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Diethyl 2-aminothiazole-4,5-dicarboxylate hemihydrate

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Key indicators

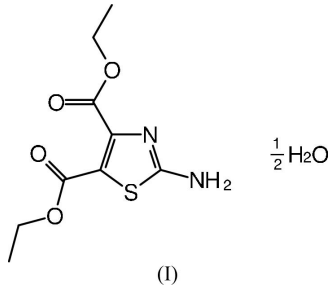
Single-crystal X-ray study
T = 120 K
Mean σ(C–C) = 0.003 Å
R factor = 0.046
wR factor = 0.122
Data-to-parameter ratio = 17.6

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

The structure of the title compound, $C_9H_{12}N_2O_4S\cdot0.5H_2O$, comprises two thiazole molecules and one water molecule in the asymmetric unit. The two thiazoles associate *via* N—H···N hydrogen-bonding interactions to form $R_2^2(8)$ graph-set dimers, while the other NH groups and water molecules construct a hydrogen-bonding network that involves three of the four ethyl ester carbonyl O atoms.

Comment

The title compound (I) was prepared as part of an ongoing investigation into the synthesis and structural properties of 2-aminothiazole derivatives.



(I)

As a part of that study, we have been examining the hydrogen-bonding networks of 2-aminothiazoles with ethyl ester substituents at the 5-position. The addition of two hydrogen-bond acceptors to the existing hydrogen-bond acceptors in 2-aminothiazole has led to some interesting

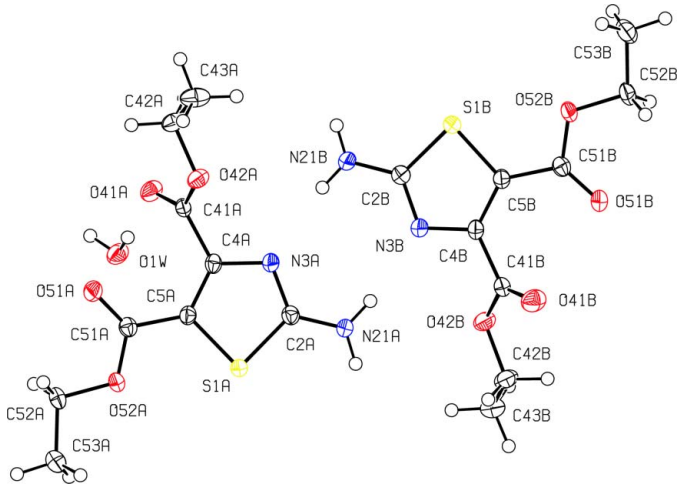


Figure 1
The structure of the asymmetric unit of (I). Displacement ellipsoids are drawn at the 50% probability level and H atoms are drawn as spheres of arbitrary radii.

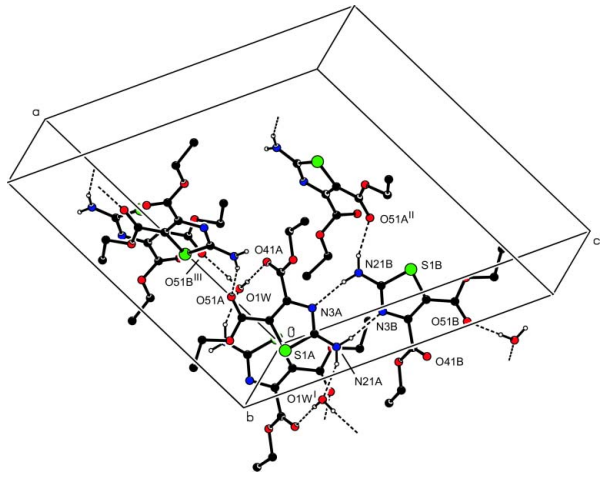


Figure 2
Partial packing diagram for (I). Hydrogen-bonding interactions are shown as dotted lines. H atoms not involved in the hydrogen-bonding network have been omitted for clarity. [Symmetry codes: (i) $-x, -y, -z$; (ii) $-x+\frac{1}{2}, y-\frac{1}{2}, -z+\frac{1}{2}$; (iii) $x+\frac{1}{2}, -y-\frac{1}{2}, z-\frac{1}{2}$.]

packing arrangements. For the three previous structures that contain a primary 2-amino group, *viz.* ethyl 2-amino-4-phenyl-1,3-thiazole-5-carboxylate (Lynch & McClenaghan, 2000), ethyl 2-amino-4-*tert*-butyl-1,3-thiazole-5-carboxylate (Lynch & McClenaghan, 2004) and ethyl 2-amino-4-isopropyl-1,3-thiazole-5-carboxylate (Kennedy *et al.*, 2004), all molecules pack with one NH group interacting with an adjacent thiazole N atom, to form an $R_2^2(8)$ graph-set (Etter, 1990) dimer, while the other NH group associates with the ethyl ester carbonyl O atom. In the other four known thiazoles with ethyl ester substituents at the 5-position and substituted 2-amino N atoms, two have tertiary amino groups, *viz.* ethyl 2-(5-ethoxycarbonyl-2-(pyrrol-1yl)thiazol-4yl-2-iminoethanoate (Brickute *et al.*, 2002) and ethyl 4-phenyl-2-(pyrrolyl)-1,3-thiazole-5-carboxylate (Lynch & McClenaghan, 2002*b*), and the other two both contain competing carbonyl O atoms with which the single NH groups associate, *viz.* ethyl 2-[*N*-(*t*-butoxycarbonyl)-L-alanylamino]-4-methyl-1,3-thiazole-5-carboxylate (Singh *et al.*, 2000) and ethyl 4-*tert*-butyl-2-(3-phenylureido)-1,3-thiazole-5-carboxylate (Lynch & McClenaghan, 2002*a*). All five structures containing at least one NH group show that an amino group prefers to interact with a carbonyl O atom, whether from the ester group or from another group in the molecule. With this in mind we decided to examine a 2-aminothiazole analogue with two ethyl ester substituents to see which substituted position would be preferred by the NH groups in the hydrogen-bonding network.

The asymmetric unit of (I) comprises two thiazole molecules; these are connected in a three-dimensional hydrogen-bonding network. The addition of the second ethyl ester and thus the increase in hydrogen-bond acceptor atoms leads to the incorporation of a water molecule to satisfy the hydrogen-bonding requirements of the packing network (Desiraju, 1991). Similarly to the three previous structures where the 2-amino group was unsubstituted, the two thiazole molecules in (I) associate *via* N—H···N interactions and form $R_2^2(8)$ graph-set dimers (Fig. 1). The hydrogen-bonding geometry is listed in Table 1. The second NH group on each thiazole molecule individually associates with different hydrogen-bond acceptor sites; on molecule *A*, the second NH group hydrogen-bonds to the water O atom, while the second NH group on molecule *B* hydrogen-bonds to the 5-substituted ester carbonyl O atom, on an adjacent molecule A. In turn, the water H atoms are donors to the 4-substituted ester carbonyl O atom on an adjacent molecule *A* and the equivalent 5-substituted O atom to molecule *B*; thus three of the four ester carbonyl O atoms are involved in the hydrogen-bonding network (Fig. 2). Separation distances of O41*B* (the remaining carbonyl O atom) of 3.208 (2) Å to C52*A*($-\frac{1}{2}+x, \frac{1}{2}-y, \frac{1}{2}+z$) and 3.365 (2) Å to C43*B*($-\frac{1}{2}-x, \frac{1}{2}+y, \frac{1}{2}-z$) indicate C—H···O interactions.

Experimental

The title compound was obtained from Key Organics Ltd and crystals were grown from an ethanol solution.

Crystal data

$C_9H_{12}N_2O_4S\cdot0.5H_2O$
$M_r = 253.28$
Monoclinic, $P2_1/n$
$a = 16.7835$ (4) Å
$b = 8.4580$ (2) Å
$c = 18.0373$ (5) Å
$\beta = 111.653$ (1)°
$V = 2379.8$ (1) Å^3
$Z = 8$
$D_x = 1.414$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 5676 reflections
$\theta = 2.9$–27.5°
$\mu = 0.28$ mm^{-1}
$T = 120$ (2) K
Prism, colourless
0.18 × 0.15 × 0.07 mm

Data collection

Nonius KappaCCD diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2003)
$T_{min} = 0.952$, $T_{max} = 0.981$
28267 measured reflections
5447 independent reflections
4260 reflections with $I > 2\sigma(I)$
$R_{int} = 0.051$
$\theta_{max} = 27.5°$
$h = -21 \rightarrow 21$
$k = -10 \rightarrow 10$
$l = -23 \rightarrow 21$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.046$
$wR(F^2) = 0.122$
$S = 1.09$
5447 reflections
309 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0594P)^2 + 0.8191P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.56$ e Å^{-3}
$\Delta\rho_{min} = -0.66$ e Å^{-3}
Extinction correction: *SHELXL97*
Extinction coefficient: 0.0145 (11)

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

D—H···A	D—H	H···A	D···A	D—H···A
N21A—H21A···N3B	0.88	2.13	2.979 (2)	161
N21A—H22A···O1W^{i}	0.88	1.97	2.805 (2)	158
N21B—H21B···N3A	0.88	2.16	3.005 (2)	160
N21B—H22B···O51A^{ii}	0.88	2.05	2.890 (2)	160
O1W—H1W···O41A	0.83 (3)	2.02 (3)	2.853 (2)	175 (2)
O1W—H2W···O51B^{iii}	0.84 (3)	1.97 (3)	2.808 (2)	175 (2)

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

All H atoms, except the two water H atoms, were included in the refinement at calculated positions, in the riding-model approximation, with C—H distances of 0.98 (CH_3 H atoms) and 0.99 Å (CH_2 H atoms) and an N—H distance of 0.88 Å. The two water H atoms were located in Fourier syntheses and positional parameters were refined. The isotropic displacement parameters for all H atoms were set equal to $1.25U_{eq}$ of the carrier atom.

Data collection: *COLLECT* (Hooft, 1998); cell refinement: *DENZO* (Otwinowski & Minor, 1997) and *COLLECT*; data reduction: *DENZO* and *COLLECT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *PLATON97* (Spek, 2003); software used to prepare material for publication: *SHELXL97*.

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$c = 18.0373\ (5)$ Å
$\beta = 111.653\ (1)°$
$V = 2379.80\ (10)$ Å^3
$Z = 8$
$F(000) = 1064$
$D_x = 1.414$ Mg m^{-3}
Melting point = 377–378 K
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 5676 reflections
$\theta = 2.9–27.5°$
$\mu = 0.28$ mm^{-1}
$T = 120$ K
Prism, colourless
0.18 × 0.15 × 0.07 mm

Data collection

Nonius KappaCCD diffractometer
Radiation source: Bruker Nonius FR591 rotating anode
10 cm confocal mirrors monochromator
Detector resolution: 9.091 pixels mm^{-1}
φ and ω scans
Absorption correction: multi-scan (SADABS; Sheldrick, 2003)
$T_{min} = 0.952$, $T_{max} = 0.981$
28267 measured reflections
5447 independent reflections
4260 reflections with $I > 2\sigma(I)$
$R_{int} = 0.051$
$\theta_{max} = 27.5°$, $\theta_{min} = 3.2°$
$h = -21\rightarrow21$
$k = -10\rightarrow10$
$l = -23\rightarrow21$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.046$
$wR(F^2) = 0.122$
$S = 1.09$
5447 reflections
309 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0594P)^2 + 0.8191P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.56$ e Å^{-3}
$\Delta\rho_{min} = -0.66$ e Å^{-3}
Extinction correction: SHELXL97, $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.0145 (11)

Special details

Experimental. The minimum and maximum absorption values stated above are those calculated in *SHELXL97* from the given crystal dimensions. The ratio of minimum to maximum apparent transmission was determined experimentally as 0.847918.

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

	x	*y*	*z*	U_{iso}*/U_{eq}
S1A	0.00585 (3)	0.19269 (5)	0.04174 (3)	0.01969 (14)
C2A	0.00612 (11)	0.0765 (2)	0.12202 (11)	0.0200 (4)
N21A	−0.06484 (10)	0.0536 (2)	0.13670 (10)	0.0251 (4)
H21A	−0.0635	−0.0053	0.1774	0.031*
H22A	−0.1133	0.0973	0.1058	0.031*
N3A	0.08201 (9)	0.01515 (19)	0.16560 (9)	0.0202 (3)
C4A	0.14150 (11)	0.0590 (2)	0.13455 (11)	0.0185 (4)
C41A	0.23107 (11)	−0.0028 (2)	0.17509 (11)	0.0187 (4)
O41A	0.26555 (8)	−0.09470 (17)	0.14581 (8)	0.0274 (3)
O42A	0.26563 (8)	0.05404 (16)	0.24851 (8)	0.0242 (3)
C42A	0.35207 (12)	−0.0022 (2)	0.29540 (12)	0.0261 (4)
H41A	0.3921	0.0256	0.2686	0.033*
H42A	0.3521	−0.1186	0.3015	0.033*
C43A	0.37871 (14)	0.0766 (3)	0.37519 (12)	0.0341 (5)
H43A	0.3802	0.1914	0.3684	0.043*
H44A	0.4358	0.0393	0.4091	0.043*
H45A	0.3375	0.0508	0.4003	0.043*
C5A	0.11454 (11)	0.1528 (2)	0.06849 (11)	0.0189 (4)
C51A	0.16686 (12)	0.2219 (2)	0.02763 (11)	0.0201 (4)
O51A	0.24464 (8)	0.21272 (17)	0.05187 (8)	0.0262 (3)
O52A	0.11926 (8)	0.29819 (16)	−0.03851 (8)	0.0236 (3)
C52A	0.16613 (13)	0.3716 (3)	−0.08358 (13)	0.0312 (5)
H51A	0.1917	0.2895	−0.1071	0.039*
H52A	0.2127	0.4396	−0.0483	0.039*
C53A	0.10264 (14)	0.4683 (3)	−0.14800 (13)	0.0350 (5)
H53A	0.0579	0.3991	−0.1833	0.044*
H54A	0.1319	0.5223	−0.1789	0.044*
H55A	0.0766	0.5469	−0.1240	0.044*
S1B	0.05291 (3)	−0.21687 (6)	0.43465 (3)	0.02324 (14)
C2B	0.04552 (12)	−0.1559 (2)	0.34028 (11)	0.0234 (4)
N21B	0.11009 (10)	−0.1726 (2)	0.31459 (10)	0.0310 (4)
H21B	0.1041	−0.1408	0.2664	0.039*
H22B	0.1587	−0.2154	0.3459	0.039*
N3B	−0.02927 (9)	−0.09242 (19)	0.29602 (9)	0.0220 (4)
C4B	−0.08278 (11)	−0.0887 (2)	0.33755 (11)	0.0196 (4)
C5B	−0.05159 (11)	−0.1509 (2)	0.41288 (11)	0.0204 (4)
C41B	−0.16709 (11)	−0.0072 (2)	0.29871 (11)	0.0213 (4)
O41B	−0.19714 (9)	0.08482 (18)	0.33210 (9)	0.0331 (4)
O42B	−0.20234 (8)	−0.04339 (17)	0.22165 (8)	0.0266 (3)
C42B	−0.28320 (12)	0.0370 (3)	0.17790 (12)	0.0312 (5)

H41B	−0.2726	0.1501	0.1707	0.039*
H42B	−0.3221	0.0298	0.2078	0.039*
C43B	−0.32269 (13)	−0.0421 (3)	0.09868 (12)	0.0300 (5)
H43B	−0.2853	−0.0285	0.0683	0.037*
H44B	−0.3788	0.0055	0.0692	0.037*
H45B	−0.3299	−0.1550	0.1065	0.037*
C51B	−0.09509 (12)	−0.1756 (2)	0.46898 (11)	0.0208 (4)
O51B	−0.17060 (8)	−0.15335 (17)	0.45389 (8)	0.0265 (3)
O52B	−0.04057 (8)	−0.23094 (17)	0.53874 (8)	0.0249 (3)
C52B	−0.07804 (13)	−0.2693 (3)	0.59773 (12)	0.0283 (5)
H51B	−0.1233	−0.3504	0.5764	0.035*
H52B	−0.1039	−0.1738	0.6114	0.035*
C53B	−0.00694 (14)	−0.3311 (3)	0.67031 (13)	0.0339 (5)
H53B	0.0398	−0.2537	0.6879	0.042*
H54B	0.0145	−0.4310	0.6572	0.042*
H55B	−0.0287	−0.3490	0.7131	0.042*
O1W	0.19277 (9)	−0.21340 (19)	−0.01321 (9)	0.0293 (3)
H1W	0.2123 (15)	−0.173 (3)	0.0322 (16)	0.037*
H2W	0.2337 (16)	−0.258 (3)	−0.0215 (15)	0.037*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1A	0.0172 (2)	0.0240 (3)	0.0185 (2)	0.00079 (18)	0.00726 (18)	0.00329 (18)
C2A	0.0211 (9)	0.0218 (10)	0.0183 (9)	−0.0003 (7)	0.0087 (7)	−0.0005 (7)
N21A	0.0179 (8)	0.0353 (10)	0.0243 (9)	0.0045 (7)	0.0103 (7)	0.0095 (7)
N3A	0.0186 (7)	0.0252 (9)	0.0187 (8)	0.0014 (6)	0.0089 (6)	0.0010 (6)
C4A	0.0188 (8)	0.0207 (10)	0.0179 (9)	−0.0014 (7)	0.0090 (7)	−0.0029 (7)
C41A	0.0181 (8)	0.0223 (10)	0.0174 (9)	−0.0022 (7)	0.0083 (7)	0.0018 (7)
O41A	0.0239 (7)	0.0358 (8)	0.0219 (7)	0.0064 (6)	0.0079 (6)	−0.0050 (6)
O42A	0.0199 (6)	0.0314 (8)	0.0193 (7)	0.0054 (6)	0.0049 (5)	−0.0042 (6)
C42A	0.0203 (9)	0.0299 (11)	0.0237 (10)	0.0042 (8)	0.0030 (8)	−0.0006 (8)
C43A	0.0345 (11)	0.0324 (12)	0.0253 (11)	0.0083 (9)	−0.0008 (9)	−0.0029 (9)
C5A	0.0170 (8)	0.0232 (10)	0.0173 (9)	−0.0013 (7)	0.0074 (7)	−0.0015 (7)
C51A	0.0212 (9)	0.0213 (10)	0.0183 (9)	−0.0024 (7)	0.0079 (7)	−0.0017 (7)
O51A	0.0181 (7)	0.0358 (8)	0.0237 (7)	−0.0039 (6)	0.0067 (6)	0.0023 (6)
O52A	0.0205 (6)	0.0314 (8)	0.0212 (7)	−0.0002 (6)	0.0103 (5)	0.0070 (6)
C52A	0.0294 (10)	0.0373 (12)	0.0337 (12)	0.0032 (9)	0.0196 (9)	0.0148 (10)
C53A	0.0362 (12)	0.0425 (14)	0.0319 (12)	0.0077 (10)	0.0190 (10)	0.0130 (10)
S1B	0.0187 (2)	0.0320 (3)	0.0195 (2)	0.00271 (19)	0.00761 (19)	0.00632 (19)
C2B	0.0214 (9)	0.0281 (11)	0.0211 (10)	0.0017 (8)	0.0084 (8)	0.0026 (8)
N21B	0.0208 (8)	0.0507 (12)	0.0245 (9)	0.0112 (8)	0.0120 (7)	0.0125 (8)
N3B	0.0194 (8)	0.0279 (9)	0.0203 (8)	0.0029 (6)	0.0092 (6)	0.0036 (7)
C4B	0.0195 (9)	0.0211 (10)	0.0211 (9)	−0.0007 (7)	0.0111 (7)	0.0001 (7)
C5B	0.0193 (9)	0.0240 (10)	0.0197 (9)	−0.0009 (7)	0.0092 (7)	0.0008 (8)
C41B	0.0204 (9)	0.0247 (10)	0.0207 (10)	−0.0008 (8)	0.0100 (8)	0.0036 (8)
O41B	0.0336 (8)	0.0369 (9)	0.0288 (8)	0.0103 (7)	0.0117 (6)	−0.0024 (7)
O42B	0.0217 (7)	0.0352 (8)	0.0214 (7)	0.0082 (6)	0.0060 (6)	−0.0005 (6)

C42B	0.0227 (10)	0.0418 (13)	0.0264 (11)	0.0117 (9)	0.0058 (8)	0.0028 (9)
C43B	0.0268 (10)	0.0293 (11)	0.0293 (11)	0.0032 (8)	0.0052 (9)	0.0019 (9)
C51B	0.0226 (9)	0.0199 (10)	0.0206 (9)	−0.0026 (7)	0.0089 (8)	−0.0002 (7)
O51B	0.0218 (7)	0.0342 (8)	0.0271 (8)	−0.0002 (6)	0.0132 (6)	0.0048 (6)
O52B	0.0219 (7)	0.0367 (8)	0.0183 (7)	−0.0014 (6)	0.0099 (6)	0.0055 (6)
C52B	0.0285 (10)	0.0392 (12)	0.0224 (10)	−0.0019 (9)	0.0155 (9)	0.0050 (9)
C53B	0.0367 (12)	0.0424 (13)	0.0243 (11)	−0.0056 (10)	0.0133 (9)	0.0072 (10)
O1W	0.0224 (7)	0.0430 (10)	0.0219 (7)	0.0070 (6)	0.0075 (6)	−0.0025 (7)

Geometric parameters (Å, º)

S1A—C5A	1.7392 (18)	S1B—C5B	1.7418 (18)
S1A—C2A	1.7485 (19)	C2B—N3B	1.327 (2)
C2A—N21A	1.326 (2)	C2B—N21B	1.334 (2)
C2A—N3A	1.331 (2)	N21B—H21B	0.88
N21A—H21A	0.88	N21B—H22B	0.88
N21A—H22A	0.88	N3B—C4B	1.366 (2)
N3A—C4A	1.364 (2)	C4B—C5B	1.368 (3)
C4A—C5A	1.363 (3)	C4B—C41B	1.496 (3)
C4A—C41A	1.502 (2)	C5B—C51B	1.465 (3)
C41A—O41A	1.201 (2)	C41B—O41B	1.203 (2)
C41A—O42A	1.325 (2)	C41B—O42B	1.330 (2)
O42A—C42A	1.463 (2)	O42B—C42B	1.462 (2)
C42A—C43A	1.497 (3)	C42B—C43B	1.493 (3)
C42A—H41A	0.99	C42B—H41B	0.99
C42A—H42A	0.99	C42B—H42B	0.99
C43A—H43A	0.98	C43B—H43B	0.98
C43A—H44A	0.98	C43B—H44B	0.98
C43A—H45A	0.98	C43B—H45B	0.98
C5A—C51A	1.460 (2)	C51B—O51B	1.209 (2)
C51A—O51A	1.217 (2)	C51B—O52B	1.338 (2)
C51A—O52A	1.332 (2)	O52B—C52B	1.458 (2)
O52A—C52A	1.462 (2)	C52B—C53B	1.503 (3)
C52A—C53A	1.497 (3)	C52B—H51B	0.99
C52A—H51A	0.99	C52B—H52B	0.99
C52A—H52A	0.99	C53B—H53B	0.98
C53A—H53A	0.98	C53B—H54B	0.98
C53A—H54A	0.98	C53B—H55B	0.98
C53A—H55A	0.98	O1W—H1W	0.83 (3)
S1B—C2B	1.739 (2)	O1W—H2W	0.84 (3)
C5A—S1A—C2A	88.82 (9)	N3B—C2B—N21B	122.89 (17)
N21A—C2A—N3A	124.00 (17)	N3B—C2B—S1B	114.77 (14)
N21A—C2A—S1A	121.29 (14)	N21B—C2B—S1B	122.34 (14)
N3A—C2A—S1A	114.70 (13)	C2B—N21B—H21B	120.0
C2A—N21A—H21A	120.0	C2B—N21B—H22B	120.0
C2A—N21A—H22A	120.0	H21B—N21B—H22B	120.0
H21A—N21A—H22A	120.0	C2B—N3B—C4B	110.29 (16)

C2A—N3A—C4A	109.72 (15)	N3B—C4B—C5B	116.59 (16)
N3A—C4A—C5A	117.41 (16)	N3B—C4B—C41B	116.98 (16)
N3A—C4A—C41A	117.27 (16)	C5B—C4B—C41B	126.28 (16)
C5A—C4A—C41A	125.31 (16)	C4B—C5B—C51B	129.44 (17)
O41A—C41A—O42A	124.75 (16)	C4B—C5B—S1B	109.36 (13)
O41A—C41A—C4A	124.68 (16)	C51B—C5B—S1B	121.04 (14)
O42A—C41A—C4A	110.53 (15)	O41B—C41B—O42B	124.43 (17)
C41A—O42A—C42A	115.77 (14)	O41B—C41B—C4B	124.24 (17)
O42A—C42A—C43A	106.94 (15)	O42B—C41B—C4B	111.25 (16)
O42A—C42A—H41A	110.3	C41B—O42B—C42B	115.04 (15)
C43A—C42A—H41A	110.3	O42B—C42B—C43B	107.72 (16)
O42A—C42A—H42A	110.3	O42B—C42B—H41B	110.2
C43A—C42A—H42A	110.3	C43B—C42B—H41B	110.2
H41A—C42A—H42A	108.6	O42B—C42B—H42B	110.2
C42A—C43A—H43A	109.5	C43B—C42B—H42B	110.2
C42A—C43A—H44A	109.5	H41B—C42B—H42B	108.5
H43A—C43A—H44A	109.5	C42B—C43B—H43B	109.5
C42A—C43A—H45A	109.5	C42B—C43B—H44B	109.5
H43A—C43A—H45A	109.5	H43B—C43B—H44B	109.5
H44A—C43A—H45A	109.5	C42B—C43B—H45B	109.5
C4A—C5A—C51A	127.36 (16)	H43B—C43B—H45B	109.5
C4A—C5A—S1A	109.35 (13)	H44B—C43B—H45B	109.5
C51A—C5A—S1A	123.15 (14)	O51B—C51B—O52B	124.24 (17)
O51A—C51A—O52A	124.18 (17)	O51B—C51B—C5B	124.79 (17)
O51A—C51A—C5A	123.96 (17)	O52B—C51B—C5B	110.94 (15)
O52A—C51A—C5A	111.85 (15)	C51B—O52B—C52B	115.76 (14)
C51A—O52A—C52A	115.84 (14)	O52B—C52B—C53B	107.19 (16)
O52A—C52A—C53A	106.78 (16)	O52B—C52B—H51B	110.3
O52A—C52A—H51A	110.4	C53B—C52B—H51B	110.3
C53A—C52A—H51A	110.4	O52B—C52B—H52B	110.3
O52A—C52A—H52A	110.4	C53B—C52B—H52B	110.3
C53A—C52A—H52A	110.4	H51B—C52B—H52B	108.5
H51A—C52A—H52A	108.6	C52B—C53B—H53B	109.5
C52A—C53A—H53A	109.5	C52B—C53B—H54B	109.5
C52A—C53A—H54A	109.5	H53B—C53B—H54B	109.5
H53A—C53A—H54A	109.5	C52B—C53B—H55B	109.5
C52A—C53A—H55A	109.5	H53B—C53B—H55B	109.5
H53A—C53A—H55A	109.5	H54B—C53B—H55B	109.5
H54A—C53A—H55A	109.5	H1W—O1W—H2W	108 (2)
C2B—S1B—C5B	88.98 (9)		

C5A—S1A—C2A—N21A	−179.15 (17)	C5B—S1B—C2B—N3B	−0.39 (16)
C5A—S1A—C2A—N3A	0.81 (15)	C5B—S1B—C2B—N21B	179.86 (19)
N21A—C2A—N3A—C4A	179.23 (18)	N21B—C2B—N3B—C4B	−179.31 (19)
S1A—C2A—N3A—C4A	−0.7 (2)	S1B—C2B—N3B—C4B	0.9 (2)
C2A—N3A—C4A—C5A	0.2 (2)	C2B—N3B—C4B—C5B	−1.2 (2)
C2A—N3A—C4A—C41A	−178.60 (16)	C2B—N3B—C4B—C41B	174.65 (17)
N3A—C4A—C41A—O41A	113.2 (2)	N3B—C4B—C5B—C51B	−174.50 (18)

C5A—C4A—C41A—O41A	−65.5 (3)	C41B—C4B—C5B—C51B	10.1 (3)
N3A—C4A—C41A—O42A	−64.7 (2)	N3B—C4B—C5B—S1B	0.9 (2)
C5A—C4A—C41A—O42A	116.6 (2)	C41B—C4B—C5B—S1B	−174.51 (15)
O41A—C41A—O42A—C42A	−0.1 (3)	C2B—S1B—C5B—C4B	−0.28 (15)
C4A—C41A—O42A—C42A	177.84 (15)	C2B—S1B—C5B—C51B	175.58 (17)
C41A—O42A—C42A—C43A	179.64 (17)	N3B—C4B—C41B—O41B	−134.5 (2)
N3A—C4A—C5A—C51A	176.18 (17)	C5B—C4B—C41B—O41B	40.9 (3)
C41A—C4A—C5A—C51A	−5.1 (3)	N3B—C4B—C41B—O42B	42.3 (2)
N3A—C4A—C5A—S1A	0.4 (2)	C5B—C4B—C41B—O42B	−142.28 (19)
C41A—C4A—C5A—S1A	179.09 (15)	O41B—C41B—O42B—C42B	−0.4 (3)
C2A—S1A—C5A—C4A	−0.64 (14)	C4B—C41B—O42B—C42B	−177.24 (16)
C2A—S1A—C5A—C51A	−176.64 (16)	C41B—O42B—C42B—C43B	−167.86 (17)
C4A—C5A—C51A—O51A	−5.6 (3)	C4B—C5B—C51B—O51B	5.6 (3)
S1A—C5A—C51A—O51A	169.63 (15)	S1B—C5B—C51B—O51B	−169.30 (16)
C4A—C5A—C51A—O52A	175.46 (18)	C4B—C5B—C51B—O52B	−176.23 (19)
S1A—C5A—C51A—O52A	−9.3 (2)	S1B—C5B—C51B—O52B	8.8 (2)
O51A—C51A—O52A—C52A	1.0 (3)	O51B—C51B—O52B—C52B	1.8 (3)
C5A—C51A—O52A—C52A	179.96 (16)	C5B—C51B—O52B—C52B	−176.36 (16)
C51A—O52A—C52A—C53A	−172.28 (17)	C51B—O52B—C52B—C53B	179.11 (17)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N21*A*—H21*A*···N3*B*	0.88	2.13	2.979 (2)	161
N21*A*—H22*A*···O1*W*^{i}	0.88	1.97	2.805 (2)	158
N21*B*—H21*B*···N3*A*	0.88	2.16	3.005 (2)	160
N21*B*—H22*B*···O51*A*^{ii}	0.88	2.05	2.890 (2)	160
O1*W*—H1*W*···O41*A*	0.83 (3)	2.02 (3)	2.853 (2)	175 (2)
O1*W*—H2*W*···O51*B*^{iii}	0.84 (3)	1.97 (3)	2.808 (2)	175 (2)

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