spond to normal van der Waals distances. The molecular-packing diagram has been deposited.

Acknowledgement is made to the donors of the Petroleum Research Fund, administered by the American Chemical Society, and to the Natural Sciences and Engineering Research Council Canada for financial support. We thank the University of British Columbia Computing Centre for assistance, and J. R. Scheffer, W. K. Appel and L. Walsh for crystals and photochemical results.

References

APPEL, W. K., GREENHOUGH, T. J., SCHEFFER, J. R., TROTTER, J. & WALSH, L. (1980). *J. Am. Chem. Soc.* **102**, 1158–1161.

APPEL, W. K., SCHEFFER, J. R. & WALSH, L. (1979). Unpublished results.

BECKER, P. J. & COPPENS, P. (1974). *Acta Cryst.* A**30**, 129–147.

BECKER, P. J. & COPPENS, P. (1975). *Acta Cryst.* A**31**, 417–425.

CROMER, D. T. & MANN, J. B. (1968). *Acta Cryst.* A**24**, 321–324.

DZAKPASU, A. A., PHILLIPS, S. E. V., SCHEFFER, J. R. & TROTTER, J. (1976). *J. Am. Chem. Soc.* **98**, 6049–6051.

GREENHOUGH, T. J. & TROTTER, J. (1980*a*). *Acta Cryst.* B**36**, 1831–1835.

GREENHOUGH, T. J. & TROTTER, J. (1980*b*). *Acta Cryst.* B**36**, 1835–1839.

GREENHOUGH, T. J. & TROTTER, J. (1980*c*). *Acta Cryst.* B**36**, 2091–2094.

LADD, M. F. C. (1978). Private communication.

MAIN, P., HULL, S. E., LESSINGER, L., GERMAIN, G., DECLERCQ, J. P. & WOOLFSON, M. M. (1978). *MULTAN 78. A System of Computer Programs for the Automatic Solution of Crystal Structures from X-ray Diffraction Data.* Univs. of York, England, and Louvain, Belgium.

PHILLIPS, S. E. V. & TROTTER, J. (1977). *Acta Cryst.* B**33**, 996–1003.

SCHEFFER, J. R. & DZAKPASU, A. A. (1978). *J. Am. Chem. Soc.* **100**, 2163–2173.

SCHEFFER, J. R., JENNINGS, B. M. & LOUWERENS, J. P. (1976). *J. Am. Chem. Soc.* **98**, 7040–7048.

SCHMIDT, G. M. J. (1971). *Pure Appl. Chem.* **27**, 647–678.

STEWART, R. F., DAVIDSON, E. R. & SIMPSON, W. T. (1965). *J. Chem. Phys.* **42**, 3175–3187.

Acta Cryst. (1980). B**36**, 2846–2848

2,2′,4,4′,6,6′-Hexa-*tert*-butylazobenzene*†

BY Y. LE PAGE, E. J. GABE AND YU WANG

Chemistry Division, National Research Council, Ottawa, Canada K1A 0R9

AND L. R. C. BARCLAY AND H. L. HOLM

Department of Chemistry, Mt Allison University, Sackville, New Brunswick, Canada

(*Received* 8 *February* 1980; *accepted* 21 *July* 1980)

Abstract. $C_{36}H_{58}N_2$, orthorhombic, *Pbca*, a = 16·537 (2), b = 17·314 (2), c = 23·074 (3) Å at 115 K, Z = 8, D_{calc} = 1·04 Mg m^{-3}, R_F = 6·3%, wR_F = 3·9%. At room temperature the space group is the same and the cell parameters are a = 16·832 (3), b = 17·343 (2), c = 23·334 (4) Å. The crystals are red-orange rectangular parallelepipeds limited by the forms {100}, {010} and {001}. Considerable distortions in the central part of the molecule are due to stresses provoked by the interaction of terminal methyl groups.

Introduction. Several azobenzene derivatives have been prepared (Holm, 1979). The structures of some of these are determined in this series in order to study the effects of crowding on the geometry of the central part of the molecule. This series is merely a presentation of the structural results while the preparation and the chemical properties of the hindered azobenzenes will be reported separately. The diffraction intensities of a roughly equidimensional sample of approximate diameter 0·18 mm of the title compound were measured. Graphite-monochromatized Cu $K\alpha$ radiation generated at 40 kV and 16 mA was used in a $\theta/2\theta$ scan with line-profile analysis (Grant & Gabe, 1978). One unique set was collected at 115 K up to 100° (2θ) giving 3391 unique measurements leading to 1743 observed [$I_{net} > 2\sigma(I_{net})$] and 1648 unobserved reflections while only 2 out of the 428 measurements of the systematic absences satisfied the observation criterion.

* Structures of Hindered Azobenzenes. I.

† NRC No. 18592.

0567-7408/80/112846-03$01.00

Table 1. *Positional parameters and isotropic thermal parameters*

E.s.d.'s refer to the last digit given.

	x	y	z	B_{iso} (Å^2)*		x	y	z	B_{iso} (Å^2)*
N	0·08980 (22)	0·24071 (22)	0·31284 (16)	1·91 (21)	N′	0·14216 (23)	0·24403 (22)	0·35226 (16)	2·22 (22)
C(1)	0·1241 (3)	0·2341 (3)	0·25453 (19)	1·31 (24)	C(1′)	0·1099 (3)	0·2259 (3)	0·40953 (17)	1·8 (3)
C(2)	0·1900 (3)	0·2766 (3)	0·23110 (19)	1·8 (3)	C(2′)	0·0467 (3)	0·2657 (3)	0·43858 (20)	1·7 (3)
C(3)	0·2270 (3)	0·2468 (3)	0·18128 (19)	1·58 (25)	C(3′)	0·0144 (3)	0·2318 (3)	0·48835 (19)	1·6 (3)
C(4)	0·2013 (3)	0·1795 (3)	0·15351 (19)	2·2 (3)	C(4′)	0·0422 (3)	0·1625 (3)	0·51041 (19)	1·9 (3)
C(5)	0·1301 (3)	0·1466 (3)	0·17360 (20)	1·9 (3)	C(5′)	0·1102 (3)	0·1292 (3)	0·48384 (20)	1·9 (3)
C(6)	0·0884 (3)	0·1739 (3)	0·22321 (18)	1·53 (25)	C(6′)	0·1465 (3)	0·1611 (3)	0·43403 (20)	1·7 (3)
C(7)	0·2168 (3)	0·3579 (3)	0·25178 (21)	2·0 (3)	C(7′)	0·0181 (3)	0·3491 (3)	0·42386 (19)	1·83 (25)
C(7*A*)	0·1406 (3)	0·4073 (3)	0·26209 (21)	2·8 (3)	C(7*A*′)	0·0939 (3)	0·4001 (3)	0·41554 (20)	2·6 (3)
C(7*B*)	0·2715 (3)	0·3580 (3)	0·30570 (21)	2·4 (3)	C(7*B*′)	−0·0386 (3)	0·3514 (3)	0·37046 (21)	2·6 (3)
C(7*C*)	0·2612 (4)	0·4013 (3)	0·20190 (20)	3·3 (3)	C(7*C*′)	−0·0275 (3)	0·3869 (3)	0·47456 (22)	3·0 (3)
C(8)	0·2469 (3)	0·1485 (3)	0·09926 (18)	2·3 (3)	C(8′)	0·0058 (3)	0·1268 (3)	0·56516 (19)	1·8 (3)
C(8*A*)	0·2394 (5)	0·2130 (3)	0·05279 (21)	6·5 (4)	C(8*A*′)	−0·0858 (3)	0·1158 (3)	0·55477 (20)	3·3 (3)
C(8*B*)	0·3341 (4)	0·1354 (4)	0·1143 (3)	6·7 (4)	C(8*B*′)	0·0177 (3)	0·1858 (3)	0·61529 (20)	3·4 (3)
C(8*C*)	0·2092 (3)	0·0749 (4)	0·07728 (22)	4·4 (4)	C(8*C*′)	0·0476 (4)	0·0495 (3)	0·58255 (20)	3·8 (3)
C(9)	0·0064 (3)	0·1362 (3)	0·23853 (19)	1·45 (25)	C(9′)	0·2281 (3)	0·1258 (3)	0·41050 (19)	2·1 (3)
C(9*A*)	−0·0596 (3)	0·1977 (3)	0·24614 (21)	2·8 (3)	C(9*A*′)	0·2929 (3)	0·1879 (3)	0·40975 (20)	2·8 (3)
C(9*B*)	0·0106 (3)	0·0854 (3)	0·29361 (21)	2·5 (3)	C(9*B*′)	0·2555 (3)	0·0589 (3)	0·45031 (21)	3·5 (3)
C(9*C*)	−0·0230 (3)	0·0831 (3)	0·18792 (22)	3·2 (3)	C(9*C*′)	0·2185 (3)	0·0888 (3)	0·34968 (21)	3·2 (3)
H(3)	0·279 (3)	0·2737 (25)	0·1660 (16)	4·8 (13)	H(3′)	−0·0214 (23)	0·2556 (24)	0·5078 (16)	2·8 (11)
H(5)	0·097 (3)	0·1030 (23)	0·1543 (16)	4·5 (13)	H(5′)	0·1325 (22)	0·0847 (22)	0·5040 (16)	2·6 (11)
H(7*A*)*A*	0·1586 (24)	0·4586 (22)	0·2725 (15)	3·1 (12)	H(7*A*)*A*′	0·1285 (19)	0·3828 (18)	0·3829 (14)	0·6 (9)
H(7*A*)*B*	0·1080 (22)	0·4181 (21)	0·2292 (14)	2·0 (10)	H(7*A*)*B*′	0·0748 (23)	0·4567 (21)	0·4031 (14)	1·8 (10)
H(7*A*)*C*	0·1056 (20)	0·3850 (18)	0·2897 (13)	0·9 (9)	H(7*A*)*C*′	0·124 (3)	0·401 (3)	0·4451 (19)	6·2 (14)
H(7*B*)*A*	0·2934 (21)	0·4066 (22)	0·3105 (15)	2·3 (10)	H(7*B*)*A*′	−0·0585 (24)	0·4038 (22)	0·3621 (16)	3·7 (12)
H(7*B*)*B*	0·3188 (23)	0·3253 (22)	0·2941 (15)	3·1 (11)	H(7*B*)*B*′	−0·0079 (23)	0·3384 (23)	0·3351 (16)	3·2 (11)
H(7*B*)*C*	0·243 (3)	0·336 (3)	0·3388 (17)	6·3 (14)	H(7*B*)*C*′	−0·093 (4)	0·303 (3)	0·3875 (25)	13·7 (23)
H(7*C*)*A*	0·2298 (23)	0·3944 (22)	0·1603 (15)	3·0 (11)	H(7*C*)*A*′	0·007 (3)	0·381 (3)	0·5174 (19)	6·4 (15)
H(7*C*)*B*	0·3137 (21)	0·3877 (19)	0·2080 (15)	1·8 (10)	H(7*C*)*B*′	−0·0783 (20)	0·3631 (19)	0·4762 (13)	0·3 (9)
H(7*C*)*C*	0·272 (3)	0·4504 (24)	0·2166 (16)	5·0 (13)	H(7*C*)*C*′	−0·0366 (24)	0·4389 (24)	0·4654 (16)	4·0 (13)
H(8*A*)*A*	0·2552 (25)	0·1929 (22)	0·0230 (14)	2·7 (11)	H(8*A*)*A*′	−0·1046 (22)	0·0887 (22)	0·5856 (14)	2·4 (11)
H(8*A*)*B*	0·272 (4)	0·267 (4)	0·063 (3)	16·7 (26)	H(8*A*)*B*′	−0·089 (3)	0·078 (3)	0·5251 (18)	6·4 (15)
H(8*A*)*C*	0·169 (3)	0·183 (3)	0·0488 (22)	10·8 (20)	H(8*A*)*C*′	−0·125 (4)	0·171 (4)	0·553 (3)	14·3 (24)
H(8*B*)*A*	0·362 (3)	0·111 (3)	0·0812 (20)	7·4 (16)	H(8*B*)*A*′	0·0005 (21)	0·1652 (20)	0·6435 (14)	1·4 (9)
H(8*B*)*B*	0·331 (3)	0·088 (3)	0·1473 (18)	6·0 (15)	H(8*B*)*B*′	0·082 (3)	0·190 (3)	0·6194 (20)	7·0 (15)
H(8*B*)*C*	0·362 (3)	0·176 (3)	0·1327 (19)	6·6 (15)	H(8*B*)*C*′	−0·019 (3)	0·240 (4)	0·6097 (23)	11·6 (20)
H(8*C*)*A*	0·2440 (23)	0·0591 (20)	0·0440 (13)	1·5 (9)	H(8*C*)*A*′	0·0152 (21)	0·0317 (19)	0·6131 (14)	1·0 (9)
H(8*C*)*B*	0·146 (3)	0·082 (3)	0·0670 (19)	6·2 (14)	H(8*C*)*B*′	0·0414 (22)	0·0119 (21)	0·5505 (15)	2·4 (11)
H(8*C*)*C*	0·213 (3)	0·024 (3)	0·1151 (19)	5·7 (14)	H(8*C*)*C*′	0·121 (3)	0·074 (4)	0·5972 (22)	13·0 (24)
H(9*A*)*A*	−0·1103 (24)	0·1762 (22)	0·2497 (16)	3·5 (11)	H(9*A*)*A*′	0·3440 (19)	0·1647 (19)	0·3960 (12)	0·2 (8)
H(9*A*)*B*	−0·0660 (21)	0·2273 (21)	0·2151 (14)	1·8 (10)	H(9*A*)*B*′	0·2973 (24)	0·2059 (23)	0·4492 (16)	4·3 (13)
H(9*A*)*C*	−0·0537 (21)	0·2293 (21)	0·2754 (14)	1·8 (10)	H(9*A*)*C*′	0·274 (3)	0·236 (3)	0·3691 (23)	10.0 (18)
H(9*B*)*A*	−0·0368 (21)	0·0573 (20)	0·3000 (14)	1·7 (10)	H(9*B*)*A*′	0·3095 (21)	0·0499 (20)	0·4388 (15)	1·6 (10)
H(9*B*)*B*	0·0491 (21)	0·0467 (20)	0·2858 (14)	1·4 (10)	H(9*B*)*B*′	0·2669 (23)	0·0807 (22)	0·4908 (14)	2·5 (10)
H(9*B*)*C*	0·0247 (21)	0·1171 (19)	0·3293 (15)	1·9 (10)	H(9*B*)*C*′	0·217 (4)	0·007 (4)	0·455 (3)	16·6 (27)
H(9*C*)*A*	0·0131 (23)	0·0430 (21)	0·1843 (16)	2·6 (11)	H(9*C*)*A*′	0·267 (3)	0·054 (3)	0·3440 (16)	5·9 (14)
H(9*C*)*B*	−0·021 (3)	0·1169 (24)	0·1546 (18)	5·0 (14)	H(9*C*)*B*′	0·2115 (20)	0·1295 (20)	0·3201 (14)	1·7 (10)
H(9*C*)*C*	−0·0680 (22)	0·0633 (21)	0·1974 (14)	2·1 (10)	H(9*C*)*C*′	0·172 (3)	0·050 (3)	0·3509 (17)	5·3 (14)

* For non-hydrogen atoms $B_{iso} = 8\pi^2(U_{11}^2 + U_{22}^2 + U_{33}^2)^{1/2}$; for H atoms $B_{iso} = 8\pi^2 U$.

No absorption correction was performed ($\mu = 0{\cdot}412$ mm^{-1}).

The positions of the non-hydrogen atoms were obtained with *MULTAN* (Germain, Main & Woolfson, 1971) on a room-temperature data set. The terminal *tert*-butyl groups are disordered at room temperature. The temperature was lowered to 115 K and no disorder was then observed. The H atoms were located on difference maps and their positional parameters were refined together with individual isotropic thermal parameters while the other atoms were refined anisotropically by block-diagonal least squares using counting-statistics weights.

The scattering curves were taken from Cromer & Waber (1974).

The final residuals are $R_F = 6{\cdot}3\%$ and $wR_F = 3{\cdot}9\%$. The atomic positional parameters are listed in Table 1.* All the calculations were performed using the NRC system of programs for the PDP8-E computer (Larson & Gabe, 1978).

* Lists of structure factors, thermal parameters and bond distances and angles have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 35499 (30 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

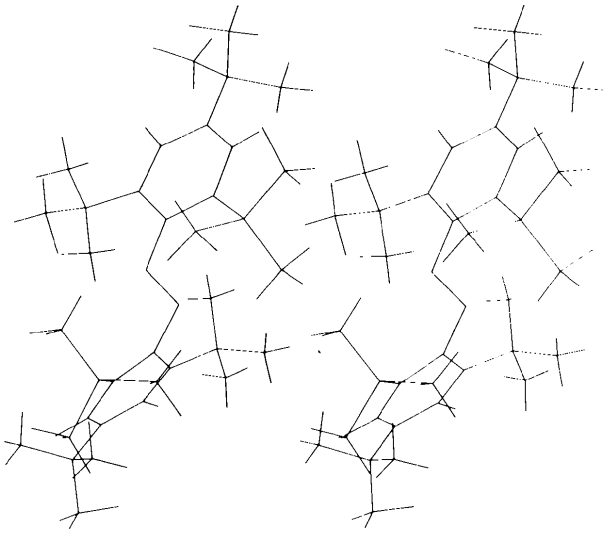


Fig. 1. Stereoscopic view of the skeleton of the molecule.

Discussion. The molecule (Fig. 1) extends approximately along *z*. The interatomic distances and angles, except those involving H atoms, are shown in Fig. 2. The angle between the mean planes of the benzene rings (68·7°) minimizes interference between the *tert*-butyl groups on C(2) and C(6) with those on C(2′) and C(6′) but, as would be expected, there are stresses in the molecule. The benzene rings are significantly non-planar with C(1) displaced 0·16 Å out of the plane of C(2), C(3), C(4), C(5) and C(6), and N displaced 0·57 (σ = 0·01) Å. The distances for the corresponding primed atoms are 0·12 and 0·40 Å. Likewise, the angles involving the C–N bonds are significantly different from 120°, with N–C(1)–C(2) and N–C(1)–C(6) being respectively 127·2 and 111·7°. Again, the values for the primed atoms are 126·3 and 112·4°. The C(1)–N–N′–C(1′) torsion angle is −161·5°.

While the central portion of the molecule is tightly packed, the intermolecular forces must be quite small, as shown by the fact that the *tert*-butyl groups on C(4) and C(4′) are disordered at room temperature, but become ordered at low temperature.

A comparative study of the geometry of the hindered azobenzenes will be reported separately.

References

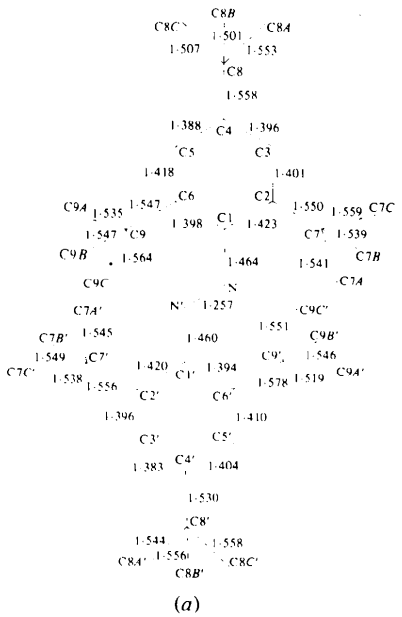
CROMER, D. T. & WABER, J. T. (1974). *International Tables for X-ray Crystallography*, Vol. IV, p. 99. Birmingham: Kynoch Press.

GERMAIN, G., MAIN, P. & WOOLFSON, M. M. (1971). *Acta Cryst.* A**27**, 368–376.

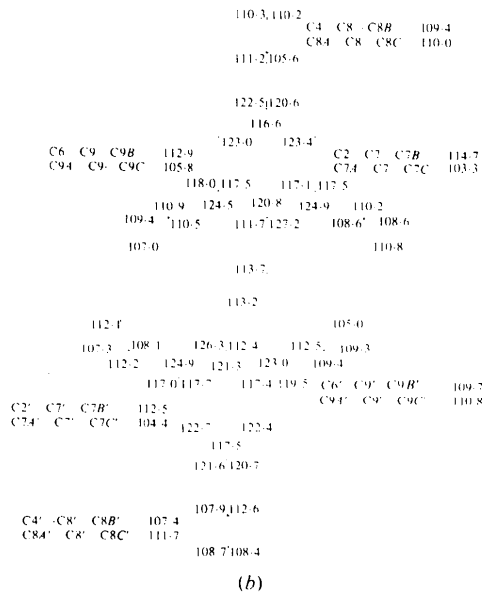
GRANT, D. F. & GABE, E. J. (1978). *J. Appl. Cryst.* **11**, 114–120.

HOLM, H. L. (1979). *Hindered Azobenzenes – Photochemistry and Structure*, BSc Honours Thesis, Mt Allison Univ., Canada.

LARSON, A. C. & GABE, E. J. (1978). *Computing in Crystallography*, edited by H. SCHENK *et al.*, p. 81. Delft Univ. Press.



(*a*)



(*b*)

Fig. 2. Schematic drawing of the molecule showing (*a*) distances (Å), (*b*) angles (°). The σ's on all the distances are 0·007 Å and on all the angles 0·4°.