Supporting Information for

$K_{0.36}(H_2O)_yWS_2$: A new layered compound for reversible hydrated potassium ion intercalation in aqueous electrolyte

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1. Experimental Section

Sample Synthesis. The reagent WS_2 was synthesized by heating a mixture of W powder and S powder at 973 K under vacuum. The obtained WS_2 powder (0.2480 g; 1 mmol) and K pieces (0.0391 g; 1 mmol) were sealed into carbon-coated fused silica tube. The tube was heated in the furnace at 1073 K for 20 hours and then cooled to room temperature. Finally, the reaction product was washed by distilled water for several times, and $K_{0.36}(H_2O)_yWS_2$ was prepared.

Single Crystal X-ray Crystallography. A suitable crystal of $K_{0.36}(H_2O)_yWS_2$ was selected for XRD data collection at 298 K. Diffraction data were collected using a Bruker D8 QUEST diffractometer equipped with a Mo $K\alpha$ radiation source. The diffraction data were integrated and corrected for absorption using a multi-scan type model by the APEX 2 and SADABS programs. The crystal data and structure refinement details are summarized in Table S3-S6.

Characterization. A Bruker D8 QUEST diffractometer equipped with Cu $K\alpha$ radiation was used to collect powder X-ray diffraction data of $K_{0.36}(H_2O)_yWS_2$ at 40 kV and 40 mA. Crystal imaging and quantitative microprobe analyses of the compound were performed by a JEOL (JSM6510) scanning electron microscope equipped with a PGT energy-dispersive X-ray analyzer. The accurate atom ratio was determined by ICP-OES. Raman measurement was performed via a thermal dispersive spectrometer using an excitation wavelength of 633 nm. Thermogravimetric analysis (TG) and differential scanning calorimetry (DSC) were conducted under Ar atmosphere at a heating rate of 5 °C/min.

Resistivity Measurement. The resistivity measurement was carried out on a Physical Properties Measurement System (PPMS). The polycrystal powders were pressed into pellets and cut into rectangular bars. Thin Cu wires were attached using silver epoxy for four probe resistivity measurements.

Electrochemical Measurements. Composite electrodes were fabricated by compressing active materials, carbon black and poly tetra fluoroethylene at a weight ratio of 8:1:1 on titanium mesh. The mass loading of working electrode was 5-6 mg/cm^2. The three-electrode system for electrochemical measurement consisted of $K_{0.36}(H_2O)_yWS_2$ composite as the working electrode, Pt as the counter-electrode, Ag/AgCl as the reference electrode, 0.5 M K_2SO_4 solution as electrolyte. The cyclic voltammetry (CV) and charge/discharge test were conducted on the electrochemical workstation (CHI660E, Shanghai Chenhua).

2. Supplementary figures

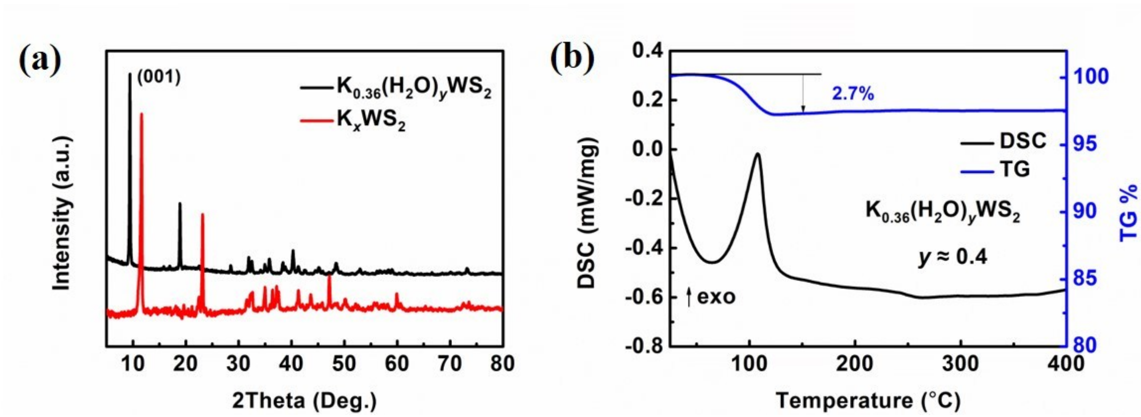

Figure S1. (a) Power X-ray diffraction pattern of $K_{0.36}(H_2O)_yWS_2$ and K_xWS_2. (b) TG-DSC curves of $K_{0.36}(H_2O)_yWS_2$.

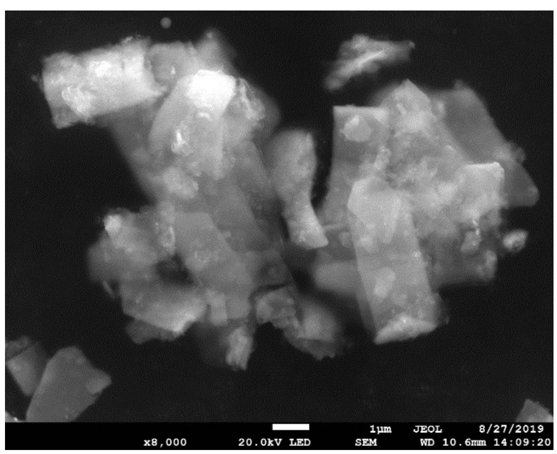

Figure S2. SEM pattern of $K_{0.36}(H_2O)_yWS_2$ hand-milled for fabricating composite electrodes.

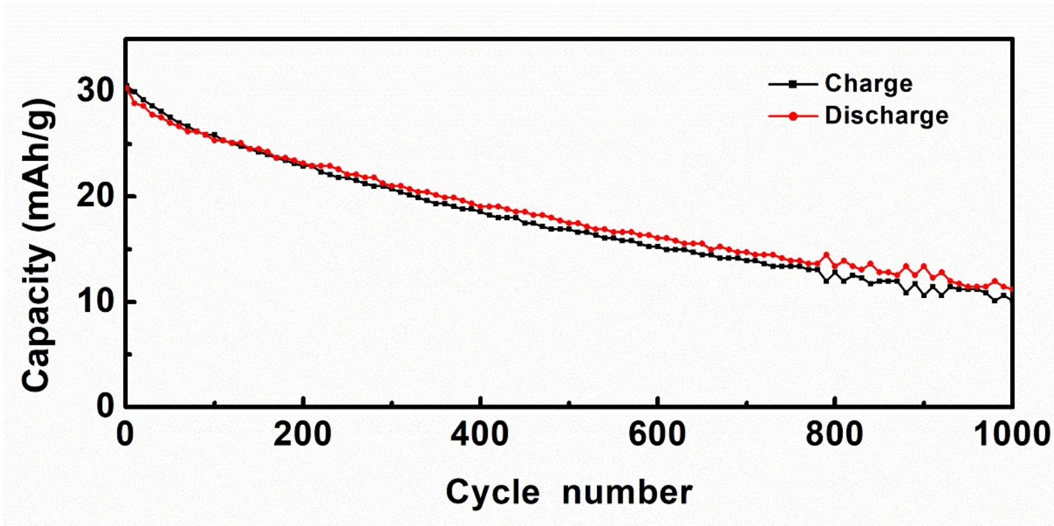

Figure S3. Cycling performance of $K_{0.36}(H_2O)_yWS_2$ electrode at 2 A g^{-1}.

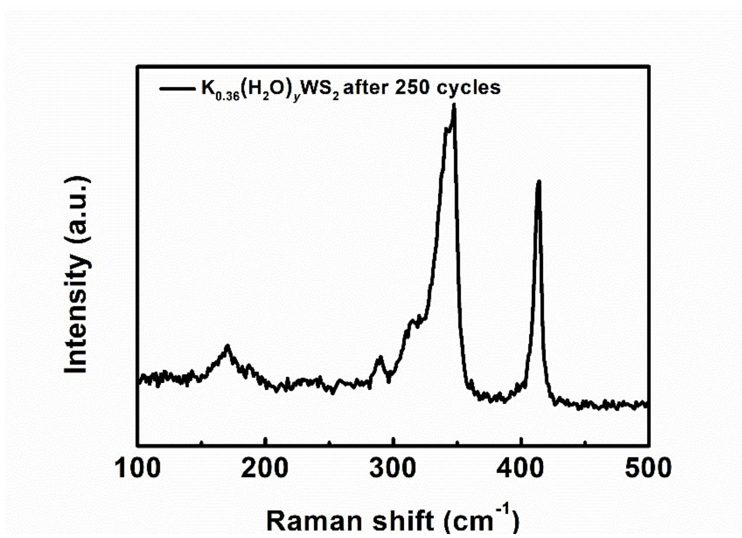

Figure S4. Raman spectrum of $K_{0.36}(H_2O)_yWS_2$ electrode after 250 cycles. The characteristic peaks at 347 and 414 cm^{-1} of 2H WS_2 were observed.

3. Supplementary tables

Table S1. ICP-OES result of $K_{0.36}(H_2O)_yWS_2$.

Element	Weight (%)	atom (%)
W	59.96	32.62
K	4.57	11.69

Table S2. EDS result for $K_{0.36}(H_2O)_yWS_2$ electrode at initial, extraction and insertion state, respectively.

State	Element	Weight (%)	atom (%)	K/W
Initial state	W	5.35	27.81	0.36
	K	68.38	10.14	
Extraction state	W	74.02	32.82	0
	K	0	0	
Insertion state	W	67.97	27.71	0.42
	K	6.13	11.75	

Table S3. Crystal data and structure refinement for $K_{0.36}(H_2O)_yWS_2$.

Crystal data	
Chemical formula	$K_{0.36}WS_2$
M_r	262.05
Crystal system, space group	Monoclinic, $P2_1/m$
Temperature (K)	298
a, *b*, *c* (Å)	5.693 (2), 3.2498 (11), 9.410 (3)
β (°)	100.317 (7)
V (Å^3)	171.28 (10)
Z	2
Radiation type	Mo $K\alpha$
μ (mm^{-1})	35.08
Crystal size (mm)	0.07 × 0.05 × 0.03
Data collection	

Diffractometer	CCD area detector
Absorption correction	Multi-scan
T_{min}, T_{max}	0.140, 0.349
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	1459, 439, 419
R_{int}	0.033
$(\sin \theta/\lambda)_{max}$ (Å^{-1})	0.649
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.031, 0.081, 1.12
No. of reflections	439
No. of parameters	33
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	1.30, -1.59

$^aR_1 = \sum||F_o| - |F_c||/\sum|F_o|$. $^bwR_2 = \{\sum[w(F_o^2 - F_c^2)^2]/\sum[w(F_o^2)^2]\}^{1/2}$, $w = 1/[\sigma^2(F_o^2) + (0.1000P)^2]$, where $P = (F_o^2 + 2F_c^2)/3$.

Table S4. Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2) for $K_{0.36}(H_2O)_yWS_2$.

	x	y	z	U_{iso}*/U_{eq}	Occ. (<1)
W	0.30202 (6)	0.7500	0.49371 (4)	0.0259 (2)	
S1	0.6435 (5)	0.7500	0.6855 (3)	0.0279 (6)	
S2	0.1253 (5)	0.2500	0.6412 (3)	0.0267 (5)	
K1	0.316 (10)	0.7500	0.958 (4)	0.129 (9)	0.177 (14)
K2	0.148 (10)	0.7500	0.964 (4)	0.129 (9)	0.183 (14)

Table S5. Atomic displacement parameters (Å^2) for $K_{0.36}(H_2O)_yWS_2$.

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
W	0.0200 (3)	0.0262 (3)	0.0314 (3)	0.000	0.0046 (2)	0.000
S1	0.0246 (13)	0.0246 (12)	0.0343 (13)	0.000	0.0044 (11)	0.000
S2	0.0230 (12)	0.0244 (12)	0.0329 (13)	0.000	0.0056 (10)	0.000
K1	0.115 (18)	0.22 (2)	0.046 (7)	0.000	0.010 (16)	0.000
K2	0.115 (18)	0.22 (2)	0.046 (7)	0.000	0.009 (16)	0.000

Table S6. Geometric parameters (Å, º) for $K_{0.36}(H_2O)_yWS_2$.

W—S1^{i}	2.402 (2)	S2—K2	3.42 (3)
W—S1^{ii}	2.402 (2)	K1—K1^{vi}	2.66 (8)
W—S1	2.404 (3)	K1—K1^{vii}	2.66 (8)
W—S2^{iii}	2.466 (2)	K1—K1^{iii}	3.2498 (12)
W—S2	2.466 (2)	K1—K1^{viii}	3.2498 (11)
W—S2^{iv}	2.534 (3)	K1—S2^{iii}	3.40 (3)
W—W^{ii}	2.7641 (9)	K1—S1^{vi}	3.70 (4)
W—W^{i}	2.7641 (9)	K1—S1^{vii}	3.70 (4)
S1—W^{i}	2.402 (2)	K1—K1^{ix}	4.15 (11)
S1—W^{ii}	2.402 (2)	K1—K1^{x}	4.15 (11)
S1—K1	3.43 (5)	K2—K2^{ix}	2.52 (8)
S1—K2^{v}	3.52 (5)	K2—K2^{x}	2.52 (8)
S1—K2^{vi}	3.68 (3)	K2—K2^{iii}	3.2498 (12)
S1—K2^{vii}	3.68 (3)	K2—K2^{viii}	3.2498 (12)
S1—K1^{vi}	3.70 (4)	K2—S2^{iii}	3.42 (3)
S1—K1^{vii}	3.70 (4)	K2—S1^{xi}	3.52 (5)
S2—W^{viii}	2.466 (2)	K2—S1^{vi}	3.68 (3)
S2—W^{iv}	2.534 (3)	K2—S1^{vii}	3.68 (3)
S2—K1^{viii}	3.40 (3)	K2—K2^{vi}	4.26 (11)
S2—K1	3.40 (3)	K2—K2^{vii}	4.26 (11)
S2—K2^{viii}	3.42 (3)		
S1^{i}—W—S1^{ii}	85.15 (11)	S2—K1—S1	58.3 (7)
S1^{i}—W—S1	109.78 (8)	S2^{iii}—K1—S1	58.3 (7)
S1^{ii}—W—S1	109.78 (8)	K1^{vi}—K1—S1^{vi}	62.9 (15)
S1^{i}—W—S2^{iii}	163.37 (11)	K1^{vii}—K1—S1^{vi}	94.8 (17)
S1^{ii}—W—S2^{iii}	93.82 (7)	K1^{iii}—K1—S1^{vi}	63.9 (3)
S1—W—S2^{iii}	86.22 (10)	K1^{viii}—K1—S1^{vi}	116.1 (3)
S1^{i}—W—S2	93.82 (7)	S2—K1—S1^{vi}	164.6 (19)
S1^{ii}—W—S2	163.37 (11)	S2^{iii}—K1—S1^{vi}	123.0 (5)
S1—W—S2	86.22 (10)	S1—K1—S1^{vi}	136.4 (13)

S2^{iii}—W—S2	82.44 (10)	K1^{vi}—K1—S1^{vii}	94.8 (17)
S1^{i}—W—S2^{iv}	83.17 (10)	K1^{vii}—K1—S1^{vii}	62.9 (15)
S1^{ii}—W—S2^{iv}	83.17 (9)	K1^{iii}—K1—S1^{vii}	116.1 (3)
S1—W—S2^{iv}	161.93 (12)	K1^{viii}—K1—S1^{vii}	63.9 (3)
S2^{iii}—W—S2^{iv}	80.23 (9)	S2—K1—S1^{vii}	123.0 (5)
S2—W—S2^{iv}	80.23 (9)	S2^{iii}—K1—S1^{vii}	164.6 (19)
S1^{i}—W—W^{ii}	102.77 (7)	S1—K1—S1^{vii}	136.4 (13)
S1^{ii}—W—W^{ii}	54.94 (7)	S1^{vi}—K1—S1^{vii}	52.2 (6)
S1—W—W^{ii}	54.85 (6)	K1^{vi}—K1—K1^{ix}	148 (3)
S2^{iii}—W—W^{ii}	90.02 (6)	K1^{vii}—K1—K1^{ix}	111.6 (15)
S2—W—W^{ii}	140.82 (7)	K1^{iii}—K1—K1^{ix}	113.0 (6)
S2^{iv}—W—W^{ii}	136.35 (4)	K1^{viii}—K1—K1^{ix}	67.0 (7)
S1^{i}—W—W^{i}	54.94 (7)	S2—K1—K1^{ix}	80.1 (10)
S1^{ii}—W—W^{i}	102.77 (7)	S2^{iii}—K1—K1^{ix}	101.6 (15)
S1—W—W^{i}	54.85 (6)	S1—K1—K1^{ix}	138.4 (13)
S2^{iii}—W—W^{i}	140.82 (7)	S1^{vi}—K1—K1^{ix}	85.2 (15)
S2—W—W^{i}	90.02 (6)	S1^{vii}—K1—K1^{ix}	64.6 (10)
S2^{iv}—W—W^{i}	136.35 (4)	K1^{vi}—K1—K1^{x}	111.6 (15)
W^{ii}—W—W^{i}	72.01 (3)	K1^{vii}—K1—K1^{x}	148 (3)
W^{i}—S1—W^{ii}	85.15 (11)	K1^{iii}—K1—K1^{x}	67.0 (6)
W^{i}—S1—W	70.22 (8)	K1^{viii}—K1—K1^{x}	113.0 (6)
W^{ii}—S1—W	70.22 (8)	S2—K1—K1^{x}	101.6 (15)
W^{i}—S1—K1	132.9 (3)	S2^{iii}—K1—K1^{x}	80.1 (10)
W^{ii}—S1—K1	132.9 (3)	S1—K1—K1^{x}	138.4 (13)
W—S1—K1	95.0 (7)	S1^{vi}—K1—K1^{x}	64.6 (10)
W^{i}—S1—K2^{v}	109.3 (6)	S1^{vii}—K1—K1^{x}	85.2 (15)
W^{ii}—S1—K2^{v}	109.3 (6)	K1^{ix}—K1—K1^{x}	46.1 (13)
W—S1—K2^{v}	179.4 (8)	K2^{ix}—K2—K2^{x}	80 (3)
W^{i}—S1—K2^{vi}	150.1 (7)	K2^{ix}—K2—K2^{iii}	130.1 (16)
W^{ii}—S1—K2^{vi}	105.7 (4)	K2^{x}—K2—K2^{iii}	49.9 (16)
W—S1—K2^{vi}	139.5 (8)	K2^{ix}—K2—K2^{viii}	49.9 (16)
K2^{v}—S1—K2^{vi}	40.9 (13)	K2^{x}—K2—K2^{viii}	130.1 (16)

W^{i}—S1—K2^{vii}	105.7 (4)	K2^{iii}—K2—K2^{viii}	179.999 (19)
W^{ii}—S1—K2^{vii}	150.1 (7)	K2^{ix}—K2—S2	90.8 (13)
W—S1—K2^{vii}	139.5 (8)	K2^{x}—K2—S2	129 (3)
K2^{v}—S1—K2^{vii}	40.9 (13)	K2^{iii}—K2—S2	118.3 (3)
K2^{vi}—S1—K2^{vii}	52.4 (5)	K2^{viii}—K2—S2	61.7 (3)
W^{i}—S1—K1^{vi}	160.0 (4)	K2^{ix}—K2—S2^{iii}	129 (3)
W^{ii}—S1—K1^{vi}	110.2 (4)	K2^{x}—K2—S2^{iii}	90.8 (13)
W—S1—K1^{vi}	126.3 (8)	K2^{iii}—K2—S2^{iii}	61.7 (3)
K1—S1—K1^{vi}	43.6 (13)	K2^{viii}—K2—S2^{iii}	118.3 (3)
W^{i}—S1—K1^{vii}	110.2 (4)	S2—K2—S2^{iii}	56.7 (6)
W^{ii}—S1—K1^{vii}	160.0 (4)	K2^{ix}—K2—S1^{xi}	73 (2)
W—S1—K1^{vii}	126.3 (8)	K2^{x}—K2—S1^{xi}	73 (2)
K1—S1—K1^{vii}	43.6 (13)	K2^{iii}—K2—S1^{xi}	90.000 (6)
K1^{vi}—S1—K1^{vii}	52.2 (6)	K2^{viii}—K2—S1^{xi}	90.000 (8)
W^{viii}—S2—W	82.44 (10)	S2—K2—S1^{xi}	56.3 (6)
W^{viii}—S2—W^{iv}	99.77 (9)	S2^{iii}—K2—S1^{xi}	56.3 (6)
W—S2—W^{iv}	99.77 (9)	K2^{ix}—K2—S1^{vi}	99.4 (18)
W^{viii}—S2—K1^{viii}	94.6 (7)	K2^{x}—K2—S1^{vi}	66.1 (14)
W—S2—K1^{viii}	135.3 (9)	K2^{iii}—K2—S1^{vi}	63.8 (2)
W^{iv}—S2—K1^{viii}	124.5 (9)	K2^{viii}—K2—S1^{vi}	116.2 (2)
W^{viii}—S2—K1	135.3 (9)	S2—K2—S1^{vi}	163.6 (18)
W—S2—K1	94.6 (7)	S2^{iii}—K2—S1^{vi}	122.7 (5)
W^{iv}—S2—K1	124.5 (9)	S1^{xi}—K2—S1^{vi}	139.1 (13)
K1^{viii}—S2—K1	57.1 (6)	K2^{ix}—K2—S1^{vii}	66.1 (14)
W^{viii}—S2—K2^{viii}	103.4 (5)	K2^{x}—K2—S1^{vii}	99.4 (18)
W—S2—K2^{viii}	149.0 (8)	K2^{iii}—K2—S1^{vii}	116.2 (2)
W^{iv}—S2—K2^{viii}	108.9 (10)	K2^{viii}—K2—S1^{vii}	63.8 (2)
W^{viii}—S2—K2	149.0 (8)	S2—K2—S1^{vii}	122.7 (5)
W—S2—K2	103.4 (5)	S2^{iii}—K2—S1^{vii}	163.6 (18)
W^{iv}—S2—K2	108.9 (9)	S1^{xi}—K2—S1^{vii}	139.1 (13)
K2^{viii}—S2—K2	56.7 (6)	S1^{vi}—K2—S1^{vii}	52.4 (5)
K1^{vi}—K1—K1^{vii}	75 (3)	K2^{ix}—K2—K2^{vi}	149 (3)

$K1^{vi}$—K1—$K1^{iii}$	52.3 (14)	$K2^{x}$—K2—$K2^{vi}$	111.4 (14)
$K1^{vii}$—K1—$K1^{iii}$	127.7 (13)	$K2^{iii}$—K2—$K2^{vi}$	67.6 (6)
$K1^{vi}$—K1—$K1^{viii}$	127.7 (13)	$K2^{viii}$—K2—$K2^{vi}$	112.4 (6)
$K1^{vii}$—K1—$K1^{viii}$	52.3 (14)	S2—K2—$K2^{vi}$	102.0 (16)
$K1^{iii}$—K1—$K1^{viii}$	180.0	$S2^{iii}$—K2—$K2^{vi}$	81.2 (11)
$K1^{vi}$—K1—S2	132 (3)	$S1^{xi}$—K2—$K2^{vi}$	137.5 (13)
$K1^{vii}$—K1—S2	94.5 (14)	$S1^{vi}$—K2—$K2^{vi}$	62.9 (8)
$K1^{iii}$—K1—S2	118.5 (3)	$S1^{vii}$—K2—$K2^{vi}$	83.2 (12)
$K1^{viii}$—K1—S2	61.4 (3)	$K2^{ix}$—K2—$K2^{vii}$	111.4 (14)
$K1^{vi}$—K1—$S2^{iii}$	94.5 (14)	$K2^{x}$—K2—$K2^{vii}$	149 (3)
$K1^{vii}$—K1—$S2^{iii}$	132 (3)	$K2^{iii}$—K2—$K2^{vii}$	112.4 (6)
$K1^{iii}$—K1—$S2^{iii}$	61.5 (3)	$K2^{viii}$—K2—$K2^{vii}$	67.6 (6)
$K1^{viii}$—K1—$S2^{iii}$	118.5 (3)	S2—K2—$K2^{vii}$	81.2 (11)
S2—K1—$S2^{iii}$	57.1 (6)	$S2^{iii}$—K2—$K2^{vii}$	102.0 (16)
$K1^{vi}$—K1—S1	74 (2)	$S1^{xi}$—K2—$K2^{vii}$	137.5 (13)
$K1^{vii}$—K1—S1	74 (2)	$S1^{vi}$—K2—$K2^{vii}$	83.2 (12)
$K1^{iii}$—K1—S1	90.000 (6)	$S1^{vii}$—K2—$K2^{vii}$	62.9 (8)
$K1^{viii}$—K1—S1	90.000 (3)	$K2^{vi}$—K2—$K2^{vii}$	44.8 (12)

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

4. References

(1) Sheldrick, G, *Acta Crystallogr., Sect C,* 2015, **71**, 3-8.