

Table 5. Bond distances in the $\text{Cu}(\text{II})(\text{en})_2 \cdot \text{H}_2\text{O}$ cation, corrected for thermal motion

For C-C bonds the motion of the copper atom has been subtracted from the carbon atoms.

Bond	Uncorrected	Lower limit	Riding	Rigid body	Upper limit
Cu(3)-N(5)	2.004 Å	2.005 Å	2.015 Å	2.014 Å	
Cu(3)-N(6)	2.034	2.034	2.036	2.042	
Cu(3)-N(7)	2.004	2.006	2.017	2.012	
Cu(3)-N(8)	2.017	2.018	2.026	2.027	
Cu(3)-O	2.441	2.442	2.449	2.450	
C(5)-N(5)	1.454	1.456	1.463	1.458	
C(6)-N(6)	1.469	1.478	1.486	1.475	
C(7)-N(7)	1.424	1.445	1.465	1.429	
C(8)-N(8)	1.448	1.469	1.499	1.452	
C(5)-C(6)	1.488	1.491		1.492	1.528 Å
C(7)-C(8)	1.408	1.408		1.411	1.586

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The Crystal Structure of Tin(II) Sulphate

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(Received 13 July 1971)

Tin(II) sulphate, SnSO_4 , crystallizes in the orthorhombic system, space group $Pnma$, with four formula units in a cell with $a=8.799$, $b=5.319$, $c=7.115$ Å. The intensities of 418 independent reflexions were obtained by counter methods and the structure refined by least-squares methods to a conventional R value of 4.4%. The structure consists of a framework of sulphate groups linked by O-Sn-O bridges. The tin(II) atoms have typical pyramidal three coordination with Sn-O bond distances of 2.27, 2.27 and 2.25 Å and O-Sn-O angles of 79.0, 77.1 and 77.1°.

Introduction

Rentzeperis (1962) described a crystal-structure determination of SnSO_4 on the basis of measurement of the intensities of 58 powder reflexions. This structure was based on the assumption that SnSO_4 had the barite structure (James & Wood, 1925) although it had previously been suggested by Donaldson & Moser (1960) that this was unlikely in view of the large difference in the ionic radii of Ba^{2+} and Sn^{2+} . This SnSO_4 structure, along with the other known tin(II) structures (Donaldson, 1967), has been used in the

interpretation of ^{119}Sn Mössbauer data. (Donaldson & Senior, 1969) It is however important to have a more accurate knowledge of the details of the environment of the tin atoms in SnSO_4 and for this reason we have carried out a full single-crystal three-dimensional structure analysis on the material.

Experimental

The crystals of SnSO_4 which were prepared by Donaldson & Moser's (1960) method are not hygroscopic as previously reported.

Crystal data

(Rentzeperis, 1962; Donaldson & Moser, 1960). Tin(II) sulphate, SnSO₄, M.W. 214.8, orthorhombic, $a=8.799 \pm 0.001$, $b=5.319 \pm 0.001$, $c=7.115 \pm 0.001$ Å, $V=333.0$ Å³, $D_c=4.185$ g.cm⁻³ for $Z=4$, $D_m=4.21$ g.cm⁻³. Total number of electrons per unit cell, $F(000)=392$. Systematic absences; $0kl$ for $k+l=2n+1$, $hk0$ for $h=2n+1$. Possible space groups; $Pnma$ or $Pn2_1a$.

The lower symmetry group ($Pn2_1a$) was at first assumed but least-squares refinement of the atomic positions showed convergence towards the centrosymmetric space-group positions, which were then used for the final refinement.

Structure determination

Data were collected from a small acicular crystal (diameter 0.133 mm) mounted along the b axis on a Hilger-Watts Y-190 single-crystal linear diffractometer using Mo $K\alpha$ radiation. Equivalent reflexions from four octants were collected to give 418 independent reflexions of above background intensity. Lorentz and polarization corrections were applied but no allowance was made for the small effects of absorption ($\mu R=0.517$) or extinction.

The positions of the Sn and S atoms were determined from a three-dimensional Patterson synthesis assuming the lower-symmetry space group $Pn2_1a$. The positions of the O atoms were obtained from a difference Fourier synthesis calculated using the Sn and S atom positions.

Full-matrix least-squares refinement of layer scale factors, atomic positions and isotropic temperature factors indicated convergence towards the positions corresponding to the centrosymmetric space group, which was then used for the final refinement. Refinement of layer scales, atomic positions and anisotropic temperature factors converged with $R = \sum ||F_o| - |F_c|| / (\sum |F_o|)$ as 0.044 using unit weights throughout. Atomic scattering factors for neutral Sn, S and O atoms were taken from *International Tables for X-ray Crystallography* (1962). A final difference Fourier map showed peaks of -18 and $+6$ as the greatest negative and positive deviations relative to $+999$ as the peak corresponding to the heaviest atom present (Sn, 50 electrons). The final observed and calculated structure factors are listed in Table 1.

The final atomic coordinates, temperature factors and their standard deviations are given in Table 2. The significant bond distances and angles based on the coordinates of Table 2 are listed in Table 3 along with their standard deviations.

The pair of diagrams in Fig. 1 provide a stereoscopic view of the unit cell as seen from a direction perpendicular to the (253) plane. Fig. 2 shows the environment of the tin atom in SnSO₄.

The computer programs used were (i) *NUCLS4*, a version of the Ibers and Doedens least-squares program, (ii) *FORDAP*, the Zalkin Fourier program, (iii) *ORFFE*, Busing and Levy's function and error pro-

gram and (iv) *CELLPLOT*, an Algol program written for the Elliott 503 computer and graph plotter. This draws ordinary or stereoscopic views of a unit cell

Table 1. Observed and calculated structure factors ($\times 10$) for SnSO₄

K=0				K=1				K=3				K=4			
H	L	F(O)	F(C)	H	L	F(O)	F(C)	H	L	F(O)	F(C)	H	L	F(O)	F(C)
0	2	1688	1992	5	6	700	717	2	1	1121	1201	10	1	436	400
0	4	947	881	5	7	181	197	2	2	573	548	10	2	391	392
0	8	331	345	6	0	1202	1219	2	3	806	772	10	3	263	274
1	1	351	286	6	1	328	294	2	4	380	369	10	4	263	273
1	2	735	654	6	2	926	908	2	5	213	217				
1	3	123	106	6	4	652	646	3	1	219	186				
1	4	905	839	6	5	147	147	3	2	322	316				
1	5	288	260	7	2	703	704	3	3	839	807	H	L	F(O)	F(C)
1	6	964	956	7	3	148	143	3	4	423	411				
1	7	213	267	7	4	741	744	3	5	839	830	0	3	593	587
1	8	479	470	7	5	101	97	3	6	518	524	0	5	441	403
1	9	267	260	7	6	691	705	3	7	481	478	0	7	290	290
2	0	1121	1163	8	0	953	946	4	0	895	890	1	2	263	254
2	2	912	874	8	1	157	142	4	1	611	595	1	3	443	430
2	3	201	211	8	2	777	774	4	2	557	526	1	4	235	212
2	4	933	870	8	3	337	339	4	3	384	378	1	5	454	439
2	5	348	360	8	4	440	431	4	4	559	559	1	6	161	158
2	6	343	317	8	5	243	237	4	5	105	106	1	7	482	444
2	7	436	429	9	2	154	157	4	6	380	382	1	9	390	392
3	1	146	130	9	3	104	93	5	2	126	116	2	1	785	844
3	2	667	583	9	4	306	315	5	4	274	287	2	2	533	517
3	3	327	327	10	0	220	226	5	5	508	549	2	3	379	353
3	4	490	440	10	1	458	455	5	7	157	171	2	4	272	243
3	5	671	618	10	2	188	185	6	0	1045	1060	2	5	157	145
3	6	647	638	10	3	414	418	6	1	187	190	2	6	184	174
3	7	577	566					6	2	800	800	3	1	126	109
3	8	579	588					6	3	155	155	3	2	347	340
4	0	300	281					6	5	131	125	3	3	487	467
4	1	1478	1551	H	L	F(O)	F(C)	6	6	117	109	3	5	510	502
4	2	332	320					7	2	572	587	3	6	350	344
4	3	1242	1213	0	2	1055	1024	7	3	180	180	3	7	300	303
4	4	410	380	0	4	627	623	7	4	635	647	3	8	240	231
4	5	226	195	0	6	86	93	7	5	139	135	4	0	452	470
4	6	212	209	0	8	135	174	7	6	588	614	4	1	556	543
5	1	236	219	1	1	438	402	8	0	741	751	4	2	307	302
5	2	289	299	1	2	879	819	8	2	609	618	4	3	339	328
5	3	838	794	1	3	358	314	8	3	254	265	4	4	387	389
5	4	531	507	1	4	1050	1037	8	4	352	352	4	6	323	311
5	5	660	631	1	5	172	88	8	5	213	214	5	2	161	155
5	6	351	345	1	6	1000	1034	9	2	165	165	5	4	290	286
5	7	548	571	1	7	94	95	9	4	295	308	5	6	484	466
6	0	681	637	1	8	466	509	10	0	165	165	6	3	376	371
6	1	1202	1173	1	9	174	208	10	1	423	427	6	5	339	349
6	2	431	379	2	0	1145	1204	10	2	148	144	6	8	646	653
6	3	909	880	2	1	844	793	10	3	364	378	6	1	233	229
6	4	106	125	2	2	944	928					6	2	510	506
6	5	681	637	2	3	391	374					6	3	371	371
6	6	257	208	2	4	841	807					7	2	392	428
6	7	163	136	2	5	339	327	H	L	F(O)	F(C)	7	4	480	485
7	2	138	126	2	6	289	277					7	6	459	457
7	3	315	306	2	7	269	269	0	2	1077	1115	8	0	618	640
7	5	508	519	3	2	165	137	0	4	617	560	8	4	507	516
7	6	212	178	3	3	550	513	0	8	244	262	8	3	193	209
7	7	550	553	3	5	456	444	1	1	124	116	8	4	279	270
8	0	698	652	3	6	296	338	1	2	457	424	8	5	150	154
8	1	515	508	3	7	438	465	1	4	650	600	8	6	168	160
8	2	358	330	4	0	898	771	1	5	714	117	8	7	292	253
8	3	436	435	4	1	1043	1055	1	6	655	670	10	1	249	254
8	4	262	209	4	2	670	644	1	7	191	189	10	3	235	252
8	5	301	319	4	3	896	909	1	8	344	353				
9	1	236	273	4	4	439	429	1	9	200	207				
9	2	277	267	4	5	175	165	2	0	661	682				
9	3	486	489	4	6	129	122	2	1	136	113	H	L	F(O)	F(C)
9	4	420	425	5	1	353	348	2	2	573	561				
9	5	521	523	5	2	232	225	2	3	189	179	0	2	476	564
9	6	726	726	5	3	959	1023	2	4	565	582	0	3	376	389
10	1	540	516	5	4	436	435	2	5	255	261	2	4	294	354
10	2	538	532	5	5	836	853	2	6	267	239	1	3	154	145
10	3	358	367	5	6	298	307	2	7	300	311	1	4	526	503
11	1	174	141	5	7	646	669	3	1	102	94	1	6	532	534
				5	8	391	374	3	2	257	257	1	8	286	283
				6	1	807	802	3	3	479	460	2	2	383	402
				6	2	136	121	3	4	248	244	2	3	229	228
				6	3	670	680	3	5	434	426	2	4	403	384
				6	4	181	182	3	6	418	429	2	5	217	200
0	1	996	961	6	5	351	360	3	7	396	407	2	6	197	168
0	3	1357	1325	6	6	167	162	3	8	418	440	2	7	203	175
0	5	684	627	6	7	194	199	4	0	261	238	3	3	274	254
0	7	371	366	7	3	229	233	4	1	994	987	3	5	266	244
1	1	130	112	7	4	122	128	4	2	251	240	3	6	203	173
1	2	760	661	7	5	393	427	4	3	761	759	3	7	232	253
1	3	931	844	8	0	684	703	4	4	289	258	3	8	246	251
1	4	443	386	8	1	665	677	4	5	142	129	4	0	300	301
1	5	724	703	8	2	393	397	4	6	142	149	4	1	578	568
1	6	393	298	8	3	515	521	5	1	179	173	4	2	274	272
1	7	741	773	8	4	298	295	5	2	218	205	4	3	443	439
1	8	131	94	8	5	289	303	5	3	571	570	4	4	223	215
1	9	556	599	9	1	141	144	5	4	397	358	5	1	168	171
2	0	1186	1251	9	2	298	303	5	5	489	483	5	2	157	149
2	1	1429	1711	9	3	390	316	5	6	253	251	5	3	506	512
2	2	616	593	9	4	463	469	5	7	404	417	5	4	232	248
2	3	1061	1007	10	0	479	490	6	0	418	419	5	5	483	466
2	4	570	510	10	1	486	506	6	1	749	759	5	6	191	176
2	5	279	261	10	2										

Table 2. *Positional and thermal atom parameters* ($\times 10^4$)

The figures given in parentheses are the standard deviations in the parameters.

The anisotropic temperature factors are of the form $\exp[-(B_{11}h^2 + B_{22}k^2 + B_{33}l^2 + 2B_{12}hk + 2B_{13}hl + 2B_{23}kl)]$.

	<i>x</i>	<i>y</i>	<i>z</i>	<i>B</i> ₁₁	<i>B</i> ₂₂	<i>B</i> ₃₃	<i>B</i> ₁₂	<i>B</i> ₁₃	<i>B</i> ₂₃
Sn	2100 (1)	2500	2165 (2)	39 (1)	114 (31)	59 (2)	0	-13 (2)	0
S	774 (4)	2500	6795 (5)	16 (4)	46 (33)	28 (7)	0	-1 (4)	0
O(1)	9240 (13)	2500	6025 (19)	14 (14)	153 (63)	109 (29)	0	-26 (16)	0
O(2)	1962 (14)	2500	5318 (16)	51 (16)	104 (59)	16 (22)	0	27 (15)	0
O(3)	1025 (9)	217	8005 (12)	55 (10)	35 (35)	79 (17)	13 (15)	11 (11)	26 (18)

Table 3. *Interatomic distances* (Å), *angles* (°) and *errors*

S-O distances		Sn-O distances	
S-O(1)	1.457 (12)	Sn-O(2) <i>A</i>	2.246 (11)
S-O(2)	1.482 (12)	Sn-O(3) <i>D</i>	2.273 (8)
S-O(3)	1.505 (9)	Sn-O(3') <i>E</i>	2.273 (8)
S-O(3')	1.505 (9)	Sn-O(1) <i>G</i>	2.949 (12)
O-S-O bond angles		Sn-O(2) <i>D</i>	3.079 (6)
O(1)-S-O(2)	112.8 (8)	Sn-O(2) <i>E</i>	3.079 (6)
O(1)-S-O(3)	110.6 (5)	Sn-O(3) <i>B</i>	3.109 (9)
O(2)-S-O(3)	107.6 (4)	Sn-O(3) <i>C</i>	3.109 (9)
O(3)-S-O(3')	107.6 (7)	Sn-O(1) <i>B</i>	3.181 (7)
O-Sn-O bond angles		Sn-O(1) <i>C</i>	3.181 (7)
O(2) A-Sn-O(3) <i>D</i>	77.1 (3)	Sn-O(3) <i>F</i>	3.336 (9)
O(2) A-Sn-O(3) <i>E</i>	77.1 (3)	Sn-O(3') <i>F</i>	3.336 (9)
O(3) D-Sn-O(3) <i>E</i>	79.0 (5)		

from any angle. Unlike previous plotting programs (Johnson, 1965; Cole & Adamson, 1969) it draws clinographic projections on crystallographic planes of space filling or 'ball and stick' models. Details of this program are available from the authors.

Discussion

The positions of the heavy Sn atoms obtained in the present work are close to those found by Rentzeperis (1962). The agreement for the S atoms is also reasonable but the changes in the oxygen atom positions lead to considerable differences in the important details of the tin environment.

Although the cell dimensions and powder diffraction data for SnSO₄ are very similar to those of BaSO₄, a comparison of their ionic radii (Table 4) suggests that the tin atom is too small to form a stable structure of the BaSO₄ type. The cell dimensions and other data for the barite minerals (SrSO₄, PbSO₄) which are isostructural with BaSO₄ are also given in Table 4.

Table 4. *A comparison of tin(II) sulphate with the barite group minerals*

	BaSO ₄	PbSO ₄	SrSO ₄	SnSO ₄
Space group	<i>Pnm</i>	<i>Pnma</i>	<i>Pnma</i>	<i>Pnma</i>
<i>a</i> (Å)	8.85	8.45	8.36	8.80
<i>b</i> (Å)	5.43	5.38	5.35	5.32
<i>c</i> (Å)	7.13	6.93	6.87	7.12
<i>V</i> (Å ³)	342.1	315.0	306.5	333.0
Cationic radius (Å)	1.35	1.21	1.13	0.85

Although this work confirms that SnSO₄ and BaSO₄ are not isostructural, tin(II) sulphate could be considered as a highly distorted form of the barite structure. In SnSO₄ the tin is surrounded by twelve oxygen atoms as in BaSO₄ but the Sn-O bond distances vary from 2.25 to 3.34 Å. Three of the oxygen atoms are much closer to the tin atom than the rest (2.25, 2.27 and 2.27 Å) and the next shortest Sn-O bond is 2.95 Å. The important feature of the SnSO₄ structure is the pyramidal three coordination of the tin atom, shown in Fig. 2. There are three Sn-O bonds of 2.25, 2.27 and 2.27 Å and pyramidal bond angles of 79.0, 77.1 and 77.1°.

This pyramidal three coordination of tin in its II⁺ oxidation state is found in a number of stannous materials (Donaldson, 1967) including SnS (Hofmann, 1935), SnSe (Okazaki & Ueda, 1956), orthorhombic SnF₂ (Donaldson & Oteng, 1967), NaSn₂F₅ (McDonald, Larson & Cromer, 1964), SnCl₂ (Rundle & Olson, 1964), KCl.SnCl₃.H₂O (Kamenar & Grdenić, 1962) and KSn(HCO₂)₃ (Jelen & Lindquist, 1969). This environment of the tin atoms can be explained either in terms of covalent bonding involving *sp*³ hybridization of the tin(II) orbitals or in terms of electrostatic crystal-field distortions (Orgel, 1959; Donaldson, 1967). In the covalent approach, the *sp*³ hybrid would produce three covalent bonds to nearest-neighbour atoms and a fourth orbital occupied by a lone-pair. The lone-pair electrons would prevent the close approach of further atoms in this direction and bond-pair-lone-pair repulsion arguments would predict X-Sn-X bond angles of less than 109°. The electrostatic treatment considers a displacement from the regular octahedral coordination, which allows mixing of the ground state (5s²) of a Sn²⁺ ion with the excited state (5s¹5p¹). In the Sn²⁺ ion, the separation of the ground state and the first excited state is of the order of 6.64 eV. In such circumstances it is possible to gain extra stabilization energy by an unsymmetrical distortion of the tin environment such as that found in SnSO₄.

The sulphate group in SnSO₄ is slightly distorted from regular tetrahedral symmetry by Sn-O interactions and the S-O bond lengths vary from 1.46 to 1.51 Å. These observations are in agreement with the infrared data of Hezel & Ross (1966) for the SO₄ group in SnSO₄.

We are grateful to Professor H. F. W. Taylor for permission to use the Aberdeen University Hilger-

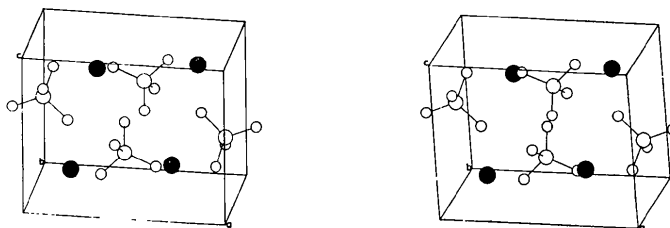


Fig. 1. Stereoscopic diagram of the unit-cell of SnSO_4 . Atom code as for Fig. 2.

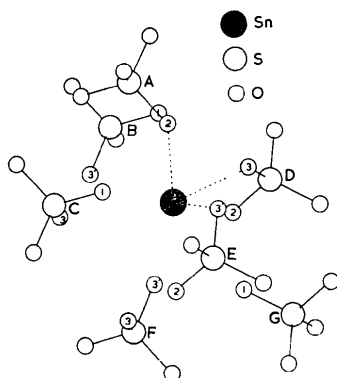


Fig. 2. The environment of the tin atoms in SnSO_4 .

Watts Y-190 linear diffractometer and to Mr A. Howie for his work in collecting the data. One of us (D.C.P.) is also grateful to the SRC for a studentship.

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A Study on the Diffraction Enhancement of Symmetry

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(Received 20 May 1971)

The condition for the diffraction enhancement of symmetry has been re-examined. From a general expression for the square of the structure amplitude for the structures composed of two kinds of parallel layers, it has been shown that the diffraction enhancement of symmetry may occur much more generally than previously supposed. Two kinds of constituent layers can have arbitrary thickness. It has been shown that the twofold rotational axis previously assumed for the local symmetry of each layer can be replaced by a twofold screw axis, a mirror plane, or a glide plane. The enhancement can take place not only from triclinic symmetry to the monoclinic Laue symmetry, but also from monoclinic symmetry to the orthorhombic Laue symmetry.

Introduction

Rose, Takeda & Wones (1966) reported that a triclinic polytype of mica produced monoclinic X-ray diffrac-

tion patterns. Sadanaga & Takeda (1968) called this phenomenon the 'diffraction enhancement of symmetry', which is caused by a particular structure of the crystal. According to the latter authors, a triclinic crys-