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Dodecan-1-aminium sulfate trihydrate

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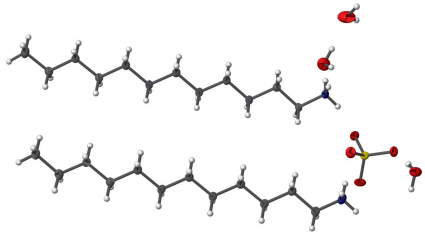
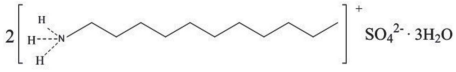
The asymmetric unit of the title salt, $2C_{12}H_{28}N^{+}\cdot SO_4^{2-}\cdot 3H_2O$, contains two *n*-dodecylammonium cations, one sulfate anion and three water molecules. In the crystal, N—H···O hydrogen bonds link the cations and anions into layers parallel to (100). These layers are further connected through O—H···O hydrogen-bonding interactions involving the sulfate ions and the isolated water molecules. The three-dimensional structure can also be considered as the superposition of thin inorganic layers of SO_4^{2-} anions and thick layers of alkylammonium cations perpendicular to the *c* axis.

3D view



Chemical scheme



Structure description

As a part of our studies on new thermal-energy storage materials (Terreros *et al.*, 2000; Li *et al.*, 1999) involving long-chain *n*-alkylammonium halides or sulfates (Zhang *et al.*, 2011*a*,*b*; Dan *et al.*, 2010), we report the synthesis and crystal structure of the title compound dodecan-1-aminium sulfate (trihydrate). *n*-Dodecylamine is a white waxy solid, which is often used in the manufacture of ore-flotation agents, surfactants, fungicides and emulsifiers (Aratono *et al.*, 1998; Törnblom *et al.*, 2000).

The asymmetric unit of the title salt contains two *n*-dodecylammonium cations, one sulfate anion and three water molecules (Figs. 1 and 2). In the crystal, N1—H1*A*···O3, N1—H1*C*···O4^{ii}, N2—H2*D*···O2 and N2—H2*E*···O1^{iv} hydrogen bonds (Table 1) link the cations and anions into layers parallel to (100). These layers are further connected through hydrogen-bonding interactions involving the sulfate or the alkylammonium ions with the isolated water molecules: O5—H5*C*···O6, O5—H5*D*···O3^{i}, O6—H6*C*···O1^{ii}, O6—H6*D*···O2^{iii}, O7—H7*C*···O1^{iv}, O7—H7*D*···O4, N1—H1*B*···O5 and N2—H2*C*···O7^{v} (Table 1). The three-dimensional structure can also be considered as the superposition of thin inorganic layers of SO_4^{2-} anions and thick layers of alkylammonium cations perpendicular to the *c* axis (Fig. 3).

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

D—H···A	D—H	H···A	D···A	D—H···A
O5—H5C···O6	0.87	1.87	2.736 (2)	176
O5—H5D···O3^{i}	0.87	1.92	2.7729 (19)	166
O6—H6C···O1^{ii}	0.87	2.02	2.849 (2)	158
O6—H6D···O2^{iii}	0.87	1.93	2.787 (2)	166
O7—H7C···O1^{iv}	0.87	1.99	2.8542 (19)	171
O7—H7D···O4	0.87	2.00	2.8665 (19)	173
N1—H1A···O3	0.91	1.97	2.8331 (19)	158
N1—H1B···O5	0.91	1.91	2.775 (2)	158
N1—H1C···O4^{ii}	0.91	2.00	2.8947 (19)	167
N2—H2C···O7^{v}	0.91	1.99	2.879 (2)	163
N2—H2D···O2	0.91	1.83	2.740 (2)	175
N2—H2E···O1^{iv}	0.91	2.11	2.9731 (19)	159

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

Synthesis and crystallization

The title compound was synthesized by reacting dodecylamine and sulfuric acid (molar ratio 2:1) in anhydrous ethanol. Slow evaporation of the solvent yielded good-quality colourless crystals over a period of about 4–8 weeks.

Refinement

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

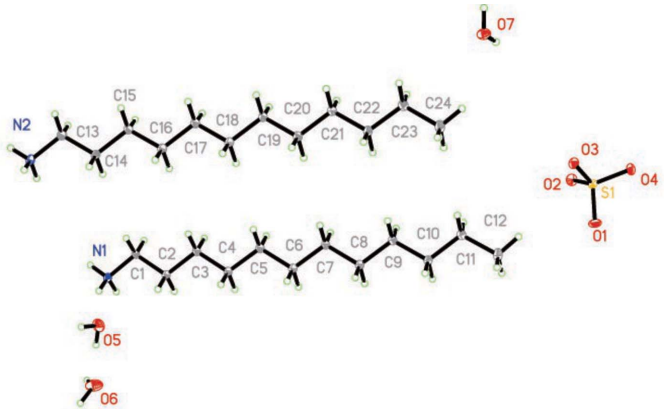


Figure 1
A view of the asymmetric unit of the title compound showing the atom numbering with 30% displacement ellipsoids.

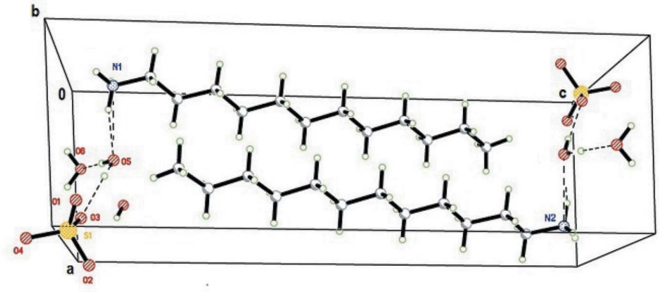


Figure 2
The expanded asymmetric unit with displacement ellipsoids drawn at the 30% probability level. Hydrogen bonds are shown as dashed lines.

Table 2
Experimental details.

Crystal data	
Chemical formula	$2C_{12}H_{28}N^{+}\cdot SO_4^{2-}\cdot 3H_2O$
M_r	522.81
Crystal system, space group	Triclinic, $P\overline{1}$
Temperature (K)	150
a, b, c (Å)	6.9477 (4), 10.8782 (7), 20.8769 (11)
α, β, γ (°)	89.110 (3), 87.052 (3), 78.876 (3)
V (Å^3)	1546.13 (16)
Z	2
Radiation type	Mo $K\alpha$
μ (mm^{-1})	0.14
Crystal size (mm)	0.05 × 0.04 × 0.03
Data collection	
Diffractometer	Bruker APEXII CCD
Absorption correction	Multi-scan (*SADABS*; Bruker, 2016)
T_{min}, T_{max}	0.682, 0.745
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	37596, 6095, 4414
R_{int}	0.074
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.617
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.047, 0.125, 1.02
No. of reflections	6095
No. of parameters	317
H-atom treatment	H-atom parameters constrained
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.42, −0.40

Computer programs: *SAINT* and *APEX2* (Bruker, 2016), *SHELXT* (Sheldrick, 2015*a*), *SHELXL* (Sheldrick, 2015*b*) and *OLEX2* (Dolomanov *et al.*, 2009).

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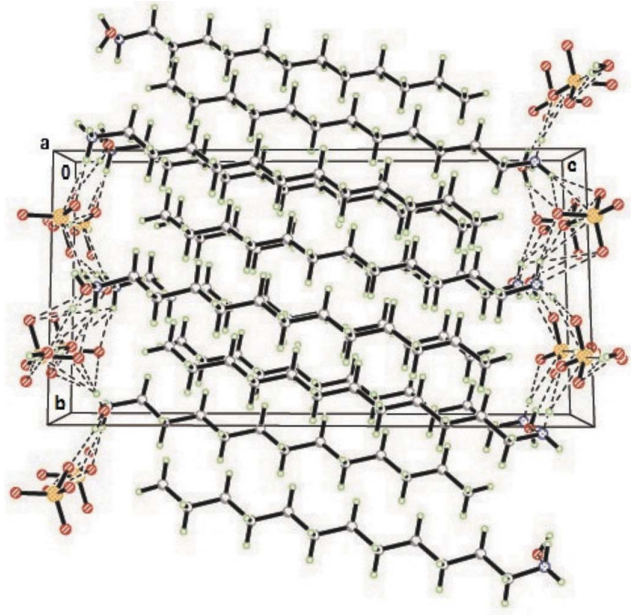

Figure 3
A packing diagram of the title compound, showing the hydrogen-bonded network (dashed lines).

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full crystallographic data

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Crystal data

$2C_{12}H_{28}N^{+}\cdot SO_4^{2-}\cdot 3H_2O$
M_r = 522.81
Triclinic, $P\overline{1}$
Hall symbol: -P 1
a = 6.9477 (4) Å
b = 10.8782 (7) Å
c = 20.8769 (11) Å
α = 89.110 (3)°
β = 87.052 (3)°
γ = 78.876 (3)°
V = 1546.13 (16) Å^3
Z = 2
$F(000)$ = 584
D_x = 1.123 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 8618 reflections
θ = 3.0–26.4°
μ = 0.14 mm^{-1}
T = 150 K
Block, colourless
0.05 × 0.04 × 0.03 mm

Data collection

Bruker APEXII CCD diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
Detector resolution: 10 pixels mm^{-1}
φ and ω scans
Absorption correction: multi-scan (SADABS; Bruker, 2016)
T_{min} = 0.682, T_{max} = 0.745
37596 measured reflections
6095 independent reflections
4414 reflections with $I > 2\sigma(I)$
R_{int} = 0.074
θ_{max} = 26.0°, θ_{min} = 2.1°
h = −8→8
k = −13→13
l = −25→25

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.047
$wR(F^2)$ = 0.125
S = 1.02
6095 reflections
317 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: mixed
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.063P)^2 + 0.384P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.42 e Å^{-3}
$\Delta\rho_{min}$ = −0.39 e Å^{-3}

Special details

Experimental. SADABS-2016/2 (Bruker, 2016) was used for absorption correction. wR2 was 0.0783 before and 0.0645 after correction. The ratio of minimum to maximum transmission is 0.915. The $\lambda/2$ correction factor is not present.′

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

Refinement. All H atoms were placed in geometrically calculated positions and constrained to ride on their parent atoms: with methylene C—H distances of 0.99 Å, methyl C—H of 0.98 Å, N—H of 0.87 Å and O–H distances of 0.87 Å, and with U_{iso}(H) values set at 1.2U_{eq}(C) for the methylene H atoms and at 1.5U_{eq}(*X*) for all other H atoms.

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

	x	*y*	*z*	U_{iso}*/U_{eq}
S1	−0.03120 (7)	0.24575 (4)	0.01517 (2)	0.01780 (13)
O1	−0.1649 (2)	0.16044 (12)	0.03328 (7)	0.0316 (3)
O2	0.15208 (19)	0.20951 (12)	0.04928 (6)	0.0271 (3)
O3	−0.12134 (19)	0.37531 (11)	0.03220 (6)	0.0229 (3)
O4	0.01304 (19)	0.23874 (11)	−0.05480 (6)	0.0237 (3)
O5	0.5349 (2)	0.51199 (14)	0.08846 (7)	0.0376 (4)
H5C	0.534570	0.579486	0.065517	0.056*
H5D	0.634794	0.458389	0.072630	0.056*
O6	0.5266 (2)	0.73077 (14)	0.02131 (8)	0.0489 (5)
H6C	0.425733	0.747683	−0.002589	0.073*
H6D	0.626213	0.738523	−0.004389	0.073*
O7	0.3276 (2)	0.03745 (12)	−0.09235 (7)	0.0285 (3)
H7C	0.278255	−0.026217	−0.078413	0.043*
H7D	0.239991	0.102613	−0.080573	0.043*
N1	0.1375 (2)	0.50460 (14)	0.09193 (6)	0.0214 (4)
H1A	0.082017	0.458202	0.064882	0.026*
H1B	0.269318	0.491922	0.082900	0.026*
H1C	0.084489	0.587208	0.086756	0.026*
C1	0.0992 (3)	0.46625 (17)	0.15913 (8)	0.0209 (4)
H1D	0.156437	0.376298	0.164712	0.025*
H1E	−0.044199	0.477890	0.168588	0.025*
C2	0.1877 (3)	0.54284 (17)	0.20554 (8)	0.0214 (4)
H2A	0.134073	0.632919	0.198629	0.026*
H2B	0.331674	0.528916	0.196774	0.026*
C3	0.1447 (3)	0.50849 (17)	0.27521 (8)	0.0215 (4)
H3A	0.000686	0.524069	0.284053	0.026*
H3B	0.195469	0.417920	0.281685	0.026*
C4	0.2360 (3)	0.58231 (17)	0.32263 (8)	0.0215 (4)
H4A	0.380637	0.562749	0.315424	0.026*
H4B	0.191563	0.672943	0.314333	0.026*
C5	0.1839 (3)	0.55424 (17)	0.39228 (8)	0.0217 (4)
H5A	0.229109	0.463675	0.400568	0.026*
H5B	0.039183	0.573130	0.399313	0.026*
C6	0.2736 (3)	0.62850 (17)	0.44023 (8)	0.0219 (4)

H6A	0.418443	0.607857	0.434110	0.026*
H6B	0.231178	0.719078	0.431322	0.026*
C7	0.2159 (3)	0.60210 (17)	0.50965 (8)	0.0208 (4)
H7A	0.258812	0.511548	0.518502	0.025*
H7B	0.070950	0.622202	0.515589	0.025*
C8	0.3039 (3)	0.67640 (17)	0.55819 (8)	0.0220 (4)
H8A	0.448837	0.656295	0.552409	0.026*
H8B	0.260877	0.767008	0.549494	0.026*
C9	0.2448 (3)	0.64894 (17)	0.62740 (8)	0.0218 (4)
H9A	0.288222	0.558317	0.635941	0.026*
H9B	0.099720	0.668408	0.632895	0.026*
C10	0.3299 (3)	0.72254 (17)	0.67674 (8)	0.0223 (4)
H10A	0.286415	0.813227	0.668366	0.027*
H10B	0.474997	0.703053	0.671430	0.027*
C11	0.2691 (3)	0.69388 (18)	0.74583 (8)	0.0260 (4)
H11A	0.123958	0.712363	0.750940	0.031*
H11B	0.313908	0.603334	0.754292	0.031*
C12	0.3515 (4)	0.7679 (2)	0.79521 (9)	0.0360 (5)
H12A	0.305746	0.857738	0.787763	0.054*
H12B	0.495274	0.748233	0.791433	0.054*
H12C	0.306512	0.745588	0.838333	0.054*
N2	0.2600 (2)	−0.03715 (14)	0.08399 (7)	0.0221 (4)
H2C	0.393411	−0.053548	0.084313	0.027*
H2D	0.217462	0.043994	0.072111	0.027*
H2E	0.220465	−0.088786	0.055626	0.027*
C13	0.1763 (3)	−0.05851 (18)	0.14901 (8)	0.0231 (4)
H13A	0.219300	−0.147372	0.161465	0.028*
H13B	0.031088	−0.040656	0.148715	0.028*
C14	0.2422 (3)	0.02499 (17)	0.19741 (8)	0.0213 (4)
H14A	0.192600	0.113701	0.185958	0.026*
H14B	0.387506	0.010803	0.195329	0.026*
C15	0.1698 (3)	0.00091 (17)	0.26579 (8)	0.0220 (4)
H15A	0.024442	0.021141	0.268585	0.026*
H15B	0.211010	−0.089188	0.276069	0.026*
C16	0.2479 (3)	0.07780 (17)	0.31513 (8)	0.0225 (4)
H16A	0.202145	0.167848	0.305645	0.027*
H16B	0.393187	0.060243	0.310807	0.027*
C17	0.1846 (3)	0.05170 (17)	0.38432 (8)	0.0220 (4)
H17A	0.039491	0.072834	0.389251	0.026*
H17B	0.225501	−0.038924	0.393387	0.026*
C18	0.2707 (3)	0.12524 (17)	0.43316 (8)	0.0224 (4)
H18A	0.228132	0.215853	0.424385	0.027*
H18B	0.415702	0.105083	0.427677	0.027*
C19	0.2107 (3)	0.09841 (17)	0.50257 (8)	0.0215 (4)
H19A	0.252310	0.007721	0.511340	0.026*
H19B	0.065765	0.119373	0.508256	0.026*
C20	0.2996 (3)	0.17204 (17)	0.55121 (8)	0.0227 (4)
H20A	0.444542	0.151211	0.545428	0.027*

H20B	0.257801	0.262723	0.542459	0.027*
C21	0.2403 (3)	0.14526 (17)	0.62060 (8)	0.0223 (4)
H21A	0.095308	0.164852	0.626256	0.027*
H21B	0.284056	0.054829	0.629599	0.027*
C22	0.3268 (3)	0.22042 (17)	0.66884 (8)	0.0213 (4)
H22A	0.284280	0.310777	0.659406	0.026*
H22B	0.471817	0.200144	0.663348	0.026*
C23	0.2670 (3)	0.19559 (18)	0.73848 (8)	0.0245 (4)
H23A	0.121890	0.213651	0.743856	0.029*
H23B	0.313123	0.105798	0.748468	0.029*
C24	0.3499 (3)	0.27414 (19)	0.78567 (9)	0.0299 (5)
H24A	0.306372	0.255006	0.829455	0.045*
H24B	0.303168	0.363214	0.776483	0.045*
H24C	0.493712	0.254871	0.781516	0.045*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.0174 (2)	0.0170 (2)	0.0192 (2)	−0.00339 (18)	−0.00217 (17)	−0.00098 (16)
O1	0.0330 (9)	0.0295 (8)	0.0378 (8)	−0.0194 (7)	−0.0034 (6)	0.0052 (6)
O2	0.0213 (8)	0.0305 (8)	0.0285 (7)	−0.0006 (6)	−0.0089 (6)	0.0029 (6)
O3	0.0251 (8)	0.0178 (7)	0.0238 (7)	0.0007 (6)	−0.0004 (5)	−0.0041 (5)
O4	0.0284 (8)	0.0227 (7)	0.0177 (7)	0.0011 (6)	−0.0007 (5)	−0.0024 (5)
O5	0.0304 (9)	0.0377 (9)	0.0411 (9)	−0.0008 (7)	0.0118 (7)	−0.0052 (7)
O6	0.0251 (9)	0.0417 (10)	0.0810 (13)	−0.0096 (8)	−0.0027 (8)	0.0064 (9)
O7	0.0242 (8)	0.0230 (7)	0.0377 (8)	−0.0050 (6)	0.0050 (6)	−0.0011 (6)
N1	0.0291 (9)	0.0206 (8)	0.0155 (8)	−0.0070 (7)	−0.0032 (6)	−0.0005 (6)
C1	0.0274 (11)	0.0235 (10)	0.0134 (8)	−0.0090 (8)	−0.0013 (7)	0.0012 (7)
C2	0.0242 (11)	0.0232 (10)	0.0180 (9)	−0.0078 (8)	−0.0007 (7)	−0.0016 (7)
C3	0.0254 (11)	0.0249 (10)	0.0157 (9)	−0.0086 (8)	−0.0014 (7)	−0.0008 (7)
C4	0.0247 (11)	0.0252 (10)	0.0160 (9)	−0.0079 (8)	−0.0027 (7)	−0.0005 (7)
C5	0.0241 (11)	0.0254 (10)	0.0170 (9)	−0.0078 (8)	−0.0036 (8)	−0.0005 (8)
C6	0.0250 (11)	0.0239 (10)	0.0182 (9)	−0.0074 (8)	−0.0021 (8)	−0.0011 (8)
C7	0.0244 (11)	0.0239 (10)	0.0154 (9)	−0.0076 (8)	−0.0024 (7)	−0.0004 (7)
C8	0.0250 (11)	0.0247 (10)	0.0173 (9)	−0.0071 (8)	−0.0017 (8)	−0.0007 (7)
C9	0.0251 (11)	0.0243 (10)	0.0174 (9)	−0.0081 (8)	−0.0005 (8)	−0.0001 (7)
C10	0.0277 (11)	0.0237 (10)	0.0176 (9)	−0.0097 (8)	−0.0020 (8)	−0.0002 (7)
C11	0.0345 (12)	0.0271 (10)	0.0187 (9)	−0.0114 (9)	−0.0015 (8)	0.0003 (8)
C12	0.0514 (15)	0.0404 (13)	0.0200 (10)	−0.0166 (11)	−0.0056 (10)	−0.0037 (9)
N2	0.0227 (9)	0.0231 (8)	0.0201 (8)	−0.0019 (7)	−0.0054 (6)	−0.0052 (6)
C13	0.0242 (11)	0.0267 (10)	0.0201 (9)	−0.0087 (8)	−0.0034 (8)	−0.0009 (8)
C14	0.0239 (11)	0.0221 (10)	0.0193 (9)	−0.0076 (8)	−0.0031 (8)	−0.0015 (7)
C15	0.0242 (11)	0.0236 (10)	0.0199 (9)	−0.0083 (8)	−0.0011 (8)	−0.0017 (8)
C16	0.0257 (11)	0.0245 (10)	0.0192 (9)	−0.0092 (8)	−0.0018 (8)	−0.0007 (8)
C17	0.0249 (11)	0.0247 (10)	0.0181 (9)	−0.0083 (8)	−0.0020 (8)	−0.0010 (8)
C18	0.0259 (11)	0.0235 (10)	0.0190 (9)	−0.0074 (8)	−0.0017 (8)	−0.0010 (8)
C19	0.0248 (11)	0.0233 (10)	0.0179 (9)	−0.0081 (8)	−0.0016 (8)	−0.0033 (7)
C20	0.0242 (11)	0.0263 (10)	0.0188 (9)	−0.0079 (9)	−0.0015 (8)	−0.0015 (8)

C21	0.0261 (11)	0.0225 (10)	0.0191 (9)	−0.0069 (8)	−0.0011 (8)	−0.0005 (8)
C22	0.0243 (11)	0.0221 (10)	0.0178 (9)	−0.0048 (8)	−0.0021 (8)	−0.0014 (7)
C23	0.0295 (11)	0.0234 (10)	0.0207 (10)	−0.0050 (9)	−0.0017 (8)	0.0006 (8)
C24	0.0393 (13)	0.0311 (11)	0.0199 (10)	−0.0072 (10)	−0.0058 (9)	−0.0007 (8)

Geometric parameters (Å, º)

S1—O1	1.4672 (14)	C11—H11B	0.9900
S1—O2	1.4747 (13)	C11—C12	1.518 (3)
S1—O3	1.4687 (12)	C12—H12A	0.9800
S1—O4	1.4773 (13)	C12—H12B	0.9800
O5—H5C	0.8701	C12—H12C	0.9800
O5—H5D	0.8700	N2—H2C	0.9100
O6—H6C	0.8702	N2—H2D	0.9100
O6—H6D	0.8700	N2—H2E	0.9100
O7—H7C	0.8702	N2—C13	1.482 (2)
O7—H7D	0.8702	C13—H13A	0.9900
N1—H1A	0.9100	C13—H13B	0.9900
N1—H1B	0.9100	C13—C14	1.514 (2)
N1—H1C	0.9100	C14—H14A	0.9900
N1—C1	1.483 (2)	C14—H14B	0.9900
C1—H1D	0.9900	C14—C15	1.525 (2)
C1—H1E	0.9900	C15—H15A	0.9900
C1—C2	1.515 (2)	C15—H15B	0.9900
C2—H2A	0.9900	C15—C16	1.522 (2)
C2—H2B	0.9900	C16—H16A	0.9900
C2—C3	1.525 (2)	C16—H16B	0.9900
C3—H3A	0.9900	C16—C17	1.527 (2)
C3—H3B	0.9900	C17—H17A	0.9900
C3—C4	1.521 (2)	C17—H17B	0.9900
C4—H4A	0.9900	C17—C18	1.520 (2)
C4—H4B	0.9900	C18—H18A	0.9900
C4—C5	1.521 (2)	C18—H18B	0.9900
C5—H5A	0.9900	C18—C19	1.527 (2)
C5—H5B	0.9900	C19—H19A	0.9900
C5—C6	1.526 (2)	C19—H19B	0.9900
C6—H6A	0.9900	C19—C20	1.528 (2)
C6—H6B	0.9900	C20—H20A	0.9900
C6—C7	1.522 (2)	C20—H20B	0.9900
C7—H7A	0.9900	C20—C21	1.525 (2)
C7—H7B	0.9900	C21—H21A	0.9900
C7—C8	1.528 (2)	C21—H21B	0.9900
C8—H8A	0.9900	C21—C22	1.524 (2)
C8—H8B	0.9900	C22—H22A	0.9900
C8—C9	1.523 (2)	C22—H22B	0.9900
C9—H9A	0.9900	C22—C23	1.526 (2)
C9—H9B	0.9900	C23—H23A	0.9900
C9—C10	1.524 (2)	C23—H23B	0.9900

C10—H10A	0.9900	C23—C24	1.519 (2)
C10—H10B	0.9900	C24—H24A	0.9800
C10—C11	1.527 (2)	C24—H24B	0.9800
C11—H11A	0.9900	C24—H24C	0.9800
O1—S1—O2	109.64 (8)	C11—C12—H12C	109.5
O1—S1—O3	110.60 (8)	H12A—C12—H12B	109.5
O1—S1—O4	109.18 (8)	H12A—C12—H12C	109.5
O2—S1—O4	109.78 (8)	H12B—C12—H12C	109.5
O3—S1—O2	108.64 (7)	H2C—N2—H2D	109.5
O3—S1—O4	108.99 (7)	H2C—N2—H2E	109.5
H5C—O5—H5D	104.5	H2D—N2—H2E	109.5
H6C—O6—H6D	104.5	C13—N2—H2C	109.5
H7C—O7—H7D	104.4	C13—N2—H2D	109.5
H1A—N1—H1B	109.5	C13—N2—H2E	109.5
H1A—N1—H1C	109.5	N2—C13—H13A	109.5
H1B—N1—H1C	109.5	N2—C13—H13B	109.5
C1—N1—H1A	109.5	N2—C13—C14	110.58 (15)
C1—N1—H1B	109.5	H13A—C13—H13B	108.1
C1—N1—H1C	109.5	C14—C13—H13A	109.5
N1—C1—H1D	109.5	C14—C13—H13B	109.5
N1—C1—H1E	109.5	C13—C14—H14A	109.1
N1—C1—C2	110.84 (14)	C13—C14—H14B	109.1
H1D—C1—H1E	108.1	C13—C14—C15	112.56 (15)
C2—C1—H1D	109.5	H14A—C14—H14B	107.8
C2—C1—H1E	109.5	C15—C14—H14A	109.1
C1—C2—H2A	109.2	C15—C14—H14B	109.1
C1—C2—H2B	109.2	C14—C15—H15A	109.0
C1—C2—C3	112.01 (15)	C14—C15—H15B	109.0
H2A—C2—H2B	107.9	H15A—C15—H15B	107.8
C3—C2—H2A	109.2	C16—C15—C14	112.75 (15)
C3—C2—H2B	109.2	C16—C15—H15A	109.0
C2—C3—H3A	109.0	C16—C15—H15B	109.0
C2—C3—H3B	109.0	C15—C16—H16A	108.8
H3A—C3—H3B	107.8	C15—C16—H16B	108.8
C4—C3—C2	112.89 (15)	C15—C16—C17	113.92 (15)
C4—C3—H3A	109.0	H16A—C16—H16B	107.7
C4—C3—H3B	109.0	C17—C16—H16A	108.8
C3—C4—H4A	108.9	C17—C16—H16B	108.8
C3—C4—H4B	108.9	C16—C17—H17A	108.9
H4A—C4—H4B	107.7	C16—C17—H17B	108.9
C5—C4—C3	113.23 (15)	H17A—C17—H17B	107.7
C5—C4—H4A	108.9	C18—C17—C16	113.26 (15)
C5—C4—H4B	108.9	C18—C17—H17A	108.9
C4—C5—H5A	108.8	C18—C17—H17B	108.9
C4—C5—H5B	108.8	C17—C18—H18A	108.8
C4—C5—C6	113.65 (15)	C17—C18—H18B	108.8
H5A—C5—H5B	107.7	C17—C18—C19	113.71 (15)

C6—C5—H5A	108.8	H18A—C18—H18B	107.7
C6—C5—H5B	108.8	C19—C18—H18A	108.8
C5—C6—H6A	109.0	C19—C18—H18B	108.8
C5—C6—H6B	109.0	C18—C19—H19A	108.9
H6A—C6—H6B	107.8	C18—C19—H19B	108.9
C7—C6—C5	113.12 (15)	C18—C19—C20	113.20 (15)
C7—C6—H6A	109.0	H19A—C19—H19B	107.8
C7—C6—H6B	109.0	C20—C19—H19A	108.9
C6—C7—H7A	108.8	C20—C19—H19B	108.9
C6—C7—H7B	108.8	C19—C20—H20A	108.9
C6—C7—C8	113.67 (15)	C19—C20—H20B	108.9
H7A—C7—H7B	107.7	H20A—C20—H20B	107.7
C8—C7—H7A	108.8	C21—C20—C19	113.33 (15)
C8—C7—H7B	108.8	C21—C20—H20A	108.9
C7—C8—H8A	109.0	C21—C20—H20B	108.9
C7—C8—H8B	109.0	C20—C21—H21A	109.0
H8A—C8—H8B	107.8	C20—C21—H21B	109.0
C9—C8—C7	113.03 (15)	H21A—C21—H21B	107.8
C9—C8—H8A	109.0	C22—C21—C20	113.11 (15)
C9—C8—H8B	109.0	C22—C21—H21A	109.0
C8—C9—H9A	108.8	C22—C21—H21B	109.0
C8—C9—H9B	108.8	C21—C22—H22A	108.8
C8—C9—C10	114.01 (15)	C21—C22—H22B	108.8
H9A—C9—H9B	107.6	C21—C22—C23	113.70 (15)
C10—C9—H9A	108.8	H22A—C22—H22B	107.7
C10—C9—H9B	108.8	C23—C22—H22A	108.8
C9—C10—H10A	108.9	C23—C22—H22B	108.8
C9—C10—H10B	108.9	C22—C23—H23A	109.0
C9—C10—C11	113.29 (15)	C22—C23—H23B	109.0
H10A—C10—H10B	107.7	H23A—C23—H23B	107.8
C11—C10—H10A	108.9	C24—C23—C22	112.87 (16)
C11—C10—H10B	108.9	C24—C23—H23A	109.0
C10—C11—H11A	108.8	C24—C23—H23B	109.0
C10—C11—H11B	108.8	C23—C24—H24A	109.5
H11A—C11—H11B	107.7	C23—C24—H24B	109.5
C12—C11—C10	113.59 (16)	C23—C24—H24C	109.5
C12—C11—H11A	108.8	H24A—C24—H24B	109.5
C12—C11—H11B	108.8	H24A—C24—H24C	109.5
C11—C12—H12A	109.5	H24B—C24—H24C	109.5
C11—C12—H12B	109.5		

N1—C1—C2—C3	178.04 (15)	N2—C13—C14—C15	−176.57 (15)
C1—C2—C3—C4	178.74 (16)	C13—C14—C15—C16	175.83 (16)
C2—C3—C4—C5	176.60 (16)	C14—C15—C16—C17	−177.64 (16)
C3—C4—C5—C6	−179.59 (16)	C15—C16—C17—C18	177.61 (16)
C4—C5—C6—C7	178.48 (16)	C16—C17—C18—C19	−179.15 (16)
C5—C6—C7—C8	−179.69 (16)	C17—C18—C19—C20	179.50 (16)
C6—C7—C8—C9	180.00 (16)	C18—C19—C20—C21	−179.89 (15)

C7—C8—C9—C10	−179.68 (16)	C19—C20—C21—C22	−179.11 (16)
C8—C9—C10—C11	179.98 (16)	C20—C21—C22—C23	179.40 (16)
C9—C10—C11—C12	−179.34 (18)	C21—C22—C23—C24	−178.35 (16)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O5—H5*C*···O6	0.87	1.87	2.736 (2)	176
O5—H5*D*···O3^{i}	0.87	1.92	2.7729 (19)	166
O6—H6*C*···O1^{ii}	0.87	2.02	2.849 (2)	158
O6—H6*D*···O2^{iii}	0.87	1.93	2.787 (2)	166
O7—H7*C*···O1^{iv}	0.87	1.99	2.8542 (19)	171
O7—H7*D*···O4	0.87	2.00	2.8665 (19)	173
N1—H1*A*···O3	0.91	1.97	2.8331 (19)	158
N1—H1*B*···O5	0.91	1.91	2.775 (2)	158
N1—H1*C*···O4^{ii}	0.91	2.00	2.8947 (19)	167
N2—H2*C*···O7^{v}	0.91	1.99	2.879 (2)	163
N2—H2*D*···O2	0.91	1.83	2.740 (2)	175
N2—H2*E*···O1^{iv}	0.91	2.11	2.9731 (19)	159

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