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Key indicators

Single-crystal X-ray study
 T = 193 K
 Mean $\sigma(\text{C}-\text{C}) = 0.006 \text{ \AA}$
 R factor = 0.027
 wR factor = 0.069
 Data-to-parameter ratio = 8.6

For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.

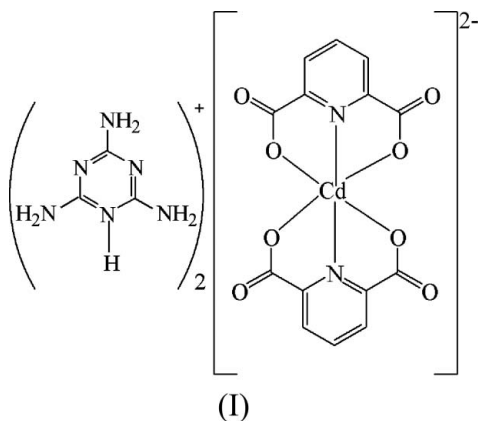
Bis(2,4,6-triamino-1,3,5-triazin-1-ium) bis(pyridine-2,6-dicarboxylato- $\kappa^3\text{O},\text{N},\text{O}'$)cadmate(II)

In the molecule of the title compound, $(\text{C}_3\text{H}_7\text{N}_6)_2\text{[Cd}(\text{C}_7\text{H}_3\text{NO}_4)_2]$, the Cd^{II} atom has a distorted octahedral coordination environment. In the crystal structure, there are some π - π stacking interactions with short distances between parallel aromatic rings, as well as ion-pairing and intramolecular $\text{N}-\text{H}\cdots\text{O}$ and $\text{N}-\text{H}\cdots\text{N}$ hydrogen-bonding interactions involving the 2,4,6-triamino-1,3,5-triazin-1-ium ions and pyridine-2,6-dicarboxylate ligands.

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Comment

We have previously reported some proton-transfer systems, using pyridine-2,6-dicarboxylic acid (pydcH_2), 2,4,6-triamino-1,3,5-triazine (tata), 2,6-pyridinediamine (pyda) and creatinine (creat), which formed the proton-transfer compounds $\text{creatH}(\text{pydcH})$ (Moghimi *et al.*, 2004), $\text{pydaH}(\text{pydcH})$ (Aghabozorg *et al.*, 2005) and $\text{tataH}_2(\text{pydc})$ (Sharif *et al.*, 2006). The crystal structures of some complexes of these systems have been reported (Aghabozorg *et al.*, 2006; Moghimi *et al.*, 2005; Aghajani *et al.*, 2006). We describe here the crystal structure of the title complex, (I), which consists of monomeric units in which the $(\text{tataH})^+$ units act as counterions and the $(\text{pydc})^{2-}$ anions as tridentate ligands.



The geometric parameters of (I) are given in Table 1. The Cd^{II} atom is located at the centre of a distorted octahedral arrangement (Fig. 1). The $\text{O}3-\text{Cd}1-\text{O}5-\text{C}13$ [$-96.2(3)^\circ$], $\text{O}3-\text{Cd}1-\text{O}7-\text{C}14$ [$106.3(3)^\circ$], $\text{O}5-\text{Cd}1-\text{O}1-\text{C}6$ [$106.8(3)^\circ$] and $\text{O}7-\text{Cd}1-\text{O}3-\text{C}7$ [$97.7(3)^\circ$] torsion angles indicate that the two dianionic $(\text{pydc})^{2-}$ units are almost perpendicular to each other.

It is of interest to note that the crystal packing diagrams indicate the layered structure of (I). The space between two layers of $[\text{Cd}(\text{pydc})_2]^{2-}$ complex anions is filled with a layer of $(\text{tataH})^+$ cations. In fact, the layers involving the Cd^{II} complex

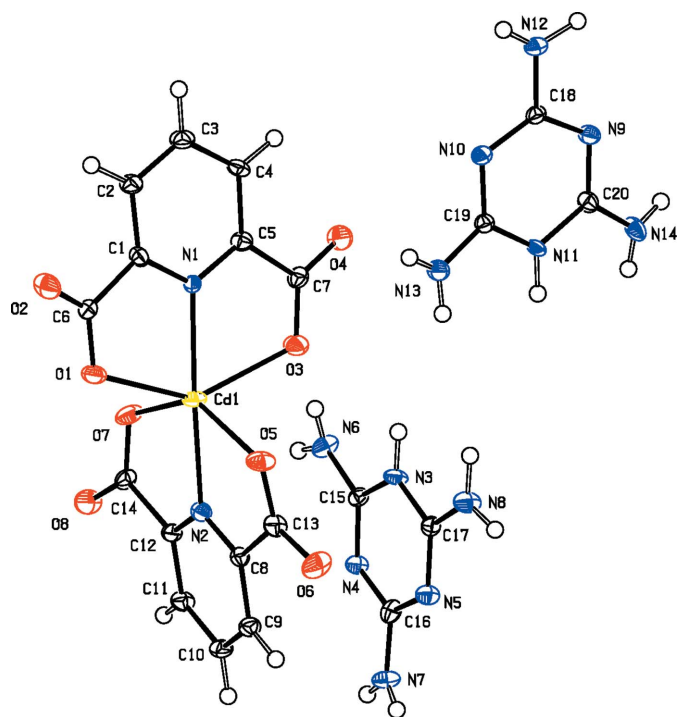


Figure 1
The asymmetric unit, with the atom-numbering scheme. Displacement ellipsoids are drawn at the 50% probability level.

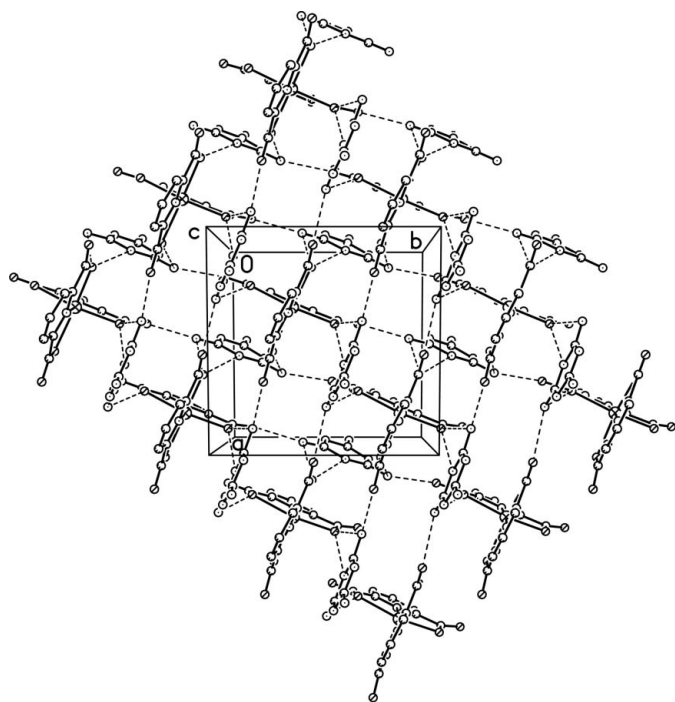


Figure 2
A packing diagram for (I). Hydrogen bonds are shown as dashed lines.

are bridged by (tataH)⁺ counter-ions *via* extensive hydrogen bonds (Fig. 2). Fig. 2 also shows the supramolecular network of the structure. Hydrogen bonds between carboxylate and (tataH)⁺ groups play important roles in stabilizing the crystal structure. The ranges of *D*–H...*A* angles and of the H...*A*

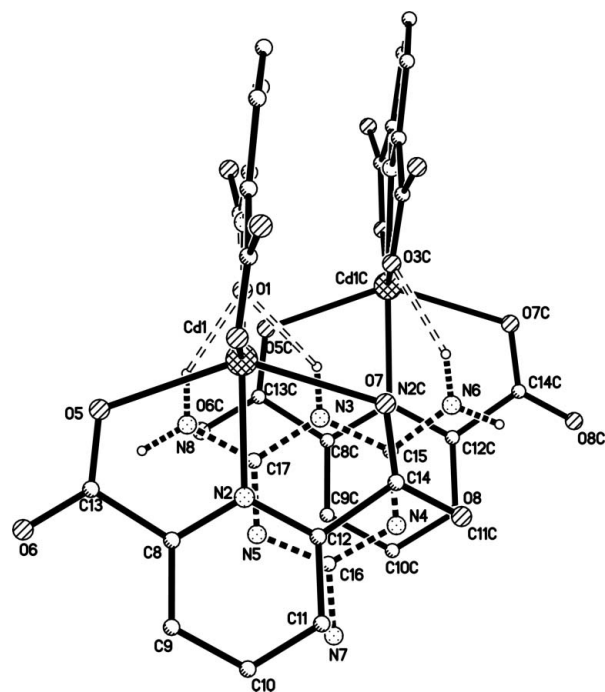


Figure 3
The shortest distances between the π -systems of the cation N3–N5/C15–C17 and the anions N2/C8–C12 and N2C/C8C–C12C. Atoms labelled with the suffix C are generated by the symmetry operator ($x, y - 1, z$). The intercentroid distances between these π -systems are 3.854 (6) and 3.643 (6) Å, respectively. The dihedral angles between the π -systems of the cation and that of the anion are both 9.7 (2)°.

and *D*...*A* distances indicate the presence of strong and weak hydrogen bonding (Table 2).

The short distances between parallel aromatic rings in neighbouring ions indicate π – π stacking interactions. The shortest distances between the π -systems of the cation N3–N5/C15–C17 and the anions N2/C8–C12 and N2C/C8C–C12C [atoms labelled with the suffix C are generated by the symmetry operator ($x, y - 1, z$)] are in the ranges 3.538–3.849 and 3.076–3.550 Å, respectively (Fig. 3). The shortest distances between the π -systems of another cation (N9–N11/C18–C20) and the anions N1/C1–C5 and N1K/C1K–C5K [atoms labelled with the suffix K are generated by the symmetry operator ($x - 1, y, z$)] are in the ranges 3.414–3.870 and 3.068–3.455 Å, respectively (Fig. 4). Therefore, in the formation of this Cd^{II} complex, ion pairing, metal–ligand coordination, hydrogen-bonding and π – π stacking play important roles in the construction of its three-dimensional network.

Experimental

A solution of Cd(NO₃)₂·2H₂O (0.022 g, 0.084 mmol) in water (3 ml) was added to a stirred aqueous solution (10 ml) of LH₂ [(LH₂ is (tataH)₂(pydc), where tata = 2,4,6-triamino-1,3,5-triazine and pydcH₂ = pyridine-2,6-dicarboxylic acid] (0.07 g, 0.168 mmol). Colourless single crystals of (I) suitable for X-ray characterization were collected at room temperature after a few days.

Crystal data

(C₃H₇N₆)₂[Cd(C₇H₃NO₄)₂]
M_r = 696.90
 Monoclinic, *Cc*
a = 10.552 (2) Å
b = 10.5640 (19) Å
c = 22.238 (4) Å
 β = 100.803 (16)°
V = 2434.9 (8) Å³

Z = 4
D_x = 1.901 Mg m⁻³
 Mo *K*α radiation
 μ = 0.98 mm⁻¹
T = 193 (2) K
 Prism, colourless
 0.20 × 0.20 × 0.15 mm

Data collection

Rebuilt Syntex *P*₂₁/Siemens *P*₃
 four-circle diffractometer
 $\omega/2\theta$ scans
 Absorption correction: none
 3581 measured reflections
 3343 independent reflections

3035 reflections with *I* > 2σ(*I*)
*R*_{int} = 0.027
 θ_{\max} = 29.1°
 2 standard reflections
 every 98 reflections
 intensity decay: 3%

Refinement

Refinement on *F*²
R[*F*² > 2σ(*F*²)] = 0.027
wR(*F*²) = 0.069
 S = 1.00
 3343 reflections
 388 parameters
 H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0469P)^2]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} = 0.001$
 $\Delta\rho_{\max} = 1.36 \text{ e \AA}^{-3}$
 $\Delta\rho_{\min} = -0.43 \text{ e \AA}^{-3}$
 Absolute structure: Flack (1983),
 with 137 Friedel pairs
 Flack parameter: 0.01 (2)

Table 1

Selected geometric parameters (Å, °).

Cd1—N2	2.203 (6)	Cd1—O7	2.384 (3)
Cd1—N1	2.202 (5)	Cd1—O5	2.391 (3)
Cd1—O1	2.381 (3)	Cd1—O3	2.394 (3)
N2—Cd1—N1	174.93 (11)	O7—Cd1—O5	143.17 (10)
N1—Cd1—O1	71.80 (13)	N1—Cd1—O3	71.32 (13)
N2—Cd1—O7	71.70 (17)	O1—Cd1—O3	143.06 (10)
O1—Cd1—O7	96.27 (12)	O7—Cd1—O3	95.54 (12)
N2—Cd1—O5	71.53 (16)	O5—Cd1—O3	95.28 (12)
O1—Cd1—O5	95.88 (11)		
O5—Cd1—O1—C6	106.8 (3)	O3—Cd1—O5—C13	−96.2 (3)
O7—Cd1—O3—C7	97.7 (3)	O3—Cd1—O7—C14	106.3 (3)

Table 2

Hydrogen-bond geometry (Å, °).

<i>D</i> — <i>H</i> ··· <i>A</i>	<i>D</i> — <i>H</i>	<i>H</i> ··· <i>A</i>	<i>D</i> ··· <i>A</i>	<i>D</i> — <i>H</i> ··· <i>A</i>
N6—H6 <i>A</i> ···O6 ⁱ	0.85	1.94	2.773 (5)	170
N6—H6 <i>B</i> ···O3	0.79	2.09	2.801 (5)	149
N8—H8 <i>A</i> ···O8 ⁱⁱ	0.85	1.92	2.759 (5)	170
N8—H8 <i>B</i> ···O1 ⁱⁱⁱ	0.90	2.09	2.850 (5)	142
N11—H11···O5 ^{iv}	0.90	2.24	2.954 (6)	136
N12—H12 <i>A</i> ···N4 ^v	0.95	2.28	3.154 (6)	152
N13—H13 <i>A</i> ···O4	0.82	1.96	2.776 (5)	174
N13—H13 <i>B</i> ···O7 ⁱⁱⁱ	0.82	2.10	2.812 (5)	144
N14—H14 <i>A</i> ···O5 ^{iv}	0.78	2.14	2.842 (5)	150
N14—H14 <i>B</i> ···O2 ^{vi}	0.85	1.92	2.755 (5)	166

Symmetry codes: (i) $x + \frac{1}{2}, y + \frac{1}{2}, z$; (ii) $x - 1, y, z$; (iii) $x - \frac{1}{2}, y + \frac{1}{2}, z$; (iv) $x, y + 1, z$; (v) $x - \frac{1}{2}, -y + \frac{3}{2}, z - \frac{1}{2}$; (vi) $x - \frac{1}{2}, y + \frac{3}{2}, z$.

The highest peak in the final difference electron-density map was located 1.23 Å from atom O1. The H atoms of the NH and NH₂ groups were located in difference syntheses and refined as riding atoms, with distance constraints of N—H = 0.87 and 0.90 Å (for NH)

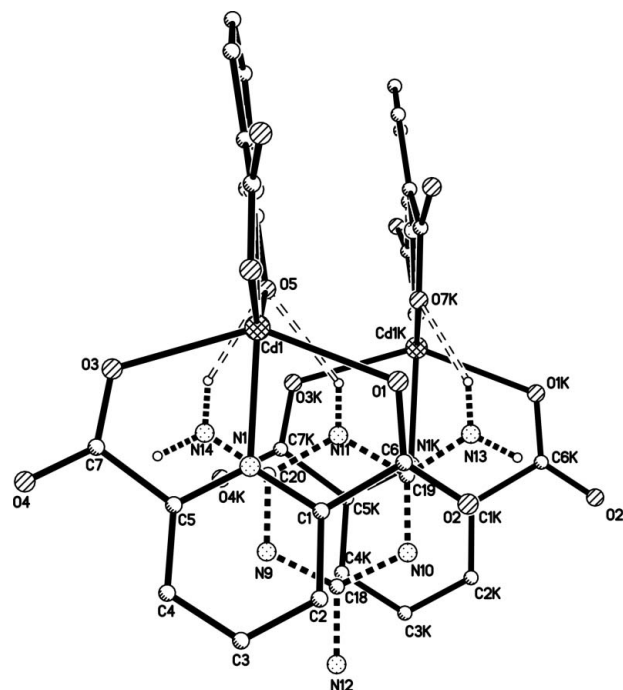


Figure 4

The shortest distances between the π-systems of the cation N9–N11/C18–C20 and the anions N1/C1–C5 and N1K/C1K–C5K. Atoms labelled with the suffix K are generated by the symmetry operator (*x* − 1, *y*, *z*). The intercentroid distances between these π-systems are 4.018 and 3.526 Å, respectively. The dihedral angles between the π-systems of the cation and that of the anion are both 10.0°.

and N—H = 0.78–0.95 Å (for NH₂), and with *U*_{iso}(H) = 1.2*U*_{eq}(N). The remaining H atoms were positioned geometrically, with C—H = 0.95 Å, and constrained to ride on their parent atoms, with *U*_{iso}(H) = 1.2*U*_{eq}(C).

Data collection: *P3/PC* (Siemens, 1989); cell refinement: *P3/PC*; data reduction: *P3/PC*; program(s) used to solve structure: *SHELXTL-Plus* (Sheldrick, 1998); program(s) used to refine structure: *SHELXTL-Plus*; molecular graphics: *SHELXTL-Plus*; software used to prepare material for publication: *SHELXTL-Plus*.

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