

Strontium Dihydrogen Diphthalate Dihydrate

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Abstract. $\text{Sr}(\text{C}_8\text{H}_5\text{O}_4)_2 \cdot 2\text{H}_2\text{O}$, $M_r = 453.9$, orthorhombic, $Cmc2_1$, $Z = 4$, $a = 28.436$ (3), $b = 8.768$ (1), $c = 6.961$ (1) Å, $V = 1735.6$ (4) Å³, $D_c = 1.737$ g cm⁻³; single-crystal diffractometer data to $\sin \theta/\lambda = 0.70$ Å⁻¹; Nb-filtered Mo $K\alpha$ radiation. The un-ionized and ionized carboxyl groups make angles of 28 and 71°, respectively, with the plane of the benzene ring. The Sr atom has a ninefold coordination of O atoms.

Introduction. Single crystals of $\text{Sr}(\text{C}_8\text{H}_5\text{O}_4)_2 \cdot 2\text{H}_2\text{O}$ were obtained from an aqueous solution of SrCO_3 and an excess of phthalic acid. A specimen with dimensions 0.36 × 0.40 × 0.50 mm was selected for data collection. Precession photographs confirmed the lattice constants given by Cingi (1959). The systematic extinctions, however, corresponded to the possible space groups $Cmc2_1$, $C2cm$ and $Cmcm$. In the course of the structure determination $Cmc2_1$ was found to be correct.

Data were collected on a computer-controlled Hilger & Watts diffractometer, in two octants of reciprocal space, to $\sin \theta/\lambda = 0.70$ Å⁻¹, yielding 2574 reflections (1388 of which were unique). Background corrections were made as described by Bartl & Schuckmann (1966). No absorption correction was applied. The related reflections were averaged. A weighting scheme, $w(I) = [\sigma^2(I)_{\text{counting}} + (0.03I)^2]^{-1}$, was used in the refinement.

The structure was determined by the Patterson method. Atomic scattering factors were from *International Tables for X-ray Crystallography* (1974), except for H for which the factors given by Stewart, Davidson & Simpson (1965) were used. The imaginary part of the dispersion factor for Sr was not included in the calculation.

Careful inspection of difference Fourier syntheses yielded positional parameters for all H atoms. They were included in the refinement but their isotropic thermal parameters were not varied. Extinction effects were found to be negligible. Refinement converged at $R(F) = 0.020$ and $R_w(F) = 0.027$.

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Table 1. Positional parameters

	x	y	z
Sr	0.0	0.60831 (3)	0.5
C(1)	0.11582 (8)	0.0821 (2)	0.4950 (9)
C(2)	0.14909 (8)	0.2097 (3)	0.4560 (3)
C(3)	0.19476 (8)	0.2019 (3)	0.5276 (5)
C(4)	0.22618 (7)	0.3202 (3)	0.4940 (8)
C(5)	0.21149 (9)	0.4481 (3)	0.3962 (5)
C(6)	0.16584 (10)	0.4593 (3)	0.3286 (4)
C(7)	0.13417 (8)	0.3395 (3)	0.3550 (4)
C(8)	0.08757 (7)	0.3511 (3)	0.2529 (4)
O(1)	0.07367 (6)	0.1025 (2)	0.5069 (10)
O(2)	0.13653 (7)	-0.0493 (2)	0.5154 (6)
O(3)	0.05803 (6)	0.4467 (2)	0.3045 (3)
O(4)	0.08210 (7)	0.2693 (2)	0.1056 (3)
O(5)	0.0	0.3114 (3)	0.6315 (4)
O(6)	0.0	0.8920 (3)	0.5005 (26)
H(1)	0.204 (1)	0.121 (3)	0.631 (7)
H(2)	0.257 (1)	0.311 (4)	0.549 (5)
H(3)	0.231 (1)	0.539 (4)	0.367 (6)
H(4)	0.161 (1)	0.560 (4)	0.253 (6)
H(5)	0.119 (1)	-0.118 (4)	0.545 (11)
H(6)	0.020 (1)	0.239 (4)	0.571 (6)
H(7)	0.022 (1)	0.951 (3)	0.480 (8)

Table 2. Bond lengths (Å) and angles (°)

C(1)–C(2)	1.488 (3)	O(2)–H(5)	0.81 (4)
C(2)–C(3)	1.394 (4)	O(5)–H(6)	0.96 (4)
C(3)–C(4)	1.391 (4)	O(6)–H(7)	0.82 (3)
C(4)–C(5)	1.381 (5)		
C(5)–C(6)	1.378 (4)	Sr–O(6)	2.488 (3)
C(6)–C(7)	1.403 (4)	2× Sr–O(3)	2.566 (2)
C(2)–C(7)	1.403 (4)	Sr–O(5)	2.659 (3)
C(7)–C(8)	1.506 (4)	2× Sr–O(4)	2.672 (2)
C(1)–O(1)	1.221 (3)	2× Sr–O(3')	2.730 (2)
C(1)–O(2)	1.297 (3)	Sr–O(5')	2.762 (3)
C(8)–O(3)	1.237 (3)		
C(8)–O(4)	1.260 (4)		
C(2)–C(1)–O(1)	121.8 (2)	C(6)–C(7)–C(2)	118.4 (2)
C(2)–C(1)–O(2)	113.7 (2)	C(6)–C(7)–C(8)	117.1 (2)
O(1)–C(1)–O(2)	124.5 (2)	C(2)–C(7)–C(8)	124.1 (2)
C(1)–C(2)–C(3)	119.1 (3)	C(7)–C(8)–O(3)	120.4 (2)
C(1)–C(2)–C(7)	120.6 (2)	C(7)–C(8)–O(4)	116.8 (2)
C(3)–C(2)–C(7)	120.2 (2)	O(3)–C(8)–O(4)	122.6 (2)
C(2)–C(3)–C(4)	120.1 (3)	C(1)–O(2)–H(5)	114 (2)
C(3)–C(4)–C(5)	119.8 (3)	H(6)–O(5)–H(6')	75 (3)
C(4)–C(5)–C(6)	120.5 (3)	H(7)–O(6)–H(7')	99 (3)
C(5)–C(6)–C(7)	120.9 (3)		



Fig. 1. Stereoscopic view of the hydrogen phthalate group. The thermal ellipsoids are the 50% probability surfaces.

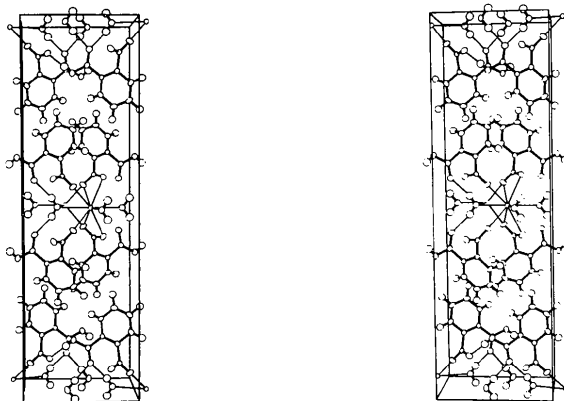


Fig. 2. Stereoscopic view of the molecular packing.

All calculations were carried out with the XRAY system (Stewart, Kruger, Ammon, Dickinson & Hall, 1972) on the Univac 1108 computer of the University of Frankfurt. Atomic coordinates are reported in Table 1, bond lengths and angles in Table 2.* Stereoscopic views of the hydrogen phthalate group and the molecular packing are shown in Figs. 1 and 2.

Discussion. The present work is part of a study of the hydrogen bonding in acid phthalates. $\text{Li}(\text{C}_8\text{H}_5\text{O}_4) \cdot 2\text{H}_2\text{O}$ (Gonschorek & Küppers, 1975) and $\text{Cu}(\text{C}_8\text{H}_5\text{O}_4)_2 \cdot 2\text{H}_2\text{O}$ (Cingi, Guastini, Musatti & Nardelli, 1969; Bartl & Küppers, 1978) were found to contain a short intramolecular hydrogen bond. No other hydrogen phthalate structures have so far been found which contain this feature.

The hydrogen phthalate proton in the title compound is involved in a relatively strong intermolecular hydro-

* Lists of structure factors and thermal parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 33548 (11 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 3. *Hydrogen bonds*

O—H...O	O...O (Å)	H...O (Å)	∠O—H—O (°)
O(2)—H(5)...O(4)	2.554 (3)	1.74 (4)	178 (4)
O(5)—H(6)...O(1)	2.911 (4)	1.98 (3)	164 (3)
O(6)—H(7)...O(1)	2.791 (3)	1.99 (3)	165 (6)

gen bond (Table 3). However, no short intramolecular hydrogen bond exists. Two additional hydrogen bonds are found and involve the water molecules. They can be classified as weak.

The benzene ring is planar and has an average C—C distance of 1.392 Å. The average C—H distance and C—C—H angle are 1.01 Å and 120° respectively. The groups [C(2),C(1),O(1),O(2)] and [C(7),C(8),O(3),O(4)] are both planar, making angles of 28 and 71°, respectively, with the plane of the benzene ring. The angle between them is 65°.

The Sr atom has a ninefold coordination of O atoms, three from water molecules and six from phthalate groups. O(6) has an exceptionally large thermal parameter in the *z* direction ($U_{33} = 0.244 \text{ Å}^2$), indicating possible disorder. This water molecule is involved in one Sr—O bond and two hydrogen bonds; all are approximately located in a plane with $z = 0.5$, allowing large thermal motion or disorder in the direction normal to this plane. *Via* the O(6)—H(7)...O(1) hydrogen bond, this effect is partly reflected in the thermal parameters of O(1), and is also apparent for O(2). Refinement with a statistical distribution of O(6) indicated disorder of this atom to be significant. However, because of large correlations, only the average position of O(6) is reported here; the disorder was found to have a minimal effect on the intermolecular distances.

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