

# Synthesis and solid-state structure of magnesium and calcium imidazole complexes

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## Abstract

Three octahedral complexes built from *N*-alkylsubstituted imidazoles and magnesium as well as calcium chloride are reported. The obtained solid-state structures differ significantly from each other, depending on the size of the metal ion and the substituent on the imidazole. A chloro-bridged structure is found in the case of *tert*-butylimidazole, while for the *iso*-propylimidazole the calcium ion is coordinated by six imidazoles. For the smaller magnesium cation, we also found a sixfold coordination, but here only three of the *iso*-propylimidazoles coordinate to the magnesium, the three other positions are taken by water molecules.

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## 1. Introduction

In the literature, complexes between nitrogen containing compounds and alkaline-earth metals are not unusual. The coordination number of these complexes is usually in between two and six [1–5]. Complex **1**, a diaqua-dichloro-bis(histamine)-calcium diaqua-tetrachloro-calcium ion pair (Fig. 1) [6], is an example for a solid-state structure which consists of calcium and imidazole, where only two imidazoles (as histamine) are coordinated to the calcium ion. Based on spectroscopic evidence, it was also reported in the literature, that calcium [7] or magnesium [7–9] ions can be coordinated by six *N*-methyl imidazoles (**2**), but no solid-state structure is available (Fig. 1).

Other related examples like the solid-state structures of complexes between magnesium and calcium halides and 2,2':6',2''-terpyridine (**3**, **4**) [3] (Fig. 2) have been presented in the last decade by White et al. [3–5]. Halut-Desportes

reported a solid-state structure where three pyridines and three water molecules are coordinated to a magnesium ion (**5**) [10].

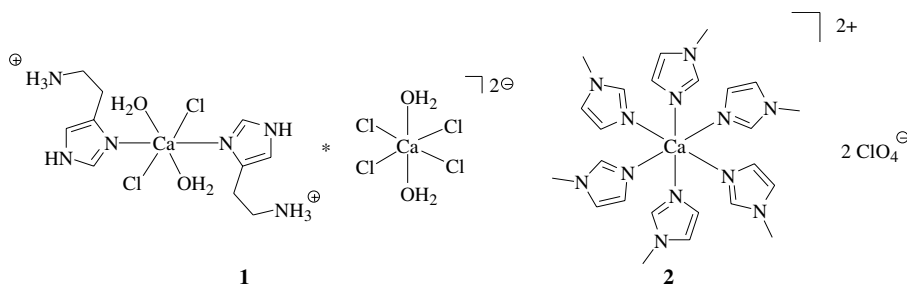
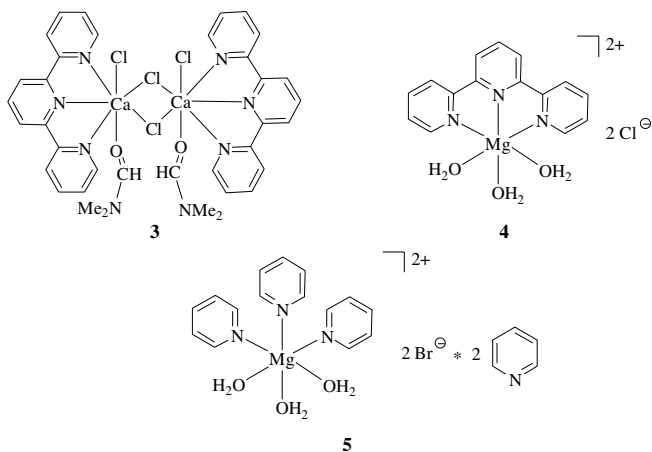
But although we conducted a thorough literature search, we did not find many examples. There are several examples of compounds where different ligands are coordinated to a central Ca(Cl<sub>2</sub>)Ca-unit [3,11–16], but to the best of our knowledge complex **3** is the only example for a solid-state structure where imidazoles are coordinated to calcium ions forming a chloro-bridged structure between two calcium ions. Three nitrogen atoms of the ligand are coordinated to each calcium ion, the sixth position is filled by a solvent molecule (DMF) [3]. The corresponding magnesium complex **4** consists of only one terpyridine ligand and three water molecules. It is a common feature of these complexes, that magnesium coordinates only three nitrogen atoms and three molecules of water (e.g. **5**) [3,10].

The reported synthesis of complex **2** is different from our preparation method. Complex **2** was prepared from calcium perchlorate tetrahydrate and *N*-methylimidazole in ethanol. For the dehydration of the calcium precursor, triethyl orthoformate was added to obtain the resulting complex [7]. Our approach may be more time-consuming, but

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<sup>1</sup> X-ray analysis.

Fig. 1. Structures of complexes **1** [6] and **2** [7].Fig. 2. Examples for different coordination modes of *N*-heterocycles to calcium and magnesium ions [3,10].

does not require a dehydration agent and it has been shown that it works with different imidazoles.

We report a new preparation method for magnesium and calcium complexes with *iso*-propylimidazole and *tert*-butylimidazole ligands and discuss their structural differences.

## 2. Experimental

### 2.1. General details

Solvents of 99.5% purity were used throughout this study. The magnesium- and calcium chloride were obtained from common suppliers and used without further purification. The *iso*-propyl- and *tert*-butylimidazoles (**6**) and (**7**) which are used for the synthesis of complexes **8–10** were prepared according to the literature procedures [17].  $^1\text{H}$  and  $^{13}\text{C}$  NMR spectra were recorded with a Bruker AC 300 P spectrometer. The spectra were referenced internally to the references of the solvent ( $\text{CDCl}_3$ ). The melting-points were measured with an Electrothermal 9100 melting-point apparatus and are uncorrected.

### 2.2. Synthesis of the complexes

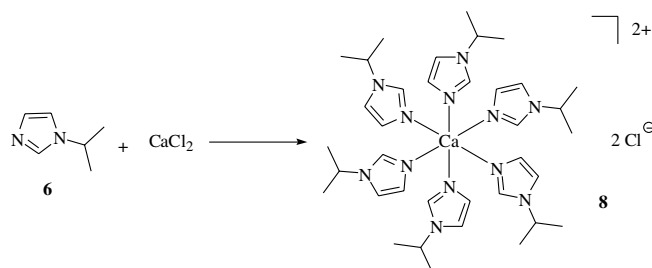
#### 2.2.1. Hexa-3-(1-*iso*-propylimidazole)calcium(II)dichloride [ $\text{Ca}(\text{iprIm})_6$ ] $\text{Cl}_2$ (**8**)

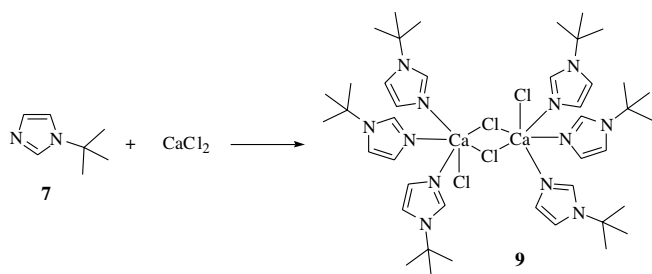
2.25 mmol calcium chloride (0.250 g) was added to 4.5 mmol 1-*iso*-propylimidazole (**6**) (0.500 g) (see Scheme 1).

This mixture has to rest until the imidazole has been absorbed by the calcium chloride. The resulting solid material was carefully washed with methylene chloride and filtrated. After removal of the solvent a white solid was obtained, which was suspended in tetrahydrofuran, filtrated, and washed with tetrahydrofuran. After removal of the solvent from the obtained filtrate, the yellow oily liquid was put in the refrigerator for crystallisation. Yield: 0.107 g (6.2%).  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , 300.13 MHz):  $\delta$  = 7.52 (s, 6H, NCHN); 6.96 (s, 6H, NCHCHN); 6.88 (s, 6H, NCHCHN); 4.27 (quint,  $J$  = 6.7 Hz, 6H, NCH( $\text{CH}_3$ ) $_2$ ); 1.38 (d,  $J$  = 6.7 Hz, 36H,  $\text{CH}_3$ -group of *iso*-prop) ppm.  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , 75.475 MHz):  $\delta$  = 135.01 (NCHN); 128.56 (NCHCHN); 116.30 (NCHCHN); 48.86 (NCH( $\text{CH}_3$ ) $_2$ ); 23.38 ( $\text{CH}_3$ -group of *iso*-prop) ppm. M.p. = 20 °C.

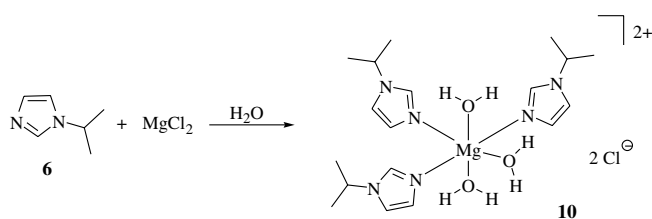
#### 2.2.2. $\mu^2$ -Dichloro-hexa-3-(1-*tert*-butylimidazole)-calcium(II)chloride [ $(\text{tBuIm})_3\text{CaCl}(\mu^2\text{-Cl})_2\text{-ClCa}(\text{tBuIm})_3$ ] (**9**)

2.0 mmol calcium chloride (0.222 g) was added to 4.0 mmol 1-*tert*-butylimidazole (**7**) (0.500 g) (see Scheme 2). This mixture has to rest until the whole imidazole has been absorbed by the calcium chloride. The resulting solid material was carefully washed with methylene chloride and filtrated. After removal of the solvent a white solid was obtained, which was suspended in tetrahydrofuran, filtrated, and washed with tetrahydrofuran. After removal of the solvent from the obtained filtrate, the yellow oily liquid was put in the refrigerator for crystallisation. Yield: 0.516 g (26.7%).  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , 300.13 MHz):  $\delta$  = 7.67 (s, 6H, NCHN); 7.03 (s, 6H, NCHCHN); 6.99 (s, 6H, NCHCHN); 1.50 (s, 54H,  $\text{CH}_3$ -group) ppm.  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ ,

Scheme 1. Preparation of hexa-3-(1-*iso*-propylimidazole)calcium(II)dichloride (**8**).



Scheme 2. Preparation of  $\mu^2$ -dichloro-hexa-3-(1-*tert*-butylimidazole)calcium(II)chloride (**9**).



Scheme 3. Synthesis of tris-aqua-tris-3-(1-*iso*-propylimidazole)magnesium(II)dichloride (**10**).

75.475 MHz):  $\delta = 134.24$  (NCHN); 128.34 (NCHCHN); 116.40 (NCHCHN); 55.08 (NC(CH<sub>3</sub>)<sub>3</sub>); 30.55 (CH<sub>3</sub>-group) ppm. M.p.: decomposition at 350 °C.

### 2.2.3. Tris-aqua-tri-3-(1-*iso*-propylimidazole)-magnesium(II)dichloride [ $\text{Mg}^{(\text{ipr})}\text{Im}_6(\text{H}_2\text{O})_3\text{Cl}_2$ ] (**10**)

To 4.5 mmol 1-*iso*-propylimidazole (**6**) (0.500 g) 2.25 mmol anhydrous magnesium chloride (0.214 g) was added (see Scheme 3). This mixture has to rest until the whole imidazole has been absorbed by the magnesium chloride. The resulting solid material was carefully washed with methylene chloride and filtrated. After removal of the solvent a white solid was obtained, which was suspended in tetrahydrofuran, filtrated, and washed with tetrahydrofuran. After removal of the solvent from the obtained filtrate, the yellow oily liquid was put in the refrigerator for crystallisation. Yield: 0.391 g (36.2%). <sup>1</sup>H NMR (CDCl<sub>3</sub>, 300.13 MHz):  $\delta = 8.14$  (s, 3H, NCHN); 7.06 (s, 3H, NCHCHN); 6.95 (s, 3H, NCHCHN); 4.41 (quint,  $J = 6.7$  Hz, 3H, NCH(CH<sub>3</sub>)<sub>2</sub>); 1.44 (d,  $J = 6.8$  Hz, 18H, CH<sub>3</sub>-group of *iso*-prop) ppm. <sup>13</sup>C NMR (CDCl<sub>3</sub>, 75.475 MHz):  $\delta = 135.39$  (NCHN); 126.62 (NCHCHN); 116.82 (NCHCHN); 49.98 (NCH(CH<sub>3</sub>)<sub>2</sub>); 23.48 (CH<sub>3</sub>-group of *iso*-prop) ppm. M.p. = 129 °C.

### 2.3. X-ray crystal-structure determination for **8–10**

Single crystals suitable for the X-ray diffraction study were grown from the oil without any solvent in the refrigerator. The crystals were stored under perfluorinated ether, transferred on a glass capillary and fixed. Preliminary

Table 1  
Crystallographic data for complexes **8–10**

	<b>8</b>	<b>9</b>	<b>10</b>
Chemical formula	C <sub>36</sub> H <sub>60</sub> N <sub>12</sub> CaCl <sub>2</sub>	C <sub>42</sub> H <sub>72</sub> N <sub>12</sub> Ca <sub>2</sub> Cl <sub>4</sub>	C <sub>18</sub> H <sub>36</sub> N <sub>6</sub> MgCl <sub>2</sub> O <sub>3</sub>
$F_w$	771.94	967.08	479.74
Color/shape	yellow/needle	colorless/plate	yellow/block
Crystal size (mm)	0.78 × 0.17 × 0.17	0.44 × 0.25 × 0.09	0.56 × 0.27 × 0.11
Crystal system	monoclinic	monoclinic	monoclinic
Space group	$P2_1/c$	$P2_1/c$	$P2_1/c$
$a$ (Å)	8.165(1)	12.507(1)	9.257(1)
$b$ (Å)	17.282(2)	10.664(1)	22.364(3)
$c$ (Å)	17.318(1)	20.210(1)	14.426(1)
$\beta$ (°)	107.37(1)	98.90(1)	117.89(1)
$V$ (Å <sup>3</sup> )	2332.3(4)	2663.0(3)	2639.6(5)
$Z$	2	2	4
$T$ (K)	198 ± 2	198 ± 2	198 ± 2
$\rho_{\text{calc}}$ (g/cm <sup>3</sup> )	1.099	1.206	1.207
$\mu$ (mm <sup>−1</sup> )	0.286	0.455	0.298
$F_{000}$	828	1032	1024
$\Theta$ range (°)	3.82–30.01	3.08–26.01	3.09–26.50
Data collected ( $h, k, l$ )	±11, ±24, −24/+23	±15, −13/+12, ±24	±11, ±28, −18/17
Number of reflections collected	51 042	53 587	59 734
Number of independent reflections / $R_{\text{int}}$	6785 (all) /0.0833	5222 (all) /0.0581	5471 (all) /0.0597
Number of observed reflections ( $I > 2\sigma(I)$ )	3909	4116	3933
Number of parameters refined	238	280	301
$R_1^a$ (observed/all)	0.0493/0.1183	0.0302/0.0521	0.0318/0.0585
$wR_2^b$ (observed/all)	0.0866/0.1040	0.0642/0.0717	0.0740/0.0816
Goodness-of-fit <sup>c</sup>	1.013	1.082	1.014
Max/min $\Delta\rho$ (e/Å <sup>3</sup> )	+0.318/−0.277	+0.215/−0.219	+0.180/−0.202

<sup>a</sup>  $R_1 = \sum(|F_o| - |F_c|) / \sum |F_o|$ .

<sup>b</sup>  $wR_2 = \left( \sum w(F_o^2 - F_c^2)^2 / \sum w(F_o^2)^2 \right)^{1/2}$ .

<sup>c</sup>  $\text{GOF} = \left( \sum w(F_o^2 - F_c^2)^2 / (\text{NO} - \text{NV}) \right)^{1/2}$ .

examination and data collection were carried out on an area detecting system (kappa-CCD; Nonius FR590) using graphite-monochromated Mo K $\alpha$  radiation ( $\lambda = 0.71073$  Å). Raw data were corrected for Lorentz, polarisation, decay and absorption effects. The absorption correction was applied using SADABS [18]. After merging the independent reflections were used for all calculations. The structures were solved by a combination of direct methods [19] and difference Fourier syntheses [20]. All non-hydrogen atoms were refined with anisotropic displacement parameters. All hydrogen atoms bound to the carbon atoms were calculated in ideal positions using the SHELXL riding model.

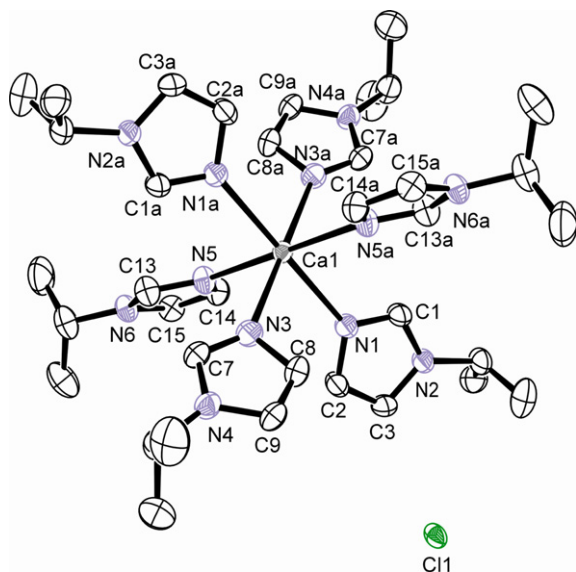


Fig. 3. ORTEP [26] representation of complex **8**; thermal ellipsoids are drawn at the 50% probability level; hydrogen atoms are omitted for clarity.

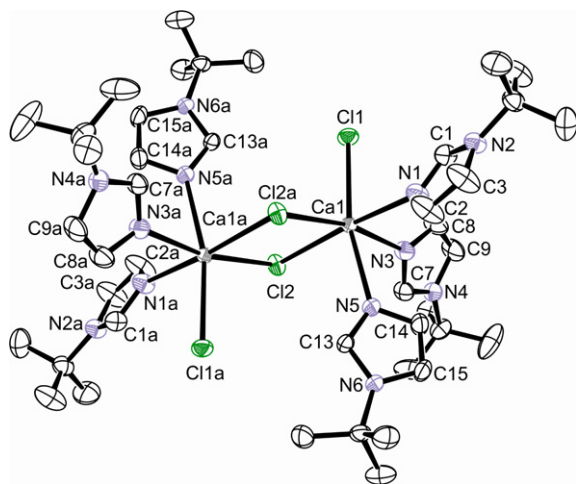


Fig. 4. ORTEP [26] representation of complex **9**; thermal ellipsoids are drawn at the 50% probability level; hydrogen atoms are omitted for clarity.

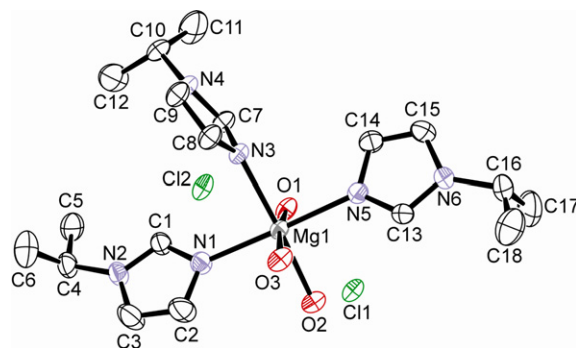


Fig. 5. ORTEP [26] representation of complex **10**; thermal ellipsoids are drawn at the 50% probability level; hydrogen atoms are omitted for clarity.

The hydrogen atoms bonded to oxygen (**10**) were refined with isotropic displacement parameters. Full-matrix least-squares refinements were carried out by minimizing  $\sum w(F_o^2 - F_c^2)^2$  with the SHELXL-97 weighting scheme and stopped at shift/err < 0.001. Details of the structure determinations are given in Table 1. Neutral-atom scattering factors for all atoms and anomalous dispersion corrections for the non-hydrogen atoms were taken from the *International Tables for Crystallography* [21]. All calculations were performed with the programs COLLECT [22], DIRAX [23], EVALCCD [24], SIR92 [19], SADABS [18], the SHELXL-97 package [20,25] and ORTEP-3 for Windows [26] (see Figs. 3–5).

### 3. Results and discussion

We studied the coordination chemistry of *N*-substituted alkylimidazoles (1-methylimidazole, 1-*iso*-propylimidazole and 1-*tert*-butylimidazole) to group II (Mg, Ca, Sr and Ba) dichlorides. While we have neither been able to crystallize the strontium and barium complexes nor any 1-methylimidazole complex, we succeeded in crystallizing three different interesting coordination complexes of magnesium and calcium with *N*-substituted imidazoles.

The preferred coordination number of magnesium is six, leading to octahedral coordination complexes, while calcium prefers coordination numbers between six and nine. Complex **8** is one of the rare examples of a solid-state structure, where six imidazoles completely fill the coordination sphere of the metal.

Complexes **8–10** all show an octahedral coordination around the metal, but very different coordination geometries. As we used the same method for the preparation of all complexes, the resulting solid-state structures allow us to study the preferred coordination geometries.

Since no specially dried chemicals or inert gas atmosphere were used in the synthesis, water could coordinate to the metal (see Scheme 3 or Fig. 5) and is obviously preferable over adding another imidazole ligand in the case of magnesium, which can only be explained by the higher oxophilicity of magnesium compared to calcium.

Table 2  
Selected bond lengths (Å) of complexes **8**–**10**

<b>8<sup>a</sup></b>		<b>9<sup>b</sup></b>		<b>10</b>	
Ca1–N5	2.4435(15)	N1–Ca1	2.4777(15)	Mg1–O3	2.0788(13)
Ca1–N3	2.4555(16)	N3–Ca1	2.4822(14)	Mg1–O1	2.0841(13)
Ca1–N1	2.4642(15)	N5–Ca1	2.5056(14)	Mg1–O2	2.1039(12)
		Ca1–Cl1	2.6987(5)	Mg1–N1	2.1714(14)
		Ca1–Cl2	2.7374(5)	Mg1–N3	2.1763(13)
		Cl2–Ca1a	2.7371(5)	Mg1–N5	2.1809(14)

<sup>a</sup> Symmetry equivalents generated by  $-x + 2, -y, -z$ .

<sup>b</sup> Symmetry equivalents generated by  $-x + 2, -y, -z + 1$ .

The solid-state structure of complex **8** shows six molecules *iso*-propylimidazole directly coordinated to the calcium. The unit cell contains a void of 108 Å<sup>3</sup> not filled by any solvent, which explains the surprisingly low melting-point of 20 °C.

But when *tert*-butylimidazole is used, only three ligands are directly bonded to the calcium cationic center (complex **9**). The unit cells of the reported solid-state structures **8** and **9** contain an inversion center. In the solid-state structure of **8** the Ca atom is located on the inversion center. Comparing the two structures **8** and **9** it can be seen that the larger substituent (*t*Bu) at the imidazole prevents a hexacoordination by imidazoles and leads to the observed structure, where the necessity of charge neutrality and the favored octahedral geometry can only be accommodated by a chloro-bridged structure, where the two calcium ions are bridged by two chlorine atoms, while one chlorine atom is directly bonded to each of the calcium ions. Steric demand of the ligand determines the structure of complex **9** where the calcium ions are separated by 4.09 Å.

When magnesium chloride is used instead of calcium chloride, we found that also in the case of the *iso*-propylimidazole ligand, the magnesium cation is only surrounded by three *iso*-propylimidazole molecules (complex **10**). The other three coordination sites are filled by water. Comparing the distances between the metal cations and the coordinating nitrogen atoms there are notable differences in the bond lengths (Table 2). While the Ca–N distances in complexes **8** and **9** are  $2.45 \pm 0.05$  Å, the Mg–N distances are significantly smaller (2.17 Å).

It is interesting to note that there is not a large difference of the Ca–N bond lengths in complexes **8** and **9**. The distances between the calcium ion and the imidazole ligands are nearly equally independent of the number of bonded imidazole molecules. This indicates that the steric requirements of the substituents are more important than electronic effects in these cases.

In Table 3 selected bond angles are presented. Due to steric and most likely packing reasons the imidazoles are not symmetrically coordinated.

#### Appendix A. Supplementary material

CCDC 639135, 639136 and 639137 contain the supplementary crystallographic data for **8**, **9** and **10**. These data can be obtained free of charge via <http://www.ccdc.cam.ac.uk/conts/retrieving.html>, or from the Cambridge Crystallographic Data Centre, 12 Union Road, Cambridge CB2 1EZ, UK, fax: (+44) 1223-336-033, or e-mail: [deposit@ccdc.cam.ac.uk](mailto:deposit@ccdc.cam.ac.uk). Supplementary data associated with this article can be found, in the online version, at [doi:10.1016/j.ica.2007.04.037](https://doi.org/10.1016/j.ica.2007.04.037).

Table 3  
Selected angles (°) of complexes **8**–**10**

8 <sup>a</sup>		9 <sup>b</sup>		10	
N1–Ca1–N1a	180.0	N1–Ca1–N3	92.97(5)	N1–Mg1–N3	86.65(5)
N3–Ca1–N1	91.32(5)	N1–Ca1–N5	83.28(5)	N1–Mg1–N5	178.30(5)
N3–Ca1–N1a	88.68(5)	N3–Ca1–N5	82.10(5)	N3–Mg1–N5	91.70(5)
N3–Ca1–N3a	180.0	N1–Ca1–Cl1	86.99(4)	O1–Mg1–N1	93.75(5)
N5–Ca1–N1	88.12(5)	N3–Ca1–Cl1	89.49(4)	O2–Mg1–N1	90.17(5)
N5–Ca1–N1a	91.88(5)	N5–Ca1–Cl1	166.76(4)	O3–Mg1–N1	89.12(5)
N5–Ca1–N3	89.08(5)	N1–Ca1–Cl2	173.23(4)	O1–Mg1–N3	90.40(5)
N5–Ca1–N3a	90.92(5)	N3–Ca1–Cl2	89.43(4)	O2–Mg1–N3	176.38(5)
N5–Ca1–N5a	180.0	N5–Ca1–Cl2	90.79(4)	O3–Mg1–N3	97.00(5)
C1–N1–Ca1	124.21(12)	N1–Ca1–Cl2a	93.16(4)	O1–Mg1–N5	86.67(5)
C2–N1–Ca1	131.39(11)	N3–Ca1–Cl2a	168.69(4)	O2–Mg1–N5	91.49(5)
C7–N3–Ca1	139.69(12)	N5–Ca1–Cl2a	89.19(4)	O3–Mg1–N5	90.68(5)
C8–N3–Ca1	115.82(11)	Cl1–Ca1–Cl2	99.368(17)	O1–Mg1–O2	88.08(5)
C13–N5–Ca1	134.26(11)	Cl1–Ca1–Cl2a	100.318(17)	O3–Mg1–O1	172.22(6)
C14–N5–Ca1	121.34(11)	Cl2a–Ca1–Cl2	83.460(15)	O3–Mg1–O2	84.68(5)
		Ca1a–Cl2–Ca1	96.541(15)	C1–N1–Mg1	130.08(11)
		C2–N1–Ca1	125.06(13)	C2–N1–Mg1	125.24(11)
		C1–N1–Ca1	129.31(12)	C7–N3–Mg1	124.99(10)
		C7–N3–Ca1	126.08(12)	C8–N3–Mg1	127.61(10)
		C8–N3–Ca1	128.68(12)	C13–N5–Mg1	128.72(11)
		C13–N5–Ca1	133.42(11)	C14–N5–Mg1	126.57(11)
		C14–N5–Ca1	121.99(11)		

<sup>a</sup> Symmetry equivalents generated by  $-x + 2, -y, -z$ .

<sup>b</sup> Symmetry equivalents generated by  $-x + 2, -y, -z + 1$ .



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