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3-*tert*-Butyl-2-hydroxy-5-methoxyazobenzene†

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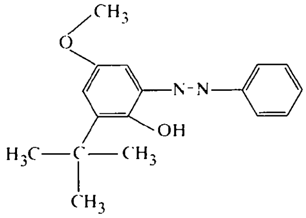
Abstract

The structure of the title compound, $C_{17}H_{20}N_2O_2$, shows features characteristic of azobenzene derivatives and is perfectly planar in the solid state with a *trans* configuration. The molecule has an intramolecular O1—H1···N1 hydrogen bond.

Comment

Azo compounds are widely used in the textile industry as synthetic colouring materials. The structure of the title compound, (I), is very similar to the structures of azo compounds studied previously (Işik *et al.*, 1997; Glowka & Olubek, 1994; Rodrigues *et al.*, 1996). The two phenyl rings are in a *trans* configuration. The lengths of the two C—N bonds are almost identical and have an average value of 1.410 (3) Å. The average value of the C—N—N angles is 115.7 (2)°. The N═N bond length is 1.274 (3) Å, in accordance with the expected value for aromatic azo compounds. The whole molecule, except for two methyls of the *tert*-butyl group, lies on a crystallographic mirror plane, so that the molecule is perfectly planar.



(I)

The planarity of the molecule facilitates an O1—H1···N1 intramolecular hydrogen bond, in which the O1 hydroxyl group adjacent to the N═N bond serves as the donor to the N1 atom. The O1—H1···N1 angle is 149.6 (2)° and the length of the hydrogen bond (H1···N1) is 1.598 (2) Å. The length of the O1···N1 bond is 2.534 (2) Å, which is shorter than a typical hydrogen bond of this type [N···O = 2.78 (10) Å; Vinogradov & Linnell, 1971]. This shows the presence of a very strong intramolecular interaction in the molecule. There are no intermolecular interactions other

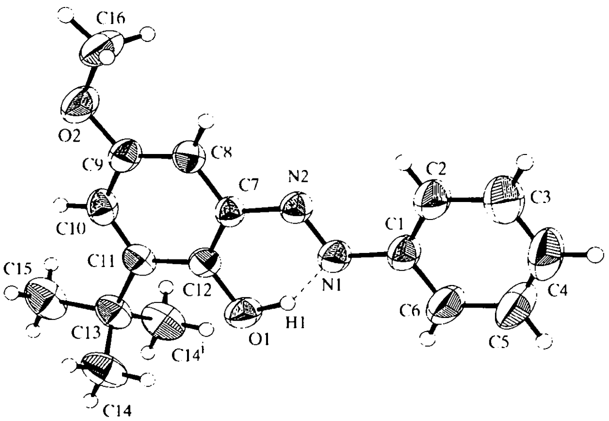


Fig. 1. *ORTEP*II (Johnson, 1976) view of the title molecule and the atomic numbering. The displacement ellipsoids are at the 50% probability level. H atoms are shown as spheres of arbitrary size. [Symmetry code: (i) $x, \frac{1}{2} - y, z$.]

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† IUPAC name: 2-*tert*-butyl-4-methoxy-6-(phenyldiazenyl)phenol.

than van der Waals contacts between the molecules, which are therefore present as isolated individuals within the crystal.

Experimental

The title compound was synthesized according to Kocaokutgen (1995). It was dissolved in hot glacial acetic acid and then left for two days at room temperature, after which time crystals were obtained.

Crystal data

$C_{17}H_{20}N_2O_2$
$M_r = 284.35$
Orthorhombic
Pnma
$a = 12.984\ (2)$ Å
$b = 6.893\ (1)$ Å
$c = 17.224\ (2)$ Å
$V = 1541.5\ (4)$ Å^3
$Z = 4$
$D_x = 1.225$ Mg m^{-3}
$D_m = 1.233$ Mg m^{-3}
D_m measured by pycnometry

Mo $K\alpha$ radiation
$\lambda = 0.71069$ Å
Cell parameters from 25 reflections
$\theta = 8.64–18.29°$
$\mu = 0.081$ mm^{-1}
$T = 293\ (2)$ K
Prismatic
0.26 × 0.25 × 0.15 mm
Dark brown

Data collection

Enraf–Nonius CAD-4 diffractometer
$\omega/2\theta$ scans
Absorption correction: none
2321 measured reflections
1451 independent reflections
953 reflections with $I > 2\sigma(I)$

$R_{\rm int} = 0.014$
$\theta_{\rm max} = 26.30°$
$h = 0 \rightarrow 16$
$k = -8 \rightarrow 8$
$l = 0 \rightarrow 21$
3 standard reflections
frequency: 120 min
intensity decay: 1.2%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.044$
$wR(F^2) = 0.113$
$S = 1.055$
1451 reflections
145 parameters
H atoms refined isotropically
$w = 1/[\sigma^2(F_o^2) + (0.058P)^2 + 0.081P]$
where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\rm max} < 0.001$
$\Delta\rho_{\rm max} = 0.147$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.159$ e Å^{-3}
Extinction correction: *SHELXL*93
Extinction coefficient: 0.0032 (9)
Scattering factors from *International Tables for Crystallography* (Vol. C)

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters (Å^2)*

$$U_{\rm eq} = (1/3)\Sigma_i\Sigma_j U^{ij}a^i a^j \mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	$U_{\rm eq}$
O1	0.41453 (14)	1/4	0.36835 (9)	0.0560 (5)
O2	0.72672 (13)	1/4	0.58460 (10)	0.0660 (6)
N1	0.29274 (15)	1/4	0.48332 (11)	0.0467 (5)
N2	0.36590 (15)	1/4	0.53261 (11)	0.0429 (5)
C1	0.1920 (2)	1/4	0.51518 (15)	0.0483 (6)
C2	0.1710 (2)	1/4	0.5939 (2)	0.0652 (8)
C3	0.0701 (2)	1/4	0.6186 (2)	0.0836 (10)
C4	−0.0088 (2)	1/4	0.5667 (2)	0.0848 (11)
C5	0.0120 (2)	1/4	0.4893 (3)	0.0896 (11)
C6	0.1125 (2)	1/4	0.4629 (2)	0.0709 (9)
C7	0.4658 (2)	1/4	0.50181 (13)	0.0383 (6)
C8	0.5432 (2)	1/4	0.55806 (13)	0.0432 (6)
C9	0.6437 (2)	1/4	0.53521 (14)	0.0460 (6)
C10	0.6674 (2)	1/4	0.45612 (15)	0.0500 (7)
C11	0.5945 (2)	1/4	0.39853 (13)	0.0450 (6)
C12	0.4900 (2)	1/4	0.42208 (12)	0.0416 (6)
C13	0.6235 (2)	1/4	0.31179 (14)	0.0545 (7)
C14	0.5803 (2)	0.0673 (3)	0.27264 (10)	0.0723 (7)
C15	0.7404 (3)	1/4	0.3004 (2)	0.0758 (10)
C16	0.7042 (3)	1/4	0.6654 (2)	0.0702 (9)

Table 2. *Selected geometric parameters (Å, °)*

O1—C12	1.348 (3)	N2—C7	1.401 (3)
O2—C9	1.374 (3)	C11—C13	1.541 (3)
O2—C16	1.422 (3)	C13—C15	1.531 (4)
N1—N2	1.274 (3)	C13—C14	1.535 (2)
N1—C1	1.419 (3)	C13—C14$^{\rm i}$	1.535 (2)
C12—O1—H1	103.8 (16)	N2—C7—C12	125.2 (2)
N2—N1—C1	115.5 (2)	C8—C9—O2	124.9 (2)
N1—N2—C7	116.0 (2)	O2—C9—C10	115.5 (2)
C6—C1—N1	116.2 (2)	O1—C12—C7	120.4 (2)
C2—C1—N1	124.1 (2)	O1—C12—C11	120.0 (2)
C8—C7—N2	113.8 (2)		
C1—N1—N2—C7	180.0	C10—C11—C13—C14$^{\rm i}$	−119.4 (2)
C10—C11—C13—C14	119.4 (2)	C12—C11—C13—C14$^{\rm i}$	60.6 (2)
C12—C11—C13—C14	−60.6 (2)		

Symmetry code: (i) $x, \frac{1}{2} - y, z$.

Data collection: *CAD*-4 *EXPRESS* (Enraf–Nonius, 1994). Cell refinement: *CAD*-4 *EXPRESS*. Data reduction: *MolEN* (Fair, 1990). Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1985). Program(s) used to refine structure: *SHELXL*93 (Sheldrick, 1993). Molecular graphics: *ORTEP*II (Johnson, 1976). Software used to prepare material for publication: *SHELXL*93.

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: SK1089). Services for accessing these data are described at the back of the journal.

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