The O atom engages in a hydrogen bond with H(2), with the interatomic distances H(2)···O(1) 2·23 and N(4)···O(1) 2·926 Å, and an NHO angle of 141°. There are no other significant intermolecular interactions.

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The Crystal Structures of the α Form of *p*-Chlorobenzamide at Room Temperature and −120°C

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Crystal structures at room temperature and −120°C have been investigated by X-rays for the α form of *p*-chlorobenzamide. Crystal data are: (room temperature) $a = 15{\cdot}027$ (7), $b = 5{\cdot}481$ (4), $c = 14{\cdot}486$ (7) Å, $\alpha = 97{\cdot}84$ (5), $\beta = 111{\cdot}99$ (5), $\gamma = 95{\cdot}17$ (5)°; (−120°C) $a = 14{\cdot}95$, $b = 5{\cdot}42$, $c = 14{\cdot}23$ Å, $\alpha = 97{\cdot}0$, $\beta = 111{\cdot}5$, $\gamma = 95{\cdot}0$°; $Z = 6$, triclinic with space group $P\bar{1}$. Intensity data were collected by the equi-inclination Weissenberg technique. The structures were refined by block-diagonal least-squares calculations. The R values for the room- and low-temperature structures based on 3090 and 3112 reflexions were 0·110 and 0·108 respectively. The crystal structures are composed of one centrosymmetric and two asymmetric dimeric units consisting of independent molecules, *A*, *B* and *C*. These units are linked by hydrogen bonds to form an endless chain along **a**. Dihedral angles between the planes of the amide group and the benzene ring for molecules *A*, *B* and *C* are 20·3, 30·3 and 33·8° (17·9, 29·1 and 33·7°) respectively, where the values in parentheses are those for the low-temperature structure. Molecules *B* and *C* are approximately parallel to each other, but the plane of the benzene ring for *A* makes angles of 34·0 and 37·8° (40·0 and 44·4°) with those for *B* and *C* respectively. A marked movement of molecules occurring gradually over a range of temperatures below the transition point (*ca* 44°C) can be described approximately in terms of rotations of molecules about the long axes of the dimeric units. The sense of rotation is the same as that of the rotation occurring drastically during the transition.

Introduction

p-Chlorobenzamide, C_7H_6ClNO, exhibits two polymorphs at room temperature (Harada, Taniguchi, Takaki & Sakurai, 1969). They are triclinic with space group $P\bar{1}$ or $P1$ (α form) and monoclinic with $P2_1/a$ (β form). Harada, Taniguchi, Takaki & Sakurai found that crystals of the α form undergo a reversible solid-phase transformation at about 44°C to the high-temperature disordered form (γ form, space group $P\bar{1}$), the *a* axis being one third that of the α form. They obtained an 'averaged structure' for the γ form by means of sharp reflexions only, but gave no solutions for the structures of the α and β forms.

In a preliminary investigation of the α form we found that reflexions with $h \neq 3n$ (n integer) were considerably less intense than those with $h = 3n$. Further we found that, by examining Weissenberg photographs taken at room temperature (*ca* 27 °C) and −120 °C, the reflexions with $h \neq 3n$ at −120 °C were generally more intense than the corresponding reflexions at room temperature even though a temperature effect was taken into account. This may suggest the existence of a gradual movement of molecules over the above mentioned temperature range. Therefore, in this paper, we deal with the structure analyses for the α form at room temperature and −120 °C to obtain information about the movement of molecules below the transition point. The detailed structure of the γ form and an explanation of the process of the phase transformation occurring between the α and γ forms are given in a separate paper (Takaki, Nakata, Taniguchi & Sakurai, 1978).

Experimental

Crystals obtained from an ethanol solution had a parallelepiped shape elongated along **b**. Cell dimensions were determined from photographs taken with a Buerger back-reflexion Weissenberg camera whose camera constant was calibrated by using Ag as a standard. The cell dimensions at room temperature and −120 °C are given in Table 1, where those for the γ form at 50 °C are also given for comparison.

Intensity data were collected on equi-inclination Weissenberg photographs for $k = 0$ to 3. Ni-filtered Cu $K\alpha$ radiation was used at room temperature but unfiltered radiation at −120 °C. The cross-sections of the crystals used for X-ray measurements at room

Table 1. *Cell dimensions of p-chlorobenzamide*

	α Form		γ Form*
	27 °C	−120 °C	50 °C
a	15·027 (7) Å	14·95 Å	5·015 Å
b	5·481 (4)	5·42	5·530
c	14·486 (7)	14·23	14·707
α	97·84 (5)°	97·0°	97·53°
β	111·99 (5)	111·5	113·03
γ	95·17 (5)	95·0	95·67
Space group	$P\bar{1}$	$P\bar{1}$	$P\bar{1}$
Z	6	6	2

* Takaki, Nakata, Taniguchi & Sakurai (1978).

Table 2. *Fractional coordinates* (× 10⁴) *for the non-hydrogen atoms for the α form at room temperature* (27 °C) *and* −120 °C

	27 °C			−120 °C		
	x	*y*	*z*	*x*	*y*	*z*
Cl(*A*)	2571 (1)	12692 (4)	4128 (1)	2547 (1)	12765 (4)	4121 (1)
N(*A*)	1140 (3)	1981 (11)	701 (3)	1141 (3)	1914 (11)	729 (3)
O(*A*)	−272 (2)	2696 (9)	792 (3)	−293 (2)	2763 (10)	766 (3)
C(1*A*)	618 (3)	3337 (12)	1078 (4)	606 (3)	3325 (13)	1071 (4)
C(2*A*)	1133 (3)	5624 (12)	1850 (3)	1118 (4)	5647 (13)	1852 (4)
C(3*A*)	2032 (4)	6837 (14)	1963 (4)	2051 (4)	6699 (14)	2011 (4)
C(4*A*)	2477 (4)	8926 (16)	2661 (5)	2496 (4)	8871 (15)	2722 (4)
C(5*A*)	2031 (4)	9893 (15)	3262 (4)	2008 (4)	9978 (14)	3274 (4)
C(6*A*)	1133 (5)	8705 (18)	3168 (5)	1094 (4)	8901 (17)	3147 (4)
C(7*A*)	700 (4)	6620 (15)	2478 (5)	646 (4)	6740 (15)	2429 (5)
Cl(*B*)	6257 (2)	11936 (5)	4370 (2)	6291 (1)	11868 (4)	4399 (1)
N(*B*)	4391 (3)	2189 (11)	526 (3)	4381 (3)	2193 (12)	526 (3)
O(*B*)	3211 (2)	1998 (9)	1127 (3)	3221 (3)	1900 (10)	1177 (3)
C(1*B*)	4009 (3)	3027 (12)	1184 (4)	3997 (3)	2971 (13)	1201 (4)
C(2*B*)	4550 (3)	5284 (12)	1954 (4)	4553 (3)	5216 (13)	1987 (3)
C(3*B*)	5145 (4)	7067 (14)	1782 (4)	5140 (4)	7028 (14)	1789 (4)
C(4*B*)	5651 (5)	9102 (15)	2508 (5)	5666 (5)	9084 (16)	2519 (5)
C(5*B*)	5584 (4)	9355 (15)	3433 (4)	5611 (4)	9336 (15)	3475 (4)
C(6*B*)	5002 (6)	7600 (16)	3630 (5)	5035 (5)	7573 (16)	3694 (4)
C(7*B*)	4481 (4)	5612 (15)	2892 (4)	4492 (4)	5503 (15)	2945 (4)
Cl(*C*)	9266 (2)	12368 (5)	4311 (2)	9248 (1)	12388 (4)	4328 (1)
N(*C*)	7648 (3)	2913 (11)	322 (3)	7621 (3)	3044 (11)	276 (3)
O(*C*)	6426 (2)	2510 (9)	865 (3)	6414 (3)	2555 (10)	856 (3)
C(1*C*)	7230 (3)	3663 (12)	961 (4)	7192 (3)	3700 (14)	929 (4)
C(2*C*)	7719 (3)	5830 (12)	1763 (4)	7696 (3)	5927 (13)	1748 (4)
C(3*C*)	8270 (4)	7829 (15)	1634 (4)	8226 (4)	7958 (15)	1604 (4)
C(4*C*)	8727 (5)	9848 (16)	2402 (5)	8697 (4)	9976 (16)	2384 (5)
C(5*C*)	8659 (4)	9879 (15)	3312 (5)	8629 (4)	9938 (15)	3336 (4)
C(6*C*)	8103 (5)	7892 (16)	3468 (5)	8099 (5)	7947 (16)	3496 (4)
C(7*C*)	7634 (4)	5969 (14)	2689 (4)	7614 (4)	5968 (15)	2693 (4)

temperature and −120°C were 0·25 × 0·25 and 0·35 × 0·35 mm respectively. Intensities of 3090 independent reflexions at room temperature and of 3112 at −120°C were estimated visually and corrected for Lorentz and polarization effects. No correction for absorption was applied.

The orientational relationship between the unit-cell axes at room temperature and −120°C was examined by taking double-exposure Weissenberg photographs about **a** and **b** (Herbstein, 1965). No significant changes in the directions of the *a* and *b* axes were observed, but the *c* axial directions at the two temperatures differed significantly from each other (*ca* 1°). Note that, since relative orientations of the unit-cell axes at room temperature and 50°C have been obtained (Takaki, Nakata, Taniguchi & Sakurai, 1978), we can examine the structural changes over the temperatures −120 to 50°C.

Weissenberg photographs taken at room temperature showed that there exists weak but significant diffuse scattering very similar to that found in the γ form (see Fig. 1 of Takaki, Nakata, Taniguchi & Sakurai, 1978). Further, a series of stationary-crystal strip photographs taken with a Weissenberg camera

Table 3. *Interatomic distances and angles*

(*a*) Bond lengths (Å)

		27°C			−120°C	
	A	*B*	*C*	*A*	*B*	*C*
N—C(1)	1·334 (9)	1·337 (9)	1·341 (9)	1·329 (10)	1·335 (10)	1·342 (10)
O—C(1)	1·243 (8)	1·246 (8)	1·260 (8)	1·247 (9)	1·238 (9)	1·232 (9)
C(1)—C(2)	1·488 (10)	1·483 (9)	1·459 (9)	1·506 (10)	1·493 (10)	1·494 (10)
C(2)—C(3)	1·391 (10)	1·376 (10)	1·389 (10)	1·385 (11)	1·383 (10)	1·379 (11)
C(2)—C(7)	1·386 (11)	1·390 (10)	1·387 (10)	1·381 (11)	1·390 (11)	1·392 (11)
C(3)—C(4)	1·349 (11)	1·363 (11)	1·375 (12)	1·389 (11)	1·379 (12)	1·385 (12)
C(4)—C(5)	1·365 (12)	1·369 (12)	1·357 (12)	1·377 (11)	1·385 (12)	1·398 (12)
C(5)—C(6)	1·395 (13)	1·373 (12)	1·407 (12)	1·377 (12)	1·370 (12)	1·373 (12)
C(6)—C(7)	1·340 (13)	1·358 (12)	1·351 (12)	1·389 (12)	1·402 (12)	1·392 (12)
C(5)—Cl	1·753 (9)	1·749 (8)	1·743 (9)	1·736 (8)	1·726 (9)	1·727 (9)

(*b*) Bond angles (°)

		27°C			−120°C	
	A	*B*	*C*	*A*	*B*	*C*
N—C(1)—O	120·7 (6)	121·0 (6)	120·3 (6)	121·7 (7)	121·9 (7)	122·0 (7)
N—C(1)—C(2)	118·1 (6)	117·5 (6)	118·9 (6)	118·0 (6)	116·3 (6)	116·8 (6)
O—C(1)—C(2)	121·2 (6)	121·5 (6)	120·8 (6)	120·4 (6)	121·8 (6)	121·2 (7)
C(1)—C(2)—C(3)	122·8 (6)	122·0 (6)	122·2 (6)	121·9 (7)	121·5 (7)	123·0 (7)
C(1)—C(2)—C(7)	119·1 (6)	119·8 (6)	120·3 (6)	118·9 (7)	119·9 (7)	118·5 (7)
C(3)—C(2)—C(7)	118·1 (7)	118·1 (7)	117·5 (7)	119·2 (7)	118·7 (7)	118·5 (7)
C(2)—C(3)—C(4)	121·6 (7)	121·1 (7)	121·1 (7)	120·5 (7)	121·6 (7)	121·6 (7)
C(3)—C(4)—C(5)	119·3 (8)	119·6 (8)	119·8 (8)	119·6 (7)	119·4 (8)	118·8 (8)
C(4)—C(5)—C(6)	120·3 (8)	120·7 (8)	120·6 (8)	120·3 (8)	120·4 (8)	120·8 (8)
C(4)—C(5)—Cl	119·9 (7)	119·3 (6)	120·6 (7)	119·5 (6)	119·4 (6)	119·3 (6)
C(6)—C(5)—Cl	119·8 (7)	120·0 (7)	118·8 (7)	120·2 (6)	120·2 (7)	119·9 (7)
C(5)—C(6)—C(7)	119·9 (9)	119·2 (8)	118·4 (8)	119·9 (8)	120·0 (8)	119·3 (8)
C(2)—C(7)—C(6)	120·8 (8)	121·3 (8)	122·5 (7)	120·4 (8)	120·0 (7)	120·9 (8)

(*c*) Intermolecular distances (Å)

	27°C	−120°C		27°C	−120°C
Cl(*A*)—Cl(*B*$^{\mathrm{vii}}$)	3·31	3·29	N(*B*)—O(*C*$^{\mathrm{v}}$)	2·89	2·90
N(*A*)—O(*B*)	2·94	2·94	O(*B*)—N(*C*$^{\mathrm{v}}$)	3·02	3·03
N(*A*)—O(*A*$^{\mathrm{iii}}$)	2·95	2·95	C(4*B*)—C(7*C*)	3·52	3·44
O(*A*)—C(1*A*$^{\mathrm{iv}}$)	3·62	3·49	Cl(*C*)—Cl(*C*$^{\mathrm{viii}}$)	3·34	3·34
C(3*A*)—N(*C*$^{\mathrm{vi}}$)	3·54	3·48	N(*C*)—O(*A*$^{\mathrm{i}}$)	2·96	2·96
C(4*A*)—O(*B*$^{\mathrm{ii}}$)	3·39	3·32	C(4*C*)—C(6*A*$^{\mathrm{i}}$)	3·51	3·47
N(*B*)—O(*C*)	2·90	2·89	C(4*C*)—C(1*C*$^{\mathrm{ii}}$)	3·51	3·44

Symmetry operators

(i)	$1+x$,	y,	z	(ii)	x,	$1+y$,	z
(iii)	$-x$,	$-y$,	$-z$	(iv)	$-x$,	$1-y$,	$-z$
(v)	$1-x$,	$-y$,	$-z$	(vi)	$1-x$,	$1-y$,	$-z$
(vii)	$1-x$,	$3-y$,	$1-z$	(viii)	$2-x$,	$3-y$,	$1-z$

(Flack, 1970) at temperatures between room temperature and −120 °C showed that the diffuse scattering associated with reflexions with $h = 3n$ weakens in intensity with decreasing temperature, while that associated with reflexions with $h \neq 3n$ remains almost unchanged over the temperature range. The above results may indicate that the latter diffuse scattering is mainly due to structural disorder whilst the former is due to the thermal effect. By considering the result of the γ form analysis, it may be inferred that the crystal of the α form contains small amounts of domains having the structure of the γ form even at −120 °C.

Determination and refinement of the structures

Patterson maps for the α form were found to be very similar to those for the γ form given by Harada, Taniguchi, Takaki & Sakurai, indicating that the arrangement of molecules in the α form may be obtained from slight shifts and reorientations of molecules in the γ form. The initial structure was then obtained, by assuming space group $P\bar{1}$, from closer examination of Patterson maps and was refined by successive Fourier and block-diagonal least-squares calculations. Non-hydrogen-atom parameters were used for refinements. Contributions of hydrogen atoms located in the calculated positions were included in the structure factors, taking $U = 0\cdot063$ and $0\cdot044$ Å^2 for the room- and low-temperature structures respectively. A weighting scheme of the type $w = 1 - \exp(-20s^2)$ with $s = \sin\theta/\lambda$ was used. The final R values for all reflexions for the room- and low-temperature structures were 0·110 and 0·108 respectively. (The R values for reflexions with $h = 3n$, $3n + 1$ and $3n + 2$ were 0·098, 0·131 and 0·133, respectively, for the room-temperature structure and 0·096, 0·127 and 0·137 for the low-temperature structure.)

Another starting structure derived by assuming space group $P1$ was refined to $R = 0\cdot102$ for the low-temperature structure, but the result was evidently unreasonable for bond lengths and thermal parameters. No further attempts were made to find alternative models for $P1$.

Positional parameters for the three crystallographically independent molecules A, B and C for the room- and low-temperature structures are given in Table 2.* Bond lengths and angles and interatomic distances are given in Table 3 and equations of the least-squares planes in Table 4.

The numerical calculations were performed on the HITAC 8700/8800 computer at the Computer Centre of Tokyo University using program *HBLS*-IV by Y. Okaya and T. Ashida for the least-squares refinement.

Discussion

The crystal structure is illustrated in Fig. 1. It consists of stacking of molecular sheets parallel to $(01\bar{3})$ with inter-layer spacing of 3·76 Å (3·68 Å; hereinafter the results for the low-temperature structure are shown in

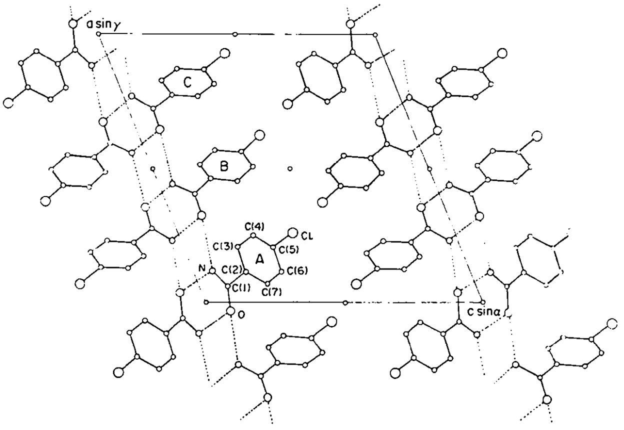

Fig. 1. Structure of the α form viewed along **b**.

Table 4. *Least-squares planes of benzene rings and planes of amide groups referred to the crystal axes*

(*a*) Benzene rings

	27 °C	−120 °C
Molecule *A*	$-4\cdot115x + 3\cdot883y - 9\cdot107z = 0\cdot029$	$-2\cdot833x + 3\cdot778y - 9\cdot724z = 0\cdot003$
Molecule *B*	$-11\cdot148x + 3\cdot523y - 1\cdot890z = -3\cdot577$	$-11\cdot199x + 3\cdot470y - 1\cdot625z = -3\cdot607$
Molecule *C*	$-11\cdot882x + 3\cdot240y - 1\cdot225z = -7\cdot486$	$-12\cdot024x + 3\cdot157y - 0\cdot844z = -7\cdot516$

(*b*) Amide groups

	27 °C	−120 °C
Molecule *A*	$1\cdot110x + 3\cdot681y - 11\cdot704z = 0\cdot035$	$1\cdot781x + 3\cdot520y - 11\cdot736z = 0\cdot021$
Molecule *B*	$-4\cdot774x + 4\cdot149y - 8\cdot055z = -1\cdot612$	$-5\cdot299x + 4\cdot011y - 7\cdot637z = -1\cdot844$
Molecule *C*	$-4\cdot928x + 4\cdot106y - 8\cdot043z = -2\cdot832$	$-5\cdot268x + 4\cdot060y - 7\cdot540z = -2\cdot987$

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* Lists of structure factors and anisotropic thermal parameters for the non-hydrogen atoms for the room- and low-temperature structures have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 33479 (27 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 5. *Coordinates* (Å) *of the centres of gravity of molecules at* 50 °C, *room temperature* (27 °C) *and* −120 °C

The coordinates are referred to the unit-cell axes for the γ form.

	Molecule *A*			Molecule *B*			Molecule *C*		
	ax	*by*	*cz*	*ax*	*by*	*cz*	*ax*	*by*	*cz*
50°C	2·302	3·976	3·572	2·302	3·976	3·572	2·302	3·976	3·572
27°C	2·344	4·011	3·493	2·586	3·768	3·699	2·276	4·065	3·473
−120°C	2·358	4·044	3·461	2·644	3·764	3·701	2·277	4·116	3·423

parentheses). Within a sheet, there are three kinds of dimeric units; one is a centrosymmetric dimer formed by NH···O hydrogen bonds of 2·954 Å (2·949 Å), while each of the other two is an asymmetric one formed by hydrogen bonds of 2·891 and 3·020 Å (2·900 and 3·030 Å). These dimeric units are linked by NH···O hydrogen bonds of 2·937, 2·896 and 2·957 Å (2·939, 2·885 and 2·959 Å) to form an endless chain along **a**. The Cl–Cl distance of 3·31 Å (3·29 Å) is the shortest distance between the neighbouring chains. A similar short distance of 3·335 Å has been found for chloranilic acid (Andersen, 1967).

Bond lengths and angles for the three independent molecules agree fairly well, but there are striking differences in the dihedral angles between the planes of the benzene ring and the amide group for each of the three molecules. The values are 20·3, 30·3 and 33·8° (17·9, 29·1 and 33·7°) for molecules *A*, *B* and *C* respectively. In addition, the values for *A* and *C* are exceedingly small and large as compared with those found in other related amides, which vary from about 23° (terephthalamide; Cobbledick & Small, 1972) to 29° (*m*-methylbenzamide; Orii, Nakamura, Takaki, Sasada & Kakudo, 1963). These unusual values of the angles, together with the unusual orientational relationship between the three independent molecules described below, may be closely related to the formation of the strange hydrogen-bonding scheme found in the α form. Note that the average value of the dihedral angles for molecules *A*, *B* and *C* at room temperature is 28·1°, the same value as for structure *V* for the γ form.

A fundamental difference between the crystal structures of the α form and structure *V* for the γ form concerns the arrangement of molecules in a chain along **a**. As shown in Fig. 2, the orientations of molecules *B* and *C* are very similar to each other, while that of *A* is remarkably different from the other two; the angle between the planes of the benzene rings for *B* and *C* is 4·4° (5·0°), while the plane of the benzene ring for *A* makes angles of 34·0 and 37·8° (40·0 and 44·4°) with those for *B* and *C* respectively. The corresponding values of the angles for the amide groups for the three molecules are rather small probably due to the correlations between the neighbouring hydrogen-bonded amide group: They are respectively 0·8° (0·8°) and 23·9 and 24·3° (28·6 and 28·7°).

The orientations of the three molecules described above can be produced approximately by rotating the molecules in structure *V* about C(2)–C(5) axes; *e.g.* the rotation angles for the benzene rings for *B* and *C* are about 9·9 and 13·5° (11·2 and 15·2°) respectively, and that for *A* is −24·3° (−29·2°). This shows that the sense of molecular rotation about the long axis of each dimer occurring drastically during the transition is the same as that of the rotation occurring gradually over a range of temperatures below the transition point. A similar feature was found in the positional changes of the centres of gravity of the molecules taking place during the transition and below the transition point (see Table 5).

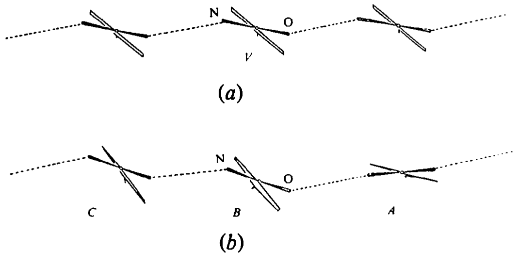

Fig. 2. Projections showing orientations of molecules. (*a*) Structure *V* at 50°C; (*b*) α form at room temperature. For clarity centrosymmetric molecules are omitted. The amide groups and the benzene rings are represented by heavy and light lines respectively.

To obtain information about the movement of molecules at the temperature near the transition point, we took Weissenberg photographs at about 40°C and compared them with those taken at room temperature. No significant differences were observed between these two sets of photographs, indicating that marked movement of the molecules does not occur even at 40°C.

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