compared to 124·4 (3)° in 5-chloro-2-pyridone where the ring nitrogen atom is protonated.

The hydrogen positions found from the difference synthesis were refined to give carbon–hydrogen distances between 0·95 (2) and 0·97 (2) Å (Table 8). As often observed the deviations from spherical electron density for the hydrogen atoms cause shifts of the apparent X-ray hydrogen positions (see *e.g.* Almlöf, Kvick & Thomas, 1973).

The hydrogen bond

Dimers are formed by hydrogen bonding between the nitrogen atoms. The $N \cdots N$ distance is 3·058 (2) Å and the bond is almost linear $[N(2)-H(1)\cdots N(1) = 179$ (2)°]. The distance $N(2)-H(1)$ is 0·81 (2) Å and $N(1)\cdots H(1)$ 2·25 (2) Å. These distances are also affected by the non-spherical charge density for the hydrogen atoms. The accepted $N(2)-H(1)$ nuclear distance is about 0·2 Å longer.

It may be observed that the $N \cdots N$ hydrogen bond, which in this case has an amino nitrogen atom as donor, is weaker than the $N \cdots N$ bond in 6-chloro-2-hydroxypyridine–2-pyridone $[N \cdots N$ 2·904 (4) Å], where the pyridine nitrogen atom serves as hydrogen bond donor. Both bonds are almost linear, so that their $N \cdots N$ distances may be taken as a measure of the relative strengths of the two bonds.

References

ALMLÖF, J., KVICK, Å. & OLOVSSON, I. (1971). *Acta Cryst.* B27, 1201–1208.
ALMLÖF, J., KVICK, Å. & THOMAS, J. O. (1973). *J. Chem. Phys.* In the press.
BUSING, W. R., MARTIN, K. O. & LEVY, H. A. (1964). *ORFFE.* Report ORNL-TM-306, Oak Ridge National Laboratory, Oak Ridge, Tennessee.
COPPENS, P. & HAMILTON, W. C. (1970). *Acta Cryst.* A26, 71–83.
COPPENS, P., LEISEROWITZ, L. & RABINOVICH, D. (1965). *Acta Cryst.* 18, 1035–1038.
CROMER, D. T. (1965). *Acta Cryst.* 18, 17–23.
HANSON, H. P., HERMAN, F., LEA, J. D. & SKILLMAN, S. (1964). *Acta Cryst.* 17, 1040–1044.
International Tables for X-ray Crystallography. (1952). Vol. I. Birmingham: Kynoch Press.
JOHNSON, C. K. (1965). *ORTEP.* Report ORNL-3794, Oak Ridge National Laboratory, Oak Ridge, Tennessee.
KARLE, I. L., DRAGONETTE, K. S. & BRENNER, S. A. (1965). *Acta Cryst.* 19, 713–716.
KVICK, Å. (1972). Unpublished.
KVICK, Å. (1973). To be published.
KVICK, Å. & BOOLES, S. (1972). *Acta Cryst.* B28, 3405–3409.
KVICK, Å. & OLOVSSON. I. (1968). *Ark. Kem.* 30, 71–80.
LONG, R. E. (1965). *The Crystal and Molecular Structures of 7,7,8,8-Tetracyanoquinodimethane and Cyclopropane Carboxamide, and a Program for Phase Determination.* Doctoral Dissertation, Univ. of California, Los Angeles.
LUNDGREN, J.-O. (1972). *UPALS, a Crystallographic Full-Matrix Least-Squares Refinement Program.* UUIC-B13-3, Inst. of Chemistry, Univ. of Uppsala, Uppsala.
PAULING, L. (1960). *The Nature of the Chemical Bond.* Ithaca: Cornell Univ. Press.
STEWART, R. F., DAVIDSON, E. R. & SIMPSON, W. T. (1965). *J. Chem. Phys.* 42, 3175–3187.

The Crystal and Molecular Structure of *cis*-1-(4-Chlorophenyl)-3-methyl-2-methylisoindoline

BY FRANÇOIS BRISSE AND JURGEN SYGUSCH

Département de Chimie, Université de Montréal, Montréal, Canada

The crystal structure of *cis*-1-(4-chlorophenyl)-3-methyl-2-methylisoindoline has been determined by direct methods, from 1851 reflexions collected on a Picker automatic diffractometer. The crystals, which have monoclinic unit-cell dimensions $a = 9·071$, $b = 12·443$, $c = 11·945$ Å and $\beta = 93·2°$, belong to the space group $P2_1/c$. The final R value is 0·040. The C(3) methyl and the C(1) chlorophenyl groups are *cis* to each other while the N(2) and the C(3) methyl groups are *trans*. The molecules are held in the crystal by van der Waals forces only.

Introduction

Isomers of 1-(4-chlorophenyl)-2,3-dimethylisoindolines were synthesized recently (Freter, Hess, Sygusch & Brisse, 1971). However, the relative orientation of the substituents at C(1) and C(3) could not be determined on the basis of standard n.m.r. techniques. Interpretation of n.m.r. data of symmetrically substituted isoindolines previously led to erroneous assignments (Carpino, 1962), while attempts to assign the stereochem-

istry of 1,3-disubstituted phthalans were uninformative (Petracek, Sugisaka, Klohs, Parker, Bordner & Roberts, 1970). It was then decided to determine the structure of one of the isomers by X-ray diffraction. This investigation, first reported in a preliminary communication (Freter et al., 1971), revealed that the isomer chosen was the *cis*-1-(4-chlorophenyl)-3-methyl-2-methylisoindoline. The purpose of this paper is to report on the determination and the description of the structure of the *cis*-isomer.

Experimental

Well developed crystals were easily obtained by slow evaporation of a methanol solution. Precession photographs indicated that the crystals were monoclinic, space group $P2_1/c$, which was uniquely determined by the systematic absences: $h0l$: $l = 2n + 1$ and $0k0$: $k = 2n + 1$. The unit-cell dimensions and the orientation of the crystal were determined on a Picker FACS-I automatic diffractometer by a least-squares fit to the settings of 12 reflexions. The unit-cell dimensions and their associated standard deviations are given below.

Crystal data

$C_{16}H_{16}NCl$, F.W. 257·76, $F(000) = 544$.
Monoclinic, $P2_1/c$, $Z = 4$,
$a = 9·071$ (2), $b = 12·443$ (3), $c = 11·945$ (4) Å,
$\beta = 93·19$ (10)°; $V = 1346·2$ Å^3,
$\varrho_{obs} = 1·26$ g cm^{-3} (pycnometry), $\varrho_{cal} = 1·272$ g cm^{-3};
$\mu(Mo\ K\alpha) = 2·68$ cm^{-1} ($\lambda Mo\ K\bar{\alpha} = 0·71069$ Å),
$T = 20$ (1) °C.

The integrated intensities of 1851 independent reflexions were collected on the Picker automatic diffractometer with zirconium-filtered molybdenum radiation for reflexions with 2θ values $\leq 45°$. The θ–2θ scan technique was used at a scan rate of $1°$ (2θ) min^{-1} and a minimum scan width of $1·5°$ (2θ). Stationary-counter, stationary-crystal background counts of 20 s were taken at each end of the scan range. The average intensity of the three standard reflexions, measured after each group of 30 reflexions, decreased by about 10% of its initial value over the duration of the data collection. All the intensities were corrected for background and placed on a common scale. Data reduction was carried out in the usual manner using the programs of Ahmed, Hall, Pippy & Huber (1966). Of the 1851 measured reflexions, there were 1631 for which $I/\sigma(I) \geq 2·0$.

The X-ray scattering factors were obtained from Cromer & Waber (1965) for C, N and Cl and from Stewart, Davidson & Simpson (1965) for H. The real and imaginary parts of the dispersion correction for Cl were taken from Cromer (1965).

Structure determination

A set of normalized structure factors $|E|$ was obtained after isotropic temperature factor correction. The sta-

tistical averages of the normalized structure factors confirm the centrosymmetric space group $P2_1/c$. The phases of 237 reflexions with $|E| \geq 1·50$ were determined by the symbolic addition procedure for non-centrosymmetric structures described by Germain, Main & Woolfson (1970). The solution with the highest figure of merit allowed us to calculate an E map which revealed 18 strong peaks which could be assigned to all non-hydrogen atoms. The set of structure factors calculated with the coordinates of these 18 peaks and an overall temperature factor of 3·5 Å^2 gave an R value of 0·39.

The refinement of the coordinates was carried out using at first Ahmed's (1966) block-diagonal least-squares program with unit weight. After 5 cycles of isotropic temperature factor refinement and two cycles of anisotropic temperature factor refinement the R index decreased to 0·099.

The 10 non-methyl hydrogen atoms were located on a Fourier difference synthesis computed at this stage. The least-squares refinement was continued to include the newly found hydrogen atoms. The program used was *NUCLS*, a modified version of *ORFLS*, further modified by J. Sygusch to include the block-diagonal approximation. This program also included the refinement of an isotropic extinction correction as described by Coppens & Hamilton (1970). Each reflexion was assigned a weight w inversely proportional to the variance based on counting statistics $w^{-1} = \sigma^2(F)$ where

$$\sigma^2(I) = t_p^2 \left[\frac{N_p}{t_p^2} + \frac{B_1 + B_2}{2t_B^2} \right] + (C \times \text{Net})^2.$$

N_p is the total peak count, B_1 and B_2 the background counts before and after the peak, Net, the net peak count, t_p the peak scan time and t_B the background preset counting time, and C the long term instability coefficient, set to 0·02.

After three more refinement cycles, another Fourier difference map revealed the remaining 6 hydrogen atoms. The H atoms were included in further refinements with isotropic temperature factors. The final values were $R = 0·040$ and $R_w = 0·035$. When all measured reflexions were included, $R = 0·056$ and $R_w = 0·037$. At the end of this refinement, the average shift to the coordinates was less than $0·3\sigma$ and the isotropic extinction correction was $g = 0·517 \times 10^4$. A final difference electron density map showed no significant residual density. The maximum and minimum densities observed were 0·10 and $-0·15$ e Å^{-3} respectively.

Results and discussion

Observed and calculated structure factors are given in Table 1. The positional and thermal parameters and their standard deviations are presented in Table 2 for the heavy atoms and in Table 3 for the hydrogen atoms. The thermal parameters are the coefficients of the expression

$$T = \exp\left[-2\pi^2(U_{11}a^{*2}h^2 + \ldots + 2U_{12}a^*b^*hk + \ldots)\right].$$

Table 1. *Observed and calculated structure amplitudes* (× 10)

Unobserved reflexions are indicated by an X.

Table 1 (cont.)

Table 2. *Positional and thermal parameters of the heavy atoms in chlorophenyldimethylisoindoline*

The coordinates are in fractions of unit-cell edges ($\times 10^5$).
The thermal parameters are given $\times 10^4$. The standard deviations refer to the least significant digits.

	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
Cl	7339 (7)	−1629 (4)	36316 (7)	890 (4)	536 (3)	1057 (5)	−83 (1)	138 (1)	−49 (1)
C(1′)	29762 (21)	30747 (16)	36993 (17)	507 (12)	469 (12)	482 (13)	13 (5)	− 8 (5)	12 (5)
C(2′)	31980 (23)	23585 (17)	45770 (17)	728 (14)	544 (14)	533 (14)	−19 (6)	−56 (6)	34 (6)
C(3′)	24941 (25)	13713 (17)	45739 (19)	797 (16)	521 (14)	600 (15)	20 (6)	14 (6)	68 (6)
C(4′)	15714 (21)	11018 (17)	36721 (17)	570 (13)	461 (13)	647 (15)	3 (5)	72 (5)	−29 (6)
C(5′)	13093 (23)	17961 (17)	27975 (19)	575 (13)	635 (14)	585 (15)	18 (5)	17 (6)	−39 (6)
C(6′)	20094 (21)	27861 (17)	28212 (19)	610 (14)	519 (14)	523 (14)	− 9 (5)	−22 (5)	30 (6)
C(1)	37423 (21)	41586 (16)	37321 (17)	536 (12)	531 (13)	512 (14)	−20 (5)	−36 (5)	18 (6)
N(2)	39722 (17)	45759 (12)	26051 (14)	561 (10)	476 (10)	553 (11)	− 1 (4)	22 (4)	20 (4)
C(3)	39549 (21)	57629 (17)	26498 (19)	552 (13)	473 (12)	613 (15)	−41 (5)	− 3 (5)	7 (6)
C(4)	30040 (21)	59699 (16)	36101 (17)	512 (12)	486 (12)	602 (15)	−36 (5)	−35 (5)	−38 (6)
C(5)	28809 (21)	50516 (16)	42437 (17)	527 (12)	531 (13)	511 (13)	−54 (5)	− 9 (5)	−32 (5)
C(6)	20753 (25)	50485 (19)	51859 (19)	729 (15)	738 (16)	576 (15)	−94 (6)	14 (6)	−38 (6)
C(7)	13673 (25)	59889 (21)	54748 (21)	734 (16)	966 (19)	722 (18)	−93 (7)	62 (7)	−172 (8)
C(8)	14688 (26)	69001 (19)	48333 (23)	707 (15)	692 (16)	968 (20)	−20 (6)	− 2 (7)	−177 (7)
C(9)	22956 (25)	69065 (17)	39029 (21)	665 (14)	556 (14)	820 (18)	−24 (5)	−21 (6)	−74 (6)
C(10)	52555 (23)	41258 (19)	21021 (19)	626 (15)	694 (16)	764 (18)	22 (6)	52 (6)	7 (7)
C(11)	33933 (25)	62388 (17)	15420 (19)	875 (17)	571 (14)	727 (18)	−28 (6)	− 8 (7)	59 (6)

Table 3. *Positional and thermal parameters of the hydrogen atoms in chlorophenyldimethylisoindoline*

The coordinates are in fractions of unit-cell edges ($\times 10^4$). The thermal parameters are given $\times 10^4$. The standard deviations refer to the least significant digits.

	x	y	z	$U(\text{iso})$
H(2′)	3873 (18)	2582 (14)	5214 (14)	590 (55)
H(3′)	2665 (18)	891 (14)	5183 (15)	672 (60)
H(5′)	645 (18)	1612 (14)	2164 (15)	638 (58)
H(6′)	1876 (16)	3277 (12)	2199 (13)	445 (50)
H(1)	4734 (17)	4073 (13)	4167 (14)	540 (53)
H(3)	4980 (17)	6066 (13)	2856 (14)	553 (54)
H(6)	1959 (18)	4406 (14)	5579 (15)	657 (59)
H(7)	797 (21)	5977 (15)	6100 (16)	814 (68)
H(8)	1014 (20)	7542 (15)	5057 (15)	794 (65)
H(9)	2345 (18)	7561 (16)	3420 (15)	742 (63)
H(10,1)	5327 (21)	4447 (17)	1353 (18)	951 (75)
H(10,2)	6181 (19)	4294 (15)	2574 (16)	690 (60)
H(10,3)	5120 (18)	3335 (14)	2022 (15)	678 (60)
H(11,1)	4007 (23)	6039 (17)	941 (17)	946 (73)
H(11,2)	2378 (19)	5969 (15)	1375 (15)	669 (60)
H(11,3)	3388 (22)	7026 (17)	1564 (17)	897 (72)

The stereochemical structure of the dimethyl chlorophenyl isoindoline molecule is shown in Fig. 1, projected on the (100) plane. The C(3) methyl and the C(1) chlorophenyl groups are *cis* to each other, while the N(2) and C(3) methyl groups are *trans*.

Bond distances and angles

The interatomic bond distances and angles are summarized in Fig. 2. The values closest to the bonds have been calculated from the final positional parameters. The other values have been corrected for rigid–body motion by the method of Schomaker & Trueblood (1968) utilizing the final thermal parameters of the non-hydrogen atoms. The root mean square error for the U_{ij}'s, $[\sum(\Delta U_{ij})^2/(n-s)]^{1/2}$ where n is the number of U_{ij} and s the number of rigid-body parameters, was only 0·005 Å^2. This correction resulted in a systematic lengthening of the bonds by an average of 0·003 Å. Since this increase is less than 1σ, the comparisons below will be made in terms of the uncorrected distances. The estimated standard deviations which have been doubled to allow for the block-diagonal approximation are of the order of 0·007 and 0·04 Å for C–C and C–H distances respectively. The average distances are compared below with the values given by Sutton (1965), and with the average distances from a very similar structure, a 1,3-disubstituted phthalan described by Petracek *et al.* (1970). (Dr J. D. Roberts kindly supplied the bond distances and angles quoted here.)

Bond type	Sutton (1965)	Isoindoline (this work)	Phthalan (Petracek *et al.*, 1970)
C*sp*³–C*sp*³	1·537 (5) Å	1·511 Å	1·521 Å
C*sp*³–C*sp*²	1·506 (5)	1·506	1·502
C*sp*²–C*sp*²	1·395 (5)	1·378	1·379
C*sp*³–N	1·472 (5)	1·465	
C*sp*²–Cl	1·70 (1)	1·746	

Petracek's bond distances and ours are in very good agreement but differ slightly from Sutton's values.

These differences are discussed below. The C*sp*²–Cl bond distance is found to be significantly longer than the value quoted by Sutton. However it agrees much better with 1·737 (16) Å which is the average of 26 C–Cl distances computed by Palenik, Donohue & Trueblood (1968).

The C*sp*²–C*sp*² aromatic distances observed in this work and Petracek's are consistently shorter than 1·395 Å. That these aromatic distances are shorter than 1·395 Å has already been reported in other papers. For example, in the structure of *p*-chlorophenyldi-
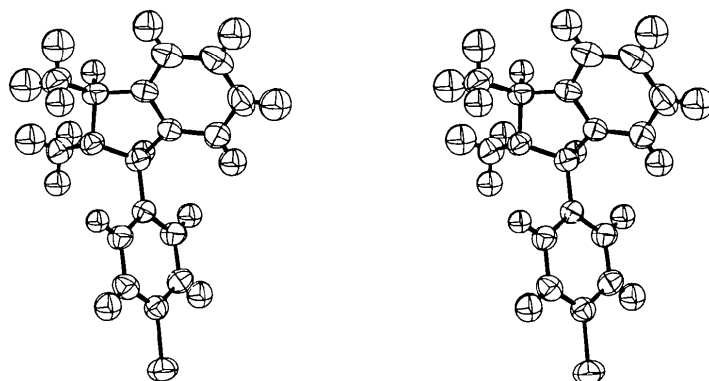
Fig. 1. Stereoscopic pair of drawings showing the structure of the chlorophenyldimethylisoindoline molecule.
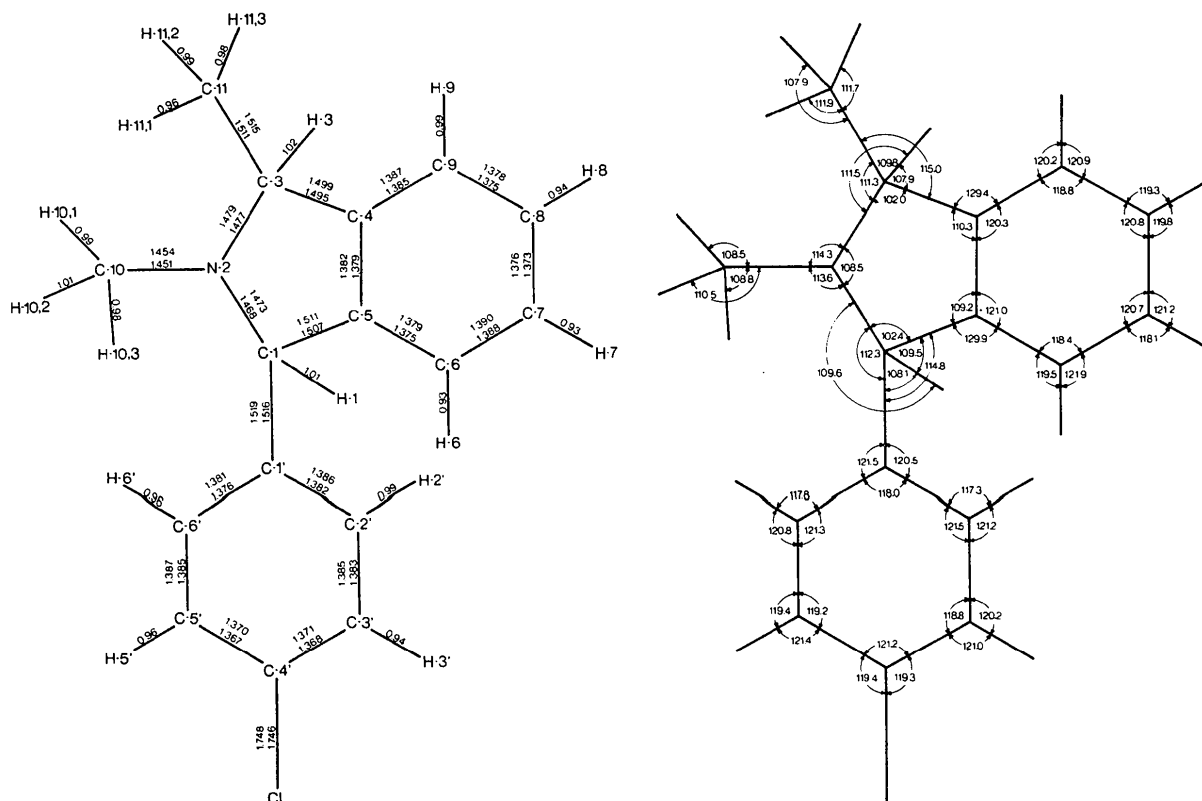
Fig. 2. Bond distances and angles.

phenylphosphine oxide (Dreissig & Plieth, 1971) there are eighteen aromatic C–C distances whose average is 1·380 Å. In the structure of an anilide hydrobromide (Singh & Ahmed, 1969) the mean of 12 C–C distances is 1·375 Å and finally in 2,2,4,4,6,6-hexaphenylcyclo-triphosphazatriene (Ahmed, Singh & Barnes, 1969), 36 aromatic C–C bonds have a mean length of 1·379 Å. The overall average of the Csp^2–Csp^2 bond distances quoted above, including our values, is 1·379 Å. Ahmed attributed the 0·015 Å difference between his values and that listed by Sutton (1965) to the omission of thermal-vibration corrections. However, in our own work a rigid-body correction has been applied and has resulted in a lengthening of the bond distances by only 0·003 Å.

Looking more closely at the chlorophenyl bond distances, it is observed that the C–C bonds adjacent to the C–Cl bond are slightly shorter than the other C–C bonds in the ring. The bond distances, bond angles and other relevant measurements from the halogeno-phenyl part of a number of recent structures are shown in Table 4. Although the observations in this Table do not apply to all halogenophenyl groups, there seem to be some real effects, in many instances, caused by the presence of the halogen atom, X = Cl or Br, on the phenyl group: a relative shortening of the bonds adjacent to the C–X bond, this shortening being of the order of 1·3–1·4% of the normal Csp^2–Csp^2 bond length; an increase of the C–CX–C ring angle from the sp^2 value of 120·0 to about 121·5° while the two X–C–C angles are reduced to nearly 119·0°; the halogen atom is usually out of the mean ring plane by 0·05 Å.

The carbon–hydrogen distances are very reasonable. There are eight Csp^3–H bonds whose lengths average 0·99₃ Å and eight Csp^2–H bonds averaging 0·95₅ Å.

The estimated standard deviations for the bond angles are 0·4° for C–C–C angles and 2·2° for C–C–H angles. The average bond angles for each of the six-membered rings is 120·0° in keeping with the trigonal coordination of these atoms while the average is 106·5° for the angles in the five-membered ring. The average bond angle around the nitrogen atom, 112·1° is half-way between the sp^2 and sp^3 angles. All the angles involving hydrogen atoms in the six-membered rings are

consistent with the sp^2 geometry. The C–C–H and N–C–H angles in the methyl groups average 109°, the usual tetrahedral value.

Mean planes

The planes passing through the chlorophenyl (plane A) and the indoline (plane B) groups of atoms are listed in Table 5. The six carbon atoms of the chloro-phenyl group deviate from the mean plane by an average of 0·006 Å. As mentioned above, the chlorine atom lies significantly above plane A. For the indoline group of atoms, only the nitrogen is above plane B, by 0·406 (2) Å. The angle between plane A and plane B is 75·7°.

Table 5. Mean planes

Plane A		Plane B	
Atoms	Deviation from plane	Atoms	Deviation from plane
C(1')	0·009 (2) Å	C(1)	0·010 (2) Å
C(2')	−0·002	C(3)	−0·009
C(3')	−0·009	C(4)	0·001
C(4')	0·009	C(5)	0·002
C(5')	−0·000	C(6)	−0·012
C(6')	−0·009	C(7)	−0·006
Cl*	0·069 (1)	C(8)	0·013 (3)
C(1)*	0·006 (2)	C(9)	0·003 (2)
		N(2)*	0·406 (2)

Equations for the least-squares planes†

Plane A $0·7902X − 0·4036Y − 0·4613Z + 1·6506 = 0$
Plane B $−0·7802X − 0·2705Y − 0·5604Z + 6·3222 = 0$

* Atoms not included in the least-squares calculation.
† The planes are expressed by $lX + mY + nZ − p = 0$ in Å. The orthogonal system of axes has $X \| a$ axis, Y in ab plane and Z along the c^* axis.

Packing of the molecules

The packing of the chlorophenyldimethylisoindo-line molecules is shown in Fig. 3. None of the intermolecular contacts is shorter than 3·30 Å. The molecules are held in the crystal by van der Waals forces only.

Table 4. Comparison of bond distances and angles in halogenophenyl groups

X	Cl	Cl	Cl	Br	Br	Br
l', average length of C–C bonds adjacent to C–X (Å)	1·368	1·379	1·368	1·373	1·369	1·367
l, average of the other C–C bonds in the ring (Å)	1·382	1·398	1·385	1·385	1·387	1·386
Δl/l, relative shortening (%)	1·42	1·36	1·23	0·87	1·30	1·37
Deviation of X from mean plane (Å)	0·068	0·055	0·052	0·051	0·006	0·053
Average C–C–X angle (°)	119·3	118·9	119·0	119·5	119·0	119·1
C–CX–C angles (°)	121·2	122·1	122·0	121·0	122·0	121·8
Final R index	0·040	0·083	0·047	0·053	0·041	0·049
Reference	This work	1	2	3	4	5

References: 1, Jensen & Jerslev (1969); 2, Dreissig & Plieth (1971); 3, 4, Calleri, Ferraris & Viterbo (1969a,b); 5, Portheine, Romers & Rutten (1972).
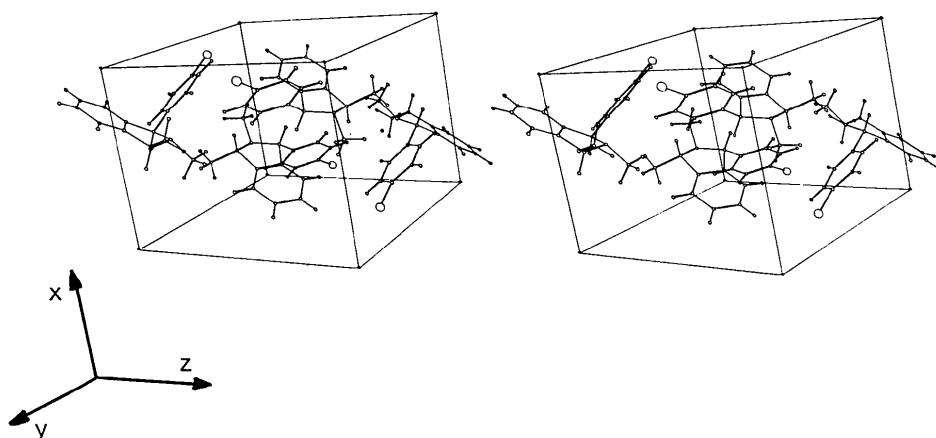
Fig. 3. Packing of the molecules in one unit cell.

References

AHMED, F. R., HALL, S. R., PIPPY, M. E. & HUBER, C. P. (1966). NRC Crystallographic Programs for the IBM/360 System. *World List of Crystallographic Computer Programs*, 2nd ed. Appendix, p. 52.

AHMED, F. R., SINGH, P. & BARNES, W. H. (1969). *Acta Cryst.* B25, 316–328.

CALLERI, M., FERRARIS, G. & VITERBO, D. (1969a). *Acta Cryst.* B25, 1126–1133.

CALLERI, M., FERRARIS, G. & VITERBO, D. (1969b). *Acta Cryst.* B25, 1133–1139.

CARPINO, L. A. (1962). *J. Amer. Chem. Soc.* 84, 2196–2201.

COPPENS, P. & HAMILTON, W. C. (1970). *Acta Cryst.* A26, 71–83.

CROMER, D. T. (1965). *Acta Cryst.* 18, 17–23.

CROMER, D. T. & WABER, J. T. (1965). *Acta Cryst.* 18, 104–109.

DREISSIG, W. & PLIETH, K. (1971). *Acta Cryst.* B27, 1146–1152.

FRETER, K. R., HESS, F. K., SYGUSCH, J. & BRISSE, F. (1971). *Canad. J. Chem.* 49, 3305–3307.

GERMAIN, G., MAIN, P. & WOOLFSON, M. N. (1970). *Acta Cryst.* B26, 274–285.

JENSEN, K. G. & JERSLEV, B. (1969). *Acta Cryst.* B25, 916–925.

PALENIK, G. J., DONOHUE, J. & TRUEBLOOD, K. N. (1968). *Acta Cryst.* B24, 1139–1146.

PETRACEK, F. J., SUGISAKA, N., KLOHS, M. W., PARKER, R. G., BORDNER, J. & ROBERTS, J. D. (1970). *Tetrahedron Lett.* 10, 707–710.

PORTHEINE, J. C., ROMERS, C. & RUTTEN, E. W. M. (1972). *Acta Cryst.* B28, 849–857.

SCHOMAKER, V. & TRUEBLOOD, K. N. (1968). *Acta Cryst.* B24, 63–76.

SINGH, P. & AHMED, F. R. (1969). *Acta Cryst.* B25, 1901–1910.

STEWART, R. F., DAVIDSON, E. R. & SIMPSON, W. T. (1965). *J. Chem. Phys.* 42, 3175–3187.

SUTTON, L. E. (1965). *Tables of Interatomic Distances and Configuration in Molecules and Ions.* Supplement 1956–1959. London: The Chemical Society.