Table 5. *Interatomic distances for nearest neighbors*

Atomic type	Nearest neighbors Number	Type	Distance*	Coordination number
I	4	II	2·811 Å	
	12	IV	2·754	16
II	1	I	2·811	
	3	III	2·572	
	3	III	2·930	
	6	IV	2·708	
	3	IV	2·895	16
III	1	II	2·572	
	1	III	2·930	
	2	III	2·622	
	4	III	2·661	
	1	IV	2·349	
	2	IV	2·524	
	2	IV	2·682	13
IV	1	I	2·754	
	2	II	2·708	
	1	II	2·895	
	1	III	2·349	
	2	III	2·524	
	2	III	2·682	
	1	IV	2·258	
	2	IV	2·424	12

* All errors are ±0·001 Å.

One might expect differences to exist in the true scattering factors of the four different types of manganese atoms, since the coordination number and symmetry of their sites are different. Although such effects were looked for in a second data set which consisted of all dependent reflections in the region $\lambda^{-1}\sin\theta < 0{\cdot}3$ Å^{-1}, no definite conclusions could be reached. This is not surprising since these inner data are most sensitive to the model for extinction and are affected more by the problems of the absorption correction.

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The Crystal and Molecular Structure of α,α′-Diselenobisformamidinium Dichloride

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The crystal structure of α,α′-diselenobisformamidinium dichloride, $[SeC(NH_2)_2]_2Cl_2$, has been determined by a three-dimensional X-ray analysis (Cu $K\alpha$). The unit cell of dimensions $a=12{\cdot}82(2)$, $b=6{\cdot}01(1)$, $c=12{\cdot}22(1)$ Å, $\beta=108{\cdot}1(0{\cdot}1)^\circ$ contains 4 stoichiometric units (space group $C2/c$). The organic cation is formed by two selenourea groups nearly perpendicular to the Se–Se = 2·380(6) Å bond, the dihedral angle $CSeSe^i\wedge SeSe^iC^i$ being 89·5°; bond distances in those groups are: Se–C = 1·94 (1), C–N = 1·32 (2) and 1·30 (2) Å. All the hydrogen atoms are involved in N···Cl hydrogen bonds. The molecular structure of the cation is compared with that of the corresponding thio-derivative.

Introduction

In an attempt to prepare silver(I)–selenourea complexes, a few crystals of α,α′-diselenobisformamidinium dichloride were obtained. They were probably formed by oxidation of selenourea by the Ag^+ ion or by atmospheric oxygen. As the crystals were too few for a conventional chemical analysis, their nature was determined by an X-ray structural study, started by assuming the product to be a silver chloride–selenourea complex. Further work showed that the compound was α,α′-diselenobisformamidinium dichloride, and so it appeared possible to correlate the structure with those of α,α′-dithiobisformamidinium dibromide and diiodide studied by Foss, Johnsen & Tvedten (1958) and to give more structural information on the selenium derivatives. A short account of this research has already been presented (Chiesi, Grossoni, Nardelli & Vidoni, 1969).

Experimental

A few stocky monoclinic crystals of the compound $[SeC(NH_2)_2]_2Cl_2$ were obtained from an aqueous solution containing AgCl and selenourea by standing in a desiccator for a few days. The forms observed were: $\{\bar{1}01\}$, $\{001\}$, $\{110\}$, $\{\bar{1}11\}$, $\{001\}$ (in decreasing order of size). The crystals are pleochroic: looking down [001] they are yellow and colourless when the electric vector is parallel to x^* and y respectively.

Crystal data from rotation and Weissenberg photographs (Ni filtered Cu radiation $\lambda = 1{\cdot}5418$ Å) are as follows: $[SeC(NH_2)_2]_2Cl_2$, $M = 316{\cdot}9$, $a = 12{\cdot}82$ (2), $b = 6{\cdot}01$ (1), $c = 12{\cdot}22$ (1) Å, $\beta = 108{\cdot}1$ (0·1)°, $Z = 4$, $V = 895{\cdot}6$ Å^3, $D_c = 2{\cdot}35$ g.cm^{-3}, $\mu = 167$ cm^{-1} (Cu $K\alpha$), $F(000) = 600$; space groups: Cc or$C2/c$. The latter space group was found from the structural analysis to be the correct one in agreement with the absence of piezoelectric effects.

Three-dimensional intensity data were collected in the usual way on multiple-film Weissenberg photographs around [100] and [010] using integration and equi-inclination techniques (Cu $K\alpha$ radiation). 856 independent reflexions were measured photometrically out of 1005 possible ones. After correction for Lorentz and polarization factors and for absorption (the samples were considered to be spherical with $\bar{r} = 0{\cdot}01$ cm as they were made roughly round by grinding), the intensities from different films were correlated following Rollett & Sparks (1960), and a trial absolute scale was then established for all the three-dimensional data by Wilson's (1942) method, assuming the formula AgCl $SeC(NH_2)_2$.

Structure analysis and refinement

In the first stages of the analysis the formula given above was assumed and on this basis the three-dimensional Patterson synthesis could be interpreted only with the Cc space group. A model for the stucture was obtained which could be refined by Fourier methods down to $R = 27{\cdot}4$%. At this point it was quite clear that the structure was much too open and there were residual peaks indicating the presence of a twofold axis which doubled the selenourea molecules. The introduction of atoms in these residual peaks greatly improved the R value (21·4%) showing that the formula assumed originally was incorrect.

Table 1. *Final atomic fractional coordinates* (× 10^4), *thermal parameters*†(× 10 Å^2) *with e.s.d.'s and ratios* (*e.s.d.*)/(*coordinate shift*)

	$x/a(\sigma)$	$y/b(\sigma)$	$z/c(\sigma)$	$B_{11}(\sigma)$	$B_{22}(\sigma)$	$B_{33}(\sigma)$	$B_{12}(\sigma)$	$B_{13}(\sigma)$	$B_{23}(\sigma)$	$\lvert r(x)\rvert$	$\lvert r(y)\rvert$	$\lvert r(z)\rvert$
Se	976 (1)	1303 (2)	2799 (1)	47 (3)	21 (0)	29 (2)	−3 (2)	8 (4)	1 (2)	3	∞	2
Cl	1447 (4)	−5621 (5)	6072 (4)	47 (8)	27 (1)	33 (6)	−3 (5)	11 (9)	2 (4)	13	7	9
N(1)	1181 (14)	−3132 (18)	3546 (12)	48 (29)	24 (5)	34 (22)	0 (18)	10 (34)	2 (16)	140	7	41
N(2)	1189 (14)	−613 (22)	4963 (13)	49 (30)	32 (8)	27 (20)	4 (22)	8 (33)	3 (18)	8	27	8
C	1149 (15)	−1080 (21)	3910 (14)	36 (25)	18 (5)	34 (25)	0 (19)	7 (34)	7 (18)	17	21	7

† The anisotropic temperature factor is: $\exp\,[-\tfrac{1}{4}(B_{11}h^2a^{*2} + B_{22}k^2b^{*2} + B_{33}l^2c^{*2} + 2B_{12}hka^*b^* + 2B_{13}hla^*c^* + 2B_{23}klb^*c^*)]$.

Chemical considerations were then applied to interpret all the observed peaks and the chemical composition of the compound was obtained in a straightforward manner assuming the $C2/c$ space group. From that point on the refinement was carried out by means of isotropic (till $R=17{\cdot}3\%$) and anisotropic cycles of Booth's differential synthesis and their regular convergence supported the correctness of the chemical interpretation. Further evidence was obtained by comparing the X-ray powder diffraction pattern of the previous crystals with that given by the oxidation product of selenourea with H_2O_2 in cooled HCl–EtOH.

After the refinement operations the final residual error indices (R for observed reflexions only, R' assuming $F_o=\frac{1}{2}F_{\min}$ when $F_c \geq F_{\min}$ for unobserved reflexions only, multiplicities not considered) were $R=9{\cdot}0$ and $R'=9{\cdot}5\%$. The introduction of hydrogen atoms in positions calculated to make N–H = 1·03 Å with trigonal hybridization for nitrogen atoms, did not change the final R indices significantly.

In Table 1 the final coordinates are given with estimated standard deviations (Cruickshank, 1949, 1956; the calculations were performed with computer programs taking into account the atomic anisotropy), together with the final anisotropic thermal parameters and the ratios $r(x)=|\sigma(x)/\varepsilon(x)|$ between e.s.d.'s and coordinate shifts. No conclusions can be drawn from the values of thermal parameters as they are probably affected by absorption and scaling effects. The observed and calculated values of the electron density and of the second derivatives at the atomic peaks, with e.s.d.'s, are compared in Table 2. The F_c values listed in Table 3 are calculated with the final parameters of Table 1 and include the contribution of hydrogen atoms, the scattering factors used being those of Thomas & Umeda (1957) for Se, those of Berghuis, Haanappel, Potters, Loopstra, MacGillavry & Veenendaal (1955) for Cl, N, C and those of McWeeny (1951) for H; the 'less than' values for F_o are calculated from the minimum observed intensity taken as $I_{\min}-1$.

The standard deviations quoted in the next section are calculated from the formulae of Ahmed & Cruickshank (1953) for bond lengths and those of Darlow (1960) for angles, the effects of errors in cell parameters being accounted for following Darlow & Cochran (1961).

The calculations were performed with the computer programs of Nardelli, Musatti, Domiano & Andreetti (1964, 1965) on the Elea 6001/S computer of the Centro di Calcolo Elettronico della Università di Parma.

Discussion

The organic cation is formed by two selenourea groups related by a twofold axis perpendicular to the Se–Se bond as shown in Fig. 1(*a*). The structures of the similar sulphur derivatives studied by Foss, Johnsen & Tvedten (1958) are also illustrated in the same Figure. The same comparison is made for distances and angles in Table 4.

The Se–Se distance is not statistically different from that found recently in trigonal selenium (2·373(5) Å)

Table 2. *Atomic peak heights* (e.Å^{-3}), curvatures (e.Å^{-5}) *and e.s.d.'s*

		ϱ	$-A_{hh}$	$-A_{kk}$	$-A_{ll}$	A_{kl}	A_{hl}	A_{hk}
Se	Obs.	76·3	610	843	755	9	192	−24
	Cals.	77·1	619	849	761	10	195	−22
Cl	Obs.	32·2	251	340	314	1	88	−14
	Calc.	32·5	256	342	318	0	89	−14
N(1)	Obs.	9·7	72	95	92	6	24	−8
	Calc.	9·6	74	96	93	6	25	−9
N(2)	Obs.	10·1	74	89	97	4	29	6
	Calc.	10·2	75	88	99	4	29	6
C	Obs.	9·8	70	103	94	16	27	−3
	Calc.	9·7	70	103	95	16	27	−1
	E.s.d.	0·4	4	5	4	2	2	2

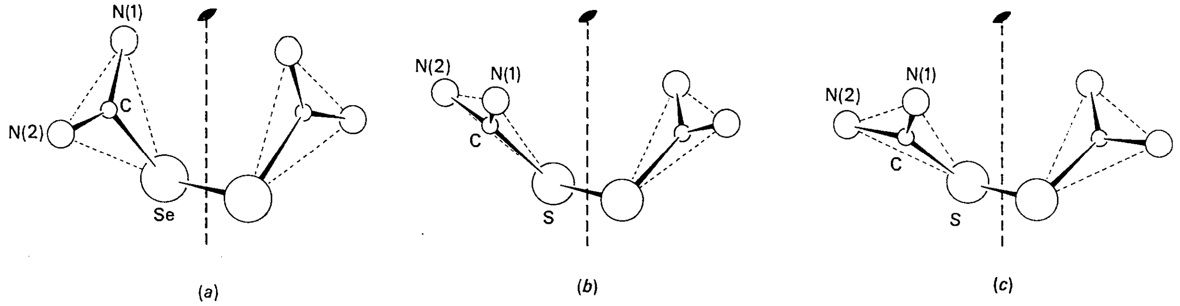

Fig. 1. Clinographic projections of the structures of the cations: (*a*) $^{+}(NH_2)_2CSeSeC(NH_2)_2^{+}$ (present paper), (*b*) $^{+}(NH_2)_2CSSC(NH_2)_2^{+}$ in the bromide derivative (Foss, Johnsen & Tvedten, 1958), (*c*) $^{+}(NH_2)_2CSSC(NH_2)_2^{+}$ in the iodide derivative (Foss, Johnsen & Tvedten, 1958).

by Cherin & Unger (1967) and can be reasonably assumed to represent a Se–Se single-bond distance. Assuming this value and the C(sp^2)–C(sp^2) = 1·483 Å single-bond distance given by Dewar & Schmeising (1960), a single bond Se(?)–C(sp^2) = 1·93 Å distance is calculated which is in good agreement with that found [1·94(1) Å] in the present case. The C–N distances in the Se cation are not significantly different and are consistent with a π delocalization along the N–C–N system. The general rule, by which the bond angle in X decreases from S to Se, is observed.

The two selenourea parts of the cation are planar

Table 3. *Observed and calculated structure factors*

A minus sign for F_o means 'less than'.

h	k	l	$\lvert 10F_o \rvert$	$10F_c$
0	2	0	748	617
0	4	0	1669	-1460
0	6	0	88	-90
0	2	1	2596	-2465
0	4	1	138	22
0	6	1	618	523
0	0	2	2038	-1580
0	2	2	625	582
0	4	2	1098	1174
0	6	2	323	-260
0	2	3	1147	1199
0	4	3	486	-448
0	6	3	929	-849
0	0	4	468	425
0	2	4	242	-225
0	4	4	764	-739
0	6	4	247	207
0	2	5	717	-680
0	4	5	92	56
0	6	5	517	490
0	0	6	806	-780
0	2	6	203	-186
0	4	6	535	467
0	6	6	100	91
0	2	7	577	533
0	4	7	361	310
0	6	7	88	57
0	0	8	735	695
0	2	8	283	247
0	4	8	109	-87
0	6	8	117	-99
0	2	9	157	113
0	4	9	117	102
0	6	9	32-	-59
0	0	10	683	641
0	2	10	324	308
0	4	10	261	-211
0	2	11	423	-365
0	4	11	147	-131
0	0	12	433	-377
0	2	12	82-	36
0	4	12	283	266
0	2	13	348	316
0	0	14	251	248
0	2	14	129	-123
1	1	0	1089	1104
1	3	0	1075	-1058
1	5	0	562	-581
1	7	0	445	507
1	1	1	943	-815
1	1	$\bar{1}$	780	921
1	3	1	244	-256
1	3	$\bar{1}$	763	870
1	5	1	834	867
1	5	$\bar{1}$	599	-670
1	7	1	243	282
1	7	$\bar{1}$	236	-260
1	1	2	303	236
1	1	$\bar{2}$	2138	-2208
1	3	2	911	936
1	3	$\bar{2}$	768	913
1	5	2	89	62
1	5	$\bar{2}$	720	812
1	7	2	342	-396
1	7	$\bar{2}$	289	-338
1	1	3	1120	1066
1	1	$\bar{3}$	1399	-1986
1	3	3	447	442
1	3	$\bar{3}$	1452	-1734
1	5	3	332	-344
1	5	$\bar{3}$	365	390
1	7	3	96	-119
1	7	$\bar{3}$	202	235
1	1	4	1246	1203
1	1	$\bar{4}$	1491	1568
1	3	4	117	-107
1	3	$\bar{4}$	843	-983
1	5	4	129	-122
1	5	$\bar{4}$	442	-439
1	7	4	109	-122
1	7	$\bar{4}$	299	371
1	1	5	424	-411
1	1	$\bar{5}$	1190	1287
1	3	5	408	-423
1	3	$\bar{5}$	374	415
1	5	5	287	-275
1	5	$\bar{5}$	862	-960
1	7	5	99	-123
1	7	$\bar{5}$	233	-300
1	1	6	81	71
1	1	$\bar{6}$	179	-258
1	3	6	74	-104
1	3	$\bar{6}$	1013	1156
1	5	6	48-	-3
1	5	$\bar{6}$	178	193
1	7	$\bar{6}$	348	-474
1	1	7	631	-633
1	1	$\bar{7}$	663	-763
1	3	7	542	-539
1	3	$\bar{7}$	281	-280
1	5	7	52	50
1	5	$\bar{7}$	535	564
1	1	8	680	-722
1	1	$\bar{8}$	749	806
1	3	8	366	327
1	3	$\bar{8}$	461	-464
1	5	8	210	182
1	5	$\bar{8}$	337	-339
1	1	9	563	569
1	1	$\bar{9}$	367	358
1	3	9	401	369
1	3	$\bar{9}$	438	436
1	5	9	214	-211
1	5	$\bar{9}$	77	-43
1	1	10	501	521
1	1	$\overline{10}$	373	-357
1	3	10	467	-447
1	3	$\overline{10}$	70-	13
1	5	10	218	-221
1	5	$\overline{10}$	176	170
1	1	11	312	-328
1	1	$\overline{11}$	99	-74
1	3	11	153	-140
1	3	$\overline{11}$	235	-215
1	5	11	240	363
1	5	$\overline{11}$	52	-49
1	1	12	82-	-96
1	1	$\overline{12}$	197	-180
1	3	12	394	391
1	3	$\overline{12}$	89	63
1	1	13	296	290
1	1	$\overline{13}$	186	-188
1	3	13	220	238
1	3	$\overline{13}$	244	-247
1	1	14	271	334
1	1	$\overline{14}$	203	206
2	0	0	629	661
2	2	0	272	-204
2	4	0	486	-439
2	6	0	134	111
2	2	1	1035	-935
2	2	$\bar{1}$	133	116
2	4	1	599	-536
2	4	$\bar{1}$	854	-881
2	6	1	59-	11
2	6	$\bar{1}$	517	-507
2	0	2	252	-205
2	0	$\bar{2}$	1432	-1329
2	2	2	778	-691
2	2	$\bar{2}$	806	839
2	4	2	175	-170
2	4	$\bar{2}$	867	952
2	6	2	194	179
2	6	$\bar{2}$	309	-295
2	2	3	304	-287
2	2	$\bar{3}$	1578	-1659
2	4	3	385	338
2	4	$\bar{3}$	297	244
2	6	3	141	137
2	6	$\bar{3}$	461	478
2	0	4	879	-921
2	0	$\bar{4}$	2604	2407
2	2	4	121	78
2	2	$\bar{4}$	106	109
2	4	4	410	393
2	4	$\bar{4}$	884	-970
2	6	4	55	-66
2	6	$\bar{4}$	55-	58
2	2	5	1069	1122
2	2	$\bar{5}$	1689	1931
2	4	5	233	214
2	4	$\bar{5}$	211	235
2	6	5	273	-272
2	6	$\bar{5}$	514	-536
2	0	6	1146	1279
2	0	$\bar{6}$	1507	-1699
2	2	6	340	336
2	2	$\bar{6}$	259	-256
2	4	6	579	-539
2	4	$\bar{6}$	824	884
2	6	6	52	-62
2	6	$\bar{6}$	48-	-16
2	2	7	924	-980
2	2	$\bar{7}$	1011	-1137
2	4	7	188	-150
2	4	$\bar{7}$	71-	16
2	6	7	339	373
2	6	$\bar{7}$	501	561
2	0	8	613	-623
2	0	$\bar{8}$	705	680
2	2	8	157	143
2	2	$\bar{8}$	261	-254
2	4	8	692	647
2	4	$\bar{8}$	784	-793
2	6	8	108	-105
2	6	$\bar{8}$	151	155
2	2	9	419	388
2	2	$\bar{9}$	427	421
2	4	9	392	-360
2	4	$\bar{9}$	409	-390
2	6	$\bar{9}$	421	-493
2	0	10	573	647
2	0	$\overline{10}$	752	-734
2	2	10	175	-161
2	2	$\overline{10}$	131	105
2	4	10	558	-546
2	4	$\overline{10}$	541	522
2	2	11	670	-675
2	2	$\overline{11}$	697	-716
2	4	11	43-	1
2	4	$\overline{11}$	122	-123
2	0	12	565	-652
2	0	$\overline{12}$	616	575
2	2	12	66-	-30
2	2	$\overline{12}$	118	91
2	4	$\overline{12}$	186	-186
2	2	13	402	457
2	2	$\overline{13}$	270	266
2	4	$\overline{13}$	39	23
2	0	14	250	312
2	0	$\overline{14}$	64-	-49
2	2	$\overline{14}$	50-	11
3	1	0	267	186
3	3	0	435	377
3	5	0	196	169
3	7	0	243	-268
3	1	1	128	-84
3	1	$\bar{1}$	60	49
3	3	1	63	52
3	3	$\bar{1}$	283	216
3	5	1	478	-406
3	5	$\bar{1}$	303	289
3	7	1	153	-146
3	7	$\bar{1}$	106	120
3	1	2	1191	1109
3	1	$\bar{2}$	741	-693
3	3	2	477	-443
3	3	$\bar{2}$	78-	-28
3	5	2	602	-529
3	5	$\bar{2}$	221	217
3	7	2	174	188
3	7	$\bar{2}$	56-	-19
3	1	3	1263	-1195
3	1	$\bar{3}$	221	192
3	3	3	1337	-1280
3	3	$\bar{3}$	93	40
3	5	3	133	109
3	5	$\bar{3}$	340	313
3	7	3	127	138
3	7	$\bar{3}$	70	80
3	1	4	984	-1325
3	1	$\bar{4}$	536	-479
3	3	4	632	603
3	3	$\bar{4}$	616	-635
3	5	4	425	390
3	5	$\bar{4}$	156	-162
3	7	4	174	-205
3	7	$\bar{4}$	372	423
3	1	5	1024	1064
3	1	$\bar{5}$	478	499
3	3	5	505	531
3	3	$\bar{5}$	225	211
3	5	5	716	-685
3	5	$\bar{5}$	635	-646
3	7	$\bar{5}$	228	-258
3	1	6	345	372
3	1	$\bar{6}$	855	-952
3	3	6	984	-981
3	3	$\bar{6}$	542	593
3	5	6	195	-200
3	5	$\bar{6}$	314	295
3	7	$\bar{6}$	180	-216
3	1	7	697	-753
3	1	$\bar{7}$	1189	-1282
3	3	7	270	-268
3	3	$\bar{7}$	918	-1015
3	5	7	747	636
3	5	$\bar{7}$	271	265
3	1	8	656	-664
3	1	$\bar{8}$	1022	1119
3	3	8	667	636
3	3	$\bar{8}$	687	-679
3	5	8	327	314
3	5	$\bar{8}$	363	-342
3	1	9	514	507
3	1	$\bar{9}$	748	771
3	3	9	441	418
3	3	$\bar{9}$	436	416
3	5	9	261	-260
3	5	$\bar{9}$	473	-449
3	1	10	563	559
3	1	$\overline{10}$	538	-539
3	3	10	270	-254
3	3	$\overline{10}$	648	639
3	5	10	234	-260
3	5	$\overline{10}$	260	268
3	1	11	329	-338
3	1	$\overline{11}$	469	-394
3	3	11	418	-408
3	3	$\overline{11}$	198	-190
3	5	$\overline{11}$	436	444
3	1	12	129	-126
3	1	$\overline{12}$	173	147
3	3	12	130	121
3	3	$\overline{12}$	416	-394
3	1	13	59-	52
3	1	$\overline{13}$	339	302
3	3	$\overline{13}$	332	302
3	1	$\overline{14}$	366	-362
3	3	$\overline{14}$	89	92
3	1	$\overline{15}$	176	-185
4	0	0	3012	-3359
4	2	0	551	-428
4	4	0	1109	950
4	6	0	116	99
4	2	1	1913	1795
4	2	$\bar{1}$	1258	-1229
4	4	1	134	105
4	4	$\bar{1}$	119	88
4	6	1	352	-312
4	6	$\bar{1}$	348	321
4	0	2	1610	1725
4	0	$\bar{2}$	251	-126
4	2	2	266	-197
4	2	$\bar{2}$	645	-587
4	4	2	1134	-994
4	4	$\bar{2}$	525	-492
4	6	2	145	118
4	6	$\bar{2}$	255	231
4	2	3	1190	-1258
4	2	$\bar{3}$	82	101
4	4	3	324	263
4	4	$\bar{3}$	375	-334
4	6	3	708	714
4	6	$\bar{3}$	502	-473
4	0	4	712	-714
4	0	$\bar{4}$	498	423
4	2	4	297	261
4	2	$\bar{4}$	56	-25
4	4	4	867	799
4	4	$\bar{4}$	102	71
4	6	4	242	-228
4	6	$\bar{4}$	52-	-20
4	2	5	822	811
4	2	$\bar{5}$	89	84
4	4	5	263	-241
4	4	$\bar{5}$	258	-253
4	6	5	602	-635
4	6	$\bar{5}$	49-	-10
4	0	6	837	877
4	0	$\bar{6}$	116-	0
4	2	6	82	-21
4	2	$\bar{6}$	348	358
4	4	6	741	-675
4	4	$\bar{6}$	141	148
4	6	6	32-	44
4	6	$\bar{6}$	238	-228
4	2	7	856	-846
4	2	$\bar{7}$	295	-308
4	4	7	180	-155
4	4	$\bar{7}$	364	334
4	6	7	175	196
4	6	$\bar{7}$	392	388
4	0	8	968	-1092
4	0	$\bar{8}$	280	271
4	2	8	214	-206
4	2	$\bar{8}$	202	-158
4	4	8	457	423
4	4	$\bar{8}$	461	-415
4	6	$\bar{8}$	113	97
4	2	9	405	405
4	2	$\bar{9}$	743	733
4	4	9	64	-44
4	4	$\bar{9}$	172	144
4	6	$\bar{9}$	242	-251
4	0	10	93-	42
4	0	$\overline{10}$	1154	-1180
4	2	10	206	-195
4	2	$\overline{10}$	340	-312
4	4	10	120	-129
4	4	$\overline{10}$	582	522
4	2	11	64-	-48
4	2	$\overline{11}$	690	-641
4	4	$\overline{11}$	57-	-53
4	0	12	60	74
4	0	$\overline{12}$	591	491
4	2	12	47-	43
4	2	$\overline{12}$	152	-136
4	4	$\overline{12}$	433	-413
4	2	$\overline{13}$	456	409
4	4	$\overline{13}$	202	-184
4	0	$\overline{14}$	371	-334
4	2	$\overline{14}$	110	125
4	0	$\overline{15}$	29-	0
4	2	$\overline{15}$	251	-319
5	1	0	1462	-1284
5	3	0	1098	878
5	5	0	775	628
5	7	0	388	-391
5	1	1	1030	859
5	1	$\bar{1}$	903	-929
5	3	1	540	429
5	3	$\bar{1}$	1064	-960
5	5	1	985	-815
5	5	$\bar{1}$	506	420
5	7	1	273	-280
5	7	$\bar{1}$	227	207
5	1	2	270	180
5	1	$\bar{2}$	1413	1494
5	3	2	1247	-1050
5	3	$\bar{2}$	686	-619
5	5	2	248	-201
5	5	$\bar{2}$	687	-607
5	7	2	448	504
5	7	$\bar{2}$	234	229
5	1	3	1148	-1023
5	1	$\bar{3}$	1127	1203
5	3	3	579	-476
5	3	$\bar{3}$	1169	1080
5	5	3	736	643
5	5	$\bar{3}$	315	-283
5	7	$\bar{3}$	182	-182
5	1	4	1111	-1043
5	1	$\bar{4}$	699	-627
5	3	4	625	536
5	3	$\bar{4}$	627	602
5	5	4	269	241
5	5	$\bar{4}$	220	175
5	7	$\bar{4}$	255	-255
5	1	5	833	787
5	1	$\bar{5}$	535	-532
5	3	5	719	656
5	3	$\bar{5}$	90	54
5	5	5	114	-105
5	5	$\bar{5}$	687	630
5	7	$\bar{5}$	189	198
5	1	6	571	534
5	1	$\bar{6}$	233	-162
5	3	6	364	-298
5	3	$\bar{6}$	599	-570
5	5	6	237	-212
5	5	$\bar{6}$	78-	-23
5	1	7	111	-79
5	1	$\bar{7}$	202	196
5	3	7	76-	-20
5	3	$\bar{7}$	87	50
5	5	7	184	153
5	5	$\bar{7}$	98	-90
5	1	8	143	101
5	1	$\bar{8}$	482	-444
5	3	8	106	89
5	3	$\bar{8}$	111	-86
5	5	8	54	35
5	5	$\bar{8}$	141	163
5	1	9	161	-130
5	1	$\bar{9}$	174	111
5	3	9	141	-121
5	3	$\bar{9}$	182	-158
5	5	$\bar{9}$	303	-282
5	1	10	248	-239
5	1	$\overline{10}$	195	-148
5	3	10	73	81
5	3	$\overline{10}$	396	330
5	5	$\overline{10}$	44	47
5	1	11	69	76
5	1	$\overline{11}$	378	-316
5	3	11	33-	12
5	3	$\overline{11}$	157	-131
5	5	$\overline{11}$	214	189
5	1	12	51-	-34
5	1	$\overline{12}$	570	477
5	3	$\overline{12}$	330	-264
5	1	$\overline{13}$	438	371
5	3	$\overline{13}$	441	376
5	1	$\overline{14}$	324	-274
5	3	$\overline{14}$	194	184
5	1	$\overline{15}$	211	-177
6	0	0	1130	-1011
6	2	0	391	331
6	4	0	911	789
6	6	0	222	-203
6	2	1	1158	1084
6	2	$\bar{1}$	664	-673
6	4	1	141	93
6	4	$\bar{1}$	750	716
6	6	1	359	-359
6	6	$\bar{1}$	632	618
6	0	2	899	900
6	0	$\bar{2}$	1524	1498
6	2	2	307	269
6	2	$\bar{2}$	462	-450
6	4	2	475	-393
6	4	$\bar{2}$	1013	-988
6	6	2	50-	-16
6	6	$\bar{2}$	222	209
6	2	3	724	-663
6	2	$\bar{3}$	1451	1618
6	4	3	290	-241
6	4	$\bar{3}$	80-	-2
6	6	3	181	187
6	6	$\bar{3}$	441	-435
6	0	4	360	-377
6	0	$\bar{4}$	1636	-1935
6	2	4	190	-128
6	2	$\bar{4}$	224	-227
6	4	4	214	194
6	4	$\bar{4}$	826	798
6	6	4	40-	34
6	6	$\bar{4}$	53-	12
6	2	5	81	-72
6	2	$\bar{5}$	1215	-1362
6	4	5	146	-126
6	4	$\bar{5}$	198	-196
6	6	5	34	-43
6	6	5	441	426
6	0	6	295	-316
6	0	$\bar{6}$	842	861
6	2	6	259	-256
6	2	$\bar{6}$	120	91
6	4	6	63-	2
6	4	$\bar{6}$	482	-573
6	6	$\bar{6}$	60	38
6	2	7	355	343
6	2	$\bar{7}$	472	479
6	4	7	245	227
6	4	$\bar{7}$	144	-101
6	6	$\bar{7}$	380	-386
6	0	8	194	180
6	0	$\bar{8}$	214	-167
6	2	8	88-	0
6	2	$\bar{8}$	217	202
6	4	8	243	-202
6	4	$\bar{8}$	461	428
6	6	$\bar{8}$	97	-78
6	2	9	91	-77
6	2	$\bar{9}$	91	-69
6	4	9	167	175
6	4	$\bar{9}$	240	209
6	6	$\bar{9}$	225	218
6	0	10	210	-235
6	0	$\overline{10}$	393	328
6	2	10	100	108
6	2	$\overline{10}$	100-	24
6	4	$\overline{10}$	176	-153
6	2	11	248	304
6	2	$\overline{11}$	356	319
6	4	$\overline{11}$	244	219
6	0	$\overline{12}$	167	-126
6	2	$\overline{12}$	139	-110
6	4	$\overline{12}$	116	-98
6	2	$\overline{13}$	138	121
6	4	$\overline{13}$	32-	-2
6	0	$\overline{14}$	363	-298
6	2	$\overline{14}$	50	-35
6	2	$\overline{15}$	236	-291
7	1	0	733	-705
7	3	0	303	227
7	5	0	187	150
7	1	1	664	535
7	1	$\bar{1}$	449	-493
7	3	1	558	474
7	3	$\bar{1}$	437	-391
7	5	1	76-	24
7	5	$\bar{1}$	250	211
7	1	2	106-	-5
7	1	$\bar{2}$	557	610
7	3	2	120	-102
7	3	$\bar{2}$	575	-524
7	5	2	141	117
7	5	$\bar{2}$	337	-319
7	1	3	231	186
7	1	$\bar{3}$	368	388
7	3	3	484	424
7	3	$\bar{3}$	215	207
7	5	3	200	186
7	5	$\bar{3}$	608	-569
7	1	4	567	498
7	1	$\bar{4}$	167	-184
7	3	4	56-	-23
7	3	$\bar{4}$	797	759
7	5	4	186	-141
7	5	$\bar{4}$	302	286
7	1	5	435	-389
7	1	$\bar{5}$	701	-764
7	3	5	323	-274
7	3	$\bar{5}$	545	-514
7	5	5	195	179
7	5	$\bar{5}$	603	561
7	1	6	141	-136
7	1	$\bar{6}$	906	1013
7	3	6	417	375
7	3	$\bar{6}$	558	-636
7	5	6	76	71
7	5	$\bar{6}$	373	-331
7	1	7	333	326
7	1	$\bar{7}$	938	1010
7	3	7	97	86
7	3	$\bar{7}$	849	845
7	5	7	320	-327
7	5	$\bar{7}$	231	-216
7	1	8	287	254
7	1	$\bar{8}$	743	-768
7	3	8	388	-363
7	3	$\bar{8}$	572	529
7	5	$\bar{8}$	285	263
7	1	9	241	-266
7	1	$\bar{9}$	449	-429
7	3	9	194	-179
7	3	$\bar{9}$	187	-164
7	5	$\bar{9}$	401	370
7	1	10	329	-363
7	1	$\overline{10}$	218	207
7	3	$\overline{10}$	448	-413
7	5	$\overline{10}$	172	-158
7	1	11	206	265
7	1	$\overline{11}$	185	169
7	3	$\overline{11}$	119	93
7	5	$\overline{11}$	248	-234
7	1	$\overline{12}$	62	-45
7	3	$\overline{12}$	153	126
7	1	$\overline{13}$	131	-105
7	3	$\overline{13}$	242	-193
7	1	$\overline{14}$	211	176
7	3	$\overline{14}$	86	78
7	1	$\overline{15}$	46-	2
8	0	0	758	809
8	2	0	227	188
8	4	0	206	-176
8	6	0	76	-90
8	2	1	582	-626
8	2	$\bar{1}$	74	-35
8	4	1	168	-140
8	4	$\bar{1}$	90	-74
8	6	1	39-	-22
8	6	$\bar{1}$	44-	-6
8	0	2	696	-743
8	0	$\bar{2}$	911	873
8	2	2	66	-49
8	2	$\bar{2}$	307	317
8	4	2	383	355
8	4	$\bar{2}$	182	-164
8	6	2	36-	29
8	6	$\bar{2}$	102	-108
8	2	3	550	561
8	2	$\bar{3}$	519	552
8	4	3	73-	-16
8	4	$\bar{3}$	102	103
8	6	3	207	-272
8	6	$\bar{3}$	58	36
8	0	4	276	286
8	0	$\bar{4}$	737	-697
8	2	4	138	-126
8	2	$\bar{4}$	159	162
8	4	4	367	-357
8	4	$\bar{4}$	379	373
8	6	$\bar{4}$	112	-117
8	2	5	378	-367
8	2	$\bar{5}$	453	-486
8	4	5	169	175
8	4	$\bar{5}$	340	338
8	6	$\bar{5}$	291	316
8	0	6	378	-382
8	0	$\bar{6}$	427	437
8	2	6	89	76
8	2	$\bar{6}$	276	-290
8	4	6	388	391
8	4	$\bar{6}$	435	-436
8	6	6	209	208
8	2	7	484	494
8	2	$\bar{7}$	598	653
8	4	7	51	44
8	4	$\bar{7}$	196	-173
8	6	$\bar{7}$	403	-425
8	0	8	580	718
8	0	$\bar{8}$	658	-681
8	2	8	117	117
8	2	$\bar{8}$	99-	9
8	4	8	247	-330
8	4	$\bar{8}$	544	508
8	6	$\bar{8}$	25-	-29
8	2	9	366	-380
8	2	$\bar{9}$	712	-731
8	4	$\bar{9}$	172	-149
8	0	10	206	-244
8	0	$\overline{10}$	933	940
8	2	$\overline{10}$	232	212
8	4	$\overline{10}$	491	-450
8	2	$\overline{11}$	469	446
8	4	$\overline{11}$	52-	-11
8	0	$\overline{12}$	297	-234
8	2	$\overline{12}$	170	155
8	4	$\overline{12}$	303	276
8	2	$\overline{13}$	270	-230
8	0	$\overline{14}$	222	188
8	2	$\overline{14}$	77	-60
9	1	0	715	636
9	3	0	350	-279
9	5	0	404	-349
9	1	1	417	-353
9	1	$\bar{1}$	309	323

Table 3 (*cont.*)

h	k	l	$\lvert 10F_{\underline{o}}\rvert$	$10F_{\underline{c}}$	h	k	l	$\lvert 10F_{\underline{o}}\rvert$	$10F_{\underline{c}}$	h	k	l	$\lvert 10F_{\underline{o}}\rvert$	$10F_{\underline{c}}$	h	k	l	$\lvert 10F_{\underline{o}}\rvert$	$10F_{\underline{c}}$	h	k	l	$\lvert 10F_{\underline{o}}\rvert$	$10F_{\underline{c}}$	h	k	l	$\lvert 10F_{\underline{o}}\rvert$	$10F_{\underline{c}}$	h	k	l	$\lvert 10F_{\underline{o}}\rvert$	$10F_{\underline{c}}$
9	3	$\underline{1}$	286	-225	9	3	$\underline{8}$	156	-176	10	2	$\overline{4}$	152	186	11	1	$\overline{1}$	318	322	11	3	$\underline{9}$	103	-89	12	4	$\underline{7}$	56-	12	13	1	$\underline{10}$	235	-271
9	3	1	463	465	9	3	$\underline{8}$	390	364	10	4	$\underline{4}$	276	-275	11	3	$\underline{1}$	449	-414	11	1	$\underline{10}$	69	71	12	0	$\underline{8}$	412	405	13	3	$\underline{10}$	156	184
9	5	$\underline{1}$	376	338	9	5	8	64-	6	10	4	$\underline{4}$	260	-255	11	3	1	209	211	11	3	$\underline{10}$	93	88	12	2	$\underline{8}$	100	65	13	1	$\underline{11}$	189	-196
9	5	1	81-	-27	9	1	$\underline{9}$	58-	-68	10	6	4	36-	-36	11	5	$\underline{1}$	97	88	11	1	$\underline{11}$	60	45	12	4	$\underline{8}$	218	-259	13	1	12	172	188
9	1	$\underline{2}$	112	-89	9	1	$\underline{9}$	353	-333	10	2	$\underline{5}$	247	-262	11	5	1	210	-249	11	3	$\underline{11}$	48-	7	12	2	$\underline{9}$	253	312	14	0	0	124	-116
9	1	2	426	-434	9	3	$\underline{9}$	117	-80	10	2	5	351	378	11	1	$\underline{2}$	233	-262	11	1	$\underline{12}$	57	-53	12	4	$\underline{9}$	97	105	14	2	0	70-	71
9	3	$\underline{2}$	595	533	9	5	$\underline{9}$	348	345	10	4	$\underline{5}$	46-	31	11	1	2	291	-282	11	3	$\underline{12}$	93	85	12	0	$\underline{10}$	384	-355	14	2	$\underline{1}$	140	133
9	3	2	105	82	9	1	$\underline{10}$	439	389	10	4	$\underline{5}$	102	111	11	3	$\underline{2}$	235	201	11	1	$\underline{13}$	57	-53	12	2	$\underline{10}$	93	-97	14	2	1	77-	-61
9	5	$\underline{2}$	152	128	9	3	$\underline{10}$	404	-374	10	6	5	109	-146	11	3	2	385	382	11	1	14	40-	-14	12	2	$\underline{11}$	85	-100	14	0	$\underline{2}$	140	147
9	5	2	205	207	9	5	$\underline{10}$	158	-162	10	0	$\underline{6}$	123	-123	11	5	$\underline{2}$	56	49	12	0	0	135-	62	12	0	$\underline{12}$	81-	-40	14	0	2	162	177
9	1	$\underline{3}$	526	488	9	1	$\underline{11}$	410	385	10	0	6	105-	22	11	5	2	182	207	12	2	0	109-	-50	12	2	$\underline{12}$	87	-100	14	2	$\underline{2}$	50-	-4
9	1	3	219	-219	9	3	$\underline{11}$	291	271	10	2	$\underline{6}$	108	121	11	1	$\underline{3}$	129	162	12	4	0	153	-122	12	2	13	36-	23	14	2	$\underline{2}$	82-	9
9	3	$\underline{3}$	258	220	9	1	$\underline{12}$	477	-430	10	2	6	103-	6	11	1	3	249	-260	12	2	$\underline{1}$	104-	-54	13	1	0	218	-179	14	2	$\underline{3}$	113	166
9	3	3	260	-245	9	3	$\underline{12}$	278	243	10	4	$\underline{6}$	28-	181	11	3	$\underline{3}$	59-	8	12	2	1	243	319	13	3	0	55-	-34	14	0	$\underline{4}$	113	-74
9	5	$\underline{3}$	418	-422	9	1	$\underline{13}$	331	-289	10	6	6	21-	-12	11	3	$\underline{3}$	159	-146	12	4	$\underline{1}$	63	71	13	1	$\underline{1}$	109-	28	14	2	$\underline{4}$	85-	-87
9	5	3	81-	27	9	3	$\underline{13}$	303	-280	10	2	$\underline{7}$	60-	13	11	5	3	301	341	12	4	1	60-	49	13	1	1	122-	17	14	2	$\underline{5}$	85-	53
9	1	$\underline{4}$	482	480	9	1	14	170	155	10	2	$\underline{7}$	126	139	11	1	$\underline{4}$	67	-50	12	0	$\underline{2}$	129	128	13	3	$\underline{1}$	70	47	14	0	$\underline{6}$	243	-245
9	1	4	161	-157	10	0	0	492	519	10	4	7	81	75	11	1	4	190	190	12	0	2	645	-670	13	3	1	89	-97	14	2	$\underline{6}$	84-	-26
9	3	$\underline{4}$	395	-371	10	2	0	223	-205	10	0	$\underline{8}$	59	65	11	3	$\underline{4}$	175	-166	12	2	$\underline{2}$	96	75	13	1	$\underline{2}$	99-	-32	14	2	$\underline{7}$	174	-233
9	3	4	85	-56	10	4	0	537	-481	10	0	8	225	-224	11	3	$\underline{4}$	368	-364	12	2	2	116	-129	13	1	2	125-	-30	14	0	$\underline{8}$	206	202
9	5	$\underline{4}$	141	-146	10	2	$\underline{1}$	608	-561	10	2	$\underline{8}$	40-	-46	11	5	4	166	-182	12	4	$\underline{2}$	43-	6	13	3	$\underline{2}$	122	-120	14	2	$\underline{8}$	73-	5
9	5	4	94	83	10	2	1	347	370	10	2	$\underline{8}$	102-	-76	11	1	$\underline{5}$	80-	21	12	4	2	213	265	13	3	2	97	109	14	2	$\underline{9}$	123	169
9	1	$\underline{5}$	505	-510	10	4	$\underline{1}$	75-	61	10	4	$\underline{8}$	69-	-26	11	1	5	371	405	12	2	$\underline{3}$	85-	-87	13	1	$\underline{3}$	109	-107	14	0	$\underline{10}$	108	-96
9	1	5	133	-119	10	4	$\underline{1}$	306	-330	10	2	$\underline{9}$	214	-204	11	3	$\underline{5}$	77	62	12	2	$\underline{3}$	317	-418	13	1	3	129-	-107	14	2	10	55	57
9	3	$\underline{5}$	442	-412	10	6	1	306	-327	10	4	$\underline{9}$	63-	-22	11	3	$\underline{5}$	361	347	12	4	3	64-	12	13	3	$\underline{3}$	29-	-30	15	1	$\underline{0}$	166	-163
9	3	5	330	-317	10	0	$\underline{2}$	549	-572	10	0	$\underline{10}$	83-	39	11	5	5	197	-211	12	0	$\underline{4}$	97-	15	13	3	3	66	-75	15	1	$\underline{1}$	74-	-80
9	5	$\underline{5}$	153	159	10	0	2	673	-714	10	2	$\underline{10}$	96	-85	11	1	$\underline{6}$	66-	-24	12	0	4	396	395	13	1	$\underline{4}$	105	-99	15	1	$\underline{2}$	80-	55
9	5	5	157	-155	10	2	$\underline{2}$	102-	-55	10	4	$\underline{10}$	135	-129	11	1	6	455	-499	12	2	$\underline{4}$	73-	18	13	1	$\underline{4}$	233	273	15	1	$\underline{3}$	85-	60
9	1	$\underline{6}$	454	-459	10	2	2	106	113	10	2	$\underline{11}$	85-	22	11	3	$\underline{6}$	31-	-17	12	2	$\underline{4}$	117	-134	13	3	$\underline{4}$	104	-108	15	3	$\underline{3}$	29-	43
9	1	6	315	340	10	4	$\underline{2}$	371	339	10	4	$\underline{11}$	178	-181	11	3	$\underline{6}$	181	178	12	4	4	241	-306	13	1	$\underline{5}$	235	202	15	1	$\underline{4}$	88-	-54
9	3	$\underline{6}$	293	272	10	4	$\underline{2}$	452	471	10	0	$\underline{12}$	214	-181	11	5	6	153	163	12	2	$\underline{5}$	58-	35	13	3	$\underline{5}$	212	237	15	3	$\underline{4}$	74	66
9	3	$\underline{6}$	91-	-12	10	6	2	36-	-31	10	2	$\underline{12}$	65	64	11	1	$\underline{7}$	51-	-38	12	2	$\underline{5}$	250	321	13	1	$\underline{6}$	206	-207	15	1	$\underline{5}$	88	-98
9	5	6	117	-97	10	2	$\underline{3}$	579	579	10	2	$\underline{13}$	278	-280	11	1	$\underline{7}$	376	-378	12	4	5	168	-202	13	3	$\underline{6}$	115	138	15	1	$\underline{6}$	103	121
9	1	$\underline{7}$	235	234	10	2	3	700	-745	10	0	$\underline{14}$	356	360	11	3	$\underline{7}$	364	-352	12	0	$\underline{6}$	59-	19	13	1	$\underline{7}$	178	-191	15	3	$\underline{6}$	29-	18
9	1	7	219	212	10	4	$\underline{3}$	124	120	10	2	14	38-	43	11	5	$\underline{7}$	44-	38	12	0	6	287	-283	13	3	$\underline{7}$	89	-94	15	1	$\underline{7}$	80-	41
9	3	$\underline{7}$	177	181	10	4	$\underline{3}$	142	-137	11	1	0	530	481	11	1	$\underline{8}$	218	213	12	2	$\underline{6}$	32-	-46	13	1	$\underline{8}$	114-	34	15	1	$\underline{8}$	72-	30
9	3	$\underline{7}$	125	113	10	6	3	121	148	11	3	0	325	-264	11	3	$\underline{8}$	180	-172	12	2	$\underline{6}$	114-	124	13	3	$\underline{8}$	216	-252	15	1	$\underline{9}$	91	94
9	5	7	265	-239	10	0	$\underline{4}$	454	525	11	5	0	182	-168	11	5	$\underline{8}$	86	-83	12	4	$\underline{6}$	250	279	13	1	$\underline{9}$	206	216	16	0	$\underline{4}$	81	-100
9	1	$\underline{8}$	147	148	10	0	4	688	718	11	1	1	501	-472	11	1	9	98-	24	12	2	7	343	-402	13	3	9	111	101	16	0	6	64	73
9	1	8	64	66	10	2	4	109	100																									

and nearly parallel (the dihedral angle between them being 8·4°). The distances of the atoms from the least-squares weighted plane $0{\cdot}9674X'+0{\cdot}0731\,Y+0{\cdot}2425Z'=1{\cdot}0284$ (X',Y',Z' are coordinates in Å referred to the orthogonal axes x,y,z^*) are: 0·000 for Se, −0·005 for N(1) and N(2), +0·015 Å for C. The chlorine atom lies in this plane.

The most significant difference between the cations of the selenium- and sulphur-derivative concerns the orientation of the CNN planes with respect to the

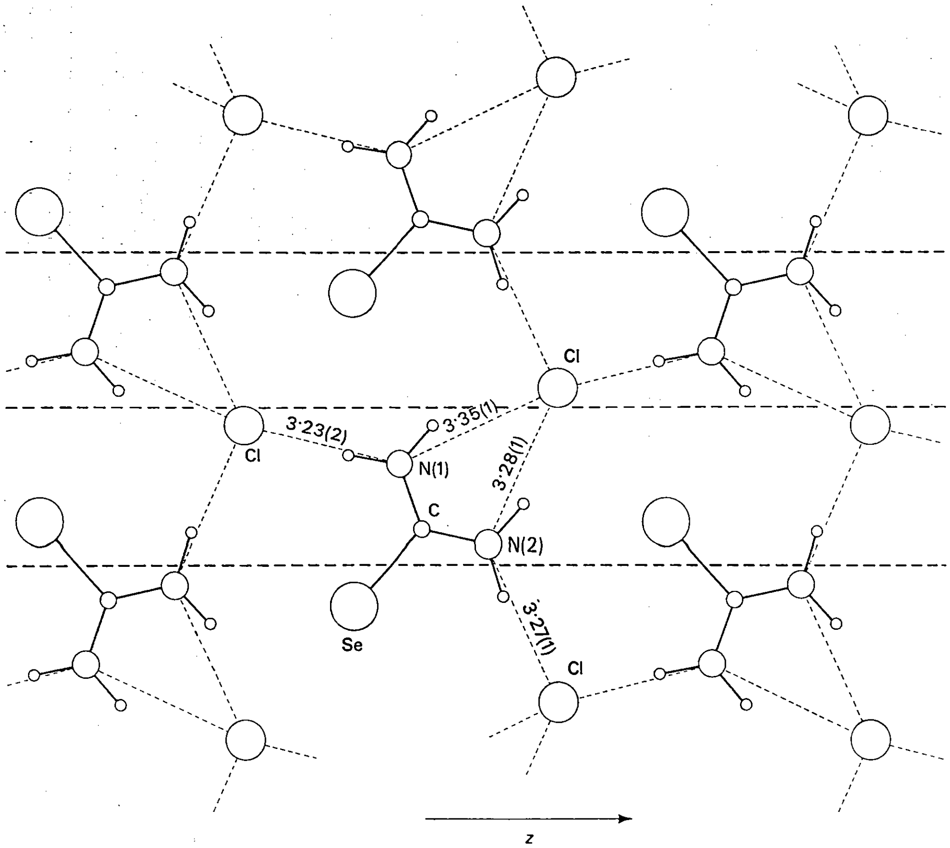

Fig. 2. Section of the structure on a plane parallel to (100) at $x=\frac{1}{8}$.

Se–Se or S–S bond; in the Se-compound these planes are nearly perpendicular to the Se–Se bond, while in the S compound they are nearly parellel.

This difference in conformation does not seem to arise from packing requirements. Thus in the two sulphur compounds, in which the environments of the cations are quite different, these units are very similar apart from some distortion probably caused by steric requirements.

The two parts of the cations are related by a twofold axis, so the cations can assume two enantiomorphous configurations; both are present in all these structures, one related to the other by a glide plane.

Packing is essentially determined by the hydrogen bonds which the NH_2 groups form with the chlorine atom: the chlorine atom is involved in four hydrogen bonds with three different selenourea groups:

Cl···N(1) = 3·35 (1) Å ClHN(1) = 146·5°
Cl···N(1^{ii}) = 3·23 (2) ClHN(1^{ii}) = 167·0
Cl···N(2) = 3·28 (1) ClHN(2) = 151·9
Cl···N(2^{iii}) = 3·27 (1) ClHN(2^{iii}) = 166·7 .

ii $x, \bar{y}-1, z+\frac{1}{2}$
iii $x, y-1, z$.

This hydrogen bond system lies in a plane nearly parallel to (100) which is shown in Fig. 2. The structure is therefore formed by double layers of selenourea groups and chlorine atoms joined together by the Se–Se bonds. These layers are packed along x^* in such a way that each chlorine atom is nearly collinear with the Se–Se bond (the angle Cl···Se–Se is 169·4°). In Fig. 3 these layers are projected on (010).

It is interesting to observe that the direction along which the electric vector vibrates when, looking along [001], the crystals appear yellow, is parallel to the Cl···Se–Se···Cl line and therefore perpendicular to the selenourea planes. The distance Se···Cl on that line is particularly short (3·19 Å) considering the van der Waals radii generally assumed (r_{Cl} = 1·80 and r_{Se} = 1·73 Å). The other van der Waals contacts (less than 3·5 Å) are as follows:

Se–C^{i} = 3·21 (2) Å N(1)–N(1^{i}) = 3.30 (2) Å
Se–N(1^{iii}) = 3.46 (1) N(1)–N(2^{iv}) = 3.38 (3)
Cl–C^{iv} = 3.24 (2)
i $\bar{x}, y, \frac{1}{2}-z$
iv $\frac{1}{2}-x, -\frac{1}{2}-y, 1-z$

Unlike thioformamidinium dibromide and diiodide which both crystallize as hydrates, the selenium com-

Table 4. *Comparison of the structural parameters for the cations* $^{+}(NH_2)_2CX\text{–}XC(NH_2)_2^{+}$, (X = Se,S)

	$Cl(NH_2)_2CSe\text{–}SeC(NH_2)_2Cl$ (present paper)	$Br(NH_2)_2CS\text{–}SC(NH_2)_2Br$ Foss *et al.* (1958)	$I(NH_2)_2CS\text{–}SC(NH_2)_2I$ Foss *et al.* (1958)
X–X^{i}	2·380 (6) Å	2·044 (10) Å	2·044 (20) Å
X–C	1·94 (1)	1·78 (3)	1·75 (4)
C–N(1)	1·32 (2)	1·33 (3)	1·33 (4)
C–N(2)	1·30 (2)	1·33 (3)	1·36 (4)
X^{i}XC	95·5 (6)°	104·0°	98·9°
XCN(1)	117·7 (1·2)°	121·2	122·9
XCN(2)	119·6 (1·0)	118·3	113·1
N(1)CN(2)	122·7 (1·4)	119·0	116·3
(X^{i}X) ∧ (XCNN)	5·1°	88·8	76·3
(X^{i}XC) ∧ ($XX^{i}C^{i}$)	89·5	89·2	104·8

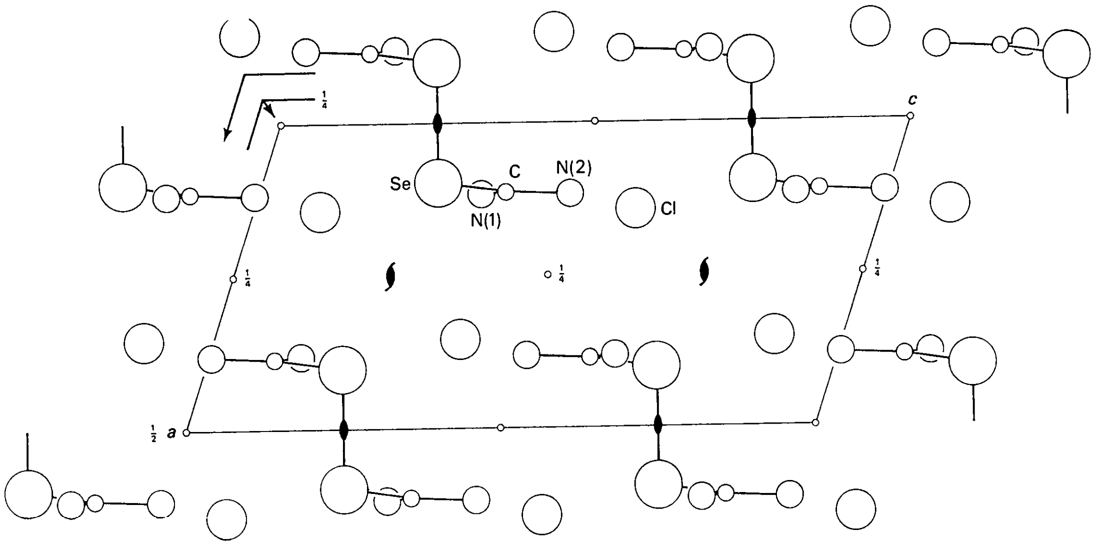

Fig. 3. Projection of the structure on (010).

pound is anhydrous. There is no room for water molecules in its crystal structure.

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Structure Cristalline de Composés Antituberculeux.
III. Structure Cristalline de la Propyl-2-thiocarbamoyl-4-pyridine

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(*Recu le* 22 *septembre* 1969)

2-Propyl-4-thiocarbamoylpyridine ($C_9N_2H_{12}S$) crystallizes in the monoclinic system, space group $P2_1/c$, with cell dimensions $a=7{\cdot}84\pm0{\cdot}02$, $b=17{\cdot}11\pm0{\cdot}02$, $c=7{\cdot}44\pm0{\cdot}02$ Å, $\beta=103°17'\pm50'$ and 4 molecules per cell. The refinement was carried out by least-squares calculations including anisotropic temperature factors. The final R value is 0·088. The structure is made up of chains of molecules parallel to the [201] direction; the molecules are linked together by hydrogen bonds.

Introduction

C'est en poursuivant toujours le même but, à savoir, la recherche d'analogies entre la structure et l'activité des molécules, que nous avons continué la détermination des structures cristallines dans la série de l'éthionamide (Colleter & Gadret ,1967, 1968 *a*, *b*).

La présente note rapporte celle du dérivé propylé en 2: la propyl-2-thiocarbamoyl-4-pyridine. Ce composé est particulièrement intéressant car il s'agit d'un des produits les plus actifs de cette série: il est d'ailleurs utilisé en thérapeutique (Colleter & Gadret, 1967).

Partie expérimentale

La propyl-2-thiocarbamoyl-4-pyridine se présente sous forme d'une poudre cristalline jaune d'or peu soluble dans l'eau, soluble dans l'alcool, l'acétone et dans la plupart des solvants organiques apolaires.

Les cristaux utilisés ont été obtenus à partir d'une solution acétonique saturée. Ils ont la forme de petits prismes allongés suivant la direction **c**, de dimensions approximatives 3 à 5 ×0·4 à 0·6 mm.

Les diagrammes de Bragg et de De Jong, la rotation du cristal s'effectuant autour de l'axe d'allongement **c**, ont été obtenus avec le rayonnement $K\alpha$ du cuivre.

Données cristallographiques

Système monoclinique; groupe spatial $P2_1/c$.

$$a=\ 7{\cdot}84\pm0{\cdot}02\ \text{Å}$$
$$b=17{\cdot}11\pm0{\cdot}02\ \text{Å}$$
$$c=\ 7{\cdot}44\pm0{\cdot}02\ \text{Å}$$
$$\beta=103°17'\pm50'$$

volume de la maille: $v=970{\cdot}939$ Å^3

nombre de molécules par maille: $z=4$

Mesure des intensités et calcul des F_o (hkl)

Ces mesures ont été faites avec un microdensitomètre Nonius sur des clichés de De Jong obtenus pour différents temps de pose.

Nous avons ainsi noté, sur les sept plans réciproques 1278 réflexions dont 979 seulement sont mesurables.

Les transmissions fournies par l'appareil, pour chaque tache, sont ensuite transcrites sur carte pour per-