Title

CRYSTAL STRUCTURE OF SODIUM GOLD(I) THIOSULPHATE DIHYDRATE, Na3Au(S2O3)2 o 2H2O

Authors

Ruben, Helena
Zalkin, Allan
Faltens, Marjorie O.
et al.

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Helena Ruben, Allan Zalkin,
Marjorie O. Faltens, and David H. Templeton

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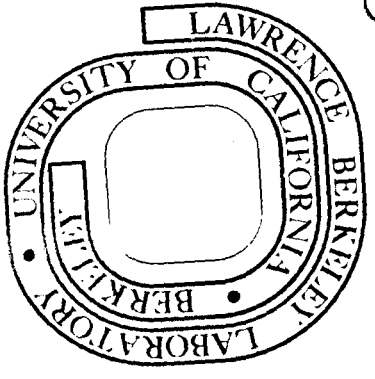

LBL-2369

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Crystal Structure of Sodium Gold(I) Thiosulphate Dihydrate, $Na_3Au(S_2O_3)_2 \cdot 2H_2O$[1]

HELENA RUBEN, ALLAN ZALKIN, MARJORIE O. FALTENS, AND DAVID H. TEMPLETON

The crystal and molecular structure of $Na_3Au(S_2O_3)_2 \cdot 2H_2O$ has been determined from a single crystal X-ray diffraction study. The monoclinic space group is $P2_1/a$ with a = 18.206(2), b = 11.355(6), c = 5.436(4) Å, β = 97.87(5)°, Z = 4, d_x = 3.14 g cm^{-3}. The structure was refined by full-matrix least-squares to a conventional R factor of 0.055 using 1811 non-zero intensities. The gold atom is bonded to two sulfur atoms from two thiosulfate groups in a nearly linear arrangement (S-Au-S, 176.5°). The average Au-S, S-S, S-O bond distances are 2.28(1), 2.06(1), and 1.46(1) Å. The three sodium atoms are coordinated to irregular polyhedra of oxygen neighbors; two of the sodium atoms have six such neighbors and one has five. Hydrogen bond lengths are O-H...S = 3.24(1) and 3.33(1) Å, O-H...O = 2.75(1) Å.

Introduction

An extensive investigation of the Mössbauer spectra of gold compounds was undertaken by Faltens and Shirley[2] to seek systematic relationships of these spectra to chemical structure. It was learned that both the isomer shift and the magnitude of the quadrupole splitting varied over wide ranges for various compounds, and neither effect alone could be used even to distinguish between gold(I) and gold(III). However, systematic relations were discovered which permitted unique discrimination between linear sp complexes of Au(I) and square-planar dsp^2 complexes of Au(III) when both effects were considered, and a partial understanding was gained of the origin of the variation of the spectra. The present study of the structure of $Na_3Au(S_2O_3)_2 \cdot 2H_2O$ was done partly to verify its structure for the Mössbauer investigation and partly as a contribution to the crystal chemistry of salt hydrates.

Gold sodium thiosulfate, known clinically as Aurocidin, Crisalbine, or Sanocrysin,[3] has been used in some medical circles for the treatment of arthritis, tuberculosis, and leprosy.

Experimental

Sodium gold(I) thiosulfate was made from $HAuCl_4$ and $Na_2S_2O_3$ and recrystallized from water-alcohol solution as described by Brown.[4] A small crystal, approximately 0.17 x 0.06 x 0.02 mm was glued to a thin pyrex glass fiber with the long dimension of the crystal (the crystallographic c axis) parallel to the fiber axis.

The crystals were aligned on a G. E. XRD-5 manual diffractometer equipped with a quarter-circle Eulerian cradle, a scintillation counter and a pulse height discriminator. X-rays were produced by a molybdenum anode tube ($\lambda_{K\alpha_1}$ = 0.70926, $\lambda_{K\alpha_2}$ = 0.71354 and $\lambda_{K\beta}$ = 0.63225 Å) operated at 45 kv and 20 ma; a 3.0 mil Zr filter was located at the receiving collimator. The tube take-off angle was 2° for the cell dimension measurements and 4° for the intensity measurements.

Cell dimensions were determined from a series of measurements along the $h00$, $0k0$, and $00l$ directions using peaks where the K_{α_1}, K_{α_2} and K_β were resolved. The β angle was measured directly from the angle between the $h00$ and $00l$ rows on the chi circle. All measurements were made at room temperature (≈23°). A total of 1966 independent reflections, all that are available in a quandrant of reciprocal space where $2\theta \leq 50°$, were counted for ten seconds each using the stationary-crystal stationary-counter technique. Background was plotted as a function of 2θ and applied routinely to the reflections with the exception of a minority for which background was measured individually because of streaking from lower orders. The most intense reflection (511) was 2303 counts per second after subtraction of the background.

The absorption parameter μ was calculated to be 145 cm^{-1}. An absorption correction was made with a program[5] that approximates the crystal with an array of grid points; it resulted in substantial improvement of the final agreement. The minimum and maximum corrections applied to the observed intensities were 1.35 and 2.62. An extinction correction was not indicated and not made. The usual Lorentz-polarization corrections were made.

The calculation of the estimated standard deviations of the intensities and the weighting scheme used in the least-squares are described elsewhere[6]. A value of p = 0.07 was found necessary to reduce the weights of the intense reflections so that their weighted residuals were comparable to those of lesser intensities. Our unpublished full-matrix least-squares program minimizes the function R_2^2 where $R_2^2 = \Sigma w(\Delta F)^2/\Sigma w F_0^2$. F_0 and F_c are observed and calculated structure factors, ΔF is the difference of their magnitudes, and w is the weighting factor. The program accommodates both the real and imaginary parts of the dispersion correction. Scattering factors for neutral Au, neutral S, neutral O and Na^+ were used[7] and modified for dispersion by adding -2.36, 0.1, 0.0 and 0.0 electrons[8] respectively. The imaginary dispersion terms are 8.89, 0.16, 0.0 and 0.04.

Results

Unit Cell and Space Group. The space group is $P2_1/a(C_{2h}^5)$ with cell dimensions at 23° of a = 18.206(6), b = 11.355(6), c = 5.436(4) Å and β = 97.87(5)°; the errors are subjective estimates.[9] With four formula units per unit cell and a cell volume of 1113 Å^3, the calculated density is 3.14 g cm^{-3}; a literature value[10] is 3.09 g cm^{-3}. The general position in $P2_1/a$ is $\pm(x,y,z;\ \frac{1}{2}+x,\frac{1}{2}-y,z)$.

Determination of the Structure. The gold atom location was deduced from a three-dimensional Patterson function. A Fourier synthesis phased by the gold atom revealed the locations of the sodium and sulfur atoms. A least-squares refinement of this partial structure using isotropic temperature factors resulted in $R = \Sigma|\Delta F|/\Sigma|F_o| = 0.21$. With anisotropic thermal parameters on the gold atom the R factor went to 0.19. A difference Fourier revealed the oxygen atom locations. Further refinement with all atoms anisotropic brought R to 0.078. After making the absorption correction the final agreement was R = 0.055 for 1811 non-zero intensities. R_2 was 0.063, and the standard deviation of an observation of unit weight was 1.19.[11]

Attempts to locate hydrogen atoms were unsuccessful. A difference Fourier showed 28 peaks larger than 1 Å^{-3}. The largest peak was 2 Å^{-3} and was near the gold atom as were several other large peaks. None of the top peaks appeared near reasonable locations for hydrogen atoms, and no attempt was made to include hydrogen in the calculations.

The final positional parameters with their standard deviations are listed in Table I. The anisotropic thermal parameters are listed in Table II.

Description of the Structure. The $Au(S_2O_3)_2^{3-}$ anion is shown in a schematic drawing in Fig. 1, and the crystal structure is shown in Fig. 2 and 3. Distances and angles are listed in Tables III and IV. The S-Au-S angle of 176.5(2)° is nearly linear, and there is no significant difference in the two Au-S bond lengths. The two S-S-Au angles are also essentially equal, and the dihedral angle of their planes is 67°. The two S-S distances are different from each other by little more than three standard deviations, but this difference is not considered to be significant. The average S-O bond distance of the six such bonds in this structure is 1.458 Å, and no individual value is more than its estimated standard deviation from this average. These dimensions of the thiosulfate group are in excellent agreement with those found by Baggio[12] for $[Co(NH_3)_5S_2O_3]Cl\cdot H_2O$, in which thiosulfate is complexed to cobalt through a sulfur atom as it is to gold in this crystal. As pointed out by Baggio,[12] the S-S bond is shorter in thiosulfates in which such complexing is absent, for example salts of Mg[13,14] and Na.[15,16]

Although the hydrogen atom positions were not determined directly from the diffraction data, one can identify three hydrogen bonds in the structure. The water molecule identified as O(7) has sulfur atom neighbors at 3.24(1) and 3.33(1) Å. These distances are within the range of O-H...S hydrogen bond distances which have been verified by nmr[17,18] and neutron diffraction[16] studies for sodium thiosulfate pentahydrate and magnesium thiosulfate hexahydrate, and the angles to these two sulfur neighbors and the two sodium neighbors are

roughly tetrahedral. On this basis we assign the two hydrogen atoms of O(7) to O-H...S bonds. Water molecule O(8) has a neighbor O(5) at 2.75(1) Å, a normal distance for an O-H...O hydrogen bond, and again the angles defined by this oxygen neighbor and the two sodium neighbors are reasonable for hydrogen bonding. The position of the other hydrogen on O(8) must complete a more or less tetrahedral coordination for this oxygen atom, but no neighbor is available close enough in a suitable direction for a hydrogen bond. Thus we conclude that O(8) makes only one hydrogen bond.

The next nearest neighbor to the gold atom, besides S(3) and S(4), is another gold atom at 3.302(1) Å. The nearest neighbors of the sodium atoms are all oxygen atoms. Na(1) is coordinated by six oxygen atoms at distances of 2.36 to 2.63 Å; the two nearest neighbors are two oxygen atoms of water molecules. Na(2) is also coordinated by six oxygen atoms at distances from 2.35 to 2.54 Å, the nearest of which comes from a water molecule. Na(3) is coordinated to five oxygen atoms ranging in distance from 2.34 to 2.53 Å, the closest of which is from a water molecule. The stereogram in Fig 3 shows some of the coordination about the sodium ions.

Table I. Positional Parameters[a]

	x	y	z
Au	.07361(3)	.06692(5)	.1479(1)
S(1)	.0006(2)	.3396(3)	.2178(5)
S(2)	.2592(2)	.0358(3)	.1207(5)
S(3)	.1591(2)	-.0366(3)	-.0338(6)
S(4)	-.0063(2)	.1713(3)	.3497(6)
Na(1)	.2176(3)	.3252(5)	.3495(10)
Na(2)	.1254(3)	-.4095(4)	.3159(9)
Na(3)	.4129(3)	-.1027(5)	.2427(10)
O(1)	.0762(5)	.3809(8)	.2892(17)
O(2)	-.0521(5)	.4044(8)	.3419(16)
O(3)	-.0179(5)	.3425(10)	-.0505(15)
O(4)	.3108(5)	-.0169(9)	-.0319(16)
O(5)	.2543(5)	.1633(8)	.0916(16)
O(6)	.2783(5)	.0010(8)	.3803(15)
O(7)[b]	.1652(5)	-.2179(9)	.4284(19)
O(8)[b]	.3387(5)	-.2627(8)	.3212(17)

[a] Standard deviations in this and subsequent tables appear in parentheses.

[b] Water molecule oxygen atom.

Table II. Anisotropic Thermal Parameters[a]

	B_{11}	B_{22}	B_{33}	B_{12}	B_{13}	B_{23}
Au	2.02(2)	1.84(3)	2.58(3)	.18(2)	.31(2)	-.23(2)
S(1)	1.82(12)	1.71(12)	1.41(11)	.02(10)	.15(9)	.03(10)
S(2)	1.99(12)	1.73(12)	1.05(11)	.03(10)	.14(9)	.04(9)
S(3)	2.10(13)	2.15(14)	2.03(13)	.38(11)	.11(10)	-.52(11)
S(4)	2.57(14)	1.88(14)	3.01(15)	.19(11)	.93(11)	.02(12)
Na(1)	4.33(28)	2.62(25)	2.47(22)	1.05(21)	.19(19)	.35(19)
Na(2)	2.70(22)	2.13(22)	1.97(20)	.07(17)	.19(17)	-.09(17)
Na(3)	2.65(23)	2.90(24)	2.57(23)	-.25(19)	.30(18)	-.22(19)
O(1)	2.13(38)	2.48(41)	3.12(43)	-.45(31)	-.17(31)	.44(34)
O(2)	2.53(38)	2.29(39)	2.71(40)	.41(31)	.98(31)	-.10(31)
O(3)	2.95(42)	4.46(52)	1.56(36)	.02(39)	-.64(30)	-.19(35)
O(4)	2.66(40)	3.00(42)	2.03(37)	.67(34)	.31(30)	-.30(33)
O(5)	3.12(41)	1.95(37)	2.15(35)	-.52(32)	-.47(30)	.40(31)
O(6)	3.20(40)	1.92(39)	1.52(34)	.08(31)	-.21(29)	.07(29)
O(7)	2.51(40)	3.25(46)	3.91(46)	-.01(35)	.85(35)	.31(39)
O(8)	4.30(50)	2.09(40)	2.40(39)	-.34(35)	-.05(35)	-.43(32)

[a] The anisotropic temperature factor has the form

$$T = \exp[-0.25(B_{11}a_1^{*2}h^2 + \ldots \quad +2B_{12}a_1^*a_2^*hk + \ldots)]$$

Table III. Interatomic Distances[a]

Atoms	Dist (Å)	Atoms	Dist (Å)	Atoms	Dist (Å)
Au -Au	3.302(1)	S(4) -O(7)[b]	3.326(10)	O(1) -O(2)	2.408(12)
Au -S(3)	2.280(3)	Na(1)-O(1)	2.626(10)	O(1) -O(3)	2.381(12)
Au -S(4)	2.272(3)	Na(1)-O(4)	2.494(11)	O(1) -O(2)	3.224(13)
S(1) -O(1)	1.457(9)	Na(1)-O(5)	2.460(11)	O(1) -O(4)	2.885(12)
S(1) -O(2)	1.458(9)	Na(1)-O(6)	2.473(10)	O(1) -O(6)	3.286(12)
S(1) -O(3)	1.451(9)	Na(1)-O(7)[b]	2.355(11)	O(1) -O(8)[b]	2.940(12)
S(1) -S(4)	2.051(5)	Na(1)-O(8)[b]	2.399(11)	O(2) -O(2)	3.221(19)
S(1) -Na(3)	3.201(6)	Na(2)-O(1)	2.540(10)	O(2) -O(3)	2.408(12)
S(1) -Na(3)	3.203(6)	Na(2)-O(2)	2.433(10)	O(2) -O(4)	3.255(12)
S(2) -O(4)	1.463(9)	Na(2)-O(3)	2.392(10)	O(2) -O(6)	3.303(13)
S(2) -O(5)	1.458(9)	Na(2)-O(3)	2.392(10)	O(2) -O(7)[b]	3.315(13)
S(2) -O(6)	1.461(9)	Na(2)-O(4)	2.390(11)	O(2) -O(8)[b]	2.940(12)
S(2) -S(3)	2.069(5)	Na(2)-O(6)	2.459(10)	O(4) -O(6)	2.403(12)
S(2) -Na(3)	3.199(6)	Na(2)-O(7)[b]	2.348(11)	O(4) -O(5)	2.426(13)
S(3) -O(4)	2.770(10)	Na(3)-O(1)	2.532(11)	O(4) -O(6)	3.176(12)
S(3) -O(5)	2.881(9)	Na(3)-O(1)	2.931(11)	O(5) -O(6)	2.421(12)
S(3) -O(6)	2.936(9)	Na(3)-O(2)	2.381(11)	O(5) -O(7)[b]	3.118(13)
S(3) -O(7)[b]	3.239(11)	Na(3)-O(2)	3.157(11)	O(5) -O(8)[b]	2.751(12)
S(4) -O(1)	2.858(10)	Na(3)-O(3)	2.383(11)	O(6) -O(7)[b]	3.260(13)
S(4) -O(2)	2.774(10)	Na(3)-O(4)	2.423(10)	O(6) -O(8)[b]	3.222(13)
S(4) -O(3)	2.904(10)	Na(3)-O(6)	2.908(11)	O(7)[b]-O(8)[b]	3.328(14)

[a]Distances have not been corrected for thermal motion. The table lists all of the distances less than 3.35 Å.

[b]Water molecule oxygen atom.

Table IV. Selected Angles

S(3) - Au -S(4)	176.5(2)	O(2)-Na(2)-O(3)	88.0(4)
O(1) -S(1)-O(2)	112.0(7)	O(2)-Na(2)-O(4)	150.7(5)
O(1) -S(1)-O(3)	109.9(7)	O(3)-Na(2)-O(6)	170.6(5)
O(2) -S(1)-O(3)	112.3(7)	O(4)-Na(2)-O(6)	81.8(4)
O(4) -S(2)-O(5)	112.2(7)	O(4)-Na(2)-O(7)	118.8(5)
O(4) -S(2)-O(6)	110.5(7)	O(6)-Na(2)-O(7)	92.4(4)
O(5) -S(2)-O(6)	112.1(7)	O(1)-Na(3)-O(2)	82.0(4)
Au -S(3)-S(2)	103.6(1)	O(1)-Na(3)-O(3)	117.7(4)
Au -S(4)-S(1)	104.1(1)	O(1)-Na(3)-O(4)	126.6(5)
O(1)-Na(1)-O(4)	68.5(3)	O(1)-Na(3)-O(8)	74.1(4)
O(1)-Na(1)-O(5)	116.6(4)	O(2)-Na(3)-O(8)	146.8(5)
O(1)-Na(1)-O(6)	80.2(4)	O(3)-Na(3)-O(8)	113.3(5)
O(1)-Na(1)-O(7)	156.6(5)	S(3)- O(7)-S(4)	103.9(2)
O(1)-Na(1)-O(8)	71.5(4)	S(3)- O(7)-Na(1)	101.4(3)
O(4)-Na(1)-O(6)	79.5(4)	S(3)- O(7)-Na(2)	114.2(3)
O(5)-Na(1)-O(7)	80.7(4)	S(4)- O(7)-Na(1)	123.6(3)
O(5)-Na(1)-O(8)	107.0(4)	S(4)- O(7)-Na(2)	89.4(2)
O(7)-Na(1)-O(8)	88.9(4)	Na(1)-O(7)-Na(2)	123.5(4)
O(1)-Na(2)-O(2)	80.8(4)	Na(1)-O(8)-Na(3)	97.4(3)
O(1)-Na(2)-O(3)	90.6(4)	Na(1)-O(8)-O(5)	117.2(4)
O(1)-Na(2)-O(7)	167.7(5)	Na(3)-O(8)-O(5)	113.2(4)

(1) Research performed under the auspices of the U. S. Atomic Energy Commission.

(2) M.O. Faltens and D.A. Shirley, *J. Chem. Phys.*, 53, 4249(1970); M.O. Faltens, Ph.D. Thesis, University of California, Berkeley, Calif., 1969.

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(9) Cell dimensions measured by J. Cabrera, and J.M. Gamboa, *Anales fis. y quim.* (Madrid), 41, 1387 (1945) are a = 18.22, b = 11.36, c = 5.45 Å, and β = 98.5°.

(10) R.C. Weast, Ed., "Handbook of Chemistry and Physics", 45th Ed., The Chemical Rubber Co., Cleveland, Ohio, (1964), p. B225.

(11) Supplementary Material Available. A listing of structure factor amplitudes will appear following these pages in the microfilm edition of this volume of the journal. Photocopies of the supplementary material from this paper only or microfiche (105 x

148 mm, 20x reduction, negatives) containing all of the supplementary material for the papers in this issue may be obtained from the Journals Department, American Chemical Society, 1155 16th St., N.W., Washington, D.C. 20036. Remit check or money order for 0.00 for photocopy or $0.00 for microfiche, referring to code number INORG-00000.

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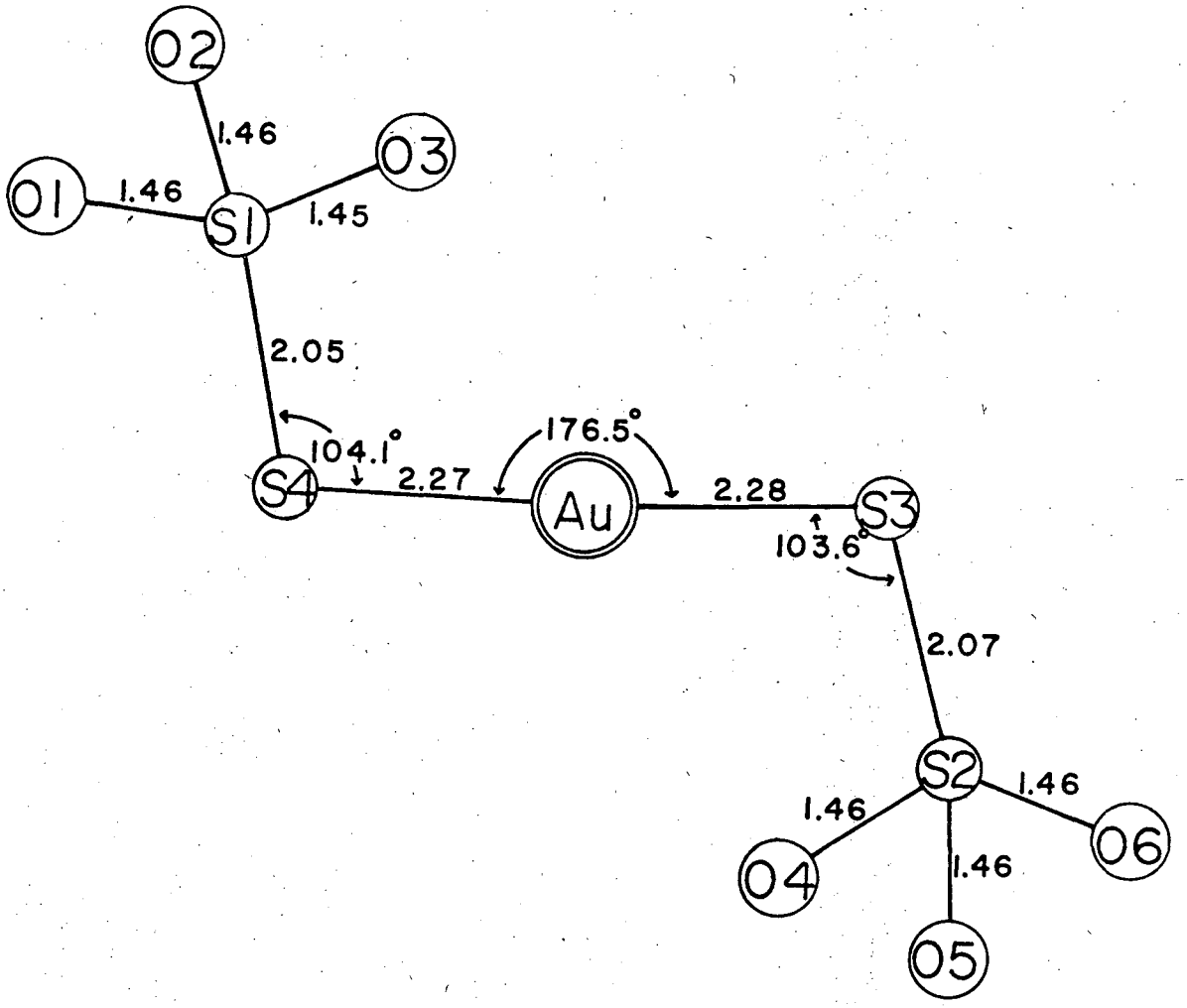
Figure Captions

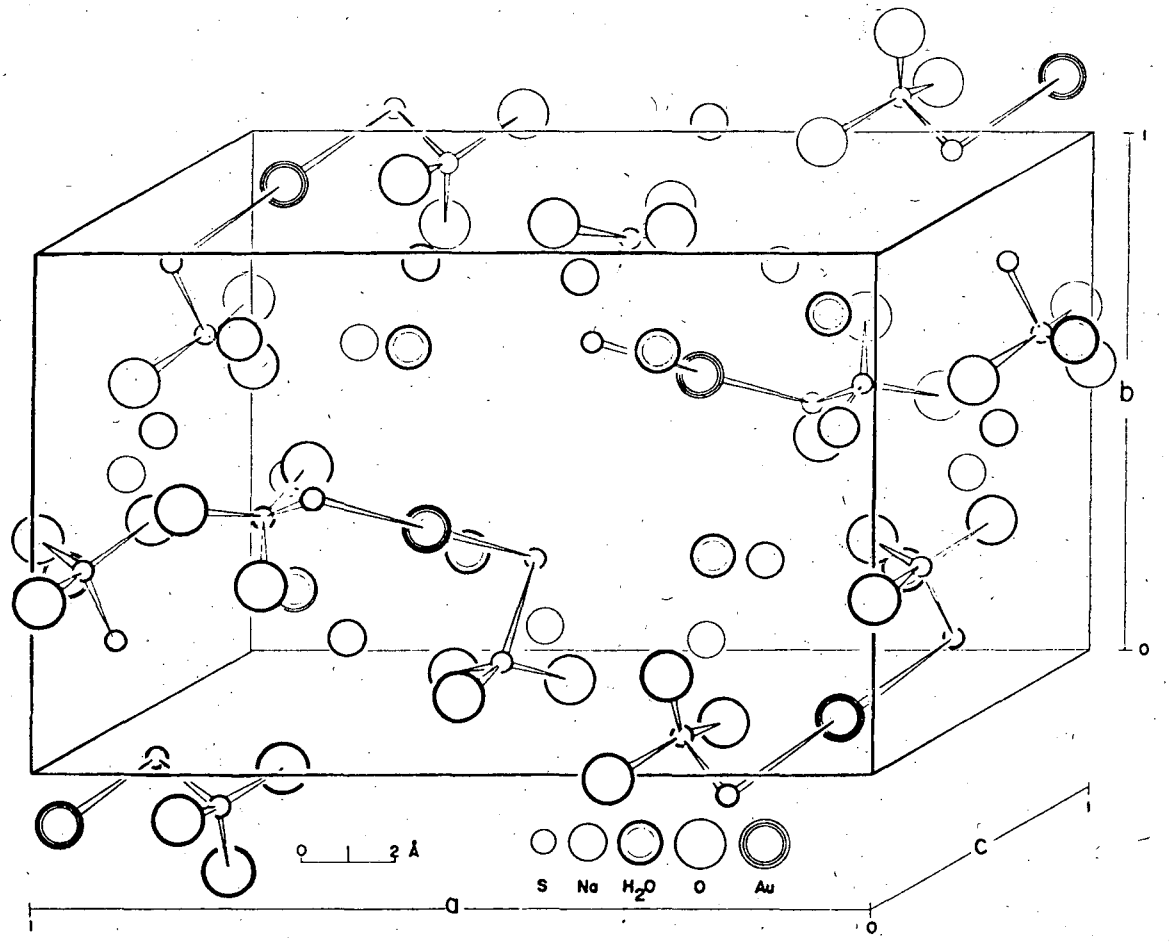
Figure 1. Schematic drawing of the $Au(S_2O_3)_2^{3-}$ anion.

Figure 2. Perspective view of the unit cell contents.

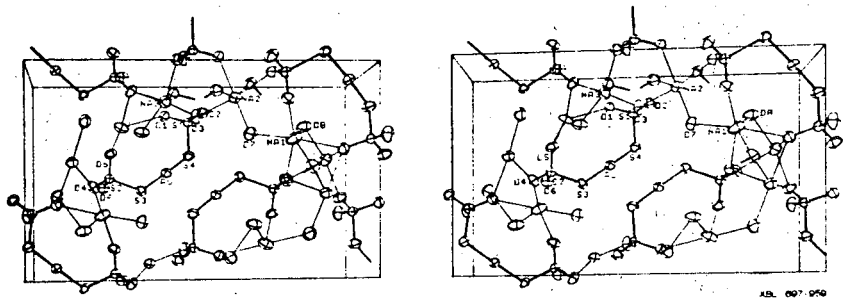
Figure 3. Stereogram showing the coordination to sodium atoms.


O2
1.46
O3
O1
1.46
S1
1.45
2.05
104.1°
176.5°
S4
2.27
Au
2.28
S3
103.6°
2.07
S2
1.46
1.46
O6
O4
1.46
O5

XBL 707-1307



MUB-11229



OBSERVED AND CALCULATED STRUCTURE FACTORS OF SODIUM GOLD THIOSULFATE DIHYDRATE
FCA(0,0,0) = 1896

H	FOB	FCA
K,L=	0,	0
2	354	324
4	113	113
6	365	360
8	225	210
10	18	20
12	524	491
14	414	387
16	177	164
18	25	40
20	149	137
K,L=	1,	0
1	309	292
2	200	191
3	74	73
4	206	208
5	365	350
6	91	90
6	90	90
7	213	228
8	246	253
9	382	381
10	225	239
11	170	171
12	123	123
13	284	283
14	0	22*
15	176	179
16	114	116
17	59	53
18	36	40
19	74	76
20	0	12*
21	207	192
K,L=	2,	0
0	450	473
1	170	174
2	15	26
3	339	333
4	176	176
5	358	339
6	492	473
7	88	94
8	299	297
9	100	103
10	130	128
11	241	241
12	136	144
13	90	92
14	131	136
15	22	21
16	57	61
17	196	201
18	107	102
19	74	73
20	125	125
21	49	38
K,L=	3,	0
1	264	271
2	331	333
3	92	80
4	569	565
5	148	138
6	192	185
7	108	107
8	299	321
9	70	75
10	450	468
11	58	55
12	156	146
13	138	138
14	73	75
15	88	88
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15	125	122
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13	205	201	-15	109	110	1	373	401	18	27	17	-2	49	50
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9	224	220	-18	27	30	-1	274	265	17	90	93	1	86	85
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2	167	167	-17	0	6*	3	340	340	-9	139	133	-15	0	19*
3	48	52	-16	169	176	4	29	17	-8	123	123	-14	147	133
4	233	229	-15	105	108	5	278	283	-7	45	43	-13	43	31
5	79	77	-14	82	83	6	67	68	-6	212	217	-12	205	189
6	158	152	-13	84	84	7	108	110	-5	57	56	-11	66	61
7	430	437	-12	78	71	8	89	94	-4	84	78	-10	0	23*
8	21	26	-11	114	110	9	75	67	-3	137	123	-9	114	101
9	144	140	-10	299	299	10	23	20	-2	34	25	-8	157	152
10	44	38	-9	31	22	11	259	262	-1	208	204	-7	125	116
11	56	52	-8	166	167	12	25	19	0	252	238	-6	292	277
12	48	41	-7	43	37	13	51	52	1	112	104	-5	0	1*
13	74	78	-6	71	70	14	64	70	2	208	200	-4	154	156
14	0	9*	-5	110	111	15	66	77	3	133	131	-3	47	36
15	165	155	-4	211	225	16	28	22	4	85	83	-2	66	59
16	76	79	-3	126	130	K,L=	5,	3	5	191	182	-1	155	150
17	56	58	-2	380	389	-18	66	71	6	142	145	0	233	224
K,L=	2,	3	-1	114	121	-17	0	9*	7	0	15*	1	25	23
-20	99	91	0	225	225	-16	170	177	8	224	226	2	168	175
-19	79	82	1	151	149	-15	77	76	9	24	21	3	83	79
-18	101	100	2	322	327	-14	93	92	10	50	48	4	0	5*
-17	164	165	3	131	123	-13	171	174	11	147	149	5	75	74
-16	0	7*	4	323	324	-12	58	66	12	58	61	6	170	177
-15	240	248	5	96	93	-11	68	72	13	27	38	7	89	96
-14	194	194	6	188	185	-10	245	246	14	187	180	8	218	227
-13	93	91	7	76	63	-9	25	21	15	0	24*	9	65	65
-12	137	136	8	22	13	-8	155	153	K,L=	7,	3	10	81	86
-11	138	130	9	22	12	-7	100	101	-16	53	60	11	48	50
-10	83	83	10	197	189	-6	35	26	-15	110	126	12	63	73
-9	237	231	11	115	117	-5	131	128	-14	30	29	K,L=	9,	3
-8	68	64	12	238	229	-4	253	251	-13	201	188	-13	82	74
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-6	266	271	14	0	17*	-2	325	324	-11	164	154	-11	74	72
-5	246	247	15	130	124	-1	220	226	-10	175	168	-10	42	37
-4	111	123	16	109	116	0	197	190	-9	28	25	-9	78	66
-3	283	296	17	49	53	1	215	214	-8	61	63	-8	137	126
-2	134	134	K,L=	4,	3	2	158	154	-7	225	217	-7	185	174
-1	295	305	-19	29	32	3	144	143	-6	46	36	-6	29	6
0	231	229	-18	0	5*	4	200	206	-5	213	203	-5	240	223
1	27	21	-17	117	122	5	119	119	-4	84	88	-4	115	112
2	148	144	-16	27	33	6	91	91	-3	26	22	-3	57	49
3	56	54	-15	136	141	7	23	24	-2	51	53	-2	226	221
4	157	159	-14	26	27	8	95	94	-1	223	217	-1	72	60
5	188	183	-13	36	39	9	94	98	0	60	49	0	38	35
6	227	223	-12	78	79	10	207	202	1	314	296	1	117	111
7	41	37	-11	327	322	11	25	25	2	130	141	2	0	17*
8	174	169	-10	24	12	12	222	234	3	180	187	3	46	42
9	157	156	-9	355	371	13	82	81	4	0	19*	4	147	154
10	84	93	-8	23	18	14	38	51	5	127	125	5	75	79
11	269	261	-7	148	139	15	91	93	6	24	29	6	60	74
12	137	136	-6	33	28	K,L=	6,	3	7	143	148	7	207	229

8 27 35	12 195 180	3 183 181
9 130 136	14 39 48	4 79 72
10 115 119	K,L= 1, 4	5 31 25
11 29 36	-18 49 42	6 241 242
K,L= 10, 3	-17 72 70	7 257 260
-11 54 56	-16 27 14	8 24 20
-10 0 2*	-15 284 273	9 202 208
-9 149 129	-14 44 51	10 50 45
-8 67 51	-13 116 119	11 178 170
-7 52 54	-12 87 91	12 133 135
-6 149 131	-11 71 75	13 38 41
-5 111 118	-10 89 95	14 39 46
-4 30 21	-9 188 195	K,L= 3, 4
-3 209 204	-8 44 38	-17 29 32
-2 50 58	-7 167 165	-16 68 71
-1 131 131	-6 43 50	-15 27 29
0 130 127	-5 121 131	-14 75 75
1 40 29	-4 75 82	-13 69 67
2 69 62	-3 307 304	-12 165 159
3 97 104	-2 23 21	-11 100 103
4 28 32	-1 351 381	-10 215 219
5 115 126	0 55 53	-9 140 138
6 84 97	1 102 98	-8 24 6
7 28 39	2 156 149	-7 120 134
8 40 53	3 64 57	-6 172 173
9 116 129	4 30 22	-5 105 99
K,L= 11, 3	5 200 198	-4 214 223
-8 173 163	6 22 31	-3 27 14
-7 0 13*	7 164 163	-2 59 60
-6 0 9*	8 33 40	-1 34 14
-5 31 30	9 24 9	0 131 127
-4 140 137	10 49 43	1 71 68
-3 86 88	11 243 230	2 235 238
-2 166 154	12 37 23	3 78 70
-1 0 7*	13 117 120	4 124 125
0 30 21	14 39 43	5 89 99
1 0 12*	K,L= 2, 4	6 69 67
2 125 107	-18 0 20*	7 120 114
3 59 59	-17 144 139	8 96 98
4 186 204	-16 118 127	9 43 42
5 0 13*	-15 26 31	10 149 145
6 135 139	-14 129 119	11 58 51
K,L= 0, 4	-13 67 68	12 53 52
-18 0 5*	-12 0 7*	13 38 35
-16 182 158	-11 202 196	14 97 93
-14 245 224	-10 132 130	K,L= 4, 4
-12 0 11*	-9 40 35	-17 110 105
-10 281 265	-8 119 122	-16 0 9*
-8 113 114	-7 185 189	-15 0 3*
-6 261 250	-6 172 174	-14 28 16
-4 190 186	-5 281 288	-13 135 147
-2 169 172	-4 138 143	-12 0 5*
0 302 294	-3 211 226	-11 288 300
2 99 93	-2 141 142	-10 0 11*
4 61 57	-1 100 108	-9 105 105
6 338 327	0 158 158	-8 96 89
8 118 106	1 165 165	-7 94 89
10 86 90	2 0 1*	-6 70 72

-5 229 238	-8 197 199	
-4 27 17	-7 69 65	
-3 72 70	-6 40 25	
-2 45 37	-5 131 125	
-1 35 18	-4 0 9*	
0 34 22	-3 0 16*	
1 351 360	-2 224 219	
2 73 73	-1 52 47	
3 307 316	0 123 124	
4 74 67	1 166 162	
5 135 137	2 66 72	
6 0 5*	3 75 80	
7 123 123	4 142 142	
8 25 20	5 25 37	
9 140 148	6 146 149	
10 0 11*	7 81 90*	
11 79 72	8 78 84	
12 27 27	9 0 23*	
13 78 85	10 77 78	
K,L= 5, 4	11 83 93	
-16 89 91	12 113 119	
-15 29 32	K,L= 7, 4	
-14 50 48	-14 32 35	
-13 80 82	-13 109 99	
-12 115 121	-12 44 41	
-11 39 24	-11 74 75	
-10 185 178	-10 84 85	
-9 82 80	-9 171 166	
-8 62 67	-8 41 43	
-7 242 252	-7 240 242	
-6 203 201	-6 50 59	
-5 62 58	-5 91 86	
-4 226 231	-4 0 24*	
-3 48 57	-3 116 114	
-2 121 115	-2 0 16*	
-1 105 102	-1 198 198	
0 99 99	0 38 24	
1 80 83	1 65 61	
2 194 198	2 131 145	
3 110 116	3 108 112	
4 128 129	4 53 55	
5 136 141	5 176 182	
6 121 130	6 46 49	
7 93 93	7 148 159	
8 110 119	8 27 39	
9 0 6*	9 0 13*	
10 179 179	10 28 32	
11 27 37	K,L= 8, 4	
12 28 36	-12 71 60	
13 113 107	-11 54 36	
K,L= 6, 4	-10 97 98	
-15 53 45	-9 68 67	
-14 109 121	-8 177 170	
-13 30 32	-7 30 21	
-12 42 33	-6 125 121	
-11 150 146	-5 0 11*	
-10 129 119	-4 89 95	
-9 40 31	-3 51 52	

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-1	94	95	-14	62	60	-11	99	93	-3	97	94	1	30	20
0	202	191	-13	27	30	-10	0	20*	-2	78	76	2	127	146
1	48	42	-12	70	68	-9	54	60	-1	0	10*	K,L=	0,	6
2	28	17	-11	149	156	-8	159	166	0	136	130	-10	122	106
3	150	149	-10	57	50	-7	54	53	1	27	30	-8	78	78
4	122	132	-9	107	112	-6	246	250	2	93	101	-6	94	77
5	47	59	-8	86	81	-5	0	18*	3	71	83	-4	150	152
6	182	185	-7	0	5*	-4	59	53	4	27	24	-2	38	42
7	28	44	-6	95	99	-3	30	20	5	76	82	0	142	135
8	56	61	-5	172	176	-2	139	143	6	195	189	2	185	176
9	81	75	-4	25	23	-1	83	86	7	28	30	4	39	44
K,L=	9,	4	-3	203	205	0	204	202	8	63	64	K,L=	1,	6
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-9	76	77	-1	139	143	2	114	110	-11	96	94	-9	0	8*
-8	44	49	0	133	133	3	51	47	-10	78	80	-8	88	88
-7	53	58	1	132	131	4	0	7*	-9	63	71	-7	83	90
-6	106	103	2	68	73	5	77	75	-8	64	70	-6	68	69
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-4	137	135	4	0	14*	7	37	33	-6	70	76	-4	28	15
-3	185	178	5	108	111	8	131	130	-5	94	95	-3	0	15*
-2	67	65	6	80	79	9	0	10*	-4	160	159	-2	30	33
-1	162	155	7	0	17*	10	48	49	-3	69	80	-1	0	20*
0	51	65	8	46	46	K,L=	4,	5	-2	168	168	0	0	8*
1	124	106	9	114	107	-14	0	13*	-1	41	45	1	197	201
2	58	67	10	0	20*	-13	88	85	0	64	63	2	39	32
3	0	14*	11	201	196	-12	41	41	1	141	135	3	72	76
4	40	28	K,L=	2,	5	-11	40	46	2	74	78	4	39	39
5	90	98	-15	143	149	-10	28	22	3	0	26*	5	28	14
6	0	4*	-14	40	46	-9	106	113	4	130	146	K,L=	2,	6
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K,L=	10,	4	-12	38	47	-7	210	218	6	0	13*	-9	95	95
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-6	110	102	-10	152	158	-5	159	169	K,L=	7,	5	-7	76	74
-5	139	134	-9	58	63	-4	53	45	-10	0	4*	-6	64	72
-4	32	24	-8	36	46	-3	0	5*	-9	161	151	-5	0	11*
-3	189	174	-7	62	59	-2	0	23*	-8	0	8*	-4	89	91
-2	89	87	-6	26	28	-1	84	82	-7	56	59	-3	144	155
-1	31	28	-5	88	87	0	0	11*	-6	46	18	-2	0	26*
0	53	58	-4	169	178	1	163	169	-5	97	99	-1	101	105
1	43	47	-3	78	76	2	52	41	-4	55	56	0	78	76
2	60	57	-2	151	164	3	0	18*	-3	166	158	1	28	38
3	104	114	-1	89	93	4	52	45	-2	0	5*	2	134	124
K,L=	0,	5	0	0	19*	5	144	135	-1	42	38	3	93	89
-14	0	11*	1	196	203	6	27	25	0	29	45	4	0	14*
-12	167	152	2	124	122	7	207	213	1	71	70	K,L=	3,	6
-10	298	285	3	69	65	8	27	22	2	0	7*	-9	42	33
-8	125	117	4	86	95	9	68	65	3	135	147	-8	115	129
-6	96	93	5	71	68	K,L=	5,	5	4	41	41	-7	67	56
-4	184	184	6	26	33	-13	0	16*	5	145	151	-6	67	77
-2	157	155	7	100	101	-12	90	100	K,L=	8,	5	-5	54	52
0	48	24	8	37	39	-11	60	60	-7	56	50	-4	98	108
2	239	245	9	0	14*	-10	0	2*	-6	32	34	-3	49	50
4	192	190	10	135	127	-9	100	96	-5	0	11*	-2	74	87
6	127	122	K,L=	3,	5	-8	141	150	-4	133	137	-1	44	42
8	124	115	-15	41	39	-7	0	10*	-3	111	115	0	103	97
10	129	119	-14	118	124	-6	183	197	-2	122	116	1	41	50
K,L=	1,	5	-13	28	37	-5	61	70	-1	31	31	2	0	6*

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K,L=   4,   6
  -7  106  113
  -6    0   15*
  -5    0   17*
  -4   34   27
  -3  146  145
  -2    0   15*
  -1  124  127
   0    0    2*
   1    0    9*
   2    0   15*
K,L=   5,   6
  -3   48   53
  -2  122  123
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TECHNICAL INFORMATION DIVISION
LAWRENCE BERKELEY LABORATORY
UNIVERSITY OF CALIFORNIA
BERKELEY, CALIFORNIA 94720