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Effects of deuteration on the structure of $NH_4H_2PO_4$ crystals characterized by neutron diffraction*

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A series of deuterated ammonium dihydrogen phosphate (DADP) crystals were grown and their structures were investigated by using powder neutron diffraction method. In the entire composition range, the deuterated level in the crystals is lower compared with the aqueous growth solution. The deuterium segregation coefficient in the crystals decreases with increasing deuterium content of the solution. The deuterium content in the NH_4^+ group is higher than that in $H_2PO_4^-$ group. In addition, the variations of lattice parameters are shown here.

Keywords: DADP crystal, neutron diffraction, deuteration level

PACS: 61.05.F–, 61.50.–f, 81.10.Dn **DOI:** 10.1088/1674-1056/28/1/016103

1. Introduction

Ammonium dihydrogen phosphate (ADP) and potassium dihydrogen phosphate (KDP) crystals possess similar structures at room temperature. These compounds are important materials which have excellent nonlinear and electric-optical properties.[1,2] Moreover, these crystals are easily grown to large sizes in an aqueous solution by using the temperature reduction method.[3] A recent study has obtained a high external conversion efficiency of 85.3% from 526 nm to 263 nm at room temperature (24 °C) by using a 60% deuterated ADP crystal.[4] This result indicates that partially deuterated ADP crystals are potential UV nonlinear materials for super-large-aperture, high-energy, and high-efficiency laser systems. Nevertheless, the inhomogeneity of deuterated ADP crystals needs to be improved. Addressing this issue would require us to determine the effect of deuterium–hydrogen substitution on crystal structure.

The substitution of deuterium for hydrogen in KDP crystals has been widely studied in the past decades.[5,6] However, insufficient research efforts have been exerted to understand such substitution effects in the ADP crystal. The crystallographic structures of ADP and KDP crystals have been well examined by x-ray analysis and neutron diffraction tests at different temperatures and pressures.[7–13] At room temperature, these two isomorphs belong to the tetragonal system with the space group $D_{2d}^{12} - I\bar{4}2d$. Each phosphate group associates with an average of two near-neighbor protons to form an H_2PO_4 ion. These connections then generate a 3D hydrogen bond network. The small difference between ADP and KDP is their distinct constituent cations (NH_4^+ with a radius of 1.42 Å and K^+ with a radius of 1.33 Å, respectively[14]), which bond to $H_2PO_4^-$ anions through hydrogen bonding. However, this difference in cation causes some properties of these two crystals to vary considerably between each other. Hydrogen bonds are different between these two crystals. One type of hydrogen bond in the KDP crystal is the oxygen–oxygen interaction between phosphate groups. In the ADP crystal, another type of hydrogen bond connects the ammonium and phosphate groups. Thus the substitution of deuterium for hydrogen in the NH_4^+ and $H_2PO_4^-$ groups of the ADP crystal is more complicated than that in the KDP crystal.

The distribution coefficient of deuterium between the deuterated KDP (DKDP) crystal and its growth solution has been frequently reported in the past.[15–18] These reports stated that the crystal can grow with a given deuteration level and that crystal properties can be analyzed from a structural perspective. For partially deuterated ADP crystals, the occupancy of deuterium in the hydrogen position may differ between the cation and anion groups inside each unit cell. Moreover, the effect of growth conditions on the occupancy and distribution of deuterium is currently unknown. Neutron diffraction, which is sensitive to light elements such as hydrogen atoms, is a powerful tool to address these issues.[19]

In this paper, a series of deuterated ADP (DADP) crys-

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tals with different deuterium concentrations are grown. Then neutron powder diffraction has been performed to determine atomic positions and occupancy factors of deuterium (hydrogen) atoms. We found the dependence of lattice parameters on deuterium content and reported the distribution equation of deuterium between the growth solutions and DADP crystals. The dissimilar distributions of deuterium between the two groups are also analyzed and explained.

2. Experiment

2.1. Crystal growth

DADP crystals were grown in an aqueous solution by using the traditional temperature reduction method, as shown in Fig. 1. Deuterated solutions were prepared by dissolving extra-pure $NH_4H_2PO_4$ salt separately into heavy and deionized water with a resistivity of 18 MΩ·cm. The highest deuterated solution was prepared by recrystallizing $NH_4H_2PO_4$ from supersaturated heavy water. The solution was filtered using a 0.22 μm microporous membrane before overheating. In accordance with the crystal growth process in traditional temperature reduction, the DADP crystals began to grow from a z-cut seed with a size of 5 cm × 5 cm × 0.5 cm. This seed was also overheated before placing into the solution. Crystallization was performed at 308–323 K and under a temperature reduction rate of 0.05–0.1 K per day. The forward-stop-backward rotation mode was adopted at 30 rpm. The growth system consisted of a 5000 mL glass crystallizer placed in a water bath. The temperature of the water bath was controlled by a programmable Shimada controller (Model FP21), with an accuracy of 0.02 K.

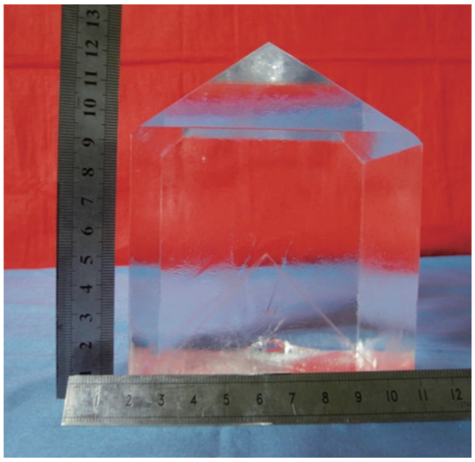


Fig. 1. DADP crystal grown in solution with deuteration level of 30% by the traditional temperature reduction method.

2.2. Neutron diffraction experiment

The neutron diffraction technique can identify the deuterium atom from its isotope hydrogen atom. To investigate the actual deuterated level in the DADP crystals, the occupancy of deuterium in the hydrogen bonds were determined by analyzing the neutron powder diffraction data using the Rietveld structural refinement method. Neutron diffraction experiments were performed on a neutron powder diffraction analyzer at the Institute of Nuclear Physics and Chemistry, China Academy of Engineering Physics (CAEP). The single crystals were ground into a fine powder for neutron diffraction experiments. Monochromatic neutron beam with wavelength of 1.5717 Å was provided by a 20 MW reactor and then monochromated with (115) germanium single crystal. The take-off angle was 92.4°. The intensity of the neutron beam was 2×10^5 n·cm^{-2}·s^{-1}, and the spot size was 24 mm×50 mm. The step was 0.1°. The measurements were conducted at room temperature. Data were refined through the Rietveld method using the Program FullProf.2k

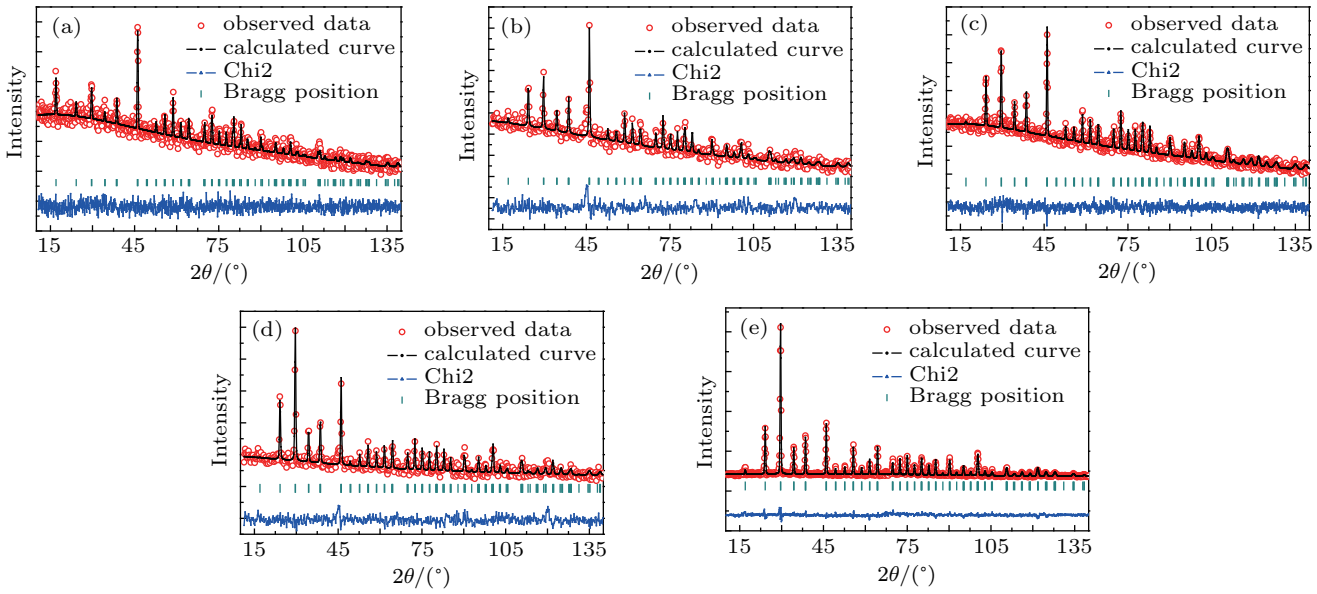


Fig. 2. Neutron diffraction patterns of DADP crystals grown in solution with different deuteration levels: (a) 0%, (b) 30%, (c) 50%, (d) 70%, and (e) 90%.

(Version 5.40).[20–22] The symmetry of all compounds is tetragonal and the space group is $I\bar{4}2d$. The neutron diffraction patterns of DADP crystals grown in solution with different deuteration levels are shown in Fig. 2.

3. Results and discussion

3.1. Distribution between solution and crystal

All of the crystals were transparent without visible macroscopic defects. A typical crystal is shown in Fig. 1. The composition exhibited in the grown DKDP crystals always differs from that of the mother solution. In general, the deuterated level in the crystal is usually lower than its growth solution.[6] The substitution of deuterium for hydrogen is slightly more sophisticated in the deuterated ADP crystal as compared with DKDP crystal. The variation in deuterated level of the crystal with deuterium content in the solution is shown in Fig. 3. In the overall composition range, the deuterated level in the crystal is lower than that in the initial growth solution, demonstrating that the segregation coefficient for deuterium is less than 1.

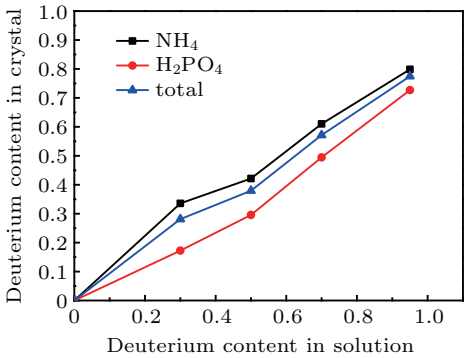


Fig. 3. Variation of deuterium content in the crystals (the mole ratio of deuterium to total hydrogen) with the change in deuterium content in the solution. The deuterium content in the group means the mole ratio of deuterium to all hydrogen in the chemical group.

Introducing the ADP crystal parameter (deuterium content is zero) to the data in Fig. 3, we achieve a linear fit of deuterium distribution for the DADP crystals. The fitting curve is shown in Fig. 4, and the equation is

$$D_c = 0.007977 + 0.8097 D_s,$$

where D_c is the deuterated level in the crystals (%) and D_s is the deuterium content in the solution (%). According to this equation, the segregation coefficient k described as $k = D_c/D_s$ would decrease with the deuterium content of the growth solution unlike that in the DKDP crystals. For the DKDP crystals, the k values increase from 0.75 to 0.99 over the deuteration range of 25%–99.8% mole D in the solution.[6] An exponential function could be employed to describe its relationship with the deuterium concentration in the solution. The segregation coefficient of deuterium is only affected by the $H_2PO_4^-$ groups in the DKDP crystals, whereas the k value for the DADP crystals is affected by both $H_2PO_4^-$ groups and NH_4^+ groups. Thus, the regular tendency of the segregation for deuterium differs between these two types of crystal.

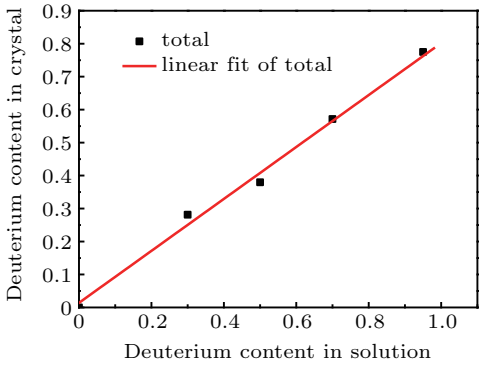


Fig. 4. Fitting of deuterium distribution in DADP crystal and the growth solution.

3.2. Lattice parameters

The detailed crystal structure refinement data for DADP are shown in Table 1 and Table 2. The crystal symmetry of

Table 1. Summary of crystal data and structure refinement for DADP with different deuterium contents.

	ADP	30% DADP	50% DADP	70% DADP	95% DADP
Wavelength	1.5717 Å	1.5717 Å	1.5717 Å	1.5764 Å	1.5717 Å
Crystal symmetry	tetragonal	tetragonal	tetragonal	tetragonal	tetragonal
Lattice parameters/Å					
a	7.4993 (14)	7.4984 (22)	7.5056 (9)	7.5107 (11)	7.5176 (4)
c	7.5459 (20)	7.5407 (34)	7.5430 (15)	7.5365 (22)	7.5353 (9)
Volume / Å^3	424.383 (0.164)	423.984 (0.260)	424.926 (0.109)	425.143 (0.151)	425.853 (0.063)
Density	1.800 g/cm^3	1.828 g/cm^3	1.834 g/cm^3	1.851 g/cm^3	1.870 g/cm^3
Angular range	$2\theta_{min}$ 10.6	$2\theta_{min}$ 11.2	$2\theta_{min}$ 10.6	$2\theta_{min}$ 3.0	$2\theta_{min}$ 11.2
	$2\theta_{max}$ 153.0	$2\theta_{max}$ 150.0	$2\theta_{max}$ 153.0	$2\theta_{max}$ 170.9	$2\theta_{max}$ 150.0
	$R_P = 3.37$	$R_P = 3.39$	$R_P = 3.06$	$R_P = 3.78$	$R_P = 4.11$
R-factors	$R_{WP} = 4.19$	$R_{WP} = 4.67$	$R_{WP} = 3.87$	$R_{WP} = 5.37$	$R_{WP} = 5.31$
	$R_E = 1.69$	$R_E = 1.48$	$R_E = 1.82$	$R_E = 3.35$	$R_E = 2.77$
Goodness-of-fit	6.17	9.91	4.51	2.57	3.68

The at% refers to the atom percentage of D/(D+H) in the solution. Here $R_P = \sum |Y_i(\text{obs}) - Y_i(\text{calc})| / \sum Y_i(\text{obs})$, $R_{WP} = \sqrt{\{\sum \omega_i [Y_i(\text{obs}) - Y_i(\text{calc})]^2\} / \sum \omega_i [Y_i(\text{obs})]^2}$, $R_E = \sqrt{(N-P) / \sum_i^N \omega_i (Y_i(\text{obs}))^2}$.

Table 2. Refined hydrogen/deuterium atomic positions, site occupancies, and uncertainties for DADP with different deuterium contents.

Crystals	Chemical group	Atom	Coordinates			Occupancy	Site
			x	*y*	*z*		
ADP	NH_4^+	H	0.10854(352)	0.01315(589)	0.57121(223)	8.000(0)	16e
		D				0	
	$H_2PO_4^-$	H	0.14053(639)	0.22707(1078)	0.12862(2023)	16.000(0)	16e
		D				0	
30%DADP	NH_4^+	H	0.16444(243)	0.08363(482)	0.55699(354)	10.630(422)	16e
		D				5.370(422)	
	$H_2PO_4^-$	H	0.16349(1557)	0.24343(3129)	0.15560(1555)	6.621(302)	16e
		D				1.379(302)	
50%DADP	NH_4^+	H	0.15194(1533)	–0.00556(2166)	0.58992(1417)	9.257(201)	16e
		D				6.743(201)	
	$H_2PO_4^-$	H	0.12560(2267)	0.19742(2382)	0.14511(3616)	5.635(119)	16e
		D				2.365(119)	
70%DADP	NH_4^+	H	0.11827(587)	0.00251(844)	0.56184(523)	5.907(656)	16e
		D				10.093(656)	
	$H_2PO_4^-$	H	0.14469(1956)	0.21777(2068)	0.12322(5512)	3.970(267)	16e
		D				4.030(267)	
98%DADP	NH_4^+	H	0.11270(209)	0.01345(348)	0.56602(213)	3.222(137)	16e
		D				12.778(137)	
	$H_2PO_4^-$	H	0.14041(336)	0.24786(1494)	0.15064(731)	2.183(115)	16e
		D				5.817(115)	

The isotropic displacement (temperature) parameter B_{iso} is set as 0.5.

ADP is not altered by substitution. Both the unit cell volume and crystal density are increased with deuterium content. The relatively small R-factors and goodness-of-fit demonstrate the close agreement between the observed and calculated profiles.

As shown in Table 1 and Fig. 5, the lattice parameters of the grown DADP crystals are highly dependent on composition. The trend of the changes in parameters along the *a* and *c* directions is distinct. In general, the lattice parameter along the *a* direction increases with increasing deuterium content in the crystals. Meanwhile, the lattice parameter along the *c* direction decreases. The difference between the lattice parameters along these two directions diminishes with the increase in deuterium content, whereas the c/a ratio shows a regular decline (Fig. 6).

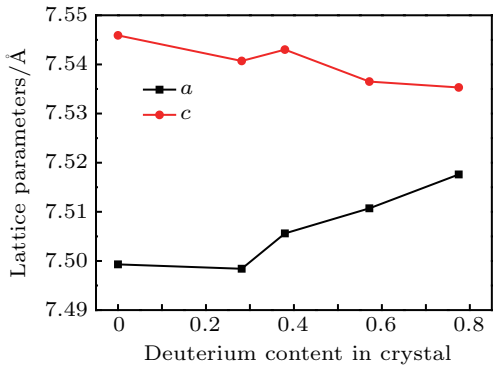


Fig. 5. Variation of unit cell parameters with composition.

The variation in lattice parameter with deuterium content is related to the distribution of hydrogen bonds. For the isomorph DKDP crystals, the hydrogen bond exists in the interaction between phosphate groups and distributes along the $a(b)$ direction. After the substitution of deuterium for hydrogen, the O–H–O bond changes to O–D–O, thereby increasing the hydrogen bond length.[7] Thus the lattice parameter along the $a(b)$ direction increases with the increase in deuterium content, and the *c*-direction parameter shows no obvious change. The *c* axis is almost perpendicular to the hydrogen bond. In the DADP crystals, the presence of the NH_4^+ group complicates the distribution of hydrogen bonds with respect to that of the DKDP crystals. Each NH_4^+ group connects with six $H_2PO_4^-$ groups through eight N–H–O bonds. Each H atom in the cation is linked with two $H_2PO_4^-$ groups by two types of hydrogen bond. Each $H_2PO_4^-$ group is connected to four neighbor $H_2PO_4^-$ groups through the O–H–O bond and six NH_4^+ groups through the O–H–N bond. Every O atom in the $H_2PO_4^-$ group is linked with two NH_4^+ groups and one neighboring $H_2PO_4^-$ group.[23] The hydrogen bonds in the (D)ADP crystals distribute along different directions. Consequently, the substitution of deuterium for hydrogen changes the *c*-direction parameter, as well as the $a(b)$ direction.

Two types of O–H(N) bond exist among the NH_4^+ and $H_2PO_4^-$ groups. The longer one (2.6 Å) is almost parallel to the *c* axis, whereas the shorter one (1.9 Å) is inclined to the $a(b)$ axis. In general, deuteration would increase the length of the hydrogen bond. The shorter O–H(N) bond and the hydrogen bonds among the $H_2PO_4^-$ groups are along the $a(b)$ direction. Hence, the *a*-direction lattice is stretched and the parameter increases. However, the O–H(N) bond along the *c* direction with the longer bond length affords the lattice of this direction enough interspace cavity, which could then admit the displaced atoms from the substitution of deuterium for hydro-

gen. Furthermore, the combined effect of the stretched hydrogen bonds along the *a* direction may twist the bonds along the *c* direction, thus leading to a slight decrease in the lattice parameter of the *c* direction.

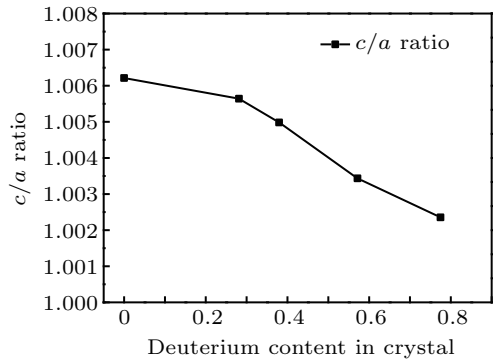


Fig. 6. The regular patterns of c/a ratio and unit cell volume change with deuterium content.

3.3. Distribution in the NH_4^+ and $H_2PO_4^-$ groups

The deuterium content in the NH_4^+ group is higher than that in the $H_2PO_4^-$ group, but the discrepancy between the two contents decreases with deuterium composition (Fig. 7). The substitution of deuterium for hydrogen in the NH_4^+ group is greater than that in the $H_2PO_4^-$ group, and the ratio of incorporation into these two groups is not a fixed constant. The ratio of the deuterium concentration of the NH_4^+ group to that of the $H_2PO_4^-$ group decreases with the increase in deuterium content in the solution. Considering the different k values obtained for the DADP and DKDP crystals, we conclude that the substitution probability of deuterium in the NH_4^+ group decreases, whereas that in the $H_2PO_4^-$ increases, with increasing deuterium content.

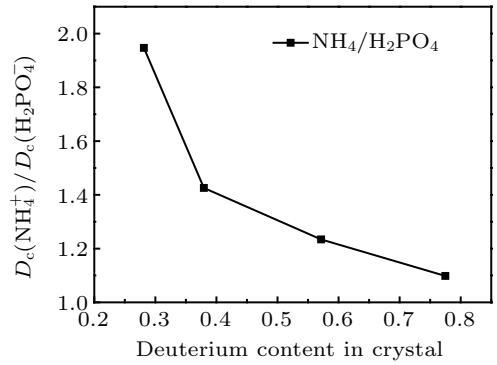


Fig. 7. The ratio of deuterium content between NH_4^+ and $H_2PO_4^-$ group.

The electronegativity of the N atom is 3.04, and the configuration of the extranuclear electron of the N atom is $2s^22p^3$. In the NH_4^+ group, these extranuclear electrons hybridize as sp^3, and the molecular is assumed to be a configuration of a regular tetrahedron. Meanwhile, the electronegativity of the O atom is 3.44, and the configuration of the extranuclear electron is $2s^22p^4$. The electronegativity differences of the N–H and O–H bonds are 0.94 and 1.34, respectively. Both are covalent bonds, but the O–H bond is more stable than the N–H bond. To a certain extent, the substitution of deuterium for hydrogen in the N–H bond is easier than that in the O–H bond. This finding can be further explained by analyzing the chemical bonding state during the crystallization of the DADP crystals in the aqueous solution. During ADP crystallization, both NH_4^+ and $H_2PO_4^-$ transform the hydrated ionic states to crystalline state. First, the free hydrated $H_2PO_4^-$ monomers transform to $(H_2PO_4^-)_n$ polymers by breaking the hydrogen bond between $H_2PO_4^-$ and H_2O. Then, the hydrated NH_4^+ ions dewater and bind to the $(H_2PO_4^-)_n$ framework by forming hydrogen bonds as in N–H$\cdots$O. In the low deuterated aqueous solution, the deuteration resistance of the $H_2PO_4^-$ group is larger than that of the NH_4^+ group. Otherwise, the formation of the $(H_2PO_4^-)_n$ framework produces numerous heavy water molecules and briefly increases the deuterated level of the solution. The deuteration of the NH_4^+ group is then improved further. When the deuterated level of the solution exceeds 90%, the segregation of deuterium for the $H_2PO_4^-$ group also exceeds 0.95. The probability of deuteration to obtain a balance in the competition between $H_2PO_4^-$ and NH_4^+ groups increases.

4. Conclusion

A series of DADP crystals were grown, and their structures were investigated using the neutron powder diffraction method. Structural information was obtained by refining the diffraction data. In the entire composition range, the deuterated level in the crystals is lower than that in the initial growth solution, showing that the segregation coefficient for deuterium is less than 1. The segregation coefficient in the DADP crystal decreases with the deuterium content of the growth solution. The deuterium content in the NH_4^+ group is higher than that in the $H_2PO_4^-$ group, but the discrepancy between the two decreases with increasing deuterium composition. In general, the lattice parameter along the *a* direction increases as the deuterium content increases in the crystal, whereas the lattice parameter along the *c* direction decreases.

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