



Reply to Kroll and Schmid-Beurmann's comment on "Water decreases displacive phase transition temperature in alkali feldspar" by Liu et al. (2018)

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Abstract. It has long been known that hydrogen impurities can be incorporated in the structure of nominally anhydrous minerals (NAMs) and substantially influence their physical properties. One of the geologically most prominent NAMs is feldspar. The hydrogen concentration in NAMs is usually expressed in parts per million of water by weight (ppm H₂O wt.) In this paper, we use the term "hydrogen" for uniformity, except when we use "water" for describing its amount expressed as parts per million of H₂O by weight. In our article (Liu et al., 2018), we carried out in situ high-temperature X-ray powder diffraction and Raman spectroscopic studies on three natural anorthoclase samples with similar Or (K-feldspar) contents (Ab₆₇Or₃₁An₂, Ab₆₆Or₃₁An₂, and Ab₆₅Or₃₃An₃) and Al–Si disordering but contrasting water contents. The spectroscopic results suggested that the displacive phase transition temperature is higher for the nearly anhydrous anorthoclase sample than the anorthoclase samples with about 200 ppm water, and we thus concluded that hydrogen is another factor impacting the displacive phase transition temperature. We thank Kroll and Schmid-Beurmann for pointing out the weakness in our interpretation that hydrogen is a possible important factor (Kroll and Schmid-Beurmann, 2020). To clarify this issue, we conducted transmission electron microscopy (TEM) experiments on the three samples to check texture effects. The TEM studies indicated that the nearly anhydrous anorthoclase sample consists of two feldspar phases, a K-poor and a K-rich one, and that the K-poor area may be responsible for the higher displacive phase transition temperature. According to the observation that the temperature of redistribution of hydrogen is accordant with the displacive phase transition temperature, the effect of hydrogen could not be ruled out. Based on these results, it can be concluded that hydrogen may not be the sole possible factor, and it was a proposition more than a definitive proof for the moment. Natural feldspars are complex, and factors affecting displacive phase transitions are multiple (e.g., Salje et al., 1991; Harrison and Salje, 1994; Hayward and Salje, 1996; Dobrovolsky et al., 2017). Therefore, to further investigate hydrogen effects on displacive phase transition in feldspar, synthetic samples with pure chemical compositions and hydrogen species are necessary. In the following, we address each issue in the same order as in the comment by Kroll and Schmidt-Beurmann (2020).

1 Reply to critique by Kroll and Schmid-Beurmann

Point (1): We have never denied the general relationship between Or content and displacive phase transition temperature in Liu et al. (2018). However, the T_c value of anorthoclase does not match with its Or content perfectly, especially when the Or content is out of the range from 2 % to 30 % (Fig. 1). Thus, there is no reason to exclude other existing factors, and the deviation of sample no. 1 in Liu et al. (2018) from

the general relationship has to be clarified. The samples used in that study have similar Or content and Al–Si disordering; thus, the deviation of sample no. 1 from the general relationship indicates that there should be other factors. It is worth noting that water content in sample no. 1 is distinctly different to that in the other two samples (60 % and 70 % lower than the other two). In addition, the displacive phase transition occurs coincidentally at the temperature of hydrogen redistribution. Based on these two facts, Liu et al. (2018) as-

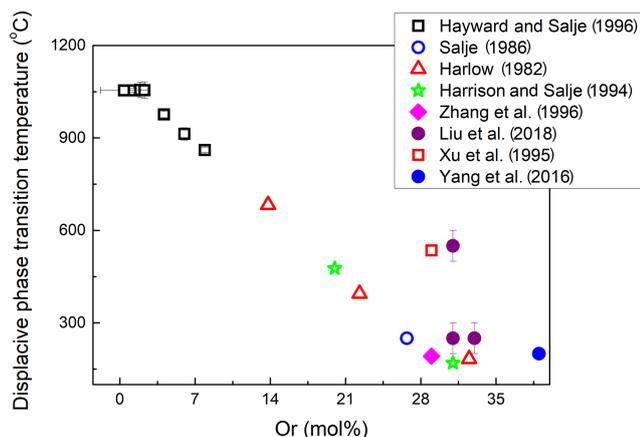


Figure 1. Relationship between displacive phase transition temperature in anorthoclase and Or content.

cribed the observed deviation from the relationship between Or content and displacive transition temperature to hydrogen effects at the time.

Hydrogen species in feldspar are very complex. There are at least five types of hydrogen species in feldspar: type I H₂O, type II H₂O, type I OH, type IIa OH, and type IIb OH, which can be distinguished from each other through Fourier transform infrared (FTIR) spectra (Johnson and Rossman, 2004). Even feldspars of similar composition and structural state may have different hydrogen species (Hofmeister and Rossman, 1985; Beran, 1986; Shuai and Yang, 2017). Furthermore, different hydrogen species locate in different sites in the structure (e.g., Johnson and Rossman, 2004; Hamada et al., 2013) and have different mobility (Kronenberg et al., 1996; Johnson and Rossman, 2013). Thus, different hydrogen species may have different impacts on the displacive phase transition in feldspar. Since hydrogen species in those samples (hydrothermally synthesized or prepared from annealing) mentioned by Kroll and Schmid-Beurmann (2020) are unclear, hydrogen effects on the displacive phase transition could not be commented on. In contrast, the three samples used in Liu et al. (2018) are anorthoclases with the type IIa OH. Although the hydrogen site of the type IIa OH in the crystal structure is still unclear, it is generally expected that multiple hydrogen sites with a range of hydrogen bond distances are involved (Johnson and Rossman, 2004). This type IIa OH will experience hydrogen redistribution from the site with stronger hydrogen bonding to the site with weaker hydrogen bonding with increasing temperature, and the displacive phase transition occurs coincidentally at the temperature of hydrogen redistribution (Fig. 4 in Liu et al., 2018). The redistribution of hydrogen among sites has also been observed during pressure-induced displacive phase transition in clinoenstatite and stishovite (Jacobsen et al., 2010; Nisr et al., 2017), and it is suggested to have an effect on the displacive phase transition in stishovite (Umamoto et al., 2016).

Point (2): The limitation of FTIR calibration seem to be ignored by Kroll and Schmid-Beurmann (2020). Hydrogen is incorporated via point defects in nominally anhydrous minerals. FTIR spectroscopy is widely used to detect trace amounts of water in nominally anhydrous minerals. About 30 % represents the uncertainty of the calibration process using FTIR for quantitative analyses, which is inevitable due to the uncertainties from sample thickness, baseline correction, absorption coefficient, etc. (Demouchy and Bolfan-Casanova, 2018). However, it does not change the conclusion that water content of sample no. 1 is much different (60 % and 70 % lower) from that of sample no. 2 and sample no. 3. Furthermore, despite the inevitable uncertainty in calculating water content using FTIR, the distinctly different water contents of sample no. 1 from the other two samples can be obviously observed from their absorbances in the IR spectra (Fig. 1 in Liu et al. 2018). In contrast, the water contents of sample nos. 2 and 3 (with 20 % difference) can be considered within the uncertainties as being on the same level, which may account for their similar transition temperature.

Point (3): We thank the authors for the notice of wrongly citing the transition temperatures. We have quoted the correct transition temperatures (183, 395, 683 °C) from Harlow (1982) in Fig. 1. The correlation is much better in the revised Fig. 1 with $R^2 = 0.90$ instead of $R^2 = 0.68$ in Fig. 9a in Liu et al. (2018). We also added more data from previous references (Salje, 1986; Harrison and Salje, 1994; Xu et al., 1995; Hayward and Salje, 1996) in Fig. 1 than those in Fig. 9a in Liu et al. (2018). Table 1 lists the data shown in Fig. 1. The different behavior of sample no. 1 can clearly be seen in Fig. 1. Obviously, the general relationship between displacive phase transition and Or content exists. However, there are exceptions. As shown in Fig. 1, in addition to the fact that this relationship does not exist when the Or content is lower than 2 % (Hayward and Salje, 1996), the relationship is less apparent when the Or content is higher than 30 %.

In addition, it should be noted that the equation suggested by Kroll et al. (1980) is derived from synthetic alkali feldspars of Or₄₀Ab₆₀–Or₀Ab₁₀₀ and several natural alkali feldspar samples (in their Fig. 8). It is not clear whether the equation reported by Kroll et al. (1980) is universal and suitable for all the complex natural chemical compositions possible.

Point (4): It is unknown whether the relationship between Or content and unit cell volume suggested by Kroll et al. (1986) is universal. The Or content of the anorthoclase reported by Yang et al. (2016) is 39 rather than 41, and it has been displayed in Fig. 1 in this paper. The X-ray diffraction (XRD) results have demonstrated that it is triclinic at ambient conditions (Yang et al., 2016). The chemical compositions of our samples are obtained from electron probe microanalyzer (EPMA). We used natural crystals of albite, rhodonite, plagioclase, orthoclase, pyrope, almandine, anhydride, and benitoite as standards. For EPMA data correction, a program based on the ZAE3 procedure was applied. We

Table 1. The data of displacive phase transition temperature of anorthoclase with different Or contents from the literature. The errors unreported in those references are listed as zero here. For the transition temperature of anorthoclase from Xu et al. (1995), it was suggested to lie between 535 and 440 °C characterized by the structural variations during heating and cooling, respectively. Thus, we here cited the critical temperature of 535 °C for comparison with our data of heating experiments.

Number	Or content (mol%)	Error (mol%)	Displacive phase transition temperature (°C)	Error (°C)	Reference
1	0.4	0.0	1054	12	Hayward and Salje (1996)
2	1.3	0.0	1054	7	Hayward and Salje (1996)
3	1.9	0.0	1056	25	Hayward and Salje (1996)
4	2.3	0.0	1055	27	Hayward and Salje (1996)
5	4.1	0.0	976	10	Hayward and Salje (1996)
6	6.0	0.0	913	20	Hayward and Salje (1996)
7	7.9	0.0	861	9	Hayward and Salje (1996)
8	26.7	2.2	250	0	Salje (1986)
9	32.5	0.0	183	0	Harlow (1982)
10	22.3	0.0	395	0	Harlow (1982)
11	13.8	0.0	683	0	Harlow (1982)
12	31.0	0.0	170	0	Harrison and Salje (1994)
13	20.0	0.0	477	0	Harrison and Salje (1994)
14	29.0	0.0	192	0	Zhang et al. (1996)
15	31.0	0.0	550	50	Liu et al. (2018)
16	31.0	0.0	250	50	Liu et al. (2018)
17	33.0	0.0	250	50	Liu et al. (2018)
18	29.0	0.0	535	0	Xu et al. (1995)
19	39.0	0.0	200	0	Yang et al. (2016)

carried out multipoint measurements to improve the accuracy. The total standard deviation is less than 0.3 %. Thus, for determination of chemical compositions of minerals, EPMA with good standards and procedures is more convincing than XRD. Additionally, the calculation of Or content from XRD parameters raises more questions than it solves. It shifts the sample in Yang et al. (2016) by more than 20 Or components, which is unrealistic and put the data point completely out of the curves deduced from Kroll et al. (1980).

Point (5): Kroll and Schmid-Beurmann argued that γ ranges from 90.1 to 90.2° based on An-poor anorthoclases (Or20-30) from references. The XRD results of sample no. 1 are really different from those of the other two. We attributed this particular behavior to different water content of sample no. 1 from the other two samples, but we agree that other parameters can influence it, even texture effects. See the following part about TEM results.

Point (6): Figure 6 in Liu et al. (2018) shows the evolutions of cell edge lengths of the three samples with increasing temperature. The unit cell edges of sample nos. 1, 2, and 3 expand with increasing temperature, with discontinuities around their transition temperatures. However, the discontinuities do not occur coincidentally for each edge of the three samples. We are also confused about the inconsistent behavior of the cell edges. It may be caused by the fact that the displacive phase transition has a weaker impact on cell edge lengths than on cell angles. Actually, the turning points at displacive phase transition temperature of the evo-

lutions of the three cell edge lengths of sample nos. 2 and 3 are less unapparent than those of sample no. 1 (Fig. 6 in Liu et al., 2018). Therefore, variations in thermal expansion coefficients of the three axes accompanied by the symmetry transition are larger for sample no. 1 than the other two. Therefore, variations in thermal expansion coefficients of the three axes accompanied by the symmetry transition are larger for sample no. 1 than the other two.

Points (7), (8) and (9): The XRD pattern of sample no. 1 at ambient conditions is indeed different from those of the other two samples. It has been reported that incorporated hydrogen can change the symmetry of the host mineral (e.g., Smyth et al., 1997). Although water contents in the samples in Liu et al. (2018) are not high enough to change the symmetry of the starting samples, the difference of the XRD patterns between sample no. 1 and the other two may be ascribed to different water contents, but other parameters such as texture can also play a role. See the following part about TEM results.

The best matched space groups were chosen by comparing diffractograms of anorthoclases with corresponding entries in an existing database of powder diffraction files (PDFs) from 2004 in the software Jade 5. The cell parameters were refined meticulously, and peaks were fitted with the reflection as precisely as possible. With regards to the reflections, about 40–50 reflections have been used in many previous studies (Harlow, 1982; Hayward and Salje, 1996; Angel et al., 2013). Angel et al. (2013) emphasized that no significant deviations were found in triclinic structure when the unit cell parameters

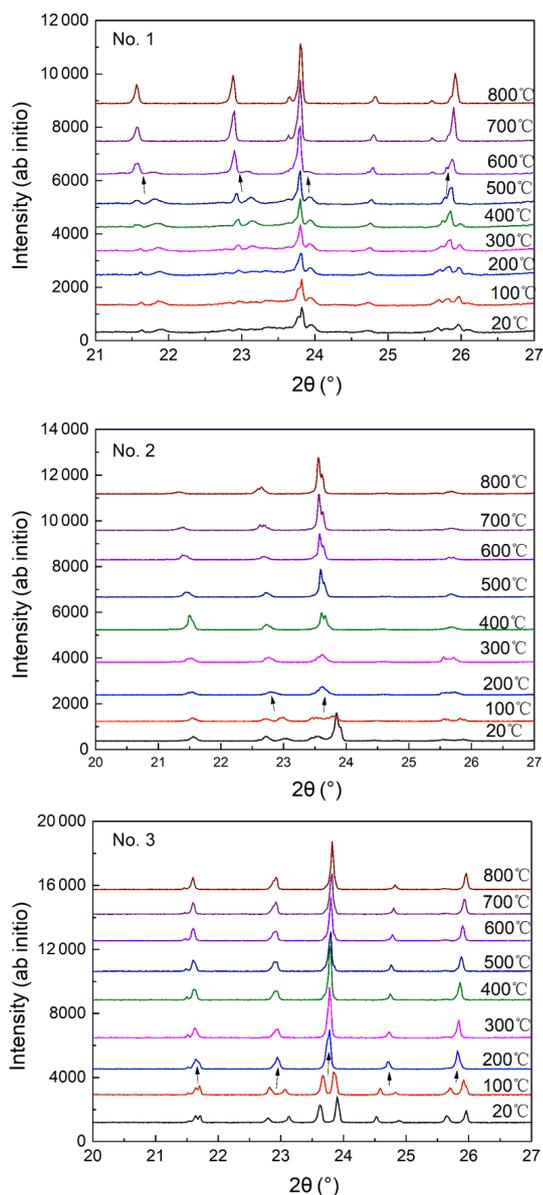


Figure 2. XRD patterns of the three samples at elevated temperatures with arrows indicating displive phase transition.

were determined from 43 reflections. We admit that we did not use an internal standard to eliminate systematic errors. But even with possibly inaccurate cell parameters obtained, the displive phase transition temperatures can also be determined from variations in the XRD patterns (Fig. 2). Figure 2 shows the evolutions of XRD peaks in the three samples at elevated temperatures up to 800 °C. The variations can be observed as the arrows. For sample no. 1, the peaks are separated in the low-temperature phase whereas the peaks coalesce at 600 °C, corresponding to the phase transition from triclinic to monoclinic symmetry (Henderson, 1979; Harrison et al., 1994). Similarly, the peaks coalesce at 200 °C for sample nos. 2 and 3 with higher water contents. Anyway, we

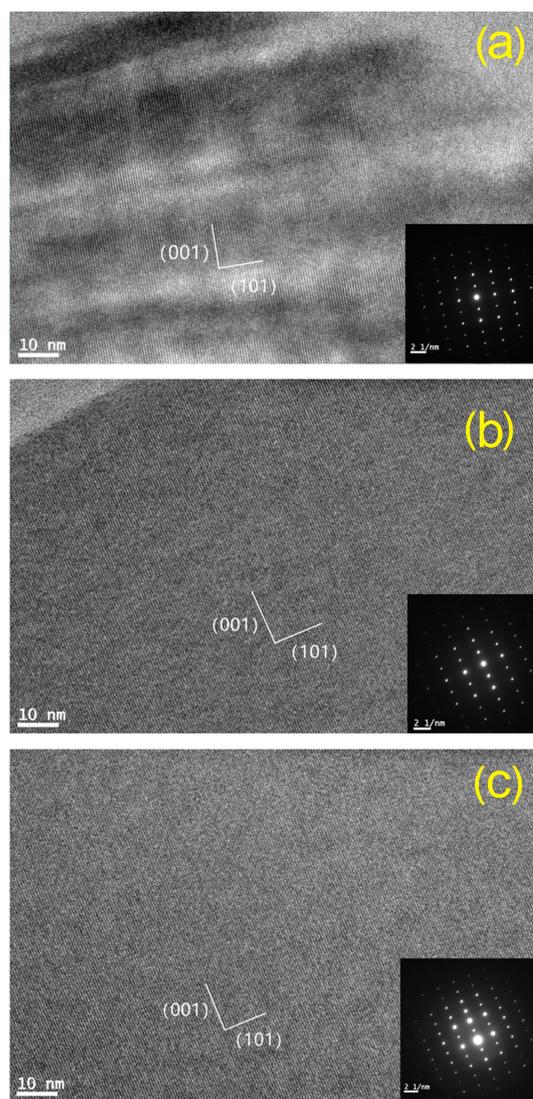


Figure 3. (a) High-resolution TEM diffractions and images of the sample no. 1, showing the wavelike lattice fringes. The heterogeneous dark and bright parts may represent the K-rich and K-poor phases. (b) High-resolution TEM diffractions and images of sample no. 2. No apparent texture effect was found. (c) High-resolution TEM diffractions and images of sample no. 3. No apparent texture effect was found.

agree with Kroll and Schmid-Beurmann (2020) that an internal standard should be applied to eliminate systematic errors and obtain accurate lattice parameters. But in this study, it will not change the main conclusions.

2 TEM results

To check if there are any texture effects, we carried out TEM measurements on the three anorthoclase samples. A FEI Quanta 3D FEG focused-ion-beam device was used for TEM sample preparation. The samples were cut into slices

around $10\ \mu\text{m} \times 2\ \mu\text{m}$ along the similar direction, and stuck on the Cu grid. Ion milling was operated for obtaining the electron-transparent area with a thickness of around 100 nm. The TEM investigations were performed with a FEI Tecnai G2 F20 S-TWIN TEM instrument operated at 200 kV. Since the samples became amorphous under the treatment of the electron beam, all the images were collected quickly.

It is evident that the TEM images of sample no. 1 are different from those of sample nos. 2 and 3. For sample no. 1 (Fig. 3a), there are apparent wavelike images as observed in Xu et al. (1995). Xu et al. (1995) have suggested that the wavelike (001) lattice fringes were thermodynamically unstable modulated structures whose symmetry was between monoclinic and triclinic. Comparatively, the TEM images of sample nos. 2 and 3 under high magnification are homogeneous. No apparent texture effect was found (Fig. 3b and c). Therefore, the heterogeneous dark and bright parts may represent two phases existing in sample no. 1: the K-rich and K-poor phases. The K-poor areas will see transition at a higher temperature like in Xu et al. (1995), and the K-rich areas will see transition at much lower temperatures. Although the three samples are all megacrysts hosted in Cenozoic basalt from the same locality, they may experience temperature decrease at different rates. Sample no. 1 may experience a slower temperature decrease than the two others during eruption, thereby inducing the start of composition modulation. The different temperature decrease rates can also explain the different water contents between the three samples. The lower water content of sample no. 1 may be caused by late degassing, while sample nos. 2 and 3 can better preserve their water contents during their fast eruption.

3 Conclusion

Liu et al. (2018) applied in situ high-temperature Raman and XRD spectroscopy to investigate the displacive phase transition in natural anorthoclase samples with similar Or contents and different water contents. The spectroscopic results suggested that the displacive phase transition temperature is higher for the nearly anhydrous anorthoclase than the anorthoclase with about 200 ppm water. Combined with previously published results, we tentatively proposed that hydrogen incorporated as defects in anorthoclase may be another factor influencing the displacive phase transition temperature. Because of the complexity of natural samples, we added TEM measurements on the three samples to check texture effects in this study. The TEM study revealed the presence of two coexisting feldspars in sample no. 1, a K-poor and a K-rich one, while nos. 2 and 3 were homogeneous. Maybe sample no. 1 experienced composition modulation during eruption, although the EPMA suggests similar chemical compositions of the three samples and although the three samples are megacrysts hosted in Cenozoic basalt from the same locality. The K-poor areas in sample no. 1 may

be responsible for the higher transition temperature. On the other hand, the temperature of hydrogen redistribution is in agreement with the displacive phase transition temperature for sample nos. 2 and 3; thus, the effect of hydrogen cannot be ruled out. To further understand this effect, experiments and characterizations on synthetic samples with end-member compositions and hydrogen-bearing lattice are necessary.

Data availability. All figures and tables presented in this manuscript are available at figshare <https://doi.org/10.6084/m9.figshare.12275498> (Liu et al., 2020).

Author contributions. WL contributed to the experiments and data analysis. YY worked on the manuscript with all authors.

Competing interests. The authors declare that they have no conflict of interest.

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