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(*E*)-*N*′-(9-Anthrylmethylidene)-*p*-toluenesulfonohydrazide

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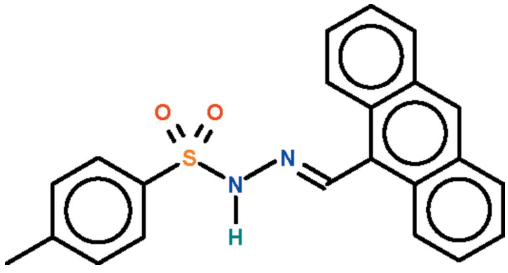
Key indicators: single-crystal X-ray study; T = 100 K; mean σ(C–C) = 0.003 Å; R factor = 0.040; wR factor = 0.104; data-to-parameter ratio = 16.9.

The S—N(H)—N=C linkage in the title molecule, $C_{22}H_{18}N_2O_2S$, is non-planar [torsion angle = 30.6 (1)°] as the amino N atom is pyramidally coordinated. In the crystal, the amino group acts as a hydrogen-bond donor to an O atom of an adjacent molecule, generating chains running parallel to the *b* axis.

Related literature

For the structure of the (*E*)-*N*′-benzylidene-*p*-toluenesulfonohydrazide analog, see: Mehrabi *et al.* (2008).



Experimental

Crystal data

$C_{22}H_{18}N_2O_2S$
M_r = 374.44
Orthorhombic, *Pbca*
a = 17.3634 (15) Å
b = 9.2438 (8) Å
c = 22.882 (2) Å
V = 3672.6 (6) Å^3
Z = 8
Mo $K\alpha$ radiation
μ = 0.20 mm^{-1}
T = 100 K
0.40 × 0.20 × 0.05 mm

Data collection

Bruker SMART APEX diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.926, T_{max} = 0.990
22220 measured reflections
4209 independent reflections
3158 reflections with $I > 2\sigma(I)$
R_{int} = 0.055

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.040
$wR(F^2)$ = 0.104
S = 1.02
4209 reflections
249 parameters
1 restraint
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.34 e Å^{-3}
$\Delta\rho_{min}$ = −0.46 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1···O1^{i}	0.86 (1)	2.07 (1)	2.911 (2)	169 (2)

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

Data collection: *APEX2* (Bruker, 2009); cell refinement: *SAINT* (Bruker, 2009); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *X-SEED* (Barbour, 2001); software used to prepare material for publication: *publCIF* (Westrip, 2010).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: PK2262).

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supporting information

Acta Cryst. (2010). E**66**, o2361 [https://doi.org/10.1107/S160053681003271X]

(*E*)-*N*′-(9-Anthrylmethylidene)-*p*-toluenesulfonohydrazide

Abdullah M. Asiri, Mohie E. M. Zayed and Seik Weng Ng

S1. Comment

p-Toluenesulfonyl hydrazide, CH_3-4-$C_6H_4SO_2NHNH_2$, condenses with carbonyl compounds to form Schiff bases, and among the plethora, nearly a hundred have had their crystal structures determined. The compounds have the azomethine double-bond in an *E*-configuration. In the Schiff base product between *p*-toluenesulfonyl hydrazide and thiophene-2-carboxaldehyde, the S–N(H)–N═C linkage is non-planar [torsion angle 30.6 (1) °] as the amino nitrogen atom (which bears a hydrogen atom) is pyramidally coordinated (Fig. 1). The amino group acts as a hydrogen-bond donor to an oxygen atom of an adjacent molecule to generate chains running parallel to the *b*-axis of the cell (Fig. 2).

S2. Experimental

p-Toluenesulfonyl hydrazide (4.66 g, 2.5 mmol) and anthracene-9-carboxaldehyde (5.162.80 g, 2.5 mmol) were heated in methanol (50 ml) for two hours. The cool solution yielded a precipitate that was recrystallized from ethanol and collected in 90% yield.

S3. Refinement

Carbon-bound H-atoms were placed in calculated positions [C–H 0.95 to 0.99 Å, U(H) 1.2 to 1.5U_{eq}(C)] and were included in the refinement in the riding model approximation. The amino H-atom was located in a difference Fourier map, and was refined with a distance restraint [N–H 0.86 (1) Å]; its temperature factor was freely refined.

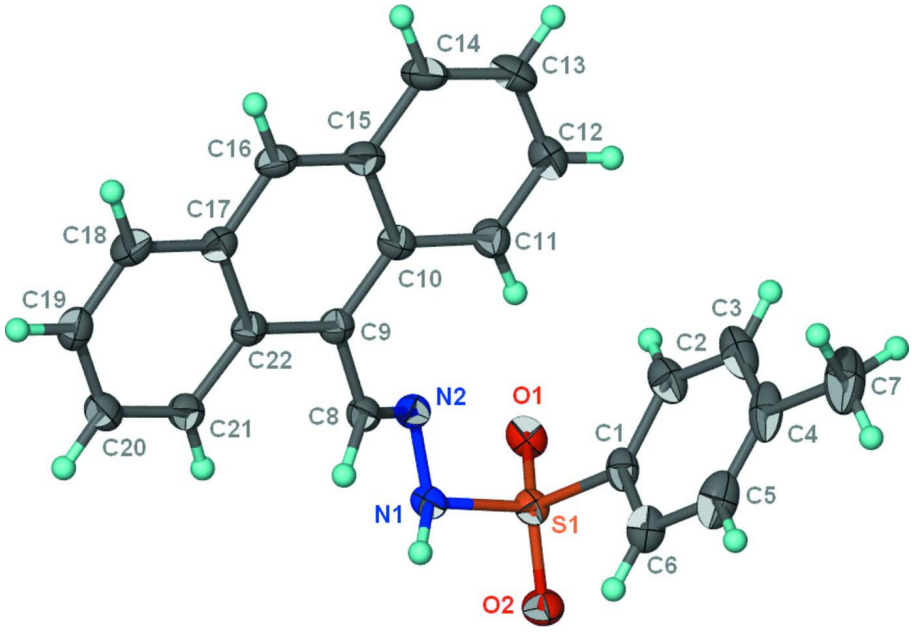

Figure 1

Thermal ellipsoid plot (Barbour, 2001) of $C_{22}H_{18}N_2O_2S$ at the 70% probability level; hydrogen atoms are drawn as spheres of arbitrary radius.

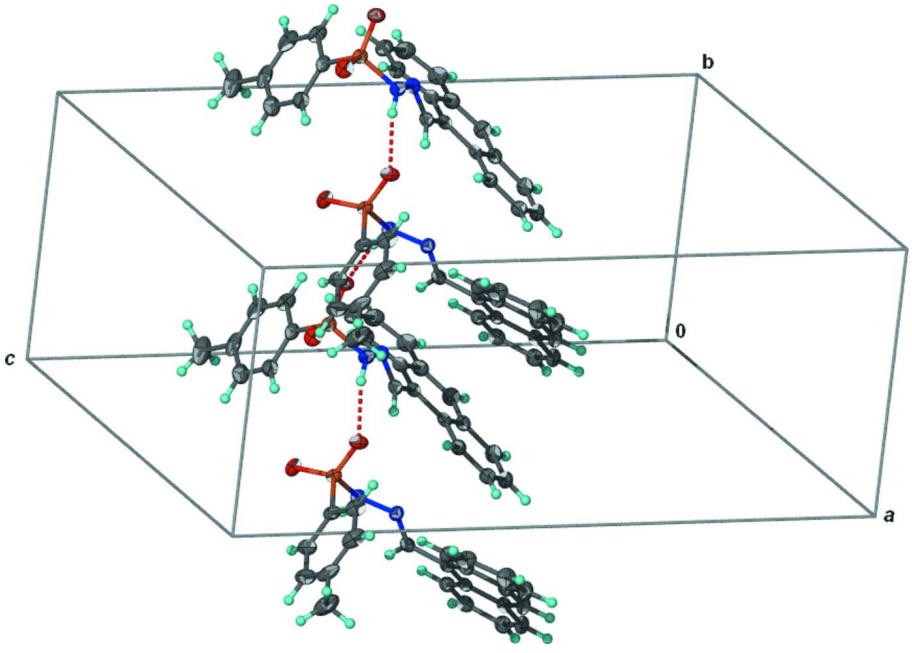

Figure 2

A view of the chain structure resulting from N—H···O hydrogen-bonding.

(*E*)-*N*′-(9-Anthrylmethylidene)-*p*-toluenesulfonohydrazide

Crystal data

$C_{22}H_{18}N_2O_2S$
$M_r = 374.44$
Orthorhombic, *Pbca*
Hall symbol: -P 2ac 2ab
$a = 17.3634\ (15)$ Å
$b = 9.2438\ (8)$ Å
$c = 22.882\ (2)$ Å
$V = 3672.6\ (6)$ Å^3
$Z = 8$
$F(000) = 1568$
$D_x = 1.354$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 3593 reflections
$\theta = 2.3–27.6°$
$\mu = 0.20$ mm^{-1}
$T = 100$ K
Prism, yellow
0.40 × 0.20 × 0.05 mm

Data collection

Bruker SMART APEX diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{min} = 0.926$, $T_{max} = 0.990$
22220 measured reflections
4209 independent reflections
3158 reflections with $I > 2\sigma(I)$
$R_{int} = 0.055$
$\theta_{max} = 27.5°$, $\theta_{min} = 1.8°$
$h = -18\rightarrow22$
$k = -12\rightarrow10$
$l = -29\rightarrow29$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.040$
$wR(F^2) = 0.104$
$S = 1.02$
4209 reflections
249 parameters
1 restraint
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0442P)^2 + 1.8913P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.34$ e Å^{-3}
$\Delta\rho_{min} = -0.46$ e Å^{-3}

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
S1	0.70021 (2)	0.57688 (5)	0.781940 (19)	0.01816 (12)
O1	0.67930 (7)	0.71367 (13)	0.75666 (6)	0.0228 (3)
O2	0.75969 (7)	0.57170 (14)	0.82533 (5)	0.0244 (3)
N1	0.73235 (8)	0.47229 (17)	0.72874 (6)	0.0188 (3)
H1	0.7581 (11)	0.4002 (17)	0.7419 (9)	0.032 (6)*
N2	0.67630 (8)	0.44447 (16)	0.68590 (6)	0.0187 (3)
C1	0.61574 (10)	0.4956 (2)	0.80808 (8)	0.0207 (4)
C2	0.54464 (11)	0.5595 (2)	0.79729 (9)	0.0263 (4)
H2	0.5412	0.6474	0.7760	0.032*
C3	0.47881 (11)	0.4919 (2)	0.81839 (9)	0.0324 (5)
H3	0.4299	0.5341	0.8111	0.039*
C4	0.48310 (12)	0.3638 (2)	0.84988 (9)	0.0319 (5)
C5	0.55497 (13)	0.3014 (2)	0.85908 (9)	0.0326 (5)

H5	0.5584	0.2129	0.8800	0.039*
C6	0.62165 (11)	0.3659 (2)	0.83825 (8)	0.0256 (4)
H6	0.6705	0.3220	0.8445	0.031*
C7	0.41088 (13)	0.2933 (3)	0.87297 (10)	0.0437 (6)
H7A	0.3755	0.3679	0.8872	0.066*
H7B	0.4242	0.2279	0.9051	0.066*
H7C	0.3861	0.2382	0.8416	0.066*
C8	0.68260 (10)	0.32154 (19)	0.66045 (7)	0.0190 (4)
H8	0.7224	0.2580	0.6727	0.023*
C9	0.63107 (10)	0.27431 (19)	0.61321 (7)	0.0181 (4)
C10	0.55246 (10)	0.31693 (19)	0.61075 (8)	0.0196 (4)
C11	0.51458 (10)	0.3970 (2)	0.65569 (8)	0.0225 (4)
H11	0.5434	0.4292	0.6885	0.027*
C12	0.43788 (11)	0.4284 (2)	0.65260 (9)	0.0256 (4)
H12	0.4139	0.4789	0.6839	0.031*
C13	0.39353 (11)	0.3870 (2)	0.60350 (9)	0.0284 (4)
H13	0.3406	0.4124	0.6013	0.034*
C14	0.42673 (11)	0.3113 (2)	0.55987 (9)	0.0274 (4)
H14	0.3968	0.2849	0.5268	0.033*
C15	0.50621 (10)	0.2698 (2)	0.56239 (8)	0.0232 (4)
C16	0.53801 (11)	0.1800 (2)	0.51995 (8)	0.0248 (4)
H16	0.5071	0.1511	0.4877	0.030*
C17	0.61374 (10)	0.1311 (2)	0.52330 (8)	0.0207 (4)
C18	0.64417 (11)	0.0329 (2)	0.48093 (8)	0.0249 (4)
H18	0.6121	0.0005	0.4500	0.030*
C19	0.71804 (11)	−0.0149 (2)	0.48399 (8)	0.0247 (4)
H19	0.7372	−0.0808	0.4557	0.030*
C20	0.76612 (11)	0.0344 (2)	0.52973 (8)	0.0229 (4)
H20	0.8179	0.0017	0.5316	0.028*
C21	0.73965 (10)	0.1276 (2)	0.57109 (8)	0.0204 (4)
H21	0.7736	0.1594	0.6010	0.025*
C22	0.66168 (10)	0.17906 (19)	0.57053 (7)	0.0184 (4)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.0166 (2)	0.0169 (2)	0.0210 (2)	−0.00194 (17)	0.00059 (17)	−0.00158 (17)
O1	0.0222 (7)	0.0163 (6)	0.0300 (7)	−0.0008 (5)	0.0016 (5)	0.0003 (5)
O2	0.0203 (7)	0.0287 (7)	0.0241 (7)	−0.0025 (6)	−0.0044 (5)	−0.0040 (6)
N1	0.0164 (7)	0.0189 (8)	0.0211 (8)	0.0014 (6)	−0.0012 (6)	−0.0008 (6)
N2	0.0167 (7)	0.0209 (8)	0.0184 (7)	−0.0022 (6)	−0.0009 (6)	−0.0003 (6)
C1	0.0204 (9)	0.0205 (9)	0.0212 (9)	−0.0052 (7)	0.0050 (7)	−0.0043 (7)
C2	0.0219 (9)	0.0249 (10)	0.0323 (10)	−0.0008 (8)	0.0037 (8)	−0.0067 (8)
C3	0.0222 (10)	0.0366 (12)	0.0386 (12)	−0.0036 (9)	0.0077 (9)	−0.0149 (10)
C4	0.0330 (11)	0.0353 (12)	0.0274 (10)	−0.0164 (9)	0.0122 (9)	−0.0173 (9)
C5	0.0449 (13)	0.0250 (11)	0.0279 (10)	−0.0115 (9)	0.0087 (9)	−0.0030 (9)
C6	0.0296 (10)	0.0220 (10)	0.0252 (9)	−0.0029 (8)	0.0049 (8)	−0.0017 (8)
C7	0.0381 (13)	0.0554 (16)	0.0375 (12)	−0.0261 (11)	0.0144 (10)	−0.0162 (11)

C8	0.0177 (9)	0.0198 (9)	0.0195 (9)	0.0003 (7)	0.0016 (7)	0.0035 (7)
C9	0.0190 (9)	0.0171 (9)	0.0182 (8)	−0.0017 (7)	0.0004 (7)	0.0026 (7)
C10	0.0187 (9)	0.0184 (9)	0.0216 (9)	−0.0010 (7)	−0.0008 (7)	0.0031 (7)
C11	0.0197 (9)	0.0241 (10)	0.0238 (9)	−0.0020 (7)	0.0005 (7)	−0.0015 (8)
C12	0.0220 (9)	0.0249 (10)	0.0300 (10)	0.0017 (8)	0.0035 (8)	−0.0020 (8)
C13	0.0184 (9)	0.0299 (11)	0.0369 (11)	0.0042 (8)	−0.0030 (8)	0.0015 (9)
C14	0.0229 (10)	0.0311 (11)	0.0282 (10)	0.0044 (8)	−0.0083 (8)	−0.0004 (9)
C15	0.0213 (9)	0.0248 (10)	0.0235 (9)	0.0015 (8)	−0.0036 (7)	0.0035 (8)
C16	0.0243 (10)	0.0297 (10)	0.0203 (9)	0.0018 (8)	−0.0067 (8)	−0.0007 (8)
C17	0.0222 (9)	0.0218 (9)	0.0182 (8)	0.0009 (7)	−0.0005 (7)	0.0022 (7)
C18	0.0274 (10)	0.0283 (10)	0.0191 (9)	−0.0004 (8)	−0.0040 (8)	−0.0010 (8)
C19	0.0289 (10)	0.0252 (10)	0.0200 (9)	0.0015 (8)	0.0039 (8)	−0.0007 (8)
C20	0.0199 (9)	0.0231 (10)	0.0258 (10)	0.0020 (7)	0.0023 (7)	0.0031 (8)
C21	0.0193 (9)	0.0210 (9)	0.0210 (8)	−0.0010 (7)	0.0005 (7)	0.0019 (7)
C22	0.0199 (9)	0.0176 (9)	0.0176 (8)	−0.0018 (7)	0.0001 (7)	0.0046 (7)

Geometric parameters (Å, º)

S1—O1	1.4370 (13)	C9—C10	1.422 (2)
S1—O2	1.4334 (13)	C10—C11	1.428 (2)
S1—N1	1.6516 (15)	C10—C15	1.435 (2)
S1—C1	1.7532 (18)	C11—C12	1.365 (3)
N1—N2	1.405 (2)	C11—H11	0.9500
N1—H1	0.857 (9)	C12—C13	1.415 (3)
N2—C8	1.281 (2)	C12—H12	0.9500
C1—C6	1.387 (3)	C13—C14	1.349 (3)
C1—C2	1.391 (3)	C13—H13	0.9500
C2—C3	1.389 (3)	C14—C15	1.434 (3)
C2—H2	0.9500	C14—H14	0.9500
C3—C4	1.388 (3)	C15—C16	1.392 (3)
C3—H3	0.9500	C16—C17	1.392 (3)
C4—C5	1.391 (3)	C16—H16	0.9500
C4—C7	1.509 (3)	C17—C18	1.429 (3)
C5—C6	1.387 (3)	C17—C22	1.434 (2)
C5—H5	0.9500	C18—C19	1.358 (3)
C6—H6	0.9500	C18—H18	0.9500
C7—H7A	0.9800	C19—C20	1.414 (3)
C7—H7B	0.9800	C19—H19	0.9500
C7—H7C	0.9800	C20—C21	1.360 (2)
C8—C9	1.470 (2)	C20—H20	0.9500
C8—H8	0.9500	C21—C22	1.435 (2)
C9—C22	1.418 (2)	C21—H21	0.9500
O1—S1—O2	119.35 (8)	C9—C10—C11	123.88 (16)
O1—S1—N1	107.68 (8)	C9—C10—C15	118.93 (16)
O2—S1—N1	104.33 (8)	C11—C10—C15	117.07 (16)
O1—S1—C1	107.65 (8)	C12—C11—C10	121.50 (18)
O2—S1—C1	110.62 (8)	C12—C11—H11	119.3

N1—S1—C1	106.45 (8)	C10—C11—H11	119.3
N2—N1—S1	112.79 (11)	C11—C12—C13	120.98 (18)
N2—N1—H1	117.7 (15)	C11—C12—H12	119.5
S1—N1—H1	111.8 (14)	C13—C12—H12	119.5
C8—N2—N1	114.84 (15)	C14—C13—C12	119.71 (17)
C6—C1—C2	121.41 (17)	C14—C13—H13	120.1
C6—C1—S1	118.58 (14)	C12—C13—H13	120.1
C2—C1—S1	120.00 (15)	C13—C14—C15	121.36 (18)
C3—C2—C1	118.52 (19)	C13—C14—H14	119.3
C3—C2—H2	120.7	C15—C14—H14	119.3
C1—C2—H2	120.7	C16—C15—C10	119.80 (16)
C4—C3—C2	121.3 (2)	C16—C15—C14	120.89 (17)
C4—C3—H3	119.3	C10—C15—C14	119.26 (17)
C2—C3—H3	119.3	C17—C16—C15	122.00 (17)
C3—C4—C5	118.72 (18)	C17—C16—H16	119.0
C3—C4—C7	120.4 (2)	C15—C16—H16	119.0
C5—C4—C7	120.9 (2)	C16—C17—C18	121.19 (17)
C6—C5—C4	121.3 (2)	C16—C17—C22	119.28 (17)
C6—C5—H5	119.4	C18—C17—C22	119.52 (16)
C4—C5—H5	119.4	C19—C18—C17	121.39 (17)
C5—C6—C1	118.73 (19)	C19—C18—H18	119.3
C5—C6—H6	120.6	C17—C18—H18	119.3
C1—C6—H6	120.6	C18—C19—C20	119.38 (17)
C4—C7—H7A	109.5	C18—C19—H19	120.3
C4—C7—H7B	109.5	C20—C19—H19	120.3
H7A—C7—H7B	109.5	C21—C20—C19	121.31 (17)
C4—C7—H7C	109.5	C21—C20—H20	119.3
H7A—C7—H7C	109.5	C19—C20—H20	119.3
H7B—C7—H7C	109.5	C20—C21—C22	121.52 (17)
N2—C8—C9	123.06 (16)	C20—C21—H21	119.2
N2—C8—H8	118.5	C22—C21—H21	119.2
C9—C8—H8	118.5	C9—C22—C21	123.60 (16)
C22—C9—C10	120.31 (16)	C9—C22—C17	119.55 (16)
C22—C9—C8	117.56 (15)	C21—C22—C17	116.85 (16)
C10—C9—C8	122.09 (16)		

O1—S1—N1—N2	−62.00 (13)	C10—C11—C12—C13	−2.3 (3)
O2—S1—N1—N2	170.23 (11)	C11—C12—C13—C14	2.1 (3)
C1—S1—N1—N2	53.21 (14)	C12—C13—C14—C15	0.9 (3)
S1—N1—N2—C8	−149.39 (13)	C9—C10—C15—C16	2.1 (3)
O1—S1—C1—C6	−175.60 (14)	C11—C10—C15—C16	−174.03 (17)
O2—S1—C1—C6	−43.59 (17)	C9—C10—C15—C14	179.46 (17)
N1—S1—C1—C6	69.17 (16)	C11—C10—C15—C14	3.4 (3)
O1—S1—C1—C2	5.81 (18)	C13—C14—C15—C16	173.73 (19)
O2—S1—C1—C2	137.82 (15)	C13—C14—C15—C10	−3.6 (3)
N1—S1—C1—C2	−109.42 (16)	C10—C15—C16—C17	1.3 (3)
C6—C1—C2—C3	1.2 (3)	C14—C15—C16—C17	−176.08 (18)
S1—C1—C2—C3	179.71 (15)	C15—C16—C17—C18	176.73 (18)

C1—C2—C3—C4	0.3 (3)	C15—C16—C17—C22	−2.6 (3)
C2—C3—C4—C5	−1.4 (3)	C16—C17—C18—C19	179.93 (18)
C2—C3—C4—C7	179.28 (18)	C22—C17—C18—C19	−0.8 (3)
C3—C4—C5—C6	1.0 (3)	C17—C18—C19—C20	−0.6 (3)
C7—C4—C5—C6	−179.68 (18)	C18—C19—C20—C21	0.7 (3)
C4—C5—C6—C1	0.4 (3)	C19—C20—C21—C22	0.6 (3)
C2—C1—C6—C5	−1.6 (3)	C10—C9—C22—C21	−177.99 (16)
S1—C1—C6—C5	179.88 (14)	C8—C9—C22—C21	−0.2 (3)
N1—N2—C8—C9	−178.04 (15)	C10—C9—C22—C17	2.8 (3)
N2—C8—C9—C22	149.92 (17)	C8—C9—C22—C17	−179.43 (15)
N2—C8—C9—C10	−32.4 (3)	C20—C21—C22—C9	178.91 (17)
C22—C9—C10—C11	171.73 (17)	C20—C21—C22—C17	−1.9 (3)
C8—C9—C10—C11	−5.9 (3)	C16—C17—C22—C9	0.5 (3)
C22—C9—C10—C15	−4.1 (3)	C18—C17—C22—C9	−178.81 (17)
C8—C9—C10—C15	178.27 (16)	C16—C17—C22—C21	−178.75 (17)
C9—C10—C11—C12	−176.38 (18)	C18—C17—C22—C21	1.9 (2)
C15—C10—C11—C12	−0.5 (3)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1···O1^{i}	0.86 (1)	2.07 (1)	2.911 (2)	169 (2)

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