Acta Cryst. (1976). B**32**, 1906

2-Chloroquinoline Iodomonochloride

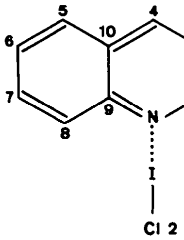
By G. Bernardinelli and R. Gerdil

Département de Chimie Organique et Laboratoire de Cristallographie aux Rayons X, Université de Genève, 30 quai Ernest Ansermet, 1211 Geneva, Switzerland

(*Received* 9 *February* 1976; *accepted* 28 *February* 1976)

Abstract. $C_9H_6Cl_2IN$, monoclinic, space group $P2_1/c$; $a=8{\cdot}297$ (1), $b=15{\cdot}257$ (8), $c=8{\cdot}831$ (4) Å, $\beta=110{\cdot}38$ (3)°; $Z=4$, $D_m=2{\cdot}068$, $D_x=2{\cdot}066$ g cm^{-3}. The structure was refined to an R of 0·03. The complex is planar with a linear N···I–Cl grouping extending along the expected orientation of the N atom lone pair.

Introduction. Yellow crystals are slowly formed at room temperature by mixing solutions of equimolecular amounts of 2-chloroquinoline and ICl in CCl_4. A crystal, of average cross-section 0·2 mm, sealed in a Lindemann capillary was used. The cell parameters and intensities were obtained at 20 °C on a Philips PW 1100 automatic diffractometer (graphite monochromator, Mo $K\alpha$ radiation). 1443 independent reflexions were recorded in the θ–2θ mode (scan width 1·5°; scan speed 0·02° s^{-1}) out to a limit of $\sin\theta/\lambda \le 0{\cdot}53$ Å^{-1}. Three monitor reflexions revealed no significant change in intensity. No absorption corrections were applied ($\mu=35{\cdot}5$ cm^{-1}). After the usual corrections, 1112 reflexions had $|F|$ greater than $2\sigma_F$ and were used in the analysis. The structure was solved by the heavy-atom method and refined by full-matrix least squares with anisotropic temperature factors for the non-hydrogen atoms. The coordinates of the H atoms were calculated with C–H = 1·09 Å and included in the model; their positions and isotropic temperature factors were refined during the latter stages. The final R based on all the observed reflexions was 0·031 with the weights $w=1/\sigma^2(F)$. The final positional and vibrational parameters are listed in Table 1. The bond lengths and angles defined by the numbering system shown below are given in Table 2.*



Discussion. This determination was undertaken to ascertain the placement of the acceptor with respect to the donor molecule to support part of a thermochemical study of the adduct formation (Biselx, 1976).

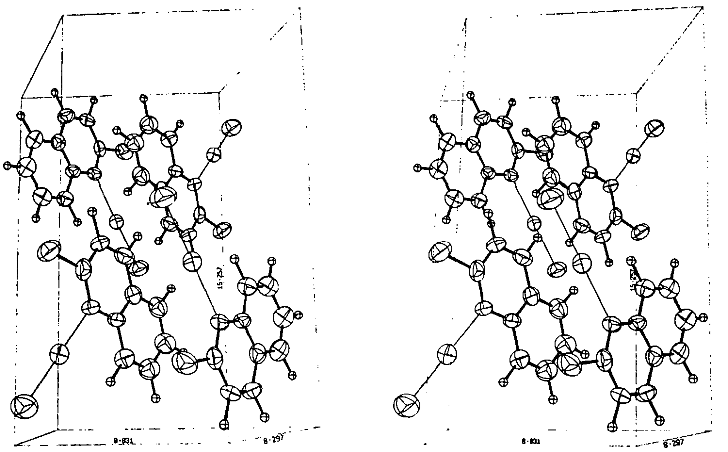


Fig. 1. Stereoscopic view of the contents of one unit cell. The origin is in the lower-right, rear corner. The lower-right molecule is the basic molecule with position x,y,z.

---

* A list of structure factors has been deposited with the British Library Lending Division as Supplementary Publication No. SUP 31707 (11 pp. 1 microfiche). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 13 White Friars, Chester CH1 1NZ, England.

Table 1. *Final positional and thermal parameters with standard deviations*

Values are $\times 10^4$ for non-hydrogen atoms and $\times 10^3$ for hydrogen atoms. The thermal parameters are of the form $\exp[-2\pi^2(a^{*2}U_{11}h^2+2a^*b^*U_{12}hk+\ldots)]$.

	x	*y*	*z*	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
I	12141 (1)	3491 (0)	5399 (1)	487 (4)	439 (4)	425 (4)	8 (4)	170 (3)	9 (4)
Cl(1)	12568 (4)	1251 (2)	5899 (3)	747 (22)	522 (19)	579 (18)	61 (14)	74 (15)	157 (14)
Cl(2)	13903 (4)	4683 (2)	6988 (4)	867 (24)	507 (18)	647 (19)	−107 (16)	−3 (17)	−59 (16)
N	10388 (11)	2306 (5)	3822 (10)	449 (56)	277 (52)	416 (53)	47 (41)	156 (46)	48 (42)
C(2)	10802 (12)	1473 (8)	4199 (11)	555 (66)	558 (73)	356 (55)	−17 (72)	240 (47)	24 (69)
C(3)	9907 (15)	758 (6)	3337 (13)	722 (88)	274 (61)	515 (75)	−11 (60)	250 (68)	87 (60)
C(4)	8532 (14)	890 (7)	2006 (14)	612 (81)	349 (70)	575 (76)	−22 (57)	251 (65)	−95 (59)
C(5)	6579 (14)	1947 (7)	94 (13)	537 (82)	613 (78)	472 (76)	−3 (56)	124 (61)	−92 (60)
C(6)	6134 (14)	2804 (8)	−327 (14)	495 (79)	630 (84)	603 (79)	73 (63)	159 (61)	−20 (71)
C(7)	7034 (14)	3483 (8)	621 (14)	571 (70)	570 (62)	634 (71)	19 (79)	229 (58)	27 (82)
C(8)	8449 (12)	3331 (7)	2005 (12)	413 (62)	453 (75)	484 (62)	−19 (56)	97 (53)	22 (56)
C(9)	8978 (13)	2463 (6)	2451 (13)	468 (68)	264 (58)	454 (68)	43 (56)	223 (58)	2 (56)
C(10)	7998 (13)	1771 (7)	1511 (13)	447 (67)	509 (79)	437 (67)	7 (56)	236 (56)	−16 (57)

Table 1 (*cont.*)

	x	*y*	*z*	*B* (Å^2)
H(3)	1014	25	371	3·3
H(4)	789	40	139	2·5
H(5)	614	149	−48	4·5
H(6)	499	314	−130	6·9
H(7)	652	420	25	10·7
H(8)	906	384	264	2·3

Table 2. *Molecular parameters*

Bond distances (Å)

N——C(2)	1·329 (15)	C(5)–C(6)	1·374 (17)
N——C(9)	1·381 (11)	C(5)–C(10)	1·414 (13)
N···I	2·432 (8)	C(6)–C(7)	1·377 (16)
C(2)–C(3)	1·388 (15)	C(7)–C(8)	1·388 (13)
C(2)–Cl(1)	1·727 (8)	C(8)–C(9)	1·407 (14)
C(3)–C(4)	1·338 (14)	C(9)–C(10)	1·413 (14)
C(4)–C(10)	1·435 (14)	I——Cl(2)	2·446 (3)

Bond angles (°) (mean e.s.d. 0·8°)

C(2)–N——C(9)	121·2	C(6)–C(7)—C(8)	121·5
C(2)–N···I	116·9	C(7)–C(8)—C(9)	119·3
C(9)–N···I	121·9	C(8)–C(9)—N	119·7
N——C(2)–C(3)	124·9	C(8)–C(9)—C(10)	118·6
N——C(2)–Cl(1)	118·2	N—C(9)—C(10)	121·6
C(3)–C(2)–Cl(1)	116·9	C(4)–C(10)–C(5)	121·5
C(2)–C(3)–C(4)	119·5	C(4)–C(10)–C(9)	117·9
C(3)–C(4)–C(10)	119·2	C(5)–C(10)–C(9)	120·6
C(6)–C(5)–C(10)	118·4	N···I——Cl(2)	179·9
C(5)–C(6)–C(7)	120·9		

The complex is essentially planar with a linear N···I–Cl arrangement extending along the expected orientation of the N atom lone pair. The line defined by the three atoms makes an angle of 3° with its projection onto the best plane through the ring atoms (the e.s.d. of the atoms from the plane is 0·015 Å). The aromatic bond lengths are very close to those found for 2-chloroquinoline itself (Merlino, 1968) and there is no clear indication of any molecular distortion by complex formation.

It seems reasonable to regard the distance between the *n* donor and acceptor atoms as reflecting the strength of their interaction; moreover it has been shown (Hassel & Rømming, 1967) that a simple relationship exists between the donor–halogen and halogen–halogen distances in complexes of halogens with *n* donors. On this basis it is seen that the strength of interaction in the present complex is relatively weaker than in pyridine.ICl, where the N···I and I–Cl distances are 2·290 and 2·510 Å, respectively (Rømming, 1972).

In the crystal the aromatic molecular planes lie closely parallel to $(10\bar{1})$; the angle between the planes of two molecules related by a screw diad is 1·6°. Adjacent molecules related by a glide plane are stacked on each other with a displacement, and the mean interplanar distance is 3·66 Å. In this situation (*e.g.* see lower two molecules in Fig. 1) contacts are made through C atoms lying at distances C(5)···C(8) = 3·602 and C(6)···C(9) = 3·579 Å, and also through I and C(1), C(2), C(3) at a mean distance I···C = 3·86 (2) Å. Molecules in adjacent layers related by a symmetry centre or a screw diad touch *via* H atoms. The packing within layers is characterized by a short distance 3·097 Å between I and H(6) of the neighbouring molecule at one unit translation along **a** and **c** respectively.

References

Biselx, C. (1976). Thesis, Chemistry Department, Univ. of Geneva, Switzerland.

Hassel, O. & Rømming, C. (1967). *Acta Chem. Scand.* **21**, 2659–2662.

Merlino, S. (1968). *Atti. Accad. Nazl. Lincei Rend. Classe Sci. Fis. Mat. Nat.* **45**, 60–71.

Rømming, C. (1972). *Acta Chem. Scand.* **26**, 1555–1560.