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Key indicators

Single-crystal X-ray study

T = 294 K

Mean $\sigma(\text{C}-\text{C}) = 0.004 \text{ \AA}$

R factor = 0.045

wR factor = 0.129

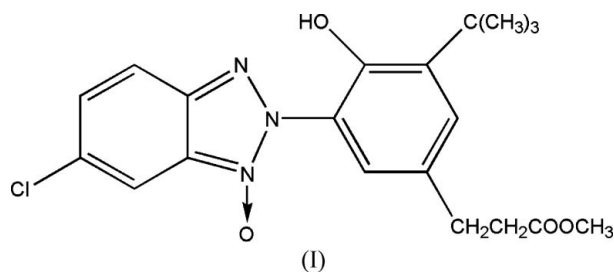
Data-to-parameter ratio = 13.8

For details of how these key indicators were
automatically derived from the article, see
<http://journals.iucr.org/e>.**Methyl 3-[3-*tert*-butyl-5-(6-chloro-1-oxybenzo-
triazol-2-yl)-4-hydroxyphenyl]propionate**

In the title compound, $\text{C}_{20}\text{H}_{22}\text{ClN}_3\text{O}_4$, the dihedral angle between the benzotriazole plane and the benzene plane is $38.2(7)^\circ$. Intramolecular $\text{O}-\text{H}\cdots\text{O}$ and $\text{O}-\text{H}\cdots\text{N}$ hydrogen bonds are present.

Comment

Benzotriazoles have long been an important class of UV absorbers and have gained wide commercial importance and acceptance for many industrial applications (Ravichandran *et al.*, 2002). *N*-oxides are a type of key intermediate in the synthesis of benzotriazoles. Synthesis of benzotriazole ultra-violet absorbers includes one-step and two-step methods. The two-step method is first to obtain the *N*-oxides with azo dyes as raw materials, then reduce the *N*-oxides to obtain the benzotriazoles. The reaction conditions of the two-step method are mild and free from dechlorination (Rosevear & Wilshire, 1982). Thus, the two-step method can help to introduce a halogen atom at the 5-position and increase the molar extinction coefficient of the benzotriazole UV absorbers (Xu & Qi, 1997). We report here the synthesis and structure of the title compound, (I). The conformation of the molecule and the atomic numbering scheme are depicted in Fig. 1. The dihedral angle between the benzotriazole plane and the phenyl plane is $38.2(7)^\circ$, and there are two intramolecular hydrogen bonds (Table 1).

**Experimental**

A mixture of 2-nitro-4-chloro-2'-hydroxy-3'-*tert*-butyl-5'-(2'-methoxycarbonyl)azobenzene (12.5 mmol), saturated NaHCO_3 solution (36 ml) and phase transfer (benzyltriethylammonium chloride) catalyst (0.42 g) was dissolved in chloroform (40 ml); to the mixture was then added sodium hydrogen sulfite (37.5 mmol) in batches over a period of 2 h under reflux; heating was continued for 1 h. The mixture was then cooled to room temperature and separated, the solvent of the organic phase was removed and the residue recrystallized from ethyl acetate. Single crystals suitable for X-ray diffraction analysis precipitated after several days (m.p. 388–389 K). Yield 77.3%. ESI-MS: $m/e = 404.89$ ($[M+H]^+$).

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Crystal data

$C_{20}H_{22}ClN_3O_4$
 $M_r = 403.86$
 Monoclinic, $P2_1/n$
 $a = 8.438$ (6) Å
 $b = 9.798$ (7) Å
 $c = 24.375$ (15) Å
 $\beta = 90.00$ (5)°
 $V = 2015$ (2) Å³

$Z = 4$
 $D_x = 1.334$ Mg m⁻³
 Mo $K\alpha$ radiation
 $\mu = 0.22$ mm⁻¹
 $T = 294$ (2) K
 Block, yellow
 $0.26 \times 0.18 \times 0.14$ mm

Data collection

Bruker SMART CCD area-detector diffractometer
 φ and ω scans
 Absorption correction: multi-scan (SADABS; Sheldrick, 1996)
 $T_{\min} = 0.640$, $T_{\max} = 1.000$

10090 measured reflections
 3567 independent reflections
 2157 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.050$
 $\theta_{\text{max}} = 25.0^\circ$

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.045$
 $wR(F^2) = 0.129$
 $S = 0.99$
 3567 reflections
 259 parameters
 H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0608P)^2 + 0.5063P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\text{max}} = 0.030$
 $\Delta\rho_{\text{max}} = 0.29$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.26$ e Å⁻³
 Extinction correction: SHELXL97
 Extinction coefficient: 0.0212 (17)

Table 1

Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$O2-H2\cdots O1$	0.82	1.77	2.558 (3)	161
$O2-H2\cdots N1$	0.82	2.33	2.978 (3)	136

The hydroxyl H atom was located in a difference Fourier map but was refined using a riding model with the O—H distance constrained to 0.82 Å and $U_{\text{iso}}(\text{H}) = 1.5U_{\text{eq}}(\text{O})$; all other H atoms were placed at geometrically idealized positions with C—H(methyl) = 0.96 Å, C—H(methylene) = 0.97 Å, C—H(aromatic) = 0.93 Å, and $U_{\text{iso}}(\text{H}) = 1.5U_{\text{eq}}(\text{C})$ for methyl and $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$ for methylene and aromatic H atoms.

Data collection: SMART (Bruker, 1998); cell refinement: SAINT (Bruker, 1998); data reduction: SAINT; program(s) used to solve

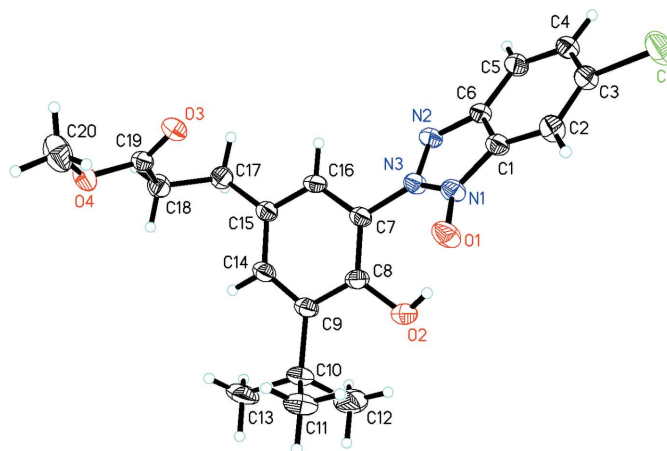


Figure 1

The molecular structure of the title compound, showing 30% probability displacement ellipsoids.

structure: SHELXS97 (Sheldrick, 1997); program(s) used to refine structure: SHELXL97 (Sheldrick, 1997); molecular graphics: SHELXTL (Bruker, 1998); software used to prepare material for publication: SHELXTL.

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References

- Bruker (1998). SHELXTL (Version 5.10), SAINT (Version 5.10) and SMART (Version 5.0). Bruker AXS Inc., Madison, Wisconsin, USA.
 Ravichandran, R., Suhadolnik, J., Wood, M. G., Debeillis, A., Detlefsen, R. E., Iyengar, R. & Wolf, J. P. (2002). US Patent No. 6 458 872.
 Rosevear, J. & Wilshire, J. F. K. (1982). *Aust. J. Chem.* **35**, 2089–2093.
 Sheldrick, G. M. (1996). SADABS. University of Göttingen, Germany.
 Sheldrick, G. M. (1997). SHELXS97 and SHELXL97. University of Göttingen, Germany.
 Xu, B. B. & Qi, G. Z. (1997). *J. East. Chin. Univ. Sci. Tech.* **23**, 321–326.