

The unit-cell parameters of an ordered K-Rb alkali feldspar series

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Abstract

Seven ordered alkali feldspars in the series $(K,Rb)AlSi_3O_8$ have been synthesized by ion-exchange techniques. Microcline was prepared from a natural low albite by ion exchange. Nearly pure $RbAlSi_3O_8$ was prepared from this microcline by ion exchange in fused $RbCl$. Intermediate compositions were prepared by homogenizing mechanical mixtures of microcline and $RbAlSi_3O_8$. The cell parameters measured at room temperature show that a , c , V , β , and γ increase nearly linearly with Rb-substitution, α decreases and b remains constant.

Plots of lattice parameters against mean ionic radii in the ordered Na-K and K-Rb alkali feldspar series show discontinuities near compositions corresponding to $K_{40-45}Na_{60-55}$, and to K_{100} . The first discontinuity occurs at near the same composition as the triclinic/monoclinic transition in disordered Na-K feldspars at room temperature. The second discontinuity occurs at a composition close to that of pure microcline and is probably related to an as yet undetermined type of phase transition at room temperature, recently described for microcline by Openshaw *et al.* (1979a).

Introduction

The discovery of a new type of phase transition in microcline near room temperature (Openshaw *et al.*, 1979a, b; Wyncke *et al.*, in preparation) led us to examine the variation of the feldspar structures in response to variation in temperature, pressure, and composition (Brown *et al.*, in preparation). No data were previously available in the literature on the effect of substitution of various amounts of Rb for K in microcline; this study fills the gap and helps in understanding the microcline phase transition.

Previous workers have studied the variation of cell parameters with composition in alkali feldspar series. Series have been prepared both by ion-exchange from natural end-members and by hydrothermal synthesis. The synthetic series analbite-sanidine was first studied by Donnay and Donnay (1952). Smith

(1956) noted a small systematic error in their data, and all parameters for this series were recalculated (Wright and Stewart, 1968). Orville (1967) and Luth and Querol-Suñé (1970) also obtained cell parameters for synthetic analbite-sanidine series.

The ion-exchange series low albite-microcline was studied by Orville (1967), using Hugo microcline. Wright and Stewart (1968) prepared an ion-exchange series from natural orthoclase P50-56F, and Waldbaum and Robie (1971) studied the low-albite to microcline ion-exchange series, starting with Amelia albite. The series of Waldbaum and Robie (1971) was re-investigated by Hovis and Peckins (1978) to resolve differences between the results of Waldbaum and Robie and those of Orville (1967). The disordered series analbite-sanidine was prepared by ion-exchange (Hovis, 1977) from heat-treated Amelia

low albite, for comparison with synthetic analbite-sanidine series.

In the system KAlSi_3O_8 - $\text{RbAlSi}_3\text{O}_8$, the disordered series sanidine- $\text{RbAlSi}_3\text{O}_8$ was prepared by Ghélin and Gasperin (1970) by hydrothermal synthesis. The ordered end-member $\text{RbAlSi}_3\text{O}_8$, reported by Weitz and Viswanathan (1971), was prepared by ion-exchange from a low albite. A full ion-exchange series between microcline and ordered $\text{RbAlSi}_3\text{O}_8$ has not previously been reported.

This work concerns the preparation of the ordered series microcline- $\text{RbAlSi}_3\text{O}_8$ by ion-exchange from Amelia low albite. Cell parameters were obtained for the series members by refinement of data from high resolution X-ray powder diffraction measurements.

Experimental

The starting material for the ion-exchange series was albite from the Rutherford Mine, Amelia County, Virginia, kindly provided by Professor G. L. Hovis (Lafayette College, Pennsylvania). An analysis of a similar sample may be found in Waldbaum and Robie (1971, p. 387).

Clean, clear fragments were selected and crushed to 170 mesh (0.090 mm). This low albite was ion-exchanged with fused KCl following the method of Waldbaum and Robie (1971) to give a microcline for which the synthesis details, major-element analysis, and cell parameters appear in Tables 1 to 3. No residual albite peaks were observed in the X-ray diffraction pattern, which was checked against the cal-

culated pattern for microcline (Borg and Smith, 1969). The cell parameters calculated from the diffractogram agree with the preferred values for microcline listed in Smith (1974, p. 258). The composition determined by XRF was $\text{K}_{97.49}\text{Na}_{2.51}$. (Note on nomenclature: for the sake of brevity, Rb is used for ordered $\text{RbAlSi}_3\text{O}_8$, K for microcline, and Na for low albite. Thus for example, $\text{Rb}_{92.76}\text{K}_{5.87}\text{Na}_{1.37}$ refers to a feldspar of composition: $\text{RbAlSi}_3\text{O}_8$ 92.76 mole percent; microcline KAlSi_3O_8 5.87 mole percent; albite $\text{NaAlSi}_3\text{O}_8$ 1.37 mole percent).

This ion-exchanged sample was taken to be maximum microcline and was used as a starting point in the synthesis of the K-Rb exchange series. A sample $\text{Rb}_{69.77}\text{K}_{28.54}\text{Na}_{1.69}$ by analysis was obtained by ion-exchange between microcline and fused RbCl at 800°C [Exchange B, Table 1(a)]. This was used to prepare the intermediate series members $\text{Rb}_{13.95}\text{K}_{83.70}\text{Na}_{2.35}$, $\text{Rb}_{27.91}\text{Rb}_{69.91}\text{Na}_{2.18}$, $\text{Rb}_{41.86}\text{K}_{56.12}\text{Na}_{2.02}$, and $\text{Rb}_{55.82}\text{K}_{42.33}\text{Na}_{1.85}$. A further run involving multiple exchanges of microcline in molten RbCl [Exchange C, Table 1(a)] yielded a product $\text{Rb}_{92.76}\text{K}_{5.87}\text{Na}_{1.37}$, which was the most rubidium-rich phase obtained.

A previous synthesis of ordered $\text{RbAlSi}_3\text{O}_8$ was reported by Weitz and Viswanathan (1971), who used albite as a direct starting material. A repeat of this synthesis was attempted, but little or no exchange was observed between albite and RbCl. The cell parameters obtained by Weitz and Viswanathan (1971) agree closely with those obtained for our most rubidium-rich composition.

Table 1. Synthesis history of samples

(a) Ion-exchange experiments				(b) Homogenization experiments				
Exchange Mixture	Duration of run	Temperature	Remarks and final product	Sample number	Duration of run	Temperature	Total time	Average temperature
A Ab 3.07g KCl 31.72g	24h00	850±10°C	K _{97.49} Na _{2.51} : see Table 2 for analysis	2 and 3	65h15	935±10°C	174h20	1000±65°C
					14h30	1050±10°C		
B Mi 1.40g RbCl 25.00g	17h40	800±10°C	Rb _{69.77} K _{28.54} Na _{1.69} : analysis-Table 2. Two exchanges; one with 15 g RbCl; one with 10 g fresh RbCl.	4 and 5	16h05	1000±10°C	249h10	950±10°C
	65h00	800±10°C			61h50	1000±10°C		
C Mi 0.69g RbCl 25.00g	21h45	800±10°C	Rb _{92.76} K _{5.87} Na _{1.37} : analysis-Table 2. Four exchanges: three with 5g RbCl; one with 10g RbCl.	6	17h30	950±10°C	240h35	875±85°C
	19h00	800±10°C			23h10	950±10°C		
	19h00	800±10°C			66h45	950±10°C		
	69h00	800±10°C			18h45	950±10°C		
					18h15	950±10°C		
		19h45	950±10°C					
		85h00	950±10°C					
			6h45	800±10°C				
			19h00	850±10°C				
			19h30	850±10°C				
			18h00	950±10°C				
			69h00	850±10°C				
			24h00	950±10°C				
			18h50	850±10°C				
			65h30	950±10°C				

Note: the products of exchanges A and B were used to prepare the composition series (see Table 2). The most rubidic product (exchange C) was synthesised later.

Table 2. Chemical analyses of ion-exchange products and compositions of series members

(a) Ion-exchange products			
Oxide	Weight percent oxide		
	Exchange A	Exchange B	Exchange C
SiO ₂	62.83	56.76	54.16
Al ₂ O ₃	19.16	17.96	17.96
Na ₂ O	0.28	0.15	0.12
K ₂ O	16.54	3.85	0.78
Rb ₂ O	-	18.68	24.48
CaO	0.19	0.14	0.20
MgO	0.17	0.31	0.08
Fe ₂ O ₃	0.24	0.42	0.28
MnO	-	0.02	-
TiO ₂	0.006	0.016	0.080
Sum	99.41	98.31	98.14

(b) Composition series			
Sample number	Composition	Remarks	
1	Rb _{0.00} K _{97.49} Na _{2.51}	Microcline, exchange A	
2	Rb _{13.95} K _{83.70} Na _{2.35}		
3	Rb _{27.91} K _{69.91} Na _{2.18}		
4	Rb _{41.86} K _{56.12} Na _{2.02}		
5	Rb _{55.82} K _{42.33} Na _{1.85}		
6	Rb _{69.77} K _{28.54} Na _{1.69}	From exchange B	
7	Rb _{92.76} K _{5.87} Na _{1.37}	From exchange C	

Two major problems were encountered during the syntheses:

(a) Homogenization: The homogenization process between potassic and rubidic phases required considerable time (more than 200 hours at 950°C on average), with removal from the furnace and grinding at approximately 24-hour intervals. Inhomogeneity was recognized by doubling of various X-ray peaks (e.g. $\bar{2}01,220$), which coalesce as homogenization proceeds. We note that this is not a sufficient test for complete homogeneity, a more sensitive method being scanning of the sample using a microbeam technique (Waldbaum and Robie, 1971). The X-ray peaks observed to coalesce were sharp, and smooth trends in cell parameters were in general noted, hence it was assumed that the compositional error due to inhomogeneity was small relative to other errors. During the homogenization process, a second problem became apparent.

(b) Breakdown: As homogenization proceeded, anomalous peaks appeared in the X-ray patterns, especially for more rubidic compositions heated

for longer periods. These peaks persisted unchanged in a sample (Rb_{55.82}K_{42.83}Na_{1.85}) which was back-exchanged to microcline. This suggests the appearance of a non-feldspathic Rb-rich phase, or one which is alkali-deficient. This may occur *via* breakdown of the RbAlSi₃O₈ during the prolonged heating at the high temperature necessary for homogenization. By visual estimation from the diffraction pattern intensities, this impurity forms a maximum of 5% of the total sample in the runs most affected due to their high Rb content and length of heating time, *i.e.*, Rb_{41.86}K_{56.12}Na_{2.02} and Rb_{55.82}K_{42.33}Na_{1.85}. This should not drastically affect the trends under study.

X-ray measurements

Both high-resolution and routine X-ray powder diffraction patterns were run on a Siemens diffractometer at the University of Nancy I, France. Radiation used was CoK α ($K\alpha_1 = 1.78892$, $K\alpha_2 = 1.79278$, $K\alpha = 1.79021\text{\AA}$). For high-resolution runs, a chart speed of 600 mm/hour with motor speed of 1/4°/min was used. Forward and reverse scans were carried out for positive and negative angles over a range of 20° to 56° 2 θ .

Peaks were assigned by first indexing the microcline pattern by comparison with the calculated pattern of Borg and Smith (1969), then following the peaks through to more rubidium-rich compositions. Ambiguous RbAlSi₃O₈ peaks were indexed by first calculating the cell parameters using known peaks, then recalculating peak positions for that phase, followed by a further calculation of cell parameters using all possible peaks. Peak positions were measured by taking an average of three half-width points between 1/2 and 3/4 peak height. Consistent with this, the wavelength value used was the weighted CoK α average ($\lambda = 1.79021\text{\AA}$). For each sample, the internal standard was semiconductor-grade Si with a cell edge $a = 5.430873\text{\AA}$ at 21°C (Hubbard *et al.*, 1975). The data were reduced by least-squares refinement program LCLSQ (Burnham, 1962). The peaks used in each refinement are given in Table 3 along with the lattice parameters obtained. The parameters obtained after back-exchanging sample number 5 [Table 2(b)] in fused KCl did not differ significantly from those of microcline, sample number 1.

Results and discussion

In Figure 1, the cell parameters are plotted *vs.* mole percent RbAlSi₃O₈ (ignoring the albite present), while the variation of these cell parameters with

Table 3. Unit-cell parameters for members of the composition series

Sample number	Mole % RbAlSi ₃ O ₈	mean cation radius (nm)	\bar{a} (nm)	\bar{b} (nm)	\bar{c} (nm)	α (°)	β (°)	γ (°)	V_3 (nm ³)
1	0.00	0.1682	0.85871 .00023*	1.29669 0.00026	0.72201 0.00014	90.646 0.038	115.929 0.010	87.643 0.028	0.72238 .00027
2	13.95	0.1694	0.86201 0.00039	1.29640 0.00041	0.72216 0.00032	90.593 0.027	115.970 0.022	87.772 0.030	0.72496 0.00061
3	27.91	0.1706	0.86632 0.00034	1.29679 0.00027	0.72324 0.00027	90.620 0.018	115.980 0.019	87.795 0.020	0.72985 0.00059
4	41.86	0.1717	0.87138 0.00018	1.29722 0.00014	0.72361 0.00010	90.558 0.008	116.037 0.010	87.873 0.008	0.73440 0.00030
5	55.82	0.1729	0.87585 0.00030	1.29671 0.00029	0.72426 0.00030	90.556 0.011	116.078 0.020	87.934 0.014	0.73832 0.00053
6	69.77	0.1741	0.88003 0.00023	1.29565 0.00022	0.72476 0.00026	90.517 0.010	116.101 0.018	88.003 0.013	0.74163 0.00048
7	92.76	0.1760	0.88431 0.00020	1.29607 0.00020	0.72558 0.00023	90.534 0.009	116.199 0.013	88.010 0.012	0.74570 0.00039

* Uncertainties as defined by BURNHAM (1962)

(Note: 1 nm = 10 Å)

Sample number	Peaks used in refinements	Total number of data points
1	$\bar{2}01$, $\bar{1}11$, $\bar{1}\bar{1}1$, 130 , $\bar{1}\bar{3}0$, $\bar{1}12$, 220 , $\bar{2}02$, 131 , 112	40
2	021 , $\bar{2}01$, $\bar{1}11$, $\bar{1}\bar{1}1$, 130 , $\bar{1}\bar{3}0$, $13\bar{1}$, 220 , $\bar{2}02$, 131 , $13\bar{2}$	44
3	021 , $\bar{2}01$, $\bar{1}11$, 130 , $\bar{1}\bar{3}0$, $13\bar{1}$, 220 , $\bar{2}02$, 131 , $13\bar{2}$	39
4	021 , $\bar{2}01$, 130 , $\bar{1}\bar{3}0$, $13\bar{1}$, $22\bar{1}$, $\bar{1}31$, 220 , 131 , $\bar{1}\bar{3}1$, $13\bar{2}$, $\bar{1}32$	48
5	021 , $\bar{2}01$, 130 , $\bar{1}\bar{3}0$, 220 , 131 , $\bar{1}\bar{3}1$, $13\bar{2}$, $\bar{1}32$	36
6	021 , $\bar{2}01$, 200 , 130 , $\bar{1}\bar{3}0$, $13\bar{1}$, $\bar{1}31$, 220 , 131 , $\bar{1}\bar{3}1$, $13\bar{2}$, $\bar{1}32$	48
7	021 , $\bar{2}01$, 130 , $\bar{1}\bar{3}0$, $13\bar{1}$, $\bar{1}31$, 220 , 131 , $\bar{1}\bar{3}1$, $13\bar{2}$, $\bar{1}32$	44

mean cation radius is shown in Figure 2. The values for the cation radii used were $\text{Na}^+ = 1.38$, $\text{K}^+ = 1.69$, $\text{Rb}^+ = 1.77\text{\AA}$, as suggested by Shannon (1976) for 9-coordination in oxides. Na^+ is too small to be in 9-coordination in feldspars (e.g. Megaw, 1974, p. 16); this is assumed here as an approximation to the average coordination of the Na^+ sites.

The use of a plot of cell parameters vs. mean cation radius (e.g. Fig. 2) gives a simple two-dimensional diagram which can be used to compare several ion-exchange series, and to take account of small amounts of impurity cations.

From Figure 1, the cell volume is seen to increase linearly with substitution of the larger cation Rb^+ . A similar increase is seen in the cell edges a and c , with a increasing much faster than c , while b remains constant. The angle α decreases slightly from 90.65 to 90.53°, while β and γ increase from 115.93 to 116.20° and 87.64 to 88.01° respectively. Note that α and γ tend toward 90°, and that $\text{RbAlSi}_3\text{O}_8$ is dimensionally only slightly triclinic due to its Al/Si order. This

is consistent with Megaw (1974, p. 16–17), who proposed that the framework geometry is controlled mainly by the relative attitude of tetrahedra within the crankshaft four-rings, and that tetrahedral site occupancy has only a second-order effect on the symmetry.

The results of the microcline– $\text{RbAlSi}_3\text{O}_8$ series alone are not surprising, but when combined with data for the low albite–microcline series (Orville, 1967; Hovis and Peckins, 1978), several interesting features become apparent (Fig. 2). Two major discontinuities in the slopes of the cell parameters vs. cation size may be seen. Not all of the individual cell parameters show both changes in slope, but all show at least one. The discontinuities in the cell edges appear sharp, and occur at 1.50–1.52 and 1.67–1.69 Å. The cell angles do not appear to change abruptly in slope; instead, a gradual change is observed in the region of the discontinuities in the cell edges.

The first discontinuity occurs near the composition $\text{K}_{40-45}\text{Na}_{60-55}$. It is well known that the variation of

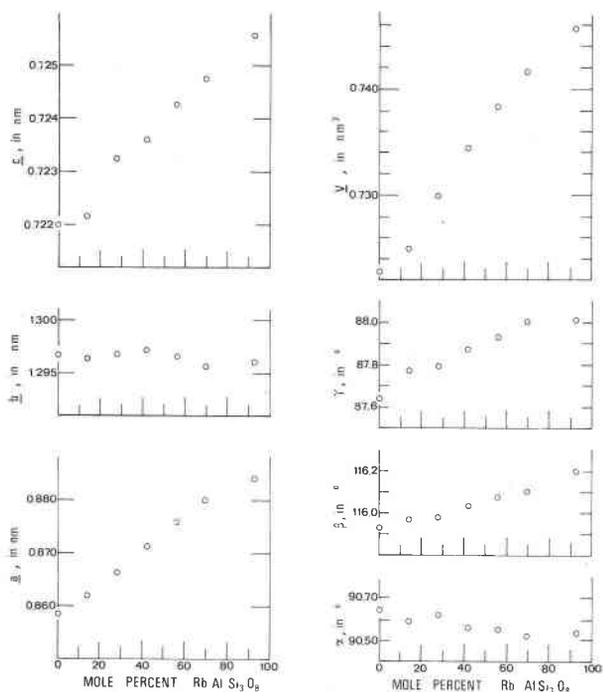


Fig. 1. Variation of the cell parameters as a function of mole percent $\text{RbAlSi}_3\text{O}_8$ in the ordered K-Rb feldspar series.

cell parameters in Na-K series is non-linear with composition. A change of symmetry occurs near $\text{K}_{35}\text{Na}_{65}$ for the disordered series (Donnay and Donnay, 1952; Orville, 1967; Wright and Stewart, 1968; Luth and Querol-Suñé, 1970; Hovis, 1977), accompanied by changes in slope of the b and c cell parameters, and cell angles α , β , and γ ; α and γ have zero slope once monoclinic symmetry is attained. In the ordered series, plots of cell parameters vs. composition show a change in the slope of b , c , α , β , and γ near $\text{K}_{40}\text{Na}_{60}$ [Orville, 1967 (Figs. 3, 4 and 5); Wright and Stewart, 1968 (Fig. 1); plot of data from Hovis and Peckins, 1978 (Table 1)], although none of these authors discussed the discontinuity in terms of the feldspar framework.

Luth (1974) discussed the behavior of cell parameters with composition for all available Na-K series. He fitted their behavior as a continuous polynomial function of composition across each entire series. Luth did not attempt to separate these functions into two or more regions separated by a discontinuity, but quoted Vogel *et al.* (1973) who had done this. Finally, Luth recognized that these polynomial functions need not be related directly to structural changes, but provided a useful tool for data comparison. Vogel *et al.* (1973) plotted the residuals of

linear relations of b and c vs. composition for five Na-K series, and showed that for both ordered and disordered series, the deviation from linearity was maximized near $\text{K}_{40}\text{Na}_{60}$. On the basis of their analysis, they concluded that regardless of the structural state of the alkali feldspars, they consist of at least two separate families (Vogel *et al.*, 1973, p. 908). They also noted that the structural rearrangements causing the triclinic-monoclinic transition in the disordered series must also occur in the ordered case. Stewart (1975, p. St6) assigned a probable cause of the change in parameter behavior to a change in the oxygen coordination around the alkali cation, but did not extend this to consider the effect on the framework as a whole.

The second discontinuity occurs at a radius of 1.67–1.69 Å, near microcline, K_{100} . Bruno and Pentinghaus (1974) listed many end-member feldspars, including $\text{NaAlSi}_3\text{O}_8$, KAlSi_3O_8 , and $\text{RbAlSi}_3\text{O}_8$, with both ordered and disordered Al/Si distributions. They plotted unit-cell parameters vs. mean cation radius, and observed a change in slope of cell edges a , b , and c near K_{100} for all framework types. Since they plotted only end-member compositions, the discontinuity near $\text{K}_{40}\text{Na}_{60}$ was not observed. From the trends observed, they proposed that the alkali feldspar lattice expanded in a regular fashion when Na, K, and Rb are substituted for each other, and the existence of a discontinuity at K_{100} was not discussed. The authors considered that for such an isostructural series with a constant anion $\text{AlSi}_3\text{O}_8^-$, the cell volume should be proportional to the cation volume, following Shannon and Prewitt (1969). This assumption must be carefully considered before being applied to a non-isotropic framework structure such as the feldspars.

The two discontinuities separate three regions in the graph of cell dimensions vs. mean cation size, characterized in Figure 2 and Table 4. Both discontinuities appear more marked for the cell edges a , b , and c than for the angles α , β , and γ . This may be due to the relative precision of the data points involved, or to the nature of the changes themselves. No single explanation is yet apparent. The first discontinuity at $\text{K}_{40}\text{Na}_{60}$ affects only edges b and c , but both parameters change in the same direction by a similar degree. Thus no discontinuity is observed on a b - c plot, and b and c may be said to be coupled. The second discontinuity involves a change in the slope of a , and the coupling between b and c is removed. We consider that this second discontinuity

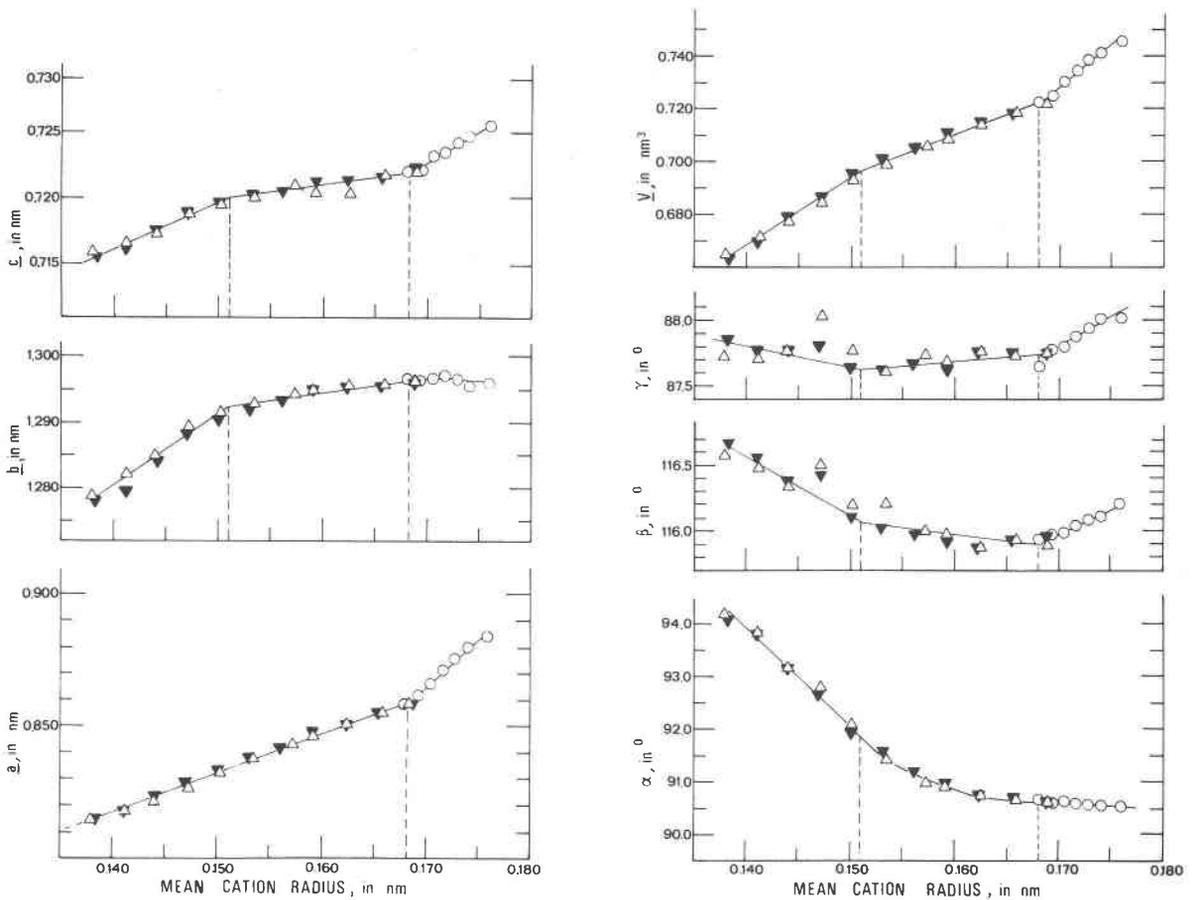


Fig. 2. Variation of the cell parameters as a function of average alkali cation radius in the ordered Na-K feldspar series (∇ from Orville, 1967; Δ from Hovis and Peckins, 1978) and K-Rb feldspar series (\circ from Table 3).

may be related to a new type of phase transition recently described for microcline at room temperature (Openshaw *et al.*, 1979a, b; Wyncke *et al.*, in preparation). We propose that these discontinuities are a

result of the expansion behavior of a framework of feldspar architecture, and will be discussed in another paper (Brown *et al.*, in preparation) in terms of the expansion of feldspar structures in general.

Table 4. Behavior of cell parameters in the three regions distinguished in Fig. 2

Region (nm)	a	b	c	α	β	γ
I 0.138 - 0.151	increases	increases	increases	decreases with greatest negative slope	decreases	decreases
II 0.151 - 0.168	increases as before with no change in slope	increase slightly, but with lesser slope	increases, but with lesser slope	decreases but slope diminishes (curvature)	remains nearly constant	remains nearly constant
III 0.168 - 0.176	increases but with greater slope	remains constant	increases with greater slope	decreases very slightly	increases	increases

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