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The ammonium chromium(III) alum $NH_4Cr(SO_4)_2\cdot12H_2O$

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Key indicators

Single-crystal X-ray study
T = 100 K
Mean σ(S–O) = 0.001 Å
Disorder in solvent or counterion
R factor = 0.019
wR factor = 0.051
Data-to-parameter ratio = 12.3

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

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The title compound, ammonium chromium(III) bis(sulfate) dodecahydrate, is composed of discrete NH_4^+ cations, $[Cr(H_2O)_6]^{3+}$ cations, SO_4^{2-} anions and water molecules. The Cr and N atoms are located on special positions of site symmetry $\overline{3}$; the S atom and one of the O atoms bonded to it are located on a threefold rotation axis. The crystal packing is stabilized by several hydrogen bonds.

Comment

The ammonium chromium alum $NH_4Cr(SO_4)_2\cdot12H_2O$, (I), is composed of discrete NH_4^+ cations, $[Cr(H_2O)_6]^{3+}$ cations, SO_4^{2-} anions and water molecules. The Cr and N atoms are located on special positions of site symmetry $\overline{3}$; the S atom and one of the O atoms bonded to it are located on a threefold rotation axis.

Alum (I) is isomorphous with $KCr(SO_4)_2\cdot12H_2O$ (Bacon & Gardner, 1958), $CsCo(SO_4)_2\cdot12H_2O$ (Beattie *et al.*, 1981), $NaAl(SO_4)_2\cdot12H_2O$ (Cromer *et al.*, 1967; Kay & Cromer, 1970), $ND_4Al(SO_4)_2\cdot12D_2O$ (Cromer & Kay, 1967), $KAl(SO_4)_2\cdot12H_2O$ (Beevers & Lipson, 1934), $RbAl(SO_4)_2\cdot12H_2O$ (Larson & Cromer, 1967), $NH_4Al(SO_4)_2\cdot12H_2O$ (Larson & Cromer, 1967; Abdeen *et al.*, 1981) and $KV(SO_4)_2\cdot12H_2O$ (Beattie *et al.*, 1996).

Although Nyburg *et al.* (2000) have discovered that the SO_4^{2-} group is disordered in many α-alums, we did not find this kind of disorder in the title compound.

Experimental

The title compound was prepared according to Gmelin (1962). Ethanol (7 ml) was dropped into a solution of 10 g $(NH_4)_2Cr_2O_7$ in 100 ml water and 11 ml H_2SO_4. Immediately, $NH_4Cr(SO_4)_2\cdot12H_2O$ precipitated from the reaction solution. Crystals of the title compound suitable for X-ray analysis were obtained by recrystallization from water at ambient temperature.

Crystal data

$NH_4Cr(SO_4)_2\cdot12H_2O$
M_r = 478.35
Cubic, $Pa\overline{3}$
a = 12.2491 (12) Å
V = 1837.9 (3) Å^3
Z = 4
D_x = 1.729 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 4846 reflections
θ = 3.7–27.6°
μ = 0.94 mm^{-1}
T = 100 (2) K
Block, violet
0.24 × 0.22 × 0.18 mm

Data collection

Stoe IPDS-II two-circle diffractometer
ω scans
Absorption correction: multi-scan (*MULABS*; Spek, 1990; Blessing, 1995)
T_{min} = 0.805, T_{max} = 0.849
5024 measured reflections
714 independent reflections
613 reflections with $I > 2\sigma(I)$
R_{int} = 0.026
θ_{max} = 27.7°
h = −12 → 15
k = −14 → 3
l = −15 → 15

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0348P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$R[F^2 > 2\sigma(F^2)] = 0.019$	$(\Delta/\sigma)_{max} < 0.001$
$wR(F^2) = 0.051$	$\Delta\rho_{max} = 0.29$ e Å^{-3}
$S = 1.04$	$\Delta\rho_{min} = -0.36$ e Å^{-3}
714 reflections	Extinction correction: *SHELXL*97
58 parameters	Extinction coefficient: 0.090 (3)
H atoms treated by a mixture of independent and constrained refinement	

Table 1
Selected bond distances (Å).

Cr1—O3^{i}	1.9602 (9)	S1—O2	1.4709 (18)
S1—O1	1.4704 (9)		

Symmetry code: (i) y, z, x.

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1···O4	0.85 (3)	2.16 (3)	3.0049 (11)	173 (3)
O3—H3A···O1^{viii}	0.80 (2)	1.84 (2)	2.6293 (13)	172 (2)
O3—H3B···O4^{ix}	0.83 (2)	1.78 (2)	2.6071 (13)	173 (2)
O4—H4B···O2^{x}	0.79 (3)	1.99 (3)	2.7752 (15)	171 (2)
O4—H4A···O1^{xi}	0.74 (2)	2.02 (2)	2.7454 (14)	169 (2)

Symmetry codes: (viii) $z, \frac{3}{2} - x, y - \frac{1}{2}$; (ix) $\frac{3}{2} - x, 1 - y, z - \frac{1}{2}$; (x) $\frac{1}{2} + x, \frac{3}{2} - y, 1 - z$; (xi) $\frac{1}{2} + z, x, \frac{3}{2} - y$.

H atoms bonded to O atoms were refined isotropically. H atoms bonded to N atoms are disordered. Their coordinates were refined with fixed individual displacement parameters [U_{iso}(H) = 1.2U_{eq}(N)].

Data collection: *X-AREA* (Stoe & Cie, 2001); cell refinement: *X-AREA*; data reduction: *X-AREA*; program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1990); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *XP* in *SHELXTL-Plus* (Sheldrick, 1991); software used to prepare material for publication: *SHELXL*97 and *PLATON* (Spek, 1990).

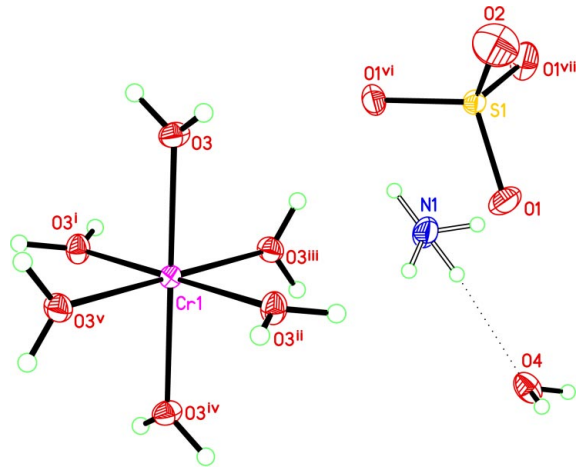

Figure 1
Perspective view of the title compound with the atom numbering; displacement ellipsoids are at the 50% probability level. Only four of the six disordered H atoms of the ammonium group are shown. [Symmetry codes: (i) y, z, x; (ii) $-y, -z, -x$; (iii) $1 - z, 1 - x, 1 - y$; (iv) $1 - x, 1 - y, 1 - z$; (v) z, x, y; (vi) $\frac{3}{2} - y, 1 - z, x - \frac{1}{2}$; (vii) $\frac{1}{2} + z, \frac{3}{2} - x, 1 - y$.]

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