metal-organic compounds

Dipotassium disulfanilamidate trihydrate

Fiona N.-F How,[a] Z. A. Rahima,[a] Hamid Khaledi[b]* and Hapipah Mohd Ali[b]

[a]Department of Biomedical Sciences, Kulliyah of Science, IIUM Kuantan, 25200 Kuantan, Malaysia, and [b]Department of Chemistry, University of Malaya, 50603 Kuala Lumpur, Malaysia

Correspondence e-mail: khaledi@siswa.um.edu.my

The asymmetric unit of the title compound, $2K^+ \cdot 2C_6H_7N_2O_2S^- \cdot 3H_2O$, consists of two potassium cations located on mirror planes, one sulfanilamidate anion in a general position and one and a half molecules of water, one of which is also located on a mirror plane. One potassium cation is seven-coordinated by six sulfonyl O atoms and one water molecule, whereas the other is surrounded by six water O atoms and two sulfonyl O atoms. In the crystal structure, the components are connected into polymeric sheets in the bc plane. The two-dimensional structure is consolidated by N—H⋯O, O—H⋯O, O—H⋯N and C—H⋯π interactions. The layers are further linked into a three-dimensional network via N—H⋯O, N—H⋯N and O—H⋯N hydrogen bonds.

Related literature

For the structures of similar potassium salts, see: Gowda *et al.* (2011) and references cited therein; Moers *et al.* (2001). For the structure of sodium sulfanilamide monohydrate, see: Moreno & Alleaume (1968).
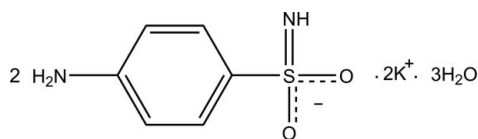
Experimental

Crystal data

$2K^+ \cdot 2C_6H_7N_2O_2S^- \cdot 3H_2O$
$M_r = 474.64$
Orthorhombic, $Cmc2_1$
$a = 23.8174\ (4)$ Å
$b = 10.9141\ (2)$ Å
$c = 7.4645\ (1)$ Å

$V = 1940.36\ (5)$ Å^3
$Z = 4$
Mo $K\alpha$ radiation
$\mu = 0.75$ mm^{-1}
$T = 100$ K
$0.33 \times 0.27 \times 0.10$ mm

Data collection

Bruker APEXII CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{min} = 0.791$, $T_{max} = 0.929$

8594 measured reflections
2157 independent reflections
2149 reflections with $I > 2\sigma(I)$
$R_{int} = 0.015$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.015$
$wR(F^2) = 0.042$
$S = 1.11$
2157 reflections
145 parameters
7 restraints

H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max} = 0.26$ e Å^{-3}
$\Delta\rho_{min} = -0.23$ e Å^{-3}
Absolute structure: Flack (1983), 985 Friedel pairs
Flack parameter: 0.04 (3)

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

*Cg*1 is the centroid of the C1–C6 ring.

D—H⋯A	D—H	H⋯A	D⋯A	D—H⋯A
N1—H1⋯O1[i]	0.88 (1)	2.17 (1)	2.9854 (15)	154 (2)
N2—H2A⋯N1[ii]	0.90 (1)	2.13 (1)	3.0109 (16)	166 (1)
N2—H2B⋯O3[iii]	0.91 (1)	2.12 (1)	3.0183 (15)	171 (1)
O3—H3A⋯N2[iv]	0.85 (2)	1.99 (2)	2.8366 (15)	172 (2)
O3—H3B⋯O2[v]	0.83 (2)	1.95 (2)	2.7561 (13)	164 (2)
O4—H4⋯N1[vi]	0.79 (1)	2.05 (1)	2.8291 (14)	175 (2)
C2—H2⋯Cg1[i]	0.95	2.98	3.5872 (14)	123
C5—H5⋯Cg1[ii]	0.95	2.66	3.4531 (14)	141

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

Data collection: *APEX2* (Bruker, 2007); cell refinement: *SAINT* (Bruker, 2007); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *X-SEED* (Barbour, 2001); software used to prepare material for publication: *SHELXL97* and *publCIF* (Westrip, 2010).

References

Barbour, L. J. (2001). *J. Supramol. Chem.* **1**, 189–191.
Bruker (2007). *APEX2* and *SAINT*. Bruker AXS Inc., Madison, Wisconsin, USA.
Flack, H. D. (1983). *Acta Cryst.* A**39**, 876–881.
Gowda, B. T., Foro, S. & Shakuntala, K. (2011). *Acta Cryst.* E**67**, m1015.
Moers, O., Blaschette, A. & Jones, P. G. (2001). *Z. Anorg. Allg. Chem.* **627**, 95–102.
Moreno, J. & Alleaume, M. (1968). *C. R. Acad. Sci. Ser. C*, **267**, 64–66.
Sheldrick, G. M. (1996). *SADABS*. University of Göttingen, Germany.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Westrip, S. P. (2010). *J. Appl. Cryst.* **43**, 920–925.

supporting information

Dipotassium disulfanilamidate trihydrate

Fiona N.-F How, Z. A. Rahima, Hamid Khaledi and Hapipah Mohd Ali

S1. Comment

The title potassium salt (Fig. 1) was obtained through the deprotonation of sulfanilamide by KOH. The structure contains two types of potassium cation, one of which, K1, is hepta-coordinated by six sulfonyl O atoms and one water molecule while the other one, K2, is octa-coordinated by six water O atoms and two sulfonyl O atoms. Both potassium atoms and one water oxygen atom, O4, are placed on a mirror plane.

The bond distance of S—N [1.5410 (12) Å] implies its double bond character and is slightly shorter than the observed values (~ 1.58 Å) in similar potassium salts (Gowda *et al.*, 2011; Moers *et al.*, 2001), but comparable with the bond length reported for sodium sulfanilamide (Moreno & Alleaume, 1968). The S1—O1 and S1—O2 distances of 1.4686 (9) and 1.4705 (9) Å attest to delocalization of the negative charge over the O—S—O fragment. These values are similar to the corresponding values in sodium sulfanilamide (1.45–1.46 Å).

In the crystal, coordination polymeric layers are formed in the *bc* plane (Fig. 2). The two dimensional structure is supplemented by N—H···O, O—H···O, O—H···N and C—H···π interactions (Table 1). The layers are further linked into a three dimensional network *via* N—H···O, N—H···N and O—H···N hydrogen bonds (Fig. 3)

S2. Experimental

Sulfanilamide (2 g) in ethanol (30 ml) was mixed with an equimolar amount of KOH in 90% ethanol (10 ml). The solution was then refluxed for 2 hr and left to cool down at room temperature. The colorless crystals of the the potassium salt were obtained within a day.

S3. Refinement

The C-bound H atoms were placed at calculated positions and were treated as riding on their parent C atoms with C—H = 0.95 Å. The N– and O-bound H atoms were located in a difference Fourier map, and refined with distance restraints of O—H = 0.84 (2) Å and N—H = 0.91 (2) Å. For all H atoms, U_{iso}(H) was set to 1.2(1.5 for hydroxyl)U_{eq}(carrier atom). An absolute structure was established using anomalous dispersion effects; 985 Friedel pairs were not merged.
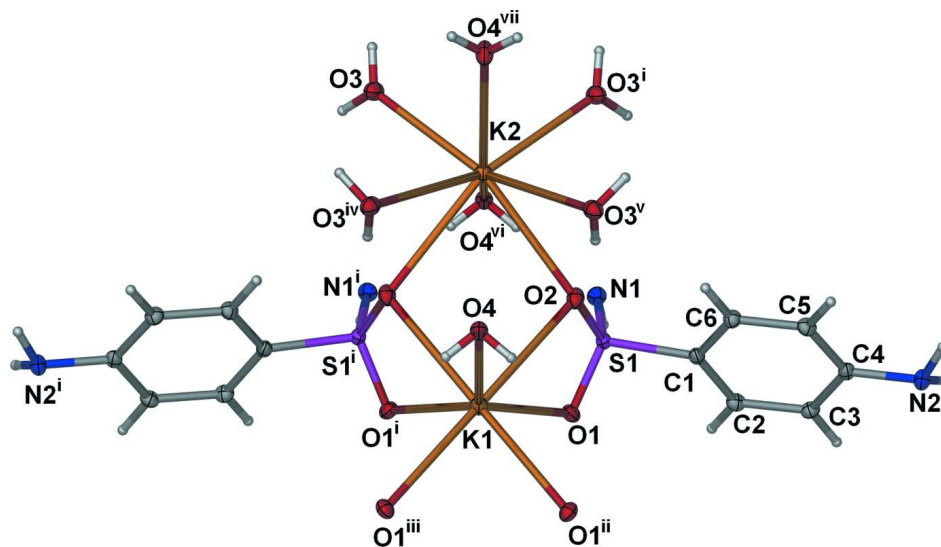
Figure 1

Thermal ellipsoid plot of the title compound at the 50% probability level. Hydrogen atoms are drawn as spheres of arbitrary radius. Symmetry code: i = -*x* + 1, *y*, *z*; ii = *x*, -*y* + 1, *z* + 1/2; iii = -*x* + 1, -*y* + 1, *z* + 1/2; iv = *x*, -*y*, *z* + 1/2; v = -*x* + 1, -*y*, *z* + 1/2; vi = *x*, *y*, *z* - 1; vii = -*x* + 1, -*y*, *z* - 1/2.
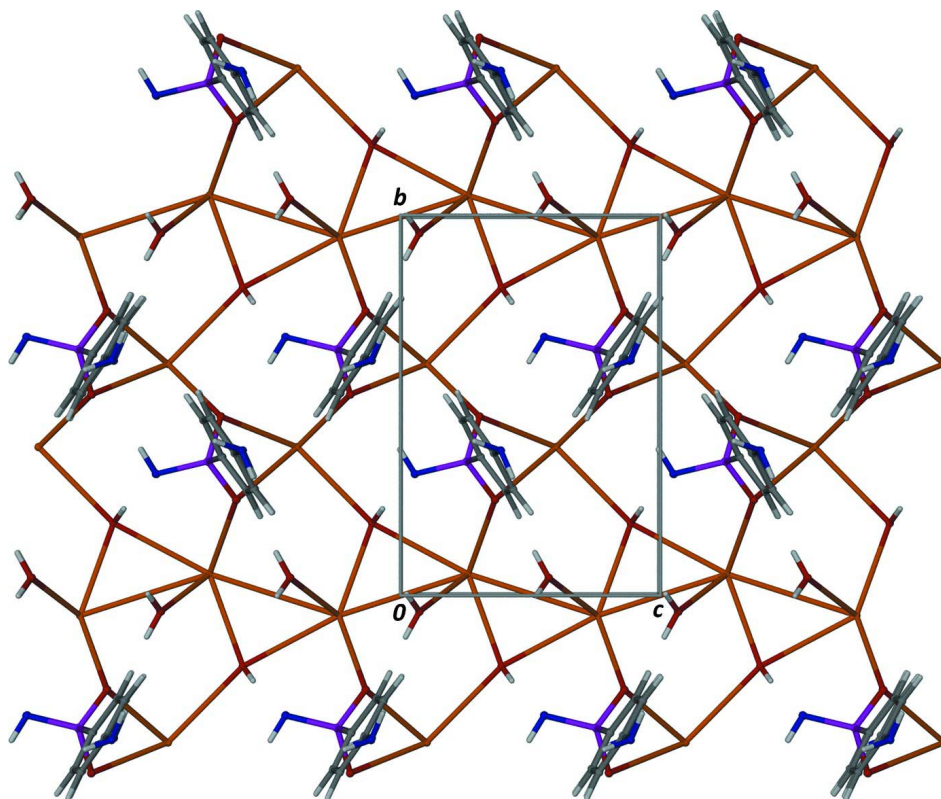
Figure 2

Packing view looking down the crystallographic *α* axis showing the two dimensional coordination polymer netwrok.
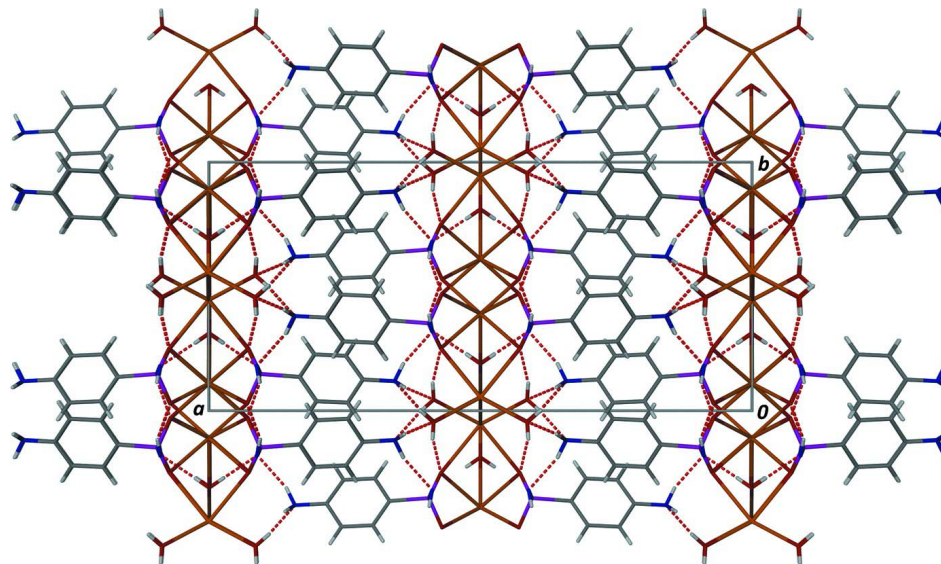
Figure 3

Packing view looking down the crystallographic *c* axis. H-bonds are shown as red dashed lines.

Dipotassium disulfanilamidate trihydrate

Crystal data

$2K^+ \cdot 2C_6H_7N_2O_2S^- \cdot 3H_2O$	$F(000) = 984$
$M_r = 474.64$	$D_x = 1.625$ Mg m^{-3}
Orthorhombic, $Cmc2_1$	Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Hall symbol: C 2c -2	Cell parameters from 9001 reflections
$a = 23.8174$ (4) Å	$\theta = 2.7$–30.5°
$b = 10.9141$ (2) Å	$\mu = 0.75$ mm^{-1}
$c = 7.4645$ (1) Å	$T = 100$ K
$V = 1940.36$ (5) Å^3	Blade, colorless
$Z = 4$	$0.33 \times 0.27 \times 0.10$ mm

Data collection

Bruker APEXII CCD diffractometer	8594 measured reflections
Radiation source: fine-focus sealed tube	2157 independent reflections
Graphite monochromator	2149 reflections with $I > 2\sigma(I)$
φ and ω scans	$R_{int} = 0.015$
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)	$\theta_{max} = 27.0°$, $\theta_{min} = 2.1°$
$T_{min} = 0.791$, $T_{max} = 0.929$	$h = -30 \rightarrow 30$
	$k = -13 \rightarrow 13$
	$l = -9 \rightarrow 9$

Refinement

Refinement on F^2	Primary atom site location: structure-invariant direct methods
Least-squares matrix: full	
$R[F^2 > 2\sigma(F^2)] = 0.015$	Secondary atom site location: difference Fourier map
$wR(F^2) = 0.042$	
$S = 1.11$	Hydrogen site location: inferred from neighbouring sites
2157 reflections	
145 parameters	H atoms treated by a mixture of independent and constrained refinement
7 restraints	

$w = 1/[\sigma^2(F_o^2) + (0.0201P)^2 + 1.0371P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.26$ e Å^{-3}

$\Delta\rho_{min} = -0.23$ e Å^{-3}
Absolute structure: Flack (1983), 985 Friedel pairs
Absolute structure parameter: 0.04 (3)

Special details

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

Refinement. Refinement of F^2 against ALL reflections. The weighted R-factor wR and goodness of fit S are based on F^2, conventional R-factors R are based on F, with F set to zero for negative F^2. The threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating R-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. R-factors based on F^2 are statistically about twice as large as those based on F, and R- factors based on ALL data will be even larger.

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

	x	*y*	*z*	U_{iso}*/U_{eq}
K1	0.5000	0.39021 (4)	0.59975 (5)	0.01535 (8)
K2	0.5000	0.05477 (3)	0.26065 (6)	0.01442 (8)
S1	0.404982 (11)	0.35192 (2)	0.25404 (4)	0.01158 (7)
O1	0.42926 (4)	0.47094 (8)	0.30427 (12)	0.01535 (19)
O2	0.42708 (4)	0.25122 (8)	0.36386 (12)	0.01634 (19)
N1	0.41122 (4)	0.31915 (11)	0.05418 (15)	0.0147 (2)
H1	0.4057 (7)	0.3869 (13)	−0.007 (2)	0.018*
N2	0.15806 (5)	0.38097 (11)	0.38621 (15)	0.0148 (2)
H2A	0.1423 (6)	0.3121 (13)	0.430 (2)	0.018*
H2B	0.1402 (6)	0.4057 (14)	0.285 (2)	0.018*
C1	0.33256 (5)	0.36379 (11)	0.30816 (17)	0.0124 (2)
C2	0.30175 (5)	0.46150 (10)	0.23864 (18)	0.0138 (2)
H2	0.3202	0.5243	0.1732	0.017*
C3	0.24417 (5)	0.46705 (10)	0.26498 (19)	0.0134 (2)
H3	0.2234	0.5342	0.2183	0.016*
C4	0.21636 (5)	0.37450 (11)	0.35982 (16)	0.0126 (2)
C5	0.24773 (5)	0.27905 (12)	0.43346 (18)	0.0144 (2)
H5	0.2295	0.2175	0.5023	0.017*
C6	0.30546 (5)	0.27342 (11)	0.40674 (17)	0.0138 (2)
H6	0.3265	0.2075	0.4561	0.017*
O3	0.58605 (4)	−0.04452 (9)	0.07181 (13)	0.0173 (2)
H3A	0.6103 (6)	0.0020 (18)	0.023 (3)	0.026*
H3B	0.5880 (8)	−0.1110 (15)	0.019 (3)	0.026*
O4	0.5000	0.18900 (13)	0.8893 (2)	0.0207 (3)
H4	0.4747 (6)	0.2267 (16)	0.929 (3)	0.031*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
K1	0.01301 (16)	0.01830 (18)	0.01474 (17)	0.000	0.000	−0.00331 (15)
K2	0.01360 (16)	0.01577 (17)	0.01391 (16)	0.000	0.000	−0.00172 (17)

S1	0.01032 (12)	0.01235 (13)	0.01206 (13)	−0.00085 (9)	0.00042 (13)	0.00013 (13)
O1	0.0144 (4)	0.0149 (4)	0.0168 (5)	−0.0035 (3)	0.0009 (3)	−0.0022 (3)
O2	0.0134 (4)	0.0169 (4)	0.0188 (5)	0.0009 (3)	−0.0002 (4)	0.0048 (4)
N1	0.0158 (5)	0.0161 (5)	0.0123 (5)	−0.0002 (4)	0.0024 (4)	−0.0005 (4)
N2	0.0118 (5)	0.0178 (5)	0.0148 (5)	0.0007 (4)	0.0004 (4)	0.0019 (4)
C1	0.0113 (6)	0.0142 (5)	0.0118 (5)	−0.0010 (5)	0.0009 (4)	−0.0027 (4)
C2	0.0163 (5)	0.0129 (5)	0.0120 (6)	−0.0015 (4)	0.0019 (5)	0.0017 (5)
C3	0.0157 (5)	0.0123 (5)	0.0121 (5)	0.0017 (4)	−0.0004 (5)	0.0008 (5)
C4	0.0121 (6)	0.0156 (6)	0.0102 (6)	−0.0001 (5)	0.0009 (4)	−0.0028 (5)
C5	0.0161 (6)	0.0136 (5)	0.0136 (5)	−0.0019 (5)	0.0017 (5)	0.0016 (5)
C6	0.0139 (6)	0.0134 (5)	0.0142 (6)	0.0010 (4)	−0.0007 (5)	0.0016 (5)
O3	0.0177 (4)	0.0155 (4)	0.0187 (5)	−0.0009 (3)	0.0033 (4)	−0.0010 (4)
O4	0.0135 (7)	0.0205 (7)	0.0280 (8)	0.000	0.000	−0.0097 (6)

Geometric parameters (Å, º)

K1—O1^i	2.7325 (9)	N1—H1	0.878 (14)
K1—O1^{ii}	2.7325 (9)	N2—C4	1.4044 (16)
K1—O2^{iii}	2.9014 (10)	N2—H2A	0.903 (13)
K1—O2	2.9014 (10)	N2—H2B	0.906 (14)
K1—O1	2.9121 (10)	C1—C6	1.3895 (17)
K1—O1^{iii}	2.9121 (10)	C1—C2	1.3947 (17)
K1—O4	3.0810 (16)	C2—C3	1.3868 (17)
K2—O3	2.7133 (10)	C2—H2	0.9500
K2—O3^{iii}	2.7133 (10)	C3—C4	1.4000 (17)
K2—O4^{iv}	2.8285 (15)	C3—H3	0.9500
K2—O2^{iii}	2.8648 (10)	C4—C5	1.3948 (18)
K2—O2	2.8648 (10)	C5—C6	1.3908 (17)
K2—O3^v	3.0996 (10)	C5—H5	0.9500
K2—O3^{vi}	3.0996 (10)	C6—H6	0.9500
K2—O4^{vii}	3.1355 (17)	O3—K2^{iv}	3.0996 (10)
S1—O2	1.4686 (9)	O3—H3A	0.852 (15)
S1—O1	1.4705 (9)	O3—H3B	0.828 (15)
S1—N1	1.5413 (12)	O4—K2^{vi}	2.8284 (15)
S1—C1	1.7763 (13)	O4—K2^{ix}	3.1355 (17)
O1—K1^{viii}	2.7324 (9)	O4—H4	0.787 (13)
O1^i—K1—O1^{ii}	76.15 (4)	O3^{vi}—K2—O4^{vii}	132.79 (2)
O1^i—K1—O2^{iii}	105.06 (3)	O2—S1—O1	112.21 (6)
O1^{ii}—K1—O2^{iii}	176.54 (3)	O2—S1—N1	109.40 (6)
O1^i—K1—O2	176.54 (3)	O1—S1—N1	114.44 (6)
O1^{ii}—K1—O2	105.06 (3)	O2—S1—C1	106.00 (6)
O2^{iii}—K1—O2	73.54 (4)	O1—S1—C1	105.03 (6)
O1^i—K1—O1	127.75 (2)	N1—S1—C1	109.31 (6)
O1^{ii}—K1—O1	84.18 (2)	S1—O1—K1^{viii}	126.15 (5)
O2^{iii}—K1—O1	92.57 (3)	S1—O1—K1	98.82 (4)
O2—K1—O1	49.62 (3)	K1^{viii}—O1—K1	103.54 (3)
O1^i—K1—O1^{iii}	84.18 (2)	S1—O2—K2	128.84 (5)

O1ⁱⁱ—K1—O1ⁱⁱⁱ	127.75 (2)	S1—O2—K1	99.32 (5)
O2ⁱⁱⁱ—K1—O1ⁱⁱⁱ	49.62 (3)	K2—O2—K1	101.04 (3)
O2—K1—O1ⁱⁱⁱ	92.58 (3)	S1—N1—H1	106.9 (11)
O1—K1—O1ⁱⁱⁱ	70.71 (4)	C4—N2—H2A	114.8 (10)
O1ⁱ—K1—O4	90.20 (3)	C4—N2—H2B	111.3 (10)
O1ⁱⁱ—K1—O4	90.20 (3)	H2A—N2—H2B	110.9 (15)
O2ⁱⁱⁱ—K1—O4	93.04 (3)	C6—C1—C2	119.69 (11)
O2—K1—O4	93.04 (3)	C6—C1—S1	121.31 (10)
O1—K1—O4	138.32 (2)	C2—C1—S1	118.83 (10)
O1ⁱⁱⁱ—K1—O4	138.32 (2)	C3—C2—C1	120.07 (11)
O3—K2—O3ⁱⁱⁱ	98.12 (4)	C3—C2—H2	120.0
O3—K2—O4ⁱᵛ	78.50 (3)	C1—C2—H2	120.0
O3ⁱⁱⁱ—K2—O4ⁱᵛ	78.50 (3)	C2—C3—C4	120.53 (11)
O3—K2—O2ⁱⁱⁱ	88.89 (3)	C2—C3—H3	119.7
O3ⁱⁱⁱ—K2—O2ⁱⁱⁱ	153.71 (3)	C4—C3—H3	119.7
O4ⁱᵛ—K2—O2ⁱⁱⁱ	127.79 (3)	C5—C4—C3	119.00 (11)
O3—K2—O2	153.71 (3)	C5—C4—N2	120.79 (11)
O3ⁱⁱⁱ—K2—O2	88.89 (3)	C3—C4—N2	120.15 (11)
O4ⁱᵛ—K2—O2	127.78 (3)	C6—C5—C4	120.40 (12)
O2ⁱⁱⁱ—K2—O2	74.64 (4)	C6—C5—H5	119.8
O3—K2—O3ᵛ	82.84 (3)	C4—C5—H5	119.8
O3ⁱⁱⁱ—K2—O3ᵛ	150.97 (2)	C1—C6—C5	120.25 (11)
O4ⁱᵛ—K2—O3ᵛ	73.24 (3)	C1—C6—H6	119.9
O2ⁱⁱⁱ—K2—O3ᵛ	54.88 (3)	C5—C6—H6	119.9
O2—K2—O3ᵛ	103.08 (3)	K2—O3—K2ⁱᵛ	84.51 (3)
O3—K2—O3ᵛⁱ	150.97 (2)	K2—O3—H3A	119.9 (14)
O3ⁱⁱⁱ—K2—O3ᵛⁱ	82.84 (3)	K2ⁱᵛ—O3—H3A	98.5 (13)
O4ⁱᵛ—K2—O3ᵛⁱ	73.24 (3)	K2—O3—H3B	129.9 (13)
O2ⁱⁱⁱ—K2—O3ᵛⁱ	103.08 (3)	K2ⁱᵛ—O3—H3B	69.3 (13)
O2—K2—O3ᵛⁱ	54.87 (3)	H3A—O3—H3B	106 (2)
O3ᵛ—K2—O3ᵛⁱ	82.79 (4)	K2ᵛⁱ—O4—K1	115.62 (5)
O3—K2—O4ᵛⁱⁱ	74.17 (3)	K2ᵛⁱ—O4—K2ⁱˣ	81.99 (4)
O3ⁱⁱⁱ—K2—O4ᵛⁱⁱ	74.17 (3)	K1—O4—K2ⁱˣ	162.39 (5)
O4ⁱᵛ—K2—O4ᵛⁱⁱ	137.70 (5)	K2ᵛⁱ—O4—H4	128.2 (13)
O2ⁱⁱⁱ—K2—O4ᵛⁱⁱ	83.57 (3)	K1—O4—H4	83.7 (15)
O2—K2—O4ᵛⁱⁱ	83.57 (3)	K2ⁱˣ—O4—H4	85.1 (16)
O3ᵛ—K2—O4ᵛⁱⁱ	132.79 (2)		

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

Hydrogen-bond geometry (Å, º)

Cg1 is the centroid of the C1–C6 ring.

D—H⋯*A*	*D*—H	H⋯*A*	*D*⋯*A*	*D*—H⋯*A*
N1—H1⋯O1ˣ	0.88 (1)	2.17 (1)	2.9854 (15)	154 (2)
N2—H2*A*⋯N1ˣⁱ	0.90 (1)	2.13 (1)	3.0109 (16)	166 (1)
N2—H2*B*⋯O3ˣⁱⁱ	0.91 (1)	2.12 (1)	3.0183 (15)	171 (1)
O3—H3*A*⋯N2ˣⁱⁱⁱ	0.85 (2)	1.99 (2)	2.8366 (15)	172 (2)

O3—H3*B*···O2^{iv}	0.83 (2)	1.95 (2)	2.7561 (13)	164 (2)
O4—H4···N1^{ix}	0.79 (1)	2.05 (1)	2.8291 (14)	175 (2)
C2—H2···*Cg*1^{x}	0.95	2.98	3.5872 (14)	123
C5—H5···*Cg*1^{xi}	0.95	2.66	3.4531 (14)	141

Symmetry codes: (iv) −*x*+1, −*y*, *z*−1/2; (ix) *x*, *y*, *z*+1; (x) *x*, −*y*+1, *z*−1/2; (xi) −*x*+1/2, −*y*+1/2, *z*+1/2; (xii) *x*−1/2, *y*+1/2, *z*; (xiii) *x*+1/2, −*y*+1/2, *z*−1/2.