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The Crystal and Molecular Structure of Tribenzylamine at −70 °C

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Crystals of tribenzylamine are monoclinic, space group $P2_1/a$ with cell dimensions $a=21{\cdot}076$ (28), $b=9{\cdot}015$ (6), $c=8{\cdot}917$ (9) Å, $\beta=93{\cdot}9$ (2)° and $Z=4$ at −70 °C. The structure was determined by three-dimensional X-ray analysis. Intensity data were collected on an equi-inclination type single-crystal automatic diffractometer with Mo $K\alpha$ radiation at −70 °C. Phases were found by the symbolic addition procedure and subsequent refinement was carried out by the block-diagonal least-squares method, the final R value being 0·092 for 821 observed reflexions. Three benzene rings surround a nitrogen atom so that the molecule has a propeller shape. The non-bonding intramolecular distances correspond to usual contact distances and the molecular conformation is determined entirely by steric hindrance.

Introduction

Tribenzylamine (TBA), $N(CH_2C_6H_5)_3$, is an aliphatic tertiary amine with bulky substituents. It may exhibit various kinds of conformations, since the molecule has six intramolecular rotation axes. In order to determine the conformation of TBA in the crystalline state, a three-dimensional X-ray analysis was carried out. This determination is considered to be important because TBA was found to form addition compounds or salts with halogens in CCl_4 and $CHCl_3$ solutions, and the molecular conformation of TBA in these compounds is expected to change depending on the difference in the molecular interactions.

Experimental

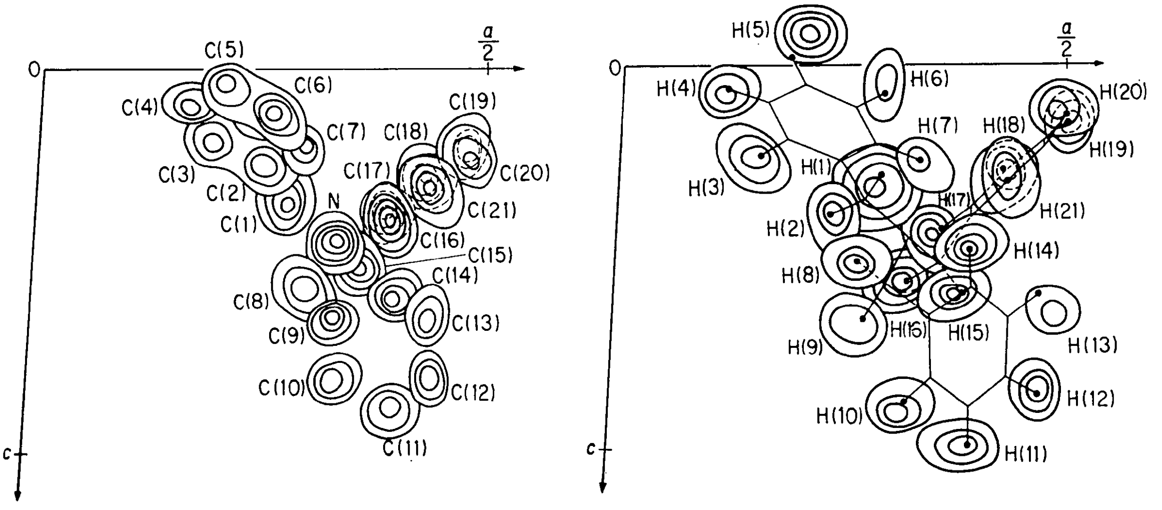
The crystals were grown by slow evaporation from an ethanol solution of commercially available TBA. They were colourless, flat plates elongated along the b axis, with the well developed form {100}, and were stable in air. They are monoclinic, space group $P2_1/a$. Some were twinned, giving pseudo-orthorhombic diffraction patterns. At room temperature the cell dimensions obtained from Weissenberg photographs with Cu $K\alpha$ radiation ($\lambda=1{\cdot}5418$ Å) are: $a=21{\cdot}86$, $b=8{\cdot}92$, $c=9{\cdot}02$ Å, $\beta=95{\cdot}0°$, $Z=4$, $D_x=1{\cdot}09$ g.cm^{-3}. Diffraction patterns obtained at room temperature suggested that unusually large thermal vibrations extended in the crystal. All subsequent data were collected at low temperature on an equi-inclination type single-crystal automatic diffractometer RIKEN-READ 1 (Sakurai, Ito & Iimura, 1970) with Mo $K\alpha$ radiation ($\lambda=0{\cdot}71069$ Å). The improved cell dimensions were obtained by least-squares fit with diffractometer 2θ data. The crystal data at about −70 °C are: $C_{21}H_{21}N$, M.W. 287·40; monoclinic, $a=21{\cdot}076$ (28), $b=9{\cdot}015$ (6), $c=8{\cdot}917$ (9) Å, $\beta=93{\cdot}9$ (2)°, $V=1960{\cdot}4$ Å^3, $Z=4$; $D_x=1{\cdot}129$ g.cm^{-3}; $F(000)=616$; $\mu=0{\cdot}75$ cm^{-1} (Mo $K\alpha$); space group, $P2_1/a$. There was no phase transition of crystals between the room temperature and liquid nitrogen temperature.

A Cryo-Tip refrigerator ACl-101 was employed to obtain low temperatures. With this equipment the specimens are placed *in vacuo*. The intensity of the monitor reflexion decreased slowly during data collection, presumably as a result of sublimation. Because of this intensity decrease three different crystals were used, the cross sections of which ranged from 0·40 × 0·20 to 0·25 × 0·25 mm. The intensity data were collected around the b and c axes, $h0l \sim h10l$ and $hk0 \sim hk1$, with Mo $K\alpha$ radiation monochromated by means of a graphite monochromator. X-ray reflexions were scanned in the ω–Y mode at 2° per min in Y, with 30 sec backgrounds at each scan side. Intensities of 821 independent reflexions were observed for $\sin\theta/\lambda < 0{\cdot}638$. They were corrected for usual Lorentz and polarization factors. No absorption or extinction corrections were applied.

Structure determination

An approximate scale factor and average temperature factor were obtained by Wilson's method. The structure was solved by the symbolic addition procedure (Karle & Karle, 1963). A program *DP*-3 written by S. R. Hall was utilized to list the $\sum_2$ relationships for each reflexion and to calculate the associated probabilities. Signs of 64 reflexions out of 189 with $|E| \geq 1{\cdot}5$ were determined manually. The phases were then refined by the tangent formula using the program *DP*-5 and additional phases were propagated until all 310 reflexions with $|E| \geq 1{\cdot}2$ were refined. An E map, shown in Fig. 1(a), was computed with these reflexions and this clearly revealed the positions of all the heavy atoms in the molecule.

Five cycles of block-diagonal least-squares refinement of these atoms with isotropic temperature factors gave an R value of 0·22. Anisotropic temperature factors were introduced and the refinement was continued, reducing the R value to 0·135. At this stage hydrogen atoms were located from a difference-Fourier synthesis. The coordinates of the hydrogen atoms, with isotropic temperature factors arbitrarily set at 3·7 Å^2, were used in four cycles of least-squares refinement for adjustment of coordinates and anisotropic temperature factors of the non-hydrogen atoms. The R value dropped to 0·112. A second difference-Fourier synthesis was calculated in an attempt to improve the hydrogen-atom positions [Fig. 1(*b*)]. Five more cycles of block-diagonal least-squares refinement were carried out with anisotropic temperature factors for carbon and nitrogen atoms and with isotropic temperature factors for hydrogen atoms. The final R value was 0·092 for all the observed reflexions. Atomic scattering factors for carbon and nitrogen were taken from *International Tables for X-ray Crystallography* (1962). For hydrogen, the



(*a*) (*b*)

Fig. 1. (*a*) Sections of the three-dimensional E map projected along the b axis. The contours are at equal intervals on an arbitrary scale. (*b*) Sections of the three-dimensional difference-Fourier synthesis through the atomic centres parallel to (010). The contours are at intervals of 0·1 e.Å^{-3}, starting at 0·1 e.Å^{-3}.

Table 1. *Final atomic coordinates* ($\times 10^4$) *and thermal parameters* ($\times 10^3$) *of non-hydrogen atoms with their estimated standard deviations*

The anisotropic temperature factors are of the form:

$$\exp \{-2\pi^2(U_{11}h^2a^{*2}+U_{22}k^2b^{*2}+U_{33}l^2c^{*2}+2U_{12}hka^*b^*+2U_{13}hla^*c^*+2U_{23}klb^*c^*)\}.$$

The B_{eq} values are the equivalent isotropic temperature factors proposed by Hamilton (1959).

Atom	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}	B_{eq}
N	3317 (4)	2213 (8)	4540 (9)	46 (6)	17 (4)	32 (5)	−10 (4)	2 (5)	−3 (4)	2·5
C(1)	2784 (6)	1782 (11)	3478 (15)	62 (9)	17 (5)	73 (9)	−2 (6)	4 (7)	10 (6)	4·0
C(2)	2535 (5)	3072 (12)	2519 (14)	49 (8)	32 (6)	57 (8)	−1 (6)	2 (7)	−6 (6)	3·6
C(3)	1913 (6)	3051 (14)	1967 (14)	52 (9)	50 (8)	60 (9)	−1 (7)	2 (7)	−19 (7)	4·3
C(4)	1682 (7)	4097 (16)	960 (14)	89 (11)	88 (11)	38 (8)	33 (9)	−7 (8)	−18 (8)	5·7
C(5)	2062 (7)	5232 (15)	547 (14)	94 (11)	67 (9)	28 (7)	41 (8)	5 (7)	8 (7)	5·0
C(6)	2674 (6)	5304 (13)	1157 (15)	66 (9)	39 (7)	63 (9)	6 (6)	19 (7)	28 (7)	4·4
C(7)	2916 (6)	4237 (13)	2135 (15)	61 (9)	39 (7)	75 (10)	−8 (7)	1 (8)	27 (7)	4·6
C(8)	3083 (7)	3118 (12)	5751 (16)	78 (10)	26 (6)	73 (9)	−2 (7)	19 (8)	−8 (7)	4·6
C(9)	3607 (6)	3956 (12)	6625 (13)	60 (9)	34 (7)	45 (7)	−2 (6)	−4 (7)	−10 (6)	3·7
C(10)	3650 (7)	3961 (13)	8201 (13)	89 (10)	40 (7)	40 (7)	11 (7)	6 (8)	0 (6)	4·5
C(11)	4113 (7)	4824 (14)	8959 (16)	97 (12)	44 (8)	64 (10)	25 (8)	−16 (9)	−25 (7)	5·5
C(12)	4527 (6)	5644 (13)	8201 (15)	55 (9)	33 (7)	88 (10)	9 (6)	−43 (8)	−24 (7)	4·8
C(13)	4500 (6)	5608 (14)	6639 (15)	48 (8)	47 (7)	76 (9)	5 (7)	−29 (8)	−8 (7)	4·6
C(14)	4055 (7)	4733 (15)	5906 (14)	79 (10)	74 (10)	37 (8)	−46 (8)	−8 (7)	−1 (7)	5·0
C(15)	3644 (6)	899 (13)	5217 (15)	72 (10)	36 (7)	75 (9)	15 (7)	6 (8)	11 (7)	4·8
C(16)	3999 (6)	81 (11)	3996 (15)	60 (9)	13 (5)	74 (9)	3 (6)	17 (8)	3 (6)	3·8
C(17)	3988 (7)	−1429 (12)	3827 (18)	107 (12)	21 (6)	103 (12)	27 (8)	14 (11)	5 (7)	6·0
C(18)	4372 (7)	−2160 (14)	2868 (18)	109 (13)	32 (8)	113 (13)	29 (8)	−33 (11)	−23 (9)	6·8
C(19)	4796 (7)	−1387 (16)	2101 (18)	86 (11)	75 (10)	76 (11)	38 (9)	3 (9)	−21 (9)	6·3
C(20)	4801 (7)	135 (16)	2158 (18)	85 (11)	68 (10)	82 (11)	0 (9)	22 (9)	−27 (8)	6·1
C(21)	4401 (7)	831 (14)	3107 (19)	72 (10)	37 (7)	134 (14)	−6 (7)	39 (10)	−31 (9)	6·3

values given by Stewart, Davidson & Simpson (1965) were used. The quantity $\sum w(kF_o-F_c)^2$ was minimized, where w is the weight function and k is the scale factor. The weighting scheme employed was: $w=(32/|F_o|)^2$ if $|F_o|>32$, $w=1$ if $32\geq|F_o|\geq 8$ and $w=0{\cdot}2$ if $|F_o|<8$.

Final atomic parameters are listed in Tables 1 and 2. Standard deviations in positional coordinates were: C=0·014, N=0·009 and H=0·1 Å. A comparison of the observed and calculated structure factors is given in Table 3.

Table 2. *Fractional coordinates* ($\times 10^3$) *and thermal parameters* (Å^2) *of hydrogen atoms*

Atom	x	y	z	B
H(1)	294 (5)	79 (13)	296 (13)	2·6 (2·9)
H(2)	243 (6)	132 (14)	397 (14)	4·3 (3·3)
H(3)	160 (5)	219 (13)	241 (13)	3·4 (3·0)
H(4)	121 (6)	407 (13)	72 (14)	4·3 (3·3)
H(5)	192 (6)	610 (14)	−22 (14)	4·6 (3·4)
H(6)	293 (5)	618 (12)	83 (13)	2·3 (2·8)
H(7)	341 (6)	426 (14)	257 (14)	4·6 (3·3)
H(8)	276 (5)	384 (13)	515 (13)	2·8 (3·0)
H(9)	287 (5)	238 (14)	670 (13)	4·0 (3·3)
H(10)	335 (5)	334 (14)	890 (13)	3·9 (3·2)
H(11)	411 (5)	481 (13)	1002 (14)	3·4 (3·2)
H(12)	485 (6)	623 (13)	875 (13)	3·1 (3·0)
H(13)	481 (6)	615 (14)	609 (13)	4·2 (3·3)
H(14)	400 (5)	467 (12)	486 (13)	2·2 (2·8)
H(15)	394 (5)	157 (13)	612 (14)	3·4 (3·2)
H(16)	329 (5)	11 (12)	569 (13)	1·8 (2·8)
H(17)	367 (5)	−203 (13)	441 (12)	3·2 (3·0)
H(18)	434 (5)	−326 (13)	274 (13)	3·7 (3·2)
H(19)	508 (5)	−202 (13)	144 (13)	2·8 (2·9)
H(20)	502 (6)	72 (14)	138 (14)	4·6 (3·4)
H(21)	439 (5)	186 (14)	313 (13)	4·0 (3·2)

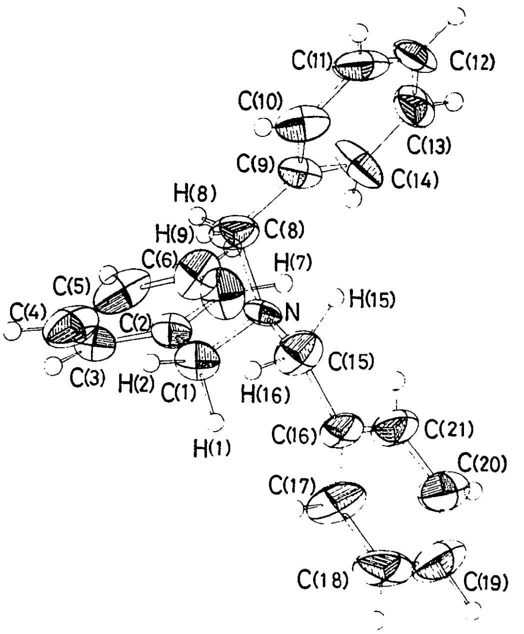
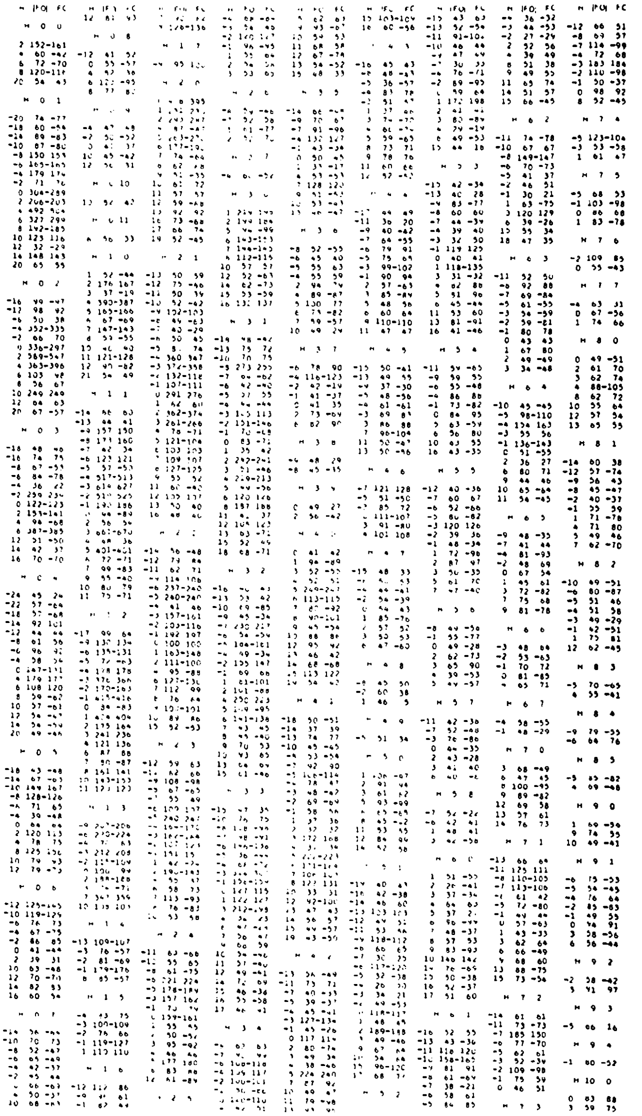


Fig. 2. Perspective drawing of the molecule showing the ellipsoids of thermal motion with a probability of 50%. Hydrogen atoms are represented by 0·1 Å radius spheres.

Table 3. *Observed and calculated structure factors* (×5)



Results and discussion

Molecular structure

Thermal-motion ellipsoids of the individual atoms and the numbering are shown in Fig. 2. Least-squares planes through various groups of atoms in the structure and the deviations from these planes are listed in Table 4. Fig. 3 shows the molecule projected onto the plane IV formed by three methylene carbon atoms, C(1), C(8) and C(15). The lone paired orbital of the nitrogen atom orients to the direction normal to this plane. The benzene rings surround the nitrogen atom so that the molecule has a propeller shape. The individual benzene rings are planar, deviations being within 0·03 Å for rings I and II, within 0·04 Å for ring III. Carbon atoms

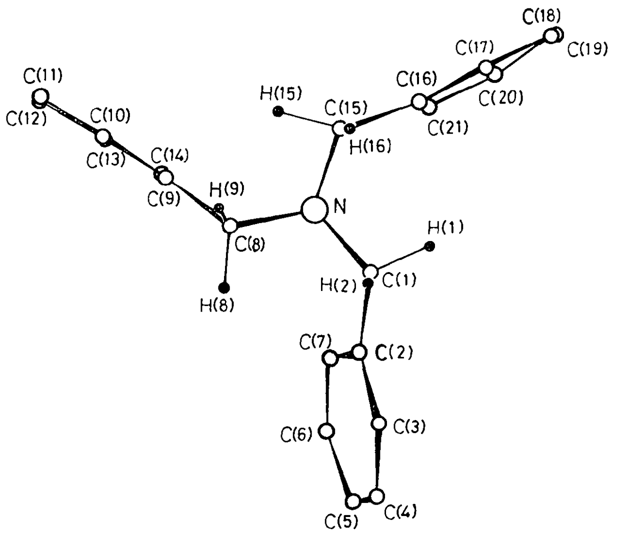


Fig. 3. Projection of the molecule parallel to the plane IV.

of methylene groups, C(1), C(8) and C(15), are slightly displaced from these benzene planes, with deviations of 0·42, 0·12 and 0·25 Å for rings I, II and III respectively. The angles between plane IV and the benzene rings are 72, 90 and 87°, all in the same sense.

Three hydrogen atoms, H(1), H(8) and H(15), are bonded to the methylene carbon atoms axially related to the plane IV, while H(2), H(9) and H(16) are bonded equatorially. The molecular conformations about the C–N bonds and C–C bonds are shown in Fig. 4. It is seen that the axial C–H bond is *gauche* to one of the two N–C bonds and *trans* to the other. On the other hand, the equatorial C–H bond is *gauche* to both the N–C bonds. For the conformation about the C–C bonds, equatorial C–H bonds are approximately *cis* to the C–C bonds of the phenyl groups.

Bond lengths and angles with their estimated standard deviations are given in Table 5. Details of the molecule are shown in Fig. 5. The mean value of the C–C bond distances in the benzene rings is 1·374 Å, which is shorter than the value found in benzene, 1·392 Å (Cox, Cruickshank & Smith, 1958). The average N–C distance, 1·472 Å, is close to the corresponding value in gaseous trimethylamine (1·472 Å) (Lide & Mann, 1958). The bond lengths of C(1)–C(2) and C(8)–C(9), 1·516 and 1·510 Å respectively, are similar to the corresponding values for toluene in the gas phase (1·51 Å) (Keidel & Bauer, 1956) and for *p*-toluic acid (1·514 Å) (Takwale & Pant, 1971). They are not significantly different from the accepted value for a single C(sp^3)–C(sp^2) bond (1·501 Å) (Stoicheff, 1962). It should be noticed, however, that the value for C(15)–C(16), 1·549 Å, is somewhat greater than for the other bonds. The average value of the C–N–C angles is 110·0° and that of the C–C–N angles is 111·5°.

Some of the non-bonding intramolecular distances are listed in Table 6. The shortest C···C distances between adjacent benzyl groups are C(1)···C(16), C(8)···C(2) and C(15)···C(9), the mean value being 3·019 Å. This is in agreement with the value of 3·0 Å that is typical for bonds of this type. The hydrogen atom coordinates have not been determined with sufficient accuracy for a meaningful discussion of their positions. However, the non-bonded H···H distances between benzyl groups are longer than 2·25 Å. These non-bonding distances correspond to contact distances and it follows that the molecular conformation is determined entirely by steric hindrance.

Table 4. *Least-squares planes*

Equation of the plane: $AX+BY+CZ=D$, where X, Y and Z are the coordinates in Å referred to the crystal axes a, b and c. D is the plane-to-origin distance in Å.

		A	*B*	*C*	*D*
I	Benzene ring C(2)∼C(7)	0·3401	−0·5435	−0·7889	1·4832
II	Benzene ring C(9)∼C(14)	−0·6268	0·7785	0·0111	−1·9497
III	Benzene ring C(16)∼C(21)	0·6597	−0·0671	0·7018	8·0302
IV	C(1), C(8) and C(15)	0·6905	0·5350	−0·5328	3·2587

Displacements (Å × 10^3) of atoms from mean planes. Bold-face deviations indicate the atoms used to define the least-squares plane.

I		II		III		IV	
N	−417	N	−835	N	−710	N	479
C(1)	158	C(8)	122	C(15)	247	C(2)	715
C(2)	**23**	C(9)	**28**	C(16)	**25**	C(9)	751
C(3)	**−24**	C(10)	**−10**	C(17)	**−4**	C(16)	702
C(4)	**6**	C(11)	**−9**	C(18)	**−27**	H(1)	−3
C(5)	**12**	C(12)	**12**	C(19)	**36**	H(2)	−980
C(6)	**−13**	C(13)	**6**	C(20)	**−13**	H(8)	162
C(7)	**−5**	C(14)	**−26**	C(21)	**−17**	H(9)	−1114
						H(15)	326
						H(16)	−1117

Angles between the plane *A* and the plane *B*.

A	I	I	II	I	II	III
B	II	III	III	IV	IV	IV
Angle (°)	127·7	108·3	119·3	71·5	90·1	86·8

Table 5. *Bond lengths* (Å) *and angles* (°) *with their estimated standard deviations in parentheses*

N——C(1)	1·472 (14)	C(9)—C(10)	1·402 (17)
N——C(8)	1·466 (16)	C(9)—C(14)	1·370 (18)
N——C(15)	1·478 (14)	C(10)–C(11)	1·387 (19)
C(1)—C(2)	1·516 (16)	C(11)–C(12)	1·359 (19)
C(8)—C(9)	1·510 (17)	C(12)–C(13)	1·391 (19)
C(15)–C(16)	1·549 (19)	C(13)–C(14)	1·359 (18)
C(2)—C(3)	1·368 (17)	C(16)–C(17)	1·369 (15)
C(2)—C(7)	1·379 (17)	C(16)–C(21)	1·376 (20)
C(3)—C(4)	1·369 (18)	C(17)–C(18)	1·384 (22)
C(4)—C(5)	1·365 (20)	C(18)–C(19)	1·354 (22)
C(5)—C(6)	1·368 (19)	C(19)–C(20)	1·373 (20)
C(6)—C(7)	1·373 (17)	C(20)–C(21)	1·386 (22)

C(1)–N——C(8)	109·9 (9)	C(8)—C(9)—C(10)	121·0 (12)
C(1)–N——C(15)	111·5 (9)	C(8)—C(9)—C(14)	121·1 (12)
C(8)–N——C(15)	108·5 (9)	C(10)–C(9)—C(14)	117·9 (12)
N——C(1)—C(2)	112·4 (11)	C(9)—C(10)–C(11)	119·0 (13)
N——C(8)—C(9)	112·8 (11)	C(10)–C(11)–C(12)	121·2 (14)
N——C(15)–C(16)	109·2 (10)	C(11)–C(12)–C(13)	120·1 (13)
C(1)–C(2)—C(3)	118·6 (11)	C(12)–C(13)–C(14)	118·4 (13)
C(1)–C(2)—C(7)	122·6 (11)	C(9)—C(14)–C(13)	123·2 (13)
C(3)–C(2)—C(7)	118·7 (12)	C(15)–C(16)–C(17)	123·0 (13)
C(2)–C(3)—C(4)	121·1 (12)	C(15)–C(16)–C(21)	121·1 (13)
C(3)–C(4)—C(5)	120·2 (13)	C(17)–C(16)–C(21)	115·6 (14)
C(4)–C(5)—C(6)	118·9 (13)	C(16)–C(17)–C(18)	122·3 (15)
C(5)–C(6)—C(7)	121·2 (13)	C(17)–C(18)–C(19)	120·1 (15)
C(2)–C(7)—C(6)	119·6 (12)	C(18)–C(19)–C(20)	120·0 (15)
		C(19)–C(20)–C(21)	118·1 (15)
		C(16)–C(21)–C(20)	123·6 (15)

	Range	Mean value
C–H (methylene)	0·99–1·19 Å	1·10 (12) Å
C–H (phenyl)	0·93–1·10	1·01 (12)
N-C-H	95–113°	106 (6)°
H-C-H	96–116	109 (8)
C–C–H (methylene)	102–120	112 (6)
C–C–H (phenyl)	113–125	120 (7)

Table 6. *Some intramolecular non-bonding distances* (Å)

C(1)····C(16)	2·99 (2)	H(2)····H(9)	2·7 (2)
C(2)····C(8)	3·03 (2)	H(2)····H(16)	2·6 (2)
C(9)····C(15)	3·03 (2)	H(9)····H(16)	2·4 (2)
H(1)····C(15)	2·4 (1)	H(1)····H(16)	2·6 (2)
H(1)····C(16)	2·4 (1)	H(8)····H(2)	2·6 (2)
H(8)····C(1)	2·4 (1)	H(15)···H(9)	2·5 (2)
H(8)····C(2)	2·5 (1)	H(2)····H(3)	2·3 (2)
H(15)···C(8)	2·3 (1)	H(9)····H(10)	2·3 (2)
H(15)···C(9)	2·3 (1)	H(16)···H(17)	2·4 (2)
		H(7)····H(14)	2·3 (2)

Crystal structure

The arrangement of the molecules is shown in Figs. 6 and 7. Intermolecular distances less than 3·8 Å are listed in Table 7. All contacts are of the van der Waals type. The molecular interaction between adjacent molecules along the *a* axis seems to be weaker than those along the *b* and *c* axes. The benzyl group III, [C(15)~C(21)], has no contact less than 3·8 Å to the neighbouring molecules. The thermal vibration of this group is larger than those of other benzyl groups. If the plane of the benzene ring III librates strongly about the long axis of the benzyl group with increasing temperature, the unit length of the *b* axis will be shortened. Although the expansion of the cell volume from −70°C to the room temperature is 3%, the length of the *b* axis is shorter at room temperature.

Table 7. *Intermolecular distances less than* 3·8 Å

i	x	y	z
ii	$\frac{1}{2}-x$	$-\frac{1}{2}+y$	$1-z$
iii	x	y	$-1+z$
iv	x	$1+y$	z
v	$1-x$	$1-y$	$2-z$

C(3^{i})····C(11^{ii})	3·69 (2) Å
C(3^{i})····C(12^{ii})	3·73 (2)
C(6^{i})····C(10^{iii})	3·66 (2)
C(6^{i})····C(11^{iii})	3·75 (2)
C(11^{i})···C(12^{v})	3·72 (2)
C(13^{i})···C(17^{iv})	3·77 (2)

Even at a low temperature the molecular motion is markedly large. The mean temperature factor of the atoms approximately corresponds to a $B\sim 5$ Å^2, which is fairly large compared with the normal B values in usual organic substances at room temperature. This suggests that the molecule of TBA is considerably flexible. Short C–C distances observed in phenyl groups may be attributed to the apparent shortening due to

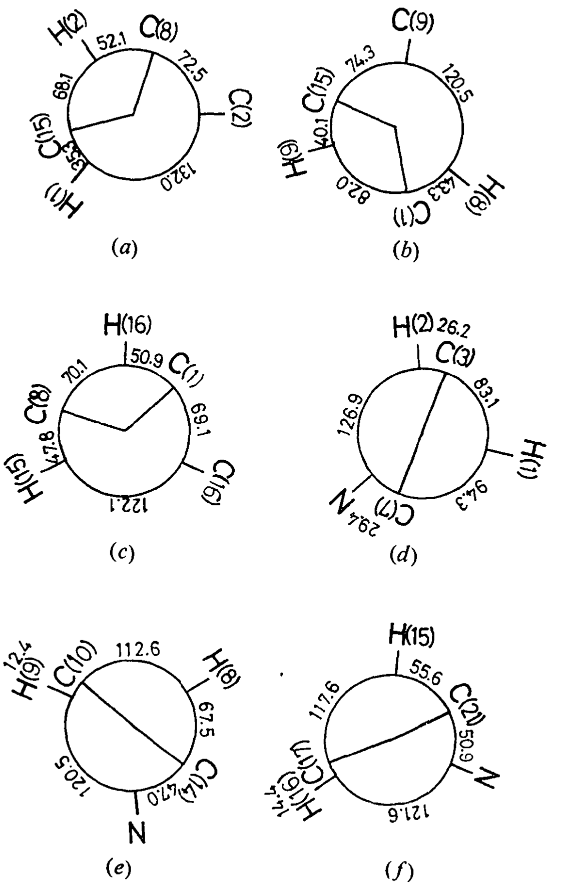


Fig. 4. Conformation angles about the C–N and C–C bonds: (*a*) N–C(1); (*b*) N–C(8); (*c*) N–C(15); (*d*) C(2)–C(1); (*e*) C(9)–C(8); (*f*) C(16)–C(15).

molecular vibration, though the correction of the bond distances for thermal motion was not applied as the TBA molecule cannot be regarded as a rigid body.

A large flexibility of the TBA molecule is consistent with the observation of the n.m.r. line width. The n.m.r. second moment for this substance has been observed to be 6·23 gauss^2 at 29°C, while the calculated value with these molecular dimensions is 7·38 gauss^2 (Aihara & Yokoi, 1972). This discrepancy can obviously be ascribed to the motional narrowing due to large thermal motion.

TBA has been found to form addition compounds or salts with halogens in CCl_4 and $CHCl_3$ solutions. However, because of the steric effects of the bulky substituents in the tertiary amine, it seems unlikely that the halogen molecule in these addition compounds is attached to the nitrogen atom without a change in the molecular conformation of TBA. It is interesting to determine to what extent the conformation in the addition compound is changed from that of TBA obtained in the present study. For this reason the structure analysis of iodine compounds is now in preparation.

Fig. 2 was obtained by using the program *ORTEP* (Johnson, 1965) on the HITAC 5020E Computer at the Computer Centre of the University of Tokyo. All other calculations were performed using the crystallographic computation system UNICS (Sakurai, Ito, Iwasaki, Watanabe & Fukuhara, 1967) on the FACOM 270-30 Computer installed in the Institute of Physical and Chemical Research.

The authors are indebted to Professor Ariyuki Aihara for his continuous interest in the problem.

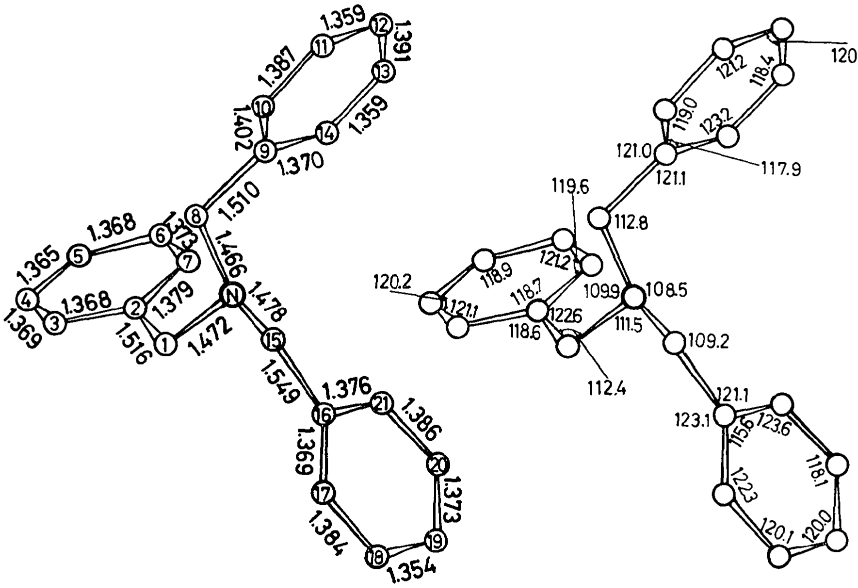


Fig. 5. Bond lengths (Å) and angles (°).

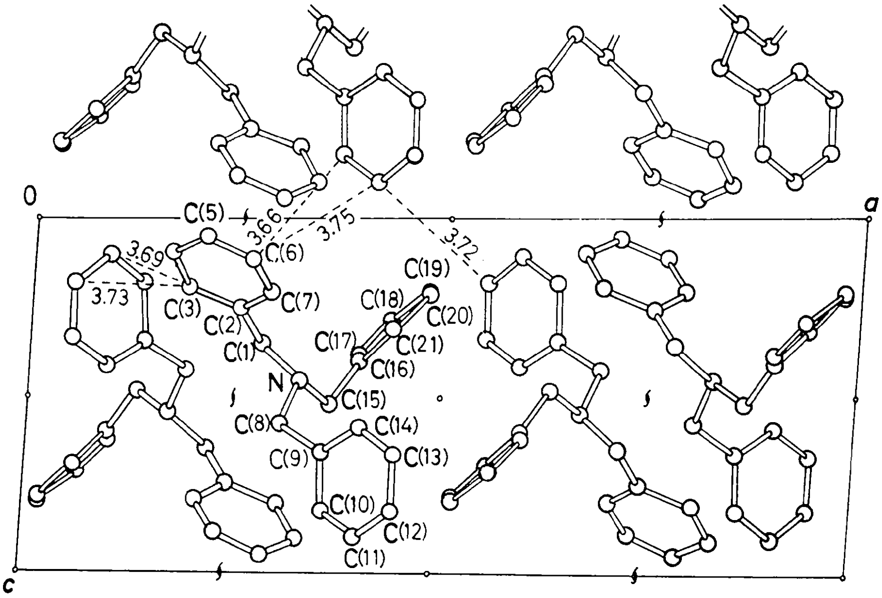


Fig. 6. Projection of the structure along the *b* axis.

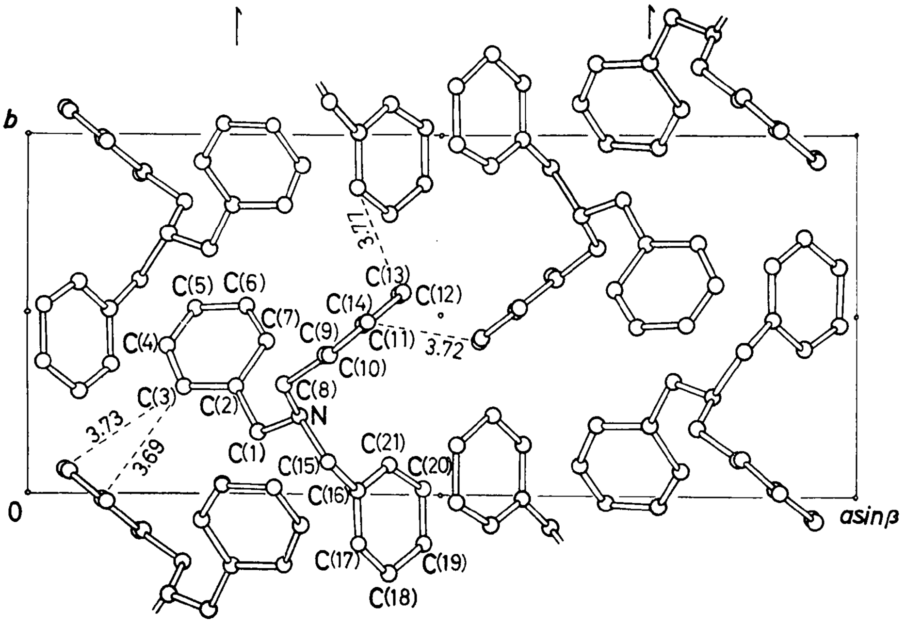


Fig. 7. Projection of the structure along the *c* axis.

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