

1 Salt forms of sulfadiazine with alkali metal and organic cations.

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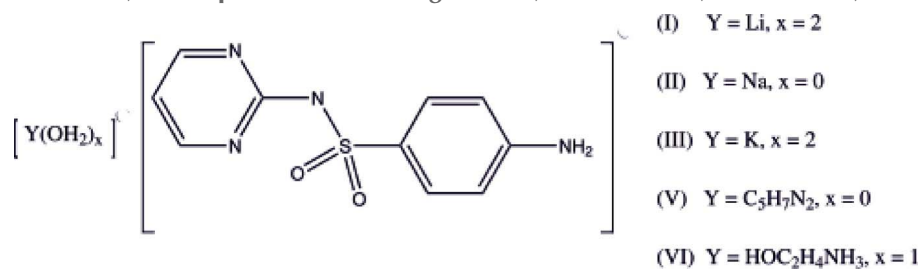
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5 Abstract

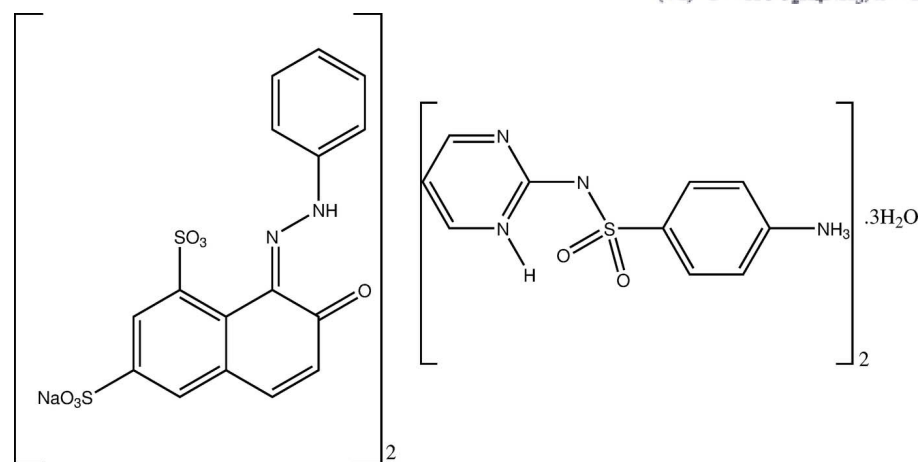
6 The structures of four salt forms of sulfadiazine (SDH) with alkali metal cations are presented. Three contain the
 7 deprotonated SD anion. These are the discrete complex $[\text{Li}(\text{SD})(\text{OH}_2)_2]$, (I), and the coordination polymers $[\text{Na}(\text{SD})]_n$,
 8 (II), and $[\text{K}(\text{SD})(\text{OH}_2)_2]_n$, (III). The Na complex (II) is a three-dimensional coordination polymer whilst the K complex
 9 (III) has two crystallographically independent $[\text{K}(\text{SD})(\text{OH}_2)_2]$ units per asymmetric unit, $Z' = 2$, and gives a two-
 10 dimensional coordination polymer whose layers propagate parallel to the crystallographic *ab* plane. The different bonding
 11 modes of the SD anion in these three complexes is discussed. Structure (IV) contains protonated SDH_2^+ cations and the
 12 Orange G (OG), $\text{C}_{16}\text{H}_{10}\text{N}_2\text{O}_7\text{S}_2$, dianion in a structure with formula $[\text{SDH}_2]_2[\text{Na}(\text{OG})(\text{OH}_2)_4]_2 \cdot 3\text{H}_2\text{O}$. The $[\text{Na}(\text{OG})(\text{OH}_2)_4]_2$
 13 dimers have antiparallel naphthol ring structures joined through two Na centres that bond to the hydrazone anions
 14 through the O atoms of the ketone and sulfonate substituents. The structures of the salts formed on reaction of SDH with
 15 2-aminopyridine and ethanolamine are also presented as $[\text{C}_5\text{H}_7\text{N}_2][\text{SD}]$, (V), and $[\text{HOCH}_2\text{CH}_2\text{NH}_3][\text{SD}] \cdot \text{H}_2\text{O}$, (VI),
 16 respectively. Structure (V) features a heterodimeric $R_2^2(8)$ hydrogen bond motif between the cation and the anion whilst
 17 structure (VI) has a tetrameric core of two cations linked by a central $R_2^2(10)$ hydrogen bonded motif which supports two
 18 anions linked to this core by $R_3^3(8)$ motifs.

19 Keywords: crystal structure, active pharmaceutical ingredients, salt selection, sulfadiazine, sulfa drugs

33scheme1.tif



33scheme2.tif



(IV)

1. Comment

The active pharmaceutical ingredient (API) 4-amino-*N*-(pyrimidin-2-yl)-benzenesulfonamide is commonly known as sulfadiazine (SDH) and is a well known antibiotic. Common variants are its Ag(I) complex which is used in creams and impregnated medical devices, and its Na salt which is used intravenously (Fisher *et al.*, 2003; Ghedini *et al.*, 2017; Mohseni *et al.*, 2016; Preskey & Kayes, 1976). SDH is amphoteric, allowing salt formation reactions to be carried out with both acids and bases. This is a pharmaceutically useful as salt formation is the commonest route used to modify the performance critical material properties (*e.g.* aqueous solubility, melting point or mechanical properties) of APIs (Stahl & Wermuth, 2008).

Structural studies of protonated SDH₂ cations as a variety of salt forms have been published, (*e.g.* Pan *et al.*, 2013; Buist *et al.*, 2014) as have studies of cocrystal phases featuring neutral SDH (*e.g.* Elacqua *et al.*, 2013). Structures of the deprotonated SD anion are also well represented in the literature. These include the structure of the commercially utilized Ag(I) complex (Baenziger & Struss, 1976) and of many transition metal complexes, especially those involving the heavier first row transition metals Co, Ni, Cu and Zn (*e.g.* Shi *et al.*, 2015; Sun *et al.*, 2016; Pan *et al.*, 2012). Salt structures of SD with organic cations are also well known (*e.g.* Elacqua *et al.*, 2013; Heren *et al.*, 2006). Somewhat strangely, there are very few structural studies of s-block metal complexes of SD. As far as we are aware, the only known s-block metal structure is that of a Ca salt form (Tommasino *et al.*, 2011). Given the ubiquity of s-block metal salt usage in pharmaceutical materials in general, and the long-standing commercial use of [Na][SD] in particular, this seemed an odd omission (Stahl & Wermuth, 2008; Preskey & Kayes, 1976). The current study adds to our knowledge of sulfadiazine structural chemistry by reporting the structures of three alkali metal salt forms of SD with Li, Na and K, structures (I), (II) and (III) respectively, as well as the structure formed when the sulfonated azo dye sodium Orange G (OG) crystallizes in the presence of SDH to give a form containing both Na and SDH₂ cations, (IV). Finally, the structures of two new organic salt forms (V) and (VI), prepared by reaction of SDH with the bases 2-aminopyridine and ethanolamine, are also presented for comparison.

The structure of the lithium salt (I) was found to consist of a simple discrete coordination compound of type [Li(SD)(OH₂)₂], see Figure 1. Extensive hydrogen bonding creates a three-dimensional hydrogen bonding network, Table 3. The SD anion acts in a chelating fashion to form a 6-membered [LiOSNCN] ring through Li to O and Li to heterocyclic N bonds. Thus although the Li coordination geometry is tetrahedral, the bond angles are considerably distorted due to the small bite angle of the chelate (range 90.91 (18) to 126.9 (2) °), see Table 2 for details of bond lengths and angles. This O,*N* chelation mode is unusual. Discrete d-block metal complexes of SD normally bond to metal centres through a N,*N* chelate utilizing both the ring and sulfamide N atoms (*e.g.* Shi *et al.*, 2015; Sun *et al.*, 2016; Pan *et al.*, 2012). The same is true of the Ca salt (Tommasino *et al.*, 2011). The polymer [Zn(SD)]_n does feature a similar O,*N* chelation mode (Yuan *et al.*, 2001) and the dimeric species [Cu₂(SD)₄] can be described as containing Cu—O bonds, but these are very long (2.55 to 2.75 Å) and form 4-membered chelate rings through interactions with the sulfamide N atom rather than the 6-membered ring involving the aromatic ring observed for (I) (Shi *et al.*, 2015). The structure of the pharmaceutically important coordination polymer [Ag(SD)]_n does contain the O,*N* chelation mode seen for (I) with the polymer propagating through both the common N,*N* chelation mode and the O,*N* mode (Baenziger & Struss, 1976).

Like the Ag complex, the structure of anhydrous [Na(SD)]_n (II) forms a coordination polymer through each SD anion making two chelating interactions with two metal centres. One such interaction forms a 6-membered NaOSNCN ring with a O,*N* bonding mode and the other forms a NaNCN ring with a N,*N* mode. The coordination polymer is further connected by NaONaO 4-membered rings, see Figs 2 and 3. Thus the atoms N1, N2, N3 and O1 bond to Na centres and form one-dimensional coordination chains that propagate parallel to the crystallographic *a* direction. Unlike the Ag

61 complex, in (II) the amine group of the ligand also takes part in bonding. This gives 6 coordinate Na centres, Table 4.
62 This last interaction type links the individual chains through amine to Na bonds and gives the overall three-dimensional
63 coordination polymer shown in Fig. 4. We are aware no other examples of anionic SD bonding to metal through its NH₂
64 tail. The coordination polymer is supported by amine to SO₂ N=H...O hydrogen bonds as described in Table 5.

65 The K salt (III) is a dihydrate with two crystallographically independent [K(SD)(OH₂)₂] units per asymmetric unit, Z' =
66 2, see Fig. 5. Of the 4 independent water molecules, two (O2W and O3W) act as terminal ligands whilst O1W bridges
67 between K1 and K1' (' = 1 - x, 1 - y, 1 - z) and O3W interestingly bridges between three K centres (K1, K2 and K1''
68 where '' = -x, 1 - y, 1 - z), see Table 6 for details. The two independent SD anions also have different bonding modes, the
69 ligand containing O3 utilizes both of its O atoms and 3 of its N atoms to bond between 4 separate K centres. Each N atom
70 bonds to only one K centre with the O atoms both bridging between two K centres. Like the Na and Ag species, above,
71 this ligand features both N,N 4-membered ring forming and O,N 6-membered ring forming chelation modes. Perhaps
72 surprisingly, the other SD anion adopts a bonding mode that forms only two donor to K contacts and bonds to only one K
73 centre. Atoms O1 and N1 (the sulfamide N atom) bond to K2. This O,N [KOSN] 4-membered ring forming bonding
74 mode is not seen for other s-block metals with SD but can be observed in the structure of [Cu₂(SD)₄] (Shi *et al.*, 2015).
75 See Table 12 for a summary of the different bonding modes adopted by SD with alkali metals. A final difference between
76 the two SD ligands is conformational. The SD anion that bonds to multiple K centres has the same conformation as that
77 found for the SD anions in all the other structures reported herein. That is, one of the SO₂ O atoms lies close to the plane
78 of the aniline ring and the pyrimidine ring is *syn* to this O atom. However, the SD anion that makes only 2 bonds to K2
79 has an alternative conformation where it is the amide N atom and not an O atom that lies closest to the plane of the
80 aniline ring (N1S1C5C6 = 17.7 (4) °). The coordination bonds combine to give two-dimensional coordination polymers
81 that propagate parallel to the *ab* plane and gives the layered structure shown in Fig. 6, with organic bilayers and inorganic
82 layers alternating along the crystallographic *c* direction. Hydrogen bonds from the amine group link between
83 neighbouring two-dimensional coordination polymers, Table 7.

84 Crystallized from SDH in the presence of acid and the sodium salt of OG, (IV) has the general formula
85 [SDH₂]₂[Na(OG)(OH₂)₄]₂·3H₂O, Fig. 7. We are unaware of any other structure containing both a metal cation and cationic
86 SDH₂. There are 4 independent water ligands bound to Na1. These are ordered but the non-coordinated water molecules
87 are not and have been modelled over 3 crystallographic sites, each with site occupancy factor of 0.5. In common with
88 other sulfonated azo species based on naphthol units, the dye adopts the hydrazone tautomeric form with protonation at
89 N6 of the N=N group (Kennedy *et al.*, 2012). This leads to characteristic lengthening (*e.g.* N=N, C—C) and shortening
90 (*e.g.* O—C, N—C) of relevant bonds compared to similar azo species. Compare values in Table 8 with *e.g.* 1.253 (2) and
91 1.418 (3) Å for N=N and N—C bond lengths in a typical azo species (Kennedy *et al.*, 2001). The dimeric unit is shown in
92 Fig. 8 and is crystallographically centrosymmetric, Z' = 0.5. The naphthol units lie antiparallel to each other with an
93 octahedral Na centre at each end. In addition to the 4 terminal water ligands, each Na centre bonds to the ketone group of
94 one anion and to atom O7 of a sulfonate group of a second anion thus creating the dimeric unit. See Tables 8 and 9 for
95 bonding parameters. There is some degree of π to π stacking across this dimer as is indicated by a minimum C...C distance
96 of 3.482 (2) Å (between C11 and the C15 atom at -x, 1 - y, -z). Orange G is exceptional amongst sulfonated azo
97 colourants in having being widely studied crystallographically. Structures are known for its s-block metal salts, for its
98 complexes with transition metals and for salt forms with organic cations (Kennedy *et al.*, 2006; Ojala *et al.*, 1994b;
99 Kennedy *et al.*, 2010; Ojala *et al.*, 1994a). However, the dimeric unit observed here is not seen for other s-block metal
100 salt forms of OG. The closest motif occurs in the Ag(I) complex of OG, where a similar dimer forms part of a larger
101 polymeric coordination network (Kennedy *et al.*, 2006). The sulfadiazine cation is found to have three H atoms bound to
102 N4, the aniline group, and one H atom bound to N3 of the pyrimidine ring. In contrast, previous work had found that

103 SDH₂ cations adopted this tautomeric form with simple anions but with sulfonate anions gave the alternative tautomer
104 with protonation at the amide N atom (Buist *et al.*, 2014). The geometry of the SDH₂ cation is somewhat different from
105 those of the SD anions in the other structures presented. Conformationally, it is again an O atom that lies closest to the
106 plane of the aniline ring, but in contrast to the SD anions this O atom is anti to the pyrimidine ring. An expected small
107 increase in S—N bond length is also seen for SDH₂ as compared to the SD anions (Elacqua *et al.*, 2013; Buist *et al.*,
108 2014). Another feature common in other structures with SDH₂ cations is a centrosymmetric $R_2^2(8)$ hydrogen bonded
109 dimer of cations formed utilizing amide and pyrimidine N atoms (Buist *et al.*, 2014). This motif is retained in (IV).
110 Structure (IV) has a layered structure with hydrophilic/inorganic layers parallel to the *bc* plane alternating with hydro-
111 phobic/organic layers.

112 Formed from the reaction of SDH with 2-aminopyridine, structure (V) is that of [C₆H₄N(H)NH₂][SD], Fig 9. It features
113 a heterodimeric $R_2^2(8)$ hydrogen bond between the cation and the amide N atom and one pyrimidine ring N atom of the
114 SD anion, Fig 10. There are also anion to anion interactions with the amine group of the SD anion donating two hydrogen
115 bonds to O atoms of two neighbouring SD anions. A final hydrogen bond is of type N—H···N and is donated by the
116 pyridine amine group to the SD amine group, see Table 10 for details. Unusually this leaves one N atom of the pyrimidine
117 ring with no hydrogen bonding interaction. The resulting structure has layers formed of heteroaromatic rings (both
118 pyridine and pyrimidine) alternating with layers of aniline groups, these layers lie parallel to the crystallographic *ac*
119 plane.

120 Structure (VI) was obtained on the reaction of SDH with ethanolamine and is that of the hydrate, [HOCH₂CH₂NH₃]
121 [SD]·H₂O, Fig 11. The dimeric $R_2^2(8)$ hydrogen bonded motif does not occur in this structure. It is replaced by a
122 tetrameric unit comprising the amide N atom and one pyrimidine ring N atom of two SD anions interacting with a
123 centrosymmetric dimer of HOCH₂CH₂NH₃ cations, see Fig 12. Thus there are two $R_3^3(8)$ motifs supported by a central
124 $R_2^2(10)$ motif. The tetramers are linked by hydrogen bonding through a bifurcated bond from H₄N to O and N acceptors
125 on a SD anion and through the water molecules. Each water molecule interacts with 4 neighbouring SD anions. Thus
126 each water molecule donates two hydrogen bonds to O atoms of SD anions and accepting two hydrogen bonds from the
127 SD amine groups, see Table 11 for details.

128 2. Synthesis and crystallization

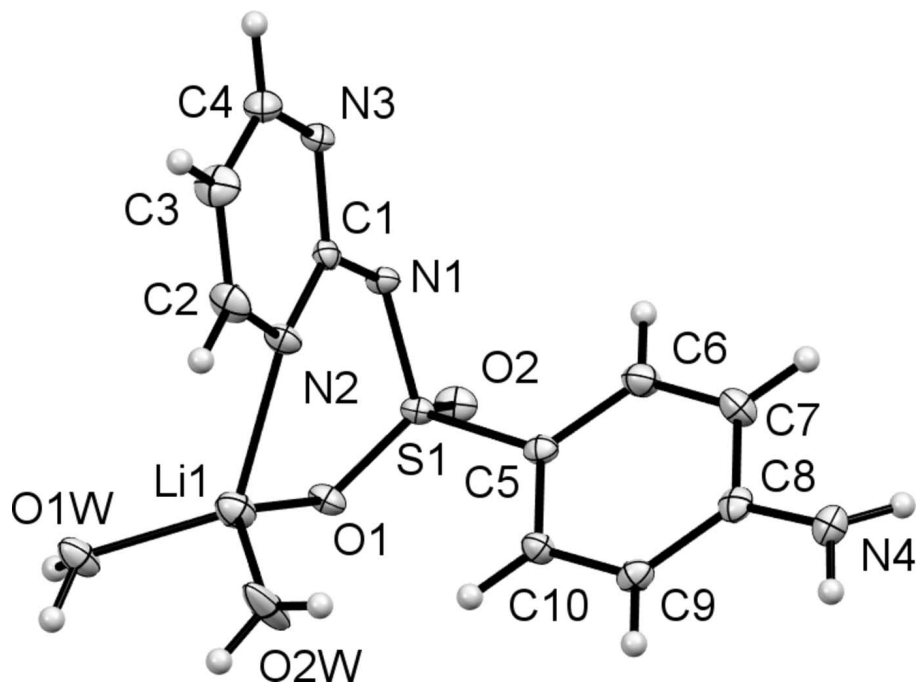
129 The simple salt forms were prepared by reacting 1:1 molar mixtures of sulfadiazine and MOH (*M* = Li, Na, K) or the
130 organic base in 50:50 water:ethanol. The mixtures were stirred and heated to give clear solutions before being left to cool
131 to room temperature. Partial evaporation of these reaction mixtures over 4 to 7 days gave suitable crystals of (I), (III), (V)
132 and (VI) but a fine powder for the Na salt. Good quality crystals of the Na salt (II) were obtained by vapour diffusion of
133 ethanol into an aqueous solution of sodium sulfadiazine. The Na OG complex (IV) was obtained by dissolving 0.20 g,
134 0.44 mmol of NaOG in the minimum amount of water. A slight excess of sulfadiazine (0.12 g, 0.48 mmol) was also
135 dissolved in the minimum amount of water. The two solutions were mixed together with stirring and acidified with
136 concentrated HCl. After 3 days, orange crystals of (IV) had grown.

137 3. Refinement

138 For all structures, H atoms bound to C atoms were placed in the expected geometric positions and treated in riding modes
139 with $U(H)_{iso} = 1.2U(C)_{eq}$. For aromatic groups C—H = 0.95 Å and for CH₂ groups C—H = 0.95 Å. All H atoms bound to N
140 were refined freely and isotropically as were H atoms bound to O in structure (VI). H atoms of water molecules in (I),
141 (III) and (IV) were located by difference syntheses and required restraints to be applied such that O—H = 0.88 (1) Å and
142 H···H = 1.33 (2) Å. For these atoms of (III) $U(H)_{iso} = 1.5U(O)_{eq}$, whilst for (I) and (IV) $U(H)_{iso}$ were refined.

143 For (IV), after several trial calculations, the three non-coordinated water molecules were given site occupancy factors
144 of 0.5. For (III) the H atoms of one water ligand were modelled as disordered over three sites.

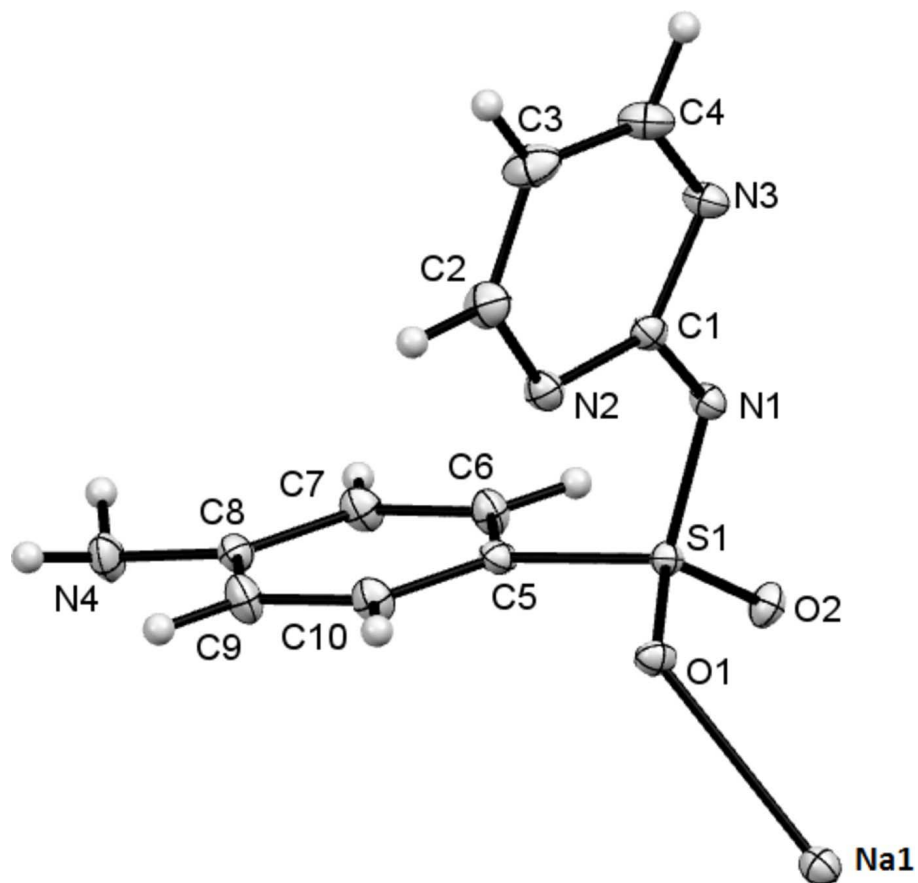
fig1.tif



145 **Figure 1**

146 Molecular structure of the Li salt (I) with non-H atoms shown as 50% probability ellipsoids.

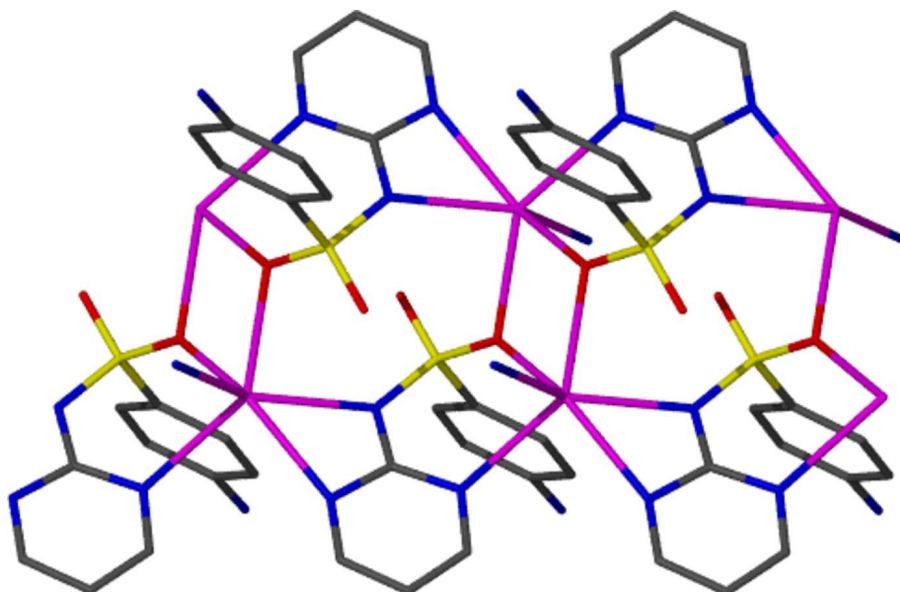
fig2.tif



147 **Figure 2**

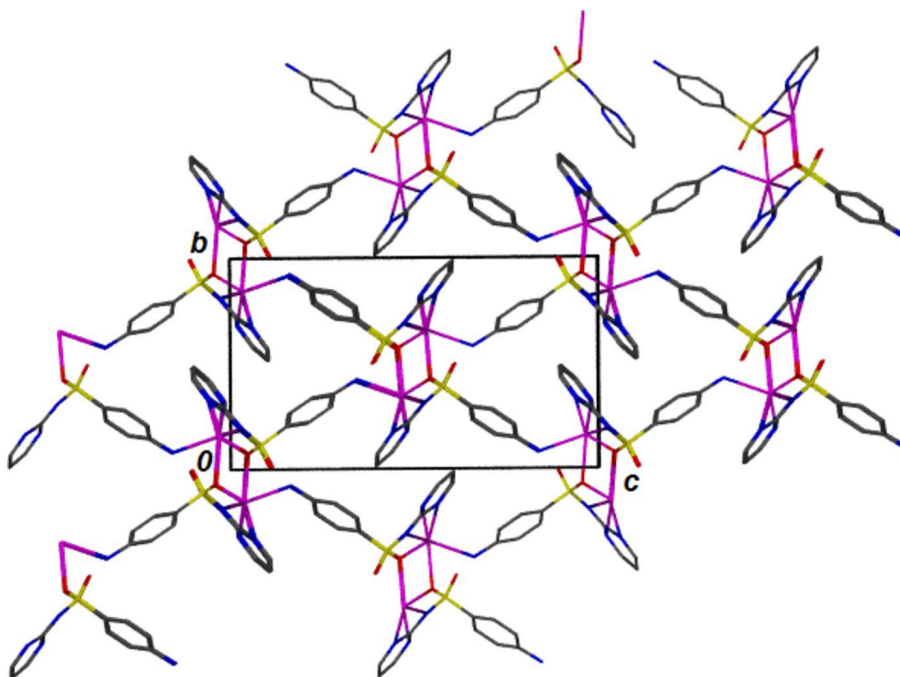
148 Contents of the asymmetric unit of the Na salt (II) with non-H atoms shown as 50% probability ellipsoids.

fig3.tif

149 **Figure 3**

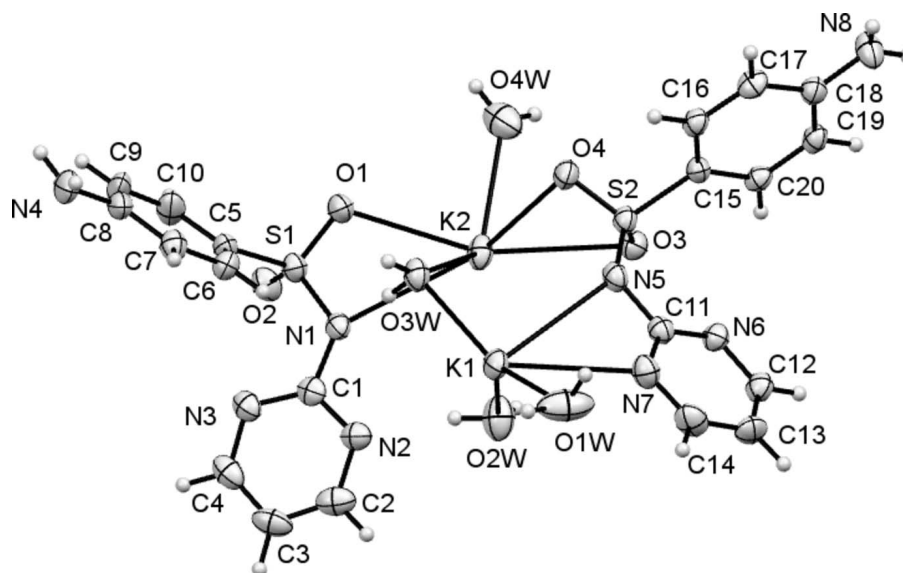
150 Part of the extended structure of (II) showing the dative bonds that give a one-dimensional chain that extends in the
151 crystallographic *a* direction. H atoms are omitted for clarity. Here and in other colour figures, black = C, blue = N, red =
152 O, yellow = S, pink = alkali metal.

fig4.tif

153 **Figure 4**

154 Packing diagram for (II) with view down the *a* axis direction.

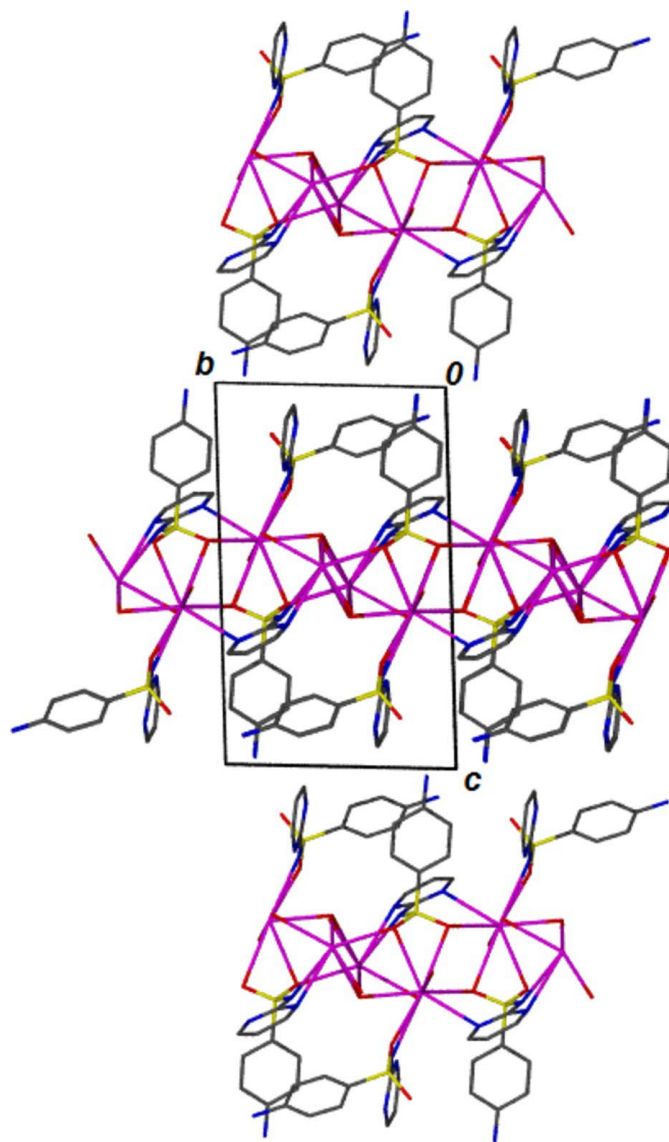
fig5.tif



155 **Figure 5**

156 Contents of the asymmetric unit of the K salt (III) with non-H atoms shown as 50% probability ellipsoids. Disordered H
157 atom positions on water ligand O4W are not shown.

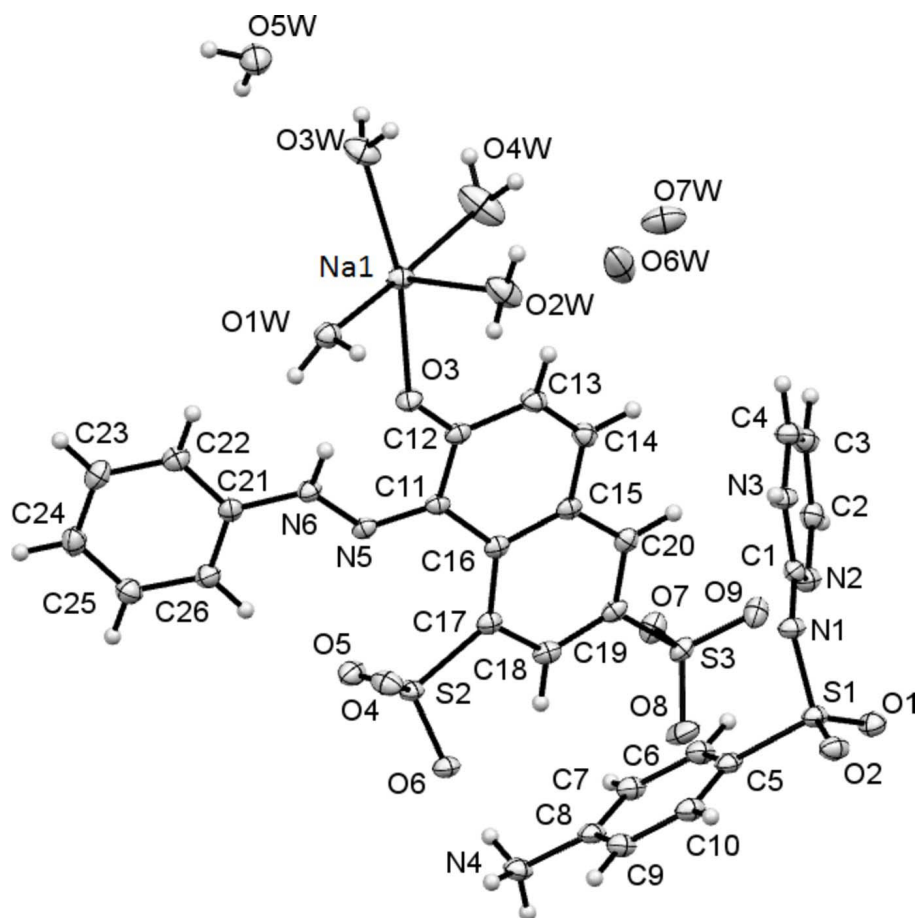
fig6.tif



158 **Figure 6**

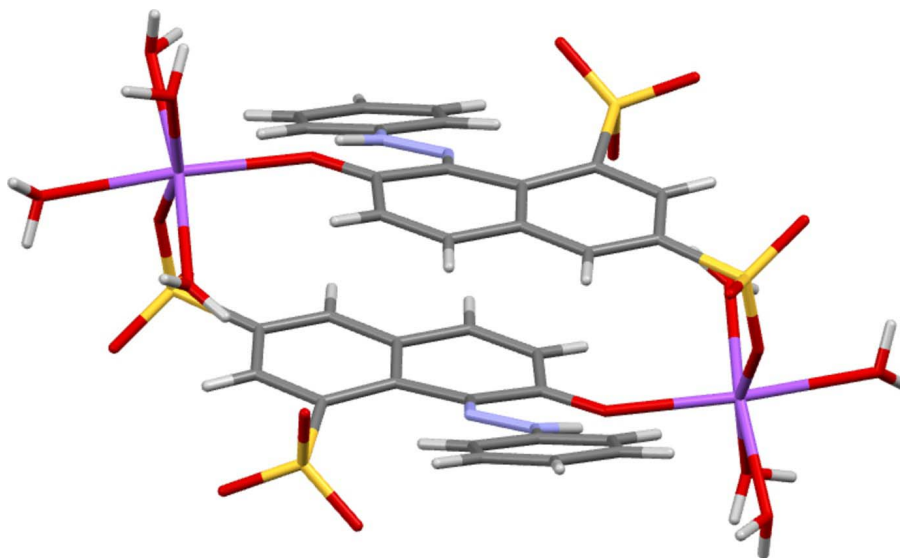
159 Packing diagram for showing the layered structure of (III) with view down the *a* axis direction.

fig7.tif

160 **Figure 7**

161 Contents of the asymmetric unit of (IV) with non-H atoms shown as 50% probability ellipsoids. Disordered H atom
162 positions on water molecules are not shown.

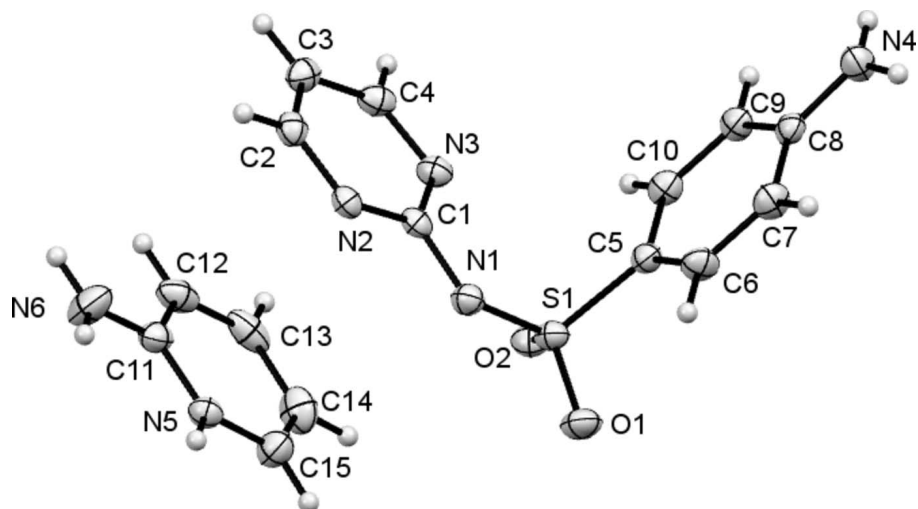
fig8.tif



163 **Figure 8**

164 View of the dimeric $[\text{Na}(\text{H}_2\text{O})_4]_2[\text{OG}]_2$ fragment found in structure (IV).

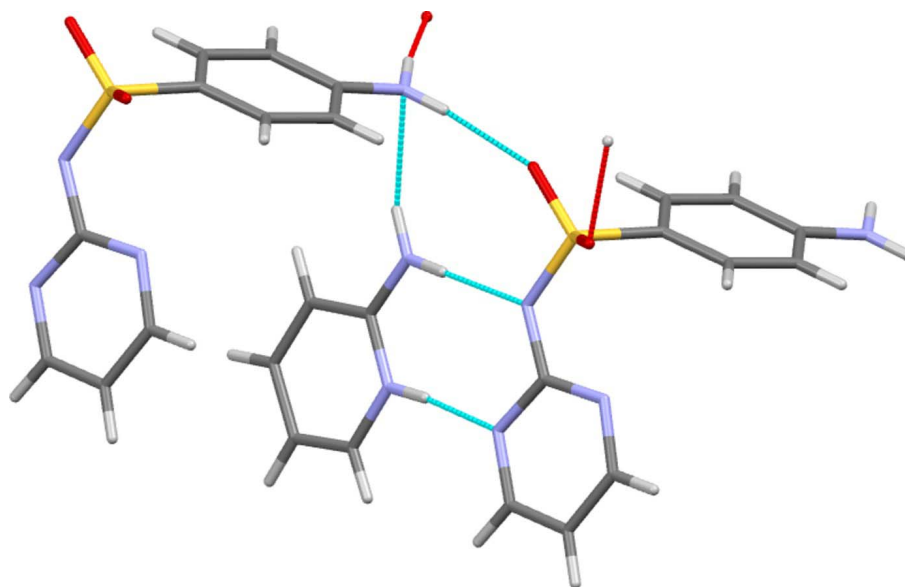
fig9.tif



165 **Figure 9**

166 Contents of the asymmetric unit of (V) with non-H atoms shown as 50% probability ellipsoids.

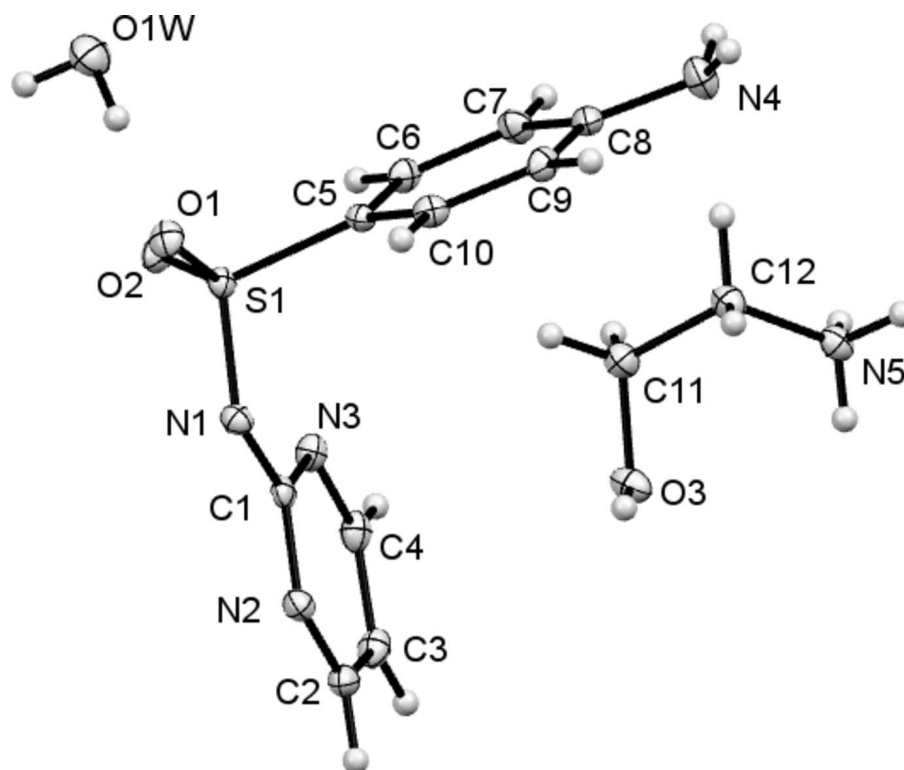
fig10.tif



167 **Figure 10**

168 Detail from the structure of (V) highlighting the $R_2^2(8)$ cation to anion hydrogen bond interaction and the role of the
 169 amine groups.

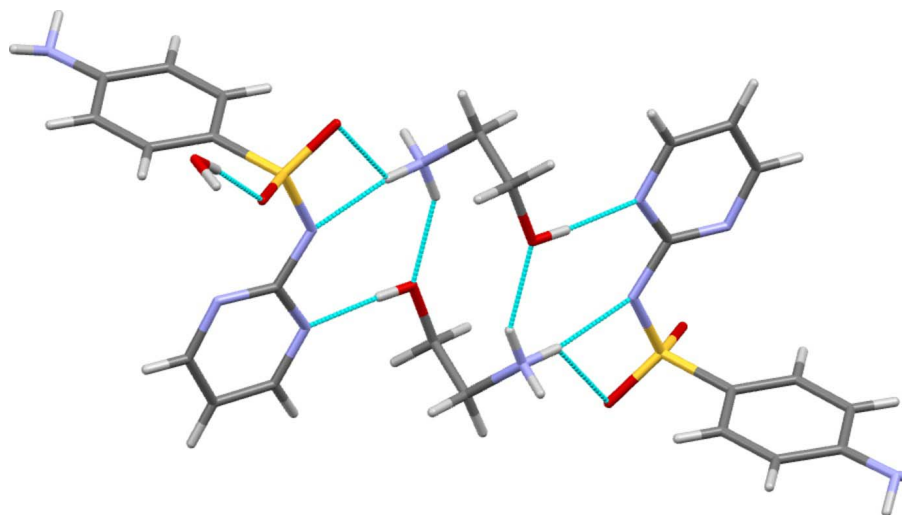
fig11.tif



170 **Figure 11**

171 Contents of the asymmetric unit of (VI) with non-H atoms shown as 50% probability ellipsoids.

fig12.tif



172 **Figure 12**

173 Detail from the structure of (VI) showing the hydrogen bonded tetramer formed by two cations and two anions.

174 **Table 1**

175 Experimental details

	(I)	(II)	(III)	(IV)
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176 Crystal data

178	Chemical formula	$C_{10}H_{13}LiN_4O_4S$	$C_{10}H_9N_4NaO_2S$	$C_{10}H_{13}KN_4O_4S$	$C_{26}H_{32}N_6NaO_{14.50}S_3$
179	M_r	292.24	272.26	324.40	779.74
180	Crystal system, space group	Monoclinic, Cc	Monoclinic, $P2_1/c$	Triclinic, $P\bar{1}$	Monoclinic, $P2_1/c$
181	Temperature (K)	123	123	123	100
182	a, b, c (Å)	11.5095 (4), 12.0788 (5), 9.5184 (4)	5.9010 (11), 10.534 (4), 18.389 (3)	8.8503 (7), 9.6385 (4), 15.9734 (7)	12.7688 (1), 16.9040 (1), 15.7411 (1)
183	α, β, γ (°)	90, 91.063 (3), 90	90, 94.216 (15), 90	92.350 (4), 95.186 (4), 94.209 (4)	90, 107.609 (1), 90
184	V (Å ³)	1323.03 (9)	1140.0 (5)	1351.79 (13)	3238.42 (4)
185	Z	4	4	4	4
186	Radiation type	Mo $K\alpha$	Mo $K\alpha$	Cu $K\alpha$	Cu $K\alpha$
187	μ (mm ⁻¹)	0.26	0.32	5.09	2.95
188	Crystal size (mm)	0.30 × 0.20 × 0.18	0.35 × 0.12 × 0.08	0.28 × 0.15 × 0.10	0.12 × 0.08 × 0.04
189					
190	Data collection				
191	Diffractometer	Oxford Diffraction Xcalibur E	Oxford Diffraction Xcalibur E	Oxford Diffraction Gemini S	XtaLAB AFC11 (RCD3)
192	Absorption correction	Multi-scan <i>CrysAlis PRO</i> , Oxford Diffraction Ltd., Version 1.171.34.40 (release 27-08-2010 <i>CrysAlis171 .NET</i>) (compiled Aug 27 2010, 11:50:40) Empirical absorption correction using spherical harmonics, implemented in SCALE3 ABSPACK scaling algorithm.	Multi-scan <i>CrysAlis PRO</i> , Oxford Diffraction Ltd., Version 1.171.34.40 (release 27-08-2010 <i>CrysAlis171 .NET</i>) (compiled Aug 27 2010, 11:50:40) Empirical absorption correction using spherical harmonics, implemented in SCALE3 ABSPACK scaling algorithm.	Multi-scan <i>CrysAlis PRO</i> , Agilent Technologies, Version 1.171.37.35 (release 13-08-2014 <i>CrysAlis171 .NET</i>) (compiled Aug 13 2014, 18:06:01) Empirical absorption correction using spherical harmonics, implemented in SCALE3 ABSPACK scaling algorithm.	Multi-scan <i>CrysAlis PRO</i> 1.171.39.34b (Rigaku Oxford Diffraction, 2017) Empirical absorption correction using spherical harmonics, implemented in SCALE3 ABSPACK scaling algorithm.
193	T_{\min}, T_{\max}	0.949, 1.000	0.961, 1.000	0.601, 1.000	0.615, 1.000
194	No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	3390, 2246, 2184	5903, 2786, 2351	15580, 5357, 3951	59125, 5913, 5686
195	R_{int}	0.016	0.029	0.050	0.027
196	$(\sin \theta/\lambda)_{\text{max}}$ (Å ⁻¹)	0.682	0.693	0.623	0.602
197					
198	Refinement				
199	$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.026, 0.066, 1.04	0.038, 0.088, 1.06	0.060, 0.157, 1.05	0.028, 0.075, 1.05
200	No. of reflections	2246	2786	5357	5913
201	No. of parameters	207	171	404	537
202	No. of restraints	8	0	14	13
203	H-atom treatment	H atoms treated by a mixture of independent and constrained refinement	H atoms treated by a mixture of independent and constrained refinement	H atoms treated by a mixture of independent and constrained refinement	H atoms treated by a mixture of independent and constrained refinement
204	$\Delta\rho_{\text{max}}, \Delta\rho_{\text{min}}$ (e Å ⁻³)	0.28, -0.25	0.43, -0.46	0.81, -0.45	0.26, -0.39

205	Absolute structure	Refined as an inversion? twin.	?	?
206	Absolute structure parameter	-0.01 (8) ?	?	?
207		(V)		(VI)
208	Crystal data			
209	Chemical formula	C ₁₅ H ₁₆ N ₆ O ₂ S		C ₁₂ H ₁₉ N ₅ O ₄ S
210	<i>M_r</i>	344.40		329.38
211	Crystal system, space group	Monoclinic, <i>P</i> 2 ₁ / <i>c</i>		Monoclinic, <i>P</i> 2 ₁ / <i>c</i>
212	Temperature (K)	123		123
213	<i>a</i> , <i>b</i> , <i>c</i> (Å)	8.5796 (2), 19.0371 (5), 11.2512 (4)		12.7755 (4), 9.8979 (4), 11.5366 (4)
214	<i>α</i> , <i>β</i> , <i>γ</i> (°)	90, 121.116 (3), 90		90, 93.508 (3), 90
215	<i>V</i> (Å ³)	1573.27 (9)		1456.08 (9)
216	<i>Z</i>	4		4
217	Radiation type	Mo <i>Kα</i>		Mo <i>Kα</i>
218	<i>μ</i> (mm ⁻¹)	0.23		0.25
219	Crystal size (mm)	0.4 × 0.3 × 0.02		0.25 × 0.24 × 0.12
220				
221	Data collection			
222	Diffractometer	Oxford Diffraction Xcalibur E		Oxford Diffraction Xcalibur E
223	Absorption correction	Multi-scan <i>CrysAlis PRO</i> , Oxford Diffraction Ltd., Version 1.171.34.40 (release 27-08-2010 <i>CrysAlis171 .NET</i>) (compiled Aug 27 2010,11:50:40) Empirical absorption correction using spherical harmonics, implemented in SCALE3 ABSPACK scaling algorithm.		Multi-scan <i>CrysAlis PRO</i> , Oxford Diffraction Ltd., Version 1.171.34.40 (release 27-08-2010 <i>CrysAlis171 .NET</i>) (compiled Aug 27 2010,11:50:40) Empirical absorption correction using spherical harmonics, implemented in SCALE3 ABSPACK scaling algorithm.
224	<i>T_{min}</i> , <i>T_{max}</i>	0.527, 1.000		0.904, 1.000
225	No. of measured, independent and observed [<i>I</i> > 2σ(<i>I</i>)] reflections	22193, 4082, 3225		7213, 3571, 2942
226	<i>R_{int}</i>	0.040		0.023
227	(sin <i>θ</i> /λ) _{max} (Å ⁻¹)	0.682		0.694
228				
229	Refinement			
230	<i>R</i> [<i>F</i> ² > 2σ(<i>F</i> ²)], <i>wR</i> (<i>F</i> ²), <i>S</i>	0.045, 0.114, 1.04		0.037, 0.095, 1.06
231	No. of reflections	4082		3571
232	No. of parameters	237		231
233	No. of restraints	0		0
234	H-atom treatment	H atoms treated by a mixture of independent and constrained refinement		H atoms treated by a mixture of independent and constrained refinement
235	Δρ _{max} , Δρ _{min} (e Å ⁻³)	0.39, -0.48		0.45, -0.47
236	Absolute structure	?		?

237 Absolute structure parameter? ?

238 Computer programs: *CrysAlis PRO* (Agilent, 2014), *CrysAlis PRO, SIR92* (Altomare *et al.*, 1994), *SHELXS* (Sheldrick, 2015), *SHELXL2014* (Sheldrick,
239 2015), *Mercury* (Macrae *et al.*, 2008), *SHELXL2014*.

240 **Table 2**

241 Selected geometric parameters (Å, °) for (I)

242	Li1—O2W	1.870 (4)	Li1—O1	1.934 (5)
243	Li1—O1W	1.910 (5)	Li1—N2	2.077 (5)
244				
245	O2W—Li1—O1W	101.5 (2)	O2W—Li1—N2	107.1 (2)
246	O2W—Li1—O1	126.9 (2)	O1W—Li1—N2	124.1 (2)
247	O1W—Li1—O1	108.5 (2)	O1—Li1—N2	90.91 (18)

248 **Table 3**

249 Hydrogen-bond geometry (Å, °) for (I)

250	<i>D</i> —H \cdots <i>A</i>	<i>D</i> —H	H \cdots <i>A</i>	<i>D</i> \cdots <i>A</i>	<i>D</i> —H \cdots <i>A</i>
251	O1W—H1W \cdots O2 ⁱ	0.875 (12)	2.051 (16)	2.881 (2)	158 (3)
252	O2W—H3W \cdots N1 ⁱⁱ	0.865 (12)	1.949 (15)	2.805 (3)	170 (4)
253	O1W—H2W \cdots N3 ⁱⁱ	0.867 (12)	1.970 (12)	2.837 (3)	178 (3)
254	O2W—H4W \cdots N4 ⁱⁱⁱ	0.863 (12)	2.006 (12)	2.866 (3)	174 (4)
255	N4—H1N \cdots O2 ^{iv}	0.84 (3)	2.40 (3)	3.070 (3)	137 (3)

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

257 **Table 4**

258 Selected bond lengths (Å) for (II)

259	Na1—O1 ⁱ	2.2897 (15)	Na1—N3 ⁱⁱ	2.4836 (18)
260	Na1—N1 ⁱⁱ	2.4533 (15)	Na1—N4 ⁱⁱⁱ	2.5771 (19)
261	Na1—O1	2.4647 (17)	Na1—N2 ⁱ	2.6670 (17)

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

263 **Table 5**

264 Hydrogen-bond geometry (Å, °) for (II)

265	<i>D</i> —H \cdots <i>A</i>	<i>D</i> —H	H \cdots <i>A</i>	<i>D</i> \cdots <i>A</i>	<i>D</i> —H \cdots <i>A</i>
266	N4—H1N \cdots O2 ^{iv}	0.86 (2)	2.06 (2)	2.907 (2)	166 (2)
267	N4—H2N \cdots O1 ^v	0.91 (2)	2.52 (2)	2.959 (2)	110.5 (17)

268 Symmetry codes: (iv) $-x, y+1/2, -z+1/2$; (v) $-x+1, y+1/2, -z+1/2$.

269 **Table 6**

270 Selected bond lengths (Å) for (III)

271	K1—O4 ⁱ	2.729 (3)	K2—O3 ⁱⁱⁱ	2.759 (3)
272	K1—O3W	2.771 (3)	K2—O4W	2.820 (4)

273	K1—O1W ⁱⁱ	2.812 (5)	K2—O3W	2.871 (3)
274	K1—O2W	2.824 (4)	K2—O1	2.883 (3)
275	K1—N7	2.932 (4)	K2—N6 ⁱⁱⁱ	2.899 (4)
276	K1—O1W	2.975 (5)	K2—O4	2.947 (3)
277	K1—O3W ⁱ	3.004 (3)	K2—O3	3.023 (3)
278	K1—N5	3.129 (4)	K2—N1	3.153 (3)

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

280 Table 7

281 Hydrogen-bond geometry ($\text{\AA}, ^\circ$) for (III)

282	$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
283	O1W—H1W \cdots O1 ⁱ	0.880 (10)	1.93 (2)	2.799 (5)	167 (8)
284	O1W—H2W \cdots O4W ⁱ	0.877 (10)	2.27 (4)	3.070 (6)	151 (6)
285	O2W—H3W \cdots O3 ⁱⁱⁱ	0.878 (10)	2.071 (19)	2.933 (5)	167 (6)
286	O2W—H4W \cdots N2	0.880 (10)	1.965 (17)	2.828 (5)	167 (6)
287	O3W—H5W \cdots N1	0.874 (10)	2.022 (18)	2.872 (5)	164 (5)
288	O3W—H6W \cdots N5 ⁱ	0.873 (10)	1.979 (14)	2.835 (5)	166 (4)
289	O4W—H7W \cdots O2W ^{iv}	0.878 (10)	2.052 (17)	2.919 (6)	169 (7)
290	O4W—H8W \cdots O4W ^v	0.879 (10)	2.05 (2)	2.920 (8)	168 (9)
291	N4—H1N \cdots N4 ^{vi}	0.87 (7)	2.50 (6)	3.054 (9)	122 (5)
292	N4—H2N \cdots O2 ^{vii}	0.93 (7)	2.57 (7)	3.431 (7)	153 (5)
293	N8—H3N \cdots O2 ^{viii}	0.87 (5)	2.38 (5)	3.046 (5)	134 (4)
294	N8—H3N \cdots N3 ^{viii}	0.87 (5)	2.61 (5)	3.283 (6)	135 (4)

295 Symmetry codes: (i) $-x, -y+1, -z+1$; (iii) $-x, -y+2, -z+1$; (iv) $x-1, y, z$; (v) $-x-1, -y+2, -z+1$; (vi) $-x-1, -y, -z$; (vii) $x, y-1, z$; (viii) $x, y, z+1$.

296 Table 8

297 Selected geometric parameters ($\text{\AA}, ^\circ$) for (IV)

298	Na1—O1W	2.3184 (13)	Na1—O2W	2.4764 (14)
299	Na1—O4W	2.3211 (15)	O3—C12	1.2626 (18)
300	Na1—O3	2.4042 (12)	N5—N6	1.3031 (18)
301	Na1—O7 ⁱ	2.4237 (12)	N5—C11	1.3322 (19)
302	Na1—O3W	2.4592 (14)	C11—C12	1.467 (2)
303				
304	O1W—Na1—O4W	169.11 (5)	O3—Na1—O3W	166.97 (5)
305	O1W—Na1—O3	90.96 (4)	O7 ⁱ —Na1—O3W	80.17 (5)
306	O4W—Na1—O3	98.12 (5)	O1W—Na1—O2W	81.14 (5)
307	O1W—Na1—O7 ⁱ	85.13 (4)	O4W—Na1—O2W	91.10 (6)
308	O4W—Na1—O7 ⁱ	101.30 (5)	O3—Na1—O2W	101.64 (5)
309	O3—Na1—O7 ⁱ	86.81 (4)	O7 ⁱ —Na1—O2W	163.94 (5)
310	O1W—Na1—O3W	87.73 (5)	O3W—Na1—O2W	90.97 (5)
311	O4W—Na1—O3W	84.75 (6)		

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

313 **Table 9**

314 Hydrogen-bond geometry (Å, °) for (IV)

315	$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
316	O1W—H1W \cdots O9 ⁱⁱ	0.88 (3)	1.99 (3)	2.8404 (16)	163 (2)
317	O1W—H2W \cdots O1 ⁱⁱ	0.91 (3)	2.24 (3)	2.8830 (17)	127 (2)
318	O1W—H2W \cdots N2 ⁱⁱ	0.91 (3)	2.07 (3)	2.8972 (18)	150 (2)
319	O2W—H3W \cdots O9 ⁱⁱ	0.86 (3)	2.05 (3)	2.8852 (18)	163 (2)
320	O2W—H4W \cdots O6 ⁱⁱⁱ	0.92 (4)	1.98 (4)	2.8932 (18)	174 (3)
321	O3W—H5W \cdots O5 ⁱⁱⁱ	0.86 (3)	1.98 (3)	2.8320 (18)	178 (3)
322	O3W—H6W \cdots O5W	0.83 (4)	2.18 (3)	2.736 (3)	124 (3)
323	O3W—H6W \cdots O6W ^{iv}	0.83 (4)	2.09 (4)	2.853 (3)	153 (3)
324	O3W—H6W \cdots O7W ^{iv}	0.83 (4)	2.06 (4)	2.799 (3)	148 (3)
325	O4W—H7W \cdots O5 ⁱ	0.879 (10)	2.242 (11)	3.1093 (19)	169 (2)
326	O4W—H8W \cdots O4W ^{iv}	0.883 (10)	2.00 (2)	2.825 (3)	154 (4)
327	O4W—H8W \cdots O6W	0.883 (10)	2.41 (5)	2.871 (3)	113 (4)
328	O4W—H14W \cdots O7W ^{iv}	0.886 (10)	2.16 (3)	2.902 (3)	140 (4)
329	O5W—H9W \cdots O4 ^v	0.883 (10)	1.845 (13)	2.710 (3)	166 (4)
330	O5W—H10W \cdots O8 ⁱ	0.885 (10)	1.986 (14)	2.843 (3)	163 (3)
331	O6W—H11W \cdots O2 ^{vi}	0.886 (10)	2.22 (2)	2.893 (3)	132 (2)
332	O6W—H12W \cdots O2W	0.881 (10)	1.850 (11)	2.731 (3)	178 (5)
333	O7W—H13W \cdots O8 ⁱⁱⁱ	0.875 (10)	1.91 (2)	2.699 (3)	150 (3)
334	N6—H1N \cdots O3	0.92 (2)	1.75 (2)	2.5342 (16)	142 (2)
335	N4—H2N \cdots O3W ^{vii}	0.92 (3)	1.93 (3)	2.839 (2)	171 (2)
336	N4—H3N \cdots O6	0.91 (2)	1.92 (2)	2.8245 (18)	170 (2)
337	N4—H4N \cdots O1 ^{viii}	0.91 (2)	2.01 (2)	2.8784 (18)	159.6 (19)
338	N4—H4N \cdots O1W ^{vii}	0.91 (2)	2.50 (2)	2.9386 (19)	110.5 (16)
339	N3—H5N \cdots O2 ^{vi}	0.84 (2)	2.57 (2)	3.0827 (17)	120.3 (18)
340	N3—H5N \cdots N1 ^{vi}	0.84 (2)	2.02 (2)	2.8655 (19)	177 (2)

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

342 **Table 10**

343 Hydrogen-bond geometry (Å, °) for (V)

344	$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
345	N4—H1N \cdots O2 ⁱ	0.86 (2)	2.04 (2)	2.895 (2)	168 (2)
346	N4—H2N \cdots O1 ⁱⁱ	0.87 (2)	2.03 (2)	2.887 (2)	174 (2)
347	N5—H3N \cdots N2 ⁱⁱⁱ	0.92 (2)	1.85 (2)	2.758 (2)	174 (2)
348	N6—H4N \cdots N4 ^{iv}	0.87 (3)	2.39 (3)	3.085 (2)	137 (2)
349	N6—H5N \cdots N1 ⁱⁱⁱ	0.92 (3)	2.00 (3)	2.918 (2)	173 (3)

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

351 **Table 11**

352 Hydrogen-bond geometry (Å, °) for (VI)

353	$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
354	N4—H1N \cdots O1W ⁱ	0.89 (2)	2.34 (2)	3.173 (2)	156 (2)
355	N4—H2N \cdots O1W ⁱⁱ	0.83 (2)	2.58 (2)	3.293 (2)	144.1 (19)
356	N5—H3N \cdots O3 ⁱⁱⁱ	0.86 (2)	2.01 (2)	2.7762 (18)	147.2 (18)
357	N5—H4N \cdots O2 ⁱⁱ	0.90 (2)	1.99 (2)	2.8488 (18)	159 (2)
358	N5—H4N \cdots N3 ⁱⁱ	0.90 (2)	2.51 (2)	3.038 (2)	118.1 (17)
359	N5—H5N \cdots O1 ^{iv}	0.93 (2)	2.44 (2)	3.1006 (18)	128.4 (17)
360	N5—H5N \cdots N1 ^{iv}	0.93 (2)	2.02 (2)	2.933 (2)	167.3 (19)
361	O3—H1H \cdots N2 ^v	0.84 (3)	1.90 (3)	2.7399 (19)	177 (2)
362	O1W—H1W \cdots O2	0.97 (3)	1.94 (3)	2.9039 (17)	174 (3)
363	O1W—H2W \cdots O1 ^{vi}	0.86 (3)	2.07 (3)	2.8997 (18)	163 (2)

364 Symmetry codes: (i) $-x, -y, -z+1$; (ii) $x, -y-1/2, z-1/2$; (iii) $-x+1, -y-1, -z+1$; (iv) $x, y-1, z$; (v) $-x+1, -y, -z+1$; (vi) $x, -y+1/2, z+1/2$.365 **Table 12**

366 Coordination modes of the SD anion, showing which atoms form bonds to alkali metal cations.

367	(I)	(II)	(III)	(III)
368	Li	Na	K L1	K L2
369 Donor atom				
370 O1	yes	yes	yes	yes
371 O2	no	no	no	yes
372 N sulfamide	no	yes	yes	yes
373 N ring 1	yes	yes	no	yes
374 N ring 2	no	yes	no	yes
375 N amine	no	yes	no	no
376				

377 **Acknowledgements**378 We thank the National Crystallography Service, University of Southampton, for data collection on (IV) (Coles & Gale,
379 2012).380 **References**

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checkCIF/PLATON results for paper uk3153

checkCIF/PLATON results

Ellipsoid plot

checkCIF/PLATON results

No syntax errors found. CIF dictionary Interpreting this report

Datablock: I

Bond precision: C-C = 0.0031 A Wavelength=0.71073
Cell: a=11.5095(4) b=12.0788(5) c=9.5184(4)
 alpha=90 beta=91.063(3) gamma=90
Temperature: 123 K

	Calculated	Reported
	-----	-----
Volume	1323.03(9)	1323.03(9)
Space group	C c	C c
Hall group	C -2yc	C -2yc
Moiety formula	C10 H13 Li N4 O4 S	?
Sum formula	C10 H13 Li N4 O4 S	C10 H13 Li N4 O4 S
Mr	292.24	292.24
Dx,g cm-3	1.467	1.467
Z	4	4
Mu (mm-1)	0.262	0.262
F000	608.0	608.0
F000'	608.76	
h,k,lmax	15,16,12	15,15,12
Nref	3512[1760]	2246
Tmin,Tmax	0.939,0.954	0.949,1.000
Tmin'	0.924	

Correction method= # Reported T Limits: Tmin=0.949 Tmax=1.000 AbsCorr = MULTI-SCAN
Data completeness= 1.28/0.64
Theta(max)= 28.976
R(reflections)= 0.0262(2184) wR2(reflections)= 0.0655(2246)
S = 1.036 Npar= 207

Alert level B

PLAT915_ALERT_3_B No Flack x Check Done: Low Friedel Pair Coverage 34 %

Alert level C

PLAT048_ALERT_1_C MoietyFormula Not Given (or Incomplete) Please Check
PLAT420_ALERT_2_C D-H Without Acceptor N4 --H2N Please Check

Alert level G

PLAT002_ALERT_2_G Number of Distance or Angle Restraints on AtSite 6 Note
PLAT172_ALERT_4_G The CIF-Embedded .res File Contains DFIX Records 2 Report
PLAT779_ALERT_4_G Suspect or Irrelevant (Bond) Angle in CIF # 18 Check
 O1 -S1 -LI1 1.555 1.555 1.555 32.38 Deg.
PLAT792_ALERT_1_G Model has Chirality at S1 (Polar SPGR) R Verify
PLAT860_ALERT_3_G Number of Least-Squares Restraints 8 Note
PLAT910_ALERT_3_G Missing # of FCF Reflection(s) Below Theta(Min). 1 Note
PLAT912_ALERT_4_G Missing # of FCF Reflections Above STh/L= 0.600 95 Note
PLAT978_ALERT_2_G Number C-C Bonds with Positive Residual Density. 10 Info

-
- 0 **ALERT level A** = Most likely a serious problem - resolve or explain
 - 1 **ALERT level B** = A potentially serious problem, consider carefully
 - 2 **ALERT level C** = Check. Ensure it is not caused by an omission or oversight
 - 8 **ALERT level G** = General information/check it is not something unexpected

- 2 ALERT type 1 CIF construction/syntax error, inconsistent or missing data
 - 3 ALERT type 2 Indicator that the structure model may be wrong or deficient
 - 3 ALERT type 3 Indicator that the structure quality may be low
 - 3 ALERT type 4 Improvement, methodology, query or suggestion
 - 0 ALERT type 5 Informative message, check
-

Datablock: II

Bond precision: C-C = 0.0030 A Wavelength=0.71073
 Cell: a=5.9010(11) b=10.534(4) c=18.389(3)
 alpha=90 beta=94.216(15) gamma=90
 Temperature: 123 K

	Calculated	Reported
	-----	-----
Volume	1140.0(5)	1140.0(5)
Space group	P 21/c	P 21/c
Hall group	-P 2ybc	-P 2ybc
Moiety formula	C10 H9 N4 Na O2 S	?
Sum formula	C10 H9 N4 Na O2 S	C10 H9 N4 Na O2 S
Mr	272.26	272.26
Dx,g cm-3	1.586	1.586
Z	4	4
Mu (mm-1)	0.320	0.320
F000	560.0	560.0
F000'	560.80	
h,k,lmax	8,14,25	7,14,24
Nref	3168	2786
Tmin,Tmax	0.955,0.975	0.961,1.000
Tmin'	0.894	

Correction method= # Reported T Limits: Tmin=0.961 Tmax=1.000 AbsCorr = MULTI-SCAN
 Data completeness= 0.879
 Theta(max)= 29.486
 R(reflections)= 0.0376(2351) wR2(reflections)= 0.0883(2786)
 S = 1.060 Npar= 171

Alert level C

PLAT048_ALERT_1_C MoietyFormula Not Given (or Incomplete)	Please Check
PLAT790_ALERT_4_C Centre of Gravity not Within Unit Cell: Resd. # C10 H9 N4 Na O2 S	1 Note
PLAT906_ALERT_3_C Large K Value in the Analysis of Variance	2.164 Check

Alert level G

PLAT004_ALERT_5_G Polymeric Structure Found with Maximum Dimension	3 Info
PLAT764_ALERT_4_G Overcomplete CIF Bond List Detected (Rep/Expd) .	1.33 Ratio
PLAT793_ALERT_4_G Model has Chirality at S1 (Centro SPGR)	S Verify
PLAT910_ALERT_3_G Missing # of FCF Reflection(s) Below Theta(Min).	3 Note
PLAT912_ALERT_4_G Missing # of FCF Reflections Above STh/L= 0.600	352 Note
PLAT978_ALERT_2_G Number C-C Bonds with Positive Residual Density.	10 Info

- 0 **ALERT level A** = Most likely a serious problem - resolve or explain
- 0 **ALERT level B** = A potentially serious problem, consider carefully

3 **ALERT level C** = Check. Ensure it is not caused by an omission or oversight

6 **ALERT level G** = General information/check it is not something unexpected

1 ALERT type 1 CIF construction/syntax error, inconsistent or missing data
1 ALERT type 2 Indicator that the structure model may be wrong or deficient
2 ALERT type 3 Indicator that the structure quality may be low
4 ALERT type 4 Improvement, methodology, query or suggestion
1 ALERT type 5 Informative message, check

Datablock: III

Bond precision: C-C = 0.0064 A Wavelength=1.54180
Cell: a=8.8503(7) b=9.6385(4) c=15.9734(7)
alpha=92.350(4) beta=95.186(4) gamma=94.209(4)
Temperature: 123 K

	Calculated	Reported
Volume	1351.79(14)	1351.79(13)
Space group	P -1	P -1
Hall group	-P 1	-P 1
Moiety formula	C20 H26 K2 N8 O8 S2	?
Sum formula	C20 H26 K2 N8 O8 S2	C10 H13 K N4 O4 S
Mr	648.81	324.40
Dx,g cm-3	1.594	1.594
Z	2	4
Mu (mm-1)	5.085	5.085
F000	672.0	672.0
F000'	676.80	
h,k,lmax	11,12,19	10,11,19
Nref	5456	5357
Tmin,Tmax	0.460,0.601	0.601,1.000
Tmin'	0.209	

Correction method= # Reported T Limits: Tmin=0.601 Tmax=1.000 AbsCorr = MULTI-SCAN
Data completeness= 0.982
Theta(max)= 73.745
R(reflections)= 0.0597(3951) wR2(reflections)= 0.1573(5357)
S = 1.045 Npar= 404

Alert level B

PLAT420_ALERT_2_B D-H Without Acceptor O4W --H9W Please Check

Alert level C

PLAT048_ALERT_1_C MoietyFormula Not Given (or Incomplete) Please Check
PLAT242_ALERT_2_C Low 'MainMol' Ueq as Compared to Neighbors of K1 Check
PLAT340_ALERT_3_C Low Bond Precision on C-C Bonds 0.00644 Ang.
PLAT420_ALERT_2_C D-H Without Acceptor N8 --H4N Please Check
PLAT906_ALERT_3_C Large K Value in the Analysis of Variance 6.059 Check
PLAT911_ALERT_3_C Missing FCF Refl Between Thmin & STh/L= 0.600 19 Report
PLAT934_ALERT_3_C Number of (Iobs-Icalc)/SigmaW > 10 Outliers 1 Check
PLAT978_ALERT_2_C Number C-C Bonds with Positive Residual Density. 0 Info

Alert level G

PLAT002_ALERT_2_G Number of Distance or Angle Restraints on AtSite 13 Note
PLAT004_ALERT_5_G Polymeric Structure Found with Maximum Dimension 2 Info
PLAT045_ALERT_1_G Calculated and Reported Z Differ by a Factor ... 0.50 Check
PLAT154_ALERT_1_G The s.u.'s on the Cell Angles are Equal ..(Note) 0.004 Degree
PLAT172_ALERT_4_G The CIF-Embedded .res File Contains DFIX Records 3 Report

PLAT300_ALERT_4_G	Atom Site Occupancy of H8W	Constrained at	0.5	Check
PLAT300_ALERT_4_G	Atom Site Occupancy of H9W	Constrained at	0.5	Check
PLAT764_ALERT_4_G	Overcomplete CIF Bond List Detected (Rep/Expd)	.	1.29	Ratio
PLAT774_ALERT_1_G	Suspect X-Y Bond in CIF: K1	--K2 ..	4.24	Ang.
PLAT774_ALERT_1_G	Suspect X-Y Bond in CIF: K1	--K1 ..	4.49	Ang.
PLAT774_ALERT_1_G	Suspect X-Y Bond in CIF: K1	--K2 ..	4.62	Ang.
PLAT774_ALERT_1_G	Suspect X-Y Bond in CIF: K2	--K1 ..	4.24	Ang.
PLAT793_ALERT_4_G	Model has Chirality at S1	(Centro SPGR)		R Verify
PLAT860_ALERT_3_G	Number of Least-Squares Restraints	14	Note
PLAT910_ALERT_3_G	Missing # of FCF Reflection(s) Below Theta(Min).		1	Note
PLAT912_ALERT_4_G	Missing # of FCF Reflections Above STh/L=	0.600	75	Note

0 **ALERT level A** = Most likely a serious problem - resolve or explain
1 **ALERT level B** = A potentially serious problem, consider carefully
8 **ALERT level C** = Check. Ensure it is not caused by an omission or oversight
16 **ALERT level G** = General information/check it is not something unexpected

7 ALERT type 1 CIF construction/syntax error, inconsistent or missing data
5 ALERT type 2 Indicator that the structure model may be wrong or deficient
6 ALERT type 3 Indicator that the structure quality may be low
6 ALERT type 4 Improvement, methodology, query or suggestion
1 ALERT type 5 Informative message, check


Datablock: IV

Bond precision: C-C = 0.0020 A Wavelength=1.54184
Cell: a=12.7688(1) b=16.9040(1) c=15.7411(1)
alpha=90 beta=107.609(1) gamma=90
Temperature: 100 K

	Calculated	Reported
	-----	-----
Volume	3238.42(4)	3238.42(4)
Space group	P 21/c	P 21/c
Hall group	-P 2ybc	-P 2ybc
Moiety formula	C32 H36 N4 Na2 O22 S4 ?	
Sum formula	C52 H64 N12 Na2 O29 S6	C26 H32 N6 Na O14.50 S3
Mr	1559.49	779.74
Dx,g cm-3	1.599	1.599
Z	2	4
Mu (mm-1)	2.946	2.946
F000	1620.0	1620.0
F000'	1629.77	
h,k,lmax	15,20,18	15,20,18
Nref	5935	5913
Tmin,Tmax	0.754,0.889	0.615,1.000
Tmin'	0.702	

Correction method= # Reported T Limits: Tmin=0.615 Tmax=1.000 AbsCorr = MULTI-SCAN
Data completeness= 0.996
Theta(max)= 68.245
R(reflections)= 0.0280(5686) wR2(reflections)= 0.0745(5913)
S = 1.048 Npar= 537

 **Alert level B**
PLAT416_ALERT_2_B Short Intra D-H..H-D H11W ..H13W 1.38 Ang.

 **Alert level C**
PLAT048_ALERT_1_C MoietyFormula Not Given (or Incomplete) Please Check

PLAT222_ALERT_3_C Non-Solv. Resd 1 H Uiso(max)/Uiso(min) Range 4.2 Ratio
 PLAT911_ALERT_3_C Missing FCF Refl Between Thmin & STh/L= 0.600 19 Report

Alert level G

PLAT002_ALERT_2_G Number of Distance or Angle Restraints on AtSite 12 Note
 PLAT045_ALERT_1_G Calculated and Reported Z Differ by a Factor ... 0.50 Check
 PLAT142_ALERT_4_G s.u. on b - Axis Small or Missing 0.00010 Ang.
 PLAT143_ALERT_4_G s.u. on c - Axis Small or Missing 0.00010 Ang.
 PLAT172_ALERT_4_G The CIF-Embedded .res File Contains DFIX Records 3 Report
 PLAT300_ALERT_4_G Atom Site Occupancy of H8W Constrained at 0.5 Check
 PLAT300_ALERT_4_G Atom Site Occupancy of H14W Constrained at 0.5 Check
 PLAT300_ALERT_4_G Atom Site Occupancy of O6W Constrained at 0.5 Check
 PLAT300_ALERT_4_G Atom Site Occupancy of O7W Constrained at 0.5 Check
 PLAT300_ALERT_4_G Atom Site Occupancy of H12W Constrained at 0.5 Check
 PLAT300_ALERT_4_G Atom Site Occupancy of H13W Constrained at 0.5 Check
 PLAT300_ALERT_4_G Atom Site Occupancy of O5W Constrained at 0.5 Check
 PLAT300_ALERT_4_G Atom Site Occupancy of H9W Constrained at 0.5 Check
 PLAT300_ALERT_4_G Atom Site Occupancy of H10W Constrained at 0.5 Check
 PLAT302_ALERT_4_G Anion/Solvent/Minor-Residue Disorder (Resd 3) 100% Note
 PLAT302_ALERT_4_G Anion/Solvent/Minor-Residue Disorder (Resd 4) 100% Note
 PLAT304_ALERT_4_G Non-Integer Number of Atoms in Resd 4 1.50 Check
 PLAT790_ALERT_4_G Centre of Gravity not Within Unit Cell: Resd. # 3 Note
 H2 O
 PLAT790_ALERT_4_G Centre of Gravity not Within Unit Cell: Resd. # 4 Note
 H2 O
 PLAT860_ALERT_3_G Number of Least-Squares Restraints 13 Note
 PLAT912_ALERT_4_G Missing # of FCF Reflections Above STh/L= 0.600 3 Note
 PLAT933_ALERT_2_G Number of OMIT Records in Embedded .res File ... 2 Note
 PLAT978_ALERT_2_G Number C-C Bonds with Positive Residual Density. 18 Info

- 0 **ALERT level A** = Most likely a serious problem - resolve or explain
- 1 **ALERT level B** = A potentially serious problem, consider carefully
- 3 **ALERT level C** = Check. Ensure it is not caused by an omission or oversight
- 23 **ALERT level G** = General information/check it is not something unexpected

- 2 ALERT type 1 CIF construction/syntax error, inconsistent or missing data
- 4 ALERT type 2 Indicator that the structure model may be wrong or deficient
- 3 ALERT type 3 Indicator that the structure quality may be low
- 18 ALERT type 4 Improvement, methodology, query or suggestion
- 0 ALERT type 5 Informative message, check

Datablock: V

Bond precision: C-C = 0.0028 A Wavelength=0.71073
 Cell: a=8.5796(2) b=19.0371(5) c=11.2512(4)
 alpha=90 beta=121.116(3) gamma=90
 Temperature: 123 K

	Calculated	Reported
	-----	-----
Volume	1573.27(9)	1573.27(9)
Space group	P 21/c	P 21/c
Hall group	-P 2ybc	-P 2ybc
Moiety formula	C10 H9 N4 O2 S, C5 H7 N2	?
Sum formula	C15 H16 N6 O2 S	C15 H16 N6 O2 S
Mr	344.40	344.40
Dx,g cm-3	1.454	1.454
Z	4	4
Mu (mm-1)	0.228	0.228
F000	720.0	720.0
F000'	720.74	
h,k,lmax	11,25,15	11,25,15
Nref	4183	4082
Tmin,Tmax	0.921,0.995	0.527,1.000

Tmin' 0.913

Correction method= # Reported T Limits: Tmin=0.527 Tmax=1.000 AbsCorr = MULTI-SCAN
Data completeness= 0.976
Theta(max)= 28.994
R(reflections)= 0.0445(3225) wR2(reflections)= 0.1139(4082)
S = 1.040 Npar= 237

Alert level C

PLAT048_ALERT_1_C MoietyFormula Not Given (or Incomplete) Please Check
PLAT906_ALERT_3_C Large K Value in the Analysis of Variance 2.533 Check

Alert level G

PLAT128_ALERT_4_G Alternate Setting for Input Space Group P21/c P21/n Note
PLAT790_ALERT_4_G Centre of Gravity not Within Unit Cell: Resd. # 2 Note
C5 H7 N2
PLAT910_ALERT_3_G Missing # of FCF Reflection(s) Below Theta(Min). 4 Note
PLAT912_ALERT_4_G Missing # of FCF Reflections Above STh/L= 0.600 98 Note
PLAT978_ALERT_2_G Number C-C Bonds with Positive Residual Density. 11 Info

-
- 0 **ALERT level A** = Most likely a serious problem - resolve or explain
0 **ALERT level B** = A potentially serious problem, consider carefully
2 **ALERT level C** = Check. Ensure it is not caused by an omission or oversight
5 **ALERT level G** = General information/check it is not something unexpected
- 1 ALERT type 1 CIF construction/syntax error, inconsistent or missing data
1 ALERT type 2 Indicator that the structure model may be wrong or deficient
2 ALERT type 3 Indicator that the structure quality may be low
3 ALERT type 4 Improvement, methodology, query or suggestion
0 ALERT type 5 Informative message, check

Datablock: VI

Bond precision: C-C = 0.0020 A Wavelength=0.71073
Cell: a=12.7755(4) b=9.8979(4) c=11.5366(4)
alpha=90 beta=93.508(3) gamma=90
Temperature: 123 K

	Calculated	Reported
	-----	-----
Volume	1456.08(9)	1456.08(9)
Space group	P 21/c	P 21/c
Hall group	-P 2ybc	-P 2ybc
Moiety formula	C10 H9 N4 O2 S, C2 H8 ?	
Sum formula	C12 H19 N5 O4 S	C12 H19 N5 O4 S
Mr	329.38	329.38
Dx,g cm-3	1.503	1.503
Z	4	4
Mu (mm-1)	0.250	0.250
F000	696.0	696.0
F000'	696.79	
h,k,lmax	17,13,16	17,12,15
Nref	4052	3571
Tmin,Tmax	0.939,0.970	0.904,1.000
Tmin'	0.939	

Correction method= # Reported T Limits: Tmin=0.904 Tmax=1.000 AbsCorr = MULTI-SCAN
Data completeness= 0.881
Theta(max)= 29.531

R(reflections)= 0.0366(2942) wR2(reflections)= 0.0953(3571)
S = 1.058 Npar= 231



Alert level C

PLAT048_ALERT_1_C MoietyFormula Not Given (or Incomplete) Please Check



Alert level G

PLAT790_ALERT_4_G Centre of Gravity not Within Unit Cell: Resd. # 2 Note
C2 H8 N O
PLAT910_ALERT_3_G Missing # of FCF Reflection(s) Below Theta(Min). 4 Note
PLAT912_ALERT_4_G Missing # of FCF Reflections Above STh/L= 0.600 432 Note
PLAT978_ALERT_2_G Number C-C Bonds with Positive Residual Density. 9 Info

-
- 0 **ALERT level A** = Most likely a serious problem - resolve or explain
0 **ALERT level B** = A potentially serious problem, consider carefully
1 **ALERT level C** = Check. Ensure it is not caused by an omission or oversight
4 **ALERT level G** = General information/check it is not something unexpected
- 1 ALERT type 1 CIF construction/syntax error, inconsistent or missing data
1 ALERT type 2 Indicator that the structure model may be wrong or deficient
1 ALERT type 3 Indicator that the structure quality may be low
2 ALERT type 4 Improvement, methodology, query or suggestion
0 ALERT type 5 Informative message, check
-

database duplication summary

Datablock: I

- **Chemical name =**
- *R* factor = 0.026
- Space group =
- Formula = C10 H13 Li N4 O4 S
- a=11.5095 b=12.0788 c=9.5184
- alpha=90 beta=91.063 gamma=90

Datablock: II

- **Chemical name =**
- *R* factor = 0.038
- Space group =
- Formula = C10 H9 N4 Na O2 S
- a=5.901 b=10.534 c=18.389
- alpha=90 beta=94.216 gamma=90

Datablock: III

- **Chemical name =**
- *R* factor = 0.060

- Space group =
- Formula = C10 H13 K N4 O4 S
- a=8.8503 b=9.6385 c=15.9734
- alpha=92.35 beta=95.186 gamma=94.209

Datablock: IV

- **Chemical name =**
- R factor = 0.028
- Space group =
- Formula = C26 H32 N6 Na O14.50 S3
- a=12.7688 b=16.904 c=15.7411
- alpha=90 beta=107.609 gamma=90

Datablock: V

- **Chemical name =**
- R factor = 0.045
- Space group =
- Formula = C15 H16 N6 O2 S
- a=8.5796 b=19.0371 c=11.2512
- alpha=90 beta=121.116 gamma=90

Datablock: VI

- **Chemical name =**
- R factor = 0.037
- Space group =
- Formula = C12 H19 N5 O4 S
- a=12.7755 b=9.8979 c=11.5366
- alpha=90 beta=93.508 gamma=90

No duplication found.

reference checking results

The following references were not checked in detail as they were not recognized as journal references

Agilent (2014). CrysAlisPro. Agilent Technologies Ltd., Yarnton, Oxfordshire, England.

Stahl, P. H. & Wermuth, C. G. (2008). Eds. *Handbook of Pharmaceutical Salts: Properties, Selection and Use*. VHCA: Zurich.

The following references may be incorrectly formatted

Ghedini, E., Pizzolitto, C., Albore, G., Menegazzo, F., Signoretto, M., Operti, L. & Cerrato, G. (2017). *J. Sol-Gel Sci. Tech.* **83**, 618--626.

[Unrecognized journal title.]

Preskey, D. & Kayes, J. B. (1976). *J. Clinical Pharm.* **1**, 39--48.
[Unrecognized journal title.]

Results of online verification of references to IUCr journals

More than one match found for Altomare, A., Cascarano, G., Giacobazzo, C., Guagliardi, A., Burla, M. C., Polidori, G. & Camalli, M. (1994). *J. Appl. Cryst.* **27**, 435--436. #ATL:SIRPOW.92 - a program for automatic solution of crystal structures by direct methods optimized for powder data. [CNOR:wi0150]# - please check.

All references appear to be cited unambiguously

Citation comments

1 date found in data_I_exptl_absorpt_process_details that could be part of a citation but not found in reference list: 2010

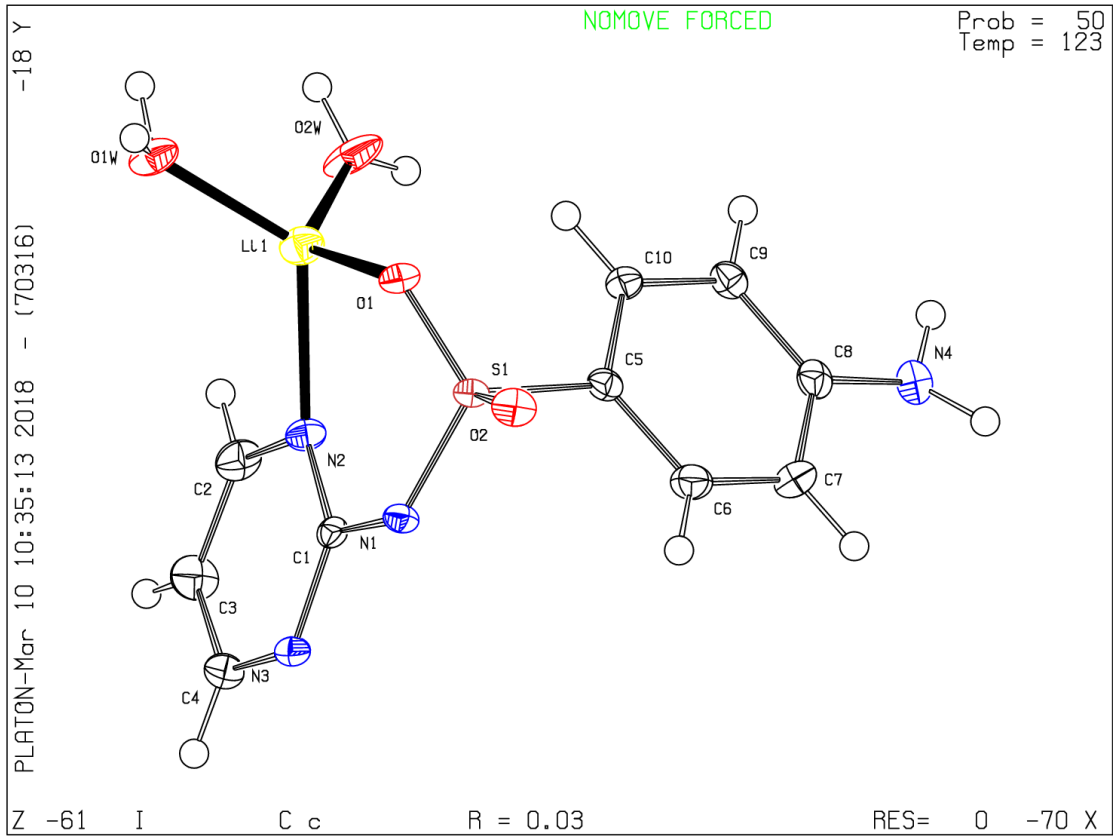
1 date found in data_II_exptl_absorpt_process_details that could be part of a citation but not found in reference list: 2010

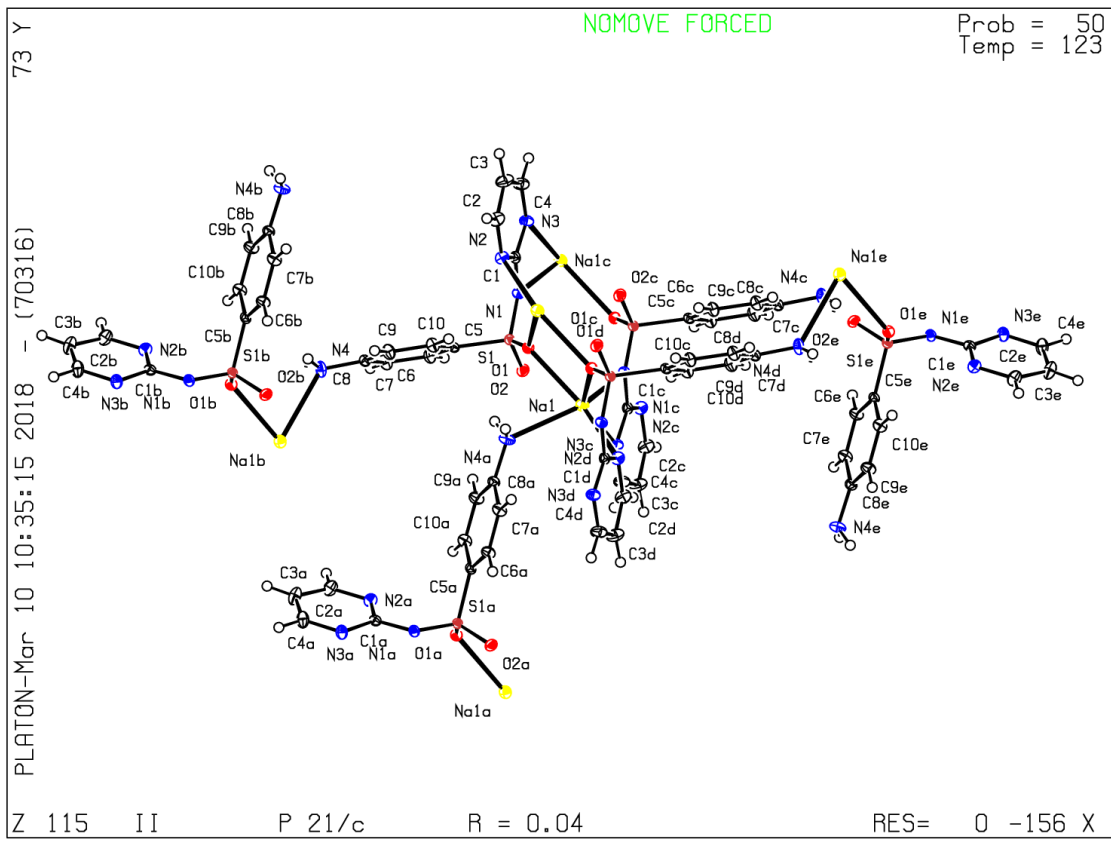
1 date found in data_III_exptl_absorpt_process_details that could be part of a citation but not found in reference list: 2014

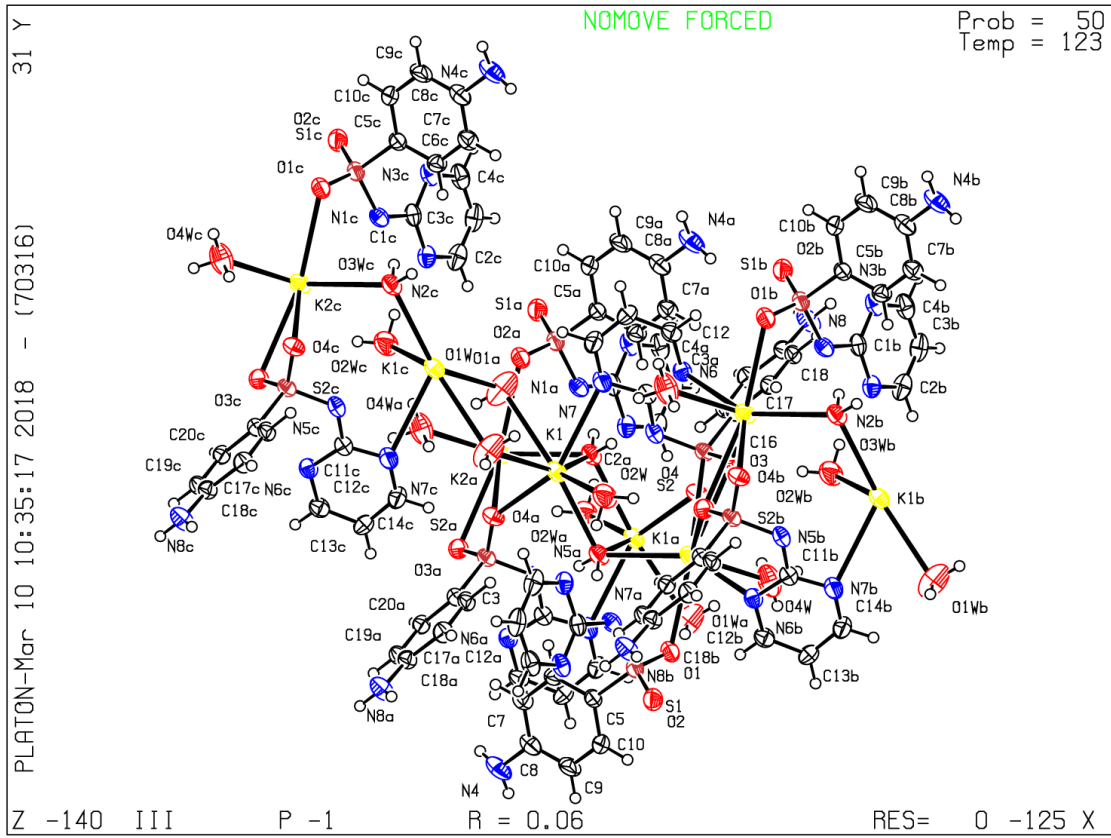
1 date found in data_IV_exptl_absorpt_process_details that could be part of a citation but not found in reference list: 2017

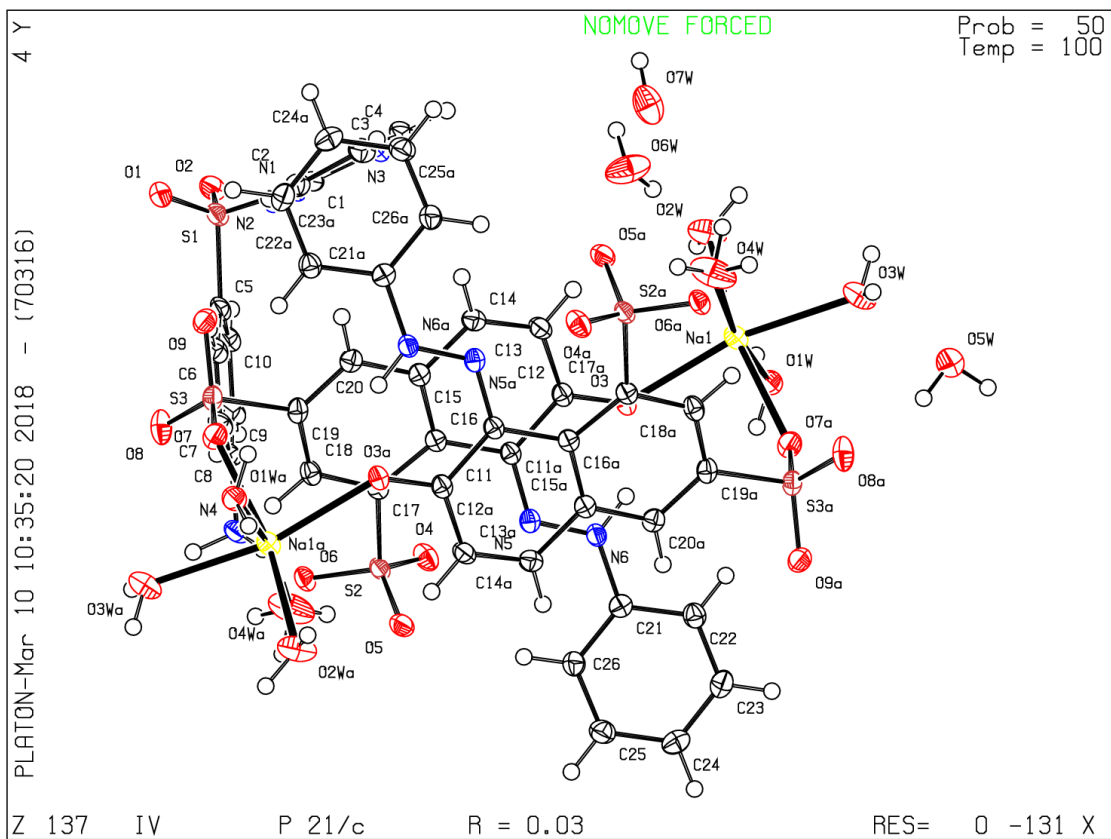
1 date found in data_V_exptl_absorpt_process_details that could be part of a citation but not found in reference list: 2010

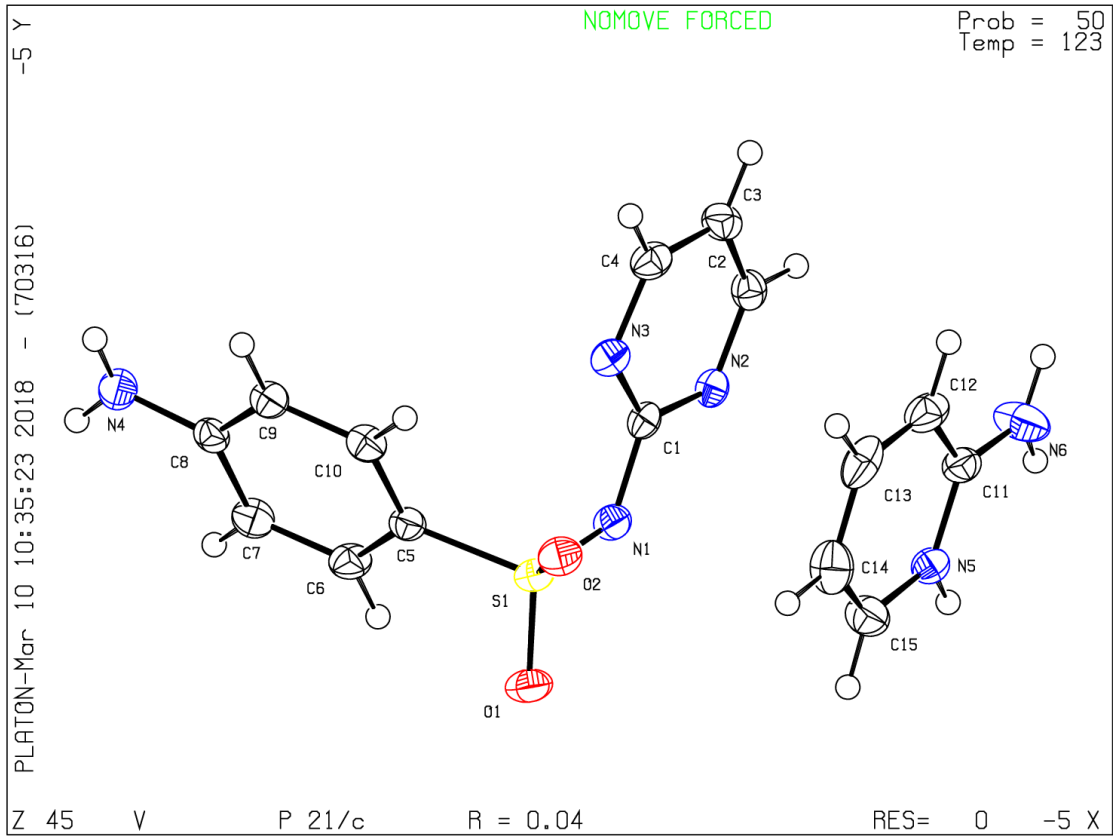
1 date found in data_VI_exptl_absorpt_process_details that could be part of a citation but not found in reference list: 2010

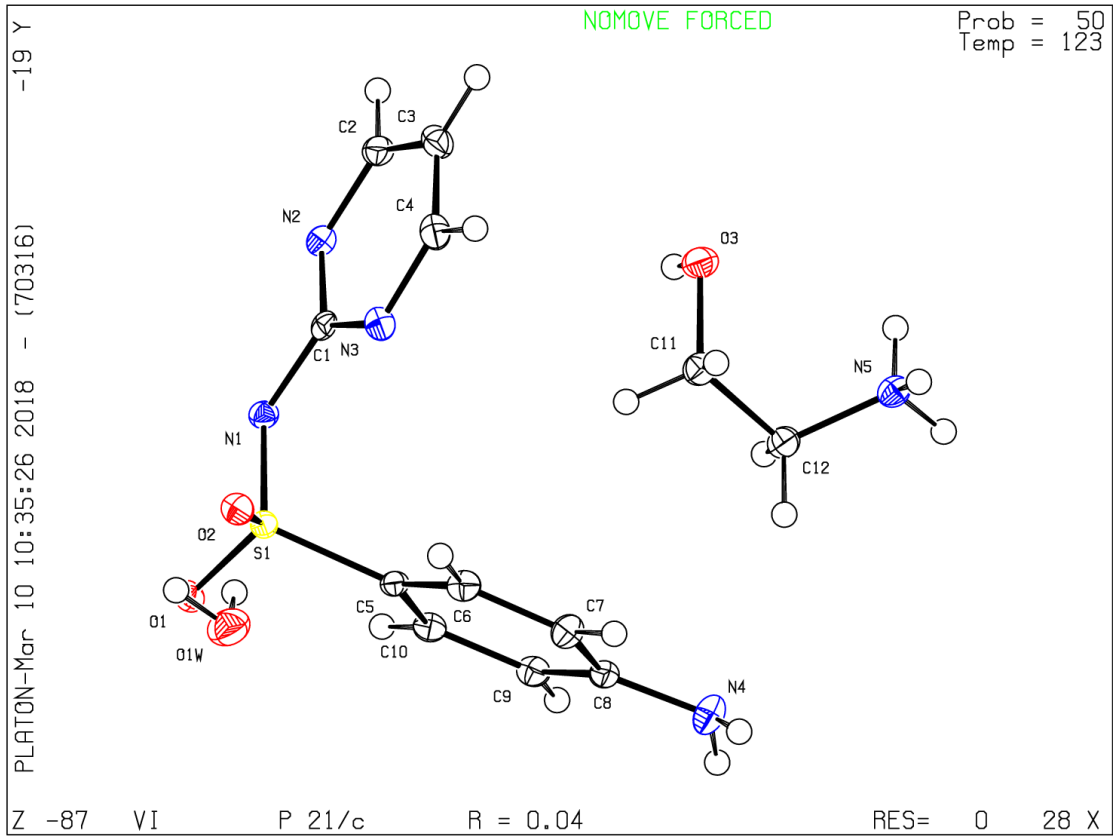












1 supporting information

2 Salt forms of sulfadiazine with alkali metal and organic cations.

3 **Gemma Campbell, Rebecca Fisher, Alan R. Kennedy,* Nathan L. C. King and Rebecca Spiteri**

4 Computing details

5 For all structures, data collection: *CrysAlis PRO* (Agilent, 2014); cell refinement: *CrysAlis PRO*; data reduction: *CrysAlis*
 6 *PRO*. Program(s) used to solve structure: *SIR92* (Altomare *et al.*, 1994) for (I), (III), (IV); *SHELXS* (Sheldrick, 2015) for
 7 (II), (V), (VI). For all structures, program(s) used to refine structure: *SHELXL2014* (Sheldrick, 2015); molecular
 8 graphics: *Mercury* (Macrae *et al.*, 2008); software used to prepare material for publication: *SHELXL2014*.

9 (I)

10 *Crystal data*

11	$C_{10}H_{13}LiN_4O_4S$	$F(000) = 608$
12	$M_r = 292.24$	$D_x = 1.467 \text{ Mg m}^{-3}$
13	Monoclinic, <i>Cc</i>	Mo $K\alpha$ radiation, $\lambda = 0.71073 \text{ \AA}$
14	$a = 11.5095 (4) \text{ \AA}$	Cell parameters from 2629 reflections
15	$b = 12.0788 (5) \text{ \AA}$	$\theta = 3.2\text{--}29.8^\circ$
16	$c = 9.5184 (4) \text{ \AA}$	$\mu = 0.26 \text{ mm}^{-1}$
17	$\beta = 91.063 (3)^\circ$	$T = 123 \text{ K}$
18	$V = 1323.03 (9) \text{ \AA}^3$	Block, colourless
19	$Z = 4$	$0.30 \times 0.20 \times 0.18 \text{ mm}$

20 *Data collection*

21	Oxford Diffraction Xcalibur E	Absorption correction: multi-scan
	diffractometer	<i>CrysAlis PRO</i> , Oxford Diffraction Ltd., Version
22	Radiation source: tube	1.171.34.40 (release 27-08-2010 CrysAlis171
23	ω scans	.NET) (compiled Aug 27 2010,11:50:40)
		Empirical absorption correction using spherical
		harmonics, implemented in SCALE3
		ABSPACK scaling algorithm.
		$T_{\min} = 0.949$, $T_{\max} = 1.000$
		3390 measured reflections
		2246 independent reflections
		2184 reflections with $I > 2\sigma(I)$
		$R_{\text{int}} = 0.016$
		$\theta_{\max} = 29.0^\circ$, $\theta_{\min} = 3.2^\circ$
		$h = -15 \rightarrow 13$
		$k = -15 \rightarrow 15$
		$l = -12 \rightarrow 10$

24 *Refinement*

25	Refinement on F^2	2246 reflections
26	Least-squares matrix: full	207 parameters
27	$R[F^2 > 2\sigma(F^2)] = 0.026$	8 restraints
28	$wR(F^2) = 0.066$	Hydrogen site location: mixed
29	$S = 1.04$	

- 30 H atoms treated by a mixture of independent
and constrained refinement
31 $w = 1/[\sigma^2(F_o^2) + (0.0365P)^2 + 0.3087P]$
where $P = (F_o^2 + 2F_c^2)/3$
32 $(\Delta/\sigma)_{\max} < 0.001$
33 $\Delta\rho_{\max} = 0.28 \text{ e } \text{\AA}^{-3}$
34 $\Delta\rho_{\min} = -0.25 \text{ e } \text{\AA}^{-3}$
- Extinction correction: *SHELXL2014* (Sheldrick, 2015), $F_c^* = kF_c[1 + 0.001x F_c^2 \lambda^3 / \sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.0054 (9)
Absolute structure: Refined as an inversion twin.
Absolute structure parameter: -0.01 (8)

35 *Special details*

- 36 **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.

- 37 **Refinement.** Refined as a 2-component inversion twin.

38 *Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (\AA^2)*

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$	
39					
40	Li1	0.8416 (4)	0.2755 (4)	0.4134 (5)	0.0198 (8)
41	S1	0.75432 (4)	0.33089 (4)	0.70030 (5)	0.01208 (13)
42	O1	0.81653 (14)	0.36761 (14)	0.57624 (17)	0.0157 (3)
43	O2	0.72513 (14)	0.42112 (14)	0.79429 (18)	0.0175 (4)
44	O1W	0.83115 (15)	0.36603 (15)	0.24923 (19)	0.0211 (4)
45	O2W	0.97330 (16)	0.19039 (18)	0.3764 (2)	0.0314 (5)
46	N1	0.63739 (15)	0.26680 (16)	0.6691 (2)	0.0129 (4)
47	N2	0.71240 (17)	0.16691 (16)	0.4758 (2)	0.0154 (4)
48	N3	0.52506 (17)	0.13140 (17)	0.5695 (2)	0.0145 (4)
49	N4	1.05266 (19)	0.00920 (18)	1.0096 (2)	0.0187 (4)
50	C1	0.62734 (19)	0.18727 (19)	0.5688 (2)	0.0121 (4)
51	C2	0.6930 (2)	0.0854 (2)	0.3827 (3)	0.0206 (5)
52	H2	0.7523	0.0684	0.3180	0.025*
53	C3	0.5921 (2)	0.0254 (2)	0.3763 (3)	0.0231 (5)
54	H3	0.5798	-0.0317	0.3091	0.028*
55	C4	0.5093 (2)	0.0531 (2)	0.4735 (3)	0.0184 (5)
56	H4	0.4376	0.0141	0.4714	0.022*
57	C5	0.84694 (19)	0.23972 (19)	0.7933 (2)	0.0137 (4)
58	C6	0.8089 (2)	0.1949 (2)	0.9188 (3)	0.0189 (5)
59	H6	0.7352	0.2151	0.9541	0.023*
60	C7	0.8785 (2)	0.1211 (2)	0.9918 (2)	0.0198 (5)
61	H7	0.8523	0.0904	1.0775	0.024*
62	C8	0.9871 (2)	0.09088 (19)	0.9410 (2)	0.0161 (5)
63	C9	1.0257 (2)	0.1391 (2)	0.8169 (2)	0.0157 (5)
64	H9	1.1005	0.1211	0.7830	0.019*
65	C10	0.95556 (18)	0.21289 (19)	0.7430 (2)	0.0154 (5)
66	H10	0.9819	0.2450	0.6582	0.018*
67	H1W	0.815 (3)	0.4368 (12)	0.251 (4)	0.050 (11)*
68	H2W	0.890 (2)	0.365 (3)	0.194 (3)	0.055 (12)*
69	H3W	1.017 (3)	0.202 (3)	0.305 (3)	0.063 (12)*
70	H4W	1.002 (3)	0.131 (2)	0.413 (4)	0.061 (12)*

71	H1N	1.122 (3)	0.003 (3)	0.985 (3)	0.025 (8)*
72	H2N	1.044 (2)	0.012 (2)	1.107 (3)	0.017 (7)*

73 *Atomic displacement parameters (\AA^2)*

74		U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
75	Li1	0.019 (2)	0.019 (2)	0.022 (2)	0.0013 (16)	0.0043 (15)	0.0013 (17)
76	S1	0.0108 (2)	0.0113 (2)	0.0143 (2)	-0.0013 (2)	0.00227 (16)	-0.0006 (2)
77	O1	0.0157 (8)	0.0145 (8)	0.0171 (8)	-0.0029 (6)	0.0045 (6)	0.0013 (7)
78	O2	0.0178 (8)	0.0133 (8)	0.0213 (9)	0.0001 (6)	0.0032 (6)	-0.0042 (7)
79	O1W	0.0208 (9)	0.0181 (9)	0.0246 (9)	0.0048 (7)	0.0098 (7)	0.0059 (8)
80	O2W	0.0274 (11)	0.0332 (11)	0.0345 (12)	0.0151 (8)	0.0209 (9)	0.0194 (9)
81	N1	0.0100 (9)	0.0144 (9)	0.0145 (9)	-0.0011 (7)	0.0020 (6)	-0.0011 (8)
82	N2	0.0140 (10)	0.0165 (10)	0.0160 (10)	0.0001 (8)	0.0049 (7)	-0.0007 (8)
83	N3	0.0135 (9)	0.0149 (10)	0.0149 (9)	-0.0035 (8)	0.0013 (7)	0.0015 (8)
84	N4	0.0194 (11)	0.0187 (11)	0.0180 (11)	0.0018 (8)	-0.0032 (8)	-0.0010 (8)
85	C1	0.0126 (10)	0.0117 (10)	0.0120 (11)	0.0003 (8)	0.0004 (7)	0.0030 (8)
86	C2	0.0230 (12)	0.0219 (12)	0.0172 (12)	0.0010 (10)	0.0071 (9)	-0.0048 (10)
87	C3	0.0262 (13)	0.0218 (12)	0.0214 (13)	-0.0044 (10)	0.0025 (9)	-0.0108 (11)
88	C4	0.0171 (11)	0.0182 (12)	0.0198 (12)	-0.0042 (9)	-0.0005 (8)	0.0008 (10)
89	C5	0.0127 (10)	0.0133 (11)	0.0150 (11)	-0.0018 (8)	0.0001 (8)	-0.0019 (9)
90	C6	0.0157 (11)	0.0212 (12)	0.0200 (13)	-0.0016 (10)	0.0029 (9)	0.0004 (10)
91	C7	0.0215 (12)	0.0208 (12)	0.0174 (12)	-0.0014 (10)	0.0049 (9)	0.0030 (10)
92	C8	0.0184 (11)	0.0142 (11)	0.0157 (12)	-0.0026 (9)	-0.0030 (8)	-0.0031 (9)
93	C9	0.0129 (11)	0.0176 (12)	0.0167 (11)	0.0018 (9)	0.0004 (8)	-0.0036 (9)
94	C10	0.0139 (11)	0.0177 (12)	0.0145 (11)	-0.0003 (9)	0.0027 (8)	0.0000 (9)

95 *Geometric parameters (\AA , $^\circ$)*

96	Li1—O2W	1.870 (4)	N4—C8	1.396 (3)
97	Li1—O1W	1.910 (5)	N4—H1N	0.84 (3)
98	Li1—O1	1.934 (5)	N4—H2N	0.93 (3)
99	Li1—N2	2.077 (5)	C2—C3	1.370 (4)
100	Li1—S1	3.002 (4)	C2—H2	0.9500
101	S1—O2	1.4533 (17)	C3—C4	1.382 (3)
102	S1—O1	1.4612 (16)	C3—H3	0.9500
103	S1—N1	1.5761 (19)	C4—H4	0.9500
104	S1—C5	1.760 (2)	C5—C10	1.385 (3)
105	O1W—H1W	0.875 (12)	C5—C6	1.390 (3)
106	O1W—H2W	0.867 (12)	C6—C7	1.378 (3)
107	O2W—H3W	0.865 (12)	C6—H6	0.9500
108	O2W—H4W	0.863 (12)	C7—C8	1.398 (3)
109	N1—C1	1.357 (3)	C7—H7	0.9500
110	N2—C2	1.340 (3)	C8—C9	1.397 (3)
111	N2—C1	1.355 (3)	C9—C10	1.386 (3)
112	N3—C4	1.325 (3)	C9—H9	0.9500
113	N3—C1	1.357 (3)	C10—H10	0.9500

114

115	O2W—Li1—O1W	101.5 (2)	C8—N4—H2N	111.7 (17)
116	O2W—Li1—O1	126.9 (2)	H1N—N4—H2N	113 (3)
117	O1W—Li1—O1	108.5 (2)	N2—C1—N3	123.4 (2)
118	O2W—Li1—N2	107.1 (2)	N2—C1—N1	122.3 (2)
119	O1W—Li1—N2	124.1 (2)	N3—C1—N1	114.3 (2)
120	O1—Li1—N2	90.91 (18)	N2—C2—C3	123.3 (2)
121	O2W—Li1—S1	125.5 (2)	N2—C2—H2	118.4
122	O1W—Li1—S1	126.84 (19)	C3—C2—H2	118.4
123	O1—Li1—S1	23.87 (8)	C2—C3—C4	115.9 (2)
124	N2—Li1—S1	68.13 (13)	C2—C3—H3	122.0
125	O2—S1—O1	113.09 (10)	C4—C3—H3	122.0
126	O2—S1—N1	106.15 (10)	N3—C4—C3	123.2 (2)
127	O1—S1—N1	115.22 (10)	N3—C4—H4	118.4
128	O2—S1—C5	107.73 (10)	C3—C4—H4	118.4
129	O1—S1—C5	107.16 (10)	C10—C5—C6	120.4 (2)
130	N1—S1—C5	107.14 (10)	C10—C5—S1	120.99 (18)
131	O2—S1—Li1	144.30 (11)	C6—C5—S1	118.58 (18)
132	O1—S1—Li1	32.38 (11)	C7—C6—C5	119.7 (2)
133	N1—S1—Li1	91.02 (11)	C7—C6—H6	120.1
134	C5—S1—Li1	96.20 (11)	C5—C6—H6	120.1
135	S1—O1—Li1	123.74 (16)	C6—C7—C8	120.7 (2)
136	Li1—O1W—H1W	124 (2)	C6—C7—H7	119.7
137	Li1—O1W—H2W	117 (2)	C8—C7—H7	119.7
138	H1W—O1W—H2W	101 (2)	N4—C8—C9	120.8 (2)
139	Li1—O2W—H3W	123 (3)	N4—C8—C7	120.1 (2)
140	Li1—O2W—H4W	134 (3)	C9—C8—C7	119.0 (2)
141	H3W—O2W—H4W	104 (2)	C10—C9—C8	120.4 (2)
142	C1—N1—S1	122.79 (16)	C10—C9—H9	119.8
143	C2—N2—C1	116.9 (2)	C8—C9—H9	119.8
144	C2—N2—Li1	112.7 (2)	C5—C10—C9	119.7 (2)
145	C1—N2—Li1	127.0 (2)	C5—C10—H10	120.1
146	C4—N3—C1	117.3 (2)	C9—C10—H10	120.1
147	C8—N4—H1N	116 (2)		
148				
149	O2—S1—O1—Li1	-167.45 (18)	C2—C3—C4—N3	-1.0 (4)
150	N1—S1—O1—Li1	-45.1 (2)	O2—S1—C5—C10	-123.58 (19)
151	C5—S1—O1—Li1	74.0 (2)	O1—S1—C5—C10	-1.6 (2)
152	O2—S1—N1—C1	169.98 (18)	N1—S1—C5—C10	122.58 (19)
153	O1—S1—N1—C1	44.0 (2)	Li1—S1—C5—C10	29.6 (2)
154	C5—S1—N1—C1	-75.1 (2)	O2—S1—C5—C6	56.4 (2)
155	Li1—S1—N1—C1	21.7 (2)	O1—S1—C5—C6	178.37 (18)
156	C2—N2—C1—N3	-1.4 (3)	N1—S1—C5—C6	-57.4 (2)
157	Li1—N2—C1—N3	156.1 (2)	Li1—S1—C5—C6	-150.4 (2)
158	C2—N2—C1—N1	179.3 (2)	C10—C5—C6—C7	-1.7 (4)
159	Li1—N2—C1—N1	-23.3 (4)	S1—C5—C6—C7	178.34 (19)
160	C4—N3—C1—N2	0.1 (3)	C5—C6—C7—C8	0.1 (4)
161	C4—N3—C1—N1	179.5 (2)	C6—C7—C8—N4	-174.7 (2)
162	S1—N1—C1—N2	-8.8 (3)	C6—C7—C8—C9	1.8 (4)

163	S1—N1—C1—N3	171.78 (16)	N4—C8—C9—C10	174.3 (2)
164	C1—N2—C2—C3	1.5 (4)	C7—C8—C9—C10	-2.1 (3)
165	Li1—N2—C2—C3	-159.1 (3)	C6—C5—C10—C9	1.4 (3)
166	N2—C2—C3—C4	-0.4 (4)	S1—C5—C10—C9	-178.66 (17)
167	C1—N3—C4—C3	1.2 (4)	C8—C9—C10—C5	0.5 (3)

168 *Hydrogen-bond geometry* (Å, °)

169	<i>D</i> —H... <i>A</i>	<i>D</i> —H	H... <i>A</i>	<i>D</i> ... <i>A</i>	<i>D</i> —H... <i>A</i>
170	O1 <i>W</i> —H1 <i>W</i> ...O2 ⁱ	0.88 (1)	2.05 (2)	2.881 (2)	158 (3)
171	O2 <i>W</i> —H3 <i>W</i> ...N1 ⁱⁱ	0.87 (1)	1.95 (2)	2.805 (3)	170 (4)
172	O1 <i>W</i> —H2 <i>W</i> ...N3 ⁱⁱ	0.87 (1)	1.97 (1)	2.837 (3)	178 (3)
173	O2 <i>W</i> —H4 <i>W</i> ...N4 ⁱⁱⁱ	0.86 (1)	2.01 (1)	2.866 (3)	174 (4)
174	N4—H1 <i>N</i> ...O2 ^{iv}	0.84 (3)	2.40 (3)	3.070 (3)	137 (3)

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

176 (II)

177 *Crystal data*

178	$C_{10}H_9N_4NaO_2S$	$F(000) = 560$
179	$M_r = 272.26$	$D_x = 1.586 \text{ Mg m}^{-3}$
180	Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation, $\lambda = 0.71073 \text{ \AA}$
181	$a = 5.9010 (11) \text{ \AA}$	Cell parameters from 2254 reflections
182	$b = 10.534 (4) \text{ \AA}$	$\theta = 3.3\text{--}29.4^\circ$
183	$c = 18.389 (3) \text{ \AA}$	$\mu = 0.32 \text{ mm}^{-1}$
184	$\beta = 94.216 (15)^\circ$	$T = 123 \text{ K}$
185	$V = 1140.0 (5) \text{ \AA}^3$	Rod, colourless
186	$Z = 4$	$0.35 \times 0.12 \times 0.08 \text{ mm}$

187 *Data collection*

188	Oxford Diffraction Xcalibur E diffractometer	Absorption correction: multi-scan
189	Radiation source: sealed tube	<i>CrysAlis PRO</i> , Oxford Diffraction Ltd., Version 1.171.34.40 (release 27-08-2010 <i>CrysAlis171</i> .NET) (compiled Aug 27 2010, 11:50:40)
190	ω scans	Empirical absorption correction using spherical harmonics, implemented in <i>SCALE3</i> ABSPACK scaling algorithm.
		$T_{\min} = 0.961, T_{\max} = 1.000$
		5903 measured reflections
		2786 independent reflections
		2351 reflections with $I > 2\sigma(I)$
		$R_{\text{int}} = 0.029$
		$\theta_{\max} = 29.5^\circ, \theta_{\min} = 3.5^\circ$
		$h = -7 \rightarrow 7$
		$k = -12 \rightarrow 14$
		$l = -24 \rightarrow 24$

191 *Refinement*

192	Refinement on F^2	$S = 1.06$
193	Least-squares matrix: full	2786 reflections
194	$R[F^2 > 2\sigma(F^2)] = 0.038$	171 parameters
195	$wR(F^2) = 0.088$	0 restraints

196 Hydrogen site location: mixed
 197 H atoms treated by a mixture of independent
 and constrained refinement

$$w = 1/[\sigma^2(F_o^2) + (0.0278P)^2 + 0.6871P]$$

where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} < 0.001$
 $\Delta\rho_{\max} = 0.43 \text{ e } \text{Å}^{-3}$
 $\Delta\rho_{\min} = -0.46 \text{ e } \text{Å}^{-3}$

198 *Special details*

199 **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.

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

	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> _{iso} [*] / <i>U</i> _{eq}	
201					
202	Na1	0.41732 (11)	-0.15699 (7)	0.03430 (4)	0.01483 (18)
203	S1	0.13569 (7)	0.11080 (4)	0.07836 (2)	0.01030 (12)
204	O1	0.34050 (19)	0.07207 (12)	0.04443 (7)	0.0130 (3)
205	O2	0.0133 (2)	0.00644 (12)	0.10838 (7)	0.0152 (3)
206	N1	-0.0383 (2)	0.18774 (14)	0.02732 (8)	0.0121 (3)
207	N2	0.2445 (2)	0.32085 (15)	-0.01969 (9)	0.0154 (3)
208	N3	-0.1495 (2)	0.33011 (16)	-0.06053 (9)	0.0155 (3)
209	N4	0.4138 (3)	0.41926 (17)	0.34001 (9)	0.0182 (4)
210	C1	0.0271 (3)	0.28102 (17)	-0.01786 (10)	0.0122 (4)
211	C2	0.2788 (3)	0.42162 (19)	-0.06182 (11)	0.0197 (4)
212	H2	0.4295	0.4527	-0.0633	0.024*
213	C3	0.1082 (3)	0.4826 (2)	-0.10308 (12)	0.0224 (4)
214	H3	0.1351	0.5566	-0.1307	0.027*
215	C4	-0.1042 (3)	0.4292 (2)	-0.10165 (11)	0.0198 (4)
216	H4	-0.2252	0.4653	-0.1318	0.024*
217	C5	0.2237 (3)	0.20545 (17)	0.15397 (10)	0.0121 (4)
218	C6	0.0767 (3)	0.21945 (18)	0.20992 (10)	0.0154 (4)
219	H6	-0.0670	0.1783	0.2059	0.019*
220	C7	0.1368 (3)	0.29163 (19)	0.27051 (10)	0.0162 (4)
221	H7	0.0348	0.3008	0.3078	0.019*
222	C8	0.3482 (3)	0.35145 (18)	0.27712 (10)	0.0141 (4)
223	C9	0.4953 (3)	0.33673 (19)	0.22135 (11)	0.0177 (4)
224	H9	0.6399	0.3768	0.2257	0.021*
225	C10	0.4342 (3)	0.26494 (19)	0.15998 (10)	0.0160 (4)
226	H10	0.5352	0.2565	0.1224	0.019*
227	H1N	0.301 (4)	0.454 (2)	0.3602 (13)	0.023 (6)*
228	H2N	0.528 (4)	0.476 (2)	0.3346 (13)	0.029 (6)*

229 *Atomic displacement parameters (Å²)*

	<i>U</i> ¹¹	<i>U</i> ²²	<i>U</i> ³³	<i>U</i> ¹²	<i>U</i> ¹³	<i>U</i> ²³	
230							
231	Na1	0.0126 (3)	0.0166 (4)	0.0152 (4)	-0.0003 (3)	0.0003 (3)	0.0012 (3)
232	S1	0.0107 (2)	0.0106 (2)	0.0097 (2)	0.00025 (15)	0.00075 (15)	-0.00029 (17)
233	O1	0.0130 (6)	0.0136 (6)	0.0126 (6)	0.0030 (5)	0.0020 (5)	-0.0009 (5)

234	O2	0.0180 (6)	0.0121 (6)	0.0159 (7)	-0.0030 (5)	0.0026 (5)	0.0004 (6)
235	N1	0.0107 (7)	0.0137 (8)	0.0118 (8)	0.0004 (6)	-0.0005 (5)	0.0013 (6)
236	N2	0.0119 (7)	0.0156 (8)	0.0188 (8)	-0.0006 (6)	0.0019 (6)	0.0007 (7)
237	N3	0.0126 (7)	0.0173 (8)	0.0163 (8)	0.0029 (6)	-0.0002 (6)	0.0032 (7)
238	N4	0.0157 (8)	0.0218 (9)	0.0168 (9)	-0.0016 (7)	-0.0016 (6)	-0.0077 (7)
239	C1	0.0116 (8)	0.0136 (9)	0.0114 (9)	0.0017 (7)	0.0013 (6)	-0.0022 (7)
240	C2	0.0165 (9)	0.0183 (10)	0.0247 (11)	-0.0013 (7)	0.0052 (7)	0.0027 (9)
241	C3	0.0243 (10)	0.0171 (10)	0.0267 (12)	0.0027 (8)	0.0084 (8)	0.0075 (9)
242	C4	0.0182 (9)	0.0212 (10)	0.0203 (10)	0.0067 (8)	0.0023 (7)	0.0053 (9)
243	C5	0.0123 (8)	0.0131 (9)	0.0106 (9)	0.0017 (7)	-0.0014 (6)	0.0002 (7)
244	C6	0.0125 (8)	0.0194 (10)	0.0143 (9)	-0.0019 (7)	0.0011 (7)	-0.0023 (8)
245	C7	0.0132 (8)	0.0212 (10)	0.0145 (9)	-0.0003 (7)	0.0027 (7)	-0.0021 (8)
246	C8	0.0146 (8)	0.0143 (9)	0.0129 (9)	0.0013 (7)	-0.0024 (7)	-0.0014 (8)
247	C9	0.0131 (8)	0.0223 (10)	0.0174 (10)	-0.0041 (7)	-0.0010 (7)	-0.0024 (9)
248	C10	0.0132 (8)	0.0204 (10)	0.0148 (9)	-0.0012 (7)	0.0031 (7)	-0.0011 (8)

249 *Geometric parameters (Å, °)*

250	Na1—O1 ⁱ	2.2897 (15)	N4—C8	1.390 (2)
251	Na1—N1 ⁱⁱ	2.4533 (15)	N4—Na1 ^{iv}	2.5772 (19)
252	Na1—O1	2.4647 (17)	N4—H1N	0.86 (2)
253	Na1—N3 ⁱⁱ	2.4836 (18)	N4—H2N	0.91 (2)
254	Na1—N4 ⁱⁱⁱ	2.5771 (19)	C1—Na1 ⁱⁱ	2.9255 (19)
255	Na1—N2 ⁱ	2.6670 (17)	C2—C3	1.375 (3)
256	Na1—C1 ⁱⁱ	2.9255 (19)	C2—H2	0.9500
257	Na1—Na1 ⁱ	3.6964 (19)	C3—C4	1.376 (3)
258	S1—O2	1.4470 (14)	C3—H3	0.9500
259	S1—O1	1.4585 (13)	C4—H4	0.9500
260	S1—N1	1.5652 (15)	C5—C10	1.388 (2)
261	S1—C5	1.7582 (18)	C5—C6	1.402 (2)
262	O1—Na1 ⁱ	2.2897 (15)	C6—C7	1.374 (3)
263	N1—C1	1.361 (2)	C6—H6	0.9500
264	N1—Na1 ⁱⁱ	2.4533 (15)	C7—C8	1.395 (2)
265	N2—C2	1.338 (3)	C7—H7	0.9500
266	N2—C1	1.353 (2)	C8—C9	1.400 (3)
267	N2—Na1 ⁱ	2.6670 (17)	C9—C10	1.384 (3)
268	N3—C4	1.328 (3)	C9—H9	0.9500
269	N3—C1	1.360 (2)	C10—H10	0.9500
270	N3—Na1 ⁱⁱ	2.4836 (18)		
271				
272	O1 ⁱ —Na1—N1 ⁱⁱ	110.47 (5)	C4—N3—C1	116.73 (16)
273	O1 ⁱ —Na1—O1	78.00 (5)	C4—N3—Na1 ⁱⁱ	147.74 (13)
274	N1 ⁱⁱ —Na1—O1	89.87 (5)	C1—N3—Na1 ⁱⁱ	94.60 (11)
275	O1 ⁱ —Na1—N3 ⁱⁱ	147.71 (6)	C8—N4—Na1 ^{iv}	130.68 (13)
276	N1 ⁱⁱ —Na1—N3 ⁱⁱ	54.64 (5)	C8—N4—H1N	113.5 (15)
277	O1—Na1—N3 ⁱⁱ	125.52 (6)	Na1 ^{iv} —N4—H1N	90.2 (15)
278	O1 ⁱ —Na1—N4 ⁱⁱⁱ	103.13 (6)	C8—N4—H2N	114.1 (15)
279	N1 ⁱⁱ —Na1—N4 ⁱⁱⁱ	137.28 (6)	Na1 ^{iv} —N4—H2N	93.5 (15)

280	O1—Na1—N4 ⁱⁱⁱ	71.83 (5)	H1N—N4—H2N	112 (2)
281	N3 ⁱⁱ —Na1—N4 ⁱⁱⁱ	105.32 (6)	N2—C1—N3	123.76 (17)
282	O1 ⁱ —Na1—N2 ⁱ	71.51 (5)	N2—C1—N1	123.45 (15)
283	N1 ⁱⁱ —Na1—N2 ⁱ	122.11 (6)	N3—C1—N1	112.79 (15)
284	O1—Na1—N2 ⁱ	141.66 (5)	N2—C1—Na1 ⁱⁱ	168.70 (13)
285	N3 ⁱⁱ —Na1—N2 ⁱ	92.06 (6)	N3—C1—Na1 ⁱⁱ	57.80 (9)
286	N4 ⁱⁱⁱ —Na1—N2 ⁱ	92.82 (6)	N1—C1—Na1 ⁱⁱ	56.52 (8)
287	O1 ⁱ —Na1—C1 ⁱⁱ	134.88 (6)	N2—C2—C3	123.65 (17)
288	N1 ⁱⁱ —Na1—C1 ⁱⁱ	27.56 (5)	N2—C2—H2	118.2
289	O1—Na1—C1 ⁱⁱ	106.06 (5)	C3—C2—H2	118.2
290	N3 ⁱⁱ —Na1—C1 ⁱⁱ	27.60 (5)	C2—C3—C4	115.45 (19)
291	N4 ⁱⁱⁱ —Na1—C1 ⁱⁱ	121.09 (6)	C2—C3—H3	122.3
292	N2 ⁱ —Na1—C1 ⁱⁱ	111.85 (6)	C4—C3—H3	122.3
293	O1 ⁱ —Na1—Na1 ⁱ	40.71 (3)	N3—C4—C3	123.66 (18)
294	N1 ⁱⁱ —Na1—Na1 ⁱ	102.42 (5)	N3—C4—H4	118.2
295	O1—Na1—Na1 ⁱ	37.29 (4)	C3—C4—H4	118.2
296	N3 ⁱⁱ —Na1—Na1 ⁱ	155.65 (5)	C10—C5—C6	119.51 (17)
297	N4 ⁱⁱⁱ —Na1—Na1 ⁱ	86.15 (5)	C10—C5—S1	121.98 (14)
298	N2 ⁱ —Na1—Na1 ⁱ	109.06 (5)	C6—C5—S1	118.51 (13)
299	C1 ⁱⁱ —Na1—Na1 ⁱ	128.42 (5)	C7—C6—C5	121.10 (16)
300	O2—S1—O1	113.78 (8)	C7—C6—H6	119.5
301	O2—S1—N1	107.35 (8)	C5—C6—H6	119.5
302	O1—S1—N1	114.59 (8)	C6—C7—C8	119.79 (17)
303	O2—S1—C5	104.83 (8)	C6—C7—H7	120.1
304	O1—S1—C5	106.92 (8)	C8—C7—H7	120.1
305	N1—S1—C5	108.86 (8)	N4—C8—C7	119.84 (17)
306	S1—O1—Na1 ⁱ	137.43 (8)	N4—C8—C9	121.08 (16)
307	S1—O1—Na1	117.97 (7)	C7—C8—C9	119.01 (17)
308	Na1 ⁱ —O1—Na1	102.00 (5)	C10—C9—C8	121.26 (16)
309	C1—N1—S1	122.48 (12)	C10—C9—H9	119.4
310	C1—N1—Na1 ⁱⁱ	95.92 (10)	C8—C9—H9	119.4
311	S1—N1—Na1 ⁱⁱ	138.08 (9)	C9—C10—C5	119.32 (17)
312	C2—N2—C1	116.40 (15)	C9—C10—H10	120.3
313	C2—N2—Na1 ⁱ	107.99 (12)	C5—C10—H10	120.3
314	C1—N2—Na1 ⁱ	121.33 (12)		
315				
316	O2—S1—O1—Na1 ⁱ	163.45 (10)	Na1 ⁱⁱ —N1—C1—N3	13.88 (16)
317	N1—S1—O1—Na1 ⁱ	39.38 (14)	S1—N1—C1—Na1 ⁱⁱ	162.33 (16)
318	C5—S1—O1—Na1 ⁱ	-81.32 (12)	C1—N2—C2—C3	-1.3 (3)
319	O2—S1—O1—Na1	5.61 (10)	Na1 ⁱ —N2—C2—C3	139.15 (18)
320	N1—S1—O1—Na1	-118.45 (8)	N2—C2—C3—C4	-3.2 (3)
321	C5—S1—O1—Na1	120.84 (8)	C1—N3—C4—C3	0.3 (3)
322	O2—S1—N1—C1	-170.13 (14)	Na1 ⁱⁱ —N3—C4—C3	-164.85 (18)
323	O1—S1—N1—C1	-42.71 (17)	C2—C3—C4—N3	3.8 (3)
324	C5—S1—N1—C1	76.92 (16)	O2—S1—C5—C10	141.97 (15)
325	O2—S1—N1—Na1 ⁱⁱ	-17.00 (15)	O1—S1—C5—C10	20.87 (18)
326	O1—S1—N1—Na1 ⁱⁱ	110.42 (13)	N1—S1—C5—C10	-103.42 (16)
327	C5—S1—N1—Na1 ⁱⁱ	-129.95 (13)	O2—S1—C5—C6	-37.32 (16)

328	C2—N2—C1—N3	5.9 (3)	O1—S1—C5—C6	-158.41 (14)
329	Na1 ⁱ —N2—C1—N3	-128.95 (16)	N1—S1—C5—C6	77.29 (16)
330	C2—N2—C1—N1	-173.70 (18)	C10—C5—C6—C7	0.3 (3)
331	Na1 ⁱ —N2—C1—N1	51.4 (2)	S1—C5—C6—C7	179.62 (15)
332	C2—N2—C1—Na1 ⁱⁱ	100.2 (7)	C5—C6—C7—C8	-0.5 (3)
333	Na1 ⁱ —N2—C1—Na1 ⁱⁱ	-34.7 (7)	Na1 ^{iv} —N4—C8—C7	81.0 (2)
334	C4—N3—C1—N2	-5.4 (3)	Na1 ^{iv} —N4—C8—C9	-95.8 (2)
335	Na1 ⁱⁱ —N3—C1—N2	166.65 (16)	C6—C7—C8—N4	-176.79 (18)
336	C4—N3—C1—N1	174.24 (17)	C6—C7—C8—C9	0.1 (3)
337	Na1 ⁱⁱ —N3—C1—N1	-13.67 (16)	N4—C8—C9—C10	177.32 (18)
338	C4—N3—C1—Na1 ⁱⁱ	-172.09 (19)	C7—C8—C9—C10	0.4 (3)
339	S1—N1—C1—N2	-4.1 (3)	C8—C9—C10—C5	-0.6 (3)
340	Na1 ⁱⁱ —N1—C1—N2	-166.45 (16)	C6—C5—C10—C9	0.2 (3)
341	S1—N1—C1—N3	176.20 (13)	S1—C5—C10—C9	-179.03 (15)

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

343 *Hydrogen-bond geometry* ($\text{\AA}, ^\circ$)

344	<i>D</i> —H \cdots <i>A</i>	<i>D</i> —H	H \cdots <i>A</i>	<i>D</i> \cdots <i>A</i>	<i>D</i> —H \cdots <i>A</i>
345	N4—H1N \cdots O2 ^v	0.86 (2)	2.06 (2)	2.907 (2)	166 (2)
346	N4—H2N \cdots O1 ^{iv}	0.91 (2)	2.52 (2)	2.959 (2)	110.5 (17)

347 Symmetry codes: (iv) $-x+1, y+1/2, -z+1/2$; (v) $-x, y+1/2, -z+1/2$.

348 (III)

349 *Crystal data*

350	C ₁₀ H ₁₃ KN ₄ O ₄ S	<i>Z</i> = 4
351	<i>M_r</i> = 324.40	<i>F</i> (000) = 672
352	Triclinic, <i>P</i> 1	<i>D_x</i> = 1.594 Mg m ⁻³
353	<i>a</i> = 8.8503 (7) \AA	Cu <i>K</i> α radiation, λ = 1.5418 \AA
354	<i>b</i> = 9.6385 (4) \AA	Cell parameters from 3605 reflections
355	<i>c</i> = 15.9734 (7) \AA	θ = 4.6–73.6 $^\circ$
356	α = 92.350 (4) $^\circ$	μ = 5.09 mm ⁻¹
357	β = 95.186 (4) $^\circ$	<i>T</i> = 123 K
358	γ = 94.209 (4) $^\circ$	Block, colourless
359	<i>V</i> = 1351.79 (13) \AA^3	0.28 \times 0.15 \times 0.10 mm

360 *Data collection*

361	Oxford Diffraction Gemini S diffractometer	3951 reflections with $I > 2\sigma(I)$
362	Radiation source: sealed tube	<i>R</i> _{int} = 0.050
363	ω scans	θ_{max} = 73.8 $^\circ$, θ_{min} = 4.6 $^\circ$
364	Absorption correction: multi-scan <i>CrysAlis PRO</i> , Agilent Technologies, Version 1.171.37.35 (release 13-08-2014 <i>CrysAlis171.NET</i>) (compiled Aug 13 2014, 18:06:01) Empirical absorption correction using spherical harmonics, implemented in <i>SCALE3</i> ABSPACK scaling algorithm.	<i>h</i> = -8 \rightarrow 10 <i>k</i> = -11 \rightarrow 11 <i>l</i> = -19 \rightarrow 19
365	<i>T</i> _{min} = 0.601, <i>T</i> _{max} = 1.000	
366	15580 measured reflections	
367	5357 independent reflections	

368 *Refinement*

369	Refinement on F^2	Hydrogen site location: mixed
370	Least-squares matrix: full	H atoms treated by a mixture of independent
371	$R[F^2 > 2\sigma(F^2)] = 0.060$	and constrained refinement
372	$wR(F^2) = 0.157$	$w = 1/[\sigma^2(F_o^2) + (0.0619P)^2 + 2.5308P]$
373	$S = 1.05$	where $P = (F_o^2 + 2F_c^2)/3$
374	5357 reflections	$(\Delta/\sigma)_{\max} < 0.001$
375	404 parameters	$\Delta\rho_{\max} = 0.81 \text{ e } \text{\AA}^{-3}$
376	14 restraints	$\Delta\rho_{\min} = -0.45 \text{ e } \text{\AA}^{-3}$

377 *Special details*

378 **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.

379 *Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (\AA^2)*

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$	Occ. (<1)	
381	K1	0.24189 (11)	0.55756 (9)	0.47536 (6)	0.0350 (2)	
382	K2	-0.15927 (12)	0.82674 (9)	0.41066 (6)	0.0365 (2)	
383	S1	-0.26644 (12)	0.67031 (10)	0.19910 (6)	0.0304 (2)	
384	S2	-0.05315 (12)	0.81132 (10)	0.61877 (6)	0.0281 (2)	
385	O1	-0.3523 (3)	0.6886 (3)	0.27240 (19)	0.0345 (7)	
386	O2	-0.3075 (4)	0.7607 (3)	0.1321 (2)	0.0398 (7)	
387	O1W	0.4746 (5)	0.4378 (5)	0.5915 (3)	0.0735 (14)	
388	H1W	0.450 (9)	0.398 (6)	0.637 (2)	0.110*	
389	H2W	0.481 (10)	0.362 (4)	0.560 (4)	0.110*	
390	O3	-0.0548 (3)	0.9494 (3)	0.58530 (18)	0.0326 (7)	
391	O2W	0.2375 (5)	0.8119 (4)	0.3937 (2)	0.0549 (10)	
392	H3W	0.196 (7)	0.891 (4)	0.398 (4)	0.082*	
393	H4W	0.199 (7)	0.785 (6)	0.3424 (17)	0.082*	
394	O4	-0.1743 (4)	0.7140 (3)	0.57871 (18)	0.0346 (7)	
395	O3W	-0.0485 (4)	0.5553 (3)	0.39403 (19)	0.0359 (7)	
396	H5W	-0.050 (6)	0.584 (4)	0.3428 (13)	0.054*	
397	H6W	-0.068 (6)	0.4658 (13)	0.384 (3)	0.054*	
398	O4W	-0.4475 (5)	0.8724 (4)	0.4641 (3)	0.0604 (11)	
399	H7W	-0.541 (3)	0.843 (7)	0.445 (4)	0.091*	
400	H8W	-0.466 (9)	0.954 (7)	0.486 (9)	0.091*	0.5
401	H9W	-0.436 (10)	0.819 (11)	0.507 (5)	0.091*	0.5
402	N1	-0.0933 (4)	0.6879 (3)	0.2369 (2)	0.0301 (8)	
403	N2	0.1612 (4)	0.7098 (4)	0.2256 (2)	0.0342 (8)	
404	N3	-0.0068 (4)	0.6541 (4)	0.1017 (2)	0.0348 (8)	
405	N4	-0.4201 (6)	0.0807 (5)	0.0779 (3)	0.0504 (12)	
406	N5	0.0982 (4)	0.7389 (4)	0.6097 (2)	0.0317 (8)	
407	N6	0.2546 (4)	0.9310 (3)	0.6755 (2)	0.0303 (8)	
408	N7	0.3546 (5)	0.7193 (4)	0.6308 (2)	0.0384 (9)	
409	N8	-0.1718 (6)	0.8726 (5)	0.9774 (3)	0.0441 (10)	
410	C1	0.0218 (5)	0.6828 (4)	0.1849 (3)	0.0309 (9)	

411	C2	0.2782 (6)	0.7163 (5)	0.1785 (3)	0.0433 (11)
412	H2	0.3781	0.7352	0.2055	0.052*
413	C3	0.2599 (6)	0.6967 (5)	0.0925 (3)	0.0447 (12)
414	H3	0.3436	0.7068	0.0593	0.054*
415	C4	0.1144 (6)	0.6617 (5)	0.0567 (3)	0.0406 (11)
416	H4	0.0987	0.6421	-0.0023	0.049*
417	C5	-0.3070 (5)	0.4959 (4)	0.1601 (3)	0.0299 (9)
418	C6	-0.2140 (5)	0.3935 (4)	0.1847 (3)	0.0312 (9)
419	H6	-0.1236	0.4172	0.2207	0.037*
420	C7	-0.2516 (5)	0.2566 (5)	0.1571 (3)	0.0353 (10)
421	H7	-0.1851	0.1873	0.1732	0.042*
422	C8	-0.3841 (5)	0.2186 (5)	0.1065 (3)	0.0377 (11)
423	C9	-0.4762 (6)	0.3219 (6)	0.0812 (3)	0.0480 (13)
424	H9	-0.5667	0.2978	0.0453	0.058*
425	C10	-0.4386 (5)	0.4598 (5)	0.1073 (3)	0.0436 (12)
426	H10	-0.5028	0.5297	0.0892	0.052*
427	C11	0.2388 (5)	0.8013 (4)	0.6399 (3)	0.0295 (9)
428	C12	0.3946 (5)	0.9760 (4)	0.7084 (3)	0.0339 (10)
429	H12	0.4095	1.0670	0.7344	0.041*
430	C13	0.5188 (5)	0.8976 (5)	0.7068 (3)	0.0384 (10)
431	H13	0.6166	0.9299	0.7326	0.046*
432	C14	0.4908 (6)	0.7693 (5)	0.6650 (3)	0.0428 (11)
433	H14	0.5740	0.7133	0.6603	0.051*
434	C15	-0.0866 (5)	0.8295 (4)	0.7260 (2)	0.0269 (8)
435	C16	-0.1369 (5)	0.7129 (4)	0.7677 (3)	0.0298 (9)
436	H16	-0.1542	0.6246	0.7382	0.036*
437	C17	-0.1615 (5)	0.7256 (4)	0.8511 (3)	0.0334 (10)
438	H17	-0.1935	0.6454	0.8794	0.040*
439	C18	-0.1399 (5)	0.8561 (4)	0.8955 (3)	0.0313 (9)
440	C19	-0.0884 (5)	0.9708 (4)	0.8523 (3)	0.0300 (9)
441	H19	-0.0723	1.0597	0.8810	0.036*
442	C20	-0.0605 (5)	0.9583 (4)	0.7689 (3)	0.0281 (9)
443	H20	-0.0236	1.0373	0.7410	0.034*
444	H1N	-0.515 (8)	0.057 (7)	0.059 (4)	0.07 (2)*
445	H2N	-0.383 (7)	0.010 (7)	0.111 (4)	0.06 (2)*
446	H3N	-0.185 (6)	0.800 (5)	1.008 (3)	0.037 (14)*
447	H4N	-0.156 (6)	0.955 (6)	1.004 (3)	0.053 (16)*

448 *Atomic displacement parameters (\AA^2)*

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}	
450	K1	0.0413 (5)	0.0269 (5)	0.0355 (5)	-0.0036 (4)	0.0044 (4)	-0.0060 (4)
451	K2	0.0504 (6)	0.0234 (4)	0.0337 (5)	-0.0016 (4)	-0.0020 (4)	-0.0007 (3)
452	S1	0.0337 (6)	0.0274 (5)	0.0301 (5)	0.0045 (4)	0.0037 (4)	-0.0023 (4)
453	S2	0.0327 (5)	0.0236 (5)	0.0269 (5)	-0.0012 (4)	0.0021 (4)	-0.0022 (4)
454	O1	0.0344 (17)	0.0338 (16)	0.0354 (16)	0.0017 (13)	0.0071 (13)	-0.0064 (13)
455	O2	0.046 (2)	0.0372 (17)	0.0375 (17)	0.0119 (14)	0.0050 (14)	0.0017 (13)
456	O1W	0.048 (2)	0.079 (3)	0.100 (4)	0.009 (2)	0.018 (2)	0.049 (3)

457	O3	0.0342 (17)	0.0296 (15)	0.0339 (16)	0.0023 (13)	0.0021 (12)	0.0019 (12)
458	O2W	0.077 (3)	0.0357 (19)	0.050 (2)	0.0102 (19)	-0.0048 (19)	-0.0027 (16)
459	O4	0.0381 (17)	0.0325 (16)	0.0310 (15)	-0.0049 (13)	0.0005 (13)	-0.0043 (12)
460	O3W	0.0464 (19)	0.0274 (15)	0.0330 (16)	0.0014 (14)	0.0028 (14)	-0.0039 (12)
461	O4W	0.079 (3)	0.039 (2)	0.067 (3)	0.0007 (19)	0.031 (2)	0.0003 (18)
462	N1	0.036 (2)	0.0247 (17)	0.0286 (18)	-0.0033 (15)	0.0060 (15)	-0.0011 (14)
463	N2	0.033 (2)	0.0271 (18)	0.043 (2)	0.0030 (15)	0.0090 (16)	0.0011 (15)
464	N3	0.045 (2)	0.0241 (18)	0.036 (2)	0.0029 (16)	0.0113 (17)	-0.0007 (15)
465	N4	0.057 (3)	0.045 (3)	0.045 (3)	-0.017 (2)	0.010 (2)	-0.020 (2)
466	N5	0.040 (2)	0.0240 (17)	0.0315 (19)	0.0023 (15)	0.0070 (15)	-0.0046 (14)
467	N6	0.033 (2)	0.0240 (17)	0.0340 (19)	-0.0005 (14)	0.0040 (15)	-0.0004 (14)
468	N7	0.045 (2)	0.034 (2)	0.037 (2)	0.0081 (17)	0.0049 (17)	-0.0048 (16)
469	N8	0.071 (3)	0.031 (2)	0.031 (2)	-0.002 (2)	0.0101 (19)	-0.0008 (17)
470	C1	0.038 (2)	0.0164 (19)	0.039 (2)	0.0026 (17)	0.0089 (19)	0.0003 (16)
471	C2	0.035 (3)	0.033 (2)	0.064 (3)	0.007 (2)	0.012 (2)	0.010 (2)
472	C3	0.043 (3)	0.041 (3)	0.057 (3)	0.015 (2)	0.023 (2)	0.011 (2)
473	C4	0.059 (3)	0.029 (2)	0.038 (2)	0.009 (2)	0.017 (2)	0.0040 (18)
474	C5	0.028 (2)	0.032 (2)	0.029 (2)	0.0021 (17)	0.0035 (16)	-0.0066 (17)
475	C6	0.032 (2)	0.029 (2)	0.031 (2)	0.0000 (18)	-0.0012 (17)	-0.0006 (17)
476	C7	0.043 (3)	0.030 (2)	0.033 (2)	0.0007 (19)	0.0027 (19)	-0.0010 (17)
477	C8	0.037 (3)	0.040 (2)	0.035 (2)	-0.008 (2)	0.0099 (19)	-0.0118 (19)
478	C9	0.031 (3)	0.060 (3)	0.048 (3)	-0.004 (2)	-0.002 (2)	-0.028 (2)
479	C10	0.030 (2)	0.054 (3)	0.046 (3)	0.016 (2)	-0.004 (2)	-0.017 (2)
480	C11	0.035 (2)	0.027 (2)	0.028 (2)	0.0019 (17)	0.0058 (17)	0.0002 (16)
481	C12	0.036 (2)	0.028 (2)	0.037 (2)	-0.0023 (18)	0.0018 (19)	-0.0017 (17)
482	C13	0.023 (2)	0.047 (3)	0.044 (3)	0.0054 (19)	-0.0007 (18)	-0.001 (2)
483	C14	0.036 (3)	0.052 (3)	0.042 (3)	0.018 (2)	0.004 (2)	0.000 (2)
484	C15	0.028 (2)	0.026 (2)	0.027 (2)	0.0014 (16)	0.0041 (16)	-0.0019 (15)
485	C16	0.030 (2)	0.022 (2)	0.036 (2)	0.0004 (16)	0.0022 (17)	-0.0044 (16)
486	C17	0.037 (2)	0.022 (2)	0.041 (2)	-0.0012 (18)	0.0028 (19)	0.0044 (17)
487	C18	0.030 (2)	0.031 (2)	0.032 (2)	0.0013 (18)	0.0016 (17)	-0.0023 (17)
488	C19	0.029 (2)	0.023 (2)	0.036 (2)	0.0024 (17)	0.0001 (17)	-0.0061 (16)
489	C20	0.025 (2)	0.0224 (19)	0.036 (2)	0.0003 (16)	0.0016 (16)	0.0004 (16)

490 *Geometric parameters (Å, °)*

491	K1—O4 ⁱ	2.729 (3)	N1—C1	1.373 (5)
492	K1—O3W	2.771 (3)	N2—C2	1.333 (6)
493	K1—O1W ⁱⁱ	2.812 (5)	N2—C1	1.343 (6)
494	K1—O2W	2.824 (4)	N3—C4	1.343 (6)
495	K1—N7	2.932 (4)	N3—C1	1.344 (6)
496	K1—O1W	2.975 (5)	N4—C8	1.393 (6)
497	K1—O3W ⁱ	3.004 (3)	N4—H1N	0.87 (7)
498	K1—N5	3.129 (4)	N4—H2N	0.93 (7)
499	K1—C11	3.457 (4)	N5—C11	1.379 (5)
500	K1—K2 ⁱ	4.2385 (13)	N6—C12	1.336 (6)
501	K1—K1 ⁱ	4.491 (2)	N6—C11	1.344 (5)
502	K1—K2	4.6167 (15)	N6—K2 ⁱⁱⁱ	2.899 (4)

503	K1—H6W	3.04 (5)	N7—C14	1.325 (6)
504	K2—O3 ⁱⁱⁱ	2.759 (3)	N7—C11	1.355 (6)
505	K2—O4W	2.820 (4)	N8—C18	1.369 (6)
506	K2—O3W	2.871 (3)	N8—H3N	0.87 (5)
507	K2—O1	2.883 (3)	N8—H4N	0.88 (6)
508	K2—N6 ⁱⁱⁱ	2.899 (4)	C2—C3	1.372 (7)
509	K2—O4	2.947 (3)	C2—H2	0.9500
510	K2—O3	3.023 (3)	C3—C4	1.373 (7)
511	K2—N1	3.153 (3)	C3—H3	0.9500
512	K2—S2	3.3840 (14)	C4—H4	0.9500
513	K2—C12 ⁱⁱⁱ	3.425 (5)	C5—C6	1.378 (6)
514	K2—S1	3.6587 (14)	C5—C10	1.388 (6)
515	K2—K1 ⁱ	4.2385 (13)	C6—C7	1.382 (6)
516	K2—H5W	2.81 (5)	C6—H6	0.9500
517	K2—H9W	3.01 (11)	C7—C8	1.379 (6)
518	S1—O2	1.445 (3)	C7—H7	0.9500
519	S1—O1	1.464 (3)	C8—C9	1.383 (7)
520	S1—N1	1.589 (4)	C9—C10	1.385 (7)
521	S1—C5	1.769 (4)	C9—H9	0.9500
522	S2—O3	1.455 (3)	C10—H10	0.9500
523	S2—O4	1.455 (3)	C12—C13	1.380 (6)
524	S2—N5	1.570 (4)	C12—K2 ⁱⁱⁱ	3.425 (5)
525	S2—C15	1.768 (4)	C12—H12	0.9500
526	O1W—K1 ⁱⁱ	2.812 (5)	C13—C14	1.376 (7)
527	O1W—H1W	0.880 (10)	C13—H13	0.9500
528	O1W—H2W	0.877 (10)	C14—H14	0.9500
529	O3—K2 ⁱⁱⁱ	2.759 (3)	C15—C20	1.387 (5)
530	O2W—H3W	0.878 (10)	C15—C16	1.395 (6)
531	O2W—H4W	0.880 (10)	C16—C17	1.372 (6)
532	O4—K1 ⁱ	2.729 (3)	C16—H16	0.9500
533	O3W—K1 ⁱ	3.004 (3)	C17—C18	1.410 (6)
534	O3W—H5W	0.874 (10)	C17—H17	0.9500
535	O3W—H6W	0.873 (10)	C18—C19	1.397 (6)
536	O4W—H7W	0.878 (10)	C19—C20	1.378 (6)
537	O4W—H8W	0.879 (10)	C19—H19	0.9500
538	O4W—H9W	0.878 (10)	C20—H20	0.9500
539				
540	O4 ⁱ —K1—O3W	76.14 (9)	C12 ⁱⁱⁱ —K2—H5W	121.5 (6)
541	O4 ⁱ —K1—O1W ⁱⁱ	91.40 (13)	S1—K2—H5W	54.1 (7)
542	O3W—K1—O1W ⁱⁱ	129.93 (13)	K1 ⁱ —K2—H5W	60.3 (5)
543	O4 ⁱ —K1—O2W	133.37 (10)	O3 ⁱⁱⁱ —K2—H9W	124 (2)
544	O3W—K1—O2W	75.00 (11)	O4W—K2—H9W	17.0 (8)
545	O1W ⁱⁱ —K1—O2W	80.19 (13)	O3W—K2—H9W	111.2 (19)
546	O4 ⁱ —K1—N7	138.88 (11)	O1—K2—H9W	87.0 (19)
547	O3W—K1—N7	125.77 (11)	N6 ⁱⁱⁱ —K2—H9W	91.0 (12)
548	O1W ⁱⁱ —K1—N7	95.36 (15)	O4—K2—H9W	55.4 (6)
549	O2W—K1—N7	87.72 (11)	O3—K2—H9W	73.4 (18)
550	O4 ⁱ —K1—O1W	83.27 (12)	N1—K2—H9W	133 (2)

551	O3W—K1—O1W	152.02 (11)	S2—K2—H9W	70.2 (11)
552	O1W ⁱⁱ —K1—O1W	68.49 (15)	C12 ⁱⁱⁱ —K2—H9W	78.2 (7)
553	O2W—K1—O1W	132.73 (12)	S1—K2—H9W	109.0 (18)
554	N7—K1—O1W	62.11 (14)	K1 ⁱ —K2—H9W	67.2 (16)
555	O4 ⁱ —K1—O3W ⁱ	76.90 (9)	H5W—K2—H9W	122 (2)
556	O3W—K1—O3W ⁱ	78.00 (10)	O2—S1—O1	113.35 (19)
557	O1W ⁱⁱ —K1—O3W ⁱ	146.61 (12)	O2—S1—N1	115.66 (19)
558	O2W—K1—O3W ⁱ	130.21 (12)	O1—S1—N1	104.21 (18)
559	N7—K1—O3W ⁱ	75.36 (10)	O2—S1—C5	108.1 (2)
560	O1W—K1—O3W ⁱ	79.04 (11)	O1—S1—C5	107.12 (18)
561	O4 ⁱ —K1—N5	130.33 (10)	N1—S1—C5	107.9 (2)
562	O3W—K1—N5	81.94 (10)	O2—S1—K2	118.44 (13)
563	O1W ⁱⁱ —K1—N5	135.34 (13)	O1—S1—K2	47.64 (12)
564	O2W—K1—N5	80.19 (11)	N1—S1—K2	59.11 (13)
565	N7—K1—N5	44.19 (10)	C5—S1—K2	132.72 (15)
566	O1W—K1—N5	97.70 (13)	O3—S2—O4	112.95 (18)
567	O3W ⁱ —K1—N5	55.02 (9)	O3—S2—N5	114.45 (19)
568	O4 ⁱ —K1—C11	145.79 (10)	O4—S2—N5	105.51 (19)
569	O3W—K1—C11	103.38 (10)	O3—S2—C15	106.97 (18)
570	O1W ⁱⁱ —K1—C11	112.40 (14)	O4—S2—C15	106.49 (18)
571	O2W—K1—C11	76.90 (10)	N5—S2—C15	110.2 (2)
572	N7—K1—C11	22.63 (11)	O3—S2—K2	63.26 (12)
573	O1W—K1—C11	83.25 (13)	O4—S2—K2	60.28 (12)
574	O3W ⁱ —K1—C11	69.73 (9)	N5—S2—K2	97.00 (14)
575	N5—K1—C11	23.50 (10)	C15—S2—K2	152.44 (15)
576	O4 ⁱ —K1—K2 ⁱ	43.70 (6)	S1—O1—K2	110.33 (15)
577	O3W—K1—K2 ⁱ	94.71 (7)	K1 ⁱⁱ —O1W—K1	111.51 (15)
578	O1W ⁱⁱ —K1—K2 ⁱ	109.14 (9)	K1 ⁱⁱ —O1W—H1W	126 (5)
579	O2W—K1—K2 ⁱ	169.33 (10)	K1—O1W—H1W	122 (6)
580	N7—K1—K2 ⁱ	96.31 (8)	K1 ⁱⁱ —O1W—H2W	69 (6)
581	O1W—K1—K2 ⁱ	57.39 (8)	K1—O1W—H2W	96 (6)
582	O3W ⁱ —K1—K2 ⁱ	42.59 (6)	H1W—O1W—H2W	98 (2)
583	N5—K1—K2 ⁱ	95.81 (7)	S2—O3—K2 ⁱⁱⁱ	133.47 (17)
584	C11—K1—K2 ⁱ	103.32 (7)	S2—O3—K2	91.28 (13)
585	O4 ⁱ —K1—K1 ⁱ	72.58 (7)	K2 ⁱⁱⁱ —O3—K2	114.35 (10)
586	O3W—K1—K1 ⁱ	40.87 (7)	K1—O2W—H3W	139 (4)
587	O1W ⁱⁱ —K1—K1 ⁱ	162.68 (12)	K1—O2W—H4W	102 (4)
588	O2W—K1—K1 ⁱ	105.79 (10)	H3W—O2W—H4W	99 (2)
589	N7—K1—K1 ⁱ	101.05 (9)	S2—O4—K1 ⁱ	143.3 (2)
590	O1W—K1—K1 ⁱ	114.70 (9)	S2—O4—K2	94.33 (14)
591	O3W ⁱ —K1—K1 ⁱ	37.13 (6)	K1 ⁱ —O4—K2	96.54 (9)
592	N5—K1—K1 ⁱ	61.97 (7)	K1—O3W—K2	109.81 (10)
593	C11—K1—K1 ⁱ	84.90 (8)	K1—O3W—K1 ⁱ	102.00 (10)
594	K2 ⁱ —K1—K1 ⁱ	63.78 (3)	K2—O3W—K1 ⁱ	92.33 (9)
595	O4 ⁱ —K1—K2	111.87 (7)	K1—O3W—H5W	114 (4)
596	O3W—K1—K2	35.80 (6)	K2—O3W—H5W	77 (3)
597	O1W ⁱⁱ —K1—K2	128.87 (9)	K1 ⁱ —O3W—H5W	144 (4)
598	O2W—K1—K2	50.39 (10)	K1—O3W—H6W	99 (4)

599	N7—K1—K2	94.90 (8)	K2—O3W—H6W	149 (4)
600	O1W—K1—K2	154.32 (11)	K1 ⁱ —O3W—H6W	71 (3)
601	O3W ⁱ —K1—K2	84.29 (7)	H5W—O3W—H6W	101 (2)
602	N5—K1—K2	56.63 (7)	K2—O4W—H7W	134 (5)
603	C11—K1—K2	72.66 (8)	K2—O4W—H8W	121 (7)
604	K2 ⁱ —K1—K2	119.23 (3)	H7W—O4W—H8W	99 (2)
605	K1 ⁱ —K1—K2	55.45 (3)	K2—O4W—H9W	94 (8)
606	O4 ⁱ —K1—H6W	59.8 (5)	H7W—O4W—H9W	99 (2)
607	O3W—K1—H6W	16.5 (5)	C1—N1—S1	120.6 (3)
608	O1W ⁱⁱ —K1—H6W	127.1 (9)	C1—N1—K2	139.9 (3)
609	O2W—K1—H6W	89.3 (5)	S1—N1—K2	95.26 (15)
610	N7—K1—H6W	136.2 (8)	C2—N2—C1	116.9 (4)
611	O1W—K1—H6W	137.8 (5)	C4—N3—C1	116.1 (4)
612	O3W ⁱ —K1—H6W	73.6 (8)	C8—N4—H1N	117 (4)
613	N5—K1—H6W	92.3 (8)	C8—N4—H2N	118 (4)
614	C11—K1—H6W	115.3 (8)	H1N—N4—H2N	110 (6)
615	K2 ⁱ —K1—H6W	80.9 (6)	C11—N5—S2	122.3 (3)
616	K1 ⁱ —K1—H6W	38.7 (9)	C11—N5—K1	91.7 (2)
617	K2—K1—H6W	52.1 (5)	S2—N5—K1	138.42 (19)
618	O3 ⁱⁱⁱ —K2—O4W	118.31 (10)	C12—N6—C11	116.1 (4)
619	O3 ⁱⁱⁱ —K2—O3W	116.43 (10)	C12—N6—K2 ⁱⁱⁱ	101.5 (3)
620	O4W—K2—O3W	123.26 (11)	C11—N6—K2 ⁱⁱⁱ	123.5 (3)
621	O3 ⁱⁱⁱ —K2—O1	130.90 (9)	C14—N7—C11	116.3 (4)
622	O4W—K2—O1	80.08 (11)	C14—N7—K1	134.6 (3)
623	O3W—K2—O1	75.53 (9)	C11—N7—K1	101.0 (3)
624	O3 ⁱⁱⁱ —K2—N6 ⁱⁱⁱ	63.92 (9)	C18—N8—H3N	121 (3)
625	O4W—K2—N6 ⁱⁱⁱ	74.67 (11)	C18—N8—H4N	120 (4)
626	O3W—K2—N6 ⁱⁱⁱ	146.26 (10)	H3N—N8—H4N	117 (5)
627	O1—K2—N6 ⁱⁱⁱ	80.79 (10)	N2—C1—N3	124.9 (4)
628	O3 ⁱⁱⁱ —K2—O4	111.22 (9)	N2—C1—N1	113.4 (4)
629	O4W—K2—O4	72.00 (12)	N3—C1—N1	121.7 (4)
630	O3W—K2—O4	75.72 (9)	N2—C2—C3	122.6 (5)
631	O1—K2—O4	117.84 (9)	N2—C2—H2	118.7
632	N6 ⁱⁱⁱ —K2—O4	137.47 (10)	C3—C2—H2	118.7
633	O3 ⁱⁱⁱ —K2—O3	65.65 (10)	C2—C3—C4	116.6 (4)
634	O4W—K2—O3	81.82 (11)	C2—C3—H3	121.7
635	O3W—K2—O3	108.46 (9)	C4—C3—H3	121.7
636	O1—K2—O3	160.24 (9)	N3—C4—C3	122.8 (5)
637	N6 ⁱⁱⁱ —K2—O3	101.97 (9)	N3—C4—H4	118.6
638	O4—K2—O3	47.93 (8)	C3—C4—H4	118.6
639	O3 ⁱⁱⁱ —K2—N1	97.57 (9)	C6—C5—C10	119.2 (4)
640	O4W—K2—N1	126.61 (12)	C6—C5—S1	121.0 (3)
641	O3W—K2—N1	56.72 (9)	C10—C5—S1	119.7 (4)
642	O1—K2—N1	46.81 (9)	C5—C6—C7	120.2 (4)
643	N6 ⁱⁱⁱ —K2—N1	89.55 (10)	C5—C6—H6	119.9
644	O4—K2—N1	131.79 (9)	C7—C6—H6	119.9
645	O3—K2—N1	151.48 (10)	C8—C7—C6	121.2 (5)
646	O3 ⁱⁱⁱ —K2—S2	85.85 (7)	C8—C7—H7	119.4

647	O4W—K2—S2	84.25 (10)	C6—C7—H7	119.4
648	O3W—K2—S2	85.87 (7)	C7—C8—C9	118.4 (4)
649	O1—K2—S2	143.14 (7)	C7—C8—N4	120.9 (5)
650	N6 ⁱⁱⁱ —K2—S2	126.55 (7)	C9—C8—N4	120.7 (5)
651	O4—K2—S2	25.39 (6)	C8—C9—C10	121.0 (4)
652	O3—K2—S2	25.46 (6)	C8—C9—H9	119.5
653	N1—K2—S2	139.86 (8)	C10—C9—H9	119.5
654	O3 ⁱⁱⁱ —K2—C12 ⁱⁱⁱ	86.35 (10)	C9—C10—C5	119.9 (5)
655	O4W—K2—C12 ⁱⁱⁱ	61.29 (12)	C9—C10—H10	120.1
656	O3W—K2—C12 ⁱⁱⁱ	136.33 (10)	C5—C10—H10	120.1
657	O1—K2—C12 ⁱⁱⁱ	62.11 (10)	N6—C11—N7	124.8 (4)
658	N6 ⁱⁱⁱ —K2—C12 ⁱⁱⁱ	22.46 (10)	N6—C11—N5	121.6 (4)
659	O4—K2—C12 ⁱⁱⁱ	132.77 (10)	N7—C11—N5	113.6 (4)
660	O3—K2—C12 ⁱⁱⁱ	114.98 (9)	N6—C11—K1	154.2 (3)
661	N1—K2—C12 ⁱⁱⁱ	85.10 (10)	N7—C11—K1	56.4 (2)
662	S2—K2—C12 ⁱⁱⁱ	135.02 (8)	N5—C11—K1	64.8 (2)
663	O3 ⁱⁱⁱ —K2—S1	113.47 (7)	N6—C12—C13	123.7 (4)
664	O4W—K2—S1	101.02 (10)	N6—C12—K2 ⁱⁱⁱ	56.0 (2)
665	O3W—K2—S1	69.16 (7)	C13—C12—K2 ⁱⁱⁱ	143.9 (3)
666	O1—K2—S1	22.03 (6)	N6—C12—H12	118.2
667	N6 ⁱⁱⁱ —K2—S1	79.88 (7)	C13—C12—H12	118.2
668	O4—K2—S1	131.84 (6)	K2 ⁱⁱⁱ —C12—H12	74.7
669	O3—K2—S1	176.98 (7)	C14—C13—C12	115.2 (4)
670	N1—K2—S1	25.63 (7)	C14—C13—H13	122.4
671	S2—K2—S1	153.19 (4)	C12—C13—H13	122.4
672	C12 ⁱⁱⁱ —K2—S1	67.56 (8)	N7—C14—C13	123.8 (5)
673	O3 ⁱⁱⁱ —K2—K1 ⁱ	140.99 (7)	N7—C14—H14	118.1
674	O4W—K2—K1 ⁱ	81.88 (9)	C13—C14—H14	118.1
675	O3W—K2—K1 ⁱ	45.08 (7)	C20—C15—C16	120.1 (4)
676	O1—K2—K1 ⁱ	82.84 (7)	C20—C15—S2	120.3 (3)
677	N6 ⁱⁱⁱ —K2—K1 ⁱ	153.33 (8)	C16—C15—S2	119.6 (3)
678	O4—K2—K1 ⁱ	39.76 (6)	C17—C16—C15	120.0 (4)
679	O3—K2—K1 ⁱ	86.84 (6)	C17—C16—H16	120.0
680	N1—K2—K1 ⁱ	94.45 (7)	C15—C16—H16	120.0
681	S2—K2—K1 ⁱ	61.95 (3)	C16—C17—C18	121.0 (4)
682	C12 ⁱⁱⁱ —K2—K1 ⁱ	131.72 (8)	C16—C17—H17	119.5
683	S1—K2—K1 ⁱ	92.52 (3)	C18—C17—H17	119.5
684	O3 ⁱⁱⁱ —K2—H5W	111.4 (10)	N8—C18—C19	120.3 (4)
685	O4W—K2—H5W	130.2 (11)	N8—C18—C17	121.9 (4)
686	O3W—K2—H5W	17.7 (2)	C19—C18—C17	117.7 (4)
687	O1—K2—H5W	65.1 (9)	C20—C19—C18	121.6 (4)
688	N6 ⁱⁱⁱ —K2—H5W	128.6 (3)	C20—C19—H19	119.2
689	O4—K2—H5W	93.3 (2)	C18—C19—H19	119.2
690	O3—K2—H5W	123.2 (7)	C19—C20—C15	119.5 (4)
691	N1—K2—H5W	39.1 (3)	C19—C20—H20	120.2
692	S2—K2—H5W	102.5 (4)	C15—C20—H20	120.2
693				
694	O2—S1—O1—K2	-107.96 (19)	C10—C5—C6—C7	0.0 (7)

695	N1—S1—O1—K2	18.6 (2)	S1—C5—C6—C7	176.6 (3)
696	C5—S1—O1—K2	132.83 (18)	C5—C6—C7—C8	-1.8 (7)
697	O4—S2—O3—K2 ⁱⁱⁱ	-161.4 (2)	C6—C7—C8—C9	2.5 (7)
698	N5—S2—O3—K2 ⁱⁱⁱ	-40.7 (3)	C6—C7—C8—N4	179.0 (4)
699	C15—S2—O3—K2 ⁱⁱⁱ	81.7 (3)	C7—C8—C9—C10	-1.4 (7)
700	K2—S2—O3—K2 ⁱⁱⁱ	-126.1 (2)	N4—C8—C9—C10	-178.0 (5)
701	O4—S2—O3—K2	-35.30 (18)	C8—C9—C10—C5	-0.3 (8)
702	N5—S2—O3—K2	85.46 (17)	C6—C5—C10—C9	1.0 (7)
703	C15—S2—O3—K2	-152.14 (15)	S1—C5—C10—C9	-175.6 (4)
704	O3—S2—O4—K1 ⁱ	143.5 (3)	C12—N6—C11—N7	-4.4 (6)
705	N5—S2—O4—K1 ⁱ	17.7 (3)	K2 ⁱⁱⁱ —N6—C11—N7	121.7 (4)
706	C15—S2—O4—K1 ⁱ	-99.4 (3)	C12—N6—C11—N5	174.9 (4)
707	K2—S2—O4—K1 ⁱ	107.0 (3)	K2 ⁱⁱⁱ —N6—C11—N5	-59.0 (5)
708	O3—S2—O4—K2	36.46 (19)	C12—N6—C11—K1	-88.0 (7)
709	N5—S2—O4—K2	-89.27 (17)	K2 ⁱⁱⁱ —N6—C11—K1	38.2 (8)
710	C15—S2—O4—K2	153.58 (16)	C14—N7—C11—N6	4.7 (7)
711	O2—S1—N1—C1	-52.2 (4)	K1—N7—C11—N6	-148.8 (4)
712	O1—S1—N1—C1	-177.3 (3)	C14—N7—C11—N5	-174.6 (4)
713	C5—S1—N1—C1	69.1 (3)	K1—N7—C11—N5	32.0 (4)
714	K2—S1—N1—C1	-161.3 (4)	C14—N7—C11—K1	153.4 (5)
715	O2—S1—N1—K2	109.16 (17)	S2—N5—C11—N6	-3.2 (6)
716	O1—S1—N1—K2	-15.96 (17)	K1—N5—C11—N6	151.5 (4)
717	C5—S1—N1—K2	-129.60 (16)	S2—N5—C11—N7	176.2 (3)
718	O3—S2—N5—C11	54.6 (4)	K1—N5—C11—N7	-29.2 (4)
719	O4—S2—N5—C11	179.4 (3)	S2—N5—C11—K1	-154.7 (3)
720	C15—S2—N5—C11	-66.0 (4)	C11—N6—C12—C13	0.4 (6)
721	K2—S2—N5—C11	118.3 (3)	K2 ⁱⁱⁱ —N6—C12—C13	-136.1 (4)
722	O3—S2—N5—K1	-85.3 (3)	C11—N6—C12—K2 ⁱⁱⁱ	136.5 (4)
723	O4—S2—N5—K1	39.5 (3)	N6—C12—C13—C14	2.8 (7)
724	C15—S2—N5—K1	154.1 (2)	K2 ⁱⁱⁱ —C12—C13—C14	-74.1 (7)
725	K2—S2—N5—K1	-21.6 (3)	C11—N7—C14—C13	-1.0 (7)
726	C2—N2—C1—N3	4.1 (6)	K1—N7—C14—C13	140.9 (4)
727	C2—N2—C1—N1	-175.5 (4)	C12—C13—C14—N7	-2.5 (7)
728	C4—N3—C1—N2	-4.1 (6)	O3—S2—C15—C20	-20.6 (4)
729	C4—N3—C1—N1	175.5 (4)	O4—S2—C15—C20	-141.6 (3)
730	S1—N1—C1—N2	175.3 (3)	N5—S2—C15—C20	104.4 (4)
731	K2—N1—C1—N2	25.0 (6)	K2—S2—C15—C20	-85.0 (4)
732	S1—N1—C1—N3	-4.4 (5)	O3—S2—C15—C16	160.5 (3)
733	K2—N1—C1—N3	-154.7 (3)	O4—S2—C15—C16	39.5 (4)
734	C1—N2—C2—C3	0.0 (7)	N5—S2—C15—C16	-74.4 (4)
735	N2—C2—C3—C4	-3.6 (7)	K2—S2—C15—C16	96.1 (4)
736	C1—N3—C4—C3	-0.1 (6)	C20—C15—C16—C17	0.4 (6)
737	C2—C3—C4—N3	3.7 (7)	S2—C15—C16—C17	179.2 (3)
738	O2—S1—C5—C6	143.5 (4)	C15—C16—C17—C18	1.4 (7)
739	O1—S1—C5—C6	-94.0 (4)	C16—C17—C18—N8	176.3 (4)
740	N1—S1—C5—C6	17.7 (4)	C16—C17—C18—C19	-1.8 (7)
741	K2—S1—C5—C6	-46.4 (4)	N8—C18—C19—C20	-177.7 (4)
742	O2—S1—C5—C10	-39.9 (4)	C17—C18—C19—C20	0.5 (6)

743	O1—S1—C5—C10	82.6 (4)	C18—C19—C20—C15	1.3 (6)
744	N1—S1—C5—C10	-165.7 (4)	C16—C15—C20—C19	-1.7 (6)
745	K2—S1—C5—C10	130.1 (3)	S2—C15—C20—C19	179.4 (3)

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

747 *Hydrogen-bond geometry* ($\text{\AA}, ^\circ$)

748	$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
749	O1W—H1W \cdots O1 ⁱ	0.88 (1)	1.93 (2)	2.799 (5)	167 (8)
750	O1W—H2W \cdots O4W ^x	0.88 (1)	2.27 (4)	3.070 (6)	151 (6)
751	O2W—H3W \cdots O3 ⁱⁱⁱ	0.88 (1)	2.07 (2)	2.933 (5)	167 (6)
752	O2W—H4W \cdots N2	0.88 (1)	1.97 (2)	2.828 (5)	167 (6)
753	O3W—H5W \cdots N1	0.87 (1)	2.02 (2)	2.872 (5)	164 (5)
754	O3W—H6W \cdots N5 ⁱ	0.87 (1)	1.98 (1)	2.835 (5)	166 (4)
755	O4W—H7W \cdots O2W ^{iv}	0.88 (1)	2.05 (2)	2.919 (6)	169 (7)
756	O4W—H8W \cdots O4W ^v	0.88 (1)	2.05 (2)	2.920 (8)	168 (9)
757	N4—H1N \cdots N4 ^{vi}	0.87 (7)	2.50 (6)	3.054 (9)	122 (5)
758	N4—H2N \cdots O2 ^{vii}	0.93 (7)	2.57 (7)	3.431 (7)	153 (5)
759	N8—H3N \cdots O2 ^{viii}	0.87 (5)	2.38 (5)	3.046 (5)	134 (4)
760	N8—H3N \cdots N3 ^{viii}	0.87 (5)	2.61 (5)	3.283 (6)	135 (4)

761 Symmetry codes: (i) $-x, -y+1, -z+1$; (iii) $-x, -y+2, -z+1$; (iv) $x-1, y, z$; (v) $-x-1, -y+2, -z+1$; (vi) $-x-1, -y, -z$; (vii) $x, y-1, z$; (viii) $x, y, z+1$.

762 **(IV)**

763 *Crystal data*

764 $\text{C}_{26}\text{H}_{32}\text{N}_6\text{NaO}_{14.50}\text{S}_3$

765 $M_r = 779.74$

766 Monoclinic, $P2_1/c$

767 $a = 12.7688 (1) \text{\AA}$

768 $b = 16.9040 (1) \text{\AA}$

769 $c = 15.7411 (1) \text{\AA}$

770 $\beta = 107.609 (1)^\circ$

771 $V = 3238.42 (4) \text{\AA}^3$

772 $Z = 4$

$F(000) = 1620$

$D_x = 1.599 \text{ Mg m}^{-3}$

Cu $K\alpha$ radiation, $\lambda = 1.54184 \text{\AA}$

Cell parameters from 35404 reflections

$\theta = 3.6\text{--}70.3^\circ$

$\mu = 2.95 \text{ mm}^{-1}$

$T = 100 \text{ K}$

Block, red

$0.12 \times 0.08 \times 0.04 \text{ mm}$

773 *Data collection*

774 XtaLAB AFC11 (RCD3)

diffractometer

775 Radiation source: rotating anode

776 ω scans

777 Absorption correction: multi-scan

CrysAlis PRO 1.171.39.34b (Rigaku Oxford

Diffraction, 2017) Empirical absorption

correction using spherical harmonics,

implemented in SCALE3 ABSPACK scaling algorithm.

$T_{\min} = 0.615, T_{\max} = 1.000$

59125 measured reflections

5913 independent reflections

5686 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.027$

$\theta_{\max} = 68.3^\circ, \theta_{\min} = 3.6^\circ$

$h = -15 \rightarrow 15$

$k = -20 \rightarrow 20$

$l = -18 \rightarrow 17$

778 *Refinement*

779 Refinement on F^2

780 Least-squares matrix: full

781 $R[F^2 > 2\sigma(F^2)] = 0.028$

$wR(F^2) = 0.075$

$S = 1.05$

5913 reflections

782	537 parameters	$w = 1/[\sigma^2(F_o^2) + (0.0364P)^2 + 2.347P]$
783	13 restraints	where $P = (F_o^2 + 2F_c^2)/3$
784	Hydrogen site location: mixed	$(\Delta/\sigma)_{\max} = 0.001$
785	H atoms treated by a mixture of independent and constrained refinement	$\Delta\rho_{\max} = 0.26 \text{ e } \text{\AA}^{-3}$ $\Delta\rho_{\min} = -0.39 \text{ e } \text{\AA}^{-3}$

786 *Special details*

787 **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.

788 *Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (\AA^2)*

789		<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$	Occ. (<1)
790	Na1	-0.36434 (5)	0.35305 (4)	0.00412 (4)	0.02103 (14)	
791	S1	0.25698 (3)	0.53159 (2)	0.56811 (2)	0.01894 (9)	
792	S2	0.28977 (3)	0.35439 (2)	0.12953 (2)	0.01723 (9)	
793	S3	0.34948 (3)	0.66079 (2)	0.22267 (2)	0.01823 (9)	
794	O1	0.30756 (9)	0.60882 (7)	0.58033 (7)	0.0231 (2)	
795	O2	0.25734 (9)	0.48924 (7)	0.64777 (7)	0.0232 (2)	
796	O1W	-0.35372 (9)	0.22020 (7)	0.04238 (8)	0.0218 (2)	
797	O3	-0.16759 (8)	0.36126 (6)	0.04200 (7)	0.0209 (2)	
798	O2W	-0.39276 (13)	0.36471 (9)	0.15242 (9)	0.0366 (3)	
799	O4	0.23184 (9)	0.28982 (6)	0.15504 (8)	0.0242 (2)	
800	O3W	-0.56243 (11)	0.33362 (8)	-0.06717 (10)	0.0324 (3)	
801	O5	0.29466 (9)	0.35183 (7)	0.03839 (7)	0.0247 (3)	
802	O4W	-0.40375 (13)	0.48626 (9)	-0.02427 (12)	0.0467 (4)	
803	H7W	-0.3673 (17)	0.5308 (9)	-0.0208 (17)	0.056*	
804	H8W	-0.449 (3)	0.501 (2)	0.006 (3)	0.056*	0.5
805	H14W	-0.463 (2)	0.499 (2)	-0.068 (2)	0.056*	0.5
806	O5W	-0.6489 (2)	0.33037 (15)	-0.24880 (18)	0.0323 (5)	0.5
807	H9W	-0.677 (3)	0.2873 (16)	-0.279 (2)	0.039*	0.5
808	H10W	-0.590 (2)	0.336 (2)	-0.267 (3)	0.039*	0.5
809	O6W	-0.3558 (3)	0.52405 (18)	0.16139 (18)	0.0459 (8)	0.5
810	H11W	-0.366 (2)	0.5317 (15)	0.2141 (10)	0.055*	
811	H12W	-0.367 (4)	0.4725 (8)	0.160 (3)	0.055*	0.5
812	O7W	-0.4311 (2)	0.54771 (18)	0.19266 (18)	0.0392 (7)	0.5
813	H13W	-0.451 (3)	0.566 (3)	0.2374 (18)	0.047*	0.5
814	O6	0.40067 (8)	0.36455 (6)	0.19268 (7)	0.0200 (2)	
815	O7	0.36235 (9)	0.69334 (7)	0.14107 (7)	0.0228 (2)	
816	O8	0.44918 (9)	0.62573 (7)	0.28078 (8)	0.0297 (3)	
817	O9	0.29864 (9)	0.71689 (7)	0.26880 (7)	0.0241 (2)	
818	N1	0.13018 (10)	0.53245 (8)	0.50886 (8)	0.0193 (3)	
819	N2	0.14382 (11)	0.63967 (8)	0.41387 (9)	0.0205 (3)	
820	N3	-0.02496 (10)	0.58329 (8)	0.41174 (9)	0.0202 (3)	
821	N4	0.47872 (12)	0.31848 (9)	0.37342 (10)	0.0228 (3)	
822	N5	0.04786 (10)	0.34343 (7)	0.02685 (8)	0.0165 (3)	
823	N6	-0.02618 (10)	0.29254 (7)	-0.01560 (8)	0.0173 (3)	

824	C1	0.08718 (12)	0.58593 (9)	0.44525 (10)	0.0185 (3)
825	C2	0.08701 (13)	0.69124 (10)	0.35332 (10)	0.0216 (3)
826	H2	0.1270	0.7293	0.3313	0.026*
827	C3	-0.02726 (13)	0.69278 (10)	0.32031 (11)	0.0232 (3)
828	H3	-0.0650	0.7314	0.2784	0.028*
829	C4	-0.08209 (13)	0.63587 (10)	0.35134 (11)	0.0238 (3)
830	H4	-0.1600	0.6334	0.3304	0.029*
831	C5	0.32606 (12)	0.47095 (9)	0.51081 (10)	0.0197 (3)
832	C6	0.35300 (12)	0.49962 (10)	0.43696 (10)	0.0223 (3)
833	H6	0.3365	0.5527	0.4176	0.027*
834	C7	0.40425 (12)	0.44943 (10)	0.39231 (10)	0.0219 (3)
835	H7	0.4236	0.4677	0.3420	0.026*
836	C8	0.42703 (12)	0.37216 (10)	0.42193 (10)	0.0205 (3)
837	C9	0.40333 (13)	0.34437 (10)	0.49661 (11)	0.0221 (3)
838	H9	0.4223	0.2919	0.5171	0.027*
839	C10	0.35154 (12)	0.39416 (10)	0.54122 (10)	0.0218 (3)
840	H10	0.3336	0.3759	0.5922	0.026*
841	C11	0.01948 (12)	0.40012 (9)	0.07444 (10)	0.0159 (3)
842	C12	-0.09441 (12)	0.41261 (9)	0.07379 (9)	0.0175 (3)
843	C13	-0.12211 (12)	0.48735 (9)	0.10541 (10)	0.0198 (3)
844	H13	-0.1955	0.4971	0.1051	0.024*
845	C14	-0.04523 (12)	0.54336 (9)	0.13538 (10)	0.0188 (3)
846	H14	-0.0671	0.5934	0.1516	0.023*
847	C15	0.06970 (12)	0.53059 (9)	0.14404 (10)	0.0166 (3)
848	C16	0.10495 (12)	0.45694 (9)	0.11881 (9)	0.0157 (3)
849	C17	0.22061 (12)	0.44559 (9)	0.13890 (10)	0.0168 (3)
850	C18	0.29200 (12)	0.50782 (9)	0.17202 (10)	0.0186 (3)
851	H18	0.3687	0.4999	0.1835	0.022*
852	C19	0.25427 (12)	0.58168 (9)	0.18898 (10)	0.0178 (3)
853	C20	0.14397 (12)	0.59221 (9)	0.17777 (10)	0.0178 (3)
854	H20	0.1182	0.6413	0.1929	0.021*
855	C21	-0.00296 (12)	0.23542 (9)	-0.07255 (10)	0.0178 (3)
856	C22	-0.09205 (13)	0.19251 (9)	-0.12561 (10)	0.0204 (3)
857	H22	-0.1643	0.2031	-0.1234	0.024*
858	C23	-0.07414 (14)	0.13423 (10)	-0.18169 (11)	0.0237 (3)
859	H23	-0.1343	0.1047	-0.2182	0.028*
860	C24	0.03150 (14)	0.11899 (10)	-0.18464 (11)	0.0243 (3)
861	H24	0.0438	0.0786	-0.2225	0.029*
862	C25	0.11952 (14)	0.16297 (10)	-0.13190 (11)	0.0242 (3)
863	H25	0.1916	0.1526	-0.1346	0.029*
864	C26	0.10358 (13)	0.22174 (10)	-0.07551 (10)	0.0211 (3)
865	H26	0.1637	0.2519	-0.0398	0.025*
866	H1W	-0.349 (2)	0.2133 (14)	0.0986 (18)	0.049 (7)*
867	H2W	-0.299 (2)	0.1888 (15)	0.0354 (16)	0.052 (7)*
868	H3W	-0.360 (2)	0.3264 (17)	0.1861 (18)	0.057 (8)*
869	H4W	-0.461 (3)	0.3655 (19)	0.161 (2)	0.089 (11)*
870	H5W	-0.607 (2)	0.3395 (16)	-0.0361 (19)	0.061 (8)*
871	H6W	-0.591 (3)	0.365 (2)	-0.109 (2)	0.086 (11)*

872	H1N	−0.0943 (19)	0.2989 (13)	−0.0086 (14)	0.039 (6)*
873	H2N	0.4668 (19)	0.2671 (15)	0.3875 (16)	0.046 (6)*
874	H3N	0.4510 (18)	0.3275 (13)	0.3137 (16)	0.036 (6)*
875	H4N	0.552 (2)	0.3289 (13)	0.3887 (14)	0.036 (6)*
876	H5N	−0.0568 (18)	0.5484 (13)	0.4332 (14)	0.034 (6)*

877 *Atomic displacement parameters (Å²)*

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}	
879	Na1	0.0167 (3)	0.0247 (3)	0.0222 (3)	0.0009 (2)	0.0066 (2)	0.0023 (2)
880	S1	0.01371 (18)	0.0258 (2)	0.01688 (18)	0.00205 (14)	0.00401 (14)	0.00107 (14)
881	S2	0.01208 (17)	0.02132 (19)	0.01829 (19)	0.00029 (13)	0.00458 (14)	0.00044 (14)
882	S3	0.01672 (18)	0.02092 (19)	0.01605 (18)	−0.00479 (14)	0.00343 (14)	−0.00034 (14)
883	O1	0.0168 (5)	0.0278 (6)	0.0235 (6)	−0.0008 (4)	0.0044 (4)	−0.0022 (5)
884	O2	0.0188 (5)	0.0335 (6)	0.0174 (5)	0.0043 (5)	0.0054 (4)	0.0033 (5)
885	O1W	0.0204 (6)	0.0252 (6)	0.0199 (6)	0.0006 (5)	0.0061 (4)	0.0006 (5)
886	O3	0.0146 (5)	0.0233 (6)	0.0249 (6)	−0.0033 (4)	0.0061 (4)	−0.0030 (4)
887	O2W	0.0409 (8)	0.0400 (8)	0.0350 (7)	0.0161 (6)	0.0208 (6)	0.0089 (6)
888	O4	0.0180 (5)	0.0200 (5)	0.0352 (6)	−0.0002 (4)	0.0090 (5)	0.0039 (5)
889	O3W	0.0246 (6)	0.0369 (7)	0.0407 (8)	0.0063 (5)	0.0171 (6)	0.0062 (6)
890	O5	0.0170 (5)	0.0378 (7)	0.0201 (6)	0.0016 (5)	0.0069 (4)	−0.0037 (5)
891	O4W	0.0522 (9)	0.0349 (7)	0.0683 (11)	0.0142 (7)	0.0413 (8)	0.0217 (7)
892	O5W	0.0279 (13)	0.0334 (14)	0.0360 (14)	−0.0018 (10)	0.0103 (11)	−0.0034 (11)
893	O6W	0.070 (2)	0.0441 (16)	0.0218 (13)	0.0273 (15)	0.0117 (13)	0.0043 (12)
894	O7W	0.0281 (14)	0.0483 (17)	0.0362 (15)	0.0008 (12)	0.0021 (11)	0.0159 (13)
895	O6	0.0129 (5)	0.0250 (6)	0.0204 (5)	0.0012 (4)	0.0026 (4)	0.0015 (4)
896	O7	0.0257 (6)	0.0247 (6)	0.0198 (6)	−0.0074 (5)	0.0098 (5)	−0.0014 (4)
897	O8	0.0205 (6)	0.0305 (6)	0.0299 (6)	−0.0045 (5)	−0.0047 (5)	0.0029 (5)
898	O9	0.0250 (6)	0.0269 (6)	0.0223 (6)	−0.0072 (5)	0.0101 (5)	−0.0067 (5)
899	N1	0.0130 (6)	0.0250 (7)	0.0192 (6)	0.0018 (5)	0.0039 (5)	0.0018 (5)
900	N2	0.0174 (6)	0.0246 (7)	0.0192 (6)	0.0006 (5)	0.0050 (5)	0.0011 (5)
901	N3	0.0142 (6)	0.0266 (7)	0.0201 (7)	0.0013 (5)	0.0058 (5)	0.0025 (5)
902	N4	0.0166 (7)	0.0317 (8)	0.0209 (7)	0.0058 (6)	0.0066 (6)	0.0019 (6)
903	N5	0.0158 (6)	0.0176 (6)	0.0149 (6)	−0.0007 (5)	0.0028 (5)	0.0023 (5)
904	N6	0.0139 (6)	0.0202 (6)	0.0172 (6)	−0.0010 (5)	0.0038 (5)	−0.0005 (5)
905	C1	0.0158 (7)	0.0226 (8)	0.0173 (7)	0.0023 (6)	0.0053 (6)	−0.0031 (6)
906	C2	0.0233 (8)	0.0234 (8)	0.0191 (8)	0.0006 (6)	0.0078 (6)	−0.0010 (6)
907	C3	0.0225 (8)	0.0274 (8)	0.0193 (8)	0.0058 (7)	0.0057 (6)	0.0030 (6)
908	C4	0.0168 (8)	0.0331 (9)	0.0202 (8)	0.0056 (6)	0.0040 (6)	0.0021 (7)
909	C5	0.0114 (7)	0.0282 (8)	0.0183 (7)	0.0010 (6)	0.0028 (6)	0.0003 (6)
910	C6	0.0180 (7)	0.0275 (8)	0.0202 (8)	0.0030 (6)	0.0042 (6)	0.0036 (6)
911	C7	0.0163 (7)	0.0316 (9)	0.0177 (7)	0.0027 (6)	0.0050 (6)	0.0039 (6)
912	C8	0.0115 (7)	0.0304 (9)	0.0186 (8)	0.0032 (6)	0.0032 (6)	0.0002 (6)
913	C9	0.0174 (7)	0.0271 (8)	0.0214 (8)	0.0041 (6)	0.0052 (6)	0.0039 (6)
914	C10	0.0159 (7)	0.0304 (9)	0.0190 (8)	0.0014 (6)	0.0050 (6)	0.0035 (6)
915	C11	0.0144 (7)	0.0181 (7)	0.0145 (7)	0.0001 (6)	0.0036 (5)	0.0024 (6)
916	C12	0.0155 (7)	0.0224 (8)	0.0140 (7)	−0.0009 (6)	0.0035 (6)	0.0024 (6)
917	C13	0.0150 (7)	0.0251 (8)	0.0203 (8)	0.0015 (6)	0.0069 (6)	0.0004 (6)

918	C14	0.0189 (7)	0.0205 (8)	0.0179 (7)	0.0021 (6)	0.0067 (6)	0.0009 (6)
919	C15	0.0165 (7)	0.0205 (7)	0.0125 (7)	-0.0002 (6)	0.0041 (6)	0.0030 (6)
920	C16	0.0153 (7)	0.0202 (7)	0.0117 (7)	-0.0006 (6)	0.0045 (6)	0.0033 (6)
921	C17	0.0157 (7)	0.0207 (7)	0.0141 (7)	-0.0002 (6)	0.0048 (6)	0.0026 (6)
922	C18	0.0148 (7)	0.0240 (8)	0.0161 (7)	-0.0016 (6)	0.0034 (6)	0.0037 (6)
923	C19	0.0175 (7)	0.0212 (7)	0.0131 (7)	-0.0032 (6)	0.0024 (6)	0.0029 (6)
924	C20	0.0199 (7)	0.0189 (7)	0.0147 (7)	-0.0010 (6)	0.0051 (6)	0.0014 (6)
925	C21	0.0201 (7)	0.0175 (7)	0.0155 (7)	0.0010 (6)	0.0049 (6)	0.0021 (6)
926	C22	0.0189 (7)	0.0225 (8)	0.0196 (8)	-0.0017 (6)	0.0055 (6)	0.0016 (6)
927	C23	0.0256 (8)	0.0234 (8)	0.0209 (8)	-0.0058 (6)	0.0051 (7)	-0.0024 (6)
928	C24	0.0312 (9)	0.0229 (8)	0.0187 (8)	0.0022 (7)	0.0077 (7)	-0.0019 (6)
929	C25	0.0208 (8)	0.0294 (9)	0.0225 (8)	0.0046 (7)	0.0065 (6)	-0.0006 (7)
930	C26	0.0179 (7)	0.0247 (8)	0.0193 (8)	0.0003 (6)	0.0034 (6)	-0.0009 (6)

931 *Geometric parameters (Å, °)*

932	Na1—O1W	2.3184 (13)	N5—C11	1.3322 (19)
933	Na1—O4W	2.3211 (15)	N6—C21	1.409 (2)
934	Na1—O3	2.4042 (12)	N6—H1N	0.92 (2)
935	Na1—O7 ⁱ	2.4237 (12)	C2—C3	1.393 (2)
936	Na1—O3W	2.4592 (14)	C2—H2	0.9500
937	Na1—O2W	2.4764 (14)	C3—C4	1.364 (2)
938	S1—O2	1.4428 (11)	C3—H3	0.9500
939	S1—O1	1.4433 (12)	C4—H4	0.9500
940	S1—N1	1.6055 (13)	C5—C10	1.388 (2)
941	S1—C5	1.7674 (16)	C5—C6	1.395 (2)
942	S2—O4	1.4419 (11)	C6—C7	1.386 (2)
943	S2—O5	1.4554 (12)	C6—H6	0.9500
944	S2—O6	1.4735 (11)	C7—C8	1.388 (2)
945	S2—C17	1.8048 (15)	C7—H7	0.9500
946	S3—O8	1.4506 (12)	C8—C9	1.382 (2)
947	S3—O7	1.4516 (11)	C9—C10	1.385 (2)
948	S3—O9	1.4605 (12)	C9—H9	0.9500
949	S3—C19	1.7769 (15)	C10—H10	0.9500
950	O1W—H1W	0.88 (3)	C11—C16	1.463 (2)
951	O1W—H2W	0.91 (3)	C11—C12	1.467 (2)
952	O3—C12	1.2626 (18)	C12—C13	1.441 (2)
953	O2W—H3W	0.86 (3)	C13—C14	1.343 (2)
954	O2W—H4W	0.92 (4)	C13—H13	0.9500
955	O3W—H5W	0.86 (3)	C14—C15	1.448 (2)
956	O3W—H6W	0.83 (4)	C14—H14	0.9500
957	O4W—H7W	0.879 (10)	C15—C20	1.401 (2)
958	O4W—H8W	0.883 (10)	C15—C16	1.421 (2)
959	O4W—H14W	0.886 (10)	C16—C17	1.427 (2)
960	O5W—H9W	0.883 (10)	C17—C18	1.386 (2)
961	O5W—H10W	0.885 (10)	C18—C19	1.393 (2)
962	O6W—H11W	0.886 (10)	C18—H18	0.9500
963	O6W—H12W	0.881 (10)	C19—C20	1.377 (2)

964	O7W—H13W	0.875 (10)	C20—H20	0.9500
965	O7—Na1 ⁱ	2.4237 (12)	C21—C22	1.394 (2)
966	N1—C1	1.337 (2)	C21—C26	1.395 (2)
967	N2—C2	1.333 (2)	C22—C23	1.387 (2)
968	N2—C1	1.345 (2)	C22—H22	0.9500
969	N3—C4	1.344 (2)	C23—C24	1.388 (2)
970	N3—C1	1.369 (2)	C23—H23	0.9500
971	N3—H5N	0.84 (2)	C24—C25	1.393 (2)
972	N4—C8	1.465 (2)	C24—H24	0.9500
973	N4—H2N	0.92 (3)	C25—C26	1.388 (2)
974	N4—H3N	0.91 (2)	C25—H25	0.9500
975	N4—H4N	0.91 (2)	C26—H26	0.9500
976	N5—N6	1.3031 (18)		
977				
978	O1W—Na1—O4W	169.11 (5)	C4—C3—C2	116.51 (15)
979	O1W—Na1—O3	90.96 (4)	C4—C3—H3	121.7
980	O4W—Na1—O3	98.12 (5)	C2—C3—H3	121.7
981	O1W—Na1—O7 ⁱ	85.13 (4)	N3—C4—C3	119.48 (15)
982	O4W—Na1—O7 ⁱ	101.30 (5)	N3—C4—H4	120.3
983	O3—Na1—O7 ⁱ	86.81 (4)	C3—C4—H4	120.3
984	O1W—Na1—O3W	87.73 (5)	C10—C5—C6	121.41 (15)
985	O4W—Na1—O3W	84.75 (6)	C10—C5—S1	117.89 (12)
986	O3—Na1—O3W	166.97 (5)	C6—C5—S1	120.71 (12)
987	O7 ⁱ —Na1—O3W	80.17 (5)	C7—C6—C5	118.90 (15)
988	O1W—Na1—O2W	81.14 (5)	C7—C6—H6	120.6
989	O4W—Na1—O2W	91.10 (6)	C5—C6—H6	120.6
990	O3—Na1—O2W	101.64 (5)	C6—C7—C8	119.27 (15)
991	O7 ⁱ —Na1—O2W	163.94 (5)	C6—C7—H7	120.4
992	O3W—Na1—O2W	90.97 (5)	C8—C7—H7	120.4
993	O2—S1—O1	116.71 (7)	C9—C8—C7	121.88 (15)
994	O2—S1—N1	104.29 (7)	C9—C8—N4	118.81 (15)
995	O1—S1—N1	113.65 (7)	C7—C8—N4	119.30 (14)
996	O2—S1—C5	106.55 (7)	C8—C9—C10	119.06 (15)
997	O1—S1—C5	108.68 (7)	C8—C9—H9	120.5
998	N1—S1—C5	106.26 (7)	C10—C9—H9	120.5
999	O4—S2—O5	115.51 (7)	C9—C10—C5	119.43 (15)
1000	O4—S2—O6	111.69 (7)	C9—C10—H10	120.3
1001	O5—S2—O6	110.45 (6)	C5—C10—H10	120.3
1002	O4—S2—C17	108.60 (7)	N5—C11—C16	116.76 (13)
1003	O5—S2—C17	105.90 (7)	N5—C11—C12	122.51 (13)
1004	O6—S2—C17	103.85 (7)	C16—C11—C12	120.25 (13)
1005	O8—S3—O7	113.59 (7)	O3—C12—C13	120.68 (13)
1006	O8—S3—O9	112.90 (7)	O3—C12—C11	121.14 (14)
1007	O7—S3—O9	112.34 (7)	C13—C12—C11	118.06 (13)
1008	O8—S3—C19	105.75 (7)	C14—C13—C12	120.66 (14)
1009	O7—S3—C19	105.76 (7)	C14—C13—H13	119.7
1010	O9—S3—C19	105.67 (7)	C12—C13—H13	119.7
1011	Na1—O1W—H1W	111.8 (16)	C13—C14—C15	122.71 (14)

1012	Na1—O1W—H2W	121.3 (16)	C13—C14—H14	118.6
1013	H1W—O1W—H2W	103 (2)	C15—C14—H14	118.6
1014	C12—O3—Na1	136.93 (10)	C20—C15—C16	121.48 (13)
1015	Na1—O2W—H3W	110.0 (18)	C20—C15—C14	118.39 (14)
1016	Na1—O2W—H4W	124 (2)	C16—C15—C14	120.13 (13)
1017	H3W—O2W—H4W	102 (3)	C15—C16—C17	116.75 (13)
1018	Na1—O3W—H5W	119.2 (19)	C15—C16—C11	117.08 (13)
1019	Na1—O3W—H6W	115 (2)	C17—C16—C11	126.18 (13)
1020	H5W—O3W—H6W	100 (3)	C18—C17—C16	120.02 (14)
1021	Na1—O4W—H7W	137.7 (16)	C18—C17—S2	113.36 (11)
1022	Na1—O4W—H8W	108 (3)	C16—C17—S2	126.58 (11)
1023	H7W—O4W—H8W	98 (2)	C17—C18—C19	121.74 (14)
1024	Na1—O4W—H14W	118 (3)	C17—C18—H18	119.1
1025	H7W—O4W—H14W	99 (2)	C19—C18—H18	119.1
1026	H9W—O5W—H10W	99 (2)	C20—C19—C18	119.40 (14)
1027	H11W—O6W—H12W	96 (2)	C20—C19—S3	121.46 (12)
1028	S3—O7—Na1 ⁱ	138.42 (7)	C18—C19—S3	119.13 (11)
1029	C1—N1—S1	123.67 (11)	C19—C20—C15	120.10 (14)
1030	C2—N2—C1	117.89 (13)	C19—C20—H20	119.9
1031	C4—N3—C1	122.12 (14)	C15—C20—H20	119.9
1032	C4—N3—H5N	121.4 (15)	C22—C21—C26	121.42 (14)
1033	C1—N3—H5N	116.4 (15)	C22—C21—N6	116.56 (13)
1034	C8—N4—H2N	109.0 (15)	C26—C21—N6	122.01 (14)
1035	C8—N4—H3N	109.8 (14)	C23—C22—C21	119.28 (15)
1036	H2N—N4—H3N	111 (2)	C23—C22—H22	120.4
1037	C8—N4—H4N	109.3 (14)	C21—C22—H22	120.4
1038	H2N—N4—H4N	111 (2)	C22—C23—C24	120.15 (15)
1039	H3N—N4—H4N	106.8 (19)	C22—C23—H23	119.9
1040	N6—N5—C11	118.99 (13)	C24—C23—H23	119.9
1041	N5—N6—C21	121.27 (13)	C23—C24—C25	119.87 (15)
1042	N5—N6—H1N	115.4 (14)	C23—C24—H24	120.1
1043	C21—N6—H1N	123.2 (14)	C25—C24—H24	120.1
1044	N1—C1—N2	125.95 (14)	C26—C25—C24	121.02 (15)
1045	N1—C1—N3	114.28 (14)	C26—C25—H25	119.5
1046	N2—C1—N3	119.77 (14)	C24—C25—H25	119.5
1047	N2—C2—C3	124.12 (15)	C25—C26—C21	118.25 (14)
1048	N2—C2—H2	117.9	C25—C26—H26	120.9
1049	C3—C2—H2	117.9	C21—C26—H26	120.9
1050				
1051	O8—S3—O7—Na1 ⁱ	81.07 (11)	C13—C14—C15—C20	-178.66 (14)
1052	O9—S3—O7—Na1 ⁱ	-149.26 (9)	C13—C14—C15—C16	1.2 (2)
1053	C19—S3—O7—Na1 ⁱ	-34.46 (12)	C20—C15—C16—C17	6.9 (2)
1054	O2—S1—N1—C1	156.92 (13)	C14—C15—C16—C17	-172.92 (13)
1055	O1—S1—N1—C1	28.75 (15)	C20—C15—C16—C11	-172.79 (13)
1056	C5—S1—N1—C1	-90.72 (14)	C14—C15—C16—C11	7.4 (2)
1057	C11—N5—N6—C21	174.83 (13)	N5—C11—C16—C15	159.68 (13)
1058	S1—N1—C1—N2	8.8 (2)	C12—C11—C16—C15	-12.5 (2)
1059	S1—N1—C1—N3	-170.92 (11)	N5—C11—C16—C17	-20.0 (2)

1060	C2—N2—C1—N1	-176.66 (15)	C12—C11—C16—C17	167.76 (14)
1061	C2—N2—C1—N3	3.0 (2)	C15—C16—C17—C18	-7.1 (2)
1062	C4—N3—C1—N1	175.88 (14)	C11—C16—C17—C18	172.62 (14)
1063	C4—N3—C1—N2	-3.8 (2)	C15—C16—C17—S2	170.58 (11)
1064	C1—N2—C2—C3	-0.2 (2)	C11—C16—C17—S2	-9.7 (2)
1065	N2—C2—C3—C4	-1.9 (2)	O4—S2—C17—C18	141.14 (11)
1066	C1—N3—C4—C3	1.6 (2)	O5—S2—C17—C18	-94.23 (12)
1067	C2—C3—C4—N3	1.1 (2)	O6—S2—C17—C18	22.15 (13)
1068	O2—S1—C5—C10	9.38 (14)	O4—S2—C17—C16	-36.66 (15)
1069	O1—S1—C5—C10	135.92 (12)	O5—S2—C17—C16	87.97 (14)
1070	N1—S1—C5—C10	-101.41 (13)	O6—S2—C17—C16	-155.65 (13)
1071	O2—S1—C5—C6	-171.14 (12)	C16—C17—C18—C19	1.8 (2)
1072	O1—S1—C5—C6	-44.60 (14)	S2—C17—C18—C19	-176.12 (11)
1073	N1—S1—C5—C6	78.07 (14)	C17—C18—C19—C20	4.0 (2)
1074	C10—C5—C6—C7	1.3 (2)	C17—C18—C19—S3	-174.87 (11)
1075	S1—C5—C6—C7	-178.11 (12)	O8—S3—C19—C20	143.56 (13)
1076	C5—C6—C7—C8	0.2 (2)	O7—S3—C19—C20	-95.67 (13)
1077	C6—C7—C8—C9	-2.2 (2)	O9—S3—C19—C20	23.63 (14)
1078	C6—C7—C8—N4	178.48 (14)	O8—S3—C19—C18	-37.62 (14)
1079	C7—C8—C9—C10	2.5 (2)	O7—S3—C19—C18	83.15 (13)
1080	N4—C8—C9—C10	-178.13 (14)	O9—S3—C19—C18	-157.55 (12)
1081	C8—C9—C10—C5	-0.9 (2)	C18—C19—C20—C15	-4.2 (2)
1082	C6—C5—C10—C9	-1.0 (2)	S3—C19—C20—C15	174.65 (11)
1083	S1—C5—C10—C9	178.47 (12)	C16—C15—C20—C19	-1.4 (2)
1084	N6—N5—C11—C16	-178.41 (12)	C14—C15—C20—C19	178.45 (13)
1085	N6—N5—C11—C12	-6.4 (2)	N5—N6—C21—C22	-169.89 (13)
1086	Na1—O3—C12—C13	6.3 (2)	N5—N6—C21—C26	10.6 (2)
1087	Na1—O3—C12—C11	-169.80 (10)	C26—C21—C22—C23	0.9 (2)
1088	N5—C11—C12—O3	13.8 (2)	N6—C21—C22—C23	-178.63 (14)
1089	C16—C11—C12—O3	-174.40 (13)	C21—C22—C23—C24	0.1 (2)
1090	N5—C11—C12—C13	-162.36 (14)	C22—C23—C24—C25	-0.7 (2)
1091	C16—C11—C12—C13	9.4 (2)	C23—C24—C25—C26	0.5 (3)
1092	O3—C12—C13—C14	-176.90 (14)	C24—C25—C26—C21	0.4 (2)
1093	C11—C12—C13—C14	-0.7 (2)	C22—C21—C26—C25	-1.1 (2)
1094	C12—C13—C14—C15	-4.7 (2)	N6—C21—C26—C25	178.40 (14)

1095 Symmetry code: (i) $-x, -y+1, -z$.

1096 *Hydrogen-bond geometry* ($\text{\AA}, ^\circ$)

1097	<i>D</i> —H \cdots <i>A</i>	<i>D</i> —H	H \cdots <i>A</i>	<i>D</i> \cdots <i>A</i>	<i>D</i> —H \cdots <i>A</i>
1098	O1 <i>W</i> —H1 <i>W</i> \cdots O9 ⁱⁱ	0.88 (3)	1.99 (3)	2.8404 (16)	163 (2)
1099	O1 <i>W</i> —H2 <i>W</i> \cdots O1 ⁱⁱ	0.91 (3)	2.24 (3)	2.8830 (17)	127 (2)
1100	O1 <i>W</i> —H2 <i>W</i> \cdots N2 ⁱⁱ	0.91 (3)	2.07 (3)	2.8972 (18)	150 (2)
1101	O2 <i>W</i> —H3 <i>W</i> \cdots O9 ⁱⁱ	0.86 (3)	2.05 (3)	2.8852 (18)	163 (2)
1102	O2 <i>W</i> —H4 <i>W</i> \cdots O6 ⁱⁱⁱ	0.92 (4)	1.98 (4)	2.8932 (18)	174 (3)
1103	O3 <i>W</i> —H5 <i>W</i> \cdots O5 ⁱⁱⁱ	0.86 (3)	1.98 (3)	2.8320 (18)	178 (3)
1104	O3 <i>W</i> —H6 <i>W</i> \cdots O5 <i>W</i>	0.83 (4)	2.18 (3)	2.736 (3)	124 (3)

1105	O3W—H6W...O6W ^{iv}	0.83 (4)	2.09 (4)	2.853 (3)	153 (3)
1106	O3W—H6W...O7W ^{iv}	0.83 (4)	2.06 (4)	2.799 (3)	148 (3)
1107	O4W—H7W...O5 ⁱ	0.88 (1)	2.24 (1)	3.1093 (19)	169 (2)
1108	O4W—H8W...O4W ^{iv}	0.88 (1)	2.00 (2)	2.825 (3)	154 (4)
1109	O4W—H8W...O6W	0.88 (1)	2.41 (5)	2.871 (3)	113 (4)
1110	O4W—H14W...O7W ^{iv}	0.89 (1)	2.16 (3)	2.902 (3)	140 (4)
1111	O5W—H9W...O4 ^v	0.88 (1)	1.85 (1)	2.710 (3)	166 (4)
1112	O5W—H10W...O8 ⁱ	0.89 (1)	1.99 (1)	2.843 (3)	163 (3)
1113	O6W—H11W...O2 ^{vi}	0.89 (1)	2.22 (2)	2.893 (3)	132 (2)
1114	O6W—H12W...O2W	0.88 (1)	1.85 (1)	2.731 (3)	178 (5)
1115	O7W—H13W...O8 ⁱⁱⁱ	0.88 (1)	1.91 (2)	2.699 (3)	150 (3)
1116	N6—H1N...O3	0.92 (2)	1.75 (2)	2.5342 (16)	142 (2)
1117	N4—H2N...O3W ⁱⁱ	0.92 (3)	1.93 (3)	2.839 (2)	171 (2)
1118	N4—H3N...O6	0.91 (2)	1.92 (2)	2.8245 (18)	170 (2)
1119	N4—H4N...O1 ^{viii}	0.91 (2)	2.01 (2)	2.8784 (18)	159.6 (19)
1120	N4—H4N...O1W ⁱⁱ	0.91 (2)	2.50 (2)	2.9386 (19)	110.5 (16)
1121	N3—H5N...O2 ^{vi}	0.84 (2)	2.57 (2)	3.0827 (17)	120.3 (18)
1122	N3—H5N...N1 ^{vi}	0.84 (2)	2.02 (2)	2.8655 (19)	177 (2)

1123 Symmetry codes: (i) $-x, -y+1, -z$; (ii) $-x, y-1/2, -z+1/2$; (iii) $x-1, y, z$; (iv) $-x-1, -y+1, -z$; (v) $x-1, -y+1/2, z-1/2$; (vi) $-x, -y+1, -z+1$; (vii) $x+1, -y+1/2, z+1/2$; (viii) $-x+1, -y+1, -z+1$.

1124 (V)

1125 *Crystal data*

1126 C₁₅H₁₆N₆O₂S

1127 $M_r = 344.40$

1128 Monoclinic, $P2_1/c$

1129 $a = 8.5796$ (2) Å

1130 $b = 19.0371$ (5) Å

1131 $c = 11.2512$ (4) Å

1132 $\beta = 121.116$ (3)°

1133 $V = 1573.27$ (9) Å³

1134 $Z = 4$

$F(000) = 720$

$D_x = 1.454$ Mg m⁻³

Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å

Cell parameters from 8493 reflections

$\theta = 3.0$ – 29.5 °

$\mu = 0.23$ mm⁻¹

$T = 123$ K

Sheet, colourless

$0.4 \times 0.3 \times 0.02$ mm

1135 *Data collection*

1136 Oxford Diffraction Xcalibur E diffractometer

1137 Radiation source: sealed tube

1138 ω scans

Absorption correction: multi-scan

CrysAlis PRO, Oxford Diffraction Ltd., Version

1.171.34.40 (release 27-08-2010 *CrysAlis171*

.NET) (compiled Aug 27 2010, 11:50:40)

Empirical absorption correction using spherical harmonics, implemented in *SCALE3*

ABSPACK scaling algorithm.

$T_{\min} = 0.527$, $T_{\max} = 1.000$

22193 measured reflections

4082 independent reflections

3225 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.040$

$\theta_{\max} = 29.0$ °, $\theta_{\min} = 3.0$ °

$h = -11 \rightarrow 11$

$k = -25 \rightarrow 25$

$l = -14 \rightarrow 15$

1139 *Refinement*

1140	Refinement on F^2	Hydrogen site location: mixed
1141	Least-squares matrix: full	H atoms treated by a mixture of independent
1142	$R[F^2 > 2\sigma(F^2)] = 0.045$	and constrained refinement
1143	$wR(F^2) = 0.114$	$w = 1/[\sigma^2(F_o^2) + (0.0493P)^2 + 0.8192P]$
1144	$S = 1.04$	where $P = (F_o^2 + 2F_c^2)/3$
1145	4082 reflections	$(\Delta/\sigma)_{\max} = 0.001$
1146	237 parameters	$\Delta\rho_{\max} = 0.39 \text{ e } \text{\AA}^{-3}$
1147	0 restraints	$\Delta\rho_{\min} = -0.48 \text{ e } \text{\AA}^{-3}$

1148 *Special details*

1149 **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.

1150 *Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (\AA^2)*

1151		<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
1152	S1	0.87444 (6)	0.84851 (2)	0.27777 (4)	0.02297 (12)
1153	O1	0.97267 (17)	0.79251 (7)	0.25677 (14)	0.0309 (3)
1154	O2	0.96376 (16)	0.87572 (7)	0.41782 (12)	0.0271 (3)
1155	N1	0.83358 (19)	0.90544 (7)	0.16260 (15)	0.0233 (3)
1156	N2	0.73473 (18)	1.01144 (7)	0.05571 (14)	0.0216 (3)
1157	N3	0.71259 (19)	0.98683 (8)	0.25488 (15)	0.0239 (3)
1158	N4	0.1507 (2)	0.73439 (9)	0.12033 (18)	0.0284 (3)
1159	N5	1.20543 (19)	1.03376 (7)	0.15032 (15)	0.0231 (3)
1160	N6	1.0916 (3)	1.14087 (10)	0.0502 (2)	0.0411 (4)
1161	C1	0.7592 (2)	0.96881 (9)	0.16105 (17)	0.0209 (3)
1162	C2	0.6662 (2)	1.07527 (9)	0.04854 (17)	0.0238 (4)
1163	H2	0.6475	1.1057	-0.0247	0.029*
1164	C3	0.6214 (2)	1.09897 (9)	0.14288 (18)	0.0258 (4)
1165	H3	0.5754	1.1449	0.1382	0.031*
1166	C4	0.6476 (2)	1.05165 (9)	0.24444 (18)	0.0258 (4)
1167	H4	0.6175	1.0661	0.3110	0.031*
1168	C5	0.6654 (2)	0.81161 (8)	0.23937 (17)	0.0215 (3)
1169	C6	0.5823 (2)	0.76230 (9)	0.13303 (18)	0.0266 (4)
1170	H6	0.6422	0.7470	0.0864	0.032*
1171	C7	0.4129 (3)	0.73551 (9)	0.09498 (18)	0.0275 (4)
1172	H7	0.3577	0.7015	0.0230	0.033*
1173	C8	0.3216 (2)	0.75825 (8)	0.16202 (17)	0.0219 (3)
1174	C9	0.4076 (2)	0.80709 (9)	0.26908 (17)	0.0235 (3)
1175	H9	0.3488	0.8224	0.3164	0.028*
1176	C10	0.5776 (2)	0.83359 (9)	0.30745 (17)	0.0235 (3)
1177	H10	0.6342	0.8669	0.3806	0.028*
1178	C11	1.1346 (2)	1.09647 (9)	0.15389 (18)	0.0251 (4)
1179	C12	1.1082 (2)	1.10992 (10)	0.2655 (2)	0.0316 (4)
1180	H12	1.0611	1.1540	0.2721	0.038*
1181	C13	1.1499 (3)	1.06005 (11)	0.3633 (2)	0.0340 (4)

1182	H13	1.1283	1.0688	0.4367	0.041*
1183	C14	1.2247 (3)	0.99557 (11)	0.3572 (2)	0.0368 (5)
1184	H14	1.2565	0.9607	0.4264	0.044*
1185	C15	1.2502 (3)	0.98455 (10)	0.2496 (2)	0.0313 (4)
1186	H15	1.3008	0.9412	0.2437	0.038*
1187	H1N	0.109 (3)	0.6980 (12)	0.067 (2)	0.036 (6)*
1188	H2N	0.100 (3)	0.7493 (12)	0.165 (2)	0.038 (6)*
1189	H3N	1.220 (3)	1.0215 (12)	0.078 (2)	0.044 (6)*
1190	H4N	1.045 (3)	1.1818 (14)	0.050 (3)	0.051 (7)*
1191	H5N	1.105 (4)	1.1256 (13)	-0.021 (3)	0.060 (8)*

1192 *Atomic displacement parameters (\AA^2)*

1193		U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
1194	S1	0.0214 (2)	0.0248 (2)	0.0239 (2)	0.00462 (16)	0.01254 (17)	0.00161 (16)
1195	O1	0.0288 (7)	0.0315 (7)	0.0364 (7)	0.0096 (5)	0.0197 (6)	0.0023 (6)
1196	O2	0.0236 (6)	0.0309 (7)	0.0230 (6)	0.0037 (5)	0.0092 (5)	0.0021 (5)
1197	N1	0.0221 (7)	0.0251 (7)	0.0242 (7)	0.0019 (6)	0.0130 (6)	0.0013 (6)
1198	N2	0.0188 (7)	0.0236 (7)	0.0215 (7)	-0.0033 (5)	0.0097 (6)	-0.0017 (5)
1199	N3	0.0220 (7)	0.0278 (7)	0.0224 (7)	0.0011 (6)	0.0119 (6)	0.0000 (6)
1200	N4	0.0302 (8)	0.0267 (8)	0.0333 (9)	-0.0041 (7)	0.0201 (7)	-0.0066 (7)
1201	N5	0.0230 (7)	0.0257 (7)	0.0230 (7)	0.0009 (6)	0.0135 (6)	-0.0012 (6)
1202	N6	0.0551 (12)	0.0302 (9)	0.0434 (11)	0.0162 (8)	0.0292 (10)	0.0084 (8)
1203	C1	0.0161 (7)	0.0247 (8)	0.0203 (8)	-0.0027 (6)	0.0082 (6)	-0.0017 (6)
1204	C2	0.0219 (8)	0.0241 (8)	0.0222 (8)	-0.0031 (7)	0.0092 (7)	0.0001 (7)
1205	C3	0.0235 (8)	0.0219 (8)	0.0267 (9)	-0.0007 (7)	0.0092 (7)	-0.0045 (7)
1206	C4	0.0215 (8)	0.0300 (9)	0.0247 (8)	-0.0001 (7)	0.0111 (7)	-0.0047 (7)
1207	C5	0.0238 (8)	0.0199 (8)	0.0227 (8)	0.0031 (6)	0.0135 (7)	0.0024 (6)
1208	C6	0.0310 (9)	0.0265 (8)	0.0288 (9)	0.0033 (7)	0.0201 (8)	-0.0030 (7)
1209	C7	0.0322 (9)	0.0250 (8)	0.0270 (9)	-0.0013 (7)	0.0166 (8)	-0.0071 (7)
1210	C8	0.0261 (8)	0.0183 (7)	0.0232 (8)	0.0027 (6)	0.0141 (7)	0.0044 (6)
1211	C9	0.0304 (9)	0.0229 (8)	0.0234 (8)	0.0022 (7)	0.0183 (7)	0.0015 (7)
1212	C10	0.0293 (9)	0.0228 (8)	0.0203 (8)	0.0014 (7)	0.0142 (7)	-0.0009 (6)
1213	C11	0.0195 (8)	0.0253 (8)	0.0286 (9)	0.0006 (7)	0.0111 (7)	-0.0029 (7)
1214	C12	0.0248 (9)	0.0353 (10)	0.0367 (10)	-0.0030 (8)	0.0173 (8)	-0.0133 (8)
1215	C13	0.0268 (9)	0.0514 (12)	0.0281 (9)	-0.0147 (9)	0.0173 (8)	-0.0168 (9)
1216	C14	0.0405 (11)	0.0416 (11)	0.0289 (10)	-0.0055 (9)	0.0185 (9)	0.0039 (8)
1217	C15	0.0368 (10)	0.0270 (9)	0.0331 (10)	0.0041 (8)	0.0202 (9)	0.0033 (8)

1218 *Geometric parameters (\AA , $^\circ$)*

1219	S1—O2	1.4458 (13)	C3—H3	0.9500
1220	S1—O1	1.4520 (12)	C4—H4	0.9500
1221	S1—N1	1.5834 (15)	C5—C10	1.388 (2)
1222	S1—C5	1.7592 (17)	C5—C6	1.393 (2)
1223	N1—C1	1.361 (2)	C6—C7	1.383 (3)
1224	N2—C2	1.334 (2)	C6—H6	0.9500
1225	N2—C1	1.360 (2)	C7—C8	1.407 (2)

1226	N3—C4	1.334 (2)	C7—H7	0.9500
1227	N3—C1	1.352 (2)	C8—C9	1.393 (2)
1228	N4—C8	1.366 (2)	C9—C10	1.384 (2)
1229	N4—H1N	0.86 (2)	C9—H9	0.9500
1230	N4—H2N	0.87 (2)	C10—H10	0.9500
1231	N5—C11	1.349 (2)	C11—C12	1.410 (3)
1232	N5—C15	1.353 (2)	C12—C13	1.354 (3)
1233	N5—H3N	0.92 (2)	C12—H12	0.9500
1234	N6—C11	1.329 (3)	C13—C14	1.403 (3)
1235	N6—H4N	0.87 (3)	C13—H13	0.9500
1236	N6—H5N	0.92 (3)	C14—C15	1.352 (3)
1237	C2—C3	1.379 (2)	C14—H14	0.9500
1238	C2—H2	0.9500	C15—H15	0.9500
1239	C3—C4	1.379 (3)		
1240				
1241	O2—S1—O1	114.52 (8)	C6—C5—S1	118.99 (13)
1242	O2—S1—N1	114.53 (8)	C7—C6—C5	120.28 (16)
1243	O1—S1—N1	105.72 (8)	C7—C6—H6	119.9
1244	O2—S1—C5	108.04 (8)	C5—C6—H6	119.9
1245	O1—S1—C5	105.70 (8)	C6—C7—C8	120.50 (16)
1246	N1—S1—C5	107.81 (8)	C6—C7—H7	119.7
1247	C1—N1—S1	121.43 (12)	C8—C7—H7	119.7
1248	C2—N2—C1	117.44 (14)	N4—C8—C9	120.93 (16)
1249	C4—N3—C1	116.41 (15)	N4—C8—C7	120.64 (16)
1250	C8—N4—H1N	119.2 (15)	C9—C8—C7	118.40 (16)
1251	C8—N4—H2N	117.6 (15)	C10—C9—C8	121.00 (16)
1252	H1N—N4—H2N	121 (2)	C10—C9—H9	119.5
1253	C11—N5—C15	121.88 (16)	C8—C9—H9	119.5
1254	C11—N5—H3N	121.5 (14)	C9—C10—C5	120.19 (16)
1255	C15—N5—H3N	116.5 (14)	C9—C10—H10	119.9
1256	C11—N6—H4N	119.3 (16)	C5—C10—H10	119.9
1257	C11—N6—H5N	117.6 (16)	N6—C11—N5	117.51 (17)
1258	H4N—N6—H5N	123 (2)	N6—C11—C12	124.49 (18)
1259	N3—C1—N2	123.66 (15)	N5—C11—C12	117.99 (17)
1260	N3—C1—N1	122.74 (15)	C13—C12—C11	119.97 (17)
1261	N2—C1—N1	113.60 (14)	C13—C12—H12	120.0
1262	N2—C2—C3	122.76 (16)	C11—C12—H12	120.0
1263	N2—C2—H2	118.6	C12—C13—C14	120.61 (17)
1264	C3—C2—H2	118.6	C12—C13—H13	119.7
1265	C2—C3—C4	115.67 (16)	C14—C13—H13	119.7
1266	C2—C3—H3	122.2	C15—C14—C13	117.98 (18)
1267	C4—C3—H3	122.2	C15—C14—H14	121.0
1268	N3—C4—C3	123.98 (16)	C13—C14—H14	121.0
1269	N3—C4—H4	118.0	C14—C15—N5	121.55 (18)
1270	C3—C4—H4	118.0	C14—C15—H15	119.2
1271	C10—C5—C6	119.61 (16)	N5—C15—H15	119.2
1272	C10—C5—S1	121.30 (13)		
1273				

1274	O2—S1—N1—C1	-46.01 (15)	C10—C5—C6—C7	0.2 (3)
1275	O1—S1—N1—C1	-173.05 (13)	S1—C5—C6—C7	-176.15 (14)
1276	C5—S1—N1—C1	74.26 (14)	C5—C6—C7—C8	0.7 (3)
1277	C4—N3—C1—N2	-3.3 (2)	C6—C7—C8—N4	176.89 (16)
1278	C4—N3—C1—N1	177.47 (15)	C6—C7—C8—C9	-1.3 (3)
1279	C2—N2—C1—N3	2.2 (2)	N4—C8—C9—C10	-177.19 (16)
1280	C2—N2—C1—N1	-178.51 (14)	C7—C8—C9—C10	1.0 (2)
1281	S1—N1—C1—N3	-1.2 (2)	C8—C9—C10—C5	-0.1 (3)
1282	S1—N1—C1—N2	179.50 (11)	C6—C5—C10—C9	-0.5 (3)
1283	C1—N2—C2—C3	0.4 (2)	S1—C5—C10—C9	175.75 (13)
1284	N2—C2—C3—C4	-1.6 (2)	C15—N5—C11—N6	-178.70 (18)
1285	C1—N3—C4—C3	2.0 (2)	C15—N5—C11—C12	0.1 (3)
1286	C2—C3—C4—N3	0.3 (3)	N6—C11—C12—C13	177.36 (19)
1287	O2—S1—C5—C10	24.88 (16)	N5—C11—C12—C13	-1.4 (3)
1288	O1—S1—C5—C10	147.91 (14)	C11—C12—C13—C14	1.9 (3)
1289	N1—S1—C5—C10	-99.39 (15)	C12—C13—C14—C15	-1.2 (3)
1290	O2—S1—C5—C6	-158.80 (13)	C13—C14—C15—N5	0.0 (3)
1291	O1—S1—C5—C6	-35.78 (16)	C11—N5—C15—C14	0.6 (3)
1292	N1—S1—C5—C6	76.93 (15)		

1293 *Hydrogen-bond geometry* (\AA , $^\circ$)

1294	<i>D</i> —H \cdots <i>A</i>	<i>D</i> —H	H \cdots <i>A</i>	<i>D</i> \cdots <i>A</i>	<i>D</i> —H \cdots <i>A</i>
1295	N4—H1N \cdots O2 ⁱ	0.86 (2)	2.04 (2)	2.895 (2)	168 (2)
1296	N4—H2N \cdots O1 ⁱⁱ	0.87 (2)	2.03 (2)	2.887 (2)	174 (2)
1297	N5—H3N \cdots N2 ⁱⁱⁱ	0.92 (2)	1.85 (2)	2.758 (2)	174 (2)
1298	N6—H4N \cdots N4 ^{iv}	0.87 (3)	2.39 (3)	3.085 (2)	137 (2)
1299	N6—H5N \cdots N1 ⁱⁱⁱ	0.92 (3)	2.00 (3)	2.918 (2)	173 (3)

1300 Symmetry codes: (i) $x-1, -y+3/2, z-1/2$; (ii) $x-1, y, z$; (iii) $-x+2, -y+2, -z$; (iv) $-x+1, -y+2, -z$.

1301 (VI)

1302 *Crystal data*

1303	$\text{C}_{12}\text{H}_{19}\text{N}_5\text{O}_4\text{S}$	$F(000) = 696$
1304	$M_r = 329.38$	$D_x = 1.503 \text{ Mg m}^{-3}$
1305	Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation, $\lambda = 0.71073 \text{ \AA}$
1306	$a = 12.7755 (4) \text{ \AA}$	Cell parameters from 3829 reflections
1307	$b = 9.8979 (4) \text{ \AA}$	$\theta = 3.2\text{--}29.5^\circ$
1308	$c = 11.5366 (4) \text{ \AA}$	$\mu = 0.25 \text{ mm}^{-1}$
1309	$\beta = 93.508 (3)^\circ$	$T = 123 \text{ K}$
1310	$V = 1456.08 (9) \text{ \AA}^3$	Fragment, colourless
1311	$Z = 4$	$0.25 \times 0.24 \times 0.12 \text{ mm}$

1312 *Data collection*

1313	Oxford Diffraction Xcalibur E diffractometer
1314	Radiation source: sealed tube
1315	ω scans

1316 Absorption correction: multi-scan
CrysAlis PRO, Oxford Diffraction Ltd., Version 1.171.34.40 (release 27-08-2010 CrysAlis171.NET) (compiled Aug 27 2010,11:50:40)
 Empirical absorption correction using spherical harmonics, implemented in SCALE3 ABSPACK scaling algorithm.
 7213 measured reflections
 3571 independent reflections
 2942 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.023$
 $\theta_{\text{max}} = 29.5^\circ$, $\theta_{\text{min}} = 3.2^\circ$
 $h = -16 \rightarrow 17$
 $k = -12 \rightarrow 12$
 $l = -15 \rightarrow 15$

1317 $T_{\text{min}} = 0.904$, $T_{\text{max}} = 1.000$

1318 *Refinement*

1319 Refinement on F^2
 1320 Least-squares matrix: full
 1321 $R[F^2 > 2\sigma(F^2)] = 0.037$
 1322 $wR(F^2) = 0.095$
 1323 $S = 1.06$
 1324 3571 reflections
 1325 231 parameters
 1326 0 restraints
 Hydrogen site location: mixed
 H atoms treated by a mixture of independent and constrained refinement
 $w = 1/[\sigma^2(F_o^2) + (0.0415P)^2 + 0.609P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\text{max}} < 0.001$
 $\Delta\rho_{\text{max}} = 0.45 \text{ e } \text{\AA}^{-3}$
 $\Delta\rho_{\text{min}} = -0.47 \text{ e } \text{\AA}^{-3}$

1327 *Special details*

1328 **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.

1329 *Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (\AA^2)*

1330		<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
1331	S1	0.22358 (3)	0.19320 (4)	0.61905 (3)	0.01162 (11)
1332	O1	0.17136 (8)	0.31689 (11)	0.58001 (10)	0.0164 (2)
1333	O2	0.20716 (8)	0.15689 (11)	0.73914 (9)	0.0150 (2)
1334	O3	0.45324 (9)	-0.36122 (12)	0.56317 (11)	0.0195 (3)
1335	O1W	0.04450 (10)	0.06888 (14)	0.88727 (13)	0.0264 (3)
1336	N1	0.34262 (10)	0.21715 (13)	0.59377 (11)	0.0131 (3)
1337	N2	0.51428 (10)	0.15350 (13)	0.58746 (11)	0.0146 (3)
1338	N3	0.39549 (10)	0.00832 (13)	0.68028 (11)	0.0150 (3)
1339	N4	0.06716 (12)	-0.25029 (16)	0.31048 (14)	0.0204 (3)
1340	N5	0.33848 (11)	-0.55735 (15)	0.42864 (13)	0.0153 (3)
1341	C1	0.41755 (11)	0.12192 (16)	0.62242 (13)	0.0125 (3)
1342	C2	0.59122 (12)	0.06494 (17)	0.61312 (14)	0.0167 (3)
1343	H2	0.6595	0.0845	0.5894	0.020*
1344	C3	0.57580 (13)	-0.05364 (17)	0.67255 (14)	0.0178 (3)
1345	H3	0.6312	-0.1156	0.6906	0.021*
1346	C4	0.47525 (12)	-0.07671 (17)	0.70408 (14)	0.0169 (3)
1347	H4	0.4617	-0.1575	0.7450	0.020*
1348	C5	0.17356 (11)	0.06143 (15)	0.53090 (13)	0.0119 (3)
1349	C6	0.14954 (12)	-0.06369 (16)	0.57665 (14)	0.0149 (3)
1350	H6	0.1589	-0.0789	0.6579	0.018*
1351	C7	0.11201 (12)	-0.16636 (17)	0.50398 (14)	0.0163 (3)
1352	H7	0.0946	-0.2513	0.5359	0.020*
1353	C8	0.09943 (11)	-0.14617 (16)	0.38370 (14)	0.0150 (3)

1354	C9	0.12428 (12)	-0.01923 (16)	0.33835 (13)	0.0150 (3)
1355	H9	0.1163	-0.0040	0.2570	0.018*
1356	C10	0.16023 (11)	0.08336 (16)	0.41125 (13)	0.0141 (3)
1357	H10	0.1760	0.1693	0.3800	0.017*
1358	C11	0.34327 (12)	-0.37296 (17)	0.57033 (15)	0.0184 (3)
1359	H11A	0.3136	-0.2833	0.5878	0.022*
1360	H11B	0.3284	-0.4349	0.6347	0.022*
1361	C12	0.29135 (12)	-0.42582 (16)	0.45860 (15)	0.0173 (3)
1362	H12A	0.2153	-0.4374	0.4673	0.021*
1363	H12B	0.3005	-0.3601	0.3953	0.021*
1364	H1N	0.0376 (17)	-0.223 (2)	0.243 (2)	0.033 (6)*
1365	H2N	0.0370 (17)	-0.313 (2)	0.3425 (19)	0.029 (6)*
1366	H3N	0.4051 (17)	-0.550 (2)	0.4209 (17)	0.023 (5)*
1367	H4N	0.3120 (17)	-0.587 (2)	0.360 (2)	0.031 (6)*
1368	H5N	0.3293 (17)	-0.625 (2)	0.483 (2)	0.032 (6)*
1369	H1H	0.4656 (19)	-0.297 (3)	0.518 (2)	0.043 (7)*
1370	H1W	0.095 (2)	0.097 (3)	0.833 (3)	0.065 (9)*
1371	H2W	0.070 (2)	0.106 (3)	0.950 (2)	0.046 (7)*

1372 *Atomic displacement parameters (\AA^2)*

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}	
1374	S1	0.01262 (17)	0.01188 (19)	0.01020 (19)	0.00106 (13)	-0.00062 (13)	-0.00008 (14)
1375	O1	0.0180 (5)	0.0138 (6)	0.0170 (6)	0.0038 (4)	-0.0017 (4)	0.0010 (5)
1376	O2	0.0167 (5)	0.0185 (6)	0.0099 (5)	0.0023 (4)	0.0007 (4)	0.0001 (4)
1377	O3	0.0162 (5)	0.0161 (6)	0.0254 (7)	-0.0015 (5)	-0.0037 (5)	0.0028 (5)
1378	O1W	0.0229 (6)	0.0330 (8)	0.0227 (7)	-0.0010 (5)	-0.0029 (5)	-0.0036 (6)
1379	N1	0.0127 (6)	0.0129 (6)	0.0135 (6)	0.0000 (5)	-0.0011 (5)	0.0009 (5)
1380	N2	0.0147 (6)	0.0149 (7)	0.0139 (6)	-0.0009 (5)	-0.0006 (5)	-0.0016 (5)
1381	N3	0.0179 (6)	0.0144 (7)	0.0126 (6)	0.0016 (5)	0.0012 (5)	0.0008 (5)
1382	N4	0.0253 (7)	0.0185 (8)	0.0171 (8)	-0.0046 (6)	-0.0014 (6)	-0.0029 (6)
1383	N5	0.0164 (7)	0.0161 (7)	0.0131 (7)	-0.0015 (5)	-0.0010 (5)	0.0014 (6)
1384	C1	0.0149 (7)	0.0137 (7)	0.0085 (7)	-0.0005 (6)	-0.0017 (5)	-0.0032 (6)
1385	C2	0.0135 (7)	0.0206 (8)	0.0158 (8)	0.0009 (6)	0.0002 (6)	-0.0049 (7)
1386	C3	0.0199 (8)	0.0184 (8)	0.0148 (8)	0.0061 (6)	-0.0016 (6)	-0.0017 (6)
1387	C4	0.0222 (8)	0.0147 (8)	0.0136 (8)	0.0026 (6)	-0.0004 (6)	-0.0002 (6)
1388	C5	0.0096 (6)	0.0138 (7)	0.0122 (7)	0.0000 (5)	0.0000 (5)	-0.0008 (6)
1389	C6	0.0149 (7)	0.0188 (8)	0.0109 (7)	0.0001 (6)	0.0013 (5)	0.0010 (6)
1390	C7	0.0172 (7)	0.0155 (8)	0.0161 (8)	-0.0031 (6)	0.0017 (6)	0.0012 (6)
1391	C8	0.0111 (7)	0.0170 (8)	0.0169 (8)	0.0007 (6)	0.0007 (6)	-0.0036 (6)
1392	C9	0.0152 (7)	0.0194 (8)	0.0104 (7)	0.0022 (6)	0.0003 (6)	0.0003 (6)
1393	C10	0.0135 (7)	0.0154 (7)	0.0134 (7)	0.0008 (6)	0.0007 (5)	0.0026 (6)
1394	C11	0.0171 (7)	0.0202 (8)	0.0178 (8)	-0.0006 (6)	0.0000 (6)	-0.0022 (7)
1395	C12	0.0168 (7)	0.0159 (8)	0.0187 (8)	-0.0007 (6)	-0.0021 (6)	-0.0002 (7)

1396 *Geometric parameters (Å, °)*

1397	S1—O1	1.4525 (11)	C2—C3	1.379 (2)
1398	S1—O2	1.4588 (11)	C2—H2	0.9500
1399	S1—N1	1.5838 (13)	C3—C4	1.376 (2)
1400	S1—C5	1.7505 (15)	C3—H3	0.9500
1401	O3—C11	1.4172 (19)	C4—H4	0.9500
1402	O3—H1H	0.84 (3)	C5—C6	1.388 (2)
1403	O1W—H1W	0.97 (3)	C5—C10	1.397 (2)
1404	O1W—H2W	0.86 (3)	C6—C7	1.384 (2)
1405	N1—C1	1.3694 (19)	C6—H6	0.9500
1406	N2—C2	1.337 (2)	C7—C8	1.401 (2)
1407	N2—C1	1.3596 (19)	C7—H7	0.9500
1408	N3—C4	1.337 (2)	C8—C9	1.405 (2)
1409	N3—C1	1.346 (2)	C9—C10	1.379 (2)
1410	N4—C8	1.379 (2)	C9—H9	0.9500
1411	N4—H1N	0.89 (2)	C10—H10	0.9500
1412	N4—H2N	0.83 (2)	C11—C12	1.507 (2)
1413	N5—C12	1.484 (2)	C11—H11A	0.9900
1414	N5—H3N	0.86 (2)	C11—H11B	0.9900
1415	N5—H4N	0.90 (2)	C12—H12A	0.9900
1416	N5—H5N	0.93 (2)	C12—H12B	0.9900
1417				
1418	O1—S1—O2	114.34 (7)	C3—C4—H4	118.2
1419	O1—S1—N1	104.16 (7)	C6—C5—C10	119.86 (14)
1420	O2—S1—N1	114.03 (7)	C6—C5—S1	121.69 (12)
1421	O1—S1—C5	107.67 (7)	C10—C5—S1	118.44 (12)
1422	O2—S1—C5	107.43 (7)	C7—C6—C5	120.08 (14)
1423	N1—S1—C5	108.97 (7)	C7—C6—H6	120.0
1424	C11—O3—H1H	108.9 (16)	C5—C6—H6	120.0
1425	H1W—O1W—H2W	101 (2)	C6—C7—C8	120.63 (15)
1426	C1—N1—S1	121.16 (11)	C6—C7—H7	119.7
1427	C2—N2—C1	116.93 (14)	C8—C7—H7	119.7
1428	C4—N3—C1	116.59 (13)	N4—C8—C7	120.77 (15)
1429	C8—N4—H1N	113.6 (15)	N4—C8—C9	120.40 (15)
1430	C8—N4—H2N	114.6 (15)	C7—C8—C9	118.78 (14)
1431	H1N—N4—H2N	116 (2)	C10—C9—C8	120.39 (14)
1432	C12—N5—H3N	111.5 (13)	C10—C9—H9	119.8
1433	C12—N5—H4N	111.2 (14)	C8—C9—H9	119.8
1434	H3N—N5—H4N	104.9 (19)	C9—C10—C5	120.26 (14)
1435	C12—N5—H5N	114.0 (13)	C9—C10—H10	119.9
1436	H3N—N5—H5N	107.1 (18)	C5—C10—H10	119.9
1437	H4N—N5—H5N	107.5 (19)	O3—C11—C12	111.39 (13)
1438	N3—C1—N2	124.11 (14)	O3—C11—H11A	109.4
1439	N3—C1—N1	122.02 (13)	C12—C11—H11A	109.4
1440	N2—C1—N1	113.87 (14)	O3—C11—H11B	109.4
1441	N2—C2—C3	122.86 (15)	C12—C11—H11B	109.3
1442	N2—C2—H2	118.6	H11A—C11—H11B	108.0

1443	C3—C2—H2	118.6	N5—C12—C11	109.89 (13)
1444	C4—C3—C2	115.90 (15)	N5—C12—H12A	109.7
1445	C4—C3—H3	122.1	C11—C12—H12A	109.7
1446	C2—C3—H3	122.1	N5—C12—H12B	109.7
1447	N3—C4—C3	123.61 (15)	C11—C12—H12B	109.7
1448	N3—C4—H4	118.2	H12A—C12—H12B	108.2
1449				
1450	O1—S1—N1—C1	178.29 (12)	N1—S1—C5—C6	109.60 (13)
1451	O2—S1—N1—C1	52.98 (14)	O1—S1—C5—C10	43.52 (13)
1452	C5—S1—N1—C1	-67.02 (14)	O2—S1—C5—C10	167.11 (11)
1453	C4—N3—C1—N2	0.3 (2)	N1—S1—C5—C10	-68.88 (13)
1454	C4—N3—C1—N1	-179.55 (14)	C10—C5—C6—C7	-0.3 (2)
1455	C2—N2—C1—N3	-0.1 (2)	S1—C5—C6—C7	-178.76 (12)
1456	C2—N2—C1—N1	179.74 (13)	C5—C6—C7—C8	1.0 (2)
1457	S1—N1—C1—N3	-4.7 (2)	C6—C7—C8—N4	176.66 (15)
1458	S1—N1—C1—N2	175.48 (10)	C6—C7—C8—C9	-0.8 (2)
1459	C1—N2—C2—C3	-0.2 (2)	N4—C8—C9—C10	-177.60 (14)
1460	N2—C2—C3—C4	0.3 (2)	C7—C8—C9—C10	-0.1 (2)
1461	C1—N3—C4—C3	-0.1 (2)	C8—C9—C10—C5	0.8 (2)
1462	C2—C3—C4—N3	-0.1 (2)	C6—C5—C10—C9	-0.6 (2)
1463	O1—S1—C5—C6	-138.00 (12)	S1—C5—C10—C9	177.88 (11)
1464	O2—S1—C5—C6	-14.40 (14)	O3—C11—C12—N5	56.48 (18)

1465 *Hydrogen-bond geometry* (\AA , $^\circ$)

1466	<i>D</i> —H \cdots <i>A</i>	<i>D</i> —H	H \cdots <i>A</i>	<i>D</i> \cdots <i>A</i>	<i>D</i> —H \cdots <i>A</i>
1467	N4—H1N \cdots O1W ⁱ	0.89 (2)	2.34 (2)	3.173 (2)	156 (2)
1468	N4—H2N \cdots O1W ⁱⁱ	0.83 (2)	2.58 (2)	3.293 (2)	144.1 (19)
1469	N5—H3N \cdots O3 ⁱⁱⁱ	0.86 (2)	2.01 (2)	2.7762 (18)	147.2 (18)
1470	N5—H4N \cdots O2 ⁱⁱ	0.90 (2)	1.99 (2)	2.8488 (18)	159 (2)
1471	N5—H4N \cdots N3 ⁱⁱ	0.90 (2)	2.51 (2)	3.038 (2)	118.1 (17)
1472	N5—H5N \cdots O1 ^{iv}	0.93 (2)	2.44 (2)	3.1006 (18)	128.4 (17)
1473	N5—H5N \cdots N1 ^{iv}	0.93 (2)	2.02 (2)	2.933 (2)	167.3 (19)
1474	O3—H1H \cdots N2 ^v	0.84 (3)	1.90 (3)	2.7399 (19)	177 (2)
1475	O1W—H1W \cdots O2	0.97 (3)	1.94 (3)	2.9039 (17)	174 (3)
1476	O1W—H2W \cdots O1 ^{vi}	0.86 (3)	2.07 (3)	2.8997 (18)	163 (2)

1477 Symmetry codes: (i) $-x, -y, -z+1$; (ii) $x, -y-1/2, z-1/2$; (iii) $-x+1, -y-1, -z+1$; (iv) $x, y-1, z$; (v) $-x+1, -y, -z+1$; (vi) $x, -y+1/2, z+1/2$.

1478 **other supporting information**

1479 Crystallographic Information File. uk3153.cif

1480 Structure factors. uk3153Isup2.hkl

1481 Structure factors. uk3153IIsup3.hkl

1482 Structure factors. uk3153IIIsup4.hkl

1483 Structure factors. uk3153IVsup5.hkl

1484 Structure factors. uk3153Vsup6.hkl

1485 Structure factors. uk3153VIsup7.hkl