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Polyol–Metal Complexes. 26.† A Three-Dimensional Triply-Connected Alkoxo–Metal Net in a Carbohydrate–Bismuth(III) Complex

PETER KLÜFERS AND PETER MAYER

Institut für Anorganische Chemie der Universität, Engesserstraße, Gebäude 30.45, D-76128 Karlsruhe, Germany. E-mail: kluefers@achibm2.chemie.uni-karlsruhe.de

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Abstract

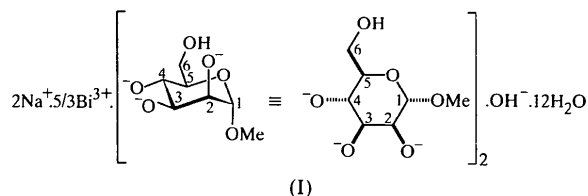
A sodium (methyl- α -D-mannopyranosidato)bismuthate(III) hydroxide hydrate of formula $\text{Na}_2[\text{Bi}_{5/3}(\text{Me-}\alpha\text{-D-Manp}2,3,4\text{H}_{-3})_2](\text{OH})\cdot 12\text{H}_2\text{O}$, (I) (where Me- α -D-Manp2,3,4H₋₃ is C₇H₁₁O₆), forms cubic crystals from alkaline aqueous solution. The carbohydrate-supported alkoxo–bismuth network of (I) resembles the silicon network in SrSi₂ [Pringle (1972). *Acta Cryst.* **B28**, 2326–2328], which is a 3,10-net according to Wells [Structural Inorganic Chemistry (1984), 5th ed., pp. 110–111, Oxford: Clarendon Press]. The mannoside residues act as trianionic triolate ligands with the O2, O3 and O4 atoms deprotonated.

Comment

Anionic carbohydrate ligands provide multidentate alkoxide patterns and may thus be used as convenient tools for the construction of alkoxo–metal clusters or networks. In this work, we report on the synthesis

† Part 25: Burger & Klüfers (1997).

and structure of an alkoxo–bismuth(III) network, (I), which is supported by trianions derived from methyl- α -D-mannopyranoside by deprotonation of the hydroxyls at the C2, C3 and C4 positions. The trianionic ligands are depicted in the scheme below.



Crystals of (I) are grown from aqueous solutions of bismuth nitrate, the mannopyranoside and sodium hydroxide in a molar ratio of about 1:3:6. The structure determination revealed the cubic crystal class 23. Accordingly, neither the crystals nor small fragments were birefracting under crossed polarizers.

The carbohydrate-supported alkoxo–metal framework of (I) (Fig. 1) may be classified topologically by taking into account only the Bi atoms at the branching points of the network. As a result, the three-dimensional triply-connected net shown in Fig. 2, which is Wells's (1984) cubic 10,3-net, is obtained. The net resembles the one found in the silicon partial structure of SrSi₂ (Pringle, 1972). Accordingly, the space group of (I) is a maximal subgroup of the SrSi₂ space group *P*4₃32, which itself is a maximal subgroup of the space group of the idealized net *I*4₁32 (Wells, 1984).

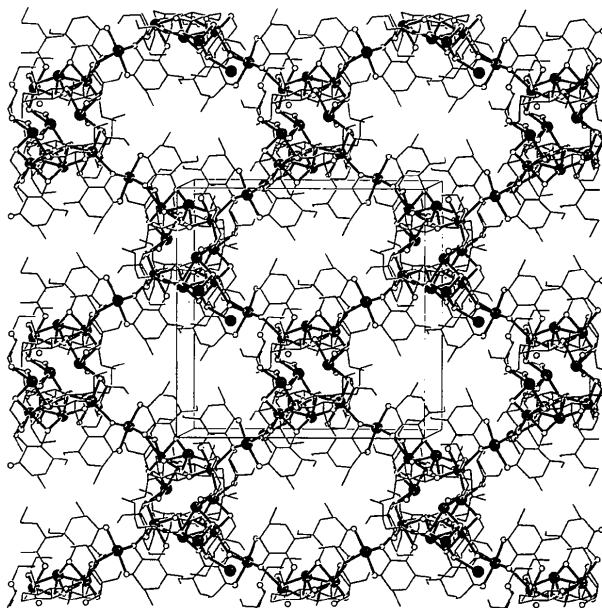


Fig. 1. The carbohydrate-supported alkoxo–bismuth network in (I). Filled circles are Bi atoms and bold bonds are Bi–O bonds. Of the mannoside ligands, which are depicted in a wire-model style, only the three deprotonated and bismuth-bonded hydroxy functions are drawn as open circles. The projection direction is the same as in Fig. 2.

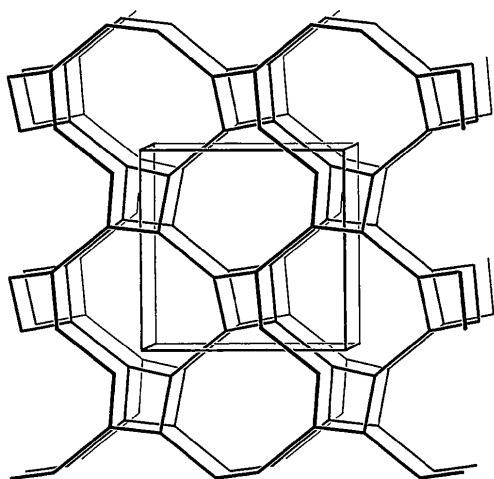


Fig. 2. The 10,3-net as constructed from Fig. 1 by replacing the chemical entity shown in Fig. 3 by a straight line connecting Bi2 and Bi3.

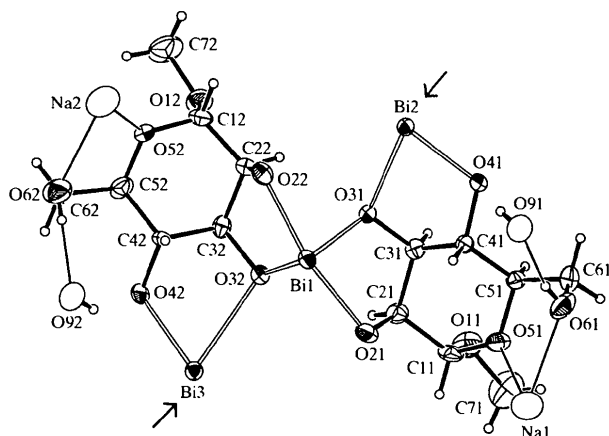


Fig. 3. The molecular structure of an Na₂Bi_{5/3}(Me- α -D-Manp2,3,4-H-₃)₂(OH)_{2/3} entity (60% probability displacement ellipsoids). Empty ellipsoids are sodium and O6—H hydrogen-bonded hydroxide ions. The hydroxide ions, Bi2 and Bi3 exhibit site symmetry 3 (the threefold axes run through Bi2/O91 and Bi3/O92). The proton of the hydroxide ion points towards the bismuth lone pair. Bi1 coordination is pseudo-trigonal-bipyramidal. The arrows denote the ends of a linear segment of the 10,3-net (*cf.* Fig. 2).

The topological frame of the 10,3-net is filled by the chemical entities shown in Fig. 3. The Bi atoms are bound solely to the mannoside, thus forming a homoleptic polyolato-bismuthate. Two branching Bi atoms of site symmetry 3 (Bi2 and Bi3) are connected by a bis(triolato)bismuthate(III) entity centred around a Bi1 atom. Bi1 is coordinated pseudo-trigonal bipyramidally, the lone pair occupying one of the equatorial positions. Both Bi2 and Bi3 adopt a 3+3 coordination, the lone pairs also being of marked stereochemical activity. The two symmetrically independent mannoside residues act as trianionic triolato ligands by forming two puckered five-ring chelates each [puckering parameters of the five-membered chelate rings according to Cremer & Pople (1975); Bi1—O21—C21—C31—O31: $Q_2 = 0.407(5) \text{ \AA}$ and $\varphi_2 = 76.7(6)^\circ$; Bi1—O22¹—C22¹—C32¹—O32¹: $Q_2 = 0.389(5) \text{ \AA}$ and $\varphi_2 = 81.3(6)^\circ$; Bi3—O32—C32—C42—O42: $Q_2 = 0.480(4) \text{ \AA}$ and $\varphi_2 = 115.2(5)^\circ$; symmetry code: (i) $\frac{1}{2} - x, 1 - y, \frac{1}{2} + z$]. The puckering parameters of the pyranose rings [O51—C11—C21—C31—C41—C51: $Q = 0.552(6) \text{ \AA}$, $\theta = 10.6(6)$ and $\varphi = 257(3)^\circ$; O52—C12—C22—C32—C42—C52: $Q = 0.569(5) \text{ \AA}$, $\theta = 12.8(5)$ and $\varphi = 267(2)^\circ$] resemble a slightly distorted ⁴C₁ conformation, as is usually found with the pyranose forms of the common aldohexoses. The typically smaller O—C—C—O torsion angle of an axial-equatorial-configured diol group of a pyranose as opposed to an equatorial-equatorial one, is retained in (I) [*cf.* the structure of a cuprate(II) with deprotonated methyl α -D-mannopyranosidate ligands; Habermann *et al.*, 1992].

The O91 and O92 atoms are hydroxide O atoms. They are triple acceptors in short hydrogen bonds, the donors being the primary O6—H alcohol functions of the two independent mannoside residues. The H atoms of both OH⁻ ions point towards the lone pair of one of the Bi

atoms located on the threefold axis each (Fig. 3). The Na atoms, together with six O-ligand atoms each, are assembled into pairs of edge-shared octahedra, which fill gaps between pairs of mannoside entities. The larger channels of the structure are filled with disordered water molecules.

Experimental

Bismuth(III) nitrate pentahydrate (0.93 g, 1.9 mmol), methyl- α -D-mannopyranoside (1.12 g, 5.8 mmol) and NaOH (0.47 g, 11.7 mmol) were stirred in 8 ml water. After addition of ethanol and ether, crystals of (I) formed within two weeks.

Crystal data

Na₂[Bi_{5/3}(C₇H₁₁O₆)₂](OH)·
12H₂O
 $M_r = 1009.792$
Cubic
 $P2_13$
 $a = 21.2245(11) \text{ \AA}$
 $V = 9561.2(9) \text{ \AA}^3$
 $Z = 12$
 $D_x = 2.1045(2) \text{ Mg m}^{-3}$
 D_m not measured

Mo K α radiation
 $\lambda = 0.71073 \text{ \AA}$
Cell parameters from 4816
reflections
 $\theta = 5-20^\circ$
 $\mu = 9.316 \text{ mm}^{-1}$
 $T = 250(2) \text{ K}$
Brick shaped
 $0.30 \times 0.22 \times 0.13 \text{ mm}$
Colourless

Data collection

Stoe IPDS diffractometer
 φ scans
Absorption correction:
numerical (IPDS software)
 $T_{\min} = 0.209, T_{\max} = 0.536$
44 099 measured reflections
6096 independent reflections
(includes Friedel pairs)

5657 reflections with
 $I > 2\sigma(I)$
 $R_{\text{int}} = 0.049$
 $\theta_{\text{max}} = 25.81^\circ$
 $h = -25 \rightarrow 25$
 $k = -23 \rightarrow 25$
 $l = -21 \rightarrow 25$

Refinement

Refinement on F^2	$\Delta\rho_{\max} = 0.618 \text{ e } \text{Å}^{-3}$
$R[F^2 > 2\sigma(F^2)] = 0.022$	$\Delta\rho_{\min} = -0.487 \text{ e } \text{Å}^{-3}$
$wR(F^2) = 0.043$	Extinction correction: none
$S = 0.983$	Scattering factors from
6096 reflections	<i>International Tables for</i>
407 parameters	<i>Crystallography (Vol. C)</i>
H atoms constrained	Absolute structure: Flack
$w = 1/[\sigma^2(F_o^2) + (0.0248P)^2]$	(1983)
where $P = (F_o^2 + 2F_c^2)/3$	Flack parameter =
$(\Delta/\sigma)_{\max} = -0.003$	-0.033 (5), 2786 Friedel
	pairs

Table 1. Fractional atomic coordinates and equivalent isotropic displacement parameters (Å^2)
$$U_{\text{eq}} = (1/3)\sum_i\sum_j U_{ij} a^i a^j \mathbf{a}_i \cdot \mathbf{a}_j$$

Occupancy	x	y	z	U_{eq}
Bi1	0.277708 (9)	0.422579 (9)	0.527744 (9)	0.01520 (5)
Bi2	0.131815 (9)	1/2-x	1/2+x	0.01454 (7)
Bi3	0.093569 (9)	1/2+x	-x	0.01290 (7)
Na1	0.08647 (15)	0.44965 (13)	0.32479 (13)	0.0457 (7)
Na2	0.00812 (13)	0.54555 (13)	0.21420 (12)	0.0425 (7)
O11	0.1768 (2)	0.2582 (2)	0.3615 (2)	0.0293 (9)
O12	0.0763 (2)	0.7420 (2)	0.1623 (2)	0.0315 (10)
O21	0.2427 (2)	0.3984 (2)	0.4325 (2)	0.0209 (8)
O22	0.1734 (2)	0.6088 (2)	0.1184 (2)	0.0219 (9)
O31	0.2182 (2)	0.3401 (2)	0.5451 (2)	0.0167 (8)
O32	0.1540 (2)	0.6462 (2)	-0.0024 (2)	0.0149 (8)
O41	0.0856 (2)	0.3404 (2)	0.5452 (2)	0.0190 (8)
O42	0.0267 (2)	0.6228 (2)	-0.0196 (2)	0.0164 (7)
O51	0.1190 (2)	0.3503 (2)	0.3753 (2)	0.0213 (8)
O52	0.0351 (2)	0.6404 (2)	0.1532 (2)	0.0224 (8)
O61	0.0205 (2)	0.4275 (2)	0.4138 (2)	0.0382 (11)
O62	-0.0482 (2)	0.5468 (2)	0.1158 (2)	0.0322 (11)
C11	0.1803 (3)	0.3238 (3)	0.3714 (3)	0.0213 (12)
C12	0.0910 (3)	0.6770 (3)	0.1611 (2)	0.0219 (12)
C21	0.2214 (3)	0.3349 (3)	0.4298 (2)	0.0191 (11)
C22	0.1408 (3)	0.6661 (3)	0.1092 (2)	0.0203 (12)
C31	0.1838 (2)	0.3208 (3)	0.4900 (2)	0.0173 (11)
C32	0.1099 (2)	0.6656 (2)	0.0443 (2)	0.0166 (11)
C41	0.1196 (2)	0.3542 (2)	0.4891 (2)	0.0147 (11)
C42	0.0523 (2)	0.6228 (2)	0.0427 (2)	0.0144 (10)
C51	0.0835 (3)	0.3332 (3)	0.4310 (3)	0.0232 (12)
C52	0.0054 (2)	0.6465 (2)	0.0916 (2)	0.0169 (11)
C61	0.0184 (3)	0.3624 (3)	0.4242 (3)	0.0324 (15)
C62	-0.0557 (3)	0.6097 (3)	0.0954 (3)	0.0293 (14)
C71	0.1484 (4)	0.2423 (4)	0.3029 (3)	0.044 (2)
C72	0.0432 (4)	0.7598 (4)	0.2176 (4)	0.054 (2)
O91	0.0215 (2)	0.4785 (2)	0.5215 (2)	0.031 (2)
O92	-0.0200 (2)	1/2+x	-x	0.030 (2)
O93	0.1549 (4)	0.1549 (4)	0.1549 (4)	0.138 (6)
O94	0.1894 (3)	0.4804 (3)	0.3502 (4)	0.088 (2)
O95	0.1059 (3)	0.5160 (3)	0.1788 (3)	0.079 (2)
O96	0.0591 (4)	0.5607 (3)	0.3142 (3)	0.090 (2)
O97	-0.0020 (3)	0.4357 (3)	0.2513 (2)	0.0494 (13)
O98	0.2526 (9)	1/2-x	1/2+x	0.251 (13)
O99	0.3340 (8)	0.1660 (8)	0.8340 (8)	0.215 (11)
O10	0.48 (3)	0.5946 (9)	0.2375 (6)	0.062 (6)
O11	0.60 (3)	0.6251 (5)	0.2266 (4)	0.2368 (4)
O12	0.67 (3)	0.3396 (5)	0.1671 (5)	0.036 (3)
O13	0.45 (3)	0.3388 (8)	0.1536 (8)	0.074 (5)
O14	0.68 (2)	0.0074 (4)	0.1122 (4)	0.088 (9)
O15	0.38 (2)	-0.0542 (9)	0.1475 (9)	0.0777 (4)
O16	0.50 (4)	-0.0774 (10)	0.6032 (13)	0.068 (4)
O17	0.47 (3)	-0.0936 (8)	0.5645 (10)	0.0961 (9)
O18	0.25 (4)	0.761 (3)	0.3694 (15)	0.2690 (8)
O19	0.51 (4)	0.7261 (14)	0.3717 (8)	0.076 (8)
O20	0.40 (3)	0.1630 (12)	0.4250 (13)	0.0978 (16)
O21	0.62 (3)	0.1374 (5)	0.3988 (5)	0.085 (16)
O22	0.44 (5)	0.6455 (16)	0.3286 (8)	0.0843 (9)
O23	0.44 (5)	0.6176 (15)	0.3376 (8)	0.099 (9)
O24	0.18 (2)	-0.1275 (11)	0.6078 (11)	0.2401 (9)
O25	0.31 (2)	-0.1580 (13)	0.5593 (14)	0.2289 (4)
O26				0.047 (4)
				0.064 (8)
				0.067 (8)
				0.060 (7)
				0.027 (9)
				0.103 (13)

Table 2. Selected geometric parameters (Å , $^\circ$)

Bi1—O32 ⁱ	2.154 (3)	O22—C22	1.412 (7)
Bi1—O31	2.191 (4)	O31—C31	1.438 (6)
Bi1—O21	2.213 (4)	O32—C32	1.424 (6)
Bi1—O22 ⁱ	2.286 (4)	O41—C41	1.421 (6)
Bi1—O42 ⁱⁱ	2.701 (3)	O42—C42	1.431 (6)
Bi2—O41	2.165 (3)	O51—C11	1.419 (7)
Bi2—O41 ⁱⁱⁱ	2.165 (3)	O51—C51	1.448 (7)
Bi2—O41 ^{iv}	2.165 (3)	O52—C12	1.428 (7)
Bi2—O31 ^v	2.665 (3)	O52—C52	1.457 (6)
Bi2—O31	2.665 (3)	O61—C61	1.400 (8)
Bi2—O31 ^{vi}	2.665 (3)	O62—C62	1.412 (8)
Bi3—O42 ^v	2.206 (3)	C11—C21	1.533 (7)
Bi3—O42	2.206 (3)	C12—C22	1.544 (7)
Bi3—O42 ^{vi}	2.206 (3)	C21—C31	1.535 (7)
Bi3—O32 ^{vi}	2.577 (4)	C22—C32	1.526 (7)
Bi3—O32	2.577 (4)	C31—C41	1.536 (7)
Bi3—O32 ^v	2.577 (4)	C32—C42	1.524 (7)
O11—C11	1.409 (7)	C41—C51	1.519 (7)
O11—C71	1.423 (7)	C42—C52	1.524 (7)
O12—C12	1.413 (7)	C51—C61	1.521 (8)
O12—C72	1.421 (8)	C52—C62	1.515 (8)
O21—C21	1.424 (6)		

O21—C21—C31—O31	-49.4 (6)	O31—C31—C41—O41	-57.4 (5)
O22—C22—C32—O32	-48.3 (6)	O32—C32—C42—O42	-55.4 (5)

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

Table 3. Hydrogen-bonding geometry (Å , $^\circ$)

D—H...A	D—H	H...A	D...A	D—H...A
O61—H861...O91	0.75 (5)	1.76 (5)	2.530 (6)	178 (5)
O62—H862...O92	0.74 (4)	1.83 (5)	2.550 (6)	166 (7)

Water and hydroxide O atoms are encoded as O9*n*. Atoms O91 and O92 (both in special positions on 3) were identified as hydroxide O atoms since they are threefold acceptors in short hydrogen bonds; of the O-bonded H atoms, only those of the hydrogen bonds of Table 2, and those bound to the hydroxide O atoms O91 and O92 (four H-atom positions in total) were located in difference Fourier maps and refined with one common O—H distance. One common isotropic displacement parameter was applied to all H atoms. The assignment of a further site of deprotonation is ambiguous. Probably a further one of the O9-type O atoms on 3 is a hydroxide ion. The water content was derived from the sum of O9-type O atoms minus one (for the hydroxide), taking disordered positions into account with the respective population parameter from the refinement. This resulted in 12.1 (5) water molecules per formula unit. The value was rounded off to 12 for subsequent calculations.

Data collection: Stoe IPDS software. Cell refinement: Stoe IPDS software. Data reduction: Stoe IPDS software. Program(s) used to solve structure: *SHELXS86* (Sheldrick, 1985). Program(s) used to refine structure: *SHELXL92* (Sheldrick, 1992). Molecular graphics: *ORTEPIII* (Burnett & Johnson, 1996) and *SCHAKAL* (Keller, 1995). Software used to prepare material for publication: *PLATON* (Spek, 1990).

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: JZ1224). Services for accessing these data are described at the back of the journal.

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N,N-Dimethylformamide Complex of Aluminium(III) Perchlorate

HONOH SUZUKI AND SHIN-ICHI ISHIGURO

Department of Chemistry, Kyushu University, 6-10-1 Hakozaeki Higashi-ku, Fukuoka 812-81, Japan. E-mail: suzükscc@mbox.nc.kyushu-u.ac.jp

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Abstract

Hexakis(*N,N*-dimethylformamide-*O*)aluminium(III) triperchlorate, [Al{(CH₃)₂NCHO}₆](ClO₄)₃, has an octahedral coordination structure with the center of symmetry at the Al³⁺ ion. One of the anions is disordered and the other centers on the twofold axis. Each *N,N*-dimethylformamide (DMF) ligand coordinates *via* its O atom in the *sp*² lone-pair direction. Although the vicinity of the Al atom is crowded with six planar ligands, no serious steric hindrance is found in the coordination structure. The intramolecular bond lengths of the coordinated DMF show clear variations from those of non-coordinated DMF and are in accord with the sequence of metal–DMF interactions: Sb^V > Si^{IV} > Al^{III} > Ca^{II}.

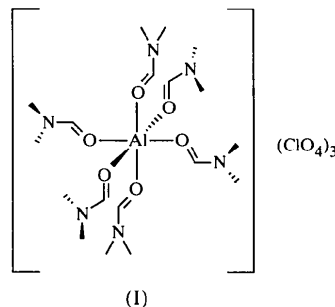
Comment

Interaction of Al ions with peptides is of crucial importance in understanding the cause and process of Alzheimer's disease (AD), and the possible role played by aluminium (Fasman, 1996; Savory *et al.*, 1996). Al, Fe and Zn ions bind to β -amyloid peptide, which is the main component of AD plaques, and enhance

aggregation (Mantyh *et al.*, 1993). Although the precise site of interaction is not known, carboxyl and hydroxyl residues in the peptide are primary candidates for metal coordination (Fasman, 1996). It is also possible that interaction with the backbone amide carbonyl O atoms promotes the β -sheet conformation of an amyloid fragment (Laczkó *et al.*, 1996).

Binding of metal ions to amides is of special relevance to conformation transitions of polypeptides and proteins. *N*-Methylacetamide has been used for model investigations (Chakrabarti *et al.*, 1981), but it was subject to the limitation that the secondary amide could be affected by anions strongly hydrogen bonded to the N—H site. Therefore, a tertiary amide, *N,N*-dimethylformamide (DMF), was proposed as the simplest model ligand to investigate the metal–peptide backbone interactions, and the systematic variation in structures of coordinated DMF molecules to alkali and alkaline earth metal ions was reported (Rao *et al.*, 1984).

Detailed structural knowledge of an aluminium–DMF complex would help to clarify the strength of the interaction of the Al³⁺ ion with the peptide backbone and possible steric hindrance in the metal environment. In view of this, we have isolated a single crystal of the title compound, (I), and performed an X-ray diffraction analysis.



The Al³⁺ ion is located at an inversion center and is surrounded by the DMF molecules in a regular octahedral arrangement (Fig. 1). One of the anions is at a general position and its O atoms (O131–O142) are disordered; the other anion (Cl2–O22) centers on a twofold axis, with four O atoms (two crystallographically independent ones and their equivalents) forming a tetrahedron.

The Al—O bond distances [1.875(2)–1.882(2) Å] fall within the range expected for octahedral aluminium complexes with neutral O-atom donors (Taylor, 1987). The DMF molecules are essentially planar. The Al—O—C angles [126.5(2)–130.2(2)°] and the Al—O—C—N torsion angles [magnitude 160.9(2)–177.6(2)°] show that the metal is approximately coplanar with each amide plane and in the lone-pair direction of each carbonyl group (*trans* to the N atom). A slight deviation of these angles from the idealized values (120 and 180°)