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1-Mesitylmethyl-1*H*benzotriazole 3-oxide

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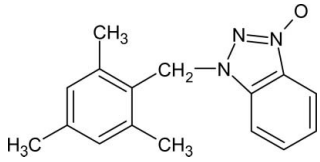
Key indicators: single-crystal X-ray study; T = 193 K; mean σ(C–C) = 0.002 Å; R factor = 0.046; wR factor = 0.132; data-to-parameter ratio = 13.8.

In the title compound, $C_{16}H_{17}N_3O$, the benzotriazole ring forms a dihedral angle of 77.25 (6)° with the phenyl ring. The benzotriazole ring is essentially planar with a maximum deviation of 0.012 (19) Å. Weak intermolecular C—H···O hydrogen bonds form $R_2^2(10)$ motifs. The crystal packing is consolidated by π—π interactions with centroid–centroid distances of 3.5994 (12) Å together with very weak C—H···π interactions.

Related literature

For bond-length data, see: Allen *et al.* (1987). For graph-set analysis of hydrogen bonding, see: Bernstein *et al.* (1995).



Experimental

Crystal data

$C_{16}H_{17}N_3O$ M_r = 267.33
Monoclinic, $P2_1/c$
a = 8.6269 (19) Å
b = 7.3422 (4) Å
c = 21.890 (5) Å
β = 103.133 (11)°
V = 1350.2 (4) Å^3
Z = 4
Cu $K\alpha$ radiation
μ = 0.67 mm^{-1}
T = 193 K
0.35 × 0.20 × 0.10 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
Absorption correction: ψ scan (*CORINC*; Draeger & Gattow (1971)
T_{min} = 0.799, T_{max} = 0.936
2722 measured reflections
2545 independent reflections
2243 reflections with $I > 2\sigma(I)$
R_{int} = 0.091
3 standard reflections every 60 min
intensity decay: 2%

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.046
$wR(F^2)$ = 0.132
S = 1.09
2546 reflections
184 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.23 e Å^{-3}
$\Delta\rho_{min}$ = −0.26 e Å^{-3}

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

*Cg*3 is the centroid of the C12–C17 ring.

D—H···A	D—H	H···A	D···A	D—H···A
C6—H6···O10^{i}	0.95	2.35	3.190 (2)	147
C16—H16···O10^{ii}	0.95	2.58	3.506 (2)	165
C18—H18*A*···*Cg*3^{iii}	0.98	2.98	3.810 (18)	144

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

Data collection: *CAD-4 EXPRESS* (Enraf–Nonius, 1994); cell refinement: *CAD-4 EXPRESS*; data reduction: *CORINC* (Draeger & Gattow, 1971); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *SHELXTL* and *PLATON* (Spek, 2009).

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BT5182).

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supporting information

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S1. Comment

The asymmetric unit of (I) comprises of one molecule of the title compound (Fig 1). The bond lengths and angles are found to have normal values (Allen *et al.*, 1987). The benzotriazole ring is essentially planar with the maximum deviation from planarity being 0.012 (19) Å for atom C5. The mean plane of the benzotriazole ring (N1/N9/N8/C2—C7) forming a dihedral angle of 77.25 (6) Å with the mean plane of the phenyl ring (C12—C17). An intermolecular weak C—H···O hydrogen bonding generates a ring of motif $R_2^2(10)$ (Bernstein *et al.*, 1995)

The crystal packing is stabilized by π—π stacking interactions [*Cg*1—*Cg*2^{i}= of 3.5994 (12) Å; *Cg*1: (N1/N9/N8/C7/C2); *Cg*2:(C2—C): Symmetry code:(i) 1-*X*, –Y, –*Z*] together with weak C—H···π interactions.

S2. Experimental

A mixture of mono(bromomethyl)mesitylene (0.213 g, 1 mmol) and sodium salt of 1-Hydroxybenzotriazole (0.157,1 mmol) in ethanol (20 ml) was heated at 333 K with stirring for 30 min. The compound formed was filtered off, and dried. The compound was dissolved in ethanol and chloroform (1: 1v/v) and allowed to undergo slow evaporation. Colourless block shaped crystals were obtained after a week.

S3. Refinement

All the H atoms were positioned geometrically (C—H=0.95 Å (aromatic); C—H=0.98 (methyl) or C—H=0.99 Å (methylene) and refined using a riding model with, U_{iso}(H)=1.2U_{equ}(C, methylene) and 1.5U_{equ}(C_{methyl}). A rotating group model was used for the methyl groups.

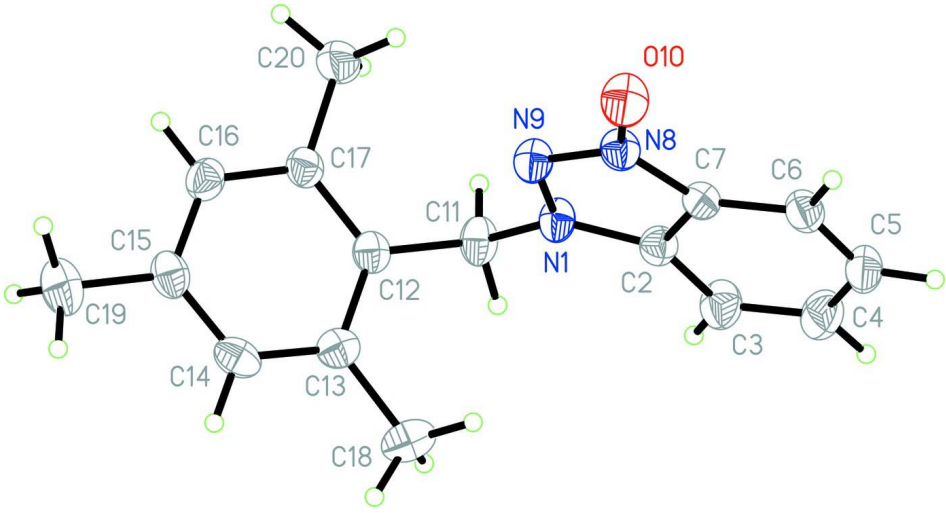

Figure 1

The molecular structure of the title compound, showing 50% probability displacement ellipsoids and the atom numbering scheme.

1-Mesitylmethyl-1*H*benzotriazole 3-oxide

Crystal data

$C_{16}H_{17}N_3O$
$M_r = 267.33$
Monoclinic, $P2_1/c$
Hall symbol: -P 2ybc
$a = 8.6269$ (19) Å
$b = 7.3422$ (4) Å
$c = 21.890$ (5) Å
$\beta = 103.133$ (11)°
$V = 1350.2$ (4) Å^3
$Z = 4$
$F(000) = 568$
$D_x = 1.315$ Mg m^{-3}
Cu $K\alpha$ radiation, $\lambda = 1.54178$ Å
Cell parameters from 25 reflections
$\theta = 35$–48°
$\mu = 0.67$ mm^{-1}
$T = 193$ K
Block, colourless
0.35 × 0.20 × 0.10 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
Radiation source: rotating anode
Graphite monochromator
$\omega/2\theta$ scans
Absorption correction: ψ scan (CORINC; Draeger & Gattow (1971))
$T_{min} = 0.799$, $T_{max} = 0.936$
2722 measured reflections
2545 independent reflections
2243 reflections with $I > 2\sigma(I)$
$R_{int} = 0.091$
$\theta_{max} = 69.9°$, $\theta_{min} = 4.2°$
$h = 0 \rightarrow 10$
$k = -8 \rightarrow 0$
$l = -26 \rightarrow 25$
3 standard reflections every 60 min
intensity decay: 2%

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.046$
$wR(F^2) = 0.132$
$S = 1.09$
2546 reflections
184 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

$w = 1/[\sigma^2(F_o^2) + (0.0705P)^2 + 0.4335P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$

$\Delta\rho_{max} = 0.23$ e Å^{-3}
$\Delta\rho_{min} = -0.26$ e Å^{-3}

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 $F^2 > \sigma(F^2)$ 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}
N1	0.70466 (16)	−0.01341 (18)	0.11411 (6)	0.0277 (3)
C2	0.71052 (18)	0.0150 (2)	0.05338 (7)	0.0272 (3)
C3	0.7611 (2)	−0.0918 (2)	0.00798 (8)	0.0369 (4)
H3	0.7999	−0.2123	0.0167	0.044*
C4	0.7513 (2)	−0.0124 (3)	−0.04902 (8)	0.0411 (4)
H4	0.7846	−0.0801	−0.0808	0.049*
C5	0.6933 (2)	0.1673 (3)	−0.06290 (8)	0.0364 (4)
H5	0.6900	0.2166	−0.1033	0.044*
C6	0.64222 (18)	0.2710 (2)	−0.01957 (7)	0.0317 (4)
H6	0.6023	0.3909	−0.0286	0.038*
C7	0.65231 (17)	0.1899 (2)	0.03868 (7)	0.0262 (3)
N8	0.61389 (15)	0.25395 (18)	0.09300 (6)	0.0285 (3)
N9	0.64505 (16)	0.13267 (18)	0.13845 (6)	0.0294 (3)
O10	0.56088 (16)	0.41370 (17)	0.10077 (6)	0.0416 (3)
C11	0.7402 (2)	−0.1818 (2)	0.15108 (7)	0.0308 (4)
H11A	0.8203	−0.2529	0.1352	0.037*
H11B	0.6421	−0.2562	0.1450	0.037*
C12	0.80200 (18)	−0.1471 (2)	0.22014 (7)	0.0258 (3)
C13	0.96499 (17)	−0.1154 (2)	0.24416 (7)	0.0278 (3)
C14	1.01987 (18)	−0.0911 (2)	0.30840 (8)	0.0307 (4)
H14	1.1305	−0.0728	0.3249	0.037*
C15	0.9183 (2)	−0.0928 (2)	0.34912 (7)	0.0311 (4)
C16	0.75646 (19)	−0.1203 (2)	0.32423 (7)	0.0307 (4)
H16	0.6850	−0.1197	0.3515	0.037*
C17	0.69733 (18)	−0.1485 (2)	0.26051 (7)	0.0272 (3)
C18	1.0814 (2)	−0.1051 (2)	0.20228 (9)	0.0396 (4)
H18A	1.0922	−0.2256	0.1844	0.059*
H18B	1.1852	−0.0648	0.2269	0.059*
H18C	1.0424	−0.0181	0.1683	0.059*
C19	0.9812 (2)	−0.0637 (3)	0.41845 (8)	0.0460 (5)
H19A	0.9922	−0.1816	0.4400	0.069*
H19B	0.9070	0.0132	0.4348	0.069*

H19C	1.0853	−0.0039	0.4257	0.069*
C20	0.52065 (19)	−0.1764 (3)	0.23637 (8)	0.0393 (4)
H20A	0.4771	−0.0771	0.2076	0.059*
H20B	0.4684	−0.1772	0.2717	0.059*
H20C	0.5017	−0.2929	0.2141	0.059*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N1	0.0360 (7)	0.0243 (7)	0.0232 (6)	0.0026 (5)	0.0075 (5)	0.0011 (5)
C2	0.0279 (7)	0.0279 (8)	0.0258 (7)	−0.0008 (6)	0.0060 (6)	−0.0003 (6)
C3	0.0467 (9)	0.0346 (9)	0.0297 (8)	0.0077 (7)	0.0096 (7)	−0.0025 (7)
C4	0.0477 (10)	0.0471 (11)	0.0304 (9)	0.0048 (8)	0.0131 (7)	−0.0050 (8)
C5	0.0394 (9)	0.0446 (10)	0.0249 (8)	−0.0020 (8)	0.0064 (7)	0.0047 (7)
C6	0.0304 (8)	0.0338 (9)	0.0290 (8)	0.0001 (6)	0.0028 (6)	0.0053 (7)
C7	0.0242 (7)	0.0283 (8)	0.0255 (7)	−0.0007 (6)	0.0042 (6)	−0.0001 (6)
N8	0.0315 (7)	0.0251 (7)	0.0284 (6)	0.0036 (5)	0.0060 (5)	0.0007 (5)
N9	0.0364 (7)	0.0255 (7)	0.0270 (6)	0.0032 (5)	0.0083 (5)	−0.0002 (5)
O10	0.0574 (8)	0.0284 (6)	0.0406 (7)	0.0164 (5)	0.0141 (6)	0.0016 (5)
C11	0.0442 (9)	0.0216 (8)	0.0261 (8)	0.0000 (6)	0.0070 (7)	0.0023 (6)
C12	0.0312 (8)	0.0195 (7)	0.0263 (7)	0.0013 (6)	0.0057 (6)	0.0023 (6)
C13	0.0304 (8)	0.0200 (7)	0.0344 (8)	0.0026 (6)	0.0099 (6)	0.0005 (6)
C14	0.0262 (7)	0.0231 (8)	0.0397 (9)	0.0012 (6)	0.0011 (6)	−0.0001 (6)
C15	0.0384 (8)	0.0250 (8)	0.0274 (8)	0.0030 (6)	0.0023 (6)	0.0019 (6)
C16	0.0349 (8)	0.0296 (8)	0.0295 (8)	0.0035 (6)	0.0112 (6)	0.0061 (6)
C17	0.0285 (7)	0.0250 (8)	0.0275 (8)	0.0003 (6)	0.0053 (6)	0.0065 (6)
C18	0.0378 (9)	0.0342 (9)	0.0523 (11)	−0.0014 (7)	0.0217 (8)	−0.0040 (8)
C19	0.0543 (11)	0.0473 (11)	0.0313 (9)	0.0008 (9)	−0.0006 (8)	−0.0014 (8)
C20	0.0290 (8)	0.0495 (11)	0.0383 (9)	−0.0042 (7)	0.0053 (7)	0.0096 (8)

Geometric parameters (Å, º)

N1—N9	1.3502 (18)	C12—C13	1.404 (2)
N1—C2	1.358 (2)	C13—C14	1.390 (2)
N1—C11	1.4713 (19)	C13—C18	1.508 (2)
C2—C7	1.390 (2)	C14—C15	1.384 (2)
C2—C3	1.411 (2)	C14—H14	0.9500
C3—C4	1.362 (2)	C15—C16	1.393 (2)
C3—H3	0.9500	C15—C19	1.506 (2)
C4—C5	1.419 (3)	C16—C17	1.388 (2)
C4—H4	0.9500	C16—H16	0.9500
C5—C6	1.365 (2)	C17—C20	1.509 (2)
C5—H5	0.9500	C18—H18A	0.9800
C6—C7	1.392 (2)	C18—H18B	0.9800
C6—H6	0.9500	C18—H18C	0.9800
C7—N8	1.387 (2)	C19—H19A	0.9800
N8—O10	1.2842 (17)	C19—H19B	0.9800
N8—N9	1.3166 (18)	C19—H19C	0.9800

C11—C12	1.506 (2)	C20—H20A	0.9800
C11—H11A	0.9900	C20—H20B	0.9800
C11—H11B	0.9900	C20—H20C	0.9800
C12—C17	1.400 (2)		

N9—N1—C2	111.49 (12)	C14—C13—C12	118.82 (14)
N9—N1—C11	120.06 (12)	C14—C13—C18	119.21 (14)
C2—N1—C11	128.19 (13)	C12—C13—C18	121.96 (15)
N1—C2—C7	106.08 (13)	C15—C14—C13	122.03 (14)
N1—C2—C3	133.66 (15)	C15—C14—H14	119.0
C7—C2—C3	120.26 (14)	C13—C14—H14	119.0
C4—C3—C2	116.27 (16)	C14—C15—C16	118.30 (14)
C4—C3—H3	121.9	C14—C15—C19	120.77 (15)
C2—C3—H3	121.9	C16—C15—C19	120.92 (15)
C3—C4—C5	122.64 (16)	C17—C16—C15	121.43 (14)
C3—C4—H4	118.7	C17—C16—H16	119.3
C5—C4—H4	118.7	C15—C16—H16	119.3
C6—C5—C4	121.56 (15)	C16—C17—C12	119.41 (14)
C6—C5—H5	119.2	C16—C17—C20	118.91 (14)
C4—C5—H5	119.2	C12—C17—C20	121.67 (14)
C5—C6—C7	115.80 (16)	C13—C18—H18A	109.5
C5—C6—H6	122.1	C13—C18—H18B	109.5
C7—C6—H6	122.1	H18A—C18—H18B	109.5
N8—C7—C2	105.00 (13)	C13—C18—H18C	109.5
N8—C7—C6	131.53 (15)	H18A—C18—H18C	109.5
C2—C7—C6	123.47 (15)	H18B—C18—H18C	109.5
O10—N8—N9	122.37 (13)	C15—C19—H19A	109.5
O10—N8—C7	125.79 (13)	C15—C19—H19B	109.5
N9—N8—C7	111.77 (13)	H19A—C19—H19B	109.5
N8—N9—N1	105.66 (12)	C15—C19—H19C	109.5
N1—C11—C12	113.08 (13)	H19A—C19—H19C	109.5
N1—C11—H11A	109.0	H19B—C19—H19C	109.5
C12—C11—H11A	109.0	C17—C20—H20A	109.5
N1—C11—H11B	109.0	C17—C20—H20B	109.5
C12—C11—H11B	109.0	H20A—C20—H20B	109.5
H11A—C11—H11B	107.8	C17—C20—H20C	109.5
C17—C12—C13	119.98 (14)	H20A—C20—H20C	109.5
C17—C12—C11	120.02 (14)	H20B—C20—H20C	109.5
C13—C12—C11	120.00 (14)		

N9—N1—C2—C7	0.47 (17)	C11—N1—N9—N8	−174.86 (13)
C11—N1—C2—C7	174.51 (14)	N9—N1—C11—C12	−35.7 (2)
N9—N1—C2—C3	−179.86 (17)	C2—N1—C11—C12	150.74 (15)
C11—N1—C2—C3	−5.8 (3)	N1—C11—C12—C17	95.32 (17)
N1—C2—C3—C4	−178.70 (17)	N1—C11—C12—C13	−85.41 (18)
C7—C2—C3—C4	0.9 (2)	C17—C12—C13—C14	1.8 (2)
C2—C3—C4—C5	−0.2 (3)	C11—C12—C13—C14	−177.43 (13)
C3—C4—C5—C6	−0.6 (3)	C17—C12—C13—C18	−177.49 (14)

C4—C5—C6—C7	0.6 (2)	C11—C12—C13—C18	3.2 (2)
N1—C2—C7—N8	−0.46 (16)	C12—C13—C14—C15	−1.6 (2)
C3—C2—C7—N8	179.81 (14)	C18—C13—C14—C15	177.75 (14)
N1—C2—C7—C6	178.74 (14)	C13—C14—C15—C16	0.1 (2)
C3—C2—C7—C6	−1.0 (2)	C13—C14—C15—C19	−179.23 (15)
C5—C6—C7—N8	179.14 (15)	C14—C15—C16—C17	1.1 (2)
C5—C6—C7—C2	0.2 (2)	C19—C15—C16—C17	−179.53 (16)
C2—C7—N8—O10	177.40 (14)	C15—C16—C17—C12	−0.8 (2)
C6—C7—N8—O10	−1.7 (3)	C15—C16—C17—C20	−179.64 (15)
C2—C7—N8—N9	0.32 (17)	C13—C12—C17—C16	−0.7 (2)
C6—C7—N8—N9	−178.79 (15)	C11—C12—C17—C16	178.61 (14)
O10—N8—N9—N1	−177.24 (13)	C13—C12—C17—C20	178.11 (15)
C7—N8—N9—N1	−0.04 (17)	C11—C12—C17—C20	−2.6 (2)
C2—N1—N9—N8	−0.28 (17)		

Hydrogen-bond geometry (Å, º)

Cg3 is the centroid of the C12–C17 ring.

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C6—H6···O10^{i}	0.95	2.35	3.190 (2)	147
C16—H16···O10^{ii}	0.95	2.58	3.506 (2)	165
C18—H18*A*···*Cg*3^{iii}	0.98	2.98	3.810 (18)	144

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