

her encouragement. They also wish to thank Dr Carol Huber and Dr S. Natarajan for helpful discussions.

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## The Crystal Structure of Ammonium Hydrogen Terephthalate

BY R. E. COBBLEDICK AND R. W. H. SMALL

*Chemistry Department, The University, Lancaster, England*

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The crystal structure of ammonium hydrogen terephthalate,  $\text{NH}_4^+\text{C}_6\text{H}_4\text{COOHCOO}^-$ , has been determined using three-dimensional X-ray data measured with a proportional counter. The space group is  $C2/c$  with four molecules in the monoclinic unit cell. Positional and anisotropic thermal vibrational parameters have been refined by the method of least-squares giving a final conventional  $R$  value of 0.076. The crystal structure consists of chains of centrosymmetric terephthalate ions linked by short hydrogen bonds across centres of symmetry ( $\text{O}\cdots\text{O}$  distance of 2.51 Å) and these chains are linked *via* the ammonium ions to form a three-dimensional hydrogen-bonding network. The carboxyl groups are twisted  $9^\circ$  out of the plane of the benzene ring to form hydrogen bonds with the ammonium ions. Bond lengths for the terephthalate ion have been corrected for librational motion and are C–C (carboxylate) =  $1.495 \pm 0.004$ , C–O =  $1.237 \pm 0.004$ , C–O =  $1.310 \pm 0.004$ , C–C =  $1.386 \pm 0.005$ , C–C =  $1.409 \pm 0.005$  and C–C =  $1.374 \pm 0.005$  Å.

### Experimental

A sample of ammonium hydrogen terephthalate was prepared by the dropwise addition of aqueous ammonia to a solution of terephthaloyl chloride in pyridine. Plate-shaped crystals were grown by recrystallization from an aqueous solution of dimethylformamide. Composition of the crystals was confirmed by quantitative elemental analysis and mass spectrometry.

Weissenberg photographs showed the crystals to be monoclinic and the systematically absent reflexions,  $hkl$  for  $(h+k)=2n+1$ ,  $h0l$  for  $l=2n+1$ , indicated the space group to be  $Cc$  or  $C2/c$ . The space group was later assumed to be  $C2/c$  on the evidence of a centric distribution of the three-dimensional intensities (Howells, Phillips & Rogers, 1950). Cell dimensions were determined by a least-squares fit to  $\theta$  values of a number of reflexions measured on a three-circle diffractometer (Small & Travers, 1961) using Cu  $K\alpha$  radiation ( $\lambda=1.5418$  Å). The density was measured by flotation

using a mixture of chlorobenzene and carbon tetrachloride. Crystal data are shown in Table 1.

Table 1. *Crystal data*

$\text{C}_8\text{H}_9\text{NO}_4$	
$a$	$18.913 \pm 0.004$ Å
$b$	$3.801 \pm 0.001$
$c$	$11.460 \pm 0.002$
$\beta$	$97.44 \pm 0.04^\circ$
$V$	$816.99$ Å <sup>3</sup>
$Z$	4
$D_{\text{obs}}$	$1.472$ g.cm <sup>-3</sup>
$D_{\text{calc}}$	1.489

### Determination and refinement of the structure

A total of 891 integrated intensities ( $833 > 1\sigma$ ) with  $\sin \theta/\lambda < 0.642$  Å<sup>-1</sup> were measured using the three-circle diffractometer. Corrections were applied to the intensities for the absorption of X-rays using the program *ABSCOR* on the Chilton ATLAS computer.

The space group was assumed to be  $C2/c$  on the basis of the centric distribution of the intensities. With four molecules in the unit cell this requires the ions to lie in special positions; for space group  $C2/c$  they are either centres of symmetry or twofold axes. An ammonium ion is not centrosymmetric and thus the nitrogen atom of the ammonium ion must lie on a twofold axis.

Two-dimensional projections of the Patterson function on to (010) and (001) were used to solve the structure. Coordinates of the non-hydrogen atoms were refined initially by two-dimensional Fourier and least-squares methods and finally by full-matrix least-squares to give an  $R$  value ( $R = \sum ||F_{\text{obs}}| - |F_{\text{calc}}|| / \sum |F_{\text{obs}}|$ ) of 0.110. A three-dimensional difference synthesis located the hydrogen atoms close to the expected positions. The hydrogen atoms were given the anisotropic temperature parameters of the atoms to which they were bonded and included in the structure model for two cycles of least-squares refinement. In a last cycle of refinement the hydrogen atom positions were refined. For the last three cycles of least-squares calculations a weighting scheme was applied with  $w^{1/2} = 0.43/(J)$  where ( $J$ ) takes the maximum value of the three functions  $0.008 |F_o| - 0.15, \sigma$  and  $0.43$  for a particular reflexion, where  $\sigma$  is the standard deviation of the structure amplitude based on counting statistics. The final  $R$  value was 0.0759, with the parameter shifts of the heavy atoms being less than one third of their standard deviations. The atomic scattering factors used throughout were those listed in *International Tables for X-ray Crystallography* (1962).

Table 2. Fractional atomic coordinates

The values of the fractional coordinates and the estimated standard deviations (in parentheses) are multiplied by  $10^4$ .

	$x$	$y$	$z$
C(1)	1078 (1)	8816 (8)	5529 (2)
C(2)	1810 (1)	8122 (7)	5229 (2)
C(3)	1997 (1)	8868 (8)	4132 (2)
C(4)	2320 (1)	6726 (8)	6101 (2)
O(1)	945 (1)	8594 (7)	6549 (2)
O(2)	599 (1)	9680 (7)	4655 (2)
N	0 (0)	5839 (14)	2500 (0)
H(1)	231 (22)	7127 (127)	3082 (34)
H(2)	338 (22)	4348 (120)	2126 (31)
H(3)	1630 (17)	198 (96)	3541 (27)
H(4)	2153 (17)	6179 (95)	6869 (29)
H(5)	0 (0)	0 (0)	5000 (0)

Table 3. Thermal vibrational parameters

The values of the vibrational parameters and the estimated standard deviations (in parentheses) are multiplied by  $10^4$ . The temperature factor expression used is of the form  $\exp \{-(b_{11}h^2 + b_{22}k^2 + b_{33}l^2 + 2b_{12}hk + 2b_{13}hl + 2b_{23}kl)\}$ .

	$b_{11}$	$b_{22}$	$b_{33}$	$b_{12}$	$b_{13}$	$b_{23}$
C(1)	17 (1)	473 (23)	61 (2)	-16 (4)	4 (1)	-33 (6)
C(2)	14 (1)	334 (19)	50 (2)	2 (3)	0 (1)	-11 (5)
C(3)	17 (1)	438 (21)	48 (2)	6 (4)	-1 (1)	-1 (6)
C(4)	19 (1)	433 (22)	42 (2)	-3 (4)	0 (1)	2 (6)
O(1)	19 (1)	973 (24)	59 (2)	7 (3)	10 (1)	0 (5)
O(2)	16 (1)	1097 (26)	60 (2)	39 (3)	1 (1)	-4 (6)
N	38 (2)	680 (40)	70 (3)	0 (0)	16 (2)	0 (0)

Table 4. Observed and calculated structure amplitudes  $\times 10^2$ 

h	k	l	Observed	Calculated
0	0	0	100	100
0	0	1	100	100
0	0	2	100	100
0	0	3	100	100
0	0	4	100	100
0	0	5	100	100
0	0	6	100	100
0	0	7	100	100
0	0	8	100	100
0	0	9	100	100
0	0	10	100	100
0	0	11	100	100
0	0	12	100	100
0	0	13	100	100
0	0	14	100	100
0	0	15	100	100
0	0	16	100	100
0	0	17	100	100
0	0	18	100	100
0	0	19	100	100
0	0	20	100	100
0	0	21	100	100
0	0	22	100	100
0	0	23	100	100
0	0	24	100	100
0	0	25	100	100
0	0	26	100	100
0	0	27	100	100
0	0	28	100	100
0	0	29	100	100
0	0	30	100	100
0	0	31	100	100
0	0	32	100	100
0	0	33	100	100
0	0	34	100	100
0	0	35	100	100
0	0	36	100	100
0	0	37	100	100
0	0	38	100	100
0	0	39	100	100
0	0	40	100	100
0	0	41	100	100
0	0	42	100	100
0	0	43	100	100
0	0	44	100	100
0	0	45	100	100
0	0	46	100	100
0	0	47	100	100
0	0	48	100	100
0	0	49	100	100
0	0	50	100	100
0	0	51	100	100
0	0	52	100	100
0	0	53	100	100
0	0	54	100	100
0	0	55	100	100
0	0	56	100	100
0	0	57	100	100
0	0	58	100	100
0	0	59	100	100
0	0	60	100	100
0	0	61	100	100
0	0	62	100	100
0	0	63	100	100
0	0	64	100	100
0	0	65	100	100
0	0	66	100	100
0	0	67	100	100
0	0	68	100	100
0	0	69	100	100
0	0	70	100	100
0	0	71	100	100
0	0	72	100	100
0	0	73	100	100
0	0	74	100	100
0	0	75	100	100
0	0	76	100	100
0	0	77	100	100
0	0	78	100	100
0	0	79	100	100
0	0	80	100	100
0	0	81	100	100
0	0	82	100	100
0	0	83	100	100
0	0	84	100	100
0	0	85	100	100
0	0	86	100	100
0	0	87	100	100
0	0	88	100	100
0	0	89	100	100
0	0	90	100	100
0	0	91	100	100
0	0	92	100	100
0	0	93	100	100
0	0	94	100	100
0	0	95	100	100
0	0	96	100	100
0	0	97	100	100
0	0	98	100	100
0	0	99	100	100
0	0	100	100	100

The final positional parameters are shown in Table 2 and the vibrational parameters of the C, N and O atoms in Table 3. Observed and calculated structure amplitudes are listed in Table 4.

### Analysis of the anisotropic thermal motion

The centre of mass of the terephthalate ion lies on a centre of symmetry and thus the rigid-body analysis method of Cruickshank (1956) in which translational and rotational displacements are represented in terms of two symmetric tensors  $T$  and  $L$  is sufficient. The thermal motion analysis program *MGTLS* of Gantzel and Trueblood was used to test the validity of rigid-

body motion of the ion as a whole. Rigid-body thermal parameters for the ion are shown in Table 5 and Table 6 gives the  $U_{ij}$  values calculated from these parameters and the 'observed' values obtained from the  $b_{ij}$  values

Table 5. Rigid-body thermal parameters referred to orthogonal axes  $a, b, c^*$

Estimated standard deviations of the elements of the T and L tensors are given in parentheses.

$$\mathbf{T} = \begin{pmatrix} 274 (9) & -47 (10) & -39 (9) \\ & 118 (17) & -47 (13) \\ & & 278 (12) \end{pmatrix} \times 10^{-4} \text{ \AA}^2$$

$$\mathbf{L} = \begin{pmatrix} 148 (13) & -29 (4) & -30 (3) \\ & 17 (2) & 6 (2) \\ & & 45 (2) \end{pmatrix} \times 10^{-4} \text{ rad}^2$$

Principal axes of T r.m.s. amplitude (Å)	Direction cosines ( $\times 10^3$ )		
0.177	-685	-11	728
0.163	-668	408	-622
0.094	-290	-913	-287

Principal axes of L r.m.s. amplitude (rad)	Direction cosines ( $\times 10^3$ )		
0.127	-946	192	262
0.061	-247	67	-967
0.033	-208	-978	-14

of the final least-squares refinement cycle. The satisfactory agreement between the observed and calculated  $U_{ij}$  values and comparison of the r.m.s.  $\Delta U_{ij}$  of  $0.0027 \text{ \AA}^2$  calculated from the rigid body model with the mean  $\sigma U_{ij}$  of  $0.0014 \text{ \AA}^2$  calculated from the estimated standard deviations of the  $b_{ij}$  values is evidence of the validity of the analysis. The atomic coordinates were corrected for the librational motion of the ion. Uncorrected and corrected bond lengths and angles are given in Tables 7 and 8.

Table 7. Bond lengths and their standard deviations

	Before libration correction	After libration correction
C(1)-C(2)	1.493 (4) Å	1.495 (4) Å
C(1)-O(1)	1.230 (3)	1.237 (4)
C(1)-O(2)	1.303 (3)	1.310 (4)
C(2)-C(3)	1.379 (4)	1.386 (5)
C(2)-C(4)	1.400 (4)	1.409 (5)
C(3)-C(4')	1.371 (5)	1.374 (5)
C(3)-H(3)	1.04 (3)	
C(4)-H(4)	1.00 (3)	
O(2)-H(5)	1.25 (-)	
N-H(1)	0.89 (4)	
N-H(2)	0.99 (4)	

Table 6. Observed and calculated values of ( $U_{ij} \times 10^4$ ) in  $\text{Å}^2$

	$U_{11}$		$U_{22}$		$U_{33}$		$U_{12}$		$U_{13}$		$U_{23}$	
	obs	calc	obs	calc	obs	calc	obs	calc	obs	calc	obs	calc
C(1)	295	288	346	414	398	365	-48	7	-4	20	-71	-48
C(2)	261	277	245	188	324	298	11	-34	-43	-34	-24	-48
C(3)	311	309	321	342	317	305	23	21	-53	-32	-3	14
C(4)	351	309	317	323	276	303	-12	-1	-32	-14	4	19
O(1)	315	326	712	687	383	398	27	46	61	32	0	-16
O(2)	288	312	803	789	394	423	144	108	-46	-49	-9	-25

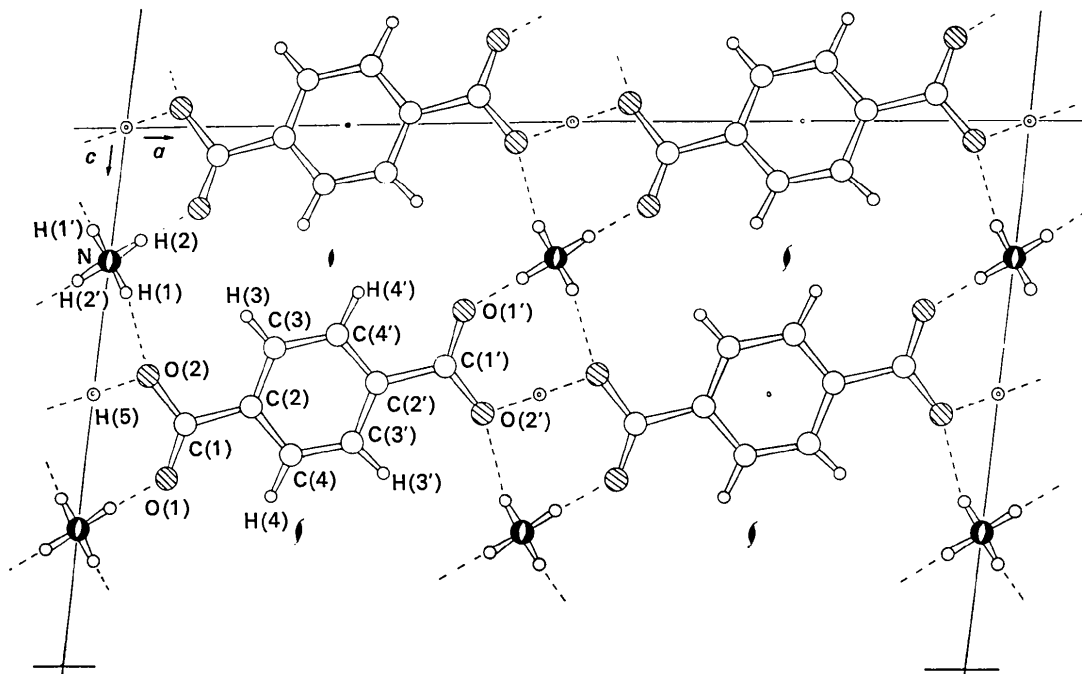


Fig. 1. The crystal structure projected on to (010).

Table 8. Bond angles and their standard deviations

	Before libration correction	After libration correction
C(2)-C(1)-O(1)	121.1 (6)°	120.9 (6)°
C(2)-C(1)-O(2)	116.2 (5)	116.1 (5)
O(1)-C(1)-O(2)	122.7 (6)	123.0 (6)
C(1)-C(2)-C(3)	121.9 (5)	121.8 (5)
C(1)-C(2)-C(4)	118.5 (5)	118.3 (5)
C(3)-C(2)-C(4)	119.6 (6)	119.9 (6)
C(2)-C(4)-C(3')	120.2 (7)	120.1 (7)
C(2)-C(3)-C(4')	120.2 (7)	120.1 (7)
C(2)-C(3)-H(3)	118 (2)	
C(2)-C(4)-H(4)	116 (2)	
H(3)-C(3)-C(4')	121 (2)	
H(4)-C(4)-C(3')	124 (2)	
H(1)-N—H(2)	111 (4)	
H(1)-N—H(1')	113 (4)	
H(2)-N—H(2')	110 (4)	
H(2)-N—H(1')	106 (4)	

### Discussion of the crystal and molecular structure

#### (a) Arrangement of the ions

Because of the space-group symmetry the ions lie in special positions. The nitrogen atom of the ammonium ion lies on a twofold axis, the acidic hydrogen atom lies on a centre of symmetry while the centre of mass of the terephthalate ion is on a centre of symmetry so that the ion is centrosymmetric.

The centrosymmetric terephthalate ions are linked into chains by short hydrogen bonds across further centres of symmetry. The hydrogen bonds are crystallographically symmetric and the O...O distance is 2.51 Å. The nitrogen atom of the ammonium ion on the twofold axis is closely surrounded by six oxygen atoms of which the four closest, from four different terephthalate ions, are linked by hydrogen bonds. These hydrogen bonds with the ammonium ions link the chains of terephthalate ions to form a complete three-dimensional network. The four shortest N...O contacts are two each of 2.77 and 2.95 Å, and the two non-hydrogen bonding contacts are each 3.05 Å. Other hydrogen-bonding geometry is given in a later section.

Fig. 1 shows a projection of the structure on to (010).

#### (b) The terephthalate ion

The benzene ring is planar within the limits of the experimental error and the plane through the carboxyl group makes an angle of 9° with this plane. The equations of these planes referred to the orthogonal axes  $a$ ,  $b$ ,  $c^*$  and the deviations of atoms from them are shown in Table 9. The two C-O bond lengths are C(1)-O(1) = 1.237 Å and C(1)-O(2) = 1.310 Å while the C-C-O angles are C(2)-C(1)-O(1) = 120.9° and C(2)-C(1)-O(2) = 116.1°. Hahn (1957) has suggested two equal C-O bond lengths of 1.260 Å and two equal C-C-O angles of 117.3° for a completely ionized carboxyl group, and bond lengths differing by at least 0.1 Å and angles by at least 9° for an un-ionized carboxyl group. The C-O lengths and C-C-O angles in ammonium hydrogen terephthalate are intermediate between these two extremes. Similar intermediate

values have been found in a number of acid salts, particularly by Speakman (1967), who has classified acid salt structures into two general types: type *A* characterized by the hydrogen bonding of two acidic radicals related by a symmetry element, and type *B* in which the acidic radicals are not crystallographically equivalent.

Table 9. Deviations of atoms from the least-squares planes

(a) Plane through the benzene ring carbon atoms  
Equation of the plane, referred to orthogonal axes  $a, b, c^*$  is  
 $0.2215x + 0.9261y + 0.3054z = 5.2585$ .

C(2)	0.002 Å	C(1)	0.034 Å
C(3)	-0.002	O(1)	0.220
C(4)	-0.002	O(2)	-0.137
H(3)	0.127	H(5)	-0.167
H(4)	-0.024		

(b) Plane through atoms C(1), C(2), O(1) and O(2)

Equation of the plane referred to orthogonal axes  $a, b, c^*$  is  
 $0.2155x + 0.9653y + 0.1478z = 4.4279$ .

C(1)	-0.002 Å	O(2)	0.001 Å
C(2)	0.001	H(5)	-0.079
O(1)	0.001		

#### (c) The ammonium ion

The ammonium ion has its hydrogen atoms approximately directed to the corners of a regular tetrahedron and these hydrogen bond to four different terephthalate ions to give a three-dimensional hydrogen-bonding network. The H-N-H angles range from 106 to 113° and the mean angle is 109.5°.

#### (d) The hydrogen bonding

The hydrogen bonds linking the terephthalate ions end-to-end to form chains are crystallographically symmetric and the O...O distance is 2.51 Å. Hydrogen bonds with the ammonium ions link these chains. The geometry of the hydrogen bonds is summarized in Table 10.

Table 10. Hydrogen-bonding geometry

#### (a) Distances

N-H(2)	0.99 Å	N-H(1)	0.89 Å
N...O(1)	2.78	N...O(2)	2.96
H(2)...O(1)	1.79	H(1)...O(2)	2.09
O(2)...H(5)	1.25		
O(2)...O(2')	2.51		

#### (b) Angles

C(1)-O(1)...N	121.9°	C(1)-O(2)...N	132.0°
N-H(2)...O(1)	175.5	N-H(1)...O(2)	167.2
C(1)-O(2)...O(2')	110.4	O(2)-H(5)...O(2')	180.0

Although the oxygen atoms of the short hydrogen bonds are related crystallographically by a centre of symmetry and require the acidic hydrogen atom to lie on the centre of symmetry, the hydrogen bond may not be truly symmetrical. Other possibilities such as statistical disorder or the oscillation of the proton be-

tween potential energy minima cannot be ruled out by the use of X-ray diffraction.

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## The Crystal and Molecular Structure of Thiamine Chloride Monohydrate

BY J. PLETCHER, M. SAX, S. SENGUPTA, J. CHU AND C. S. YOO

*Biocrystallography Laboratory, Veterans Administration Hospital, Pittsburgh, Pennsylvania 15240 and the Department of Crystallography, University of Pittsburgh, Pittsburgh, Pennsylvania 15213, U.S.A.*

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The crystal structure of thiamine chloride monohydrate ( $C_{12}H_{17}ClN_4OS \cdot H_2O$ ) has been determined from three-dimensional diffractometer data. The crystals are monoclinic, space group  $P2_1/c$ , with four molecules in the unit cell;  $a = 8.914$  (3),  $b = 16.775$  (3),  $c = 10.405$  (1) Å,  $\beta = 94.28$  (2)°. The structure was refined by the full-matrix least-squares technique to a final  $R$  value of 0.033 for the 2368 observed reflections. The conformation found for the free base of thiamine is very similar to that which is observed for the protonated form. The absence of the proton on the pyrimidine ring reduces the latter's 'quinoidal' character, but the bond to the amino group, although longer in the unprotonated compound, still exhibits considerable double-bond character.

### Introduction

The pyrophosphate ester of thiamine is a coenzyme for a number of different enzyme systems. Since thiamine alone catalyzes many of the same reactions as the enzyme systems, we have been studying thiamine and its derivatives in an effort to correlate its structural features with its chemical properties. The present structure, which contains the free base of thiamine, is the only one studied in which the pyrimidine ring is not protonated.

### Experimental

The free base of thiamine was prepared by titrating a 2M solution of thiamine chloride hydrochloride (Eastman Organic Chemicals) with sodium hydroxide to pH 8. The compound was crystallized by adding an equal volume of acetone to the resulting 1M solution and then maintaining it at approximately  $-15^\circ\text{C}$ . Colorless, tabular crystals grew slowly over several days' time. The space group was determined from Weissenberg photographs. The unit-cell parameters were determined from the axial reflections using a Picker four-circle diffractometer with Cu  $K\alpha$  radiation. The  $\alpha_1$  peak was used whenever the  $\alpha_1\alpha_2$  doublet was resolved. The e.s.d.'s are the r.m.s. deviations for the various determinations of the cell parameters. The crystal data are summarized in Table 1.

Table 1. *Crystal data*

Thiamine chloride monohydrate	$C_{12}H_{17}ClN_4OS \cdot H_2O$
M.W. 318.83	Data measured at ambient temperature, $22^\circ\text{C}$
$a = 8.914$ (3) Å	$\lambda(\text{Cu } K\alpha_1\alpha_2) = 1.54178$ Å
$b = 16.775$ (3)	$\lambda(\text{Cu } K\alpha_1) = 1.54051$
$c = 10.405$ (1)	$Z = 4$
$\beta = 94.28$ (2)°	Systematic absences $h0l$ $l = 2n + 1$
$V = 1551.55$ Å <sup>3</sup>	$0k0$ $k = 2n + 1$
Space group $P2_1/c$	
$F(000) = 672$	
$\mu(\text{Cu } K\alpha) = 34.47$ cm <sup>-1</sup>	
$\rho_o = 1.361$ g.cm <sup>-3</sup> (by flotation in benzene- $\text{CCl}_4$ )	
$\rho_c = 1.365$	
M.p. $194\text{--}196^\circ\text{C}$ unc. (Thermolyne melting point apparatus)	
begins losing water of hydration at $\sim 110^\circ\text{C}$	

Coordinates of crystal corners ( $\times 10^5$ ) in cm in reference coordinate system:

$X$  along  $-c^*$ ,  $Y$  along  $a$ ,  $Z$  along  $-b^*$ .

$X$	$Y$	$Z$	$X$	$Y$	$Z$
1453	108	2540	2830	734	318
-922	-68	2540	2102	679	1497
-1572	404	1497	-1572	-640	1497
-2830	312	-527	-2830	-734	-527
-2217	357	-1497	-2217	-688	-1497
-1568	-117	-2540	1696	-396	-1497
1045	78	-2540	2830	-312	318
1696	648	-1497	2102	-366	1497

The intensity data were collected (Shiono, 1968a) from a crystal that was mounted about the  $b$  axis