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## Key indicators

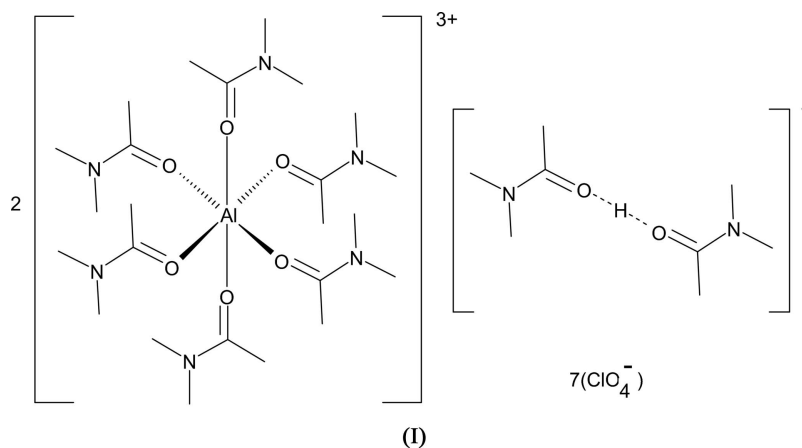
Single-crystal X-ray study  
 $T = 297$  K  
Mean  $\sigma(\text{C}-\text{C}) = 0.009$  Å  
Disorder in solvent or counterion  
 $R$  factor = 0.078  
 $wR$  factor = 0.244  
Data-to-parameter ratio = 14.0For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.*N,N*-Dimethylacetamide complex of aluminium(III) perchlorate

In the title compound, bis[hexakis(*N,N*-dimethylacetamide)aluminium(III)] bis(*N,N*-dimethylacetamide)ium heptakis(perchlorate),  $[\text{H}(\text{C}_4\text{H}_9\text{NO})_2][\text{Al}(\text{C}_4\text{H}_9\text{NO})_6]_2(\text{ClO}_4)_7$ , the  $\text{Al}^{\text{III}}$  ion has an octahedral coordination environment. The singly charged cation lies on a centre of inversion. One of the anions lies on an inversion centre and is disordered; a second anion is also disordered. Three  $\text{Al}-\text{O}-\text{C}-\text{N}$  torsion angles deviate significantly from  $180^\circ$ , indicating that the  $\text{Al}-\text{O}$  bonds are displaced from the direction of the lone pair of the  $sp^2$  O atom. This is ascribed to the steric hindrance of the ligand in an octahedral environment.

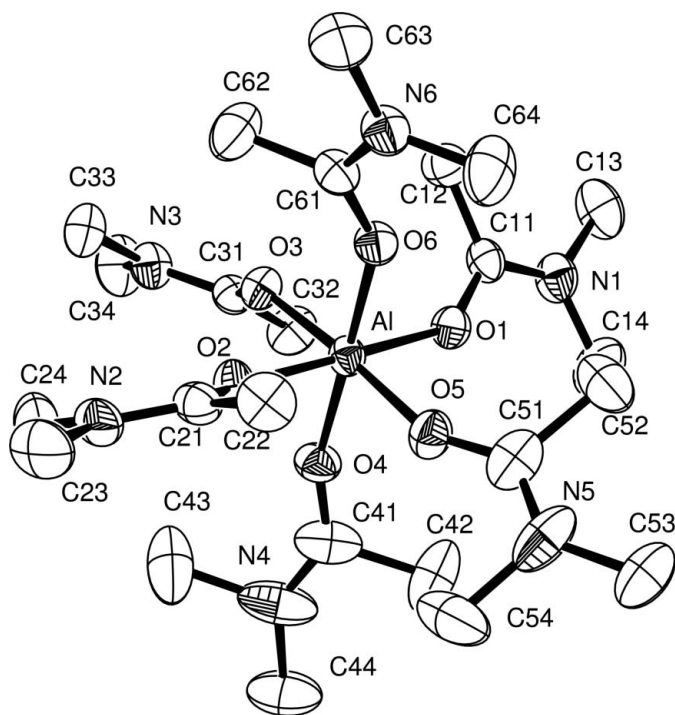
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## Comment

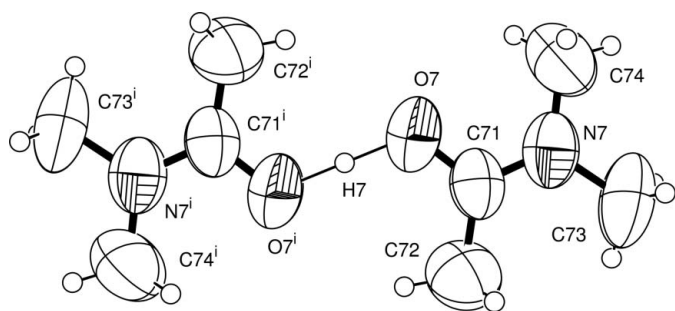
Complexation equilibria in *N,N*-dimethylacetamide (DMA) solution have revealed unusual coordination structures and the importance of sterically hindered solvation (Suzuki & Ishiguro, 1992). DMA is also considered as a simple model ligand in studying metal-peptide interactions. In particular, interaction of the aluminium ion with peptides is of crucial importance in understanding the possible role of aluminium in Alzheimer's disease (Fasman, 1996; Savory *et al.*, 1996). We report here the structure of  $[\text{Al}^{\text{III}}(\text{DMA})_6]_2[\text{H}(\text{DMA})_2]-(\text{ClO}_4)_7$ .



The  $\text{Al}^{3+}$  ion is surrounded by DMA molecules in an octahedral arrangement (Fig. 1). The DMA molecules are essentially planar. Three  $\text{Al}-\text{O}-\text{C}-\text{N}$  torsion angles  $[138.8(8)-149.3(4)^\circ]$  are found to deviate significantly from  $180^\circ$  (Table 1). They serve as a measure of coplanarity between the metal and the molecular plane of DMA, and indicate that  $\text{Al}^{3+}$  is displaced from three DMA molecular planes. Such a deviation is totally absent in the analogous aluminium complex of *N,N*-dimethylformamide (Suzuki &



**Figure 1**  
Molecular structure of the  $[\text{Al}(\text{DMA})_6]^{3+}$  complex cation, showing the labelling of the non-H atoms. Displacement ellipsoids are drawn at the 30% probability level. H atoms have been omitted for clarity.



**Figure 2**  
The structure of the  $[\text{H}(\text{DMA})_2]^+$  cation. Displacement ellipsoids are drawn at the 50% probability level. [Symmetry code: (i)  $-x, -y, -z$ .]

Ishiguro, 1998). It is therefore ascribed to the steric hindrance of the acetyl methyl group of DMA in the octahedral coordination structure.

The centrosymmetric cation (Fig. 2) has the bridging hydrogen at the centre of inversion. The planar structure is essentially the same as those reported for  $[\text{H}(\text{DMA})_2]^+$  cations; the  $\text{O} \cdots \text{O}$  distance [2.386 (8) Å] is within the range (2.31–2.63 Å) found for short hydrogen bonds (Hussain & Schlemper, 1980; Behmel *et al.*, 1981). One of the anions is disordered over an inversion centre and a second perchlorate is also disordered.

## Experimental

$\text{Al}(\text{ClO}_4)_3$  DMA solvate was prepared by dissolving the  $\text{Al}(\text{ClO}_4)_3$  in DMA and removing the excess solvent in a rotary evaporator. A suitable single crystal was obtained by recrystallization from a

mixture of DMA and ethyl orthoformate, the latter acting both as the co-solvent and as a dehydrating reagent. The hygroscopic crystal was sealed in a capillary tube in a glove-box over  $\text{P}_2\text{O}_5$ .

## Crystal data

$(\text{C}_8\text{H}_{19}\text{N}_2\text{O}_2)[\text{Al}(\text{C}_4\text{H}_9\text{NO})_6](\text{ClO}_4)_7$   
 $M_r = 1970.82$   
 Monoclinic,  $P2_1/n$   
 $a = 22.413$  (2) Å  
 $b = 10.862$  (2) Å  
 $c = 22.416$  (3) Å  
 $\beta = 118.80$  (1)°  
 $V = 4782.2$  (12) Å<sup>3</sup>  
 $Z = 2$

$D_x = 1.369$  Mg m<sup>-3</sup>  
 Cu  $K\alpha$  radiation  
 Cell parameters from 25 reflections  
 $\theta = 67.2$ – $69.9^\circ$   
 $\mu = 2.86$  mm<sup>-1</sup>  
 $T = 297$  (2) K  
 Prism, colourless  
 $0.40 \times 0.40 \times 0.40$  mm

## Data collection

Enraf-Nonius CAD-4 diffractometer  
 $\omega$ - $2\theta$  scans  
 Absorption correction:  $\psi$  scan (North *et al.*, 1968)  
 $T_{\min} = 0.318$ ,  $T_{\max} = 0.318$   
 8959 measured reflections  
 8716 independent reflections  
 5548 reflections with  $I > 2\sigma(I)$

$R_{\text{int}} = 0.034$   
 $\theta_{\text{max}} = 69.9^\circ$   
 $h = -27 \rightarrow 23$   
 $k = 0 \rightarrow 13$   
 $l = 0 \rightarrow 23$   
 3 standard reflections  
 frequency: 60 min  
 intensity decay: 2.7%

## Refinement

Refinement on  $F^2$   
 $R[F^2 > 2\sigma(F^2)] = 0.078$   
 $wR(F^2) = 0.244$   
 $S = 1.05$   
 8716 reflections  
 623 parameters  
 H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.1096P)^2 + 5.6858P]$   
 where  $P = (F_o^2 + 2F_c^2)/3$   
 $(\Delta\rho)_{\text{max}} = 0.001$   
 $\Delta\rho_{\text{max}} = 0.57$  e Å<sup>-3</sup>  
 $\Delta\rho_{\text{min}} = -0.33$  e Å<sup>-3</sup>

**Table 1**

Selected geometric parameters (Å, °).

Al—O1	1.878 (3)	Al—O4	1.870 (3)
Al—O2	1.883 (3)	Al—O5	1.864 (3)
Al—O3	1.905 (3)	Al—O6	1.887 (3)
O1—Al—O2	178.31 (15)	O3—Al—O6	93.57 (13)
O1—Al—O3	92.67 (13)	O4—Al—O5	89.63 (16)
O1—Al—O4	91.33 (14)	O4—Al—O6	178.61 (15)
O1—Al—O5	89.21 (14)	O5—Al—O6	90.12 (15)
O1—Al—O6	90.03 (14)	Al—O1—C11	141.3 (3)
O2—Al—O3	85.74 (13)	Al—O2—C21	145.7 (3)
O2—Al—O4	87.98 (14)	Al—O3—C31	138.6 (3)
O2—Al—O5	92.33 (14)	Al—O4—C41	153.3 (4)
O2—Al—O6	90.66 (13)	Al—O5—C51	152.9 (4)
O3—Al—O4	86.65 (14)	Al—O6—C61	143.6 (3)
O3—Al—O5	175.86 (15)		
Al—O1—C11—N1	-177.3 (3)	Al—O4—C41—N4	138.8 (8)
Al—O1—C11—C12	2.3 (7)	Al—O4—C41—C42	-44.5 (11)
Al—O2—C21—N2	-180.0 (4)	Al—O5—C51—N5	-164.4 (5)
Al—O2—C21—C22	1.2 (8)	Al—O5—C51—C52	17.1 (10)
Al—O3—C31—N3	149.3 (4)	Al—O6—C61—N6	146.8 (4)
Al—O3—C31—C32	-33.1 (7)	Al—O6—C61—C62	-33.9 (8)

The bridging H7 atom of the  $[\text{H}(\text{DMA})_2]^+$  cation was located in a difference map and its  $U_{\text{iso}}$  value was refined. Methyl H atoms were placed at idealized positions with fixed C—H distances (0.96 Å) and H—C—H angles, and refined using a rotating model, with displacement parameters set as 1.5 times the equivalent isotropic displacement parameter of the methyl C atom. Disorder of four perchlorate O atoms (O23, O24, O33 and O34) was suggested by their highly

anisotropic displacement parameters, so that two positions for each atom were calculated by moving along the direction of the largest eigenvalue of the displacement tensor, with a fixed site-occupation factor of 0.5. After the refinement, the displacement parameters for some perchlorate O atoms were still somewhat anisotropic and the Cl—O distances showed variation, but further attempts with more disordered positions did not converge. The Cl—O distances around Cl4 were restrained to be equal. One of the perchlorate anions (Cl4/O41—O44) lies on an inversion center and is therefore disordered with equal occupancies for both components. Atom N4 also shows high anisotropy and possibility of disorder, which is a likely cause for the longer N4—C43 and C41—C42 distances; an attempt to use disordered positions was unsuccessful.

Data collection: *CAD-4 Software* (Enraf-Nonius, 1989); cell refinement: *CAD-4 Software*; data reduction: *CAD-4 Software*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1990); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3* (Farrugia, 1997); software used to prepare material for publication: *SHELXL97*.

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