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L-Glutaminium 4-methylbenzenesulfonate

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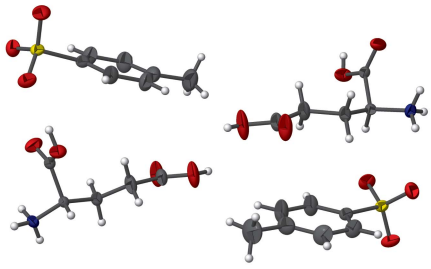
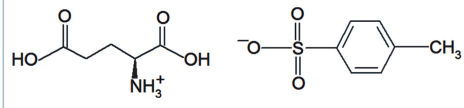
Structural data: full structural data are available from iucrdata.iucr.org

The asymmetric unit of the title salt, $C_5H_{10}NO_4^+\cdot C_7H_7O_3S^-$, contains two glutaminium cations and two 4-methylbenzenesulfonate anions. The crystal packing features weak intermolecular C—H···O, O—H···O and N—H···O hydrogen bonds.

3D view



Chemical scheme



Structure description

Glutamic acid is a proteinogenic and non-essential amino acid which acts as a neurotransmitter and is essential in the pathology of neurological and psychiatric disorders (Haxhiu *et al.*, 2000; Liu *et al.*, 2016). Glutamate (L-Glu) is the main excitatory transmitter within the mammalian central nervous system (Tamborini *et al.*, 2016). L-Glutamate plays an important role in chronic neurodegenerative and acute diseases such as cerebral ischemia, traumatic brain injury, spinal injury and epilepsy (Hynd *et al.*, 2004).

The asymmetric unit of the title compound comprises two glutaminium cations and two 4-methylbenzenesulfonate anions (Fig. 1). The geometric parameters of the title compound agree well with those reported for a similar structure (Thayanithi *et al.*, 2016). The crystal packing is controlled by weak intermolecular C—H···O, O—H···O and N—H···O hydrogen bonds (Table 1, Fig. 2), forming a three-dimensional structure.

Synthesis and crystallization

The title compound was synthesized by dissolving L-Glutamic acid (0.1 mmol) and *p*-toluene sulfonic acid monohydrate (0.1 mmol) in 30 ml of deionized water. The solution was stirred for 4 h, filtered into a beaker and kept dust free. Colourless block-shaped crystals were obtained from the mother solution in 93% yield.

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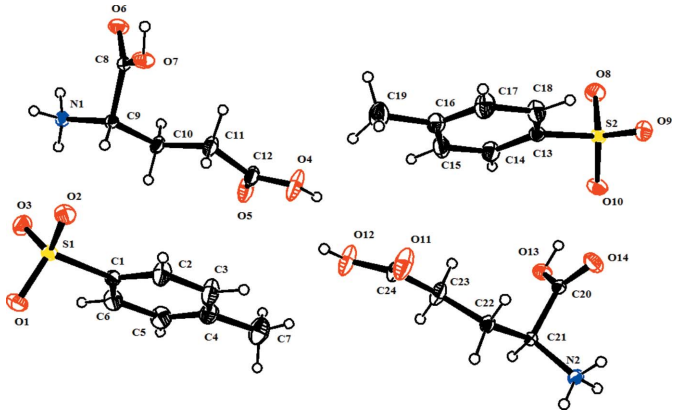

Figure 1
The asymmetric unit of the title compound, showing atom labels and 30% probability displacement ellipsoids for non-H atoms.

Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2.

Acknowledgements

The authors acknowledge the SAIF, IIT Madras, Chennai.

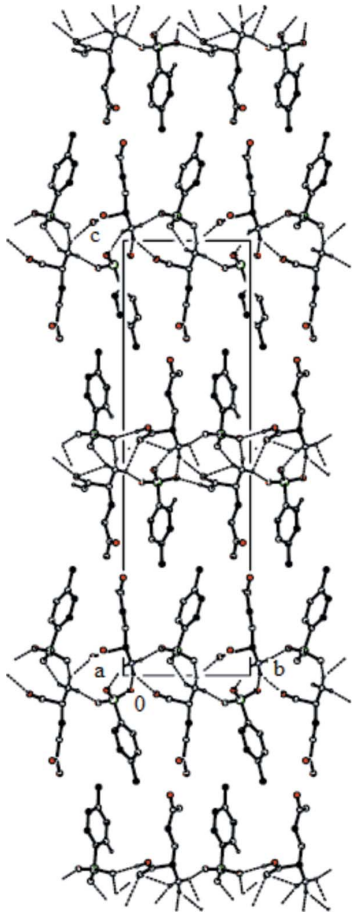

Figure 2
The packing of the title compound, viewed along the *a* axis. Hydrogen bonds are shown as dashed lines.

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1*C*···O6	0.86 (1)	2.46 (3)	2.7527 (17)	100 (2)
C2—H2···O2	0.93	2.49	2.864 (2)	104
C18—H18···O9	0.93	2.53	2.904 (2)	104
N1—H1*B*···O2^{i}	0.87 (2)	2.01 (2)	2.8301 (16)	157 (2)
N2—H2*B*···O8^{ii}	0.86 (1)	2.00 (1)	2.8524 (17)	170 (2)
O7—H7···O1^{iii}	0.82 (1)	1.84 (1)	2.6592 (16)	176 (3)
C6—H6···O6^{iv}	0.93	2.55	3.316 (2)	140
N2—H2*C*···O14	0.86 (1)	2.50 (2)	2.7803 (18)	100 (2)
N1—H1*C*···O3^{v}	0.86 (1)	2.07 (1)	2.8969 (18)	160 (2)
N2—H2*C*···O9^{vi}	0.86 (1)	2.18 (2)	2.9369 (18)	147 (2)
N1—H1*A*···O1^{vii}	0.88 (2)	2.05 (2)	2.9150 (16)	167 (2)
N2—H2*A*···O10^{viii}	0.87 (1)	2.11 (2)	2.8947 (17)	150 (2)

Symmetry codes: (i) $x-1, y, z$; (ii) $x+1, y+1, z$; (iii) $x, y-1, z$; (iv) $x, y+1, z$; (v) $-x, y-\frac{1}{2}, -z+1$; (vi) $-x+2, y+\frac{1}{2}, -z+2$; (vii) $-x+1, y-\frac{1}{2}, -z+1$; (viii) $-x+1, y+\frac{1}{2}, -z+2$.

Table 2
Experimental details.

Crystal data	
Chemical formula	$C_5H_{10}NO_4^+\cdot C_7H_7O_3S^-$
M_r	319.33
Crystal system, space group	Monoclinic, $P2_1$
Temperature (K)	295
a, b, c (Å)	5.4540 (3), 8.8013 (5), 30.2297 (18)
β (°)	92.835 (1)
V (Å^3)	1449.32 (14)
Z	4
Radiation type	Mo $K\alpha$
μ (mm^{-1})	0.26
Crystal size (mm)	0.25 × 0.20 × 0.20
Data collection	
Diffractometer	Bruker APEXII CCD
Absorption correction	Multi-scan (*SADABS*; Sheldrick, 1996)
T_{min}, T_{max}	0.956, 0.975
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	22234, 9991, 8841
R_{int}	0.019
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.769
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.036, 0.092, 1.03
No. of reflections	9991
No. of parameters	414
No. of restraints	9
H-atom treatment	H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.35, −0.29
Absolute structure	Flack (1983), 4563 Friedel pairs
Absolute structure parameter	0.01 (4)

Computer programs: *APEX2* (Bruker, 2008), *SAINT* (Bruker, 2008), *SHELXS97* (Sheldrick, 2008), *SHELXL97* (Sheldrick, 2008), *PLATON* (Spek, 2009).

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full crystallographic data

IUCrData (2017). **2**, x170162 [https://doi.org/10.1107/S2414314617001626]

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L-Glutaminium 4-methylbenzenesulfonate

Crystal data

$C_5H_{10}NO_4^+·C_7H_7O_3S^-$
M_r = 319.33
Monoclinic, $P2_1$
Hall symbol: P 2yb
a = 5.4540 (3) Å
b = 8.8013 (5) Å
c = 30.2297 (18) Å
β = 92.835 (1)°
V = 1449.32 (14) Å^3
Z = 4
$F(000)$ = 672
D_x = 1.463 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 5436 reflections
θ = 0.7–33.1°
μ = 0.26 mm^{-1}
T = 295 K
Block, colourless
0.25 × 0.20 × 0.20 mm

Data collection

Bruker APEXII CCD diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
Detector resolution: 0 pixels mm^{-1}
ω and φ scans
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)
T_{min} = 0.956, T_{max} = 0.975
22234 measured reflections
9991 independent reflections
8841 reflections with $I > 2\sigma(I)$
R_{int} = 0.019
θ_{max} = 33.1°, θ_{min} = 0.7°
h = −8→8
k = −13→13
l = −44→45

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.036
$wR(F^2)$ = 0.092
S = 1.03
9991 reflections
414 parameters
9 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
$w = 1/[\sigma^2(F_o^2) + (0.0469P)^2 + 0.186P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.003
$\Delta\rho_{max}$ = 0.35 e Å^{-3}
$\Delta\rho_{min}$ = −0.29 e Å^{-3}
Absolute structure: Flack (1983), 4563 Friedel pairs
Absolute structure parameter: 0.01 (4)

Special details

Refinement. H atoms were positioned geometrically and refined using riding model with C—H = 0.97 Å and U_{iso}(H) = 1.2Ueq(C) for CH_2, C—H = 0.96 Å and U_{iso}(H) = 1.5Ueq(C) for CH_3, C—H = 0.98 Å and U_{iso}(H) = 1.2Ueq(C) for C—H, C—H = 0.93 Å and U_{iso}(H) = 1.2Ueq(C) for aromatic C—H.
One hydroxyl H atom was freely refined with the O—H distance restrained to 0.82 (1) Å, the remaining three using a riding model with O—H = 0.82 Å and U_{iso}(H) = 1.5Ueq(O). The displacement parameter of one riding H atom was refined.
H atoms bonded to N were freely refined with the N—H distances restrained to 0.86 (2) Å.

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

	x	*y*	*z*	U_{iso}*/U_{eq}
S1	0.51194 (6)	0.76994 (3)	0.554874 (11)	0.02313 (7)
O1	0.6281 (2)	0.91527 (13)	0.54506 (4)	0.0359 (3)
O2	0.6862 (2)	0.64604 (13)	0.55531 (4)	0.0373 (3)
O3	0.2927 (2)	0.74121 (15)	0.52729 (4)	0.0388 (3)
O6	0.1221 (2)	0.13029 (13)	0.54256 (4)	0.0357 (3)
O7	0.4978 (2)	0.19277 (13)	0.56959 (5)	0.0347 (2)
O4	0.2597 (3)	0.3680 (3)	0.72528 (4)	0.0665 (5)
H4	0.1858	0.3789	0.7480	0.100*
O5	−0.0963 (3)	0.4398 (2)	0.69465 (4)	0.0541 (4)
N1	0.0337 (2)	0.43370 (15)	0.52645 (4)	0.0244 (2)
C10	0.0652 (3)	0.40515 (18)	0.60781 (5)	0.0281 (3)
H10A	0.0238	0.5114	0.6117	0.034*
H10B	−0.0868	0.3478	0.6057	0.034*
C9	0.1981 (2)	0.38670 (14)	0.56476 (4)	0.0199 (2)
H9	0.3463	0.4499	0.5659	0.024*
C1	0.4162 (3)	0.78387 (17)	0.60965 (5)	0.0250 (3)
C8	0.2681 (3)	0.22182 (15)	0.55723 (4)	0.0217 (2)
C4	0.2677 (4)	0.7993 (2)	0.69645 (6)	0.0445 (4)
C2	0.5542 (3)	0.7157 (2)	0.64356 (6)	0.0426 (4)
H2	0.6975	0.6640	0.6376	0.051*
C12	0.1120 (3)	0.3917 (2)	0.69141 (5)	0.0369 (4)
C5	0.1322 (4)	0.8685 (2)	0.66220 (7)	0.0459 (4)
H5	−0.0092	0.9218	0.6685	0.055*
C6	0.2021 (3)	0.8605 (2)	0.61848 (6)	0.0370 (4)
H6	0.1070	0.9056	0.5957	0.044*
C11	0.2190 (3)	0.3513 (2)	0.64802 (5)	0.0370 (4)
H11A	0.2368	0.2418	0.6464	0.044*
H11B	0.3815	0.3956	0.6472	0.044*
C3	0.4782 (4)	0.7246 (3)	0.68669 (6)	0.0544 (5)
H3	0.5726	0.6788	0.7094	0.065*
C7	0.1889 (6)	0.8037 (4)	0.74348 (7)	0.0708 (7)
H7A	0.3014	0.7453	0.7620	0.106*
H7B	0.0270	0.7616	0.7447	0.106*
H7C	0.1879	0.9070	0.7537	0.106*
H7	0.532 (5)	0.1074 (16)	0.5614 (9)	0.069 (9)*
H1C	−0.074 (4)	0.368 (2)	0.5168 (8)	0.056 (7)*

H1B	−0.048 (4)	0.516 (2)	0.5314 (7)	0.040 (6)*
H1A	0.120 (3)	0.438 (3)	0.5027 (6)	0.043 (6)*
S2	0.47952 (6)	−0.07804 (4)	0.944471 (11)	0.02329 (7)
O13	0.4998 (2)	0.34191 (13)	0.94161 (4)	0.0325 (2)
H13	0.4630	0.2548	0.9483	0.060 (8)*
O9	0.7071 (2)	−0.13081 (14)	0.96616 (4)	0.0345 (2)
O8	0.2987 (2)	−0.19844 (14)	0.93747 (5)	0.0408 (3)
O14	0.8813 (2)	0.26035 (14)	0.95742 (5)	0.0424 (3)
O10	0.3770 (2)	0.05506 (13)	0.96595 (4)	0.0355 (3)
N2	1.0071 (2)	0.56332 (15)	0.97136 (4)	0.0251 (2)
O11	1.0828 (3)	0.4383 (3)	0.80447 (4)	0.0597 (4)
O12	0.7164 (3)	0.4973 (2)	0.77510 (5)	0.0599 (5)
H12	0.7879	0.4869	0.7521	0.115 (14)*
C21	0.8273 (3)	0.51769 (15)	0.93531 (4)	0.0205 (2)
H21	0.6884	0.5885	0.9340	0.025*
C13	0.5501 (3)	−0.01743 (16)	0.89089 (5)	0.0256 (3)
C20	0.7386 (3)	0.35883 (16)	0.94638 (5)	0.0241 (3)
C22	0.9485 (3)	0.51528 (17)	0.89077 (5)	0.0269 (3)
H22A	1.0358	0.6100	0.8869	0.032*
H22B	1.0670	0.4331	0.8906	0.032*
C23	0.7601 (3)	0.4941 (2)	0.85277 (5)	0.0395 (4)
H23A	0.6608	0.4058	0.8588	0.047*
H23B	0.6523	0.5818	0.8514	0.047*
C24	0.8682 (3)	0.4742 (2)	0.80852 (5)	0.0370 (4)
C14	0.3910 (3)	0.0751 (2)	0.86642 (6)	0.0368 (3)
H14	0.2494	0.1112	0.8788	0.044*
C18	0.7653 (3)	−0.0665 (2)	0.87305 (6)	0.0380 (3)
H18	0.8775	−0.1248	0.8899	0.046*
C16	0.6499 (4)	0.0616 (2)	0.80408 (6)	0.0404 (4)
C15	0.4420 (4)	0.1144 (2)	0.82343 (6)	0.0438 (4)
H15	0.3341	0.1776	0.8073	0.053*
C17	0.8114 (4)	−0.0275 (3)	0.82973 (6)	0.0470 (5)
H17	0.9545	−0.0621	0.8176	0.056*
C19	0.6998 (5)	0.1001 (3)	0.75667 (7)	0.0622 (6)
H19A	0.6648	0.0133	0.7382	0.093*
H19B	0.8691	0.1281	0.7548	0.093*
H19C	0.5972	0.1835	0.7469	0.093*
H2C	1.114 (3)	0.496 (2)	0.9792 (8)	0.051 (7)*
H2B	1.101 (4)	0.637 (2)	0.9645 (8)	0.058 (7)*
H2A	0.937 (4)	0.576 (3)	0.9961 (5)	0.054 (7)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.02595 (16)	0.01870 (14)	0.02521 (16)	0.00330 (12)	0.00599 (12)	−0.00040 (12)
O1	0.0419 (6)	0.0222 (5)	0.0456 (7)	−0.0002 (5)	0.0214 (5)	0.0001 (5)
O2	0.0428 (6)	0.0295 (6)	0.0406 (6)	0.0157 (5)	0.0118 (5)	−0.0005 (5)
O3	0.0410 (6)	0.0429 (7)	0.0316 (6)	0.0009 (5)	−0.0061 (5)	−0.0024 (5)

O6	0.0406 (6)	0.0229 (5)	0.0423 (6)	−0.0007 (5)	−0.0114 (5)	−0.0041 (5)
O7	0.0291 (5)	0.0253 (5)	0.0493 (7)	0.0082 (4)	−0.0030 (5)	−0.0074 (5)
O4	0.0642 (10)	0.1150 (15)	0.0196 (6)	0.0341 (10)	−0.0059 (6)	−0.0049 (7)
O5	0.0538 (8)	0.0846 (11)	0.0241 (6)	0.0257 (8)	0.0052 (5)	0.0033 (7)
N1	0.0299 (6)	0.0234 (5)	0.0197 (5)	0.0069 (5)	0.0005 (4)	0.0019 (5)
C10	0.0332 (7)	0.0324 (7)	0.0186 (6)	0.0081 (6)	0.0019 (5)	−0.0023 (5)
C9	0.0228 (6)	0.0180 (5)	0.0189 (5)	0.0016 (4)	0.0000 (5)	−0.0013 (4)
C1	0.0276 (6)	0.0226 (6)	0.0254 (6)	0.0007 (5)	0.0066 (5)	−0.0004 (5)
C8	0.0293 (6)	0.0183 (5)	0.0175 (5)	0.0026 (5)	0.0011 (5)	0.0003 (4)
C4	0.0560 (11)	0.0462 (10)	0.0325 (8)	−0.0029 (8)	0.0146 (8)	−0.0039 (7)
C2	0.0388 (9)	0.0578 (11)	0.0312 (8)	0.0176 (8)	0.0029 (7)	0.0016 (8)
C12	0.0464 (9)	0.0440 (9)	0.0199 (6)	0.0086 (7)	−0.0010 (6)	0.0000 (6)
C5	0.0456 (10)	0.0503 (10)	0.0438 (10)	0.0121 (8)	0.0215 (8)	−0.0010 (8)
C6	0.0329 (8)	0.0409 (9)	0.0379 (9)	0.0101 (6)	0.0096 (7)	0.0044 (7)
C11	0.0439 (9)	0.0463 (9)	0.0205 (7)	0.0143 (7)	−0.0005 (6)	−0.0018 (6)
C3	0.0629 (13)	0.0717 (14)	0.0284 (8)	0.0182 (11)	0.0001 (8)	0.0041 (9)
C7	0.0957 (19)	0.0847 (19)	0.0346 (10)	−0.0002 (16)	0.0273 (12)	−0.0037 (12)
S2	0.02485 (15)	0.01980 (15)	0.02554 (16)	−0.00294 (12)	0.00467 (12)	0.00029 (12)
O13	0.0313 (5)	0.0265 (5)	0.0398 (6)	−0.0075 (4)	0.0030 (5)	0.0005 (5)
O9	0.0326 (6)	0.0406 (6)	0.0301 (6)	0.0033 (5)	−0.0009 (5)	0.0021 (5)
O8	0.0439 (7)	0.0346 (6)	0.0439 (7)	−0.0192 (5)	0.0029 (6)	0.0038 (5)
O14	0.0424 (7)	0.0277 (5)	0.0557 (8)	−0.0020 (5)	−0.0122 (6)	0.0121 (6)
O10	0.0439 (6)	0.0264 (5)	0.0379 (6)	0.0024 (5)	0.0199 (5)	−0.0007 (5)
N2	0.0313 (6)	0.0243 (5)	0.0194 (5)	−0.0073 (5)	−0.0003 (5)	−0.0022 (4)
O11	0.0579 (9)	0.0969 (13)	0.0246 (6)	0.0214 (9)	0.0033 (6)	−0.0027 (8)
O12	0.0609 (9)	0.0978 (13)	0.0202 (6)	0.0163 (9)	−0.0061 (6)	−0.0025 (7)
C21	0.0261 (6)	0.0183 (5)	0.0167 (5)	−0.0018 (4)	−0.0015 (5)	−0.0003 (4)
C13	0.0288 (7)	0.0227 (6)	0.0256 (7)	−0.0025 (5)	0.0039 (5)	−0.0006 (5)
C20	0.0326 (7)	0.0217 (6)	0.0178 (6)	−0.0042 (5)	−0.0008 (5)	0.0002 (5)
C22	0.0328 (7)	0.0296 (7)	0.0183 (6)	−0.0035 (5)	0.0022 (5)	0.0009 (5)
C23	0.0399 (9)	0.0594 (11)	0.0190 (7)	−0.0061 (8)	−0.0003 (6)	−0.0016 (7)
C24	0.0485 (10)	0.0425 (9)	0.0195 (6)	0.0005 (7)	−0.0018 (7)	−0.0011 (6)
C14	0.0343 (8)	0.0421 (9)	0.0342 (8)	0.0102 (7)	0.0023 (6)	0.0011 (7)
C18	0.0365 (8)	0.0452 (9)	0.0329 (8)	0.0109 (7)	0.0082 (6)	0.0064 (7)
C16	0.0540 (10)	0.0374 (8)	0.0302 (8)	−0.0039 (8)	0.0057 (7)	0.0024 (7)
C15	0.0520 (10)	0.0450 (10)	0.0337 (8)	0.0105 (8)	−0.0030 (8)	0.0086 (8)
C17	0.0456 (10)	0.0625 (12)	0.0339 (9)	0.0077 (9)	0.0132 (8)	0.0030 (9)
C19	0.0857 (17)	0.0677 (15)	0.0342 (10)	0.0071 (13)	0.0117 (10)	0.0102 (10)

Geometric parameters (Å, º)

S1—O3	1.4460 (12)	S2—O8	1.4558 (12)
S1—O2	1.4461 (11)	S2—O10	1.4637 (12)
S1—O1	1.4642 (12)	S2—C13	1.7657 (15)
S1—C1	1.7650 (14)	O13—C20	1.3115 (18)
O6—C8	1.2021 (18)	O13—H13	0.8200
O7—C8	1.3144 (18)	O14—O14	0.000 (4)
O7—H7	0.816 (10)	O14—C20	1.2012 (19)

O4—C12	1.288 (2)	N2—C21	1.4849 (18)
O4—H4	0.8200	N2—H2C	0.855 (9)
O5—C12	1.221 (2)	N2—H2B	0.858 (7)
N1—C9	1.4877 (17)	N2—H2A	0.866 (10)
N1—H1C	0.863 (10)	O11—C24	1.224 (2)
N1—H1B	0.867 (15)	O12—C24	1.290 (2)
N1—H1A	0.878 (15)	O12—H12	0.8200
C10—C11	1.518 (2)	C21—C20	1.5221 (19)
C10—C9	1.5295 (19)	C21—C22	1.5293 (19)
C10—H10A	0.9700	C21—H21	0.9800
C10—H10B	0.9700	C13—C14	1.378 (2)
C9—C8	1.5205 (18)	C13—C18	1.385 (2)
C9—H9	0.9800	C20—O14	1.2012 (19)
C1—C2	1.379 (2)	C22—C23	1.514 (2)
C1—C6	1.385 (2)	C22—H22A	0.9700
C4—C3	1.368 (3)	C22—H22B	0.9700
C4—C5	1.383 (3)	C23—C24	1.499 (2)
C4—C7	1.506 (3)	C23—H23A	0.9700
C2—C3	1.390 (3)	C23—H23B	0.9700
C2—H2	0.9300	C14—C15	1.386 (2)
C12—C11	1.504 (2)	C14—H14	0.9300
C5—C6	1.395 (3)	C18—C17	1.389 (2)
C5—H5	0.9300	C18—H18	0.9300
C6—H6	0.9300	C16—C15	1.382 (3)
C11—H11A	0.9700	C16—C17	1.387 (3)
C11—H11B	0.9700	C16—C19	1.510 (3)
C3—H3	0.9300	C15—H15	0.9300
C7—H7A	0.9600	C17—H17	0.9300
C7—H7B	0.9600	C19—H19A	0.9600
C7—H7C	0.9600	C19—H19B	0.9600
S2—O9	1.4511 (12)	C19—H19C	0.9600

O3—S1—O2	113.44 (8)	O8—S2—O10	112.10 (7)
O3—S1—O1	112.93 (8)	O9—S2—C13	106.90 (7)
O2—S1—O1	111.71 (7)	O8—S2—C13	105.28 (7)
O3—S1—C1	106.17 (7)	O10—S2—C13	105.70 (7)
O2—S1—C1	105.59 (7)	C20—O13—H13	109.5
O1—S1—C1	106.28 (7)	O14—O14—C20	0 (10)
C8—O7—H7	108.7 (19)	C21—N2—H2C	115.5 (17)
C12—O4—H4	109.5	C21—N2—H2B	114.2 (16)
C9—N1—H1C	117.0 (18)	H2C—N2—H2B	100 (2)
C9—N1—H1B	113.1 (15)	C21—N2—H2A	111.4 (16)
H1C—N1—H1B	106 (2)	H2C—N2—H2A	100 (2)
C9—N1—H1A	108.7 (14)	H2B—N2—H2A	114 (2)
H1C—N1—H1A	98 (2)	C24—O12—H12	109.5
H1B—N1—H1A	114 (2)	N2—C21—C20	107.03 (11)
C11—C10—C9	112.44 (12)	N2—C21—C22	110.56 (11)
C11—C10—H10A	109.1	C20—C21—C22	109.67 (11)

C9—C10—H10A	109.1	N2—C21—H21	109.8
C11—C10—H10B	109.1	C20—C21—H21	109.8
C9—C10—H10B	109.1	C22—C21—H21	109.8
H10A—C10—H10B	107.8	C14—C13—C18	119.89 (15)
N1—C9—C8	107.16 (11)	C14—C13—S2	120.71 (12)
N1—C9—C10	109.87 (11)	C18—C13—S2	119.38 (12)
C8—C9—C10	111.21 (11)	O14—C20—O14	0.00 (15)
N1—C9—H9	109.5	O14—C20—O13	125.09 (14)
C8—C9—H9	109.5	O14—C20—O13	125.09 (14)
C10—C9—H9	109.5	O14—C20—C21	121.01 (14)
C2—C1—C6	120.18 (15)	O14—C20—C21	121.01 (14)
C2—C1—S1	119.42 (12)	O13—C20—C21	113.88 (12)
C6—C1—S1	120.39 (12)	C23—C22—C21	111.28 (13)
O6—C8—O7	125.40 (13)	C23—C22—H22A	109.4
O6—C8—C9	121.88 (13)	C21—C22—H22A	109.4
O7—C8—C9	112.70 (12)	C23—C22—H22B	109.4
C3—C4—C5	118.25 (17)	C21—C22—H22B	109.4
C3—C4—C7	119.8 (2)	H22A—C22—H22B	108.0
C5—C4—C7	121.9 (2)	C24—C23—C22	114.17 (15)
C1—C2—C3	119.73 (16)	C24—C23—H23A	108.7
C1—C2—H2	120.1	C22—C23—H23A	108.7
C3—C2—H2	120.1	C24—C23—H23B	108.7
O5—C12—O4	122.72 (16)	C22—C23—H23B	108.7
O5—C12—C11	123.67 (15)	H23A—C23—H23B	107.6
O4—C12—C11	113.60 (15)	O11—C24—O12	122.82 (17)
C4—C5—C6	121.72 (17)	O11—C24—C23	122.67 (15)
C4—C5—H5	119.1	O12—C24—C23	114.51 (16)
C6—C5—H5	119.1	C13—C14—C15	120.04 (16)
C1—C6—C5	118.65 (17)	C13—C14—H14	120.0
C1—C6—H6	120.7	C15—C14—H14	120.0
C5—C6—H6	120.7	C13—C18—C17	119.12 (16)
C12—C11—C10	113.67 (13)	C13—C18—H18	120.4
C12—C11—H11A	108.8	C17—C18—H18	120.4
C10—C11—H11A	108.8	C15—C16—C17	117.71 (17)
C12—C11—H11B	108.8	C15—C16—C19	121.06 (19)
C10—C11—H11B	108.8	C17—C16—C19	121.23 (19)
H11A—C11—H11B	107.7	C16—C15—C14	121.30 (17)
C4—C3—C2	121.45 (18)	C16—C15—H15	119.4
C4—C3—H3	119.3	C14—C15—H15	119.4
C2—C3—H3	119.3	C16—C17—C18	121.84 (18)
C4—C7—H7A	109.5	C16—C17—H17	119.1
C4—C7—H7B	109.5	C18—C17—H17	119.1
H7A—C7—H7B	109.5	C16—C19—H19A	109.5
C4—C7—H7C	109.5	C16—C19—H19B	109.5
H7A—C7—H7C	109.5	H19A—C19—H19B	109.5
H7B—C7—H7C	109.5	C16—C19—H19C	109.5
O9—S2—O8	113.01 (8)	H19A—C19—H19C	109.5
O9—S2—O10	113.11 (8)	H19B—C19—H19C	109.5

C11—C10—C9—N1	177.06 (13)	O10—S2—C13—C14	−41.80 (16)
C11—C10—C9—C8	58.59 (17)	O9—S2—C13—C18	18.89 (16)
O3—S1—C1—C2	137.98 (15)	O8—S2—C13—C18	−101.54 (15)
O2—S1—C1—C2	17.26 (16)	O10—S2—C13—C18	139.65 (14)
O1—S1—C1—C2	−101.53 (15)	O14—O14—C20—O13	0.0 (6)
O3—S1—C1—C6	−40.99 (15)	O14—O14—C20—C21	0.0 (6)
O2—S1—C1—C6	−161.71 (14)	N2—C21—C20—O14	−47.36 (18)
O1—S1—C1—C6	79.50 (15)	C22—C21—C20—O14	72.62 (18)
N1—C9—C8—O6	−40.23 (18)	N2—C21—C20—O14	−47.36 (18)
C10—C9—C8—O6	79.86 (17)	C22—C21—C20—O14	72.62 (18)
N1—C9—C8—O7	141.59 (12)	N2—C21—C20—O13	134.14 (13)
C10—C9—C8—O7	−98.32 (14)	C22—C21—C20—O13	−105.88 (14)
C6—C1—C2—C3	−0.2 (3)	N2—C21—C22—C23	−170.78 (13)
S1—C1—C2—C3	−179.20 (17)	C20—C21—C22—C23	71.43 (16)
C3—C4—C5—C6	1.6 (3)	C21—C22—C23—C24	−173.86 (14)
C7—C4—C5—C6	−178.1 (2)	C22—C23—C24—O11	18.2 (3)
C2—C1—C6—C5	0.8 (3)	C22—C23—C24—O12	−162.05 (18)
S1—C1—C6—C5	179.78 (15)	C18—C13—C14—C15	2.4 (3)
C4—C5—C6—C1	−1.5 (3)	S2—C13—C14—C15	−176.17 (15)
O5—C12—C11—C10	11.8 (3)	C14—C13—C18—C17	−3.2 (3)
O4—C12—C11—C10	−169.55 (18)	S2—C13—C18—C17	175.38 (16)
C9—C10—C11—C12	170.29 (14)	C17—C16—C15—C14	−2.5 (3)
C5—C4—C3—C2	−0.9 (4)	C19—C16—C15—C14	177.8 (2)
C7—C4—C3—C2	178.8 (2)	C13—C14—C15—C16	0.5 (3)
C1—C2—C3—C4	0.3 (4)	C15—C16—C17—C18	1.6 (3)
O9—S2—C13—C14	−162.57 (14)	C19—C16—C17—C18	−178.6 (2)
O8—S2—C13—C14	77.01 (15)	C13—C18—C17—C16	1.2 (3)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1*C*···O6	0.86 (1)	2.46 (3)	2.7527 (17)	100 (2)
C2—H2···O2	0.93	2.49	2.864 (2)	104
C18—H18···O9	0.93	2.53	2.904 (2)	104
N1—H1*B*···O2^{i}	0.87 (2)	2.01 (2)	2.8301 (16)	157 (2)
N2—H2*B*···O8^{ii}	0.86 (1)	2.00 (1)	2.8524 (17)	170 (2)
O7—H7···O1^{iii}	0.82 (1)	1.84 (1)	2.6592 (16)	176 (3)
C6—H6···O6^{iv}	0.93	2.55	3.316 (2)	140
N2—H2*C*···O14	0.86 (1)	2.50 (2)	2.7803 (18)	100 (2)
N1—H1*C*···O3^{v}	0.86 (1)	2.07 (1)	2.8969 (18)	160 (2)
N2—H2*C*···O9^{vi}	0.86 (1)	2.18 (2)	2.9369 (18)	147 (2)
N1—H1*A*···O1^{vii}	0.88 (2)	2.05 (2)	2.9150 (16)	167 (2)
N2—H2*A*···O10^{viii}	0.87 (1)	2.11 (2)	2.8947 (17)	150 (2)

Symmetry codes: (i) *x*−1, *y*, *z*; (ii) *x*+1, *y*+1, *z*; (iii) *x*, *y*−1, *z*; (iv) *x*, *y*+1, *z*; (v) −*x*, *y*−1/2, −*z*+1; (vi) −*x*+2, *y*+1/2, −*z*+2; (vii) −*x*+1, *y*−1/2, −*z*+1; (viii) −*x*+1, *y*+1/2, −*z*+2.