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Supporting information for article:

**Temperature-induced reversible structural phase transition and X-ray diffuse scattering in 2-amino-3-nitropyridinium hydrogen sulfate**

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**Table S1** Selected geometric parameters ( $\text{\AA}$ ,  $^\circ$ ) for **I**.

O1—H1B	0.8200	S1—O3	1.280 (10)
S1—O1	1.547 (3)	S1—O3A	1.610 (11)
S1—O2	1.563 (15)	S1—O4	1.510 (6)
S1—O2A	1.28 (2)	S1—O4A	1.435 (7)
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O1—S1—O2	105.6 (9)	O3—S1—O2	115.3 (9)
O1—S1—O3A	103.4 (6)	O3—S1—O4	118.7 (6)
O4—S1—O1	98.5 (3)	O2A—S1—O1	111.8 (13)
O4—S1—O2	105.5 (7)	O2A—S1—O4A	121.8 (11)
O4A—S1—O1	108.2 (3)	O2A—S1—O3A	108.3 (12)
O4A—S1—O3A	101.3 (6)	S1—O1—H1B	109.5
O3—S1—O1	111.3 (6)		
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**Table S2** Selected hydrogen-bond parameters for **I**.

$D—H\cdots A$	$D—H$ ( $\text{\AA}$ )	$H\cdots A$ ( $\text{\AA}$ )	$D\cdots A$ ( $\text{\AA}$ )	$D—H\cdots A$ ( $^\circ$ )
N2—H2A $\cdots$ O3	0.86	2.04	2.897 (13)	177.1
N2—H2B $\cdots$ O11	0.86	2.12	2.682 (14)	122.4
N2—H2B $\cdots$ O2 <sup>i</sup>	0.86	2.44	3.01 (2)	123.8
N1—H1 $\cdots$ O2	0.86	1.85	2.704 (15)	170.5
C6—H6 $\cdots$ O3 <sup>ii</sup>	0.93	2.37	3.207 (13)	148.9
O1—H1B $\cdots$ O4 <sup>iii</sup>	0.82	1.85	2.652 (7)	164.3
O1—H1B $\cdots$ O4A <sup>iii</sup>	0.82	1.83	2.612 (7)	160.1
N2A—H2AA $\cdots$ O3A	0.86	2.13	2.923 (12)	153.5
N2A—H2AB $\cdots$ O11A	0.86	2.11	2.676 (17)	123.1
N1A—H1A $\cdots$ O3A	0.86	2.12	2.916 (18)	154.5
N1A—H1A $\cdots$ O2A	0.86	2.35	3.08 (32)	142.0

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

**Table S3** Selected geometric parameters ( $\text{\AA}$ ,  $^\circ$ ) for **II**.

S1—O11	1.563 (2)	S3—O31	1.563 (2)
S1—O14	1.454 (2)	S3—O32	1.464 (2)
S1—O12	1.462 (2)	S3—O33	1.444 (2)
S1—O13	1.453 (2)	S3—O34	1.458 (2)
O11—H11A	0.8400	O31—H31A	0.8400
S2—O21	1.556 (2)	S4—O41	1.575 (2)
S2—O24	1.450 (2)	S4—O42	1.451 (3)
S2—O22	1.446 (3)	S4—O43	1.459 (2)
S2—O23	1.461 (2)	S4—O44	1.445 (2)
O21—H21A	0.8400	O41—H41A	0.8400
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O14—S1—O11	103.57 (13)	O32—S3—O31	107.10 (13)
O14—S1—O12	111.73 (13)	O33—S3—O31	108.60 (13)
O12—S1—O11	107.26 (14)	O33—S3—O32	111.66 (13)
O13—S1—O11	107.74 (13)	O33—S3—O34	114.21 (14)
O13—S1—O14	113.79 (14)	O34—S3—O31	102.86 (13)
O13—S1—O12	112.10 (14)	O34—S3—O32	111.74 (14)
S1—O11—H11A	109.5	S3—O31—H31A	109.5
O24—S2—O21	103.32 (14)	O42—S4—O41	107.25 (14)
O24—S2—O23	111.54 (14)	O42—S4—O43	110.79 (14)
O22—S2—O21	107.88 (15)	O43—S4—O41	106.77 (14)
O22—S2—O24	114.97 (15)	O44—S4—O41	104.43 (14)
O22—S2—O23	111.21 (14)	O44—S4—O42	115.18 (15)
O23—S2—O21	107.28 (14)	O44—S4—O43	111.78 (14)
S2—O21—H21A	109.5	S4—O41—H41A	109.5

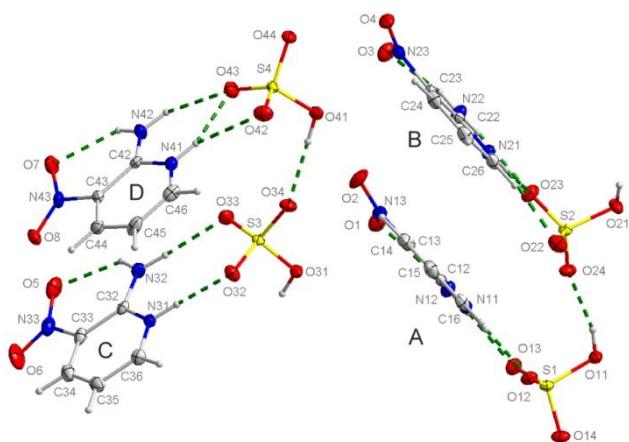
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**Table S4** Selected hydrogen-bond parameters for **II**.

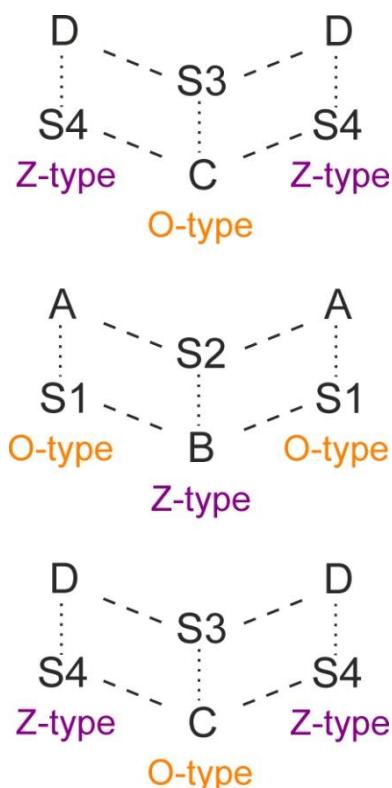
$D-H\cdots A$	$D-H$ (Å)	$H\cdots A$ (Å)	$D\cdots A$ (Å)	$D-H\cdots A$ (°)
N11—H11···O12	0.88	1.84	2.710 (3)	171.3
N12—H12A···O13	0.88	2.04	2.922 (3)	177.8
N12—H12B···O1	0.88	2.09	2.691 (3)	124.6
N12—H12B···O22 <sup>i</sup>	0.88	2.12	2.808 (4)	135.1
C16—H16···O24 <sup>ii</sup>	0.95	2.43	3.186 (4)	135.8
N21—H21···O22	0.88	2.48	3.187 (4)	137.5
N21—H21···O23	0.88	1.93	2.767 (4)	157.4
N22—H22A···O23	0.88	2.21	2.969 (3)	143.7
N22—H22B···O3	0.88	2.09	2.686 (3)	124.1
N22—H22B···O14 <sup>i</sup>	0.88	2.17	2.919 (3)	143.5
C24—H24···O5 <sup>iii</sup>	0.95	2.55	3.419 (4)	152.0
C26—H26···O13 <sup>ii</sup>	0.95	2.32	3.093 (4)	137.6
N31—H31···O32	0.88	1.84	2.719 (3)	172.1
N32—H32A···O33	0.88	2.03	2.912 (3)	177.4
N32—H32B···O5	0.88	2.08	2.678 (3)	124.4
N32—H32B···O42 <sup>iv</sup>	0.88	2.13	2.816 (4)	134.5
C36—H36···O44 <sup>iii</sup>	0.95	2.44	3.223 (4)	139.9
N41—H41···O42	0.88	2.43	3.141 (4)	138.2
N41—H41···O43	0.88	1.95	2.777 (4)	155.6
N42—H42A···O32 <sup>iv</sup>	0.88	2.37	2.875 (3)	117.1
N42—H42A···O43	0.88	2.20	2.955 (3)	143.3
N42—H42B···O7	0.88	2.08	2.678 (3)	124.5
N42—H42B···O34 <sup>iv</sup>	0.88	2.19	2.950 (3)	144.0
C46—H46···O33 <sup>iii</sup>	0.95	2.32	3.106 (4)	139.4
O11—H11A···O24	0.84	1.80	2.599 (3)	159.2

O21—H21A···O14 <sup>v</sup>	0.84	1.87	2.696 (3)	166.3
O31—H31A···O44 <sup>vi</sup>	0.84	1.81	2.628 (3)	165.1
O41—H41A···O34	0.84	1.84	2.646 (3)	159.8

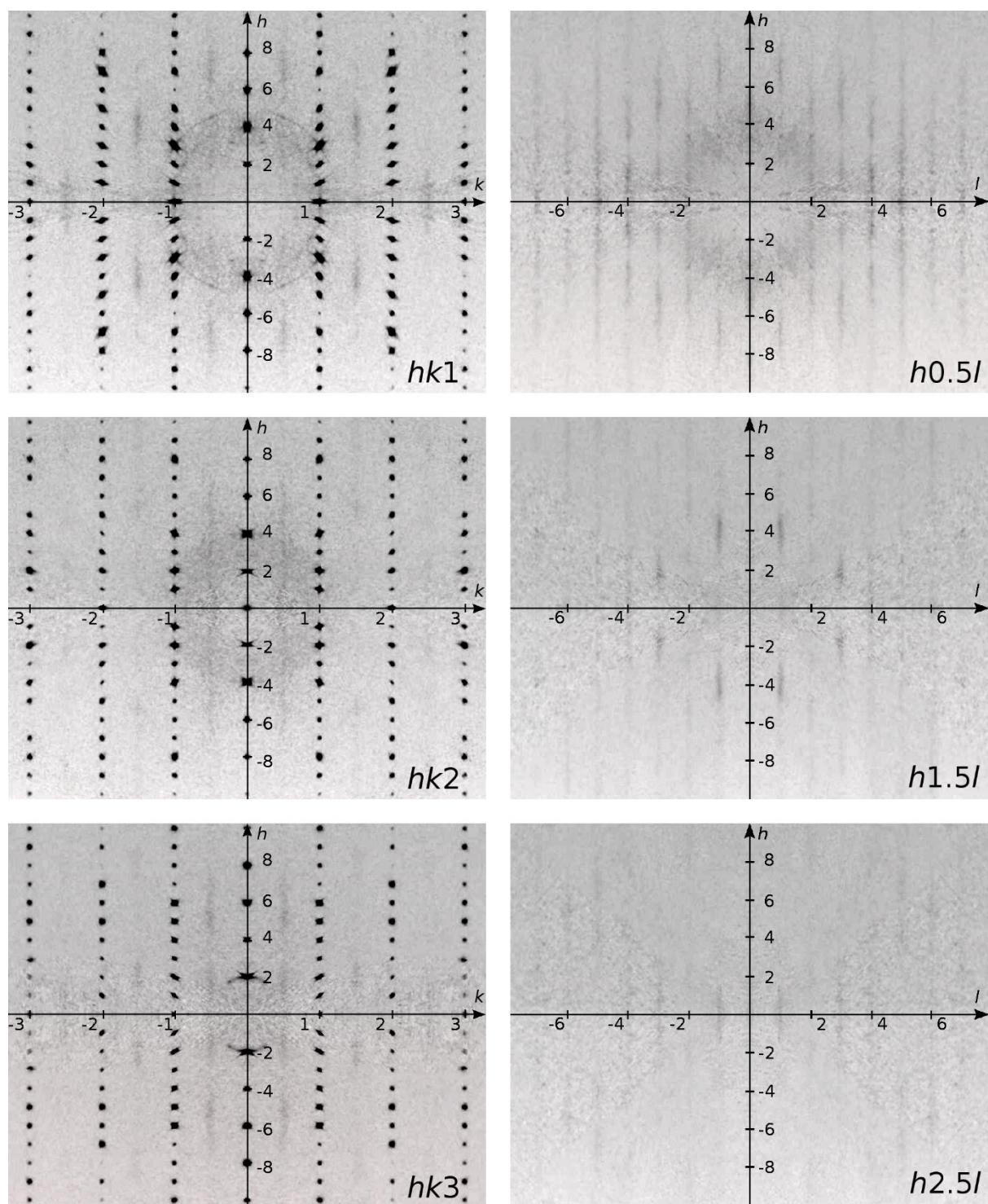
Symmetry code(s): (i)  $-x+1, y-1/2, -z+1$ ; (ii)  $-x+1, y+1/2, -z+1$ ; (iii)  $-x+1, y+1/2, -z$ ; (iv)  $-x+1, y-1/2, -z$ ; (v)  $x+1, y, z$ ; (vi)  $x-1, y, z$ .



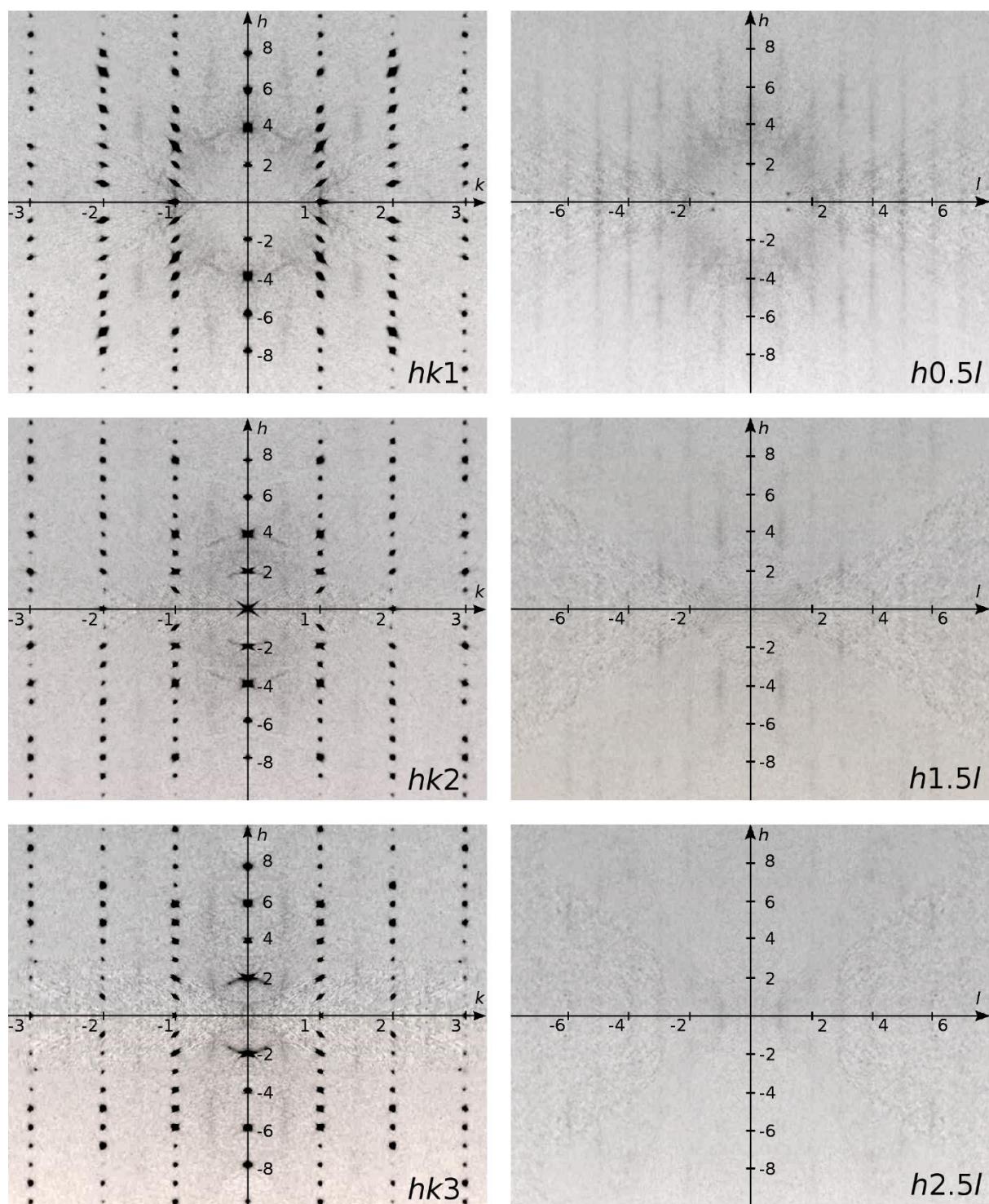
**Figure S1** The asymmetric unit of **II**, showing the atom-numbering scheme and hydrogen-bonding interactions (green dashed lines). Displacement ellipsoids are drawn at the 50 % probability level.



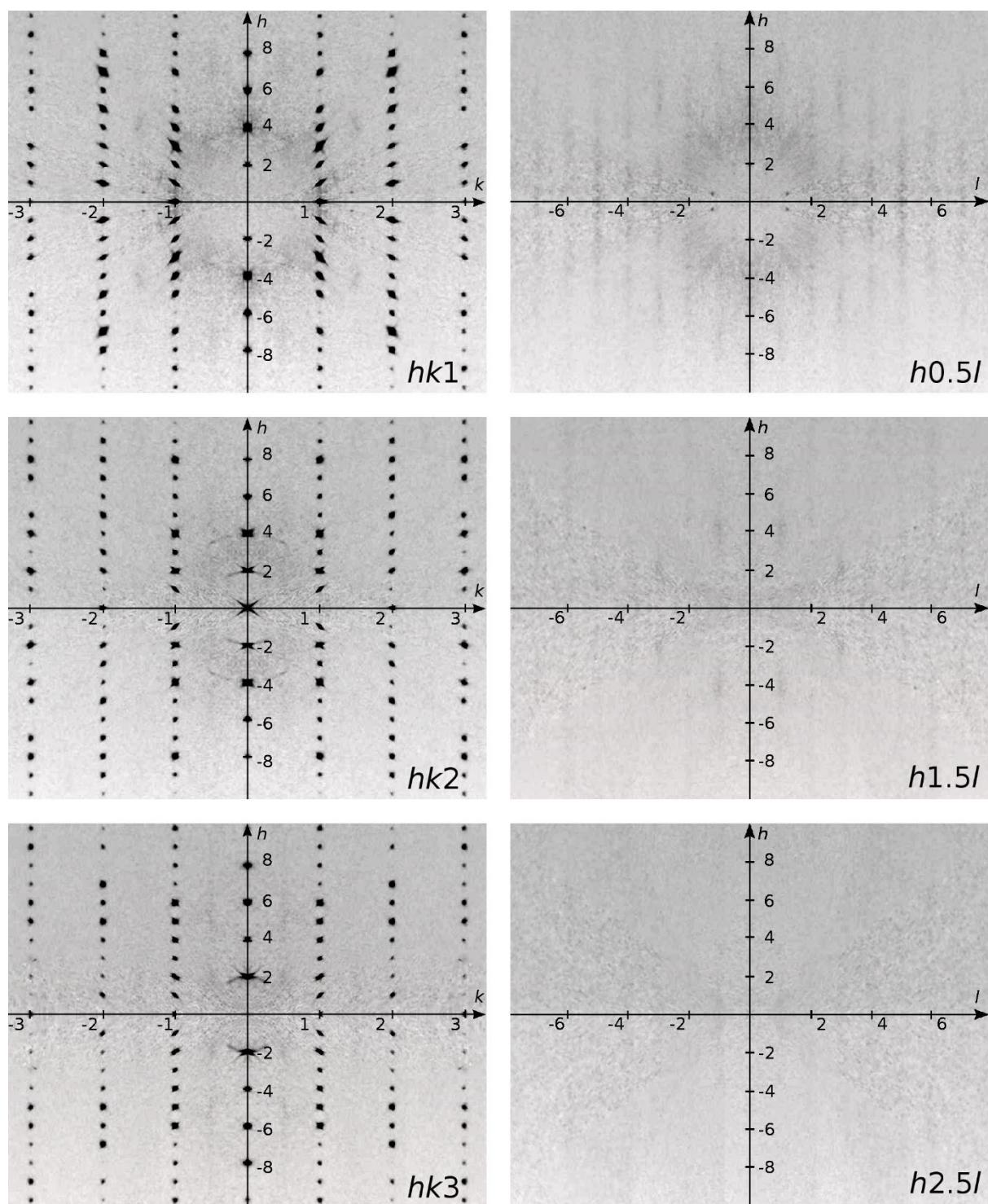
**Figure S2** The mutual arrangement of two types of pairs (Z- and O-types, black dotted lines stand for the hydrogen bonds within the pairs), which consist of four kinds of symmetrically nonequivalent 2A3NP cations (denoted as A, B, C and D) and four types of  $\text{HSO}_4^-$  anions (labeled S1, S2, S3 and S4) in one layer of the phase **II**. The H-bonds between different pairs are shown as black dashed lines.



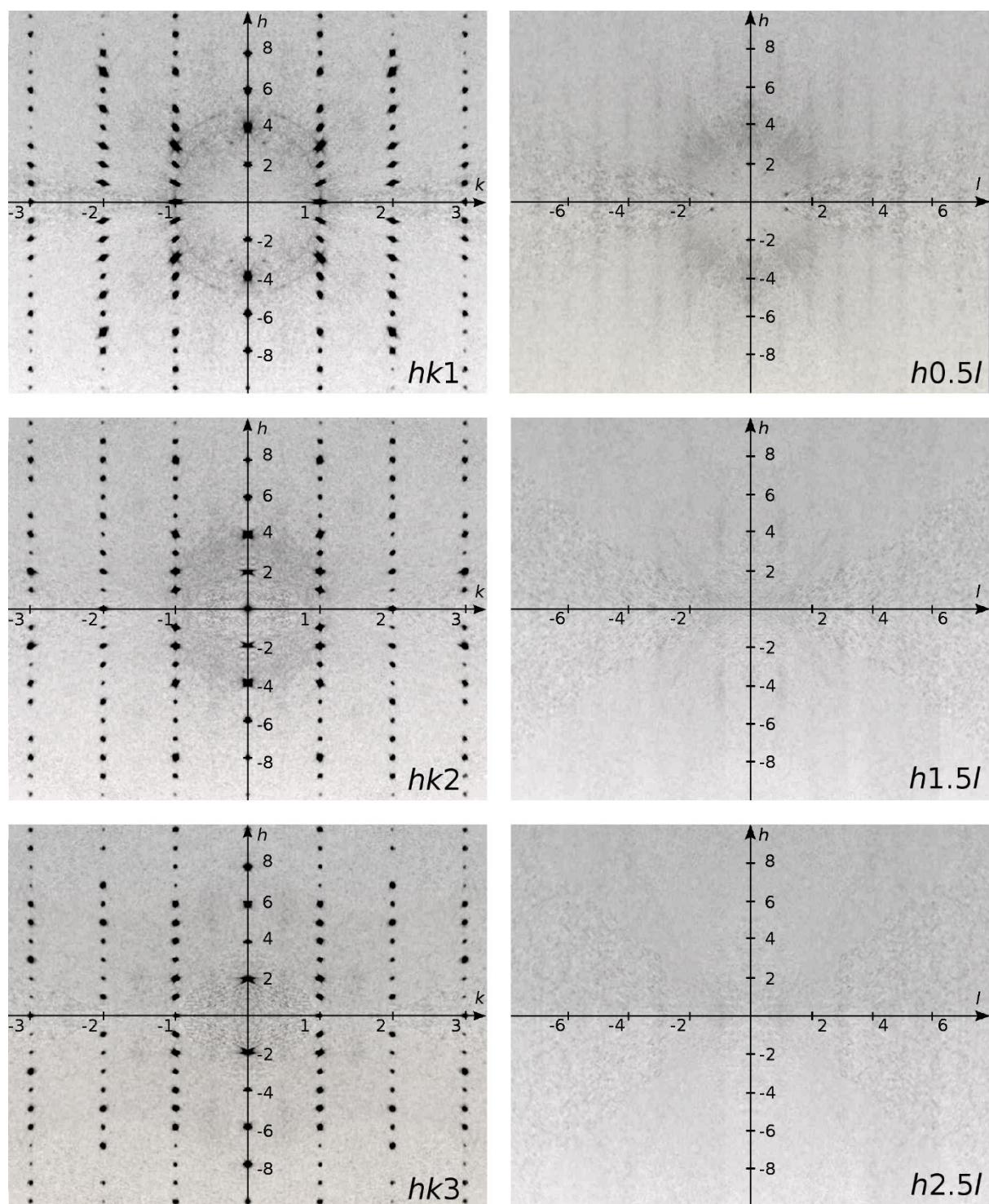
**Figure S3** Experimental sections of the reciprocal space of 2A3NP-HS in phase **I** ( $T = 315\text{ K}$ ).



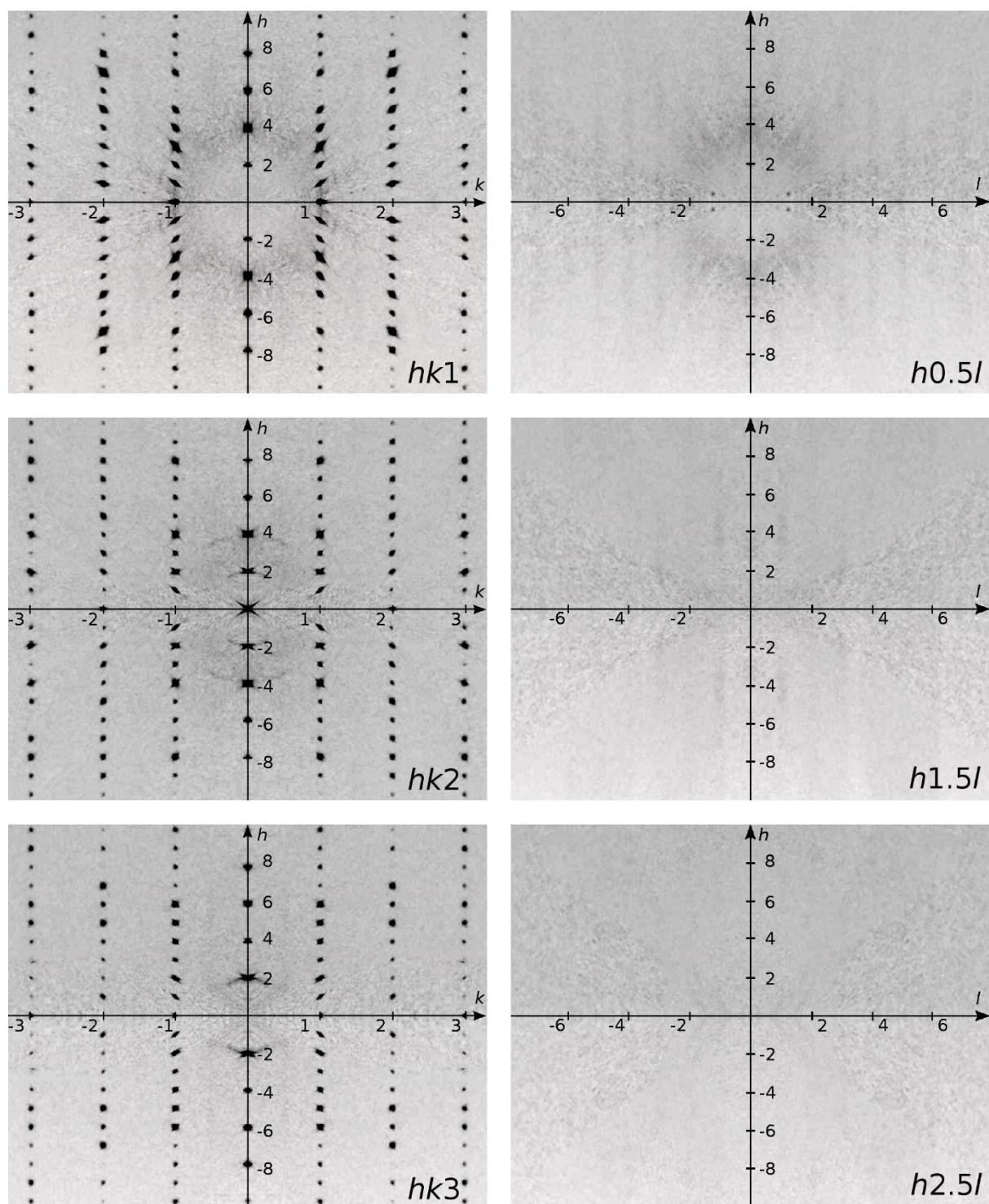
**Figure S4** Experimental sections of the reciprocal space of 2A3NP-HS in phase **I** (T = 325 K).



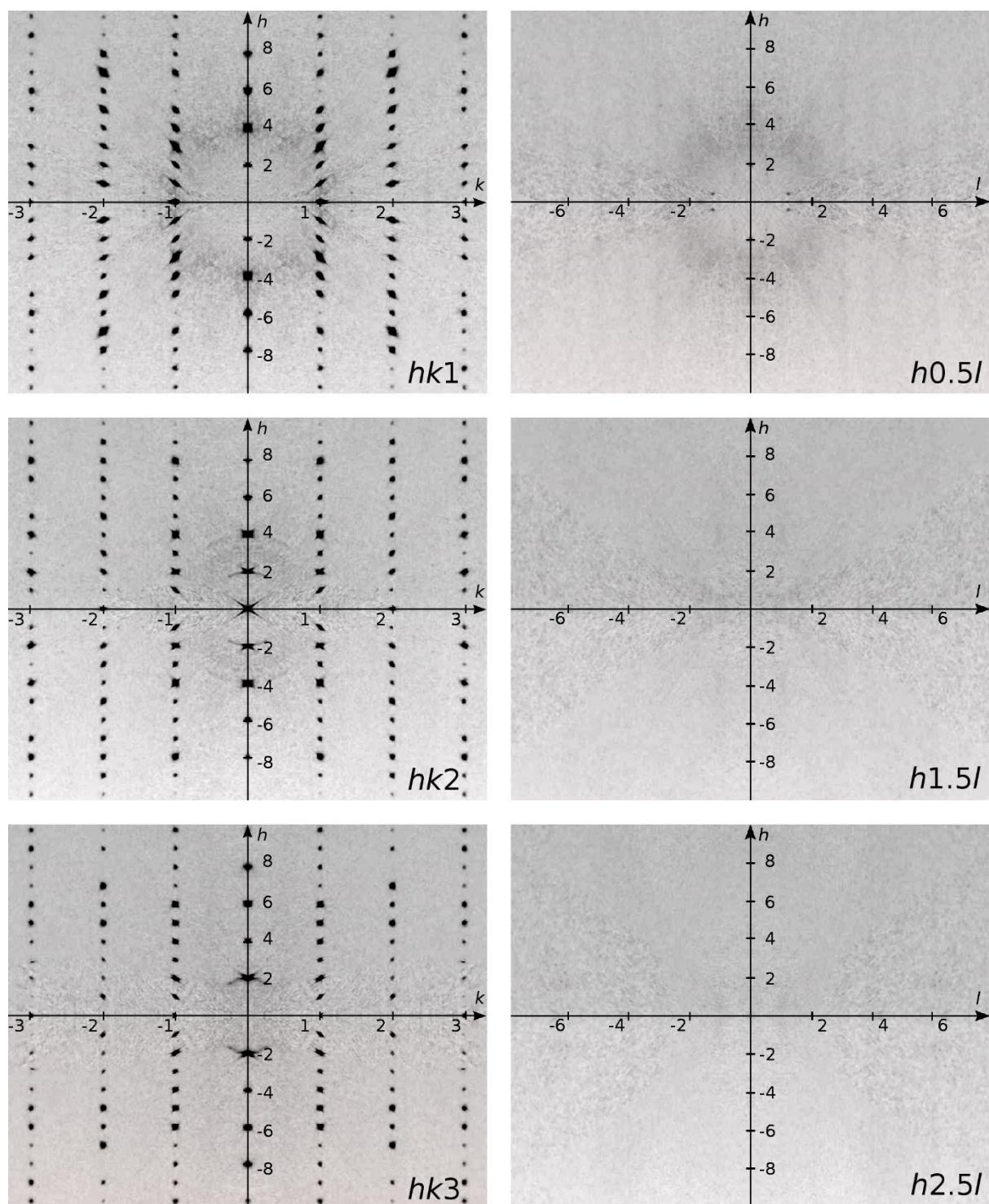
**Figure S5** Experimental sections of the reciprocal space of 2A3NP-HS in phase **I** ( $T = 335\text{ K}$ ).



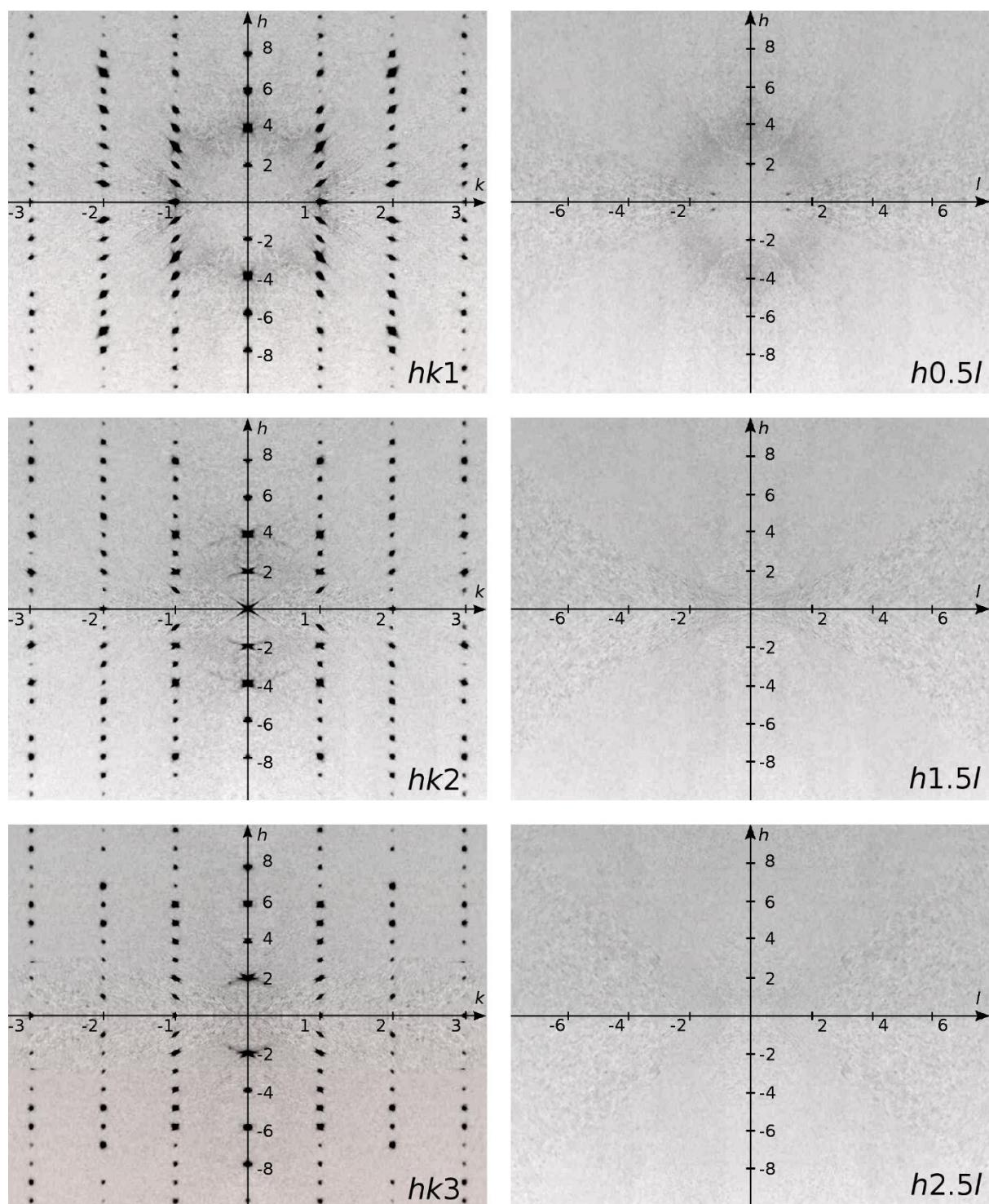
**Figure S6** Experimental sections of the reciprocal space of 2A3NP-HS in phase **I** ( $T = 345\text{ K}$ ).



**Figure S7** Experimental sections of the reciprocal space of 2A3NP-HS in phase **I** ( $T = 355\text{ K}$ ).



**Figure S8** Experimental sections of the reciprocal space of 2A3NP-HS in phase **I** ( $T = 365\text{ K}$ ).



**Figure S9** Experimental sections of the reciprocal space of 2A3NP-HS in phase **I** ( $T = 375\text{ K}$ ).