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2-Oxo-2-(2-oxo-2*H*-chromen-3-yl)ethyl diethyldithiocarbamate

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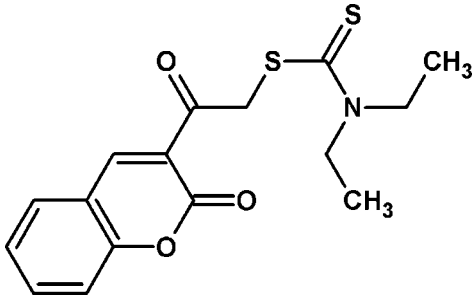
Key indicators: single-crystal X-ray study; T = 296 K; mean σ(C–C) = 0.003 Å; R factor = 0.035; wR factor = 0.090; data-to-parameter ratio = 14.2.

In the title compound, $C_{16}H_{17}NO_3S_2$, the dihedral angles between the O/C/C/S group and the 2*H*-chromene ring system and the thiocarbamate group are 14.46 (9) and 83.30 (9)°, respectively. The bond-angle sum at the N atom is 360.0°. One of the methyl C atoms lies above the thiocarbamate plane and one lies below it [deviations = 1.264 (3) and −1.147 (3) Å, respectively]. In the crystal, inversion dimers linked by pairs of C—H···O hydrogen bonds generate $R_2^2(10)$ loops. Weak aromatic π–π stacking interactions [shortest centroid–centroid distance = 3.8138 (11) Å] are also observed.

Related literature

For backgrond to chromenes, a related structure and the synthesis of the title compound, see: Kumar *et al.* (2012).



Experimental

Crystal data

$C_{16}H_{17}NO_3S_2$
M_r = 335.43
Orthorhombic, *Pbcn*
a = 16.3379 (5) Å
b = 9.6445 (3) Å
c = 20.5078 (6) Å
V = 3231.43 (17) Å^3
Z = 8
Mo $K\alpha$ radiation
μ = 0.34 mm^{-1}
T = 296 K
0.24 × 0.20 × 0.12 mm

Data collection

Bruker SMART CCD diffractometer
Absorption correction: ψ scan (*SADABS*; Bruker, 2001)
T_{min} = 0.770, T_{max} = 1.000
12047 measured reflections
2831 independent reflections
2129 reflections with $I > 2\sigma(I)$
R_{int} = 0.030

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.035
$wR(F^2)$ = 0.090
S = 1.05
2831 reflections
199 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.21 e Å^{-3}
$\Delta\rho_{min}$ = −0.18 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
C9—H9···O5^{i}	0.93	2.49	3.198 (2)	134

Symmetry code: (i) $-x, -y+1, -z+1$.

Data collection: *SMART* (Bruker, 2001); cell refinement: *SAINT* (Bruker, 2001); data reduction: *SAINT*; 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, 2012); software used to prepare material for publication: *SHELXL97*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HB7115).

References

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Kumar, K. M., Devarajegowda, H. C., Jeyaseelan, S., Mahabaleshwaraiah, N. M. & Kotresh, O. (2012). *Acta Cryst.* E**68**, o1657.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.

supplementary materials

Acta Cryst. (2013). E**69**, o1454 [doi:10.1107/S1600536813021806]

2-Oxo-2-(2-oxo-2*H*-chromen-3-yl)ethyl diethyldithiocarbamate

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1. Comment

As part of our ongoing structural studies of chromene derivatives with possible biological activity (Kumar *et al.*, 2012), we now describe the structure of the title compound, (I), (Fig. 1).

The 2*H*-chromene ring system is close to planar, with a maximum deviation of 0.031 (1) Å for atom C8. In the crystal, C9—H9···O5 hydrogen bonds (Table 1) and π-π interactions between fused benzene rings of chromene [shortest centroid–centroid distance = 3.8138 (11) Å] occur. The C—H···O hydrogen bonds generate an $R_2^2(10)$ loop.

2. Experimental

This compound was prepared according to the reported method (Kumar *et al.*, 2012). Colourless blocks of the title compound were grown from a mixed solution of EtOH/$CHCl_3$ (V/V = 2/1) by slow evaporation at room temperature. Yield= 90%, m.p. 380 K.

3. Refinement

All H atoms were positioned geometrically, with C—H = 0.93 Å for aromatic H, C—H = 0.97 Å for methylene H and C—H = 0.96 Å for methyl H,and refined using a riding model with U_{iso}(H) = 1.5U_{eq}(C) for methyl H and U_{iso}(H) = 1.2U_{eq}(C) for all other H.

Computing details

Data collection: *SMART* (Bruker, 2001); cell refinement: *SAINT* (Bruker, 2001); data reduction: *SAINT* (Bruker, 2001); 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, 2012); software used to prepare material for publication: *SHELXL97* (Sheldrick, 2008).

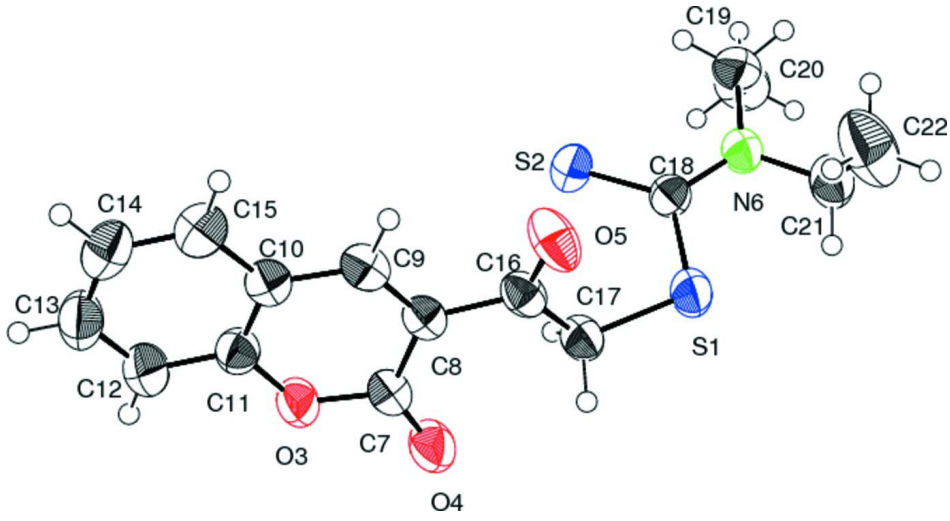

Figure 1

The molecular structure of the title compound. Displacement ellipsoids are drawn at the 50% probability level.

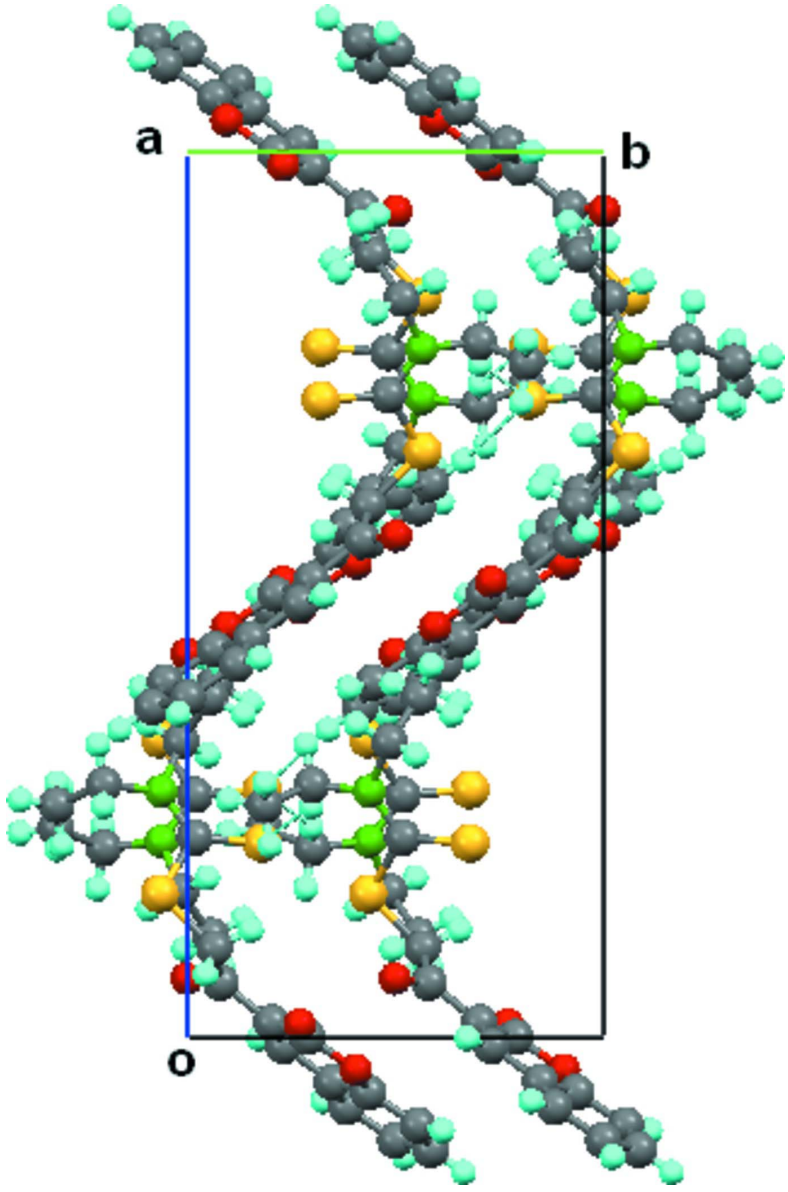

Figure 2

The packing of molecule shows when view along *c* axis.

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Crystal data

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Orthorhombic, *Pbcn*
Hall symbol: -P 2n 2ab
a = 16.3379 (5) Å
b = 9.6445 (3) Å
c = 20.5078 (6) Å
V = 3231.43 (17) Å^3
Z = 8
F(000) = 1408

D_x = 1.379 Mg m^{-3}
Melting point: 380 K
Mo *Kα* radiation, λ = 0.71073 Å
Cell parameters from 2831 reflections
θ = 2.0–25.0°
μ = 0.34 mm^{-1}
T = 296 K
Block, colourless
0.24 × 0.20 × 0.12 mm

Data collection

Bruker SMART CCD diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
ω and φ scans
Absorption correction: ψ scan (*SADABS*; Bruker, 2001)
T_{min} = 0.770, T_{max} = 1.000
12047 measured reflections
2831 independent reflections
2129 reflections with $I > 2\sigma(I)$
R_{int} = 0.030
θ_{max} = 25.0°, θ_{min} = 2.0°
h = −19→16
k = −11→11
l = −21→23

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.035
$wR(F^2)$ = 0.090
S = 1.05
2831 reflections
199 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
$w = 1/[\sigma^2(F_o^2) + (0.0414P)^2 + 0.5615P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.21 e Å^{-3}
$\Delta\rho_{min}$ = −0.18 e Å^{-3}

Special details

Experimental. IR (KBr): 634 cm^{-1} (C—S), 1266 cm^{-1} (C=S), 1069 cm^{-1} (C—O), 859 cm^{-1} (C—N),1170 cm^{-1} (C—O—C), 1696 cm^{-1} (C=O), 1725 cm^{-1} (Coumarin C=O). GCMS: m/e: 335. 1H NMR (400 MHz, $CDCl_3$, \?,. p.p.m): 1.24 (m, 3H, C_{12}), 1.35 (m, 3H, C_1), 3.80 (t, 2H, C_2),3.97(t, 2H, C_{13}), 4.80(s,2*H*, C_4), 7.27 (s, 1H, C_{16}), 7.37 (m, 1H, C_{10}),7.66 (s, 1H, C_{11}), 8.49(s, 1H, C_9).13 C NMR (400 MHz, CDCl3, \?,. p.p.m): 194(C_3), 191(C_5), 159(C_{14}), 155(C_{15}), 147(C_7), 134(C_6), 130(C_{11}), 125(C_9), 125(C_{10}), 118(C_8), 116(C_{16}), 50(C_4), 47(C_2),46(C_{13}), 12(C_1), 11(C_{12}).Elemental analysis for $C_{16}H_{17}NO_3S_2$: C, 57.22; H, 5.06; N, 4.11.

Geometry. All s.u.'s (except the s.u. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell s.u.'s are taken into account individually in the estimation of s.u.'s in distances, angles and torsion angles; correlations between s.u.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell s.u.'s is used for estimating s.u.'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 $F^2 > 2\sigma(F^2)$ 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}
S1	0.20407 (3)	0.56622 (6)	0.33761 (2)	0.05998 (19)
S2	0.11615 (3)	0.32066 (6)	0.28013 (3)	0.05692 (18)
O3	0.17542 (8)	0.09457 (14)	0.53601 (7)	0.0570 (4)
O4	0.26174 (8)	0.23027 (17)	0.48512 (7)	0.0714 (5)
O5	0.07374 (9)	0.50120 (16)	0.43332 (7)	0.0685 (4)
N6	0.13841 (10)	0.56284 (17)	0.22176 (7)	0.0501 (4)
C7	0.19123 (12)	0.2115 (2)	0.49934 (9)	0.0490 (5)
C8	0.12118 (10)	0.2984 (2)	0.48279 (8)	0.0417 (4)
C9	0.04753 (11)	0.2690 (2)	0.50922 (9)	0.0468 (5)
H9	0.0038	0.3278	0.5004	0.056*
C10	0.03396 (11)	0.1524 (2)	0.54993 (9)	0.0449 (5)
C11	0.09952 (12)	0.0653 (2)	0.56141 (9)	0.0481 (5)

C12	0.09278 (14)	−0.0535 (2)	0.59863 (11)	0.0628 (6)
H12	0.1376	−0.1114	0.6050	0.075*
C13	0.01817 (15)	−0.0843 (2)	0.62606 (11)	0.0658 (6)
H13	0.0126	−0.1631	0.6518	0.079*
C14	−0.04852 (14)	0.0007 (3)	0.61575 (11)	0.0631 (6)
H14	−0.0987	−0.0217	0.6344	0.076*
C15	−0.04160 (12)	0.1179 (2)	0.57822 (10)	0.0577 (5)
H15	−0.0869	0.1744	0.5715	0.069*
C16	0.12926 (12)	0.4194 (2)	0.43773 (9)	0.0468 (5)
C17	0.20748 (12)	0.4352 (2)	0.39882 (9)	0.0517 (5)
H17A	0.2200	0.3473	0.3782	0.062*
H17B	0.2518	0.4563	0.4287	0.062*
C18	0.14928 (10)	0.4828 (2)	0.27391 (9)	0.0445 (5)
C19	0.08964 (13)	0.5153 (3)	0.16571 (9)	0.0585 (6)
H19A	0.0624	0.5941	0.1459	0.070*
H19B	0.0478	0.4515	0.1808	0.070*
C20	0.14183 (16)	0.4445 (3)	0.11560 (11)	0.0759 (7)
H20A	0.1082	0.4155	0.0797	0.114*
H20B	0.1677	0.3650	0.1348	0.114*
H20C	0.1829	0.5077	0.1003	0.114*
C21	0.17643 (16)	0.7006 (2)	0.21421 (11)	0.0694 (7)
H21A	0.1894	0.7154	0.1686	0.083*
H21B	0.2273	0.7027	0.2385	0.083*
C22	0.1227 (2)	0.8147 (3)	0.23727 (15)	0.1102 (11)
H22A	0.1500	0.9019	0.2312	0.165*
H22B	0.1108	0.8019	0.2827	0.165*
H22C	0.0726	0.8141	0.2128	0.165*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.0785 (4)	0.0517 (4)	0.0498 (3)	−0.0153 (3)	−0.0134 (3)	0.0112 (3)
S2	0.0549 (3)	0.0488 (3)	0.0670 (4)	−0.0095 (2)	0.0006 (2)	0.0070 (3)
O3	0.0494 (8)	0.0523 (9)	0.0693 (9)	0.0053 (6)	−0.0001 (7)	0.0199 (8)
O4	0.0480 (8)	0.0799 (12)	0.0864 (11)	0.0065 (8)	0.0037 (7)	0.0350 (9)
O5	0.0805 (10)	0.0611 (10)	0.0639 (9)	0.0304 (9)	0.0120 (8)	0.0156 (8)
N6	0.0563 (9)	0.0491 (11)	0.0450 (9)	−0.0060 (8)	−0.0037 (7)	0.0071 (8)
C7	0.0536 (12)	0.0485 (13)	0.0448 (10)	0.0028 (10)	−0.0023 (9)	0.0057 (10)
C8	0.0506 (11)	0.0390 (11)	0.0356 (9)	0.0048 (9)	−0.0019 (8)	−0.0038 (8)
C9	0.0534 (11)	0.0435 (12)	0.0434 (10)	0.0095 (9)	−0.0031 (9)	−0.0056 (10)
C10	0.0523 (11)	0.0425 (12)	0.0400 (10)	0.0011 (9)	0.0001 (8)	−0.0056 (9)
C11	0.0524 (11)	0.0464 (13)	0.0455 (11)	−0.0041 (10)	−0.0007 (9)	0.0004 (10)
C12	0.0667 (14)	0.0510 (14)	0.0707 (14)	−0.0007 (11)	−0.0040 (11)	0.0134 (12)
C13	0.0846 (17)	0.0540 (15)	0.0588 (13)	−0.0160 (13)	0.0022 (12)	0.0081 (12)
C14	0.0659 (14)	0.0632 (16)	0.0602 (13)	−0.0158 (13)	0.0136 (11)	−0.0075 (12)
C15	0.0561 (12)	0.0583 (14)	0.0586 (12)	−0.0002 (10)	0.0072 (10)	−0.0059 (12)
C16	0.0591 (12)	0.0428 (12)	0.0384 (10)	0.0064 (10)	−0.0055 (9)	−0.0019 (9)
C17	0.0594 (12)	0.0510 (13)	0.0445 (10)	−0.0019 (10)	−0.0054 (9)	0.0073 (10)
C18	0.0390 (9)	0.0486 (12)	0.0460 (11)	0.0000 (9)	0.0043 (8)	0.0051 (10)
C19	0.0561 (11)	0.0673 (15)	0.0522 (12)	−0.0056 (11)	−0.0109 (10)	0.0059 (12)

C20	0.0916 (17)	0.0818 (19)	0.0544 (13)	0.0017 (14)	−0.0066 (12)	−0.0065 (13)
C21	0.0908 (16)	0.0625 (16)	0.0550 (13)	−0.0156 (13)	−0.0106 (12)	0.0138 (12)
C22	0.177 (3)	0.068 (2)	0.085 (2)	0.024 (2)	−0.017 (2)	0.0027 (17)

Geometric parameters (Å, º)

S1—C18	1.7762 (19)	C13—C14	1.380 (3)
S1—C17	1.7818 (19)	C13—H13	0.9300
S2—C18	1.660 (2)	C14—C15	1.372 (3)
O3—C11	1.374 (2)	C14—H14	0.9300
O3—C7	1.380 (2)	C15—H15	0.9300
O4—C7	1.202 (2)	C16—C17	1.514 (3)
O5—C16	1.206 (2)	C17—H17A	0.9700
N6—C18	1.331 (2)	C17—H17B	0.9700
N6—C19	1.472 (2)	C19—C20	1.500 (3)
N6—C21	1.474 (3)	C19—H19A	0.9700
C7—C8	1.459 (3)	C19—H19B	0.9700
C8—C9	1.350 (2)	C20—H20A	0.9600
C8—C16	1.494 (3)	C20—H20B	0.9600
C9—C10	1.418 (3)	C20—H20C	0.9600
C9—H9	0.9300	C21—C22	1.485 (4)
C10—C11	1.381 (3)	C21—H21A	0.9700
C10—C15	1.404 (3)	C21—H21B	0.9700
C11—C12	1.382 (3)	C22—H22A	0.9600
C12—C13	1.375 (3)	C22—H22B	0.9600
C12—H12	0.9300	C22—H22C	0.9600
C18—S1—C17	102.29 (9)	C8—C16—C17	118.65 (16)
C11—O3—C7	122.91 (15)	C16—C17—S1	114.62 (14)
C18—N6—C19	121.27 (17)	C16—C17—H17A	108.6
C18—N6—C21	123.41 (16)	S1—C17—H17A	108.6
C19—N6—C21	115.27 (16)	C16—C17—H17B	108.6
O4—C7—O3	115.77 (17)	S1—C17—H17B	108.6
O4—C7—C8	127.52 (19)	H17A—C17—H17B	107.6
O3—C7—C8	116.71 (16)	N6—C18—S2	124.38 (14)
C9—C8—C7	119.04 (18)	N6—C18—S1	113.30 (14)
C9—C8—C16	119.41 (17)	S2—C18—S1	122.31 (11)
C7—C8—C16	121.55 (16)	N6—C19—C20	111.66 (17)
C8—C9—C10	122.84 (17)	N6—C19—H19A	109.3
C8—C9—H9	118.6	C20—C19—H19A	109.3
C10—C9—H9	118.6	N6—C19—H19B	109.3
C11—C10—C15	117.86 (19)	C20—C19—H19B	109.3
C11—C10—C9	117.47 (17)	H19A—C19—H19B	107.9
C15—C10—C9	124.65 (18)	C19—C20—H20A	109.5
O3—C11—C10	120.69 (17)	C19—C20—H20B	109.5
O3—C11—C12	116.85 (18)	H20A—C20—H20B	109.5
C10—C11—C12	122.46 (19)	C19—C20—H20C	109.5
C13—C12—C11	118.4 (2)	H20A—C20—H20C	109.5
C13—C12—H12	120.8	H20B—C20—H20C	109.5
C11—C12—H12	120.8	N6—C21—C22	112.7 (2)

C12—C13—C14	120.6 (2)	N6—C21—H21A	109.1
C12—C13—H13	119.7	C22—C21—H21A	109.1
C14—C13—H13	119.7	N6—C21—H21B	109.1
C15—C14—C13	120.7 (2)	C22—C21—H21B	109.1
C15—C14—H14	119.7	H21A—C21—H21B	107.8
C13—C14—H14	119.7	C21—C22—H22A	109.5
C14—C15—C10	120.0 (2)	C21—C22—H22B	109.5
C14—C15—H15	120.0	H22A—C22—H22B	109.5
C10—C15—H15	120.0	C21—C22—H22C	109.5
O5—C16—C8	119.41 (18)	H22A—C22—H22C	109.5
O5—C16—C17	121.94 (18)	H22B—C22—H22C	109.5
C11—O3—C7—O4	173.66 (18)	C13—C14—C15—C10	−0.1 (3)
C11—O3—C7—C8	−5.8 (3)	C11—C10—C15—C14	−0.1 (3)
O4—C7—C8—C9	−172.6 (2)	C9—C10—C15—C14	178.54 (19)
O3—C7—C8—C9	6.8 (3)	C9—C8—C16—O5	11.3 (3)
O4—C7—C8—C16	6.9 (3)	C7—C8—C16—O5	−168.29 (18)
O3—C7—C8—C16	−173.64 (15)	C9—C8—C16—C17	−168.69 (17)
C7—C8—C9—C10	−3.5 (3)	C7—C8—C16—C17	11.7 (3)
C16—C8—C9—C10	176.94 (17)	O5—C16—C17—S1	−9.5 (3)
C8—C9—C10—C11	−1.1 (3)	C8—C16—C17—S1	170.48 (13)
C8—C9—C10—C15	−179.82 (18)	C18—S1—C17—C16	−78.83 (16)
C7—O3—C11—C10	1.4 (3)	C19—N6—C18—S2	4.5 (3)
C7—O3—C11—C12	−178.37 (18)	C21—N6—C18—S2	−172.67 (17)
C15—C10—C11—O3	−178.91 (17)	C19—N6—C18—S1	−175.84 (14)
C9—C10—C11—O3	2.3 (3)	C21—N6—C18—S1	7.0 (2)
C15—C10—C11—C12	0.8 (3)	C17—S1—C18—N6	179.60 (14)
C9—C10—C11—C12	−177.98 (19)	C17—S1—C18—S2	−0.70 (14)
O3—C11—C12—C13	178.51 (18)	C18—N6—C19—C20	−91.4 (2)
C10—C11—C12—C13	−1.2 (3)	C21—N6—C19—C20	86.0 (2)
C11—C12—C13—C14	1.0 (3)	C18—N6—C21—C22	−93.0 (2)
C12—C13—C14—C15	−0.3 (3)	C19—N6—C21—C22	89.7 (2)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C9—H9···O5^{i}	0.93	2.49	3.198 (2)	134

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