markedly longer, 2·827 (4) Å. The three shorter bonds do not deviate by more than about 10° from linearity, whereas the fourth [O(1)—H(21)···Br] deviates by 41·3°. It may thus be concluded that three of the H atoms are engaged in bonds of approximately equal strength and the fourth, H(21), is engaged in a much weaker bond. This agrees with the conclusion drawn by Schiffer & Hornig (1969) from the vibrational spectrum. This remarkable difference in hydrogen-bond strength is also reflected in the thermal-vibration parameters of the H atoms (cf. Table 1): the root-mean-square amplitudes of vibration of H(21) are considerably larger than those of the other three H atoms, which have very similar vibration ellipsoids. The mean dimensions of an isolated water molecule in the ground vibrational state are: r(O—H) = 0·974 Å, r(H—H) = 1·537 Å and ∠HOH = 104·54° (Kuchitsu & Bartell, 1962; Shibata & Bartell, 1965). From the data in Table 3, it can be seen that the O—H distances are somewhat shorter here. This tendency is opposite to the generally expected lengthening of the O—H bond on hydrogen-bond formation. We also see that the HOH angle in $H_2O(1)$ is smaller, 102·5°, and in $H_2O(2)$ nearly equal to the value in the isolated molecule. However, the dimensions here have been calculated directly from the final mean atomic positions. These positions differ from the true mean positions because of the inadequate treatment of the vibrations in the analysis of the diffraction data. In particular, the presence of large-amplitude librations is suggested by the appearance of the thermal ellipsoids. Such librations would lead to a shift in the apparent H positions towards the O atom when the structure is refined using standard anisotropic thermal parameters. This is consistent with the observations that (1) the O(1)—H(21) bond, 0·938 Å, is significantly shorter than the three other O—H bonds, 0·955–

0·958 Å, and (2) the amplitudes of vibration of H(21) are significantly larger than those of the remaining three H atoms. A discussion of the internal geometry of the water molecule will therefore have to be postponed until a more realistic description of the vibrational motions has been obtained.

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Ammonium Thiosulphate

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Abstract. $(NH_4)_2S_2O_3$, monoclinic, $C2$, $a = 10·230$ (4), $b = 6·500$ (2), $c = 8·820$ (3) Å, $\beta = 94·66$ (2)°, $V = 585$ Å^3, $Z = 4$, $D_c = 1·684$ Mg m^{-3}, Nb-filtered Mo $K\alpha$ radiation, $\mu = 0·80$ mm^{-1}; 936 independent diffractometer data up to sin $\theta/\lambda = 0·71$ Å^{-1} at room temperature; final $R(F) = 0·032$. The structure consists of NH_4 and S_2O_3 tetrahedra interconnected by hydrogen bonds. All but two H atoms were located by difference methods. One of the NH_4 groups is possibly disordered at room temperature.

Introduction. Crystals of $(NH_4)_2S_2O_3$ were grown from aqueous solution. A crystal 0·5 × 0·6 × 0·65 mm was selected. Precession photographs confirmed the lattice

constants reported by Brunt (1946) and Elerman, Uraz, Armağan & Aka (1978). They were refined from 15 reflections centred on a Syntex $P2_1$ diffractometer. The possible space groups for the systematic extinctions are: $C2$, Cm and $C2/m$, of which $C2$ was confirmed as correct after the refinement. 3878 reflections were measured up to $\sin\theta/\lambda = 0.71$ Å^{-1} on the diffractometer by the $\theta/2\theta$ scan technique. Three standard reflections observed after 50 reflections did not show any intensity fluctuations. An absorption correction was applied, $\mu = 0.8$ mm^{-1}; the correction factor ranged from 1.057 to 1.157. Background corrections were made by profile analysis (Blessing, Coppens & Becker, 1974). The equivalent reflections were weight-averaged, resulting in 936 independent reflections, of which 910 had $I > 2\sigma(I)$. This set was used in the subsequent refinements carried out with the XRAY system (Stewart, Kruger, Ammon, Dickinson & Hall, 1972).

An attempt to refine the structure from the atomic coordinates of Brunt (1946) was unsuccessful. Positional parameters for S were obtained by the Patterson method. Subsequent Fourier syntheses revealed the positions of all non-hydrogen atoms. Refinement of anisotropic temperature factors and of an isotropic extinction parameter yielded $R(F) = 0.038$. The H atoms were located from difference syntheses and refined with isotropic temperature factors to a final

$R(F) = 0.032$, $R_w(F) = 0.029$ and $S = [\sum w(F_o - F_c)^2/(NO - NV)]^{1/2} = 2.33$. Scattering factors were as listed in International Tables for X-ray Crystallography (1974), except for H (Stewart, Davidson & Simpson, 1965). The atomic coordinates are reported in Table 1, bond lengths and angles in Table 2.* A stereoview of the unit cell is given in Fig. 1.

Discussion. Brunt (1946) reported the S_2O_3 group in $(NH_4)_2S_2O_3$ as planar but the present study shows that an almost regular tetrahedron (with angles between 108.9 and 111.3°) exists, in agreement with the structure of $Na_2S_2O_3$ (Sándor & Csordás, 1961). N(1) in a general position is hydrogen bonded (Table 3) to three O atoms and to S. The other two N atoms are in nonequivalent special positions on the twofold axis along [010]. N(3) has a tetrahedral environment of four hydrogen-bonded O atoms while six O atoms form a distorted octahedron around N(2). The corresponding H atoms could not be located and we conclude that this NH_4 group is disordered at room temperature, which explains the large thermal motion of N(2). The hydrogen-bonding scheme (Table 3) consists of medium to weak bonds. They are comparable to

Table 1. *Atomic positional parameters*

	x	y	z
S(1)	0.3443 (1)	0.0000	0.1050 (1)
S(2)	0.2972 (1)	0.8236 (2)	0.2752 (1)
O(1)	0.1580 (2)	0.7643 (4)	0.2491 (2)
O(2)	0.3791 (2)	0.6374 (4)	0.2798 (3)
O(3)	0.3166 (3)	0.9401 (4)	0.4176 (3)
N(1)	0.1471 (3)	0.3260 (6)	0.2747 (3)
N(2)	0.0000	0.812 (2)	0.5000
N(3)	0.0000	0.9547 (7)	0.0000
H(11)	0.133 (5)	0.452 (9)	0.235 (5)
H(12)	0.055 (5)	0.254 (7)	0.264 (5)
H(13)	0.176 (3)	0.345 (7)	0.378 (4)
H(14)	0.197 (4)	0.251 (8)	0.225 (5)
H(32)	−0.053 (4)	1.057 (7)	0.053 (4)
H(31)	0.043 (4)	0.883 (7)	0.065 (5)

Table 2. *Interatomic distances (Å) and bond angles (°)*

S_2O_3 tetrahedron

S(2)–S(1)	1.979 (1)	S(1)–S(2)–O(1)	108.9 (1)
S(2)–O(1)	1.476 (2)	S(1)–S(2)–O(2)	108.9 (1)
S(2)–O(2)	1.472 (3)	S(1)–S(2)–O(3)	109.1 (1)
S(2)–O(3)	1.465 (2)		
O(1)–O(2)	2.403 (3)	O(1)–S(2)–O(2)	109.2 (1)
O(1)–O(3)	2.400 (3)	O(1)–S(2)–O(3)	109.4 (2)
O(2)–O(3)	2.425 (4)	O(2)–S(2)–O(3)	111.3 (2)
S(1)–O(1)	2.827 (2)		
S(1)–O(2)	2.824 (3)		
S(1)–O(3)	2.821 (3)		

Table 3. *Hydrogen-bonding scheme*

N(1) coordination

N–H···O	N···O,S	N–H	H···O,S	∠N–H···O,S
N(1)–H(11)···O(1)	2.860 (5) Å	0.90 (5) Å	2.05 (6) Å	150 (4)°
N(1)–H(12)···O(2)	3.006 (4)	1.06 (4)	1.96 (4)	170 (4)
N(1)–H(13)···O(3)	2.809 (4)	0.94 (3)	1.91 (4)	160 (3)
N(1)–H(14)···S(1)	3.361 (3)	0.86 (4)	2.51 (4)	173 (4)

N(2) coordination

N(2)···O(1)	2.861 (3) Å	N(2)···O(3)	3.107 (7) Å
N(2)···O(2)	3.065 (7)		

N(3) coordination

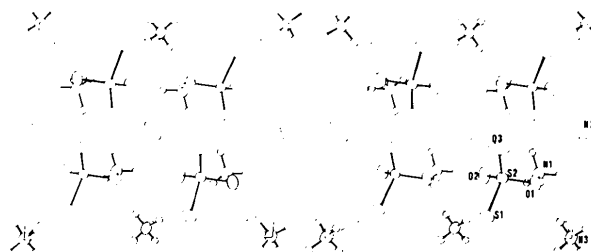| N–H···O	N···O	N–H	H···O
N(3)–H(31)···O(1)	2.897 (3) Å	0.84 (4) Å	2.07 (4) Å
N(3)–H(32)···O(2)	3.086 (3)	1.00 (4)	2.23 (4)

Fig. 1. Stereoscopic view of the unit cell of $(NH_4)_2S_2O_3$.

N—H$\cdots$O distances in $(NH_4)_2S_2O_5$ (Baggio, 1971) and to several interactions in $(NH_4)_2S_2O_8$ (Sivertsen & Sørum, 1969). The S—S distance of 1·979 (1) Å is shorter than the 2·01 (2) Å in $Na_2S_2O_3$ and the 2·024 (4) Å (Lisensky & Levy, 1978) and 2·015 (3) Å (Uraz & Armağan, 1977) in $Na_2S_2O_3.5H_2O$. The average S—O distance in $(NH_4)_2S_2O_3$ (1·471 Å) is slightly longer than the average of 1·467 Å in $Na_2S_2O_3.5H_2O$. The hydrogen-bond system may, via the S—O bonds, have an influence on the S—S bond distances.

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Diammonium Tellurium(VI) Dioxide Tetrahydroxide

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Abstract. $(NH_4)_2TeO_2(OH)_4$, monoclinic, $C2/m$, $a = 8·019$ (2), $b = 6·568$ (1), $c = 6·352$ (2) Å, $\beta = 103·89$ (2)°, $Z = 4$, $V = 324·8$ (1) Å^3, $D_x = 2·70$ Mg m^{-3}, $M_r = 263·70$. Final $R = 0·028$. The $TeO_2(OH)_4^{2-}$ ions are monomeric and connected via hydrogen bonds and the NH_4^+ ions. The coordination of the Te^{VI} atom is octahedral, with Te—OH and Te—O distances of 1·958 (2) and 1·879 (2) Å, respectively.

Introduction. As part of an investigation of the three-component system, $Te(OH)_6$–TeO_2–$NH_3(aq.)$, crystals of $(NH_4)_2TeO_2(OH)_4$ have been prepared. The structures of $(NH_4)_2Te_2O_5.2H_2O$ and $(NH_4)_2Te_4O_9.4H_2O$ (Johansson, 1978a) have been determined. Crystals of ammonium tellurate(VI) were prepared by hydrothermal synthesis. Monoclinic $Te(OH)_6$ was dissolved in an excess of concentrated aqueous ammonia (25%). The solution was placed in an autoclave at 373 K for

48 h; colourless, plate-shaped crystals were obtained. The crystals are not stable in air but could be mounted in glass capillaries in a glove box containing an ammonia atmosphere (Johansson, 1978b). A Syntex $P2_1$ four-circle diffractometer was used for data collection and the integrated intensities were evaluated with the Lehmann & Larsen (1974) profile-analysis method ($LELA$; Lindqvist & Ljungström, 1979). The intensities were corrected for Lorentz and polarization effects with a local program (SYN). Experimental data are listed in Table 1.

The systematic absences $h + k = 2n$ for h, k, l indicated the space group to be $C2$, Cm or $C2/m$. The Te positions were found from a Patterson synthesis and the N and O atoms from successive electron density calculations (DRF; A. Zalkin, Berkeley) in accordance with space group $C2/m$. A preliminary least-squares refinement of the structure with a block-diagonal