6,6'-Dibromo-2,2',4,4'-tetra-*tert*-butylazobenzene*†

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Abstract. $C_{28}H_{40}Br_2N_2$, orthorhombic, *Pbcn*, $a = 12\cdot891$ (2), $b = 11\cdot277$ (2), $c = 20\cdot039$ (5) Å at room temperature, giving $Z = 4$ and $D_c = 1\cdot29$ Mg m^{-3}. The crystals are rectangular orange-red (010) tablets. The refinement of 1759 observed reflections gave $R_F = 0\cdot035$. A twofold axis passes through the center of the N=N bond. The Br atom is 0·22 Å out of the plane of a reasonably planar benzene ring.

Introduction. The structures of a series of hindered azobenzenes have been determined (Gabe, Wang, Barclay & Dust, 1981).

The diffraction intensities of a crystal, 0·21 × 0·36 × 0·35 mm, of the title compound were measured with graphite-monochromatized Cu $K\alpha$ radiation generated at 40 kV, 10 mA. Line-profile analysis (Grant & Gabe, 1978) of the $\theta/2\theta$ scan was used. One unique set was collected up to 110° 2θ giving 1831 reflections of which 1759 were considered to be observed [$I_{net} > 2\sigma(I_{net})$] while 72 reflections were unobserved. An absorption correction using Gaussian integration was applied ($\mu = 4\cdot03$ mm^{-1}).

The position of the Br atom was found from a Patterson map. The remaining 14 non-hydrogen atoms

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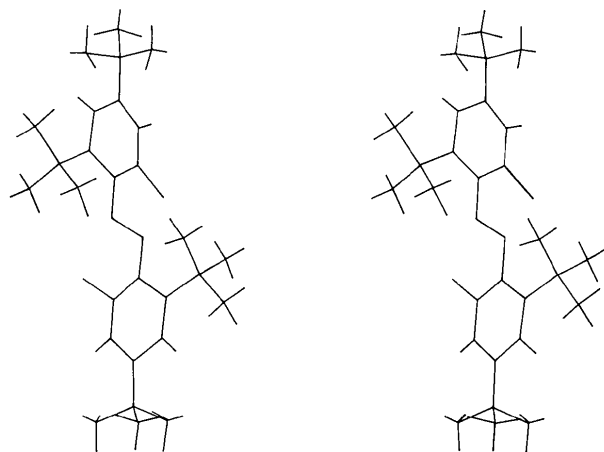* Structures of Hindered Azobenzenes. IV.
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Fig. 1. Stereoscopic view of the skeleton of the molecule along the y direction. A twofold axis in the middle of the N—N bond relates the two halves of the molecule.

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

E.s.d.'s refer to the last digit printed. B_{iso} is the arithmetic mean of the principal axes of the thermal ellipsoid.

	x	y	z	B_{iso} (Å²)
Br	0·09488 (3)	0·10360 (4)	0·165380 (21)	5·512 (22)
N	−0·04589 (17)	0·29859 (20)	0·23973 (12)	3·04 (12)
C(1)	−0·05435 (23)	0·2954 (3)	0·16872 (14)	2·87 (14)
C(2)	−0·13168 (23)	0·3664 (3)	0·13813 (16)	3·12 (15)
C(3)	−0·1395 (3)	0·3601 (3)	0·06956 (16)	3·76 (17)
C(4)	−0·07975 (24)	0·2871 (3)	0·02931 (15)	3·72 (17)
C(5)	−0·0086 (3)	0·2153 (3)	0·06058 (15)	3·74 (17)
C(6)	0·00370 (24)	0·2194 (3)	0·12875 (15)	3·23 (15)
C(7)	−0·20498 (25)	0·4461 (3)	0·17955 (16)	3·78 (17)
C(8)	−0·0969 (3)	0·2840 (4)	−0·04653 (16)	5·07 (20)
C(9)	−0·1455 (3)	0·5303 (3)	0·22460 (18)	4·97 (20)
C(10)	−0·2750 (3)	0·5227 (4)	0·13500 (19)	5·78 (22)
C(11)	−0·2763 (3)	0·3662 (3)	0·22139 (19)	5·32 (22)
C(12)	−0·1120 (4)	0·4071 (5)	−0·07413 (21)	8·9 (3)
C(13)	−0·1948 (4)	0·2058 (5)	−0·05979 (21)	8·7 (3)
C(14)	−0·0068 (4)	0·2203 (4)	−0·08319 (21)	9·2 (3)
H(3)	−0·1850 (17)	0·4048 (20)	0·0513 (11)	2·2 (6)
H(5)	0·0264 (21)	0·1538 (24)	0·0379 (13)	4·4 (7)
H(91)	−0·0992 (20)	0·5763 (24)	0·2072 (14)	4·5 (7)
H(92)	−0·1867 (20)	0·5842 (23)	0·2465 (15)	4·9 (7)
H(93)	−0·1063 (20)	0·492 (3)	0·2613 (16)	6·0 (8)
H(101)	−0·3212 (23)	0·474 (3)	0·1067 (15)	6·2 (8)
H(102)	−0·2379 (22)	0·572 (3)	0·1053 (13)	5·3 (8)
H(103)	−0·320 (3)	0·572 (3)	0·1639 (17)	8·0 (11)
H(111)	−0·3129 (24)	0·318 (3)	0·1915 (15)	6·7 (9)
H(112)	−0·3231 (23)	0·408 (3)	0·2454 (17)	6·5 (9)
H(113)	−0·2423 (22)	0·3210 (25)	0·2539 (15)	5·7 (8)
H(121)	−0·175 (3)	0·432 (3)	−0·0580 (17)	8·7 (11)
H(122)	−0·045 (3)	0·442 (3)	−0·0678 (17)	7·9 (10)
H(123)	−0·1170 (25)	0·400 (3)	−0·1187 (19)	7·3 (10)
H(131)	−0·2369 (23)	0·261 (3)	−0·0473 (13)	5·6 (8)
H(132)	−0·160 (3)	0·133 (4)	−0·0467 (23)	13·7 (15)
H(133)	−0·201 (3)	0·220 (4)	−0·1134 (22)	13·1 (15)
H(141)	0·0339 (22)	0·2983 (24)	−0·0763 (14)	5·4 (8)
H(142)	−0·016 (4)	0·129 (3)	−0·0658 (22)	12·5 (13)
H(143)	−0·024 (3)	0·237 (3)	−0·1315 (19)	10·7 (13)

were located on successive cycles of structure-factor and difference-map calculations. The R_F value was reduced to 0·086 when a difference map showed the 20 H atoms. Final cycles of least squares in which all parameters were refined keeping the H atoms isotropic gave $R_F = 0\cdot035$ and $wR_F = 0\cdot035$. The atomic positional parameters are listed in Table 1.‡

The scattering curves for neutral atoms from *International Tables for X-ray Crystallography* (1974) were used. All the calculations were performed using the NRC system of programs for the PDP8-E computer (Larson & Gabe, 1978).

Discussion. The two halves of the molecule (Fig. 1) are related by a twofold axis along *y*. The distances and angles are shown on Fig. 2.

The distances in the molecule are quite regular but some angles in the benzene ring are notably different from 120°, especially C(2)—C(3)—C(4) with a value of 124·7 (3) and C(1)—C(2)—C(3) with 116·9 (3)°. The Br atom is displaced 0·22 Å out of the plane of the benzene ring. The ring itself is reasonably planar, atoms C(1) to C(6) being respectively 0·021, −0·018, 0·001, 0·013, −0·010 and −0·007 Å ($\sigma = 0.003$ Å for all distances) out of their least-squares plane. The angle between the planes of the rings is 92·7 (6)°. The C(1)—N(1)—N(1')—C(1') torsion angle is 176·9 (6)°.

A comprehensive study of the molecular geometries of the hindered azobenzenes will be reported separately.

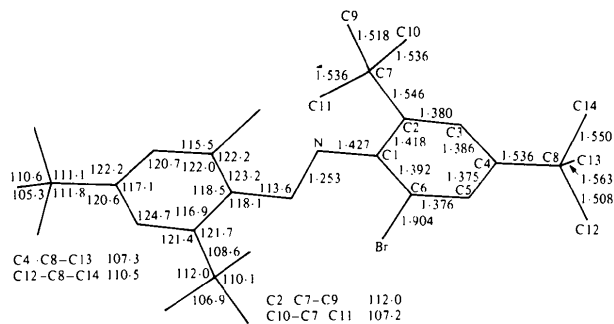We thank Professor H. Rau of the Universität Hohenheim, Germany, for supplying the crystals.

Fig. 2. Distances (Å) and angles (°) in the molecule. The e.s.d. on the C(6)—Br distance is 0·003 Å while it is 0·006 Å on the other distances shown, and 0·3° on the angles shown. The C—H bond lengths range from 0·86 (3) to 1·10 (4) Å.

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N-[(Ethyl-1 pyrrolidinyl-2) méthyl] Méthoxy-2 Sulfamoyl-5 Benzamide (Sulpiride)

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Abstract. $C_{15}H_{23}N_3O_4S$, triclinic, $P\bar{1}$, $Z = 2$, $a = 11·173$ (2), $b = 9·082$ (2), $c = 11·295$ (2) Å, $\alpha = 90·5$ (1), $\beta = 130·9$ (1), $\gamma = 100·1$ (1)°; $D_c = 1·34$, $D_m = 1·34$ Mg m^{-3}. The structure was solved by direct methods and refined by a full-matrix least-squares procedure to a final R value of 0·077 for 1622 observed reflexions using Cu $K\alpha$ radiation. The molecule of sulpiride is in extended configuration. A partial disorder was observed around the amide function. Hydrogen bonds occur between the H atoms of the sulfamoyl group and the amide function and the pyrrolidinyl group of two different molecules.