

C(333)	0.2614 (5)	0.5508 (10)	0.7609 (4)	5.8 (3)
C(334)	0.3008 (5)	0.5101 (10)	0.7277 (4)	5.6 (3)
C(335)	0.3514 (4)	0.4297 (10)	0.7434 (3)	4.0 (2)
O(m)	1.2312 (5)	-0.2829 (10)	0.8951 (4)	12.4 (3)
C(m)	1.2496 (9)	-0.3262 (3)	0.9458 (7)	16.2 (7)

Table 4. Selected geometric parameters (Å, °) for (2)

Co(1)—S(11)	2.214 (2)	S(21)—C(211)	1.728 (8)
Co(1)—S(12)	2.203 (2)	S(22)—C(221)	1.724 (8)
Co(1)—S(13)	2.202 (2)	S(23)—C(231)	1.731 (9)
Co(1)—O(11)	1.976 (5)	O(21)—N(21)	1.336 (8)
Co(1)—O(12)	1.954 (5)	O(22)—N(22)	1.360 (8)
Co(1)—O(13)	1.951 (6)	O(23)—N(23)	1.352 (8)
S(11)—C(111)	1.710 (8)	S(21)—S(31)	2.205 (2)
S(12)—C(121)	1.735 (7)	Co(3)—S(32)	2.206 (2)
S(13)—C(131)	1.724 (8)	Co(3)—S(33)	2.207 (2)
O(11)—N(11)	1.348 (8)	Co(3)—O(31)	1.945 (6)
O(12)—N(12)	1.357 (8)	Co(3)—O(32)	1.927 (6)
O(13)—N(13)	1.343 (8)	Co(3)—O(33)	1.943 (5)
O(22)···O(m)	2.91 (1)	S(31)—C(311)	1.734 (8)
Co(2)—S(21)	2.190 (2)	S(32)—C(325)	1.728 (9)
Co(2)—S(22)	2.204 (2)	S(33)—C(331)	1.718 (9)
Co(2)—S(23)	2.215 (2)	O(31)—N(31)	1.349 (7)
Co(2)—O(21)	1.938 (5)	O(32)—N(32)	1.348 (7)
Co(2)—O(22)	1.967 (5)	O(33)—N(33)	1.348 (7)
Co(2)—O(23)	1.946 (5)		
S(11)—Co(1)—O(11)	87.0 (2)	Co(2)—S(23)—C(231)	96.9 (3)
S(12)—Co(1)—O(12)	87.9 (2)	Co(2)—O(21)—N(21)	115.8 (5)
S(13)—Co(1)—O(13)	86.6 (2)	Co(2)—O(22)—N(22)	115.7 (4)
Co(1)—S(11)—C(111)	96.9 (3)	Co(2)—O(23)—N(23)	116.9 (4)
Co(1)—S(12)—C(121)	96.9 (2)	S(31)—Co(3)—O(31)	87.3 (2)
Co(1)—S(13)—C(131)	97.5 (3)	S(32)—Co(3)—O(32)	88.0 (2)
Co(1)—O(11)—N(11)	115.1 (5)	S(33)—Co(3)—O(33)	87.1 (2)
Co(1)—O(12)—N(12)	116.1 (4)	Co(3)—S(31)—C(311)	97.1 (3)
Co(1)—O(13)—N(13)	114.7 (4)	Co(3)—S(32)—C(325)	96.3 (2)
S(21)—Co(2)—O(21)	87.8 (2)	Co(3)—S(33)—C(331)	96.8 (3)
S(22)—Co(2)—O(22)	87.5 (2)	Co(3)—O(31)—N(31)	116.4 (5)
S(23)—Co(2)—O(23)	87.5 (2)	Co(3)—O(32)—N(32)	115.6 (4)
Co(2)—S(21)—C(211)	97.4 (3)	Co(3)—O(33)—N(33)	116.1 (4)
Co(2)—S(22)—C(221)	98.1 (2)		

The H atoms, except those of H₂O and MeOH for (1) and MeOH for (2), were placed at calculated positions and given isotropic displacement factors derived from those of the parent atoms. The H atoms were included in the structure-factor calculations but not refined. Both structures were solved by direct methods (Main *et al.*, 1982) and refined by full-matrix least squares (Frenz, 1985).

We appreciate very much the financial support from the Climbing Program – National Key Project for Fundamental Research, the NNSF of China and the NSF of the Province of Fujian.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: MU1088). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Two-Dimensional Open-Frame Host Structure of the Inclusion Compound [Cd(tenH)₂{Ni(CN)₄}]₂.4C₆H₅NH₂ (ten = 1,4-Diazabicyclo[2.2.2]octane)

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(Received 10 May 1994; accepted 11 August 1994)

Abstract

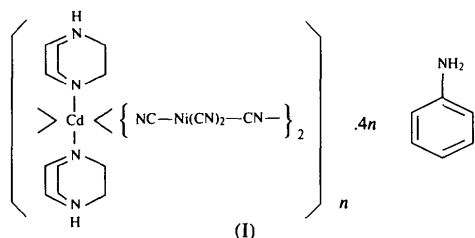
In the title inclusion compound, bis(1-azonia-4-azabicyclo[2.2.2]octane)cadmium(II) bis[tetracyanonickelate(II)]-aniline (1/4), [Cd(C₆H₁₃N₂)₂{Ni(CN)₄}]₂.4C₆H₇N, the host contains Cd²⁺ and [Ni(CN)₄]²⁻ in a ratio of 1:2. The two crystallographically independent [Ni(CN)₄]²⁻ anions behave as bidentate bridging ligands, spanning the Cd²⁺ cations with the N atoms of the cyano groups in *trans* positions along both the *a* and *b* axes, building up a two-dimensional network [Cd(tenH)₂{NC—Ni(CN)₂—CN—}]_n (ten = 1,4-diazabicyclo[2.2.2]octane). Two unidentate tenH ligands coordinate to the Cd in axial positions, the other N-atom end being protonated. The guest aniline molecules accommodated in the interlayer space are hydrogen

bonded *via* the amino groups to the non-bridged N atoms of the cyano groups and the protonated N-atom end of the tenH ligand.

Comment

The three-dimensional host structures of the Hofmann-en-type clathrates $[\text{Cd}(\text{en})\text{Ni}(\text{CN})_4] \cdot 2G$ ($G = \text{C}_6\text{H}_6$, $\text{C}_4\text{H}_4\text{S}$ or $\text{C}_4\text{H}_5\text{N}$) (Iwamoto, 1984) are substantially analogous to those of the two-dimensional Hofmann-type clathrates $[\text{Cd}(\text{NH}_3)_2\text{Ni}(\text{CN})_4] \cdot 2G$ (Iwamoto, 1984) in that the guest molecules are accommodated in the interlayer space between the two-dimensional networks of $[\text{Cd}(\text{—NC—Ni}_{1/4})_4]_n$. In the former, the networks are spanned by the ambidentate 1,2-diaminoethane (en) ligands; in the latter they are supported by the NH_3 ligands protruding from the networks. However, the structures of the aniline and phenol clathrates $[\text{Cd}(\text{en})_2\text{Ni}(\text{CN})_4] \cdot 2\text{C}_6\text{H}_5\text{NH}_2$ and $\{[\text{Cd}(\text{en})]_2(\text{en})\{\text{Ni}(\text{CN})_4\}_2\} \cdot 4\text{C}_6\text{H}_5\text{OH}$ (Yuge & Iwamoto, 1994) are different, not only from those of the Hofmann and Hofmann-en types, as shown by their compositions, but also from each other. The host complex of the aniline clathrate has the one-dimensional structure $[\text{—Cd}(\text{en})_2\text{—NC—Ni}(\text{CN})_2\text{—CN—}]_n$ in which each en group is chelated to Cd, and the cyano groups at *trans* positions produce the catenation. The three-dimensional host of the phenol clathrate is built by doubly bridged $[\text{Ni}(\text{CN—Cd—NC—})_2]_n$ and $[\text{—Cd—en—}]_n$ catenations sharing the en-chelated Cd atoms and additionally spanned by $\mu\text{-trans-NC—Ni}(\text{CN})_2\text{—CN—}$ bridges.

We attempted to use the ligand 1,4-diazabicyclo-[2.2.2]octane (ten: triethylenediamine) to produce metal-complex hosts for the aromatic guests, but only the aniline clathrate was obtained from the $\text{Cd}^{2+}\text{—}[\text{Ni}(\text{CN})_4]^{2-}\text{—ten—G}$ systems. So far, the title compound $[\text{Cd}(\text{tenH})_2\{\text{Ni}(\text{CN})_4\}_2] \cdot 4\text{C}_6\text{H}_5\text{NH}_2$ (I), is the only example of a cadmium tetracyanonickelate host with a $\text{Cd}^{2+}:\text{[Ni}(\text{CN})_4]^{2-}$ ratio of 1:2 that has been prepared during our attempts to develop multi-dimensional structures formed between the Lewis acid Cd^{2+} and the Lewis base N-atom end of $[\text{Ni}(\text{CN})_4]^{2-}$. Attempts to obtain crystals of the compounds with Ni, Cu or Zn in place of Cd have been unsuccessful.



The Cd and the two Ni atoms are situated on inversion centres (Fig. 1). Both $[\text{Ni}(\text{CN})_4]^{2-}$ moieties behave as bidentate bridging ligands spanning the Cd

atoms at the N-atom ends of the cyano groups in *trans* positions; the one-dimensional $[\text{—Cd—}\{\text{trans-NC—Ni}(\text{CN})_2\text{—CN—}\}]_n$ chains run along the *a* and *b* axes, sharing the Cd atoms to form the two-dimensional $[\text{Cd}(\text{tenH})_2\{\text{NC—Ni}(\text{CN})_2\text{—CN—}\}_2]_n$ network (Fig. 2). This type of one-dimensional chain has been found in the following compounds: $[\text{Ni}(\text{en})_2\text{Pd}(\text{CN})_4]$ (Rüegg & Ludi, 1971), $[\text{M}(\text{en})_2\text{Ni}(\text{CN})_4]$ ($M = \text{Ni}$, Cu or Zn) (Černák, Chomič, Baloghová & Dunaj-Jurčo, 1988; Černák, Potočňák, Chomič & Dunaj-Jurčo, 1990; Lokaj, Gyerová, Sopková, Sivý, Kettmann & Vrabel, 1991) and $[\text{M}(\text{en})_2\text{Ni}(\text{CN})_4] \cdot 2\text{C}_6\text{H}_5\text{NH}_2$ ($M = \text{Ni}$, Cu, Zn or Cd) (Yuge & Iwamoto, 1994). The axially elongated octahedral coordination about Cd is directed along the $\text{Cd}(\text{tenH})_2$ unit in the present structure [equatorial Cd—N bonds 2.325 (3) and 2.334 (3), axial 2.446 (3) Å]. By contrast, the coordination in $[\text{Cd}(\text{en})_2\text{Ni}(\text{CN})_4] \cdot 2\text{C}_6\text{H}_5\text{NH}_2$ is along the $\text{Cd}(\text{NC})_2$ chain [equatorial bonds 2.345 (5) and 2.313 (5) Å, axial 2.454 (5) Å].

The stacking of the $\mu\text{-trans-NC—Ni}(\text{CN})_2\text{—CN—}$ linked networks is determined by the *n* glide; the guest aniline molecules are accommodated in the interlayer space. Each unidentate tenH ligand is protonated at 1-N; this end protrudes toward the square mesh of an adjacent

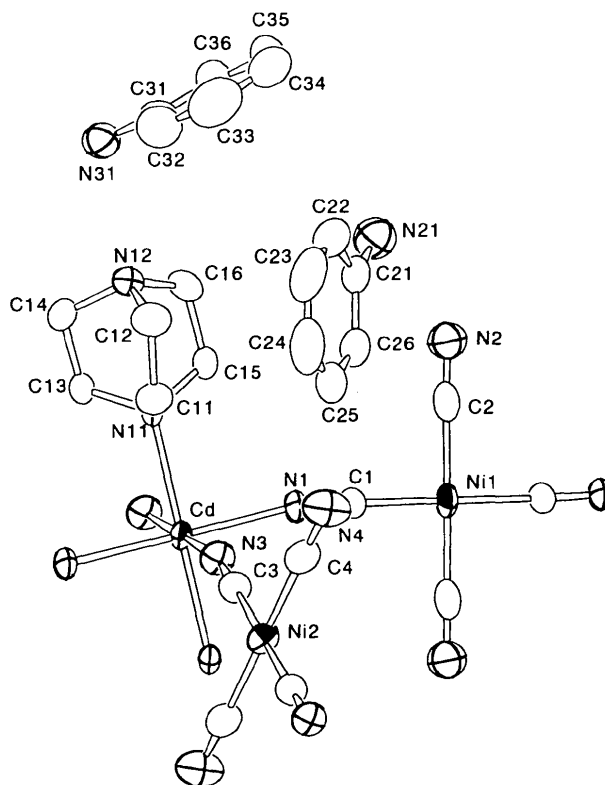


Fig. 1. Partial structure of $[\text{Cd}(\text{tenH})_2\{\text{Ni}(\text{CN})_4\}_2] \cdot 4\text{C}_6\text{H}_5\text{NH}_2$ showing atomic labelling. Displacement ellipsoids are drawn at the 50% probability level; H atoms are omitted for clarity.

network. The unidentate cyano groups of the μ -*trans*-NC—Ni(CN)₂—CN— moieties also protrude into the interlayer space to form hydrogen bonds with the ammonio groups of the tenH ligands and the amino groups of the guest aniline molecules. One of the crystallographically independent aniline molecules, whose molecular plane is vertical to the layer, appears to form hydrogen bonds with the N-atom ends of two unidentate CN moieties from adjacent layers. The other aniline, whose molecular plane is almost parallel to the layer, forms hydrogen bonds with the N-atom ends of two unidentate CN groups from the same layer and with a tenH ligand protruding from an adjacent layer. These hydrogen bonds reinforce the three-dimensional crystal structure (Fig. 2).

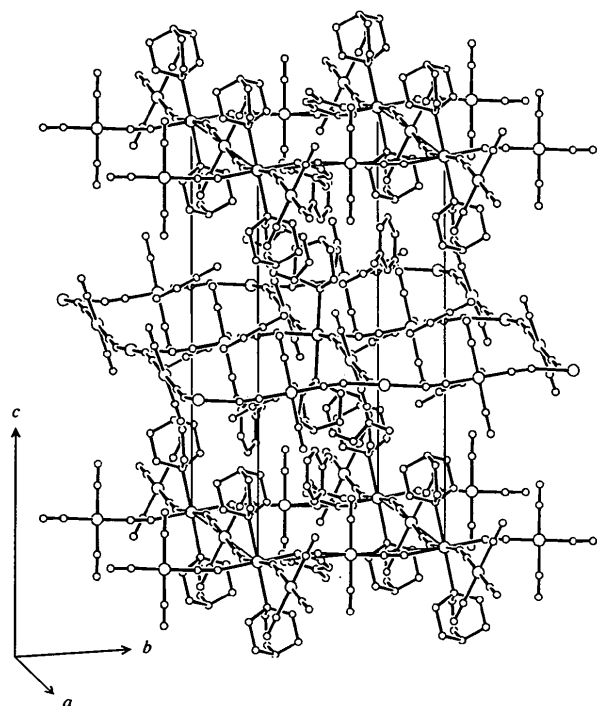


Fig. 2. Packing diagram for [Cd(tenH)₂{Ni(CN)₄}₂].4C₆H₅NH₂. H atoms are omitted for clarity.

Experimental

To an aqueous solution containing 1.14 g (5 mmol) of CdCl₂·2.5H₂O, 2.60 g (10 mmol) of K₂[Ni(CN)₄]·H₂O, 1.12 g (10 mmol) of ten and 5 ml of 2-aminoethanol was added an appropriate amount of citric acid to adjust the pH to 9 in ca 100 ml of the final volume. The small amount of the precipitate which formed immediately was filtered off and the solution was covered with a layer of neat aniline and allowed to stand in a refrigerator at ca 278 K for a few weeks. The density D_m was measured by flotation in mesitylene-bromoform.

Crystal data

[Cd(C₆H₁₃N₂)₂{Ni(CN)₄}₂].4C₆H₇N

$M_r = 1036.82$

Monoclinic

$P2_1/n$

$a = 10.537(2) \text{ \AA}$

$b = 10.615(3) \text{ \AA}$

$c = 21.101(4) \text{ \AA}$

$\beta = 92.24(2)^\circ$

$V = 2358.4(9) \text{ \AA}^3$

$Z = 2$

$D_x = 1.47 \text{ Mg m}^{-3}$

$D_m = 1.47(1) \text{ Mg m}^{-3}$

Data collection

Rigaku AFC-5R diffractometer

$\omega/2\theta$ scans

Absorption correction: none

7697 measured reflections

5554 independent reflections

3654 observed reflections

$[F > 4\sigma(F)]$

Mo $K\alpha$ radiation

$\lambda = 0.71069 \text{ \AA}$

Cell parameters from 25 reflections

$\theta = 16.00\text{--}17.45^\circ$

$\mu = 1.287 \text{ mm}^{-1}$

$T = 293 \text{ K}$

Plate

$0.24 \times 0.20 \times 0.18 \text{ mm}$

Yellow

$R_{\text{int}} = 0.011$

$\theta_{\text{max}} = 30.00^\circ$

$h = 0 \rightarrow 14$

$k = 0 \rightarrow 14$

$l = -29 \rightarrow 29$

3 standard reflections

monitored every 200

reflections

intensity decay: 3.7%

Refinement

Refinement on F

$R = 0.0417$

$wR = 0.0338$

$S = 1.369$

3654 reflections

397 parameters

All H-atom parameters

refined

$w = 1/[\sigma^2(F_o) + 0.00005F_o^2]$

$(\Delta/\sigma)_{\text{max}} = 0.013$

$\Delta\rho_{\text{max}} = 0.60 \text{ e \AA}^{-3}$

$\Delta\rho_{\text{min}} = -0.46 \text{ e \AA}^{-3}$

Extinction correction: none

Atomic scattering factors

from SHELX76

(Sheldrick, 1976) for C,

H, N and International

Tables for X-ray Crystal-

lography (1974, Vol. IV,

Table 2.2B) for Cd, Ni

Table 1. Fractional atomic coordinates and equivalent isotropic displacement parameters (\AA^2)

$$U_{\text{eq}} = (1/3)\sum_i\sum_j U_{ij}a_i^*a_j$$

	x	y	z	U_{eq}
Cd	0	0	0	0.0234 (1)
Ni1	0	1/2	0	0.0334 (2)
Ni2	1/2	0	0	0.0323 (2)
N1	-0.0060 (4)	0.2176 (3)	0.0118 (2)	0.040 (1)
N2	-0.0864 (4)	0.5334 (4)	0.1334 (2)	0.060 (2)
N3	0.2190 (3)	0.0019 (5)	0.0214 (2)	0.039 (1)
N4	0.5626 (4)	0.1063 (5)	0.1303 (2)	0.061 (2)
C1	-0.0053 (5)	0.3249 (4)	0.0083 (2)	0.034 (1)
C2	-0.0534 (4)	0.5193 (4)	0.0828 (2)	0.043 (2)
C3	0.3260 (3)	0.0012 (5)	0.0147 (2)	0.033 (1)
C4	0.5366 (4)	0.0669 (4)	0.0811 (2)	0.038 (2)
N11	-0.0537 (3)	-0.0314 (3)	0.1106 (2)	0.027 (1)
C11	0.0644 (5)	-0.0631 (5)	0.1478 (2)	0.040 (2)
C12	0.0431 (5)	-0.0591 (6)	0.2189 (3)	0.047 (2)
C13	-0.1462 (5)	-0.1342 (5)	0.1202 (2)	0.037 (2)
C14	-0.1579 (6)	-0.1638 (5)	0.1908 (2)	0.044 (2)
C15	-0.1085 (5)	0.0854 (4)	0.1372 (2)	0.034 (2)
C16	-0.1499 (5)	0.0639 (5)	0.2051 (3)	0.042 (2)
N12	-0.0969 (4)	-0.0583 (4)	0.2279 (2)	0.036 (1)
N21	-0.0113 (5)	0.3800 (6)	0.2568 (3)	0.066 (2)
C21	0.0993 (5)	0.3101 (5)	0.2488 (3)	0.045 (2)
C22	0.1588 (6)	0.2480 (6)	0.2995 (3)	0.059 (2)
C23	0.2624 (7)	0.1718 (7)	0.2901 (4)	0.076 (3)

C24	0.3053 (7)	0.1540 (7)	0.2308 (5)	0.082 (3)
C25	0.2478 (7)	0.2145 (6)	0.1803 (3)	0.066 (3)
C26	0.1458 (6)	0.2915 (5)	0.1887 (3)	0.052 (2)
N31	-0.1329 (5)	-0.0909 (5)	0.3566 (2)	0.053 (2)
C31	-0.0492 (5)	-0.0093 (6)	0.3924 (2)	0.047 (2)
C32	0.0733 (7)	-0.0448 (7)	0.4075 (3)	0.068 (3)
C33	0.1547 (8)	0.0364 (9)	0.4416 (4)	0.087 (4)
C34	0.1134 (9)	0.1528 (8)	0.4600 (3)	0.079 (3)
C35	-0.0070 (9)	0.1871 (7)	0.4437 (3)	0.081 (3)
C36	-0.0892 (6)	0.1093 (6)	0.4102 (3)	0.058 (2)

Table 2. Selected geometric parameters (Å, °)

Cd—N1	2.325 (3)	C4—N4	1.142 (5)
Cd—N3	2.334 (3)	N11—C11	1.483 (5)
Cd—N11	2.446 (3)	N11—C13	1.483 (5)
Ni1—C1	1.868 (4)	N11—C15	1.486 (5)
Ni1—C2	1.868 (5)	N12—C12	1.495 (6)
Ni2—C3	1.871 (4)	N12—C14	1.497 (6)
Ni2—C4	1.879 (5)	N12—C16	1.485 (6)
C1—N1	1.141 (5)	C11—C12	1.528 (7)
C2—N2	1.145 (6)	C13—C14	1.531 (7)
C3—N3	1.142 (4)	C15—C16	1.532 (7)
N2...N21	3.146 (8)	N4...N31 ⁱⁱⁱ	3.308 (7)
N2...N31 ⁱ	3.254 (7)	N12...N31	2.779 (7)
N4...N21 ⁱⁱ	3.442 (8)		
N1—Cd—N3	90.1 (2)	Cd—N11—C15	110.7 (2)
N1—Cd—N11	91.4 (1)	C11—N11—C13	107.7 (4)
N3—Cd—N11	94.6 (1)	C13—N11—C15	107.2 (4)
C1—Ni1—C2	90.6 (2)	C15—N11—C11	108.7 (4)
C3—Ni2—C4	90.8 (2)	N11—C11—C12	111.1 (4)
Cd—N1—C1	169.8 (4)	N11—C15—C16	111.0 (4)
Cd—N3—C3	161.8 (3)	N11—C13—C14	111.2 (4)
Ni1—C1—N1	177.9 (5)	C11—C12—N12	108.0 (4)
Ni1—C2—N2	178.8 (4)	C13—C14—N12	107.8 (4)
Ni2—C3—N3	177.6 (3)	C15—C16—N12	108.4 (4)
Ni2—C4—N4	177.9 (4)	C12—N12—C14	109.6 (4)
Cd—N11—C11	108.3 (3)	C14—N12—C16	109.6 (4)
Cd—N11—C13	114.1 (3)	C16—N12—C12	108.9 (4)

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

All non-H atoms were refined anisotropically. All H atoms were found from the $\Delta\rho$ maps and refined isotropically to clarify the hydrogen-bond network.

Data collection: *MSC/AFC Diffractometer Control Software* (Molecular Structure Corporation, 1988). Cell refinement: *MSC/AFC Diffractometer Control Software*. Data reduction: *SHELX76* (Sheldrick, 1976). Program(s) used to solve structure: *SHELX76*. Program(s) used to refine structure: *SHELX76*. Molecular graphics: *ORTEPII* (Johnson, 1976). All calculations were carried out on a HITAC M-680H computer at the Institute for Molecular Science, Okazaki.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: AB1195). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Complexe entre un Carboxylate de Rhodium(II) et un Dérivé de la [1,4]Thiazépine: Synthèse et Structure Cristalline de Tétrakis(μ -acétato)bis-[[11-aminodibenzo[*b,f*]][1,4]thiazépine]-rhodium(II)}(*Rh—Rh*)

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(Received 20 March 1994; accepted 31 May 1994)

Abstract

The unit cell of the title compound, tetrakis(μ -acetato-*O:O'*)bis[[11-aminodibenzo[*b,f*]][1,4]thiazépine-*N*¹⁰]rhodium(II)}(*Rh—Rh*), $[\text{Rh}(\text{C}_2\text{H}_3\text{O}_2)_4(\text{C}_{13}\text{H}_{10}\text{N}_2\text{S}_2)]_2$, contains four discrete dimer molecules which have an inversion centre. The Rh—Rh distance [2.402 (2) Å] and other parameters for the rhodium acetate cage show no major deviations from those reported in the literature. The thiazépine is in a boat conformation. The dihedral angle between the two aromatic rings is 96.7 (5)°. There are no hydrogen bonds.