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4-[(4-Methylbenzenesulfonamido)-methyl]cyclohexanecarboxylic acid

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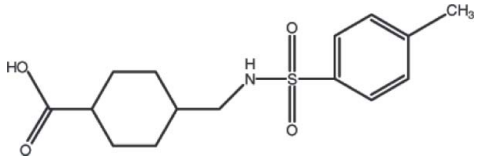
Key indicators: single-crystal X-ray study; T = 296 K; mean σ(C–C) = 0.007 Å; R factor = 0.076; wR factor = 0.227; data-to-parameter ratio = 19.4.

The title compound, $C_{15}H_{21}NO_4S$, crystallized with two independent molecules in the asymmetric unit in which the dihedral angles between the mean planes of the benzene and cyclohexane rings are 78.3 (2) and 67.6 (2)°. The substituents of the cyclohexyl rings are in equatorial orientations. In the crystal, both molecules form $R_2^2(6)$ carboxylic acid inversion dimers *via* pairs of O—H···O hydrogen bonds. Further N—H···O and C—H···O interactions generate a three-dimensional network.

Related literature

For background to tranexamic acid, see: Boylan *et al.* (1996); Nilsson (1980); Khan *et al.* (2002); Shah *et al.* (2010); Shahzadi *et al.* (2007); Svahn *et al.* (1986); Vávrová *et al.* (2005).



Experimental

Crystal data

$C_{15}H_{21}NO_4S$
M_r = 311.40
Triclinic, $P\overline{1}$
a = 6.0655 (5) Å
b = 10.3999 (9) Å
c = 26.468 (2) Å
α = 98.947 (3)°
β = 90.001 (4)°
γ = 104.880 (3)°
V = 1592.6 (2) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.22 mm^{-1}
T = 296 K
0.24 × 0.18 × 0.07 mm

Data collection

Bruker APEXII CCD diffractometer
18232 measured reflections
7641 independent reflections
3536 reflections with $I > 2\sigma(I)$
R_{int} = 0.059

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.076
$wR(F^2)$ = 0.227
S = 1.02
7641 reflections
394 parameters
5 restraints
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.53 e Å^{-3}
$\Delta\rho_{min}$ = −0.35 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O4—H4O···O3^{i}	0.84 (4)	1.81 (4)	2.638 (6)	171 (4)
O7—H7O···O8^{ii}	0.81 (4)	1.87 (4)	2.664 (5)	166 (5)
N1—H1N···O6^{iii}	0.86 (3)	2.15 (3)	3.001 (4)	171 (4)
N2—H2N···O2	0.84 (3)	2.09 (4)	2.924 (4)	171 (4)
C10—H10B···O6^{iii}	0.97	2.57	3.446 (6)	151
C19—H19···O2	0.93	2.59	3.490 (5)	164

Symmetry codes: (i) $-x+3, -y, -z+1$; (ii) $-x+3, -y+2, -z+2$; (iii) $x+1, y, z$.

Data collection: *APEX2* (Bruker, 2007); cell refinement: *SAINT* (Bruker, 2007); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *PLATON* (Spek, 2009); software used to prepare material for publication: *WinGX* (Farrugia, 1999) and *PLATON*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HB5893).

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4-[(4-Methylbenzenesulfonamido)methyl]cyclohexanecarboxylic acid

Muhammad Ashfaq, Samina Iram, Mehmet Akkurt, Islam Ullah Khan, Ghulam Mustafa and Shahzad Sharif

S1. Comment

Tranexamic acid, is a synthetic amino acid that is used commonly for curing abnormal bleeding in a variety of diseases (Boylan *et al.*, 1996; Nilsson, 1980). Number of Scientists derivatized this drug, evaluated their activities and found most of them superior to the parent drug (Svahn *et al.*, 1986; Khan *et al.*, 2002; Vávrová *et al.*, 2005; Shahzadi *et al.*, 2007; Shah *et al.*, 2010).

As seen in Fig. 1, the title compound (I) crystalizes in the triclinic space group P-1 with two independent molecules in the asymmetric unit. The dihedral angle is 64.7 (2) ° between the benzene rings (C2–C7 and C17–C22) of two independent molecules in the asymmetric unit. The cyclohexane rings (C9–C14 and C24–C29) of two independent molecules have a chair configuration [the puckering parameters are Q_T = 0.561 (5) Å, θ = 174.4 (5) °, φ = 353 (6) ° and Q_T = 0.563 (5) Å, θ = 0.8 (5) °, φ = 139 (29) °, respectively].

In the crystal structure, intermolecular N—H···O, O—H···O and C—H···O interactions help to stabilize the crystal structure (Table 1, Fig. 2).

S2. Experimental

4-toluenesulfonyl chloride (1.21 g, 6.36 mmol) was added to the solution of tranexamic acid (1.0 g, 6.36 mmol) in distilled water (10 ml). The reaction mixture was stirred at room temperature at pH 8–9, which was adjusted by 1 *M* sodium carbonate solution. After completion of the reaction which was observed by the consumption of 4-toluenesulfonyl chloride, the pH was adjusted at 2–3 using 1 N HCl solution, which results the formation of precipiates, which were filtered off and dried. The product was recrystallized using methanol to yield colourless plates of (I).

S3. Refinement

The N and O-bound H atoms were located in a difference Fourier map and their positional parameters were restrained [N1—H1N = 0.86 (3), N2—H2N = 0.84 (3), O4—H4O = 0.84 (4) and O7—H7O = 0.81 (4) Å]. Their displacement parameters were refined using a riding model, with U_{iso}(H) = 1.2U_{eq}(N) or 1.5U_{eq}(O). The remaining H atoms were placed at calculated positions and allowed to ride on their carrier atoms with C—H = 0.93 - 0.98 Å, and U_{iso} = 1.2U_{eq}(C) for CH and CH_2 groups and U_{iso} = 1.5U_{eq}(C) for CH_3 group.

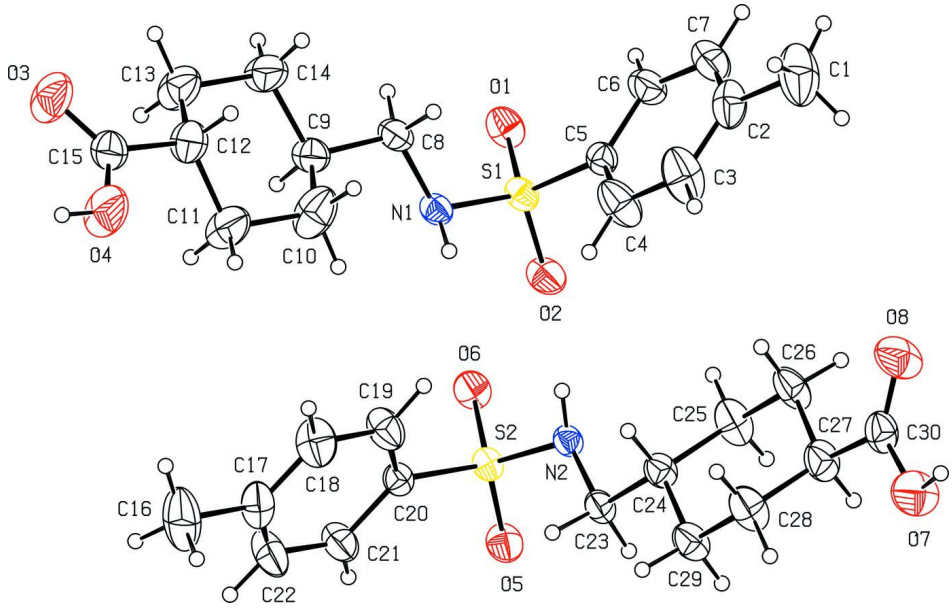

Figure 1

View of the two independent molecules in the asymmetric unit with displacement ellipsoids for non-H atoms drawn at the 30% probability level.

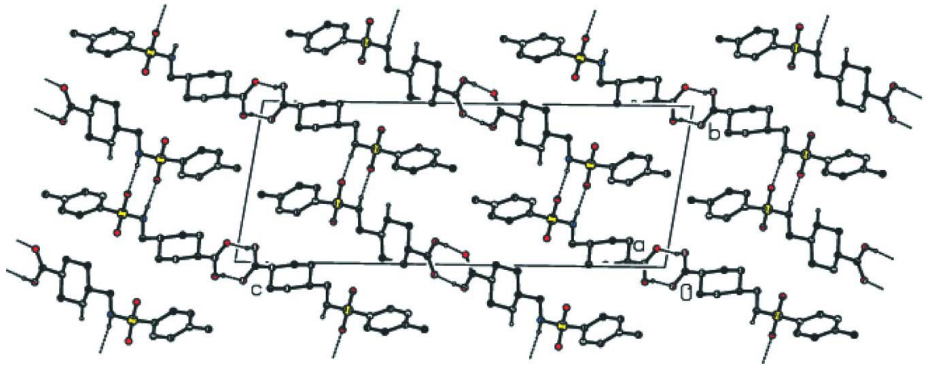

Figure 2

The packing and hydrogen bonding of (I), viewed down *a* axis. Hydrogen atoms not involved in hydrogen bonding have been omitted for clarity.

4-[(4-Methylbenzenesulfonamido)methyl]cyclohexanecarboxylic acid

Crystal data

$C_{15}H_{21}NO_4S$
$M_r = 311.40$
Triclinic, $P\overline{1}$
Hall symbol: -P 1

$a = 6.0655\ (5)$ Å
$b = 10.3999\ (9)$ Å
$c = 26.468\ (2)$ Å
$\alpha = 98.947\ (3)°$

β = 90.001 (4)°
γ = 104.880 (3)°
V = 1592.6 (2) Å^3
Z = 4
F(000) = 664
D_x = 1.299 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å

Cell parameters from 2057 reflections
θ = 2.8–21.5°
μ = 0.22 mm^{-1}
T = 296 K
Plate, colourless
0.24 × 0.18 × 0.07 mm

Data collection

Bruker APEXII CCD diffractometer
Radiation source: sealed tube
Graphite monochromator
φ and ω scans
18232 measured reflections
7641 independent reflections

3536 reflections with $I > 2\sigma(I)$
R_{int} = 0.059
θ_{max} = 28.3°, θ_{min} = 2.3°
h = −5→8
k = −13→13
l = −33→35

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.076
$wR(F^2)$ = 0.227
S = 1.02
7641 reflections
394 parameters
5 restraints
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
$w = 1/[\sigma^2(F_o^2) + (0.1014P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.53 e Å^{-3}
$\Delta\rho_{min}$ = −0.35 e Å^{-3}

Special details

Geometry. Bond distances, angles *etc*. have been calculated using the rounded fractional coordinates. All su's are estimated from the variances of the (full) variance-covariance matrix. The cell e.s.d.'s are taken into account in the estimation of distances, angles and torsion angles

Refinement. Refinement on F^2 for ALL reflections except those flagged by the user for potential systematic errors. Weighted R-factors wR and all goodnesses 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 observed criterion of $F^2 > \sigma(F^2)$ is used only for calculating -R-factor-obs *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	0.59294 (16)	0.36920 (10)	0.78560 (4)	0.0462 (3)
O1	0.4029 (4)	0.2571 (3)	0.78800 (10)	0.0609 (10)
O2	0.5563 (5)	0.4991 (3)	0.78533 (10)	0.0647 (11)
O3	1.2671 (7)	−0.0636 (4)	0.53183 (13)	0.1013 (17)
O4	1.4863 (7)	0.1415 (4)	0.54321 (14)	0.1083 (17)
N1	0.7227 (5)	0.3365 (3)	0.73371 (11)	0.0450 (11)
C1	1.2352 (11)	0.4191 (6)	0.96605 (19)	0.114 (3)
C2	1.0735 (9)	0.4061 (5)	0.92107 (17)	0.0733 (19)
C3	1.1413 (8)	0.4766 (6)	0.88168 (19)	0.093 (2)
C4	0.9963 (8)	0.4656 (5)	0.84050 (17)	0.0814 (19)

C5	0.7805 (6)	0.3824 (4)	0.83802 (13)	0.0442 (12)
C6	0.7115 (8)	0.3108 (4)	0.87682 (15)	0.0656 (17)
C7	0.8597 (10)	0.3241 (5)	0.91804 (17)	0.082 (2)
C8	0.7808 (7)	0.2069 (4)	0.72344 (14)	0.0562 (16)
C9	0.8747 (8)	0.1807 (4)	0.67113 (16)	0.0592 (17)
C10	1.0961 (9)	0.2721 (5)	0.6634 (2)	0.092 (2)
C11	1.1912 (10)	0.2368 (4)	0.61059 (19)	0.093 (2)
C12	1.2054 (9)	0.0906 (4)	0.60319 (17)	0.0717 (19)
C13	0.9806 (9)	−0.0001 (5)	0.60706 (19)	0.084 (2)
C14	0.8840 (10)	0.0355 (4)	0.65955 (18)	0.086 (2)
C15	1.3243 (9)	0.0523 (5)	0.55579 (17)	0.0653 (17)
S2	0.25305 (15)	0.71011 (9)	0.71862 (3)	0.0417 (3)
O5	0.1844 (5)	0.8290 (3)	0.71441 (10)	0.0600 (10)
O6	0.0813 (4)	0.5889 (3)	0.72280 (9)	0.0562 (9)
O7	1.3989 (6)	1.0912 (4)	0.95942 (13)	0.0918 (17)
O8	1.2587 (6)	0.8845 (3)	0.97377 (12)	0.0874 (12)
N2	0.4188 (5)	0.7396 (3)	0.76897 (10)	0.0416 (10)
C16	0.7982 (10)	0.5973 (6)	0.53281 (18)	0.110 (3)
C17	0.6599 (8)	0.6246 (5)	0.57895 (16)	0.0673 (17)
C18	0.6913 (8)	0.5789 (5)	0.62363 (18)	0.0771 (19)
C19	0.5698 (8)	0.6030 (5)	0.66593 (16)	0.0675 (17)
C20	0.4129 (6)	0.6759 (4)	0.66451 (13)	0.0429 (11)
C21	0.3779 (8)	0.7221 (4)	0.61988 (15)	0.0649 (17)
C22	0.4988 (10)	0.6941 (5)	0.57743 (17)	0.084 (2)
C23	0.6137 (6)	0.8615 (4)	0.77586 (13)	0.0464 (12)
C24	0.7819 (6)	0.8593 (4)	0.81700 (13)	0.0487 (12)
C25	0.6862 (7)	0.8384 (5)	0.86771 (15)	0.0705 (18)
C26	0.8735 (7)	0.8386 (5)	0.90713 (15)	0.0674 (16)
C27	1.0635 (7)	0.9679 (4)	0.91159 (15)	0.0641 (17)
C28	1.1607 (7)	0.9884 (5)	0.86120 (15)	0.0728 (19)
C29	0.9747 (7)	0.9873 (4)	0.82208 (14)	0.0597 (14)
C30	1.2480 (7)	0.9766 (5)	0.95100 (15)	0.0604 (16)
H1A	1.27680	0.51080	0.98320	0.1720*
H1B	1.36980	0.39360	0.95410	0.1720*
H1C	1.16170	0.36110	0.98940	0.1720*
H1N	0.831 (6)	0.408 (3)	0.7338 (16)	0.0790*
H3	1.28830	0.53290	0.88290	0.1110*
H4	1.04500	0.51480	0.81430	0.0980*
H4O	1.555 (6)	0.119 (3)	0.5172 (13)	0.0980*
H6	0.56550	0.25330	0.87550	0.0790*
H7	0.81110	0.27530	0.94440	0.0990*
H8A	0.64520	0.13520	0.72630	0.0670*
H8B	0.89300	0.20490	0.74920	0.0670*
H9	0.76490	0.19270	0.64630	0.0710*
H10A	1.20500	0.26930	0.68980	0.1100*
H10B	1.07990	0.36340	0.66720	0.1100*
H11A	1.09210	0.24870	0.58390	0.1120*
H11B	1.34170	0.29630	0.60840	0.1120*

H12	1.29950	0.08310	0.63210	0.0860*
H13A	0.99250	−0.09240	0.60260	0.1010*
H13B	0.87800	0.00720	0.58020	0.1010*
H14A	0.73120	−0.02250	0.66060	0.1030*
H14B	0.97800	0.01770	0.68590	0.1030*
H2N	0.442 (7)	0.666 (3)	0.7742 (15)	0.0720*
H7O	1.502 (6)	1.085 (5)	0.9778 (16)	0.0900*
H16A	0.71020	0.59260	0.50210	0.1650*
H16B	0.83640	0.51320	0.53270	0.1650*
H16C	0.93570	0.66870	0.53430	0.1650*
H18	0.79890	0.52980	0.62520	0.0930*
H19	0.59380	0.56980	0.69550	0.0810*
H21	0.27220	0.77250	0.61850	0.0780*
H22	0.46980	0.72310	0.54720	0.1010*
H23A	0.55680	0.94080	0.78460	0.0560*
H23B	0.69050	0.86770	0.74380	0.0560*
H24	0.84850	0.78400	0.80520	0.0580*
H25A	0.57100	0.75300	0.86400	0.0840*
H25B	0.61330	0.90940	0.88010	0.0840*
H26A	0.80760	0.83020	0.94020	0.0810*
H26B	0.93570	0.76190	0.89670	0.0810*
H27	0.99440	1.04250	0.92290	0.0770*
H28A	1.23410	0.91740	0.84910	0.0880*
H28B	1.27560	1.07390	0.86490	0.0880*
H29A	0.91360	1.06450	0.83240	0.0720*
H29B	1.04130	0.99550	0.78900	0.0720*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.0430 (5)	0.0572 (6)	0.0446 (6)	0.0194 (5)	0.0095 (4)	0.0158 (4)
O1	0.0422 (15)	0.080 (2)	0.0572 (17)	0.0050 (14)	0.0078 (13)	0.0194 (14)
O2	0.079 (2)	0.0739 (19)	0.0602 (18)	0.0466 (17)	0.0198 (15)	0.0231 (15)
O3	0.116 (3)	0.082 (3)	0.093 (3)	0.018 (2)	0.046 (2)	−0.012 (2)
O4	0.123 (3)	0.091 (3)	0.099 (3)	0.019 (2)	0.064 (3)	−0.006 (2)
N1	0.0477 (19)	0.051 (2)	0.0425 (17)	0.0195 (15)	0.0098 (15)	0.0149 (15)
C1	0.113 (5)	0.165 (6)	0.072 (4)	0.063 (4)	−0.031 (3)	−0.006 (3)
C2	0.074 (3)	0.095 (4)	0.054 (3)	0.039 (3)	−0.010 (2)	−0.007 (3)
C3	0.051 (3)	0.151 (5)	0.067 (3)	0.010 (3)	−0.007 (2)	0.018 (3)
C4	0.053 (3)	0.123 (4)	0.064 (3)	0.002 (3)	0.004 (2)	0.038 (3)
C5	0.045 (2)	0.049 (2)	0.041 (2)	0.0157 (18)	0.0083 (17)	0.0083 (17)
C6	0.071 (3)	0.064 (3)	0.056 (3)	−0.001 (2)	0.000 (2)	0.023 (2)
C7	0.105 (4)	0.089 (4)	0.053 (3)	0.018 (3)	−0.006 (3)	0.025 (3)
C8	0.073 (3)	0.057 (3)	0.047 (2)	0.025 (2)	0.012 (2)	0.0202 (19)
C9	0.068 (3)	0.058 (3)	0.060 (3)	0.027 (2)	0.023 (2)	0.017 (2)
C10	0.071 (3)	0.086 (4)	0.105 (4)	0.014 (3)	0.026 (3)	−0.016 (3)
C11	0.111 (4)	0.061 (3)	0.098 (4)	0.014 (3)	0.064 (4)	0.004 (3)
C12	0.082 (4)	0.073 (3)	0.065 (3)	0.033 (3)	0.022 (3)	0.004 (2)

C13	0.090 (4)	0.067 (3)	0.093 (4)	0.027 (3)	0.030 (3)	−0.002 (3)
C14	0.119 (5)	0.059 (3)	0.086 (4)	0.032 (3)	0.050 (3)	0.017 (3)
C15	0.075 (3)	0.069 (3)	0.058 (3)	0.029 (3)	0.020 (3)	0.011 (3)
S2	0.0352 (5)	0.0511 (6)	0.0402 (5)	0.0129 (4)	0.0003 (4)	0.0085 (4)
O5	0.0653 (18)	0.0715 (19)	0.0554 (17)	0.0386 (15)	−0.0008 (14)	0.0119 (14)
O6	0.0385 (14)	0.0642 (18)	0.0570 (16)	−0.0050 (13)	−0.0009 (12)	0.0134 (13)
O7	0.080 (3)	0.092 (3)	0.093 (3)	0.000 (2)	−0.045 (2)	0.0209 (19)
O8	0.077 (2)	0.094 (2)	0.085 (2)	−0.0004 (19)	−0.0317 (18)	0.0339 (19)
N2	0.0432 (17)	0.0455 (19)	0.0356 (16)	0.0095 (15)	−0.0033 (13)	0.0088 (14)
C16	0.086 (4)	0.163 (6)	0.064 (3)	0.018 (4)	0.027 (3)	−0.007 (3)
C17	0.053 (3)	0.086 (3)	0.050 (3)	0.003 (2)	0.005 (2)	−0.003 (2)
C18	0.062 (3)	0.105 (4)	0.071 (3)	0.038 (3)	0.010 (3)	0.007 (3)
C19	0.072 (3)	0.094 (3)	0.053 (3)	0.046 (3)	0.017 (2)	0.021 (2)
C20	0.0376 (19)	0.048 (2)	0.040 (2)	0.0055 (17)	−0.0024 (16)	0.0070 (17)
C21	0.077 (3)	0.080 (3)	0.049 (3)	0.035 (3)	0.007 (2)	0.020 (2)
C22	0.102 (4)	0.114 (4)	0.044 (3)	0.036 (3)	0.012 (3)	0.022 (3)
C23	0.047 (2)	0.053 (2)	0.037 (2)	0.0067 (18)	−0.0012 (16)	0.0109 (17)
C24	0.043 (2)	0.056 (2)	0.045 (2)	0.0076 (18)	−0.0065 (17)	0.0111 (18)
C25	0.047 (2)	0.103 (4)	0.054 (3)	0.003 (2)	0.000 (2)	0.018 (2)
C26	0.054 (3)	0.094 (3)	0.046 (2)	−0.004 (2)	−0.009 (2)	0.026 (2)
C27	0.057 (3)	0.076 (3)	0.051 (3)	0.003 (2)	−0.015 (2)	0.009 (2)
C28	0.052 (3)	0.098 (4)	0.057 (3)	−0.007 (2)	−0.003 (2)	0.023 (2)
C29	0.048 (2)	0.072 (3)	0.052 (2)	−0.003 (2)	−0.0085 (19)	0.020 (2)
C30	0.054 (3)	0.073 (3)	0.046 (2)	0.005 (2)	−0.005 (2)	0.004 (2)

Geometric parameters (Å, º)

S1—O1	1.423 (3)	C10—H10B	0.9700
S1—O2	1.425 (3)	C10—H10A	0.9700
S1—N1	1.617 (3)	C11—H11A	0.9700
S1—C5	1.760 (4)	C11—H11B	0.9700
S2—N2	1.611 (3)	C12—H12	0.9800
S2—O5	1.422 (3)	C13—H13A	0.9700
S2—O6	1.434 (3)	C13—H13B	0.9700
S2—C20	1.771 (4)	C14—H14B	0.9700
O3—C15	1.234 (6)	C14—H14A	0.9700
O4—C15	1.251 (7)	C16—C17	1.513 (7)
O4—H4O	0.84 (4)	C17—C18	1.372 (7)
O7—C30	1.290 (6)	C17—C22	1.360 (8)
O8—C30	1.223 (6)	C18—C19	1.366 (7)
O7—H7O	0.81 (4)	C19—C20	1.364 (6)
N1—C8	1.465 (5)	C20—C21	1.379 (5)
N1—H1N	0.86 (3)	C21—C22	1.376 (7)
N2—C23	1.482 (5)	C23—C24	1.499 (5)
N2—H2N	0.84 (3)	C24—C29	1.517 (6)
C1—C2	1.510 (8)	C24—C25	1.487 (5)
C2—C3	1.370 (7)	C25—C26	1.542 (6)
C2—C7	1.351 (8)	C26—C27	1.518 (6)

C3—C4	1.373 (7)	C27—C28	1.482 (6)
C4—C5	1.366 (6)	C27—C30	1.506 (6)
C5—C6	1.365 (5)	C28—C29	1.528 (6)
C6—C7	1.381 (7)	C16—H16A	0.9600
C8—C9	1.509 (6)	C16—H16B	0.9600
C9—C14	1.509 (6)	C16—H16C	0.9600
C9—C10	1.468 (7)	C18—H18	0.9300
C10—C11	1.540 (7)	C19—H19	0.9300
C11—C12	1.527 (6)	C21—H21	0.9300
C12—C15	1.498 (7)	C22—H22	0.9300
C12—C13	1.459 (7)	C23—H23A	0.9700
C13—C14	1.537 (7)	C23—H23B	0.9700
C1—H1A	0.9600	C24—H24	0.9800
C1—H1B	0.9600	C25—H25A	0.9700
C1—H1C	0.9600	C25—H25B	0.9700
C3—H3	0.9300	C26—H26A	0.9700
C4—H4	0.9300	C26—H26B	0.9700
C6—H6	0.9300	C27—H27	0.9800
C7—H7	0.9300	C28—H28A	0.9700
C8—H8B	0.9700	C28—H28B	0.9700
C8—H8A	0.9700	C29—H29A	0.9700
C9—H9	0.9800	C29—H29B	0.9700

O1—S1—O2	119.56 (18)	C14—C13—H13A	110.00
O1—S1—N1	107.94 (16)	C12—C13—H13A	110.00
O1—S1—C5	107.47 (18)	C12—C13—H13B	110.00
O2—S1—N1	105.84 (17)	C14—C13—H13B	110.00
O2—S1—C5	107.52 (18)	H13A—C13—H13B	108.00
N1—S1—C5	108.07 (17)	C13—C14—H14B	109.00
N2—S2—C20	108.08 (17)	H14A—C14—H14B	108.00
O5—S2—N2	108.46 (16)	C9—C14—H14B	109.00
O5—S2—C20	107.58 (18)	C13—C14—H14A	109.00
O5—S2—O6	118.87 (18)	C9—C14—H14A	109.00
O6—S2—C20	107.80 (17)	C16—C17—C18	121.1 (5)
O6—S2—N2	105.65 (15)	C16—C17—C22	121.2 (4)
C15—O4—H4O	117 (2)	C18—C17—C22	117.7 (4)
C30—O7—H7O	109 (4)	C17—C18—C19	122.2 (5)
S1—N1—C8	118.0 (2)	C18—C19—C20	119.7 (4)
S1—N1—H1N	104 (3)	S2—C20—C19	121.1 (3)
C8—N1—H1N	118 (2)	S2—C20—C21	119.9 (3)
S2—N2—C23	118.2 (2)	C19—C20—C21	119.0 (4)
S2—N2—H2N	108 (3)	C20—C21—C22	120.3 (4)
C23—N2—H2N	119 (3)	C17—C22—C21	121.1 (4)
C3—C2—C7	118.0 (5)	N2—C23—C24	112.1 (3)
C1—C2—C3	120.6 (5)	C25—C24—C29	110.7 (3)
C1—C2—C7	121.4 (5)	C23—C24—C29	109.0 (3)
C2—C3—C4	121.2 (5)	C23—C24—C25	115.8 (3)
C3—C4—C5	120.1 (4)	C24—C25—C26	111.5 (4)

S1—C5—C6	120.5 (3)	C25—C26—C27	110.6 (4)
C4—C5—C6	119.3 (4)	C26—C27—C28	111.3 (3)
S1—C5—C4	120.3 (3)	C26—C27—C30	113.2 (4)
C5—C6—C7	119.7 (4)	C28—C27—C30	111.1 (4)
C2—C7—C6	121.8 (4)	C27—C28—C29	110.9 (4)
N1—C8—C9	113.0 (3)	C24—C29—C28	112.0 (3)
C10—C9—C14	111.0 (4)	O7—C30—O8	121.4 (4)
C8—C9—C14	109.4 (4)	O7—C30—C27	114.5 (4)
C8—C9—C10	115.1 (4)	O8—C30—C27	124.2 (4)
C9—C10—C11	113.3 (4)	C17—C16—H16A	110.00
C10—C11—C12	109.2 (4)	C17—C16—H16B	110.00
C13—C12—C15	113.7 (4)	C17—C16—H16C	109.00
C11—C12—C15	113.0 (4)	H16A—C16—H16B	109.00
C11—C12—C13	110.5 (5)	H16A—C16—H16C	109.00
C12—C13—C14	110.3 (4)	H16B—C16—H16C	109.00
C9—C14—C13	113.0 (4)	C17—C18—H18	119.00
O4—C15—C12	116.8 (4)	C19—C18—H18	119.00
O3—C15—O4	122.6 (5)	C18—C19—H19	120.00
O3—C15—C12	120.5 (5)	C20—C19—H19	120.00
C2—C1—H1C	109.00	C20—C21—H21	120.00
H1A—C1—H1B	110.00	C22—C21—H21	120.00
H1A—C1—H1C	110.00	C17—C22—H22	119.00
H1B—C1—H1C	109.00	C21—C22—H22	119.00
C2—C1—H1B	109.00	N2—C23—H23A	109.00
C2—C1—H1A	109.00	N2—C23—H23B	109.00
C4—C3—H3	119.00	C24—C23—H23A	109.00
C2—C3—H3	119.00	C24—C23—H23B	109.00
C3—C4—H4	120.00	H23A—C23—H23B	108.00
C5—C4—H4	120.00	C23—C24—H24	107.00
C7—C6—H6	120.00	C25—C24—H24	107.00
C5—C6—H6	120.00	C29—C24—H24	107.00
C2—C7—H7	119.00	C24—C25—H25A	109.00
C6—C7—H7	119.00	C24—C25—H25B	109.00
H8A—C8—H8B	108.00	C26—C25—H25A	109.00
N1—C8—H8B	109.00	C26—C25—H25B	109.00
C9—C8—H8B	109.00	H25A—C25—H25B	108.00
N1—C8—H8A	109.00	C25—C26—H26A	109.00
C9—C8—H8A	109.00	C25—C26—H26B	110.00
C10—C9—H9	107.00	C27—C26—H26A	110.00
C14—C9—H9	107.00	C27—C26—H26B	110.00
C8—C9—H9	107.00	H26A—C26—H26B	108.00
C9—C10—H10A	109.00	C26—C27—H27	107.00
C9—C10—H10B	109.00	C28—C27—H27	107.00
C11—C10—H10A	109.00	C30—C27—H27	107.00
C11—C10—H10B	109.00	C27—C28—H28A	109.00
H10A—C10—H10B	108.00	C27—C28—H28B	109.00
C10—C11—H11B	110.00	C29—C28—H28A	109.00
C12—C11—H11B	110.00	C29—C28—H28B	110.00

C12—C11—H11A	110.00	H28A—C28—H28B	108.00
C10—C11—H11A	110.00	C24—C29—H29A	109.00
H11A—C11—H11B	108.00	C24—C29—H29B	109.00
C13—C12—H12	106.00	C28—C29—H29A	109.00
C15—C12—H12	106.00	C28—C29—H29B	109.00
C11—C12—H12	106.00	H29A—C29—H29B	108.00

O1—S1—N1—C8	−50.3 (3)	C9—C10—C11—C12	−55.7 (6)
O2—S1—N1—C8	−179.4 (3)	C10—C11—C12—C13	58.8 (5)
C5—S1—N1—C8	65.7 (3)	C10—C11—C12—C15	−172.5 (4)
O1—S1—C5—C4	168.2 (3)	C11—C12—C15—O3	−147.3 (5)
O1—S1—C5—C6	−12.5 (4)	C11—C12—C15—O4	35.0 (7)
O2—S1—C5—C4	−61.9 (4)	C13—C12—C15—O4	162.0 (5)
O2—S1—C5—C6	117.5 (4)	C15—C12—C13—C14	172.9 (4)
N1—S1—C5—C4	51.9 (4)	C13—C12—C15—O3	−20.3 (7)
N1—S1—C5—C6	−128.7 (3)	C11—C12—C13—C14	−58.8 (5)
O5—S2—C20—C21	−25.3 (4)	C12—C13—C14—C9	55.4 (6)
O6—S2—C20—C19	−75.4 (4)	C16—C17—C18—C19	179.5 (5)
O6—S2—C20—C21	104.0 (4)	C22—C17—C18—C19	−1.1 (8)
N2—S2—C20—C19	38.4 (4)	C16—C17—C22—C21	−178.1 (5)
N2—S2—C20—C21	−142.3 (3)	C18—C17—C22—C21	2.5 (8)
O5—S2—N2—C23	−50.6 (3)	C17—C18—C19—C20	−0.7 (8)
O6—S2—N2—C23	−179.1 (3)	C18—C19—C20—S2	−179.7 (4)
C20—S2—N2—C23	65.7 (3)	C18—C19—C20—C21	1.0 (7)
O5—S2—C20—C19	155.3 (4)	S2—C20—C21—C22	−179.0 (4)
S1—N1—C8—C9	172.9 (3)	C19—C20—C21—C22	0.4 (7)
S2—N2—C23—C24	−166.7 (2)	C20—C21—C22—C17	−2.2 (7)
C1—C2—C3—C4	179.9 (5)	N2—C23—C24—C25	−54.0 (5)
C7—C2—C3—C4	0.6 (8)	N2—C23—C24—C29	−179.6 (3)
C3—C2—C7—C6	−0.2 (8)	C23—C24—C25—C26	−179.7 (4)
C1—C2—C7—C6	−179.4 (5)	C29—C24—C25—C26	−55.0 (5)
C2—C3—C4—C5	−0.5 (8)	C23—C24—C29—C28	−176.6 (3)
C3—C4—C5—S1	179.4 (4)	C25—C24—C29—C28	55.0 (4)
C3—C4—C5—C6	0.0 (7)	C24—C25—C26—C27	55.8 (5)
S1—C5—C6—C7	−178.9 (4)	C25—C26—C27—C28	−56.1 (5)
C4—C5—C6—C7	0.4 (7)	C25—C26—C27—C30	177.9 (3)
C5—C6—C7—C2	−0.4 (8)	C26—C27—C28—C29	56.0 (5)
N1—C8—C9—C10	65.8 (5)	C30—C27—C28—C29	−176.9 (4)
N1—C8—C9—C14	−168.5 (4)	C26—C27—C30—O7	−173.0 (4)
C14—C9—C10—C11	52.0 (5)	C26—C27—C30—O8	7.0 (6)
C8—C9—C14—C13	−179.3 (4)	C28—C27—C30—O7	60.9 (5)
C8—C9—C10—C11	176.9 (4)	C28—C27—C30—O8	−119.1 (5)
C10—C9—C14—C13	−51.3 (6)	C27—C28—C29—C24	−55.5 (5)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O4—H4*O*···O3^{i}	0.84 (4)	1.81 (4)	2.638 (6)	171 (4)

O7—H7*O*···O8^{ii}	0.81 (4)	1.87 (4)	2.664 (5)	166 (5)
N1—H1*N*···O6^{iii}	0.86 (3)	2.15 (3)	3.001 (4)	171 (4)
N2—H2*N*···O2	0.84 (3)	2.09 (4)	2.924 (4)	171 (4)
C10—H10*B*···O6^{iii}	0.97	2.57	3.446 (6)	151
C19—H19···O2	0.93	2.59	3.490 (5)	164

Symmetry codes: (i) $-x+3, -y, -z+1$; (ii) $-x+3, -y+2, -z+2$; (iii) $x+1, y, z$.