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Hydrochlorides, hydrates, hydronitrate, and an unanticipated hydrolysis product of famotidine

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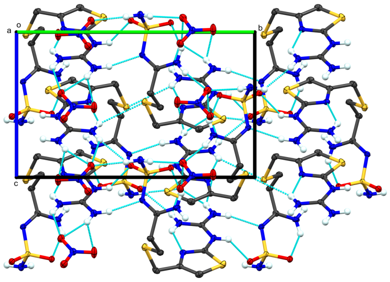
This article contributes to the development of Paper-Based Analytical Devices (PADs), a low-cost field-friendly platform for screening low-quality medicines. Our investigation focuses on famotidine, the active pharmaceutical ingredient (API) in Pepcid AC [an over-the-counter medicine used to treat gastroesophageal reflux disease (GERD)]. We report the successful isolation and characterization of several new crystalline forms of famotidine, focusing on the API itself rather than its PAD-activated colored complex. These forms include a famotidine hydrochloride polymorph, $C_6H_{14}N_7O_2S^+\cdot Cl^-$ (**I**), a famotidine hydrochloride hemihydrate salt, $C_6H_{14}N_7O_2S^+\cdot Cl^-\cdot 0.5H_2O$ (**II**), and a famotidine nitrate salt, $C_6H_{14}N_7O_2S^+\cdot NO_3^-$ (**III**). Unexpectedly, we also characterized a hydrolyzed famotidine complex, *N*-(diaminomethylene)-4-({[3-oxo-3-(sulfamoylamino)propyl]sulfanyl}methyl)thiazol-2-aminium chloride sesquihydrate, $C_8H_{15}N_6O_3S_3^+\cdot Cl^-\cdot 1.5H_2O$ (**IV**). The crystal structures reveal significant solid-state diversity: hemihydrate salt **II** exhibits two symmetry-independent famotidine hydrochloride molecules per asymmetric unit, while sesquihydrate salt **IV** shows four crystallographically-independent hydrochloride molecules and six symmetry-independent water molecules per standard unit. All four complexes display extensive hydrogen-bonded networks in the solid state. The detailed structural characterization of these crystalline complexes generates fundamental solid-state chemistry data; this knowledge is essential for predicting and controlling the drug performance and formulation stability of famotidine and crucially informs our development of PADs.



1. Introduction

The active pharmaceutical ingredient (API) famotidine is a histamine H_2-receptor antagonist that is available as an over-the-counter (OTC) medicine with the brand name of Pepcid. It is classified as an antigastroesophageal reflux disease (anti-GERD) medicine that decreases the amount of gastric acid produced by the stomach (Kapoor *et al.*, 2005).

We have developed an antigastroesophageal reflux disease (anti-GERD) paper-based analytical device (PAD) as a low-cost field-friendly reliable tool that allows untrained users to screen for low-quality medicines (Barstis *et al.*, 2016), including an anti-GERD PAD to screen for low-quality Pepcid. On these PADs, we have incorporated three key colorimetric tests for the APIs present in various anti-GERD medicines; however, the chemistry of these tests is not well understood. Our research goal is to better understand the chemistry occurring on the anti-GERD PAD by elucidating the chemical structures of the colored famotidine–metal complexes *via* X-ray crystallography. We began with a modest study of the chemical structures of the polymorphs, salts, and solvates of the API famotidine, the parent compound.

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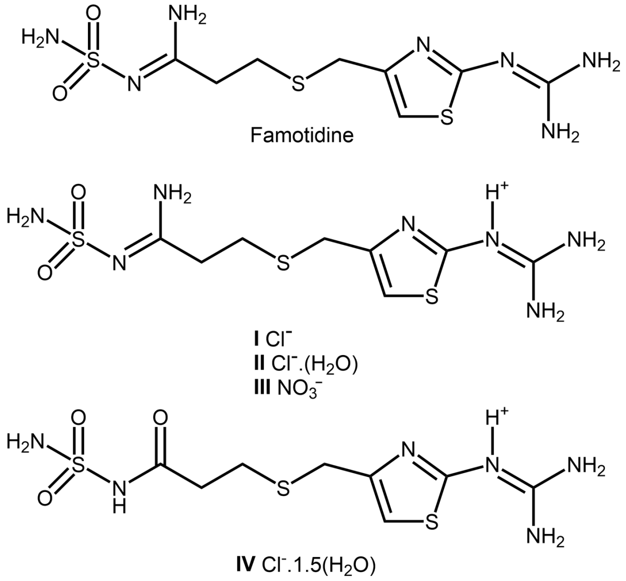
Chemical structures of polymorphic APIs, and their salts, are of interest to pharmaceutical manufacturing companies, because of potential variation in bioactivities and synthesis efficiencies. Simultaneously, polymorphism in APIs, including famotidine, is a serious concern for pharmaceutical manufacturing companies. Formerly, APIs were thought to exist in only one form; however, different polymorphs of these APIs are known to exist. These different polymorphs have varying packing properties, as well as physical properties (*e.g.* melting point, solubility, dissolution rate, and thermal stability), so the full characterization of the APIs, including their crystal structure, must be included as part of the pharmaceutical industry's drug discovery, development, and optimization processes. One case that illustrates the importance of the full characterization of API polymorphs was the high-profile case of the API Ritonavir (Norvir), an antiretroviral medicine manufactured by Abbott Laboratories (now AbbVie, Inc.) (Bauer *et al.*, 2001; Morissette *et al.*, 2003; Bučar *et al.*, 2015). An excellent treatise on polymorphism is detailed in Bernstein's monograph (Bernstein, 2023).



Scheme 1

Polymorphism occurs when the same solid material packs in different orientations. Famotidine crystallizes in three forms (Form A, Form B, and Form C) (Yanagisawa *et al.*, 1987; Golič *et al.*, 1989; Ferenczy *et al.*, 2000; Shankland *et al.*, 2002; Florence *et al.*, 2003; Overgaard & Hibbs, 2004; Saikia *et al.*, 2019), depending on the cooling rates and solvents used (Hassan *et al.*, 1997; Lu *et al.*, 2007*a*; Lu *et al.*, 2007*b*; Takebayashi *et al.*, 2021; Soto & Svärd, 2021), but Form A and Form B are the most commonly discussed polymorphs in the literature. Form C is a metastable form that has not been structurally characterized. Powder diffraction patterns of this form display broad low-intensity peaks indicative of nanocrystalline material (Hassan *et al.*, 1997). Form A is more thermodynamically stable than the metastable polymorph B; however, Form B is kinetically favored (Német *et al.*, 2009; Lin *et al.*, 2006). Metastable polymorph B is the most bioactive and thus used as the famotidine API in commercial anti-GERD medicines, such as Pepcid (Lin, 2014; Upadhyay *et al.*, 2022).

Our work began with the successful crystallization and structural analysis of the known famotidine polymorph, Form B. This preliminary work validated our experimental methodology, providing the necessary confidence to tackle the structural elucidation of more complex famotidine compounds, particularly those relevant to the activated PADs. We significantly expanded the solid-state chemistry of famotidine by determining the crystal structures of three new salts/polymorphs: a famotidine hydrochloride polymorph, a famotidine hydrochloride hemihydrate, and a famotidine nitrate salt. Furthermore, we report an unexpected chemical transformation of the API. Under the crystallization conditions employed, we observed the acidic hydrolysis of the amidine N atom, resulting in its replacement by a carbonyl group.

2. Experimental

2.1. Chemicals and materials

Famotidine (CAS No. 76824-35-6, >98% pure, HPLC grade) was purchased from TCI America (Portland, OR) and 200 proof ethyl alcohol (CAS No. 64-17-5, >99.98% ACS grade) was purchased from Pharmo-Aaper (Brookfield, CT); both were used as received. Methanol (CAS No 67-56-1, ≥99.8% ACS grade) was purchased from Sigma–Aldrich, Inc. (St Louis, MO) and used as received. Hydrochloric acid (CAS No. 7647-01-0, Fisher Chemical, 33-38%, technical grade) and nitric acid (CAS No. 7697-37-2, 69–70%, technical grade) were purchased from Fisher Scientific (Hanover Park, IL) and diluted to 3 and 0.1 *M*, respectively. Kimble 20 ml scintillation vials were purchased from Avantar–VWR (Allentown, PA) and Fisherbrand Shell Type 1 glass vials (15 × 45 mm) were purchased from Fisher Scientific (Hanover Park, IL).

2.2. General crystallization procedure

Following the solubility products outlined by Takebayashi and co-workers (Takebayashi *et al.*, 2021), that famotidine is less soluble in an ethanolic solution than in a methanolic solution, we prepared our acidified methanolic solutions in a 20 ml vial, carefully layered ethanol onto this mixture, and allowed the solution to equilibrate. Upon standing for several days, crystals were found to form and were inspected under a microscope and on the diffractometer to determine what species were present. Unit-cell determinations yielding known parameters were discarded.

2.2.1. Famotidine hydrochloride (I)

Single crystals of famotidine hydrochloride (**I**) were grown by dissolving famotidine (88 mg, 0.36 mmol) in methanol (3 ml) and acidifying with 3 *M* hydrochloric acid until a pH of 2 was obtained. Ethanol (7 ml) was layered on the methanol solution in a capped 20 ml vial at room temperature and allowed to stand, yielding the colorless block-like crystals that were analyzed. Colorless needle-like crystals of the known

Table 1
Experimental details.

Experiments were carried out at 120 K. Absorption was corrected for by numerical methods (*SADABS*; Krause *et al.*, 2015).

	I	**II**	**III**	**IV**
Crystal data				
Chemical formula	$C_8H_{16}N_7O_2S_3^+\cdot Cl^-$	$C_8H_{16}N_7O_2S_3^+\cdot Cl^-\cdot 0.5H_2O$	$C_8H_{16}N_7O_2S_3^+\cdot NO_3^-$	$C_8H_{15}N_6O_3S_3^+\cdot Cl^-\cdot 1.5H_2O$
M_r	373.91	382.91	400.47	401.91
Crystal system, space group	Monoclinic, $P2_1/n$	Triclinic, $P\overline{1}$	Monoclinic, $P2_1/c$	Triclinic, $P\overline{1}$
a, b, c (Å)	8.8712 (14), 8.4069 (13), 21.423 (3)	8.5104 (3), 13.8816 (4), 14.1753 (5)	13.5616 (16), 13.9590 (17), 8.504 (1)	5.1449 (6), 25.285 (2), 26.306 (2)
α, β, γ (°)	90, 90.357 (3), 90	92.178 (1), 92.655 (1), 107.297 (1)	90, 90.597 (2), 90	89.017 (7), 87.760 (8), 86.803 (8)
V (Å^3)	1597.7 (4)	1594.84 (9)	1609.8 (3)	3413.9 (6)
Z	4	4	4	8
Radiation type	Mo $K\alpha$	Mo $K\alpha$	Mo $K\alpha$	Cu $K\alpha$
μ (mm^{-1})	0.65	0.65	0.50	5.69
Crystal size (mm)	0.20 × 0.11 × 0.06	0.18 × 0.12 × 0.07	0.21 × 0.16 × 0.08	0.20 × 0.06 × 0.02
Data collection				
Diffractometer	Bruker D8	Bruker D8	Bruker D8	Bruker Venture
$T_{\min}$, $T_{\max}$	0.945, 0.989	0.830, 0.933	0.927, 0.989	0.475, 0.757
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	23845, 3963, 3237	40692, 7938, 6530	24503, 4017, 3502	82198, 12581, 8070
$R_{\rm int}$	0.040	0.044	0.029	0.219
$(\sin \theta/\lambda)_{\max}$ (Å^{-1})	0.667	0.667	0.668	0.608
Refinement				
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.028, 0.069, 1.04	0.062, 0.130, 1.09	0.027, 0.068, 1.04	0.074, 0.190, 1.04
No. of reflections	3963	7938	4017	12581
No. of parameters	224	398	223	876
H-atom treatment	H atoms treated by a mixture of independent and constrained refinement	H-atom parameters constrained	H atoms treated by a mixture of independent and constrained refinement	H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{\max}$, $\Delta\rho_{\min}$ (e Å^{-3})	0.33, −0.37	1.37, −1.79	0.35, −0.37	0.48, −0.52

Computer programs: *APEX4* (Bruker, 2021), *SAINT* (Bruker, 2021), *SHELXT2018* (Sheldrick, 2015*a*), *SHELXL2019* (Sheldrick, 2015*b*), *Mercury* (Macrae *et al.*, 2020), and *FinalCIF* (Kratzert, 2025).

polymorph of famotidine hydrochloride (Ishida *et al.*, 1989) were also identified and characterized from the bulk sample.

2.2.2. Famotidine hydrochloride hydrate (II) and hydrolyzed famotidine hydrochloride sesquihydrate (IV)

Famotidine hydrochloride (18 mg, 0.06 mmol) was dissolved in 3 ml methanol and was acidified with 3 *M* hydrochloric acid until a pH of 1 was obtained. Single crystals of **II** and **IV** were both grown by liquid diffusion of ethanol (7 ml) into the acidified methanolic solution of famotidine hydrochloride in a sealed 20 ml vial, at room temperature upon standing over one week.

2.2.3. Famotidine nitrate (III)

Colorless block-like crystals of famotidine nitrate (**III**) were obtained by liquid diffusion of ethanol (7 ml) into a 3 ml methanol solution of famotidine hydrochloride (16 mg, 0.05 mmol) in a capped 20 ml vial that was mildly acidified with 0.1 *M* nitric acid at room temperature.

2.3. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 1. H atoms bonded to C atoms were included in geometrically calculated positions with a riding model [C—H = 0.95 (aromatic) and 0.99 Å (methylene); $U_{\rm iso}({\rm H}) = 1.2U_{\rm eq}({\rm C})$]. H atoms bonded to N and O atoms were located from a difference Fourier map. For complex **I**, these H atoms were refined freely. A riding model for these H atoms was used for **II**; when freely refined, some of the H-atom positions refined to unreasonable positions. H atoms bonded to N atoms in complex **III** were treated with a mixture of freely refined and riding models, depending on how they behaved during refinement. Because of the lower quality of the data for complex **IV**, all H atoms were refined with a riding model. The disordered amidinate N atom (N14/N14*A*) in complex **II** was modeled over two positions at 50% occupancy. The positions for the two sites were observed in a difference Fourier map. It should be noted that the crystals for compound **IV** were particularly challenging. Multiple attempts were made to obtain a suitably diffracting sample that still required the extra X-ray intensity provided by a Diamond micro-focus copper source. Many of these crystallizations were serendipitous. Furthermore, this compound suffers from solvent loss during mounting that reduced the data quality. The structural model remains accurate, as atom types were differentiated during refinement, most significantly in the exchange of nitrogen for oxygen.

3. Results and discussion

Famotidine hydrochloride (**I**) is a new polymorph of the salt (Fig. 1). The reported structure crystallizes in the *C*-centered monoclinic space group *Cc* (Ishida *et al.*, 1989), in contrast with the primitive monoclinic $P2_1/n$ system reported here (Table 1). The significant structural difference between the two molecules is the orientation of the sulfamolylpropionamidine moiety. This moiety is rotated ~112° at the α-carbon (Fig. S1 in the supporting information) with respect to the thiazole moiety. In both cases, protonation has occurred at guanidine atom N3. In contrast with the two known forms of famotidine, the sulfamolylpropionamidine chain in **I** is extended away from the thiazole ring. In Form A, the sulfamolylpropionamidine group extends away from the thiazole then curves back around forming a 'spoon'-like shape when viewed edge on. In Form B, the chain curves back toward the thiazole ring forming a 'C'-shape when viewed edge on (Fig. S2).

Table 2
Hydrogen-bond geometry (Å, °) for **I**.

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1*A*···Cl1	0.84 (2)	2.44 (2)	3.1828 (15)	149 (2)
N1—H1*B*···N5$^{\text{i}}$	0.87 (2)	2.14 (2)	2.999 (2)	169 (2)
N2—H2*A*···O1$^{\text{ii}}$	0.83 (2)	2.09 (2)	2.9253 (18)	174 (2)
N2—H2*B*···N4	0.82 (2)	2.18 (2)	2.801 (2)	131.9 (19)
N3—H3···O2$^{\text{i}}$	0.802 (19)	2.02 (2)	2.8115 (19)	168.2 (19)
N6—H6*A*···Cl1$^{\text{iii}}$	0.87 (2)	2.42 (2)	3.1863 (16)	148.6 (19)
N6—H6*B*···Cl1$^{\text{ii}}$	0.76 (2)	2.47 (2)	3.2230 (18)	174 (2)
N7—H7*A*···Cl1$^{\text{ii}}$	0.82 (2)	2.66 (2)	3.3584 (16)	144.1 (17)
N7—H7*A*···O1	0.82 (2)	2.48 (2)	2.9627 (19)	118.4 (16)
N7—H7*B*···Cl1$^{\text{iv}}$	0.86 (2)	2.40 (2)	3.2644 (16)	176.7 (18)

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

Regarding the extended structure of **I**, all donors and acceptors, except the S atoms, are involved in hydrogen bonding (Table 2 and Fig. 2). There is one intramolecular hydrogen bond from guanidine atom N2 to thiazole atom N4. The guanidine atoms N1 and N3 form a hydrogen bond to the sulfamolyl group of a neighboring cation related by inversion symmetry [N1···N5$^{\text{i}}$ and N3···O2$^{\text{i}}$; symmetry code: (i) $-x+1, -y+1, -z+1$]. The neighboring cation necessarily has reciprocating hydrogen bonds from its guanidine to the standard molecule's sulfamolyl group. Guanidine atom N2 forms a hydrogen bond to sulfamolyl atom O2$^{\text{ii}}$ of a different neighboring cation [symmetry code: (ii) $-x+2, -y+1, -z+1$]. Atom N1 completes its hydrogen bonding with a contact to the chloride ion (N1···Cl1). The chloride ion serves as an acceptor for five hydrogen bonds from four different cations: the noted N1···Cl1 contact, interactions from N6 of two different symmetry-related cations, and from N7 of two other different famotidine cations. The result of these interactions is a hydrogen-bonded chain of chloride ions along the screw axis

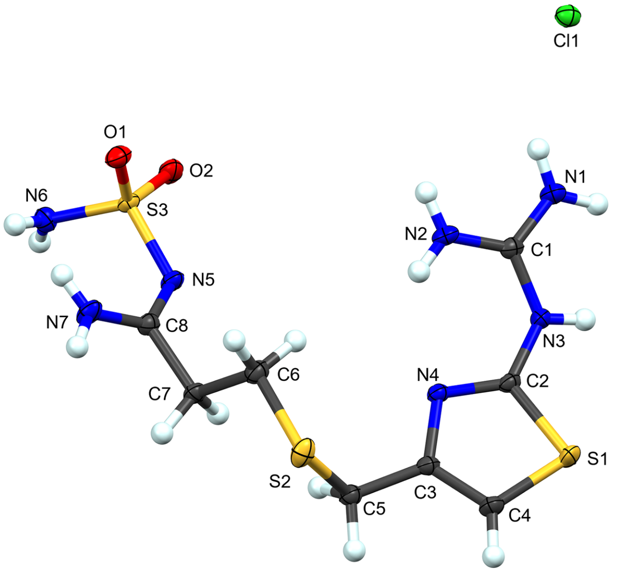
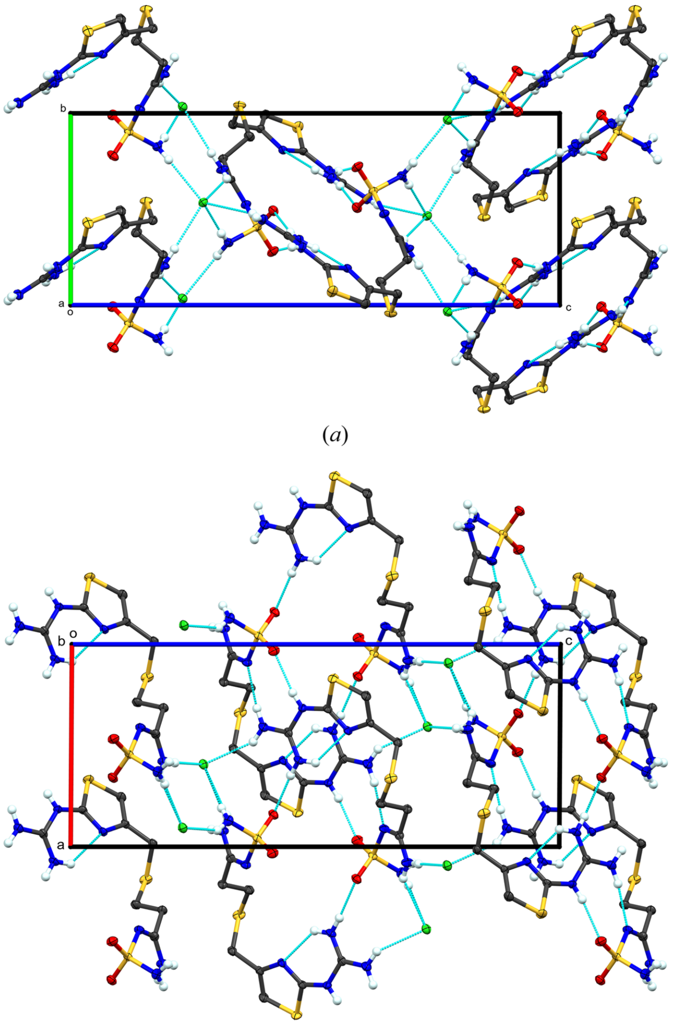


Figure 1
The atom-labeling scheme for **I**. Atomic displacement ellipsoids for non-H atoms are depicted at the 50% probability level and H atoms are shown as spheres of an arbitrary radius.



(*a*)

(*b*)

Figure 2
Packing diagram of **I**, viewed along (*a*) the *a* axis and (*b*) the *b* axis. Blue dashed lines represent hydrogen-bond interactions. Atomic displacement ellipsoids are shown at the 50% probability level. Only H atoms involved in hydrogen bonding are shown.

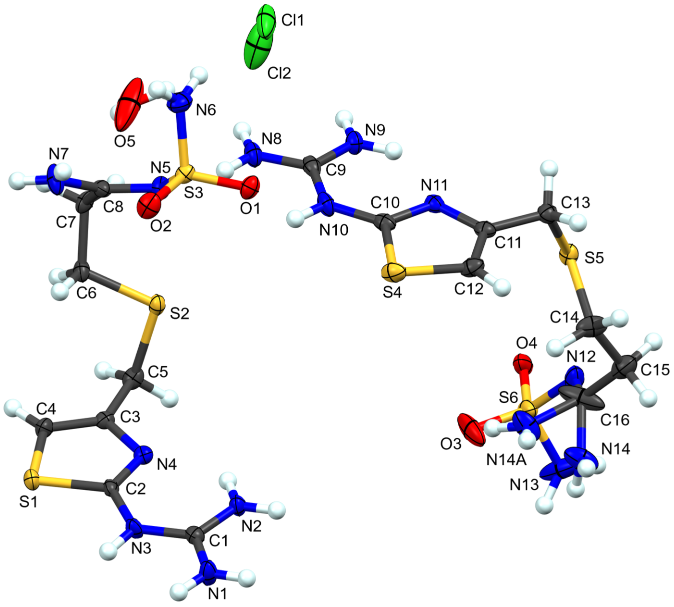


Figure 3
The atom-labeling scheme for **II**. Atomic displacement ellipsoids for non-H atoms are depicted at the 50% probability level and H atoms are shown as spheres of an arbitrary radius.

Table 3
Hydrogen-bond geometry (Å, °) for **II**.

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1*A*···Cl1^{i}	0.88	2.31	3.177 (3)	170
N1—H1*B*···N12^{ii}	0.88	2.08	2.959 (4)	176
N2—H2*A*···O1^{i}	0.88	2.57	3.233 (4)	132
N2—H2*B*···S2^{iii}	0.88	2.92	3.591 (3)	134
N2—H2*B*···N4	0.88	2.07	2.728 (4)	131
N3—H3···O4^{ii}	0.88	2.01	2.801 (4)	150
N6—H6*A*···O5^{iv}	0.88	2.12	2.826 (5)	137
N6—H6*B*···Cl1	0.88	2.44	3.303 (4)	167
N7—H7*A*···O2	0.88	2.30	2.882 (4)	123
N7—H7*A*···O5^{iv}	0.88	2.63	3.315 (5)	136
N7—H7*B*···Cl1^{iv}	0.88	2.58	3.411 (3)	157
N8—H8*A*···Cl2	0.88	2.42	3.226 (3)	152
N8—H8*B*···N5	0.88	2.10	2.959 (4)	166
N9—H9*A*···Cl2	0.88	2.64	3.388 (3)	144
N9—H9*B*···S5^{v}	0.88	2.97	3.634 (3)	134
N9—H9*B*···N11	0.88	2.07	2.727 (4)	131
N10—H10···O1	0.88	2.15	2.821 (4)	133
N13—H13*C*···Cl2^{vi}	0.88	2.71	3.466 (5)	145
N13—H13*D*···Cl2^{vii}	0.88	2.48	3.250 (4)	147
N14—H14*E*···Cl1^{vi}	0.88	2.46	3.131 (6)	134
N14—H14*F*···Cl2^{viii}	0.88	2.81	3.531 (7)	140
N14—H14*F*···O5^{viii}	0.88	2.46	3.167 (8)	138
N14*A*—H14*C*···O3	0.88	1.82	2.539 (7)	138
N14*A*—H14*D*···O2^{i}	0.88	2.35	2.999 (7)	131
O5—H5*C*···Cl2	0.87	2.27	3.126 (5)	167
O5—H5*D*···Cl1^{ix}	0.87	2.30	3.168 (4)	175

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

parallel to the *b* axis. The hydrogen bonds to the neighboring famotidine cations extend this into a three-dimensional network. Graph-set analysis reveals 40 different interactions in the solid state, which are beyond utility to discuss here (Etter *et al.*, 1990).

Complex **II** represents the first structural characterization of a hydrochloride hydrate of famotidine (Fig. 3). Formally the structure is a hemihydrate with one water molecule present in the standard unit per two famotidine hydrochloride salts. One of the two cations has positional disorder at the amidine N atom (N14/N14*A*). In all other respects, the famotidine cations are essentially identical, with only small deviations in the sulfamolylpropionamidine chain when overlaid at the thiazole group (Fig. S3).

In contrast with the hydrochloride **I**, there are several intramolecular hydrogen bonds within the famotidine cations in **II**: the same guanidine-to-thiazole N-atom hydrogen bond exists (N2···N4/N9···N11), and a second is a hydrogen bond from amidine atom N7/N14*A* to nearby sulfonamide atom O2/O3, respectively, of the same cation (Table 3). The disordered

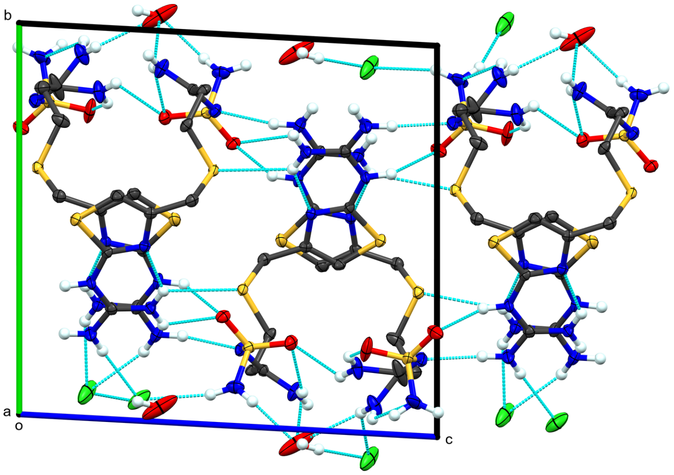


Figure 4
Packing diagram of **II**, viewed along the *a* axis. Blue dashed lines represent hydrogen-bond interactions. Atomic displacement ellipsoids are shown at the 50% probability level. Only H atoms involved in hydrogen bonding are shown.

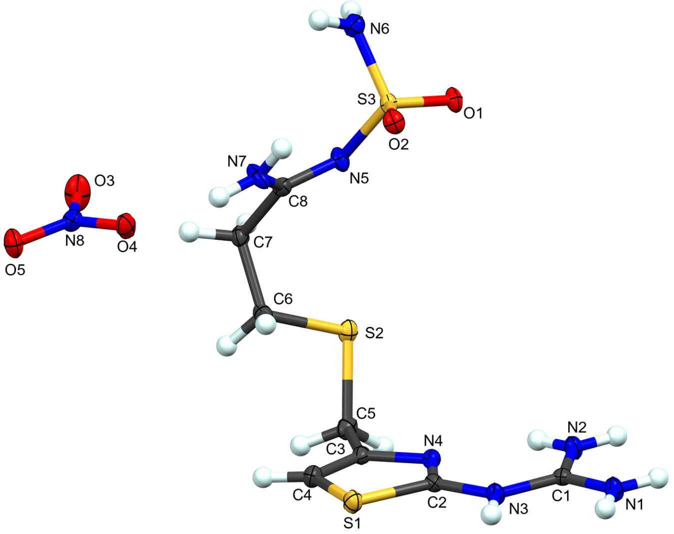


Figure 5
The atom-labeling scheme for **III**. Atomic displacement ellipsoids for non-H atoms are depicted at the 50% probability level and H atoms are shown as spheres of an arbitrary radius.

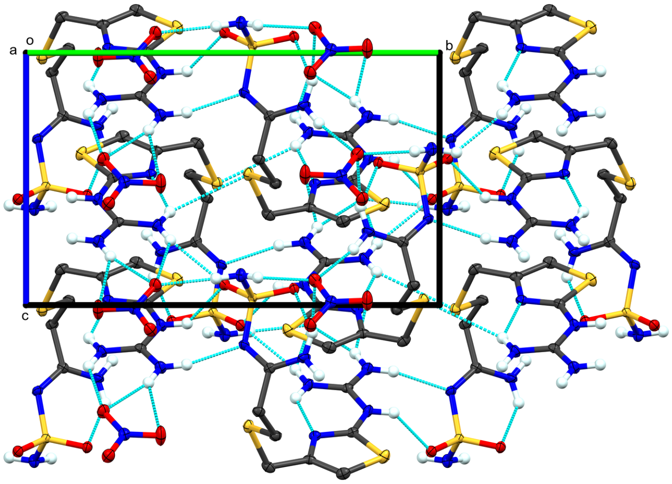


Figure 6
Packing diagram of **III**, viewed along the *a* axis. Blue dashed lines represent hydrogen-bond interactions. Atomic displacement ellipsoids are shown at the 50% probability level. Only H atoms involved in hydrogen bonding are shown.

Table 4
Hydrogen-bond geometry (Å, °) for **III**.

$D—\text{H}\cdots A$	$D—\text{H}$	$\text{H}\cdots A$	$D\cdots A$	$D—\text{H}\cdots A$
$\text{N1—H1}A\cdots\text{O3}^{\text{i}}$	0.88	2.40	3.1392 (16)	141
$\text{N1—H1}A\cdots\text{O5}^{\text{i}}$	0.88	2.08	2.9302 (16)	161
$\text{N1—H1}A\cdots\text{N8}^{\text{i}}$	0.88	2.59	3.4547 (17)	170
$\text{N1—H1}B\cdots\text{N5}^{\text{ii}}$	0.88	2.11	2.9647 (17)	164
$\text{N2—H2}A\cdots\text{O1}^{\text{iii}}$	0.88	2.37	3.0669 (15)	137
$\text{N2—H2}A\cdots\text{O5}^{\text{i}}$	0.88	2.62	3.3299 (15)	138
$\text{N2—H2}B\cdots\text{S2}^{\text{iv}}$	0.88	2.95	3.5664 (13)	128
$\text{N2—H2}B\cdots\text{N4}$	0.88	2.05	2.7199 (16)	132
$\text{N3—H3}\cdots\text{O1}^{\text{ii}}$	0.88	2.10	2.7914 (15)	135
$\text{N7—H7}A\cdots\text{O2}$	0.88	2.18	2.8042 (15)	127
$\text{N7—H7}A\cdots\text{O4}^{\text{v}}$	0.88	2.39	3.0515 (16)	133
$\text{N7—H7}B\cdots\text{O4}$	0.88	2.03	2.8997 (16)	168
$\text{N6—H6}A\cdots\text{O5}^{\text{vi}}$	0.861 (18)	2.085 (18)	2.8992 (17)	157.6 (16)
$\text{N6—H6}B\cdots\text{O4}^{\text{v}}$	0.856 (18)	2.115 (19)	2.9184 (16)	156.1 (16)

Symmetry codes: (i) $x+1, -y+\frac{3}{2}, z-\frac{1}{2}$; (ii) $-x+1, y+\frac{1}{2}, -z+\frac{1}{2}$; (iii) $-x+1, -y+1, -z$; (iv) $-x+1, -y+1, -z+1$; (v) $x, -y+\frac{3}{2}, z-\frac{1}{2}$; (vi) $-x, y-\frac{1}{2}, -z+\frac{1}{2}$.

Table 5
Selected torsion angles (°) for **IV**.

N4—C3—C5—S2	−77.7 (6)	N16—C19—C21—S8	−75.3 (6)
C5—S2—C6—C7	−85.3 (5)	C21—S8—C22—C23	157.5 (5)
N10—C11—C13—S5	−59.9 (6)	C26—S10—C28—C27	−0.9 (5)
C13—S5—C14—C15	−88.8 (5)	N22—C27—C29—S11	−60.7 (7)

amidine atom N14/N14*A* satisfies several different hydrogen-bond interactions. The N14 position forms two pairs of bifurcated hydrogen bonds. The first is an intramolecular hydrogen bond to sulfonamide atom N13 and an intermolecular contact with Cl1$^{\text{vi}}$ [symmetry code: (vi) $x+1, y+1, z$]. However, the former is less likely to be a firm electrostatic interaction due to directionality. The second H atom forms contacts with the water of crystallization (O5$^{\text{viii}}$) and the second chloride (Cl2$^{\text{viii}}$) [symmetry code: (viii) $x, y+1, z$]. When the N atom is at the N14*A* site, it forms the hydrogen bond previously noted, and an intermolecular hydrogen bond to O2$^{\text{i}}$ of a sulfonamide group on a neighboring cation [symmetry code: (i) $-x+1, -y+1, -z+1$].

The presence of two symmetry-independent famotidine hydrochloride complexes and a water of crystallization create

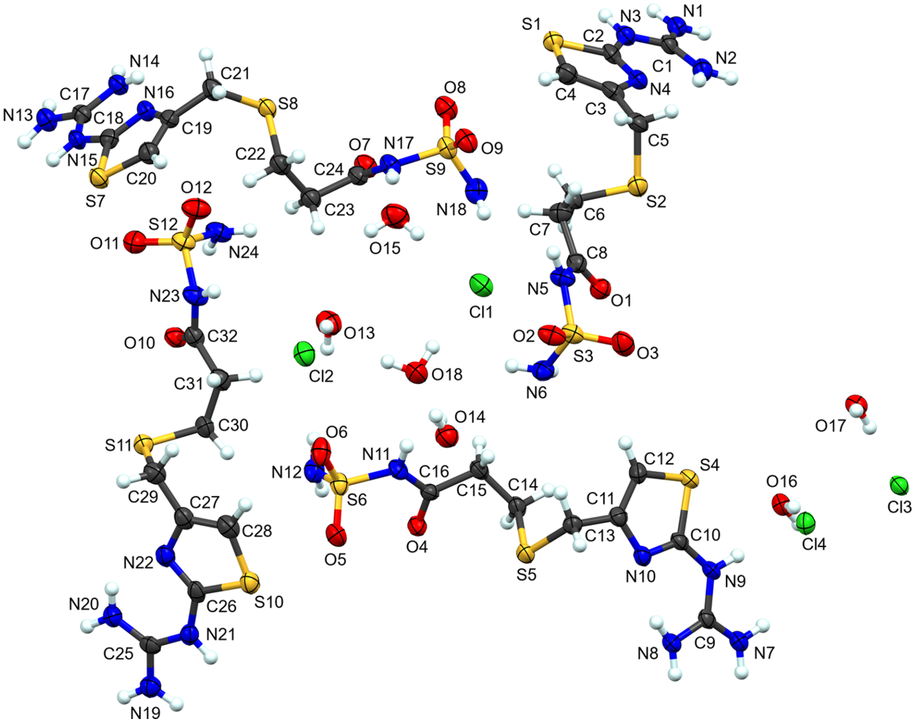


Figure 7
The atom-labeling scheme for **IV**. Atomic displacement ellipsoids for non-H atoms are depicted at the 50% probability level and H atoms are shown as spheres of an arbitrary radius.

a plethora of hydrogen-bond interactions (Table 3 and Fig. 4). This discussion will focus on the significant differences across this series of materials. Examining the chloride ions and water molecule, Cl1 serves as an acceptor for five hydrogen bonds and Cl2 accepts six hydrogen bonds. The water of crystallization is a donor in two hydrogen bonds to each chloride and is an acceptor of three hydrogen bonds from the two sulfamolyl N atoms of one cation and the sulfonamide N atom of a second cation (N6, N7, and N14, successively). The guanidine intramolecular hydrogen bond (above) is bifurcated with a contact to an S atom on an adjacent cation [N2···S2$^{\rm i}$ and N9···S5$^{\rm ii}$; symmetry code: (i) $-x + 1$, $-y + 1$, $-z + 1$; (ii) x, y, $z + 1$]. This is in contrast with **I**, in which the S atoms are not part of the hydrogen-bonding network. These N—H···S hydrogen bonds are self-complementary related across inversion centers. The sulfonamide N atoms (N6 and N13) differ, in their intermolecular contacts. Atom N6 forms hydrogen bonds with water O5$^{\rm iv}$ and Cl1, whereas N13 forms hydrogen bonds to both chloride ions, like that of the sulfamido N atom in **I** [symmetry code: (iv) $-x + 1$, $-y$, $-z + 1$].

Complex **III** is formally the hydronitrate salt of famotidine (Fig. 5). As with the hydrochloride salts, protonation occurs at atom N3 of the guanidine moiety. With regard to the crystal morphology, this nitrate forms large block-like crystals compared with the rod-like crystals observed for the other complexes presented here. This implies potential utility in separations with this different larger morphology. Nitrates are also not inherently hazardous to biological systems and may present an alternative for API development. Like famotidine Form B, the sulfonamide group is curved back towards the thiazole ring forming a slightly open 'C' shape in the solid state.

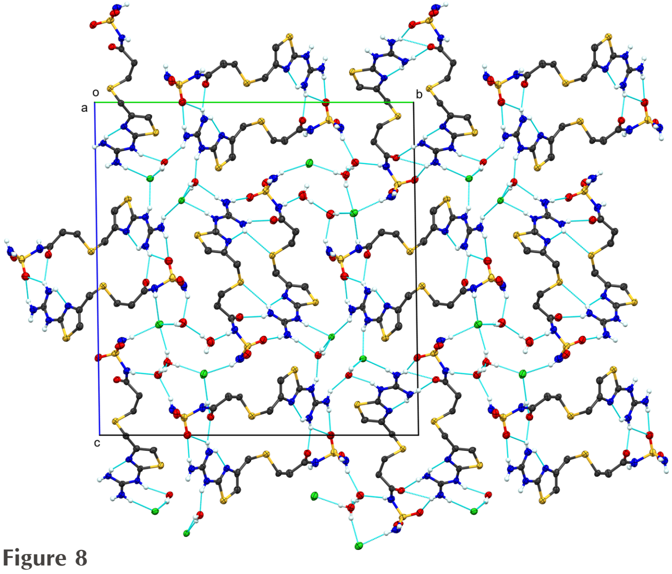


Figure 8
Packing diagram of **IV**, viewed along the *a* axis. Blue dashed lines represent hydrogen-bond interactions. Atomic displacement ellipsoids are shown at the 50% probability level. Only H atoms involved in hydrogen bonding are shown.

Table 6
Hydrogen-bond geometry (Å, °) for **IV**.

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1*A*···O16$^{\rm i}$	0.87	2.02	2.821 (7)	152
N1—H1*B*···O3$^{\rm ii}$	0.87	2.08	2.913 (8)	160
N2—H2*A*···N4	0.87	2.05	2.678 (8)	128
N2—H2*B*···O1$^{\rm ii}$	0.87	2.06	2.871 (7)	155
N2—H2*B*···O3$^{\rm ii}$	0.87	2.61	3.240 (8)	130
N3—H3···O17$^{\rm ii}$	0.87	1.96	2.781 (7)	156
N5—H5···Cl1$^{\rm iii}$	0.87	2.61	3.262 (6)	133
N6—H6*A*···O2$^{\rm iv}$	0.87	1.98	2.810 (7)	159
N6—H6*B*···O14$^{\rm iii}$	0.87	2.17	3.007 (8)	162
N7—H7*C*···O4$^{\rm v}$	0.87	2.48	3.138 (6)	133
N7—H7*C*···O5$^{\rm v}$	0.87	2.18	2.966 (7)	150
N7—H7*D*···Cl4$^{\rm iv}$	0.87	2.39	3.245 (5)	168
N8—H8*A*···O4$^{\rm v}$	0.87	2.05	2.904 (7)	167
N8—H8*B*···N10	0.87	1.93	2.709 (8)	149
N9—H9···O16	0.87	1.98	2.817 (6)	161
N11—H11···O14$^{\rm iii}$	0.87	2.10	2.862 (7)	146
N12—H12*A*···O6$^{\rm iv}$	0.87	1.97	2.798 (7)	158
N12—H12*B*···Cl2	0.87	2.41	3.278 (6)	174
N13—H13*C*···O8$^{\rm vi}$	0.87	1.94	2.811 (7)	175
N13—H13*D*···O17$^{\rm vii}$	0.87	2.14	3.011 (7)	175
N14—H14*C*···S8$^{\rm viii}$	0.87	3.00	3.680 (5)	136
N14—H14*C*···N16	0.87	2.10	2.750 (7)	131
N14—H14*D*···O7$^{\rm vi}$	0.87	2.13	2.932 (7)	154
N15—H15···Cl3$^{\rm ix}$	0.87	2.25	3.083 (5)	159
N17—H17···O15	0.87	1.90	2.756 (8)	168
N18—H18*A*···O9$^{\rm iv}$	0.87	2.05	2.890 (7)	162
N18—H18*B*···Cl1	0.87	2.42	3.257 (6)	162
N19—H19*A*···Cl3$^{\rm v}$	0.87	2.48	3.213 (6)	142
N19—H19*B*···O11$^{\rm x}$	0.87	2.06	2.876 (8)	155
N20—H20*A*···N22	0.87	2.07	2.700 (8)	129
N20—H20*B*···O10$^{\rm x}$	0.87	2.26	2.964 (7)	137
N20—H20*B*···O11$^{\rm x}$	0.87	2.42	3.167 (8)	144
N21—H21···Cl4$^{\rm xi}$	0.87	2.34	3.110 (5)	148
N23—H23···Cl2$^{\rm iii}$	0.87	2.49	3.294 (6)	154
N24—H24*A*···O12$^{\rm iv}$	0.87	1.99	2.807 (8)	157
N24—H24*B*···O13$^{\rm iii}$	0.87	2.15	2.933 (8)	150
O13—H13*E*···Cl2$^{\rm iv}$	0.85	2.29	3.142 (6)	175
O13—H13*F*···Cl2	0.85	2.37	3.200 (6)	164
O14—H14*E*···O18	0.85	1.95	2.798 (8)	172
O14—H14*F*···O18$^{\rm iv}$	0.85	2.20	2.884 (8)	138
O15—H15*C*···Cl1	0.85	2.85	3.335 (6)	118
O15—H15*D*···O13$^{\rm iii}$	0.85	1.97	2.813 (8)	175
O16—H16*A*···Cl4$^{\rm iv}$	0.85	2.18	2.992 (5)	160
O16—H16*B*···Cl4	0.85	2.37	3.195 (5)	164
O17—H17*A*···Cl3$^{\rm iii}$	0.85	2.30	3.100 (5)	158
O17—H17*B*···Cl3	0.85	2.32	3.165 (5)	173
O18—H18*C*···Cl1	0.85	2.20	3.045 (5)	174
O18—H18*D*···Cl2	0.85	2.32	3.148 (5)	165

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

Germane to these complexes, there is an intramolecular hydrogen bond from guanidine atom N2 to thiazole atom N4 (Table 4). Like **II**, atom N2 also forms a bifurcated hydrogen bond to S2$^{\rm iv}$ on a neighboring cation [symmetry code: (iv) $-x + 1$, $-y + 1$, $-z + 1$]. Another similarity with **II** is the intramolecular hydrogen bond from N7 to O2, in contrast with **I**. Predictably, the nitrate anion serves as a hydrogen-bond hub in this structure. Atom O3 is an acceptor of one hydrogen bond that is shared (bifurcated) with O5. Atoms O4 and O5 both accept three hydrogen bonds. Atom O4 is an acceptor for hydrogen bonds from sulfamolyl atoms N6 and N7 of one cation, and N7 of a second famotidine cation. The bifurcated hydrogen bond between O3 and O5 originates from guanidine atom N1 on a neighboring cation. Both H atoms on guanidine atom N2 form bifurcated hydrogen bonds. One is the intramolecular hydrogen bond described above, that is shared with S2^{iv}. The second H atom forms contacts with nitrate atom O5^{i} and sulfonamide atom O1^{iii} of a second neighboring cation, that also accepts a second hydrogen bond from N3 from a different cation [symmetry codes: (i) $x + 1, -y + \frac{3}{2}, z - \frac{1}{2}$; (iii) $-x + 1, -y + 1, -z$]. The third hydrogen bond to nitrate atom O5 is from sulfonamide atom N6. These various interactions are highlighted in Fig. 6.

Complex **IV** presents an unusual modification of the parent famotidine compound. The amidine N atom (N7 in the parent compound) has been replaced with an O atom and sulfonamide atom N6 has been protonated (Fig. 7). Evidence for this modification appears in the form of the hydrogen bonds in which these atoms are involved (see below for details). Given that hydrochloric acid is present in the crystallization medium, presumably this is an acid hydrolysis. Furthermore, this is an example of a high-Z' structure with four crystallographically independent cations and associated anions in the asymmetric unit. It is also a sesquihydrate, with six unique water molecules in the standard unit (1.5 water molecules per salt). Inspection of the differences between the four cations is highlighted in the overlay (Fig. S4). Molecules 2 (S4) and 4 (S10) are remarkably similar. In contrast, molecules 1 (S1) and 3 (S7) have a similar orientation along the propionamide chain and deviate at C6/C22. The torsion angles along the propionamide C—S—C—C chain and N(thiazole)—C(thiazole)—C—S highlight these differences (Table 5).

Complex **IV** appears to be structurally similar to Famotidine Related Compound C (or Famotidine Impurity C), a known degradation product of the API famotidine (USP-NF, 2020). The compound's chemical name is 3-[({2-[(diaminomethylidene)amino]-1,3-thiazol-4-yl}methyl)sulfanyl]-*N*-sulfamoylpropanamide ($C_8H_{14}N_6O_3S_3$). Famotidine Impurity C is primarily formed through the hydrolysis of famotidine (Junnarkar & Stavchansky, 1995; Suleiman *et al.*, 1989), which distinguishes it from a synthetic impurity. Therefore, it is reasonable to conclude that **IV** is a hydrated salt of the freebase Famotidine Impurity C, a known and previously characterized degradation product of the API famotidine.

With four crystallographically-independent cations, associated anions, and solvent molecules, the extended structure of **IV** has numerous intermolecular interactions (Table 6 and Fig. 8). Thus, discussion will be restricted to the more salient features of the packing. From one perspective, each of the cations forms a centrosymmetric self-dimer. The dimers are stacked along the *a* axis. Ignoring the anion and waters of crystallization, these four stacks of molecules form a herringbone pattern. Located within channels formed between the herringbone array are two channels. These channels are populated with hydrogen-bonded chains of water molecules and Cl atoms. One chain consists of chloride ions Cl1 and Cl2, along with water molecules O13, O14, O15, and O18. The second channel contains the two remaining chloride ions (Cl3 and Cl4) and water molecules O16 and O17. The ubiquitous guanidine-to-thiazole N-atom hydrogen bond is present in all four cations. The hydrogen-bonded self-dimers, for three of the four modified famotidine molecules, are formed by hydrogen bonds from the guanidine moiety on one molecule to a sulfonamide O atom and the adjacent carbonyl O atom that has replaced the amidinate N atom by hydrolysis. The outlier is the chain formed by the fourth molecule (S7) that forms hydrogen-bond contacts with two different centrosymmetric cations. The guanidine moiety forms hydrogen bonds to the sulfonamide and adjacent carbonyl O atom on one cation (N13$\cdots$O8^{vi} and N14$\cdots$O7^{vi}). Unlike the other three cations, one guanidine N atom (N14) forms a hydrogen bond to S8^{viii} of a second cation [symmetry codes: (vi) $-x + 2, -y + 1, -z + 1$; (viii) $-x + 1, -y + 1, -z + 1$]. This results in the sulfonamide and carbonyl O atoms at the terminus of the standard cation accepting hydrogen bonds from the guanidine of this second hydrogen-bonded inversion-related cation. Despite the lack of translation symmetry that is typical for such formations, this chain of hydrogen-bonded molecules adopts a helical motif.

4. Conclusion

Famotidine, a widely distributed anti-GERD drug typically formulated as the hydrochloride salt, was systematically investigated across a range of crystallization conditions and acidities. As a result of this study, we successfully characterized the crystal structures of four new complexes: a polymorph of the famotidine hydrochloride salt, a hemihydrate of the famotidine hydrochloride salt, and a famotidine hydronitrate salt. The fourth structure represents a hydrolyzed salt of a known famotidine degradation product; it is formed through the replacement of the amidine N atom by a carbonyl group, accompanied by protonation at a neighboring N atom. All characterized structures are stabilized *via* extensive intra- and intermolecular hydrogen-bonded networks. These structural elucidations provide the necessary foundation for characterizing more complex systems, including the colored famotidine–metal complexes found on the activated anti-GERD PADs.

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Hydrochlorides, hydrates, hydronitrate, and an unanticipated hydrolysis product of famotidine

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Computing details

N-(Diaminomethylidene)-4-({[2-(*N*′-sulfamoylcarbamimidoyl)ethyl]sulfanyl}methyl)-1,3-thiazol-2-aminium chloride (I)

Crystal data

$C_8H_{16}N_7O_2S_3^+\cdot Cl^-$
$M_r = 373.91$
Monoclinic, $P2_1/n$
$a = 8.8712\ (14)$ Å
$b = 8.4069\ (13)$ Å
$c = 21.423\ (3)$ Å
$\beta = 90.357\ (3)°$
$V = 1597.7\ (4)$ Å^3
$Z = 4$
$F(000) = 776$
$D_x = 1.554$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 6463 reflections
$\theta = 2.5–28.3°$
$\mu = 0.65$ mm^{-1}
$T = 120$ K
Block, colourless
0.20 × 0.11 × 0.06 mm

Data collection

Bruker D8 diffractometer
Radiation source: fine-focus sealed tube, Siemens
Bruker TRIUMPH curved-graphite monochromator
Detector resolution: 8.33 pixels mm^{-1}
combination of ω and φ–scans
Absorption correction: numerical (SADABS; Krause *et al.*, 2015)
$T_{min} = 0.945$, $T_{max} = 0.989$
23845 measured reflections
3963 independent reflections
3237 reflections with $I > 2\sigma(I)$
$R_{int} = 0.040$
$\theta_{max} = 28.3°$, $\theta_{min} = 1.9°$
$h = -11\rightarrow11$
$k = -10\rightarrow11$
$l = -28\rightarrow28$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.028$
$wR(F^2) = 0.069$
$S = 1.04$
3963 reflections
224 parameters
0 restraints
Primary atom site location: dual
Secondary atom site location: difference Fourier map
Hydrogen site location: mixed
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0325P)^2 + 0.5785P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.33$ e Å^{-3}
$\Delta\rho_{min} = -0.36$ e Å^{-3}

Special details

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

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

	x	y	z	$U_{\rm iso}$*/$U_{\rm eq}$
Cl1	0.59332 (4)	0.53484 (5)	0.27098 (2)	0.01882 (9)
S1	0.16869 (5)	0.07142 (5)	0.53532 (2)	0.02274 (10)
S2	0.66278 (5)	−0.04290 (5)	0.65447 (2)	0.02166 (10)
S3	1.04458 (4)	0.59458 (5)	0.61870 (2)	0.01517 (9)
O1	1.15917 (13)	0.50802 (14)	0.58541 (5)	0.0206 (2)
O2	0.96733 (13)	0.72088 (14)	0.58636 (5)	0.0224 (3)
N1	0.40872 (18)	0.44606 (17)	0.39305 (7)	0.0207 (3)
H1A	0.482 (3)	0.477 (3)	0.3722 (10)	0.038 (6)*
H1B	0.317 (3)	0.457 (3)	0.3794 (10)	0.038 (6)*
N2	0.57262 (16)	0.33924 (18)	0.46602 (7)	0.0193 (3)
H2A	0.648 (3)	0.379 (3)	0.4491 (10)	0.036 (6)*
H2B	0.585 (2)	0.295 (3)	0.5000 (10)	0.031 (6)*
N3	0.31561 (16)	0.29937 (17)	0.47297 (6)	0.0169 (3)
H3	0.240 (2)	0.303 (2)	0.4525 (9)	0.020 (5)*
N4	0.42614 (15)	0.19158 (16)	0.56595 (6)	0.0157 (3)
N5	0.91090 (15)	0.47578 (16)	0.64146 (6)	0.0175 (3)
N6	1.12835 (18)	0.67398 (18)	0.67697 (7)	0.0187 (3)
H6A	1.081 (2)	0.742 (3)	0.7003 (10)	0.037 (6)*
H6B	1.191 (2)	0.627 (3)	0.6918 (9)	0.025 (6)*
N7	1.07811 (17)	0.29294 (18)	0.68949 (7)	0.0211 (3)
H7A	1.156 (2)	0.341 (2)	0.6810 (9)	0.025*
H7B	1.085 (2)	0.208 (3)	0.7121 (9)	0.025*
C1	0.43631 (18)	0.36178 (18)	0.44374 (7)	0.0162 (3)
C2	0.31921 (17)	0.19826 (18)	0.52431 (7)	0.0152 (3)
C3	0.38764 (17)	0.08105 (19)	0.61165 (7)	0.0156 (3)
C4	0.25459 (19)	0.0072 (2)	0.60262 (8)	0.0204 (3)
H4	0.213684	−0.070309	0.629974	0.025*
C5	0.48493 (18)	0.0578 (2)	0.66802 (7)	0.0183 (3)
H5A	0.427340	−0.003969	0.699167	0.022*
H5B	0.506524	0.163365	0.686509	0.022*
C6	0.78602 (19)	0.1205 (2)	0.63459 (8)	0.0205 (3)
H6C	0.883950	0.076954	0.620722	0.025*
H6D	0.741263	0.179748	0.599106	0.025*
C7	0.81400 (18)	0.23608 (19)	0.68857 (7)	0.0172 (3)
H7C	0.722313	0.300267	0.696045	0.021*
H7D	0.837646	0.175946	0.727136	0.021*
C8	0.94386 (18)	0.34390 (19)	0.67254 (7)	0.0166 (3)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl1	0.0212 (2)	0.01681 (19)	0.01845 (18)	−0.00155 (15)	0.00180 (14)	0.00123 (14)
S1	0.0186 (2)	0.0264 (2)	0.0232 (2)	−0.01265 (17)	−0.00296 (16)	0.00456 (17)
S2	0.0207 (2)	0.01272 (19)	0.0315 (2)	−0.00312 (16)	−0.00312 (17)	−0.00172 (16)
S3	0.01501 (19)	0.01413 (19)	0.01640 (18)	−0.00395 (14)	0.00120 (14)	0.00186 (14)
O1	0.0193 (6)	0.0216 (6)	0.0210 (6)	−0.0050 (5)	0.0058 (4)	−0.0035 (5)
O2	0.0194 (6)	0.0208 (6)	0.0269 (6)	−0.0045 (5)	−0.0038 (5)	0.0088 (5)
N1	0.0197 (8)	0.0219 (7)	0.0207 (7)	−0.0057 (6)	0.0025 (6)	0.0035 (6)
N2	0.0161 (7)	0.0201 (7)	0.0218 (7)	−0.0037 (6)	0.0053 (6)	0.0024 (6)
N3	0.0146 (7)	0.0208 (7)	0.0154 (6)	−0.0047 (6)	−0.0011 (5)	0.0027 (5)
N4	0.0162 (6)	0.0155 (7)	0.0155 (6)	−0.0048 (5)	0.0025 (5)	−0.0008 (5)
N5	0.0154 (7)	0.0161 (7)	0.0209 (7)	−0.0045 (5)	0.0018 (5)	0.0023 (5)
N6	0.0226 (8)	0.0151 (7)	0.0184 (7)	−0.0012 (6)	−0.0019 (6)	−0.0008 (6)
N7	0.0173 (7)	0.0153 (7)	0.0307 (8)	−0.0036 (6)	−0.0006 (6)	0.0052 (6)
C1	0.0197 (8)	0.0128 (7)	0.0163 (7)	−0.0041 (6)	0.0037 (6)	−0.0042 (6)
C2	0.0153 (7)	0.0140 (7)	0.0165 (7)	−0.0048 (6)	0.0032 (6)	−0.0020 (6)
C3	0.0166 (8)	0.0144 (7)	0.0159 (7)	−0.0030 (6)	0.0039 (6)	−0.0004 (6)
C4	0.0209 (8)	0.0203 (8)	0.0201 (8)	−0.0072 (7)	0.0018 (6)	0.0036 (7)
C5	0.0188 (8)	0.0186 (8)	0.0175 (7)	−0.0048 (6)	0.0027 (6)	−0.0011 (6)
C6	0.0184 (8)	0.0210 (8)	0.0221 (8)	−0.0046 (7)	0.0022 (6)	−0.0032 (7)
C7	0.0180 (8)	0.0153 (8)	0.0183 (7)	−0.0054 (6)	0.0010 (6)	0.0011 (6)
C8	0.0186 (8)	0.0145 (8)	0.0167 (7)	−0.0038 (6)	0.0018 (6)	−0.0024 (6)

Geometric parameters (Å, º)

S1—C4	1.7138 (18)	N4—C3	1.3938 (19)
S1—C2	1.7262 (16)	N5—C8	1.325 (2)
S2—C6	1.8080 (17)	N6—H6A	0.87 (2)
S2—C5	1.8155 (17)	N6—H6B	0.76 (2)
S3—O2	1.4392 (12)	N7—C8	1.315 (2)
S3—O1	1.4424 (12)	N7—H7A	0.82 (2)
S3—N6	1.5952 (15)	N7—H7B	0.86 (2)
S3—N5	1.6275 (13)	C3—C4	1.347 (2)
N1—C1	1.318 (2)	C3—C5	1.493 (2)
N1—H1A	0.84 (2)	C4—H4	0.9500
N1—H1B	0.87 (2)	C5—H5A	0.9900
N2—C1	1.311 (2)	C5—H5B	0.9900
N2—H2A	0.83 (2)	C6—C7	1.530 (2)
N2—H2B	0.82 (2)	C6—H6C	0.9900
N3—C1	1.350 (2)	C6—H6D	0.9900
N3—C2	1.390 (2)	C7—C8	1.507 (2)
N3—H3	0.802 (19)	C7—H7C	0.9900
N4—C2	1.299 (2)	C7—H7D	0.9900
C4—S1—C2	88.24 (8)	N4—C2—S1	116.20 (11)
C6—S2—C5	102.17 (8)	N3—C2—S1	118.15 (12)

O2—S3—O1	117.97 (7)	C4—C3—N4	115.14 (14)
O2—S3—N6	106.63 (8)	C4—C3—C5	123.90 (14)
O1—S3—N6	105.77 (8)	N4—C3—C5	120.83 (14)
O2—S3—N5	104.56 (7)	C3—C4—S1	111.13 (12)
O1—S3—N5	110.83 (7)	C3—C4—H4	124.4
N6—S3—N5	111.05 (7)	S1—C4—H4	124.4
C1—N1—H1A	117.9 (16)	C3—C5—S2	115.49 (11)
C1—N1—H1B	120.3 (15)	C3—C5—H5A	108.4
H1A—N1—H1B	121 (2)	S2—C5—H5A	108.4
C1—N2—H2A	121.7 (15)	C3—C5—H5B	108.4
C1—N2—H2B	120.3 (15)	S2—C5—H5B	108.4
H2A—N2—H2B	118 (2)	H5A—C5—H5B	107.5
C1—N3—C2	126.17 (14)	C7—C6—S2	113.58 (11)
C1—N3—H3	113.4 (14)	C7—C6—H6C	108.8
C2—N3—H3	118.1 (14)	S2—C6—H6C	108.8
C2—N4—C3	109.27 (13)	C7—C6—H6D	108.8
C8—N5—S3	120.34 (11)	S2—C6—H6D	108.8
S3—N6—H6A	120.2 (14)	H6C—C6—H6D	107.7
S3—N6—H6B	116.6 (16)	C8—C7—C6	109.30 (12)
H6A—N6—H6B	117 (2)	C8—C7—H7C	109.8
C8—N7—H7A	122.6 (14)	C6—C7—H7C	109.8
C8—N7—H7B	118.9 (13)	C8—C7—H7D	109.8
H7A—N7—H7B	118.4 (19)	C6—C7—H7D	109.8
N2—C1—N1	122.94 (15)	H7C—C7—H7D	108.3
N2—C1—N3	120.49 (15)	N7—C8—N5	127.46 (15)
N1—C1—N3	116.57 (15)	N7—C8—C7	115.69 (14)
N4—C2—N3	125.64 (14)	N5—C8—C7	116.81 (14)

O2—S3—N5—C8	179.14 (12)	N4—C3—C4—S1	0.34 (19)
O1—S3—N5—C8	51.02 (14)	C5—C3—C4—S1	176.25 (12)
N6—S3—N5—C8	−66.23 (15)	C2—S1—C4—C3	−0.94 (13)
C2—N3—C1—N2	−5.4 (2)	C4—C3—C5—S2	112.27 (16)
C2—N3—C1—N1	175.28 (15)	N4—C3—C5—S2	−72.04 (17)
C3—N4—C2—N3	177.55 (14)	C6—S2—C5—C3	87.83 (13)
C3—N4—C2—S1	−1.50 (17)	C5—S2—C6—C7	66.00 (13)
C1—N3—C2—N4	26.6 (2)	S2—C6—C7—C8	166.90 (11)
C1—N3—C2—S1	−154.34 (13)	S3—N5—C8—N7	1.1 (2)
C4—S1—C2—N4	1.46 (13)	S3—N5—C8—C7	−176.57 (11)
C4—S1—C2—N3	−177.66 (13)	C6—C7—C8—N7	−90.06 (17)
C2—N4—C3—C4	0.72 (19)	C6—C7—C8—N5	87.86 (17)
C2—N4—C3—C5	−175.32 (14)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1*A*···Cl1	0.84 (2)	2.44 (2)	3.1828 (15)	149 (2)
N1—H1*B*···N5^{i}	0.87 (2)	2.14 (2)	2.999 (2)	169 (2)
N2—H2*A*···O1^{ii}	0.83 (2)	2.09 (2)	2.9253 (18)	174 (2)

N2—H2*B*···N4	0.82 (2)	2.18 (2)	2.801 (2)	131.9 (19)
N3—H3···O2$^{\mathrm{i}}$	0.802 (19)	2.02 (2)	2.8115 (19)	168.2 (19)
N6—H6*A*···Cl1$^{\mathrm{iii}}$	0.87 (2)	2.42 (2)	3.1863 (16)	148.6 (19)
N6—H6*B*···Cl1$^{\mathrm{ii}}$	0.76 (2)	2.47 (2)	3.2230 (18)	174 (2)
N7—H7*A*···Cl1$^{\mathrm{ii}}$	0.82 (2)	2.66 (2)	3.3584 (16)	144.1 (17)
N7—H7*A*···O1	0.82 (2)	2.48 (2)	2.9627 (19)	118.4 (16)
N7—H7*B*···Cl1$^{\mathrm{iv}}$	0.86 (2)	2.40 (2)	3.2644 (16)	176.7 (18)

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

N-(Diaminomethylidene)-4-({[2-(*N′*-sulfamoylcarbamimidoyl)ethyl]sulfanyl}methyl)-1,3-thiazol-2-aminium chloride hemihydrate (II)

Crystal data

$C_8H_{16}N_7O_2S_3^+\cdot Cl^-\cdot 0.5H_2O$
$M_r = 382.91$
Triclinic, $P\overline{1}$
$a = 8.5104\ (3)$ Å
$b = 13.8816\ (4)$ Å
$c = 14.1753\ (5)$ Å
$\alpha = 92.178\ (1)°$
$\beta = 92.655\ (1)°$
$\gamma = 107.297\ (1)°$
$V = 1594.84\ (9)$ Å^3

$Z = 4$
$F(000) = 796$
$D_x = 1.595$ Mg m^{-3}
Mo *Kα* radiation, $\lambda = 0.71073$ Å
Cell parameters from 9920 reflections
$\theta = 2.8–28.3°$
$\mu = 0.65$ mm^{-1}
$T = 120$ K
Block, pink
0.18 × 0.12 × 0.07 mm

Data collection

Bruker D8 diffractometer
Radiation source: fine-focus sealed tube, Siemens
Bruker TRIUMPH curved-graphite monochromator
Detector resolution: 8.33 pixels mm^{-1}
combination of ω and φ–scans
Absorption correction: numerical (SADABS; Krause *et al.*, 2015)

$T_{\min} = 0.830$, $T_{\max} = 0.933$
40692 measured reflections
7938 independent reflections
6530 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.044$
$\theta_{\max} = 28.3°$, $\theta_{\min} = 1.4°$
$h = -11\rightarrow 11$
$k = -18\rightarrow 17$
$l = -18\rightarrow 18$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.062$
$wR(F^2) = 0.130$
$S = 1.09$
7938 reflections
398 parameters
0 restraints
Primary atom site location: dual

Secondary atom site location: difference Fourier map
Hydrogen site location: mixed
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.029P)^2 + 5.5883P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} = 0.001$
$\Delta\rho_{\max} = 1.37$ e Å^{-3}
$\Delta\rho_{\min} = -1.79$ e Å^{-3}

Special details

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

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}	Occ. (<1)
S1	1.11861 (11)	0.49380 (6)	0.86445 (6)	0.02074 (17)	
S2	0.92217 (10)	0.34981 (6)	0.53476 (5)	0.01849 (17)	
S3	0.34507 (10)	0.20370 (6)	0.55068 (6)	0.01703 (16)	
O1	0.3215 (3)	0.27816 (18)	0.48783 (17)	0.0223 (5)	
O2	0.3282 (3)	0.22041 (19)	0.65016 (17)	0.0265 (5)	
N1	0.8992 (4)	0.7847 (2)	0.8295 (2)	0.0273 (7)	
H1A	0.855354	0.826442	0.800753	0.033*	
H1B	0.911626	0.787905	0.891647	0.033*	
N2	0.9299 (3)	0.7101 (2)	0.68642 (19)	0.0201 (6)	
H2A	0.886343	0.751023	0.656012	0.024*	
H2B	0.962315	0.664474	0.654645	0.024*	
N3	1.0119 (3)	0.6543 (2)	0.82821 (19)	0.0199 (6)	
H3	1.026735	0.665584	0.889967	0.024*	
N4	1.0585 (3)	0.5528 (2)	0.69975 (18)	0.0164 (5)	
N5	0.5287 (3)	0.1969 (2)	0.53012 (19)	0.0192 (6)	
N6	0.2090 (4)	0.0995 (2)	0.5171 (2)	0.0292 (7)	
H6A	0.142636	0.065095	0.557949	0.035*	
H6B	0.200571	0.076703	0.457709	0.035*	
N7	0.5459 (4)	0.0964 (3)	0.6589 (2)	0.0297 (7)	
H7A	0.448022	0.094836	0.677576	0.036*	
H7B	0.605700	0.064783	0.690227	0.036*	
C1	0.9465 (4)	0.7172 (2)	0.7795 (2)	0.0190 (6)	
C2	1.0577 (4)	0.5740 (2)	0.7894 (2)	0.0155 (6)	
C3	1.1120 (4)	0.4678 (2)	0.6857 (2)	0.0169 (6)	
C4	1.1511 (4)	0.4275 (2)	0.7652 (2)	0.0212 (7)	
H4	1.191026	0.370478	0.766592	0.025*	
C5	1.1210 (4)	0.4298 (2)	0.5866 (2)	0.0200 (6)	
H5A	1.201298	0.390825	0.586123	0.024*	
H5B	1.162067	0.488280	0.546931	0.024*	
C6	0.9091 (4)	0.2378 (2)	0.5997 (2)	0.0199 (6)	
H6C	1.015956	0.223096	0.599124	0.024*	
H6D	0.888204	0.251077	0.666399	0.024*	
C7	0.7725 (4)	0.1455 (2)	0.5581 (2)	0.0201 (6)	
H7C	0.775945	0.143031	0.488365	0.024*	
H7D	0.793482	0.083668	0.580886	0.024*	
C8	0.6032 (4)	0.1462 (2)	0.5839 (2)	0.0180 (6)	
S4	0.39140 (12)	0.51951 (7)	0.37230 (6)	0.02549 (19)	
S5	0.54026 (10)	0.63345 (6)	0.04453 (6)	0.01952 (17)	
S6	1.13854 (12)	0.80411 (7)	0.07084 (6)	0.0263 (2)	
O3	1.1569 (4)	0.7858 (3)	0.16885 (19)	0.0479 (9)	
O4	1.1714 (4)	0.7328 (2)	0.00409 (19)	0.0344 (7)	
N8	0.5994 (4)	0.2225 (2)	0.3286 (2)	0.0250 (6)	
H8A	0.634820	0.177351	0.298140	0.030*	
H8B	0.595759	0.222570	0.390572	0.030*	
N9	0.5563 (3)	0.2912 (2)	0.18856 (19)	0.0213 (6)	

H9A	0.591470	0.246511	0.157191	0.026*	
H9B	0.524184	0.336749	0.157982	0.026*	
N10	0.5013 (4)	0.3595 (2)	0.33219 (19)	0.0227 (6)	
H10	0.502571	0.355033	0.393969	0.027*	
N11	0.4346 (3)	0.4515 (2)	0.20662 (19)	0.0185 (5)	
N12	0.9525 (4)	0.8043 (2)	0.0380 (2)	0.0308 (7)	
N13	1.2600 (6)	0.9139 (3)	0.0543 (3)	0.0581 (14)	
H13C	1.321935	0.950530	0.101929	0.070*	
H13D	1.264733	0.937771	−0.002536	0.070*	
N14	0.9190 (8)	0.9288 (6)	0.1496 (5)	0.0352 (16)	0.5
H14E	1.024303	0.963310	0.154689	0.042*	0.5
H14F	0.848239	0.949027	0.182449	0.042*	0.5
N14A	0.8961 (8)	0.8354 (6)	0.1906 (4)	0.0342 (16)	0.5
H14C	0.971149	0.807659	0.210733	0.041*	0.5
H14D	0.840555	0.859282	0.231494	0.041*	0.5
C9	0.5518 (4)	0.2901 (3)	0.2815 (2)	0.0205 (7)	
C10	0.4481 (4)	0.4365 (3)	0.2958 (2)	0.0200 (6)	
C11	0.3756 (4)	0.5335 (2)	0.1940 (2)	0.0193 (6)	
C12	0.3443 (4)	0.5788 (3)	0.2747 (2)	0.0241 (7)	
H12	0.302425	0.635112	0.277018	0.029*	
C13	0.3501 (4)	0.5630 (3)	0.0957 (2)	0.0209 (7)	
H13A	0.273787	0.604695	0.095976	0.025*	
H13B	0.296940	0.501153	0.055084	0.025*	
C14	0.5628 (4)	0.7570 (3)	0.0995 (3)	0.0294 (8)	
H14A	0.456469	0.772164	0.092508	0.035*	
H14B	0.591819	0.756814	0.167962	0.035*	
C15	0.6949 (5)	0.8382 (3)	0.0556 (3)	0.0329 (9)	
H15A	0.682994	0.826069	−0.014004	0.039*	
H15B	0.678482	0.904677	0.070561	0.039*	
C16	0.8638 (5)	0.8416 (4)	0.0892 (3)	0.0541 (15)	
Cl1	0.21375 (12)	0.05390 (9)	0.28703 (9)	0.0468 (3)	
Cl2	0.6294 (2)	0.06502 (11)	0.16223 (9)	0.0657 (5)	
O5	0.8476 (4)	0.0370 (3)	0.3364 (4)	0.0837 (16)	
H5C	0.801847	0.048375	0.283537	0.125*	
H5D	0.946117	0.037175	0.322437	0.125*	

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.0256 (4)	0.0206 (4)	0.0167 (4)	0.0085 (3)	−0.0041 (3)	0.0037 (3)
S2	0.0254 (4)	0.0192 (4)	0.0134 (3)	0.0103 (3)	0.0018 (3)	0.0028 (3)
S3	0.0183 (4)	0.0161 (4)	0.0182 (4)	0.0068 (3)	0.0054 (3)	0.0010 (3)
O1	0.0238 (12)	0.0214 (12)	0.0255 (12)	0.0113 (10)	0.0069 (10)	0.0051 (10)
O2	0.0327 (14)	0.0283 (13)	0.0226 (12)	0.0135 (11)	0.0137 (10)	0.0038 (10)
N1	0.0430 (19)	0.0274 (16)	0.0190 (14)	0.0227 (14)	−0.0018 (13)	0.0001 (12)
N2	0.0255 (15)	0.0212 (14)	0.0167 (13)	0.0122 (12)	−0.0029 (11)	0.0007 (11)
N3	0.0270 (15)	0.0235 (14)	0.0114 (12)	0.0120 (12)	−0.0027 (10)	−0.0029 (10)
N4	0.0157 (12)	0.0170 (13)	0.0167 (12)	0.0050 (10)	0.0027 (10)	0.0007 (10)

N5	0.0196 (14)	0.0233 (14)	0.0178 (13)	0.0103 (11)	0.0058 (10)	0.0045 (11)
N6	0.0233 (15)	0.0217 (15)	0.0395 (18)	0.0024 (12)	−0.0009 (13)	0.0009 (13)
N7	0.0240 (15)	0.0447 (19)	0.0238 (15)	0.0131 (14)	0.0052 (12)	0.0176 (14)
C1	0.0174 (15)	0.0197 (16)	0.0187 (15)	0.0042 (12)	−0.0012 (12)	−0.0004 (12)
C2	0.0127 (14)	0.0168 (14)	0.0163 (14)	0.0038 (11)	−0.0020 (11)	0.0032 (11)
C3	0.0142 (14)	0.0164 (15)	0.0201 (15)	0.0044 (12)	0.0021 (11)	0.0010 (12)
C4	0.0240 (17)	0.0173 (15)	0.0238 (16)	0.0090 (13)	−0.0022 (13)	0.0014 (13)
C5	0.0200 (16)	0.0202 (16)	0.0226 (16)	0.0087 (13)	0.0092 (13)	0.0039 (13)
C6	0.0164 (15)	0.0242 (17)	0.0202 (15)	0.0070 (13)	0.0006 (12)	0.0082 (13)
C7	0.0201 (16)	0.0178 (15)	0.0256 (17)	0.0092 (13)	0.0055 (13)	0.0059 (13)
C8	0.0171 (15)	0.0188 (15)	0.0159 (14)	0.0026 (12)	−0.0011 (12)	0.0001 (12)
S4	0.0333 (5)	0.0228 (4)	0.0166 (4)	0.0022 (4)	0.0066 (3)	−0.0023 (3)
S5	0.0247 (4)	0.0203 (4)	0.0162 (4)	0.0103 (3)	0.0049 (3)	0.0019 (3)
S6	0.0382 (5)	0.0206 (4)	0.0148 (4)	0.0011 (4)	−0.0016 (3)	0.0025 (3)
O3	0.0347 (16)	0.078 (2)	0.0174 (13)	−0.0044 (16)	−0.0026 (11)	0.0165 (14)
O4	0.0481 (17)	0.0248 (13)	0.0327 (14)	0.0189 (12)	−0.0205 (13)	−0.0059 (11)
N8	0.0266 (15)	0.0308 (16)	0.0184 (14)	0.0091 (13)	0.0009 (11)	0.0087 (12)
N9	0.0237 (14)	0.0252 (15)	0.0171 (13)	0.0100 (12)	0.0026 (11)	0.0046 (11)
N10	0.0250 (15)	0.0319 (16)	0.0118 (12)	0.0088 (12)	0.0020 (11)	0.0060 (11)
N11	0.0173 (13)	0.0205 (14)	0.0159 (13)	0.0029 (11)	0.0008 (10)	0.0011 (10)
N12	0.053 (2)	0.0274 (16)	0.0183 (14)	0.0217 (15)	−0.0006 (14)	0.0013 (12)
N13	0.096 (3)	0.0242 (18)	0.036 (2)	−0.0147 (19)	0.041 (2)	−0.0097 (15)
N14	0.023 (3)	0.053 (4)	0.029 (3)	0.012 (3)	0.003 (3)	−0.018 (3)
N14A	0.030 (3)	0.061 (5)	0.018 (3)	0.024 (3)	0.004 (2)	−0.006 (3)
C9	0.0149 (15)	0.0260 (17)	0.0180 (15)	0.0019 (13)	−0.0009 (12)	0.0057 (13)
C10	0.0159 (15)	0.0229 (16)	0.0175 (15)	0.0002 (13)	0.0024 (12)	−0.0011 (12)
C11	0.0160 (15)	0.0189 (15)	0.0200 (15)	0.0009 (12)	0.0011 (12)	−0.0014 (12)
C12	0.0278 (18)	0.0181 (16)	0.0243 (17)	0.0029 (14)	0.0076 (14)	0.0010 (13)
C13	0.0191 (16)	0.0211 (16)	0.0221 (16)	0.0057 (13)	0.0002 (13)	0.0007 (13)
C14	0.0213 (17)	0.0252 (18)	0.041 (2)	0.0065 (14)	0.0097 (15)	−0.0091 (16)
C15	0.044 (2)	0.0223 (18)	0.032 (2)	0.0080 (17)	0.0091 (17)	0.0027 (15)
C16	0.026 (2)	0.085 (4)	0.030 (2)	−0.014 (2)	0.0118 (17)	−0.029 (2)
Cl1	0.0253 (5)	0.0515 (6)	0.0620 (7)	0.0071 (4)	−0.0079 (5)	0.0336 (6)
Cl2	0.1141 (12)	0.0728 (9)	0.0441 (6)	0.0693 (9)	0.0528 (7)	0.0303 (6)
O5	0.039 (2)	0.080 (3)	0.148 (4)	0.029 (2)	0.036 (2)	0.084 (3)

Geometric parameters (Å, º)

S1—C4	1.729 (3)	S5—C13	1.823 (3)
S1—C2	1.739 (3)	S6—O3	1.432 (3)
S2—C6	1.815 (3)	S6—O4	1.440 (3)
S2—C5	1.828 (3)	S6—N13	1.600 (3)
S3—O2	1.441 (2)	S6—N12	1.630 (4)
S3—O1	1.442 (2)	N8—C9	1.318 (4)
S3—N6	1.600 (3)	N8—H8A	0.8800
S3—N5	1.632 (3)	N8—H8B	0.8800
N1—C1	1.320 (4)	N9—C9	1.321 (4)
N1—H1A	0.8800	N9—H9A	0.8800

N1—H1B	0.8800	N9—H9B	0.8800
N2—C1	1.317 (4)	N10—C9	1.360 (4)
N2—H2A	0.8800	N10—C10	1.387 (4)
N2—H2B	0.8800	N10—H10	0.8800
N3—C1	1.360 (4)	N11—C10	1.295 (4)
N3—C2	1.389 (4)	N11—C11	1.389 (4)
N3—H3	0.8800	N12—C16	1.272 (5)
N4—C2	1.294 (4)	N13—H13C	0.8800
N4—C3	1.396 (4)	N13—H13D	0.8800
N5—C8	1.319 (4)	N14—C16	1.402 (8)
N6—H6A	0.8800	N14—H14E	0.8800
N6—H6B	0.8800	N14—H14F	0.8800
N7—C8	1.323 (4)	N14A—C16	1.462 (8)
N7—H7A	0.8800	N14A—H14C	0.8800
N7—H7B	0.8800	N14A—H14D	0.8800
C3—C4	1.349 (4)	C11—C12	1.359 (5)
C3—C5	1.495 (4)	C11—C13	1.492 (5)
C4—H4	0.9500	C12—H12	0.9500
C5—H5A	0.9900	C13—H13A	0.9900
C5—H5B	0.9900	C13—H13B	0.9900
C6—C7	1.527 (5)	C14—C15	1.513 (5)
C6—H6C	0.9900	C14—H14A	0.9900
C6—H6D	0.9900	C14—H14B	0.9900
C7—C8	1.506 (4)	C15—C16	1.480 (6)
C7—H7C	0.9900	C15—H15A	0.9900
C7—H7D	0.9900	C15—H15B	0.9900
S4—C12	1.728 (4)	O5—H5C	0.8700
S4—C10	1.738 (3)	O5—H5D	0.8700
S5—C14	1.809 (4)		

C4—S1—C2	88.15 (15)	O3—S6—N13	107.6 (2)
C6—S2—C5	98.39 (15)	O4—S6—N13	108.38 (18)
O2—S3—O1	117.83 (15)	O3—S6—N12	113.05 (18)
O2—S3—N6	106.86 (17)	O4—S6—N12	103.61 (16)
O1—S3—N6	106.38 (16)	N13—S6—N12	107.3 (2)
O2—S3—N5	111.59 (15)	C9—N8—H8A	120.0
O1—S3—N5	103.97 (14)	C9—N8—H8B	120.0
N6—S3—N5	110.00 (16)	H8A—N8—H8B	120.0
C1—N1—H1A	120.0	C9—N9—H9A	120.0
C1—N1—H1B	120.0	C9—N9—H9B	120.0
H1A—N1—H1B	120.0	H9A—N9—H9B	120.0
C1—N2—H2A	120.0	C9—N10—C10	126.3 (3)
C1—N2—H2B	120.0	C9—N10—H10	116.9
H2A—N2—H2B	120.0	C10—N10—H10	116.9
C1—N3—C2	125.9 (3)	C10—N11—C11	110.0 (3)
C1—N3—H3	117.0	C16—N12—S6	123.6 (3)
C2—N3—H3	117.0	S6—N13—H13C	120.0
C2—N4—C3	109.9 (3)	S6—N13—H13D	120.0

C8—N5—S3	121.6 (2)	H13C—N13—H13D	120.0
S3—N6—H6A	120.0	C16—N14—H14E	120.0
S3—N6—H6B	120.0	C16—N14—H14F	120.0
H6A—N6—H6B	120.0	H14E—N14—H14F	120.0
C8—N7—H7A	120.0	C16—N14A—H14C	120.0
C8—N7—H7B	120.0	C16—N14A—H14D	120.0
H7A—N7—H7B	120.0	H14C—N14A—H14D	120.0
N2—C1—N1	121.7 (3)	N8—C9—N9	120.9 (3)
N2—C1—N3	121.2 (3)	N8—C9—N10	117.7 (3)
N1—C1—N3	117.1 (3)	N9—C9—N10	121.4 (3)
N4—C2—N3	125.0 (3)	N11—C10—N10	124.5 (3)
N4—C2—S1	115.9 (2)	N11—C10—S4	115.9 (3)
N3—C2—S1	119.1 (2)	N10—C10—S4	119.6 (2)
C4—C3—N4	115.3 (3)	C12—C11—N11	115.4 (3)
C4—C3—C5	126.1 (3)	C12—C11—C13	126.1 (3)
N4—C3—C5	118.6 (3)	N11—C11—C13	118.5 (3)
C3—C4—S1	110.7 (2)	C11—C12—S4	110.3 (3)
C3—C4—H4	124.6	C11—C12—H12	124.8
S1—C4—H4	124.6	S4—C12—H12	124.8
C3—C5—S2	113.0 (2)	C11—C13—S5	113.5 (2)
C3—C5—H5A	109.0	C11—C13—H13A	108.9
S2—C5—H5A	109.0	S5—C13—H13A	108.9
C3—C5—H5B	109.0	C11—C13—H13B	108.9
S2—C5—H5B	109.0	S5—C13—H13B	108.9
H5A—C5—H5B	107.8	H13A—C13—H13B	107.7
C7—C6—S2	112.4 (2)	C15—C14—S5	111.4 (3)
C7—C6—H6C	109.1	C15—C14—H14A	109.3
S2—C6—H6C	109.1	S5—C14—H14A	109.3
C7—C6—H6D	109.1	C15—C14—H14B	109.3
S2—C6—H6D	109.1	S5—C14—H14B	109.3
H6C—C6—H6D	107.9	H14A—C14—H14B	108.0
C8—C7—C6	113.0 (3)	C16—C15—C14	112.9 (4)
C8—C7—H7C	109.0	C16—C15—H15A	109.0
C6—C7—H7C	109.0	C14—C15—H15A	109.0
C8—C7—H7D	109.0	C16—C15—H15B	109.0
C6—C7—H7D	109.0	C14—C15—H15B	109.0
H7C—C7—H7D	107.8	H15A—C15—H15B	107.8
N5—C8—N7	127.1 (3)	N12—C16—N14	126.4 (4)
N5—C8—C7	116.7 (3)	N12—C16—N14A	113.6 (5)
N7—C8—C7	116.2 (3)	N12—C16—C15	121.0 (4)
C12—S4—C10	88.35 (17)	N14—C16—C15	104.6 (5)
C14—S5—C13	98.85 (16)	N14A—C16—C15	117.7 (4)
O3—S6—O4	116.5 (2)	H5C—O5—H5D	104.5

O2—S3—N5—C8	42.6 (3)	O4—S6—N12—C16	165.3 (4)
O1—S3—N5—C8	170.6 (3)	N13—S6—N12—C16	−80.2 (4)
N6—S3—N5—C8	−75.9 (3)	C10—N10—C9—N8	179.5 (3)
C2—N3—C1—N2	−4.4 (5)	C10—N10—C9—N9	0.9 (5)

C2—N3—C1—N1	175.0 (3)	C11—N11—C10—N10	179.0 (3)
C3—N4—C2—N3	179.6 (3)	C11—N11—C10—S4	−0.1 (4)
C3—N4—C2—S1	−0.6 (3)	C9—N10—C10—N11	2.0 (5)
C1—N3—C2—N4	6.7 (5)	C9—N10—C10—S4	−178.9 (3)
C1—N3—C2—S1	−173.1 (3)	C12—S4—C10—N11	0.4 (3)
C4—S1—C2—N4	1.0 (3)	C12—S4—C10—N10	−178.7 (3)
C4—S1—C2—N3	−179.1 (3)	C10—N11—C11—C12	−0.5 (4)
C2—N4—C3—C4	−0.4 (4)	C10—N11—C11—C13	−179.6 (3)
C2—N4—C3—C5	179.6 (3)	N11—C11—C12—S4	0.8 (4)
N4—C3—C4—S1	1.2 (4)	C13—C11—C12—S4	179.8 (3)
C5—C3—C4—S1	−178.8 (3)	C10—S4—C12—C11	−0.6 (3)
C2—S1—C4—C3	−1.2 (3)	C12—C11—C13—S5	102.8 (4)
C4—C3—C5—S2	97.6 (4)	N11—C11—C13—S5	−78.2 (3)
N4—C3—C5—S2	−82.3 (3)	C14—S5—C13—C11	−80.4 (3)
C6—S2—C5—C3	−73.7 (3)	C13—S5—C14—C15	−168.8 (3)
C5—S2—C6—C7	−167.4 (2)	S5—C14—C15—C16	−78.0 (4)
S2—C6—C7—C8	−77.5 (3)	S6—N12—C16—N14	36.9 (9)
S3—N5—C8—N7	−0.5 (5)	S6—N12—C16—N14A	−30.7 (6)
S3—N5—C8—C7	−179.9 (2)	S6—N12—C16—C15	−179.2 (3)
C6—C7—C8—N5	84.9 (4)	C14—C15—C16—N12	102.5 (5)
C6—C7—C8—N7	−94.5 (4)	C14—C15—C16—N14	−106.9 (5)
O3—S6—N12—C16	38.3 (5)	C14—C15—C16—N14A	−44.8 (6)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1*A*···Cl1^{i}	0.88	2.31	3.177 (3)	170
N1—H1*B*···N12^{ii}	0.88	2.08	2.959 (4)	176
N2—H2*A*···O1^{i}	0.88	2.57	3.233 (4)	132
N2—H2*B*···S2^{iii}	0.88	2.92	3.591 (3)	134
N2—H2*B*···N4	0.88	2.07	2.728 (4)	131
N3—H3···O4^{ii}	0.88	2.01	2.801 (4)	150
N6—H6*A*···O5^{iv}	0.88	2.12	2.826 (5)	137
N6—H6*B*···Cl1	0.88	2.44	3.303 (4)	167
N7—H7*A*···O2	0.88	2.30	2.882 (4)	123
N7—H7*A*···O5^{iv}	0.88	2.63	3.315 (5)	136
N7—H7*B*···Cl1^{iv}	0.88	2.58	3.411 (3)	157
N8—H8*A*···Cl2	0.88	2.42	3.226 (3)	152
N8—H8*B*···N5	0.88	2.10	2.959 (4)	166
N9—H9*A*···Cl2	0.88	2.64	3.388 (3)	144
N9—H9*B*···S5^{v}	0.88	2.97	3.634 (3)	134
N9—H9*B*···N11	0.88	2.07	2.727 (4)	131
N10—H10···O1	0.88	2.15	2.821 (4)	133
N13—H13*C*···Cl2^{vi}	0.88	2.71	3.466 (5)	145
N13—H13*D*···Cl2^{vii}	0.88	2.48	3.250 (4)	147
N14$^{\mathrm{a—H14}E}$a···Cl1^{vi}	0.88	2.46	3.131 (6)	134
N14$^{\mathrm{a—H14}F}$a···Cl2^{viii}	0.88	2.81	3.531 (7)	140
N14$^{\mathrm{a—H14}F}$a···O5^{viii}	0.88	2.46	3.167 (8)	138

N14$A^{\text{b—H14}C}$b···O3	0.88	1.82	2.539 (7)	138
N14$A^{\text{b—H14}D}$b···O2$^{\text{i}}$	0.88	2.35	2.999 (7)	131
O5—H5*C*···Cl2	0.87	2.27	3.126 (5)	167
O5—H5*D*···Cl1$^{\text{ix}}$	0.87	2.30	3.168 (4)	175

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

N-(Diaminomethylidene)-4-({[2-(*N′*-sulfamoylcarbamimidoyl)ethyl]sulfanyl}methyl)-1,3-thiazol-2-aminium chloride (III)

Crystal data

$C_8H_{16}N_7O_2S_3^+\cdot NO_3^-$	$F(000) = 832$
$M_r = 400.47$	$D_x = 1.652$ Mg m^{-3}
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
$a = 13.5616$ (16) Å	Cell parameters from 9778 reflections
$b = 13.9590$ (17) Å	$\theta = 2.8–28.3°$
$c = 8.504$ (1) Å	$\mu = 0.50$ mm^{-1}
$\beta = 90.597$ (2)°	$T = 120$ K
$V = 1609.8$ (3) Å^3	Needle, colourless
$Z = 4$	0.21 × 0.16 × 0.08 mm

Data collection

Bruker D8 diffractometer	$T_{\min} = 0.927$, $T_{\max} = 0.989$
Radiation source: fine-focus sealed tube, Siemens	24503 measured reflections
Bruker TRIUMPH curved-graphite monochromator	4017 independent reflections
Detector resolution: 8.33 pixels mm^{-1}	3502 reflections with $I > 2\sigma(I)$
combination of ω and φ–scans	$R_{\text{int}} = 0.029$
Absorption correction: numerical (SADABS; Krause *et al.*, 2015)	$\theta_{\max} = 28.4°$, $\theta_{\min} = 1.5°$
	$h = -18\rightarrow18$
	$k = -18\rightarrow18$
	$l = -11\rightarrow11$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: mixed
$R[F^2 > 2\sigma(F^2)] = 0.027$	H atoms treated by a mixture of independent and constrained refinement
$wR(F^2) = 0.068$	$w = 1/[\sigma^2(F_o^2) + (0.0319P)^2 + 0.8958P]$ where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.04$	$(\Delta/\sigma)_{\max} = 0.001$
4017 reflections	$\Delta\rho_{\max} = 0.35$ e Å^{-3}
223 parameters	$\Delta\rho_{\min} = -0.37$ e Å^{-3}
0 restraints	
Primary atom site location: dual	

Special details

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

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
S1	0.47526 (2)	0.86642 (3)	0.60140 (4)	0.01841 (8)
S2	0.34369 (2)	0.53840 (2)	0.48103 (4)	0.01691 (8)
S3	0.20634 (2)	0.54809 (2)	−0.02951 (4)	0.01266 (8)
O1	0.27969 (7)	0.48027 (7)	−0.08103 (11)	0.0165 (2)
O2	0.22344 (7)	0.64776 (7)	−0.06387 (11)	0.0167 (2)
N1	0.76416 (9)	0.82574 (9)	0.25311 (14)	0.0183 (2)
H1A	0.805961	0.795864	0.191678	0.022*
H1B	0.767379	0.888379	0.263686	0.022*
N2	0.68932 (9)	0.68296 (8)	0.31681 (14)	0.0171 (2)
H2A	0.730096	0.651116	0.256252	0.020*
H2B	0.643529	0.652140	0.369119	0.020*
N3	0.63313 (8)	0.82707 (8)	0.42090 (13)	0.0161 (2)
H3	0.642511	0.889335	0.427363	0.019*
N4	0.53607 (8)	0.69882 (8)	0.51894 (13)	0.0145 (2)
N5	0.19643 (9)	0.52773 (8)	0.15869 (13)	0.0162 (2)
N6	0.10529 (9)	0.51902 (9)	−0.11874 (14)	0.0173 (2)
N7	0.11214 (10)	0.67223 (9)	0.20927 (14)	0.0219 (3)
H7A	0.115677	0.691463	0.110943	0.026*
H7B	0.082935	0.708400	0.279590	0.026*
C1	0.69684 (9)	0.77676 (9)	0.32914 (15)	0.0140 (2)
C2	0.55501 (9)	0.78948 (10)	0.50494 (15)	0.0141 (2)
C3	0.45374 (9)	0.68574 (10)	0.61306 (15)	0.0151 (3)
C4	0.41262 (10)	0.76712 (10)	0.66853 (16)	0.0186 (3)
H4	0.356923	0.769325	0.735267	0.022*
C5	0.41907 (10)	0.58607 (10)	0.64222 (16)	0.0184 (3)
H5A	0.380095	0.585040	0.739992	0.022*
H5B	0.477064	0.543992	0.658349	0.022*
C6	0.23051 (10)	0.60362 (10)	0.51883 (16)	0.0168 (3)
H6C	0.215180	0.599749	0.632222	0.020*
H6D	0.239829	0.671958	0.491628	0.020*
C7	0.14364 (10)	0.56281 (10)	0.42247 (15)	0.0163 (3)
H7C	0.142878	0.492177	0.432811	0.020*
H7D	0.081011	0.587763	0.465044	0.020*
C8	0.15060 (10)	0.58920 (10)	0.25123 (15)	0.0145 (2)
O3	−0.08030 (9)	0.67462 (8)	0.48230 (15)	0.0311 (3)
O4	−0.00300 (7)	0.80085 (7)	0.40225 (12)	0.0214 (2)
O5	−0.11729 (8)	0.81307 (7)	0.57880 (12)	0.0220 (2)
N8	−0.06717 (8)	0.76214 (8)	0.48781 (14)	0.0163 (2)
H6A	0.0914 (13)	0.4594 (13)	−0.105 (2)	0.020*
H6B	0.0592 (13)	0.5604 (13)	−0.109 (2)	0.020*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.01507 (16)	0.01719 (16)	0.02303 (17)	0.00182 (12)	0.00265 (13)	−0.00513 (13)

S2	0.01586 (16)	0.01440 (16)	0.02055 (17)	0.00001 (12)	0.00343 (12)	−0.00049 (12)
S3	0.01421 (15)	0.01124 (15)	0.01258 (15)	0.00149 (11)	0.00328 (11)	0.00075 (11)
O1	0.0179 (5)	0.0150 (4)	0.0166 (5)	0.0050 (4)	0.0054 (4)	0.0009 (4)
O2	0.0206 (5)	0.0121 (4)	0.0176 (5)	−0.0001 (4)	0.0032 (4)	0.0014 (4)
N1	0.0187 (6)	0.0169 (6)	0.0196 (6)	−0.0026 (4)	0.0069 (5)	−0.0020 (4)
N2	0.0183 (6)	0.0144 (5)	0.0186 (6)	−0.0012 (4)	0.0057 (5)	−0.0030 (4)
N3	0.0164 (5)	0.0124 (5)	0.0196 (6)	−0.0028 (4)	0.0052 (4)	−0.0036 (4)
N4	0.0130 (5)	0.0167 (5)	0.0138 (5)	−0.0008 (4)	0.0010 (4)	−0.0006 (4)
N5	0.0209 (6)	0.0146 (5)	0.0133 (5)	0.0031 (4)	0.0046 (4)	0.0022 (4)
N6	0.0164 (6)	0.0139 (5)	0.0215 (6)	0.0009 (5)	0.0002 (5)	−0.0005 (5)
N7	0.0311 (7)	0.0220 (6)	0.0125 (5)	0.0118 (5)	0.0023 (5)	−0.0016 (5)
C1	0.0132 (6)	0.0167 (6)	0.0120 (6)	−0.0011 (5)	−0.0010 (5)	−0.0017 (5)
C2	0.0133 (6)	0.0169 (6)	0.0120 (6)	0.0003 (5)	0.0001 (5)	−0.0023 (5)
C3	0.0121 (6)	0.0209 (7)	0.0122 (6)	−0.0009 (5)	−0.0002 (5)	0.0012 (5)
C4	0.0137 (6)	0.0237 (7)	0.0184 (6)	−0.0011 (5)	0.0025 (5)	−0.0022 (5)
C5	0.0160 (6)	0.0216 (7)	0.0175 (6)	−0.0016 (5)	0.0012 (5)	0.0055 (5)
C6	0.0191 (6)	0.0183 (6)	0.0129 (6)	0.0038 (5)	0.0012 (5)	−0.0013 (5)
C7	0.0157 (6)	0.0192 (6)	0.0140 (6)	0.0004 (5)	0.0037 (5)	0.0007 (5)
C8	0.0126 (6)	0.0163 (6)	0.0147 (6)	−0.0009 (5)	0.0005 (5)	−0.0015 (5)
O3	0.0320 (6)	0.0129 (5)	0.0487 (7)	−0.0013 (4)	0.0145 (5)	0.0000 (5)
O4	0.0198 (5)	0.0212 (5)	0.0233 (5)	−0.0075 (4)	0.0086 (4)	−0.0036 (4)
O5	0.0238 (5)	0.0181 (5)	0.0242 (5)	0.0024 (4)	0.0097 (4)	−0.0016 (4)
N8	0.0136 (5)	0.0165 (5)	0.0189 (6)	0.0004 (4)	0.0006 (4)	0.0008 (4)

Geometric parameters (Å, º)

S1—C4	1.7259 (15)	N6—H6A	0.861 (18)
S1—C2	1.7362 (13)	N6—H6B	0.856 (18)
S2—C6	1.8162 (14)	N7—C8	1.3186 (18)
S2—C5	1.8269 (15)	N7—H7A	0.8800
S3—O2	1.4409 (10)	N7—H7B	0.8800
S3—O1	1.4446 (10)	C3—C4	1.353 (2)
S3—N6	1.6115 (12)	C3—C5	1.4903 (19)
S3—N5	1.6324 (12)	C4—H4	0.9500
N1—C1	1.3158 (17)	C5—H5A	0.9900
N1—H1A	0.8800	C5—H5B	0.9900
N1—H1B	0.8800	C6—C7	1.5375 (19)
N2—C1	1.3175 (17)	C6—H6C	0.9900
N2—H2A	0.8800	C6—H6D	0.9900
N2—H2B	0.8800	C7—C8	1.5060 (18)
N3—C1	1.3647 (17)	C7—H7C	0.9900
N3—C2	1.3872 (17)	C7—H7D	0.9900
N3—H3	0.8800	O3—N8	1.2354 (16)
N4—C2	1.2971 (17)	O4—N8	1.2617 (15)
N4—C3	1.3926 (17)	O5—N8	1.2558 (15)
N5—C8	1.3237 (17)		
C4—S1—C2	88.32 (7)	C4—C3—N4	115.23 (12)

C6—S2—C5	98.74 (7)	C4—C3—C5	126.45 (13)
O2—S3—O1	117.29 (6)	N4—C3—C5	118.31 (12)
O2—S3—N6	106.59 (6)	C3—C4—S1	110.67 (10)
O1—S3—N6	106.08 (6)	C3—C4—H4	124.7
O2—S3—N5	112.46 (6)	S1—C4—H4	124.7
O1—S3—N5	104.31 (6)	C3—C5—S2	112.97 (9)
N6—S3—N5	109.85 (6)	C3—C5—H5A	109.0
C1—N1—H1A	120.0	S2—C5—H5A	109.0
C1—N1—H1B	120.0	C3—C5—H5B	109.0
H1A—N1—H1B	120.0	S2—C5—H5B	109.0
C1—N2—H2A	120.0	H5A—C5—H5B	107.8
C1—N2—H2B	120.0	C7—C6—S2	111.36 (9)
H2A—N2—H2B	120.0	C7—C6—H6C	109.4
C1—N3—C2	126.25 (12)	S2—C6—H6C	109.4
C1—N3—H3	116.9	C7—C6—H6D	109.4
C2—N3—H3	116.9	S2—C6—H6D	109.4
C2—N4—C3	109.96 (11)	H6C—C6—H6D	108.0
C8—N5—S3	120.93 (10)	C8—C7—C6	111.68 (11)
S3—N6—H6A	111.4 (12)	C8—C7—H7C	109.3
S3—N6—H6B	113.9 (12)	C6—C7—H7C	109.3
H6A—N6—H6B	118.6 (16)	C8—C7—H7D	109.3
C8—N7—H7A	120.0	C6—C7—H7D	109.3
C8—N7—H7B	120.0	H7C—C7—H7D	107.9
H7A—N7—H7B	120.0	N7—C8—N5	126.56 (13)
N1—C1—N2	122.06 (12)	N7—C8—C7	116.62 (12)
N1—C1—N3	117.40 (12)	N5—C8—C7	116.80 (12)
N2—C1—N3	120.54 (12)	O3—N8—O5	120.29 (12)
N4—C2—N3	124.71 (12)	O3—N8—O4	120.11 (12)
N4—C2—S1	115.80 (10)	O5—N8—O4	119.60 (12)
N3—C2—S1	119.49 (10)		

O2—S3—N5—C8	38.07 (13)	N4—C3—C4—S1	1.00 (15)
O1—S3—N5—C8	166.20 (11)	C5—C3—C4—S1	−178.54 (11)
N6—S3—N5—C8	−80.47 (12)	C2—S1—C4—C3	−1.08 (11)
C2—N3—C1—N1	177.26 (13)	C4—C3—C5—S2	98.24 (15)
C2—N3—C1—N2	−1.8 (2)	N4—C3—C5—S2	−81.28 (14)
C3—N4—C2—N3	178.95 (12)	C6—S2—C5—C3	−75.41 (11)
C3—N4—C2—S1	−0.64 (15)	C5—S2—C6—C7	−167.65 (10)
C1—N3—C2—N4	6.1 (2)	S2—C6—C7—C8	−73.57 (13)
C1—N3—C2—S1	−174.33 (11)	S3—N5—C8—N7	−2.7 (2)
C4—S1—C2—N4	1.02 (11)	S3—N5—C8—C7	178.78 (9)
C4—S1—C2—N3	−178.59 (11)	C6—C7—C8—N7	−86.68 (15)
C2—N4—C3—C4	−0.25 (17)	C6—C7—C8—N5	91.97 (15)
C2—N4—C3—C5	179.33 (12)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1*A*···O3^{i}	0.88	2.40	3.1392 (16)	141
N1—H1*A*···O5^{i}	0.88	2.08	2.9302 (16)	161
N1—H1*A*···N8^{i}	0.88	2.59	3.4547 (17)	170
N1—H1*B*···N5^{ii}	0.88	2.11	2.9647 (17)	164
N2—H2*A*···O1^{iii}	0.88	2.37	3.0669 (15)	137
N2—H2*A*···O5^{i}	0.88	2.62	3.3299 (15)	138
N2—H2*B*···S2^{iv}	0.88	2.95	3.5664 (13)	128
N2—H2*B*···N4	0.88	2.05	2.7199 (16)	132
N3—H3···O1^{ii}	0.88	2.10	2.7914 (15)	135
N7—H7*A*···O2	0.88	2.18	2.8042 (15)	127
N7—H7*A*···O4^{v}	0.88	2.39	3.0515 (16)	133
N7—H7*B*···O4	0.88	2.03	2.8997 (16)	168
N6—H6*A*···O5^{vi}	0.861 (18)	2.085 (18)	2.8992 (17)	157.6 (16)
N6—H6*B*···O4^{v}	0.856 (18)	2.115 (19)	2.9184 (16)	156.1 (16)

Symmetry codes: (i) *x*+1, −*y*+3/2, *z*−1/2; (ii) −*x*+1, *y*+1/2, −*z*+1/2; (iii) −*x*+1, −*y*+1, −*z*; (iv) −*x*+1, −*y*+1, −*z*+1; (v) *x*, −*y*+3/2, *z*−1/2; (vi) −*x*, *y*−1/2, −*z*+1/2.

N-(Diaminomethylidene)-4-({[3-oxo-3-(sulfamoylamino)propyl]sulfanyl}methyl)-1,3-thiazol-2-aminium chloride sesquihydrate (IV)

Crystal data

$C_8H_{15}N_6O_3S_3^+\cdot Cl^-\cdot 1.5H_2O$	$Z = 8$
$M_r = 401.91$	$F(000) = 1672$
Triclinic, $P\bar{1}$	$D_x = 1.564$ Mg m^{-3}
$a = 5.1449$ (6) Å	Cu *Kα* radiation, $\lambda = 1.54178$ Å
$b = 25.285$ (2) Å	Cell parameters from 2407 reflections
$c = 26.306$ (2) Å	$\theta = 2.4$–68.8°
$\alpha = 89.017$ (7)°	$\mu = 5.69$ mm^{-1}
$\beta = 87.760$ (8)°	$T = 120$ K
$\gamma = 86.803$ (8)°	Needle, colourless
$V = 3413.9$ (6) Å^3	0.20 × 0.06 × 0.02 mm

Data collection

Bruker Venture diffractometer	$T_{min} = 0.475$, $T_{max} = 0.757$
	82198 measured reflections
Radiation source: micro-focus, Incoatec Diamond	12581 independent reflections
	8070 reflections with $I > 2\sigma(I)$
HELIOS Multi-layer monochromator	$R_{int} = 0.219$
Detector resolution: 7.41 pixels mm^{-1}	$\theta_{max} = 69.7°$, $\theta_{min} = 1.7°$
combination of *ω* and *φ*–scans	$h = -6 \rightarrow 6$
Absorption correction: numerical (SADABS; Krause *et al.*, 2015)	$k = -30 \rightarrow 30$
	$l = -31 \rightarrow 31$

Refinement

Refinement on F^2	12581 reflections
Least-squares matrix: full	876 parameters
$R[F^2 > 2\sigma(F^2)] = 0.074$	0 restraints
$wR(F^2) = 0.190$	Primary atom site location: dual
$S = 1.04$	

Secondary atom site location: difference Fourier map
Hydrogen site location: mixed
H atoms treated by a mixture of independent and constrained refinement

$w = 1/[\sigma^2(F_o^2) + (0.0871P)^2]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.48$ e Å^{-3}
$\Delta\rho_{min} = -0.52$ e Å^{-3}

Special details

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

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
S1	0.2993 (3)	0.61440 (6)	0.80328 (6)	0.0378 (3)
S2	0.2380 (3)	0.47821 (6)	0.94274 (6)	0.0395 (4)
S3	−0.1721 (3)	0.26614 (6)	0.94736 (6)	0.0348 (3)
O1	0.1985 (9)	0.34892 (17)	0.94553 (17)	0.0362 (9)
O2	−0.4074 (9)	0.24259 (17)	0.9356 (2)	0.0438 (11)
O3	−0.1208 (11)	0.27536 (18)	0.99943 (19)	0.0482 (12)
N1	0.9347 (11)	0.7130 (2)	0.8984 (2)	0.0376 (12)
H1A	0.972914	0.725288	0.868089	0.056*
H1B	1.021045	0.720202	0.924950	0.056*
N2	0.6855 (12)	0.6576 (2)	0.9486 (2)	0.0414 (13)
H2A	0.557014	0.636924	0.953568	0.035 (18)*
H2B	0.756583	0.662490	0.977509	0.05 (2)*
N3	0.6196 (11)	0.66585 (19)	0.8620 (2)	0.0354 (11)
H3	0.646106	0.682573	0.833255	0.07 (3)*
N4	0.3407 (11)	0.60338 (19)	0.9000 (2)	0.0357 (11)
N5	−0.1868 (11)	0.3241 (2)	0.9174 (2)	0.0379 (12)
H5	−0.319488	0.340409	0.903346	0.09 (4)*
N6	0.0590 (11)	0.2300 (2)	0.9239 (2)	0.0372 (12)
H6A	0.212403	0.242257	0.926843	0.031 (17)*
H6B	0.056997	0.224446	0.891369	0.05 (2)*
C1	0.7468 (13)	0.6787 (2)	0.9041 (2)	0.0332 (13)
C2	0.4306 (13)	0.6286 (2)	0.8613 (2)	0.0342 (13)
C3	0.1502 (13)	0.5699 (2)	0.8861 (3)	0.0377 (14)
C4	0.1037 (14)	0.5706 (2)	0.8358 (3)	0.0412 (15)
H4	−0.019361	0.549819	0.820499	0.049*
C5	0.0286 (14)	0.5357 (2)	0.9264 (3)	0.0406 (14)
H5A	−0.137997	0.523769	0.914434	0.049*
H5B	−0.010674	0.557014	0.957333	0.049*
C6	0.2151 (13)	0.4404 (2)	0.8855 (2)	0.0328 (13)
H6C	0.206974	0.465256	0.856070	0.039*
H6D	0.376164	0.417368	0.880908	0.039*
C7	−0.0154 (13)	0.4063 (3)	0.8849 (3)	0.0396 (14)
H7A	−0.174106	0.427521	0.896172	0.048*
H7B	−0.039281	0.394996	0.849649	0.048*

C8	0.0148 (13)	0.3581 (2)	0.9187 (2)	0.0350 (13)
S4	0.5629 (3)	0.19072 (6)	1.09152 (6)	0.0387 (4)
S5	0.5203 (3)	0.04961 (5)	0.95824 (6)	0.0366 (3)
S6	0.3471 (3)	0.05295 (6)	0.75400 (6)	0.0338 (3)
O4	0.7283 (8)	0.05689 (16)	0.83327 (16)	0.0332 (9)
O5	0.4152 (10)	−0.00043 (18)	0.76722 (18)	0.0447 (11)
O6	0.1050 (9)	0.0675 (2)	0.73196 (17)	0.0447 (11)
N7	1.1799 (11)	0.0659 (2)	1.17968 (19)	0.0339 (11)
H7C	1.264019	0.037605	1.190949	0.023 (15)*
H7D	1.217955	0.093820	1.196226	0.026 (16)*
N8	0.9320 (11)	0.02558 (19)	1.12239 (19)	0.0344 (11)
H8A	1.016192	−0.002538	1.134137	0.04 (2)*
H8B	0.799712	0.035068	1.104055	0.031 (17)*
N9	0.8725 (11)	0.11601 (18)	1.13671 (19)	0.0325 (11)
H9	0.896837	0.142551	1.156277	0.049*
N10	0.5879 (10)	0.09083 (19)	1.07284 (18)	0.0319 (11)
N11	0.3354 (10)	0.0884 (2)	0.8072 (2)	0.0332 (11)
H11	0.193229	0.108330	0.811741	0.029 (17)*
N12	0.5695 (10)	0.0738 (2)	0.7161 (2)	0.0389 (12)
H12A	0.725236	0.064262	0.725667	0.031 (17)*
H12B	0.529353	0.105871	0.705321	0.04 (2)*
C9	0.9954 (12)	0.0683 (2)	1.1462 (2)	0.0308 (12)
C10	0.6844 (12)	0.1258 (2)	1.1010 (2)	0.0300 (12)
C11	0.4103 (12)	0.1150 (2)	1.0404 (2)	0.0319 (12)
C12	0.3708 (14)	0.1678 (3)	1.0451 (2)	0.0377 (14)
H12	0.253745	0.189448	1.025592	0.045*
C13	0.2815 (12)	0.0827 (2)	1.0026 (2)	0.0327 (12)
H13A	0.159081	0.106064	0.983125	0.039*
H13B	0.179564	0.055738	1.020827	0.039*
C14	0.6585 (13)	0.1074 (2)	0.9280 (2)	0.0345 (13)
H14A	0.656229	0.136364	0.952893	0.041*
H14B	0.842229	0.098524	0.917349	0.041*
C15	0.5080 (13)	0.1263 (2)	0.8818 (2)	0.0339 (13)
H15A	0.321144	0.132002	0.891803	0.041*
H15B	0.572131	0.160563	0.869426	0.041*
C16	0.5380 (12)	0.0867 (2)	0.8397 (2)	0.0287 (12)
S7	0.4163 (3)	0.31636 (6)	0.39894 (6)	0.0369 (3)
S8	0.3966 (3)	0.44961 (6)	0.54035 (6)	0.0371 (3)
S9	0.4242 (3)	0.46818 (6)	0.73313 (6)	0.0341 (3)
O7	0.8010 (9)	0.44429 (18)	0.64613 (18)	0.0431 (11)
O8	0.5113 (10)	0.51764 (18)	0.71430 (19)	0.0449 (11)
O9	0.1757 (9)	0.46694 (18)	0.75809 (19)	0.0422 (10)
N13	1.1090 (11)	0.4074 (2)	0.3014 (2)	0.0381 (12)
H13C	1.223589	0.430882	0.294803	0.05 (2)*
H13D	1.158861	0.378449	0.285605	0.037 (19)*
N14	0.9172 (11)	0.45838 (19)	0.3661 (2)	0.0338 (11)
H14C	0.783626	0.464411	0.386886	0.035 (18)*
H14D	1.036412	0.480213	0.357288	0.033 (17)*

N15	0.7754 (10)	0.37457 (19)	0.35051 (19)	0.0317 (11)
H15	0.818677	0.346608	0.332616	0.05 (2)*
N16	0.5088 (10)	0.41298 (19)	0.41761 (19)	0.0310 (10)
N17	0.4053 (11)	0.4280 (2)	0.6840 (2)	0.0366 (11)
H17	0.282489	0.407774	0.694736	0.037 (19)*
N18	0.6338 (10)	0.4433 (2)	0.7709 (2)	0.0402 (12)
H18A	0.784185	0.455562	0.762285	0.024 (15)*
H18B	0.571412	0.417253	0.788728	0.06 (2)*
C17	0.9358 (12)	0.4148 (2)	0.3400 (2)	0.0315 (12)
C18	0.5818 (12)	0.3739 (2)	0.3888 (2)	0.0335 (13)
C19	0.3060 (13)	0.3992 (2)	0.4509 (2)	0.0330 (13)
C20	0.2286 (14)	0.3493 (3)	0.4458 (3)	0.0391 (14)
H20	0.090517	0.334477	0.465272	0.047*
C21	0.1882 (13)	0.4391 (3)	0.4870 (2)	0.0386 (14)
H21A	0.018224	0.427191	0.500319	0.046*
H21B	0.155713	0.473110	0.468499	0.046*
C22	0.3428 (13)	0.3884 (2)	0.5766 (3)	0.0382 (14)
H22A	0.177747	0.392585	0.597143	0.046*
H22B	0.327535	0.358868	0.552843	0.046*
C23	0.5664 (14)	0.3754 (2)	0.6114 (3)	0.0391 (14)
H23A	0.728349	0.368789	0.590336	0.047*
H23B	0.532224	0.342398	0.630688	0.047*
C24	0.6077 (13)	0.4190 (2)	0.6486 (2)	0.0358 (13)
S10	0.1396 (3)	−0.09985 (6)	0.70887 (6)	0.0386 (3)
S11	0.1439 (3)	0.01797 (6)	0.55173 (6)	0.0378 (3)
S12	−0.1939 (3)	0.23587 (6)	0.52787 (6)	0.0373 (3)
O10	0.2135 (9)	0.15245 (16)	0.54147 (17)	0.0376 (10)
O11	−0.1027 (11)	0.2247 (2)	0.4768 (2)	0.0510 (12)
O12	−0.4437 (9)	0.26093 (18)	0.5370 (2)	0.0479 (12)
N19	0.8800 (11)	−0.2022 (2)	0.6230 (2)	0.0395 (12)
H19A	0.914557	−0.210966	0.654223	0.016 (14)*
H19B	0.968227	−0.217281	0.597773	0.034 (18)*
N20	0.6279 (12)	−0.1527 (2)	0.5681 (2)	0.0385 (12)
H20A	0.495944	−0.130911	0.561628	0.018 (14)*
H20B	0.725214	−0.166343	0.543309	0.04 (2)*
N21	0.5216 (11)	−0.1538 (2)	0.65473 (19)	0.0347 (11)
H21	0.590419	−0.167673	0.681916	0.031 (17)*
N22	0.2329 (11)	−0.0927 (2)	0.6118 (2)	0.0364 (11)
N23	−0.2046 (11)	0.1790 (2)	0.5601 (2)	0.0392 (12)
H23	−0.338006	0.181393	0.581512	0.028 (16)*
N24	0.0127 (11)	0.2713 (2)	0.5520 (2)	0.0414 (13)
H24A	0.170622	0.259958	0.543263	0.05 (2)*
H24B	−0.007597	0.282406	0.583112	0.04 (2)*
C25	0.6755 (12)	−0.1691 (2)	0.6145 (2)	0.0331 (13)
C26	0.3147 (14)	−0.1170 (2)	0.6532 (2)	0.0352 (13)
C27	0.0223 (14)	−0.0579 (2)	0.6238 (3)	0.0378 (14)
C28	−0.0555 (14)	−0.0574 (3)	0.6735 (3)	0.0426 (15)
H28	−0.199287	−0.036212	0.687018	0.051*

C29	−0.0940 (14)	−0.0252 (3)	0.5819 (3)	0.0404 (14)
H29A	−0.244802	−0.003183	0.595714	0.049*
H29B	−0.157854	−0.048753	0.556026	0.049*
C30	0.1930 (14)	0.0614 (2)	0.6042 (2)	0.0380 (14)
H30A	0.192321	0.040247	0.636197	0.046*
H30B	0.366633	0.076166	0.599457	0.046*
C31	−0.0106 (13)	0.1064 (2)	0.6091 (3)	0.0382 (14)
H31A	−0.185854	0.092136	0.609023	0.046*
H31B	0.006197	0.123964	0.642123	0.046*
C32	0.0143 (13)	0.1466 (2)	0.5669 (2)	0.0349 (13)
Cl1	0.4620 (4)	0.32940 (7)	0.81670 (7)	0.0535 (4)
Cl2	0.4460 (3)	0.19231 (7)	0.66722 (6)	0.0446 (4)
Cl3	0.7820 (3)	0.26816 (5)	1.29489 (5)	0.0339 (3)
Cl4	0.3990 (3)	0.17045 (6)	1.22937 (5)	0.0362 (3)
O13	0.9337 (10)	0.2657 (2)	0.6630 (2)	0.0503 (12)
H13E	1.069660	0.245370	0.666138	0.023 (15)*
H13F	0.788161	0.251540	0.667145	0.036 (19)*
O14	1.0308 (11)	0.18595 (19)	0.81895 (19)	0.0472 (11)
H14E	0.888809	0.195828	0.805298	0.07 (3)*
H14F	1.124519	0.207788	0.802856	0.08 (4)*
O15	0.0733 (12)	0.3526 (2)	0.7193 (2)	0.0572 (14)
H15C	0.043760	0.338576	0.748535	0.05 (2)*
H15D	0.040250	0.326096	0.701660	0.11 (5)*
O16	0.9201 (9)	0.21592 (16)	1.18011 (16)	0.0374 (9)
H16A	1.042612	0.207989	1.200195	0.056*
H16B	0.785322	0.208539	1.197998	0.056*
O17	0.2735 (9)	0.31094 (16)	1.24012 (17)	0.0367 (9)
H17A	0.132428	0.295871	1.246887	0.030 (17)*
H17B	0.400758	0.298641	1.257202	0.034 (18)*
O18	0.5394 (12)	0.21510 (19)	0.7822 (2)	0.0532 (13)
H18C	0.520046	0.247753	0.789368	0.014 (13)*
H18D	0.508126	0.215563	0.750732	0.05 (2)*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.0482 (9)	0.0326 (7)	0.0324 (7)	−0.0034 (6)	0.0016 (7)	−0.0015 (6)
S2	0.0525 (9)	0.0331 (7)	0.0329 (8)	−0.0039 (7)	0.0018 (7)	−0.0014 (6)
S3	0.0332 (7)	0.0287 (7)	0.0423 (8)	−0.0017 (6)	0.0021 (6)	−0.0012 (6)
O1	0.037 (2)	0.035 (2)	0.036 (2)	−0.0022 (18)	0.001 (2)	0.0007 (17)
O2	0.035 (2)	0.032 (2)	0.065 (3)	−0.0022 (18)	0.001 (2)	−0.001 (2)
O3	0.062 (3)	0.039 (2)	0.044 (3)	−0.011 (2)	0.010 (2)	−0.004 (2)
N1	0.046 (3)	0.033 (3)	0.033 (3)	0.004 (2)	−0.005 (2)	−0.006 (2)
N2	0.051 (3)	0.039 (3)	0.034 (3)	−0.004 (3)	0.001 (3)	−0.006 (2)
N3	0.043 (3)	0.029 (2)	0.034 (3)	−0.001 (2)	0.005 (2)	−0.004 (2)
N4	0.045 (3)	0.028 (2)	0.034 (3)	0.003 (2)	0.002 (2)	−0.006 (2)
N5	0.034 (3)	0.027 (2)	0.053 (3)	−0.001 (2)	−0.004 (3)	0.000 (2)
N6	0.033 (3)	0.036 (3)	0.042 (3)	0.000 (2)	−0.006 (2)	−0.001 (2)

C1	0.038 (3)	0.029 (3)	0.031 (3)	0.003 (2)	0.005 (3)	−0.008 (2)
C2	0.039 (3)	0.030 (3)	0.034 (3)	0.005 (3)	0.004 (3)	−0.011 (2)
C3	0.039 (3)	0.025 (3)	0.049 (4)	0.003 (2)	0.006 (3)	−0.009 (3)
C4	0.049 (4)	0.032 (3)	0.042 (4)	−0.003 (3)	0.007 (3)	−0.002 (3)
C5	0.046 (4)	0.030 (3)	0.044 (4)	0.008 (3)	0.004 (3)	−0.007 (3)
C6	0.039 (3)	0.027 (3)	0.032 (3)	0.004 (2)	0.005 (3)	−0.006 (2)
C7	0.040 (3)	0.034 (3)	0.045 (4)	−0.002 (3)	−0.003 (3)	0.002 (3)
C8	0.035 (3)	0.034 (3)	0.035 (3)	0.003 (3)	0.000 (3)	−0.005 (2)
S4	0.0533 (9)	0.0266 (7)	0.0359 (8)	0.0070 (6)	−0.0065 (7)	−0.0084 (6)
S5	0.0546 (9)	0.0266 (6)	0.0285 (7)	−0.0021 (6)	0.0006 (7)	−0.0044 (5)
S6	0.0302 (7)	0.0413 (8)	0.0297 (7)	−0.0004 (6)	0.0021 (6)	−0.0063 (6)
O4	0.033 (2)	0.036 (2)	0.031 (2)	0.0041 (18)	−0.0011 (18)	−0.0054 (17)
O5	0.058 (3)	0.035 (2)	0.042 (3)	−0.004 (2)	−0.006 (2)	−0.0097 (19)
O6	0.031 (2)	0.068 (3)	0.034 (2)	−0.003 (2)	0.0033 (19)	−0.011 (2)
N7	0.041 (3)	0.029 (2)	0.032 (3)	0.000 (2)	−0.001 (2)	−0.007 (2)
N8	0.043 (3)	0.029 (2)	0.031 (3)	0.006 (2)	0.000 (2)	−0.005 (2)
N9	0.043 (3)	0.022 (2)	0.032 (3)	0.002 (2)	−0.003 (2)	−0.0075 (19)
N10	0.039 (3)	0.031 (2)	0.026 (2)	0.000 (2)	0.006 (2)	−0.0046 (19)
N11	0.030 (2)	0.035 (3)	0.034 (3)	0.006 (2)	0.007 (2)	−0.007 (2)
N12	0.029 (3)	0.053 (3)	0.034 (3)	0.003 (2)	0.001 (2)	−0.004 (2)
C9	0.037 (3)	0.028 (3)	0.026 (3)	0.001 (2)	0.008 (3)	−0.005 (2)
C10	0.036 (3)	0.024 (3)	0.029 (3)	0.003 (2)	0.008 (3)	−0.004 (2)
C11	0.034 (3)	0.032 (3)	0.030 (3)	0.004 (2)	0.005 (3)	−0.002 (2)
C12	0.045 (4)	0.039 (3)	0.028 (3)	0.004 (3)	−0.005 (3)	−0.008 (2)
C13	0.038 (3)	0.029 (3)	0.030 (3)	−0.005 (2)	0.001 (3)	0.000 (2)
C14	0.045 (3)	0.030 (3)	0.028 (3)	−0.005 (3)	0.005 (3)	−0.006 (2)
C15	0.041 (3)	0.027 (3)	0.034 (3)	0.000 (2)	0.004 (3)	−0.006 (2)
C16	0.032 (3)	0.023 (2)	0.029 (3)	0.003 (2)	0.005 (2)	0.001 (2)
S7	0.0473 (8)	0.0275 (6)	0.0360 (8)	−0.0050 (6)	0.0055 (7)	−0.0058 (6)
S8	0.0489 (9)	0.0278 (6)	0.0344 (8)	−0.0030 (6)	0.0033 (7)	−0.0073 (6)
S9	0.0334 (7)	0.0344 (7)	0.0346 (7)	−0.0028 (6)	0.0009 (6)	−0.0082 (6)
O7	0.043 (3)	0.040 (2)	0.046 (3)	−0.003 (2)	0.006 (2)	−0.006 (2)
O8	0.049 (3)	0.038 (2)	0.049 (3)	−0.005 (2)	0.000 (2)	−0.008 (2)
O9	0.036 (2)	0.044 (2)	0.048 (3)	−0.001 (2)	−0.003 (2)	−0.014 (2)
N13	0.040 (3)	0.035 (3)	0.040 (3)	−0.006 (2)	0.005 (3)	−0.008 (2)
N14	0.037 (3)	0.028 (2)	0.036 (3)	−0.003 (2)	0.003 (2)	−0.004 (2)
N15	0.039 (3)	0.026 (2)	0.029 (2)	−0.001 (2)	0.006 (2)	−0.0074 (19)
N16	0.034 (3)	0.029 (2)	0.030 (2)	−0.002 (2)	0.000 (2)	−0.0071 (19)
N17	0.033 (3)	0.035 (3)	0.041 (3)	−0.003 (2)	0.004 (2)	−0.009 (2)
N18	0.030 (3)	0.051 (3)	0.040 (3)	−0.007 (2)	0.005 (2)	−0.003 (2)
C17	0.031 (3)	0.028 (3)	0.035 (3)	0.000 (2)	−0.003 (3)	−0.004 (2)
C18	0.037 (3)	0.033 (3)	0.031 (3)	−0.002 (2)	−0.006 (3)	−0.001 (2)
C19	0.038 (3)	0.031 (3)	0.029 (3)	0.006 (2)	−0.002 (3)	−0.002 (2)
C20	0.043 (3)	0.038 (3)	0.037 (3)	−0.004 (3)	0.001 (3)	−0.004 (3)
C21	0.040 (3)	0.038 (3)	0.037 (3)	−0.003 (3)	0.006 (3)	−0.006 (3)
C22	0.044 (3)	0.033 (3)	0.038 (3)	−0.003 (3)	0.007 (3)	−0.003 (3)
C23	0.046 (4)	0.033 (3)	0.038 (3)	0.001 (3)	0.004 (3)	−0.005 (3)
C24	0.038 (3)	0.033 (3)	0.036 (3)	0.005 (3)	−0.003 (3)	−0.005 (2)

S10	0.0466 (9)	0.0376 (7)	0.0310 (7)	−0.0023 (7)	0.0068 (7)	−0.0033 (6)
S11	0.0493 (9)	0.0321 (7)	0.0317 (7)	−0.0018 (6)	0.0015 (7)	−0.0027 (6)
S12	0.0336 (7)	0.0319 (7)	0.0458 (9)	0.0012 (6)	0.0030 (7)	−0.0018 (6)
O10	0.039 (2)	0.033 (2)	0.040 (2)	0.0021 (18)	0.008 (2)	−0.0002 (18)
O11	0.059 (3)	0.048 (3)	0.044 (3)	0.009 (2)	−0.001 (2)	0.000 (2)
O12	0.038 (2)	0.038 (2)	0.066 (3)	0.000 (2)	0.006 (2)	0.006 (2)
N19	0.046 (3)	0.036 (3)	0.036 (3)	−0.004 (2)	0.001 (3)	−0.001 (2)
N20	0.045 (3)	0.038 (3)	0.031 (3)	0.004 (2)	0.003 (2)	−0.004 (2)
N21	0.043 (3)	0.033 (3)	0.028 (3)	−0.007 (2)	−0.001 (2)	−0.002 (2)
N22	0.045 (3)	0.033 (2)	0.031 (3)	−0.006 (2)	0.005 (2)	−0.003 (2)
N23	0.033 (3)	0.033 (3)	0.051 (3)	0.003 (2)	0.014 (3)	0.001 (2)
N24	0.037 (3)	0.030 (3)	0.056 (4)	0.001 (2)	0.010 (3)	−0.003 (2)
C25	0.039 (3)	0.021 (2)	0.040 (3)	−0.004 (2)	−0.002 (3)	−0.003 (2)
C26	0.049 (4)	0.027 (3)	0.030 (3)	−0.012 (3)	0.004 (3)	−0.006 (2)
C27	0.045 (4)	0.032 (3)	0.037 (3)	−0.007 (3)	0.007 (3)	−0.006 (2)
C28	0.045 (4)	0.036 (3)	0.047 (4)	−0.003 (3)	0.003 (3)	−0.008 (3)
C29	0.042 (3)	0.034 (3)	0.045 (4)	−0.003 (3)	0.001 (3)	−0.003 (3)
C30	0.049 (4)	0.033 (3)	0.033 (3)	−0.003 (3)	−0.005 (3)	−0.007 (2)
C31	0.041 (3)	0.035 (3)	0.038 (3)	−0.002 (3)	0.007 (3)	−0.007 (3)
C32	0.038 (3)	0.031 (3)	0.035 (3)	−0.005 (3)	0.006 (3)	−0.006 (2)
Cl1	0.0683 (11)	0.0386 (8)	0.0532 (10)	0.0060 (8)	−0.0076 (9)	−0.0103 (7)
Cl2	0.0440 (8)	0.0472 (8)	0.0421 (8)	−0.0002 (7)	0.0032 (7)	−0.0074 (7)
Cl3	0.0392 (7)	0.0283 (6)	0.0340 (7)	0.0003 (5)	0.0019 (6)	−0.0065 (5)
Cl4	0.0405 (7)	0.0391 (7)	0.0292 (7)	−0.0049 (6)	0.0018 (6)	−0.0041 (5)
O13	0.043 (3)	0.051 (3)	0.057 (3)	0.002 (2)	−0.001 (2)	−0.006 (2)
O14	0.054 (3)	0.042 (2)	0.044 (3)	0.002 (2)	0.005 (2)	−0.001 (2)
O15	0.065 (3)	0.042 (3)	0.065 (4)	−0.011 (3)	0.002 (3)	0.001 (3)
O16	0.044 (2)	0.035 (2)	0.032 (2)	0.0005 (19)	0.0006 (19)	−0.0076 (17)
O17	0.038 (2)	0.032 (2)	0.040 (2)	−0.0014 (18)	−0.001 (2)	−0.0025 (18)
O18	0.080 (4)	0.036 (2)	0.043 (3)	0.003 (2)	−0.002 (3)	−0.005 (2)

Geometric parameters (Å, º)

S1—C4	1.732 (7)	S9—N18	1.593 (6)
S1—C2	1.744 (6)	S9—N17	1.668 (5)
S2—C6	1.809 (6)	O7—C24	1.211 (9)
S2—C5	1.817 (7)	N13—C17	1.333 (9)
S3—O2	1.425 (5)	N13—H13C	0.8700
S3—O3	1.431 (6)	N13—H13D	0.8700
S3—N6	1.571 (6)	N14—C17	1.306 (8)
S3—N5	1.651 (5)	N14—H14C	0.8699
O1—C8	1.212 (8)	N14—H14D	0.8700
N1—C1	1.336 (9)	N15—C17	1.363 (8)
N1—H1A	0.8700	N15—C18	1.389 (9)
N1—H1B	0.8700	N15—H15	0.8699
N2—C1	1.314 (9)	N16—C18	1.287 (8)
N2—H2A	0.8700	N16—C19	1.392 (9)
N2—H2B	0.8700	N17—C24	1.380 (9)

N3—C1	1.360 (9)	N17—H17	0.8701
N3—C2	1.391 (9)	N18—H18A	0.8699
N3—H3	0.8701	N18—H18B	0.8702
N4—C2	1.279 (9)	C19—C20	1.353 (9)
N4—C3	1.394 (9)	C19—C21	1.485 (9)
N5—C8	1.385 (9)	C20—H20	0.9500
N5—H5	0.8700	C21—H21A	0.9900
N6—H6A	0.8700	C21—H21B	0.9900
N6—H6B	0.8703	C22—C23	1.516 (10)
C3—C4	1.352 (10)	C22—H22A	0.9900
C3—C5	1.498 (10)	C22—H22B	0.9900
C4—H4	0.9500	C23—C24	1.516 (9)
C5—H5A	0.9900	C23—H23A	0.9900
C5—H5B	0.9900	C23—H23B	0.9900
C6—C7	1.506 (9)	S10—C28	1.717 (8)
C6—H6C	0.9900	S10—C26	1.737 (6)
C6—H6D	0.9900	S11—C30	1.811 (6)
C7—C8	1.500 (9)	S11—C29	1.833 (7)
C7—H7A	0.9900	S12—O12	1.415 (5)
C7—H7B	0.9900	S12—O11	1.433 (6)
S4—C12	1.728 (7)	S12—N24	1.586 (6)
S4—C10	1.742 (6)	S12—N23	1.659 (5)
S5—C14	1.818 (6)	O10—C32	1.216 (8)
S5—C13	1.838 (6)	N19—C25	1.331 (9)
S6—O5	1.417 (5)	N19—H19A	0.8702
S6—O6	1.421 (5)	N19—H19B	0.8699
S6—N12	1.595 (6)	N20—C25	1.312 (9)
S6—N11	1.672 (5)	N20—H20A	0.8698
O4—C16	1.210 (7)	N20—H20B	0.8702
N7—C9	1.317 (9)	N21—C25	1.345 (9)
N7—H7C	0.8700	N21—C26	1.376 (9)
N7—H7D	0.8701	N21—H21	0.8699
N8—C9	1.321 (8)	N22—C26	1.314 (9)
N8—H8A	0.8702	N22—C27	1.386 (9)
N8—H8B	0.8699	N23—C32	1.371 (9)
N9—C9	1.355 (8)	N23—H23	0.8701
N9—C10	1.383 (8)	N24—H24A	0.8701
N9—H9	0.8702	N24—H24B	0.8702
N10—C10	1.295 (8)	C27—C28	1.351 (10)
N10—C11	1.384 (8)	C27—C29	1.492 (10)
N11—C16	1.372 (8)	C28—H28	0.9500
N11—H11	0.8700	C29—H29A	0.9900
N12—H12A	0.8700	C29—H29B	0.9900
N12—H12B	0.8699	C30—C31	1.509 (9)
C11—C12	1.348 (9)	C30—H30A	0.9900
C11—C13	1.494 (9)	C30—H30B	0.9900
C12—H12	0.9500	C31—C32	1.499 (9)
C13—H13A	0.9900	C31—H31A	0.9900

C13—H13B	0.9900	C31—H31B	0.9900
C14—C15	1.522 (9)	O13—H13E	0.8500
C14—H14A	0.9900	O13—H13F	0.8499
C14—H14B	0.9900	O14—H14E	0.8498
C15—C16	1.505 (8)	O14—H14F	0.8500
C15—H15A	0.9900	O15—H15C	0.8518
C15—H15B	0.9900	O15—H15D	0.8501
S7—C20	1.732 (7)	O16—H16A	0.8515
S7—C18	1.737 (6)	O16—H16B	0.8504
S8—C21	1.832 (7)	O17—H17A	0.8499
S8—C22	1.833 (6)	O17—H17B	0.8500
S9—O9	1.416 (5)	O18—H18C	0.8498
S9—O8	1.426 (5)	O18—H18D	0.8481

C4—S1—C2	88.0 (3)	O9—S9—N18	107.9 (3)
C6—S2—C5	99.4 (3)	O8—S9—N18	108.3 (3)
O2—S3—O3	119.0 (3)	O9—S9—N17	104.0 (3)
O2—S3—N6	107.1 (3)	O8—S9—N17	108.3 (3)
O3—S3—N6	108.4 (3)	N18—S9—N17	109.1 (3)
O2—S3—N5	104.7 (3)	C17—N13—H13C	119.8
O3—S3—N5	107.7 (3)	C17—N13—H13D	129.5
N6—S3—N5	109.8 (3)	H13C—N13—H13D	108.5
C1—N1—H1A	118.9	C17—N14—H14C	119.4
C1—N1—H1B	118.1	C17—N14—H14D	113.1
H1A—N1—H1B	122.8	H14C—N14—H14D	127.0
C1—N2—H2A	123.2	C17—N15—C18	126.1 (5)
C1—N2—H2B	127.7	C17—N15—H15	112.2
H2A—N2—H2B	109.0	C18—N15—H15	121.4
C1—N3—C2	124.6 (5)	C18—N16—C19	110.5 (5)
C1—N3—H3	120.6	C24—N17—S9	122.8 (5)
C2—N3—H3	114.7	C24—N17—H17	130.8
C2—N4—C3	110.8 (6)	S9—N17—H17	101.5
C8—N5—S3	121.5 (5)	S9—N18—H18A	108.2
C8—N5—H5	109.1	S9—N18—H18B	111.0
S3—N5—H5	128.7	H18A—N18—H18B	139.0
S3—N6—H6A	114.6	N14—C17—N13	122.1 (6)
S3—N6—H6B	116.6	N14—C17—N15	121.4 (6)
H6A—N6—H6B	101.9	N13—C17—N15	116.5 (5)
N2—C1—N1	121.7 (6)	N16—C18—N15	125.5 (6)
N2—C1—N3	120.4 (6)	N16—C18—S7	115.9 (5)
N1—C1—N3	117.9 (6)	N15—C18—S7	118.6 (4)
N4—C2—N3	125.6 (6)	C20—C19—N16	114.9 (6)
N4—C2—S1	115.7 (5)	C20—C19—C21	125.7 (6)
N3—C2—S1	118.7 (5)	N16—C19—C21	119.4 (5)
C4—C3—N4	114.8 (6)	C19—C20—S7	110.6 (5)
C4—C3—C5	126.9 (6)	C19—C20—H20	124.7
N4—C3—C5	118.3 (6)	S7—C20—H20	124.7
C3—C4—S1	110.6 (5)	C19—C21—S8	112.4 (5)

C3—C4—H4	124.7	C19—C21—H21A	109.1
S1—C4—H4	124.7	S8—C21—H21A	109.1
C3—C5—S2	112.4 (5)	C19—C21—H21B	109.1
C3—C5—H5A	109.1	S8—C21—H21B	109.1
S2—C5—H5A	109.1	H21A—C21—H21B	107.9
C3—C5—H5B	109.1	C23—C22—S8	110.5 (5)
S2—C5—H5B	109.1	C23—C22—H22A	109.6
H5A—C5—H5B	107.8	S8—C22—H22A	109.6
C7—C6—S2	114.9 (5)	C23—C22—H22B	109.6
C7—C6—H6C	108.5	S8—C22—H22B	109.6
S2—C6—H6C	108.5	H22A—C22—H22B	108.1
C7—C6—H6D	108.5	C22—C23—C24	113.3 (5)
S2—C6—H6D	108.5	C22—C23—H23A	108.9
H6C—C6—H6D	107.5	C24—C23—H23A	108.9
C8—C7—C6	112.7 (6)	C22—C23—H23B	108.9
C8—C7—H7A	109.0	C24—C23—H23B	108.9
C6—C7—H7A	109.0	H23A—C23—H23B	107.7
C8—C7—H7B	109.0	O7—C24—N17	123.7 (6)
C6—C7—H7B	109.0	O7—C24—C23	121.8 (6)
H7A—C7—H7B	107.8	N17—C24—C23	114.5 (6)
O1—C8—N5	121.5 (6)	C28—S10—C26	88.5 (3)
O1—C8—C7	123.7 (6)	C30—S11—C29	99.9 (3)
N5—C8—C7	114.8 (6)	O12—S12—O11	119.9 (3)
C12—S4—C10	88.3 (3)	O12—S12—N24	107.8 (3)
C14—S5—C13	99.6 (3)	O11—S12—N24	107.1 (3)
O5—S6—O6	121.1 (3)	O12—S12—N23	104.2 (3)
O5—S6—N12	108.2 (3)	O11—S12—N23	108.4 (3)
O6—S6—N12	106.8 (3)	N24—S12—N23	109.2 (3)
O5—S6—N11	108.0 (3)	C25—N19—H19A	118.7
O6—S6—N11	103.1 (3)	C25—N19—H19B	120.7
N12—S6—N11	109.1 (3)	H19A—N19—H19B	120.3
C9—N7—H7C	127.0	C25—N20—H20A	121.7
C9—N7—H7D	121.4	C25—N20—H20B	117.7
H7C—N7—H7D	111.2	H20A—N20—H20B	120.4
C9—N8—H8A	110.7	C25—N21—C26	125.3 (5)
C9—N8—H8B	106.8	C25—N21—H21	107.6
H8A—N8—H8B	141.3	C26—N21—H21	126.4
C9—N9—C10	125.3 (5)	C26—N22—C27	109.9 (5)
C9—N9—H9	119.5	C32—N23—S12	121.8 (4)
C10—N9—H9	114.9	C32—N23—H23	124.4
C10—N10—C11	110.4 (5)	S12—N23—H23	108.1
C16—N11—S6	121.9 (4)	S12—N24—H24A	110.7
C16—N11—H11	123.8	S12—N24—H24B	121.4
S6—N11—H11	114.3	H24A—N24—H24B	114.8
S6—N12—H12A	112.5	N20—C25—N19	120.1 (6)
S6—N12—H12B	111.0	N20—C25—N21	122.0 (6)
H12A—N12—H12B	121.9	N19—C25—N21	118.0 (6)
N7—C9—N8	121.3 (6)	N22—C26—N21	124.8 (6)

N7—C9—N9	118.0 (5)	N22—C26—S10	115.1 (5)
N8—C9—N9	120.7 (6)	N21—C26—S10	120.0 (5)
N10—C10—N9	126.2 (5)	C28—C27—N22	115.2 (6)
N10—C10—S4	115.3 (5)	C28—C27—C29	126.6 (6)
N9—C10—S4	118.6 (4)	N22—C27—C29	118.1 (6)
C12—C11—N10	115.6 (6)	C27—C28—S10	111.1 (5)
C12—C11—C13	124.3 (6)	C27—C28—H28	124.4
N10—C11—C13	120.1 (5)	S10—C28—H28	124.4
C11—C12—S4	110.5 (5)	C27—C29—S11	111.5 (5)
C11—C12—H12	124.8	C27—C29—H29A	109.3
S4—C12—H12	124.8	S11—C29—H29A	109.3
C11—C13—S5	111.7 (4)	C27—C29—H29B	109.3
C11—C13—H13A	109.3	S11—C29—H29B	109.3
S5—C13—H13A	109.3	H29A—C29—H29B	108.0
C11—C13—H13B	109.3	C31—C30—S11	113.6 (5)
S5—C13—H13B	109.3	C31—C30—H30A	108.9
H13A—C13—H13B	107.9	S11—C30—H30A	108.9
C15—C14—S5	111.7 (4)	C31—C30—H30B	108.9
C15—C14—H14A	109.3	S11—C30—H30B	108.9
S5—C14—H14A	109.3	H30A—C30—H30B	107.7
C15—C14—H14B	109.3	C32—C31—C30	112.2 (5)
S5—C14—H14B	109.3	C32—C31—H31A	109.2
H14A—C14—H14B	107.9	C30—C31—H31A	109.2
C16—C15—C14	111.4 (5)	C32—C31—H31B	109.2
C16—C15—H15A	109.4	C30—C31—H31B	109.2
C14—C15—H15A	109.4	H31A—C31—H31B	107.9
C16—C15—H15B	109.4	O10—C32—N23	121.2 (6)
C14—C15—H15B	109.4	O10—C32—C31	124.0 (6)
H15A—C15—H15B	108.0	N23—C32—C31	114.6 (6)
O4—C16—N11	122.0 (5)	H13E—O13—H13F	116.7
O4—C16—C15	123.5 (6)	H14E—O14—H14F	96.2
N11—C16—C15	114.5 (5)	H15C—O15—H15D	97.5
C20—S7—C18	88.1 (3)	H16A—O16—H16B	102.4
C21—S8—C22	99.0 (3)	H17A—O17—H17B	113.7
O9—S9—O8	118.9 (3)	H18C—O18—H18D	101.9

O2—S3—N5—C8	−177.9 (5)	O9—S9—N17—C24	178.3 (5)
O3—S3—N5—C8	54.5 (6)	O8—S9—N17—C24	50.9 (6)
N6—S3—N5—C8	−63.3 (6)	N18—S9—N17—C24	−66.7 (6)
C2—N3—C1—N2	−3.6 (9)	C18—N15—C17—N14	−0.8 (10)
C2—N3—C1—N1	176.2 (6)	C18—N15—C17—N13	−179.9 (6)
C3—N4—C2—N3	178.9 (6)	C19—N16—C18—N15	178.4 (6)
C3—N4—C2—S1	−0.8 (7)	C19—N16—C18—S7	−0.5 (7)
C1—N3—C2—N4	3.7 (10)	C17—N15—C18—N16	6.2 (10)
C1—N3—C2—S1	−176.6 (5)	C17—N15—C18—S7	−174.9 (5)
C4—S1—C2—N4	0.5 (5)	C20—S7—C18—N16	1.0 (5)
C4—S1—C2—N3	−179.2 (5)	C20—S7—C18—N15	−177.9 (5)
C2—N4—C3—C4	0.8 (8)	C18—N16—C19—C20	−0.6 (8)

C2—N4—C3—C5	178.1 (5)	C18—N16—C19—C21	−178.7 (6)
N4—C3—C4—S1	−0.4 (7)	N16—C19—C20—S7	1.4 (7)
C5—C3—C4—S1	−177.4 (5)	C21—C19—C20—S7	179.3 (5)
C2—S1—C4—C3	0.0 (5)	C18—S7—C20—C19	−1.3 (5)
C4—C3—C5—S2	99.2 (8)	C20—C19—C21—S8	106.8 (7)
N4—C3—C5—S2	−77.7 (6)	N16—C19—C21—S8	−75.3 (6)
C6—S2—C5—C3	−71.3 (5)	C22—S8—C21—C19	−74.7 (5)
C5—S2—C6—C7	−85.3 (5)	C21—S8—C22—C23	157.5 (5)
S2—C6—C7—C8	−74.0 (6)	S8—C22—C23—C24	58.2 (7)
S3—N5—C8—O1	−7.0 (9)	S9—N17—C24—O7	−10.1 (9)
S3—N5—C8—C7	173.5 (5)	S9—N17—C24—C23	171.7 (5)
C6—C7—C8—O1	3.7 (9)	C22—C23—C24—O7	−112.8 (7)
C6—C7—C8—N5	−176.9 (5)	C22—C23—C24—N17	65.3 (7)
O5—S6—N11—C16	52.0 (5)	O12—S12—N23—C32	−170.4 (5)
O6—S6—N11—C16	−178.7 (5)	O11—S12—N23—C32	60.9 (6)
N12—S6—N11—C16	−65.5 (5)	N24—S12—N23—C32	−55.5 (6)
C10—N9—C9—N7	176.6 (6)	C26—N21—C25—N20	−5.6 (9)
C10—N9—C9—N8	−3.1 (9)	C26—N21—C25—N19	174.9 (6)
C11—N10—C10—N9	−178.1 (6)	C27—N22—C26—N21	−179.6 (6)
C11—N10—C10—S4	1.2 (6)	C27—N22—C26—S10	0.8 (7)
C9—N9—C10—N10	3.2 (10)	C25—N21—C26—N22	2.4 (9)
C9—N9—C10—S4	−176.1 (5)	C25—N21—C26—S10	−178.1 (5)
C12—S4—C10—N10	−0.8 (5)	C28—S10—C26—N22	0.0 (5)
C12—S4—C10—N9	178.6 (5)	C28—S10—C26—N21	−179.5 (5)
C10—N10—C11—C12	−1.1 (8)	C26—N22—C27—C28	−1.5 (8)
C10—N10—C11—C13	177.4 (5)	C26—N22—C27—C29	178.1 (5)
N10—C11—C12—S4	0.5 (7)	N22—C27—C28—S10	1.6 (7)
C13—C11—C12—S4	−178.0 (5)	C29—C27—C28—S10	−178.0 (5)
C10—S4—C12—C11	0.1 (5)	C26—S10—C28—C27	−0.9 (5)
C12—C11—C13—S5	118.5 (6)	C28—C27—C29—S11	118.9 (7)
N10—C11—C13—S5	−59.9 (6)	N22—C27—C29—S11	−60.7 (7)
C14—S5—C13—C11	−62.6 (5)	C30—S11—C29—C27	−66.1 (5)
C13—S5—C14—C15	−88.8 (5)	C29—S11—C30—C31	−83.2 (5)
S5—C14—C15—C16	−67.2 (6)	S11—C30—C31—C32	−70.4 (7)
S6—N11—C16—O4	−4.8 (8)	S12—N23—C32—O10	−14.9 (9)
S6—N11—C16—C15	172.5 (4)	S12—N23—C32—C31	162.1 (5)
C14—C15—C16—O4	−28.8 (8)	C30—C31—C32—O10	−22.0 (9)
C14—C15—C16—N11	153.9 (5)	C30—C31—C32—N23	161.0 (5)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1*A*···O16^{i}	0.87	2.02	2.821 (7)	152
N1—H1*B*···O3^{ii}	0.87	2.08	2.913 (8)	160
N2—H2*A*···N4	0.87	2.05	2.678 (8)	128
N2—H2*B*···O1^{ii}	0.87	2.06	2.871 (7)	155
N2—H2*B*···O3^{ii}	0.87	2.61	3.240 (8)	130
N3—H3···O17^{ii}	0.87	1.96	2.781 (7)	156

N5—H5···Cl1^{iii}	0.87	2.61	3.262 (6)	133
N6—H6*A*···O2^{iv}	0.87	1.98	2.810 (7)	159
N6—H6*B*···O14^{iii}	0.87	2.17	3.007 (8)	162
N7—H7*C*···O4^{v}	0.87	2.48	3.138 (6)	133
N7—H7*C*···O5^{v}	0.87	2.18	2.966 (7)	150
N7—H7*D*···Cl4^{iv}	0.87	2.39	3.245 (5)	168
N8—H8*A*···O4^{v}	0.87	2.05	2.904 (7)	167
N8—H8*B*···N10	0.87	1.93	2.709 (8)	149
N9—H9···O16	0.87	1.98	2.817 (6)	161
N11—H11···O14^{iii}	0.87	2.10	2.862 (7)	146
N12—H12*A*···O6^{iv}	0.87	1.97	2.798 (7)	158
N12—H12*B*···Cl2	0.87	2.41	3.278 (6)	174
N13—H13*C*···O8^{vi}	0.87	1.94	2.811 (7)	175
N13—H13*D*···O17^{vii}	0.87	2.14	3.011 (7)	175
N14—H14*C*···S8^{viii}	0.87	3.00	3.680 (5)	136
N14—H14*C*···N16	0.87	2.10	2.750 (7)	131
N14—H14*D*···O7^{vi}	0.87	2.13	2.932 (7)	154
N15—H15···Cl3^{ix}	0.87	2.25	3.083 (5)	159
N17—H17···O15	0.87	1.90	2.756 (8)	168
N18—H18*A*···O9^{iv}	0.87	2.05	2.890 (7)	162
N18—H18*B*···Cl1	0.87	2.42	3.257 (6)	162
N19—H19*A*···Cl3^{v}	0.87	2.48	3.213 (6)	142
N19—H19*B*···O11^{x}	0.87	2.06	2.876 (8)	155
N20—H20*A*···N22	0.87	2.07	2.700 (8)	129
N20—H20*B*···O10^{x}	0.87	2.26	2.964 (7)	137
N20—H20*B*···O11^{x}	0.87	2.42	3.167 (8)	144
N21—H21···Cl4^{xi}	0.87	2.34	3.110 (5)	148
N23—H23···Cl2^{iii}	0.87	2.49	3.294 (6)	154
N24—H24*A*···O12^{iv}	0.87	1.99	2.807 (8)	157
N24—H24*B*···O13^{iii}	0.87	2.15	2.933 (8)	150
O13—H13*E*···Cl2^{iv}	0.85	2.29	3.142 (6)	175
O13—H13*F*···Cl2	0.85	2.37	3.200 (6)	164
O14—H14*E*···O18	0.85	1.95	2.798 (8)	172
O14—H14*F*···O18^{iv}	0.85	2.20	2.884 (8)	138
O15—H15*C*···Cl1	0.85	2.85	3.335 (6)	118
O15—H15*D*···O13^{iii}	0.85	1.97	2.813 (8)	175
O16—H16*A*···Cl4^{iv}	0.85	2.18	2.992 (5)	160
O16—H16*B*···Cl4	0.85	2.37	3.195 (5)	164
O17—H17*A*···Cl3^{iii}	0.85	2.30	3.100 (5)	158
O17—H17*B*···Cl3	0.85	2.32	3.165 (5)	173
O18—H18*C*···Cl1	0.85	2.20	3.045 (5)	174
O18—H18*D*···Cl2	0.85	2.32	3.148 (5)	165

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