

N—H...O distances in $(\text{NH}_4)_2\text{S}_2\text{O}_5$ (Baggio, 1971) and to several interactions in $(\text{NH}_4)_2\text{S}_2\text{O}_8$ (Sivertsen & Sørum, 1969). The S—S distance of 1.979 (1) Å is shorter than the 2.01 (2) Å in $\text{Na}_2\text{S}_2\text{O}_3$ and the 2.024 (4) Å (Lisensky & Levy, 1978) and 2.015 (3) Å (Uraz & Armağan, 1977) in $\text{Na}_2\text{S}_2\text{O}_3 \cdot 5\text{H}_2\text{O}$. The average S—O distance in $(\text{NH}_4)_2\text{S}_2\text{O}_3$ (1.471 Å) is slightly longer than the average of 1.467 Å in $\text{Na}_2\text{S}_2\text{O}_3 \cdot 5\text{H}_2\text{O}$. 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. $(\text{NH}_4)_2\text{TeO}_2(\text{OH})_4$, 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) Å³, $D_x = 2.70$ Mg m⁻³, $M_r = 263.70$. Final $R = 0.028$. The $\text{TeO}_2(\text{OH})_4^{2-}$ 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, $\text{Te}(\text{OH})_6\text{—TeO}_2\text{—NH}_3(\text{aq.})$, crystals of $(\text{NH}_4)_2\text{TeO}_2(\text{OH})_4$ have been prepared. The structures of $(\text{NH}_4)_2\text{Te}_2\text{O}_5 \cdot 2\text{H}_2\text{O}$ and $(\text{NH}_4)_2\text{Te}_4\text{O}_9 \cdot 4\text{H}_2\text{O}$ (Johansson, 1978*a*) have been determined. Crystals of ammonium tellurate(VI) were prepared by hydrothermal synthesis. Monoclinic $\text{Te}(\text{OH})_6$ 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, 1978*b*). 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

approximation (*BLOCK*; Lindgren, 1977) gave $R = 0.068$. After correction for absorption (*DATAP2*; Coppens, Leiserowitz & Rabinovich, 1965) R dropped to 0.050. In the final refinement anisotropic temperature factors and an isotropic extinction parameter were included and the full-matrix least-squares program *LINUS* (Coppens & Hamilton, 1970) was used, giving $R = 0.028$. The positional parameters are given in Table 2.* Scattering factors from Cromer & Waber (1965) were used for Te, those of Doyle & Turner (1968) for O and N. Correction was made for the real part of the anomalous scattering (Cromer, 1965). The

* Lists of structure factors and anisotropic thermal parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 34342 (12 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 1. *Experimental data*

Crystal dimensions	0.04 × 0.31 × 0.31 mm
Radiation	Mo $K\alpha$, $\lambda = 0.71069 \text{ \AA}$
μ	4.73 mm^{-1}
Monochromator	Graphite crystal
Scan type	$\omega/2\theta$
Maximum 2θ angle	50.0°
Scan interval ($\Delta\omega$)	1.2–1.4°
Scan speed (in ω)	1.0–4.0° min^{-1}
Standard reflection	020 at intervals of 25 reflections 860 000 ± 20 000 counts; stable
Recorded reflections	2387
Independent reflections	1714
Significant reflections	1687 $\{ I/\sigma(I) > 3.0\}$
Refinement of cell parameters	15 reflections with $14.1 < 2\theta < 36.7^\circ$

Table 2. *Final atomic parameters in $(\text{NH}_4)_2\text{TeO}_2(\text{OH})_4$*

E.s.d.'s are given in parentheses.

Isotropic extinction parameter (Coppens & Hamilton, 1970)			
$g = 0.24(2) \times 10^4$.			
	x	y	z
Te	0	0	0
O(1)	0.2348 (3)	0	0.1335 (4)
O(2)	-0.0316 (3)	0.2066 (3)	0.2090 (3)
N	0.2079 (5)	$\frac{1}{2}$	0.4187 (5)

Table 3. *Distances (\AA) and angles ($^\circ$) in the $\text{TeO}_2(\text{OH})_4^{2-}$ ion*

Te—O(1)	1.870 (2) × 2	O(1)—Te—O(2)	88.5 (1) × 4
Te—O(2)	1.958 (2) × 4	O(2)—Te—O(2)'	87.8 (1) × 2

Table 4. *Hydrogen bonds (Fig. 1)*

O(2)—H...O(1)	2.649 (3) \AA	Te—O(2)...O(1)	125.6 (1)°	O(1)...N...O(2)	114.3 (1)° × 2
N—H...O(1)	2.771 (4)	Te—O(1)...O(2)	132.3 (1) × 2	O(2)...N...O(2)	86.2 (1)
N—H...O(2)	2.882 (3) × 2	Te—O(1)...N	111.5 (1)	O(2)...N...O(2)'	87.2 (1) × 2
N—H...O(2)'	3.050 (4) × 2 bifurcated	N—O(1)...O(2)	83.0 (1) × 2	O(2)...N...O(2)'	123.9 (1) × 2
		O(2)—O(1)...O(2)	93.3 (1)	O(1)...N...O(2)'	119.1 (1) × 2
				O(2)'\dots N...O(2)'	52.8 (1)

observed reflections were weighted according to Cruickshank (1970): $w = (20.0 + F_o + 0.004F_o^2)^{-1}$.

Discussion. $(\text{NH}_4)_2\text{TeO}_2(\text{OH})_4$ consists of monomeric, octahedral $\text{TeO}_2(\text{OH})_4^{2-}$ ions and NH_4^+ ions held together by hydrogen bonds. The tellurate(VI) octahedra are arranged in layers.

Bond distances and angles (Table 3) were calculated with *DISTAN* (A. Zalkin, Berkeley). The $\text{TeO}_2(\text{OH})_4^{2-}$ octahedron is nearly regular with respect to the O—Te—O angles. Other tellurates(VI) with almost regular octahedral coordination are the monoclinic (Lindqvist & Lehmann, 1973) and cubic (Falck & Lindqvist, 1978) phases of telluric acid, in which the Te—OH distances are 1.909 (1) and 1.913 (6) \AA , respectively. The Te—O_{terminal} and Te—OH bonds in $(\text{NH}_4)_2\text{TeO}_2(\text{OH})_4$ (Table 3) compare reasonably well with the values 1.86 and 1.98 \AA predicted by Lindqvist (1973) for the $\text{TeO}_2(\text{OH})_4^{2-}$ ion. Similar bond lengths have also been found in $\text{Ag}_2\text{TeO}_2(\text{OH})_4$, in which Te—O_{terminal} = 1.85 (9) and Te—OH = 1.95 (8) and 2.00 (5) \AA (Fischer, 1969).

Probable hydrogen bonds are indicated in Table 4. The terminal O(1) acts as a strong hydrogen-bond acceptor giving rise to short O(2)...O(1) and N...O(1) distances. The N—H...O(1) hydrogen bond, 2.771 (4) \AA , is shorter than those found in $(\text{NH}_4)_2\text{SO}_4$,

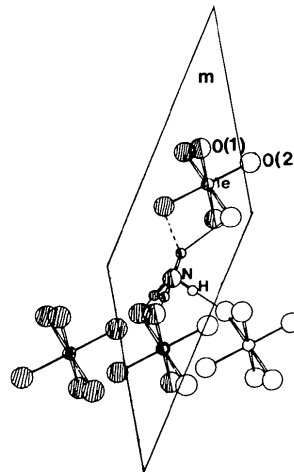


Fig. 1. The mirror plane, $y = \frac{1}{2}$, with the ammonium hydrogen bonds (ORTEP; Johnson, 1965).

2.87–3.04 Å (Hamilton & Ibers, 1968). Because of the mirror plane through N the two N...O contacts of 3.050 (4) Å represent a case of bifurcation (*cf.* Fig. 1).

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Tricarbonyl(1,1-dimethoxy-2,4,6-triphenyl- λ^5 -phosphorin)chrom und (4-*tert*-Butyl-1,1-difluor-2,6-diphenyl- λ^5 -phosphorin)tricarbonylchrom

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Abstract. $C_{28}H_{23}CrO_5P$, $Cr(C_{25}H_{23}O_2P)(CO)_3$, orthorhombic, $P2_12_12_1$, $Z = 4$, $a = 18.655$ (8), $b = 15.550$ (7), $c = 8.529$ (5) Å, $R = 0.114$ for 1247 observed reflexions. $C_{24}H_{21}CrF_2O_3P$, $Cr(C_{21}H_{21}F_2P)(CO)_3$, monoclinic, $P2_1/a$, $Z = 4$, $a = 15.092$ (6), $b = 12.442$ (5), $c = 13.119$ (6) Å, $\beta = 115.1$ (1)°, $R = 0.059$ for 3457 observed reflexions; $\lambda = 0.7107$ Å. In both molecules the P atom is out of the plane of the ring and the $Cr(CO)_3$ group is so oriented that one of the carbonyl groups is above the P atom.

Einleitung. In einer vorangehenden Veröffentlichung haben Lückoff & Dimroth (1976) über bisher unbekannte π -Komplexe einiger λ^5 -Phosphorringe mit Chrom-, Molybdän- und Wolframtricarbonyl berichtet. In dieser Arbeit wird, anhand der Röntgenstrukturanalysen der Tricarbonylchrom-Komplexe von 1,1-Dimethoxy-2,4,6-triphenyl- λ^5 -phosphorin (I) und von 4-*tert*-Butyl-1,1-difluor-2,6-diphenyl- λ^5 -phosphorin (II), der Einfluss verschiedener Substituenten auf die Verschiebung der Elektronen in diesen π -Komplexen untersucht.

Die Gitterkonstante beider Kristalle wurde durch das Suchprogramm des Philips PW 1100 Automatischen

Vier-Kreis-Diffraktometers gefunden und anhand von 25 starken Reflexen weiter verfeinert. 1991 Reflexe von Kristall (I) und 3817 Reflexe von Kristall (II) wurden bei Zimmertemperatur auf einem automatischen Philips PW 1100 Vier-Kreis-Diffraktometer gemessen (Mo $K\alpha$ Strahlung, Graphit-Monochromator, θ - 2θ Abtastung), davon hatten 1247 Reflexe ($\theta_{\max} = 23^\circ$) für (I) und 3457 Reflexe ($\theta_{\max} = 25^\circ$) für (II) eine Intensität grösser als $1,5\sigma(I)$ und wurden als beobachtet gekennzeichnet. Die Strukturbestimmung erfolgte in beiden Fällen nach den direkten Methoden anhand des *MULTAN*-Programmes von Declercq, Germain, Main & Woolfson (1973). Die *E*-Fourier-Synthesen, gerechnet mit den Zeichen und Phasen der wahrscheinlichsten Vorschläge, zeigten in beiden Fällen vollständige Molekülskelette. Die erhaltenen Strukturmodelle wurden anhand der beobachteten Intensitäten und mit Einheitsgewichten nach der Methode der kleinsten Fehlerquadratsumme, mit Hilfe des Blockdiagonalverfahrens des Programms von Ahmed, Hall, Pippy & Huber (1966), so lange verfeinert, bis die Parameterverschiebungen kleiner als die dazugehörigen Standardabweichungen waren. Der auffallend schlechte *R*-Wert für (I) ist durch die schlechte Qualität des Kristalls bedingt.

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