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# [Tris(2-aminoethyl)amine- $\kappa^4N$ ]-chloro(dimethyl sulfoxide-O)cobalt(III) diperchlorate

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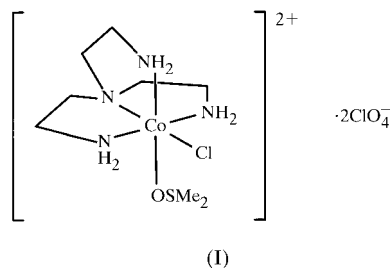
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The title compound, [CoCl(tren)(dmsO)](ClO<sub>4</sub>)<sub>2</sub> [tren is tris(2-aminoethyl)amine, C<sub>6</sub>H<sub>18</sub>N<sub>4</sub>; dmsO is dimethyl sulfoxide, C<sub>2</sub>H<sub>6</sub>OS], is the first crystal structure reported with dmsO coordinated to Co<sup>III</sup>. It crystallizes with two independent molecules in the asymmetric unit. A localized non-crystallographic inversion centre is observed between the two cations.

## Comment

The title compound was obtained during the hydrolysis study of alanine methyl ester catalyzed by [Co(tren)Cl<sub>2</sub>]Cl (Kimura *et al.*, 1970). Although crystal structures of several Co<sup>II</sup>-dmsO complexes have been determined (Kinoshita & Ouchi, 1988; Amari *et al.*, 1994; Lu *et al.*, 1998; Baidina *et al.*, 1991; Ciccamese *et al.*, 1993; Tkachev *et al.*, 1994), the title compound, (I), represents the first crystal structure with dmsO coordinated



to Co<sup>III</sup>. It is noteworthy that in all of these Co<sup>II</sup>-dmsO complexes, the dmsO ligands always lie *trans* to each other. However, in the title complex, the dmsO ligand is *trans* to an N atom of the tren ligand. There are two independent molecules in the asymmetric unit. The coordination geometries of these two molecules are very similar. Cobalt(III) is six-coordinate with four N atoms from tren, one chloro, and one O atom from dmsO. Chloro is in a *trans* position to the tertiary N atom of tren, with Co—Cl = 2.2500 (9) Å for Co1 and 2.2422 (9) Å for

Co2. The distances between Co and the O atom of dmsO are 1.952 (2) and 1.949 (2) Å in the two molecules. Both cations possess the same, and only, intramolecular hydrogen-bonding interaction between the amine proton and sulfur, N2—H2D···S1 and N7—H7D···S2. All the other amine protons are involved in extensive intermolecular hydrogen-bonding interactions with perchlorate O atoms.

## Experimental

[Co(tren)Cl<sub>2</sub>]Cl (0.5 g, 1.60 mmol) in water was mixed with alanine methyl ester in DMSO (15 ml). Diethylamine (0.8 ml) was added to the resulting solution and kept at 333 K for 30 min. The precipitate was collected and redissolved in water to which extra NaClO<sub>4</sub> had been added. Suitable crystals were obtained after a few days.

## Crystal data

[CoCl(C<sub>6</sub>H<sub>18</sub>N<sub>4</sub>)(C<sub>2</sub>H<sub>6</sub>OS)](ClO<sub>4</sub>)<sub>2</sub>  
*M<sub>r</sub>* = 517.65  
 Monoclinic, *P*2<sub>1</sub>/*c*  
*a* = 20.0177 (10) Å  
*b* = 15.7193 (8) Å  
*c* = 12.9074 (7) Å  
 $\beta$  = 103.235 (1)°  
*V* = 3953.6 (4) Å<sup>3</sup>  
*Z* = 8  
*D<sub>x</sub>* = 1.739 Mg m<sup>-3</sup>  
 Mo *K*α radiation  
 Cell parameters from 8192 reflections  
 $\theta$  = 1.66–27.02°  
 $\mu$  = 1.427 mm<sup>-1</sup>  
*T* = 293 (2) K  
 Column, red  
 0.3 × 0.2 × 0.15 mm

## Data collection

Bruker SMART CCD area-detector  
 diffractometer  
 $\varphi$  and  $\omega$  scans  
 Absorption correction: empirical (Blessing, 1995)  
*T<sub>min</sub>* = 0.674, *T<sub>max</sub>* = 0.814  
 23 159 measured reflections  
 8616 independent reflections  
 6484 reflections with *I* > 2σ(*I*)  
*R<sub>int</sub>* = 0.025  
 $\theta_{max}$  = 27.02°  
*h* = −25 → 25  
*k* = −15 → 20  
*l* = −16 → 15

## Refinement

Refinement on *F*<sup>2</sup>  
*R*(*F*) = 0.044  
*wR*(*F*<sup>2</sup>) = 0.130  
*S* = 1.036  
 8616 reflections  
 466 parameters  
 H-atom parameters constrained  
 $w = 1/[\sigma^2(F_o^2) + (0.0977P)^2 + 1.2289P]$   
 where  $P = (F_o^2 + 2F_c^2)/3$   
 $(\Delta/\sigma)_{max} = 0.002$   
 $\Delta\rho_{max} = 0.99 \text{ e \AA}^{-3}$   
 $\Delta\rho_{min} = -0.84 \text{ e \AA}^{-3}$

Table 1

Hydrogen-bonding geometry (Å, °).

<i>D</i> —H··· <i>A</i>	<i>D</i> —H	H··· <i>A</i>	<i>D</i> ··· <i>A</i>	<i>D</i> —H··· <i>A</i>
N2—H2C···O10 <sup>i</sup>	0.90	2.13	3.009 (5)	167
N2—H2D···O5 <sup>ii</sup>	0.90	2.24	3.021 (4)	145
N2—H2D···S1	0.90	2.85	3.379 (3)	119
N3—H3C···O9 <sup>iii</sup>	0.90	2.65	3.201 (5)	121
N3—H3D···Cl1 <sup>iv</sup>	0.90	2.51	3.399 (3)	169
N4—H4C···O3	0.90	2.65	3.314 (5)	131
N4—H4C···Cl1 <sup>iv</sup>	0.90	2.70	3.462 (3)	143
N4—H4D···O9 <sup>i</sup>	0.90	2.18	3.051 (5)	162
N6—H6C···Cl2 <sup>v</sup>	0.90	2.53	3.421 (3)	168
N6—H6D···O17A <sup>vi</sup>	0.90	2.44	3.040 (9)	124
N7—H7D···O6	0.90	2.21	3.052 (5)	156
N7—H7D···S2	0.90	2.87	3.402 (3)	120
N7—H7E···O18A <sup>iii</sup>	0.90	2.07	2.932 (10)	161
N8—H8D···O17B <sup>iii</sup>	0.90	2.17	2.940 (7)	144
N8—H8D···O17B <sup>iii</sup>	0.90	2.17	2.940 (7)	144
N8—H8D···O17A <sup>iii</sup>	0.90	2.31	3.178 (9)	163
N8—H8D···O18A <sup>iii</sup>	0.90	2.61	3.334 (10)	139
N8—H8E···O11 <sup>vii</sup>	0.90	2.47	3.181 (5)	136
N8—H8E···Cl2 <sup>v</sup>	0.90	2.73	3.472 (3)	141

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

All H atoms were placed in idealized positions and refined as riding atoms with relative isotropic displacement parameters. The O atoms of the two perchlorate anions are disordered.

Data collection: *SMART* (Bruker, 1998); cell refinement: *SMART* (Bruker, 1998); data reduction: *SAINTE-Plus* (Bruker, 1999); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); software used to prepare material for publication: *SHELXTL* (Bruker, 1998).

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