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Acta Cryst. (1999). C**55**, 1260–1262

Sodium (nitrilotriacetato)magnesate pentahydrate

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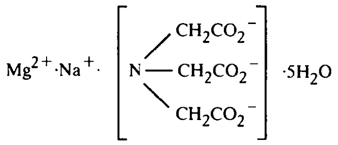
(*Received 8 February 1999; accepted 1 April 1999*)

Abstract

In the title compound, triaqua-μ-nitrilotriacetato-magnesiumsodium dihydrate, $[MgNa(C_6H_6NO_6)(H_2O)_3]\cdot 2H_2O$, a nitrilotriacetate ligand chelates one Mg atom *via* three Mg—O bonds and one Mg—N bond. Each ligand also binds to two Na atoms and another Mg atom such that an infinite layer of stoichiometry $MgNa(C_6H_6NO_6)(H_2O)_3$ is formed. Adjacent layers are bound *via* hydrogen bonds with two interlayer water molecules.

Comment

Nitrilotriacetic acid (NTA) is found as a multidentate ligand in many metal–chelate compounds (Skrzypczak-Jankun & Smith, 1994; Martell, 1975). A magnesium-chelated NTA complex has been proposed recently as a guest species within a layered double hydroxide (Kaneyoshi & Jones, 1999). The crystal structure of the title compound, (I), contains Mg atoms chelated by NTA molecules *via* three Mg—O bonds [2.042 (2)–2.096 (2) Å] and one Mg—N bond [Mg1—N1 2.232 (2) Å]. In addition, each Mg atom is bound to one O atom of a neighbouring NTA molecule [Mg1—O5^{i} 2.029 (2) Å; symmetry code: (i) $2-x, y-\frac{1}{2}, \frac{3}{2}-z$] and one O atom of a water molecule [Mg1—O7 2.066 (2) Å], resulting in a distorted octahedral coordination geometry. The Na atoms are also octahedrally coordinated by four O atoms of different NTA molecules [2.385 (2)–2.453 (2) Å] and two O atoms of water molecules [Na1—O8 2.402 (2) and Na1—O9 2.331 (2) Å].



(I)

The three carboxylate groups of each NTA molecule adopt different bonding patterns. The first (C2, O1 and O2) binds to the chelated Mg atom and one Na atom *via* O2, and is involved in interlayer hydrogen bonding through O1 [O1$^{ii}\cdots$O8 2.762 (3) Å and O1$^{ii}\cdots$H8*A*—O8 167 (3)°; symmetry code: (ii) $2-x, \frac{1}{2}+y, \frac{3}{2}+z$]. The second (C4, O3 and O4) binds to one Mg atom and one Na atom *via* O3 and O4, respectively. In the third carboxylate group (C6, O5 and O6), O5 and O6 each bind to both an Mg atom and an Na atom, acting as bridges between adjacent $MgNa(C_6H_6NO_6)(H_2O)_3$ units.

The interactions between the metal atoms and the NTA ligands, together with hydrogen-bonding interactions, give rise to infinite $MgNa(C_6H_6NO_6)(H_2O)_3$ lay-

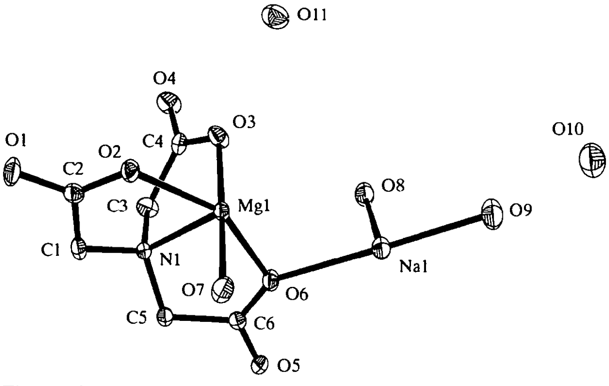


Fig. 1. The asymmetric unit of (I), showing displacement ellipsoids at the 50% probability level. H atoms have been omitted for clarity.

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ers parallel to the (001) plane (Fig. 2). Adjacent layers are bound *via* hydrogen bonds (Table 2) with two water molecules which are not coordinated to any metal atom (O10 and O11).

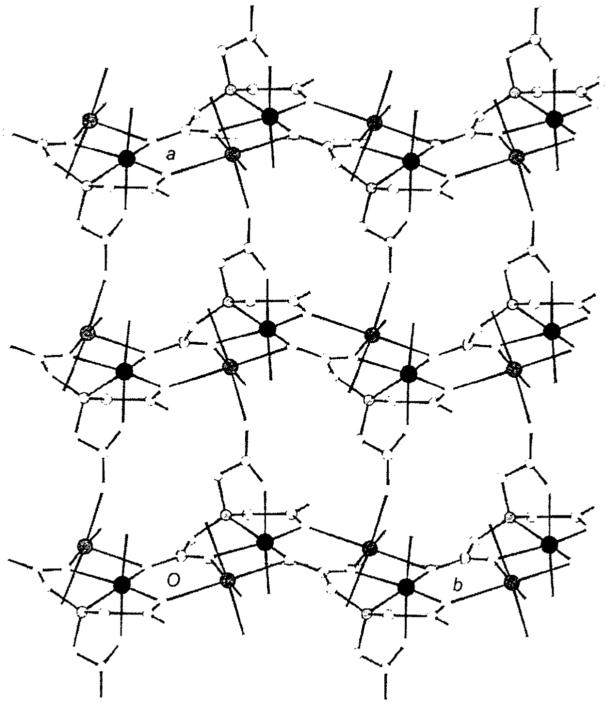

Fig. 2. View of the (001) plane, showing an infinite $MgNa(C_6H_6NO_6)(H_2O)_3$ layer. H atoms and interlayer water molecules have been omitted.

The structure of $NaMg(NTA)(H_2O)_3{\cdot}2H_2O$ may be compared with those of other M^{II} analogues reported previously. In $NaCu(NTA){\cdot}H_2O$, the Cu atoms are also octahedrally coordinated, but in this case, solely by N and O atoms of NTA ligands (without any coordinated water molecules) (Whitlow, 1973). In NaCa(NTA), each Ca atom adopts sevenfold coordination *via* one tetradentate NTA molecule and the O atoms of three neighbouring NTA molecules (Barnett & Uchtman, 1979). The differences between the structures of these compounds and that of the title compound may be attributed to the amount of water of crystallization.

Experimental

Single crystals of the title compound were grown by slow vapour diffusion of acetone into an aqueous solution of $Na_3(NTA)$, $H_3(NTA)$ and $Mg(OH)_2$ in stoichiometric amounts at approximately 278 K.

Crystal data

$[MgNa(C_6H_6NO_6)(H_2O)_3]{\cdot}2H_2O$
$M_r = 325.50$
Orthorhombic
Pbca
Cu *Kα* radiation
$\lambda = 1.54178$ Å
Cell parameters from 25 reflections
$\theta = 32.2–49.6°$
$a = 14.623\ (7)$ Å
$b = 9.729\ (5)$ Å
$c = 19.228\ (10)$ Å
$V = 2735\ (2)$ Å^3
$Z = 8$
$D_x = 1.581$ Mg m^{-3}
D_m not measured
$\mu = 1.994$ mm^{-1}
$T = 180\ (2)$ K
Plate
0.60 × 0.25 × 0.10 mm
Colourless

Data collection

Stoe Stadi-4 four-circle diffractometer
ω–θ scans
Absorption correction: ψ scan (North *et al.*, 1968)
$T_{min} = 0.522$, $T_{max} = 0.798$
3421 measured reflections
1718 independent reflections
1435 reflections with $I > 2\sigma(I)$
$R_{int} = 0.041$
$\theta_{max} = 54.92°$
$h = -15 \rightarrow 15$
$k = -10 \rightarrow 10$
$l = -20 \rightarrow 20$
3 standard reflections every 100 reflections intensity decay: none

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.030$
$wR(F^2) = 0.077$
$S = 1.062$
1718 reflections
212 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0376P)^2 + 0.7644P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.012$
$\Delta\rho_{max} = 0.194$ e Å^{-3}
$\Delta\rho_{min} = -0.183$ e Å^{-3}
Extinction correction: *SHELXL97* (Sheldrick, 1997*a*)
Extinction coefficient: 0.0012 (1)
Scattering factors from *International Tables for Crystallography* (Vol. C)

Table 1. *Selected bond lengths (Å)*

Mg1—O5^{i}	2.0292 (18)	Na1—O9	2.331 (2)
Mg1—O3	2.0420 (17)	Na1—O6	2.385 (2)
Mg1—O7	2.0660 (19)	Na1—O8	2.402 (2)
Mg1—O2	2.0773 (18)	Na1—O2^{ii}	2.415 (2)
Mg1—O6	2.0964 (18)	Na1—O4^{iii}	2.4471 (19)
Mg1—N1	2.232 (2)	Na1—O5^{i}	2.4526 (19)

Symmetry codes: (i) $2 - x, y - \frac{1}{2}, \frac{3}{2} - z$; (ii) $2 - x, \frac{1}{2} + y, \frac{3}{2} - z$; (iii) $\frac{1}{2} + x, y, \frac{3}{2} - z$.

Table 2. *Hydrogen-bonding geometry (Å, °)*

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O7—H7*B*···O4^{i}	0.84 (3)	1.93 (3)	2.758 (3)	165 (3)
O7—H7*A*···O8^{ii}	0.88 (3)	1.91 (3)	2.783 (3)	173 (3)
O8—H8*B*···O11^{iii}	0.83 (3)	2.14 (3)	2.904 (3)	153 (3)
O8—H8*A*···O1^{iv}	0.90 (4)	1.88 (4)	2.762 (3)	167 (3)
O9—H9*B*···O1^{v}	0.86 (3)	1.91 (3)	2.746 (3)	162 (3)
O10—H10*B*···O1^{i}	0.80 (3)	2.07 (3)	2.863 (3)	177 (3)
O10—H10*A*···O11^{vi}	0.93 (4)	1.89 (4)	2.821 (3)	172 (2)
O9—H9*A*···O10	0.84 (3)	1.94 (3)	2.771 (3)	173 (3)
O11—H11*A*···O3	0.92 (3)	1.90 (3)	2.792 (3)	165 (3)

Symmetry codes: (i) $\frac{1}{2} + x, y, \frac{3}{2} - z$; (ii) $2 - x, y - \frac{1}{2}, \frac{3}{2} - z$; (iii) $\frac{3}{2} - x, \frac{1}{2} + y, z$; (iv) $x, \frac{1}{2} - y, \frac{1}{2} + z$; (v) $2 - x, \frac{1}{2} + y, \frac{3}{2} - z$; (vi) $2 - x, -y, 2 - z$.

All H atoms of the CH_2 groups were positioned geometrically (C—H 0.99 Å) and refined using a riding model with the U_{iso} values for each H atom taken as $1.2U_{eq}$ of the carrier atom. Water H atoms were located in difference Fourier maps and refined with a fixed isotropic displacement parameter of 0.05 Å^2. The attachment of a low-temperature device to the diffractometer prohibited collection of data to a higher angle.

Data collection: *DIF*4 (Stoe & Cie, 1992*a*). Cell refinement: *DIF*4. Data reduction: *REDU*4 (Stoe & Cie, 1992*b*). Program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997*b*). Program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997*a*). Molecular graphics: *XP* in *SHELXTL/PC* (Sheldrick, 1994) and *CAMERON* (Pearce *et al.*, 1993). Software used to prepare material for publication: *SHELXL*97.

The authors would like to thank Shin-Etsu Chemical Co. Ltd for granting study leave to MK, the EPSRC and Zeneca Specialties for funding (AB), and Dr Neil Feeder for assistance.

Supplementary data for this paper are available from the IUCr electronic archives (Reference: CF1309). Services for accessing these data are described at the back of the journal.

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Acta Cryst. (1999). C**55**, 1262–1263

Dichlorobis(1-methyl-1*H*-benzimidazole-*N*^{3})-cobalt(II)

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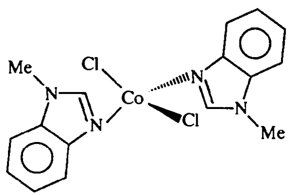
(*Received 13 July 1998; accepted 6 April 1999*)

Abstract

The metal atom in the title complex, $[CoCl_2(C_8H_8N_2)_2]$, has a slightly distorted tetrahedral coordination involving two Cl^- ions and two N atoms from the aromatic groups. The dihedral angle between the planes of the two 1-methylbenzimidazole ligands is 117.7 (7)°.

Comment

Benzimidazoles are an important class of compounds in biological systems and in coordination chemistry. This fused two-ring system occurs in many biologically active compounds, such as antinematodal and antitumor drugs. It also appears to bind to cobalt in vitamin B12. Metal complexes with benzimidazoles have been investigated in the search for new modes of biological activity (Allan *et al.*, 1981; Golič & Mirčeva, 1988). In order to investigate the role of the 1-alkyl substituent of benzimidazole in the coordinating behavior of this ligand, we have synthesized Co^{II} complexes of the type $CoX_2(L_2)$, where $X = Cl^-$ or NO_3^- and L = 1-methylbenzimidazole, 1,2-dimethylbenzimidazole, 1-isopropyl-2-methylbenzimidazole or 2-methyl-1-propenylbenzimidazole. In addition, the effect of the 1-alkyl group upon the donor and acceptor ability of benzimidazole is of special interest with regard to the electrochemical properties of the Co^{II} complexes. The title compound, (I), has been synthesized as a simple model for complexes with increased steric hindrance in the vicinity of the coordinating N atom of benzimidazole.



(I)

The X-ray crystallographic analysis of (I) revealed that the Co^{II} ion has a slightly distorted tetrahedral coordination, involving two Cl^- ions and two N atoms

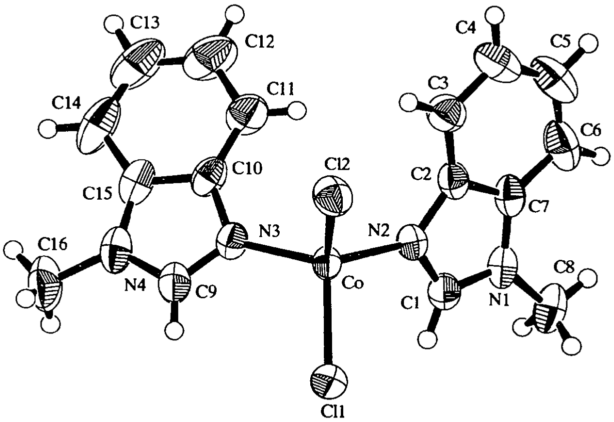


Fig. 1. An *ORTEP* (Johnson, 1965) representation of the title compound, showing the atom-numbering scheme and 50% probability displacement ellipsoids. H atoms are drawn as spheres of arbitrary radii.