
<th>x</th>
<th>y</th>
<th>z</th>
<th>Uiso*</th>
<th>Ueq</th>
</tr>
</thead>
<tbody>
<tr>
<td>Cd1</td>
<td>0.52764(2)</td>
<td>0.38396(2)</td>
<td>0.30756(2)</td>
<td>0.03019(14)</td>
<td></td>
</tr>
<tr>
<td>O1</td>
<td>0.8422(2)</td>
<td>0.4108(3)</td>
<td>0.22148(15)</td>
<td>0.05486(6)</td>
<td></td>
</tr>
<tr>
<td>O2</td>
<td>0.7733(2)</td>
<td>0.2711(3)</td>
<td>0.29949(16)</td>
<td>0.06517(1)</td>
<td></td>
</tr>
<tr>
<td>O3</td>
<td>0.66637(16)</td>
<td>0.4534(2)</td>
<td>0.24203(12)</td>
<td>0.0430(4)</td>
<td></td>
</tr>
<tr>
<td>O4</td>
<td>0.50244(19)</td>
<td>0.1582(2)</td>
<td>0.23162(12)</td>
<td>0.0381(4)</td>
<td></td>
</tr>
<tr>
<td>O5</td>
<td>0.40642(19)</td>
<td>0.3261(2)</td>
<td>0.15234(12)</td>
<td>0.0451(5)</td>
<td></td>
</tr>
<tr>
<td>O6</td>
<td>0.3991(2)</td>
<td>0.1034(2)</td>
<td>0.11000(15)</td>
<td>0.0424(6)</td>
<td></td>
</tr>
<tr>
<td>N1</td>
<td>0.6817(2)</td>
<td>0.0746(3)</td>
<td>0.49964(16)</td>
<td>0.0337(5)</td>
<td></td>
</tr>
<tr>
<td>N2</td>
<td>0.58974(19)</td>
<td>0.2501(2)</td>
<td>0.41944(13)</td>
<td>0.0345(5)</td>
<td></td>
</tr>
<tr>
<td>N3</td>
<td>0.7624(2)</td>
<td>0.3769(2)</td>
<td>0.25672(15)</td>
<td>0.0358(6)</td>
<td></td>
</tr>
<tr>
<td>N4</td>
<td>0.4365(2)</td>
<td>0.1971(2)</td>
<td>0.16328(13)</td>
<td>0.0311(4)</td>
<td></td>
</tr>
<tr>
<td>N5</td>
<td>0.3328(2)</td>
<td>0.4021(2)</td>
<td>0.31408(13)</td>
<td>0.0338(5)</td>
<td></td>
</tr>
<tr>
<td>N6</td>
<td>0.16172(19)</td>
<td>0.4862(2)</td>
<td>0.33563(13)</td>
<td>0.0339(5)</td>
<td></td>
</tr>
<tr>
<td>C1</td>
<td>1.0928(2)</td>
<td>0.0670(3)</td>
<td>0.55878(16)</td>
<td>0.0366(6)</td>
<td></td>
</tr>
<tr>
<td>H1</td>
<td>1.1559</td>
<td>0.1133</td>
<td>0.5990</td>
<td>0.044*</td>
<td></td>
</tr>
<tr>
<td>H2</td>
<td>0.9663</td>
<td>0.0641</td>
<td>0.6247</td>
<td>0.046*</td>
<td></td>
</tr>
<tr>
<td>C3</td>
<td>0.8869(2)</td>
<td>−0.0275(3)</td>
<td>0.51526(15)</td>
<td>0.0333(5)</td>
<td></td>
</tr>
<tr>
<td>H3</td>
<td>0.9064</td>
<td>0.0350</td>
<td>0.52929(16)</td>
<td>0.0375(6)</td>
<td></td>
</tr>
<tr>
<td>H4A</td>
<td>0.7249</td>
<td>−0.1385</td>
<td>0.5007</td>
<td>0.045*</td>
<td></td>
</tr>
<tr>
<td>H4B</td>
<td>0.7712</td>
<td>−0.0635</td>
<td>0.5881</td>
<td>0.045*</td>
<td></td>
</tr>
<tr>
<td>C5</td>
<td>0.6727(3)</td>
<td>0.0146(3)</td>
<td>0.43015(18)</td>
<td>0.0402(6)</td>
<td></td>
</tr>
<tr>
<td>H5</td>
<td>0.7201</td>
<td>0.1253</td>
<td>0.3932</td>
<td>0.048*</td>
<td></td>
</tr>
<tr>
<td>C6</td>
<td>0.5985(3)</td>
<td>0.1138(3)</td>
<td>0.5534(2)</td>
<td>0.0386(7)</td>
<td></td>
</tr>
<tr>
<td>H6</td>
<td>0.5829</td>
<td>0.1062</td>
<td>0.5852</td>
<td>0.046*</td>
<td></td>
</tr>
<tr>
<td>C7</td>
<td>0.5428(2)</td>
<td>0.2428(3)</td>
<td>0.48597(15)</td>
<td>0.0391(6)</td>
<td></td>
</tr>
<tr>
<td>H7</td>
<td>0.4808</td>
<td>0.3043</td>
<td>0.4956</td>
<td>0.047*</td>
<td></td>
</tr>
<tr>
<td>C8</td>
<td>0.2771(2)</td>
<td>0.5164(3)</td>
<td>0.33535(16)</td>
<td>0.0373(6)</td>
<td></td>
</tr>
<tr>
<td>H8</td>
<td>0.3147</td>
<td>0.6082</td>
<td>0.3487</td>
<td>0.045*</td>
<td></td>
</tr>
<tr>
<td>C9</td>
<td>0.2457(3)</td>
<td>0.2948(3)</td>
<td>0.29974(17)</td>
<td>0.0379(6)</td>
<td></td>
</tr>
<tr>
<td>H9</td>
<td>0.2581</td>
<td>0.1991</td>
<td>0.2833</td>
<td>0.046*</td>
<td></td>
</tr>
<tr>
<td>C10</td>
<td>0.1405(3)</td>
<td>0.3464(3)</td>
<td>0.31235(18)</td>
<td>0.0401(6)</td>
<td></td>
</tr>
<tr>
<td>H10</td>
<td>0.0663</td>
<td>0.2922</td>
<td>0.3063</td>
<td>0.048*</td>
<td></td>
</tr>
<tr>
<td>C11</td>
<td>0.0749(3)</td>
<td>0.5872(3)</td>
<td>0.3567(2)</td>
<td>0.0426(6)</td>
<td></td>
</tr>
</tbody>
</table>

Received April 30, 2019; accepted June 4, 2019; available online June 19, 2019

Abstract

C₁₆H₁₄CdN₆O₆, monoclinic, P2₁/c (no. 14), a = 11.2983(5) Å, b = 9.2539(4) Å, c = 170604(7) Å, β = 105.398(1)°, Z = 4, V = 1719.69(13) Å³, Rgt(F) = 0.0321, wRref(F²) = 0.1060, T = 193(2) K.

CCDC no.: 1907940

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 materials

All solvents and other reagents were of analytical grade. The method of synthesizing the title compound has been improved [4]. A mixture of cadmium nitrate, 1,4-bis((1H-imidazol-1-yl)methyl)benzene and water was introduced into a 25 mL Teflon reactor autoclave and heated to 130 °C for 3 days. After cooling down to room temperature, colourless block crystals suitable for single crystal X-ray crystallographic analysis were obtained. Elemental analysis-Anal.
imidazole groups have a strong coordination ability, so (1) used to construct MOFs with different topologies [5–7]. Flexible ligands have variety of configurations that can be positions and constrained to ride on their parent atoms.

Experimental details

<table>
<thead>
<tr>
<th>Atom</th>
<th>x</th>
<th>y</th>
<th>z</th>
<th>Uiso/(\text{Ueq})</th>
</tr>
</thead>
<tbody>
<tr>
<td>H11A</td>
<td>0.0012</td>
<td>0.5956</td>
<td>0.3101</td>
<td>0.051*</td>
</tr>
<tr>
<td>H11B</td>
<td>0.1134</td>
<td>0.6839</td>
<td>0.3669</td>
<td>0.051*</td>
</tr>
<tr>
<td>C12</td>
<td>0.0361(2)</td>
<td>0.5397(3)</td>
<td>0.43093(17)</td>
<td>0.0357(6)</td>
</tr>
<tr>
<td>C13</td>
<td>0.1223(2)</td>
<td>0.4932(3)</td>
<td>0.50065(16)</td>
<td>0.0398(6)</td>
</tr>
<tr>
<td>H13</td>
<td>0.2066</td>
<td>0.4889</td>
<td>0.5014</td>
<td>0.048*</td>
</tr>
<tr>
<td>C14</td>
<td>0.0863(2)</td>
<td>0.4532(3)</td>
<td>0.56892(17)</td>
<td>0.0394(6)</td>
</tr>
<tr>
<td>H14</td>
<td>0.1458</td>
<td>0.4206</td>
<td>0.6160</td>
<td>0.047*</td>
</tr>
</tbody>
</table>

calcd. for C_{30}H_{34}CdN_{4}O_{6}: C, 35.42%; H, 2.97%; N, 17.70%; Found: C, 35.26%; H, 2.85%; N, 17.59%.

Discussion

Hydrogen atoms were placed in their geometrically idealized positions and constrained to ride on their parent atoms.

Experimental details

Before us, Li et al. [12] used the same ligand (L) with Cd(NO\(_3\))\(_2\) obtaining a different structure. The structure obtained by Li et al. is a 2D framework, in which the Cd(II) atom is six-coordinated, and the nitrate ions participate in coordination as a counter anion only. The complex we reported is different, forming a three-dimensional (3D) 2-fold interpenetrating structure, in which the Cd(II) atom is seven-coordinated, and the nitrate ions not only acts as a counter anion, but also participates in structural construction as a bridging group.

In detail the title crystal structure shows that the title complex consists of one Cd(II) atom, one L\(_1\) ligand and two nitrate anions. The Cd(II) atom is seven-coordinated with two nitrogen atoms (N2 and N5) from two L\(_1\) ligands and five oxygen atoms (O3, O4, O4\(_\text{a}\), O5 and O6) from two nitrate ions.

In the framework, Cd(II) is bridged by nitrate anions, forming a one-dimensional chain. Adjacent one-dimensional chains are connected by L\(_4\) to form a two-dimensional network structure. The two-dimensional network layer is further connected by L\(_1\) to form a three-dimensional framework. Due to the large pores in the three-dimensional structure, the adjacent three-dimensional structures form one stable double-penetrating three-dimensional framework [13].

References