

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 *C2/c* (Loub, 1979).

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

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**Abstract.** [(NH<sub>2</sub>)<sub>2</sub>CSSC(NH<sub>2</sub>)<sub>2</sub>][ReCl<sub>4</sub>(H<sub>2</sub>O)O]·Cl·H<sub>2</sub>O, C<sub>2</sub>H<sub>8</sub>N<sub>4</sub>S<sub>2</sub><sup>2+</sup>·Cl<sub>4</sub>H<sub>2</sub>O<sub>2</sub>Re<sup>-</sup>·Cl<sup>-</sup>·H<sub>2</sub>O, monoclinic, *P*2<sub>1</sub>/*c*, *a* = 13.512 (8), *b* = 7.025 (4), *c* = 16.230 (9) Å, β = 100.68 (4)°, *M<sub>r</sub>* = 567.8, *V* = 1513.9 Å<sup>3</sup>, *Z* = 4, *D<sub>m</sub>* = 2.49, *D<sub>x</sub>* = 2.49 Mg m<sup>-3</sup>, λ(Mo *K*α) = 0.71069 Å, μ = 9.63 mm<sup>-1</sup>; final *R* = 0.038 and *R<sub>w</sub>* = 0.032 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<sub>2</sub>O 2.20 (2) and Re—Cl 2.335–2.373 Å; the O—Re—H<sub>2</sub>O 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<sub>2</sub>)<sub>2</sub>CSSC(NH<sub>2</sub>)<sub>2</sub>]Cl<sub>2</sub> 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<sub>2</sub> + ReO<sub>4</sub><sup>-</sup> + (NH<sub>2</sub>)<sub>2</sub>CS + 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<sub>4</sub><sup>-</sup> + (NH<sub>2</sub>)<sub>2</sub>CS + 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<sub>2</sub>(H<sub>2</sub>O)Otu<sub>2</sub>]Cl and [ReCl<sub>3</sub>(H<sub>2</sub>O)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<sub>4</sub> and 0.1 g (NH<sub>2</sub>)<sub>2</sub>CS, 10–20 ml of concentrated HCl was added. The solution was left in a desiccator over P<sub>2</sub>O<sub>5</sub>. After one day dark-green crystals

of [ReCl<sub>3</sub>(H<sub>2</sub>O)Otu] and white crystals of [(NH<sub>2</sub>)<sub>2</sub>CSSC(NH<sub>2</sub>)<sub>2</sub>]Cl<sub>2</sub> precipitated. Upon further standing the dark-green crystals of [ReCl<sub>3</sub>(H<sub>2</sub>O)Otu] dissolved and after 1–2 weeks other (yellow-green and yellow) crystalline compounds precipitated. The yellow-green platy crystals were investigated in this work; they are rather unstable in air but quite stable in paraffin oil. Examination of Weissenberg photographs showed that the crystals are monoclinic; systematic extinctions determined the space group to be *P*2<sub>1</sub>/*c*. A crystal of approximate dimensions 0.07 × 0.11 × 0.06 mm was selected for the data collection. A Syntex P2<sub>1</sub> diffractometer and Mo *K*α radiation with a graphite monochromator were used for lattice-parameter and intensity measurements by the  $\theta$ -2 $\theta$  scan technique. After each group of 40 reflexions, the intensity of a standard reflexion was measured; no significant change in intensity was observed. The data were corrected for Lorentz and polarization effects but not for absorption or extinction. Of 3402 reflexions accessible below 2 $\theta$  = 55°, 1656 with  $I > 3\sigma(I)$  were used for structure determination. All calculations were performed on a Nova 1200 computer with the Syntex XTL structure determination system. The scattering factors used for neutral atoms were those tabulated by D. T. Cromer and J. T. Waber (*International Tables for X-ray Crystallography*, 1974).

The approximate coordinates of the Re atom were determined from the Patterson map. A three-dimensional electron density map calculated with signs based on Re showed peaks which were identified as Cl and S atoms. A difference-Fourier synthesis at this point revealed the positions of nine peaks of similar density which were used for determination of the light-atom positions; *R* then dropped to 0.10. The compound was identified as [(NH<sub>2</sub>)<sub>2</sub>CSSC(NH<sub>2</sub>)<sub>2</sub>][ReCl<sub>4</sub>(H<sub>2</sub>O)O]Cl · H<sub>2</sub>O. This structure was then refined by least-squares techniques, first with isotropic, then with anisotropic thermal parameters, to *R* = 0.039 and *R*<sub>w</sub> = 0.034. Eight H atoms (bonded to N atoms) were placed in geometrically calculated positions at a distance of 1.0 Å from the bonded atoms. Four remaining H atoms were found from a difference-Fourier synthesis. Further refinement with fixed parameters for H atoms reduced *R* to 0.038 and *R*<sub>w</sub> to 0.032. The final three-dimensional difference-Fourier synthesis was featureless.\*

**Discussion.** The final atomic coordinates and their estimated standard deviations are listed in Table 1. Crystals of [(NH<sub>2</sub>)<sub>2</sub>CSSC(NH<sub>2</sub>)<sub>2</sub>][ReCl<sub>4</sub>(H<sub>2</sub>O)O]Cl · H<sub>2</sub>O are composed of *trans*-aquatetrachlorooxo-

\* Lists of structure factors and anisotropic thermal parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 34678 (35 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 1. Final positional parameters of the title compound

The H-atom positions are unrefined (all *B* = 3.0 Å<sup>2</sup>).

	<i>x</i>	<i>y</i>	<i>z</i>
Re	0.20018 (4)	0.25924 (13)	0.47864 (3)
Cl(1)	0.2519 (3)	0.2384 (8)	0.6263 (2)
Cl(2)	0.1841 (3)	0.2377 (9)	0.3320 (2)
Cl(3)	0.1254 (3)	-0.0457 (6)	0.4793 (3)
Cl(4)	0.3166 (4)	0.5052 (6)	0.4790 (3)
Cl(5)	0.5152 (3)	0.1561 (5)	0.3959 (2)
O(1)	0.0963 (7)	0.3834 (15)	0.4814 (6)
O(2)	0.3328 (8)	0.0794 (14)	0.4774 (7)
O(3)	0.0301 (7)	0.1003 (16)	0.1585 (6)
S(1)	0.2491 (2)	0.7515 (8)	0.1701 (2)
S(2)	0.3526 (3)	0.8654 (6)	0.2638 (3)
C(1)	0.1435 (9)	0.6991 (18)	0.2188 (8)
C(2)	0.4197 (9)	0.6566 (21)	0.3096 (9)
N(1)	0.1433 (7)	0.7478 (25)	0.2965 (6)
N(2)	0.0690 (8)	0.6133 (18)	0.1706 (8)
N(3)	0.4956 (8)	0.6974 (17)	0.3702 (7)
N(4)	0.3952 (8)	0.4889 (15)	0.2855 (7)
H(11)	0.084	0.717	0.323
H(12)	0.203	0.816	0.330
H(21)	0.007	0.579	0.192
H(22)	0.074	0.582	0.111
H(31)	0.538	0.592	0.400
H(32)	0.511	0.832	0.387
H(41)	0.436	0.373	0.313
H(42)	0.334	0.465	0.237
H(1)	0.350	0.100	0.425
H(2)	0.375	-0.050	0.500
H(3)	0.075	0.150	0.150
H(4)	0.000	0.050	0.100

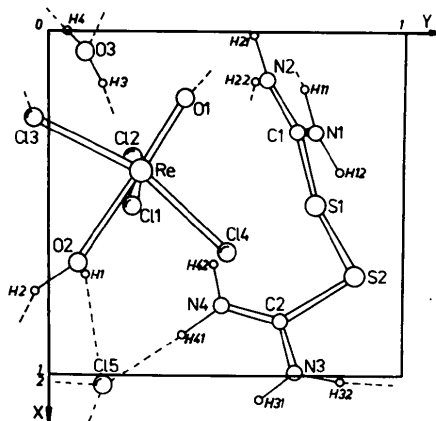


Fig. 1. The crystal structure projected on the (001) plane.

rhenate(V) anions, Cl<sup>-</sup> anions, dithiobisformamidinium cations and water of crystallization. The arrangement of the molecules in projection on the (001) plane is shown in Fig. 1. The more important interatomic distances and bond angles are listed in Table 2.

In the *trans*-aquatetrachlorooxorhenate(V) anion, the six bonded ligand atoms form a distorted octahedron about the Re atom. The Re atom does not lie in

the 'best plane' defined by the four Cl atoms but is displaced in the direction of the oxo O atom by 0.32 Å. This distortion is also indicated by the O—Re—Cl and H<sub>2</sub>O—Re—Cl apical angles which vary within the ranges 95.6 (4)–100.6 (4)° and 80.2 (3)–83.4 (3)° respectively. The O—Re—H<sub>2</sub>O bond angle of 176.4 (5)° deviates slightly from linearity. Furthermore, the Re—Cl distances are not quite identical and vary from 2.335 (4) to 2.373 (3) Å, but all interatomic

distances and angles in the [ReCl<sub>4</sub>(H<sub>2</sub>O)O]<sup>−</sup> complex anion are unexceptional and agree well with values reported for other monooxo complexes of rhenium(V) (see Table 3).

A comparison of the bond-distance and bond-angle values in the *trans*-aquatetrachlorooxorhenate(V) ion, [ReCl<sub>4</sub>(H<sub>2</sub>O)O]<sup>−</sup>, and the *trans*-aquatetrachlorooxorhenium(VI) molecule, [ReCl<sub>4</sub>(H<sub>2</sub>O)O] (Frais & Lock, 1972), is informative (see Table 3). Both are distorted octahedra with an oxo O atom and a water molecule in *trans* positions and vary only in the oxidation state of Re. Although the difference in the Re—O(oxo) distances may be classified as experimental uncertainty, the difference of 0.07 Å in the average Re—Cl and Re—O(aqua) bond lengths is significant. A similar significant difference in the Re—Cl distances was observed earlier for another pair: [ReCl<sub>4</sub>O] (Edwards, 1972) and [ReCl<sub>4</sub>O]<sup>−</sup> (Lis & Jezowska-Trzebiatowska, 1977).

The dimensions of the formamidine disulphide unit are listed in Table 2. The S—S bond length of 2.030 (5) Å is in agreement with those found in [(NH<sub>2</sub>)<sub>2</sub>CSSC(NH<sub>2</sub>)<sub>2</sub>]Br<sub>2</sub>·H<sub>2</sub>O and [(NH<sub>2</sub>)<sub>2</sub>CSSC(NH<sub>2</sub>)<sub>2</sub>]I<sub>2</sub>·H<sub>2</sub>O (Foss, Johnsen & Tvedten, 1958) and [(NH<sub>2</sub>)<sub>2</sub>CSSC(NH<sub>2</sub>)<sub>2</sub>]Cl<sub>2</sub> (Villa, Manfredotti, Nardelli & Tani, 1972). Also, the S valency angles and the dihedral angle of the disulphide group (88.8°) lie in the normal range. The thiourea groups are quite planar. The mean value for S—C, 1.80 (2) Å, is similar to that in the bromide, iodide and chloride salts. The C—N distances [1.307 (16), 1.302 (17), 1.315 (17), 1.266 (18) Å] do

Table 2. Selected intramolecular distances (Å) and angles (°) in the title compound

Re—O(1)	1.660 (9)	Re—O(2)	2.195 (10)
Re—Cl(1)	2.373 (3)	Re—Cl(2)	2.354 (3)
Re—Cl(3)	2.369 (4)	Re—Cl(4)	2.335 (4)
S(1)—C(1)	1.793 (13)	S(2)—C(2)	1.809 (14)
C(1)—N(1)	1.307 (16)	C(2)—N(3)	1.315 (17)
C(1)—N(2)	1.302 (17)	C(2)—N(4)	1.266 (18)
S(1)—S(2)	2.030 (5)		
O(1)—Re—O(2)	176.4 (5)	O(1)—Re—Cl(2)	98.1 (4)
O(1)—Re—Cl(1)	95.6 (4)	O(1)—Re—Cl(4)	100.6 (4)
O(1)—Re—Cl(3)	96.4 (4)	O(2)—Re—Cl(2)	82.9 (3)
O(2)—Re—Cl(1)	83.4 (3)	O(2)—Re—Cl(4)	82.8 (3)
O(2)—Re—Cl(3)	80.2 (3)	Cl(1)—Re—Cl(3)	89.2 (2)
Cl(1)—Re—Cl(2)	166.3 (2)	Cl(2)—Re—Cl(3)	89.3 (2)
Cl(1)—Re—Cl(4)	88.3 (2)	Cl(3)—Re—Cl(4)	163.0 (2)
Cl(2)—Re—Cl(4)	89.2 (2)	S(1)—S(2)—C(2)	102.2 (5)
S(2)—S(1)—C(1)	104.2 (5)	S(2)—C(2)—N(3)	113.1 (10)
S(1)—C(1)—N(1)	121.1 (10)	S(2)—C(2)—N(4)	123.1 (11)
S(1)—C(1)—N(2)	114.8 (10)	N(3)—C(2)—N(4)	123.9 (13)
N(1)—C(1)—N(2)	124.1 (13)		

Table 3. Comparison of bond distances (Å) in six coordinated monooxo complexes of rhenium(V) and [ReCl<sub>4</sub>(H<sub>2</sub>O)O]

Compound	Re—O	Re—H <sub>2</sub> O	Re—Cl	Reference
(C <sub>2</sub> H <sub>8</sub> N <sub>4</sub> S <sub>2</sub> )[ReCl <sub>4</sub> (H <sub>2</sub> O)O]Cl·H <sub>2</sub> O	1.660 (9)	2.195 (10)	2.335–2.373	This paper
[ReCl <sub>4</sub> O(PEt <sub>2</sub> Ph) <sub>2</sub> ]	1.60		2.41–2.47	Ehrlich & Owston (1963)
[As(C <sub>2</sub> H <sub>5</sub> ) <sub>4</sub> ][ReBr <sub>4</sub> (CH <sub>3</sub> CN)O]	1.73 (6)			Cotton & Lippard (1966)
[N(C <sub>2</sub> H <sub>5</sub> ) <sub>4</sub> ][ReBr <sub>4</sub> (H <sub>2</sub> O)O]	1.71 (4)	2.32 (4)		Cotton & Lippard (1965)
[Re(C <sub>2</sub> H <sub>5</sub> N <sub>2</sub> Cl <sub>2</sub> O(OEt))]	1.684 (7)		2.366–2.441	Lock & Turner (1977)
[Re(C <sub>2</sub> H <sub>5</sub> O <sub>2</sub> )Cl <sub>2</sub> O{P(C <sub>6</sub> H <sub>5</sub> ) <sub>3</sub> }]	1.69 (1)		2.339–2.376	Lock & Che'ng Wan (1975)
[ReCl <sub>3</sub> (H <sub>2</sub> O)Ot <sub>u</sub> ]	1.713 (19)	2.291 (20)	2.347–2.425	Lis (1977)
[ReCl <sub>2</sub> (H <sub>2</sub> O)Ot <sub>u</sub> ] <sub>2</sub> Cl	1.654 (10)	2.231 (10)	2.390–2.408	Lis (1976)
K <sub>2</sub> [ReCl <sub>5</sub> O]	1.651 (21)		2.372–2.503	Głowiak (1976)
[H <sub>2</sub> en][ReCl <sub>5</sub> O]	1.689 (16)		2.373–2.510	Głowiak (1976)
[ReCl <sub>4</sub> (H <sub>2</sub> O)O]	1.63 (2)	2.27 (2)	2.28–2.30	Frais & Lock (1972)

Table 4. Probable hydrogen bonds

X—H...Y	X...Y	X—H	H...Y	∠X—H...Y
N(1)—H(11)...O(3)[−x, ½ + y, ½ − z]	2.781 (14) Å	1.00 Å	1.82 Å	160°
N(2)—H(22)...Cl(3)[x, ½ − y, z − ½]	3.364 (12)	1.01	2.38	165
N(3)—H(32)...Cl(5)[x, 1 + y, z]	3.253 (12)	1.00	2.28	166
N(4)—H(41)...Cl(5)	3.198 (11)	1.04	2.18	168
O(2)—H(1)...Cl(5)	3.053 (11)	0.93	2.40	127
O(2)—H(2)...Cl(5)[1 − x, −y, 1 − z]	3.102 (11)	1.10	2.16	142
O(3)—H(3)...Cl(1)[x, ½ − y, z − ½]	3.334 (10)	0.74	2.61	169
O(3)—H(4)...O(1)[−x, y − ½, ½ − z]	2.993 (13)	1.02	2.04	153

not differ significantly. It can be assumed that formamidine disulphide exists as a diprotonated cation, and not in the monoprotated form, and that the crystals contain water of crystallization and not H<sub>3</sub>O<sup>+</sup> ions, as was reported earlier (Lis, 1978).

The structure is held together by weak hydrogen bonds; their lengths and angles are given in Table 4.

In the present and earlier investigations (Lis, 1976, 1977, 1978) it was found that in the system ReO<sub>4</sub><sup>-</sup> + (NH<sub>2</sub>)<sub>2</sub>CS + HCl there is a redox reaction between ReO<sub>4</sub><sup>-</sup> ions and thiourea molecules. It was observed that, depending on the thiourea concentrations, quite different Re complexes may be obtained. The reaction is postulated to proceed stepwise. At first, if the HCl concentration is relatively small, the octahedral yellow-brown complex of Re<sup>VII</sup> is formed. Next, as the HCl concentration increases, the Re<sup>7+</sup> ions are reduced by thiourea to Re<sup>5+</sup> ions and thiourea is transformed into dithiobisformamidinium dichloride. At this stage, some coloured Re complexes may be easily isolated in the crystalline state, depending on the HCl and thiourea concentrations. Further, at an extreme concentration of HCl and with excess thiourea the reduction most probably stops at the Re<sup>4+</sup> complexes.

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## Tetramethylammonium 2-( $\eta$ -Cyclopentadienyl)-1-carba-2-cobalta-closo-undecahydro-dodecaborate(1-)

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**Abstract.** [N(CH<sub>3</sub>)<sub>4</sub>][( $\eta$ -C<sub>5</sub>H<sub>5</sub>)CoCB<sub>10</sub>H<sub>11</sub>], C<sub>4</sub>H<sub>12</sub>N<sup>+</sup>.C<sub>6</sub>H<sub>16</sub>B<sub>10</sub>Co<sup>-</sup>, *M<sub>r</sub>* = 329.48, orthorhombic, *Pbcm*, *a* = 8.891 (1), *b* = 15.934 (6), *c* = 12.808 (1) Å, *V* = 1814.5 (7) Å<sup>3</sup>, *Z* = 4, *D<sub>x</sub>* = 1.206, *D<sub>m</sub>* = 1.204 Mg m<sup>-3</sup> (by flotation),  $\mu$ (Mo *K*α) = 0.93 mm<sup>-1</sup>. The

structure was refined to *R* = 0.045 for 1568 counter reflections. The anion can be described as an icosahedron with one Co, one C and ten B atoms at its vertices. The anion and cation exhibit crystallographic symmetry *m* and 2 respectively.