C(25), C(26), O(3), C(6). [Deviations of these nine atoms from the least-squares plane are, respectively, −0·02 (1), −0·01 (1), −0·03 (1), −0·01 (1), −0·00 (1), 0·02 (1), 0·00 (1), 0·01 (1), 0·05 (1) Å.] The phenyl ring on N(2) intersects the Mo, N(2), C(6) plane at 88·8°. The Mo–N(2) bond *trans* to allyl at 2·222 (10) Å is very much shorter than Mo–N(1) *trans* to carbonyl at 2·334 (11) Å.

The room-temperature ^1H NMR spectrum of [Mo(η-allyl)(CO)$_2$(py)(salNPh)] dissolved in $CDCl_3$ is given in Table 3 and is consistent with any of the three possible octahedral structures. However, it is not consistent with a fluxional molecule as reported for the related pd complex, nor with a mixture of isomers. On raising the temperature, the ^1H NMR spectrum simplified and at 343 K the allyl protons H_a and H_s were equivalent in pairs thus indicating a stereochemically non-rigid structure. Hence the ligand arrangement in Fig. 1(*b*) is not peculiar to the pd complex and must reflect the electronic and/or steric requirements of these complexes.

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Structure of an Adduct of Orthotelluric Acid and Urea

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Abstract. $Te(OH)_6.2CO(NH_2)_2$, monoclinic, $C2/c$, a = 14·828 (8), b = 8·891 (6), c = 10·023 (7) Å, β = 129·13 (3)°, Z = 4, D_m = 2·31, D_c = 2·27 Mg m^{-3}. The structure consists of infinite $Te(OH)_6.2CO(NH_2)_2$ layers parallel to (100). Layers are connected through hydrogen bonds (Te–O–H···O–C). Within the layers the $Te(OH)_6$ molecules are hydrogen bonded to neighbouring $Te(OH)_6$ molecules and to urea molecules.

Introduction. The title compound was studied as part of an investigation of oxygen-containing Te compounds. The adduct was prepared by crystallization from a concentrated aqueous solution of orthotelluric acid and urea in a molar ratio of 1:3. Clear crystals were obtained, some as large, nearly regular prisms, of maximum dimensions 12 × 10 × 6 mm; the crystals had well developed faces, (100), (010) and (10$\bar{1}$), the largest being (100), and the smallest (10$\bar{1}$). Intensities were collected ($\omega/2\theta$ scan) in the range $3 \le 2\theta \le 62°$ on a Stoe four-circle diffractometer with graphite-monochromated Mo $K\alpha$ radiation. 1523 reflections were measured; those with $I \le 3\sigma(I)$ were treated as unobserved. The remaining 1498 reflections were corrected for Lp and absorption. The positions of Te atoms were obtained from a Patterson synthesis. The remaining non-H atoms were located by Fourier and ΔF syntheses and refined by least squares to R = 0·095 with isotropic and to R = 0·070 with anisotropic temperature factors. The positions of the H atoms have not been determined. The final positional parameters are listed in Table 1.* The calculations were performed with *SHELX* 76 (Sheldrick, 1976) on an IBM 370/168 computer. The scattering factor for Te, which is not

* Lists of structure factors and anisotropic thermal parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 34621 (11 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Table 1. *Fractional atomic coordinates* ($\times 10^4$) *with e.s.d.'s in parentheses*

	x	y	z
Te	0	4322 (0)	2500
O(1)	1202 (6)	4274 (4)	4993 (8)
O(2)	798 (6)	2872 (6)	2187 (8)
O(3)	805 (6)	5895 (7)	2328 (9)
O(4)	1620 (4)	341 (5)	3907 (6)
C	1426 (4)	−215 (8)	4852 (7)
N(1)	1267 (7)	−1705 (7)	4893 (10)
N(2)	1413 (10)	708 (5)	5950 (13)

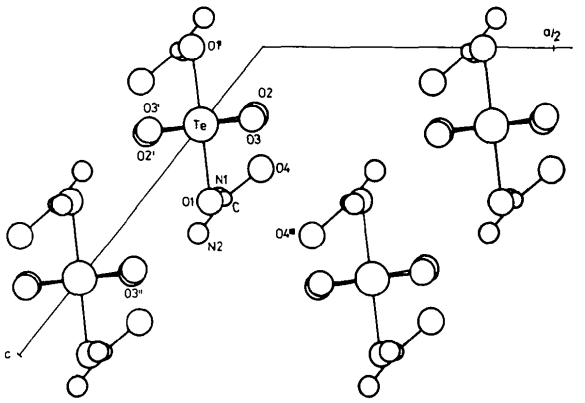

Fig. 1. Projection of the structure along [010].

stored in the program, was taken from Cromer & Mann (1968). The projection of the structure along [010] is depicted in Fig. 1.

Discussion. The intermolecular distances and angles, shown in Table 2, appear normal. The orthotellurate group forms a distorted octahedron, average Te–O distance 1·92 Å, O–Te–O angle 90°. For comparison the mean Te–O distance in cubic orthotelluric acid is 1·913 Å (X-ray data; Falck & Lindqvist, 1978), or 1·93 Å (neutron data; Cohen-Addad, 1971); in monoclinic orthotelluric acid it is 1·916 Å (X-ray data; Lindqvist, 1970), or 1·909 Å (neutron data; Lindqvist & Lehmann, 1973). The practically planar urea group shows a shortened C–O distance and lengthened C–N distances; in urea itself the C–O and C–N distances are 1·257 and 1·340 Å respectively (Mullen & Hellner, 1978).

The hydrogen-bond network, which is responsible for the close packing in this structure, is very involved. Each tellurate O atom forms two hydrogen bonds. In the layer the tellurate O atoms form hydrogen bonds (2·755 Å) similar to those in the orthotelluric acids (2·58–2·773 Å). Another tellurate O atom forms the shortest hydrogen bond (2·621 Å) with the O atom of the urea, and one with a N atom (2·977 Å). A further

Table 2. *Interatomic distances* (Å) *and angles* (°) *and intermolecular distances* (Å) <3·36 Å *with e.s.d.'s in parentheses*

Te–O(1)	1·950 (4)	O(1)–Te–O(2)	92·0 (3)
–O(2)	1·905 (6)	–O(3)	90·8 (3)
–O(3)	1·916 (6)	–O(2^{i})	86·2 (4)
C–O(4)	1·252 (7)	–O(3^{i})	91·0 (3)
–N(1)	1·351 (7)	–O(1^{i})	177·5 (4)
–N(2)	1·383 (10)	O(2)–Te–O(3)	89·5 (4)
O(1)–O(3^{ii})	2·755 (10)	–O(2^{i})	94·8 (4)
–O(4^{iii})	2·696 (7)	–O(3^{i})	174·8 (4)
–N(2)	3·270 (5)	O(3)–Te–O(3^{i})	86·2 (4)
O(2)–O(4)	2·621 (5)	O(4)–C–N(1)	122·5 (5)
–N(1^{iv})	2·977 (11)	–N(2)	119·5 (5)
O(3)–N(1^{v})	3·068 (8)	N(1)–C–N(2)	117·9 (6)
–N(1^{vi})	3·212 (6)		
O(4)–N(2^{iv})	2·933 (11)		

Symmetry code

(i)	$\bar{x}, \quad y, \quad \frac{1}{2}-z$	(iv)	$x, \quad \bar{y}, \quad z-\frac{1}{2}$
(ii)	$x, \quad 1-y, \quad \frac{1}{2}+z$	(v)	$x, \quad 1+y, \quad z$
(iii)	$\frac{1}{2}-x, \quad \frac{1}{2}-y, \quad 1-z$	(vi)	$\bar{x}, \quad 1+y, \quad \frac{1}{2}-z$

tellurate O atom forms one hydrogen bond with a tellurate O atom of the nearest tellurate group (2·755 Å) and one with a N atom (3·068 Å). The urea O atom forms the following hydrogen bonds: one with a tellurate O atom (2·621 Å) and one with a N atom of another urea molecule (2·933 Å). All these hydrogen bonds bind the urea molecules and the orthotelluric acid molecules in the layer parallel to (100). The layers are connected through the hydrogen bond from the urea O atom to the tellurate O atom (2·696 Å).

Urea forms analogous compounds with nitric and phosphoric acids: $HNO_3.CO(NH_2)_2$ and $H_3PO_4.CO(NH_2)_2$. The O–C, C–N(1), C–N(2) distances in urea nitrate are 1·30, 1·30, 1·31 Å (mean values, X-ray data; Harkema & Feil, 1969), or 1·298, 1·312, 1·315 Å (neutron data; Worsham & Busing, 1969). In urea phosphate the distances are 1·290, 1·323, 1·340 Å (X-ray data; Sundera-Rao, Turley & Pepinsky, 1957), 1·27, 1·35, 1·41 Å (X-ray data; Wolfram, Arutunian, Antishkina & Porai-Koshits, 1967), or 1·278, 1·312, 1·320 Å (neutron data; Nozik, Fykin, Bukin & Muradjan, 1976).

Orthotelluric acid forms adducts with alkali fluorides which possess short O–H···F hydrogen bonds. In $Te(OH)_6.2KF$ (Allmann & Haase, 1976) the $Te(OH)_6$ octahedron is nearly ideal: the average Te–O distance is 1·905 Å and the average O–Te–O angle 90°. In $Te(OH)_6.NaF$ (Allmann, 1976) the $Te(OH)_6$ octahedra are slightly distorted, mean Te–O distance 1·92 Å. In $Te(OH)_6.2CsF.2H_2O$ (Allmann & Rius, 1978) the average Te–O distance is 1·91 Å.

From systematic absences it is not possible to distinguish between the space groups $C2/c$ and Cc. Therefore in parallel with calculations for $C2/c$, calculations for Cc were carried out. The temperature

factors of some atoms in *Cc* were not positive definite and several interatomic distances were improbable. The non-coincidence of the Raman and IR spectra also indicates *C*2/*c* (Loub, 1979).

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Structure of Dithiobisformamidinium *trans*-Aquatetrachlorooxorhenate(V) Chloride Monohydrate

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Abstract. $[(NH_2)_2CSSC(NH_2)_2][ReCl_4(H_2O)O]Cl.H_2O$, $C_2H_8N_4S_2^{2+}.Cl_4H_2O_2Re^-.Cl^-.H_2O$, monoclinic, $P2_1/c$, $a = 13{\cdot}512\,(8)$, $b = 7{\cdot}025\,(4)$, $c = 16{\cdot}230\,(9)$ Å, $\beta = 100{\cdot}68\,(4)°$, $M_r = 567{\cdot}8$, $V = 1513{\cdot}9$ Å^3, $Z = 4$, $D_m = 2{\cdot}49$, $D_x = 2{\cdot}49$ Mg m^{-3}, λ(Mo $K\alpha$) = 0·71069 Å, $\mu = 9{\cdot}63$ mm^{-1}; final $R = 0{\cdot}038$ and $R_w = 0{\cdot}032$ for 1656 reflexions. The crystals are composed of dithiobisformamidinium cations, *trans*-aquatetrachlorooxorhenate(V) anions, chlorine anions and water molecules. The bond lengths in the complex anion are Re—O 1·66 (1), Re—H_2O 2·20 (2) and Re—Cl 2·335–2·373 Å; the O—Re—H_2O bond angle is 176·4 (5)°. The disulphide group has the normal configuration and dimensions, with an S—S distance of 2·03 (1) Å and CSS/SSC dihedral angle of 89°. The thiourea groups are planar. Isolation of the title compound as well as $[(NH_2)_2CSSC(NH_2)_2]Cl_2$ indicates that salts of formamidine disulphide can be obtained from thiourea by the action of an oxidizing agent such as perrhenate anions in acidic solutions.

Introduction. The interactions between perrhenate ions and thiourea have been studied in the system (I), $SnCl_2$ + ReO_4^- + $(NH_2)_2CS$ + HCl, by several authors (Ryabchikov & Lazarev, 1955; Ryabchikov, 1962; Borisova & Kariakin, 1967; Morpurgo, 1968; Borisova, 1969; Kuznetsov, Novitskaia, Koz'min & Borisova, 1973). The present author (Lis, 1976, 1977) has reported that in the system (II), ReO_4^- + $(NH_2)_2CS$ + HCl, the redox reaction between the perrhenate ions and thiourea also proceeds. In this simpler system several coloured Re compounds can be easily isolated depending on the HCl concentration. In addition to two earlier characterized complexes of formulae $[ReCl_2(H_2O)Otu_2]Cl$ and $[ReCl_3(H_2O)Otu]$ the author has isolated five further complexes. One of these is the yellow-green title compound, for which preliminary data have been reported (Lis, 1978); full single-crystal X-ray data are reported here.

In system (II) thiourea is the reducing agent and is oxidized to formamidine disulphide. Formamidine disulphide either precipitates in crystalline form as a by-product (dihydrochloride) or forms mixed crystals with Re complexes.

The title compound was obtained as follows: to 0·1 g $KReO_4$ and 0·1 g $(NH_2)_2CS$, 10–20 ml of concentrated HCl was added. The solution was left in a desiccator over P_2O_5. After one day dark-green crystals

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