

**Table 33B-1-001.** K<sub>1-x</sub>(NH<sub>4</sub>)<sub>x</sub>H<sub>2</sub>PO<sub>4</sub> (KDP-ADP, x = 0.89). Unit cell parameters in phase I [94Bou]. Parameter: *T*.

<i>T</i> [K]	295	192	164	154
<i>a</i> = <i>b</i> [Å]	7.4971(12)	7.4731(16)	7.4707(12)	7.4694(14)
<i>c</i> [Å]	7.471(3)	7.462(3)	7.456(3)	7.456(3)

**Table 33B-1-002.** K<sub>1-x</sub>(NH<sub>4</sub>)<sub>x</sub>H<sub>2</sub>PO<sub>4</sub> (KDP-ADP, x = 0.23). Unit cell parameters, interatomic distances and bond angles [91Ono]. As to the superscripts a, b, c, see Table 33B-1-006.

<i>T</i> [K]	91.8	131.3	215.3	293
<i>a</i> = <i>b</i> [Å]	7.4270(24)	7.4294(23)	7.4419(21)	7.4540(23)
<i>c</i> [Å]	7.0461(23)	7.0545(19)	7.0724(19)	7.0782(29)
Distances [Å]				
P–O	1.5393(22)	1.5397(24)	1.5399(25)	1.5346(13)
K–O <sup>a</sup>	2.8212(24)	2.8222(25)	2.8321(27)	2.8429(16)
K–O	2.9351(26)	2.9386(28)	2.9418(29)	2.9501(17)
O–O <sup>b</sup>	2.4828(30)	2.4851(31)	2.4881(34)	2.4921(18)
Angles (degree)				
O–P–O <sup>c</sup>	110.71(13)	110.66(13)	110.34(14)	110.69(7)
O–K–O <sup>c</sup>	51.12(6)	51.05(6)	50.89(7)	50.67(4)

**Table 33B-1-003.** K<sub>1-x</sub>(NH<sub>4</sub>)<sub>x</sub>H<sub>2</sub>PO<sub>4</sub> (KDP-ADP). Unit cell parameters for several x [51Nit]. *T* = 15...25 °C.

<i>x</i>	<i>a<sub>x</sub></i>	<i>c<sub>x</sub></i>	( <i>a<sub>x</sub></i> – <i>a<sub>0</sub></i> )/ <i>a<sub>0</sub></i>	( <i>c<sub>x</sub></i> – <i>c<sub>0</sub></i> )/ <i>c<sub>0</sub></i>
0	7.449	6.973	0	0
1/5	7.47	7.15...7.25	0.0035	0.033
2/3	7.482	7.375	0.0044	0.058
4/5	7.48	7.39	0.0048	0.060
1	7.492	7.522	0.0058	0.079

**Table 33B-1-004.** K<sub>1-x</sub>(NH<sub>4</sub>)<sub>x</sub>H<sub>2</sub>PO<sub>4</sub> (KDP-ADP, x = 0.89). Fractional coordinates and temperature parameters in phase I [94Bou].  $U_{ij}$  [Å<sup>2</sup>] is defined by Eq. (d) in Introduction.

Atom	x	y	z	$U_{11}$	$U_{22}$	$U_{33}$	$U_{12}$	$U_{13}$	$U_{23}$
154 K	N	0.0000	0.0000	0.5000	0.0169(2)	0.0169(2)	0.0135(4)	0.0000	0.0000
	K	0.0000	0.0000	0.5000	0.0185(4)	0.0185(4)	0.0125(6)	0.0000	0.0000
	P	0.0000	0.0000	0.0000	0.00725(5)	0.00725(5)	0.0186(1)	0.0000	0.0000
	O	0.14767(5)	0.08446(5)	0.11662(7)	0.0124(1)	0.0110(1)	0.0204(1)	0.0018(1)	-0.0034(1)
	H(O)	0.151(4)	0.238(1)	0.120(1)	0.046(9)				
	H(N)	0.089(2)	0.012(2)	0.565(1)	0.019(2)				
164 K	N	0.0000	0.0000	0.5000	0.0183(2)	0.0183(2)	0.0134(4)	0.0000	0.0000
	K	0.0000	0.0000	0.5000	0.0187(4)	0.0187(4)	0.0134(6)	0.0000	0.0000
	P	0.0000	0.0000	0.0000	0.00763(5)	0.00763(5)	0.0190(1)	0.0000	0.0000
	O	0.14759(5)	0.08437(5)	0.11663(7)	0.0131(1)	0.0114(1)	0.0213(2)	0.0021(1)	-0.0037(1)
	H(O)	0.154(4)	0.243(1)	0.121(1)	0.056(1)				
	H(N)	0.097(2)	0.014(2)	0.568(1)	0.017(2)				
192 K	N	0.0000	0.0000	0.5000	0.0198(2)	0.0198(2)	0.0155(4)	0.0000	0.0000
	K	0.0000	0.0000	0.5000	0.0228(4)	0.0228(4)	0.0160(7)	0.0000	0.0000
	P	0.0000	0.0000	0.0000	0.00887(5)	0.00887(5)	0.0210(1)	0.0000	0.0000
	O	0.14738(5)	0.08445(5)	0.11663(6)	0.0150(1)	0.0132(1)	0.0233(1)	0.0022(1)	-0.0041(1)
	H(O)	0.147(3)	0.238(1)	0.119(1)	0.047(7)				
	H(N)	0.091(1)	0.010(2)	0.567(1)	0.019(2)				
295 K	N	0.0000	0.0000	0.5000	0.0285(2)	0.0285(2)	0.0231(5)	0.0000	0.0000
	K	0.0000	0.0000	0.5000	0.0342(7)	0.0342(7)	0.0222(9)	0.0000	0.0000
	P	0.0000	0.0000	0.0000	0.01341(6)	0.01341(6)	0.0260(1)	0.0000	0.0000
	O	0.14649(6)	0.08441(6)	0.11697(8)	0.0221(1)	0.0191(1)	0.0333(2)	0.0037(1)	-0.0065(1)
	H(O)	0.161(2)	0.234(3)	0.118(1)	0.071(9)				
	H(N)	0.105(2)	0.006(2)	0.568(1)	0.019(3)				

**Table 33B-1-005.** K<sub>1-x</sub>(NH<sub>4</sub>)<sub>x</sub>H<sub>2</sub>PO<sub>4</sub> (KDP-ADP, x = 0.23). Fractional coordinates and temperature parameters [91Ono]. Parameter:  $T$ .  $U_{ij}$  [ $\cdot 10^{-5} \text{ \AA}^2$ ] is defined by Eq. (d) in Introduction.

		91.8 K	131.3 K	215.3 K	293 K
P	$U_{11}$	621(61)	712(67)	1112(73)	1372(22)
	$U_{22}$	= $U_{11}$	= $U_{11}$	= $U_{11}$	= $U_{11}$
	$U_{33}$	2327(60)	2393(68)	2702(79)	2615(20)
K	$U_{11}$	1167(73)	1348(81)	1957(95)	2356(31)
	$U_{22}$	= $U_{11}$	= $U_{11}$	= $U_{11}$	= $U_{11}$
	$U_{33}$	1409(58)	1627(65)	2087(79)	2035(23)
NH <sub>4</sub>	$U_{11}$	392(746)	399(867)	1919(1083)	3837(707)
	$U_{22}$	= $U_{11}$	= $U_{11}$	= $U_{11}$	= $U_{11}$
	$U_{33}$	548(614)	696(693)	1230(843)	3093(487)
O	$x$	14902(26)	14902(27)	14828(30)	14770(12)
	$y$	8284(26)	8275(28)	8283(31)	8284(13)
	$z$	12419(35)	12414(38)	12437(40)	12329(18)
	$U_{11}$	818(75)	873(75)	1323(87)	1733(14)
	$U_{22}$	868(75)	968(84)	1447(93)	1803(14)
	$U_{33}$	2120(73)	2278(80)	2692(89)	3153(38)
	$U_{12}$	82(39)	35(42)	44(46)	110(15)
	$U_{13}$	-108(49)	-144(54)	-246(59)	-335(21)
	$U_{23}$	-130(41)	-108(45)	-228(51)	-215(19)

**Table 33B-1-006.** K<sub>1-x</sub>(NH<sub>4</sub>)<sub>x</sub>H<sub>2</sub>PO<sub>4</sub> (KDP-ADP, x = 0.89). Interatomic distances [Å] and bond angles [°] in phase I [94Bou]. Parameter: *T*.

	<i>T</i> [K]			
	154	164	192	295
P–O	1.5397(4)	1.5391(4)	1.5390(4)	1.5395(5)
O–O <sup>b)</sup>	2.5410(7)	2.5396(7)	2.5388(7)	2.5350(9)
O–O <sup>f)</sup>	2.4761(7)	2.4780(7)	2.4775(8)	2.4858(9)
O–H(O)	1.13(1)	1.15(1)	1.15(1)	1.13(2)
H(O)–O <sup>f)</sup>	1.35(1)	1.32(1)	1.33(1)	1.36(2)
H(O) H(O) <sup>f)</sup>	0.24(3)	0.18(2)	0.20(2)	0.26(4)
N–H(N)	0.83(1)	0.88(1)	0.85(1)	0.94(1)
N–O <sup>a)</sup>	3.1279(5)	3.1279(5)	3.1297(4)	2.1298(6)
N–O <sup>h)</sup>	2.8833(4)	2.8842(4)	2.8867(4)	2.9004(4)
H(N) <sup>c)</sup> –O <sup>h)</sup>	2.10(1)	2.04(2)	2.09(1)	2.01(2)
H(N)–O <sup>f)</sup>	2.65(1)	2.64(1)	2.64(1)	2.62(1)
O–P–O <sup>b)</sup>	111.21(3)	111.18(3)	111.13(3)	110.83(4)
O–P–O <sup>c)</sup>	108.61(2)	108.62(2)	108.64(2)	108.80(2)
P–O–H(O)	117(1)	119(1)	117(1)	121(1)
P–O–N	100.43(2)	100.46(2)	100.51(2)	100.69(2)
O–H(O)–O <sup>f)</sup>	173(2)	173(2)	176(2)	169(2)
H(N)–N–H(N) <sup>b)</sup>	108(1)	110(2)	108(1)	115(1)
H(N)–N–H(N) <sup>c)</sup>	110(1)	109(1)	110(1)	107(1)
N–H(N) <sup>c)</sup> –O <sup>h)</sup>	157(1)	157(1)	156(1)	157(1)
$\theta$	60.22(1)	60.24(1)	60.18(1)	60.04(1)
$\beta$	34.39(2)	34.41(2)	34.43(2)	34.58(2)

<sup>a)</sup>  $x, y, z.$       <sup>c)</sup>  $y, \frac{1}{2} + x, \frac{1}{4} - z.$ <sup>b)</sup>  $\bar{x}, \bar{y}, z.$       <sup>f)</sup>  $x, \frac{1}{2} - y, \frac{1}{4} - z.$ <sup>c)</sup>  $\bar{y}, x, \bar{z}.$       <sup>g)</sup>  $y, \frac{1}{2} + x, \frac{1}{4} + z.$ <sup>d)</sup>  $y, \bar{x}, \bar{z}.$       <sup>h)</sup>  $\bar{y}, \frac{1}{2} - x, \frac{1}{4} + z.$