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5-*tert*-Butyl-2-methoxy-1,3-dinitrobenzene

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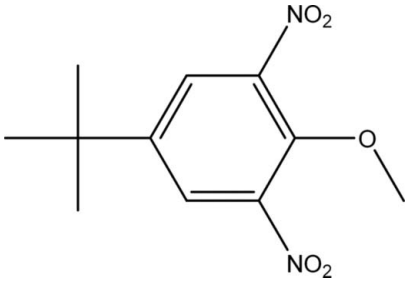
Key indicators: single-crystal X-ray study; T = 298 K; mean σ(C–C) = 0.003 Å; R factor = 0.051; wR factor = 0.162; data-to-parameter ratio = 18.7.

The title compound, $C_{11}H_{14}N_2O_5$, was prepared by the reaction of HNO_3 and 1-*tert*-butyl-4-methoxybenzene. The atoms of the benzene ring, the nitro N atoms, the methoxy O atom and the *tert*-butyl tertiary C atom are coplanar; the dihedral angles between this plane and the NO_2 planes are 50.41 (18) and 20.2 (3)°. There are weak C—H···O intermolecular interactions.

Related literature

For related literature, see: Kwiatkowski *et al.* (1997).



Experimental

Crystal data

$C_{11}H_{14}N_2O_5$
M_r = 254.24
Monoclinic, $C2/c$
a = 21.7800 (18) Å
b = 12.3756 (10) Å
c = 10.0521 (8) Å
β = 110.686 (3)°
V = 2534.8 (4) Å^3
Z = 8
Mo $K\alpha$ radiation
μ = 0.11 mm^{-1}
T = 298 (2) K
0.30 × 0.25 × 0.20 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
Absorption correction: multi-scan (*ABSCOR*; Higashi, 1995)
T_{min} = 0.956, T_{max} = 0.979
16809 measured reflections
3063 independent reflections
2411 reflections with $I > 2\sigma(I)$
R_{int} = 0.024
3 standard reflections
frequency: 60 min
intensity decay: 0.3%

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.051
$wR(F^2)$ = 0.162
S = 1.03
3063 reflections
164 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.53 e Å^{-3}
$\Delta\rho_{min}$ = −0.34 e Å^{-3}

Table 1
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
C9—H9A···O5^{i}	0.96	2.67	3.626 (4)	175
C9—H9C···O4^{ii}	0.96	2.67	3.610 (3)	167

Symmetry codes: (i) $-x+\frac{1}{2}, y-\frac{1}{2}, -z+\frac{3}{2}$; (ii) $-x+\frac{1}{2}, -y+\frac{1}{2}, -z+1$.

Data collection: *CAD-4 EXPRESS* (Enraf–Nonius, 1994); cell refinement: *CAD-4 EXPRESS*; data reduction: *XCAD4* (Harms & Wocadlo, 1995); 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); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: KP2103).

References

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supplementary materials

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5-*tert*-Butyl-2-methoxy-1,3-dinitrobenzene

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Comment

5-tert-Butyl-2-methoxy-1,3-dinitrobenzene, (I), is an important intermediate in synthesis of Butralin which is a herbicide (Kwiatkowski *et al.*, 1997) and was obtained by the reaction of HNO_3 and 1-tert-butyl-4-methoxybenzene, as colourless crystals suitable for X-ray structure analysis.

The molecular structure of (I) is illustrated in Fig. 1. Atoms C1, C2, C3, C4, C5, C6, C7, N1, N2 and O1 are coplanar, the largest deviation being 0.0578 (13) Å for C3. The dihedral angles between the C1—C7/N1/N2/O1 plane and the O2/O3/N1 and O4/O5/N2 planes are 50.41 (18) and 20.21 (29)^{o}, respectively.

Experimental

The title compound was prepared from HNO_3 and 1-tert-butyl-4-methoxybenzene, according to the procedure of Kwiatkowski *et al.* (1997).

Refinement

H atoms were added at calculated positions and refined using a riding model. H atoms were given isotropic displacement parameters equal to 1.2 (or 1.5 for methyl H atoms) times the equivalent isotropic displacement parameters of their parent atoms and C—H distances were restrained to 0.93 Å for those bonded to phenyl ring, 0.96 Å for those bonded to methyl.

Figures

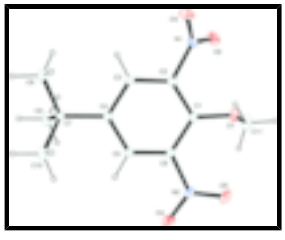

Fig. 1. The structure of (I) shown with the 30% probability displacement ellipsoids.

5-tert-Butyl-2-methoxy-1,3-dinitrobenzene

Crystal data

$C_{11}H_{14}N_2O_5$	F_{000} = 1072
M_r = 254.24	D_x = 1.332 Mg m^{-3}
Monoclinic, *C*2/*c*	Mo *K*α radiation λ = 0.71073 Å

Hall symbol: -C 2yc	Cell parameters from 25 reflections
a = 21.7800 (18) Å	θ = 10.0–14.8º
b = 12.3756 (10) Å	μ = 0.11 mm^{-1}
c = 10.0521 (8) Å	T = 298 (2) K
β = 110.686 (3)º	Prism, colourless
V = 2534.8 (4) $Å^3$	0.30 × 0.25 × 0.20 mm
Z = 8	

Data collection

Enraf–Nonius CAD-4 diffractometer	R_{int} = 0.024
Radiation source: fine-focus sealed tube	θ_{max} = 28.2º
Monochromator: graphite	θ_{min} = 1.9º
T = 298(2) K	h = −28→27
ω/2–θ scans	k = −16→16
Absorption correction: multi-scan (ABSCOR; Higashi, 1995)	l = −12→13
T_{min} = 0.956, T_{max} = 0.979	3 standard reflections
16809 measured reflections	every 60 min
3063 independent reflections	intensity decay: 0.3%
2411 reflections with $I > 2\sigma(I)$	

Refinement

Refinement on F^2	H-atom parameters constrained
Least-squares matrix: full	$w = 1/[\sigma^2(F_o^2) + (0.0801P)^2 + 1.6942P]$ where $P = (F_o^2 + 2F_c^2)/3$
$R[F^2 > 2\sigma(F^2)]$ = 0.051	$(\Delta/\sigma)_{max} < 0.001$
$wR(F^2)$ = 0.162	$\Delta\rho_{max}$ = 0.53 e $Å^{-3}$
S = 1.03	$\Delta\rho_{min}$ = −0.34 e $Å^{-3}$
3063 reflections	Extinction correction: SHELXL97, $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
164 parameters	Extinction coefficient: 0.0056 (9)
Primary atom site location: structure-invariant direct methods	
Secondary atom site location: difference Fourier map	
Hydrogen site location: inferred from neighbouring sites	

Special details

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

Refinement. Refinement of F^2 against ALL reflections. The weighted R-factor wR and goodness of fit S are based on F^2, conventional R-factors R are based on F, with F set to zero for negative F^2. The threshold expression of $F^2 > 2\text{sigma}(F^2)$ is used only for calculating R-factors(gt) etc. and is not relevant to the choice of reflections for refinement. R-factors based on F^2 are statistically about twice as large as those based on F, and R- factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
C9	0.41557 (11)	0.0960 (2)	0.7960 (2)	0.0732 (6)
H9A	0.4019	0.0243	0.8093	0.110*
H9B	0.4614	0.0955	0.8091	0.110*
H9C	0.3907	0.1204	0.7016	0.110*
C10	0.42528 (11)	0.2866 (2)	0.8843 (3)	0.0831 (7)
H10A	0.4001	0.3116	0.7904	0.125*
H10B	0.4710	0.2866	0.8967	0.125*
H10C	0.4181	0.3337	0.9533	0.125*
O4	0.19977 (9)	0.33505 (15)	0.55134 (17)	0.0828 (5)
O5	0.12433 (9)	0.3218 (2)	0.6367 (3)	0.1169 (8)
C11	0.08562 (11)	0.0967 (2)	0.6871 (3)	0.0795 (7)
H11A	0.0417	0.0987	0.6877	0.119*
H11B	0.0997	0.0230	0.6896	0.119*
H11C	0.0868	0.1308	0.6022	0.119*
C1	0.19258 (7)	0.15762 (12)	0.82339 (16)	0.0407 (3)
O1	0.12844 (6)	0.15281 (11)	0.80883 (14)	0.0561 (4)
C4	0.33051 (7)	0.16977 (12)	0.88046 (15)	0.0382 (3)
C3	0.30526 (8)	0.10427 (12)	0.96067 (15)	0.0406 (3)
H3	0.3336	0.0645	1.0359	0.049*
C2	0.23849 (8)	0.09782 (12)	0.92962 (16)	0.0408 (3)
C5	0.28576 (8)	0.22938 (12)	0.77189 (16)	0.0410 (3)
H5	0.3010	0.2733	0.7152	0.049*
N1	0.21527 (8)	0.02213 (12)	1.01381 (17)	0.0525 (4)
C6	0.21916 (8)	0.22504 (12)	0.74620 (15)	0.0414 (4)
O3	0.24253 (9)	0.02423 (13)	1.14221 (15)	0.0738 (5)
N2	0.17748 (8)	0.29694 (13)	0.63483 (17)	0.0568 (4)
C7	0.40392 (8)	0.17216 (13)	0.90428 (17)	0.0444 (4)
O2	0.17149 (9)	−0.04078 (14)	0.95125 (19)	0.0815 (5)
C8	0.44524 (9)	0.1347 (2)	1.0536 (2)	0.0700 (6)
H8A	0.4333	0.0620	1.0677	0.105*
H8B	0.4376	0.1814	1.1224	0.105*
H8C	0.4909	0.1369	1.0648	0.105*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C9	0.0524 (11)	0.1024 (17)	0.0703 (13)	0.0098 (11)	0.0283 (10)	−0.0150 (12)
C10	0.0492 (12)	0.0739 (14)	0.1155 (19)	−0.0102 (10)	0.0157 (12)	0.0228 (13)
O4	0.0792 (11)	0.0996 (12)	0.0663 (9)	0.0261 (9)	0.0217 (8)	0.0410 (9)

O5	0.0628 (11)	0.1358 (18)	0.1556 (19)	0.0521 (11)	0.0429 (12)	0.0776 (15)
C11	0.0450 (11)	0.0950 (17)	0.0886 (16)	−0.0054 (10)	0.0113 (10)	−0.0152 (13)
C1	0.0381 (8)	0.0423 (8)	0.0438 (8)	0.0050 (6)	0.0172 (6)	−0.0052 (6)
O1	0.0383 (6)	0.0667 (8)	0.0674 (8)	0.0048 (5)	0.0239 (6)	−0.0072 (6)
C4	0.0383 (8)	0.0390 (7)	0.0372 (7)	0.0044 (6)	0.0133 (6)	−0.0008 (5)
C3	0.0428 (8)	0.0411 (7)	0.0360 (7)	0.0048 (6)	0.0115 (6)	0.0023 (6)
C2	0.0456 (8)	0.0400 (7)	0.0403 (7)	0.0008 (6)	0.0194 (6)	−0.0007 (6)
C5	0.0427 (8)	0.0416 (8)	0.0400 (8)	0.0039 (6)	0.0164 (6)	0.0039 (6)
N1	0.0566 (9)	0.0500 (8)	0.0576 (9)	0.0001 (7)	0.0284 (7)	0.0061 (6)
C6	0.0420 (8)	0.0419 (8)	0.0385 (7)	0.0110 (6)	0.0120 (6)	0.0030 (6)
O3	0.1015 (12)	0.0722 (9)	0.0528 (8)	−0.0061 (8)	0.0336 (8)	0.0140 (7)
N2	0.0479 (9)	0.0588 (9)	0.0581 (9)	0.0158 (7)	0.0116 (7)	0.0138 (7)
C7	0.0361 (8)	0.0512 (9)	0.0446 (8)	0.0028 (6)	0.0127 (6)	0.0025 (7)
O2	0.0743 (10)	0.0813 (11)	0.0888 (11)	−0.0284 (8)	0.0287 (9)	0.0089 (8)
C8	0.0400 (9)	0.1079 (17)	0.0564 (11)	0.0121 (10)	0.0099 (8)	0.0133 (11)

Geometric parameters (Å, °)

C9—C7	1.527 (3)	C1—C6	1.397 (2)
C9—H9A	0.9600	C4—C3	1.387 (2)
C9—H9B	0.9600	C4—C5	1.391 (2)
C9—H9C	0.9600	C4—C7	1.531 (2)
C10—C7	1.526 (3)	C3—C2	1.378 (2)
C10—H10A	0.9600	C3—H3	0.9300
C10—H10B	0.9600	C2—N1	1.467 (2)
C10—H10C	0.9600	C5—C6	1.382 (2)
O4—N2	1.204 (2)	C5—H5	0.9300
O5—N2	1.204 (2)	N1—O3	1.216 (2)
C11—O1	1.429 (3)	N1—O2	1.219 (2)
C11—H11A	0.9600	C6—N2	1.467 (2)
C11—H11B	0.9600	C7—C8	1.525 (2)
C11—H11C	0.9600	C8—H8A	0.9600
C1—O1	1.3535 (19)	C8—H8B	0.9600
C1—C2	1.391 (2)	C8—H8C	0.9600
C7—C9—H9A	109.5	C3—C2—C1	123.94 (14)
C7—C9—H9B	109.5	C3—C2—N1	117.24 (14)
H9A—C9—H9B	109.5	C1—C2—N1	118.81 (14)
C7—C9—H9C	109.5	C6—C5—C4	121.65 (14)
H9A—C9—H9C	109.5	C6—C5—H5	119.2
H9B—C9—H9C	109.5	C4—C5—H5	119.2
C7—C10—H10A	109.5	O3—N1—O2	124.12 (16)
C7—C10—H10B	109.5	O3—N1—C2	117.40 (15)
H10A—C10—H10B	109.5	O2—N1—C2	118.44 (15)
C7—C10—H10C	109.5	C5—C6—C1	122.22 (14)
H10A—C10—H10C	109.5	C5—C6—N2	116.42 (14)
H10B—C10—H10C	109.5	C1—C6—N2	121.35 (14)
O1—C11—H11A	109.5	O5—N2—O4	122.47 (17)
O1—C11—H11B	109.5	O5—N2—C6	118.85 (17)
H11A—C11—H11B	109.5	O4—N2—C6	118.45 (15)

O1—C11—H11C	109.5	C8—C7—C10	108.10 (18)
H11A—C11—H11C	109.5	C8—C7—C9	108.96 (17)
H11B—C11—H11C	109.5	C10—C7—C9	110.11 (18)
O1—C1—C2	119.63 (14)	C8—C7—C4	111.64 (14)
O1—C1—C6	125.50 (14)	C10—C7—C4	110.33 (14)
C2—C1—C6	114.70 (14)	C9—C7—C4	107.69 (14)
C1—O1—C11	116.77 (14)	C7—C8—H8A	109.5
C3—C4—C5	117.03 (14)	C7—C8—H8B	109.5
C3—C4—C7	121.81 (13)	H8A—C8—H8B	109.5
C5—C4—C7	121.08 (13)	C7—C8—H8C	109.5
C2—C3—C4	120.38 (14)	H8A—C8—H8C	109.5
C2—C3—H3	119.8	H8B—C8—H8C	109.5
C4—C3—H3	119.8		
C2—C1—O1—C11	−108.9 (2)	C4—C5—C6—C1	−2.7 (2)
C6—C1—O1—C11	76.2 (2)	C4—C5—C6—N2	176.15 (14)
C5—C4—C3—C2	1.6 (2)	O1—C1—C6—C5	176.70 (14)
C7—C4—C3—C2	−175.09 (14)	C2—C1—C6—C5	1.5 (2)
C4—C3—C2—C1	−2.8 (2)	O1—C1—C6—N2	−2.0 (2)
C4—C3—C2—N1	176.56 (13)	C2—C1—C6—N2	−177.23 (14)
O1—C1—C2—C3	−174.29 (14)	C5—C6—N2—O5	−157.4 (2)
C6—C1—C2—C3	1.2 (2)	C1—C6—N2—O5	21.5 (3)
O1—C1—C2—N1	6.3 (2)	C5—C6—N2—O4	17.3 (2)
C6—C1—C2—N1	−178.18 (13)	C1—C6—N2—O4	−163.92 (18)
C3—C4—C5—C6	1.0 (2)	C3—C4—C7—C8	−22.9 (2)
C7—C4—C5—C6	177.75 (14)	C5—C4—C7—C8	160.45 (16)
C3—C2—N1—O3	47.5 (2)	C3—C4—C7—C10	−143.18 (18)
C1—C2—N1—O3	−133.07 (17)	C5—C4—C7—C10	40.2 (2)
C3—C2—N1—O2	−130.37 (18)	C3—C4—C7—C9	96.62 (18)
C1—C2—N1—O2	49.1 (2)	C5—C4—C7—C9	−79.98 (19)

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C9—H9A···O5^{i}	0.96	2.67	3.626 (4)	175
C9—H9C···O4^{ii}	0.96	2.67	3.610 (3)	167

Symmetry codes: (i) −*x*+1/2, *y*−1/2, −*z*+3/2; (ii) −*x*+1/2, −*y*+1/2, −*z*+1.

Fig. 1