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The Crystal Structures of Tris(selenourea) Dichloride Hydrate and Tris(selenourea) Dibromide Hydrate

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The crystal structures of tris(selenourea) dichloride hydrate, $(su)_3Cl_2.H_2O$, and tris(selenourea) dibromide hydrate, $(su)_3Br_2.H_2O$, where $su = SeC(NH_2)_2$, have been determined by X-ray methods, and refined by full-matrix least squares procedures. The dichloride and the dibromide are isomorphous, space group *Pbca* (No. 61) with eight formula units per unit cell. The dichloride has the cell dimensions, $a = 12.202(5)$ Å, $b = 18.142(7)$ Å, $c = 12.232(5)$ Å. The cell dimensions of the dibromide are, $a = 12.604(5)$ Å, $b = 18.268(7)$ Å and $c = 12.512(5)$ Å.

The tris(selenourea) ion possesses a nearly linear three-selenium system with Se – Se – Se bond angle 173.8°, and Se – Se bond lengths from 2.597(2) Å to 2.717(2) Å.

The atoms in each selenourea group are co-planar, and the three selenourea groups of the ion are nearly parallel, with dihedral angles of 5° to 15°. The three selenium atoms and the carbon atoms of the two terminal selenourea groups are nearly co-planar, and so are the three selenium atoms and the carbon atom of the middle selenourea group. These two planes make an angle of about 75° with each other. The selenium-carbon bonds are nearly normal to the three-selenium sequence.

Tris(selenourea) dichloride hydrate, $(su)_3Cl_2.H_2O$, and the analogous dibromide, $(su)_3Br_2.H_2O$, were first prepared by Verneuil [1–3] by air oxidation of an aqueous solution of selenourea containing hydrochloric or hydrobromic acid, respectively. The cation may be regarded as the adduct of selenourea and its oxidative dimerization product, the formamidinium diselenide cation:

$$2\ SeC(NH_2)_2 = [SeC(NH_2)_2]^{2+} + 2\ e,\ E^\circ = 0.24\ V\ [4]$$

$$[SeC(NH_2)_2]_2^{2+} + SeC(NH_2)_2 = [SeC(NH_2)_2]_3^{2+}$$

The reaction is analogous to the formation reaction of the trihalide ions and the pseudo-trihalide ion, $(SeCN)_3^-$.[5,6]

Preparative and crystallographic data on the compounds have been reported earlier.[7]

CRYSTAL DATA

The crystals of tris(selenourea) dichloride hydrate, $(su)_3Cl_2.H_2O$, and of the analogous dibromide, $(su)_3Br_2.H_2O$, are isomorphous. Both crystallized as red brown orthorhombic prisms elongated along the *a* axis. The unit cell dimensions were determined from zero-layer Weissenberg photographs around the three axes. Seventy observations for each compound were measured and evaluated by means of a least squares program. Intensities were estimated visually from integrated Weissenberg photographs around the *a* and *c* axes, taken with $CuK\alpha$ radiation using the multi-film technique.

Data of $(su)_3Cl_2.H_2O$:
Space group *Pbca* (No. 61), $a = 12.202(5)$ Å, $b = 18.142(7)$ Å, $c = 12.232(5)$ Å, $Z = 8$. $D_m = 2.25$ g/cm³, $D_x = 2.26$ g/cm³.
Crystal I: $0.154 \times 0.096 \times 0.096$ mm³, $0kl - 7kl$,
Crystal II: $0.250 \times 0.144 \times 0.176$ mm³, $hk0 - hk1$,
number of reflections 1731 of 2129 possible, $\mu = 146$ cm⁻¹.

Data of $(su)_3Br_2.H_2O$:
Space group *Pbca* (No. 61), $a = 12.604(5)$ Å, $b = 18.268(7)$ Å, $c = 12.512(5)$ Å, $Z = 8$. $D_m = 2.52$ g/cm³, $D_x = 2.55$ g/cm³.
Crystal I: $0.132 \times 0.110 \times 0.086$ mm³, $0kl - 7kl$
Crystal II: $0.080 \times 0.110 \times 0.090$ mm³, $hk0 - hk1$
number of reflections 1701 of 2401 possible, $\mu = 177.8$ cm⁻¹.

Intensities were corrected for absorption [8] and extinction.[9]

THE STRUCTURE ANALYSES

The two structures were solved by Patterson and Fourier methods, and refined by full-matrix least-squares program minimizing the

function $r = \sum W(|F_o| - K|F_c|)$,[2] where K is the scale factor and $W = 1/[Ka_1 + (a_2F_o)^2/4W_o + (a_3F_o)^4]$. The weight W_o is based on the estimated reliability of the film readings. At the start of the refinement the values of a_1, a_2, and a_3 were chosen to be 0.8, 0.05, and 0.004, respectively, for both structures. Cruickshank[10] has suggested to check the weighting scheme by checking that $\sum W(\Delta F)^2/N$ is constant in groups of increasing $|F|$ or $\sin\theta/\lambda$. In this work the structure factors were divided into five groups with $\sin^3\theta_n - \sin^3\theta_{n-1}$ equal for all groups, and the weighting parameters were varied. In the structure of the dichloride the final choice of the parameters were: $a_1 = 0.8$, $a_2 = 0.15$, and $a_3 = 0.004$. The difference between the highest and the lowest value of $\sum W(\Delta F)^2/N$ was then 15 %. In the case of the dibromide the original weighting parameters seemed to fit well and were not changed. The difference between the highest and the lowest value of $\sum W(\Delta F)^2/N$ was 16 %.

The final refinements brought the reliability index, R, to 0.055 for the dichloride and to 0.062 for the dibromide. The difference map of the dibromide showed a peak of 4e/Å^3 at the position of one of the selenium atoms, Se_1. Apart from this peak, the peaks in both difference maps were 0.4 – 0.8 e/Å^3 and were found in positions expected for hydrogen atoms.

Computational procedures and programs used have been described earlier.[11]

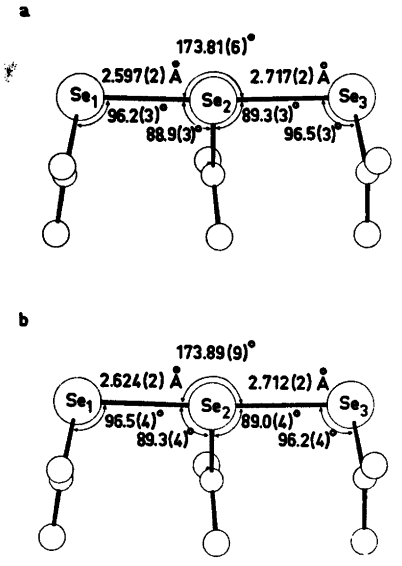

Fig. 1. The triselenourea ion in $(su)_3Cl_2.H_2O$ (a) and in $(su)_3Br_2.H_2O$ (b), as seen normal to the plane passing through Se_1, Se_3, and a point which is equidistant from C_2 and the middle point between C_1 and C_3.

Table 1. Atomic coordinates for tris(selenourea) dichloride hydrate and for tris(selenourea) dibromide hydrate, in fractions of orthorhombic cell edges, with origin at a centre of symmetry. Standard deviations from least squares are given in parentheses.

	x	y	z
$[SeC(NH_2)_2]_3Cl_2.H_2O$			
Se_1	0.28088(9)	0.27125(4)	0.16569(7)
Se_2	0.25241(8)	0.41281(5)	0.17943(6)
Se_3	0.24471(8)	0.56245(5)	0.18459(6)
Cl_1	0.06050(18)	0.10427(11)	0.47303(15)
Cl_2	0.05636(19)	0.39622(11)	0.46484(16)
C_1	0.1878(8)	0.2458(4)	0.2866(6)
C_2	0.3534(8)	0.4146(3)	0.3019(6)
C_3	0.1424(8)	0.5702(4)	0.3017(5)
N_1	0.0830(7)	0.2377(4)	0.2691(6)
N_2	0.2349(7)	0.2363(4)	0.3815(5)
N_3	0.4593(7)	0.4155(3)	0.2819(6)
N_4	0.3161(6)	0.4166(3)	0.4001(5)
N_5	0.0375(7)	0.5761(4)	0.2806(6)
N_6	0.1761(6)	0.5689(4)	0.4021(5)
O	0.4661(6)	0.2472(4)	0.4327(5)
$[SeC(NH_2)_2]_3Br.H_2O$			
Se_1	0.27860(13)	0.27396(7)	0.16532(10)
Se_2	0.25235(12)	0.41610(7)	0.17960(8)
Se_3	0.24599(12)	0.56445(7)	0.18479(9)
Br_1	0.06170(11)	0.10175(7)	0.47453(9)
Br_2	0.05851(11)	0.39559(7)	0.46983(9)
C_1	0.1895(11)	0.2486(5)	0.2842(9)
C_2	0.3526(11)	0.4181(5)	0.2980(9)
C_3	0.1447(11)	0.5718(5)	0.2973(8)
N_1	0.0871(11)	0.2425(6)	0.2667(9)
N_2	0.2333(9)	0.2392(5)	0.3757(7)
N_3	0.4547(9)	0.4184(5)	0.2766(8)
N_4	0.3179(9)	0.4190(5)	0.3957(7)
N_5	0.0419(9)	0.5770(5)	0.2762(8)
N_6	0.1769(9)	0.5703(5)	0.3978(7)
O	0.4576(8)	0.2494(5)	0.4233(7)

THE TRIS(SELENOUREA) ION

Bond lengths and angles in the tris(selenourea), ion, based on the atomic coordinates in Table 1, are given in Fig. 1 and Table 3. The uncertainties in cell dimensions are taken into account in the given standard deviations.

The tris(selenourea) ion, in both compounds, has an approximate linear three-selenium sequence with a Se – Se – Se bond angle of 173.8°. The Se – Se bond lengths in the dichloride are 2.597(2) Å and 2.717(2) Å, and in the dibromide 2.624(2) Å and 2.712(2) Å; the mean values of

Table 2. Anisotropic temperature parameters (Å²) in the form $\exp-[2\pi^2(U_{11}h^2a^{*2}+\cdots+2U_{23}klb^*c^*)+\cdots]$. The values are multiplied with 10^3. Standard deviations are given in parentheses.

	U_{11}	U_{22}	U_{33}	U_{12}	U_{23}	U_{13}
$[SeC(NH_2)]_3Cl_2.H_2O$						
Se_1	28.3(11)	37.5 (4)	25.3(4)	1.8(4)	−1.0(3)	3.2(4)
Se_2	12.3(10)	51.7(5)	17.3(4)	0.7(4)	1.5(3)	−0.4(4)
Se_3	19.3(11)	38.2(4)	24.4(4)	1.5(4)	0.6(3)	2.4(4)
Cl_1	20.2(15)	44.3(10)	26.3(9)	−5.0(9)	2.0(8)	6.0(10)
Cl_2	21.3(15)	49.4(11)	29.8(10)	1.1(10)	1.3(9)	5.9(11)
C_1	21(6)	32(3)	26(3)	−3(3)	2(3)	8(5)
C_2	11(6)	26(3)	27(4)	0(3)	3(3)	−7(4)
C_3	21(6)	23(3)	20(3)	1(3)	−2(3)	5(4)
N_1	20(5)	57(4)	44(4)	4(4)	−1(4)	6(5)
N_2	50(7)	42(4)	23(3)	−1(4)	12(3)	0(4)
N_3	25(5)	44(4)	29(3)	−1(3)	8(3)	2(4)
N_4	17(6)	50(4)	19(3)	3(3)	−1(3)	2(3)
N_5	26(5)	52(4)	31(4)	1(4)	−1(3)	−6(4)
N_6	15(5)	45(4)	28(3)	−3(3)	−5(3)	−2(4)
O	43(5)	61(4)	34(3)	−1(3)	1(3)	3(4)
$[SeC(NH_2)_2]_3Br_2.H_2O$						
Se_1	47.7(15)	44.7(7)	33.8(6)	1.9(6)	−1.8(5)	3.1(7)
Se_2	23.4(13)	54.2(7)	20.8(5)	0.7(5)	2.5(4)	−0.8(6)
Se_3	30.4(13)	44.1(6)	26.7(5)	0.9(5)	−1.0(5)	3.8(6)
Br_1	28.9(14)	50.6(7)	27.9(6)	−3.7(6)	−0.2(5)	4.2(6)
Br_2	29.7(14)	55.5(8)	32.3(6)	0.1(6)	2.4(5)	5.3(6)
C_1	37(9)	23(4)	35(5)	5(5)	−2(4)	0(7)
C_2	21(9)	38(6)	30(5)	−1(5)	5(4)	−9(7)
C_3	29(9)	31(5)	23(5)	3(5)	−2(4)	5(6)
N_1	41(8)	56(6)	51(6)	2(6)	−1(5)	0(7)
N_2	50(9)	49(6)	32(5)	1(5)	6(4)	2(5)
N_3	28(7)	51(6)	33(5)	3(5)	1(4)	−7(6)
N_4	23(8)	47(5)	22(4)	2(4)	−2(4)	−4(5)
N_5	29(8)	56(6)	35(5)	−1(5)	−3(4)	5(6)
N_6	27(7)	43(5)	27(4)	−3(4)	−2(4)	−3(5)
O	67(8)	53(5)	43(5)	1(5)	1(4)	−4(5)

Table 3. Dimensions of the selenourea groups. Bond lengths (Å) and angles (°). Standard deviations are given in parentheses.

	Dichloride	Dibromide		Dichloride	Dibromide
Se_1-C_1	1.921(8)	1.921(12)	$Se_1-C_1-N_1$	118.7(5)	117.7(8)
C_1-N_1	1.304(13)	1.341(19)	$Se_1-C_1-N_2$	117.1(6)	118.2(9)
C_1-N_2	1.308(11)	1.283(15)	$N_1-C_1-N_2$	124.2(7)	124.1(10)
Se_2-C_2	1.940(8)	1.947(12)	$Se_2-C_2-N_3$	118.7(5)	118.7(7)
C_2-N_3	1.315(13)	1.315(18)	$Se_2-C_2-N_4$	119.8(6)	119.9(8)
C_2-N_4	1.285(10)	1.298(14)	$N_3-C_2-N_4$	121.5(7)	121.4(10)
Se_3-C_3	1.905(8)	1.906(12)	$Se_3-C_3-N_5$	119.9(5)	120.8(7)
C_3-N_5	1.310(13)	1.325(18)	$Se_3-C_3-N_6$	120.3(6)	119.7(8)
C_3-N_6	1.295(10)	1.321(14)	$N_5-C_3-N_6$	119.9(7)	119.5(10)

the Se–Se bond lengths are 2.657 Å and 2.668 Å, respectively.

In the triiodide ion [12,13] the total length of the three-center system increases with increasing asymmetry, and there is a relationship between total length and the asymmetry of the system. The same trend has also been found in the three-selenium sequence in the triselenocyanate ion.[14] The ion is symmetrical in the rubidium and the cesium salts, with Se–Se bond lengths of 2.656(3) Å and 2.650(3) Å, respectively. In the potassium salt the ion is unsymmetrical, with bond lengths 2.689(4) Å and 2.648(4) Å, mean value 2.669 Å. In the structures of the tris(selenourea) ion the total length of the three-selenium sequence is largest in the dibromide salt, but the asymmetry of the ion is largest in the dichloride salt.

The Se–Se–Se angle in the triselenocyanate ion has been found to vary between 176.0(3)° in the potassium salt to 178.3(1) in the cesium salt. The bending occurs in a such way that the –CN groups are coming closer to each other. In the tris(selenourea) ion the bending of the

Table 4. Nitrogen-halide and nitrogen-oxygen distances (Å) and angles (°). Standard deviations are given in parentheses.

Distance	Length N···X	Angle C–N···X	Distance from plane
$[SeC(NH_2)_2]_3Cl_2.H_2O$			
$N_1\cdots Cl_{1A}$	3.487(8)	92.2(5)	−1.806
$N_1\cdots Cl_{2A}$	3.756(8)	83.9(5)	3.316
$N_1\cdots M$	2.473	93.1	0.755
$N_1\cdots O_G$	2.856(10)	128.6(6)	−0.133
$N_2\cdots Cl_{1A}$	3.393(8)	96.3(5)	−1.806
$N_2\cdots Cl_{2A}$	3.769(8)	83.3(5)	3.316
$N_2\cdots M$	2.417	90.7	0.755
$N_2\cdots O_A$	2.897(11)	127.7(5)	−0.071
$N_3\cdots Cl_{1B}$	3.262(7)	101.5(5)	−0.430
$N_3\cdots Cl_{2C}$	3.261(7)	121.9(5)	−0.288
$N_4\cdots Cl_{1B}$	3.384(8)	96.5(5)	−0.430
$N_4\cdots Cl_{2A}$	3.287(8)	124.3(5)	−0.360
$N_5\cdots Cl_{1D}$	3.363(8)	122.8(5)	0.446
$N_5\cdots Cl_{2E}$	3.356(8)	99.4(5)	0.346
$N_6\cdots Cl_{1F}$	3.391(8)	122.7(5)	0.917
$N_6\cdots Cl_{2E}$	3.331(8)	100.9(6)	0.346
$N_6\cdots Cl_{2A}$	3.540(7)	95.2(5)	−3.265
$[SeC(NH_2)_2]_3Br_2.H_2O$			
$N_1\cdots Br_{1A}$	3.672(11)	91.5(7)	−1.914
$N_1\cdots Br_{2A}$	3.796(11)	85.4(7)	3.288
$N_1\cdots M$	2.596	92.3	0.687
$N_1\cdots O_G$	2.886(15)	133.6(8)	−0.303
$N_2\cdots Br_{1A}$	3.537(11)	98.3(7)	−1.914
$N_2\cdots Br_{2A}$	3.795(11)	85.8(7)	3.288
$N_2\cdots M$	2.500	87.4	0.687
$N_2\cdots O_{1A}$	2.896(16)	126.5(8)	−0.038
$N_3\cdots Br_{1B}$	3.413(10)	101.6(7)	−0.433
$N_3\cdots Br_{2C}$	3.376(10)	124.4(7)	−0.374
$N_4\cdots Br_{1B}$	3.496(10)	98.0(8)	−0.433
$N_4\cdots Br_{2A}$	3.425(11)	125.1(8)	−0.417
$N_5\cdots Br_{1D}$	3.428(10)	124.4(6)	0.380
$N_5\cdots Br_{2E}$	3.457(10)	100.6(6)	0.346
$N_6\cdots Br_{1F}$	3.480(11)	123.4(8)	0.833
$N_6\cdots Br_{2E}$	3.454(10)	100.9(8)	0.346
$N_6\cdots Br_{2A}$	3.637(10)	97.4(7)	−3.320

M is the midpoint between Cl_{1A} and Cl_{2A}, and between Br_{1A} and Br_{2A}.

Se – Se – Se sequence is opposite, so that the $-C(NH_2)_2$ groups are coming further away from each other. The main reason for this and for the asymmetry of the Se – Se – Se sequence is probably an approach of a halide ion to the terminal selenium atom which has the longest bond to the central selenium atom. The $Se_3\cdots Cl_2'$ $(\frac{1}{2}-x,\ 1-y,\ z-\frac{1}{2})$ distance is 3.699(2) Å and the $Se_3\cdots Br_2'$ distance is 3.719(2) Å. The $C_3-Se_3\cdots Cl_2'$ angle is 164.0(2)° and the $C_3-Se_3\cdots Br_2'$ angle is 164.6(3)°. The angle between the central selenium atom, the terminal selenium atom and the chloride ion, $Se_2-Se_3\cdots Cl_2'$, is 99.38(4)° and the $Se_2-Se_3\cdots Br_2'$ is 99.16(5)°.

Each of the selenourea groups in both structures of the tris(selenourea) ion is planar within the error, the largest deviation of an atom from the least square plane of the group being 0.009 Å.

Bond lengths and angles in the selenourea groups do not deviate significantly from the values found in the crystals of selenourea.[15] As seen from Table 3 the differences in the individual Se – C bond lengths are not significant, nor are the differences in the individual C – N bond lengths. The mean value of the Se – C bond lengths is 1.92 Å, and the mean value of the C – N bond lengths is 1.30 Å. The N – C – N angles are from 119.5(9)° to 124.2(7)° and the Se – C – N angles are beteeen 117.1(6)° and 120.8(7)°.

Since the atoms in each selenourea group of the tris(selenourea) ion are co-planar, as in the structure of selenourea, and the bond lengths and angles have not changed, the oxydation:

$$3\ SeC(NH_2)_2 = [SeC(NH_2)_2]_3^{2+} + 2e$$

has not altered the dimensions of the selenourea groups.

Corresponding Se – Se – C angles in the two structures are equal within the error. The mean value of the angles at the central selenium atom, Se_2, is 89.1°, and the mean value of the angles at the terminal selenium atoms, Se_1 and Se_3, is 96.3°.

The three selenium atoms and the two carbon atoms of the terminal selenourea groups are approximately co-planar, the largest deviation of an atom from a least squares plane being 0.05 Å in the dichloride and 0.07 Å in the dibromide. So are the three selenium atoms and the carbon atom of the middle selenourea group, the largest deviation of an atom from a least squares plane being 0.09 Å in both compounds. The angle between these two planes is 74.9° in the dichloride and 76.3° in the dibromide.

The plane of the terminal selenourea groups, Se_1, and of the central selenourea group, Se_2, make an angle of 14.9° with each other in the dichloride and 14.6° in the dibromide. The dihedral angle between the planes of the Se_2 and the Se_3 groups is 5.4° in the dichloride and 5.3° in the dibromide.

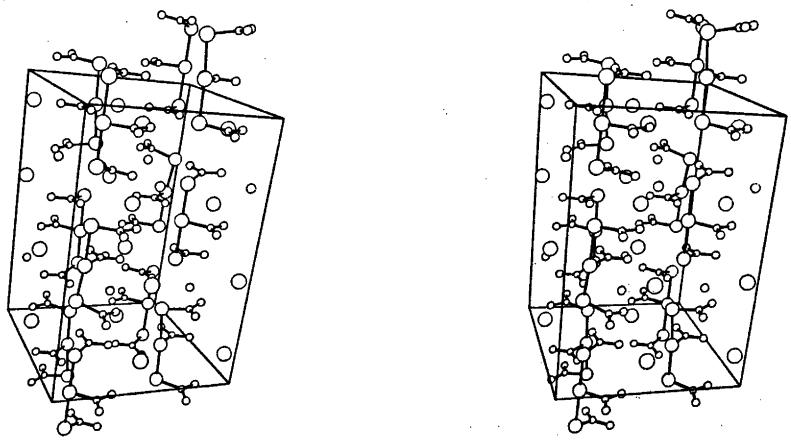

Fig. 2. A stereoscopic pair of drawing showings the content of the unit cell in $(su)_3Br_2.H_2O$.

THE PACKING IN THE CRYSTALS

The central selenium atom, Se_2, in the three-selenium sequence has a close contact to a halide ion. The $Se_2\cdots Cl_1''$ (x, $\frac{1}{2}-y$, $\frac{1}{2}+z$) distance is 3.457(3) Å and the $Se_2\cdots Br_1''$ (x, $\frac{1}{2}-y$, $\frac{1}{2}+z$) distance is 3.530(3) Å. Taking the difference in the ionic radii of the bromide ion and chloride ion to be 0.14 Å, the selenium atom should be more engaged in the contact to the bromide ion than to the chloride ion. The angle $C_2-Se_2\cdots Cl_1''$ is 174.6(2)° and the angle $C_2-Se_2\cdots Br_1''$ is 175.0(3)°. The distance from Cl_1'' to the least squares plane through Se_1, Se_2, Se_3, and C_2 is 0.523 Å. The distance from Br_1'' to the corresponding plane is 0.489 Å. From these data it appears that the halide ion approaches the fourth coordination site of square-planar four-coordination at Se_2. This is a tendency encountered also in other primarily three-coordinated complexes of selenium(II) [16,17] and of tellurium(II).[18,19]

The amino nitrogen atoms have short distances, probably involving hydrogen bonds, to halide ions and to water oxygen atoms. The nitrogen atoms are assumed to have a trigonal-planar bonding system, *i.e.*, the hydrogen atoms lie in or close to the planes through the selenourea groups. The interatomic distances and angles, and the distances of the halide ions and the oxygen atoms from the selenourea plane, are listed in Table 4. In the table an atom marked with A denotes an atom at (x, y, z), B at ($x+\frac{1}{2}$, $\frac{1}{2}-y$, $1-z$), C at ($x+\frac{1}{2}$, y, $\frac{1}{2}-z$), D at ($\bar{x}$, $y+\frac{1}{2}$, $\frac{1}{2}-z$), E at ($\bar{x}$, $1-y$, $1-z$), F at ($\frac{1}{2}-x$, $\frac{1}{2}+y$, z), and G at ($x-\frac{1}{2}$, y, $\frac{1}{2}-z$), where x, y, z are the atomic coordinates of Table 1. Since the halide ions X_{1A} and X_{2A} are rather far out of the selenourea plane, the N_1-H and the N_2-H bonds are probably directed not towards X_{1A} and X_{2A} but towards a point somewhere between X_{1A} and X_{2A}, probably closer to X_{1A} than X_{2A}. Something like this could also be the case with the N_6-H bonds.

Hydrogen bonds are also found from the water molecule to the halide ion. The $O\cdots Cl_{1B}$ distance is 3.149(7) Å and the $O\cdots Cl_{2B}$ is 3.091(7) Å. The $Cl\cdots O\cdots Cl$ angle is 116.2(2)°. The $O\cdots Br_{1B}$ is 3.278(10) Å and the $O\cdots Br_{2B}$ is 3.228(10) Å with $Br\cdots O\cdots Br$ angle of 111.2(2)°. The hydrogen atoms located in the difference electron density maps are in accordance with such hydrogen bonds.

The water molecule is surrounded by two nitrogen atoms, *cf.* Table 4, and, as mentioned above, by two halide ions. The arrangement is approximately tetrahedral. The angles are from 97.0° to 119.6° in the dichloride, and from 94.7° to 121.6° in the dibromide.

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