

2-*tert*-Butyl-4,6-dinitrophenolDaniel E. Lynch<sup>a\*</sup> and  
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## Key indicators

Single-crystal X-ray study  
 $T = 120$  K  
Mean  $\sigma(\text{C}-\text{C}) = 0.003$  Å  
 $R$  factor = 0.056  
 $wR$  factor = 0.165  
Data-to-parameter ratio = 37.0For details of how these key indicators were  
automatically derived from the article, see  
<http://journals.iucr.org/e>.

The structure of the title compound,  $\text{C}_{10}\text{H}_{12}\text{N}_2\text{O}_5$ , has been determined and is found to have an intramolecular hydrogen bond between the phenol group and one of the 6-nitro O atoms. The molecule packs in a zigzag hydrogen-bonded chain, consisting of an intermolecular hydrogen bond, parallel to the  $c$  axis, between the phenol group and a 4-nitro O atom. The dihedral angle between adjacent molecules in the chain is  $82(3)^\circ$ .

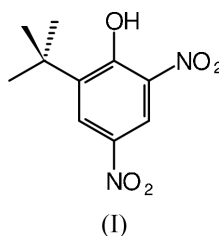
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## Comment

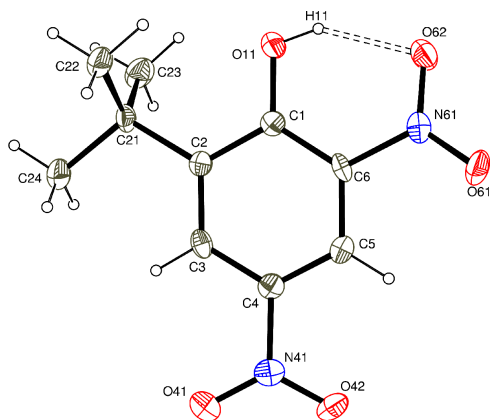
The title compound, (I), was first prepared in 1938 (Ipatieff *et al.*, 1938) by the nitration of 2,4-di-*tert*-butylphenol, with cleavage of the 4-*tert*-butyl group. It was later found that (I) could also be prepared by the nitration of 2,6-di-*tert*-butylphenol, with cleavage of the 6-*tert*-butyl group (Hart & Cassis, 1951). Attempts by Hart and Cassis at nitration without cleavage yielded small quantities of 3,3',5,5'-tetra-*tert*-butyl-*p*-diphenoquinone. The Cambridge Structural Database (Version of April 2004; Allen, 2002) reveals only one structure of a molecule similar to (I), that being musk ambrette, 4-*tert*-butyl-3-methoxy-2,6-dinitrotoluene (De Ridder *et al.*, 1990). In a series of studies to prepare organic salts of 2,6-disubstituted phenols, such as the title compound and 2,6-di-*tert*-butyl-4-nitrophenol, with simple organic bases, we characterized the structure of the title compound and report it here.



Compound (I) exists with an intramolecular hydrogen bond between the phenol group and one of the 6-nitro O atoms (Fig. 1) and packs in a zigzag hydrogen-bonded chain, parallel to the  $c$  axis, consisting of an intermolecular hydrogen bond between the phenol group and a 4-nitro O atom (Fig. 2). Hydrogen-bonding associations are listed in Table 1 and the dihedral angle between adjacent molecules in the chain is  $82(3)^\circ$ .

## Experimental

The title compound, (I), was obtained from Key Organics Ltd. Crystals of (I) were grown from a methanol solution.



**Figure 1**  
The molecular structure (ORTEP-3; Farrugia, 1997) and atom-numbering scheme for (I). Displacement ellipsoids are drawn at the 50% probability level. Dashed lines indicate the intramolecular hydrogen bond.

#### Crystal data

$C_{10}H_{12}N_2O_5$	$D_x = 1.422 \text{ Mg m}^{-3}$
$M_r = 240.22$	Mo $K\alpha$ radiation
Monoclinic, $P2_1/c$	Cell parameters from 52 reflections
$a = 9.974 (2) \text{ \AA}$	$\theta = 4.3\text{--}18.1^\circ$
$b = 10.575 (2) \text{ \AA}$	$\mu = 0.12 \text{ mm}^{-1}$
$c = 11.547 (2) \text{ \AA}$	$T = 120 (2) \text{ K}$
$\beta = 112.90 (3)^\circ$	Needle, yellow
$V = 1122.0 (4) \text{ \AA}^3$	$0.20 \times 0.06 \times 0.06 \text{ mm}$
$Z = 4$	

#### Data collection

Bruker–Nonius KappaCCD area-detector diffractometer	4502 reflections with $I > 2\sigma(I)$
$\varphi$ and $\omega$ scans	$R_{\text{int}} = 0.070$
Absorption correction: none	$\theta_{\text{max}} = 25.0^\circ$
5908 measured reflections	$h = -11 \rightarrow 11$
5912 independent reflections	$k = -12 \rightarrow 12$
	$l = -12 \rightarrow 12$

#### Refinement

Refinement on $F^2$	$w = 1/[\sigma^2(F_o^2) + (0.1108P)^2]$
$R[F^2 > 2\sigma(F^2)] = 0.056$	where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.165$	$(\Delta/\sigma)_{\text{max}} = 0.027$
$S = 1.04$	$\Delta\rho_{\text{max}} = 0.36 \text{ e \AA}^{-3}$
5912 reflections	$\Delta\rho_{\text{min}} = -0.34 \text{ e \AA}^{-3}$
160 parameters	Extinction correction: SHELXL97
H-atom parameters constrained	Extinction coefficient: 0.033 (5)

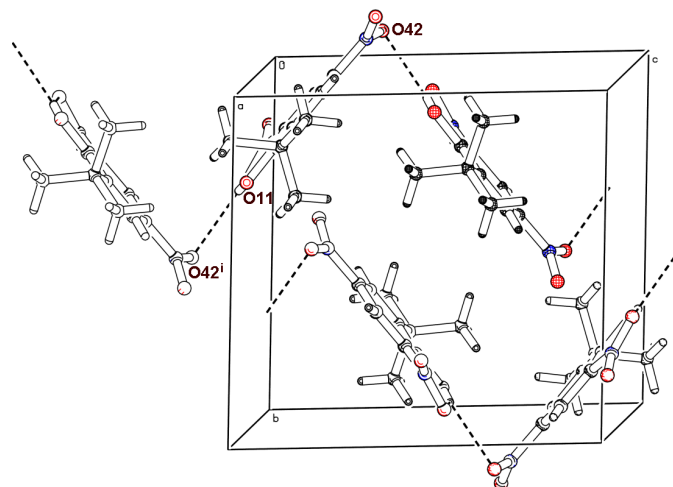
**Table 1**

Hydrogen-bonding geometry ( $\text{\AA}$ ,  $^\circ$ ).

$D\text{--}H\cdots A$	$D\text{--}H$	$H\cdots A$	$D\cdots A$	$D\text{--}H\cdots A$
O11–H11 $\cdots$ O62	0.82	1.85	2.5743 (17)	146
O11–H11 $\cdots$ O42 <sup>i</sup>	0.82	2.42	2.8926 (19)	118

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

All H atoms were included in the refinement at calculated positions in the riding-model approximation, with C–H distances of 0.93 (aromatic H atoms) and 0.96  $\text{\AA}$  (CH<sub>3</sub> H atoms), and O–H distance of 0.82  $\text{\AA}$ . The isotropic displacement parameters were set equal to



**Figure 2**

Packing diagram for (I). [Symmetry code: (i)  $x, \frac{1}{2} - y, z - \frac{1}{2}$ .] Dashed lines indicate intermolecular hydrogen bonds.

$1.5U_{\text{eq}}$  of the carrier atom for the methyl groups and  $1.2U_{\text{eq}}$  of the carrier for aromatic CH and hydroxyl OH groups. Crystals of (I) were twinned with two nearly equal components related by a  $180^\circ$  rotation about the [100] reciprocal lattice direction. Combined data with complete or no overlap were used for refinement, and the twinning prevented merging of equivalent reflections before refinement. The partially overlapped reflections were rejected, resulting in a low data-completeness value of 89.9%.

Data collection: DENZO (Otwinowski & Minor, 1997) and COLLECT (Hooft, 1998); cell refinement: DENZO and COLLECT; data reduction: DENZO and SCALEPACK (Otwinowski & Minor, 1997), and COLLECT; program(s) used to solve structure: SHELXS97 (Sheldrick, 1997); program(s) used to refine structure: SHELXL97 (Sheldrick, 1997); molecular graphics: ORTEP-3 for Windows (Farrugia, 1997) and PLUTON94 (Spek, 1994); software used to prepare material for publication: SHELXL97.

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