

References

- Charpin, P., Folcher, M., Nierlich, M., Lance, M., Vigner, D., Navaza, A. & de Rango, C. (1990). *Acta Cryst.* **C46**, 1778–1781.
- Hämäläinen, R., Turpeinen, U. & Mutikainen, I. (1996). *Acta Cryst.* **C52**, 16–17.
- Howatson, J., Grew, D. M. & Morosin, B. (1975). *J. Inorg. Nucl. Chem.* **37**, 1933–1935.
- Nicolet XRD Corporation (1980). *Nicolet P3/R3 Data Collection Operators Manual*. Nicolet XRD Corporation, 10061 Bubb Road, Cupertino, California 95014, USA.
- Nierlich, M., Iroulart, G., Vigner, D., Keller, N. & Lance, M. (1990). *Acta Cryst.* **C46**, 2459–2460.
- North, A. C. T., Phillips, D. C. & Mathews, F. S. (1968). *Acta Cryst.* **A24**, 351–359.
- Sheldrick, G. M. (1990). *SHELXTL/PC*. Version 4.1. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.
- Sheldrick, G. M. (1993). *SHELXL93. Program for the Refinement of Crystal Structures*. University of Göttingen, Germany.

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Isomorphous Polymeric Complexes of Ca^{2+} and Pb^{2+} with 9,10-Dihydro-9-oxo-10-acridineacetate (CMA^-) Ligands: $[\text{M}(\text{CMA})_2(\text{H}_2\text{O})_2]$

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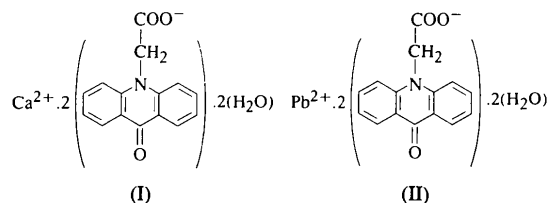
Abstract

The title compounds, *catena*-poly[*diaquacalcium-bis-μ*-(9,10-dihydro-9-oxo-10-acridineacetato-*O,O'*:*O*)], $[\text{Ca}(\text{CMA})_2(\text{H}_2\text{O})_2]$, and the analogous lead(II) compound, $[\text{Pb}(\text{CMA})_2(\text{H}_2\text{O})_2]$ ($\text{CMA} = \text{C}_{15}\text{H}_{10}\text{NO}_3$), are carboxylate-bridged linear polymers in the solid state. Each ligand chelates one metal ion and bridges to the next metal ion. Two water molecules complete eightfold coordination around the Ca and Pb atoms. Adjacent polymer chains show stacking interactions of parallel acridone groups and there are hydrogen bonds linking aqua ligands to carboxylate and ketone O atoms.

Comment

This investigation is a continuation of our earlier studies on the ligating properties of the 9,10-dihydro-9-oxo-10-acridineacetate anion, a powerful interferon inducer (Inglot, Młochowski, Szulc, Inglot & Albin,

1985). We have found that the CMA^- anion in its metal complexes exhibits a variety of carboxylate bonding modes. Bridges through a single O atom have been observed in $[\text{Cu}(\text{CMA})_2(\text{H}_2\text{O})_2]$ (Miernik, Lis, Palus & Reedijk, 1993), whereas both μ_3 single-atom bridging and *syn-syn* bridging by the OCO group are present in $[\text{Na}_2\text{Zn}(\text{CMA})_4(\text{EtOH})_2(\text{H}_2\text{O})_7]$ (Miernik & Lis, 1994). In this article we describe the preparation and crystal structure of two isomorphous complexes of CMA^- , namely $[\text{Ca}(\text{CMA})_2(\text{H}_2\text{O})_2]$, (I) and $[\text{Pb}(\text{CMA})_2(\text{H}_2\text{O})_2]$, (II), in which the CMA^- anions act as both chelating and single-atom bridging ligands.



The atomic numbering scheme for the repeating unit of the Ca compound is shown in Fig. 1. The Pb complex is essentially the same, except for a twofold disorder of one aqua ligand. A packing diagram is shown in Fig. 2. The crystal structures consist of $[\text{Ca}(\text{CMA})_2(\text{H}_2\text{O})_2]$ or $[\text{Pb}(\text{CMA})_2(\text{H}_2\text{O})_2]$ repeating units. The Ca and Pb atoms are eight-coordinate, being bonded to six O atoms from carboxylate groups and two water O atoms. Each carboxylate group forms a chelate ring with one metal ion and is also coordinated to a centrosymmetrically related neighbouring metal ion in a monodentate manner. Thus, the repeating units are linked by monatomic carboxylate bridges to form polymer chains. The van der Waals interactions between ring systems of CMA^- result in interleaving of adjacent polymer chains, with stacks of ligands along the *a* axis, separated by about 3.5 Å.

The main features of coordination around the metal ions observed in compounds (I) and (II), namely a co-

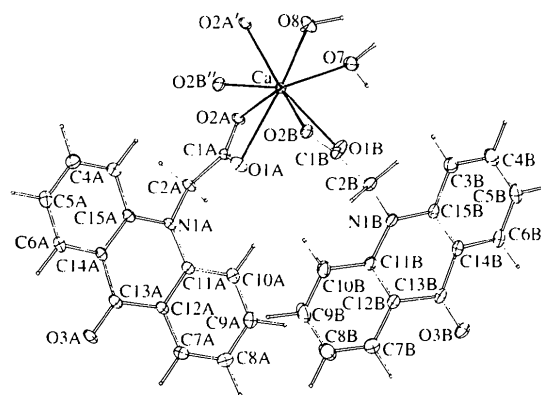


Fig. 1. Molecular geometry around the Ca atom in (I). Displacement ellipsoids are plotted at the 50% probability level.

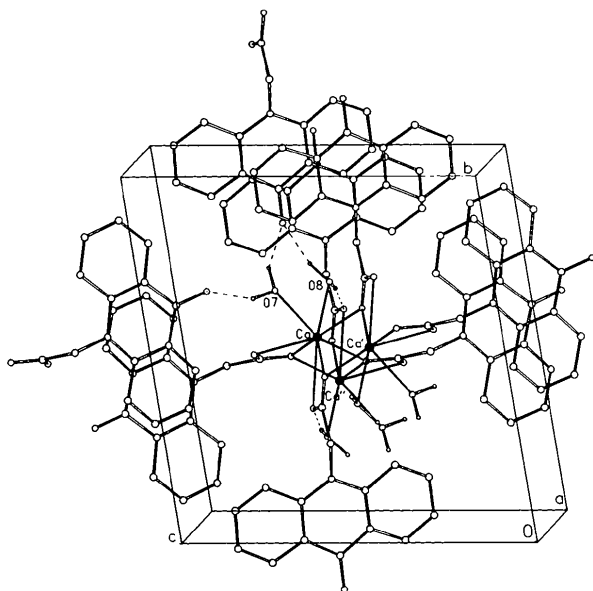


Fig. 2. The crystal packing of the Ca complex.

ordination number of eight as well as the chelate and bridging mode of the carboxylate ligands, are typical for calcium and are known for lead(II) complexes. Furthermore, a polymeric structure is commonly observed for Ca²⁺ and Pb²⁺ carboxylates. The bond lengths and bond angles for the metal coordination in [Ca(CMA)₂(H₂O)₂] are in agreement with the average values found for a series of calcium bidentate carboxylates (Einspahr & Bugg, 1980). The distances found in the PbO₈ polyhedron are characteristic for lead(II) carboxylates (Bensch & Gunter, 1987; Blake, Gould, Grant, Milne, Reed & Winpenny, 1994; Bryant, Chacko & Etter, 1984; Chandler, Hampden-Smith & Duesler, 1992; Głowiak, Kozłowski, Erre, Micera & Gulianti, 1992; Jones, Schelbach, Schwarzmann, Thone & Vielmader, 1988; Lis, 1984; Mak, Yip, O'Reilly, Smith & Kennard, 1985).

The successive MO₈ polyhedra share a common edge, which is defined by atoms O2A' and O2B. The deviations of the metal cations from the carboxylate group planes are not significant: 0.41 (1) (ligand A) and 0.19 (1) Å (ligand B), and 0.31 (1) (ligand A) and 0.35 (1) Å (ligand B) for the calcium and lead complex, respectively.

The angles between the planes through the carboxylate groups and the average acridone ring planes are 92.2 (A) and 76.3° (B) in [Ca(CMA)₂(H₂O)₂], and 97.4 (A) and 79.9° (B) in [Pb(CMA)₂(H₂O)₂], so that each carboxylate is approximately perpendicular to the corresponding acridone moiety.

The greater steric freedom in the lead complex due to the longer M—O distances as compared with the Ca complex, is reflected by the presence of a disordered water molecule, O7/O70 (site occupancy 50/50%) in [Pb(CMA)₂(H₂O)₂].

The water molecules are involved in hydrogen bonding within and between the polymer chains. The O8...O1A hydrogen bond is formed between the adjacent coordination polyhedra in one polymer chain, whereas the O8...O3A, O7...O3A and O7...O3B hydrogen bonds are to the 9-oxo O atoms of CMA⁻ in neighbouring polymer chains (see Tables 3 and 5).

The geometries of the acridone part of the ligands in (I) and (II) as well as in previously studied CMA⁻ complexes (Miernik, Lis, Palus & Reedijk 1993; Miernik & Lis, 1994) are very similar. In all the compounds the N atom lies out of the mean plane of the three coupled rings. The distortion from planarity is largest in the calcium and lead complexes (maximum deviation 0.20 Å).

Experimental

[Ca(CMA)₂(H₂O)₂] and [Pb(CMA)₂(H₂O)₂] were obtained from aqueous solutions (150 cm³), after Na(CMA) (1.0 mmol) and the metal salt [Ca(NO₃)₂ or Pb(NO₃)₂] (0.05 mmol) were mixed. After 2 days yellow crystals appeared.

Compound (I)

Crystal data

[Ca(C₁₅H₁₀NO₃)₂(H₂O)₂]

M_r = 580.59

Triclinic

*P*1̄

a = 7.402 (5) Å

b = 13.172 (6) Å

c = 13.686 (6) Å

α = 82.90 (4)°

β = 77.85 (4)°

γ = 86.53 (4)°

V = 1293.7 (12) Å³

Z = 2

D_s = 1.490 (2) Mg m⁻³

D_m = 1.50 Mg m⁻³

D_m measured by flotation in aqueous KI solution

Cu Kα radiation

λ = 1.5418 Å

Cell parameters from 25 reflections

θ = 10–21°

μ = 2.60 mm⁻¹

T = 287 (2) K

Needle like

0.15 × 0.10 × 0.04 mm

Yellow

Data collection

Kuma KM4 automated four-circle κ-axis diffractometer

ω/2θ scans

Absorption correction:

refined from Δ*F*

(*DIFABS*; Walker &

Stuart, 1983)

T_{min} = 0.73, *T_{max}* = 0.89

6809 measured reflections

5456 independent reflections

4130 observed reflections

[*I* > 3σ(*I*)]

R_{int} = 0.0152

θ_{max} = 75°

h = -8 → 2

k = -16 → 16

l = -16 → 17

3 standard reflections

monitored every 100

reflections

intensity decay: 1%

Refinement

Refinement on *F*²

R(*F*) = 0.0307

w*R*(*F*²) = 0.0847

S = 1.066

(Δ/σ)_{max} = -0.177

Δρ_{max} = 0.39 e Å⁻³

Δρ_{min} = -0.31 e Å⁻³

Extinction correction: none

4130 reflections
387 parameters
 $w = 1/[\sigma^2(F_o^2) + (0.05P)^2 + 0.38P]$
where $P = (F_o^2 + 2F_c^2)/3$

Atomic scattering factors
from *International Tables for Crystallography* (1992), Vol. C, Tables 4.2.6.8 and 6.1.1.4)

Compound (II)*Crystal data*[Pb(C₁₅H₁₀NO₃)₂(H₂O)₂] $M_r = 747.7$

Triclinic

P1

 $a = 7.637$ (5) Å $b = 13.174$ (7) Å $c = 13.749$ (5) Å $\alpha = 82.35$ (4)° $\beta = 78.00$ (4)° $\gamma = 85.04$ (5)° $V = 1338.5$ (12) Å³ $Z = 2$ $D_x = 1.855$ (3) Mg m⁻³ $D_m = 1.90$ Mg m⁻³ D_m measured by flotation in CH₂Br₂/CCl₄ solutionMo $K\alpha$ radiation $\lambda = 0.71069$ Å

Cell parameters from 25 reflections

 $\theta = 9-12^\circ$ $\mu = 6.36$ mm⁻¹ $T = 300$ (2) K

Irregular polyhedron

 $0.15 \times 0.15 \times 0.1$ mm

Yellow

Table 1. Fractional atomic coordinates and equivalent isotropic displacement parameters (Å²) for (I)
$$U_{eq} = (1/3)\sum_i \sum_j U_{ij} a_i^* a_j^* \mathbf{a}_i \cdot \mathbf{a}_j$$

	<i>x</i>	<i>y</i>	<i>z</i>	U_{eq}
Ca	0.22300 (4)	0.53862 (2)	0.54372 (2)	0.02236 (9)
O1A	0.2062 (2)	0.34694 (9)	0.58703 (11)	0.0398 (3)
O2A	0.4834 (2)	0.40790 (8)	0.56774 (9)	0.0325 (3)
O3A	0.1549 (2)	-0.14123 (9)	0.58053 (10)	0.0414 (3)
O1B	0.0236 (2)	0.50888 (14)	0.71135 (10)	0.0528 (4)
O2B	-0.1343 (2)	0.51843 (10)	0.59204 (9)	0.0375 (3)
O3B	-0.3339 (2)	0.34502 (12)	1.15529 (9)	0.0491 (4)
O7†	0.2956 (3)	0.66123 (11)	0.64875 (10)	0.0432 (6)
O70†	0.392 (3)	0.6100 (16)	0.6410 (13)	0.027 (6)
O8	0.1043 (2)	0.70143 (9)	0.47085 (11)	0.0397 (3)
N1A	0.3412 (2)	0.14437 (10)	0.58174 (11)	0.0290 (3)
N1B	-0.2861 (2)	0.46434 (11)	0.86077 (10)	0.0312 (3)
C1A	0.3749 (2)	0.33508 (11)	0.57817 (11)	0.0253 (3)
C2A	0.4644 (2)	0.22811 (12)	0.58008 (13)	0.0291 (4)
C3A	0.3733 (3)	0.17812 (14)	0.40095 (14)	0.0377 (4)
C4A	0.3622 (3)	0.1454 (2)	0.31096 (15)	0.0435 (5)
C5A	0.2983 (3)	0.0487 (2)	0.30745 (15)	0.0433 (5)
C6A	0.2438 (3)	-0.01374 (14)	0.39462 (15)	0.0373 (4)
C7A	0.1222 (3)	-0.06393 (14)	0.7662 (2)	0.0417 (5)
C8A	0.1143 (4)	-0.0242 (2)	0.8548 (2)	0.0511 (5)
C9A	0.1823 (4)	0.0729 (2)	0.8524 (2)	0.0526 (6)
C10A	0.2562 (3)	0.12863 (15)	0.76364 (15)	0.0427 (5)
C11A	0.2653 (3)	0.08905 (12)	0.67186 (13)	0.0299 (4)
C12A	0.1951 (3)	-0.00829 (12)	0.67352 (13)	0.0307 (4)
C13A	0.1991 (3)	-0.05172 (12)	0.58050 (14)	0.0313 (4)
C14A	0.2545 (2)	0.01647 (12)	0.48845 (13)	0.0294 (3)
C15A	0.3230 (2)	0.11346 (12)	0.49139 (13)	0.0290 (3)
C1B	-0.1233 (3)	0.51077 (13)	0.68280 (12)	0.0309 (4)
C2B	-0.3050 (3)	0.50828 (15)	0.75973 (12)	0.0337 (4)
C3B	-0.2002 (3)	0.6280 (2)	0.8982 (2)	0.0420 (4)
C4B	-0.1634 (3)	0.6877 (2)	0.9671 (2)	0.0502 (5)
C5B	-0.1728 (3)	0.6490 (2)	1.0672 (2)	0.0507 (5)
C6B	-0.2209 (3)	0.5501 (2)	1.09813 (15)	0.0417 (5)
C7B	-0.3447 (3)	0.2124 (2)	1.0120 (2)	0.0486 (5)
C8B	-0.3449 (4)	0.1496 (2)	0.9397 (2)	0.0622 (7)
C9B	-0.3194 (4)	0.1919 (2)	0.8394 (2)	0.0591 (6)
C10B	-0.2994 (3)	0.2942 (2)	0.81237 (15)	0.0450 (5)
C11B	-0.3026 (3)	0.36041 (14)	0.88646 (12)	0.0322 (4)
C12B	-0.3203 (3)	0.31732 (14)	0.98716 (13)	0.0339 (4)
C13B	-0.3087 (3)	0.38129 (15)	1.06548 (12)	0.0339 (4)
C14B	-0.2616 (3)	0.48672 (14)	1.03067 (13)	0.0327 (4)
C15B	-0.2508 (2)	0.52638 (14)	0.92904 (12)	0.0308 (4)

† Occupancy factors for O7 and O70 are 0.941(6) and 0.059(6), respectively.

Table 2. Selected geometric parameters (Å) for (I)

Ca—O2B ⁱ	2.316 (2)	Ca—O1A	2.526 (2)
Ca—O70	2.32 (2)	Ca—O2A	2.552 (2)
Ca—O7	2.443 (2)	Ca—O2B	2.609 (2)
Ca—O8	2.445 (2)	Ca...Ca ⁱ	3.967 (2)
Ca—O1B	2.453 (2)	Ca...Ca ⁱⁱ	4.125 (3)
Ca—O2A ⁱⁱ	2.464 (2)		

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

Table 3. Hydrogen-bonding geometry (Å, °) for (I)

<i>D</i> —H... <i>A</i>	H... <i>A</i>	<i>D</i> ... <i>A</i>	<i>D</i> —H... <i>A</i>
O7—H7...O3A ⁱ	1.93 (2)	2.865 (2)	160 (2)
O7—H71...O3B ⁱⁱ	1.82 (2)	2.746 (3)	158 (2)
O8—H8...O1A ⁱⁱⁱ	1.76 (2)	2.718 (3)	170 (2)
O8—H81...O3A ⁱ	1.88 (2)	2.791 (2)	156 (2)

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

*Data collection*Kuma KM4 automated four-circle κ -axis diffractometer $\omega/2\theta$ scans

Absorption correction:

refined from ΔF

(DIFABS; Walker &

Stuart, 1983)

 $T_{min} = 0.402$, $T_{max} =$

0.461

3727 measured reflections

3697 independent reflections

*Refinement*Refinement on F^2 $R(F) = 0.0284$ $wR(F^2) = 0.0724$ $S = 1.063$

3007 reflections

369 parameters

 $w = 1/[\sigma^2(F_o^2) + (0.035P)^2$ $+ 5.2P]$ where $P = (F_o^2 + 2F_c^2)/3$

3007 observed reflections

 $[I > 3\sigma(I)]$ $R_{int} = 0.018$ $\theta_{max} = 23^\circ$ $h = -8 \rightarrow 0$ $k = -14 \rightarrow 14$ $l = -14 \rightarrow 15$

3 standard reflections

monitored every 50

reflections

intensity decay: 10%

Table 4. Fractional atomic coordinates and equivalent isotropic displacement parameters (Å²) for (II)
$$U_{eq} = (1/3)\sum_i \sum_j U_{ij} a_i^* a_j^* \mathbf{a}_i \cdot \mathbf{a}_j$$

	<i>x</i>	<i>y</i>	<i>z</i>	U_{eq}
Pb	0.21218 (4)	0.54208 (2)	0.54888 (2)	0.02733 (11)
O1A	0.2281 (9)	0.3485 (5)	0.5722 (6)	0.048 (2)
O2A	0.4759 (9)	0.4036 (5)	0.6004 (5)	0.046 (2)
O3A	0.1482 (9)	-0.1254 (5)	0.5680 (5)	0.049 (2)
O1B	0.0233 (9)	0.4804 (6)	0.7214 (5)	0.054 (2)
O2B	-0.1189 (8)	0.5237 (5)	0.5969 (4)	0.037 (2)
O3B	-0.3349 (11)	0.3406 (6)	1.1567 (5)	0.060 (2)
N1A	0.3541 (9)	0.1461 (5)	0.5832 (6)	0.034 (2)
N1B	-0.2897 (10)	0.4535 (6)	0.8607 (5)	0.035 (2)
C1A	0.3837 (12)	0.3343 (6)	0.5880 (6)	0.032 (2)
C2A	0.4715 (11)	0.2265 (7)	0.5879 (7)	0.037 (2)
C3A	0.4012 (13)	0.1854 (7)	0.4013 (7)	0.042 (3)

C4A	0.3961 (14)	0.1581 (8)	0.3109 (8)	0.049 (3)
C5A	0.3243 (14)	0.0673 (8)	0.3030 (7)	0.052 (3)
C6A	0.2622 (13)	0.0041 (7)	0.3866 (7)	0.043 (3)
C7A	0.1060 (13)	-0.0531 (7)	0.7562 (7)	0.047 (3)
C8A	0.0932 (17)	-0.0163 (8)	0.8454 (9)	0.064 (3)
C9A	0.1668 (17)	0.0724 (9)	0.8482 (8)	0.067 (4)
C10A	0.2532 (14)	0.1275 (8)	0.7629 (7)	0.048 (3)
C11A	0.2667 (11)	0.0925 (6)	0.6695 (6)	0.033 (2)
C12A	0.1899 (11)	0.0005 (6)	0.6666 (7)	0.037 (2)
C13A	0.1977 (11)	-0.0388 (6)	0.5716 (7)	0.033 (2)
C14A	0.2663 (11)	0.0294 (6)	0.4820 (7)	0.034 (2)
C15A	0.3422 (11)	0.1206 (7)	0.4901 (7)	0.034 (2)
C1B	-0.1166 (12)	0.4978 (6)	0.6888 (6)	0.030 (2)
C2B	-0.2993 (11)	0.4958 (7)	0.7591 (6)	0.035 (2)
C3B	-0.2077 (13)	0.6174 (7)	0.8953 (7)	0.045 (3)
C4B	-0.1724 (15)	0.6775 (9)	0.9633 (9)	0.058 (3)
C5B	-0.1854 (15)	0.6404 (10)	1.0617 (9)	0.062 (3)
C6B	-0.2310 (14)	0.5424 (9)	1.0956 (8)	0.051 (3)
C7B	-0.3432 (15)	0.2035 (8)	1.0174 (8)	0.057 (3)
C8B	-0.3474 (18)	0.1398 (9)	0.9462 (9)	0.070 (4)
C9B	-0.3197 (18)	0.1803 (9)	0.8449 (10)	0.070 (4)
C10B	-0.3016 (14)	0.2815 (8)	0.8172 (7)	0.048 (3)
C11B	-0.3033 (11)	0.3498 (7)	0.8886 (6)	0.036 (2)
C12B	-0.3221 (12)	0.3092 (7)	0.9903 (6)	0.038 (3)
C13B	-0.3120 (12)	0.3729 (8)	1.0656 (7)	0.043 (3)
C14B	-0.2678 (11)	0.4796 (7)	1.0301 (7)	0.037 (3)
C15B	-0.2583 (11)	0.5177 (7)	0.9269 (7)	0.036 (2)
O8	0.0841 (9)	0.7262 (5)	0.4496 (6)	0.055 (2)
O7†	0.163 (3)	0.6875 (11)	0.6801 (11)	0.052 (4)
O71†	0.249 (2)	0.6800 (10)	0.6620 (10)	0.041 (4)

† The assumed occupancy factor for atoms O7 and O71 is 0.5.

Table 5. Selected geometric parameters (Å) for (II)

Pb—O2B	2.501 (6)	Pb···Pb ⁱ	4.046 (3)
Pb—O1A	2.523 (6)	Pb···Pb ⁱⁱ	4.411 (3)
Pb—O2B ⁱ	2.538 (6)	O8···O1A ⁱ	2.743 (9)
Pb—O1B	2.576 (6)	O8···O3A ⁱⁱⁱ	2.838 (9)
Pb—O71	2.611 (13)	O7···O1B	2.957 (16)
Pb—O2A	2.739 (6)	O7···O3A ⁱⁱⁱ	2.735 (15)
Pb—O7	2.753 (14)	O7···O3B ^{iv}	2.795 (16)
Pb—O8	2.824 (6)	O71···O3A ⁱⁱⁱ	2.832 (14)
Pb—O2A ⁱⁱ	2.878 (6)	O71···O3B ^{iv}	2.682 (14)

Symmetry codes: (i) $-x, 1-y, 1-z$; (ii) $1-x, 1-y, 1-z$; (iii) $x, 1+y, z$; (iv) $-x, 1-y, 2-z$.

Data collection, cell refinement and data reduction were performed using Kuma KM4 diffractometer software. In the case of (II), the standard reflections decreased in intensity by approximately 10% over the period of data collection; the data were therefore rescaled using the intensities of the standards. The structure of [Ca(CMA)₂(H₂O)₂] was solved by direct methods. The refinement of [Pb(CMA)₂(H₂O)₂] was initiated using the heavy-atom coordinates of [Ca(CMA)₂(H₂O)₂]. In both structures the C-bonded H atoms were included in geometrically calculated positions. The water H atoms in the Ca salt were found in a difference map and were refined with the restraint O—H = 0.97 Å. In the case of the Ca crystal, after refinement an additional significant peak was found on a difference Fourier map near the O(7) water molecule. It was admitted as a partially disordered O7 atom and assigned as O70. In the case of the Pb crystal, one water molecule was found to be disordered over two positions with occupancy factors of 0.5; the water H atoms were not resolvable. Refinement was performed using SHELXL93 (Sheldrick, 1993).

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: CF1027). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

References

- Bensch, W. & Gunter, J. R. (1987). *Z. Kristallogr.* **178**, 257–262.
 Blake, A. J., Gould, R. O., Grant, C. M., Milne, P. Y. E., Reed, D. & Winpenny, R. E. P. (1994). *Angew. Chem.* **33**, 195–197.
 Bryant, R. G., Chacko, V. P. & Etter, M. C. (1984). *Inorg. Chem.* **23**, 3580–3584.
 Chandler, C. D., Hampden-Smith, M. J. & Duesler E. N. (1992). *Inorg. Chem.* **31**, 4891–4893.
 Einspahr, H. & Bugg, C. E. (1980). *Acta Cryst.* **B37**, 1044–1052.
 Glowiak, T., Kozłowski, H., Erre, L. S., Micera, G. & Gulinati, B. (1992). *Inorg. Chim. Acta*, **202**, 43–47.
 Inglot, A. D., Młochowski, J., Szulc, Z., Inglot, O. & Albin, M. (1985). *Arch. Immun. Ther. Exp.* **33**, 275–285.
 Jones, P. G., Schelbach, R., Schwarzmann, E., Thone, C. & Vielmader, A. (1988). *Acta Cryst.* **C44**, 1198–1200.
 Lis, T. (1984). *Acta Cryst.* **C40**, 374–376.
 Mak, T. C. W., Yip, W.-H., O'Reilly, E. J., Smith, G. & Kennard, C. H. L. (1985). *Inorg. Chim. Acta*, **100**, 267–273.
 Miernik D. & Lis T. (1994). *J. Chem. Cryst.* **24**, 731–737.
 Miernik, D., Lis, T., Palus, J. & Reedijk, J. (1993). *Inorg. Chim. Acta*, **205**, 231–237.
 Sheldrick, G. M. (1993). *SHELXL93. Program for the Refinement of Crystal Structures*. University of Göttingen, Germany.
 Walker, N. & Stuart, D. (1983). *Acta Cryst.* **A39**, 158–166.

Acta Cryst. (1996). **C52**, 1174–1177

Tris(1,10-phenanthroline)ruthenium(II) Bis(hexafluorophosphate)

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Abstract

[Ru(phen)₃](PF₆)₂ (phen = 1,10-phenanthroline), [Ru(C₁₂H₈N₂)₃](PF₆)₂, is homostructural with the perchlorate monohydrates of the Cu^{II}, Co^{II}, Os^{II} and Fe^{II} analogues. This homomorphism is notable in view of the widely differing metal-atom environments and counterion sizes, and suggests that the packing arrangement is a general and accommodating one among tris(1,10-phenanthroline)metal(II) complexes. While [Ru(bpy)₃](PF₆)₂ is built up from homochiral layers, [Ru(phen)₃](PF₆)₂ is composed of