however, we suggest that when all atoms are included in an interaction energy calculation, relative conformational energies may be realistically assigned. This treatment tends to indicate that for our system the borate configuration is relatively stable in its packing arrangement with the $[Ni_2(tren)_2(OCN)_2]^{2+}$ dimer, and this indeed may contribute to the driving force enabling us to isolate this compound.

The packing diagrams (see Figure 3) illustrate the facile dimer-tetraphenylborate packing. Two rings of each anion (those roughly perpendicular to the *b* and *c* unit cell axes) effectively isolate chains of dimer molecules in two directions. That these rings include an angle (see Table VIII) of 87.2° which is considerably different from the expected average ring-ring dihedral angle (~70° in ref 21) again points out the considerable conformational mobility of this anion.

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Supplementary Material Available. The final values of $|F_o|$ and $|F_c|$ for 2773 reflections 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 Street, 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-74-2056.

Contribution from the Division of Natural Sciences and Mathematics, University of Regina, Regina, Saskatchewan, Canada, and the Department of Chemistry and Chemical Engineering, University of Saskatchewan, Saskatoon, Saskatchewan, Canada

Nitrosodisulfonates and Hydroxylamine-*N,N*-disulfonates. II. Crystal Structure of a Rubidium Hydroxylamine-*N,N*-disulfonate

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The crystal structure of $Rb_5\{[ON(SO_3)_2]_2H\}\cdot 3H_2O$ has been determined by direct methods and refined by the full-matrix least-squares method to a weighted residue of 0.071, using 3256 independent reflections measured by diffractometer. The crystals are triclinic: $a = 9.357$ (6) Å, $b = 11.109$ (7) Å, $c = 11.206$ (5) Å, $\alpha = 102.03$ (3)°, $\beta = 99.09$ (4)°, $\gamma = 115.53$ (4)°. The structure contains 2 formula units in the space group $P\bar{1}$, and the two $\{[ON(SO_3)_2]_2H\}^{5-}$ anions occupy each of two independent centers of symmetry. The two $[ON(SO_3)_2]^{3-}$ groups in each anion are joined by a symmetric hydrogen bond; the O–H–O distances are 2.41 (2) and 2.43 (2) Å. The $\{[ON(SO_3)_2]_2H\}^{5-}$ anions are separated by the Rb^+ cations and water molecules.

A future article[1] discusses the preparation, characterization, and magnetic susceptibility of a series of hydroxylamine-*N,N*-disulfonates. A number of crystalline modifications of rubidium hydroxylamine-*N,N*-disulfonate have been prepared and identified. One has been characterized by an X-ray diffraction study and is reported here.

Experimental Section

Crystals of modification II[1] of rubidium hydroxylamine-*N,N*-disulfonate $\{[HON(SO_3)_2]^{2-}\cdot 2Rb^+\cdot {}^1/_2RbOH\cdot H_2O\}$ were larger than required for crystallographic studies and it was therefore necessary to fracture them. The fracture surfaces were curved. An attempt was made to grind the crystals to spheres but they shattered immediately in the sphere grinder. A sample of approximate dimensions 0.03 cm with irregular shape was finally selected for data collection. The approximate lattice constants and orientation of the crystal were first determined by film methods. The Laue symmetry indicated a triclinic space group.

The crystal was placed on a Picker FACS-I automated diffractometer equipped with a highly oriented graphite monochromator. The lattice constants were determined by a least-squares fit to the positions of 12 reflections with 2θ values between 39 and 42° as observed using Mo $K\alpha_1$ radiation. The lattice constants determined in this way were $a = 9.357$ (6) Å, $b = 11.109$ (7) Å, $c = 11.206$ (5) Å, $\alpha = 102.03$ (3)°, $\beta = 99.09$ (4)°, $\gamma = 115.53$ (4)°, and $V = 986.6$ Å^3, at 20°. Assuming 4 formula units per unit cell and a formula weight of 432.1, based on the stoichiometry given above, the calculated density is 2.93 g cm^{-3} which compares with a measured value of 2.85 g cm^{-3} obtained by picnometry. The linear absorption coefficient for Mo $K\alpha$ radiation is 50.1 cm^{-1}. The integrated intensities of 4574 independent reflections were measured using the θ-2θ scan mode and Mo $K\alpha$ radiation. Data were collected between 0 and 38° in 2θ with a scan rate of 2° min^{-1} and 10-sec background counts on each side of the peak. Data were also collected between 38 and 55° in 2θ with a 1° min^{-1} scan rate and 40-sec background counts. The intensities of three standard reflections were checked after each set of 98 data points was collected. These reflections decreased in intensity by roughly 30% over the time of the data collection. The average fluctuations between the intensities of successive standard reflections were of the order of magnitude expected from counting statistical errors. The data were corrected for the decrease in intensity using the averaged intensities of the three standards at each point at which standards were collected and interpolating linearly between each successive set of standards.

The data were then reduced to observed structure factors by standard methods. The polarization factor used was $(\cos^2 2\theta_m + \cos^2 2\theta_s)/2 \sin \theta_s$ where θ_m and θ_s are the monochromator and sample Bragg angles, respectively. The scattering factors were calculated from the series coefficients given by Cromer and Mann.[2]

Normalized structure factors were calculated, and from the average values of $|E|$ and $|E^2 - 1|$ (0.793 and 0.926) the space group was inferred to be $P\bar{1}$. The 396 reflections with $|E| > 1.71$ were used with the logical symbolic addition method (LSAM) programs of Germain and Woolfson.[3] None of the E maps generated using these programs were chemically reasonable. The convergence elimination algorithm, which is part of the MULTAN direct-methods

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[1] B. D. Perlson, D. B. Russell, and B. E. Robertson, *Inorg. Chem.*, in press.

[2] D. T. Cromer and J. B. Mann, *Acta Crystallogr., Sect. A*, **24**, 321 (1968).

[3] G. Germain and M. M. Woolfson, *Acta Crystallogr., Sect. B*, **24**, 91 (1968).

procedure,[4] was used to obtain an improved starting point. The sign of the 10,–10,2 reflection was determined to be positive by Harker-Kasper inequalities. Symbols were assigned to the reflections 2,–7,7, 3,–12,7, 444, 7,–11,2, and 7,0,–12. The probabilities that the sign of the reflection 2,–7,7 and the product of the signs of the reflections 3,–12,7 and 444 were positive were given as 1.0000 and 0.9993. These relations were used to determine the phases of the remaining reflections. A solution with the sign of the 2,–7,7 reflection positive and the other five signs negative gave the position of the five independent Rb atoms and three of the eight sulfur atoms. The rest of the 28 nonhydrogen atoms were then found by standard Fourier techniques.[5] The positions of these atoms were first refined by the full-matrix least-squares method using individual isotropic temperature factors, with all weights set at unity. The temperature factors were next allowed to vary anisotropically. Absorption corrections were made based on the approximation that the crystal was spherical with a radius of 0.016 cm. The curved shape of the crystal did not permit the estimation of exact anisotropic absorption corrections. Based on rather crude approximations, this procedure would lead to errors of up to 8% in $|F_o|$. The calculated intensities of a number of strong reflections were significantly higher than the corresponding observed values. It was assumed that these reflections were affected by secondary extinction and seven were removed from the refinement. The weighting scheme was changed to

$$w^{-1/2} = 7.453 - 0.1529|F_o| + 1.098|F_o|^2 \times 10^{-3} - 5.59|F_o|^3 \times 10^{-6}$$

The coefficients in the expansion were calculated from a fit of $||F_o| - |F_c||$ to $|F_o|$ and are based on the assumption that this gives the best available approximation to $\sigma(F_o)$. The fourth term was added after it was discovered that the curve intersected the abscissa when a three-parameter equation was used. Reflections for which $|F_o|/w^{-1/2}$ was less than 2.38 were excluded from the least-squares calculations, leaving 3256 independent reflections in the refinement. The anisotropic temperature factors of three atoms within the hydroxylamine-*N*,*N*-disulfonate groups were nonpositive definite, presumably because of the limited accuracy of the absorption corrections. The standard deviations for the thermal parameters of the atoms are probably underestimated in the least-squares procedure.

Extensive efforts were made to find the hydrogen atoms by difference Fourier methods after the best positions had been found for the nonhydrogen atoms. Possible positions were found in this manner for six hydrogen positions but they did not represent the highest peaks on the difference map and did not remain in chemically reasonable positions when their coordinates were allowed to vary in the least-squares procedure. Therefore all hydrogen atoms were excluded from the final cycles of least squares and the calculation of F_c. The agreement between $|F_o|$ and $|F_c|$ for the weaker reflections is not good. These reflections may be affected both by Renninger scattering and by an inadequate treatment of background.[6] The weighting scheme which was used gives these reflections low weight. The final weighted least-squares residue, R_w, that is, $[\Sigma w(|F_o| - |F_c|)^2/w|F_o|^2]^{1/2}$, was 0.071 and the traditional R factor was 0.089.

The observed and calculated structure factors are listed in Table I.[7] The final positional and thermal parameters are given in Table II.

Table II

Fractional Atomic Coordinates[a,b]

Atom	x/a	y/b	z/c
N(1)	1133 (14)	441 (10)	8625 (9)
N(2)	2029 (13)	6420 (9)	4634 (9)
O(1)	368 (14)	8557 (9)	6527 (9)
O(2)	1633 (12)	810 (11)	2942 (10)
O(3)	625 (14)	788 (9)	6489 (8)
O(4)	3508 (13)	9863 (10)	8763 (10)
O(5)	3789 (14)	1908 (11)	8195 (10)
O(6)	3688 (14)	1793 (10)	327 (9)
O(7)	130 (12)	6318 (10)	2784 (9)
O(8)	2033 (14)	5539 (9)	2326 (9)
O(9)	3006 (12)	8010 (8)	3333 (8)
O(10)	5075 (16)	3433 (13)	5208 (13)
O(11)	4675 (11)	8528 (8)	5935 (9)
O(12)	3762 (14)	6452 (11)	6581 (10)
O(13)	466 (12)	9412 (9)	9252 (8)
O(14)	1038 (13)	4972 (9)	4481 (9)
W(15)	4147 (17)	4667 (12)	8506 (11)
W(16)	507 (17)	2885 (12)	9509 (11)
W(17)	2797 (18)	6274 (15)	130 (12)
S(1)	21 (4)	9655 (3)	7032 (3)
S(2)	3190 (4)	1034 (3)	8989 (3)
S(3)	1814 (4)	6600 (3)	3153 (3)
S(4)	4011 (4)	7025 (3)	5542 (3)
Rb(1)	1441 (2)	6906 (1)	7923 (2)
Rb(2)	2124 (2)	3407 (1)	5874 (1)
Rb(3)	3024 (2)	3485 (2)	2243 (2)
Rb(4)	3200 (2)	9553 (1)	1494 (1)
Rb(5)	3089 (2)	180 (1)	5448 (1)

Thermal Parameters[b-d]

Atom	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
N(1)[e]	34 (6)	22 (5)	11 (5)	15 (5)	12 (4)	17 (4)
N(2)	23 (6)	13 (5)	17 (5)	4 (4)	8 (4)	11 (4)
O(1)	55 (7)	20 (5)	30 (5)	24 (5)	0 (5)	6 (4)
O(2)	21 (6)	47 (6)	48 (7)	10 (5)	8 (5)	24 (5)
O(3)[e]	59 (7)	26 (5)	21 (5)	19 (5)	12 (5)	23 (4)
O(4)	27 (6)	26 (5)	53 (7)	15 (5)	3 (5)	–3 (5)
O(5)	39 (7)	41 (6)	41 (6)	3 (5)	21 (5)	25 (5)
O(6)	46 (7)	27 (5)	24 (5)	7 (5)	5 (5)	2 (4)
O(7)	29 (6)	33 (5)	35 (5)	18 (5)	–2 (4)	16 (4)
O(8)	54 (7)	22 (5)	29 (5)	25 (5)	13 (5)	3 (4)
O(9)	35 (6)	11 (4)	30 (5)	2 (4)	11 (4)	14 (4)
O(10)	44 (8)	66 (8)	71 (9)	43 (7)	13 (6)	–3 (7)
O(11)	22 (5)	6 (4)	41 (5)	2 (4)	–4 (4)	9 (4)
O(12)	45 (7)	37 (6)	31 (5)	3 (5)	–4 (5)	25 (5)
O(13)[e]	40 (6)	36 (5)	30 (5)	18 (5)	30 (5)	32 (4)
O(14)	40 (6)	13 (4)	32 (5)	–4 (4)	4 (5)	16 (4)
W(15)	68 (9)	47 (7)	54 (7)	23 (7)	18 (7)	28 (6)
W(16)	73 (10)	38 (6)	46 (7)	31 (7)	4 (6)	12 (5)
W(17)	62 (9)	83 (10)	53 (8)	23 (8)	16 (7)	42 (8)
S(1)	24 (2)	15 (1)	17 (1)	11 (1)	5 (1)	9 (1)
S(2)	27 (2)	18 (1)	24 (2)	8 (1)	9 (1)	8 (1)
S(3)	28 (2)	9 (1)	17 (1)	9 (1)	9 (1)	8 (1)
S(4)	23 (2)	10 (1)	22 (2)	6 (1)	5 (1)	8 (1)
Rb(1)	58 (1)	19 (1)	51 (1)	11 (1)	–8 (1)	8 (1)
Rb(2)	41 (1)	20 (1)	33 (1)	11 (1)	11 (1)	9 (1)
Rb(3)	51 (1)	35 (1)	60 (1)	29 (1)	23 (1)	24 (1)
Rb(4)	40 (1)	36 (1)	27 (1)	22 (1)	13 (1)	19 (1)
Rb(5)	40 (1)	32 (1)	33 (1)	24 (1)	22 (1)	17 (1)

[a] $\times 10^4$. [b] Numbers in parentheses are estimated standard deviations. [c] $\times 10^3$. [d] The temperature factor expression is $\exp[-2\pi^2(h^2b_1{}^2U_{11} + \cdots + 2hkb_1b_2U_{12} + \cdots)]$. [e] Nonpositive definite.

Results and Discussion

The structure contains two independent $\{[ON(SO_3)_2]_2H\}^{5-}$ anions at the centers of symmetry at [0, 0, 0] and [0, 1/2, 1/2]. The $[ON(SO_3)_2]^{3-}$ groups in each anion appear to be joined by a symmetric hydrogen bond. Modification II of rubidium hydroxylamine-*N*,*N*-disulfonate is crystallized from aqueous solution brought to pH 12-13 with rubidium hydroxide and has the stoichiometry $[HON(SO_3Rb)]_2 \cdot {}^1/_2RbOH \cdot H_2O$. It appears that at pH 12-13 the solution contains both hydroxylamine-*N*,*N*-disulfonate dianions $[HON(SO_3)_2]^{2-}$ and nitrosyl-*N*,*N*-disulfonate trianions $[ON(SO_3)_2]^{3-}$ and that these ions condense to form the dimeric anions $\{[ON(SO_3)_2]_2H\}^{5-}$. The dimeric anions are separated by the Rb^+ cations and the water molecules. The correct

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[4] G. Germain, P. Main, and M. M. Woolfson, *Acta Crystallogr., Sect. B*, 26, 274 (1970).

[5] All Fourier calculations were carried out with a modified version of the program FORDAP written by Zalkin. All least-squares calculations were married out with a modified version of the program ORFLS written by Busing, Martin, and Levy. Stereoscopic drawings were prepared with the aid of the program ORTEP written by Johnson. Estimated standard deviations were calculated using the program ORFFE written by Busing, Martin, and Levy.

[6] P. Coppens, P. Becker, R. H. Blessing, and F. K. Ross, American Crystallographic Association Program and Abstracts, Series 2 1, Storrs, Conn., 1973, paper N5.

[7] See paragraph at end of paper regarding supplementary material.

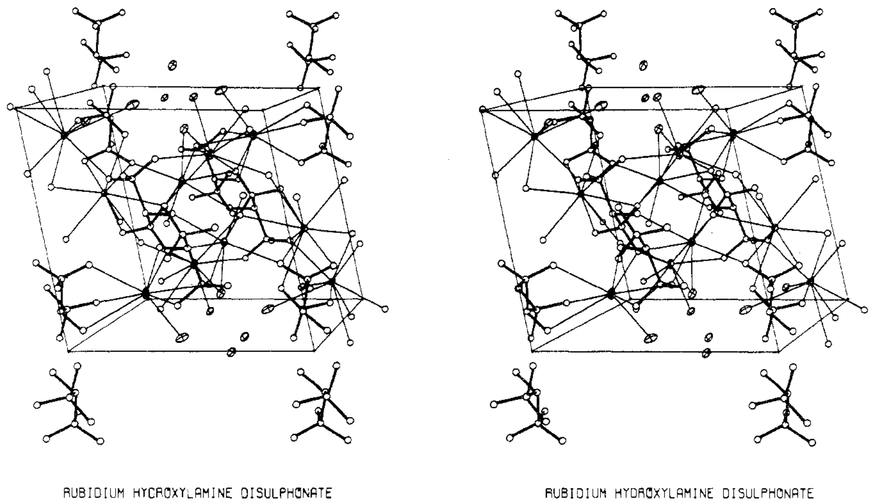

Figure 1. Stereoscopic view of $Rb_5\{[ON(SO_3)_2]_2H\}\cdot 3H_2O$. The atoms making up the hydroxylamine-*N,N*-disulfonate groups are shown as spheres and the bonds in those groups are shown as heavier lines. The water oxygen atoms appear as closed ellipsoids and the Rb^+ ions appear as ellipsoids with one octant removed. Rb–O distances less than 3.6 Å are shown as light lines. The outline of the unit cell is also included.

formula for the compound is then $Rb_5\{[ON(SO_3)_2]_2H\}\cdot 3H_2O$. A stereoscopic view of the structure is shown in Figure 1.

The cations do not have well-defined coordination spheres; even though distances greater than the sum of the ionic radii of the Rb and oxygen atoms are included, Figure 1 shows voids with no neighbors to the cations. The distances to Rb^+ ions are listed in Table III.

The three independent water molecules are all located near the face of the unit cell defined by the vectors **a** and **b**. The centers of symmetry at $[^1/_2, ^1/_2, 0]$ and $[0, ^1/_2, 0]$ repeat these groups to form continuous rods running in the **a** direction. The atoms with closest contacts to the water oxygen atoms, other than the Rb^+ ions, are the oxygen and nitrogen atoms of the dimeric anions. The shortest contact is the 2.85 (3) Å distances to the atom O(8) from W(17) (see Table II). The shortest contact between water oxygen atoms is the 3.07 (5) Å contact between W(15) and W(17) (see Table II). It is not possible to infer the presence or absence of hydrogen bonds from these distances.

An attempt was made to determine if the halves of the dimeric anions might be joined by asymmetric disordered hydrogen bonds. If this were the case the bonding between the nonhydrogen atoms of the donor and acceptor sides of the dimeric anions might be expected to be different, in which case atoms other than the hydrogen atom, particularly the nitrogen and hydroxyl oxygen atoms, might be expected to show disorder or thermal ellipsoids with anomalously long axes. It was for this reason that the structure was refined anisotropically, despite the inaccurate absorption corrections. Although the thermal parameters of three atoms became nonpositive definite, it was not possible to interpret the results of the refinement as showing disorder consistent with asymmetric hydrogen bonding.

The bond lengths and bond angles of the two dimeric anions are given in Figure 2. The distances between the nitroso oxygen atoms (2.41 (2) and 2.43 (2) Å) within the dimeric anions are characteristic of symmetric hydrogen bonding. Speakman and coworkers have reported symmetric hydrogen bonds in a number of acid salts of carboxylic acids.[8] A symmetric hydrogen bond has also been reported to join two molecules of the *N*-oxide alkaloid coccinellin[9] with an oxygen atom to oxygen atom distance of 2.43 Å. It has been suggested that crystals of the compound $Na_3H(CO_3)_2\cdot 2H_2O$ may contain symmetric hydrogen bonds with a slightly longer O–O distance of 2.53 Å.[10] The compound has also been studied by neutron diffraction[11] and the results suggest the possibility that the hydrogen bond is asymmetric and disordered.

Table III. Rb–O Distances Less Than 3.59 Å (Å)

Rb(1)–O(4)	2.842 (10)	Rb(2)–O(7)	2.859 (11)
O(14)	2.915 (9)	O(12)	2.907 (10)
O(17)	2.954 (12)	O(15)	2.911 (12)
O(12)	2.982 (13)	O(3)	2.920 (8)
O(1)	3.017 (9)	O(1)	2.936 (10)
O(7)	3.086 (9)	O(14)	2.941 (9)
O(8)	3.132 (11)	O(10)	2.962 (12)
O(13)	3.420 (12)		
Rb(3)–O(8)	2.799 (10)	Rb(4)–O(2)	2.882 (12)
O(6)	2.876 (11)	O(9)	2.924 (8)
O(15)	2.944 (12)	O(6)	2.945 (10)
O(2)	3.015 (10)	O(11)	2.962 (9)
O(12)	3.052 (12)	O(16)	3.167 (13)
O(13)	3.285 (10)	O(4)	3.193 (12)
O(16)	3.300 (12)	O(13)	3.215 (10)
O(10)	3.481 (12)	O(17)	3.473 (12)
Rb(5)–O(11)	2.864 (9)	Rb(5)–O(5)	3.067 (10)
O(11)	2.895 (8)	O(9)	3.194 (10)
O(9)	2.972 (9)	O(2)	3.266 (11)
O(3)	2.999 (11)	O(3)	3.371 (12)
O(1)	3.020 (11)		

The structure of a triclinic modification of potassium nitrosodisulfonate (Fremy's salt) has previously been reported,[12] and it was found that the nitrosodisulfonate anions crystallized in pairs with their nitroso groups in close proximity. The nitroso oxygen atom to nitroso oxygen atom distance was determined to be 3.10 (4) Å and the nitroso nitrogen atom of each nitrosodisulfonate anion was found to be at a distance of 2.86 (4) Å from the nitroso oxygen atom of the other nitrosodisulfonate anion. In addition the N–O

(8) W. C. Hamilton and J. H. Ibers, "Hydrogen Bonding in Solids," W. A. Benjamin, New York, N. Y., 1968, pp 181–182.

(9) R. Karlsson and D. Losman, *J. Chem. Soc., Chem. Commun.*, 629 (1972).

(10) C. J. Brown, J. S. Peiser, and A. Turner-Jones, *Acta Crystallogr.*, 2, 167 (1949).

(11) G. E. Bacon and N. A. Curry, *Acta Crystallogr.*, 9, 82 (1956).

(12) R. A. Howie, L. S. D. Glasser, and W. Moser, *J. Chem. Soc. A*, 3043 (1968).

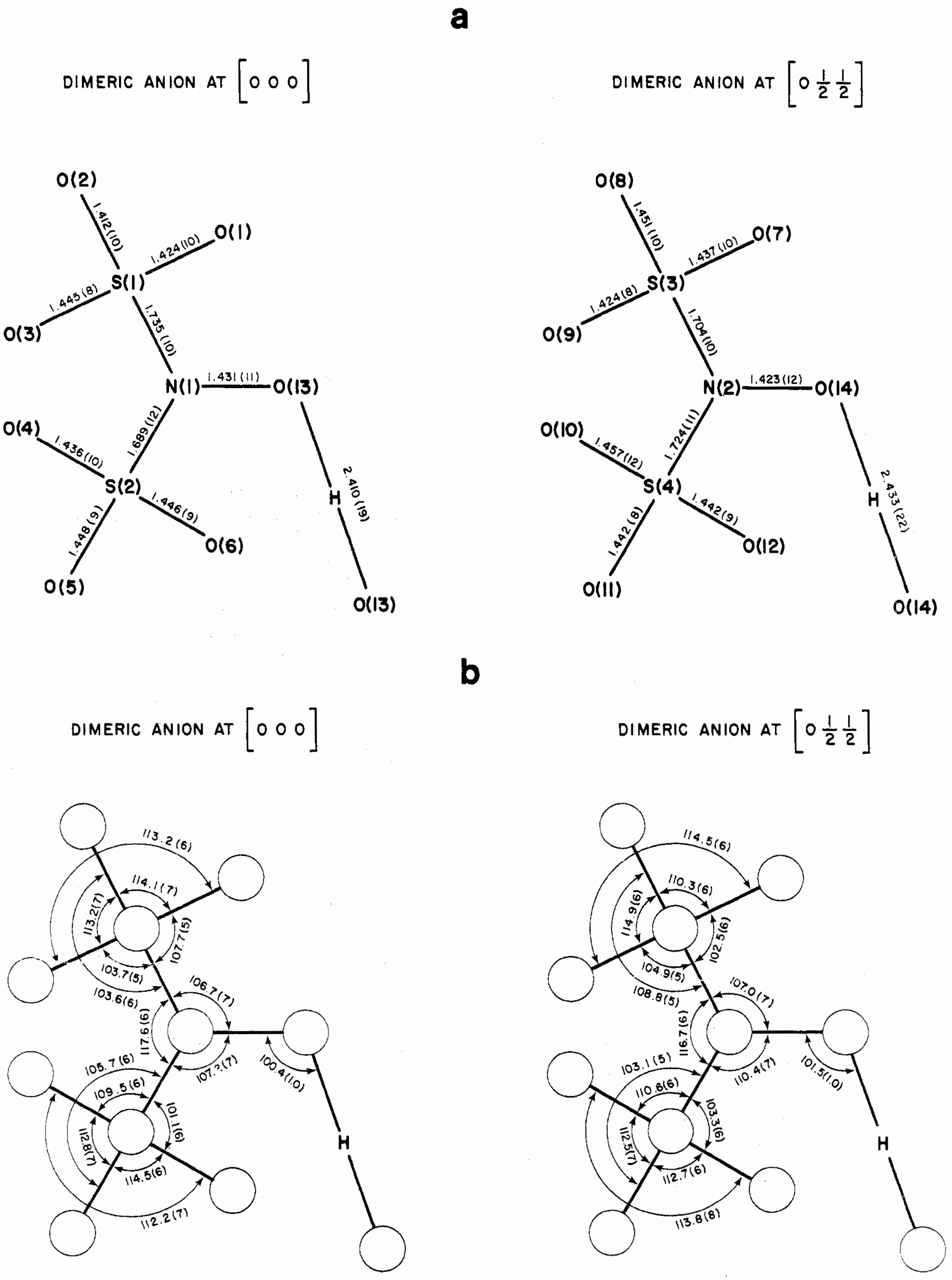

Figure 2. (a) Bond lengths in angstroms for hydroxylamine-*N*,*N*-disulfonate groups. (b) Bond angles in degrees for hydroxylamine-*N*,*N*-disulfonate groups. Numbers in parentheses are estimated standard deviations.

distance was 1.28 Å and the nitrogen atom was found to be approximately 0.10 Å out of the plane of the nitroso oxygen and sulfur atoms. The bond length of 1.28 Å for the N-O bond suggests that this bond has about 50% double-bond character and the fact that the nitrogen bonds are almost coplanar suggests that the nitrogen atom is essentially sp^2 hybridized. The N-O bond length together with electron paramagnetic resonance results of the unpaired spin densities on the nitrogen and nitroso oxygen atoms[13] suggest that the bonding between the nitrogen and nitroso oxygen atoms involves two electrons in a σ MO, two electrons in a π MO, and one electron in a π^* MO. In the present case the nitro-

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(13) Z. Luz, B. L. Silver, and C. Eden, *J. Chem. Phys.*, **44**, 4421 (1966).

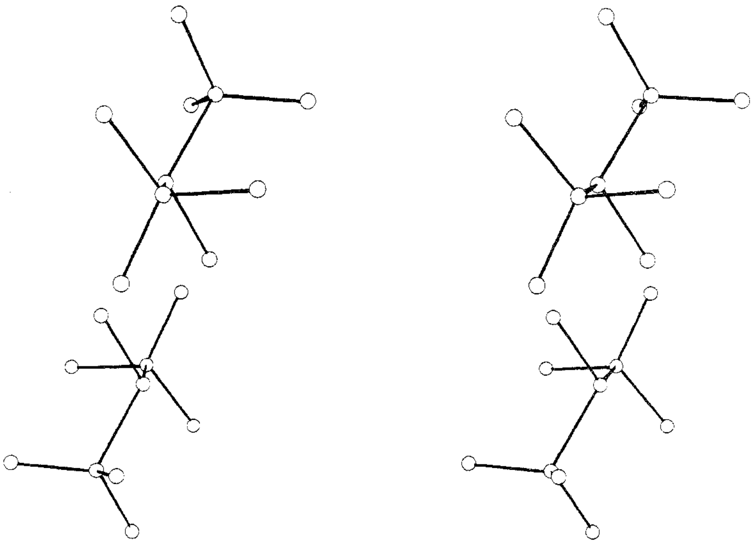

Figure 3. Stereoscopic view of the dimeric unit located at [0, 0, 0].

gen atoms are 0.51 and 0.48 Å out of the plane of the nitroso oxygen and two sulfur atoms for the dimeric anions at [0, 0, 0] and [0, $^1/_2$, $^1/_2$], respectively. The N-O distances are increased from 1.28 Å in the nitrosodisulfonate to 1.431 (11) and 1.423 (12) Å in the hydroxylamine-*N*,*N*-disulfonate. This may be compared with the N-O distance of 1.425 (7) Å[9] in coccinellin. These bond lengths are only slightly shorter than the length observed for an N-O single bond (1.46 Å), and the S-N-O and S-N-S bond angles (Figure 2) are close to the tetrahedral bond angles, suggesting that the nitrogen atom is essentially sp^3 hybridized in the hydroxylamine-*N*,*N*-disulfonate.

The structure of $K_3[SO_3ON(SO_3)_2]\cdot{}^3/_2H_2O$ has recently been determined by Brown and Strydom.[14] It shows a similar geometry to that reported here. However, the N-S and N-O bonds are slightly longer and the nitrogen atom is slightly further removed from the plane of the two sulfur atoms and the oxygen atom.

In modification II of rubidium hydroxylamine-*N*,*N*-disulfonate the plane formed by the N-O· · ·H· · ·O-N group and the plane of the nitrogen atom and the two sulfur atoms attached to it are nearly orthogonal (88.1 and 89.4° for the dimeric anions at [0, 0, 0] and [0, $^1/_2$, $^1/_2$], respectively). Considering half of the dimeric anion as a substituted hydroxylamine, the conformations about the N-O bond in the two independent groups are both trans, as is the conformation in the closely related anion in $K_3[SO_3ON(SO_3)_2]\cdot{}^3/_2H_2O$.[14] This would suggest that the trans conformation may be the stable one for hydroxylamines. The structure of hydroxylamine itself, as determined by Meyers and Lipscomb,[15] does not clearly indicate which conformation exists in the solid state. The geometry of one of the dimeric anions of $Rb_5\{[ON(SO_3)_2]_2H\}\cdot 3H_2O$ is shown in Figure 3.

The S-O distances average 1.438 Å, are similar to the values found for the nitrosodisulfonate, and are consistent with the analysis of $R(SO_3)_2$ groups given by Cruickshank.[16]

The structure of hydroxylamine shows an N-O distance of 1.48 (3) Å[15] while the equivalent distance in the monosulfonate is 1.51 (2) Å.[17] It seems unusual that the N-O bond should become longer in the monosulfonate and shorter in the disulfonate. The structure of both hydroxylamine and the monosulfonate were determined by film methods and refined to relatively large *R* factors. The quoted errors on these bond lengths may be somewhat optimistic.

Jeffrey and Jones[18] have predicted that the stability of aminesulfonates to acid hydrolysis parallels the bond order of the N-S bonds. Naiditch and Yost[19] have shown that the rate of acid hydrolysis of hydroxylamine-*N*,*N*-disulfonate is faster than that of the monosulfonate. Belt and Baenziger[17] have therefore predicted that the N-S bond lengths in the disulfonate should be longer than the value of 1.67 (2) Å which they found for the monosulfonate. The average value of the N-S bond length found here is 1.71 Å which is only marginally longer.

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Registry No. $Rb_5\{[ON(SO_3)_2]_2H\}\cdot 3H_2O$, 51795-71-2.

Supplementary Material Available. Table I, containing the observed and calculated structure factors, 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 $4.00 for photocopy or $2.00 for microfiche, referring to code number INORG-74-2062.

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