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Crystal structure of rubidium sulfanilate sesquihydrate, $RbH_2NC_6H_4SO_3 \cdot 1.5H_2O$

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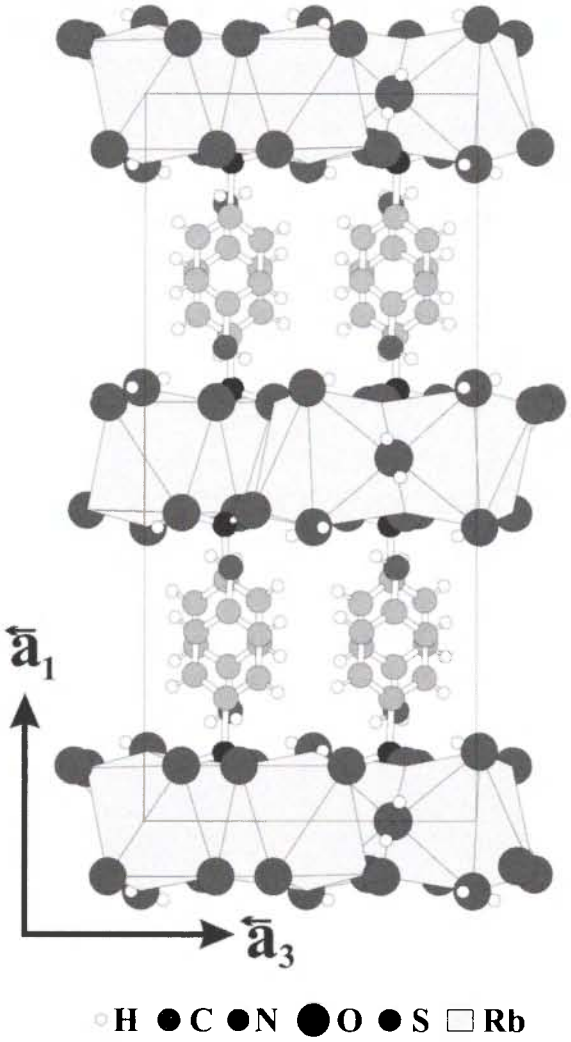

Abstract

$C_6H_9NO_{4.5}RbS$, orthorhombic, *Pbcm* (No. 60), a =23.271(5) Å, b =7.844(2) Å, c =10.635(2) Å, V =1941.3 Å^3, Z =8, $R_{gt}(F)$ =0.024, $R_w(F^2)$ =0.050, T = 293 K.

Source of material

Large single crystals were obtained from aqueous solutions of the title compound by controlled lowering of temperature and by slow evaporation in the range between 315 K and 290 K [1].

Discussion

The structures of rubidium, RBSH, and potassium sulfanilate sesquihydrate [2], KSH, are isotypic. In comparison with the K–O distances observed in KSH the Rb–O distances of the RbO_8 coordination polyhedra are slightly larger and vary between 2.825 Å and 3.292 Å. The larger size of the rubidium cation is also reflected by the thicknesses of the four-layer packages abcd which are periodically repeated along [100], i.e. a_1(RBSH) > a_1(KSH).

Table 1. Data collection and handling.

Crystal:	colorless prism, size 0.30 x 0.28 x 0.35 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	53.05 cm^{-1}
Diffractometer, scan mode:	Nonius CAD4, $\omega/2\theta$
$2\theta_{max}$:	55.92°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	5762, 2334
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 1669
$N(param)_{refined}$:	160
Programs:	PARST95 [3], SHELXS-86 [4], SHELXL-93 [5], ATOMS [6]

Table 2. Atomic coordinates and displacement parameters (in Å^2).

Atom	Site	x	y	z	U_{iso}
H(1)	8*d*	0.177(1)	0.752(4)	0.402(3)	0.037(8)
H(3)	8*d*	0.280(1)	1.010(4)	0.091(3)	0.032(7)
H(4)	8*d*	0.184(1)	1.016(4)	0.084(3)	0.037(8)
H(2)	8*d*	0.274(1)	0.749(4)	0.408(3)	0.039(8)
H(5)	8*d*	0.360(2)	0.778(5)	0.276(3)	0.05(1)
H(6)	8*d*	0.364(2)	0.916(5)	0.199(3)	0.05(1)
H(1W1)	8*d*	−0.026(1)	0.551(5)	0.231(4)	0.07(1)
H(1W2)	8*d*	−0.108(2)	0.468(6)	0.057(4)	0.09(2)
H(2W2)	8*d*	−0.097(1)	0.377(5)	−0.039(3)	0.04(1)

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Table 3. Atomic coordinates and displacement parameters (in $Å^2$).

Atom	Site	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
Rb	8*d*	0.01691(1)	0.23147(3)	0.05993(2)	0.0370(2)	0.0376(2)	0.0336(2)	−0.0030(1)	−0.0018(1)	−0.0014(1)
S	8*d*	0.09281(3)	0.89869(8)	0.24121(6)	0.0265(3)	0.0288(3)	0.0285(3)	−0.0023(3)	0.0012(3)	−0.0011(3)
O(1)	8*d*	0.07626(8)	0.8735(3)	0.1107(2)	0.037(1)	0.053(1)	0.032(1)	−0.0014(9)	−0.0061(8)	−0.005(1)
O(2)	8*d*	0.07263(8)	0.7650(3)	0.3239(2)	0.037(1)	0.049(1)	0.049(1)	−0.0094(9)	0.0044(9)	0.015(1)
O(3)	8*d*	0.07629(8)	1.0673(3)	0.2850(2)	0.0310(9)	0.038(1)	0.057(1)	0.0045(8)	0.0020(9)	−0.013(1)
C(1)	8*d*	0.1685(1)	0.8879(3)	0.2455(2)	0.028(1)	0.024(1)	0.026(1)	−0.000(1)	0.001(1)	−0.003(1)
C(2)	8*d*	0.1966(1)	0.8051(3)	0.3434(3)	0.037(1)	0.029(1)	0.028(1)	−0.002(1)	0.001(1)	0.002(1)
C(3)	8*d*	0.2560(1)	0.8011(4)	0.3468(3)	0.040(2)	0.031(1)	0.031(1)	0.002(1)	−0.008(1)	0.002(1)
C(4)	8*d*	0.2882(1)	0.8772(3)	0.2526(3)	0.029(1)	0.024(1)	0.039(1)	0.002(1)	−0.002(1)	−0.007(1)
C(5)	8*d*	0.2599(1)	0.9594(4)	0.1548(3)	0.033(2)	0.034(2)	0.034(1)	−0.001(1)	0.008(1)	0.003(1)
C(6)	8*d*	0.2007(1)	0.9645(3)	0.1513(3)	0.035(1)	0.032(2)	0.028(1)	0.004(1)	0.001(1)	0.005(1)
N	8*d*	0.3484(1)	0.8760(4)	0.2592(3)	0.032(1)	0.036(2)	0.061(2)	0.003(1)	−0.003(1)	0.002(1)
O(1W1)	4*c*	0	0.4936(4)	1/4	0.048(2)	0.033(2)	0.058(2)	0	−0.002(2)	0
O(1W2)	8*d*	−0.0961(1)	0.4654(3)	−0.0102(3)	0.062(2)	0.045(2)	0.047(2)	−0.004(1)	0.004(1)	−0.009(1)

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