

The ratios of the height to base in the trigonal prisms around Ga(1), Ga(5) and Ga(6) show values much higher than unity (elongated prisms), so that the distances of Ga to Ca at the vertices of the prism are larger than the waist contacts.

The calcium atoms, distributed over fifteen site sets, have polyhedra that are identical or closely related to each other, with coordination numbers from 11 to 15. For example, Ca(1) and Ca(3) are surrounded by 4Ga + 9Ca in the same manner, while Ca(15) is lacking a Ca atom; an identical polyhedron formed by 4Ga + 10Ca is found around Ca(2), Ca(4) and Ca(12); the same applies to Ca(6), Ca(10) and Ca(13) coordinated with 3Ga + 10Ca, and to Ca(8) and Ca(11) coordinated with 3Ga + 11Ca.

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## Structure of Hexakis(1-methylimidazole)cadmium(II) Nitrate and its <sup>113</sup>Cd Solution and Solid-State NMR Spectrum

BY P. F. RODESILER\*

*Department of Chemistry, Columbia College, Columbia, South Carolina 29203, USA*

AND N. G. CHARLES, E. A. H. GRIFFITH, K. LEWINSKI AND E. L. AMMA†

*Department of Chemistry, University of South Carolina, Columbia, South Carolina 29208, USA*

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**Abstract.** [Cd(C<sub>4</sub>H<sub>6</sub>N<sub>2</sub>)<sub>6</sub>](NO<sub>3</sub>)<sub>2</sub>, *M<sub>r</sub>* = 729.04, monoclinic, *P*2<sub>1</sub>/*n*, *a* = 11.335 (3), *b* = 7.726 (1), *c* = 19.059 (3) Å, β = 95.60 (2)°, *V* = 1661 (1) Å<sup>3</sup>, *Z* = 2, *D<sub>m</sub>* = 1.47 (2), *D<sub>x</sub>* = 1.46 g cm<sup>-3</sup>, λ(Mo *K*α) = 0.71073 Å, μ = 7.18 cm<sup>-1</sup>, *F*(000) = 748, *T* ~ 293 K, *R* = 0.051 for 3060 observed reflections. The structure may be described as isolated hexakis(1-methylimidazole)Cd<sup>II</sup> ions and disordered nitrate ions separated by ordinary van der Waals distances. The coordination polyhedron of Cd<sup>II</sup> may be described as a slightly tetragonally compressed octahedron of N atoms from the non-methylated N atoms of the imidazoles. The Cd atom is on a center of symmetry

with Cd–N distances of 2.366 (6), 2.362 (6) and 2.331 (5) Å. The solid-state cross-polarization/magic-angle spinning, CP/MAS, <sup>113</sup>Cd NMR signal is observed at 230 p.p.m. and the methanol solution signal is observed at 177 p.p.m., both deshielded relative to the 0.1 mol dm<sup>-3</sup> Cd(ClO<sub>4</sub>)<sub>2</sub> in D<sub>2</sub>O standard.

**Introduction.** <sup>113</sup>Cd NMR has been shown to be a versatile probe of the metal-ion site in compounds ranging from Cd-containing organometallics to Zn, Ca enzymes in which the naturally occurring metal has been replaced by Cd. In some of these enzymes the chemical shifts are such that few, if any, model compounds are known. For example, the values found for calmodulin and troponin C are at ~–100 p.p.m. relative to the 0.1 *M* Cd(ClO<sub>4</sub>)<sub>2</sub> in D<sub>2</sub>O standard. A

\* Deceased May 1985.

† To whom correspondence should be addressed.

review (Ellis, 1983) and a publication (Rodesiler, Turner, Charles, Griffith & Amma, 1984) are available in which extensive and comprehensive up-to-date references are available. The development of solid-state cross-polarization magic-angle spinning (CP/MAS)  $^{113}\text{Cd}$  NMR has broadened the scope of this probe (Ackerman, Orr, Bartuska & Maciel, 1979; Mennitt, Shatlock, Bartuska & Maciel, 1981). The combination of X-ray crystallography and solid-state (CP/MAS) data offers an excellent opportunity to study and correlate metal-ion geometry with  $^{113}\text{Cd}$  NMR chemical shift and by this means to convert  $^{113}\text{Cd}$  NMR into a much more useful chemical tool. The ultimate goal of such studies is to be able to observe the  $^{113}\text{Cd}$  NMR signal in the solid or in solution and to be able to describe the ligands and the stereochemistry of these ligands about the Cd atom or the stereochemistry of a metal ion which has been substituted by Cd. To this end we have been actively synthesizing compounds, determining their crystal structures, and measuring the CP/MAS  $^{113}\text{Cd}$  NMR and the solution  $^{113}\text{Cd}$  NMR as well. We report here a portion of this ongoing research program.

**Experimental.** A solution containing 1.64 g (0.02 mol) 1-methylimidazole (Aldrich) was added slowly with stirring to a solution of 1.54 g (0.005 mol)  $\text{Cd}(\text{NO}_3)_2 \cdot 2\text{H}_2\text{O}$  (Fisher). The total volume of approximately 145 ml was heated gently (330 K) for 45 min, then allowed to evaporate at ambient temperature. Diffraction-quality crystals were collected after one week and protected from moisture since they are somewhat hygroscopic. The crystals were collected by gravity filtration and either sealed in thin-walled capillaries for X-ray diffraction studies or stored for NMR measurements. Enraf-Nonius CAD-4 diffractometer interfaced to a PDP-11/60, crystal aligned and intensity data collected by standard techniques (Enraf-Nonius, 1982).  $D_m$  by flotation.  $h0l$ ,  $h + l = 2n + 1$ ,  $0k0$ ,  $k = 2n + 1$  absent; crystal  $0.30 \times 0.69 \times 0.30$  mm; faces: (001), (00 $\bar{1}$ ), (010), (1 $\bar{1}0$ ), ( $\bar{1}\bar{1}0$ ), (100), ( $\bar{1}00$ ); absorption correction, max., min. transmission factors 0.831, 0.805 (from *SDP* package, see below); graphite monochromator,  $\theta = 6.1^\circ$ ;  $p = 0.030$  in  $\sigma(F_o^2) = [\sigma(I_{\text{raw}})^2 + (pI_{\text{raw}})^2]^{1/2}/Lp$  and  $w = 1/\sigma(F_o^2)$ ; data considered non-zero if  $F_o^2 > 4\sigma(F_o^2)$ , 4306 independent  $hkl$ 's measured in  $\omega$ - $2\theta$  mode to  $2\theta_{\text{max}} = 60^\circ$ ,  $hkl$  range 0-14, 0-10, -23-24; 3060 reflections used; 25 general reflections used in orientation matrix (checked every 24 h) and used for all parameter measurements; three standard reflections monitored every 100 reflections ( $255$ ,  $4,2,10$ ,  $354$ ), decay  $< 1\%$   $I$ ; variable scan speed with preliminary scan speed of  $4^\circ \text{ min}^{-1} 2\theta$ ; structure solved by standard heavy-atom methods; structure refined by full-matrix least squares, anisotropic temperature factors (except for disordered nitrate groups, see below) and anomalous-dispersion

corrections with weights based upon intensity statistics; function minimized  $\sum_i w_i [|F_o|_i - |F_c|_i]^2$ ; final least squares performed on a VAX-780 with *SHELX76* (Sheldrick, 1976) with  $f, f', f''$  from Ibers & Hamilton (1974) for  $\text{Cd}^{2+}$  and all others as neutral atoms; largest shift at end of refinement (except for nitrate groups)  $= 0.04\sigma$ , av. shift  $< 0.02\sigma$ , No. of variables 190, final  $R = 0.051$ ,  $wR = 0.062$ ;  $S = 1.31$ ; H-atom contributions calculated from idealized positions; highest peak in final difference Fourier map  $0.85 \text{ e } \text{\AA}^{-3}$  and deepest hole  $0.53 \text{ e } \text{\AA}^{-3}$ ; all computer programs used either those in Enraf-Nonius *SDP* package (Frenz, 1982) on the PDP-11/60 or the *SHELX* system on the VAX-780; *ORTEPII* (Johnson, 1976) calculated on an Amdahl V6.

The atomic coordinate parameters and e.s.d.'s are listed in Table 1.\* Selected interatomic distances and angles and e.s.d.'s are listed in Table 2. The nitrate groups were found to be disordered, and our best approximation to the disorder and refinement thereof was in terms of two rigid, planar idealized nitrate groups: N(7a), O(1a), O(2a), O(3a); N(7b), O(1b), O(2b), O(3b) with a dihedral angle between planes of  $51.1(5)^\circ$  and a relative occupancy of 0.68 and 0.32 respectively. A figure showing this disorder is in the supplementary data. Individual atoms were allowed variable isotropic temperature factors. Some small peaks remained in the final difference electron density map indicating a more complex disorder, but it was deemed unprofitable to pursue a more complex disordered model. The experimental conditions and equipment for the  $^{113}\text{Cd}$  NMR have been described elsewhere (Charles, Griffith, Rodesiler & Amma, 1983).

**Discussion.** The crystal structure of hexakis(1-methylimidazole) $\text{Cd}^{\text{II}}$  nitrate may be described as isolated complex ions of  $\text{Cd}^{\text{II}}$  and disordered nitrate ions with only normal van der Waals distances between these entities, Fig. 1 (the disordered groups are not shown for the sake of clarity). The  $\text{Cd}^{\text{II}}$  complex ion, with the Cd atom on a center of symmetry, is almost regular octahedral with N donor atoms from the 3 position of each of the six 1-methylimidazole ligands, Fig. 2. The three independent Cd-N distances are 2.366 (6), 2.362 (6) and 2.331 (5) Å. The last Cd-N distance corresponds to a slight axial compression of the octahedron. These distances are the expected values for Cd-N in this type of Cd complex ion (Rodesiler *et al.*, 1984; Mighell & Santoro, 1971).

\* Lists of structure factors, anisotropic thermal parameters, H-atom parameters, details of least-squares planes and a figure detailing the nitrate disorder have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 42641 (21 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 1. Fractional atomic coordinates with *e.s.d.*'s in parentheses and equivalent isotropic temperature factors

$$B_{eq} = \frac{1}{3} \pi^2 \sum_i \sum_j U_{ij} a_i^* a_j^* a_i \cdot a_j$$

	x	y	z	$B_{eq}(\text{\AA}^2)$
Cd*	0.0000	0.0000	0.0000	4.49
N(1)	0.3541 (3)	0.2464 (6)	0.0480 (2)	5.88
N(2)	0.1700 (3)	0.1615 (4)	0.0339 (2)	4.94
C(1)	0.2784 (4)	0.1304 (7)	0.0186 (3)	6.46
C(2)	0.1785 (4)	0.3042 (7)	0.0743 (3)	5.96
C(3)	0.2924 (5)	0.3593 (7)	0.0840 (3)	6.60
C(4)	0.4839 (4)	0.2472 (11)	0.0420 (4)	8.53
N(3)	0.1435 (4)	-0.3144 (5)	0.1895 (2)	6.21
N(4)	0.0469 (4)	-0.1804 (5)	0.0997 (2)	5.56
C(5)	0.1523 (5)	-0.2055 (6)	0.1363 (3)	5.83
C(6)	-0.0313 (5)	-0.2830 (7)	0.1304 (3)	6.93
C(7)	0.0268 (7)	-0.3668 (8)	0.1862 (4)	7.83
C(8)	0.2419 (8)	-0.3741 (10)	0.2391 (4)	9.25
N(5)	0.2246 (3)	-0.2716 (6)	-0.1481 (2)	6.16
N(6)	0.1128 (3)	-0.1791 (5)	-0.0693 (2)	5.32
C(9)	0.1485 (5)	-0.1483 (7)	-0.1312 (3)	6.69
C(10)	0.1693 (5)	-0.3274 (7)	-0.0461 (3)	6.31
C(11)	0.2378 (4)	-0.3862 (7)	-0.0948 (4)	6.52
C(12)	0.2893 (8)	-0.2728 (13)	-0.2106 (4)	10.93
N(7a)†	0.4697 (5)	0.2226 (8)	0.8485 (3)	8.53
O(1a)	0.3731 (5)	0.1803 (8)	0.8701 (3)	11.68
O(2a)	0.4900 (5)	0.3774 (8)	0.8349 (3)	14.28
O(3a)	0.5469 (5)	0.1106 (8)	0.8405 (3)	12.85
N(7b)	0.4826 (13)	0.2191 (21)	0.8547 (8)	8.53
O(1b)	0.5879 (13)	0.2088 (21)	0.8808 (8)	10.79
O(2b)	0.4234 (13)	0.3514 (21)	0.8652 (8)	16.05
O(3b)	0.4376 (13)	0.0970 (21)	0.8183 (8)	22.68

\* Cd is on an invariant special position.

† N(7a), O(1a), O(2a), O(3a) comprise one part of a disordered nitrate group with occupancy 0.68 and the other part is N(7b), O(1b), O(2b), O(3b) with occupancy 0.32.

Table 2. Interatomic distances ( $\text{\AA}$ ) and bond angles ( $^\circ$ ) with *e.s.d.*'s in parentheses

Cd*—N(2)	2.333 (3)	N(3)—C(8)	1.464 (9)
Cd—N(4)	2.375 (4)	N(4)—C(5)	1.337 (7)
Cd—N(6)	2.371 (4)	N(4)—C(6)	1.363 (7)
N(1)—C(1)	1.327 (6)	C(6)—C(7)	1.359 (9)
N(1)—C(3)	1.347 (7)	N(5)—C(9)	1.345 (7)
N(1)—C(4)	1.487 (5)	N(5)—C(11)	1.345 (8)
N(2)—C(1)	1.312 (5)	N(5)—C(12)	1.459 (9)
N(2)—C(2)	1.343 (6)	N(6)—C(9)	1.306 (7)
C(2)—C(3)	1.355 (7)	N(6)—C(10)	1.365 (7)
N(3)—C(5)	1.329 (7)	C(10)—C(11)	1.346 (8)
N(3)—C(7)	1.379 (8)		
N(2)—Cd—N(4)	88.7 (1)	Cd—N(4)—C(5)	128.4 (3)
N(2)—Cd—N(6)	89.5 (1)	Cd—N(4)—C(6)	125.5 (3)
N(4)—Cd—N(6)	90.7 (1)	C(5)—N(4)—C(6)	106.1 (4)
C(1)—N(1)—C(3)	107.8 (4)	N(3)—C(5)—N(4)	111.1 (4)
C(1)—N(1)—C(4)	125.1 (4)	N(4)—C(6)—C(7)	109.1 (5)
C(3)—N(1)—C(4)	127.1 (4)	N(3)—C(7)—C(6)	106.6 (6)
Cd—N(2)—C(1)	127.3 (3)	C(9)—N(5)—C(11)	108.2 (4)
Cd—N(2)—C(2)	127.5 (3)	C(9)—N(5)—C(12)	125.8 (5)
C(1)—N(2)—C(2)	105.2 (4)	C(11)—N(5)—C(12)	125.8 (5)
N(1)—C(1)—N(2)	111.3 (4)	Cd—N(6)—C(9)	128.9 (3)
N(2)—C(2)—C(3)	110.5 (4)	Cd—N(6)—C(10)	124.8 (3)
N(1)—C(3)—C(2)	105.2 (4)	C(9)—N(6)—C(10)	105.6 (4)
C(5)—N(3)—C(7)	107.1 (5)	N(5)—C(9)—N(6)	110.5 (4)
C(5)—N(3)—C(8)	125.7 (5)	N(6)—C(10)—C(11)	110.1 (5)
C(7)—N(3)—C(8)	127.1 (5)	N(5)—C(11)—C(10)	105.6 (5)

\* All distances and angles are calculated with *e.s.d.*'s for Cd = 0 and consequently are somewhat optimistic.

The sums of the bond angles around N(2), N(4) and N(6) show that the donor orbital of the ring is a nitrogen  $sp^2$ -hybridized orbital. It is interesting to note that the Cd is out of the imidazole ring plane for only the imidazoles defined by N(2) and N(6) by 0.05 (1) and 0.29 (1)  $\text{\AA}$  respectively. This displacement is most likely due to some steric interactions between these rings. The dihedral angles between the planes are: N(2) and N(4), 87.7 (1); N(2) and N(6), 84.4 (1); and N(4) and N(6), 61.8 (1) $^\circ$ .

The bond distances and angles within the 1-methylimidazole ring are normal and the expected values relative to imidazole (Martinez-Carrera, 1966).

The solid-state cross-polarization magic-angle spinning (CP/MAS)  $^{113}\text{Cd}$  NMR shows a single peak at 230 p.p.m. and the methanol solution spectrum shows a single peak at 177 p.p.m., both deshielded relative to the 0.1M Cd(ClO<sub>4</sub>)<sub>2</sub> in D<sub>2</sub>O standard. In addition, the

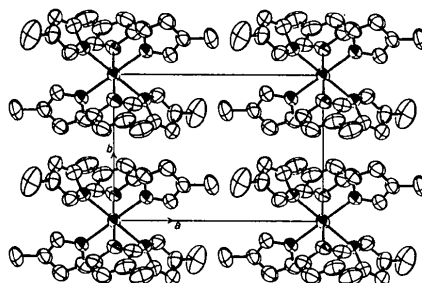


Fig. 1. A projection of part of the contents of the unit cell onto the *ab* plane. For simplicity the disordered nitrate groups are not shown. In addition, the Cd and associated ligands at  $\frac{1}{2}, \frac{1}{2}, \frac{1}{2}$  are not shown because the overlap of atoms is such that little of utility can be seen. For the same reason the rest of the unit cell is not shown. The Cd atoms and the six N atoms bound to them are shown as cross-hatched ellipses, whereas the remaining atoms are shown only by the ellipse boundaries. The ellipses are drawn at the 50% probability level.

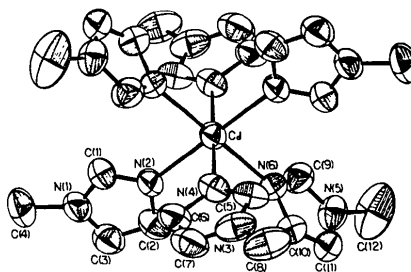


Fig. 2. An ORTEPII view of an isolated hexakis(1-methylimidazole)Cd<sup>II</sup> species with atom notation consistent with tables and text. The Cd atom resides on a center of symmetry and consequently only the independent atoms are labeled. The thermal ellipsoids are drawn at the 50% probability level.

solution spectrum shows some concentration dependence. This is to be compared with the 238 p.p.m. observed by Mennitt *et al.* (1981) for hexakis(imidazole)Cd<sup>II</sup> in the solid state. Clearly, the species in methanol is not the species that exists in the solid state, and either the coordination sphere is expanded with one or more solvent molecules or some of the nitrogen ligands are replaced by solvent molecules. The former is the more likely since we have shown (Rodesiler, Griffith, Charles & Amma, 1985) a regular six-coordinate Cd species can readily add a seventh ligand to make the metal geometry seven-coordinate pentagonal bipyramidal. We have also shown that the addition of the oxygen donor from the solvent methanol shifts the <sup>113</sup>Cd NMR signal to more shielded values by approximately 70 p.p.m. (Rodesiler & Amma, 1982) as is observed in this case. However, the latter possibility cannot be completely ruled out because of the concentration dependence.

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## Structures of Bis[tricarbonyl(trimethylphosphine)cobalt] (1) and Dicarbonylchlorobis(trimethylphosphine)cobalt (2)

BY R. A. JONES,\* M. H. SEEBERGER, A. L. STUART, B. R. WHITTLESEY AND T. C. WRIGHT

*Department of Chemistry, University of Texas, Austin, Texas 78712, USA*

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**Abstract.** (1), [Co<sub>2</sub>(CO)<sub>6</sub>{P(CH<sub>3</sub>)<sub>3</sub>}]<sub>2</sub>, *M<sub>r</sub>* = 438.08, cubic, *Pa*3, *a* = 12.4539 (18) Å, *V* = 1931.6 Å<sup>3</sup>, *Z* = 4, *D<sub>x</sub>* = 1.507 g cm<sup>-3</sup>, λ(Mo *K*α) = 0.71069 Å, μ = 18.977 cm<sup>-1</sup>, *F*(000) = 888, *T* = 296 (2) K, final *R* = 0.0272 for 435 unique observations. The structure consists of discrete molecules of [Co<sub>2</sub>(CO)<sub>6</sub>{P(CH<sub>3</sub>)<sub>3</sub>}]<sub>2</sub>. There is a Co–Co bond of 2.669 (1) Å. The geometry about each Co atom is trigonal bipyramidal with three equatorial carbonyls and an apical PMe<sub>3</sub> molecule *trans* to the other Co atom. (1) is a product of the reaction of [Ni{*μ-tert*-Bu(H)P}(PMe<sub>3</sub>)<sub>2</sub>]<sub>2</sub> and [Co<sub>2</sub>(CO)<sub>8</sub>] in toluene solution. (2),

[CoCl(CO)<sub>2</sub>{P(CH<sub>3</sub>)<sub>3</sub>}]<sub>2</sub>, *M<sub>r</sub>* = 302.57, orthorhombic, *Pnma*, *a* = 9.793 (7), *b* = 10.602 (4), *c* = 13.590 (1) Å, *V* = 1411.2 (2) Å<sup>3</sup>, *Z* = 4, *D<sub>x</sub>* = 1.424 g cm<sup>-3</sup>, λ(Mo *K*α) = 0.71069 Å, μ = 16.06 cm<sup>-1</sup>, *F*(000) = 624, *T* = 296 (2) K, final *R* = 0.0512 for 673 unique observed reflections. The structure of (2) has an overall trigonal-bipyramidal configuration with *trans* PMe<sub>3</sub> groups and two carbonyls and a Cl in the equatorial plane. The Cl and two CO groups which occupy the equatorial positions are disordered and the structure was refined successfully using partial occupancies of 1/3 Cl, 2/3 C and 2/3 O at the appropriate sites. (2) is the product of the reaction of (CO)<sub>5</sub>Mo(*tert*-Bu<sub>2</sub>P)Li with [CoCl<sub>2</sub>(PMe<sub>3</sub>)<sub>2</sub>] in THF at 183 K.

\* To whom correspondence should be addressed.

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