

The Crystal and Molecular Structure of *anti*- α -Bromoacetophenone Oxime

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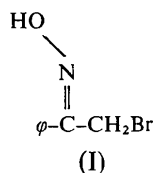
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The structure of *anti*- α -bromoacetophenone oxime (C_8H_8NOBr) was solved by Fourier methods and refined by full-matrix least-squares analysis to a final $R_1=0.046$; space group: $I\bar{4}$, $Z=8$. The distances and angles are similar to those observed in other oxime structures. The phenyl group and the oxime function are not coplanar, the angle between their normals being 54.3° . Molecules are linked into tetramers by $O-H \cdots N$ hydrogen bonds about an S_4 axis at $\frac{1}{2}, 0, z$, with both the oxygens and nitrogens assuming nearly tetrahedral arrangements. A cluster of four bromine atoms make van der Waals contacts of 3.681 \AA about an S_4 axis at $0, 0, z$.

Introduction

In 1901 Korten & Scholl (1901) reported synthesis of α -bromoacetophenone oxime by reaction of α -bromoacetophenone with hydroxylamine hydrochloride in aqueous methanol. They made the assignment of *anti* (I)* to the isomer of m.p. 92° based on the then current misconception that the Beckmann rearrangement occurred by *syn* migration. Fischer & Grob (1962) repeated the synthesis of Korten & Scholl and confirmed their assignment of *anti* based on u.v. spectra and complexing reactions of derivatives of (I).



However, Masaki, Fukui & Ohta (1967) and Blumbergs, Thanawalla, Ash, Lieske & Steinberg (1971) found the synthesis of Korten & Scholl produced not (I), but a mixture of α -bromo- and α -chloroacetophenone oxime, consisting principally of the *syn* isomers (Smith, Heidema, Kaiser, Wetherington & Moncrief, 1972).

Synthesis of the previously unknown *anti* isomer *via* solvolysis of *syn*- α -bromoacetophenone oxime to *anti*- α -morpholinoacetophenone oxime and subsequent conversion to (I) has been reported (Smith *et al.*, 1972). In light of past misassignments of the stereochemistry of this oxime, an X-ray structure determination was undertaken. A preliminary report was published with the synthesis (Smith *et al.*, 1972).

Structure determination

Crystals of (I), obtained by recrystallization from $CHCl_3$ /petroleum spirit,† were found to be tetragonal with

* *anti* refers to the isomer having the alkyl group *trans* to the oxime oxygen.

† Crystals of (I) were supplied by E. T. Kaiser, Searle Chemistry Laboratory, University of Chicago.

cell dimensions $a=12.056$ (13), $c=12.109$ (23) \AA ; Laue group $4/m$. An experimental density of 1.63 (3) g cm^{-3} indicated the space group to be $I4$ or $I\bar{4}$ with 8 molecules per unit cell. Physical and crystallographic data are summarized in Table 1. The absence of power satellites in the Harker section of the Patterson map subsequently identified the space group as $I\bar{4}$ (Buerger, 1959).

Table 1. Summary of physical and crystallographic data

C_8H_8NOBr	
Melting point	114.0-114.5°C
Space group	$I\bar{4}$
a	12.056 (13) \AA
c	12.109 (23) \AA
Density (obs)	1.63 (3) g cm^{-3}
Density (calc)	1.62
Z	8
Reflections:	
$F^2 > 2\sigma$	808
total	853
R_1 ($F^2 > 2\sigma$)	4.6%
R_1 (all reflections)	5.9%

Data were taken on three crystals with an automated Supper-Pace diffractometer and $Cu K\alpha$ radiation ($\lambda=1.5418 \text{ \AA}$), corrected for Lorentz and polarization effects, and correlated to give 853 independent reflections, 808 of which were above 2σ .‡

The position of the bromine atom was obtained from the Patterson map and used as a phasing model for the calculation of an electron-density map which revealed the positions of the ten remaining non-hydrogen atoms.

Beginning with $R_1=0.21$, the heavy-atom parameters were refined by full-matrix least-squares methods first isotropically to $R_1=0.091$, then anisotropically to $R_1=0.050$ with the weighting scheme $w=1/\sigma^2$. A difference-Fourier map was calculated and peaks cor-

‡ $\sigma(I)=[(0.05I)^2 + N_0 + K^2 N_b]^{1/2}$, N_0 =gross count, N_b =background count, K =scan time/background time (Parker, Flynn & Boer, 1968).

Table 2. Final atomic positions and anisotropic temperature factors for anti- α -bromoacetophenone oxime
$$T = \exp -(\beta_{11}h^2 + \beta_{22}k^2 + \beta_{33}l^2 + \beta_{12}hk + \beta_{13}hl + \beta_{23}kl)$$

	x/a	y/a	z/c	β_{11}	β_{22}	β_{33}	β_{12}	β_{13}	β_{23}
Br	0.4586 (1)	1.2989 (1)	0.5469 (1)	91 (1)	73 (1)	105 (1)	-23 (1)	30 (1)	10 (1)
C(2)	0.3228 (6)	1.2313 (6)	0.6045 (7)	64 (5)	60 (5)	88 (6)	-16 (7)	22 (9)	-32 (9)
C(3)	0.3223 (5)	1.1094 (5)	0.5800 (5)	53 (4)	55 (4)	56 (5)	-8 (7)	2 (7)	-3 (7)
N	0.3600 (5)	1.0502 (5)	0.6612 (5)	71 (4)	69 (4)	59 (4)	-12 (6)	-26 (7)	-4 (7)
O	0.3603 (5)	0.9365 (4)	0.6410 (5)	104 (4)	62 (4)	74 (4)	-3 (6)	-52 (8)	32 (6)
C(6)	0.2806 (5)	1.0670 (5)	0.4724 (5)	50 (4)	45 (4)	57 (5)	4 (6)	-3 (7)	23 (7)
C(7)	0.1798 (6)	1.1091 (6)	0.4331 (8)	61 (5)	64 (5)	84 (7)	15 (8)	-12 (9)	18 (9)
C(8)	0.1365 (7)	1.0711 (7)	0.3356 (8)	90 (6)	97 (7)	85 (7)	-14 (11)	-75 (12)	36 (12)
C(9)	0.1921 (9)	0.9914 (7)	0.2745 (7)	127 (9)	92 (7)	65 (6)	-61 (13)	-37 (13)	19 (10)
C(10)	0.2926 (8)	0.9517 (7)	0.3117 (7)	107 (7)	71 (5)	71 (6)	-45 (10)	57 (12)	-23 (9)
C(11)	0.3366 (6)	0.9871 (6)	0.4116 (7)	63 (5)	57 (5)	74 (6)	-17 (8)	29 (8)	2 (8)

Table 2 (cont.)

H(12)	0.2422	1.2545	0.5956
H(13)	0.3056	1.2504	0.6895
H(14)	0.1367	1.1795	0.4660
H(15)	0.0655	1.0966	0.3047
H(16) [†]	0.1500	0.9610	0.1990
H(17)	0.3316	0.9067	0.2667
H(18)	0.4248	0.9667	0.4304
H(19)	0.3943	0.8942	0.6913

[†] Calculated position.

responding to expected hydrogen positions were found for all but one of the eight hydrogens, H(16).

Several of the hydrogen peaks were of low residual electron density. Attempts were made to improve the hydrogen location by refinement of the parameters of those hydrogens most clearly defined in the difference maps, and by exclusion of certain data as a function

of $\sin \theta/\lambda$ (La Placa & Ibers 1965). However, these procedures did not significantly improve the appearance of the difference maps. The final hydrogen coordinates reported are those from the original difference Fourier with the exception of H(12), H(13), H(14), and H(15) which were refined isotropically and of H(16) which were calculated.

Two cycles of refinement of the heavy-atom parameters with the hydrogens included but not varied resulted in a final $R_1 = 0.046$ ($R_2 = 0.059$ for all accessible data). The final atomic coordinates and temperature factors are listed in Table 2. A final difference-Fourier map contained no residual electron density above $0.15 \text{ e } \text{ \AA}^{-3}$ except in the region about the point 0,0,0 where there is a cluster of four closely packed bromine atoms (see Fig. 2). A listing of the observed and calculated structure factors appears in Table 3.

Discussion

The observed bond distances and angles and their estimated standard errors are shown in Table 4. Fig. 1 is an ORTEP II (Johnson, 1964) drawing of (I) based on 50% probability thermal ellipsoids. As indicated by Fig. 1, the assignment of *anti* to this α -bromoacetophenone oxime was confirmed. The Br-C(2) distance (1.957 Å) is somewhat longer than the average value of 1.94 Å reported by Sutton (1965). The difficulty with the location of the hydrogens mentioned previously is reflected in the distances and angles associated with them. The H(12)-C(2)-H(13) angle of 82° found in the alkyl group is unreasonable despite the presence of the bromine atom. A distorted methyl group (H-C-H angle of 88°) was reported by Hamilton (1961) in a neutron diffraction study of dimethyl glyoxime; we do not, however, attach any significance to this similarity.

In general, distances and angles found in (I) are similar to those found in other oximes. Table 5 presents for comparison selected bond distances and angles from several oxime structure determinations. Both the C(3)-N and N-O distances in the present structure are intermediate in value. The N-C(3)-C(6) angle (126.4°) is widened at the expense of the N-C(3)-C(2) angle

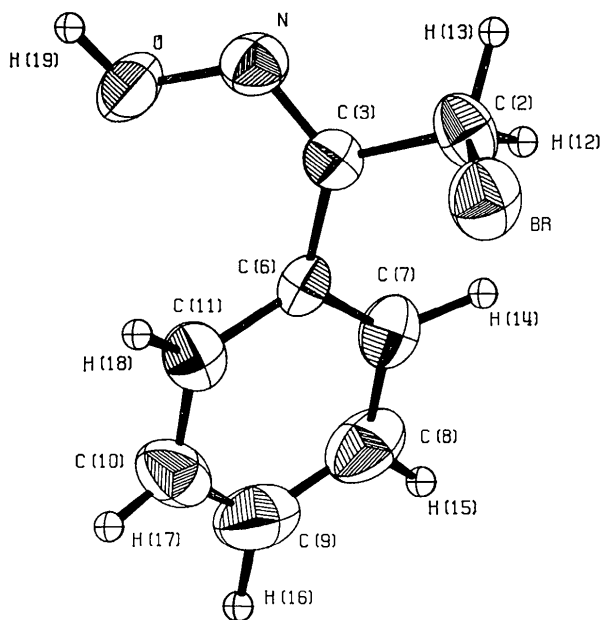


Fig. 1. *anti*- α -Bromoacetophenone oxime. Thermal ellipsoids represent 50% probability.

Table 4. Bond distances and angles and their estimated standard errors for anti- α -bromoacetophenone oxime

Br—C(2)	1.957 (7) Å	Br—C(2)—C(3)	109.9 (5)°
C(2)—C(3)	1.499 (9)	C(2)—C(3)—N	112.8 (6)
C(3)—N	1.297 (9)	C(2)—C(3)—C(6)	120.8 (6)
C(3)—C(6)	1.487 (9)	N—C(3)—C(6)	126.4 (6)
N—O	1.392 (8)	C(3)—N—O	114.2 (6)
C(6)—C(7)	1.401 (9)	C(3)—C(6)—C(7)	117.8 (6)
C(6)—C(11)	1.388 (8)	C(3)—C(6)—C(11)	122.6 (6)
C(7)—C(8)	1.370 (13)	C(7)—C(6)—C(11)	119.6 (6)
C(8)—C(9)	1.387 (13)	C(6)—C(7)—C(8)	120.1 (7)
C(9)—C(10)	1.379 (14)	C(7)—C(8)—C(9)	120.5 (8)
C(10)—C(11)	1.388 (11)	C(8)—C(9)—C(10)	119.5 (8)
C(2)—H(12)	1.017	C(9)—C(10)—C(11)	120.9 (8)
C(2)—H(13)	1.075	C(6)—C(11)—C(10)	119.3 (7)
C(7)—H(14)	1.072	Br—C(2)—H(12)	130.3
C(8)—H(15)	0.983	C(3)—C(2)—H(12)	104.2
*C(9)—H(16)	1.081	Br—C(2)—H(13)	114.4
C(10)—H(17)	0.901	C(3)—C(2)—H(13)	113.5
C(11)—H(18)	1.114	H(12)—C(2)—H(13)	81.8
O—H(19)	0.894	C(6)—C(7)—H(14)	125.5
		C(8)—C(7)—H(14)	113.6
		C(7)—C(8)—H(15)	123.7
		C(9)—C(8)—H(15)	115.8
		*C(8)—C(9)—H(16)	119.0
		*C(10)—C(9)—H(16)	121.5
		C(9)—C(10)—H(17)	118.0
		C(11)—C(10)—H(17)	120.9
		C(6)—C(11)—H(18)	120.6
		C(10)—C(11)—H(18)	118.3
		N—O—H(19)	116.4

* H(16) is a calculated position.

Table 6. Equations of least-squares planes and atomic displacements

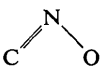
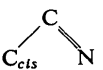
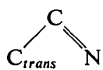
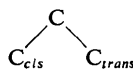
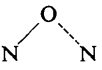
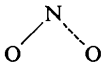
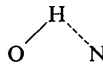
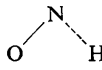
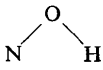
$$\alpha: 0.9224X + 0.0696Y - 0.3798Z = 1.8444$$

$$\beta: 0.4871X + 0.7237Y - 0.4890Z = 8.1606$$

	Displacement (Å)
α { C(2)	-0.001
C(3)	-0.003
C(6)	-0.001
N	-0.001
O	0.000
β { C(6)	-0.002
C(7)	0.007
C(8)	-0.002
C(9)	-0.009
C(10)	0.015
C(11)	-0.009

Molecules of (I) are linked into tetramers by a network of O—H...N hydrogen bonds about an S_4 axis at $\frac{1}{2}, 0, z$ (see Figs. 2 and 3). The distances and angles involved in the hydrogen bonding network are quite reasonable (Tables 5 and 7), the O...N distance of 2.784 Å being indicative of a rather strong hydrogen bond. The location of H(19) from the difference-Fourier map was not completely unambiguous. The region between the oxygen and nitrogen of an adjacent molecule consisted of a blurred region of low residual electron density with a maximum 0.89 Å from the oxygen atom. The

Table 5. Summary of selected distances and angles from several oxime structures

	C=N	N—O					O—H
1. (I)	1.297 (9)	1.392 (8)	114.2 (6)	126.4 (6)	112.8 (6)	120.8 (6)	0.894
2. Dimethylglyoxime (neutron)	1.253 (11)	1.321 (21)	111.4 (9)	126.0 (10)	113.8 (9)	120.0 (9)	1.020 (36)
3. Dimethylglyoxime (X-ray)	1.27 (5)	1.38 (2)	113.9 (20)	124.6 (20)	115.1 (20)	120.4 (20)	
4. <i>syn-p</i> -Chlorobenzaldoxime	1.260 (8)	1.408 (7)	110.8 (4)	—	120.8 (5)	—	
5. <i>anti-p</i> -Chlorobenzaldoxime	1.26	1.39	118	130	—	—	
6. Formamidoxime	1.288	1.415	109.7	—	—	—	
7. Acetoxime	1.29 (3)	1.36 (3)	111	131	113	117	
8. α -5-(2'-Chloroethoxy)- <i>o</i> -quinone-2-oxime	1.306 (7)	1.365 (6)	112.5 (8)	125.9 (8)	114.3 (8)	119.6 (8)	0.89†
	O...N	N...H					
1.	2.784 (8)	1.952 (6)	100.2 (4)	122.1 (4)	154.3	119.8	116.4
2.	2.766 (20)	1.906 (20)	86.0 (7)	94.0 (7)	140.0 (25)	106.2 (15)	109.6 (21)
3.	2.83		79.5 (20)	79.5 (20)			
4.	2.825 (6)		84.7*	95.3			
5.	2.82		109				
6.	2.82	2.05					101.9
7.	2.78		111	129			
8.	—	—	—	—	—	—	—

* Jerslev (1957)

† O—H...O rather than O—H...N.

space between the oxygen and nitrogen available for hydrogen occupation is severely restricted by the presence of the S_4 axis; thus we feel reasonably confident in the position reported for H(19). Positioning of H(19) closer to the nitrogen than to the oxygen would imply a zwitterionic structure for this oxime. Several investigators have proposed zwitterionic structures for other oximes based on oxygen–nitrogen angles, but no direct structural evidence has been found to confirm a zwitterionic structure for an oxime in the solid state [see Hamilton (1961) for a summary].

Table 7. Selected intramolecular distances and angles of nearly tetrahedral groups

O ⁱ ...N ⁱⁱ	2.784 (8) Å	O ⁱ ...P(1)...O ⁱⁱⁱ	109.0°
O ⁱ ...N ⁱⁱⁱ	3.386 (8)	O ⁱ ...P(1)...O ⁱⁱ	109.7
O ⁱ ...O ⁱⁱⁱ	3.700 (10)	N ⁱ ...P(1)...N ⁱⁱⁱ	118.1
O ⁱ ...O ⁱⁱ	3.716 (11)	N ⁱ ...P(1)...N ⁱⁱ	105.3
N ⁱ ...N ⁱⁱⁱ	3.588 (11)	Br ⁱ ...P(1)...Br ⁱⁱⁱ	111.9
N ⁱ ...N ⁱⁱ	3.326 (10)	Br ⁱ ...P(1)...Br ⁱⁱ	108.3
Br ⁱ ...Br ^{iv}	3.681 (1)	Br ⁱ ...P(2)...Br ^v	154.2
Br ⁱ ...Br ^v	4.951 (1)	Br ⁱ ...P(2)...Br ^{iv}	92.9
Br ⁱ ...Br ⁱⁱⁱ	7.275 (0)		
Br ⁱ ...Br ⁱⁱ	7.117 (0)		

ⁱ (1)* x, y, z

ⁱⁱ (8) $\bar{y} + \frac{1}{2}, x - \frac{1}{2}, \bar{z} + \frac{1}{2}$

ⁱⁱⁱ (10) $\bar{x} + 1, \bar{y}, z$

^{iv} (6) $\bar{y} + 1, x, \bar{z} + 1$

^v (5) $\bar{x} + 1, \bar{y} + 1, z$

P(1) has coordinates $(\frac{1}{2}, 0, \frac{3}{4})$

P(2) has coordinates $(0, 0, 0)$

* Numbers in parentheses refer to labeling of molecules in Fig. 2.

The molecular packing is perhaps the most unusual feature of the crystal structure. The unit cell, while possessing tetragonal symmetry, is almost cubic (see Table 1). Although not required by the space group, it is observed that several atoms are very nearly tetrahedrally arranged (see Figs. 2 and 3 for a stereoscopic view of the hydrogen-bonding network and molecular packing). As a measure of how closely tetrahedral a set

of four atoms are, the angles $A_i \cdots P_j \cdots A_k$ are compared with the tetrahedral angle of 109.47° in Table 7, for $A = O, N,$ and Br . (The points P_j are the special positions $0, 0, 0$ and $\frac{1}{2}, 0, \frac{3}{4}$ of the space group.) It is interesting to note that both the oxygen and the nitrogen are very nearly tetrahedrally arranged in the hydrogen-bond network. Also, while the four bromines in closest contact [clustered about $0, 0, 0$] are definitely not tetrahedral, there are four bromines about the center of the hydrogen bond network $(\frac{1}{2}, 0, \frac{3}{4})$ which are close to being tetrahedral.

Four bromines cluster about the point $0, 0, 0$. These bromines make van der Waals contacts of 3.681 \AA , in close agreement with the van der Waals radius of 1.85 \AA suggested by Bondi (1964). Several other investigators have noted close Br–Br contacts [1,4-dibromocycl(3,2,2)azine, 3.59 \AA , Hanson, 1961]; *p*-dibromobenzene, 3.76 \AA , (Croatto, Bezzi & Bua, 1952); 1,3,5-tribromobenzene, $3.76, 3.77 \text{ \AA}$, (Milledge & Pant, 1960)].

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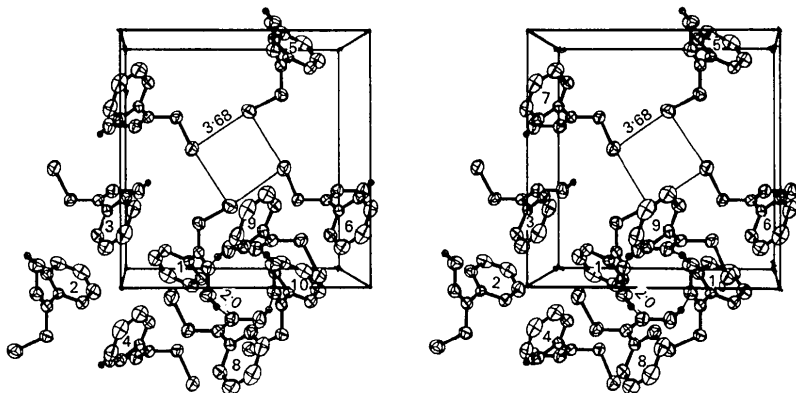


Fig. 2. Stereoscopic view illustrating molecular packing and hydrogen bond network about central molecule (1). Note arrangement of phenyl groups, the four closely packed bromines, and the hydrogen bond network.

(1) x, y, z

(2) \bar{x}, \bar{y}, z

(3) $\bar{y}, x, \bar{z} + 1$

(4) $y, \bar{x}, \bar{z} + 1$

(5) $\bar{x} + 1, \bar{y} + 1, z$

(6) $\bar{y}, + 1, x, \bar{z} + 1$

(7) $y, \bar{x} + 1, \bar{z} + 1$

(8) $\bar{y} + \frac{1}{2}, x - \frac{1}{2}, \bar{z} + \frac{1}{2}$

(9) $y + \frac{1}{2}, \bar{x} + \frac{1}{2}, \bar{z} + \frac{1}{2}$

(10) $\bar{x} + 1, \bar{y}, z$

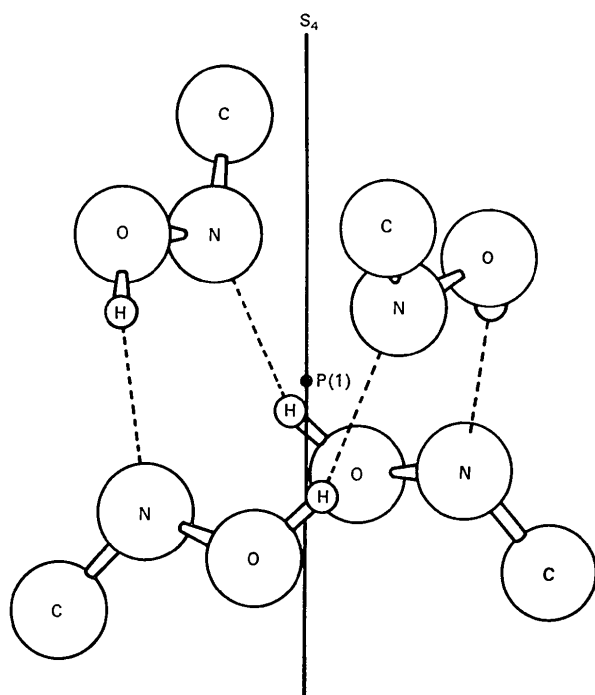


Fig. 3. Tetrameric hydrogen bond network of *anti*- α -bromoacetophenone oxime. The point $P(1)$ has coordinates $(\frac{1}{2}, 0, \frac{3}{4})$ and is the invariant point of the S_4 operation. The nitrogens and oxygens occupy nearly tetrahedral positions.

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Perdeuterated Biuret Hydrate $C_2D_5N_3O_2 \cdot (0.77D_2O)$

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Biuret hydrate $C_2D_5N_3O_2 \cdot nD_2O$; monoclinic, space group $P2_1/c$ (except for the water molecules); $a = 3.6518$ (6), $b = 17.767$ (5), $c = 9.048$ (1) Å, $\beta = 118.91$ (1)°; $d_{meas} = 1.596$ (1) g cm⁻³ at 25°C, corresponding to $Z = 4$, $n = 0.769$ (4). The crystal structure as determined from X-ray (Cu $K\alpha$) diffractometer data is the same as for the hydrogenated material, except in detail. Full-matrix least-squares refinement gave $n = 0.769$ (15), and e.s.d.'s of 0.003 Å in C–N, C–O bond lengths, 0.05 Å in N–D bond lengths and 0.2° in bond angles at nitrogen and carbon atoms. The water molecules appear to be disordered, forming zigzag hydrogen bonds of finite length, as in the hydrates of caffeine and thymine.

Experimental

Biuret was prepared by gently heating urea (Werner, 1923) and purified by several crystallizations from water and finally ethanol. Anhydrous biuret (8g) was then distilled almost to dryness three times with 50 ml of 99.5 mol.% heavy water, supplied by the U. S. Atomic Energy Commission. Final crystallization of deuterated biuret hydrate was by slow evaporation of

a heavy-water solution at room temperature in an atmosphere of nitrogen. Crystal-density measurements were made on several large, clear crystals using the method of flotation in a mixture of carbon tetrachloride and dibromoethane. A long needle was cleaved parallel to (100) to give a crystal of dimensions 0.30, 0.15, 0.30 mm along a^* , b and c . The crystal was immediately

$$* R = \frac{(\sum_h |AF|)}{(\sum_h |F_{meas}|)}$$