The Crystal Structure of $\text{Hg}(\text{OH})_2 \cdot 2\text{HgSO}_4 \cdot \text{H}_2\text{O}$

GERT BJÖRNLUND

Inorganic Chemistry 1, Chemical Center, University of Lund, P. O. B. 740, S-220 07 Lund 7, Sweden

The crystal structure of $\text{Hg}(\text{OH})_2 \cdot 2\text{HgSO}_4 \cdot \text{H}_2\text{O}$ has been determined from X-ray intensity data collected with a four-circle single crystal diffractometer. The crystals are monoclinic, space group $C2/c$, with $a = 7.152$, $b = 8.919$, $c = 14.488$ Å and $\beta = 98.94^\circ$. The unit cell contains four formula units.

The structure is built up by finite zig-zag chains $\text{O1}-\text{Hg}-(\text{OH})-\text{Hg}-(\text{OH})-\text{Hg}-\text{O1}$, where O1 is an oxygen atom in a sulphate group. The chains run approximately parallel to the bc -plane in the b -direction. There probably exist hydrogen bonds directly between the finite chains as well as between the chains *via* water molecules. In this way the structure constitutes a three-dimensional network.

According to Hoitsema there exist four solid phases in the system $\text{HgO}-\text{SO}_3-\text{H}_2\text{O}$.¹ One of these was described by the formula $3\text{HgO} \cdot 2\text{SO}_3 \cdot 2\text{H}_2\text{O}$. The crystal structure of this phase was later studied by Bonefačić, who presented the formula $2\text{HgSO}_4 \cdot \text{HgO} \cdot 2\text{H}_2\text{O}$.² However, only the locations of the mercury and the sulphur atoms were reported. The purpose of the present investigation was to study the coordination of the mercury atoms and thus a complete structure determination was performed. In a manner consistent with this examination the formula of the compound should be written $\text{Hg}(\text{OH})_2 \cdot 2\text{HgSO}_4 \cdot \text{H}_2\text{O}$.

EXPERIMENTAL

Crystals of $\text{Hg}(\text{OH})_2 \cdot 2\text{HgSO}_4 \cdot \text{H}_2\text{O}$ were formed when diluted sulphuric acid was allowed to be in contact with solid, freshly precipitated, yellow mercury(II) oxide. Transparent crystals grew out of the solid HgO . The homogeneity of the crystals was checked by X-ray Guinier-Hägg photographs.

X-Ray powder diffraction photographs were recorded in a Guinier-Hägg focusing camera using $\text{CuK}\alpha_1$ radiation and potassium chloride

as an internal standard. The powder pattern could be interpreted by means of a monoclinic unit cell with the following lattice parameters, with standard deviations in brackets: $a = 7.152(1)$, $b = 8.919(2)$, $c = 14.488(2)$ Å and $\beta = 98.94(2)^\circ$.

The density of the crystals, 6.18 g cm^{-3} , was determined from the apparent loss of weight in benzene. Assuming a unit cell content of four formula units, the calculated density is 6.17 g cm^{-3} .

A single crystal, elongated along b , of the approximate dimensions $0.03 \times 0.09 \times 0.01 \text{ mm}^3$, mounted along the b -axis, was used for recording the layers $h0l-h7l$ with a Weissenberg camera using Ni-filtered $\text{CuK}\alpha$ radiation. The multifilm technique was used. The intensities were estimated visually by comparing with a calibrated scale and corrected for Lorentz and polarization effects in the usual way. As the linear absorption coefficient³ was 1002 cm^{-1} , an absorption correction was carried out, the crystal being described by means of six planes. These film data were used for the preliminary determination of the structure.

The computer controlled four-circle diffractometer CAD-4 (manufactured by Enraf-Nonius, Delft, Holland) was employed for the refinement of the structure. The diffractometer was equipped with a graphite monochromator. $\text{MoK}\alpha$ radiation and the same crystal as above were used.

The intensities were recorded at a take off angle of 5° . The $\omega-2\theta$ scan technique was employed with the scan interval $\Delta\omega = 0.9 + 0.5 \tan \theta$. A fast pre-scan was used in order to determine the speed at which a predetermined minimum number of counts (2000) were received. The background intensity was measured for one fourth of the scan time at both ends of the interval. The intensities of 867 independent reflections were recorded in the range $3^\circ < \theta < 25^\circ$. The intensities of 658 reflections were considered significantly different from the background as they had $I > 3\sigma_0(I)$. The values of $\sigma_0(I)$ were based on counting statistics.

The stability of the crystal and of the electronics was checked by measuring two control reflections, 224 and 353, every 90 min. The

values of I and $\sigma_o(I)$ were corrected for Lorentz, polarization and absorption effects. The expression $p = (1 + \cos^2 2\theta_M \cos^2 2\theta)/(1 + \cos^2 2\theta_M)$ with $\theta_M = 6.08^\circ$ was used for the polarization factor. The linear absorption coefficient³ was calculated to be 518 cm^{-1} and the transmission factor, evaluated by numerical integration, varied in the interval 0.26–0.57.

The computer UNIVAC 1108 in Lund was used for the calculations. The programmes employed were CELSIUS, DATAPC, DRF, LINUS, WAL, ORTEP, LALS, DISTAN and SACTA. Information about the programmes is given elsewhere.^{4,5}

DETERMINATION AND REFINEMENT OF THE STRUCTURE

The systematic extinctions of the reflections hkl for $h+k$ odd and $h0l$ for h,l odd suggest one of the space groups Cc (No. 9) or $C2/c$ (No. 15) in both cases b as unique axis.⁶

Film data. A three-dimensional Patterson function was used to find preliminary positions of the mercury atoms. The space group was assumed to be $C2/c$. Least-squares refinements followed by three-dimensional difference Fourier-sections revealed the positions of the

sulphur and oxygen atoms. The positional parameters of all the atoms, the anisotropic temperature factors for the mercury atoms, the isotropic factors for the other atoms and the inter-layer scale factors were then refined using a full matrix least-squares programme. The resulting discrepancy factor $R = \sum ||F_o| - |F_c|| / \sum |F_o|$ was 0.114. The parameters of the mercury and sulphur atoms were in good agreement with those earlier given by Bonefačić.²

Diffraction data. For the refinement of the crystal structure there were 658 non-zero, independent reflections available. Employing the preliminary parameters as input for the diffractometer data the least-squares calculations performed in the same way as above resulted in $R = 0.037$. The quantity minimized was $w(|F_o| - |F_c|)^2$ with weighting according to $w = 1/(\sigma_o^2 + a|F_o|^2 + b)$, where a and b were chosen to make the quantity minimized equal in the different $|F_o|$ and $\sin \theta$ intervals. The convergence of the least-squares refinements was checked

by $R_w = [\sum w(|F_o| - |F_c|)^2 / \sum w|F_o|^2]^{\frac{1}{2}}$

as well as by the R -factor.

Table 1. Final positional and thermal parameters with standard deviations (in brackets). The temperature factor expression used is $\exp -[(\beta_{11}h^2 + \beta_{22}k^2 + \beta_{33}l^2 + 2\beta_{12}hk + 2\beta_{13}hl + 2\beta_{23}kl)]$.

Atom	Point position	x	y	z
Hg1	4(c)	1/4	1/4	0
Hg2	8(f)	0.25210(10)	0.93488(7)	0.14825(5)
S	8(f)	0.2167(5)	0.5923(4)	0.1050(3)
O1	8(f)	0.1579(16)	0.2109(12)	0.3381(9)
O2	8(f)	0.3764(18)	0.0037(14)	0.3311(8)
O3	8(f)	0.3490(18)	0.5019(15)	0.0611(9)
O4	8(f)	0.0809(18)	0.6684(15)	0.0361(10)
O5	8(f)	0.2190(16)	0.1639(12)	0.1353(8)
O6	4(e)	0	0.8354(17)	1/4

	$\beta_{11} \times 10^3$	$\beta_{22} \times 10^3$	$\beta_{33} \times 10^3$	$\beta_{12} \times 10^3$	$\beta_{13} \times 10^3$	$\beta_{23} \times 10^3$
Hg1	9.1(2)	4.1(1)	1.29(5)	0.0(1)	-0.45(7)	0.23(6)
Hg2	8.8(2)	3.4(1)	1.85(4)	-0.15(9)	-0.10(6)	0.03(5)
S	3.7(7)	3.5(6)	1.2(2)	-0.4(5)	-0.1(3)	0.0(3)
O1	6(2)	1(1)	2.5(6)	-0(1)	-1(1)	0.0(7)
O2	11(3)	5(1)	1.2(6)	2(2)	0(1)	0.4(8)
O3	10(3)	8(2)	2.5(7)	-1(2)	3(1)	-2.6(9)
O4	9(3)	8(2)	3.7(8)	-2(2)	-3(1)	3(1)
O5	8(3)	2(1)	1.9(6)	-3(1)	-1(1)	0.3(7)
O6	6(3)	2(2)	4(1)	0	-3(2)	0

Table 2. Observed and calculated structure factors for $\text{Hg}(\text{OH})_2 \cdot 2\text{HgSO}_4 \cdot \text{H}_2\text{O}$. The columns are l , $|F_o|$ and $|F_c|$.

h=0 k=0 l=0	6 333 335	5 441 504	15 112 119	4 288 281	4 143 139	-6 131 132	5 239 249
-12 233 237	8 289 283	4 148 152	15 156 162	5 235 227	6 342 326	-5 207 203	7 73 74
-10 203 194	9 164 155	5 258 262	15 2 K=4	4 188 187	8 281 283	-4 139 133	8 102 80
-8 432 404	11 90 89	7 148 136	7 199 198	7 199 198	10 78 86	-3 266 263	9 141 149
-6 404 432	12 167 161	9 234 225	-15 229 228	8 388 387	12 204 201	-2 300 306	
-4 353 320	14 216 226	0 117 117	-14 124 116	9 249 244		-1 85 85	h=6 k=4
-2 367 320	h=0 k=4	12 157 154	-12 149 144	11 170 164	h=4 k=2	1 00 87	-12 123 119
0 479 432	-10 363 345	-11 273 277	-11 164 155	-15 173 128	-15 157 156	2 317 320	-11 148 147
2 413 404	-6 385 395	h=1 k=7	-10 154 155	-13 200 282	-12 136 137	3 275 268	-10 126 123
10 105 104	-2 234 268	-9 116 124	-9 202 307	-12 90 87	4 219 227	4 219 227	-9 174 171
12 236 237	0 168 168	-12 231 222	-9 116 124	-9 92 80	5 174 163	5 174 163	-8 92 91
14 310 327	2 244 242	-11 86 73	-7 79 92	-7 512 535	-7 109 105	-7 109 105	-6 76 77
h=0 k=2	4 304 305	-9 75 49	-6 189 198	-6 154 157	-7 90 80	-7 90 80	-5 197 193
-16 207 218	6 76 74	-7 256 254	-5 387 417	-5 136 138	-5 172 179	-5 172 179	-4 153 150
-15 182 181	9 81 60	-7 204 208	-6 339 354	-4 605 415	11 166 166	11 166 166	-3 102 97
-13 94 109	10 357 345	-6 103 116	-3 140 151	-3 212 224	12 202 217	12 202 217	-2 152 151
-12 174 114	h=0 k=10	-5 181 182	-2 262 257	-2 167 164	-2 270 274	-2 270 274	-1 177 176
-11 225 222	-5 191 188	-4 176 176	-1 363 363	-1 442 475	0 54 52	-13 219 232	1 222 227
-10 401 399	-1 157 154	-2 269 272	0 166 162	1 551 539	1 251 248	-8 73 66	2 81 81
-9 274 228	0 206 215	1 125 188	1 373 376	2 104 101	-2 249 255	-7 434 450	3 66 59
-8 152 146	1 152 156	2 165 106	4 243 238	3 153 147	4 397 403	-5 95 103	4 155 151
-7 94 99	5 188 188	2 295 285	5 440 435	4 138 140	5 287 285	-3 184 188	5 242 238
-5 313 311	h=1 k=1	3 92 90	6 204 188	5 106 115	7 104 114	-2 102 112	6 125 128
-4 587 591	h=1 k=1	4 173 170	7 111 115	7 473 465	9 231 236	-1 361 356	8 114 123
-2 338 360	-17 189 182	5 225 224	8 156 147	13 525 530	11 153 153	2 117 113	h=6 k=4
-1 221 231	-14 194 200	6 193 182	9 163 157		12 90 82	3 108 109	-9 120 129
0 176 144	-15 140 147	7 234 228	10 128 130	h=1 k=5	5 127 125	5 127 125	-8 181 189
1 229 231	-14 124 131	8 278 270	11 225 223	h=4 k=4	7 342 345	-6 215 213	-6 215 213
2 352 360	-13 79 76	12 249 237	12 149 147	-11 200 190	8 103 98	-5 168 160	-5 168 160
4 416 491	-12 332 322	h=1 k=9	13 115 117	-9 266 260	-13 120 118	-1 127 133	0 295 297
5 332 311	7 262 255	h=1 k=9	14 123 133	-8 138 136	-12 111 111	h=5 k=5	1 134 127
7 94 99	-10 304 291	-7 251 245	h=2 k=4	-7 151 155	-11 193 190	-12 87 91	5 125 128
9 247 228	-7 68 59	-6 170 164	-14 202 212	-5 200 214	-10 139 150	-11 256 266	h=7 k=1
10 404 399	-6 324 332	-5 83 71	-13 81 54	-5 408 405	-9 255 244	-8 203 198	-11 150 152
11 217 222	-5 355 354	-4 123 131	-12 140 151	-2 73 62	-8 124 124	-7 71 83	-10 106 111
12 113 114	-4 205 199	-2 260 262	-11 100 93	0 77 76	-7 73 81	-7 109 103	-9 159 156
13 109 109	-3 479 508	-1 254 252	-9 145 151	1 63 62	-6 120 125	-6 67 56	-8 159 156
15 171 181	-2 362 371	1 240 243	-8 295 307	2 110 106	-5 318 325	-5 166 167	-7 101 99
16 211 218	-1 90 97	2 300 299	-6 312 321	3 475 482	-4 245 246	-3 405 407	-5 125 125
h=0 k=2	1 96 82	4 117 127	-5 206 207	4 157 157	-3 122 123	-2 67 72	-4 77 75
-15 226 228	2 349 346	5 96 90	-4 90 105	5 199 183	-2 240 240	0 85 85	-3 227 219
-14 136 140	3 504 489	6 149 152	-3 92 81	6 83 82	-1 208 208	2 64 73	-2 195 187
-13 138 144	4 340 345	7 228 227	-2 109 115	7 86 94	0 61 69	3 355 357	-2 195 187
-12 181 176	5 204 201	8 165 174	-1 206 210	8 156 144	1 362 305	4 123 113	-1 70 67
-11 250 252	6 261 236	h=2 k=0	0 500 512	9 256 256	2 144 145	5 149 153	0 86 86
-10 331 332	7 83 79	-14 343 332	1 181 180	11 279 279	3 49 81	6 65 63	2 208 215
-9 334 332	8 303 317	-13 150 153	2 90 93	12 136 128	4 212 216	7 74 85	3 159 163
-8 102 101	9 274 263	-12 143 132	3 112 108	h=3 k=7	5 353 348	8 125 122	4 142 143
-7 164 169	10 72 65	-10 154 154	4 211 209	-12 193 191	6 172 171	9 185 203	5 119 111
-6 241 242	11 212 221	-9 396 412	5 305 306	-11 90 79	7 147 153	h=5 k=7	6 149 146
-5 459 456	12 364 368	-8 577 588	6 214 207	-9 87 74	9 235 230	-8 144 174	h=7 k=5
-4 258 268	13 76 59	-6 324 323	0 167 151	-8 219 217	10 160 151	-7 139 138	-7 139 138
-3 162 161	14 149 160	-5 140 106	11 108 107	-7 166 171	11 177 172	-6 141 136	-7 329 331
-2 218 228	15 151 165	0 923 950	12 175 171	-6 127 128	12 93 93	-5 109 108	-5 79 73
-1 381 386	h=1 k=3	2 179 165	h=2 k=8	-5 159 158	h=4 k=6	-4 113 112	-3 126 127
0 202 205	-13 374 331	4 562 541	-10 317 332	-4 152 150	-12 113 101	-3 117 110	-1 244 242
1 382 396	-13 374 331	5 903 882	-9 68 70	-3 103 104	-11 85 89	-2 207 205	1 244 247
2 226 228	-12 97 96	1 96 85	-4 421 421	-2 219 211	-11 85 89	-1 168 170	2 67 68
3 169 161	-9 88 87	12 243 249	-2 213 220	-1 183 186	-9 161 137	1 115 119	3 86 80
4 270 268	-8 87 89	14 320 340	0 155 155	1 127 124	-8 219 217	2 206 199	5 96 112
5 479 464	-7 565 585	h=2 k=2	2 241 238	2 241 242	-6 273 274	4 170 169	h=7 k=5
6 557 542	-6 115 112	-16 195 192	4 356 349	3 61 63	-5 195 202	5 137 140	-7 93 80
7 182 169	-5 144 147	-15 189 188	6 93 91	4 183 185	-4 114 118	6 124 125	-5 105 111
8 114 101	-3 202 211	-11 102 94	0 76 79	5 170 167	-2 126 86	h=8 k=8	-3 278 271
9 553 532	-2 217 217	-13 102 94	h=2 k=10	6 183 179	h=10 k=12	-14 240 232	-2 84 84
10 147 142	-1 546 583	-12 154 150	-2 93 71	7 220 216	1 157 161	-12 87 82	2 82 86
11 254 252	-5 597 629	-11 194 186	-1 123 115	8 278 231	2 120 122	-10 103 103	3 261 269
12 183 176	3 189 182	-10 360 365	-9 219 214	h=3 k=9	3 95 87	h=8 k=8	h=8 k=8
13 146 148	4 111 108	-9 194 204	1 162 155	-7 238 237	5 196 182	-4 362 361	-4 362 361
14 135 140	5 127 119	-8 111 110	h=3 k=1	-6 136 137	6 286 294	-4 160 161	-8 178 172
15 228 228	7 589 557	-7 151 149	-1 123 115	-4 112 109	8 144 154	-6 247 242	-6 247 242
h=0 k=6	8 190 128	-5 263 266	-16 175 168	-2 230 228	9 124 131	2 131 129	-4 91 97
-14 217 226	9 94 74	-5 46 43	-15 108 109	-1 218 217	h=4 k=8	6 204 293	0 300 297
-12 162 161	15 122 128	-2 350 356	-14 128 125	1 221 214	-4 341 329	8 174 174	h=8 k=2
-10 159 155	h=1 k=5	-1 142 144	-12 308 305	4 85 80	-2 187 184	h=4 k=2	-5 96 91
-8 278 283	-15 82 84	1 270 269	-11 241 241	5 88 92	0 151 150	-12 91 79	-4 223 228
-6 335 335	-13 84 66	2 293 296	-10 176 180	h=4 k=0	2 109 200	-11 124 123	-3 79 85
-5 208 212	-12 162 162	3 78 72	-9 341 340	-14 294 292	4 313 308	-10 255 250	-2 129 124
-3 93 90	-11 320 324	4 507 581	-8 209 214	-12 105 106	6 76 60	-5 160 161	1 176 172
-2 98 112	-9 271 279	5 328 315	-7 296 293	-10 117 119	h=5 k=1	-4 346 349	2 134 134
-1 182 188	-5 204 203	6 63 75	-4 221 223	-8 434 431	-15 75 75	-2 207 207	h=8 k=8
0 434 452	-7 161 163	7 164 159	-3 430 437	-6 673 471	-12 273 210	-1 89 95	
1 188 188	-5 210 215	8 91 83	-2 645 460	-7 74 71	-11 194 197	1 202 204	
2 114 112	-4 127 120	9 245 239	-1 93 97	0 613 623	-9 153 143	2 222 233	
3 96 90	-3 575 570	10 416 404	1 298 204	2 80 83	-8 249 246	4 254 260	
4 87 97	-2 107 124	11 193 190	2 578 373				
5 209 212	2 165 170	12 104 98	3 381 384				

A refinement using anisotropic thermal parameters for all the atoms resulted in $R=0.034$ and $R_w=0.043$. Correction for isotropic secondary extinction and the use of anomalous scattering factors⁷ for the mercury and sulphur atoms affected the refinement little. The ex-

inction parameter⁸ $g=2.1(2) \times 10^3$ corresponds to a mosaic spread of 28.2 seconds or a domain size of 1.47×10^{-5} cm. The finally obtained agreement factors were $R=0.032$ and $R_w=0.041$. The average value of the quantity minimized in different $|F_o|$ and $\sin \theta$ intervals was fairly

constant. The values $a=0.0018$ and $b=0$ were used for the weighting. The atomic scattering factors used for the neutral mercury were those given by Cromer and Waber,⁹ and those for sulphur and oxygen by Hanson *et al.*¹⁰

As the peaks in the Fourier sections were distinct, the least-squares refinements converged in a normal way and the interatomic distances and angles were reasonable it was concluded that space group $C2/c$ was the correct one.

A three-dimensional difference Fourier calculation based on the final least-squares refinement showed no peaks of importance. Thus, it was not possible to locate the hydrogen atoms.

The positional and anisotropic thermal parameters of all the atoms with estimated standard deviations are listed in Table 1 and the observed and calculated structure factors in Table 2.

DESCRIPTION AND DISCUSSION OF THE STRUCTURE

A fundamental building element of the structure of $\text{Hg}(\text{OH})_2 \cdot 2\text{HgSO}_4 \cdot \text{H}_2\text{O}$ is finite zig-zag chains $\text{O1}-\text{Hg}-(\text{OH})-\text{Hg}-(\text{OH})-\text{Hg}-\text{O1}$,

where O1 is an oxygen atom in a sulphate group. This is to the authors knowledge the first example of a mercury compound with finite chains as a structural element. The distances and angles in the chains show good agreement with the values found in other chains containing mercury and oxygen.¹

Selected interatomic distances and angles in $\text{Hg}(\text{OH})_2 \cdot 2\text{HgSO}_4 \cdot \text{H}_2\text{O}$ are given in Table 3 and a stereoscopic pair of drawings of the structure is shown in Fig. 1 (notations of the atoms, *cf.* Table 1). As seen from Table 1 the atoms Hg1 occupy the point position 4(c) with point symmetry $\bar{1}$ and the atoms Hg2 the general point position 8(f). Each mercury atom Hg1 is bonded to two oxygen atoms O5 with distances $\text{Hg1}-\text{O5}$ 2.14(1) Å. The mercury atoms Hg1 are also surrounded by two other pairs of oxygen atoms, at distances of 2.48(1) and 2.45(1) Å, belonging to sulphate groups. The six oxygen atoms form a deformed octahedron. The mercury atoms Hg2 are each bonded to two oxygen atoms, *viz.* the atoms O1 and O5, with distances 2.10(1) and 2.06(1) Å, and the angle $\text{O1}-\text{Hg2}-\text{O5}$ 168.8(4)°. Around Hg2 there are further oxygen atoms at distances ≥ 2.65 Å.

The sulphate groups are slightly distorted,

Table 3. Distances (Å) and angles (°) with standard deviations in brackets.

Distances mercury to oxygen

Hg1-2 O5	2.14(1)	Hg2-O1	2.10(1)
Hg1-2 O3	2.48(1)	Hg2-O5	2.06(1)
Hg1-2 O4	2.45(1)		

Angles in the chains

$\angle \text{O5}-\text{Hg1}-\text{O5}$	180	$\angle \text{Hg2}-\text{O1}-\text{S}$	117.3(7)
$\angle \text{O1}-\text{Hg2}-\text{O5}$	168.8(4)		
$\angle \text{Hg1}-\text{O5}-\text{Hg2}$	114.2(5)		

Distances and angles within the sulphate tetrahedron

S-O1	1.54(1)	S-O3	1.46(1)
S-O2	1.46(1)	S-O4	1.45(1)
$\angle \text{O1}-\text{S}-\text{O2}$	108.5(7)	$\angle \text{O2}-\text{S}-\text{O3}$	111.6(8)
$\angle \text{O1}-\text{S}-\text{O3}$	104.4(7)	$\angle \text{O2}-\text{S}-\text{O4}$	111.7(8)
$\angle \text{O1}-\text{S}-\text{O4}$	108.6(7)	$\angle \text{O3}-\text{S}-\text{O4}$	111.7(8)

Distances and angles indicating hydrogen bonding

O1-O5	2.81(1)	O5-O6	2.85(1)
$\angle \text{Hg2}-\text{O1}-\text{O5}$	114.5(5)	$\angle \text{Hg2}-\text{O5}-\text{O1}$	103.5(5)
$\angle \text{S}-\text{O1}-\text{O5}$	108.3(6)	$\angle \text{Hg2}-\text{O5}-\text{O6}$	114.7(5)
$\angle \text{Hg1}-\text{O5}-\text{O1}$	108.0(5)	$\angle \text{O1}-\text{O5}-\text{O6}$	116.4(5)
$\angle \text{Hg1}-\text{O5}-\text{O6}$	100.2(4)	$\angle \text{O5}-\text{O6}-\text{O5}$	115.0(7)

Fig. 1. A stereoscopic pair of drawings showing the structure of $\text{Hg}(\text{OH})_2 \cdot 2\text{HgSO}_4 \cdot \text{H}_2\text{O}$. The hydrogen bonds in the cell are drawn with thin lines.

Fig. 2. A projection of a unit cell of the structure on the ac -plane. The hydrogen bonds are dotted and other bonds full lined.

giving an extra long bond, 1.54(1) Å, between S and O1, probably caused by the bonding O1 to Hg2. The same effect is found in the chromate group in the structure of $\text{HgCrO}_4 \cdot \frac{1}{2}\text{H}_2\text{O}$.¹³

The oxygen atoms O5 may form hydrogen bonds to the sulphate oxygen atoms O1 outside their own finite groups since the closest distance O1–O5 is 2.81(1) Å, which is a possible hydrogen bond distance.¹³ The angles involved are a further support for hydrogen bonding. For chemical reasons the hydrogen atom is closer to O5 than to O1, which means that the hydrogen bonding should be described O5–H...O1. The chains therefore resemble those found in $\text{Hg}(\text{OH})\text{BrO}_3$,⁵ and in $\text{Hg}(\text{OH})\text{F}$.¹⁴ In this way two-dimensional networks are built up parallel to each other, which is shown in a projection, Fig. 2.

Between the networks there are water molecules. The two-dimensional networks are probably knitted together by hydrogen bonds *via* the water molecules. The closest distance O5–O6, where O6 is the water oxygen atom, is 2.85(1) Å. The angles involved indicate that this is a hydrogen bond as well.

Acknowledgements. The author thanks Dr. Karin Aurivillius for valuable discussions and comments on this work. The author is also indebted to Professor Sture Fronæus for his kind interest in this work. This investigation is part of a research project, supported by the Swedish Natural Science Research Council.

REFERENCES

1. Hoitsema, C. Z. *Phys. Chem.* 17 (1895) 651.
2. Bonefačić, A. *Croat. Chem. Acta* 34 (1962) 119.
3. *International Tables for X-Ray Crystallography*, 2nd Ed., Kynoch Press, Birmingham 1968, Vol III, p. 162.
4. Oscarsson, Å. *Acta Crystallogr. B* 29 (1973) 1747.
5. Björnlund, G. *Acta Chem. Scand.* 25 (1971) 1645.
6. *International Tables for X-Ray Crystallography*, 3rd Ed., Kynoch Press, Birmingham 1969, Vol I.
7. *Ibid.* Vol III, p. 213.
8. Zachariasen, W. H. *Acta Crystallogr.* 23 (1967) 558–564.
9. Cromer, D. T. and Waber, I. T. *Acta Crystallogr.* 18 (1965) 104.
10. Hanson, H. P., Herman, F., Lea, I. D. and Skillman, S. *Acta Crystallogr.* 17 (1964) 1040.
11. Aurivillius, K. *Ark. Kemi* 24 (1965) 151.

12. Aurivillius, K. *Acta Chem. Scand.* 26 (1972) 2113.
13. Hamilton, W. C. and Ibers, J. A. *Hydrogen Bonding in Solids*, Benjamin, New York 1968, p. 259.
14. Grdenic, D. and Sikirica, M. *Inorg. Chem.* 12 (1973) 544.

Received October 20, 1973.