

mentaires en plaçant le strontium et l'aluminium donne  $R=0,25$ . L'introduction du fluor ne nous a pas permis d'obtenir un facteur de fiabilité inférieur à 0,20. Ceci s'explique par le fait que le nombre des paramètres à affiner est beaucoup trop élevé par rapport à celui des facteurs de structure observés. Un calcul général effectué sur l'ensemble des strates observées, y compris les strates supplémentaires, donne encore  $R=0,137$ . Les valeurs de  $F_o$  et  $F_c$  des plans supplémentaires sont donc très faibles, et affectent peu le résultat final.

Les déplacements des atomes par rapport aux positions calculées précédemment en faisant abstraction des distortions sont de l'ordre de 0,10 Å pour l'aluminium et de 0,07 Å pour le strontium (Fig. 9). Seule la cote  $z$  varie, les cotes  $x$  et  $y$  restent inchangées. Si les déplacements proposés pour les atomes de strontium peuvent être considérés comme raisonnables, d'après les résultats de l'affinement, les déplacements calculés pour l'aluminium sont donnés à titre de simple indication (Tableau 8). L'amplitude des facteurs de structure observés et calculés est donnée au Tableau 9. A la suite de ce travail nous procédons à une détermination précise des structures de  $Sr_2Fe_2F_9$  et  $Ba_3(FeF_6)_2$ , de manière à pouvoir interpréter les mécanismes de passage de ces phases à  $BaFeF_5$ .

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## The Crystal Structure of Pentabromotoluene

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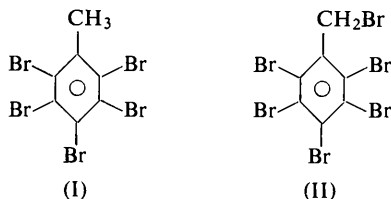
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Pentabromotoluene ( $C_6Br_5CH_3$ ) forms monoclinic crystals having the symmetry of space group  $P2_1/c$ . The reported density,  $2.97 \text{ g.cm}^{-3}$ , indicates two molecules in the unit cell with  $a=8.3798 \pm 0.0008$ ,  $b=4.0080 \pm 0.0007$ ,  $c=17.2526 \pm 0.0009$  Å, and  $\beta=116.84^\circ$ . The structure was determined from three-dimensional  $Cu K\alpha$  diffractometer data by Patterson, electron density, and least-squares methods, which yielded a final  $R=0.074$  for 750 reflections. Molecules are disordered in the crystal, which permits each molecule to occupy a site of  $\bar{1}$  symmetry. The indicated random nature of the disorder, with the methyl group occupying any of the six substituent positions with equal probability, is confirmed by comparing the entropy of fusion with that of pentabromobenzyl bromide, which forms an ordered crystal.

### Introduction

Our attention was directed to pentabromotoluene (I),



an intermediate in the synthesis of pentabromobenzyl bromide (II), when it was discovered that the latter would not cause termination of the very reactive styryl anion in a Szwarc polymerization of styrene. This unusual inertness of the substituted benzyl bromide suggests that it, and in all probability also its precursor (I), are sterically crowded molecules. The  $Br \cdots Br$  separation calculated for an idealized planar  $C_6Br_6$  molecule is only  $3.27$  Å, which is considerably shorter than twice the van der Waals radius of bromine,  $3.9$  Å. The extent of overcrowding might be expected to manifest itself in some measurable distortion of the planar hexagonal structure. The literature does not

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permit a very reliable estimate of either the type, or extent, of expected distortion. Thus, significant deviations from planarity were reported by Bastiansen & Hassel (1947) in an early electron-diffraction study of *o*-dibromobenzene. However, a more recent electron-diffraction study of the same compound by Strand (1966) indicated a planar molecule exhibiting  $C_{2v}$  symmetry, but with an enlarged angle of  $63.5^\circ$  between the carbon-bromine bonds. Turning to the tetrasubstituted derivatives, Gafner & Herstein (1960) found a  $\text{Br}\cdots\text{Br}$  distance of  $3.385 \text{ \AA}$  from a crystallographic study of 1,2,4,5-tetrabromobenzene, which corresponds to a deformation of only  $1.5^\circ$  of the C-Br bond directions. More steric hindrance would be anticipated in the hexasubstituted benzenes. Tulinsky & White (1958) and Strel'tsova & Struchkov (1961) concluded from X-ray studies that hexachlorobenzene is planar and has normal bond lengths. This was confirmed by Strand & Cox (1966), who found from an electron-diffraction study that hexachlorobenzene possesses planar  $D_{6h}$  symmetry. On the other hand, Strand (1966) assigned  $S_6$  symmetry to hexabromobenzene, and reported that alternate bromine atoms lie above and below the plane of the ring, as judged from the  $\text{Br}\cdots\text{Br}$  separation of *ortho* substituents. However, he also reported that the other  $\text{Br}\cdots\text{Br}$  distances, and the non-bonded  $\text{C}\cdots\text{Br}$  distances, were in better agreement with an undistorted planar conformation. Coulson & Stocker (1959) performed theoretical calculations which predicted that the *o*-disubstituted benzenes should be planar, but their analysis did not permit a definite conclusion with regard to distortion in the hexasubstituted benzenes. We have undertaken a crystallographic study of pentabromotoluene to gain additional insight into the deformations resulting from steric overcrowding.

### Experimental

We selected a crystal of pentabromotoluene (I) grown from  $\text{CCl}_4$  solution which was  $40\mu$  by  $70\mu$  in cross section and  $100\mu$  in length along the  $b$  axis. It was mounted with the  $b$  axis along the  $\varphi$  spindle axis. The crystal class was monoclinic, with unit-cell dimensions, measured at  $23^\circ\text{C}$ , of  $a = 8.3798 \pm 0.0008$ ,  $b = 4.0080 \pm 0.0007$ ,  $c = 17.2526 \pm 0.0009 \text{ \AA}$ , and  $\beta = 116.84^\circ \pm 0.02^\circ$  using  $\text{Cu } K\alpha = 1.5418 \text{ \AA}$ . Unit-cell parameters were evaluated by measuring with a narrow counter slit both positive and negative  $2\theta$  values for all available orders. Above  $2\theta = 15^\circ$ , the  $\alpha_1$  and  $\alpha_2$  components were resolved. The  $d$  spacings were extrapolated to  $\theta = 90^\circ$  using the function  $\frac{1}{2}[(\cos^2 \theta/\sin \theta) + (\cos^2 \theta/\theta)]$ . The error limits listed represent estimates based on this plot. The density,  $2.97 \text{ g.cm}^{-3}$ , reported by Groth (1915) indicates there are two molecules per unit cell (calculated density  $3.13 \text{ g.cm}^{-3}$ ). Extinctions were observed in precession photographs for  $h0l$  with  $l$  odd, and  $0k0$  with  $k$  odd. However, diffractometer examination appeared to show very weak 010 and 030 reflect-

ions. Unfortunately, only four orders could be examined with copper radiation, and no source of shorter wavelength was available to us. Therefore, possible space groups are  $Pc$ ,  $P2_1/c$ , or  $P2_1/c$ , if the additional extinction rule involving  $0k0$  reflections applies. Since there are two molecules in the unit cell, and the pentabromotoluene molecule has neither a center of symmetry nor a twofold axis perpendicular to the ring, only space group  $Pc$  is compatible with the true molecular symmetry. However, numerous examples of compounds of this type exhibit disorder in the crystalline state, and in this case the molecule can occupy a site of symmetry higher than the molecular symmetry. The  $N(z)$  test of Howells, Phillips, & Rogers (1950) indicated a centrosymmetric space group, but this test sometimes fails when heavy atoms are present. Although the bulk of the evidence favored  $P2_1/c$ , we decided to begin our analysis with the space group of lowest symmetry,  $Pc$ .

A data set, consisting of 750 unique reflections within the sphere  $2\theta < 125^\circ$ , was collected with nickel-filtered copper radiation using a Picker four-circle diffractometer having a scintillation counter and pulse-height analyzer. Integrated intensities were measured by the  $\theta$ - $2\theta$  scan technique with a scan rate of  $1^\circ \text{ min}^{-1}$  over a  $2\theta$  range of  $2^\circ$ , plus an allowance for dispersion. The background was counted for 40 sec at both extremes of the scan range. Eleven reflections were too weak to observe, but these were included in the analysis with  $|F_{\text{obs}}| = 0.5|F_{\text{min}}|$ , where  $|F_{\text{min}}|$  is the structure amplitude of the weakest observed reflection. Attempts to measure the 010 and 030 reflections at  $\chi = 90^\circ$  revealed only weak intensity at a few particular  $\varphi$  values, so these were treated as extinctions. No correction was applied for absorption. F. R. Ahmed's program, 'Absorption Corrections for the Three-circle Goniostat Geometry Using the Gaussian Integration Formula', was used to obtain a qualitative assessment of the effect of absorption. The linear-absorption coefficient for  $\text{Cu } K\alpha$  radiation is  $258 \text{ cm}^{-1}$ , and the average value of the absorption factor,  $A^*$  was  $2.54$  for the crystal examined. For 6.5% of the reflections measured,  $A^* \geq 3.00$ , while for 5.4% of the reflections,  $A^* \leq 2.20$ .

### Structure analysis

A two-dimensional trial structure suggested by the strong reflections  $\bar{1}06$ ,  $202$ , and  $\bar{3}04$ , which intersect to form angles of approximately  $60^\circ$ , was confirmed by the 16 strongest vectors in the three-dimensional Patterson map. However, the latter did not reveal the location of the methyl group. Hence, two-dimensional analyses were performed using an overall isotropic temperature factor,  $B = 3.74$ , and placing the methyl group in each of the six possible locations. All of these trials resulted in  $R(h0l) = 0.35$  after two  $F_o$  synthesis iterations, with nearly the same electron density appearing in each substituent position. On the other hand, a randomly disordered model having at each substituent position  $\frac{2}{3}$  of a bromine atom, and  $\frac{1}{3}$  carbon



tion so obtained is

$$0.3762 X + 0.9130 Y - 0.1580 Z = 0,$$

where  $X$ ,  $Y$ , and  $Z$  are distances in Å along the orthogonal axes  $a$ ,  $b$ , and  $c^*$ , respectively. Displacements from this plane and their standard deviations in Å are:

$$\begin{aligned} C(1) &= -0.038 \text{ (10)} \\ C(2) &= -0.002 \text{ (10)} \\ C(3) &= -0.003 \text{ (10)} \\ Br(1) &= -0.018 \text{ (2)} \\ Br(2) &= +0.021 \text{ (2)} \\ Br(3) &= -0.021 \text{ (2)}. \end{aligned}$$

There appears to be a slight tendency for the bromine atoms to alternate above and below the plane of the ring, but the deviations from planarity are within  $\pm 0.02$  Å. We must recognize the possibility that the bromine atoms may deviate from planarity by substantially more than is indicated above. If the molecule is actually puckered and, due to the random disorder, the bromine locations are effectively an average of the 'up' and 'down' positions, the diffraction data would give an erroneous indication of planarity. The same consideration applies to the hexahalobenzenes, although

in this case the crystallographic data would not reveal the presence of disorder. If pentabromotoluene is actually puckered, the thermal ellipsoids for the bromine atoms should be considerably elongated in the direction perpendicular to the ring. The thermal ellipsoids in Fig. 2 at the 50% probability level are some-

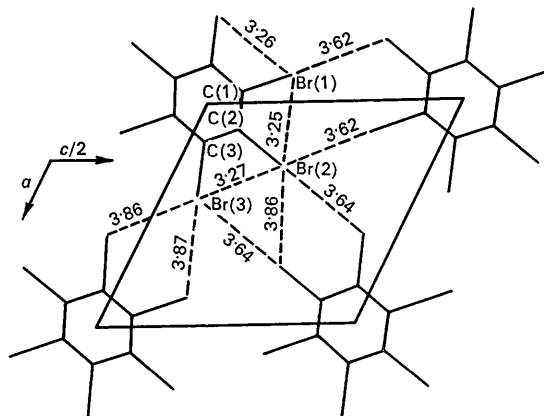


Fig. 1. The shortest interatomic distances in pentabromotoluene shown in projection on the  $a$ - $c$  plane.

Table 2. *Positional parameters and temperature factors of pentabromotoluene*

Temperature factors are in the form:  $\exp[-B \sin^2 \theta/\lambda^2]$  or  $\exp[-(B_{11}h^2 + B_{12}hk + \dots)]$ .

	$x/a$	$y/b$	$z/c$
C(1)	-0.0553 (12)*	0.1260 (24)	0.0605 (6)
C(2)	0.1181 (13)	0.0263 (22)	0.0877 (6)
C(3)	0.1752 (13)	-0.1088 (26)	0.0294 (6)
Br(1)	-0.13226 (19)	0.30942 (38)	0.13665 (8)
Br(2)	0.27938 (16)	0.06568 (52)	0.20510 (8)
Br(3)	0.41100 (19)	-0.25820 (43)	0.06929 (9)

	$B$	$B_{11}$	$B_{22}$	$B_{33}$	$B_{23}$	$B_{13}$	$B_{12}$
C(1)	2.61 (16)						
C(2)	2.58 (16)						
C(3)	2.85 (16)						
Br(1)		0.01489 (26)	0.0653 (11)	0.00286 (6)	-0.00364 (37)	0.00858 (21)	0.00728 (78)
Br(2)		0.01328 (31)	0.0965 (14)	0.00256 (6)	-0.00137 (46)	0.00345 (22)	-0.00493 (95)
Br(3)		0.00953 (25)	0.0874 (12)	0.00373 (6)	0.00804 (40)	0.00575 (21)	0.01760 (75)

\* Numbers in parentheses give the standard deviations in the last significant figures.

Table 3. *Bond distances and angles*

Standard deviations are in parentheses. Primed atoms are related to unprimed atoms by a center of symmetry.

C(1)-C(2)	1.370 (16) Å	C(2)-C(1)-Br(1)	121.8 (8)°
C(2)-C(3)	1.404 (15)	C(2)-C(1)-C(3')	118.6 (9)
C(1)-C(3')	1.419 (13)	Br(1)-C(1)-C(3')	119.4 (8)
C(1)-Br(1)	1.857 (10)	C(1)-C(2)-C(3)	121.3 (10)
C(2)-Br(2)	1.866 (10)	C(1)-C(2)-Br(2)	118.9 (8)
C(3)-Br(3)	1.873 (12)	C(3)-C(2)-Br(2)	119.8 (8)
(Nonbonded atoms)		C(2)-C(3)-Br(3)	120.3 (8)
Br(1)···Br(2)	3.251 (2)	C(2)-C(3)-C(1')	120.0(10)
Br(2)···Br(3)	3.275 (2)	Br(3)-C(3)-C(1')	119.7 (8)
Br(1)···Br(3')	3.260 (2)	(Nonbonded atoms)	
		Br(2)-Br(1)-Br(3')	119.84 (6)
		Br(1)-Br(2)-Br(3)	120.27 (6)
		Br(2)-Br(3)-Br(1')	119.85 (6)

what elongated in this direction. Table 4, which gives the principal-axis transformations of the thermal ellipsoids, reveals that the bromine thermal ellipsoids are oblate, with the larger principal axes directed approximately normal to the plane and tangential to the ring, respectively.

Table 4. Root-mean-square amplitudes of vibration for the carbon atoms and r.m.s. displacements and principal axes of thermal vibrational ellipsoids for the bromine atoms

Direction cosines ( $H$ ) are referred to the axes  $a$ ,  $b$ , and  $c^*$

	Axis ( $i$ )	$U$ (Å <sup>2</sup> )	$H_{ia}$	$H_{ib}$	$H_{ic}^*$
C(1)		0.182			
C(2)		0.181			
C(3)		0.190			
Br(1)	1	0.142	-0.709	0.296	0.640
	2	0.210	-0.624	0.160	-0.765
	3	0.238	-0.329	-0.942	0.071
Br(2)	1	0.171	0.296	0.060	0.953
	2	0.212	-0.952	-0.069	0.299
	3	0.281	-0.083	0.996	-0.037
Br(3)	1	0.151	0.922	-0.243	0.301
	2	0.206	-0.363	-0.279	0.889
	3	0.278	0.131	0.929	0.346

The disorder in pentabromotoluene involves the random occupancy of the methyl group of one of the substituent positions. Since X-ray diffraction gives a time-averaged result, it cannot distinguish between a static model in which each molecule is permanently fixed in one of the possible orientations, and a dynamic model in which each molecule undergoes discontinuous librations of  $\pi/3$ , but spends most of the time near one of the six orientations of lowest energy (Kauzmann, 1942). Tulinsky & White (1958) concluded from X-ray and dielectric constant measurements that the disorder in 1,2-dichlorotetramethylbenzene is best described by the dynamic model. Charbonneau & Trotter (1967) also proposed that the disorder in 1,2,4-trichloro-3,5,6-trimethylbenzene is of the dynamic type, after eliminating the static model by nuclear magnetic resonance second-moment analysis. We note, in passing, that the large tangential component of the bromine thermal

ellipsoids shown in Table 4 is consistent with the hypothesis of kinetic disorder for pentabromotoluene as well. Khotsyanova (1966) collected unit-cell parameters for 13 hexasubstituted benzene derivatives containing Br and Cl. Crystals of all these compounds are isomorphous, belonging to space group  $P2_1/c$  with two molecules per unit cell; hence, those having no true center of symmetry must be disordered. Khotsyanova, Babushkina, Kuznetsov & Semin (1969) point out that the statistical character of the disorder in this type of compound cannot be predicted *a priori* from the fact that the molecule occupies the special position  $\bar{1}$ . They found the disorder to be nonrandom in pentabromoaniline and pentachloroaniline, both of which crystallize in  $P2_1/c$  with two molecules in the unit cell. The probability of finding the  $\text{NH}_2$  group in each of the three nonsymmetry related positions was 16.3, 16.3, and 67.4% for the pentabromo compound, as evaluated from the electron density in two-dimensional projection. The corresponding values deduced from a three-dimensional analysis of the pentachloro derivative were 23.5, 23.5, and 53.0%. The authors substantiated the latter values by nuclear quadrupole resonance of  $^{35}\text{Cl}$ .

The statistical occupancy of the  $\text{CH}_3$  group in pentabromotoluene appears to be very nearly random, since the three substituent peaks in the final three-dimensional electron-density diagram are of about the same magnitude. This conclusion finds support in a comparative thermodynamic study of pentabromotoluene (I) and pentabromobenzyl bromide (II) using a Perkin Elmer DSC-1B differential scanning calorimeter. Thermograms were obtained using a heating rate of  $10^\circ \text{min}^{-1}$  for an indium standard and for several samples ranging in weight from 5 to 10 mg. The melting temperatures, enthalpies, and entropies of fusion were:

Compound	$T_m$ (°K)	$\Delta H_f$ (kcal.mol <sup>-1</sup> )	$\Delta S_f$ (eu)
I	556	$6.37 \pm 0.07$	$11.5 \pm 0.1$
II	456	$6.79 \pm 0.09$	$14.9 \pm 0.2$

We observe that these two compounds have nearly the same enthalpy of fusion, but their melting points differ by  $100^\circ$ . Crystals of (II) are triclinic, and we can infer

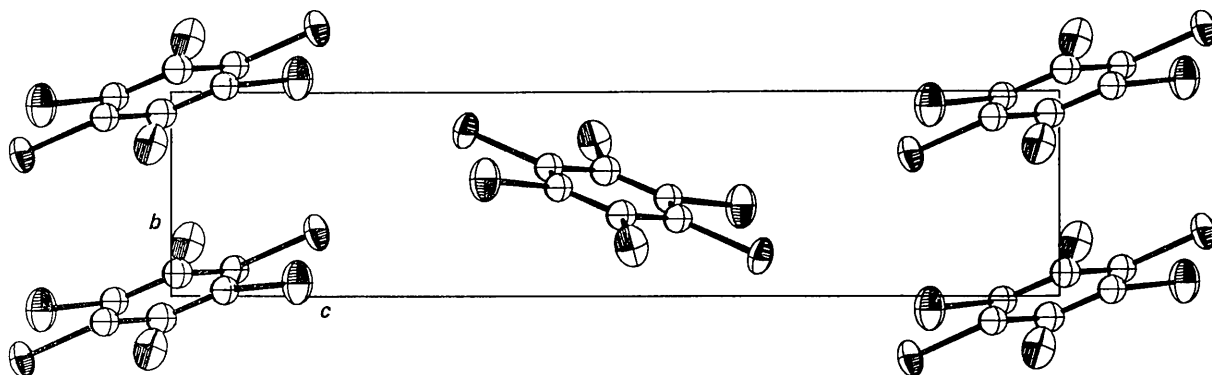


Fig. 2. Molecular packing of pentabromotoluene shown on the  $b$ - $c$  plane.

that molecules of pentabromobenzyl bromide enter the crystal lattice in an ordered manner due to their asymmetrical shape. If we ascribe the difference in  $\Delta S_f$  solely to entropy differences in the solid state, then for random disorder in (I) we expect an entropy difference of  $R \ln 6 = 3.54$  e.u. This stands in reasonable agreement with the 3.4 e.u. difference in the experimental  $\Delta S_f$  values, thereby supporting the deduction of random disorder in pentabromotoluene from the crystal-structure study.

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## The Crystal Structure of Guanine Monohydrate

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The crystal structure of guanine monohydrate ( $C_5H_5N_5O \cdot H_2O$ ) has been determined from three-dimensional diffractometer and visual data and refined by full-matrix least-squares to an  $R$  index of 0.101 for 530 reflexions. The crystals are monoclinic, space group  $P2_1/n$ , with cell dimensions  $a = 16.510$  (8),  $b = 11.277$  (8),  $c = 3.645$  (5) Å,  $\beta = 96.8$  (1)° and  $Z = 4$ . The hydrogen bonding between bases is similar to that found in the crystal structures of guanosine and inosine. The molecules are stacked along  $c$  with an interplanar spacing of 3.30 Å. The crystal structure of guanine is almost identical to that of 8-azaguanine.

#### Introduction

The determination of the crystal structure of guanine was long a goal of this Laboratory. Guanine has properties, such as low solubility in most solvents, that are not characteristic of the other nucleic acid bases.

Of particular interest to us was the tautomeric form that guanine might choose – that is, which two of the four ring nitrogen atoms are protonated. It has been assumed that the favored positions for these two protons are at N(1) and N(9); however, other tautomeric forms can be represented by satisfactory valence-bond structures, and could lead to interesting hydrogen bonding and base pairing arrangements.

We have recently been successful in obtaining small crystals of guanine. Although the size of these crystals did not allow us to obtain highly accurate intensity data, we have been able to carry out a moderately

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