5-(3,4-Dimethylphenylsulfonyl)-8-methoxy-2-methyl-2,3,5,6-tetrahydro-4*H*-2,6-methano-1,3-benzoxazocin-4-one

Vyacheslav N. Baumer,[a]* Svitlana S. Kovalenko,[b] Olexiy V. Silin[b] and Segriy M. Kovalenko[b]

[a]STC 'Institute for Single Crystals', National Academy of Sciences of Ukraine, 60 Lenin Avenue, Kharkiv 61001, Ukraine, and [b]National University of Pharmacy, 4 Blyukher Street, Kharkiv 61002, Ukraine

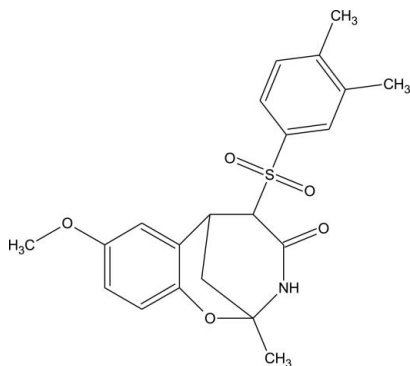Correspondence e-mail: baumer@xray.isc.kharkov.com

Molecules of the title molecule, $C_{21}H_{23}NO_5S$, form centrosymmetric dimers *via* intermolecular N—H···O bonds. In addition, there are intermolecular C—H···O bonds. The eight-membered ring and both six-membered rings belonging to the tricyclic ring system adopt a conformation which is intermediate between a sofa and a half-chair.

Related literature

For related literature, see: Bürgi & Dunitz (1994); Bilokin *et al.* (1998); Bondi (1964); Kovalenko & Victorova (2005); Konovalova *et al.* (2007).

Experimental

Crystal data

$C_{21}H_{23}NO_5S$	c = 11.724 (6) Å
M_r = 401.46	α = 94.77 (4)°
Triclinic, $P\bar{1}$	β = 106.22 (4)°
a = 8.077 (4) Å	γ = 98.36 (4)°
b = 11.153 (6) Å	V = 994.8 (9) Å^3

Z = 2	T = 293 (2) K
Mo $K\alpha$ radiation	$0.4 \times 0.15 \times 0.05$ mm
μ = 0.20 mm^{-1}	

Data collection

Siemens P3/PC diffractometer	R_{int} = 0.085
Absorption correction: none	2 standard reflections
3856 measured reflections	every 98 reflections
3673 independent reflections	intensity decay: 1%
1634 reflections with $I > 2\sigma(I)$	

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.045	258 parameters
$wR(F^2)$ = 0.092	H-atom parameters constrained
S = 1	$\Delta\rho_{max}$ = 0.17 e Å^{-3}
3673 reflections	$\Delta\rho_{min}$ = −0.16 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N10—H10···O11[i]	0.86	2.03	2.867 (4)	166
C20—H20···O2[ii]	0.93	2.53	3.442 (5)	166

Symmetry codes: (i) $-x + 1, -y + 1, -z + 2$; (ii) $x - 1, y, z$.

Data collection: *P3* (Siemens, 1989); cell refinement: *P3*; data reduction: *XDISK* (Siemens, 1991); program(s) used to solve structure: *SIR97* (Altomare *et al.*, 1999); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *XP* (Siemens, 1998); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

References

Altomare, A., Burla, M. C., Camalli, M., Cascarano, G. L., Giacovazzo, C., Guagliardi, A., Moliterni, A. G. G., Polidori, G. & Spagna, R. (1999). *J. Appl. Cryst.* **32**, 115–119.
Bilokin, Y. V., Kovalenko, S. N. & Chernykh, V. P. (1998). *Heterocycl. Commun.* **4**, 169–170.
Bondi, A. (1964). *J. Phys. Chem.* **68**, 441–446.
Bürgi, H.-B. & Dunitz, J. D. (1994). Editors. *Structure Correlation*, Vol. 2, pp. 771–777. Weinheim: VCH.
Farrugia, L. J. (1999). *J. Appl. Cryst.* **32**, 837–838.
Konovalova, I. S., Baumer, V. N., Shishkin, O. V., Vlasenko, Y. D., Parkhomenko, O. O. & Kovalenko, S. M. (2007). *Acta Cryst.* E**63**, o3011.
Kovalenko, V. N. & Victorova, A. P. (2005). *Compendium of medicinal preparation*, p. 1920. http://www.medbook.net.ru/013216.shtml
Sheldrick, G. M. (1997). *SHELXL97*. University of Göttingen, Germany.
Siemens (1989). *P3*. Siemens Analytical X-ray Instruments Inc., Karlsruhe, Germany.
Siemens (1991). *XDISK*. Siemens Analytical X-ray Instruments Inc., Karlsruhe, Germany.
Siemens (1998). *XP*. Siemens Analytical X-ray Instruments Inc., Karlsruhe, Germany.

supplementary materials

5-(3,4-Dimethylphenylsulfonyl)-8-methoxy-2-methyl-2,3,5,6-tetrahydro-4*H*-2,6-methano-1,3-benzoxazocin-4-one

V. N. Baumer, S. S. Kovalenko, O. V. Silin and S. M. Kovalenko

Comment

The 2,3,5,6-tetrahydro-4*H*-2,6-methano-1,3-benzoxazocin-4-one derivatives have been studied for their biological activity (Kovalenko & Victorova, 2005). One of such derivatives, Lortalamine, is used as an antidepressant. Moreover, it is well known that sulfur containing pharmacophores such as sulfonyl and thiophene are used for synthesis of many medications *e.g.* antibiotics, anaesthetics, antiparasitics, resolvents, antihelmintic drugs, anticholinergic drugs, antihistamines *etc*. (Kovalenko & Victorova, 2005). Thus, the developement of synthetic procedures and the study of structural peculiarities connected with the biological activity of these compounds seems to be very important. The present work is a continuation of our previous investigation (Konovalova *et al.*, 2007).

Bilokin *et al.* (1998) have reported the very similar structure $C_{13}H_{14}N_2O_2S$, (II), in which the atom C4 has no substituent and thiocarboxamide group is attached to the atom C12 instead xylenesulfonyl one. Bond distances and angles in the title compound are in a good agreement with tabulated data (Bürgi & Dunitz, 1994) and those for the parent compound (II) as well, so these values will not be discussed here. The tricyclic part of the title compound consist of three approximately planar fragments namely hydroquinone (denoted hereafter as P1) including atoms O4, O8 and aromatic ring (C2 to C7) with r.m.s. deviation of 0.027 Å and maximum deviation of +0.045 Å for atom O4; *N*-methylpropanamide fragment (P2, atoms C1, C9, N10, O11, C11, C12) for which r.m.s.d. is 0.076Å and maximum deviations are −0.110 Å for C1 and +0.133 Å for C12; and butane fragment (P3, atoms C1, C13, C9, C14), r.m.s.d. of 0.022 Å, maximum deviations are observed for C9 (−0.025 Å) and C14 (+0.022 Å). Planes P1 and P2 make a dihedral angle of 82.18 (8)° one to another and 129.90 (19)° and 130.83 (12)° to the plane P3, respectively. Such noticeable distinction between the former value and latter two ones is caused by a sterical hindrance between hydrogen atoms attached to C13 and atoms C7 and C11 (C7···H13*a* 2.85 Å, C11···H13*b* 3.00 Å, the sum of van der Waals radii is 2.90 Å (Bondi, 1964)). A position of the sulfonyl group with respect to S1–C12 bond is fixed by intermolecular contact O1···H21$^{2-x,2-y,-z}$ 2.70Å and intermolecular hydrogen bond O2···H20$^{x-1,y,z}$ 2.53 Å (the sum of van der Waals radii is 2.72 Å. Aromatic ring of xylene fragment is attached slightly asymmetrically to the sulfonyl group, so that torsion angles O1—S1—C16—C21 and O2—S1—C16—C17 are 39.8 (2)° and 15.4 (3)°, resp. Torsion position of this ring is fixed by the hydrogen bond C20–H20···O2$^{x+1,y,z}$ 2.53 Å (C···O 3.44 Å, C—H···O 166°) and intermolecular contacts C20···H14*c*$^{x+1,y,z}$ of 2.86 Å and C21···H14*c*$^{x+1,y,z}$ of 2.76 Å. Also it should be noted that methyl atom C22 lies in the plane of the ring (its deviation is +0.020 (5) Å) whereas atom C23 has a deviation of −0.134 Å due to intermolecular contact H23*b*···H23*b*$^{2-x,1-y,-1-z}$ of 2.37 Å (sum of radii is 2.40 Å).

In addition to the mentioned C–H···O bond, the strong intermolecular H-bond is observed in (I) namely N10—H10···O11$^{1-x,1-y,-z}$ (N—H 0.86 Å, H···O 2.03 Å, N···O 2.869 (3) Å, N—H···O 166°). This bond calls forth formation of centrosymmetrical molecular dimers in the structure. It should be noted that similar dimers are observed in the structure (II) as well in which the parent molecule have not so bulk substituents.

supplementary materials

Experimental

Title compound was obtained by reaction of 3-[(3,4- dimethylphenyl)sulfonyl]-6-methoxy-2*H*-1-benzopyran-2-ones with acetone and ammonia according to a Michael-type condensation at the molar ratio of components 1:1:1 in methanol medium.

Refinement

All hydrogen atoms were located from the difference electron density map and refined using the riding model approximation with U_{iso} constrained to be 1.5 times U_{eq} of the carrier atom for the methyl groups and 1.2 times U_{eq} of the carrier atom for the other atoms. Rotation of metyl groups was refined using AFIX 137 instruction (Sheldrick, 1997).

Figures
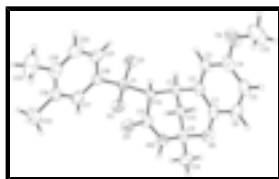
Fig. 1. A view of the title molecule, showing displacement ellipsoids at the 40% probability level and the atom-numbering scheme.

5-[(3,4-Dimethylphenyl)sulfonyl]-8-methoxy-2-methyl-2,3,5,6-tetrahydro-4*H*- 2,6-methano-1,3-benzoxazocin-4-one

Crystal data

$C_{21}H_{23}NO_5S$	$Z = 2$
$M_r = 401.46$	$F_{000} = 424$
Triclinic, $P\bar{1}$	$D_x = 1.340$ Mg m^{-3}
Hall symbol: -P 1	Mo $K\alpha$ radiation $\lambda = 0.71073$ Å
$a = 8.077$ (4) Å	Cell parameters from 26 reflections
$b = 11.153$ (6) Å	$\theta = 10–11°$
$c = 11.724$ (6) Å	$\mu = 0.20$ mm^{-1}
$\alpha = 94.77$ (4)°	$T = 293$ (2) K
$\beta = 106.22$ (4)°	Plate, colourless
$\gamma = 98.36$ (4)°	$0.4 \times 0.15 \times 0.05$ mm
$V = 994.8$ (9) Å^3	

Data collection

Siemens P3/PC diffractometer	$R_{int} = 0.085$
Radiation source: sealed tube	$\theta_{max} = 25.6°$
Monochromator: graphite	$\theta_{min} = 1.8°$
$T = 293(2)$ K	$h = -9 \rightarrow 9$
2θ/θ scans	$k = 0 \rightarrow 13$
Absorption correction: none	$l = -14 \rightarrow 14$

3856 measured reflections

3673 independent reflections

1634 reflections with $I > 2\sigma(I)$

2 standard reflections

every 98 reflections

intensity decay: 1%

Refinement

Refinement on F^2	H-atom parameters constrained
Least-squares matrix: full	$[\exp(2.00(\sin\theta/\lambda)^2)]/[\sigma^2(F_o^2) + (0.024P)^2]$ where $P = 0.33333F_o^2 + 0.66667F_c^2$
$R[F^2 > 2\sigma(F^2)] = 0.045$	$(\Delta/\sigma)_{max} = 0.001$
$wR(F^2) = 0.092$	$\Delta\rho_{max} = 0.17$ e Å^{-3}
$S = 1$	$\Delta\rho_{min} = -0.16$ e Å^{-3}
3673 reflections	Extinction correction: SHELXL, $Fc^* = kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
258 parameters	Extinction coefficient: 0.0098 (9)

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 > 2sigma(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	0.28829 (8)	0.15966 (6)	1.10431 (6)	0.04256 (18)
O1	0.2056 (2)	0.03322 (15)	1.08861 (15)	0.0548 (5)
O2	0.4723 (2)	0.19048 (18)	1.16111 (15)	0.0598 (5)
C1	0.2693 (3)	0.1128 (2)	0.86480 (19)	0.0389 (6)
H1	0.2239	0.0302	0.8777	0.047*
C2	0.1706 (3)	0.1375 (2)	0.7398 (2)	0.0415 (6)
C3	−0.0064 (3)	0.0977 (2)	0.6899 (2)	0.0482 (7)
H3	−0.0692	0.0535	0.7328	0.058*
C4	−0.0929 (4)	0.1221 (3)	0.5771 (2)	0.0597 (8)
O4	−0.2693 (3)	0.0809 (2)	0.54010 (18)	0.0884 (7)
C5	0.0010 (4)	0.1849 (3)	0.5121 (2)	0.0642 (9)
H5	−0.0553	0.1998	0.4353	0.077*
C6	0.1770 (4)	0.2253 (2)	0.5611 (2)	0.0612 (8)
H6	0.2396	0.2674	0.5168	0.073*
C7	0.2633 (3)	0.2045 (2)	0.6751 (2)	0.0500 (7)
O8	0.4397 (2)	0.25171 (16)	0.71831 (15)	0.0539 (5)
C9	0.5216 (3)	0.2484 (2)	0.8446 (2)	0.0450 (7)

N10	0.4723 (2)	0.34304 (19)	0.91249 (17)	0.0483 (6)
H10	0.5326	0.4152	0.9212	0.058*
C11	0.3452 (3)	0.3316 (2)	0.9628 (2)	0.0437 (7)
O11	0.3084 (2)	0.42068 (16)	1.01614 (16)	0.0607 (5)
C12	0.2401 (3)	0.2072 (2)	0.95643 (19)	0.0380 (6)
H12	0.116	0.2142	0.931	0.046*
C13	0.4611 (3)	0.1244 (2)	0.8754 (2)	0.0473 (7)
H13A	0.4796	0.0608	0.821	0.057*
H13B	0.5269	0.116	0.9565	0.057*
C14	0.7147 (3)	0.2781 (3)	0.8582 (2)	0.0644 (8)
H14A	0.7423	0.3588	0.838	0.097*
H14B	0.7447	0.2199	0.8057	0.097*
H14C	0.7801	0.2747	0.9396	0.097*
C15	−0.3674 (4)	0.1109 (4)	0.4273 (3)	0.1047 (13)
H15A	−0.488	0.0735	0.4097	0.157*
H15B	−0.3212	0.0813	0.3654	0.157*
H15C	−0.3587	0.198	0.4312	0.157*
C16	0.1791 (3)	0.2475 (2)	1.1781 (2)	0.0416 (7)
C17	0.2650 (3)	0.3499 (3)	1.2567 (2)	0.0580 (8)
H17	0.3866	0.3687	1.2777	0.07*
C18	0.1733 (4)	0.4271 (3)	1.3063 (2)	0.0599 (8)
C19	−0.0046 (4)	0.3982 (3)	1.2718 (2)	0.0631 (8)
C20	−0.0919 (4)	0.2945 (3)	1.1951 (3)	0.0688 (9)
H20	−0.2133	0.2752	1.1751	0.083*
C21	−0.0011 (3)	0.2191 (3)	1.1477 (2)	0.0571 (8)
H21	−0.0608	0.1491	1.0953	0.068*
C22	0.2765 (5)	0.5395 (3)	1.3931 (3)	0.1008 (13)
H22A	0.2199	0.6084	1.3744	0.151*
H22B	0.3931	0.5562	1.3863	0.151*
H22C	0.2818	0.5249	1.4735	0.151*
C23	−0.1140 (5)	0.4851 (4)	1.3137 (3)	0.1136 (14)
H23A	−0.086	0.5644	1.2906	0.17*
H23B	−0.0878	0.4918	1.3993	0.17*
H23C	−0.2364	0.4533	1.2774	0.17*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.0389 (3)	0.0465 (4)	0.0418 (4)	0.0117 (3)	0.0078 (3)	0.0107 (3)
O1	0.0729 (11)	0.0329 (10)	0.0642 (11)	0.0113 (9)	0.0253 (9)	0.0169 (8)
O2	0.0371 (9)	0.0860 (14)	0.0542 (11)	0.0170 (9)	0.0046 (8)	0.0188 (10)
C1	0.0551 (14)	0.0187 (12)	0.0436 (14)	0.0128 (11)	0.0105 (12)	0.0112 (10)
C2	0.0529 (14)	0.0329 (14)	0.0411 (14)	0.0161 (12)	0.0151 (12)	0.0007 (11)
C3	0.0545 (15)	0.0431 (16)	0.0415 (15)	0.0067 (13)	0.0062 (12)	0.0054 (12)
C4	0.0668 (18)	0.0549 (18)	0.0457 (17)	0.0202 (15)	−0.0033 (15)	−0.0055 (14)
O4	0.0738 (14)	0.1019 (18)	0.0651 (14)	0.0055 (13)	−0.0129 (11)	0.0076 (13)
C5	0.086 (2)	0.064 (2)	0.0349 (16)	0.0161 (17)	0.0030 (15)	0.0109 (14)
C6	0.090 (2)	0.0587 (19)	0.0454 (16)	0.0247 (16)	0.0253 (15)	0.0251 (14)

C7	0.0604 (16)	0.0527 (17)	0.0408 (15)	0.0222 (14)	0.0167 (13)	0.0000 (13)
O8	0.0563 (10)	0.0601 (12)	0.0524 (11)	0.0148 (9)	0.0230 (9)	0.0143 (9)
C9	0.0482 (14)	0.0475 (16)	0.0436 (15)	0.0245 (12)	0.0106 (12)	0.0120 (12)
N10	0.0501 (11)	0.0389 (13)	0.0603 (13)	0.0080 (10)	0.0264 (10)	−0.0041 (11)
C11	0.0476 (14)	0.0389 (15)	0.0515 (15)	0.0187 (12)	0.0202 (13)	0.0064 (12)
O11	0.0782 (11)	0.0403 (11)	0.0771 (12)	0.0091 (9)	0.0466 (10)	0.0029 (9)
C12	0.0336 (12)	0.0425 (15)	0.0322 (13)	0.0025 (12)	0.0036 (10)	0.0020 (11)
C13	0.0661 (16)	0.0351 (15)	0.0413 (14)	0.0247 (13)	0.0098 (12)	0.0025 (12)
C14	0.0562 (16)	0.077 (2)	0.0691 (19)	0.0209 (16)	0.0259 (15)	0.0165 (16)
C15	0.096 (3)	0.107 (3)	0.079 (3)	0.009 (2)	−0.021 (2)	0.006 (2)
C16	0.0446 (14)	0.0446 (16)	0.0293 (13)	−0.0013 (12)	0.0045 (11)	0.0073 (12)
C17	0.0581 (16)	0.074 (2)	0.0455 (16)	0.0100 (15)	0.0182 (13)	0.0178 (15)
C18	0.0865 (19)	0.0551 (19)	0.0373 (15)	−0.0021 (16)	0.0257 (14)	0.0007 (13)
C19	0.0770 (19)	0.071 (2)	0.0484 (17)	0.0197 (17)	0.0264 (15)	0.0092 (15)
C20	0.0514 (16)	0.095 (3)	0.0666 (19)	0.0134 (17)	0.0242 (15)	0.0206 (18)
C21	0.0584 (17)	0.0618 (19)	0.0603 (17)	0.0133 (15)	0.0273 (14)	0.0213 (15)
C22	0.152 (3)	0.072 (2)	0.072 (2)	−0.008 (2)	0.044 (2)	−0.0143 (19)
C23	0.150 (3)	0.127 (3)	0.093 (3)	0.073 (3)	0.060 (2)	0.012 (2)

Geometric parameters (Å, °)

S1—O2	1.4252 (18)	C11—C12	1.502 (3)
S1—O1	1.4434 (19)	C12—H12	0.98
S1—C16	1.739 (3)	C13—H13A	0.97
S1—C12	1.809 (2)	C13—H13B	0.97
C1—C13	1.504 (3)	C14—H14A	0.96
C1—C2	1.529 (3)	C14—H14B	0.96
C1—C12	1.531 (3)	C14—H14C	0.96
C1—H1	0.98	C15—H15A	0.96
C2—C3	1.374 (3)	C15—H15B	0.96
C2—C7	1.394 (3)	C15—H15C	0.96
C3—C4	1.382 (3)	C16—C17	1.368 (3)
C3—H3	0.93	C16—C21	1.380 (3)
C4—O4	1.364 (3)	C17—C18	1.405 (4)
C4—C5	1.379 (4)	C17—H17	0.93
O4—C15	1.433 (4)	C18—C19	1.360 (4)
C5—C6	1.367 (4)	C18—C22	1.521 (4)
C5—H5	0.93	C19—C20	1.371 (4)
C6—C7	1.380 (3)	C19—C23	1.541 (4)
C6—H6	0.93	C20—C21	1.373 (4)
C7—O8	1.378 (3)	C20—H20	0.93
O8—C9	1.449 (3)	C21—H21	0.93
C9—N10	1.438 (3)	C22—H22A	0.96
C9—C13	1.506 (3)	C22—H22B	0.96
C9—C14	1.507 (3)	C22—H22C	0.96
N10—C11	1.315 (3)	C23—H23A	0.96
N10—H10	0.86	C23—H23B	0.96
C11—O11	1.246 (3)	C23—H23C	0.96
O2—S1—O1	118.40 (12)	S1—C12—H12	107.6

O2—S1—C16	109.80 (12)	C1—C13—C9	107.98 (19)
O1—S1—C16	108.70 (12)	C1—C13—H13A	110.1
O2—S1—C12	108.85 (11)	C9—C13—H13A	110.1
O1—S1—C12	106.35 (12)	C1—C13—H13B	110.1
C16—S1—C12	103.70 (12)	C9—C13—H13B	110.1
C13—C1—C2	109.1 (2)	H13A—C13—H13B	108.4
C13—C1—C12	110.50 (18)	C9—C14—H14A	109.5
C2—C1—C12	107.85 (18)	C9—C14—H14B	109.5
C13—C1—H1	109.8	H14A—C14—H14B	109.5
C2—C1—H1	109.8	C9—C14—H14C	109.5
C12—C1—H1	109.8	H14A—C14—H14C	109.5
C3—C2—C7	118.9 (2)	H14B—C14—H14C	109.5
C3—C2—C1	122.4 (2)	O4—C15—H15A	109.5
C7—C2—C1	118.7 (2)	O4—C15—H15B	109.5
C2—C3—C4	121.3 (3)	H15A—C15—H15B	109.5
C2—C3—H3	119.3	O4—C15—H15C	109.5
C4—C3—H3	119.3	H15A—C15—H15C	109.5
O4—C4—C5	125.5 (3)	H15B—C15—H15C	109.5
O4—C4—C3	115.2 (3)	C17—C16—C21	119.3 (3)
C5—C4—C3	119.3 (3)	C17—C16—S1	121.8 (2)
C4—O4—C15	117.6 (3)	C21—C16—S1	118.6 (2)
C6—C5—C4	119.8 (3)	C16—C17—C18	121.3 (3)
C6—C5—H5	120.1	C16—C17—H17	119.3
C4—C5—H5	120.1	C18—C17—H17	119.3
C5—C6—C7	121.1 (3)	C19—C18—C17	117.8 (3)
C5—C6—H6	119.5	C19—C18—C22	123.5 (3)
C7—C6—H6	119.5	C17—C18—C22	118.7 (3)
O8—C7—C6	117.0 (2)	C18—C19—C20	121.4 (3)
O8—C7—C2	123.6 (2)	C18—C19—C23	120.5 (3)
C6—C7—C2	119.5 (2)	C20—C19—C23	118.1 (3)
C7—O8—C9	116.25 (19)	C19—C20—C21	120.4 (3)
N10—C9—O8	108.54 (19)	C19—C20—H20	119.8
N10—C9—C13	110.9 (2)	C21—C20—H20	119.8
O8—C9—C13	108.7 (2)	C20—C21—C16	119.7 (3)
N10—C9—C14	109.6 (2)	C20—C21—H21	120.1
O8—C9—C14	103.7 (2)	C16—C21—H21	120.1
C13—C9—C14	114.9 (2)	C18—C22—H22A	109.5
C11—N10—C9	127.2 (2)	C18—C22—H22B	109.5
C11—N10—H10	116.4	H22A—C22—H22B	109.5
C9—N10—H10	116.4	C18—C22—H22C	109.5
O11—C11—N10	122.3 (2)	H22A—C22—H22C	109.5
O11—C11—C12	118.4 (2)	H22B—C22—H22C	109.5
N10—C11—C12	119.3 (2)	C19—C23—H23A	109.5
C11—C12—C1	112.5 (2)	C19—C23—H23B	109.5
C11—C12—S1	109.45 (16)	H23A—C23—H23B	109.5
C1—C12—S1	111.78 (16)	C19—C23—H23C	109.5
C11—C12—H12	107.6	H23A—C23—H23C	109.5
C1—C12—H12	107.6	H23B—C23—H23C	109.5
C13—C1—C2—C3	159.1 (2)	C2—C1—C12—C11	−75.0 (2)

C12—C1—C2—C3	−80.8 (3)	C13—C1—C12—S1	−79.3 (2)
C13—C1—C2—C7	−21.9 (3)	C2—C1—C12—S1	161.46 (16)
C12—C1—C2—C7	98.2 (2)	O2—S1—C12—C11	−42.5 (2)
C7—C2—C3—C4	0.5 (4)	O1—S1—C12—C11	−171.12 (16)
C1—C2—C3—C4	179.5 (2)	C16—S1—C12—C11	74.33 (19)
C2—C3—C4—O4	−177.8 (2)	O2—S1—C12—C1	82.76 (18)
C2—C3—C4—C5	1.6 (4)	O1—S1—C12—C1	−45.85 (18)
C5—C4—O4—C15	−3.6 (4)	C16—S1—C12—C1	−160.39 (16)
C3—C4—O4—C15	175.9 (3)	C2—C1—C13—C9	54.8 (2)
O4—C4—C5—C6	177.6 (3)	C12—C1—C13—C9	−63.6 (2)
C3—C4—C5—C6	−1.8 (4)	N10—C9—C13—C1	51.4 (2)
C4—C5—C6—C7	−0.1 (4)	O8—C9—C13—C1	−67.9 (2)
C5—C6—C7—O8	−178.1 (2)	C14—C9—C13—C1	176.4 (2)
C5—C6—C7—C2	2.3 (4)	O2—S1—C16—C17	15.4 (2)
C3—C2—C7—O8	178.0 (2)	O1—S1—C16—C17	146.4 (2)
C1—C2—C7—O8	−1.0 (3)	C12—S1—C16—C17	−100.8 (2)
C3—C2—C7—C6	−2.4 (4)	O2—S1—C16—C21	−170.7 (2)
C1—C2—C7—C6	178.5 (2)	O1—S1—C16—C21	−39.8 (2)
C6—C7—O8—C9	169.4 (2)	C12—S1—C16—C21	73.1 (2)
C2—C7—O8—C9	−11.0 (3)	C21—C16—C17—C18	−0.5 (4)
C7—O8—C9—N10	−75.8 (3)	S1—C16—C17—C18	173.3 (2)
C7—O8—C9—C13	45.0 (3)	C16—C17—C18—C19	−1.2 (4)
C7—O8—C9—C14	167.7 (2)	C16—C17—C18—C22	180.0 (3)
O8—C9—N10—C11	96.9 (3)	C17—C18—C19—C20	2.6 (4)
C13—C9—N10—C11	−22.6 (3)	C22—C18—C19—C20	−178.6 (3)
C14—C9—N10—C11	−150.5 (2)	C17—C18—C19—C23	−174.4 (3)
C9—N10—C11—O11	−177.2 (2)	C22—C18—C19—C23	4.3 (4)
C9—N10—C11—C12	3.0 (3)	C18—C19—C20—C21	−2.3 (5)
O11—C11—C12—C1	166.5 (2)	C23—C19—C20—C21	174.9 (3)
N10—C11—C12—C1	−13.7 (3)	C19—C20—C21—C16	0.4 (4)
O11—C11—C12—S1	−68.6 (2)	C17—C16—C21—C20	0.9 (4)
N10—C11—C12—S1	111.2 (2)	S1—C16—C21—C20	−173.1 (2)
C13—C1—C12—C11	44.3 (3)		

Hydrogen-bond geometry (Å, °)

D—H⋯*A*	*D*—H	H⋯*A*	*D*⋯*A*	*D*—H⋯*A*
N10—H10⋯O11[i]	0.86	2.03	2.867 (4)	166
C20—H20⋯O2[ii]	0.93	2.53	3.442 (5)	166

Symmetry codes: (i) −*x*+1, −*y*+1, −*z*+2; (ii) *x*−1, *y*, *z*.

Fig. 1