final $R = \sum||F_o| - |F_c||/\sum|F_o| = 0{\cdot}048$ for the 2093 observed reflexions. The final coordinates are given in Table 2. Fig. 1 gives bond lengths and angles and the atom numbering. Fig. 2 shows the packing.*

Discussion. Compared to the corresponding base, the main differences observed are the inversion of the oxyimino and carboxyl groups by half-rotation around C(5)–C(7) (Table 3) and the coplanarity of the three groups aminothiazolyl, oxyimino and carboxyl (Table 4). This coplanarity allows the formation of an intramolecular hydrogen bond between the protonated N(4) of the thiazolyl ring and O(9) of the oxyimino group; moreover an interaction [C(6)–H(61)···O(12)] occurs between the thiazolyl and carbonyl groups. This conformation leads to a close approach of O(13) and N(8), 2·548 (5) compared to 2·90 Å for the sum of the van der Waals radii (Pauling, 1960).

Each molecule is bonded to two neighbours by N–H···Br⁻ bonds: N(3)···$\text{Br}^-(x, -y + \frac{3}{2}, z - \frac{1}{2})$ 3·272 (5), N(3)···$\text{Br}^-(-x + 1, -y + 1, -z + 1)$ 3·333 (5) Å.

We thank Dr J. Perronnet (Roussel–Uclaf, Romainville, France) for providing the sample.

Table 4. *Deviations from coplanarity and angles between planes*

Deviations (Å)

(1) Plane through S(1), C(2), N(3), N(4), C(5) and C(6): aminothiazolyl group

S(1)	−0·000	N(4)	−0·005
C(2)	0·005	C(5)	0·005
N(3)	−0·001	C(6)	−0·003

(e.s.d. 0·004 Å)

(2) Plane through C(5), C(7), N(8), O(9) and C(11): oxyimino group

C(5)	−0·004	O(9)	−0·002
C(7)	0·015	C(11)	−0·005
N(8)	−0·003		

(e.s.d. 0·008 Å)

(3) Plane through C(7), C(11), O(12) and O(13): carboxyl group

C(7)	−0·001	O(12)	−0·002
C(11)	0·005	O(13)	−0·002

(e.s.d. 0·003 Å)

Angles (°)

Planes	This work	Corresponding base
1–2	0·6	13·4
2–3	5·5	77·1
1–3	5·3	82·3

(e.s.d. <0·3°)

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* Lists of structure factors and anisotropic thermal parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 35828 (11 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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2,2′,4,4′,6,6′-Hexaisopropylazobenzene*†

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Abstract. $C_{30}H_{46}N_2$, orthorhombic, *Pbnn*, $a = 5{\cdot}9952$ (6), $b = 19{\cdot}630$ (2), $c = 23{\cdot}929$ (2) Å at 220 K giving $Z = 4$ and $D_c = 1{\cdot}03$ Mg m^{-3}. At room temperature, the space group is the same and the cell parameters are approximately 6·016, 19·68 and 24·08 Å. At 115 K the sample is a monoclinic twin with $\gamma = 90{\cdot}2°$. The transition temperature of this reversible transformation is about 200 K. The crystals are yellow to orange needles elongated parallel to **a** and limited by the {010} and {001} forms. A twofold axis relates the two parts of the molecule which extends

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* Structures of Hindered Azobenzenes. II.

† NRC No. 19079.

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along z. The final R_F value is 6·6% for 898 observed data.

Introduction. The structures of a series of hindered azobenzenes have been determined (Le Page, Gabe, Wang, Barclay & Holm, 1980). The diffraction intensities of a rectangular parallelepiped crystal, 0·15 × 0·20 × 0·35 mm, of the title compound were measured.

Graphite-monochromatized Cu $K\alpha$ radiation generated at 40 kV and 20 mA was used in a $\theta/2\theta$ scan with line-profile analysis (Grant & Gabe, 1978). One unique set was collected up to 110° 2θ giving 2114 unique measurements leading to 898 observed [I net $> 3\sigma(I$ net$)$] and 888 unobserved reflections while only 1 out of the remaining 328 measurements of the systematic absences satisfied the observation criterion. No absorption correction was performed ($\mu = 0{\cdot}445$ mm^{-1}).

Table 1. *Atomic parameters x,y,z and $B_{\rm iso}$*

The equivalent positions in *Pbnn* are $\pm(x,y,z;\ -x,\ \tfrac{1}{2}+y,\ z;\ \tfrac{1}{2}+x,\ -y,\ \tfrac{1}{2}+z;\ \tfrac{1}{2}-x,\ \tfrac{1}{2}-y,\ \tfrac{1}{2}+z)$. E.s.d.'s refer to the last digit printed. $B_{\rm iso}$ is the arithmetic mean of the principal axes of the thermal ellipsoid.

	x	y	z	$B_{\rm iso}$ (Å^2)
N	0·8393 (5)	0·12350 (13)	0·23641 (12)	3·57 (17)
C(1)	0·8054 (6)	0·12013 (18)	0·17629 (13)	3·56 (19)
C(2)	0·9326 (7)	0·16871 (18)	0·14540 (16)	4·54 (22)
C(3)	0·9088 (8)	0·16626 (21)	0·08785 (16)	6·0 (3)
C(4)	0·7801 (8)	0·11813 (23)	0·06137 (15)	6·5 (3)
C(5)	0·6679 (7)	0·07047 (21)	0·09239 (15)	5·5 (3)
C(6)	0·6752 (6)	0·06979 (17)	0·15129 (14)	3·77 (20)
C(7)	1·0723 (7)	0·22222 (20)	0·17497 (16)	5·48 (24)
C(71)	1·2921 (7)	0·23524 (22)	0·14406 (21)	7·8 (3)
C(72)	0·9427 (8)	0·28807 (24)	0·18257 (20)	8·7 (3)
C(8)	0·7560 (8)	0·1196 (3)	−0·00313 (17)	10·9 (4)
C(81)	0·5582 (11)	0·1547 (3)	−0·02133 (18)	12·5 (5)
C(82)	0·9249 (12)	0·0897 (3)	−0·03226 (19)	13·5 (5)
C(9)	0·5564 (6)	0·01390 (18)	0·18258 (15)	4·18 (21)
C(91)	0·3035 (7)	0·01894 (22)	0·17465 (18)	6·5 (3)
C(92)	0·6390 (7)	−0·05672 (19)	0·16495 (18)	7·0 (3)
H(7)	1·119 (5)	0·2058 (13)	0·2138 (11)	5·0 (9)
H(711)	1·356 (5)	0·2631 (15)	0·1664 (12)	6·3 (10)
H(712)	1·351 (6)	0·1862 (17)	0·1310 (15)	10·4 (13)
H(713)	1·265 (7)	0·2746 (19)	0·1059 (17)	13·5 (15)
H(721)	0·803 (5)	0·2734 (15)	0·2002 (13)	8·1 (11)
H(722)	0·908 (6)	0·3045 (17)	0·1454 (16)	11·8 (14)
H(723)	1·059 (9)	0·3149 (21)	0·2069 (18)	18·8 (20)
H(823)	0·902 (6)	0·0893 (15)	−0·0743 (14)	9·0 (12)
H(811)	0·480 (9)	0·1905 (21)	−0·0036 (22)	18·8 (19)
H(812)	0·550 (6)	0·1167 (17)	−0·0144 (14)	9·8 (13)
H(813)	0·561 (8)	0·1592 (19)	−0·0540 (18)	15·4 (17)
H(821)	1·054 (7)	0·0569 (19)	−0·0177 (16)	13·1 (15)
H(822)	0·751 (7)	0·0372 (20)	−0·0184 (17)	16·0 (17)
H(8)	0·964 (11)	0·167 (3)	−0·0229 (24)	28·3 (29)
H(9)	0·594 (5)	0·0190 (14)	0·2236 (11)	6·8 (10)
H(911)	0·232 (6)	−0·0271 (17)	0·1907 (14)	10·8 (12)
H(912)	0·261 (5)	0·0150 (15)	0·1357 (13)	7·8 (10)
H(913)	0·267 (7)	0·0728 (16)	0·1907 (15)	12·4 (14)
H(921)	0·560 (8)	−0·0973 (19)	0·1961 (17)	15·2 (16)
H(922)	0·792 (5)	−0·0576 (13)	0·1752 (11)	5·2 (9)
H(923)	0·649 (7)	−0·0704 (19)	0·1291 (17)	14·6 (17)
H(3)	1·006 (6)	0·2065 (14)	0·0667 (12)	5·6 (9)
H(5)	0·577 (5)	0·0310 (13)	0·0769 (10)	4·0 (8)

The 16 non-hydrogen atoms were obtained with *MULTAN* (Germain, Main & Woolfson, 1971). The H atoms were located on difference maps and their positional parameters were refined together with iso-

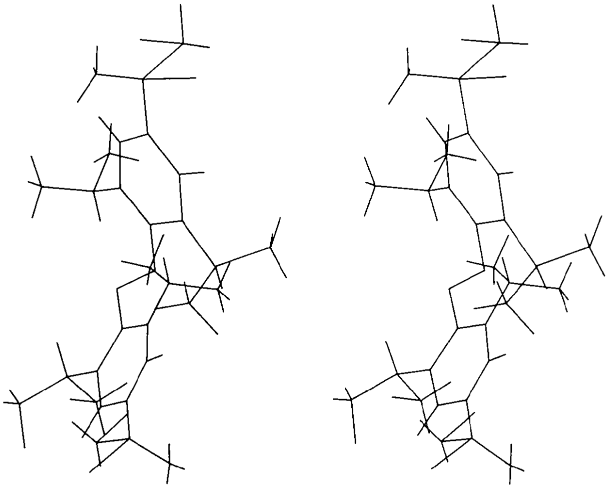

Fig. 1. Stereoscopic view of the skeleton of the molecule.

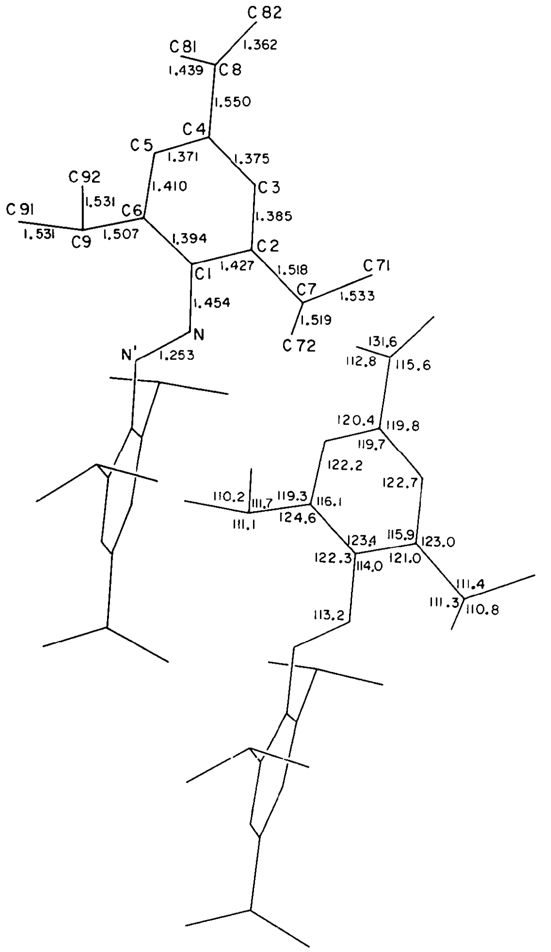

Fig. 2. Perspective drawings showing the distances (Å) and angles (°). The standard deviations are 0·006 Å and 0·4°.

tropic thermal parameters while the other atoms were refined anisotropically by block-diagonal least-squares calculations using counting-statistics weights. The scattering factors from Cromer & Waber (1974) were used. The final residuals are $R_F = 6{\cdot}6\%$ and $wR_F = 4{\cdot}4\%$. 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).

Discussion. The molecule (Fig. 1) extends along z while the two parts of the dimer are related by the twofold axis parallel to y and passing through the middle of the N=N bond at $x = z = \frac{1}{4}$. The interatomic distances and angles except those involving H atoms are shown in Fig. 2. The high thermal motion observed at the C(81) and C(82) positions probably indicates some degree of disorder at these positions. Consequently the H atoms bonded to C(8), C(81) and C(82) are not considered to be located with certainty. Their refined positions are nevertheless given in Table 1. The average C—H distance is 1·05 Å.

The 76·2° angle between the planes of the benzene rings is such that little interference occurs between the isopropyl groups on C(2) and C(6) and their equivalents on the symmetry-related part of the dimer. Consequently, contrary to what is observed in the hexa-*tert*-butyl derivative, the C(1) and N atoms lie approximately in the plane defined by the C(2), C(3), C(4), C(5) and C(6) atoms.

The torsion angle between the N—C(1) and N′—C(1′) bonds is 174·3°. A comparative study of the geometry of hindered azobenzenes will be reported separately.

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* The tables of anisotropic thermal parameters, observed and calculated structure factors and distances and angles in the molecule have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 35856 (16 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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2,2′,4,4′,6,6′-Hexamethylazobenzene*†

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Abstract. $C_{18}H_{22}N_2$, monoclinic, $P2_1/c$, $a = 4{\cdot}870$ (2), $b = 10{\cdot}017$ (2), $c = 16{\cdot}431$ (4) Å, $\beta = 108{\cdot}05$ (2)° at room temperature, giving $Z = 2$ and $D_c = 1{\cdot}16$ Mg m^{-3}. The molecule is planar, utilizes a symmetry center and is relatively undistorted. The final R_F value is 3·8% for 708 observed data.

Introduction. The structures of a series of hindered azobenzenes have been determined (Le Page, Gabe, Barclay & Dust, 1981). The diffraction intensities of a crystal, 0·3 × 0·3 × 0·3 mm, of the title compound were measured with graphite-monochromatized Cu $K\alpha$ radiation using profile analysis (Grant & Gabe, 1978) with the $\theta/2\theta$ scan technique. Three unique sets of 776 reflections for $2\theta \leq 100°$ were collected and averaged to give 708 with significant and 68 with insignificant

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* Structure of Hindered Azobenzenes. III.

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