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Redetermination of the Structure of Caesium Hydrogensulfate

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Abstract. CsHSO₄, $M_r = 229.98$, monoclinic, $P2_1/c$, $a = 8.214$ (2), $b = 5.809$ (2), $c = 10.984$ (2) Å, $\beta = 119.39$ (2)°, $V = 456.7$ (2) Å³, $Z = 4$, $D_x = 3.346$ Mg m⁻³, $\lambda(\text{Mo } K\alpha) = 0.71069$ Å, $\mu(\text{Mo } K\alpha) = 8.318$ mm⁻¹, $F(000) = 416$, $R = 0.057$, $wR = 0.062$, 2172 unique reflections, $T = 296$ K (low-temperature phase). The structure deviates slightly from the previously reported structure with space group $P2_1/m$, and $Z = 2$. The unit cell is composed of two pseudocells. Two kinds of similar hydrogen-bonded chains, in which hydrogens are located opposite each other with respect to the center of the hydrogen bond, run along the b axis one dimensionally.

Introduction. It is known that many compounds with the formula $AHBO_4$ ($A =$ monovalent atoms or groups, $B =$ S, Se) exhibit the following properties: (1) they undergo (successive) phase transitions, (2) a good number of them display superionic character in

the high-temperature phase, (3) the phase transition is often irreversible, and (4) moisture sometimes affects the character of the phase transition. CsHSO₄, a typical compound of this type, undergoes successive phase transitions from a low-temperature phase to an intermediate phase in the temperature range from 333 to 370 K on heating (Belushkin, Natkaniec, Pakida, Shuvalov & Wasicki, 1987), and then to a superionic high-temperature phase at $T_i = 410$ –414 K (Komukae, Osaka, Makita, Ozaki, Itoh & Nakamura, 1981; Baranov, Fedosyuk, Schagina & Shuvalov, 1984). The low-temperature phase, however, does not appear on cooling the high-temperature phase (Belushkin *et al.*, 1987). The X-ray structure analysis has been carried out at room temperature on the low-temperature phase (Itoh, Ozaki & Nakamura, 1981). According to the aforementioned work, the space group of CsHSO₄ is $P2_1/m$ with unit-cell parameters $a = 7.304$, $b = 5.810$,

$c = 5.491 \text{ \AA}$, $\beta = 101.5^\circ$ and $Z = 2$. After this study, Balagurov, Belushkin, Beskrovnyi, Vratslav, Wasicki, Dutt, Dlouha, Jirak, Natkaniec, Savenko & Shuvalov (1985) reported from their neutron diffraction study that the space group is $P2_1/c$ and the unit cell is twice as large along the c axis. The reported unit-cell parameters are $a = 8.223$, $b = 5.814$, $c = 10.99 \text{ \AA}$, $\beta = 119.4^\circ$ and $Z = 4$. Following this result, we have redetermined the structure of CsHSO_4 at room temperature on the basis of newly measured reflection data.

Experimental. The compound was prepared by slowly cooling an aqueous solution containing equimolar amounts of Cs_2SO_4 and H_2SO_4 . An untwinned spherical specimen, 0.26 mm in diameter, was mounted on a Rigaku AFC-5 automatic four-circle diffractometer; graphite-monochromated $\text{Mo K}\alpha$ radiation was used. Measurements were carried out by the θ - 2θ scanning method with a scan speed of 8° min^{-1} in 2θ . Lattice parameters were determined by least-squares refinement of 24 reflections ranging from $2\theta = 23.7$ to 26.5° . An absorption correction was made in view of the considerable absorption effect ($\mu r = 1.08$). Maximum value of $\sin\theta/\lambda$ reached 1.0 \AA^{-1} in intensity measurements ($2\theta \leq 90^\circ$). 417 reflections in a quadrant with Miller indices $-17 \leq h \leq 17$, $0 \leq k \leq 12$, $0 \leq l \leq 22$ were collected; of these, 3185 were observed with $|F_o| > \sigma(|F_o|)$. The intensities of the 656, 1,1,10 and 544 reflections were monitored after every 200 measurements and showed variations less than 3.6%. The space group is $P2_1/c$ from the systematic extinctions. The positions of the Cs and S atoms were found from a three-dimensional Patterson map and those of the O and H atoms were found on a difference Fourier map phased with the heavier atoms at respective stages with the use of the Fourier synthesis program *UNICS* (Sakurai, 1967). Complex neutral atomic scattering factors for Cs, S and O, and the neutral atomic scattering factor for H were taken from *International Tables for X-ray Crystallography* (1974). The function minimized was $\sum w(|F_o| - |F_c|)^2$ with $w = 1/\sigma^2(|F_o|)$ by the full-matrix least-squares program *RADIEL* (Coppens, Guru Row, Leung, Stevens, Becker & Yang, 1979). The final discrepancy factors were $R = 0.057$ and $wR = 0.062$ and a goodness of fit $S = 1.76$ for 2172 unique reflections. The isotropic secondary-extinction coefficient is $g = 11.6(5) \times 10^{-4}$. The smallest extinction factor in the reflections used is $\gamma = 0.18$ for $\bar{1}02$. $(\Delta/\sigma)_{\text{max}} = 0.003$. The total number of parameters, including a scale parameter and the extinction coefficient, was 61. The final residual electron density showed an irregular maximum of $\Delta\rho = 3.1 \text{ e \AA}^{-3}$ near Cs, maxima of 1.4 e \AA^{-3} midway between Cs and S, 1.2 e \AA^{-3} near S and O, and peaks less than

Table 1. Fractional positional parameters and thermal parameters [$U_{\text{eq}} (\times 10^2)$ for Cs, S and O; $U_{\text{iso}} (\times 10^2)$ for H], with *e.s.d.*'s in parentheses

The equivalent isotropic thermal parameters, U_{eq} , have been calculated from $U_{\text{eq}} = (U_{11} + U_{22} + U_{33} + 2U_{13}\cos\beta)/3$.

	<i>x</i>	<i>y</i>	<i>z</i>	U_{eq} or U_{iso} (\AA^2)
Cs	0.28808 (4)	0.74303 (5)	0.15233 (3)	2.34 (1)
S	0.2459 (2)	0.2359 (2)	0.3974 (1)	1.84 (3)
O(1)	0.3008 (8)	0.2480 (6)	0.2917 (6)	3.2 (1)
O(2)	0.3929 (8)	0.2404 (6)	0.5392 (5)	3.4 (1)
O(3)	0.1158 (7)	0.0444 (8)	0.3707 (5)	3.3 (1)
O(4)	0.1304 (7)	0.4599 (8)	0.3820 (5)	3.4 (2)
$\text{H}^{0.3-}$	0.026 (7)	0.494 (7)	0.276 (6)	0.5 (17)*

* The neutral H model gives a negative thermal parameter. We refined the charge of H so as to minimize wR leading to the charged model of $\text{H}^{0.3-}$. It seems, however, that the U_{iso} is still very small.

Table 2. Atomic distances (\AA) and angles ($^\circ$), with *e.s.d.*'s in parentheses

Cs—O(1)	3.235 (5)	O(1)—O(2 ⁱⁱⁱ)	3.639 (6)
—O(1 ⁱ)	3.122 (7)	—O(3)	2.399 (10)
—O(2 ⁱⁱ)	3.350 (5)	—O(3 ^{iv})	3.444 (7)
—O(2 ⁱⁱⁱ)	3.105 (4)	—O(4)	2.412 (10)
—O(3 ⁱⁱ)	3.172 (5)	—O(4 ^{viii})	3.515 (7)
—O(3 ^{iv})	3.402 (6)	O(2)—O(2 ⁱⁱⁱ)	3.798 (7)
—O(3 ^v)	3.763 (7)	—O(2 ⁱⁱⁱ)	3.623 (8)
—O(4)	3.736 (7)	—O(3)	2.402 (6)
—O(4 ^v)	3.503 (6)	—O(4)	2.367 (6)
—O(4 ^{vii})	3.114 (5)	O(3)—C(3 ^v)	3.760 (6)
S—O(1)	1.439 (8)	—O(4)	2.416 (6)
—O(2)	1.428 (8)	—O(4 ^{iv})	3.398 (6)
—O(3)	1.469 (5)	—O(4 ^{viii})	2.555 (6)
—O(4)	1.570 (5)	—H ^{viii}	1.479 (47)
O(1)—O(2)	2.438 (8)	O(4)—O(4 ^v)	3.917 (6)
—O(2 ⁱⁱ)	3.214 (10)	O(4)—H	1.075 (47)
—O(2 ⁱⁱⁱ)	3.745 (6)		
O(1)—S—O(2)	116.6 (4)	O(2)—S—O(4)	104.2 (3)
O(1)—S—O(3)	111.2 (3)	O(3)—S—O(4)	105.3 (3)
O(1)—S—O(4)	106.5 (3)	O(3)—H ^{viii} —O(4 ^{viii})	179.2 (41)
O(2)—S—O(3)	112.0 (3)		

Symmetry code: (i) $1 - x, 0.5 + y, 0.5 - z$; (ii) $x, 0.5 - y, z - 0.5$; (iii) $1 - x, 1 - y, 1 - z$; (iv) $-x, 0.5 + y, 0.5 - z$; (v) $x, 1 + y, z$; (vi) $x, 1.5 - y, z - 0.5$; (vii) $1 - x, -y, 1 - z$; (viii) $-x, y - 0.5, 0.5 - z$; (ix) $x, y - 1, z$.

0.6 e \AA^{-3} elsewhere. The final positional and thermal parameters are given in Table 1.*

Discussion. The bond distances and angles are shown in Table 2. The present lattice vectors $\mathbf{a}_n, \mathbf{b}_n, \mathbf{c}_n$ are related to the previous ones $\mathbf{a}_o, \mathbf{b}_o, \mathbf{c}_o$ (Itoh *et al.*, 1981) by $\mathbf{a}_n = \mathbf{a}_o + \mathbf{c}_o$, $\mathbf{b}_n = -\mathbf{b}_o$ and $\mathbf{c}_n = -2\mathbf{c}_o$. It turns out that intrinsic reflections were neglected in the previous study through a mistake at the stage of

* Lists of structure factors and anisotropic atomic displacement parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 52349 (9 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

peak hunting, together with several unindexed weak reflections, which are observed again for some of the prepared specimens in the present study. It seems that the appearance of these undesirable reflections depends on the conditions with respect to the presence of water during sample preparation in accordance with the remarks on the effect of moisture (Belushkin *et al.*, 1987; Baranowski, Friesel & Lundén, 1989). In this study, we used a specimen from which no such parasitic reflections could be detected.

It is easily understood from Fig. 1 that the unit cell is composed of two pseudo-unit cells resulting in the slightly modulated form of the previously reported structure, which corresponds to the average structure of two pseudo-unit cells. Two kinds of the hydrogen-bonded chains which are related to each other by symmetry elements run along the *b* axis one dimensionally. It turns out that the low-temperature structure of CsHSO_4 has no analogue among the AHBO_4 -type structures, *i.e.* CsDSO_4 (Merinov, Baranov, Maksimov & Shuvalov, 1986), CsHSeO_4 (Baran & Lis, 1987), $\text{CsH}_{1-x}\text{D}_x\text{SO}_4$ ($x=0.67$) and $\text{CsH}_{1-x}\text{D}_x\text{SeO}_4$ ($x=0.70$) (Balagurov, Beskrovnyi, Savenko, Berinov, Dlouha, Vratislav & Jirak, 1987) together with RbHSO_4 (Ashmore & Petch, 1975), NH_4HSO_4 (Nelmes, 1972), NaHSO_4 and KHSO_4 (Sonneveld & Visser, 1978), and is rather similar to the paraelectric structure of CsH_2PO_4 (Matsunaga, Itoh & Nakamura, 1980) apart from an H atom

which combines O(1) and O(2) in the case of CsH_2PO_4 . Although the shape of SO_4 is fairly regular, we find that the bond lengths S—O(3) and S—O(4) are a little longer than S—O(1) and S—O(2) as shown in Table 2. This is due to the formation of the hydrogen bond between O(3) and O(4).

It is quite natural to consider from this study that the double-peaked electron density at the position of H found in the previous study results not from a disordering of H but from a consequence of the averaging of two pseudo-unit cells. It follows, therefore, that the phase transition associated with an ordering of H, which is predicted in the previous study, should be rejected. In accordance with this conclusion of the electron density distribution, it has been confirmed by the dielectric measurements that no phase transition takes place down to 4 K from room temperature (Döbler & Happ, 1987).

In conclusion, it is worth paying attention to the following characteristics of the low-temperature phase of CsHSO_4 . Firstly, this phase never appears on cooling from the high-temperature phase as mentioned above (Belushkin *et al.*, 1987). Secondly, the transition temperature to this phase decreases strongly with the degree of deuterization and no phase transition takes place for the highly deuterated salt (Balagurov *et al.*, 1987). These results imply that the low-temperature phase of CsHSO_4 is the metastable phase corresponding to the fact that this structure has no analogue among the other AHBO_4 -type structures as far as we know.

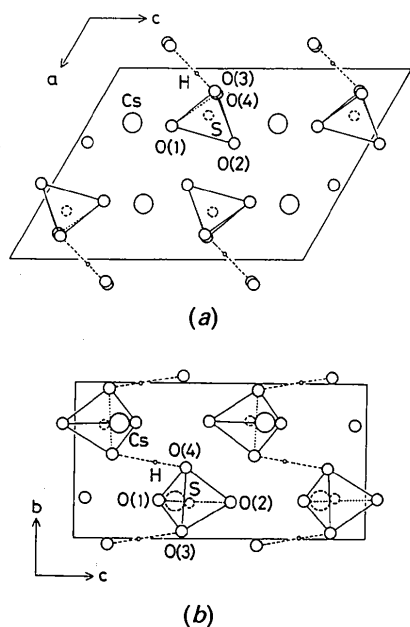


Fig. 1. (a) The *b*-axis and (b) *a*-axis views of the CsHSO_4 structure. The hydrogen bonds are drawn with broken lines.

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Hydrogen Bonding in Barium Hydroxide Trihydrate by Neutron Diffraction

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Abstract. $\text{Ba}(\text{OH})_2 \cdot 3\text{H}_2\text{O}$, $M_r = 225.40$, orthorhombic, $Pnma$, $a = 7.640$ (2), $b = 11.403$ (5), $c = 5.965$ (1) Å, $V = 519.7$ (5) Å³, $Z = 4$, $D_x = 2.88$ g cm⁻³, neutron radiation, $\lambda = 0.8495$ Å, $\mu(\text{calc.}) = 2.14$ cm⁻¹, $F(000) = 17.39$, $T = 295$ K, $R = 0.044$ for 558 observed independent reflections. The water molecules exhibit strong hydrogen bonds to the hydroxide ion with H...O distances of 1.696 (3) Å (H₂O I) and 1.667 (3) and 1.672 (2) Å (H₂O II). The hydroxide ions are not hydrogen-bonded. The OH bond lengths are 0.99–1.01 Å (H₂O) and 0.948 (3) Å (OH⁻); the HOH angles are 102.0 (2)° (H₂O I) and 107.6 (2)° (H₂O II).

Introduction. As part of our structural and spectroscopic studies on alkaline-earth hydroxides (Buchmeier & Lutz, 1986; Kuske, Engelen, Henning, Lutz, Fuess & Gregson, 1988; Lutz, Eckers, Schneider & Haeuseler, 1981; Lutz, Henning & Haeuseler, 1987; Lutz, Kuske & Henning, 1988) we performed a neutron diffraction experiment on $\text{Ba}(\text{OH})_2 \cdot 3\text{H}_2\text{O}$ in order to gain more insight into the hydrogen-bond system of this compound than is available by X-ray structure determination (Buchmeier & Lutz, 1986) and to correlate previous IR and Raman spectroscopic results (Lutz *et al.*, 1981; Lutz & Lange, 1989) with structural data.

Experimental. Single crystals of $\text{Ba}(\text{OH})_2 \cdot 3\text{H}_2\text{O}$ (prisms) were prepared as described by Bauer (1903). A suitable crystal (0.50 × 0.71 × 2.15 mm, $V = 0.51$ mm³) was mounted in a sealed thin-walled quartz capillary on an Enraf–Nonius CAD-4 diffractometer to obtain the orientation matrix. Neutron data were collected at the ILL, Grenoble, France,

using the D9 four-circle diffractometer with a beryllium (110) monochromator. The intensities of 1030 reflections, 840 unique, were collected in two shells with the step-scan method [$2\theta \leq 30^\circ$, ω - $x\theta$ scans with $x \leq 2$, $(\sin\theta)/\lambda < 0.305$ Å⁻¹, range of hkl : $0 \leq h \leq 6$, $-8 \leq k \leq 8$, $0 \leq l \leq 5$; $30 \leq 2\theta \leq 76^\circ$, ω - 2θ scans, $0.305 \leq (\sin\theta)/\lambda \leq 0.725$ Å⁻¹, range of hkl : $0 \leq h \leq 12$, $0 \leq k \leq 18$, $0 \leq l \leq 10$]. An intensity decrease of the standard reflection (060) was not observed during measuring time. Data reduction was performed according to Lehmann & Larsen (1974) with the program *COLL5N*. Merging of symmetry-equivalent reflections of the inner shell gave an internal $R = 0.0067$. 282 reflections with $I < 4.5\sigma_I$ (σ_I from counting statistics) were considered unobserved. A numerical absorption correction did not improve the results. The Ba and O positions from X-ray data (Buchmeier & Lutz, 1986) were taken as starting values. Scattering lengths for all atoms were taken from Sears (1986): $b_{\text{Ba}} = 5.25$ (4), $b_{\text{O}} = 5.803$ (4), $b_{\text{H}} = -3.739$ (1) fm. Full-matrix least-squares refinement with a neutron-diffraction-adapted *SHELX76* program (Sheldrick, 1976) of positional and anisotropic thermal parameters, extinction coefficient, error weight, and scale factor based on the F^2 magnitudes of 558 reflections gave a final $R = 0.044$, $wR = 0.024$ [$w = 1.068/\sigma^2(F)$], and an extinction coefficient of 9.29×10^{-7} . The ratio of maximum least-squares shift to e.s.d. in the final cycle was 0.002. The final atomic coordinates are given in Table 1, anisotropic thermal parameters in Table 2.†

† Lists of structure factors have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 52293 (5 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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