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Crystal structure of 1-nitro-4-(trimethylsilylethynyl)naphthalene

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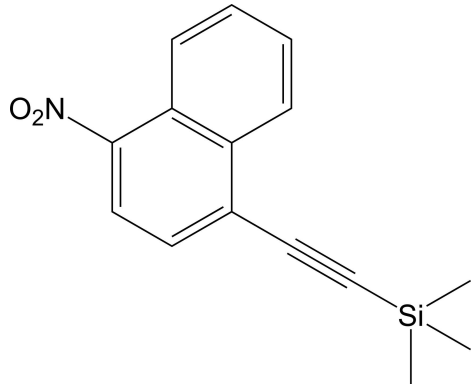
In the title compound, $C_{15}H_{15}NO_2Si$, the dihedral angle between the nitro group and the mean plane of the naphthalene system is 22.04 (11)°. In the crystal, π–π interactions generate supramolecular chains propagating along the *a*-axis direction; the centroid-to-centroid distances range from 3.5590 (12) to 3.8535 (12) Å.

Keywords: crystal structure; trialkylsilylacetylene; nitroarene; π–π interactions.

CCDC reference: 1058939

1. Related literature

For the syntheses of arylalkynes by Sonogashira coupling, see: Takahashi *et al.* (1980). For desilylation of the related 1-nitro-4-(trimethylsilylethynyl)benzene and its use in the construction of metal alkynyl complexes with enhanced non-linear optical properties, see: McDonagh *et al.* (1996*a*,*b*, 2003); Garcia *et al.* (2002). For related structures, see: Squadrito *et al.* (1990); Khan *et al.* (2004).



2. Experimental

2.1. Crystal data

$C_{15}H_{15}NO_2Si$
M_r = 269.37
Triclinic, $P\overline{1}$
a = 6.9679 (9) Å
b = 9.2425 (12) Å
c = 11.799 (1) Å
α = 100.242 (9)°
β = 99.698 (9)°
γ = 107.127 (12)°
V = 694.62 (15) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.17 mm^{-1}
T = 150 K
0.23 × 0.07 × 0.04 mm

2.2. Data collection

Agilent SuperNova (Dual, Cu at zero, EosS2) diffractometer
Absorption correction: analytical [*CrysAlis PRO* (Agilent, 2014), based on expressions derived by Clark & Reid (1995)]
T_{min} = 0.986, T_{max} = 0.996
4695 measured reflections
3112 independent reflections
2621 reflections with $I > 2\sigma(I)$
R_{int} = 0.021

2.3. Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.044
$wR(F^2)$ = 0.114
S = 1.07
3112 reflections
175 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.36 e Å^{-3}
$\Delta\rho_{min}$ = −0.23 e Å^{-3}

Data collection: *CrysAlis PRO* (Agilent, 2014); cell refinement: *CrysAlis PRO*; data reduction: *CrysAlis PRO*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL2013* (Sheldrick, 2015); molecular graphics: *OLEX2* (Dolomanov *et al.*, 2009); software used to prepare material for publication: *OLEX2*.

Acknowledgements

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Supporting information for this paper is available from the IUCr electronic archives (Reference: XU5846).

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supporting information

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Crystal structure of 1-nitro-4-(trimethylsilylethynyl)naphthalene

Jun Du and Graeme J. Moxey

S1. Synthesis and crystallization

1-Iodo-4-nitronaphthalene (1.196 g, 4.00 mmol) was added to triethylamine (30 mL) and the mixture deoxygenated and charged with nitrogen. $PdCl_2(PPh_3)_2$ (12 mg, 0.016 mmol), CuI (6 mg, 0.03 mmol) and trimethylsilylacetylene (0.7 mL, 5.00 mmol) were added and the reaction heated to 35 °C overnight. The solution was filtered through filter paper, washing with triethylamine (10 mL), and the solvent was removed from the filtrate. The residue was then passed through a short pad of silica, eluting with 4:1 petrol:CH_2Cl_2. Reduction in volume of the eluate afforded the product as a yellow solid (1.034 g, 96%). Anal. Calc. for $C_{15}H_{15}NO_2Si$: C, 66.88; H, 5.61; N, 5.20. Found: C, 66.67; H, 5.68; N, 5.28%. ^{1}H NMR (δ, 400 MHz, $CDCl_3$): 8.55 (d, J_{HH} = 8.0 Hz, 1H, H_8), 8.47 (d, J_{HH} = 8.0 Hz, 1H, H_5), 8.15 (d, J_{HH} = 8.0 Hz, 1H, H_{11}), 7.79 – 7.65 (m, 3H, H_4, H_9, H_{10}), 0.36 (s, 9H, Me); ^{13}C NMR (δ, 101 MHz, $CDCl_3$): 146.3 (C_6), 134.4 (C_{12}), 129.8 (C_9), 128.9 (C_4), 128.2 (C_{11}), 127.7 (C_3), 127.1 (C_{10}), 125.1 (C_7), 123.5 (C_8), 123.3 (C_5), 105.1 (C_2), 101.4 (C_1), 0.1 (s, Me); IR (ATR, cm^{-1}): 2956, 2156, 1507, 1323. Bright yellow crystals of the title compound were obtained by diffusion of methanol into a dichloromethane solution.

S2. Refinement

Crystal data, data collection and structure refinement details are summarized below.

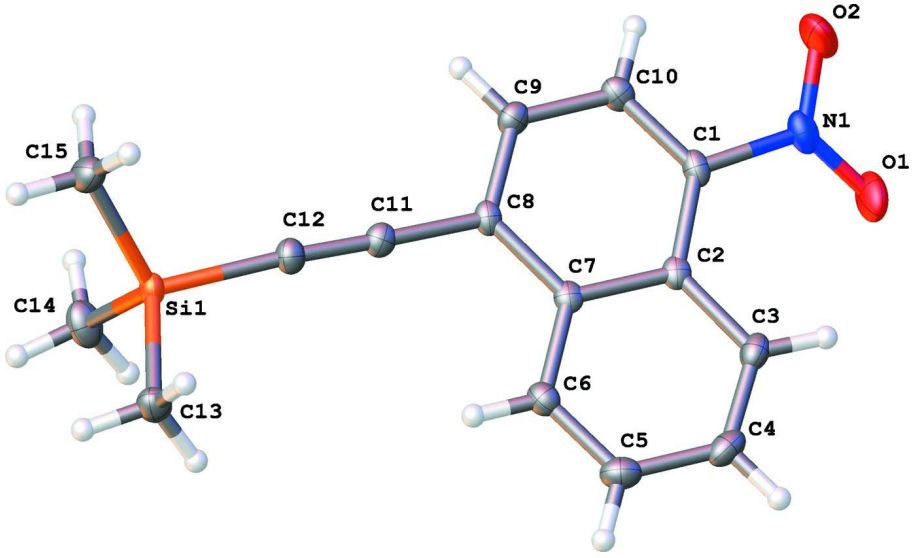

Figure 1

Molecular structure of 1-nitro-4-(trimethylsilylethynyl)naphthalene, with displacement ellipsoids set at the 40% probability level.

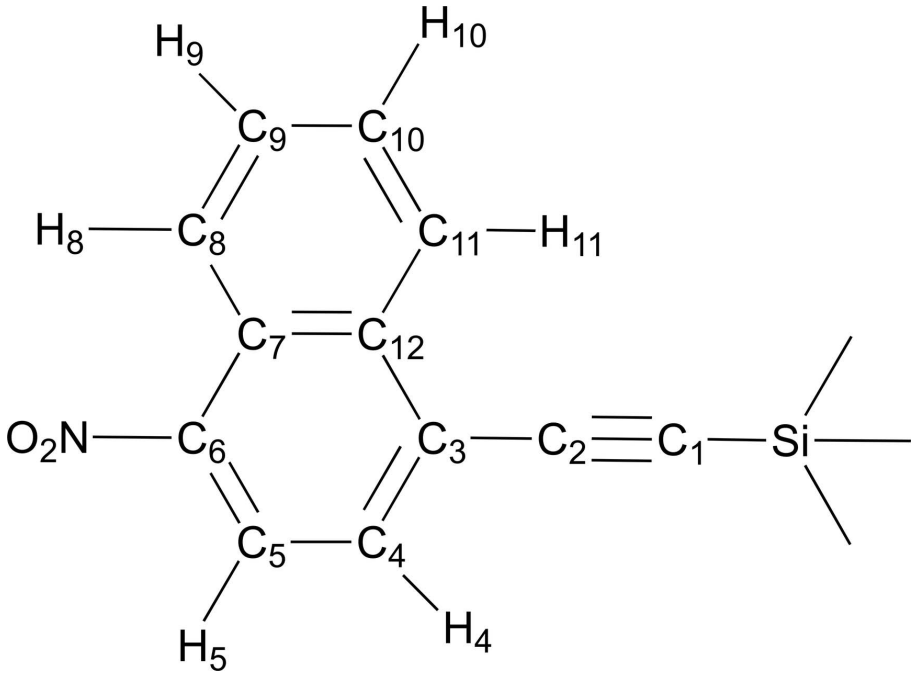

Figure 2

Atom numbering scheme of 1-nitro-4-(trimethylsilylethynyl)naphthalene for ^{1}H and ^{13}C NMR assignments.

1-Nitro-4-(trimethylsilylethynyl)naphthalene

Crystal data

$C_{15}H_{15}NO_2Si$
$M_r = 269.37$
Triclinic, $P\overline{1}$
$a = 6.9679$ (9) Å
$b = 9.2425$ (12) Å
$c = 11.799$ (1) Å
$\alpha = 100.242$ (9)°
$\beta = 99.698$ (9)°
$\gamma = 107.127$ (12)°
$V = 694.62$ (15) Å^3
$Z = 2$
$F(000) = 284$
$D_x = 1.288$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 1967 reflections
$\theta = 2.6$–$28.3°$
$\mu = 0.17$ mm^{-1}
$T = 150$ K
Needle, yellow
0.23 × 0.07 × 0.04 mm

Data collection

Agilent SuperNova (Dual, Cu at zero, EosS2) diffractometer
Radiation source: SuperNova (Mo) X-ray Source
Mirror monochromator
Detector resolution: 8.1297 pixels mm^{-1}
ω scans
Absorption correction: analytical [*CrysAlis PRO* (Agilent, 2014), based on expressions derived by Clark & Reid (1995)]
$T_{min} = 0.986$, $T_{max} = 0.996$
4695 measured reflections
3112 independent reflections
2621 reflections with $I > 2\sigma(I)$
$R_{int} = 0.021$
$\theta_{max} = 29.2°$, $\theta_{min} = 1.8°$
$h = -6 \rightarrow 9$
$k = -11 \rightarrow 12$
$l = -15 \rightarrow 15$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.044$
$wR(F^2) = 0.114$
$S = 1.07$
3112 reflections
175 parameters
0 restraints

Primary atom site location: structure-invariant direct methods
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0415P)^2 + 0.3469P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.36$ e Å^{-3}
$\Delta\rho_{min} = -0.23$ e Å^{-3}

Special details

Experimental. Absorption correction: CrysAlis Pro (Agilent Technologies, 2014) Analytical numeric absorption correction using a multifaceted crystal model based on expressions derived by R.C. Clark & J.S. Reid. (Clark & Reid, 1995). Empirical absorption correction using spherical harmonics, implemented in SCALE3 ABSPACK scaling algorithm.

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 (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
C1	0.2485 (3)	0.6318 (2)	0.44838 (15)	0.0204 (4)
C2	0.1929 (2)	0.4663 (2)	0.41137 (15)	0.0188 (4)
C3	0.1112 (3)	0.3754 (2)	0.29263 (16)	0.0247 (4)
H3	0.0875	0.4239	0.2316	0.030*
C4	0.0675 (3)	0.2178 (2)	0.26757 (17)	0.0293 (4)
H4	0.0147	0.1606	0.1893	0.035*
C5	0.1001 (3)	0.1400 (2)	0.35659 (18)	0.0293 (4)
H5	0.0661	0.0321	0.3376	0.035*
C6	0.1819 (3)	0.2230 (2)	0.47120 (17)	0.0234 (4)
H6	0.2048	0.1711	0.5301	0.028*
C7	0.2324 (2)	0.3874 (2)	0.50191 (15)	0.0182 (4)
C8	0.3257 (3)	0.4749 (2)	0.62159 (15)	0.0187 (4)
C9	0.3745 (3)	0.6352 (2)	0.65052 (15)	0.0220 (4)
H9	0.4329	0.6912	0.7288	0.026*
C10	0.3369 (3)	0.7131 (2)	0.56329 (16)	0.0225 (4)
H10	0.3720	0.8211	0.5832	0.027*
C11	0.3756 (3)	0.3974 (2)	0.71220 (15)	0.0215 (4)
C12	0.4216 (3)	0.3354 (2)	0.78877 (16)	0.0235 (4)
C13	0.4757 (3)	0.0381 (2)	0.83030 (17)	0.0300 (4)
H13A	0.3530	−0.0142	0.7682	0.045*
H13B	0.4823	−0.0237	0.8873	0.045*
H13C	0.5949	0.0523	0.7973	0.045*
C14	0.2518 (3)	0.2089 (3)	0.97894 (19)	0.0361 (5)
H14A	0.1234	0.1699	0.9203	0.054*
H14B	0.2647	0.3083	1.0269	0.054*

H14C	0.2547	0.1369	1.0281	0.054*
C15	0.7217 (3)	0.3443 (2)	1.00937 (18)	0.0327 (5)
H15A	0.7507	0.2860	1.0658	0.049*
H15B	0.7161	0.4421	1.0501	0.049*
H15C	0.8287	0.3633	0.9667	0.049*
N1	0.2165 (3)	0.7285 (2)	0.36481 (15)	0.0275 (4)
O1	0.0903 (2)	0.6698 (2)	0.27072 (14)	0.0437 (4)
O2	0.3213 (3)	0.86743 (19)	0.39546 (15)	0.0532 (5)
Si1	0.47002 (8)	0.23164 (6)	0.90416 (4)	0.02062 (14)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0176 (8)	0.0279 (10)	0.0240 (9)	0.0126 (7)	0.0093 (7)	0.0138 (7)
C2	0.0125 (7)	0.0262 (9)	0.0212 (8)	0.0085 (7)	0.0063 (7)	0.0086 (7)
C3	0.0193 (9)	0.0349 (11)	0.0191 (9)	0.0081 (8)	0.0036 (7)	0.0076 (8)
C4	0.0228 (9)	0.0372 (12)	0.0213 (9)	0.0066 (8)	0.0030 (8)	−0.0012 (8)
C5	0.0256 (10)	0.0240 (10)	0.0350 (11)	0.0071 (8)	0.0065 (8)	0.0016 (8)
C6	0.0211 (9)	0.0239 (10)	0.0280 (9)	0.0093 (7)	0.0067 (8)	0.0093 (8)
C7	0.0123 (7)	0.0238 (9)	0.0212 (8)	0.0077 (7)	0.0064 (7)	0.0074 (7)
C8	0.0160 (8)	0.0264 (9)	0.0199 (8)	0.0114 (7)	0.0078 (7)	0.0100 (7)
C9	0.0217 (9)	0.0262 (10)	0.0193 (8)	0.0103 (7)	0.0065 (7)	0.0033 (7)
C10	0.0233 (9)	0.0229 (9)	0.0267 (9)	0.0124 (8)	0.0105 (8)	0.0073 (7)
C11	0.0196 (8)	0.0267 (10)	0.0208 (9)	0.0105 (7)	0.0070 (7)	0.0054 (7)
C12	0.0248 (9)	0.0274 (10)	0.0217 (9)	0.0110 (8)	0.0078 (7)	0.0083 (7)
C13	0.0396 (11)	0.0241 (10)	0.0272 (10)	0.0113 (9)	0.0099 (9)	0.0058 (8)
C14	0.0410 (12)	0.0438 (13)	0.0373 (11)	0.0215 (10)	0.0217 (10)	0.0197 (10)
C15	0.0364 (11)	0.0296 (11)	0.0282 (10)	0.0100 (9)	−0.0004 (9)	0.0066 (8)
N1	0.0294 (9)	0.0352 (10)	0.0313 (9)	0.0193 (8)	0.0158 (7)	0.0186 (8)
O1	0.0390 (9)	0.0558 (11)	0.0400 (9)	0.0167 (8)	−0.0007 (7)	0.0290 (8)
O2	0.0889 (14)	0.0287 (9)	0.0447 (10)	0.0188 (9)	0.0129 (9)	0.0198 (8)
Si1	0.0252 (3)	0.0222 (3)	0.0169 (2)	0.0097 (2)	0.00518 (19)	0.00766 (19)

Geometric parameters (Å, º)

C1—C2	1.427 (3)	C10—H10	0.9300
C1—C10	1.366 (3)	C11—C12	1.201 (2)
C1—N1	1.476 (2)	C12—Si1	1.8403 (19)
C2—C3	1.422 (3)	C13—H13A	0.9600
C2—C7	1.430 (2)	C13—H13B	0.9600
C3—H3	0.9300	C13—H13C	0.9600
C3—C4	1.363 (3)	C13—Si1	1.860 (2)
C4—H4	0.9300	C14—H14A	0.9600
C4—C5	1.399 (3)	C14—H14B	0.9600
C5—H5	0.9300	C14—H14C	0.9600
C5—C6	1.362 (3)	C14—Si1	1.862 (2)
C6—H6	0.9300	C15—H15A	0.9600
C6—C7	1.418 (3)	C15—H15B	0.9600

C7—C8	1.430 (2)	C15—H15C	0.9600
C8—C9	1.382 (3)	C15—Si1	1.853 (2)
C8—C11	1.439 (2)	N1—O1	1.215 (2)
C9—H9	0.9300	N1—O2	1.228 (2)
C9—C10	1.390 (2)		

C2—C1—N1	122.38 (16)	C12—C11—C8	178.5 (2)
C10—C1—C2	122.82 (16)	C11—C12—Si1	175.42 (17)
C10—C1—N1	114.80 (16)	H13A—C13—H13B	109.5
C1—C2—C7	116.38 (15)	H13A—C13—H13C	109.5
C3—C2—C1	125.72 (16)	H13B—C13—H13C	109.5
C3—C2—C7	117.84 (17)	Si1—C13—H13A	109.5
C2—C3—H3	119.7	Si1—C13—H13B	109.5
C4—C3—C2	120.51 (17)	Si1—C13—H13C	109.5
C4—C3—H3	119.7	H14A—C14—H14B	109.5
C3—C4—H4	119.2	H14A—C14—H14C	109.5
C3—C4—C5	121.65 (18)	H14B—C14—H14C	109.5
C5—C4—H4	119.2	Si1—C14—H14A	109.5
C4—C5—H5	120.1	Si1—C14—H14B	109.5
C6—C5—C4	119.74 (18)	Si1—C14—H14C	109.5
C6—C5—H5	120.1	H15A—C15—H15B	109.5
C5—C6—H6	119.6	H15A—C15—H15C	109.5
C5—C6—C7	120.89 (17)	H15B—C15—H15C	109.5
C7—C6—H6	119.6	Si1—C15—H15A	109.5
C2—C7—C8	119.87 (16)	Si1—C15—H15B	109.5
C6—C7—C2	119.33 (16)	Si1—C15—H15C	109.5
C6—C7—C8	120.79 (16)	O1—N1—C1	119.96 (17)
C7—C8—C11	120.25 (16)	O1—N1—O2	123.02 (17)
C9—C8—C7	120.30 (16)	O2—N1—C1	117.02 (17)
C9—C8—C11	119.42 (16)	C12—Si1—C13	107.97 (9)
C8—C9—H9	119.8	C12—Si1—C14	106.63 (9)
C8—C9—C10	120.33 (16)	C12—Si1—C15	109.92 (9)
C10—C9—H9	119.8	C13—Si1—C14	110.88 (10)
C1—C10—C9	120.29 (17)	C15—Si1—C13	109.63 (10)
C1—C10—H10	119.9	C15—Si1—C14	111.70 (10)
C9—C10—H10	119.9		

C1—C2—C3—C4	178.69 (16)	C5—C6—C7—C8	−177.53 (16)
C1—C2—C7—C6	−179.69 (14)	C6—C7—C8—C9	179.94 (15)
C1—C2—C7—C8	−0.9 (2)	C6—C7—C8—C11	1.9 (2)
C2—C1—C10—C9	−0.7 (3)	C7—C2—C3—C4	1.7 (2)
C2—C1—N1—O1	21.6 (2)	C7—C8—C9—C10	−1.2 (2)
C2—C1—N1—O2	−158.72 (17)	C8—C9—C10—C1	0.9 (3)
C2—C3—C4—C5	0.2 (3)	C10—C1—C2—C3	−176.32 (16)
C2—C7—C8—C9	1.2 (2)	C10—C1—C2—C7	0.7 (2)
C2—C7—C8—C11	−176.86 (14)	C10—C1—N1—O1	−158.99 (17)
C3—C2—C7—C6	−2.4 (2)	C10—C1—N1—O2	20.7 (2)
C3—C2—C7—C8	176.36 (15)	C11—C8—C9—C10	176.88 (15)

C3—C4—C5—C6	−1.5 (3)	N1—C1—C2—C3	3.1 (3)
C4—C5—C6—C7	0.7 (3)	N1—C1—C2—C7	−179.89 (14)
C5—C6—C7—C2	1.2 (2)	N1—C1—C10—C9	179.83 (14)