ally influencing the structure determination as described in the paper, confirmed in principle that these machines may be capable of aiding the evaluation of structures with marked pseudo-symmetry (actual or in projection).

Finally, the authors wish to thank Mr E. W. Colbeck, Metallurgical Director of Messrs Hadfields Limited, for his continued interest and encouragement, and for permission to publish this communication.

References

A. S. T. M. *Index of X-ray Diffraction Data* (1950a). Card 3-0825.
A. S. T. M. *Index of X-ray Diffraction Data* (1950b). Card 1-1027.
BUNN, C. W. (1945). *Chemical Crystallography*, p. 277. Oxford: Clarendon Press.
FORD, W. F. & REES, W. J. (1949). *Trans. Brit. Ceram. Soc.* **48**, 291.
HARGREAVES, A. (1955). *Acta Cryst.* **8**, 12.
HOWELLS, E. R., PHILLIPS, D. C. & ROGERS, D. (1950). *Acta Cryst.* **3**, 210.
MACLENNAN, G. & BEEVERS, C. A. (1955). *Acta Cryst.* **8**, 579.
MALQUORI, G. L. & CIRILLI, V. (1952). *Third International Symposium on the Chemistry of Cement.* London: Cement and Concrete Association.
TAKÉUCHI, Y., WATANABÉ, T. & ITO, T. (1950). *Acta Cryst.* **3**, 98.
ZEMANN, J. (1947). *Experientia*, **3**, 452.

The Crystal Structure of an Unstable Form of Chloroacetamide

BY MIKIO KATAYAMA

Department of Chemistry, The University of Tokyo, Bunkyo-ku, Tokyo, Japan

Chloroacetamide, $CH_2Cl.CONH_2$, was found to have two modifications, both of which belong to the same space group ($P2_1/a$, 4 molecules per unit cell) and have almost the same cell dimensions. The difference is only in the value of the angle β: $102.5°$ in the unstable, and $98° 49'$ in the stable form. The crystal and molecular structure of the unstable form have been determined from a study of the two-dimensional projections of ϱ and $(\varrho_o - \varrho_c)$ along the a and b axes. Bond lengths are almost the same as those in the related molecules: C–Cl $= 1.76$ Å, C–C $= 1.51$ Å, C–O $= 1.25$ Å and C–N $= 1.40$ Å (the estimated standard deviation of each atom is about 0.03 Å). The C–Cl bond takes approximately the *cis* position to the C–N bond. An interesting rule is found in the bond lengths and bond angles of the terminal amide group. N–H $\cdots$ O hydrogen bonds are relatively weak in this crystal, the shortest N–H $\cdots$ O distance being 3.05 Å and others about 3.37 Å and 3.39 Å.

Introduction

For some years past the protein structure has been approached by means of detailed X-ray analysis of some amino acids and related compounds which constitute the polypeptide chain of proteins. Much information has already been obtained concerning not only the dimensions and configurations of these molecules but also the nature and strength of the forces which bind them together. The structure of the terminal amide group has been studied for oxamide (Romers, 1953; Ayerst & Duke, 1954), succinamide (Pasternak, 1953), nicotin amide (Wright & King, 1954), glycyl-L-asparagine (Pasternak, Katz & Corey, 1954), L-glutamine (Cochran & Penfold, 1952), acetamide (Senti & Harker, 1940), 6-amido-3-pyridazone (Cucka & Small, 1954), and tetradecanamide (Turner & Lingafelter, 1955), but the dimensions reported for the group are appreciably different from each other. For this reason an accurate analysis of the crystal

structure of simple amides and the comparison of the results with those already reported would be of interest.

Recently Dejace (1955) reported an X-ray analysis of the crystal structure of chloroacetamide. He prepared the crystals from alcoholic or benzene solution. The present author obtained another form of the crystal by slow evaporation of its aqueous solution; for convenience, this will be termed the unstable form in this article. An accurate analysis of the structure of this crystal and the comparison of the results with the structure of the stable form are reported here.

Crystal data and experimental measurements

Chloroacetamide, $Cl–CH_2CO.NH_2$, was prepared by Dr I. Nakagawa, and its purity was examined by use of the Raman and infra-red spectra. Single crystals were obtained from aqueous solution in plate form or

needles elongated along the b axis. The (001) faces were predominant and there was cleavage parallel to the (10$\bar{1}$).

Rotation and oscillation and Weissenberg photographs were taken with Cu $K\alpha$ radiation. The cell dimensions, determined from the oscillation photographs, are

$$a = 7\cdot45, \quad b = 5\cdot15, \quad c = 10\cdot27 \text{ Å}, \quad \beta = 102\cdot5°.$$

Absent spectra: ($h0l$) when h is odd, and ($0k0$) when k is odd. Space group: C_{2h}^5–$P2_1/a$. It should be noted that the length of the c axis is one-half of the value given by Groth (1906–19), and β is larger by about 4° than his value.

Dejace prepared his crystals of chloroacetamide from ethyl alcohol or benzene solution and gave the following cell dimensions: $a=7\cdot41$, $b=5\cdot13$, $c=10\cdot26$ Å, $\beta=98°$ 49'. The angle β is the same as the value of Groth in this case. Single crystals of the stable form were also found among our crystals prepared from aqueous solution and kept in a desiccator for a long time. Oscillation photographs of these crystals were taken around the c axis and the intensities of the ($hk0$) reflexions were measured. The electron density projected on ($00l$), calculated with these data, gave the same structure as that obtained by Dejace, and β was found to be 98·5° by the Weissenberg photographs around the b axis.

The intensities of the ($h00$) and ($0k0$) reflexions were found to be almost the same in crystals of both forms, except the (200) reflexion: for the stable form this was nearly zero and hard to observe, while for the unstable form it was very strong.

Debye–Scherrer patterns of the crystals prepared from water and chloroform solutions were also examined with Cu $K\alpha$ radiation. There seem to exist slight differences in the scattering angles and a large difference in relative intensities. Some of the samples prepared from aqueous solution showed double peaks at the scattering angles of about 27° and 24°, while the others have only single peaks at these angles. This seems to indicate that the former was a mixture of the stable and unstable forms. If we assume that the crystals prepared from aqueous solution are ready to change to the stable form, it can be easily understood why the reflexions of the mixture were observed and why the patterns of the stable form were found with the lapse of time. Of course, there is also a possibility that under certain conditions both forms appear simultaneously on crystallization from aqueous solution.

As the unstable crystals sublimed easily at ordinary temperature and pressure, they had to be coated with paraffin. The crystals used for the b-axis photographs were 0·2 mm. in diameter. Those for the a-axis photographs were cut from larger crystals, the dimensions of the section being about 0·25×0·4 mm.². Special care was taken to secure specimens of approximately square cross-section. This attempt, however, was un-

successful in the case of the crystal for the a-axis photographs.

The intensity data used in the present analysis were confined to a complete survey of the ($h0l$) and ($0kl$) reflexions. They were estimated visually, using the multiple-film technique to correlate strong and weak reflexions.

Structure determination

Structure determination using Harker–Kasper inequalities was found to be unsuccessful; it gave the signs of only a few structure factors. Since the molecule has one heavy atom (chlorine), the position of this atom is relatively easily determined by the method of Patterson projection. Thus the approximate position of the chlorine atom was found to be $x \approx \frac{1}{8}$, $y \approx \frac{1}{4}$, and $z \approx \frac{1}{8}$ or $\frac{3}{8}$ from Patterson projections upon (010) and (100). It is now possible to assume the crystal structure from the known bond lengths and angles by packing atoms in a unit cell. Starting from this trial structure, improved parameters of all atoms were obtained by trial and error. The signs of the majority of ($h0l$) and ($0kl$) structure factors were obtained and used with suitable temperature factors to evaluate electron-density projections. The final electron-density projection on (100) is shown in Fig. 1. At this stage
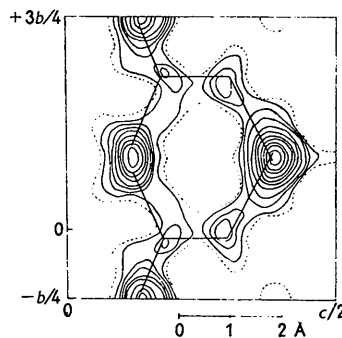
Fig. 1. Electron density projected on (100). Contours at every 2 e.Å^{-2} for C, O, N atoms and at 4 e.Å^{-2} for Cl atom. Broken lines represent 1 e.Å^{-2}.

the percentage discrepancies $(100\Sigma|F_o-F_c| \div \Sigma|F_o|)$ were 25% for ($0kl$) and 23% for ($h0l$).

Refinement of structure and estimation of accuracy

Further refinement of this projection of the structure was carried out by evaluating successive Fourier difference synthesis with (F_o-F_c) as coefficients. Coordinate adjustments were first made until the electron-density slope at the atomic centres became zero. Next the temperature factors were adjusted, assuming that they are isotropic. The coordinates of the atoms (except the four hydrogen atoms) and their isotropic temperature factors are shown in Table 1. At this stage the percentage discrepancies were 17·8% for ($0kl$) and 15·2% for ($h0l$).

Table 1. *Coordinates and temperature factors*

	Cl	C_1	C_2	O	N
x	0.113_3	0.085_0	-0.051_7	-0.136_7	-0.098_3
y	0.233_3	0.533_3	0.533_3	0.716_7	0.295_0
z	0.385_0	0.305_0	0.180_0	0.138_3	0.118_3
β (Å²)	4.5	0.5	0.5	1.5	1.0

The final $(\varrho_o-\varrho_c)$ maps are shown in Fig. 2. The significant peaks on these maps may be partly due to the hydrogen atoms and partly due to anisotropic vibration of atoms. The coordinates of the four hydrogen atoms were tentatively assumed as shown

Table 2. *Assumed coordinates for hydrogen atoms*

	H_1	H_2	H_3	H_4
x	0.039	0.190	-0.143	-0.215
y	0.668	0.480	0.350	0.175
z	0.370	0.275	0.038	0.190

in Table 2 from these $(\varrho_o-\varrho_c)$ maps by taking into account the known C–H and N–H bond lengths and angles. In Table 3 t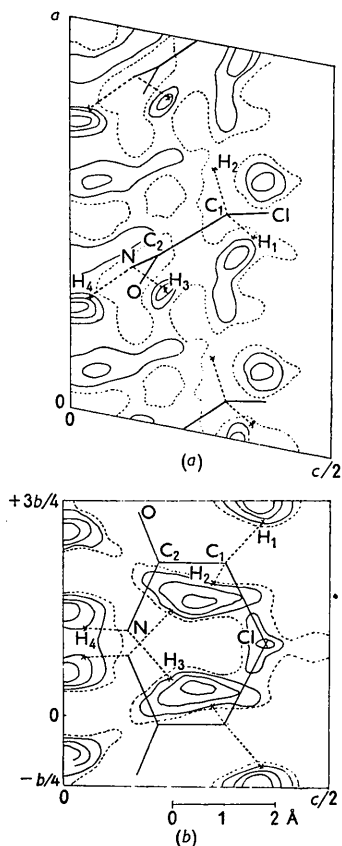here are listed the observed and calculated structure factors including the contribution of the hydrogen atoms. The final percentage discrepancies are 15·6% and 15·0% for the $(0kl)$ and $(h0l)$ respectively.

The standard deviations of the atomic coordinates were estimated by the method of Booth & Britten (1948), using both the assumptions that $\sigma(F_o)=0.2F_o$ and $\sigma(F_o)=3.2$. These values of the standard deviations of the structure factors were computed by the comparison of the independent measurements made on the oscillation and Weissenberg photographs. Larger deviations of the atomic coordinates were found when the relation $\sigma(F_o) = 3.2$ was used. The results are shown in Table 4. The probable errors of the bond lengths are also given in Table 4.

Table 3. *Observed and calculated structure factors*

hkl	$|F_o|$	F_c	hkl	$|F_o|$	F_c	hkl	$|F_o|$	F_c
000	—	192	$\bar{4}$09	7	5	0,1,11	10	11
200	29	−29	$\bar{4}$,0,10	6	− 3	0,1,12	0	2
400	56	−54	$\bar{4}$,0,11	6	11	021	5	− 6
600	13	−16	$\bar{4}$,0,12	4	1	022	28	19
800	< 7	6	601	7	12	023	9	1
201	7	−12	602	15	−15	024	50	51
202	80	80	603	7	− 5	025	10	−11
203	10	− 7	604	5	0	026	10	13
204	16	−16	605	0	2	027	0	− 2
205	6	− 7	606	15	15	028	37	33
206	31	−28	607	8	− 1	029	< 9	1
207	13	7	608	4	4	0,2,10	0	3
208	7	10	$\bar{6}$01	0	− 4	0,2,11	7	7
209	7	7	$\bar{6}$02	23	28	0,2,12	10	9
2,0,10	16	17	$\bar{6}$03	7	− 9	031	8	21
2,0,11	8	− 5	$\bar{6}$04	10	15	032	18	−25
2,0,12	10	8	$\bar{6}$05	< 7	4	033	18	14
$\bar{2}$01	9	8	$\bar{6}$06	19	−23	034	0	− 1
$\bar{2}$02	113	−113	$\bar{6}$07	< 7	− 2	035	0	− 6
$\bar{2}$03	8	8	$\bar{6}$08	0	− 2	036	14	14
$\bar{2}$04	3	2	$\bar{6}$09	0	9	037	9	− 5
$\bar{2}$05	10	13	$\bar{6}$,0,10	< 5	4	038	0	3
$\bar{2}$06	41	41	001	2	− 4	039	0	− 6
$\bar{2}$07	11	− 7	002	22	−24	0,3,10	0	− 6
$\bar{2}$08	6	1	003	4	− 4	0,3,11	8	9
$\bar{2}$09	7	−13	004	70	−72	041	10	− 4
$\bar{2}$,0,10	7	6	005	12	10	042	9	− 6
$\bar{2}$,0,11	10	12	006	5	5	043	7	− 1
$\bar{2}$,0,12	11	−10	007	8	13	044	9	−20
401	5	4	008	19	16	045	< 9	4
402	17	20	009	12	−11	046	0	3
403	0	− 4	0,0,10	13	12	047	<10	4
404	25	25	0,0,11	< 6	− 4	048	8	3
405	6	− 2	0,0,12	7	−10	049	0	− 5
406	7	5	020	58	−48	0,4,10	4	5
407	7	− 5	040	31	35	051	0	5
408	19	−19	060	9	− 3	052	13	11
409	10	3	011	27	−25	053	9	− 9
4,0,10	10	− 8	012	50	−49	054	0	1
$\bar{4}$01	5	10	013	42	42	055	0	− 1
$\bar{4}$02	21	−20	014	0	2	056	7	− 8
$\bar{4}$03	5	0	015	4	2	057	0	12
$\bar{4}$04	52	52	016	26	−25	061	4	3
$\bar{4}$05	5	− 2	017	25	−25	062	0	− 2
$\bar{4}$06	6	2	018	0	2	063	< 6	2
$\bar{4}$07	7	− 9	019	0	0	064	< 5	4
$\bar{4}$08	17	−16	0,1,10	6	6	065	4	− 3

(a)

Fig. 2. $(\varrho_o - \varrho_c)$ projection (a) on (010), (b) on (100). Contours at every 0·3 e.Å^{-2}, zero contour being broken.

Table 4. *Standard deviations of atomic coordinates, assuming constant* $\sigma(F_o) = 3·2$, *and the probable errors of bond lengths*

Atom	Cl	C_1	C_2	O	N
$\sigma(r)$ (Å)	0·025	0·027	0·027	0·033	0·023

Bond	C–Cl	C–C	C–O	C–N
δr (Å)	0·03	0·03	0·04	0·03

Discussion of structure and molecular dimensions

The dimensions of the molecule are shown in Fig. 3(a). The values in parentheses are those obtained by electron diffraction experiments by Kimura, Aoki & Kurita (1954) in which only the C–Cl and Cl $\cdots$ N lengths and the angle C_2–C_1–Cl were obtained by assuming other bond lengths and angles. It is, therefore, clear that the values obtained by X-rays are more reliable than those by electron diffraction. The largest discrepancy was found in the N–C_2 distance: 1·40 Å by X-rays and 1·32 Å by electron diffraction. This disagreement is probably due to the difference in the experimental conditions: the electron diffraction observations were made in the gaseous state, while the X-ray diffraction observations were made in the crystalline state.
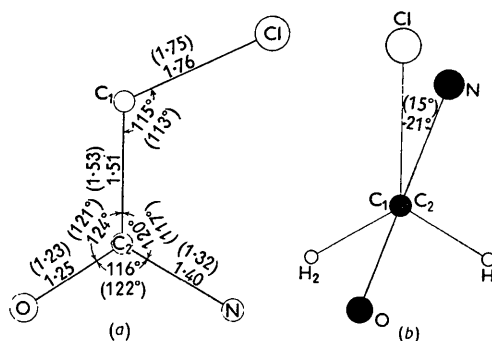
Fig. 3. (a) Bond lengths and angles of chloroacetamide. The values obtained by electron diffraction are written in parentheses. (b) The molecule viewed in the direction of the C_1–C_2 bond. The angle of twist from the cis form determined by electron diffraction is written in parenthesis.

The difference of molecular structures between the stable and unstable crystals is relatively large. In the stable crystals C_1–Cl is 1·81 Å and C_1–C_2 is 1·47 Å, values which differ considerably from those found in the unstable crystals. The dimensions of the amide group also show large difference (Table 5). Coincidence was found only in the angles C_2–C_1–Cl and C_1–C_2–N.

The C_1–Cl length (1·76 Å) is in satisfactory agreement with the accepted value in other molecules. The C_1–C_2 length (1·51 Å) is a little shorter than the standard single-bond value of 1·54 Å. A shorter distance, however, has frequently been found in crystals of amino acids and other related compounds. It would be reasonable for this molecule to have such a length because the conjugation effect of π-electrons in the adjacent C_2–O double bond would reduce the length of the C_1–C_2 bond. The bond angle C_2–C_1–Cl (115°) is larger than the tetrahedral angle because of the strong repulsive force between Cl and C_2 and other atoms. It may be said that the atoms C_1, C_2, O and N are in the same plane, since the deviation of the C_2 atom from the plane C_1–O–N is only about 0·02 Å.

The projection of the molecule along the C_1–C_2 bond is shown in Fig. 3(b). The angle of twist from the cis configuration is about 21°, 'cis' denoting the configuration in which C_1–Cl and C_2–N bonds take the cis position. This angle is in good agreement with the value of 15° obtained by electron diffraction, and the value of about 14° calculated from the atomic coordinates determined in the stable crystal. This indicates that the angle of twist is fixed mainly by the intra-molecular forces. Kimura et al. suggested that the hydrogen bond N–H $\cdots$ Cl makes this position stable. It should, however, be mentioned that we have no definite evidence of the existence of a hydrogen bond between N–H $\cdots$ Cl. Even if a hydrogen bond exists, it is not a simple linear one because it is clearly shown in the $(\varrho_o - \varrho_c)$ projection that the hydrogen atom deviates distinctly from the line of N $\cdots$ Cl.

The electrostatic force between the two large dipoles of the C=O and C–Cl bonds would bring the molecule

to the *cis* position, but the steric repulsion between the chlorine and nitrogen atoms would prevent it assuming the exact *cis* position. The net result would produce the angle of twist of 21° for the most stable configuration.

The bond lengths and angles of the terminal amide group of various compounds are listed in Table 5 from

Table 5. *Bond lengths and angles for amide group*

	C–O (Å)	C–N (Å)	C–C–O (°)	C–C–N (°)	O–C–N (°)
Chloroacetamide (unstable form)	1·25	1·40	124	120	116
Acetamide	1·28	1·38	129	109	122
Glycyl-ᴅ-asparagine	1·22	1·39	125	115	120
Succinamide	1·22	1·40	123	114	123
Oxamide	1·25	1·31	120	115	126
Nicotin amide	1·22	1·34	118	117	125
ʟ-Glutamine	1·27	1·28	118	118	123
6-Amido-3-pyridazone	1·25	1·33	119	118	123
Tetradecanamide	1·23	1·26	130	113	116
Chloroacetamide (stable form)	1·23	1·33	111	119	130

which we find an interesting correlation of the bond lengths with the bond angles. The first four molecules, which have larger C–N lengths of about 1·38 Å, have the angles C–C–O larger than the angles O–C–N and C–C–N, while the remaining molecules, which have shorter C–N lengths of about 1·30 Å, have the largest angles O–C–N, except in the case of tetradecanamide. The same relation was found by the author in carboxyl groups (to be published). These relations may be explained simply by the existence of repulsion between the non-bonded atoms. The change of the bond lengths of the amide group is due to the conjugation effect of π-electrons and the effect is smaller in the former group than in the latter. Three molecules of the latter six have conjugated groups in the neighbouring bond of the amide group. These have large resonance effect and, hence, the bond lengths and angles would be remarkably affected. The other three are ʟ-glutamine, tetradecanamide, and the stable form of chloroacetamide. The molecule of ʟ-glutamine has a very short C–C length of about 1·47 Å in the next neighbour of the amide group, and tetradecanamide has short C–C bonds and normal C–C bonds alternately. The molecule of chloroacetamide of the stable crystal has also a very short C–C length of 1·47 Å. Thus strong resonance effect is brought about in these molecules. This is the reason why the molecules in the latter part in Table 5 have shorter C–N lengths and the largest O–C–N angles. No relation is found between the structure of amide group and N–H ··· O hydrogen bond distances.

The packing of the molecules in the lattice is shown in Fig. 4. The distances Cl ··· Cl and Cl ··· C_2 between the neighbouring molecules are nearly equal to the sum of the van der Waals radii. The hydrogen bonds formed between amide groups are relatively
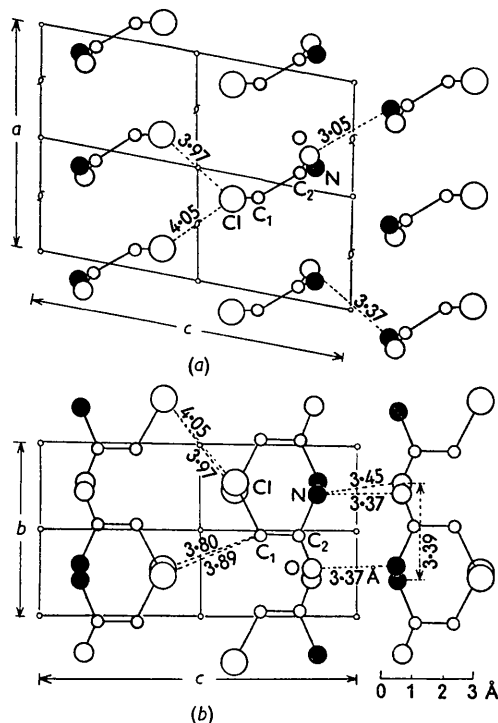

Fig. 4. A view of chloroacetamide structure (a) down the b axis, (b) down the a axis.

weak, as is easily seen from the N–H ··· O bond length of 3·05 Å and the other N ··· O lengths of 3·37 and 3·39 Å. The weakness of the hydrogen bonds may be the reason why the crystal sublimes easily.

It is very interesting to compare the packing arrangements of molecules in the stable and unstable crystals. In the latter the chains of molecules are formed in the direction of the b axis by weak hydrogen bonds (N–H ··· O length 3·05 Å). In the stable form these hydrogen bonds are broken and the atom pairs of longer N ··· O lengths (3·37 and 3·39 Å) in the unstable form become shorter (2·82 Å and 3·01 Å) to form new hydrogen bonds. This is an essential feature in the mechanism of the transformation from the unstable to the stable crystal.

References

AYERST, E. M. & DUKE, J. R. C. (1954). *Acta Cryst.* **7**, 588.

BOOTH, A. D. & BRITTEN, K. H. V. (1948). *Proc. Roy. Soc.* A, **197**, 241.

COCHRAN, W. & PENFOLD, B. R. (1952). *Acta Cryst.* **5**, 644.

CUCKA, P. & SMALL, R. W. H. (1954). *Acta Cryst.* **7**, 199.

DEJACE, J. (1955). *Acta Cryst.* **8**, 851.

GROTH, P. (1906–19). *Chemische Krystallographie.* Leipzig: Engelmann.

KIMURA, M., AOKI, M. & KURITA, Y. (1954). *Bull. Chem. Soc., Japan,* **27**, 360.

PASTERNAK, R. A. (1953). *Acta Cryst.* **6**, 808.

PASTERNAK, R. A., KATZ, L. & COREY, R. B. (1954). *Acta Cryst.* **7**, 225.

ROMERS, C. (1953). *Acta Cryst.* **6**, 429.

SENTI, F. & HARKER, D. (1940). *J. Amer. Chem. Soc.* **62**, 2008.

TURNER, J. D. & LINGAFELTER, E. C. (1955). *Acta Cryst.* **8**, 551.

WRIGHT, W. B. & KING, G. S. D. (1954). *Acta Cryst.* **7**, 283.

The Crystal Structure of Magnesium Phosphite Hexahydrate, MgHPO₃.6H₂O

BY D. E. C. CORBRIDGE

Research Department, Albright and Wilson Limited, Oldbury, Birmingham, England

The crystal structure of magnesium phosphite hexahydrate, $MgHPO_3.6H_2O$, has been determined by Fourier methods. The lattice is rhombohedral, with unit-cell dimensions $a_R = 5\cdot96$ Å, $\alpha = 96° 24'$. The space group is $R3$ and the Mg, P and H atoms lie on the unique axis. The structure is built from discrete tetrahedral $[HPO_3]^{2-}$ ions and octahedral $Mg^{2+}(H_2O)_6$ groups, which are linked together in three dimensions by a continuous system of hydrogen bonds. The crystals are isomorphous with those of magnesium sulphite hexahydrate, $MgSO_3.6H_2O$, which probably contain an isostructural arrangement.

Introduction

Phosphorous acid, H_3PO_3, is dibasic, and its crystalline salts are usually assumed to contain discrete tetrahedral $[HPO_3]^{2-}$ ions in which the hydrogen atom is linked directly to phosphorus. Evidence for this configuration is provided by infra-red spectra (Corbridge & Lowe, 1954) and Raman spectra (Simon & Fehér, 1937), but no detailed X-ray studies of crystalline phosphites have been reported. The existence of approximately tetrahedral $[H_2PO_2]^-$ ions in hypophosphites has been demonstrated in the crystal structures of ammonium hypophosphite $(NH_4)H_2PO_2$ (Zachariasen & Mooney, 1934) and magnesium hypophosphite $Mg(H_2PO_2)_2$ (Pedrazuela, Garcia-Blanco & Rivoir, 1953).

Unit cell and space group

Small, hemimorphic, trigonal pyramidal crystals were obtained by evaporation of an aqueous solution of the salt. The dimensions of the hexagonal unit cell, determined from rotation photographs, were

$$[a] = 8\cdot88, \quad [c] = 9\cdot10 \text{ Å} .$$

The corresponding rhombohedral constants are

$$[a_R] = 5\cdot96 \text{ Å}, \quad \alpha = 96° 24' .$$

The density, determined by flotation, was $1\cdot708$ g.cm.$^{-3}$, while that calculated for the hexagonal cell, assuming 3 units of $MgHPO_3.6H_2O$, was $1\cdot703$ g.cm.$^{-3}$.

The symmetry of $[c]$-axis Weissenberg and oscillation photographs indicated a rhombohedral lattice. Indexing of zero and 1st layer $[c]$- and $[a]$-axis Weissenberg photographs revealed that reflexions were absent for $-h+k+l = 3n$, and the intensities of spots on the $[c]$-axis zero-layer-line photograph were such that $I_{hki0} \neq I_{khi0}$. The crystals were found to be strongly pyroelectric and space group $R3$ was adopted.

Intensity data

Experimental intensity data were obtained from zero-layer-line $[c]$- and $[a]$-axis Weissenberg photographs taken with Cu $K\alpha$ radiation. A multiple-film technique employing six films for each layer line was used. The intensities were estimated visually, and Lorentz and polarization corrections were made in the usual way. Absorption effects were minimized by using small equant crystals $\sim 0\cdot3$ mm. The structure amplitudes were put on an absolute scale by comparison with the calculated values during later stages of the analysis. A total of 180 different spots were estimated, giving a final list of 74 non-equivalent $\{hki0\}$- and $\{0kil\}$-type reflexions.

Structure determination

In space group $R3$ the special and general positions in the hexagonal cell are: