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N-Ethyl-*N*-phenyl-*N*′-tosylformamidine

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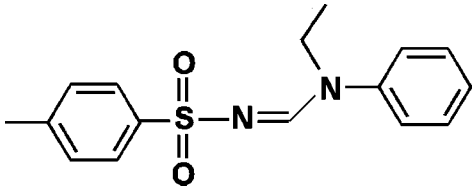
Key indicators: single-crystal X-ray study; T = 291 K; mean σ(C–C) = 0.005 Å; R factor = 0.054; wR factor = 0.154; data-to-parameter ratio = 15.2.

The title compound, $C_{16}H_{18}N_2O_2S$, was obtained as an unexpected product while attempting to form carbon–nitrogen bonds by catalytic amidation. The molecule displays an E conformation about the C=N double bond. The planes of the two aromatic rings in the molecule form a dihedral angle of 47.06 (9)°.

Related literature

For the crystal structures of related compounds, see: Cole *et al.* (2005, 2007). For the synthesis of substituted sulfanilamides by catalytic amidation, see: Liu *et al.* (2008); Xu *et al.* (2007, 2008).



Experimental

Crystal data

$C_{16}H_{18}N_2O_2S$ M_r = 302.38
Monoclinic, $P2_1/c$
a = 16.306 (5) Å
b = 8.122 (4) Å
c = 12.674 (4) Å
β = 108.22 (2)°
V = 1594.3 (10) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.21 mm^{-1}
T = 291 K
0.60 × 0.46 × 0.42 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
Absorption correction: spherical (*WinGX*; Farrugia, 1999)
T_{min} = 0.885, T_{max} = 0.918
3769 measured reflections
2928 independent reflections
1958 reflections with $I > 2\sigma(I)$
R_{int} = 0.005
3 standard reflections
every 200 reflections
intensity decay: 2.7%

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.054
$wR(F^2)$ = 0.154
S = 1.09
2928 reflections
192 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.34 e Å^{-3}
$\Delta\rho_{min}$ = −0.44 e Å^{-3}

Data collection: *DIFRAC* (Gabe & White, 1993); cell refinement: *DIFRAC*; data reduction: *NRCVAX* (Gabe *et al.*, 1989); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *SHELXL97*.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: RZ2331).

References

Cole, M. L., Deacon, G. B., Forsyth, C. M., Junk, P. C., Konstas, K. & Wang, J. (2007). *Chem. Eur. J.* **13**, 8092–8110.
Cole, M. L., Deacon, G. B., Junk, P. C. & Konstas, K. (2005). *Chem. Commun.* pp. 1581–1583.
Farrugia, L. J. (1997). *J. Appl. Cryst.* **30**, 565.
Farrugia, L. J. (1999). *J. Appl. Cryst.* **32**, 837–838.
Gabe, E. J., Le Page, Y., Charland, J.-P., Lee, F. L. & White, P. S. (1989). *J. Appl. Cryst.* **22**, 384–387.
Gabe, E. J. & White, P. S. (1993). *DIFRAC*. American Crystallographic Association Meeting, Pittsburgh, Abstract PA 104.
Liu, X.-W., Zhang, Y.-M., Wang, L., Fu, H., Jiang, Y.-Y. & Zhao, Y.-F. (2008). *J. Org. Chem.* **73**, 6207–6212.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Xu, X.-L., Cheng, D.-P., Li, J.-H., Guo, H.-Y. & Yan, J. (2007). *Org. Lett.* **9**, 1585–1587.
Xu, X.-L., Li, X.-N., Ma, L., Ye, N. & Weng, B.-J. (2008). *J. Am. Chem. Soc.* **130**, 14048–14049.

supporting information

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N-Ethyl-*N*-phenyl-*N*′-tosylformamidine

Heng-Shui Shen, Nan Liu, Zi-Cheng Li and Wen-Cai Huang

S1. Comment

In the course of our studies aimed to prepare a substituted sulfanilamide from the corresponding tertiary amines by catalytic amidation using a transition metal salt (Xu *et al.*, 2008; Xu *et al.*, 2007; Liu *et al.*, 2008), the title compound was unexpectedly obtained in about 54% yield.

The molecule of the title compound (Fig. 1) dispays an *E* conformation about the C8═N1 double bond. The values of the N1-C8 (1.301 (3) Å) and N2-C8 (1.326 (3) Å) bonds indicate some degree of conjugation, which was not observed in the related compounds N,N′-bis(2,6-diisopropylphenyl)-N-(4-(3′,4′,5′-trifluorophenoxy)butyl)formamidine (Cole *et al.*, 2007) and N-(4-(2,3,4,5-tetrafluorophenoxy)butyl)-N,N′-bis(2,6-diisopropylphenyl)formamidine (Cole *et al.*, 2005). The dihedral angle formed by the phenyl and benzene rings is 47.06 (9)°. The crystal structure (Fig. 2) is enforced only by van der Waals interactions.

S2. Experimental

N,*N*-Diethylaniline (149 mg, 1 mmol), *p*-toluenesulfonyl azide (591 mg, 3 mmol), copper(I) chloride(20 mg, 0.2 mmol), TEBA (triethylbenzylammonium chloride) (22.7 mg, 0.1 mmol) and acetonitrile (5 mL) were added into a 25 mL round-bottom flask. The resulting mixture was stirred and refluxed for 8 h, then it was evaporated to almost dryness under reduced pressure. Purification was performed by column chromatography on silica gel with petroleum ether/ethyl acetate (7:1–6:1, *v*/*v*) as eluent to give the pure product (163 mg, yield 54%). Colourless crystals suitable for X-ray analysis were obtained by slow evaporation of a cyclohexane/acetyl acetate solution (5:1 *v*/*v*) at room temperature.

S3. Refinement

H atoms were positioned geometrically and refined using a riding model, with C—H = 0.93–0.97 Å and with U_{iso}(H) = 1.2U_{eq}(C) or 1.5U_{eq}(C) for methyl H atoms.

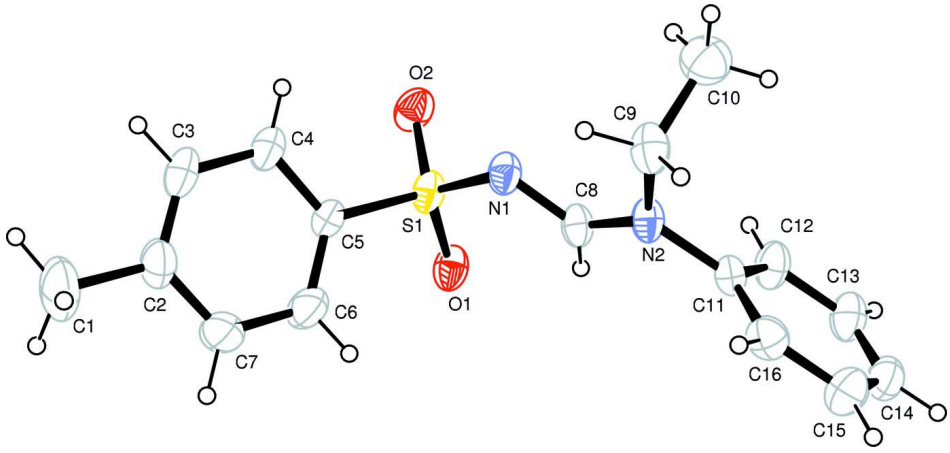

Figure 1

The molecular structure of the title compound, with displacement ellipsoids drawn at the 50% probability level.

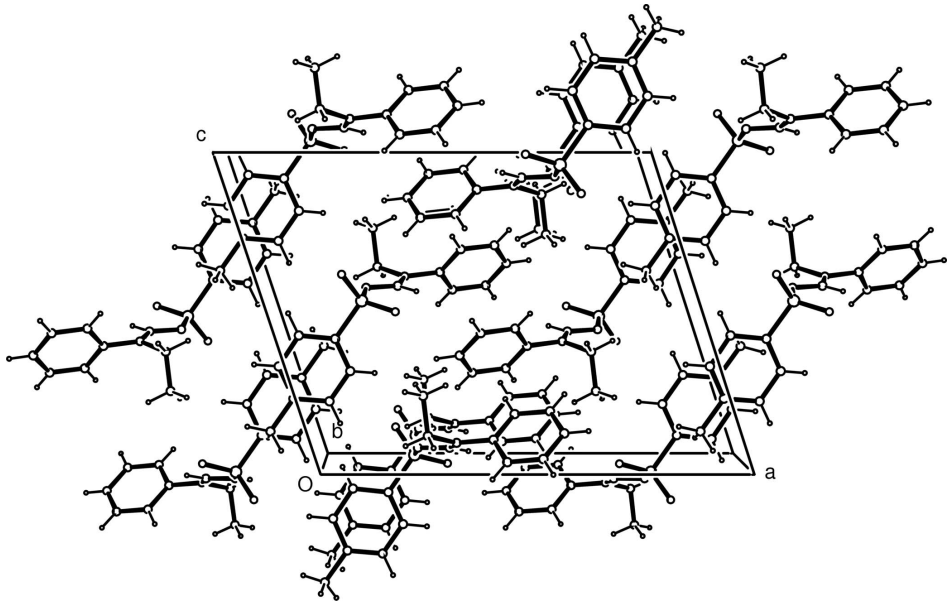

Figure 2

A packing diagram of the title compound approximately viewed along the *b* axis.

N-Ethyl-*N*-phenyl-*N*′-tosylformamidine

Crystal data

$C_{16}H_{18}N_2O_2S$
$M_r = 302.38$
Monoclinic, $P2_1/c$
Hall symbol: -P 2ybc
$a = 16.306\ (5)$ Å
$b = 8.122\ (4)$ Å
$c = 12.674\ (4)$ Å
$\beta = 108.22\ (2)°$
$V = 1594.3\ (10)$ Å^3
$Z = 4$

$F(000) = 640$
$D_x = 1.260$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 25 reflections
$\theta = 4.7–7.7°$
$\mu = 0.21$ mm^{-1}
$T = 291$ K
Block, colourless
0.60 × 0.46 × 0.42 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
ω–2θ scans
Absorption correction: for a sphere (*WinGX*; Farrugia, 1999)
T_{min} = 0.885, T_{max} = 0.918
3769 measured reflections
2928 independent reflections
1958 reflections with $I > 2\sigma(I)$
R_{int} = 0.005
θ_{max} = 25.5°, θ_{min} = 1.3°
h = −6→19
k = −9→0
l = −15→14
3 standard reflections every 200 reflections
intensity decay: 2.7%

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.054
$wR(F^2)$ = 0.154
S = 1.09
2928 reflections
192 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
$w = 1/[\sigma^2(F_o^2) + (0.0973P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.34 e Å^{-3}
$\Delta\rho_{min}$ = −0.44 e Å^{-3}

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.

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 (Å²)

	x	y	z	U_{iso}*/U_{eq}
S1	0.22480 (4)	0.35989 (7)	0.03686 (5)	0.0502 (2)
O1	0.29488 (10)	0.4299 (2)	0.00582 (16)	0.0589 (5)
O2	0.20033 (12)	0.4417 (2)	0.12215 (16)	0.0671 (5)
N1	0.24137 (12)	0.1667 (2)	0.07283 (17)	0.0519 (5)
N2	0.33809 (12)	−0.0487 (2)	0.10219 (16)	0.0499 (5)
C1	−0.0882 (2)	0.3571 (5)	−0.3731 (3)	0.1032 (13)
H1A	−0.1362	0.4031	−0.3547	0.155*
H1B	−0.1020	0.2469	−0.4003	0.155*
H1C	−0.0763	0.4232	−0.4294	0.155*
C2	−0.00939 (19)	0.3541 (4)	−0.2704 (3)	0.0693 (8)
C3	−0.01841 (18)	0.3645 (4)	−0.1664 (3)	0.0808 (9)
H3	−0.0734	0.3725	−0.1595	0.097*
C4	0.05244 (17)	0.3632 (4)	−0.0724 (3)	0.0706 (8)
H4	0.0452	0.3694	−0.0026	0.085*
C5	0.13384 (15)	0.3529 (3)	−0.0822 (2)	0.0488 (6)

C6	0.14362 (18)	0.3421 (4)	−0.1855 (3)	0.0739 (8)
H6	0.1985	0.3331	−0.1928	0.089*
C7	0.0719 (2)	0.3447 (5)	−0.2784 (3)	0.0844 (10)
H7	0.0791	0.3399	−0.3483	0.101*
C8	0.31639 (14)	0.1071 (3)	0.07690 (19)	0.0465 (5)
H8	0.3564	0.1767	0.0614	0.056*
C9	0.27942 (17)	−0.1647 (3)	0.1310 (3)	0.0654 (8)
H9A	0.2849	−0.2722	0.1006	0.078*
H9B	0.2203	−0.1278	0.0979	0.078*
C10	0.2990 (2)	−0.1785 (4)	0.2552 (3)	0.0852 (9)
H10A	0.3586	−0.2073	0.2887	0.128*
H10B	0.2631	−0.2620	0.2716	0.128*
H10C	0.2877	−0.0749	0.2844	0.128*
C11	0.42519 (15)	−0.1012 (3)	0.11568 (19)	0.0459 (6)
C12	0.49282 (16)	−0.0216 (3)	0.1901 (2)	0.0597 (7)
H12	0.4828	0.0657	0.2320	0.072*
C13	0.57586 (17)	−0.0723 (4)	0.2023 (3)	0.0689 (8)
H13	0.6220	−0.0188	0.2529	0.083*
C14	0.59129 (19)	−0.1996 (4)	0.1413 (3)	0.0700 (8)
H14	0.6477	−0.2328	0.1505	0.084*
C15	0.5245 (2)	−0.2778 (4)	0.0673 (3)	0.0753 (9)
H15	0.5352	−0.3637	0.0249	0.090*
C16	0.44046 (19)	−0.2311 (3)	0.0543 (2)	0.0611 (7)
H16	0.3946	−0.2867	0.0047	0.073*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.0381 (3)	0.0471 (4)	0.0608 (4)	0.0024 (2)	0.0089 (3)	−0.0001 (3)
O1	0.0414 (9)	0.0505 (9)	0.0797 (12)	−0.0026 (7)	0.0118 (9)	0.0065 (9)
O2	0.0571 (11)	0.0710 (12)	0.0682 (12)	0.0060 (9)	0.0124 (9)	−0.0151 (10)
N1	0.0370 (10)	0.0499 (11)	0.0639 (13)	0.0017 (8)	0.0084 (9)	0.0074 (9)
N2	0.0409 (11)	0.0454 (11)	0.0538 (12)	0.0006 (8)	0.0010 (9)	0.0040 (9)
C1	0.067 (2)	0.126 (3)	0.089 (2)	0.013 (2)	−0.0152 (19)	−0.004 (2)
C2	0.0511 (15)	0.0738 (18)	0.0694 (19)	0.0089 (13)	−0.0011 (14)	−0.0040 (15)
C3	0.0391 (14)	0.114 (3)	0.084 (2)	0.0170 (16)	0.0112 (15)	0.0069 (18)
C4	0.0431 (14)	0.099 (2)	0.0668 (17)	0.0185 (14)	0.0139 (13)	0.0050 (16)
C5	0.0368 (12)	0.0470 (12)	0.0596 (15)	0.0059 (10)	0.0108 (11)	0.0012 (11)
C6	0.0446 (15)	0.107 (2)	0.0693 (19)	0.0033 (15)	0.0162 (14)	−0.0107 (17)
C7	0.0657 (19)	0.125 (3)	0.0575 (18)	0.0086 (19)	0.0119 (16)	−0.0106 (18)
C8	0.0403 (12)	0.0481 (13)	0.0452 (13)	−0.0006 (10)	0.0047 (10)	0.0019 (10)
C9	0.0468 (14)	0.0541 (14)	0.083 (2)	−0.0065 (11)	0.0022 (14)	0.0118 (14)
C10	0.092 (2)	0.082 (2)	0.091 (2)	−0.0002 (18)	0.042 (2)	0.0094 (19)
C11	0.0457 (13)	0.0448 (12)	0.0417 (12)	0.0060 (10)	0.0058 (10)	0.0057 (10)
C12	0.0460 (13)	0.0597 (15)	0.0631 (16)	0.0036 (12)	0.0022 (12)	−0.0096 (12)
C13	0.0440 (14)	0.0769 (18)	0.0763 (19)	0.0044 (13)	0.0054 (14)	0.0058 (16)
C14	0.0548 (16)	0.0751 (18)	0.085 (2)	0.0171 (14)	0.0291 (16)	0.0245 (17)
C15	0.087 (2)	0.0697 (19)	0.079 (2)	0.0193 (17)	0.0405 (19)	0.0017 (16)

C16	0.0689 (17)	0.0584 (15)	0.0520 (15)	−0.0008 (13)	0.0132 (13)	−0.0049 (12)

Geometric parameters (Å, º)

S1—O2	1.428 (2)	C6—H6	0.9300
S1—O1	1.4371 (18)	C7—H7	0.9300
S1—N1	1.633 (2)	C8—H8	0.9300
S1—C5	1.755 (3)	C9—C10	1.510 (5)
N1—C8	1.301 (3)	C9—H9A	0.9700
N2—C8	1.326 (3)	C9—H9B	0.9700
N2—C11	1.441 (3)	C10—H10A	0.9600
N2—C9	1.467 (3)	C10—H10B	0.9600
C1—C2	1.517 (4)	C10—H10C	0.9600
C1—H1A	0.9600	C11—C12	1.369 (3)
C1—H1B	0.9600	C11—C16	1.379 (4)
C1—H1C	0.9600	C12—C13	1.377 (4)
C2—C7	1.363 (4)	C12—H12	0.9300
C2—C3	1.373 (4)	C13—C14	1.361 (4)
C3—C4	1.376 (4)	C13—H13	0.9300
C3—H3	0.9300	C14—C15	1.353 (4)
C4—C5	1.374 (3)	C14—H14	0.9300
C4—H4	0.9300	C15—C16	1.381 (4)
C5—C6	1.371 (4)	C15—H15	0.9300
C6—C7	1.376 (4)	C16—H16	0.9300
O2—S1—O1	117.23 (12)	N1—C8—N2	122.7 (2)
O2—S1—N1	107.27 (12)	N1—C8—H8	118.6
O1—S1—N1	112.35 (10)	N2—C8—H8	118.6
O2—S1—C5	107.76 (11)	N2—C9—C10	111.4 (2)
O1—S1—C5	107.95 (12)	N2—C9—H9A	109.3
N1—S1—C5	103.32 (11)	C10—C9—H9A	109.3
C8—N1—S1	116.09 (17)	N2—C9—H9B	109.3
C8—N2—C11	119.30 (19)	C10—C9—H9B	109.3
C8—N2—C9	121.8 (2)	H9A—C9—H9B	108.0
C11—N2—C9	118.42 (19)	C9—C10—H10A	109.5
C2—C1—H1A	109.5	C9—C10—H10B	109.5
C2—C1—H1B	109.5	H10A—C10—H10B	109.5
H1A—C1—H1B	109.5	C9—C10—H10C	109.5
C2—C1—H1C	109.5	H10A—C10—H10C	109.5
H1A—C1—H1C	109.5	H10B—C10—H10C	109.5
H1B—C1—H1C	109.5	C12—C11—C16	120.1 (2)
C7—C2—C3	118.3 (3)	C12—C11—N2	119.6 (2)
C7—C2—C1	121.3 (3)	C16—C11—N2	120.3 (2)
C3—C2—C1	120.4 (3)	C11—C12—C13	119.2 (3)
C2—C3—C4	121.2 (3)	C11—C12—H12	120.4
C2—C3—H3	119.4	C13—C12—H12	120.4
C4—C3—H3	119.4	C14—C13—C12	120.9 (3)
C5—C4—C3	119.7 (3)	C14—C13—H13	119.5

C5—C4—H4	120.2	C12—C13—H13	119.5
C3—C4—H4	120.2	C15—C14—C13	119.9 (3)
C6—C5—C4	119.6 (3)	C15—C14—H14	120.0
C6—C5—S1	120.3 (2)	C13—C14—H14	120.0
C4—C5—S1	120.1 (2)	C14—C15—C16	120.5 (3)
C5—C6—C7	119.7 (3)	C14—C15—H15	119.8
C5—C6—H6	120.2	C16—C15—H15	119.8
C7—C6—H6	120.2	C11—C16—C15	119.4 (3)
C2—C7—C6	121.6 (3)	C11—C16—H16	120.3
C2—C7—H7	119.2	C15—C16—H16	120.3
C6—C7—H7	119.2		

O2—S1—N1—C8	−125.18 (19)	C5—C6—C7—C2	−1.5 (5)
O1—S1—N1—C8	5.1 (2)	S1—N1—C8—N2	−178.33 (18)
C5—S1—N1—C8	121.1 (2)	C11—N2—C8—N1	−173.9 (2)
C7—C2—C3—C4	−0.9 (5)	C9—N2—C8—N1	−1.9 (4)
C1—C2—C3—C4	−179.5 (3)	C8—N2—C9—C10	−96.1 (3)
C2—C3—C4—C5	0.6 (5)	C11—N2—C9—C10	76.0 (3)
C3—C4—C5—C6	−0.7 (4)	C8—N2—C11—C12	55.5 (3)
C3—C4—C5—S1	177.3 (2)	C9—N2—C11—C12	−116.7 (3)
O2—S1—C5—C6	154.6 (2)	C8—N2—C11—C16	−124.7 (3)
O1—S1—C5—C6	27.1 (3)	C9—N2—C11—C16	63.1 (3)
N1—S1—C5—C6	−92.1 (2)	C16—C11—C12—C13	0.3 (4)
O2—S1—C5—C4	−23.4 (2)	N2—C11—C12—C13	−179.9 (2)
O1—S1—C5—C4	−150.9 (2)	C11—C12—C13—C14	0.2 (4)
N1—S1—C5—C4	89.9 (2)	C12—C13—C14—C15	0.2 (5)
C4—C5—C6—C7	1.1 (5)	C13—C14—C15—C16	−1.0 (5)
S1—C5—C6—C7	−176.9 (3)	C12—C11—C16—C15	−1.2 (4)
C3—C2—C7—C6	1.3 (5)	N2—C11—C16—C15	179.1 (2)
C1—C2—C7—C6	179.9 (3)	C14—C15—C16—C11	1.5 (4)