tices and 24 edges to the decahedron, but as basal faces are shared there are 12 vertices and 20 edges per unit of the column. There are then four hydrogen atoms excess to each unit, one *per* three water molecules, and these form hydrogen bonds to the phenolic oxygen atoms of the nickel complex. The column of water molecules, infinite in one-dimension, represents an extremely compact assembly of water molecules provided that hydrogen acceptors of appropriate number and dimension are available, but it could not be extended to a three-dimensional structure in their absence. The structure is then in no sense a clathrate or potential clathrate, but is an interesting intermediate between these compounds and more conventional hydrates in which the arrangement of water molecules, *per se*, is less extensive.

References

CRADWICK, P. D. & HALL, D. (1971). *Acta Cryst.* **B27**, 1468.

DAS SARMA, B. & BAILAR, J. C. (1955). *J. Amer. Chem. Soc.* **77**, 5476.

DODSON, G. G. & HALL, D. (1961). *J. Inorg. Nucl. Chem.* **23**, 33.

FOX, M. R. & LINGAFELTER, E. C. (1967). *Acta Cryst.* **22**, 943.

JEFFREY, G. A. & MCMULLAN, R. K. (1967). *Progr. Inorg. Chem.* **8**, 43.

LINGAFELTER, E. C. & BRAUN, R. L. (1966). *J. Amer. Chem. Soc.* **88**, 2951.

MAIN, P. & WOOLFSON, M. M. (1963). *Acta Cryst.* **16**, 731.

RAE, A. D. (1965). *Acta Cryst.* **19**, 683.

STEWART, J. M., LINGAFELTER, E. C. & BREAZEALE, J. D. (1961). *Acta Cryst.* **14**, 888.

Acta Cryst. (1972). B**28**, 49

The Crystal and Molecular Structure of 2-Dimethylaminoethyl Selenolbenzoate Hydrochloride

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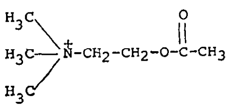
The crystal structure of the local anesthetic 2-dimethylaminoethyl selenolbenzoate hydrochloride has been determined. The crystals are monoclinic, $P2_1/c$ with $a=12{\cdot}287$ (3), $b=6{\cdot}945$ (1), $c=15{\cdot}523$ (3) Å and $\beta=94{\cdot}57$ (2)°. Least-squares refinement on 2411 observed reflections resulted in an R value of 0·051. The $\overset{+}{N}$–C–C–Se group adopts the *trans* conformation with a torsion angle about the C–C bond of 174·4°. The two Se–C bond distances are both 1·945 Å with estimated standard deviations of 0·005 Å. The C–Se–C angle is 96·4 (2)°. Cell constants for four related compounds are also given.

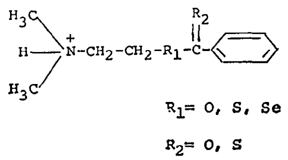
Introduction

The conformations of biologically active molecules related to acetylcholine (I) have been extensively studied (Chothia, 1970; Chothia & Pauling, 1970; Shefter & Mautner, 1969). These molecules all incorporate the $\overset{+}{N}$–C–C–R_1 group where R_1 may be oxygen, sulfur or selenium. While these short chains are formally flexible, the $\overset{+}{N}$–C–C–O group usually adopts a *gauche* conformation (Pauling, 1968; Sundaralingam, 1968; Chothia & Pauling, 1970). Other studies show that the $\overset{+}{N}$–C–C–S and $\overset{+}{N}$–C–C–Se groups take up the *trans* conformation (Shefter & Mautner, 1969).

In this work five members of a series of local anesthetics related to the tertiary analog of benzoylcholine, 2-dimethylaminoethyl benzoate (II, with $R_1=O$, $R_2=O$) were investigated. These compounds are obtained by systematically replacing the ether oxygen in the parent molecule (position R_1) with sulfur or selenium and the carboxyl oxygen (position R_2) with sulfur. Only the selenolester 2-dimethylaminoethyl selenolbenzoate hydrochloride ($R_1=Se$, $R_2=O$) was selected for complete structure determination.



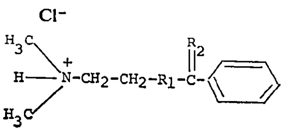
(I)



(II)

Crystals of the parent compound ($R_1=O$, $R_2=O$) proved highly deliquescent and were not investigated. The structure of the closely related 2-diethylaminoethyl *p*-aminobenzoate HCl (procaine HCl) was determined instead and a discussion of these results will appear subsequently (Dexter, 1972).

Table 1. *Cell Constants for*



$R_1 = O, S, Se$
$R_2 = O, S$
Space Group $P2_1/c$

	$R_1=S, R_2=S$	$R_1=Se, R_2=S$	$R_1=Se, R_2=O$	$R_1=S, R_2=O$	$R_1=O, R_2=S$
a (Å)	11·81 (1)	11·95 (1)	12·26 (1)	12·15 (1)	9·73 (1)
b (Å)	6·90 (1)	6·85 (1)	6·98 (1)	6·93 (1)	9·22 (1)
c (Å)	16·68 (1)	16·66 (1)	15·43 (2)	15·34 (1)	12·74 (1)
β(°)	92·8 (2)	92·9 (2)	94·8 (2)	94·4 (2)	115·2 (2)

Experimental

Shown in Table 1 are the approximate cell dimensions of the five compounds as measured from Weissenberg and precession photographs. In all cases systematic absences *h*0*l* for *l* odd and 0*k*0 for *k* odd uniquely determine the space group as $P2_1/c$.

A crystal of the selenolester, approximately 0·18 × 0·20 × 0·14 mm, was cut from a large, colorless plate and mounted with **b** along the φ axis of a General Electric computer-controlled diffractometer. The 2θ values (Cu $K\alpha_1$, $\lambda = 1{\cdot}54050$ Å) of 15 carefully centered high angle reflections were used in a least-squares refinement of the lattice constants. (Crystal data are presented in Table 2.) The intensities of the 2810 independent reflections with $2\theta < 155°$ (Cu $K\bar{\alpha}$, $\lambda = 1{\cdot}5418$ Å) were measured using θ–2θ scans. The 399 reflections for which $I_{\text{obs}} < 2\sigma(I_{\text{obs}})$ were all treated as unobserved and assigned an intensity equal to 2σ (I_{obs}). The intensities were corrected for Lorentz and polarization factors but not for absorption.

During data collection the intensities of the three standard reflections decreased linearly at rates which seemed to be a function of 2θ. The crystal also became yellow-brown in color (possibly due to liberation of free selenium). Although the losses in intensity (9 to 33%) were significant, the solution of the structure proceeded uneventfully and exploratory refinement cycles led to a chemically reasonable structure.

Table 2. *Crystal data for 2-dimethylaminoethyl selenolbenzoate hydrochloride*

Formula	$(CH_3)_2N(CH_2)_2Se\ COC_6H_5.HCl$
M.W.	292·7
Space group	$P2_1/c$
a	12·387 (3) Å
b	6·945(1)
c	15·523(3)
β	94·57(2)°
Volume	1331·2 Å³
ϱ(X-ray)	1·460 g.cm⁻³
ϱ(flotation)	1·47 g.cm⁻³
Z	4
μ	59·3 cm⁻¹

The estimated standard deviations of the lattice constants are three times those obtained from the least-squares calculation.

Structure determination and refinement

The selenium atom and chloride ion were readily located from an $E^2 - 1$ vector map. Coordinates and

Table 3. *Final positional and anisotropic thermal parameters for the non-hydrogen atoms*

The positional parameters for Cl⁻ and Se are × 10⁵. All others are × 10⁴. The estimated standard deviations are those obtained from the least-squares and refer to the last decimal place given. The form of the anisotropic temperature factor is

$$\exp\left[-\tfrac{1}{4}(B_{11}h^2a^{*2} + B_{22}k^2b^{*2} + B_{33}l^2c^{*2} + 2B_{12}hka^*b^* + 2B_{13}hla^*c^* + 2B_{23}klb^*c^*)\right].$$

	x	*y*	*z*	B_{11}	B_{22}	B_{33}	B_{12}	B_{13}	B_{23}
C(1)	3598 (4)	1948 (6)	7904 (3)	4·9 (2)	4·4 (2)	3·1 (1)	−0·2 (1)	−0·7 (2)	0·1 (1)
C(2)	5371 (3)	2051 (7)	8721 (3)	3·8 (1)	4·6 (2)	5·0 (2)	0·3 (1)	0·7 (2)	0·4 (1)
C(3)	3744 (2)	1892 (4)	9518 (2)	3·7 (1)	2·7 (1)	3·0 (1)	0·0 (1)	—0·2 (1)	0·6 (1)
C(4)	2635 (3)	2699 (6)	9633 (2)	3·4 (1)	4·2 (2)	3·7 (1)	0·4 (1)	0·2 (1)	1·0 (1)
C(5)	8458 (2)	4451 (5)	4650 (2)	3·4 (1)	4·5 (2)	2·9 (1)	0·3 (1)	0·2 (1)	0·2 (1)
C(6)	8747 (2)	3128 (5)	3950 (2)	3·0 (1)	3·9 (1)	2·9 (1)	−0·3 (1)	0·2 (1)	0·1 (1)
C(7)	8210 (3)	3159 (5)	3128 (2)	3·0 (1)	4·4 (2)	3·1 (1)	−0·0 (1)	−0·1 (1)	0·0 (1)
C(8)	8510 (3)	1892 (5)	2500 (2)	3·7 (1)	5·1 (2)	3·4 (1)	−0·8 (1)	−0·1 (1)	−0·7 (1)
C(9)	9353 (3)	0642 (6)	2680 (2)	4·6 (2)	4·5 (2)	4·5 (2)	−0·1 (1)	0·5 (2)	−1·0 (1)
C(10)	9894 (3)	0580 (6)	3504 (3)	4·2 (1)	4·4 (2)	5·2 (2)	0·8 (1)	−0·0 (2)	−0.3 (1)
C(11)	9587 (3)	1804 (5)	4133 (2)	4·1 (1)	4·3 (2)	3·7 (2)	0·2 (1)	—0·6 (1)	0·2 (1)
N	4207 (2)	2611 (4)	8711 (2)	3·5 (1)	2·4 (1)	2·9 (1)	−0·1 (1)	−0·1 (1)	0·4 (1)
O	8635 (3)	4106 (5)	5406 (2)	7·3 (2)	6·2 (2)	2·6 (2)	1·6 (1)	0·1 (1)	0·1 (1)
Cl⁻	58321 (8)	19387 (1)	62870 (6)	5·35 (4)	2·22 (3)	4·45 (4)	−0·08 (2)	−0·62 (3)	−0·02 (2)
Se	21848 (3)	18784 (6)	07473 (2)	4·98 (2)	4·76 (2)	3·64 (2)	−0·51 (1)	0·47 (2)	−0·25 (1)

isotropic thermal parameters for the heavy atoms and the scale factor were adjusted with one cycle of full-matrix least squares (with unit weights) to give an R value of 0·35. The subsequent electron density map revealed the other 13 non-hydrogen atoms. After several least-squares cycles with individual isotropic temperature factors R decreased to 0·12. At this point the hydrogen atoms were located from a difference map and included in the refinement with fixed isotropic temperature factors. After allowing the heavy atoms to refine anisotropically R was 0·075. Weights were then assigned from a plot of $|\Delta F|$ *vs.* $|F_o|$. Several intense low angle reflections appeared to be affected by extinction and were excluded from the remainder of the refinement. Using anisotropic thermal parameters for all non-hydrogen atoms and isotropic thermal parameters for hydrogen atoms, the refinement converged with $R=0{\cdot}051$. In the last cycle for the 200 parameters refined, the average change was $0{\cdot}04\sigma$ and the maximum change was $0{\cdot}4\sigma$. The final difference map showed a peak of about 0·5 electron at the position of the chloride ion and a pattern of peaks and holes about the selenium atom position with heights of about ± 1·0 electron. While this pattern might suggest incomplete refinement of the anisotropic thermal parameters, it also could be a manifestation of the crystal decay noted during the data collection.

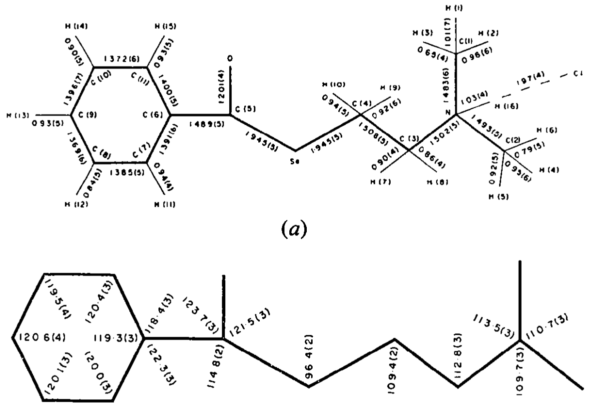


Fig. 1.(*a*) Bond distances in 2-dimethylaminoethyl selenolbenzoate hydrochloride. (*b*) Bond angles involving non-hydrogen atoms. (Angles involving H atoms are within expected limits.)

Scattering factors for carbon, nitrogen and oxygen were taken from *International Tables for X-ray Crystallography* (1962), for selenium and chlorine (Cl^-) from Cromer & Waber (1964) and for hydrogen from Stewart, Davidson & Simpson (1965). The bulk of the computations was carried out with the *X-ray 67 System* programs (Stewart, 1967). The mean plane program used was that of Ahmed, Hall, Pippy & Saunderson (1966).

Table 4. *Final coordinates and isotropic thermal parameters for the hydrogen atoms*

The positional parameters are all × 10^3.

	x	y	z	B
H(1)	356 (5)	050 (9)	792 (4)	8 (1)
H(2)	379 (4)	239 (8)	736 (4)	7 (1)
H(3)	309 (3)	217 (6)	787 (3)	3 (1)
H(4)	545 (4)	070 (9)	866 (3)	6 (1)
H(5)	574 (4)	262 (7)	919 (3)	5 (1)
H(6)	560 (4)	245 (8)	830 (3)	5 (1)
H(7)	377 (3)	061 (6)	957 (2)	3 (1)
H(8)	420 (2)	217 (5)	995 (2)	3 (1)
H(9)	205 (5)	242 (9)	926 (4)	7 (1)
H(10)	269 (3)	405 (7)	962 (3)	4 (1)
H(11)	761 (3)	398 (6)	303 (3)	4 (1)
H(12)	825 (4)	194 (7)	198 (3)	6 (1)
H(13)	956 (4)	−018 (7)	225 (3)	6 (1)
H(14)	046 (4)	−018 (8)	369 (3)	7 (1)
H(15)	002 (4)	188 (6)	465 (3)	6 (1)
H(16)	418 (3)	410 (6)	871 (2)	3 (1)

Table 5. *Torsion angles*

	Angle (τ)
C(11)-C(6)-C(5)-Se	153·2°
C(6)—C(5)-Se—C(4)	169·4
O——C(5)-Se—C(4)	166·9
C(5)—Se—C(4)-C(3)	87·4
Se——C(4)-C(3)-N	174·4
C(4)—C(3)-N——C(2)	169·7
C(4)—C(3)-N——C(1)	65·9

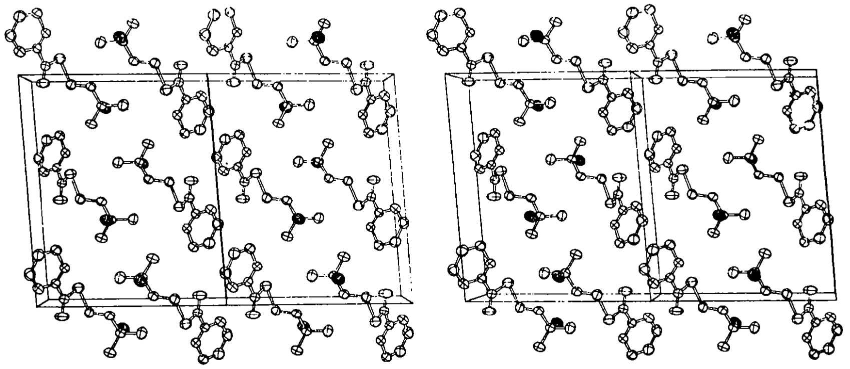


Fig. 2. Stereo diagrams (Johnson, 1965) showing molecular packing along *b*. *a*→, *c* ↑ . Hydrogen atoms have been omitted. Chloride ions have been shaded.

Results and discussion

Final coordinates and thermal parameters for non-hydrogen atoms are given in Table 3 and for hydrogen atoms in Table 4. Fig. 1 shows bond distances, bond angles and the numbering system. Some torsion angles along the side chain are listed in Table 5. Table 6 is the list of observed and calculated structure factors.

Table 6. *Observed and calculated structure factors*

The columns are l, $10F_o$ and $10F_c$. Unobserved reflections are marked with an asterisk. Reflections marked with an E were excluded from the final refinement cycles.

0,0,L		
2	840	820
4	823	-784
6	1085	-1028
8	281	-271
10	285	-267
12	63	-51
14	252	225
16	142	136
18	193	-164
1,0,L		
0	144	137
-2	709	739
2	475	-462
-4	1467	1480
4	41	-48
-6	287	-271
6	635	-597
-8	1152	-1136
8	200	199
-10	972	-878
10	883	841
-12	210	195
12	563	525
-14	148	156
14	342	-324
-16	105	102
16	371	-360
-18	139	148
2,0,L		
0	452	-477
-2	717	728
2	1887E	-2398
-4	334	-297
4	876	-820
-6	650	673
6	857	811
-8	731	720
8	1310	1249
-10	25*	17
10	115	-96
-12	469	-475
12	261	-248
-14	267	-263
14	74	-69
-16	79	81
16	133	-128
18	23*	-10
-18	172	191
3,0,L		
0	834	-888
-2	1440E	-1676
2	673	686
-4	480	-475
4	718	724
-6	552	512
6	314	318
-8	389	428
8	100	90
-10	323	323
10	166	-168
-12	156	136
12	451	-430
-14	132	142
14	150	-146
-16	339	-352
16	127	125
-18	72	-76
4,0,L		
0	73	-62
-2	219	221
2	494	542
-4	757	-842
4	181	177
-6	954	-992
6	365	276
-8	259	-245
8	647	-636
-10	624	622
10	495	-486
-12	471	488
12	374	377
-14	246	235
14	379	371
-16	117	-120
16	117	112
-18	127	-128
5,0,L		
0	1268	1383
2	23*	2
-2	615	687
-4	199	-220
4	988	-989
-6	20*	-12
6	746	-730
-8	629	-634
8	71	66
-10	424	-426
10	277	275
-12	76	-81
12	298	300
-14	407	420
14	174	175
-16	262	274
16	44	56
-18	73	-79
6,0,L		
0	451	-449
-2	206	216
2	658	-711
-4	1006	1098
4	124	-134
6	26*	-27
-6	551	563
-8	369	-371
8	41	48
-10	446	-467
10	423	439
-12	218	-213
12	222	229
-14	55	-37
14	247	-265
-16	111	108
16	226	-250
-18	153	148
7,0,L		
0	61	-86
-2	162	138
2	516	-494
-4	156	-150
4	114	-107
-6	572	597
6	546	553
-8	821	846
8	426	429
-10	69	53
10	284	-295
-12	333	-359
12	326	-334
-14	391	-409
14	144	-161
16	21*	23
-16	65	-56
-18	19*	19
8,0,L		
0	25*	13
-2	416	-448
2	782	809
-4	318	-335
4	601	633
-6	70	63
6	155	-140
-8	56	61
8	217	-221
-10	105	103
10	154	-156
-12	186	180
12	192	-203
-14	41	-41
14	107	-124
-16	226	-236
9,0,L		
0	288	313
2	29*	28
-2	443	433
-4	395	-422
4	220	-222
-6	716	-737
6	167	166
-8	173	-170
8	170	-170
-10	181	218
10	124	-131
-12	198	211
12	129	130
-14	195	189
14	190	215
-16	74	64
10,0,L		
0	29*	10
-2	208	219
2	92	-85
-4	204	214
4	592	-592
-6	183	182
6	224	-224
-8	253	-252
8	121	124
-10	275	-288
10	271	280
-12	28*	-12
12	94	94
14	19*	-13
-14	208	218
-16	126	129
11,0,L		
0	313	-312
-2	85	91
2	417	-411
-4	296	297
4	50	56
-6	193	180
6	370	369
-8	51	-55
8	46	45
-10	59	-73
10	44	43
-12	168	-164
12	32	-32
-14	105	-100
12,0,L		
0	99	-98
2	31*	-5
-2	277	-289
-4	165	-176
4	154	152
-6	145	142
6	279	274
-8	244	246
8	102	105
-10	26*	6
10	57	-58
-12	30	-38
13,0,L		
0	45	36
-2	93	-93
2	178	162
-4	90	-72
4	239	238
-6	67	-66
6	111	-112
-8	25*	27
8	144	-155
-10	22*	10
-12	117	118
14,0,L		
0	165	173
-2	131	141
2	44	-35
-4	26*	20
4	48	-48
-6	131	-135
6	76	-72
-8	22*	8
-10	35	-33
15,0,L		
0	22*	1
-2	21*	3
2	22*	-7
-4	119	122
-6	92	99
0,1,L		
1	706	643
2	142	-126
3	605	543
4	885	-832
5	61	29
6	171	142
7	603	-531
8	266	277
9	297	-271
10	537	517
11	90	87
12	369	346
13	29*	-23
14	64	-60
15	29*	15
16	252	-259
17	46	-45
18	198	-194
19	26	-18
1,1,L		
0	632E	-748
1	14*	3
-1	361	357
-2	949	-1023
2	837	-837
-3	874	877
3	340	-376
-4	152	168
4	229	-217
-5	130	-112
5	90	-89
-6	1118	1089
6	362	361
-7	52	-58
7	43	74
-8	806	771
8	731	671
-9	278	-275
9	192	179
-10	76	-63
10	149	133
-11	121	-114
11	87	85
-12	361	-372
12	195	-183
-13	129	-122
13	114	-110
-14	410	-413
14	364	-345
-15	95	90
15	70	-63
-16	27*	18
16	41	-32
-17	97	105
17	107	-108
-18	48	49
18	63	64
-19	20*	10
19	53	-58
2,1,L		
0	484	492
1	509	-535
-1	772	-832
-2	1034	-1155
2	533	524
-3	361	351
3	93	64
-4	174	-178
4	1506	1546
-5	314	329
5	117	105
-6	56	53
6	115	128
-7	425	403
7	120	117
-8	453	457
8	486	-451
-9	161	-155
9	376	333
-10	557	557
10	546	-526
-11	52	-51
11	27*	-9
-12	158	149
12	397	-386
-13	131	-130
13	177	-169
-14	398	-394
14	91	-89
-15	29*	-16
15	111	-106
-16	291	-303
16	115	113
-17	26*	29
17	28	21
-18	95	-94
18	146	151
-19	70	72
19	47	50
3,1,L		
0	1357E	1577
1	423	-421
-1	893	-970
-2	215	210
2	1318	1043
-3	102	-112
3	76	81
-4	1158	-1218
4	54	47
-5	500	-479
5	713	681
-6	1182	-1190
6	272	-270
-7	37	-29
7	179	173
-8	26	-28
8	321	-332
-9	347	345
9	33	-78
-10	232	204
10	316	-302
-11	233	226
11	174	-166
-12	432	433
12	39	-42
-13	41	-34
13	28*	6
-14	143	128
14	242	233
-15	48	-54
15	130	98
-16	63	-63
16	179	184
-17	26*	11
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11	273	-265
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7	219	216
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11	74	-77
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12	111	-117
-13	195	-142
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14	26*	25
15	157	154
16	35	36
17	137	133
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13	97	69
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3	186	-203
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6	28*	20
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8	28*	9
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9	143	-127
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11	358	369
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14	77	73
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16	22*	15
-16	23*	29
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6	122	-113
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15	109	-109
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-17	53	55
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-5	30	16
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7	87	85
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13	232	-209
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Table 6 (*cont.*)

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4	68	62
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9	212	-224
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-7	47	40
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0	311	297
1	50	36
2	98	93
3	63	-64
4	189	-165
5	271	-250
6	236	-221
7	31*	1
8	108	-106
9	196	194
10	76	70
11	222	219
12	102	101
13	58	-56
14	73	87
	1,6,L	
0	35	-22
1	458	-428
-1	27*	-26
-2	204	207
2	184	-177
-3	154	139
3	104	-95
-4	201	196
4	241	-231
-5	111	100
5	235	212
-6	31	10
6	156	-141
-7	78	85
7	255	236
-8	142	-133
8	56	48
-9	111	119
9	28*	-18
-10	88	-82
10	222	214
-11	26*	-10
11	25*	20
-12	52	48
12	36	93
-13	172	-195
13	30	-20
-14	36	30
14	25	-36
	2,6,L	
0	166	-173
1	183	170
-1	50	-47
-2	136	139
2	237	-220
-3	326	-341
3	159	148
-4	142	142
4	64	-59
-5	56	-58
5	102	100
-6	164	161
6	140	120
-7	178	173
7	60	52
-8	72	69
8	165	151
-9	108	116
9	27*	25
-10	54	-44
10	25*	22
-11	25*	-4
11	140	-147
-12	118	-112
12	99	-97
-13	56	-57
13	142	-145
-14	91	-95
	3,6,L	
0	96	-102
1	44	54
-1	28*	-3
-2	166	-160
2	211	206
-3	61	63
3	166	143
-4	73	-85
4	199	186
-5	195	-214
5	73	53
-6	26*	11
6	119	119
-7	148	-155
7	204	-200
-8	78	79
8	37	-26
-9	28*	8
9	172	-170
-10	55	54
10	80	-75
-11	190	200
11	37	37
-12	23*	-7
12	145	-147
-13	107	115
13	53	53
-14	36	-32
	4,6,L	
0	50	54
1	32*	14
-1	302	324
-2	41	38
2	176	173
-3	209	233
3	230	-225
-4	80	-77
4	51	43
-5	84	-77
5	242	-239
-6	184	-180
6	31*	-28
-7	27*	-10
7	43	-47
-8	34	-36
8	165	-157
-9	73	-73
9	27*	-5
-10	110	99
10	74	-65
-11	25*	-15
11	23*	-10
-12	109	113
12	40	41
-13	26	9
13	62	80
	5,6,L	
0	201	203
1	109	-111
-1	104	-105
-2	159	169
2	30*	4
-3	138	147
3	64	-61
-4	97	110
4	171	-173
-5	205	210
5	30*	-24
-6	63	-58
6	164	-165
-7	27*	19
7	46	44
-8	136	-135
8	53	-50
-9	139	-140
9	133	138
-10	87	-86
10	62	65
-11	129	-137
11	87	89
-12	21*	-7
12	56	54
-13	32	-32
	6,6,L	
0	63	-68
1	275	-268
-1	194	-194
-2	28*	1
2	146	-145
-3	29*	-6
3	41	-30
-4	174	180
4	29*	-11
-5	27*	-24
5	233	240
-6	50	50
6	29*	33
-7	57	53
7	218	220
-8	66	-57
8	85	84
-9	104	117
9	53	-55
-10	122	-123
10	120	125
-11	23*	9
11	20*	1
-12	50	-45
	7,6,L	
0	149	-145
1	134	137
-1	60	-56
-2	121	-133
2	130	-125
-3	227	-236
3	153	158
-4	28*	-33
4	70	74
-5	51	-53
5	28*	13
-6	62	56
6	100	94
-7	82	80
7	38	-31
-8	135	139
8	125	129
-9	58	53
9	39	-34
-10	22*	24
10	17*	9
-11	21*	19
-12	30	-19
	8,6,L	
0	27*	12
-1	60	69
1	56	47
-2	112	-107
2	95	89
-3	84	87
3	107	97
-4	85	-88
4	97	99
-5	71	-74
5	86	89
-6	49	-51
6	56	-65
-7	155	-157
7	151	-151
-8	63	57
8	21*	-15
-9	28	-27
9	110	-107
-10	67	67
-11	56	56
	9,6,L	
0	72	78
-1	115	117
1	63	52
-2	86	92
2	47	33
-3	85	78
3	139	-127
-4	25*	15
4	25*	-32
-5	24*	-8
5	132	-129
-6	131	-124
6	53	-52
-7	62	66
7	20*	0
-8	47	-52
-9	35	-32
	10,6,L	
0	36	27
-1	91	-93
1	113	-102
-2	105	104
2	44	-38
-3	84	83
3	23*	-8
-4	105	107
4	109	-101
-5	102	94
5	28	27
-6	24	27
-7	37	38
	11,6,L	
0	77	-82
-1	72	-62
1	39	-28
-2	20*	-13
2	74	-64
-3	54	-54
-4	72	71
-5	38	-49
	0,7,L	
1	41	-31
2	229	-211
3	120	-111
4	150	-137
5	30*	-22
6	129	125
7	154	155
8	55	45
9	79	76
10	45	36
11	53	-55
	1,7,L	
0	62	-66
1	31*	-14
-1	119	-137
-2	267	-276
2	30*	24
-3	117	-117
3	180	179
-4	27*	8
4	71	-65
-5	73	78
5	82	77
-6	290	293
6	29*	13
-7	25*	-25
7	63	-60
-8	158	164
8	200	201
-9	24*	-14
9	33	-26
-10	103	-100
10	92	97
-11	30	32
11	19*	15
	2,7,L	
0	99	-91
1	52	40
-1	140	156
-2	176	-134
2	52	45
-3	47	-42
3	68	-65
-4	28*	-31
4	355	340
-5	148	-155
5	37	39
-6	83	-91
6	105	101
-7	103	-100
7	42	43
-8	31	22
8	166	-162
-9	78	83
9	100	-102
-10	165	176
10	116	-115
-11	20*	8
11	39	-43
	3,7,L	
0	289	306
1	119	121
-1	52	54
-2	28*	-17
2	135	131
-3	31	-28
3	36	34
-4	291	-306
4	128	-132
-5	114	128
5	204	-204
-6	153	-152
6	89	-92
-7	62	60
7	100	-101
-8	112	109
8	23*	7
-9	88	-95
9	44	46
-10	75	76
10	58	-57
-11	67	-72
	4,7,L	
0	28*	-18
1	128	-134
-1	111	120
-2	146	168
2	41	32
-3	149	156
3	70	-66
-4	198	221
4	86	-83
-5	62	56
5	96	102
-6	23*	18
6	273	-274
-7	55	-56
7	41	39
-8	194	-207
8	104	-104
-9	22*	4
9	41	-45
-10	71	-72
10	105	105
-11	18*	-12
	5,7,L	
0	27*	13
1	54	63
-1	53	-56
-2	166	167
2	222	-210
-3	142	-160
3	28*	-31
-4	67	67
4	137	-136
-5	25*	-21
5	43	-46
-6	22*	-14
6	163	170
-7	122	128
7	101	106
-8	23*	9
8	118	122
-9	33	32
9	82	87
-10	38	-37
	6,7,L	
0	157	-165
1	90	-84
-1	59	-60
-2	264	-279
2	116	117
-3	25*	8
3	92	84
-4	25*	-15
4	62	63
-5	27	-18
5	57	58
-6	167	168
6	52	52
-7	36	-38
7	42	-38
-8	50	50
8	70	72
-9	20*	11
	7,7,L	
0	24*	24
1	83	77
-1	109	121
-2	24*	2
2	25*	14
-3	47	-47
3	47	-51
-4	88	-89
4	154	148
-5	83	-89
5	37	-39
-6	97	-102
6	55	55
-7	30	-24
7	43	40
-8	19*	-12
	8,7,L	
0	216	223
-1	22*	-27
1	24*	0
-2	22*	-9
2	114	108
-3	33	27
3	22*	15
-4	104	-102
4	71	-77
-5	45	51
5	47	-60
-6	52	-44
-7	22	-15
	9,7,L	
0	40	-48
-1	29*	23
1	92	-80
-2	104	100
2	60	-63
-3	34	40
-4	124	128
	0,8,L	
0	355	-370
1	27*	-25
2	75	-61
3	25*	-6
4	121	120
5	48	51
6	124	120
7	41	40
8	18*	16
	1,8,L	
0	24*	16
1	27*	19
-1	35	-32
-2	83	-79
2	80	73
-3	33	-32
3	25*	-1
-4	170	-176
4	61	59
-5	22*	5
5	24*	-5
-6	19*	-16
6	196	197
-7	19*	4
7	20*	3
-8	109	115
8	20	-7
	2,8,L	
0	35	41
1	34	-33
-1	24*	4
-2	104	-108
2	242	233
-3	37	42
3	24*	-13
-4	38	-23
4	83	81
-5	21*	-2
5	22*	-2
-6	81	-82
6	95	-96
-7	19*	5
7	25	-17
-8	73	-86
	3,8,L	
0	70	65
1	25*	6
-1	30	32
-2	179	189
2	115	-122
-3	23*	2
3	23*	-7
-4	77	76
4	106	-105
-5	21*	2
5	21*	-2
-6	63	-58
6	42	-42
-7	27	26
	4,8,L	
0	22*	16
1	36	-25
-1	32	-24
-2	21*	-26
2	46	-29
-3	21*	12
3	22*	-1
-4	85	86
4	47	-46
-5	19*	16
5	20*	6
-6	123	127
6	42	-46
	5,8,L	
0	169	-176
1	38	-31
-1	26	-19
-2	85	-89
2	28	-21
-3	20*	7
3	21*	-8
-4	29	16
4	135	136
-5	19*	-6
	6,8,L	
0	50	57
1	37	33
-1	18*	2
-2	30	-27
2	72	64
-3	18*	-11

As expected, the $\overset{+}{\mathrm{N}}$–C–C–Se group in 2-dimethylaminoethyl selenolbenzoate hydrochloride adopts the *trans* conformation. The torsion angle about the C(3)–C(4) bond is 174·4°. In structures of related selenol- and thiolesters the corresponding torsion angles range from 171 to 176° (Shefter & Mautner, 1969). Karle & Estlin (1969) found that the conformation of the $\overset{+}{\mathrm{N}}$–C–C–Se chain in 2-aminoethylselenic acid HCl is also *trans* with a torsion angle of 171·6°. It is very probable that the *trans* conformation is maintained in the four nearly isomorphous crystals (Table 1) investigated here.

The two chemically different C–Se distances are both 1·945 (5) Å and the C(5)–Se–C(4) angle is 96·4 (2)°. These distances are in good agreement with the others which have been tabulated by Hope, Knobler & McCullough (1970). They determined a weighted average Se–C distance for nonaromatic compounds of 1·98 (2) Å and 1·92 (2) Å for aromatic compounds. A comparison of the selenolester group found here with that reported for acetylselenolcholine iodide (Shefter & Kennard, 1966) would not be very meaningful because of the large standard deviations quoted for the latter structure.

The benzene ring is significantly nonplanar. For the mean plane of all six atoms C(6)···C(11), $0{\cdot}6616X+0{\cdot}6922Y-0{\cdot}2882Z-1{\cdot}497=0$, $\chi^2=300$. (The coefficients refer to an orthogonal set of axes where X lies along **a**, Y is in the (**a**,**b**) plane and Z lies along **c***.) $\chi^2=2{\cdot}6$ for the mean plane $0{\cdot}6682X-0{\cdot}6887Y-0{\cdot}2811Z-1{\cdot}4241=0$ through the four most nearly coplanar atoms C(8)···C(11). C(6) is 0·035 (3) Å below and C(7) 0·052 (4) Å above this plane.

There is little evidence of resonance interaction between the selenolester and phenyl groups. The C(5)–C(6) bond distance of 1·489 (5) Å is only 3·8σ shorter than the other C–C single bond in the molecule. The angle between the plane of the selenolester group O–C(5)–Se, $0{\cdot}9163X-0{\cdot}4000Y-0{\cdot}0166Z-1{\cdot}8542=0$, and the mean plane of the whole phenyl ring is 25·4°. This twist places the Se atom on the opposite side of the mean plane from C(7).

The conformation about the Se–C(4) bond ($\tau=87{\cdot}4°$) is not that normally found in esters (τ about 180°) (Mathieson, 1965) or in other selenol- and thiolesters (τ about 120°) (Shefter & Mautner, 1969). A similar conformation ($\tau=79°$) has been found in acetylcholine bromide (Canepa, Pauling & Sörum, 1966). Liquori, Damiani & deCoen (1968) concluded on the basis of total van der Waals energy calculations that in acetylcholine the observed *gauche* conformation was not significantly less stable than the *trans*. In the present structure, the observed torsion angle probably arises to accommodate the formation of different environments for the alkyl and aromatic parts of the molecule. A packing diagram (Fig. 2) shows that the protonated nitrogen atoms are hydrogen bonded to chloride ions (N–Cl, 3·07 Å) so that layers of charged groups alternate with layers of interlocking phenyl rings.

The smoothness of the refinement and the reasonableness of the final structure prompted an attempt to assess the effect of the crystal decay on the results.

The $hk0$ reflections were measured twice: at the beginning of the experiment as part of the hkl reflections (I_o) and later as part of the $hk\bar{l}$ reflections ($I_{1/2}$). Fig. 3 is a plot of $\ln(I_{1/2}/I_0)$ *versus* $\sin^2\theta/\lambda^2$. Assuming that the decrease in intensity can be described in terms of an apparent B parameter, by the end of the data collection the total effect of the decomposition was an increase in the overall temperature factor by about 1·5 Å^2.

The effect of a larger temperature factor is to increase the uncertainty in atomic positions, not to displace them (Cruickshank, 1949). It seems reasonable, therefore, to accept as valid the positional parameters obtained from the data affected by crystal decay – with the usual warning that the standard deviations obtained from the least-squares probably will be too small.

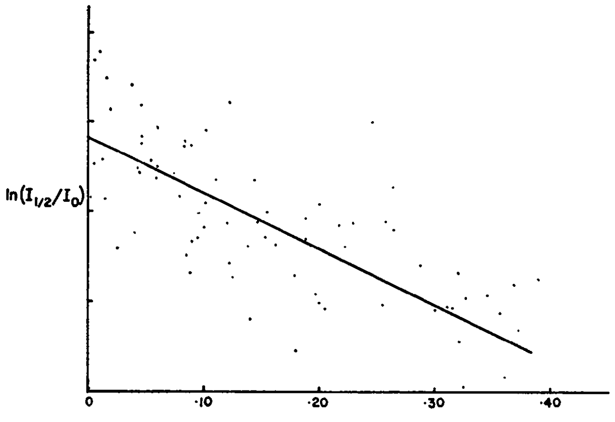

Fig. 3. Plot of $\ln(I_{1/2}/I_0)$ versus $(\sin^2\theta)/\lambda^2$ showing loss of intensity at midpoint of data collection for 75 $hk0$ reflections. The slope of the least-squares straight line corresponds to a B parameter of 0·8 (1) Å^2.

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References

Ahmed, F. R., Hall, S. R., Pippy, M. E. & Saunderson, C. P. (1966). *NRC Crystallographic Programs for the IBM/360 System. IUC World List of Crystallographic Computer Programs*. 2nd ed. Appendix, p. 52.

Canepa, F. G., Pauling, P. J. & Sörum, H. (1966). *Nature, Lond.* **210**, 907.

Chothia, C. H. (1970). *Nature, Lond.* **225**, 36.

Chothia, C. H. & Pauling, P. (1970). *Proc. Nat. Acad. Sci.* **65**, 477.

Cromer, D. T. & Waber, J. T. (1965). *Acta Cryst.* **18**, 104.

Cruickshank, D. W. J. (1949). *Acta Cryst.* **2**, 154.

Dexter, D. D. (1972). *Acta Cryst.* **B28**, 77.

Hope, H., Knobler, C. & McCullough, J. D. (1970). *Acta Cryst.* **B26**, 628.

International Tables for X-ray Crystallography (1962). Vol. III. Birmingham: Kynoch Press.

Johnson, C. K. (1965). *A Fortran Ellipsoid Plot Program for Crystal Structure Illustrations*. ORNL-3794, Oak Ridge National Laboratory, Oak Ridge, Tennessee.

Karle, I. L. & Estlin, J. A. (1969). *Z. Kristallogr.* **129**, 147.

Liquori, A. M., Damiani, A. & deCoen, J. L. (1968). *J. Mol. Biol.* **33**, 445.

Mathieson, A. McL. (1965). *Tetrahedron Letters*, p. 4137.

Pauling, P. (1968). In *Structural Chemistry and Molecular Biology*. Edited by A. Rich and N. Davidson, p. 555. San Francisco, London: Freeman.

Shefter, E. & Kennard, O. (1966). *Science*, **153**, 1389.

Shefter, E. & Mautner, H. G. (1969). *Proc. Nat. Acad. Sci.* **63**, 1253.

Stewart, J. M. (1967). Technical Report 67-58, Computer Science Center, University of Maryland.

Stewart, R. F., Davidson, R. E. & Simpson, W. T. (1965). *J. Chem. Phys.* **42**, 3175.

Sundaralingam, M. (1968). *Nature, Lond.* **217**, 35.