

2-*tert*-Butyl-4,6-dinitrophenolDaniel E. Lynch<sup>a\*</sup> and  
Ian McClenaghan<sup>b</sup><sup>a</sup>School of Science and the Environment,  
Coventry University, Coventry CV1 5FB,  
England, and <sup>b</sup>Key Organics Ltd, Highfield  
Industrial Estate, Camelford, Cornwall  
PL32 9QZ, EnglandCorrespondence e-mail:  
apx106@coventry.ac.uk

## Key indicators

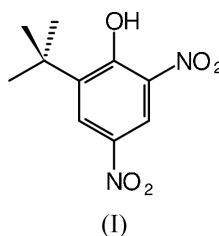
Single-crystal X-ray study  
 $T = 120$  K  
Mean  $\sigma(\text{C}-\text{C}) = 0.003$  Å  
 $R$  factor = 0.056  
 $wR$  factor = 0.165  
Data-to-parameter ratio = 37.0For details of how these key indicators were  
automatically derived from the article, see  
<http://journals.iucr.org/e>.

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  $82(3)^\circ$ .

Received 10 June 2004  
Accepted 29 June 2004  
Online 9 July 2004

## Comment

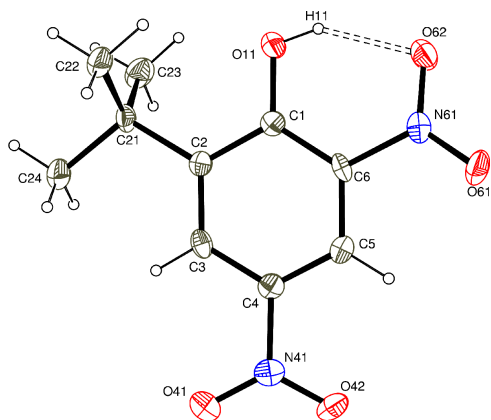
The title compound, (I), was first prepared in 1938 (Ipatieff *et al.*, 1938) by the nitration of 2,4-di-*tert*-butylphenol, with cleavage of the 4-*tert*-butyl group. It was later found that (I) could also be prepared by the nitration of 2,6-di-*tert*-butylphenol, with cleavage of the 6-*tert*-butyl 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)

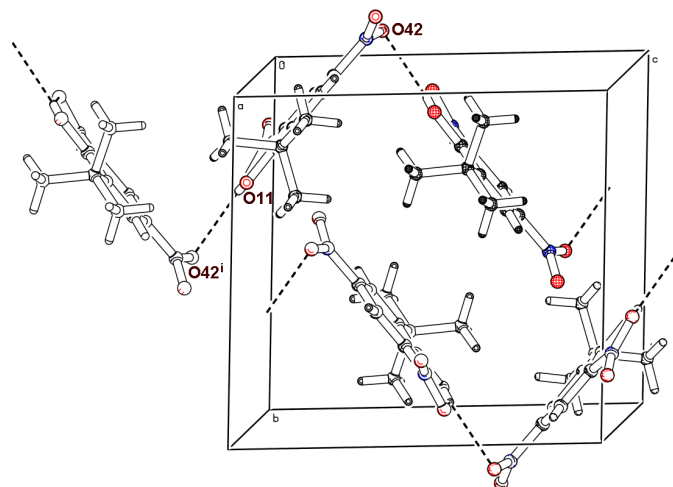
**Table 1**

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.

The authors thank the EPSRC National Crystallography Service (Southampton, England).

#### References

- Allen, F. H. (2002). *Acta Cryst.* **B58**, 380–388.  
 De Ridder, D. J. A., Goubitz, K. & Schenk, H. (1990). *Acta Cryst.* **C46**, 468–470.  
 Farrugia, L. J. (1997). *J. Appl. Cryst.* **30**, 565.  
 Hart, H. & Cassis, F. A. Jr (1951). *J. Org. Chem.* **73**, 3179–3182.  
 Hooft, R. (1998). COLLECT. Nonius BV, Delft, The Netherlands.  
 Ipatieff, V. N., Pines, H. & Friedman, B. S. (1938). *J. Am. Chem. Soc.* **60**, 2495–2497.  
 Otwinowski, Z. & Minor, W. (1997). *Methods in Enzymology*, Vol. 276, *Macromolecular Crystallography*, Part A, edited by C. W. Carter Jr and R. M. Sweet, pp. 307–326. New York: Academic Press.  
 Sheldrick, G. M. (1997). SHELXS97 and SHELXL97. University of Göttingen, Germany.  
 Spek, A. L. (1994). PLUTON94. University of Utrecht, The Netherlands.

## supporting information

*Acta Cryst.* (2004). E60, o1288–o1289 [https://doi.org/10.1107/S1600536804015855]

2-*tert*-Butyl-4,6-dinitrophenol

Daniel E. Lynch and Ian McClenaghan

2-*tert*-Butyl-4,6-dinitrophenol*Crystal data*

$C_{10}H_{12}N_2O_5$

$M_r = 240.22$

Monoclinic,  $P2_1/c$

Hall symbol: -P 2ybc

$a = 9.974$  (2) Å

$b = 10.575$  (2) Å

$c = 11.547$  (2) Å

$\beta = 112.90$  (3)°

$V = 1122.0$  (4) Å<sup>3</sup>

$Z = 4$

$F(000) = 504$

$D_x = 1.422$  Mg m<sup>-3</sup>

Mo  $K\alpha$  radiation,  $\lambda = 0.71073$  Å

Cell parameters from 52 reflections

$\theta = 4.3$ – $18.1$ °

$\mu = 0.12$  mm<sup>-1</sup>

$T = 120$  K

Plate, yellow

$0.20 \times 0.06 \times 0.06$  mm

*Data collection*

Bruker–Nonius KappaCCD area-detector diffractometer

Radiation source: Bruker–Nonius FR591 rotating anode

Graphite monochromator

Detector resolution: 9.091 pixels mm<sup>-1</sup>

$\varphi$  and  $\omega$  scans

5908 measured reflections

5912 independent reflections

4502 reflections with  $I > 2\sigma(I)$

$R_{int} = 0.000$

$\theta_{max} = 25.0$ °,  $\theta_{min} = 2.7$ °

$h = -11 \rightarrow 11$

$k = -12 \rightarrow 12$

$l = -12 \rightarrow 12$

*Refinement*

Refinement on  $F^2$

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.056$

$wR(F^2) = 0.165$

$S = 1.04$

5912 reflections

160 parameters

0 restraints

Primary atom site location: structure-invariant direct methods

Secondary atom site location: difference Fourier map

Hydrogen site location: inferred from neighbouring sites

H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.1108P)^2]$

where  $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{max} = 0.027$

$\Delta\rho_{max} = 0.36$  e Å<sup>-3</sup>

$\Delta\rho_{min} = -0.34$  e Å<sup>-3</sup>

Extinction correction: SHELXL97,

$F_c^* = kFc[1 + 0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$

Extinction coefficient: 0.033 (5)

*Special details*

**Geometry.** All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters ( $\text{\AA}^2$ )

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
C1	0.08422 (16)	0.24756 (16)	0.01103 (17)	0.0175 (4)
O11	0.08686 (12)	0.34527 (12)	-0.06166 (12)	0.0252 (4)
H11	0.0059	0.3778	-0.0918	0.038*
C2	0.21649 (17)	0.17828 (16)	0.07515 (17)	0.0167 (4)
C21	0.35876 (16)	0.21422 (16)	0.05852 (18)	0.0199 (5)
C22	0.33651 (18)	0.19937 (18)	-0.08108 (18)	0.0273 (5)
H22A	0.2582	0.2530	-0.1322	0.041*
H22B	0.3133	0.1129	-0.1064	0.041*
H22C	0.4242	0.2229	-0.0913	0.041*
C23	0.40487 (19)	0.35174 (18)	0.1040 (2)	0.0311 (5)
H23A	0.4939	0.3718	0.0944	0.047*
H23B	0.4193	0.3591	0.1909	0.047*
H23C	0.3299	0.4093	0.0547	0.047*
C24	0.48590 (18)	0.12544 (18)	0.1379 (2)	0.0291 (5)
H24A	0.4601	0.0393	0.1130	0.044*
H24B	0.5040	0.1349	0.2254	0.044*
H24C	0.5720	0.1474	0.1243	0.044*
C3	0.21111 (17)	0.07892 (16)	0.15038 (17)	0.0182 (4)
H3	0.2946	0.0313	0.1915	0.022*
C4	0.08200 (17)	0.04759 (15)	0.16662 (18)	0.0187 (4)
N41	0.08388 (15)	-0.05678 (14)	0.25181 (15)	0.0225 (4)
O41	0.18873 (14)	-0.13023 (13)	0.28710 (14)	0.0362 (4)
O42	-0.02105 (13)	-0.06679 (11)	0.28282 (13)	0.0284 (4)
C5	-0.04623 (18)	0.11166 (16)	0.10471 (17)	0.0176 (4)
H5	-0.1311	0.0898	0.1152	0.021*
C6	-0.04398 (16)	0.20999 (15)	0.02624 (17)	0.0179 (4)
N61	-0.18396 (15)	0.27369 (15)	-0.04410 (15)	0.0231 (4)
O61	-0.29647 (11)	0.22493 (13)	-0.04595 (12)	0.0320 (4)
O62	-0.18238 (12)	0.37669 (12)	-0.09758 (13)	0.0306 (4)

Atomic displacement parameters ( $\text{\AA}^2$ )

	$U^{11}$	$U^{22}$	$U^{33}$	$U^{12}$	$U^{13}$	$U^{23}$
C1	0.0198 (9)	0.0146 (9)	0.0188 (12)	-0.0009 (9)	0.0085 (8)	-0.0030 (8)
O11	0.0178 (6)	0.0247 (8)	0.0322 (9)	0.0036 (6)	0.0087 (6)	0.0090 (6)
C2	0.0177 (9)	0.0173 (9)	0.0157 (12)	-0.0001 (8)	0.0071 (8)	-0.0029 (8)
C21	0.0120 (8)	0.0214 (10)	0.0258 (13)	0.0017 (8)	0.0067 (8)	-0.0004 (8)
C22	0.0183 (9)	0.0354 (12)	0.0305 (14)	0.0011 (9)	0.0122 (9)	0.0051 (9)
C23	0.0267 (10)	0.0317 (12)	0.0373 (15)	-0.0066 (10)	0.0151 (10)	-0.0009 (10)
C24	0.0185 (10)	0.0364 (12)	0.0338 (14)	0.0041 (9)	0.0118 (9)	0.0052 (9)
C3	0.0154 (9)	0.0186 (10)	0.0180 (12)	0.0030 (8)	0.0036 (8)	-0.0051 (7)
C4	0.0212 (9)	0.0140 (9)	0.0214 (12)	-0.0030 (8)	0.0088 (8)	-0.0044 (8)
N41	0.0255 (8)	0.0208 (8)	0.0196 (11)	-0.0044 (8)	0.0072 (8)	-0.0028 (7)
O41	0.0333 (8)	0.0328 (8)	0.0439 (11)	0.0107 (7)	0.0163 (7)	0.0148 (7)
O42	0.0298 (7)	0.0277 (7)	0.0330 (9)	-0.0074 (6)	0.0180 (7)	0.0012 (6)

C5	0.0168 (9)	0.0208 (10)	0.0159 (12)	-0.0030 (8)	0.0072 (8)	-0.0054 (8)
C6	0.0129 (8)	0.0181 (9)	0.0191 (12)	0.0056 (8)	0.0022 (8)	-0.0012 (8)
N61	0.0176 (8)	0.0264 (10)	0.0249 (11)	0.0027 (8)	0.0077 (7)	-0.0015 (7)
O61	0.0148 (6)	0.0453 (9)	0.0369 (10)	-0.0020 (7)	0.0113 (6)	0.0051 (7)
O62	0.0221 (7)	0.0243 (8)	0.0430 (10)	0.0055 (6)	0.0102 (7)	0.0087 (7)

*Geometric parameters (Å, °)*

C1—O11	1.338 (2)	C24—H24A	0.96
C1—C6	1.414 (2)	C24—H24B	0.96
C1—C2	1.437 (2)	C24—H24C	0.96
O11—H11	0.82	C3—C4	1.411 (2)
C2—C3	1.378 (2)	C3—H3	0.93
C2—C21	1.551 (2)	C4—C5	1.376 (2)
C21—C22	1.547 (3)	C4—N41	1.474 (2)
C21—C23	1.554 (3)	N41—O42	1.2355 (18)
C21—C24	1.557 (3)	N41—O41	1.2375 (18)
C22—H22A	0.96	C5—C6	1.385 (2)
C22—H22B	0.96	C5—H5	0.93
C22—H22C	0.96	C6—N61	1.477 (2)
C23—H23A	0.96	N61—O61	1.2276 (16)
C23—H23B	0.96	N61—O62	1.2552 (18)
C23—H23C	0.96		
O11—C1—C6	122.31 (14)	C21—C24—H24A	109.5
O11—C1—C2	118.60 (14)	C21—C24—H24B	109.5
C6—C1—C2	119.08 (15)	H24A—C24—H24B	109.5
C1—O11—H11	109.5	C21—C24—H24C	109.5
C3—C2—C1	117.42 (15)	H24A—C24—H24C	109.5
C3—C2—C21	121.79 (15)	H24B—C24—H24C	109.5
C1—C2—C21	120.79 (15)	C2—C3—C4	121.63 (16)
C22—C21—C2	109.68 (14)	C2—C3—H3	119.2
C22—C21—C23	110.52 (15)	C4—C3—H3	119.2
C2—C21—C23	110.63 (15)	C5—C4—C3	121.80 (16)
C22—C21—C24	107.82 (15)	C5—C4—N41	118.90 (15)
C2—C21—C24	110.72 (15)	C3—C4—N41	119.30 (15)
C23—C21—C24	107.41 (15)	O42—N41—O41	123.60 (15)
C21—C22—H22A	109.5	O42—N41—C4	117.74 (14)
C21—C22—H22B	109.5	O41—N41—C4	118.66 (14)
H22A—C22—H22B	109.5	C4—C5—C6	117.45 (16)
C21—C22—H22C	109.5	C4—C5—H5	121.3
H22A—C22—H22C	109.5	C6—C5—H5	121.3
H22B—C22—H22C	109.5	C5—C6—C1	122.54 (15)
C21—C23—H23A	109.5	C5—C6—N61	116.73 (15)
C21—C23—H23B	109.5	C1—C6—N61	120.72 (15)
H23A—C23—H23B	109.5	O61—N61—O62	122.85 (14)
C21—C23—H23C	109.5	O61—N61—C6	118.70 (15)
H23A—C23—H23C	109.5	O62—N61—C6	118.44 (14)

---

H23B—C23—H23C 109.5

---

*Hydrogen-bond geometry (Å, °)*

<i>D</i> —H··· <i>A</i>	<i>D</i> —H	H··· <i>A</i>	<i>D</i> ··· <i>A</i>	<i>D</i> —H··· <i>A</i>
O11—H11···O62	0.82	1.85	2.5743 (17)	146
O11—H11···O42 <sup>i</sup>	0.82	2.42	2.8926 (19)	118

---

Symmetry code: (i)  $x, -y+1/2, z-1/2$ .