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THE CRYSTAL STRUCTURE OF AN ADULARIA AND A REFINEMENT OF THE STRUCTURE OF ORTHOCLASE

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Abstract

The crystal structures of an adularia (Spencer B) and orthoclase (Spencer C) have been refined by least-squares methods. Although both specimens show diffraction evidence of triclinic domains and weak, diffuse reflections $h+k=2n+1$, they are monoclinic on the average and were refined in space group $C2/m$, giving "average" structures. Summary results are:

Tetrahedral Site	Mean T-O distance, Å		Average Al content of site	
	Spencer B	Spencer C	Spencer B	Spencer C
T_1	1.664	1.656	0.39	0.35
T_2	1.622	1.628	0.11	0.15

Together with the structures of four previously determined K-rich feldspars, these make it possible to better relate unit-cell parameters, optic axial angle and birefringence "b"-α to Al/Si distribution. These physical properties can now be used to estimate the structural state of potassium feldspars.

Introduction

The structural states of potassium feldspars may be related to the petrogenesis and thermal history of the rocks in which they occur. Crystal structure analyses have shown that their structural states are differentiated on the basis of Al/Si distribution in the nonequivalent tetrahedral sites; Al and Si are disordered in high sanidine and ordered in maximum microcline. The structures of five potassium-rich feldspars previously have been refined: sanidine, (heated Spencer C) $C2/m$, Cole *et al.* (1949); Ribbe (1963); orthoclase, (Spencer C) $C2/m$, Jones and Taylor (1961a, b); intermediate microcline, (Spencer U) $C\bar{1}$, Bailey and Taylor (1955); maximum microcline, (igneous) $C\bar{1}$, Brown and Bailey (1946); maximum microcline, (authigenic) $C\bar{1}$, Finney and Bailey (1964).

The results of these analyses have made it possible to establish relationships between Al/Si distribution and more-readily-determined physical properties, such as unit-cell parameters *b* and *c*, γ^*, optic axial angle, extinction angle on (001) and birefringence "*b*"-α (see Finney and Bailey, 1964). The purpose of this paper is to provide additional precise data for the monoclinic feldspars intermediate between sanidine and microcline so that the correlations of physical properties and structural state will be known well enough to obviate the need for further tedious structure analyses.

The first part of this paper presents the crystal structure of an adularia; it is specimen B of Spencer (1937), henceforth referred to as Spencer B. Optical properties and cell parameters (Wright and Stewart, 1968, their Figs. 2b and 4) indicate that Spencer B has a structural state intermediate between orthoclase and intermediate microcline which is about as low a structural state as is observed in "monoclinic" potassium feldspars (compare Laves and Viswanathan [1967] who indicate that the expected largest optic axial angle for monoclinic potassium feldspar is 63°; 2V for Spencer B is ~65° corrected for Na content).

The second part of this paper is a reexamination of "type" orthoclase (Spencer's specimen C) which previously had been refined using four two-dimensional difference syntheses (Jones and Taylor, 1961b). To make meaningful comparisons, Spencer C was re-refined in the same manner as Spencer B, using the same atomic scattering factors, but using the observed structure amplitudes listed by Jones and Taylor. The data are combined in the tables to permit direct comparison of chemical composition, unit cell and structural parameters.

SYMMETRY

In these refinements the space group $C2/m$ was assumed for both Spencer B and Spencer C. That the true symmetry is lower than this is evidenced by weak, diffuse reflections $h+k=2n+1$ elongated parallel to *b* which were observed in a *b*-axis oscillation photograph of our crystal; Laves and Goldsmith (1961) show a similar photograph (their Fig. 5) and state that orthoclases of this sort contain unit-cell scale "antiphase" domains within which triclinic symmetry has been established by increased Al/Si ordering. These domains nucleate in four different orientations, and if they grow large enough they are expected to eventually adopt albite or pericline twin orientations. The nucleation and growth processes have been eloquently described and illustrated by Laves (1950, 1952), Bambauer and Laves (1960), Laves and Goldsmith (1961), Brown (1962) and Brown and Bailey (1964).

The X-ray diffraction photographs of our Spencer B indicate that the triclinic domains have grown larger than unit-cell scale within the host. Symmetrically disposed about certain strong reflections with $h+k=2n$

are faint streaks whose elongation is either normal to or parallel to the *b* axis (Fig. 1). McConnell (1965) has reported similar streaks in oscillation photographs of Spencer C. These streaks indicate the existence of domains whose orientations are already tending to those of the opposed members of albite or pericline twins, respectively. Using phase-contrast electron microscopy McConnell observed an example of domains of this sort in an optically monoclinic adularia (his Fig. 3). They are not twins in the normal sense of the word, because they are separated by regions of monoclinic host; but it is certain that were Al/Si ordering allowed to continue, they would be nuclei for the albite and pericline twins that make up the familiar cross-hatched twinning in microcline (compare Laves and Goldsmith, 1961, their Fig. 11).

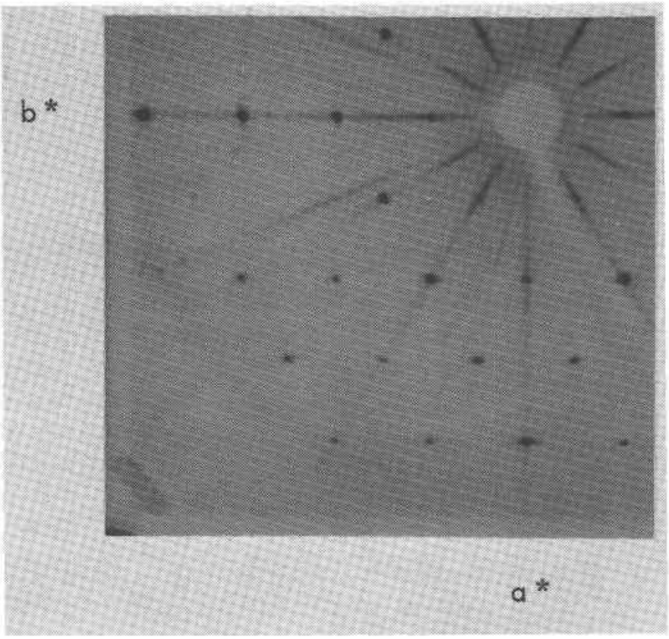

FIG. 1. A zero-level *c*-axis precession photograph of Spencer B, showing diffuse streaks through strong reflections $h+k=2n$. The streaks are either parallel or normal to the $b^*(=b)$ axis. Cu$K\alpha$ radiation.

Spencer B and Spencer C are nonetheless monoclinic on the average. That is to say, their morphologic and optical properties as well as their cell dimensions indicate that *on the average* the Al/Si distribution is monoclinic: for every left-oriented triclinic domain there is a symmetrically opposed right-oriented domain. In Spencer B careful measurement of each of 25 pairs of medium intensity reflections *hkl* and $h\bar{k}l$ shows agreement within 2.3 percent without correcting for absorption. These measurements support the presence of the mirror plane. It was expedient to ignore the few extremely diffuse reflections $h+k=2n+1$ and refine using the space group $C2/m$. Jones and Taylor made similar judgements in deciding to refine Spencer C in this space group.

The primary purpose of these structure analyses is to determine the average Al/Si distribution, because local departures from the average are spatially too small to affect the measured optical properties and the unit-cell parameters as determined by powder diffraction methods, and because the relationships between structural state and measured physical properties depend not on the short-range Al/Si order established within the monoclinic host and symmetry-related triclinic domains, but on the degree of long-range order averaged over the entire single crystal.

EXPERIMENTAL PROCEDURES

A colorless cleavage fragment 0.29×0.21×0.24 mm was chosen for analysis from specimen B in Spencer's suite of alkali feldspars (Spencer, 1930; 1937). The specimen is described as an adularia from St. Gotthard, Switzerland. J. V. Smith of the University of Chicago provided the material, and D. B. Stewart of the U. S. Geological Survey determined the unit-cell parameters using a least-squares refinement of the powder pattern. Table 1 contains the chemical analyses and unit-cell parameters.

The crystal was elongate parallel to the *a*-axis and was mounted on this axis for collection of intensities. Over 1000 reflections *0kl*-*5kl* with $\sin\theta<0.6$ were measured using Mo$K\alpha$ radiation and an equi-inclination single-crystal diffractometer. An uncollimated scintillation counter was used in order to record the intensity contribution of both the sharp diffraction maxima $h+k=2n$ and their associated diffuse streaks. Diffraction peaks were scanned at 2°/min. and recorded on a strip chart, later to be integrated and placed on the same relative scale. Lorentz-polarization and absorption corrections were computed. In the latter a cylindrical approximation of the crystal shape was used with $r=0.127$ mm; $\mu_l=0.19$. No corrections for primary or secondary extinction were made, because a weighting scheme was adopted which minimizes these effects.

The details of data collection and correction for Spencer C are found in Jones and Taylor (1961b).

The structures of Spencer B and Spencer C were refined using the full-matrix least-squares program ORFLS (Busing, Martin and Levy, 1962). Atomic scattering factors of K, Al, Si and O were taken from Volume III of the International Tables for X-ray Crystallography and

TABLE 1. CHEMICAL COMPOSITION AND CELL PARAMETERS OF SPENCER B ADULARIA AND SPENCER C ORTHOCLASE

CHEMICAL COMPOSITION (Smith and Ribbe, 1966)		
Oxides	Spencer B	Spencer C
K_2O	15.1 wt. %	15.3 wt. %
Na_2O	1.1	0.9
CaO	0.0_1	0.1
BaO	0.7	0.1
SrO	0.0_5	0.1
Fe_2O_3	0.0	0.0
Al_2O_3	18.5	18.3
SiO_2	63.8	64.2
Total	99.3	99.0
Feldspar molecules		
orthoclase	89.2 wt. %	90.1 wt. %
albite	9.1	7.9
anorthite	0.1	0.7
celsian	1.6	0.3
Sr-feldspar	0.1	0.2
Total	100.1	99.2
Cell parameters (D. B. Stewart, private comm.) Standard errors are in brackets and refer to last decimal place.		
a	8.554(2) Å	8.561(2) Å
b	12.970(4) Å	12.996(4) Å
c	7.207(2) Å	7.192(2) Å
β	116° 0.4(6)′	116° 0.6(6)′
Volume	718.7 Å	718.9 $Å^3$
Specific gravity (Spencer, 1937)	2.566	2.563

modified for half-ionized atoms. The tetrahedral atoms were given a weighted scattering factor $[1/4f_{Al}+3/4f_{Si}.]$ in order to obviate *a priori* assumptions of tetrahedral ordering.

In the refinement of Spencer B we used 750 reflections ($F_{obs}>10$) weighted according to the scheme of Cruickshank (1965). Convergence was rapid; the atomic coordinates from the isotropic refinement ($R=8.4\%$) were not significantly different than those from the subsequent anisotropic refinement ($R=5.5\%$).[1] In the refinement of Spencer C we used 465 reflections ($F_{obs}>10$), having averaged those which Jones and Taylor measured on more than one photograph. The weighting scheme

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[1] The final observed and calculated structure amplitudes for Spencer B are available upon request.

TABLE 2. ATOMIC COORDINATES IN FRACTIONS OF THE CELL EDGES[a]

Atom	Spencer B			Spencer C		
	x	y	z	x	y	z
O_A(1)	0	0.1448(4)	0	0	0.1459(8)	0
O_A(2)	0.6354(9)	0	0.2840(6)	0.6346(11)	0	0.2851(13)
O_B	.8276(7)	.1448(3)	.2277(5)	.8280(7)	.1470(7)	.2282(9)
O_C	.0351(6)	.3116(2)	.2590(5)	.0349(6)	.3106(5)	.2607(9)
O_D	.1818(6)	.1254(2)	.4075(4)	.1815(6)	.1258(5)	.4065(7)
T_1	.0094(2)	.1843(1)	.2247(1)	.0095(3)	.1844(2)	.2239(3)
T_2	.7079(2)	.1176(1)	.3443(1)	.7089(2)	.1178(2)	.3443(3)
K	.2835(3)	0	.1369(2)	.2838(3)	0	.1373(4)

[a] Standard errors $\times 10^{-4}$, are in parentheses.

and details of the refinement procedures are identical to those employed for Spencer B. For Spencer C the final *R*-factors were 7.2 percent for the isotropic refinement and 5.4 percent for the anisotropic refinement.

The final atomic coordinates are listed in Table 2; interatomic distances are in Table 3, bond angles in Table 4 and isotropic and anisotropic thermal parameters in Table 5. The standard errors in atomic parameters were calculated from the variance-covariance matrices and include the effects of the estimated errors in the cell parameters.

TABLE 3. INTERATOMIC DISTANCES IN Å[a]

	Spencer B	Spencer C		Spencer B	Spencer C
T_1-O_A(1)	1.667(2)	1.654(4)	O_A(1)-O_B	2.664(5)	2.642(6)
-O_B	1.646(6)	1.641(6)	O_A(1)-O_C	2.787(5)	2.774(11)
-O_C	1.670(3)	1.661(7)	O_A(1)-O_D	2.665(3)	2.655(5)
-O_D	1.671(4)	1.666(5)	O_B-O_C	2.745(6)	2.712(10)
			O_B-O_D	2.735(8)	2.735(7)
Mean T_1-O	1.664(2)	1.656(3)	O_C-O_D	2.716(5)	2.700(7)
T_2-O_A(2)	1.632(2)	1.641(3)	Mean O-O tetrahedron 1	2.715(2)	2.703(3)
-O_B	1.623(6)	1.620(6)			
-O_C	1.615(5)	1.631(6)			
-O_D	1.618(3)	1.621(5)	O_A(2)-O_B	2.642(7)	2.674(10)
			O_A(2)-O_C	2.569(4)	2.586(7)
Mean T_2-O	1.622(2)	1.628(3)	O_A(2)-O_D	2.643(5)	2.648(8)
			O_B-O_C	2.669(8)	2.677(8)
K-O_A(1)	2.878(4)	2.893(7)	O_B-O_D	2.677(5)	2.678(8)
-O_A(2)	2.717(8)	2.711(9)	O_C-O_D	2.684(6)	2.685(7)
-O_B	3.025(4)	3.045(7)			
-O_C	3.117(4)	3.129(6)			
-O_D	2.949(4)	2.945(7)	Mean O-O tetrahedron 2	2.647(2)	2.658(3)

[a] Standard errors, $\times 10^{-3}$, are in parentheses.

Table 4. Interatomic Angles in Degrees[a]

	Spencer B	Spencer C
$O_A(1)$-T_1-O_B	105.9(2)	106.6(3)
$O_A(1)$ O_C	113.3(2)	113.6(4)
$O_A(1)$ O_D	106.0(2)	106.2(3)
O_B O_C	111.7(2)	110.4(4)
O_B O_D	111.1(2)	111.5(3)
O_C O_D	108.8(2)	108.5(3)
Mean O-T_1-O	109.5(1)	109.5(1)
$O_A(2)$-T_2-O_B	108.5(2)	110.2(5)
$O_A(2)$ O_C	104.6(3)	104.4(4)
$O_A(2)$ O_D	108.8(2)	108.5(4)
O_B O_C	111.0(2)	110.8(4)
O_B O_D	111.3(3)	111.4(3)
O_C O_D	112.2(2)	111.3(3)
Mean O-T_2-O	109.4(1)	109.4(2)
T_1-$O_A(1)$-T_1	144.1(3)	144.8(7)
T_2-$O_A(2)$-T_2	138.3(4)	137.8(5)
T_1-O_B -T_2	152.7(3)	153.3(4)
T_1-O_C -T_2	131.2(3)	131.1(4)
T_1-O_D -T_2	141.8(3)	141.6(4)
Mean T-O-T	141.6(1)	141.7(2)

[a] Standard errors, $\times 10^{-1}$, are in parentheses.

Discussion

Wright and Stewart (1968) have shown that the *b* and *c* cell parameters and certain optical properties of the isochemical polymorphs of any alkali feldspar vary with structural state, and they point out on the basis of known crystal structures that the systematic change of these properties must be related to changes in the alumino-silicate framework. This involves a progressive disordering of Al and Si over the symmetrically non-equivalent tetrahedral sites. The degree of disorder is directly measurable by structure analysis, using a relationship determined by Smith and Bailey (1963) which shows that the average Al/Si content of individual tetrahedra is linearly related to their mean T-O distances.

The chemical compositions of Spencer's specimens B and C are very similar; therefore, the highly significant differences in the *b* and *c* cell parameters and optical properties (Table 1 and Figs. 2b and 4 of Wright

TABLE 5. THERMAL PARAMETERS
Isotropic B (Å^2)[a]

Atom	Spencer B	Spencer C
$O_A(1)$	1.7(1)	1.8(1)
$O_A(2)$	1.3(1)	1.8(2)
O_B	2.2(1)	2.2(1)
O_C	1.6(1)	1.7(1)
O_D	1.2(1)	1.6(1)
T_1	0.83(3)	0.91(3)
T_2	0.65(3)	0.80(3)
K	2.26(5)	2.23(6)

RMS COMPONENT OF THERMAL DISPLACEMENT ALONG PRINCIPAL AXES, IN Å[b]

Atom	Axis	Spencer B	Spencer C
$O_A(1)$	1	0.121(7)	0.132(9)
	2	.146(8)	.142(13)
	3	.172(15)	.144(10)
$O_A(2)$	1	.080(8)	.106(13)
	2	.132(6)	.129(11)
	3	.202(14)	.170(13)
O_B	1	.131(11)	.110(10)
	2	.167(5)	.160(10)
	3	.176(8)	.178(10)
O_C	1	.106(5)	.116(10)
	2	.149(4)	.133(7)
	3	.158(14)	.154(9)
O_D	1	.103(4)	.102(10)
	2	.121(5)	.114(9)
	3	.197(12)	.161(7)
T_1	1	.085(2)	.089(4)
	2	.103(2)	.092(4)
	3	.129(6)	.116(4)
T_2	1	.061(3)	.080(4)
	2	.100(8)	.094(3)
	3	.109(2)	.105(3)
K	1	.150(3)	.131(4)
	2	.162(2)	.170(4)
	3	.176(5)	.172(4)

[a] Standard errors refer to last decimal place.

[b] Standard errors, $\times 10^{-3}$, are in parentheses.

and Stewart, 1968) must be due to real differences in the distribution of Al and Si between the tetrahedral sites T_1 and T_2, although these differences are small.

Site	Mean T-O (Å)		Avg. Al content[1]		Isotropic temp. factor (Å^2)	
	B	*C*	*B*	*C*	*B*	*C*
T_1	1.664(2)	1.656(3)	0.39	0.35	0.83	0.91
T_2	1.622(2)	1.628(3)	0.11	0.15	0.65	0.80

It is interesting to notice that the differences between $(\overline{T_1-O})$ and $(\overline{T_2-O})$ is 0.042 Å in B and 0.028 Å in C, and that these are proportional to the corresponding differences in the isotropic temperature factors 0.18 Å^2 for B and 0.11 Å^2 for C. Individual temperature factors adjust to the difference in site occupancy (*cf.* Ribbe *et al.*, in press) because the same average scattering factor was used for each tetrahedral atom.

As far as the other interatomic parameters are concerned, the mean O-O distance for each tetrahedron (Table 3) shows the influence of site occupancy in the same manner as the mean T-O distance. The individual bond angles within or between tetrahedra (Table 4) are distorted from the average value in exactly the same way as those in all other feldspar structures (*cf.* Megaw *et al.*, 1962). The K-O bond lengths are not sufficiently different from one another or from those of sanidine (Ribbe, 1963) to merit further discussion. The isotropic temperature factors for K in the three structures are also very similar: Spencer B, 2.26(5); Spencer C, 2.23(6); sanidine, 2.0(1) Å^2.

At the end of the isotropic refinement of Spencer B, $(\rho_o-\rho_c)$ maps were calculated through each atom parallel to (100). The anisotropy of electron density observed at the site of each T and O atom (see Table 5) is an expression of the average nature of the structure determination and may result from small positional differences of similar atoms in the symmetry-related triclinic domains and in the monoclinic host (see *Symmetry*), as well as thermal vibration. A study of the extremely diffuse reflections $h+k=2n+1$ and the diffuse streaks associated with the intense $h+k=2n$ reflections might provide details of short-range order within the monoclinic and triclinic domains which would explain in part the observed anisotropy.

Because the intensities of the extremely diffuse reflections $h+k=2n+1$ could not be easily measured, they were ignored in this refinement of the average structure. Nonetheless, their presence indicates that the true space group is probably $P2_1/a$ (Laves and Goldsmith, 1961), if indeed it is proper to speak of the "true" space group of a material with such

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[1] Determined from Curve I of Ribbe and Gibbs (1967) and normalized to total Al = 1.00.

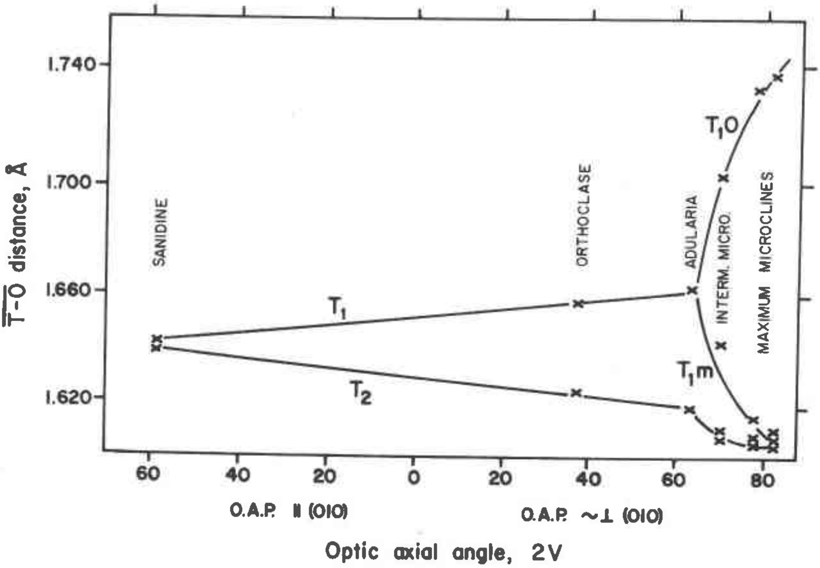

FIG. 2. Plot of optic axial angle for the six structurally determined potassic feldspars against the mean T-O distance of their non-equivalent tetrahedra. Optic angle has been corrected for the effect of Na-content using Figure 4 of Wright and Stewart (1968); therefore, this plot is valid for pure potassium feldspars.

a well-documented domain texture. In any case, our justification for refining Spencer B in space group $C2/m$ is based on the argument that it is consistent with the gross physical properties with which we are attempting to correlate the *average* Al/Si distribution.

CONCLUSIONS

With the completion of the refinements of Spencer B and Spencer C the systematic structural variations of the potassic feldspar polymorphs are known sufficiently well to permit excellent correlations of structural state with physical properties. Figures 2 and 3 show the variation of optic axial angle and birefringence "b"-α with structural state in the potassic feldspars. We have chosen to plot structural state as a function of the (T-O) distance of individual tetrahedra in deference to a recent re-evaluation of the relationship between $(\overline{\text{T-O}})$ and Al-content in feldspars (Ribbe and Gibbs, 1967; *cf.* Jones, in press).[1]

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[1] The comparison of physical properties to any one of several long-range order parameters (Bragg and Williams, 1934; Smith and MacKenzie, 1961; also personal communication from O. H. J. Christie, 1967) depends on a knowledge of the precise Al/Si distribution is regarded as unnecessarily subjective. This will be discussed in detail by Ribbe and Gibbs (forthcoming).

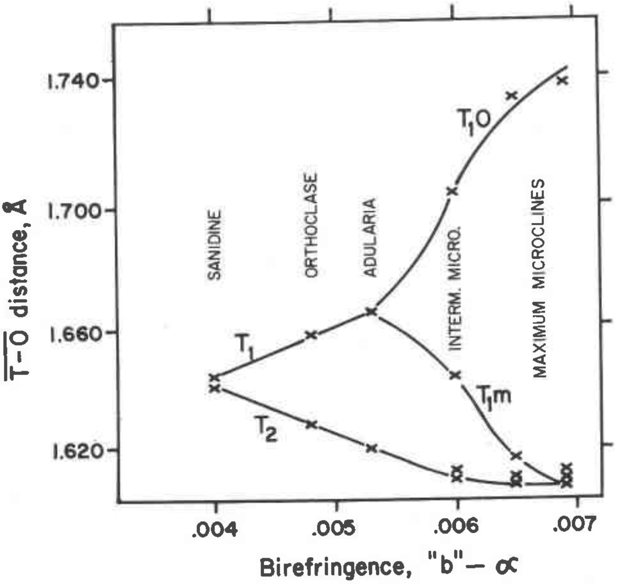

FIG. 3. The relationship of birefringence "*b*"-α to the mean bond lengths of the nonequivalent tetrahedra in six structurally-determined potassic feldspars. "*b*" is the refractive index whose vibration direction is parallel or most nearly parallel to the *b* crystallographic axis (Hewlett, 1959).

Figure 2 shows that in monoclinic potassic feldspars the optic axial angle is very sensitive to Al/Si distribution, varying 120° between sanidine and adularia; however, the variation is only 20° for the microclines. Unfortunately, 2V is dependent on the Na-content of the potassic feldspar. Composition must be known and corrected for, using Figure 4 of Wright and Stewart (1968), before this method can be used to determine the structural state. A more satisfactory optical property to use is birefringence "*b*"-α (Fig. 3),[1] which is independent of Na-content in potassium-rich feldspars and appears to be an excellent indicator of the sizes of the nonequivalent tetrahedra. Thus it may be used as a direct and simple measure of the Al/Si distribution.

For further details of the relationships of structural state, physical properties and chemical composition of the alkali feldspars the reader is referred to Wright and Stewart (1968) and to forthcoming papers by Ribbe and Gibbs and Stewart and Ribbe.

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[1] Birefringence "*b*"-α is defined (Hewlett, 1959; Finney and Bailey 1964) in terms of the index "*b*" of the ray that vibrates most nearly parallel to the *b* crystallographic axis, which may be either γ or β.

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