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2,6-Diazidotoluene

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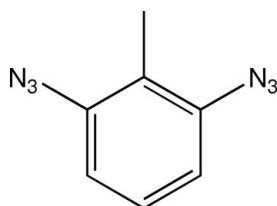
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Key indicators: single-crystal X-ray study; $T = 200$ K; mean $\sigma(\text{C}–\text{C}) = 0.002$ Å; disorder in main residue; R factor = 0.030; wR factor = 0.061; data-to-parameter ratio = 11.0.

The structure of the title compound, $\text{C}_7\text{H}_6\text{N}_6$, consists of almost planar molecules with $\text{C}–\text{N}$ distances of 1.429 (2) and 1.428 (2) Å. The H atoms of the methyl group are disordered over two sites with occupancy factors of 0.69 and 0.31. The azide groups show typical geometry for covalently bound azides.

Related literature

The preparation of the title compound by a slightly different procedure was reported by Chapyshev & Tomioka (2003). For the comparable compound, 2-azidobenzyl bromide, see: Klapötke *et al.* (2003).



Experimental

Crystal data

$\text{C}_7\text{H}_6\text{N}_6$
 $M_r = 174.16$
Orthorhombic, $Pccn$
 $a = 12.298$ (5) Å
 $b = 25.896$ (5) Å
 $c = 5.085$ (5) Å

$V = 1619.4$ (18) Å³
 $Z = 8$
Mo $K\alpha$ radiation
 $\mu = 0.10$ mm⁻¹
 $T = 200$ (2) K
0.29 × 0.14 × 0.13 mm

Data collection

Oxford Xcalibur3 CCD area-detector diffractometer
Absorption correction: multi-scan (*CrysAlis RED*; Oxford Diffraction, 2007)
 $T_{\min} = 0.899$, $T_{\max} = 0.990$

5871 measured reflections
1578 independent reflections
956 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.040$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.030$
 $wR(F^2) = 0.061$
 $S = 0.94$
1578 reflections
143 parameters

H atoms treated by a mixture of independent and constrained refinement
 $\Delta\rho_{\text{max}} = 0.10$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.12$ e Å⁻³

Data collection: *CrysAlis CCD* (Oxford Diffraction, 2007); cell refinement: *CrysAlis RED* (Oxford Diffraction, 2007); data reduction: *CrysAlis RED*; program(s) used to solve structure: *SIR97* (Altomare *et al.*, 1999); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *DIAMOND* (Brandenburg, 1996); software used to prepare material for publication: *SHELXL97*.

The University of Munich, the Fonds der Chemischen Industrie, and the Deutsche Forschungsgemeinschaft (KL 636/10–1) are gratefully acknowledged for financial support.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: FJ2092).

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supporting information

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2,6-Diazidotoluene

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S1. Comment

The structure of the title compound exhibits C–N distances [C2–N1 1.429 (2) and C6–N4 1.428 (2) Å] similar to the distance in 2-N₃C₆H₄CH₂Br [1.428 (5) Å, Klapötke *et al.*, 2003]. The values of both azide groups are in the common range for covalent azide groups with longer N_α–N_β distances [N1–N2 1.241 (2) and N4–N5 1.253 (2) Å] and shorter terminal N_β–N_γ distances [N2–N3 1.133 (2) and N5–N6 1.122 (2) Å] with more triple bond character. The azide angles are slightly bent [N1–N2–N3 172.98 (18) and N4–N5–N6 172.36 (17)°].

S2. Experimental

The title compound was prepared according the literature [Chapyshev & Tomioka (2003)], slightly modified, *e.g.* the column chromatography was performed with hexane/chloroform (1:4) as an eluent. Colorless crystals were obtained by slow evaporation of a chloroform solution. ¹H NMR (400 MHz, CDCl₃, Me₄Si): δ 7.23 (tq, 4-H, ³J_{H–H} = 8.1 Hz, ⁶J_{H–H} = 0.5 Hz, 1H), 6.89 (d, 3-H, 2H), 2.05 (m, CH₃, 3H) p.p.m.; ¹³C NMR (400 MHz, CDCl₃, Me₄Si): δ 140.0/127.2/121.3/114.0 (Ar–C), 11.2 (CH₃) p.p.m.; ¹⁵N NMR (400 MHz, CDCl₃, MeNO₂) δ -139.5 (N_β), -149.0 (N_γ), -291.4 (N_α) p.p.m..

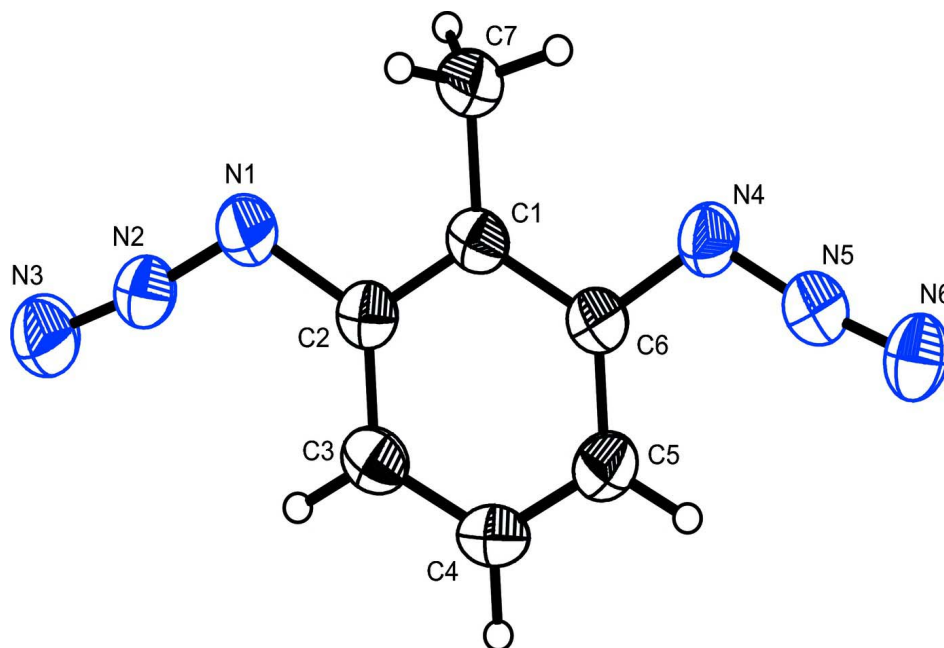


Figure 1

Molecular structure of C₇H₆N₆ with displacement ellipsoids drawn at the 50% probability level. The minor disorder component of the methyl hydrogen atoms has been omitted.

2,6-Diazidotoluene

Crystal data

C₇H₆N₂ $M_r = 174.16$ Orthorhombic, *Pccn*

Hall symbol: -P 2ab 2ac

 $a = 12.298 (5) \text{ \AA}$ $b = 25.896 (5) \text{ \AA}$ $c = 5.085 (5) \text{ \AA}$ $V = 1619.4 (18) \text{ \AA}^3$ $Z = 8$ $F(000) = 720$ $D_x = 1.429 (1) \text{ Mg m}^{-3}$ Mo $K\alpha$ radiation, $\lambda = 0.71073 \text{ \AA}$

Cell parameters from 1382 reflections

 $\theta = 3.9\text{--}30.0^\circ$ $\mu = 0.10 \text{ mm}^{-1}$ $T = 200 \text{ K}$

Needle, colorless

 $0.29 \times 0.14 \times 0.13 \text{ mm}$

Data collection

Oxford Xcalibur3 CCD area-detector
diffractometer

Radiation source: fine-focus sealed tube

Graphite monochromator

Detector resolution: 15.9809 pixels mm^{-1} ω scans

Absorption correction: multi-scan

(CrysAlis RED; Oxford Diffraction, 2007) $T_{\min} = 0.899$, $T_{\max} = 0.990$

5871 measured reflections

1578 independent reflections

956 reflections with $I > 2\sigma(I)$ $R_{\text{int}} = 0.040$ $\theta_{\max} = 26.0^\circ$, $\theta_{\min} = 4.3^\circ$ $h = -15 \rightarrow 9$ $k = -19 \rightarrow 31$ $l = -5 \rightarrow 6$

Refinement

Refinement on F^2

Least-squares matrix: full

 $R[F^2 > 2\sigma(F^2)] = 0.030$ $wR(F^2) = 0.061$ $S = 0.94$

1578 reflections

143 parameters

0 restraints

Primary atom site location: structure-invariant
direct methodsSecondary atom site location: difference Fourier
mapHydrogen site location: inferred from
neighbouring sitesH atoms treated by a mixture of independent
and constrained refinement $w = 1/[\sigma^2(F_o^2) + (0.0396P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$ $(\Delta/\sigma)_{\max} < 0.001$ $\Delta\rho_{\max} = 0.11 \text{ e \AA}^{-3}$ $\Delta\rho_{\min} = -0.12 \text{ e \AA}^{-3}$

Special details

Refinement. Aromatic H atoms were placed in idealized positions and allowed to ride on their respective parent atoms, with $C\text{--}H = 0.95$ ($C_{\text{arom}}\text{--}H$) and with $U_{\text{iso}}(H) = kU_{\text{eq}}(\text{carrier atom})$, where $k = 1.2$ for CH. The H atoms attached to methyl C7 are disordered over two sites. These were freely refined; the occupancy of the major disorder component is 0.69 (5). The highest peak and deepest hole in the final difference map were located 0.93 \AA from atom H7B and 0.32 \AA from atom H7A, respectively.

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

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$	Occ. (<1)
C1	0.45618 (11)	0.14719 (5)	0.0665 (3)	0.0348 (4)	
C2	0.45326 (12)	0.10499 (5)	-0.1031 (3)	0.0353 (4)	
C3	0.53072 (12)	0.06637 (6)	-0.0938 (3)	0.0415 (4)	
H3	0.5271	0.0383	-0.2136	0.050*	
C4	0.61302 (13)	0.06857 (6)	0.0888 (3)	0.0426 (4)	
H4	0.6662	0.0420	0.0957	0.051*	

C5	0.61819 (12)	0.10957 (5)	0.2622 (3)	0.0405 (4)	
H5	0.6745	0.1111	0.3899	0.049*	
C6	0.54110 (12)	0.14841 (5)	0.2495 (3)	0.0353 (4)	
C7	0.37338 (17)	0.18935 (8)	0.0524 (5)	0.0462 (4)	
H7A	0.297 (2)	0.1729 (9)	0.075 (5)	0.042 (10)*	0.69 (5)
H7B	0.385 (3)	0.2132 (11)	0.190 (8)	0.073 (11)*	0.69 (5)
H7C	0.381 (3)	0.2054 (10)	-0.129 (7)	0.060 (11)*	0.69 (5)
H7D	0.323 (4)	0.1895 (16)	-0.102 (10)	0.010 (16)*	0.31 (5)
H7E	0.334 (4)	0.193 (2)	0.231 (14)	0.04 (2)*	0.31 (5)
H7F	0.410 (4)	0.2239 (18)	0.031 (10)	0.015 (18)*	0.31 (5)
N1	0.36418 (10)	0.10504 (5)	-0.2837 (2)	0.0440 (4)	
N2	0.35797 (10)	0.06789 (5)	-0.4371 (3)	0.0419 (3)	
N3	0.34235 (11)	0.03628 (5)	-0.5865 (3)	0.0564 (4)	
N4	0.54156 (10)	0.19271 (4)	0.4164 (3)	0.0440 (3)	
N5	0.61378 (11)	0.19404 (4)	0.5897 (3)	0.0409 (3)	
N6	0.67279 (12)	0.19998 (5)	0.7553 (3)	0.0528 (4)	

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0329 (8)	0.0333 (8)	0.0382 (9)	-0.0034 (8)	0.0065 (8)	0.0026 (8)
C2	0.0346 (9)	0.0381 (8)	0.0332 (9)	-0.0052 (8)	0.0037 (8)	0.0030 (8)
C3	0.0440 (9)	0.0367 (8)	0.0436 (10)	0.0011 (9)	0.0053 (9)	-0.0017 (8)
C4	0.0415 (9)	0.0384 (8)	0.0480 (10)	0.0065 (9)	0.0043 (10)	0.0052 (9)
C5	0.0379 (9)	0.0444 (9)	0.0393 (9)	-0.0016 (9)	-0.0016 (9)	0.0050 (8)
C6	0.0367 (9)	0.0352 (8)	0.0340 (8)	-0.0039 (8)	0.0038 (8)	0.0013 (8)
C7	0.0428 (11)	0.0422 (10)	0.0537 (13)	0.0029 (11)	-0.0036 (12)	-0.0064 (12)
N1	0.0455 (8)	0.0406 (7)	0.0460 (8)	-0.0019 (7)	-0.0033 (7)	-0.0088 (7)
N2	0.0408 (8)	0.0462 (8)	0.0387 (8)	-0.0031 (8)	-0.0005 (7)	0.0000 (8)
N3	0.0611 (9)	0.0572 (9)	0.0510 (9)	-0.0003 (8)	-0.0051 (8)	-0.0148 (8)
N4	0.0455 (8)	0.0477 (8)	0.0388 (8)	-0.0006 (7)	-0.0065 (8)	-0.0063 (7)
N5	0.0455 (8)	0.0416 (8)	0.0357 (8)	-0.0052 (7)	0.0065 (8)	0.0007 (7)
N6	0.0545 (8)	0.0650 (10)	0.0389 (9)	-0.0080 (8)	-0.0047 (8)	-0.0028 (7)

Geometric parameters (Å, °)

C1—C2	1.393 (2)	C6—N4	1.4270 (19)
C1—C6	1.399 (2)	C7—H7A	1.04 (3)
C1—C7	1.495 (2)	C7—H7B	0.94 (3)
C2—C3	1.382 (2)	C7—H7C	1.02 (3)
C2—N1	1.4294 (19)	C7—H7D	1.00 (5)
C3—C4	1.375 (2)	C7—H7E	1.04 (7)
C3—H3	0.9500	C7—H7F	1.01 (5)
C4—C5	1.382 (2)	N1—N2	1.2410 (17)
C4—H4	0.9500	N2—N3	1.1331 (17)
C5—C6	1.3836 (19)	N4—N5	1.2518 (19)
C5—H5	0.9500	N5—N6	1.1222 (17)

C2—C1—C6	116.67 (13)	H7A—C7—H7C	111 (2)
C2—C1—C7	121.72 (16)	H7B—C7—H7C	113 (2)
C6—C1—C7	121.60 (15)	C1—C7—H7D	118 (2)
C3—C2—C1	121.93 (15)	H7A—C7—H7D	62 (2)
C3—C2—N1	123.42 (13)	H7B—C7—H7D	132 (3)
C1—C2—N1	114.65 (14)	H7C—C7—H7D	49 (2)
C4—C3—C2	120.03 (15)	C1—C7—H7E	110 (2)
C4—C3—H3	120.0	H7A—C7—H7E	61 (3)
C2—C3—H3	120.0	H7B—C7—H7E	50 (3)
C3—C4—C5	119.80 (15)	H7C—C7—H7E	143 (3)
C3—C4—H4	120.1	H7D—C7—H7E	113 (3)
C5—C4—H4	120.1	C1—C7—H7F	110 (2)
C4—C5—C6	119.80 (14)	H7A—C7—H7F	141 (2)
C4—C5—H5	120.1	H7B—C7—H7F	55 (2)
C6—C5—H5	120.1	H7C—C7—H7F	60 (2)
C5—C6—C1	121.76 (13)	H7D—C7—H7F	101 (3)
C5—C6—N4	123.63 (14)	H7E—C7—H7F	103 (3)
C1—C6—N4	114.61 (13)	N2—N1—C2	116.77 (13)
C1—C7—H7A	108.3 (11)	N3—N2—N1	172.70 (16)
C1—C7—H7B	109.8 (17)	N5—N4—C6	116.34 (13)
H7A—C7—H7B	109 (3)	N6—N5—N4	172.29 (15)
C1—C7—H7C	106.0 (15)		
C6—C1—C2—C3	-0.6 (2)	C4—C5—C6—N4	-178.50 (14)
C7—C1—C2—C3	178.86 (16)	C2—C1—C6—C5	-0.2 (2)
C6—C1—C2—N1	179.09 (12)	C7—C1—C6—C5	-179.69 (16)
C7—C1—C2—N1	-1.5 (2)	C2—C1—C6—N4	179.16 (13)
C1—C2—C3—C4	0.8 (2)	C7—C1—C6—N4	-0.3 (2)
N1—C2—C3—C4	-178.84 (13)	C3—C2—N1—N2	0.2 (2)
C2—C3—C4—C5	-0.2 (2)	C1—C2—N1—N2	-179.43 (13)
C3—C4—C5—C6	-0.6 (2)	C5—C6—N4—N5	-3.5 (2)
C4—C5—C6—C1	0.8 (2)	C1—C6—N4—N5	177.14 (13)
