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Bis(2-amino-2-thiazolinium) dichromate(VI)

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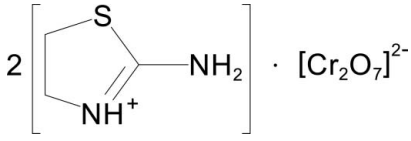
Key indicators: single-crystal X-ray study; T = 298 K; mean σ(C–C) = 0.003 Å; R factor = 0.023; wR factor = 0.071; data-to-parameter ratio = 15.7.

The crystal structure of the title compound, $(C_3H_7N_2S)_2[Cr_2O_7]$, consists of 2-amino-2-thiazolinium cations and discrete dichromate anions linked together by N—H···O hydrogen bonds to form a one-dimensional ribbon structure lying parallel to the (102) plane and running along the b axis. The dichromate anion is located on a twofold axis that passes through its central O atom.

Related literature

For related structure, see: Sieroń (2007). For related literature, see: Allen *et al.* (1987); Etter *et al.* (1990).



Experimental

Crystal data

$(C_3H_7N_2S)_2[Cr_2O_7]$
M_r = 422.35
Monoclinic, $P2/n$
a = 8.0304 (1) Å
b = 6.5332 (1) Å
c = 14.1019 (2) Å
β = 95.065 (2)°
V = 736.96 (2) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 1.79 mm^{-1}
T = 298 K
0.60 × 0.40 × 0.25 mm

Data collection

Kuma KM-4 CCD diffractometer
Absorption correction: multi-scan (Oxford Diffraction, 2007)
T_{min} = 0.410, T_{max} = 0.636
9894 measured reflections
1708 independent reflections
1604 reflections with $I > 2\sigma(I)$
R_{int} = 0.022

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.023
$wR(F^2)$ = 0.071
S = 1.12
1708 reflections
109 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.39 e Å^{-3}
$\Delta\rho_{min}$ = −0.31 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Cr1—O1	1.6231 (13)	Cr1—O3	1.7656 (6)
Cr1—O2	1.6099 (13)	Cr1—O4	1.6033 (17)
O1—Cr1—O2	109.03 (7)	O2—Cr1—O4	109.99 (9)
O1—Cr1—O3	108.80 (6)	O3—Cr1—O4	108.07 (8)
O1—Cr1—O4	109.10 (8)	Cr1—O3—Cr1^{i}	155.81 (17)
O2—Cr1—O3	111.81 (10)		

Symmetry code: (i) $-x+\frac{3}{2}, y, -z+\frac{3}{2}$.

Table 2
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1···O1	0.83 (3)	2.00 (3)	2.8233 (19)	171 (2)
N2—H21···O2	0.83 (2)	2.08 (2)	2.900 (3)	174 (2)
N2—H22···O1^{ii}	0.78 (3)	2.15 (3)	2.897 (2)	159 (2)

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

Data collection: *CrysAlis CCD* (Oxford Diffraction, 2007); cell refinement: *CrysAlis RED* (Oxford Diffraction, 2007); data reduction: *CrysAlis RED*; program(s) used to solve structure: *SHELXTL* (Sheldrick, 2001); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL* and *Mercury* (Macrae *et al.*, 2006); software used to prepare material for publication: *PLATON* (Spek, 2003).

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: IS2186).

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  doi:10.1107/S1600536807032023

supplementary materials

Acta Cryst. (2007). E**63**, m2068 [**doi:10.1107/S1600536807032023**]

Bis(2-amino-2-thiazolinium) dichromate(VI)

L. Sieron

Comment

The title compound, (I), was investigated as part of a structural study on hydrogen-bonding patterns in 2-amino-2-thiazolinium salts (Sieroń, 2007).

In (I), the asymmetric unit is composed of one 2-amino-2-thiazolinium cation and half dichromate anion (Fig. 1). The cation occupies a general position whereas the anion is located on a twofold axis. The bond lengths and angles are within normal ranges (Allen *et al.*, 1987).

The dichromate anion links the cation *via* intermolecular N—H···O hydrogen bonds forming eight-membered ring with graph-set descriptor $R_2^2(8)$ (Etter *et al.*, 1990). The other N—H···O hydrogen bond associates adjacent cations *via* O1 atom into $C_2^2(6)$ chains. The combination of these motifs results in the formation of a one-dimensional ribbon structure lying parallel to the (102) plane and running along the *b* axis, as shown in Fig. 2. The same motifs are found in bis(2-amino-2-thiazolinium) tetra-μ-formato-κ^8O:*O*′-bis[(formato-κ*O*)copper(II)] structure (Sieroń, 2007).

Experimental

The title compound was prepared by dissolving 2-amino-2-thiazoline (1 mmol) and chromic anhydride (1 mmol) in hot water (25 ml). After a few days, prism-shaped orange crystals were obtained at room temperature.

Refinement

All H atoms were initially located in a difference Fourier map. C-bonded H atoms were placed in geometrically idealized positions and constrained to ride on their parent atoms, with C–H distances of 0.97 Å and U_{iso}(H) = 1.2U_{eq}(C). Amine H atoms were refined freely.

Figures

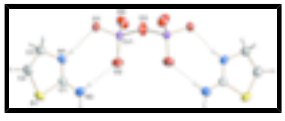

Fig. 1. The molecular structure of (I), showing the atom-numbering scheme. Displacement ellipsoids for non-H atoms are drawn at the 50% probability level. Unlabelled atoms are related to labelled atoms by symmetry code (−*x* + 3/2, *y*, −*z* + 3/2). Dotted lines indicate hydrogen bonds.

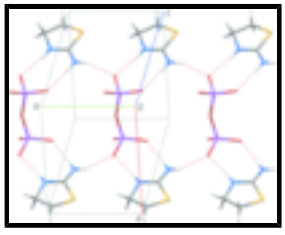

Fig. 2. A packing view of (I), showing a ribbon of hydrogen-bonded (dashed lines) cations and anions running along the *b* axis.

Bis(2-amino-2-thiazolinium) dichromate(VI)

Crystal data

$(C_3H_7N_2S)_2[Cr_2O_7]$
$M_r = 422.35$
Monoclinic, $P2/n$
Hall symbol: -P 2yac
$a = 8.0304$ (1) Å
$b = 6.5332$ (1) Å
$c = 14.1019$ (2) Å
$\beta = 95.065$ (2)º
$V = 736.96$ (2) Å^3
$Z = 2$
$F_{000} = 428$
$D_x = 1.903$ Mg m^{-3}
Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å
Cell parameters from 8378 reflections
$\theta = 2.5$–30.0º
$\mu = 1.79$ mm^{-1}
$T = 298$ K
Prism, orange
0.60 × 0.40 × 0.25 mm

Data collection

Kuma KM-4 CCD diffractometer
Monochromator: graphite
Detector resolution: 8.2356 pixels mm^{-1}
$T = 298$ K
ω scans
Absorption correction: multi-scan (Oxford Diffraction, 2007)
$T_{min} = 0.410$, $T_{max} = 0.636$
9894 measured reflections
1708 independent reflections
1604 reflections with $I > 2\sigma(I)$
$R_{int} = 0.022$
$\theta_{max} = 27.5$º
$\theta_{min} = 2.8$º
$h = -10 \rightarrow 10$
$k = -8 \rightarrow 8$
$l = -18 \rightarrow 18$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.023$
$wR(F^2) = 0.071$
$S = 1.12$
1708 reflections
109 parameters
Primary atom site location: structure-invariant direct methods
Hydrogen site location: difference Fourier map
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0408P)^2 + 0.3039P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.39$ e Å^{-3}
$\Delta\rho_{min} = -0.31$ e Å^{-3}
Extinction correction: SHELXTL, $Fc^* = kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.0186 (18)

Special details

Geometry. Bond distances, angles *etc*. have been calculated using the rounded fractional coordinates. All su's are estimated from the variances of the (full) variance-covariance matrix. The cell e.s.d.'s are taken into account in the estimation of distances, angles and torsion angles

Refinement. Refinement on F^2 for ALL reflections except those flagged by the user for potential systematic errors. Weighted *R*-factors *wR* and all goodnesses of fit S are based on F^2, conventional *R*-factors *R* are based on F, with F set to zero for negative F^2. The observed criterion of $F^2 > 2\text{sigma}(F^2)$ is used only for calculating –*R*-factor-obs *etc*. and is not relevant to the choice of reflections for refinement. *R*-factors based on F^2 are statistically about twice as large as those based on F, and *R*-factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
S1	0.21595 (6)	−0.12790 (7)	1.02709 (4)	0.0414 (2)
N1	0.31040 (19)	0.2021 (2)	0.95161 (10)	0.0331 (4)
N2	0.3482 (3)	−0.0856 (3)	0.86045 (13)	0.0426 (5)
C1	0.30017 (19)	0.0056 (3)	0.93613 (11)	0.0294 (4)
C2	0.2114 (3)	0.1018 (3)	1.09950 (13)	0.0393 (5)
C3	0.2295 (2)	0.2811 (3)	1.03239 (13)	0.0366 (5)
Cr1	0.53421 (3)	0.42057 (4)	0.74127 (2)	0.0263 (1)
O1	0.45721 (16)	0.4958 (2)	0.83802 (9)	0.0373 (4)
O2	0.4996 (2)	0.1793 (2)	0.72715 (11)	0.0523 (5)
O3	0.75000	0.4772 (4)	0.75000	0.0558 (7)
O4	0.4462 (2)	0.5453 (3)	0.65261 (11)	0.0542 (5)
H1	0.351 (3)	0.280 (4)	0.9131 (18)	0.052 (7)*
H2A	0.10670	0.11100	1.12860	0.0470*
H2B	0.30260	0.10060	1.14940	0.0470*
H3A	0.12060	0.33720	1.01140	0.0440*
H3B	0.29690	0.38820	1.06420	0.0440*
H21	0.386 (3)	−0.014 (4)	0.8192 (17)	0.043 (6)*
H22	0.352 (3)	−0.205 (5)	0.8556 (19)	0.053 (7)*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.0513 (3)	0.0294 (2)	0.0450 (3)	−0.0049 (2)	0.0127 (2)	0.0094 (2)
N1	0.0397 (7)	0.0273 (7)	0.0336 (7)	−0.0038 (6)	0.0102 (6)	0.0044 (6)
N2	0.0566 (10)	0.0313 (9)	0.0418 (9)	0.0075 (7)	0.0150 (8)	0.0013 (7)
C1	0.0263 (7)	0.0287 (8)	0.0330 (7)	0.0022 (6)	0.0019 (6)	0.0053 (6)
C2	0.0429 (10)	0.0434 (10)	0.0322 (8)	−0.0022 (7)	0.0065 (7)	0.0038 (7)
C3	0.0441 (9)	0.0314 (9)	0.0350 (8)	−0.0036 (7)	0.0072 (7)	−0.0029 (7)
Cr1	0.0251 (2)	0.0267 (2)	0.0280 (2)	−0.0017 (1)	0.0076 (1)	−0.0007 (1)
O1	0.0405 (6)	0.0331 (6)	0.0409 (7)	−0.0021 (5)	0.0190 (5)	−0.0040 (5)
O2	0.0727 (10)	0.0310 (7)	0.0556 (8)	−0.0064 (7)	0.0197 (7)	−0.0111 (6)
O3	0.0263 (9)	0.0703 (14)	0.0725 (14)	0.0000	0.0148 (9)	0.0000

O4	0.0521 (9)	0.0639 (10)	0.0454 (8)	−0.0076 (7)	−0.0029 (6)	0.0207 (7)

Geometric parameters (Å, °)

Cr1—O1	1.6231 (13)	N1—H1	0.83 (3)
Cr1—O2	1.6099 (13)	N2—H21	0.83 (2)
Cr1—O3	1.7656 (6)	N2—H22	0.78 (3)
Cr1—O4	1.6033 (17)	C2—C3	1.521 (3)
S1—C1	1.7369 (17)	C2—H2B	0.97
S1—C2	1.817 (2)	C2—H2A	0.97
N1—C1	1.304 (2)	C3—H3A	0.97
N1—C3	1.455 (2)	C3—H3B	0.97
N2—C1	1.310 (3)		
O1—Cr1—O2	109.03 (7)	S1—C1—N2	122.36 (16)
O1—Cr1—O3	108.80 (6)	N1—C1—N2	124.45 (17)
O1—Cr1—O4	109.10 (8)	S1—C2—C3	106.18 (13)
O2—Cr1—O3	111.81 (10)	N1—C3—C2	106.73 (15)
O2—Cr1—O4	109.99 (9)	C3—C2—H2A	110
O3—Cr1—O4	108.07 (8)	S1—C2—H2A	111
C1—S1—C2	91.65 (9)	S1—C2—H2B	110
Cr1—O3—Cr1^{i}	155.81 (17)	C3—C2—H2B	110
C1—N1—C3	117.04 (14)	H2A—C2—H2B	109
C3—N1—H1	121.5 (18)	N1—C3—H3A	110
C1—N1—H1	121.1 (18)	N1—C3—H3B	110
C1—N2—H21	118.0 (18)	C2—C3—H3A	110
C1—N2—H22	122.7 (19)	C2—C3—H3B	110
H21—N2—H22	119 (3)	H3A—C3—H3B	109
S1—C1—N1	113.19 (12)		

Symmetry codes: (i) −*x*+3/2, *y*, −*z*+3/2.

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1···O1	0.83 (3)	2.00 (3)	2.8233 (19)	171 (2)
N2—H21···O2	0.83 (2)	2.08 (2)	2.900 (3)	174 (2)
N2—H22···O1^{ii}	0.78 (3)	2.15 (3)	2.897 (2)	159 (2)

Symmetry codes: (ii) *x*, *y*−1, *z*.

Fig. 1

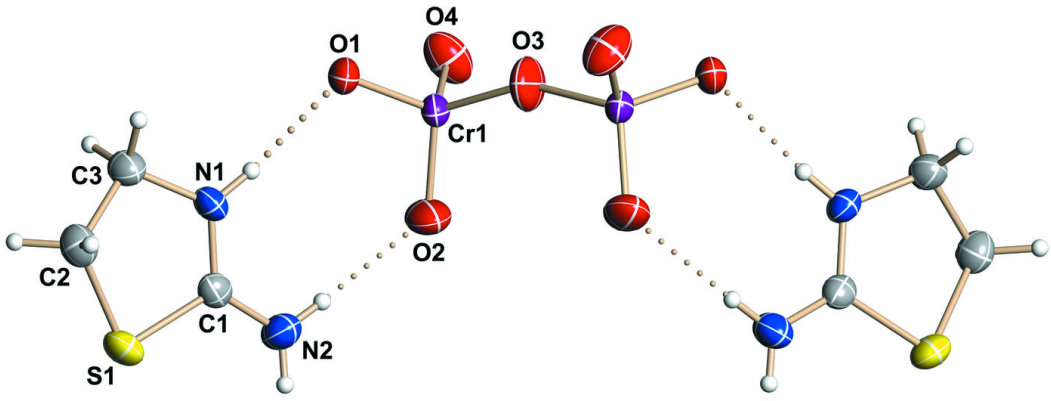

Fig. 2