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Crystal Structure of Sodium Gold(I) Thiosulfate Dihydrate, $\text{Na}_3\text{Au}(\text{S}_2\text{O}_3)_2 \cdot 2\text{H}_2\text{O}^1$

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The crystal and molecular structure of $\text{Na}_3\text{Au}(\text{S}_2\text{O}_3)_2 \cdot 2\text{H}_2\text{O}$ has been determined from a single-crystal X-ray diffraction study. The monoclinic space group is $P2_1/a$ with $a = 18.206$ (6) Å, $b = 11.355$ (6) Å, $c = 5.436$ (4) Å, $\beta = 97.87$ (5)°, $Z = 4$, and $d_x = 3.14$ g cm⁻³. The structure was refined by full-matrix least squares to a conventional R factor of 0.055 using 1811 nonzero intensities. The gold atom is bonded to two sulfur atoms from two thiosulfate groups in a nearly linear arrangement ($\text{S}-\text{Au}-\text{S} = 176.5^\circ$). The average Au-S, S-S, and 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 $\text{O}-\text{H}\cdots\text{S} = 3.24$ (1) and 3.33 (1) Å and $\text{O}-\text{H}\cdots\text{O} = 2.75$ (1) Å.

Introduction

An extensive investigation of the Mossbauer spectra of gold compounds was undertaken by Faltens and Shirley² 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 $\text{Na}_3\text{Au}(\text{S}_2\text{O}_3)_2 \cdot 2\text{H}_2\text{O}$ was done partly to verify its structure for the Mossbauer investigation and partly as a contribution to the crystal chemistry of salt hydrates.

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

Experimental Section

Sodium gold(I) thiosulfate was made from HAuCl_4 and $\text{Na}_2\text{S}_2\text{O}_3$ and recrystallized from water-alcohol solution as described by Brown.⁴ A small crystal, approximately $0.17 \times 0.06 \times 0.02$ 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 GE 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, $\lambda_{K\beta}$ 0.63225 Å) operated at 45 kV and 20 mA; a 3.0-mil Zr filter was located at the receiving collimator. The tube takeoff 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 χ circle. All measurements were made at room temperature ($\approx 23^\circ$). A total of 1966 independent reflections, all that are available in a quadrant of reciprocal space where $2\theta \leq 50^\circ$, were counted for 10 sec 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/sec after subtraction of the background.

(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.

(3) R. E. Kirk and D. F. Othmer, "Encyclopedia of Chemical Technology," Vol. 7, Interscience Encyclopedia Inc., New York, N. Y., 1951, pp 287-288.

(4) H. Brown, *J. Amer. Chem. Soc.*, **49**, 958 (1927).

Table I. Positional Parameters^a

	x	y	z
Au	0.07361 (3)	0.06692 (5)	0.1479 (1)
S(1)	0.0006 (2)	0.3396 (3)	0.2178 (5)
S(2)	0.2592 (2)	0.0358 (3)	0.1207 (5)
S(3)	0.1591 (2)	-0.0366 (3)	-0.0338 (6)
S(4)	-0.0063 (2)	0.1713 (3)	0.3497 (6)
Na(1)	0.2176 (3)	0.3252 (5)	0.3495 (10)
Na(2)	0.1254 (3)	-0.4095 (4)	0.3159 (9)
Na(3)	0.4129 (3)	-0.1027 (5)	0.2427 (10)
O(1)	0.0762 (5)	0.3809 (8)	0.2892 (17)
O(2)	-0.0521 (5)	0.4044 (8)	0.3419 (16)
O(3)	-0.0179 (5)	0.3425 (10)	-0.0505 (15)
O(4)	0.3108 (5)	-0.0169 (9)	-0.0319 (16)
O(5)	0.2543 (5)	0.1633 (8)	0.0916 (16)
O(6)	0.2783 (5)	0.0010 (8)	0.3803 (15)
O(7) ^b	0.1652 (5)	-0.2179 (9)	0.4284 (19)
O(8) ^b	0.3387 (5)	-0.2627 (8)	0.3212 (17)

^a Standard deviations in this and subsequent tables appear in parentheses. ^b Water molecule oxygen atom.

The absorption parameter μ was calculated to be 145 cm⁻¹. An absorption correction was made with a program⁵ 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 procedure 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 $R_2^2 = \sum w(\Delta F)^2 / \sum w F_o^2$. F_o 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 used⁷ 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) Å, $b = 11.355$ (6) Å, $c = 5.436$ (4) Å, and $\beta = 97.87$ (5)°; the errors are subjective estimates.⁹ With four formula units per unit cell and a cell volume of 1113 Å³, the calculated density is

(5) W. C. Hamilton, absorption program HORSE, private communication, 1969.

(6) A. Zalkin, D. H. Templeton, and T. Ueki, *Inorg. Chem.*, **12**, 1641 (1973).

(7) D. T. Cromer and J. T. Waber, *Acta Crystallogr.*, **18**, 104 (1965).

(8) D. T. Cromer, *Acta Crystallogr.*, **18**, 17 (1965).

(9) Cell dimensions measured by J. Cabrera, and J. M. Gamboa, *An. Fis. Quim.*, **41**, 1387 (1945), are $a = 18.22$, $b = 11.36$, $c = 5.45$ Å, and $\beta = 98.5^\circ$.

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)	0.18 (2)	0.31 (2)	-0.23 (2)
S(1)	1.82 (12)	1.71 (12)	1.41 (11)	0.02 (10)	0.15 (9)	0.03 (10)
S(2)	1.99 (12)	1.73 (12)	1.05 (11)	0.03 (10)	0.14 (9)	0.04 (9)
S(3)	2.10 (13)	2.15 (14)	2.03 (13)	0.38 (11)	0.11 (10)	-0.52 (11)
S(4)	2.57 (14)	1.88 (14)	3.01 (15)	0.19 (11)	0.93 (11)	0.02 (12)
Na(1)	4.33 (28)	2.62 (25)	2.47 (22)	1.05 (21)	0.19 (19)	0.35 (19)
Na(2)	2.70 (22)	2.13 (22)	1.97 (20)	0.07 (17)	0.19 (17)	-0.09 (17)
Na(3)	2.65 (23)	2.90 (24)	2.57 (23)	-0.25 (19)	0.30 (18)	-0.22 (19)
O(1)	2.13 (38)	2.48 (41)	3.12 (43)	-0.45 (31)	-0.17 (31)	0.44 (34)
O(2)	2.53 (38)	2.29 (39)	2.71 (40)	0.41 (31)	0.98 (31)	-0.10 (31)
O(3)	2.95 (42)	4.46 (52)	1.56 (36)	0.02 (39)	-0.64 (30)	-0.19 (35)
O(4)	2.66 (40)	3.00 (42)	2.03 (37)	0.67 (34)	0.31 (30)	-0.30 (33)
O(5)	3.12 (41)	1.95 (37)	2.15 (35)	-0.52 (32)	-0.47 (30)	0.40 (31)
O(6)	3.20 (40)	1.92 (39)	1.52 (34)	0.08 (31)	-0.21 (29)	0.07 (29)
O(7)	2.51 (40)	3.25 (46)	3.91 (46)	-0.01 (35)	0.85 (35)	0.31 (39)
O(8)	4.30 (50)	2.09 (40)	2.40 (39)	-0.34 (35)	-0.05 (35)	-0.43 (32)

^a The anisotropic temperature factor has the form $T = \exp[-0.25(B_{11}a^*h^2 + \dots + 2B_{12}a^*b^*hk + \dots)]$.

Table III. Interatomic Distances (Å)^a

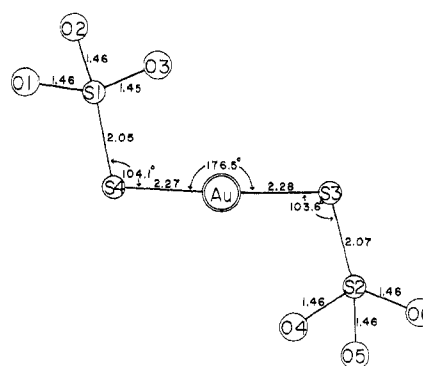
Au-Au	3.302 (1)	S(4)-O(7) ^b	3.326 (10)	O(1)-O(2)	2.408 (12)
Au-S(3)	2.280 (3)	Na(1)-O(1)	2.626 (10)	O(1)-O(3)	2.381 (12)
Au-S(4)	2.272 (3)	Na(1)-O(4)	2.494 (11)	O(1)-O(2)	3.224 (13)
S(1)-O(1)	1.457 (9)	Na(1)-O(5)	2.460 (11)	O(1)-O(4)	2.885 (12)
S(1)-O(2)	1.458 (9)	Na(1)-O(6)	2.473 (10)	O(1)-O(6)	3.286 (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.

3.14 g cm⁻³; a literature value¹⁰ is 3.09 g cm⁻³. The general position in $P2_1/a$ is $\pm(x, y, z; 1/2 + x, 1/2 - y, z)$.

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 map 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 nonzero 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 map 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.

Figure 1. Schematic drawing of the $\text{Au}(\text{S}_2\text{O}_3)_2^{3-}$ anion.

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 $\text{Au}(\text{S}_2\text{O}_3)_2^{3-}$ anion is shown in a schematic drawing in Figure 1, and the crystal structure is shown in Figures 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°.

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

(11) In the last cycle no parameter shifted more than 2% of its estimated standard deviation.

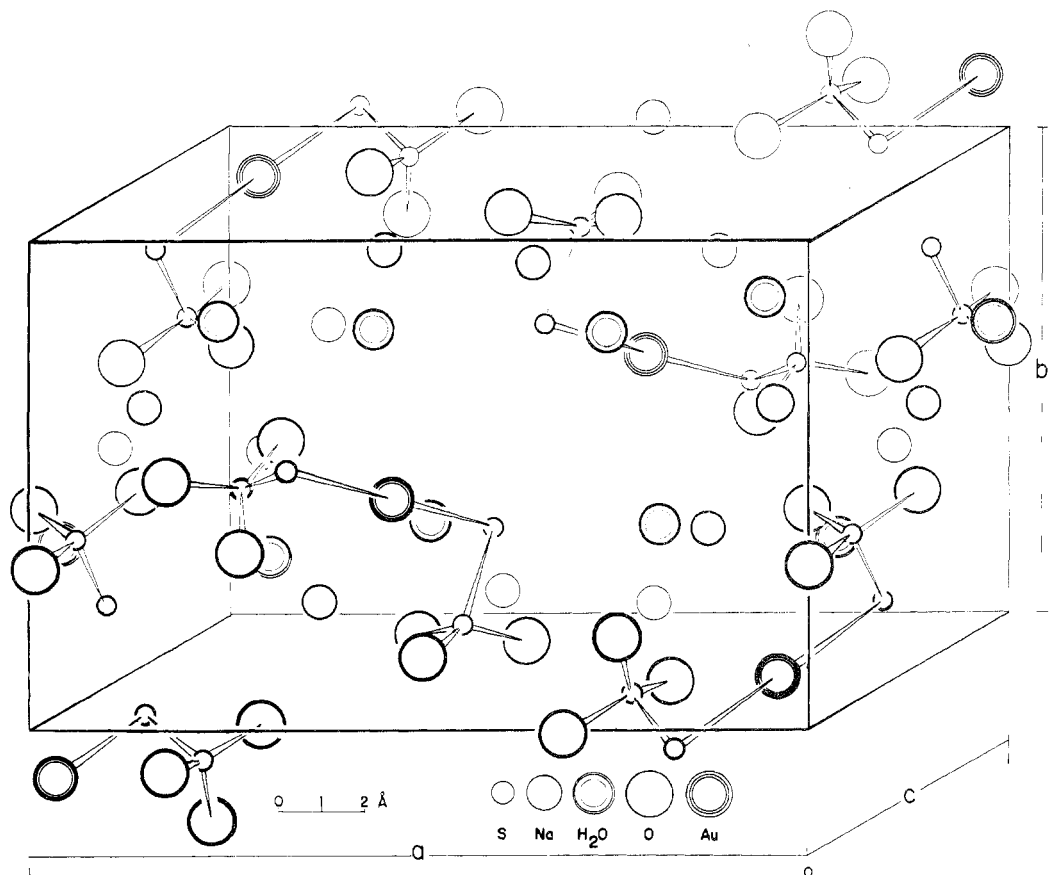


Figure 2. Perspective view of the unit cell contents.

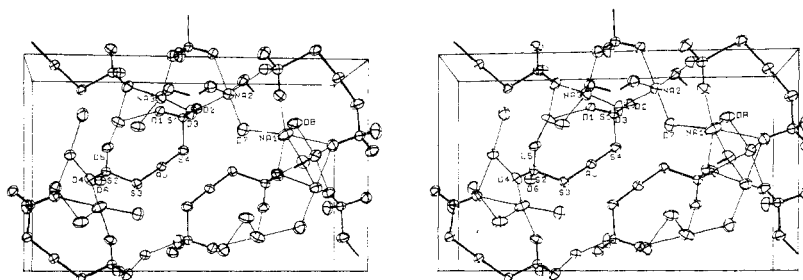


Figure 3. Stereogram showing the coordination to sodium atoms.

Table IV. Selected Angles (deg)

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)

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 Baggio¹² for [Co(NH₃)₅S₂O₃]Cl·H₂O, 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

(12) S. Baggio, *J. Chem. Soc. A*, 2384 (1970).

(13) S. Baggio, L. M. Amzel, and L. N. Becka, *Acta Crystallogr., Sect. B*, 25, 2650 (1969).

(14) M. Nardelli, G. Fava, and G. Giraldi, *Acta Crystallogr.*, 15, 227 (1962).

(15) P. G. Taylor and C. A. Beevers, *Acta Crystallogr.*, 6, 311 (1952).

(16) V. M. Padmanabhan, V. S. Yadava, Q. O. Navarro, A. Garcia, L. Karsono, I. H. Suh, and L. S. Chien, *Acta Crystallogr., Sect. B*, 27, 253 (1971).

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 diffraction¹⁶ 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 and 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 nearest neighbors of the sodium atoms are all oxygen atoms. Na(1) is coordinated by six oxygen atoms at distances of 2.36–2.63 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

(17) C. R. K. Murty and Z. M. El Saffar, *Acta Crystallogr.*, **15**, 536 (1962).

(18) Z. M. El Saffar, *Acta Crystallogr., Sect. B*, **25**, 1708 (1969).

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 Figure 3 shows some of the coordination about the sodium ions.

The next nearest neighbor to the gold atom, besides S(3) and S(4), is another gold atom at 3.302 (1) Å. This distance may be compared with 2.884 Å in metallic gold¹⁹ and values ranging from 2.67 to 3.27 Å in various gold cluster complexes.²⁰ If metallic bonding is present in our complex, it must be relatively weak.

Registry No. Na₃Au(S₂O₃)₂·2H₂O, 33847-99-3.

Supplementary Material Available. The table of observed and calculated 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 × 148 mm, 24× 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 \$3.00 for photocopy or \$2.00 for microfiche, referring to code number INORG-74-1836.

(19) D. N. Batchelder and R. O. Simmons, *J. Appl. Phys.*, **36**, 2864 (1965).

(20) S. L. Lawton, W. J. Rohrbough, and G. T. Kokotailo, *Inorg. Chem.*, **11**, 2227 (1972), and references cited therein.

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Crystal and Molecular Structure of μ -Carbonyl-bis- μ -[bis(bis(trifluoromethyl)phosphino)sulfur-*P, P'*]-bis(carbonylnickel)¹

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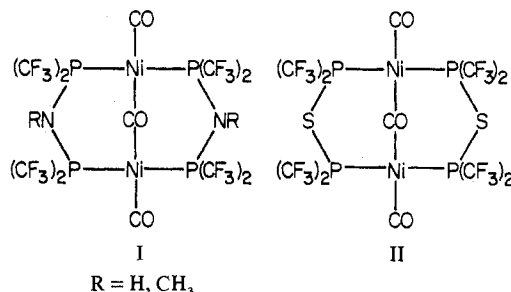
AIC40156M

The crystal structure of Ni₂ [(CF₃)₂PSP(CF₃)₂]₂(CO)₃ was determined from visually estimated intensity data from single crystals. The unit cell is orthorhombic, space group *Pbcn*, with *a* = 10.49 ± 0.01 Å, *b* = 14.26 ± 0.01 Å, and *c* = 18.77 ± 0.01 Å, and contains four molecules. A trial structure was obtained by direct methods and Fourier techniques and refined by full-matrix least squares. The final value of the *R* index is 0.087. The molecular structure includes a nickel-nickel bond of length 2.577 (5) Å and five-membered nickel-phosphorus-sulfur rings.

Introduction

The synthesis of Ni₂ [(CF₃)₂PSP(CF₃)₂]₂(CO)₃ was first reported as one of a series of syntheses of nickel carbonyl complexes with bifunctional phosphine ligands.^{2,3} With the exception of the thiobisphosphine product, all were fairly well characterized by standard physicochemical methods. The infrared spectra of two of the products, Ni₂ [(CF₃)₂PN(R)P(CF₃)₂]₂(CO)₃, R = H, CH₃, suggested the presence of both terminal and bridging carbonyl groups. On the basis of this and other evidence (molecular weights, decomposition products), bicyclic structures (I and II) were proposed for both. ¹⁹F nmr spectra of both amine products³ precluded the presence of significant paramagnetic behavior in either compound.

Attempts to measure the molecular weight of the thiobisphosphine product were without success. Its infrared spec-



trum, however, also gave evidence of bridging carbonyl groups and a structure similar to those of the aminobisphosphines was postulated. The thiobisphosphine product is assumed to exhibit magnetic properties similar to those of aminobisphosphines.

The presence of a bridging carbonyl group suggested the possible presence of metal-metal bonding. Among the population of known metal-metal bonds, examples of bonds between zerovalent nickel atoms are relatively rare. At the time this investigation was initiated only one such example

(1) Supported by the National Science Foundation and the Advanced Research Projects Agency, Office of the Secretary of Defense.

(2) A. B. Burg and R. A. Sinclair, *J. Amer. Chem. Soc.*, **88**, 5354 (1966).

(3) R. A. Sinclair and A. B. Burg, *Inorg. Chem.*, **7**, 2160 (1968).