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Acta Cryst. (1999). C**55**, 636–637

N,N'-Bis(2-ammonioethyl)oxamide diperchlorate

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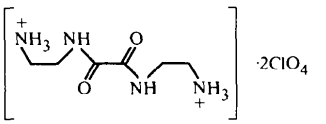
(*Received 26 May 1998; accepted 9 November 1998*)

Abstract

The title compound, $C_6H_{16}N_4O_2^{2+}\cdot 2ClO_4^-$, was obtained by an acid-catalyzed hydrolysis of the *N,N'*-bis[2-(salicylideneamino)ethyl]oxamide Schiff base. The oxamides are in a *trans*-conformation with all six non-H atoms essentially coplanar. Both primary N atoms are protonated to form the diperchlorate salt.

Comment

N,N'-disubstituted oxamides are used in the synthesis of polymetallic species with peculiar magneto-optical properties and in the design of a synthetic strategy for the development of molecular-based devices (Ojima & Nonoyama, 1988; Aguiari *et al.*, 1997). One of the advantages of these ligands is their easy *cis-trans* conformational interconversion affording symmetric and asymmetric oxamidato bridges (Benelli *et al.*, 1993). Since much research interest is focused on their conformation and bridging behaviour, it was considered useful to report the structure of the ligand itself.



(I)

The structure of the title compound, (I), consists of a doubly protonated *N,N'*-bis(2-ammonioethyl)oxamide cation and two perchlorate anions, which are joined together by hydrogen bonding. There is a crystallographically imposed centre of symmetry lying in the middle of the C1—C1($3-x, 1-y, 1-z$) bond. A drawing of the doubly protonated *N,N'*-bis(2-ammonioethyl)oxamide cation with the numbering scheme is shown in Fig. 1 and relevant distances and angles are given in Table 1. The oxamide groups take a *trans*-conformation and the six atoms are planar to ±0.002 Å. The C1—O1 and C1—N1 bonds display some double-bond character while the C—C bonds are typical for single bonds (Orpen *et al.*, 1989), suggesting electronic delocalization on the OCN group. The terminal primary N atom is protonated to form hydrogen bonds with the perchlorate anions [N1···O13 2.932 (5) Å].

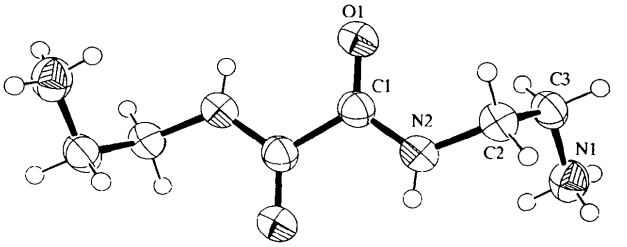


Fig. 1. Molecular structure of the *N,N'*-bis(2-ammonioethyl)oxamide cation with displacement ellipsoids at the 30% probability level.

Experimental

The compound was obtained as a by-product of the reaction of the *N,N'*-bis[2-(salicylideneamino)ethyl]oxamide Schiff base with hydrated lanthanide perchlorates in a methanol–acetonitrile medium. It was evident that the trace amount of free perchloric acid in the lanthanide salt resulted in this acid-catalyzed hydrolysis.

Crystal data

$C_6H_{16}N_4O_2^{2+}\cdot 2ClO_4^-$
M_r = 375.13
Monoclinic
$P2_1/n$
a = 8.517 (2) Å
b = 7.731 (2) Å
c = 10.830 (2) Å
β = 90.38 (3)°
V = 713.1 (3) Å^3
Z = 2
D_x = 1.747 Mg m^{-3}
D_m not measured
Mo $K\alpha$ radiation
λ = 0.71073 Å
Cell parameters from 478 reflections
θ = 6.05–25.00°
μ = 0.516 mm^{-1}
T = 293 (2) K
Prismatic
0.25 × 0.20 × 0.15 mm
Yellow

Data collection

Rigaku R-AXIS IIC IP diffractometer	R_{int} = 0.064
Oscillation frame scans	θ_{max} = 25.62°
Absorption correction: none	$h = -10 \rightarrow 0$
2158 measured reflections	$k = -9 \rightarrow 9$
1186 independent reflections	$l = -13 \rightarrow 13$
1104 reflections with $I > 2\sigma(I)$	Intensity decay: none

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0948P)^2 + 0.2199P]$ where $P = (F_o^2 + 2F_c^2)/3$
$R[F^2 > 2\sigma(F^2)]$ = 0.066	$(\Delta/\sigma)_{max} < 0.001$
$wR(F^2)$ = 0.175	$\Delta\rho_{max}$ = 0.394 e Å^{-3}
S = 1.115	$\Delta\rho_{min}$ = −0.648 e Å^{-3}
1186 reflections	Extinction correction: none
124 parameters	Scattering factors from *International Tables for Crystallography* (Vol. C)
Only positional coordinates of H atoms refined	

Table 1. *Selected geometric parameters (Å, °)*

N1—C3	1.485 (5)	C2—C3	1.504 (5)
N2—C1	1.328 (4)	C11—O12	1.415 (3)
N2—C2	1.449 (5)	C11—O11	1.417 (3)
O1—C1	1.234 (4)	C11—O13	1.439 (3)
C1—C1^{i}	1.536 (6)	C11—O14	1.445 (3)
C1—N2—C2	122.4 (3)	O12—C11—O11	110.0 (3)
O1—C1—N2	125.0 (3)	O12—C11—O13	109.5 (2)
O1—C1—C1^{i}	121.3 (4)	O11—C11—O13	108.9 (2)
N2—C1—C1^{i}	113.7 (4)	O12—C11—O14	108.8 (2)
N2—C2—C3	114.0 (3)	O11—C11—O14	110.4 (2)
N1—C3—C2	112.0 (3)	O13—C11—O14	109.2 (2)

Symmetry code: (i) $3 - x, 1 - y, 1 - z$.

Diffraction intensities were collected on a Rigaku R-AXIS IIC image-plate diffractometer by taking oscillation photographs (total oscillation range ϕ = 0–180°, 20 frames in total; oscillation angle $\Delta\phi$ = 9° per frame; exposure time = 8 min per frame). The data set is complete only to 82% due to a blind region in the experimental set-up. H atoms were located in a difference map and the positional coordinates were refined.

Cell refinement: *BIOTEX* (Pflugrath *et al.*, 1996). Data reduction: *BIOTEX*. Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1990). Program(s) used to refine structure: *SHELXL*93 (Sheldrick, 1993). Molecular graphics: *SHELXS*86. Software used to prepare material for publication: *SHELXL*93.

This work was supported by the NNSF of China and the Postdoctoral Fund of Guangdong Province for C-YS.

Supplementary data for this paper are available from the IUCr electronic archives (Reference: OS1034). Services for accessing these data are described at the back of the journal.

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Acta Cryst. (1999). C**55**, 637–639

3,17-Dioxo-4-oxaandrostane-5α-carbaldehyde

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(*Received 27 July 1998; accepted 24 November 1998*)

Abstract

The title compound, $C_{19}H_{26}O_4$, has a C5 atom with an unusual environment, which leads to a molecular 5α configuration. Ring *A* is highly flattened. The carbaldehyde group is slightly disordered, with interchange of the H and O positions.

Comment

This work is part of an ongoing project of the structure determination of steroids with clinical interest and their precursors (Ramos Silva *et al.*, 1996; Andrade *et al.*, 1997; Paixão *et al.*, 1997; Paixão, Andrade, de Almeida, Costa *et al.*, 1998; Paixão, Andrade, de Almeida, Tavares da Silva *et al.*, 1998). During former studies leading to an improved synthesis of formestane (Tavares da Silva *et al.*, 1996), an aromatase inhibitor used to treat breast cancer, the title compound, (I), has been isolated as one of the products obtained through oxidation of androst-4-ene-3,17-dione with potassium permanganate. Knowing that *trans*-fused aldehydo lactones of this type can be important intermediates in preparing 4-cyclooctenone derivatives, *e.g.* in steroids to increase a biological response (Philippo *et al.*, 1991), we have successfully increased the amount of the title compound in the product distribution by changing the reaction conditions.