

C(11)	0.2983 (15)	0.7708 (10)	-0.2207 (17)	0.070 (6)
C(12)	-0.0802 (18)	0.8716 (11)	-0.0066 (22)	0.087 (7)
Ru(2)	0.22941 (8)	0.11876 (5)	-0.36635 (10)	0.0384 (3)
Cl(2)	0.3490 (3)	0.20042 (19)	-0.1298 (4)	0.0516 (10)
O(1a)	0.2097 (8)	0.0409 (6)	-0.1876 (12)	0.053 (3)
O(2a)	0.3185 (9)	-0.0404 (6)	0.0115 (12)	0.060 (3)
N(1a)	0.4229 (10)	0.0575 (7)	-0.3040 (14)	0.047 (4)
C(1a)	0.4461 (11)	0.0037 (6)	-0.1602 (14)	0.044 (4)
C(2a)	0.3176 (11)	0.0020 (6)	-0.1052 (14)	0.038 (3)
C(3a)	0.5765 (14)	0.0225 (13)	-0.0174 (21)	0.097 (8)
C(4a)	0.0085 (12)	0.1546 (8)	-0.4785 (16)	0.050 (4)
C(5a)	0.0925 (12)	0.2160 (7)	-0.4952 (15)	0.046 (4)
C(6a)	0.2018 (12)	0.2025 (7)	-0.5663 (15)	0.045 (4)
C(7a)	0.2217 (10)	0.1256 (7)	-0.6208 (10)	0.041 (3)
C(8a)	0.1314 (13)	0.0680 (8)	-0.6112 (18)	0.057 (5)
C(9a)	0.0288 (12)	0.0790 (8)	-0.5355 (17)	0.055 (5)
C(10a)	0.0781 (16)	0.2946 (8)	-0.4331 (19)	0.063 (5)
C(11a)	0.3369 (14)	0.1155 (11)	-0.6972 (16)	0.065 (5)
C(12a)	-0.0589 (19)	0.0143 (10)	-0.5047 (26)	0.088 (8)

Table 2. Selected geometric parameters (Å, °)

Cl(1)—Ru(1)	2.439 (3)	Cl(2)—Ru(2)	2.428 (3)
O(1)—Ru(1)	2.085 (8)	O(1a)—Ru(2)	2.094 (9)
N(1)—Ru(1)	2.113 (9)	N(1a)—Ru(2)	2.123 (10)
C(4)—Ru(1)	2.188 (11)	C(4a)—Ru(2)	2.194 (11)
C(5)—Ru(1)	2.206 (10)	C(5a)—Ru(2)	2.222 (11)
C(6)—Ru(1)	2.198 (12)	C(6a)—Ru(2)	2.185 (11)
C(7)—Ru(1)	2.155 (9)	C(7a)—Ru(2)	2.146 (9)
C(8)—Ru(1)	2.158 (12)	C(8a)—Ru(2)	2.173 (13)
C(9)—Ru(1)	2.165 (11)	C(9a)—Ru(2)	2.163 (11)
C(2)—O(1)	1.262 (13)	C(2a)—O(1a)	1.271 (14)
C(2)—O(2)	1.233 (15)	C(2a)—O(2a)	1.234 (14)
C(1)—N(1)	1.456 (14)	C(1a)—N(1a)	1.495 (15)
C(2)—C(1)	1.538 (15)	C(2a)—C(1a)	1.514 (15)
C(3)—C(1)	1.532 (15)	C(3a)—C(1a)	1.496 (18)
C(5)—C(4)	1.401 (18)	C(5a)—C(4a)	1.396 (18)
C(9)—C(4)	1.414 (18)	C(9a)—C(4a)	1.436 (18)
C(6)—C(5)	1.416 (16)	C(6a)—C(5a)	1.441 (16)
C(10)—C(5)	1.513 (16)	C(10a)—C(5a)	1.486 (17)
C(7)—C(6)	1.415 (16)	C(7a)—C(6a)	1.448 (16)
C(8)—C(7)	1.413 (18)	C(8a)—C(7a)	1.371 (18)
C(11)—C(7)	1.495 (17)	C(11a)—C(7a)	1.516 (16)
C(9)—C(8)	1.386 (20)	C(9a)—C(8a)	1.400 (20)
C(12)—C(9)	1.510 (19)	C(12a)—C(9a)	1.503 (20)
O(1)—Ru(1)—Cl(1)	86.6 (3)	O(1a)—Ru(2)—Cl(2)	85.1 (3)
N(1)—Ru(1)—Cl(1)	85.3 (3)	N(1a)—Ru(2)—Cl(2)	85.8 (3)
N(1)—Ru(1)—O(1)	76.4 (3)	N(1a)—Ru(2)—O(1a)	78.2 (3)
C(2)—O(1)—Ru(1)	119.1 (7)	C(2a)—O(1a)—Ru(2)	118.3 (7)
C(1)—N(1)—Ru(1)	111.0 (7)	C(1a)—N(1a)—Ru(2)	113.4 (7)
C(2)—C(1)—N(1)	108.2 (8)	C(2a)—C(1a)—N(1a)	110.5 (9)
C(3)—C(1)—N(1)	113.4 (10)	C(3a)—C(1a)—N(1a)	112.6 (10)
C(3)—C(1)—C(2)	112.5 (10)	C(3a)—C(1a)—C(2a)	111.0 (12)
O(2)—C(2)—O(1)	124.9 (11)	O(2a)—C(2a)—O(1a)	122.5 (10)
C(1)—C(2)—O(1)	115.3 (10)	C(1a)—C(2a)—O(1a)	118.5 (10)
C(1)—C(2)—O(2)	119.8 (10)	C(1a)—C(2a)—O(2a)	118.9 (10)
C(9)—C(4)—C(5)	119.0 (11)	C(9a)—C(4a)—C(5a)	120.2 (12)
C(6)—C(5)—C(4)	120.4 (11)	C(6a)—C(5a)—C(4a)	119.4 (11)
C(10)—C(5)—C(4)	118.8 (11)	C(10a)—C(5a)—C(4a)	122.0 (12)
C(10)—C(5)—C(6)	120.6 (12)	C(10a)—C(5a)—C(6a)	118.5 (12)
C(7)—C(6)—C(5)	120.0 (11)	C(7a)—C(6a)—C(5a)	119.4 (11)
C(8)—C(7)—C(6)	118.9 (11)	C(11a)—C(7a)—Ru(2)	130.7 (6)
C(11)—C(7)—C(6)	118.6 (12)	C(11a)—C(7a)—C(6a)	116.8 (12)
C(11)—C(7)—C(8)	122.5 (12)	C(11a)—C(7a)—C(8a)	123.9 (12)
C(9)—C(8)—C(7)	120.7 (12)	C(9a)—C(8a)—C(7a)	122.2 (12)
C(8)—C(9)—C(4)	120.9 (12)	C(8a)—C(9a)—C(4a)	119.3 (11)
C(12)—C(9)—C(4)	119.6 (14)	C(12a)—C(9a)—C(4a)	117.7 (14)
C(12)—C(9)—C(8)	119.4 (14)	C(12a)—C(9a)—C(8a)	123.0 (14)

The H atoms of the NH<sub>2</sub> groups were located from a difference synthesis and their positional parameters were refined. All other H atoms were included in calculated positions (C—H = 0.95 Å) with refinement of their isotropic displacement parameters. The structure was solved by direct methods using the *TREF* option of *SHELXS86* (Sheldrick, 1986). All subsequent calculations used *SHELX76* (Sheldrick, 1976).

We thank the SERC for a studentship (LCC) and Leicester University Computer Centre who provided support for the X-ray single-crystal calculations.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry, including H-atom geometry, have been deposited with the IUCr (Reference: MU1096). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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## *trans*-Tetraaquabis(*p*-nitrohippurato)-cobalt(II) Dihydrate

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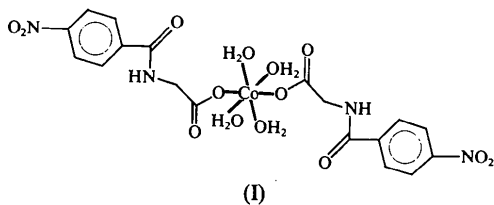
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## Abstract

Crystals of [Co(4-NO<sub>2</sub>-hip)<sub>2</sub>(H<sub>2</sub>O)<sub>4</sub>].2H<sub>2</sub>O (4-NO<sub>2</sub>-hip = *p*-NO<sub>2</sub>.C<sub>6</sub>H<sub>4</sub>CO.NHCH<sub>2</sub>CO<sub>2</sub><sup>-</sup>) contain molecules of the centrosymmetric octahedral cobalt(II) complex and lattice water molecules which are interlinked by hydrogen bonds. The Co atom lies on a crystallographic symmetry centre and has a slightly elongated octahedral environment arising from coordination by the carboxylic O atoms of two monodentate 4-nitrohippurate anions and the O atoms of four water molecules.

### Comment

The coordination of the Co atom in the title compound (I) (see Fig. 1) is very similar to that found in Co<sup>II</sup> complexes of other N-protected amino acids, such as *N*-tosyl-β-glycinate (Battaglia *et al.*, 1985) and *N*-acetyl-glycinate (Menabue *et al.*, 1987). The Co—O bond distances and angles (Table 2) are also comparable with those found in [Co(bzgly)<sub>2</sub>(H<sub>2</sub>O)<sub>3</sub>].2H<sub>2</sub>O (bzgly = *N*-benzoylglycinate) (Eichelberger *et al.*, 1977; Morelock *et al.*, 1979) and [Co(4-NH<sub>2</sub>-hip)<sub>2</sub>(H<sub>2</sub>O)<sub>2</sub>].2H<sub>2</sub>O (Forti, Menabue & Saladini, 1991), although in the first case one water molecule bridges two Co<sup>II</sup> ions giving rise to infinite one-dimensional chains, while in the second, a polymeric arrangement results from the bridging coordination of the 4-aminohippurate anions.



In the title complex (I) the planar C<sup>α</sup>—COO group forms a dihedral angle of 77.2(2)° with the amidic group, which in turn makes a dihedral angle of 18.0(2)° with the phenyl ring. The conformation of the amino acid moiety can be described by the torsion angles O(1)—C(1)—C(2)—N(1) and C(1)—C(2)—N(1)—C(3) of 146.4(4) and 59.7(5)°, respectively. The carboxylic and amidic O atoms appear to be involved in intramolecular hydrogen bonds with O(W1) and O(W2) with contact distances ranging from 2.562 to 2.961 Å, while the amidic N atom and O(W2) form intermolecular hydrogen bonds with contact distances of 2.694–2.971 Å. The water H atoms were not located, precluding a detailed description of the hydrogen-bond arrangement

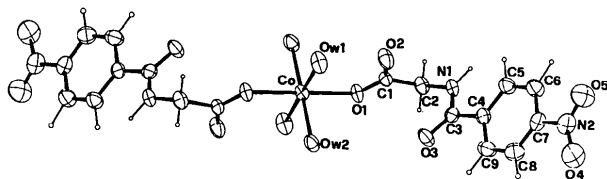


Fig. 1. ORTEP (Johnson, 1965) view of *trans*-[Co(4-NO<sub>2</sub>-hip)<sub>2</sub>(H<sub>2</sub>O)<sub>4</sub>], with 40% probability ellipsoids. H atoms are represented as spheres of arbitrary radii.

### Experimental

A methanolic solution (0.05 dm<sup>3</sup>) of cobalt(II) acetate (0.02 mol dm<sup>-3</sup>) was added to a methanolic solution (0.02 dm<sup>3</sup>) of the amino acid (0.1 mol dm<sup>-3</sup>). After a few days a pink crystalline compound separated {[Co(4-NO<sub>2</sub>-hip)<sub>2</sub>(H<sub>2</sub>O)<sub>4</sub>].2H<sub>2</sub>O, yield 80%; analysis: found, C 35.21, H

4.35, N 9.06%; calculated for C<sub>18</sub>H<sub>26</sub>CoN<sub>4</sub>O<sub>16</sub>, C 35.23, H 4.27, N 9.14%}. The density, *D<sub>m</sub>*, was measured by flotation in CHCl<sub>3</sub>—CHCl<sub>2</sub>CCl<sub>3</sub>.

### Crystal data

[Co(C<sub>9</sub>H<sub>7</sub>N<sub>2</sub>O<sub>5</sub>)<sub>2</sub>(H<sub>2</sub>O)<sub>4</sub>].-  
2H<sub>2</sub>O

*M<sub>r</sub>* = 613.3

Monoclinic

*P*2<sub>1</sub>/*a*

*a* = 13.113 (1) Å

*b* = 5.351 (2) Å

*c* = 18.757 (2) Å

β = 109.54 (1)°

*V* = 1240.3 (5) Å<sup>3</sup>

*Z* = 2

*D<sub>x</sub>* = 1.64 Mg m<sup>-3</sup>

*D<sub>m</sub>* = 1.60 Mg m<sup>-3</sup>

Mo Kα radiation

λ = 0.71069 Å

Cell parameters from 25 reflections

θ = 5.5–12°

μ = 0.77 mm<sup>-1</sup>

*T* = 298 (2) K

Irregular prism

0.20 × 0.15 × 0.10 mm

Pink

### Data collection

Enraf–Nonius CAD-4 diffractometer

ω/2θ scans

Absorption correction:

empirical

*T<sub>min</sub>* = 0.96, *T<sub>max</sub>* = 1.00

2531 measured reflections

2439 independent reflections

1363 observed reflections

[*I* > 2σ(*I*)]

*R<sub>int</sub>* = 0.023

θ<sub>max</sub> = 25°

*h* = -15 → 15

*k* = 0 → 6

*l* = 0 → 22

3 standard reflections

frequency: 180 min

intensity variation: 10%

### Refinement

Refinement on *F*

*R* = 0.040

*wR* = 0.041

1363 reflections

178 parameters

Unit weights applied

(Δ/σ)<sub>max</sub> = 0.002

Δρ<sub>max</sub> = 0.40 e Å<sup>-3</sup>

Δρ<sub>min</sub> = -0.37 e Å<sup>-3</sup>

Extinction correction: none

Atomic scattering factors

from *International Tables*

for *X-ray Crystallography*

(1974, Vol. IV)

Table 1. Fractional atomic coordinates and equivalent isotropic displacement parameters (Å<sup>2</sup>)

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

Co	<i>x</i>	<i>y</i>	<i>z</i>	<i>U<sub>eq</sub></i> (3)
O(1)	0	0	0	0.0234 (3)
O(1)	-0.0655 (2)	-0.1350 (6)	0.0825 (2)	0.030 (1)
O(2)	-0.1845 (3)	0.1664 (7)	0.0809 (2)	0.046 (1)
C(1)	-0.1347 (3)	-0.0349 (8)	0.1066 (2)	0.028 (2)
C(2)	-0.1624 (4)	-0.1700 (8)	0.1696 (3)	0.033 (2)
N(1)	-0.1875 (3)	0.0015 (8)	0.2212 (2)	0.031 (1)
C(3)	-0.1129 (4)	0.1664 (9)	0.2592 (2)	0.032 (2)
O(3)	-0.0226 (2)	0.1741 (7)	0.2514 (2)	0.044 (1)
C(4)	-0.1414 (3)	0.3478 (9)	0.3105 (3)	0.033 (2)
C(5)	-0.0566 (4)	0.470 (1)	0.3626 (3)	0.046 (2)
C(6)	-0.0760 (4)	0.650 (1)	0.4104 (3)	0.051 (2)
C(7)	-0.1810 (4)	0.6989 (9)	0.4040 (3)	0.037 (2)
C(8)	-0.2680 (4)	0.580 (1)	0.3532 (3)	0.043 (2)
C(9)	-0.2469 (4)	0.401 (1)	0.3057 (3)	0.042 (2)
N(2)	-0.2037 (4)	0.8961 (8)	0.4524 (3)	0.046 (2)
O(4)	-0.2943 (3)	0.9667 (8)	0.4388 (2)	0.065 (2)
O(5)	-0.1269 (4)	0.9615 (8)	0.5067 (3)	0.072 (2)
O(W1)	-0.0936 (3)	0.3178 (6)	-0.0128 (2)	0.042 (1)
O(W2)	0.1247 (2)	0.1646 (6)	0.0862 (2)	0.039 (1)
O(W3)	0.0731 (2)	0.5020 (7)	0.1797 (2)	0.042 (1)

Table 2. Selected geometric parameters (Å, °)

Co—O(1)	2.132 (3)	C(4)—C(5)	1.376 (6)
Co—O(W1)	2.064 (3)	C(5)—C(6)	1.393 (8)
Co—O(W2)	2.072 (3)	C(6)—C(7)	1.367 (8)
O(1)—C(1)	1.260 (6)	C(7)—C(8)	1.375 (6)
O(2)—C(1)	1.268 (5)	C(8)—C(9)	1.397 (8)
C(1)—C(2)	1.530 (7)	C(9)—C(4)	1.385 (7)
C(2)—N(1)	1.449 (6)	C(7)—N(2)	1.486 (7)
N(1)—C(3)	1.333 (6)	N(2)—O(4)	1.190 (7)
C(3)—O(3)	1.240 (6)	N(2)—O(5)	1.219 (6)
C(3)—C(4)	1.500 (7)		
O(W1)—Co—O(W2)	91.4 (1)	C(3)—C(4)—C(5)	116.6 (4)
O(1)—Co—O(W2)	89.0 (1)	C(5)—C(4)—C(9)	120.0 (4)
O(1)—Co—O(W1)	89.7 (1)	C(4)—C(5)—C(6)	120.4 (5)
Co—O(1)—C(1)	129.0 (3)	C(5)—C(6)—C(7)	118.2 (5)
O(1)—C(1)—O(2)	124.5 (4)	C(6)—C(7)—N(2)	119.1 (5)
O(2)—C(1)—C(2)	118.1 (4)	C(6)—C(7)—C(8)	123.3 (5)
O(1)—C(1)—C(2)	117.4 (4)	C(8)—C(7)—N(2)	117.6 (5)
C(1)—C(2)—N(1)	112.5 (4)	C(7)—C(8)—C(9)	117.6 (5)
C(2)—N(1)—C(3)	119.1 (4)	C(4)—C(9)—C(8)	120.4 (5)
N(1)—C(3)—C(4)	118.4 (4)	C(7)—N(2)—O(5)	115.8 (5)
N(1)—C(3)—O(3)	121.2 (4)	C(7)—N(2)—O(4)	118.9 (4)
O(3)—C(3)—C(4)	120.4 (4)	O(4)—N(2)—O(5)	125.1 (5)
C(3)—C(4)—C(9)	123.3 (4)		

The structure was solved by conventional Patterson and Fourier techniques, and was refined by full-matrix least-squares calculations. All non-H atoms were refined anisotropically. H atoms, except those of water molecules, were located on a Fourier difference map and were treated as fixed contributors. Data collection, cell refinement, data reduction: *CAD-4 Software* (Enraf-Nonius, 1989). Program(s) used to solve structure: *SHELX76* (Sheldrick, 1976). Program(s) used to refine structure: *SHELX76*. Molecular graphics: *ORTEP* (Johnson, 1965). Software used to prepare material for publication: *PARST* (Nardelli, 1983).

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: MU1099). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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## Bis[tris(bipyridine)nickel(II)] Tris[dicyanoargentate(I)] Chloride *n*-Hydrate (*n* = 9)

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## Abstract

The structure of bis[tris(bipyridine)nickel(II)] tris[dicyanoargentate(I)] chloride nonahydrate,  $[\text{Ni}(\text{C}_{10}\text{H}_8\text{N}_2)_3]_2[\text{Ag}(\text{CN})_2]_3 \cdot 9\text{H}_2\text{O}$ , is ionic. It comprises  $[\text{Ni}(\text{bpy})_3]^{2+}$  cations with threefold symmetry placed in the cavities of a three-dimensional hydrogen-bonded network formed by  $\text{N}\cdots\text{O}$  and  $\text{O}\cdots\text{O}$  type contacts between linear  $[\text{Ag}(\text{CN})_2]^-$  anions and a hexagonal prismatic cluster of water molecules formed by the packing of two crystallographically independent water molecules. The  $\text{Cl}^-$  anion is partially distributed over the positions of the O atoms. The Ni atom is in a six-coordinate  $\text{N}_6$  deformed octahedral environment with the Ni—N distances being almost equal: 2.093 (3) (3 $\times$ ) and 2.096 (3) Å (3 $\times$ ). The Ag—C distance is 2.052 (5) Å.