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N,*N*′-Bis[2-(dimethylammonio)ethyl]oxamide dinitrate

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Key indicators

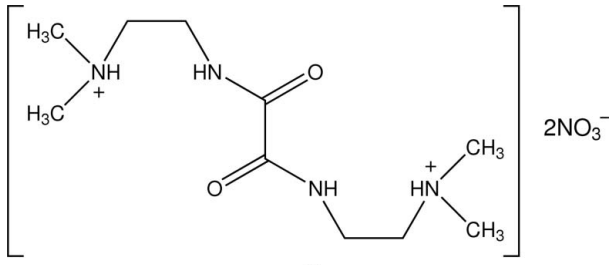
Single-crystal X-ray study
T = 298 K
Mean σ(C–C) = 0.002 Å
R factor = 0.041
wR factor = 0.110
Data-to-parameter ratio = 18.7

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

In the crystal structure of the title compound, $C_{10}H_{24}N_4O_2^{2+}\cdot 2NO_3^-$ or $[H_4dmaeoxd](NO_3)_2$ {$H_2dmaeoxd$ is *N*,*N*′-bis[2-(dimethylamino)ethyl]oxamide}, the diprotonated $H_4dmaeoxd$ dication occupies a special position on an inversion centre and exhibits a *transoid* conformation. The six non-H atoms of the oxamide group are almost exactly coplanar. Two symmetry-independent NH groups of the dication form hydrogen bonds with two O atoms belonging to one NO_3^- anion. Four independent C—H···O interactions link dications and anions into an infinite three-dimensional system.

Comment

Thanks to their bioactivities (Barta-Szalai *et al.*, 2004) and their versatile bridging function (Ojima & Nonoyama, 1988), many oxamide compounds and their complexes have been the subject of extensive investigation (Ruiz *et al.*, 1999). The original goal of this work was to prepare a dinuclear imidazole complex of Cr^{III}, in which the $H_2dmaeoxd$ ligand {$H_2dmaeoxd$ is *N*,*N*′-bis[2-(dimethylamino)ethyl]oxamide} has a bridging function and the imidazoles act as terminal ligands. However, in the course of the synthesis of the complex, the title compound, $(H_4dmaeoxd)(NO_3)_2$, (I), was obtained. In this paper, we report the synthesis of this compound and the results of its X-ray crystallographic study.



(I)

The crystal structure of (I) is built up of $H_4dmaeoxd^{2+}$ dications and NO_3^- anions. The cation has a *transoid* conformation and occupies a special position on an inversion centre (Fig. 1). The six non-H atoms of the oxamide group are almost coplanar, which is similar to other oxamide compounds (Su *et al.*, 1999; Perić *et al.*, 2001). The C5—C5^{i} distance [symmetry code: (i) 1 − *x*, 1 − *y*, 2 − *z*] of 1.541 (3) Å is somewhat longer than the analogous bond in the copper complex of the deprotonated parent oxamide [1.518 (5) Å; Real *et al.*, 1993].

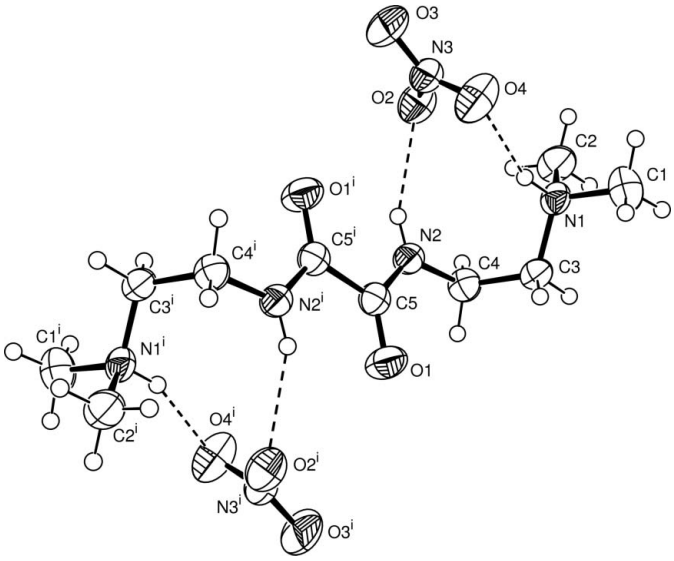


Figure 1
The dication and anions in the structure of (I). Displacement ellipsoids are drawn at the 50% probability level. Dashed lines indicate hydrogen bonds. [Symmetry code: (i) $1-x, 1-y, 2-z$.]

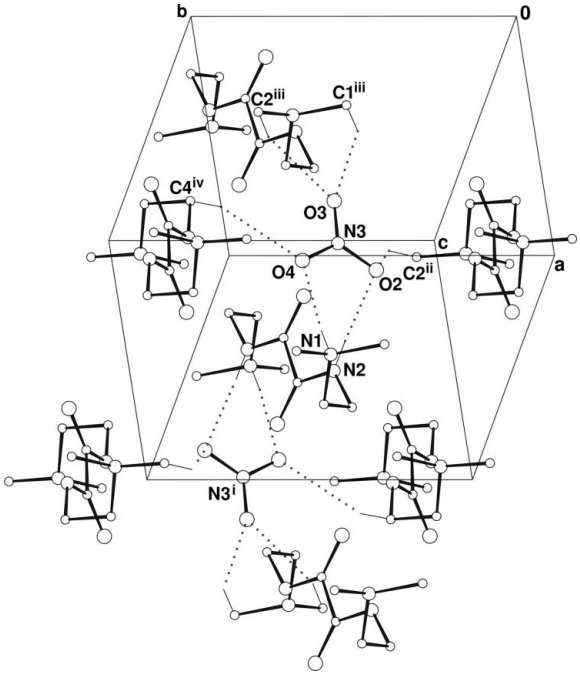


Figure 2
A packing diagram for (I), viewed approximately down [13$\bar{1}$]. Hydrogen bonds are shown as dotted lines. H atoms not involved in hydrogen bonding have been omitted for clarity. [Symmetry codes: (i) $1-x, 1-y, 2-z$; (ii) $1-x, y-\frac{1}{2}, \frac{3}{2}-z$; (iii) $x-1, y, z$; (iv) $1-x, \frac{1}{2}+y, \frac{3}{2}-z$.]

As shown in Fig. 1, two symmetry-independent 'active' H atoms (those bonded to atoms N1 and N2) on each flank of the cation form two hydrogen bonds with atoms O4 and O2 of the same nitrate anion, which gives rise to an ion-triplet, H_4dmaeoxd$^{2+}\cdot 2NO_3^-$. Thanks to four additional non-classical C—H···O hydrogen bonds (Fig. 2; Table 1), a three-dimensional supramolecular hydrogen-bonded network is formed in the crystal structure of (I). It is noteworthy that each nitrate anion interacts with four H_4dmaeoxd^{2+} dications and forms six hydrogen bonds, which is consistent with six equivalent low-energy regions on the electrostatic potential surface of nitrate (Hay *et al.*, 2002).

Experimental

All reagents were of AR grade and were used without further purification. The ligand, H_2dmaeoxd, was prepared according to the method of Ojima & Yamada (1970). To a solution of H_2dmaeoxd (0.0230 g, 0.1 mmol) in methanol (10 ml) were added successively piperidine (0.2 mmol) and a solution of $Cr(NO_3)_3\cdot 9H_2O$ (0.0800 g, 0.2 mmol) in methanol (10 ml). The mixture was stirred quickly until the solution became clear and then imidazole (0.0272 g, 0.4 mmol) in methanol (10 ml) was added. Stirring of the reaction mixture was continued at 333 K for 2 h. Colourless crystals of the title compound suitable for X-ray analysis precipitated on the third day, after the solution had been left to stand at room temperature.

Crystal data

$C_{10}H_{24}N_4O_2^{2+}\cdot 2NO_3^-$
$M_r = 356.35$
Monoclinic, $P2_1/c$
$a = 7.9809$ (19) Å
$b = 8.703$ (2) Å
$c = 12.214$ (3) Å
$\beta = 99.522$ (3)°
$V = 836.7$ (3) Å^3
$Z = 2$
$D_x = 1.414$ Mg m^{-3}
Mo $K\alpha$ radiation
$\mu = 0.12$ mm^{-1}
$T = 298$ (2) K
Flake, colourless
0.27 × 0.16 × 0.09 mm

Data collection

Bruker APEX area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2003)
$T_{min} = 0.968$, $T_{max} = 0.989$
5342 measured reflections
2043 independent reflections
1237 reflections with $I > 2\sigma(I)$
$R_{int} = 0.025$
$\theta_{max} = 28.4°$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.041$
$wR(F^2) = 0.110$
$S = 1.00$
2043 reflections
109 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0388P)^2 + 0.2517P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.20$ e Å^{-3}
$\Delta\rho_{min} = -0.16$ e Å^{-3}

Table 1
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1···O4	0.859 (19)	1.998 (19)	2.820 (2)	159.7 (17)
N2—H2···O2	0.842 (19)	2.23 (2)	3.023 (2)	156.1 (16)
C1—H1*C*···O3^{i}	0.96	2.42	3.272 (2)	147
C2—H2*C*···O3^{i}	0.96	2.49	3.321 (3)	145
C2—H2*A*···O2^{ii}	0.96	2.54	3.267 (3)	133
C4—H4*A*···O4^{iii}	0.97	2.58	3.329 (3)	135

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

The N-bound H atoms were located in a difference Fourier map and refined isotropically [N—H 0.85 (2) and 0.84 (2) Å]. The C-bound H atoms were placed in calculated positions, with C—H

distances of 0.97 Å (methylene) and 0.96 Å (methyl), and refined as riding, with $U_{iso}(H) = 1.2U_{eq}$ of the carrier atom ($1.5U_{eq}$ for methyl H atoms).

Data collection: *SMART* (Bruker, 2002); cell refinement: *SAINT* (Bruker, 2002); data reduction: *SAINT*; program(s) used to solve structure: *SIR92* (Altomare *et al.*, 1993); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997) and *CAMERON* (Watkin *et al.*, 1993); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

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