Table 1. *Positional parameters and equivalent isotropic thermal parameters with estimated standard deviations in parentheses*

Anisotropically refined atoms are given in the form of the equivalent isotropic displacement parameter defined as $\frac{4}{3}[a^2\beta(1,1) + b^2\beta(2,2) + c^2\beta(3,3) + ab(\cos\gamma)\beta(1,2) + ac(\cos\beta)\beta(1,3) + bc(\cos\alpha)\beta(2,3)]$.

	x	y	z	$B_{eq}(\text{Å}^2)$
N1	0·1914 (1)	0·5908 (2)	0·1245 (2)	2·75 (4)
C2	0·2921 (2)	0·5032 (3)	0·1756 (2)	2·58 (4)
C3	0·2780 (2)	0·2971 (3)	0·1918 (2)	2·65 (5)
N4	0·2028 (1)	0·2696 (2)	0·2602 (2)	2·68 (4)
C5	0·1007 (2)	0·3583 (3)	0·2048 (3)	4·00 (6)
C6	0·1170 (2)	0·5613 (3)	0·1934 (2)	3·83 (5)
N7	0·1915 (1)	0·7648 (3)	0·0732 (2)	3·39 (4)
O8	0·2713 (1)	0·8161 (2)	0·0545 (2)	4·28 (4)
O9	0·1110 (1)	0·8544 (2)	0·0469 (2)	4·94 (5)
O10	0·3471 (1)	0·5819 (2)	0·3019 (1)	2·86 (3)
C11	0·4481 (2)	0·6338 (3)	0·3239 (2)	3·04 (5)
O12	0·4937 (1)	0·5970 (3)	0·2507 (2)	5·08 (4)
C13	0·4899 (2)	0·7396 (4)	0·4488 (2)	3·84 (6)
O14	0·2431 (1)	0·2238 (2)	0·0645 (1)	3·77 (4)
N15	0·2000 (1)	0·0920 (3)	0·3056 (2)	3·15 (4)
O16	0·1201 (1)	0·0413 (2)	0·3227 (2)	4·31 (4)
O17	0·2796 (1)	0·0004 (2)	0·3293 (2)	4·53 (4)

2·14 (2), O14···O8 = 2·998 (4) Å and $\angle$O—H···O = 169·4 (1·1)°.

Related literature. For the structures of some other substituted 1,4-diazacyclohexanes see Sekido, Okamoto & Hirokawa (1985) and Ko Tien-Ming & Moncrief (1975) and references therein. The first article referenced above relates to the structure of 1,4-dinitrosopiperazine, which has a chair-shaped ring with essentially planar amino nitrogen atoms, in contrast to the pyramidal nitrogens in the title compound.

Table 2. *Bond distances (Å) and angles (°)*

N1—C2	1·440 (2)	C5—C6	1·505 (3)
N1—C6	1·462 (3)	N7—O8	1·226 (3)
N1—N7	1·383 (3)	N7—O9	1·221 (2)
C2—C3	1·529 (3)	O10—C11	1·364 (2)
C2—O10	1·437 (2)	C11—O12	1·191 (3)
C3—N4	1·464 (3)	C11—C13	1·492 (3)
C3—O14	1·403 (2)	N15—O16	1·218 (3)
N4—C5	1·463 (3)	N15—O17	1·221 (2)
N4—N15	1·387 (3)		
C2—N1—C6	118·1 (2)	N4—C5—C6	108·7 (2)
C2—N1—N7	115·5 (2)	N1—C6—C5	109·4 (2)
C6—N1—N7	115·7 (2)	N1—N7—O8	117·8 (2)
N1—C2—C3	109·8 (2)	N1—N7—O9	117·4 (2)
N1—C2—O10	108·1 (2)	O8—N7—O9	124·9 (2)
C3—C2—O10	109·1 (2)	C2—O10—C11	116·5 (2)
C2—C3—N4	109·1 (2)	O10—C11—O12	122·7 (2)
C2—C3—O14	105·9 (2)	O10—C11—C13	110·7 (2)
N4—C3—O14	112·5 (2)	O12—C11—C13	126·6 (2)
C3—N4—C5	117·4 (2)	N4—N15—O16	117·9 (2)
C3—N4—N15	114·5 (2)	N4—N15—O17	117·0 (2)
C5—N4—N15	114·8 (2)	O16—N15—O17	125·0 (2)

References

Enraf–Nonius (1985). *Structure Determination Package.* Enraf–Nonius, Delft, The Netherlands.
International Tables for X-ray Crystallography (1974). Vol. IV. Birmingham: Kynoch Press. (Present distributor D. Reidel, Dordrecht.)
Ko Tien-Ming & Moncrief, J. W. (1975). *Acta Cryst.* B31, 2544–2546.
Sekido, K., Okamoto, K. & Hirokawa (1985). *Acta Cryst.* C41, 741–743.

Structure of *N*-(*N*-Chlorobenzimidoyl)benzamidinium Decachlorodiselenate(IV) Acetonitrile Solvate

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Abstract. $[C_{14}H_{13}ClN_3]_2[Se_2Cl_{10}].2CH_3CN$, $M_r = 1112\cdot0$, triclinic, $P\bar{1}$, $a = 10\cdot457$ (1), $b = 10\cdot860$ (1), $c = 11\cdot762$ (2) Å, $\alpha = 64\cdot49$ (1), $\beta = 74\cdot61$ (1), $\gamma = 74\cdot52$ (1)°, $V = 1144$ (1) Å^3, $Z = 1$, $D_x = 1\cdot61$ g cm^{-3}, $\lambda(\text{Mo } K\alpha) = 0\cdot71073$ Å, $\mu = 23\cdot4$ cm^{-1}, $F(000) = 552$, $T = 293$ K, $R = 0\cdot060$ for 1767 unique observed reflections. The structure of the centrosymmetric decachlorodiselenate(IV) ion can be characterized as two $SeCl_6$ octahedra sharing an edge with average Se—Cl distances of 2·729, 2·209, and 2·382 Å for the

Table 1. *Fractional atomic coordinates and equivalent isotropic thermal parameters* (Å^2) *for the non-H atoms*

	x	y	z	B_{eq}
Se	0.03789 (9)	0.36853 (8)	0.41420 (7)	2.81 (2)
Cl(1)	0.2494 (3)	0.2695 (3)	0.4845 (2)	5.14 (8)
Cl(2)	−0.0131 (3)	0.1616 (2)	0.4780 (2)	5.54 (8)
Cl(3)	0.1389 (3)	0.3768 (3)	0.2211 (2)	4.49 (7)
Cl(4)	0.0773 (2)	0.6305 (2)	0.3486 (2)	3.41 (6)
Cl(5)	−0.1724 (2)	0.4729 (2)	0.3415 (2)	4.04 (6)
Cl(6)	0.4914 (3)	0.1245 (3)	0.3076 (2)	5.52 (8)
N(1)	0.6035 (8)	0.0300 (8)	0.2284 (7)	4.2 (2)
N(2)	0.7216 (7)	0.0148 (7)	0.0372 (6)	3.1 (2)
N(3)	0.8100 (8)	−0.1691 (7)	0.2071 (6)	3.5 (2)
N(4)	0.339 (1)	0.8945 (9)	0.2165 (8)	5.8 (3)
C(1)	0.5543 (9)	0.2208 (9)	0.0203 (8)	3.2 (2)
C(2)	0.415 (1)	0.2437 (9)	0.0232 (9)	3.9 (3)
C(3)	0.359 (1)	0.365 (1)	−0.0655 (9)	4.4 (3)
C(4)	0.433 (1)	0.461 (1)	−0.1525 (9)	4.6 (3)
C(5)	0.569 (1)	0.440 (1)	−0.154 (1)	4.9 (3)
C(6)	0.6292 (9)	0.319 (1)	−0.0692 (9)	4.0 (3)
C(7)	0.6238 (9)	0.0862 (9)	0.1059 (8)	3.4 (2)
C(8)	0.8125 (9)	−0.0987 (8)	0.0844 (7)	2.8 (2)
C(9)	0.9204 (8)	−0.1399 (8)	−0.0110 (7)	2.8 (2)
C(10)	0.9838 (9)	−0.2786 (9)	0.0225 (8)	3.4 (2)
C(11)	1.084 (1)	−0.3140 (9)	−0.0659 (8)	4.1 (3)
C(12)	1.125 (1)	−0.2193 (9)	−0.1822 (8)	4.1 (3)
C(13)	1.063 (1)	−0.082 (1)	−0.2155 (8)	3.9 (3)
C(14)	0.9617 (9)	−0.0426 (9)	−0.1289 (8)	3.3 (2)
C(15)	0.373 (1)	0.828 (1)	0.307 (1)	7.7 (4)
C(16)	0.425 (3)	0.738 (2)	0.424 (2)	14.5 (7)*

The starred atom was refined isotropically. Anisotropically refined atoms are given in the form of the equivalent isotropic thermal parameter defined as $B_{eq} = \frac{4}{3}[a^2B(1,1) + b^2B(2,2) + c^2B(3,3) + ab(\cos\gamma)B(1,2) + ac(\cos\beta)B(1,3) + bc(\cos\alpha)B(2,3)]$.

bridging, *trans*-to-bridging, and *cis*-to-bridging bonds. The bridging Se—Cl—Se angle is 93.35 (6)°. All cation bond angles and lengths are within a normal range.

Experimental. The title compound was prepared by the reaction of $SeCl_4$ with benzimidoylbenzamidinium chloride in acetonitrile in the presence of excess chlorine. Crystals obtained from CH_3CN solutions as the CH_3CN solvate. Pale-yellow air-sensitive parallelepiped data crystal $0.22 \times 0.18 \times 0.54$ mm sealed in a glass capillary. Intensities measured with an Enraf–Nonius CAD-4 diffractometer using ω–2θ scans of 4 to 16° min^{-1} in θ. Unit cell determined from least-squares analysis of angle data for 25 reflections with $16 < 2\theta < 22°$. Analytical absorption correction based on crystal shape varied from 0.60 to 1.00. Data collected to $(\sin\theta)/\lambda$ of 0.50 Å^{-1}, $-10 \le h \le 10$, $-10 \le k \le 10$, $-11 \le l \le 6$. Three standard reflections (402, 22$\bar{3}$, $\bar{2}4\bar{1}$) decreased 35.4% in intensity over 35.0 h of data collection; anisotropic drift correction applied. 4103 reflections measured, 2442 unique ($R_{int} = 0.02$), 675 reflections with $I < 3\sigma(I)$ considered unobserved. Solved by direct methods using *MULTAN*11/82 (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1982) and Fourier methods. Full-matrix least squares minimized $\sum w(\Delta F)^2$. Cation H atoms constrained to idealized positions with isotropic $B = 1.2 \times B$ of the bonded atom with the exceptions of the N(1) H and the H atoms of the acetonitrile solvent molecule, which

Table 2. *Selected bond distances* (Å) *and bond angles* (°)

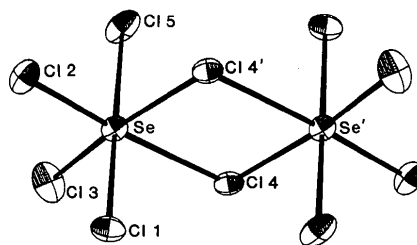|			
Se—Cl(1)	2.380 (2)	Se—Cl(4)	2.725 (2)
Se—Cl(2)	2.208 (2)	Se—Cl(4)'	2.733 (2)
Se—Cl(3)	2.210 (2)	Se—Cl(5)	2.384 (2)
Cl(1)—Se—Cl(2)	91.53 (9)	Cl(2)—Se—Cl(5)	89.81 (9)
Cl(1)—Se—Cl(3)	88.97 (8)	Cl(3)—Se—Cl(4)	90.22 (7)
Cl(1)—Se—Cl(4)	91.90 (8)	Cl(3)—Se—Cl(4)'	176.42 (8)
Cl(1)—Se—Cl(4)'	89.41 (7)	Cl(3)—Se—Cl(5)	90.76 (8)
Cl(1)—Se—Cl(5)	178.65 (9)	Cl(4)—Se—Cl(4)'	86.65 (6)
Cl(2)—Se—Cl(3)	94.18 (9)	Cl(4)—Se—Cl(5)	86.78 (7)
Cl(2)—Se—Cl(4)	174.47 (9)	Cl(4)'—Se—Cl(5)	90.79 (7)
Cl(2)—Se—Cl(4)'	89.05 (8)	Se—Cl(4)—Se'	93.35 (6)

Fig. 1. *ORTEP* diagram (Johnson, 1976) and atom-numbering scheme for the anion. All ellipsoids at 30% probability level.
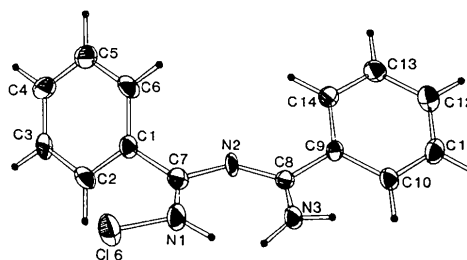

Fig. 2. *ORTEP* diagram (Johnson, 1976) and atom-numbering scheme for the cation. Non-H ellipsoids at 30% probability level, H atoms given arbitrary radii.

were located on difference maps. All non-H atoms except the methyl C of the solvent refined anisotropically for a total of 239 variables. $R = 0.060$, $wR = 0.072$, $S = 1.70$, where non-Poisson $w^{-1} = [\sigma^2(I) + 0.0049I^2]/4F^2$. Final $(\Delta/\sigma)_{max} < 0.07$, $\Delta\rho_{max} = +1.80$ (4) and $\Delta\rho_{min} = -1.25$ (4) e Å^{-3} on final difference map. Atomic scattering factors and anomalous-dispersion corrections from *International Tables for X-ray Crystallography* (1974) and programs used were those of Enraf–Nonius (1982) *SDP*.* Table 1 gives the atom coordinates and Table 2 selected bond distances and angles. Figs. 1 and 2 show the anion and cation with the numbering scheme.

Related literature. The structure of an analogous ion, $[TeBr_{10}]^{2-}$, has been reported (Krebs & Buscher, 1980). Other dihalogen-bridged structures of selenium that have been determined include $[SeBr_6]^{2-}$ (Krebs, Schaffer & Pohl, 1984), $[Se_2(CF_3)_2Cl_6]$ (Marsden, Sheldrick & Taylor, 1977), and the dimeric $[SeOCl_3]^-$ and $[SeOBr_3]^-$ ions (Krebs, Schaffer & Hucke, 1982).

References

Enraf–Nonius (1982). *Structure Determination Package.* Enraf–Nonius, Delft, The Netherlands.

International Tables for X-ray Crystallography (1974). Vol. IV. Birmingham: Kynoch Press. (Present distributor D. Reidel, Dordrecht.)

Johnson, C. K. (1976). *ORTEPII.* Report ORNL-5138. Oak Ridge National Laboratory, Tennessee, USA.

Krebs, B. & Buscher, K. (1980). *Z. Anorg. Allg. Chem.* **463**, 56–64.

Krebs, B., Schaffer, A. & Hucke, M. (1982). *Z. Naturforsch. Teil B*, **37**, 1410–1417.

Krebs, B., Schaffer, A. & Pohl, S. (1984). *Z. Naturforsch. Teil B,* **39**, 1633–1641.

Main, P., Fiske, S. J., Hull, S. E., Lessinger, L., Germain, G., Declercq, J.-P. & Woolfson, M. M. (1982). *MULTAN11/82. A System of Computer Programs for the Automatic Solution of Crystal Structures from X-ray Diffraction Data.* Univs. of York, England, and Louvain, Belgium.

Marsden, C. J., Sheldrick, G. M. & Taylor, R. (1977). *Acta Cryst.* B33, 139–141.

Structure of [2-(2-Chloroethoxy)ethoxy]-2-methyl-6-morpholinoquinoline*

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Abstract. $C_{18}H_{23}ClN_2O_3$, $M_r = 350.8$, triclinic, $P\bar{1}$, $a = 6.037$ (1), $b = 10.208$ (3), $c = 15.499$ (3) Å, $\alpha = 103.98$ (2), $\beta = 82.68$ (2), $\gamma = 108.56$ (2)°, $V = 877.4$ (4) Å^3, $Z = 2$, D_m (flotation, KI solution) = 1.34, $D_x = 1.33$ g cm^{-3}, λ(Mo $K\alpha$) = 0.7107 Å, $\mu = 2.41$ cm^{-1}, $F(000) = 372$, $T = 293$ K, $R = 0.062$ for 882 observed reflections. The morpholine ring adopts a chair conformation while the quinoline ring is planar. The morpholine ring and the side chain are *cis* with respect to the quinoline ring. The sum of the angles around the morpholine N atom is 346.4°.

Experimental. The title compound (Fig. 1) was prepared as part of a programme to make compounds with pharmaceutical and insecticidal value. It was prepared by refluxing 6-amino-4-hydroxyquinaldine hydrochloride with 2,2'-dichlorodiethyl ether. Crystals were grown from petroleum ether.

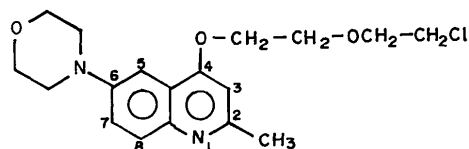Crystal of approximate dimensions $0.18 \times 0.25 \times 0.58$ mm; lattice parameters from 20 reflections (18 <

Fig. 1. The title compound.

$2\theta < 34°$); intensity data collected on an Enraf–Nonius CAD-4F-11M single-crystal X-ray diffractometer; graphite-monochromated Mo $K\alpha$ radiation; $\omega/2\theta$ scan mode; scan speed 1° min^{-1}; $\theta < 24°$, index range h 0 to 7, k −12 to 12, l −18 to 18; 3040 reflections measured; 882 significant ($|F_o| > 3\sigma|F_o|$). Three standard reflections ($00\bar{4}$, $\bar{1}1\bar{3}$ and $1\bar{3}\bar{7}$) measured every 2000 s; 4% variation in intensity; no correction for absorption. Structure solved by direct methods (*MULTAN*78; Main, Hull, Lessinger, Germain, Declercq & Woolfson, 1978) using a modified procedure (Tavale & Guru Row, 1986); full-matrix refinement of scale factor and non-H-atom positional and anisotropic thermal parameters (positional parameters for H atoms geometrically fixed) converged to $R = 0.062$ and $wR =$