

6333 measured reflections  
1640 independent reflections

3 standard reflections  
frequency: 80 min  
intensity decay: <1%

### Refinement

Refinement on  $F^2$   
 $R[F^2 > 2\sigma(F^2)] = 0.051$   
 $wR(F^2) = 0.111$   
 $S = 1.08$   
1640 reflections  
81 parameters  
H atoms treated by a  
mixture of independent  
and constrained refinement

$w = 1/[\sigma^2(F_o^2) + (0.0590P)^2]$   
where  $P = (F_o^2 + 2F_c^2)/3$   
 $(\Delta/\sigma)_{\max} = 0.001$   
 $\Delta\rho_{\max} = 1.31 \text{ e } \text{Å}^{-3}$   
 $\Delta\rho_{\min} = -1.61 \text{ e } \text{Å}^{-3}$   
Extinction correction: none  
Scattering factors from  
*International Tables for  
Crystallography* (Vol. C)

Table 1. Selected bond lengths (Å)

Cu1—N4 <sup>i</sup>	1.933 (7)	Cu3—C3	1.916 (7)
Cu1—C1	2.017 (5)	Cu3—N14 <sup>iii</sup>	2.114 (6)
Cu1...Cu3	2.4008 (13)	Cu3—C1	2.176 (5)
Cu1—S11	2.474 (2)	C1—N2	1.140 (7)
Cu2—N2	1.881 (5)	C3—N4	1.151 (9)
Cu2—S11 <sup>ii</sup>	2.377 (2)		

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

The six highest peaks and the four deepest holes in the final difference density map all had magnitudes greater than 1. All of these peaks and holes were between 0.7 and 1.0 Å from one or another of the Cu atoms. The H atoms were placed in idealized positions, except for the amine hydrogen (H14), for which the positional parameters were refined [N—H = 0.89 (8) Å].

Data collection: *CAD-4 Operations Manual* (Enraf–Nonius, 1977). Cell refinement: *CAD-4 Operations Manual*. Data reduction: *TEXSAN* (Molecular Structure Corporation, 1985). Program(s) used to solve structure: *TEXSAN*. Program(s) used to refine structure: *SHELXTL/PC* (Sheldrick, 1994). Molecular graphics: *SHELXTL/PC* and *TEXSAN*.

We thank Dr Victor G. Young Jr of the X-ray Crystallographic Laboratory at the University of Minnesota for help with the *SHELXTL* system.

Supplementary data for this paper are available from the IUCr electronic archives (Reference: BK1494). Services for accessing these data are described at the back of the journal.

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## [Cd(4,4'-bipy)<sub>2</sub>(NO<sub>3</sub>)<sub>2</sub>](2-nitroaniline)<sub>2</sub>, a novel two-dimensional lattice inclusion compound

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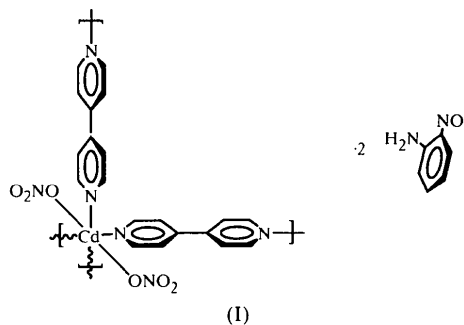
(Received 1 March 1999; accepted 25 August 1999)

### Abstract

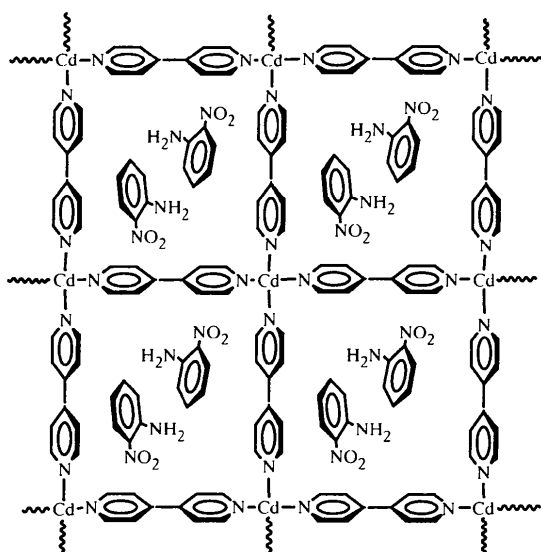
In the title complex, poly[[[bis(nitrato-*O*)cadmium(II)]-bis( $\mu$ -4,4'-bipyridyl-*N:N'*)] bis(2-nitroaniline)], [Cd(NO<sub>3</sub>)<sub>2</sub>(C<sub>10</sub>H<sub>8</sub>N<sub>2</sub>)<sub>2</sub>]<sub>2</sub>·2C<sub>6</sub>H<sub>6</sub>N<sub>2</sub>O<sub>2</sub>, octahedrally coordinated Cd<sup>2+</sup> ions are connected by linear rigid 4,4'-bipyridine ligands to form infinite two-dimensional square grids with the nitrate anions occupying the axial positions. The 2-nitroaniline guest molecules fill the space between the grids through  $\pi$ - $\pi$  stacking interactions. The average Cd—N and Cd—O bond distances are 2.347 (3) and 2.352 (3) Å, respectively.

### Comment

Self-assembled metal coordination polymers have emerged as an important class of materials which show potential applications in the fields of host–guest molecular recognition, heterogeneous catalysis and gas adsorption. As part of our continuing studies of organizing non-linear optical chromophores inside metal coordination polymer hosts, we synthesized a novel two-dimensional lattice inclusion compound with a repeat unit of [Cd(4,4'-bipy)<sub>2</sub>(NO<sub>3</sub>)<sub>2</sub>](2-nitroaniline)<sub>2</sub>, (I) (4,4'-bipy is 4,4'-bipyridyl).



The lattice formed by (I) is shown schematically over and the molecular structure is shown in Fig. 1.



The asymmetric unit of (I) consists of a  $\text{Cd}^{2+}$  cation, two half molecules of 4,4'-bipy, a coordinated nitrate anion and a 2-nitroaniline molecule. The  $\text{Cd}^{2+}$  cation and one 4,4'-bipy fragment are both situated on the twofold rotation axis. Another 4,4'-bipy fragment and the nitrate anion are bound to the  $\text{Cd}^{2+}$  cation perpendicular to this

direction. The octahedral coordination of the metal ion is generated by a combination of the twofold rotation and the cell translation along the  $b$  direction. This also gives rise to a two-dimensional layered structure with  $\text{Cd}^{2+}$ -4,4'-bipy square grids.

The  $\text{NO}_3^-$  groups occupy the axial positions of the metal ions through their O atoms. The 4,4'-bipy ligands and the metal ions form a two-dimensional host network with the 2-nitroaniline guest molecules situated between the networks. Two nitroaniline molecules, related by an inversion center, are closely associated with each grid. The driving force for such inclusion appears to be the  $\pi$ - $\pi$  stacking interactions because the shortest distance between the nitroaniline molecule to one of the pyridine rings is *ca* 3.56 Å.

The layers are stacked in such a fashion that one edge of a square grid runs across the center of a square grid from the next layer. The  $\text{Cd}\cdots\text{Cd}$  separation is 11.7687(7) Å, which coincides with the  $b$ -axis length. The  $\text{Cd}-\text{N}$  bond distances range from 2.341(4) to 2.349(2) Å. The  $\text{Cd}-\text{O}$  bond distance is 2.352(3) Å. These are all comparable with the other similar inclusion compounds reported in the literature (Abrahams *et al.*, 1990; Fujita *et al.*, 1994; Huang & Xiong, 1997; Wang *et al.*, 1999). All other bond distances and angles are in the normal ranges.

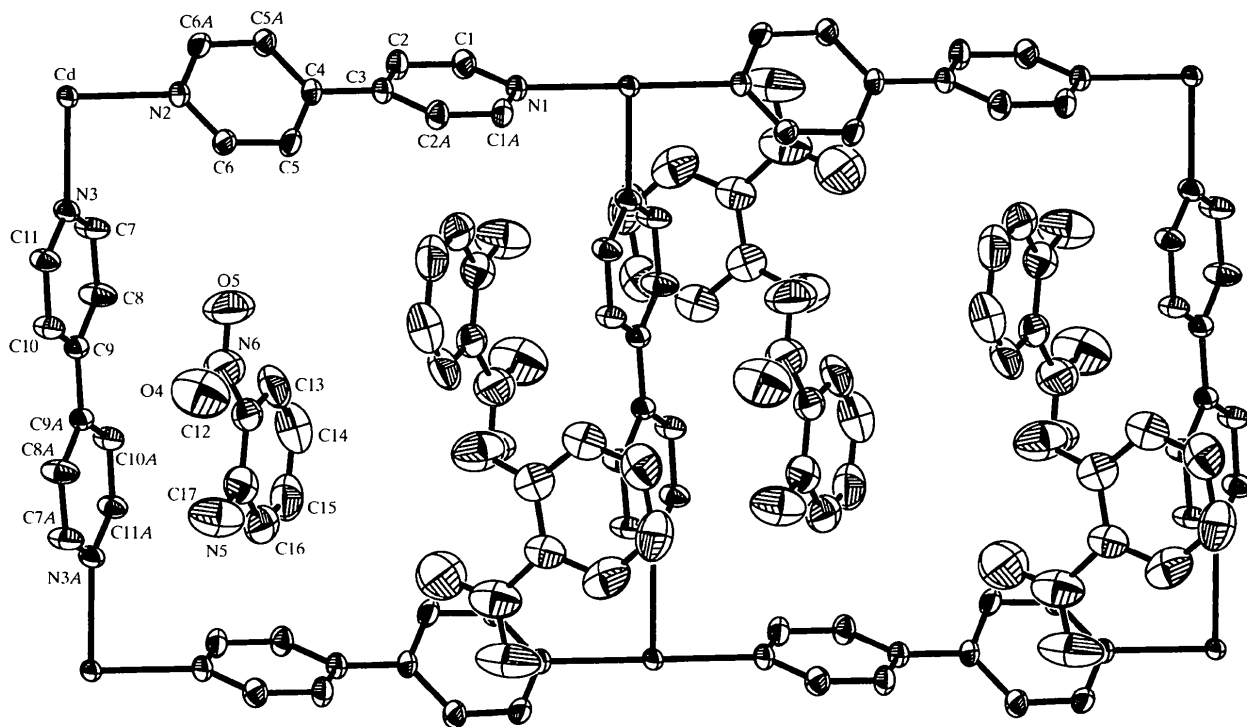


Fig. 1. A view (ORTEPII; Johnson, 1976) of two of the square grids formed by (I) with the associated 2-nitroaniline molecules. The nitrate ligands are not shown. Displacement ellipsoids are shown at the 35% probability level and H atoms have been omitted for clarity.

## Experimental

The title compound was synthesized from Cd(NO<sub>3</sub>)<sub>2</sub>·4H<sub>2</sub>O, 4,4'-bipyridine and 2-nitroaniline in ethanol by a molecular self-assembly reaction. X-ray quality single crystals were obtained by slow evaporation of the solvent.

### Crystal data

[Cd(NO<sub>3</sub>)<sub>2</sub>(C<sub>10</sub>H<sub>8</sub>N<sub>2</sub>)<sub>2</sub>]<sub>2</sub>·  
2C<sub>6</sub>H<sub>6</sub>N<sub>2</sub>O<sub>2</sub>  
M<sub>r</sub> = 825.04  
Monoclinic  
C2/c  
a = 21.850 (2) Å  
b = 11.7687 (7) Å  
c = 13.3256 (8) Å  
β = 100.544 (2)°  
V = 3368.8 (3) Å<sup>3</sup>  
Z = 4  
D<sub>x</sub> = 1.627 Mg m<sup>-3</sup>  
D<sub>m</sub> not measured

Mo Kα radiation

λ = 0.7107 Å

Cell parameters from 1096 reflections

θ = 1.34–27.09°

μ = 0.724 mm<sup>-1</sup>

T = 298.2 K

Prism

0.3 × 0.2 × 0.1 mm

Yellow

### Data collection

Bruker SMART CCD  
diffractometer  
ω scans  
Absorption correction:  
multi-scan (SADABS;  
Sheldrick, 1996)  
T<sub>min</sub> = 0.800, T<sub>max</sub> = 0.930  
9000 measured reflections  
3519 independent reflections

2733 reflections with

I &gt; 2.5σ(I)

R<sub>int</sub> = 0.024θ<sub>max</sub> = 27.09°

h = 0 → 27

k = -14 → 14

l = -17 → 15

Intensity decay: none

### Refinement

Refinement on F<sup>2</sup>  
R(F) = 0.051  
wR(F<sup>2</sup>) = 0.083  
S = 1.09  
2733 reflections  
242 parameters  
H atoms not refined  
w = 1/[σ<sup>2</sup>(F<sub>o</sub>) +  
0.00055|F<sub>o</sub>|<sup>2</sup>]

(Δ/σ)<sub>max</sub> < 0.001Δρ<sub>max</sub> = 0.48 e Å<sup>-3</sup>Δρ<sub>min</sub> = -0.41 e Å<sup>-3</sup>

Extinction correction: none

Scattering factors from  
International Tables for  
Crystallography (Vol. C)

Table 1. Selected geometric parameters (Å, °)

Cd—O2	2.352 (3)	N1—C1	1.342 (4)
Cd—O2 <sup>i</sup>	2.352 (3)	N2—C6	1.339 (4)
Cd—N1 <sup>ii</sup>	2.341 (4)	N3—C7	1.331 (4)
Cd—N2	2.348 (4)	N5—C17	1.336 (7)
Cd—N3	2.349 (2)	N6—C12	1.442 (7)
Cd—N3 <sup>i</sup>	2.349 (2)	C1—C2	1.381 (4)
O1—N4	1.152 (5)	C2—C3	1.391 (4)
O2—N4	1.249 (4)	C3—C4	1.485 (5)
O3—N4	1.218 (4)	C4—C5	1.383 (4)
O4—N6	1.244 (7)	C5—C6	1.381 (4)
O5—N6	1.223 (6)		
O2—Cd—O2 <sup>i</sup>	178.9 (1)	N1 <sup>ii</sup> —Cd—N2	180.0
O2—Cd—N3	97.3 (1)	N1 <sup>ii</sup> —Cd—N3	94.71 (6)
O2 <sup>i</sup> —Cd—N1 <sup>ii</sup>	90.55 (7)	N2—Cd—N3	85.29 (6)
O2 <sup>i</sup> —Cd—N3	82.6 (1)	Cd—O2—N4	128.1 (2)

Symmetry codes: (i) 2 - x, y, ½ - z; (ii) x, y - 1, z.

All H atoms were calculated and included in the structural model, but were fixed and not refined.

Data collection: SMART (Siemens, 1994). Cell refinement: SAINT (Siemens, 1994). Data reduction: SAINT. Program(s) used to solve structure: SIR92 (Altomare *et al.*, 1993). Program(s) used to refine structure: TEXSAN (Molecular Structure Corporation, 1995). Software used to prepare material for publication: TEXSAN.

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: DA1076). Services for accessing these data are described at the back of the journal.

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## [1,1'-Bis(diphenylphosphino)ferrocene]trichloro(phenylimido)rhenium(V) dichloromethane solvate

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## Abstract

In the title compound, [Re(C<sub>6</sub>H<sub>5</sub>N)Cl<sub>3</sub>(C<sub>34</sub>H<sub>28</sub>FeP<sub>2</sub>)]·CH<sub>2</sub>Cl<sub>2</sub>, the coordination sphere of the Re atom can be described as a distorted octahedron. The two cyclopentadienyl ligands in the 1,1'-bis(diphenylphosphino)ferrocene moiety adopt a *gauche* conformation.