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Crystal structure of cesium sulfanilate hydrate, $CsH_2NC_6H_4SO_3 \cdot H_2O$

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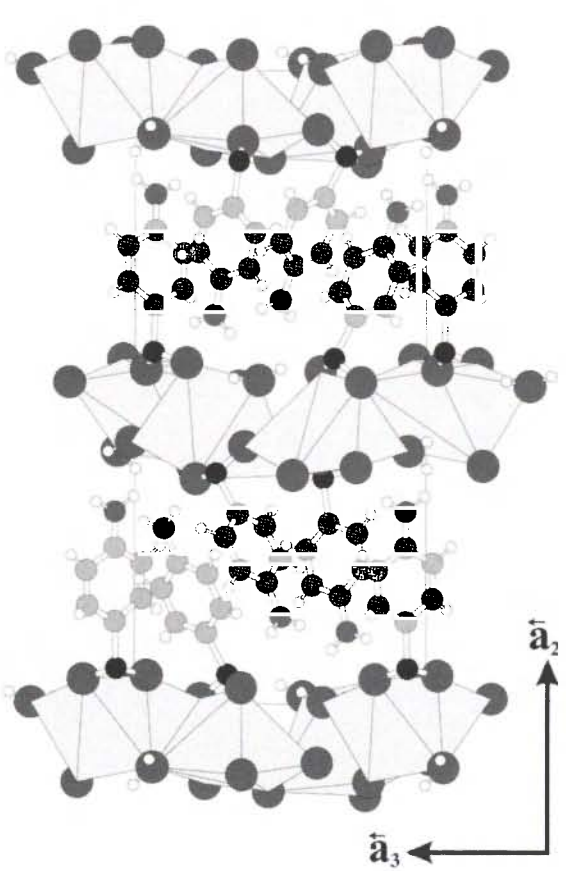

Abstract

$C_6H_8CsNO_4S$, monoclinic, $P12_11$ (No. 4), $a = 7.980(2)$ Å, $b = 23.420(5)$ Å, $c = 10.789(2)$ Å, $\beta = 91.52(3)°$, $V = 2015.7$ Å^3, $Z = 8$, $R_{gt}(F) = 0.028$, $R_w(F^2) = 0.065$, $T = 293$ 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 structure is built up by irregular CsO_6 and CsO_7 polyhedra with Cs–O distances between 3.048 Å and 3.352 Å. These polyhedra are linked by common vertices, common edges and by hydrogen bonds between the oxygen atoms of the hydrate molecules and oxygen atoms of the SO_3 groups to form layers parallel (010). The S–N axes of the sulfanilate anions located between the cation layers join the [010] direction at angles between 12° and 25°. Therefore, the distance of 11.71 Å between adjacent cation sheets is slightly smaller than the value one would expect from the structures of other alkali sulfanilates in combination with the ionic radius of cesium.

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Table 1. Data collection and handling.

Crystal:	colorless prism, size 0.25 x 0.25 x 0.30 mm
Wavelength:	Mo K_α radiation (0.71073 A)
μ:	38.64 cm^{-1}
Diffractometer, scan mode:	Nonius CAD4, $\omega/2\theta$
$2\theta_{max}$:	49.94°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	6961, 6491
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 6055
$N(param)_{refined}$:	564
Programs:	PARST95 [2], SHELXS-97 [3], SHELXL-97 [4], ATOMS [5]

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

Atom	Site	x	y	z	U_{iso}
H(15)	2a	0.3327	0.3049	0.8615	0.12(1)
H(16)	2a	0.2008	0.2877	0.9700	0.12(1)
H(11)	2a	0.377(6)	0.143(2)	0.653(4)	0.03(1)
H(12)	2a	0.443(9)	0.236(3)	0.726(6)	0.08(2)
H(13)	2a	0.087(8)	0.196(3)	0.983(6)	0.07(2)
H(14)	2a	0.077(7)	0.105(2)	0.895(5)	0.05(2)
H(25)	2a	0.1346	0.6129	0.4693	0.12(1)
H(26)	2a	0.2947	0.6246	0.5653	0.12(1)
H(21)	2a	0.076(8)	0.777(3)	0.228(6)	0.07(2)
H(22)	2a	0.045(8)	0.684(3)	0.323(6)	0.07(2)
H(23)	2a	0.425(6)	0.715(2)	0.569(5)	0.04(1)
H(24)	2a	0.475(6)	0.807(2)	0.472(4)	0.03(1)
H(35)	2a	0.8020	0.3284	0.0048	0.12(1)
H(36)	2a	0.9151	0.3198	0.1332	0.12(1)
H(31)	2a	0.859(8)	0.141(3)	0.187(6)	0.05(2)
H(32)	2a	0.91(1)	0.234(4)	0.219(9)	0.12(3)
H(33)	2a	0.643(8)	0.249(3)	−0.087(6)	0.07(2)
H(34)	2a	0.59(1)	0.149(4)	−0.074(8)	0.11(3)
H(45)	2a	0.7143	0.5857	0.6771	0.12(1)
H(46)	2a	0.6004	0.6032	0.7962	0.12(1)
H(41)	2a	0.883(7)	0.752(3)	0.518(5)	0.05(2)
H(42)	2a	0.844(9)	0.661(3)	0.564(6)	0.08(2)
H(43)	2a	0.578(7)	0.709(2)	0.841(5)	0.04(2)
H(44)	2a	0.636(9)	0.799(3)	0.812(7)	0.08(2)
H(1W1)	2a	0.569(4)	−0.090(4)	−0.051(8)	0.12(1)
H(2W1)	2a	0.46(1)	−0.129(2)	0.001(5)	0.12(1)
H(1W2)	2a	0.09(1)	0.011(4)	0.274(3)	0.12(1)
H(2W2)	2a	0.09(1)	0.020(3)	0.429(5)	0.12(1)
H(1W3)	2a	−0.073(3)	−0.084(4)	0.002(9)	0.12(1)
H(2W3)	2a	−0.16(1)	−0.101(4)	−0.093(4)	0.12(1)
H(1W4)	2a	0.57(1)	0.016(4)	0.508(3)	0.12(1)
H(2W4)	2a	0.61(1)	0.016(4)	0.337(2)	0.12(1)

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}
Cs(1)	2*a*	0.22170(5)	−0.00102(2)	0.05922(4)	0.0575(2)	0.0757(3)	0.0633(2)	−0.0005(2)	−0.0035(2)	0.0145(2)
Cs(2)	2*a*	0.28674(4)	0.88987(2)	0.66963(4)	0.0398(2)	0.0601(2)	0.0781(3)	−0.0016(2)	0.0054(2)	−0.0055(2)
Cs(3)	2*a*	0.71565(5)	0.01706(2)	0.70313(4)	0.0467(2)	0.0808(3)	0.0790(3)	−0.0038(2)	0.0024(2)	0.0053(2)
Cs(4)	2*a*	0.18460(7)	0.41141(3)	0.70919(4)	0.0877(3)	0.1088(4)	0.0672(3)	0.0214(3)	−0.0112(2)	−0.0152(3)
S(11)	2*a*	0.1843(2)	0.04610(7)	0.6835(1)	0.0398(7)	0.0477(9)	0.0608(8)	0.0060(6)	−0.0044(6)	0.0000(7)
O(11)	2*a*	0.0621(6)	0.0573(2)	0.5839(4)	0.063(3)	0.063(3)	0.061(2)	0.005(2)	−0.017(2)	−0.011(2)
O(12)	2*a*	0.1150(6)	0.0087(2)	0.7757(4)	0.074(3)	0.055(3)	0.079(3)	−0.004(2)	−0.008(2)	0.005(2)
O(13)	2*a*	0.3408(5)	0.0255(2)	0.6363(5)	0.049(2)	0.073(3)	0.100(3)	0.015(2)	0.001(2)	−0.029(3)
C(11)	2*a*	0.2208(6)	0.1123(2)	0.7563(5)	0.035(3)	0.046(3)	0.043(3)	0.005(2)	−0.005(2)	0.005(2)
C(12)	2*a*	0.3310(7)	0.1507(3)	0.7052(5)	0.042(3)	0.058(4)	0.037(3)	0.000(2)	0.007(2)	0.005(2)
C(13)	2*a*	0.3470(8)	0.2052(3)	0.7526(5)	0.057(3)	0.063(4)	0.045(3)	−0.011(3)	−0.002(3)	0.007(3)
C(14)	2*a*	0.2563(6)	0.2221(3)	0.8546(5)	0.038(3)	0.054(4)	0.046(3)	0.001(2)	−0.006(2)	−0.002(3)
C(15)	2*a*	0.1510(7)	0.1821(3)	0.9080(5)	0.046(3)	0.057(4)	0.041(3)	0.005(3)	0.004(2)	0.000(3)
C(16)	2*a*	0.1321(7)	0.1285(3)	0.8594(5)	0.042(3)	0.055(4)	0.047(3)	−0.004(3)	0.007(2)	0.010(3)
N(11)	2*a*	0.2641(7)	0.2774(3)	0.9002(5)	0.076(4)	0.062(4)	0.069(3)	−0.008(3)	0.000(3)	−0.013(3)
S(21)	2*a*	0.3206(2)	0.86484(7)	0.2768(1)	0.0526(8)	0.0523(9)	0.0608(9)	0.0025(7)	−0.0015(7)	0.0035(7)
O(21)	2*a*	0.1782(6)	0.8768(2)	0.1954(4)	0.068(3)	0.072(3)	0.081(3)	0.007(2)	−0.020(2)	0.016(2)
O(22)	2*a*	0.4727(6)	0.8559(2)	0.2070(5)	0.074(3)	0.073(3)	0.105(4)	0.004(3)	0.026(3)	0.017(3)
O(23)	2*a*	0.3476(7)	0.9069(2)	0.3738(5)	0.112(4)	0.051(3)	0.088(3)	−0.012(3)	−0.009(3)	−0.012(2)
C(21)	2*a*	0.2775(6)	0.8003(2)	0.3513(5)	0.038(3)	0.047(3)	0.045(3)	0.002(2)	−0.001(2)	−0.006(2)
C(22)	2*a*	0.1493(7)	0.7639(3)	0.3088(5)	0.041(3)	0.066(4)	0.043(3)	0.000(3)	−0.009(2)	−0.006(3)
C(23)	2*a*	0.1282(7)	0.7118(3)	0.3603(5)	0.044(3)	0.057(4)	0.049(3)	−0.008(3)	0.002(2)	−0.008(3)
C(24)	2*a*	0.2360(7)	0.6920(3)	0.4542(5)	0.049(3)	0.051(4)	0.042(3)	0.001(3)	0.008(2)	−0.011(3)
C(25)	2*a*	0.3610(7)	0.7288(3)	0.4991(5)	0.047(3)	0.065(4)	0.039(3)	0.005(3)	−0.002(2)	−0.006(3)
C(26)	2*a*	0.3829(7)	0.7815(3)	0.4468(5)	0.043(3)	0.048(3)	0.052(3)	−0.003(2)	−0.007(2)	−0.012(3)
N(21)	2*a*	0.2201(7)	0.6375(2)	0.5011(6)	0.074(4)	0.062(4)	0.079(4)	−0.010(3)	−0.005(3)	0.006(3)
S(31)	2*a*	0.6910(2)	0.05609(8)	0.0634(2)	0.0562(9)	0.080(1)	0.067(1)	0.0131(8)	0.0133(8)	0.0140(9)
O(31)	2*a*	0.5724(7)	0.0451(3)	−0.0404(6)	0.083(4)	0.109(5)	0.131(5)	−0.027(3)	−0.025(4)	0.014(4)
O(32)	2*a*	0.8437(7)	0.0284(3)	0.0378(9)	0.061(3)	0.110(5)	0.238(8)	0.006(3)	0.023(4)	−0.052(5)
O(33)	2*a*	0.615(1)	0.0392(3)	0.1750(6)	0.199(7)	0.098(5)	0.093(4)	0.018(5)	0.052(5)	0.032(3)
C(31)	2*a*	0.7322(7)	0.1299(3)	0.0684(5)	0.046(3)	0.082(5)	0.044(3)	0.010(3)	0.004(2)	0.006(3)
C(32)	2*a*	0.830(1)	0.1548(4)	0.1615(7)	0.058(4)	0.107(6)	0.047(3)	0.018(4)	−0.003(3)	0.015(4)
C(33)	2*a*	0.8643(9)	0.2111(4)	0.1658(6)	0.057(4)	0.112(6)	0.050(4)	−0.004(4)	−0.009(3)	−0.020(4)
C(34)	2*a*	0.8059(7)	0.2475(3)	0.0691(6)	0.044(3)	0.081(5)	0.060(4)	0.005(3)	0.005(3)	−0.011(3)
C(35)	2*a*	0.7036(8)	0.2230(3)	−0.0231(6)	0.061(4)	0.062(4)	0.053(3)	0.011(3)	0.000(3)	0.009(3)
C(36)	2*a*	0.6683(7)	0.1663(3)	−0.0232(5)	0.045(3)	0.074(4)	0.046(3)	0.004(3)	−0.003(3)	0.007(3)
N(31)	2*a*	0.8450(8)	0.3044(3)	0.0690(7)	0.089(5)	0.071(4)	0.114(5)	−0.003(4)	−0.010(4)	−0.025(4)
S(41)	2*a*	0.8212(2)	0.85741(7)	0.6466(2)	0.0379(7)	0.057(1)	0.078(1)	0.0009(7)	−0.0017(7)	0.0091(8)
O(41)	2*a*	0.9276(6)	0.8617(2)	0.5406(4)	0.078(3)	0.098(4)	0.073(3)	−0.021(3)	0.005(2)	0.028(3)
O(42)	2*a*	0.9135(6)	0.8741(2)	0.7596(5)	0.068(3)	0.070(3)	0.087(3)	−0.019(2)	0.012(2)	−0.014(2)
O(43)	2*a*	0.6658(5)	0.8883(2)	0.6331(7)	0.044(2)	0.069(3)	0.211(7)	0.001(3)	−0.005(3)	0.034(4)
C(41)	2*a*	0.7716(6)	0.7852(3)	0.6666(5)	0.038(3)	0.058(4)	0.038(3)	0.001(2)	−0.007(2)	−0.003(2)
C(42)	2*a*	0.8319(7)	0.7441(3)	0.5896(5)	0.043(3)	0.071(4)	0.043(3)	0.003(3)	0.006(2)	0.001(3)
C(43)	2*a*	0.7969(8)	0.6874(3)	0.6103(6)	0.051(3)	0.065(4)	0.061(4)	0.013(3)	0.002(3)	−0.020(3)
C(44)	2*a*	0.6993(8)	0.6710(3)	0.7069(7)	0.050(3)	0.054(4)	0.081(4)	−0.003(3)	−0.007(3)	−0.002(3)
C(45)	2*a*	0.6360(8)	0.7131(3)	0.7831(6)	0.047(3)	0.077(5)	0.054(4)	0.001(3)	0.010(3)	0.013(3)
C(46)	2*a*	0.6694(6)	0.7693(3)	0.7648(5)	0.039(3)	0.066(4)	0.035(3)	0.003(3)	0.005(2)	0.003(3)
N(41)	2*a*	0.668(1)	0.6141(3)	0.7290(8)	0.105(5)	0.058(4)	0.154(7)	−0.009(4)	0.024(5)	0.003(4)
O(1W1)	2*a*	0.4543(8)	−0.1001(3)	−0.0612(6)	0.082(3)	0.141(6)	0.096(4)	−0.005(4)	0.001(3)	0.002(4)
O(1W2)	2*a*	0.129(1)	0.0028(3)	0.3547(5)	0.158(6)	0.100(4)	0.061(3)	0.015(4)	0.001(3)	−0.014(3)
O(1W3)	2*a*	−0.1906(8)	−0.0864(3)	−0.0136(6)	0.115(4)	0.107(5)	0.081(4)	0.016(4)	0.013(3)	0.001(3)
O(1W4)	2*a*	0.6031(9)	0.0123(5)	0.4240(6)	0.113(5)	0.27(1)	0.072(4)	−0.041(6)	−0.008(3)	0.043(5)

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