3-tert-Butyl-2'-chloro-2-hydroxy-5-methylazobenzene

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Abstract

The crystal structure of the title compound, 6-tert-butyl-
2-(2-chlorophenyldiazenyl)-4-methylphenol, $C_{17}H_{19}Cl$-
N_2O, determined at room temperature, shows that
the molecule is essentially planar, as found for other
azobenzene derivatives, and features a significant in-
tramolecular H···N interaction of 1.75 (3) Å resulting
in the formation of a six-membered ring.

Comment

The synthesis and spectroscopic characterization of
the title compound, (I), has been reported previously
(Kocaokutgen & Gumrukcuoglu, 1996). The present
report details the X-ray crystal structure of (I), which
was undertaken in order to determine the conformation
of the molecule.
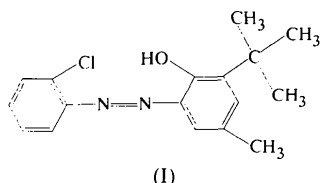
A view of a molecule of (I), with the atom labelling,
is shown in Fig. 1. Bond lengths and angles associated
with the phenyl portions of the molecule are as expected.
The N2—C1 and N1—C7 bond lengths of 1.405 (3) Å
and 1.417 (3) Å, respectively, indicate single-bond char-
acter, a result consistent with those found for other azo-
benzene derivatives studied recently (Işik et al., 1997,
1998). The N=N bond length of 1.266 (2) Å is in-
dicative of significant double-bond character; the C—Cl
bond distance is 1.735 (3) Å.

The aromatic rings, which adopt a trans configuration
about the N=N double bond, are essentially planar.
The dihedral angle between the C1—N2=N1—C7 azo
moiety and the chloro-substituted ring is 1.88 (4)°, that
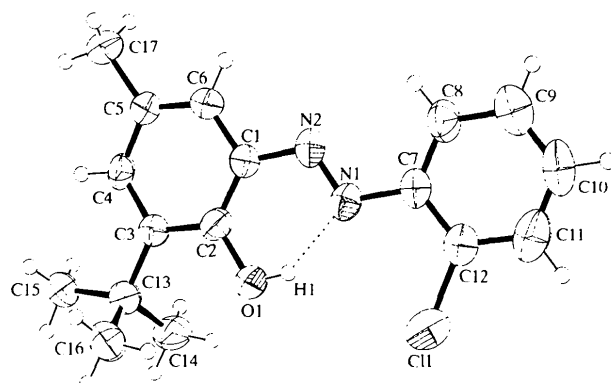between the azo group and the other aromatic ring is

Fig. 1. ORTEPII (Johnson, 1976) view of the title molecule showing
the atomic numbering scheme. The displacement ellipsoids are at
the 50% probability level and H atoms are shown as spheres of
arbitrary size.

3.98 (1)°, and that between the two rings is 5.85 (4)°.
These results indicate that the molecule as a whole is
essentially planar.

A significant intramolecular interaction involving the
hydroxy H1 atom and the N1 atom is noted such
that a six-membered ring is formed [H1···N1 1.75 (3),
O1···N1 2.54 (3) Å and O1—H1···N1 148(2)°].

Experimental

The title compound was obtained as described previously
(Kocaokutgen & Gumrukcuoglu, 1996) and recrystallized from
ethanol to produce crystals of suitable quality for X-ray
diffraction analysis.

Crystal data

$C_{17}H_{19}ClN_2O$	Mo $K\alpha$ radiation
$M_r = 302.79$	$\lambda = 0.71069$ Å
Triclinic	Cell parameters from 25
$P\bar{1}$	reflections
$a = 7.1610 (1)$ Å	$\theta = 11.31–20.83°$
$b = 9.445 (2)$ Å	$\mu = 0.242$ mm^{-1}
$c = 12.479 (3)$ Å	$T = 293 (2)$ K
$\alpha = 80.630 (2)°$	Prismatic
$\beta = 72.441 (3)°$	$0.35 \times 0.25 \times 0.20$ mm
$\gamma = 83.272 (2)°$	Red
$V = 791.9 (3)$ Å^3	
$Z = 2$	
$D_x = 1.270$ Mg m^{-3}	
$D_m = 1.27$ Mg m^{-3}	
D_m measured by pycnometry	

Data collection

Enraf–Nonius CAD-4	$R_{int} = 0.010$
diffractometer	$\theta_{max} = 26.85°$
ω/θ scans	$h = 0 \rightarrow 8$
Absorption correction: none	$k = -11 \rightarrow 11$
3418 measured reflections	$l = -14 \rightarrow 15$
3158 independent reflections	3 standard reflections
2411 reflections with	frequency: 120 min
$I > 2\sigma(I)$	intensity decay: 0.3%

$C_{17}H_{19}ClN_2O$

Refinement

Table 1. *Selected geometric parameters (Å, °)*

Cl1—C12	1.735 (3)	C3—C13	1.537 (3)
O1—C2	1.345 (3)	C5—C17	1.506 (3)
N2—N1	1.266 (2)	C13—C15	1.532 (3)
N2—C1	1.405 (3)	C13—C14	1.535 (4)
N1—C7	1.417 (3)	C13—C16	1.536 (4)
C2—O1—H1	106.1 (19)	C14—C13—C16	110.1 (2)
N1—N2—C1	115.8 (2)	C15—C13—C3	111.8 (2)
N2—N1—C7	115.1 (2)	C14—C13—C3	110.2 (2)
C15—C13—C14	106.7 (2)	C16—C13—C3	109.7 (2)
C15—C13—C16	108.3 (2)		
C1—N2—N1—C7	177.3 (2)	N2—N1—C7—C12	−179.0 (2)
N1—N2—C1—C6	−179.4 (2)	N2—N1—C7—C8	−0.1 (3)
N1—N2—C1—C2	−1.0 (3)		

Data were corrected for Lorentz–polarization factors. The structure was solved by direct methods using *SHELXS*86 (Sheldrick, 1990) and refined by full-matrix least squares using *SHELXL*93 (Sheldrick, 1993). Non-H atoms were refined anisotropically. The H atoms were located from difference-Fourier maps and refined isotropically. C—H distances were in the range 0.88 (6)–1.03 (3) Å, and U_{iso} values for H atoms were in the range 0.044 (6)–0.18 (2) $Å^2$.

Data collection: *CAD-4 EXPRESS* (Enraf–Nonius, 1993). Cell refinement: *CAD-4 EXPRESS*. Data reduction: *MolEN* (Fair, 1990). Molecular graphics: *ORTEP*II (Johnson, 1976). Software used to prepare material for publication: *SHELXL*93.

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N-(2-Dimethoxymethyl-3-hydroxy-2-methyl-6-nitro-3,4-dihydro-2H-1-benzopyran-4-yl)-acetamide

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Abstract

The benzopyran-based title compound, $C_{15}H_{20}N_2O_7$, is a potential potassium-channel opener with cardiovascular therapeutic activities. The 2-dimethoxymethyl and 3-hydroxyl groups are in axial positions while the 4-acetamide group is in a pseudo-equatorial position. The 4-acetamide group is nearly perpendicular to the benzopyran plane. There are two intermolecular hydrogen bonds; one between the hydroxyl O and acetamide O atoms [2.725 (5) Å], and the other between the acetamide N and methoxy O atoms [3.139 (5) Å].

Comment

ATP-sensitive potassium-channel openers are promising in cardiovascular therapy as antihypertensives and coronary vasodilators and consist of several structural groups including benzopyran compounds such as cromakalim, (II) (Robertson & Steinberg, 1990). The title compound, (I), is one of a series of benzopyran-based

(I)

(II)