The crystal structure of tetragonal leucite

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Abstract

Low temperature leucite is known to have a tetragonal structure which is similar to the high temperature cubic structure. This tetragonal structure has never been studied because of the fine twinning, which makes the selection of untwinned single crystals difficult. The best crystal chosen for this research is made up of three individuals: one, comprising 79 per cent of the total, is merohedrically twinned with respect to the second, and pseudo-merohedrically with respect to the third. The crystal structure of tetragonal leucite, $a = 13.09$, $c = 13.75$ A, space group $I4_1/a$, can be derived by displacing the cubic structure (some atoms must be shifted as much as 1 Å). There is no (Si,Al) order. The K atoms are coordinated by six oxygens at the average distance of 3.01 A, six other being at $\sim$3.7 Å. In cubic leucite these values are 3.35 and 3.54 Å; this is the most striking difference between the crystal structures.

Introduction

Leucite $K[AlSi_2O_6]$ was shown by Wyart (1940) to have a structure with the same topology as analcime, as determined by Taylor (1930). Leucite undergoes a phase transition, also described by Wyart (1940), from a cubic phase (space group $Ia3d$) stable above 630°C, to a tetragonal one (space group $I4_1/a$) stable at lower temperature. This polymorphic inversion was investigated in detail by Faust (1963), who also listed previous studies of the problem. Recently Peacor (1968) refined the structure of the cubic phase of leucite at high temperature. The same author pointed out the difficulty of obtaining a suitable single crystal of the tetragonal phase due to the fine twins which always develop in tetragonal leucite during its inversion from the high temperature cubic phase.

Experimental

A sample of leucite from Roccamonfina, Caserta, Italy (specimen n. 16-3-3-24 of the Museo di Mineralogia dell'Università di Modena) was selected for this study (see next section). A chemical analysis of this material yields unit cell contents, calculated on the basis of 6 oxygens, of:

$$16([K_{0.94}Na_{0.05}Ca_{0.01}][Fe^{3+}_{0.01}Al_{0.96}]Si_{2.02}O_6);$$

the chemical analysis also revealed the presence of 0.002 Mg and Ti.

The unit cell dimensions and the intensities of the reflections were measured with a Philips PW_{1100} single crystal automatic diffractometer using graphite monochromatized MoKα radiation at room temperature. The cell dimensions thus determined—$a = 13.09(1)$ and $c = 13.75(1)$ A—are in good agreement with those determined by the least-squares refinement of powder diffraction data and with those quoted by Faust (1963) for a sample from the same locality. The space group determined from the systematic absences is $I4_1/a$, the same as determined by Wyart (1940) and by Náray-Szabó (1942). The intensities of 1040 independent reflections in the range 4°–50° 2θ were inspected using θ–2θ scans with a symmetrical scan range of 1.20° in 2θ from the calculated diffraction angle. The scan rate was 0.04° θ/sec. For each reflection, repeated scans were made until the preset maximum number (3) or the preset number of counts (1000) was reached. Due to the small dimensions (0.05 × 0.05 × 0.08 mm) of the crystal, 529 weak reflections were skipped by the diffractometer program during the data collection, because their intensities in counts/sec measured at the top of each reflection (namely, I_t) and the mean intensities in counts/sec of both background

measurements (namely, I_b) gave: $I_t - 2\sqrt{I_t} < I_b$. These "unobserved" reflections were assigned values of half the minimum observed intensity. Three standard reflections, monitored at three hour intervals, showed no variation in intensity greater than 1 percent. Processing of the data was carried out in the manner described by Davies and Gatehouse (1973) to yield $|F_{obs}|$ and $\sigma|F_{obs}|$. No absorption or extinction corrections were applied. The atomic scattering factors used are those listed by Hanson *et al* (1964).

The leucite twins

Natural leucite crystals always grow as the cubic phase (Faust, 1963). When a cubic leucite single-crystal inverts to the tetragonal form, a complex twin develops. All six planes of the cubic form {110} may become twin planes in the tetragonal phase (point group $4/m$). Two cases must be considered:

I. Merohedric twins develop on the tetragonal planes (110) and ($\bar{1}$10), the two individuals having exactly parallel crystallographic axes, but with a and b interchanged. X-ray diffraction of such twins gives hkl diffractions of one individual exactly superposed with khl diffractions of the other. The two individuals of the twin show simultaneous extinction under crossed nicols.

II. Peudomerohedric twins develop on the tetragonal planes (101), (011), ($\bar{1}$01), ($0\bar{1}$1), the two individuals having parallel a (or b) axes, but the remaining two axes are not parallel. The two individuals of such a twin give close but distinct spots in X-ray photographs and do not extinguish simultaneously when viewed under crossed nicols.

In short, if single crystal X-ray photographs reveal any splitting of spots, at least a pseudomerohedric twin is present; if no splitting appears, the presence of a merohedric twin may still be suspected, but it can be detected only by studying the intensities of the diffractions.

The different orientations of the individuals in leucite pseudomerohedric twins were described in detail by Sadanaga and Ozawa (1968) and by Korekawa (1969).

Since the observation of the pseudomerohedric twins is properly made using the polarizing microscope, thick sections were prepared from our leucite specimen to allow the selection of some apparently untwinned fragments. Most of these fragments were discarded after a first X-ray test with oscillation photographs, but two were thus selected. Measurements using the single crystal diffractometer revealed the first to be composed of three pseudo-

merohedrically twinned individuals, the first comprising 88 percent, the second 8 percent, and the third 4 percent; the second fragment, composed of only two pseudomerohedrically twinned individuals, comprising 96 and 4 percent respectively, was satisfactory for further measurements. The first individual of the twin was actually affected by merohedric twinning, which was detected during the refinement of the crystal structure (see next section).

If a twinned crystal of tetragonal leucite is heated above 630°C, it inverts to an untwinned cubic crystal which, upon lowering the temperature, gives a twinned crystal again with the same proportion of twin domains and with the same twin boundaries as before. This "memory" displayed by leucite twins was demonstrated by optical and/or X-ray methods by Wyart (1938), Peacor (1968), Sadanaga and Ozawa (1968), and Korekawa (1969). This "memory" was verified once more during this research, on the following three twins:

I. Two individuals with common a axis, kept at 800°C overnight.

II. Four individuals with common a axis, kept at 700°C overnight, and then for three hours at ~630°C before cooling to room temperature.

III. Four individuals, the a axis being common for two individuals at a time, were kept at 700°C for one day, with four interspersed fast quenchings to room temperature.

The intensities of a small set of diffractions were measured at room temperature before and after heating for each twin individual; there was no significant variation of these intensities.

Determination and refinement of the crystal structure

The structural study started with the atomic parameters given by Peacor (1968) for the cubic phase, properly adapted to the tetragonal space group. Least squares refinement with a modified version of the program ORFLS (Busing, Martin and Levy, 1962) allowed the reduction of R from 0.54 to only 0.32, without possibility of further improvement. Direct methods (program MULTAN described by Germain, Main, and Woolfson, 1971) were then applied, and new starting parameters were obtained. In this case, R dropped to 0.11 in four least-squares cycles.

At this stage one point emerged: the largest differences between $|F_{obs}|$ and $|F_{calc}|$'s were observed for pairs of F_{hkl} and F_{khl} when $|F_{calc}|$'s of the two were markedly different. This was ascribed to

TABLE 1. Observed and Calculated Structure Factors

Column headings throughout: | h | k | $|F_o|$ | $|F_c|$ |

Column group 1

ℓ = 0

h	k	$|F_o|$	$|F_c|$
2	0	48.5	49.9
4	0	685.4	-685.6
6	0*	16.1	-10.2
8	0	194.5	196.2
10	0	114.5	-119.0
12	0	65.9	-68.5
14	0*	26.8	38.8
2	2	48.8	46.8
4	2	397.4	388.3
6	2	87.5	94.0
8	2	296.3	-300.7
10	2	106.1	105.3
12	2*	24.4	43.9
14	2	74.6	-77.6
2	4	130.3	130.1
4	4	221.2	228.3
6	4	25.0	-18.4
8	4	23.8	13.4
10	4	113.0	110.2
12	4	63.5	-73.1
14	4*	27.5	-21.9
2	6	186.5	-186.7
4	6	212.8	-207.6
6	6*	20.3	-43.0
8	6	139.4	140.2
10	6*	23.7	46.6
12	6*	26.0	29.2
14	6*	28.5	-39.5
2	8*	25.0	-7.1
4	8	139.9	-140.4
6	8	116.1	-121.1
8	8*	23.3	-25.5
10	8	57.5	58.2
12	8*	27.4	51.5
2	10*	25.0	-11.8
4	10	76.6	70.0
6	10	79.0	76.7
8	10	101.2	-106.2
10	10	122.6	-119.6
2	12*	24.4	-25.1
4	12	152.8	155.6
6	12*	26.0	5.9
8	12*	27.4	-22.8
2	14	40.3	41.6
4	14	87.0	-87.4
6	14	66.4	-72.2

ℓ = 1

h	k	$|F_o|$	$|F_c|$
1	0	38.5	-40.5
3	0*	11.5	19.6
5	0*	14.7	0.6
7	0*	17.6	14.8
9	0	130.2	-134.8
11	0*	22.9	1.7
13	0*	25.5	46.8
15	0*	28.2	-5.2
2	1	110.5	106.5
4	1	69.6	-69.2
6	1	71.9	71.6
8	1	111.1	110.7
10	1	46.2	49.9
12	1	40.6	-34.1
14	1	78.7	-88.2
1	2	157.2	-155.4
3	2	128.6	129.6
5	2	105.1	-94.2
7	2	130.5	-133.2
9	2	44.9	33.7
11	2	104.7	110.5
13	2*	25.7	-54.7
15	2	81.8	-80.8
2	3	73.0	-68.4
4	3*	14.7	6.0
6	3	39.5	26.0
8	3	55.3	58.0
10	3	154.1	155.9
12	3	55.3	62.7
14	3	91.3	-87.6
1	4	87.9	89.3
3	4	31.0	-23.1
5	4	101.3	-98.0
7	4*	19.0	17.7
9	4	140.4	139.5
11	4	23.8	-39.7
13	4*	26.3	1.5
15	4*	28.9	3.9
2	5	26.6	-22.5
4	5	70.6	71.0
6	5	71.0	-68.2
8	5	148.9	-144.8
10	5	20.3	11.7
12	5	80.2	84.0
14	5	71.5	62.7
1	6	27.9	20.7
3	6	115.9	-118.3
5	6	72.2	73.3
7	6	136.0	135.0
9	6	83.0	-76.1
11	6	82.3	-80.2
13	6	120.5	115.4
2	7*	25.0	14.2
4	7	19.0	10.2
6	7*	25.0	-6.8
8	7	163.4	-160.2
10	7	90.0	-97.6
12	7*	26.7	10.5
1	8	13.5	-16.9
3	8	55.0	49.5
5	8	21.4	-11.6
7	8	24.2	-36.8
9	8*	24.3	-29.5
11	8*	26.3	19.5
13	8*	28.5	-7.2

Column group 2

h	k	$|F_o|$	$|F_c|$
2	9	20.6	47.7
4	9	79.2	-85.9
6	9	57.1	53.7
8	9	59.6	59.2
10	9	58.5	-57.7
12	9*	28.2	-45.2
1	10	232.4	227.8
3	10	103.1	-103.9
5	10	159.8	-160.9
7	10	19.1	12.5
9	10	85.5	83.3
11	10*	28.0	1.7
2	11	28.4	20.5
4	11*	23.8	18.9
6	11	42.5	40.6
8	11	79.6	74.9
10	11*	28.0	36.0
1	12*	24.3	-58.1
3	12*	24.7	19.4
5	12	67.9	64.0
7	12*	26.7	-44.5
9	12*	28.2	-20.8
2	13*	25.7	-14.5
4	13*	26.3	23.8
6	13*	27.3	-24.2
8	13*	28.5	-11.9
1	14	99.0	-102.9
3	14	85.7	88.2
5	14*	28.0	55.9
7	14*	28.4	-20.0
2	15*	28.4	-20.0
4	15*	28.9	7.8

ℓ = 2

h	k	$|F_o|$	$|F_c|$
2	0	82.5	-83.3
4	0	95.5	-93.6
6	0*	16.5	23.3
8	0	69.2	69.4
10	0	21.8	-3.9
12	0	24.4	-12.0
14	0*	27.0	18.3
1	1	110.8	-105.8
3	1	84.2	-83.1
5	1	66.8	68.1
7	1	121.0	121.2
9	1	61.5	-62.5
11	1*	23.1	-8.7
13	1	45.8	43.5
15	1*	28.3	1.8
2	2	12.0	10.9
4	2	42.4	41.0
6	2	62.6	-61.5
8	2	99.8	-98.1
10	2	22.0	20.5
12	2	75.6	72.0
14	2*	27.2	6.2
1	3	69.5	68.9
3	3	273.4	-268.1
5	3	25.0	13.1
7	3	129.6	126.3
9	3*	21.1	-35.2
11	3	68.5	-68.2
13	3	26.1	45.1
15	3*	28.7	36.0
2	4	68.8	61.7
4	4	32.5	-31.0
6	4*	18.1	-16.0
8	4*	20.4	9.4
10	4*	22.8	-23.0
12	4*	25.2	-33.7
14	4*	27.7	15.0
1	5*	15.3	-6.6
3	5	253.6	237.2
5	5	107.4	-106.1
7	5	189.7	-189.3
9	5	47.4	42.3
11	5	104.9	105.8
13	5	52.1	-50.4
2	6	27.4	-28.2
4	6	18.1	26.2
6	6	69.5	69.1
8	6	21.8	21.8
10	6	23.9	-36.0
12	6	26.2	-29.9
14	6*	28.6	-7.5
1	7	11.2	21.2
3	7	328.7	333.9
5	7	139.4	-138.9
7	7	156.8	-154.2
9	7	109.5	108.4
11	7	25.7	-24.3
13	7*	28.0	-41.8
2	8	48.7	42.6
4	8	20.4	35.7
6	8	21.8	-22.6
8	8	23.5	-0.2
10	8	25.4	36.6
12	8*	27.5	8.8
1	9	20.5	20.4
3	9	21.1	-29.1
5	9	71.5	62.3
7	9	79.2	79.9
9	9	60.9	-54.1
11	9	116.9	-116.3
1	10*	22.0	-28.8
3	10*	22.8	-30.1
5	10	23.9	1.9
7	10	25.4	2.5
9	10*	27.2	1.7
1	11*	23.1	9.2
3	11	66.4	-63.4
5	11	41.1	38.6
7	11	25.7	11.6
9	11*	27.3	-27.4

ℓ = 4

h	k	$|F_o|$	$|F_c|$
0	0	732.6	-744.9
2	0	96.6	95.1
4	0	309.8	321.0

Column group 3

h	k	$|F_o|$	$|F_c|$
2	12*	24.6	-42.7
4	12*	25.2	-17.6
6	12*	26.2	3.8
8	12*	27.5	-1.4
1	13*	25.7	10.3
3	13*	26.1	-36.9
5	13*	26.9	-19.4
7	13*	28.0	-18.9
2	14*	27.2	33.8
4	14*	27.7	4.5
6	14*	28.6	-13.2
1	15*	28.3	4.1
3	15*	28.7	1.2

ℓ = 3

h	k	$|F_o|$	$|F_c|$
1	0	43.0	-46.6
3	0	86.6	-82.7
5	0	88.3	75.8
7	0	135.1	133.2
9	0	148.2	149.9
11	0*	23.3	-26.6
13	9*	25.9	-25.4
15	0*	28.5	0.6
2	1	12.7	-2.7
4	1	58.7	-49.2
6	1	45.5	-43.8
8	1*	19.6	4.1
10	1	73.4	79.3
12	1*	24.6	-6.3
14	1	72.4	-65.2
1	2	108.2	-112.2
3	2	274.1	-266.3
5	2	137.4	122.8
7	2	179.4	182.7
9	2*	21.1	2.3
11	2	83.4	-77.1
13	2	49.0	49.7
15	2	84.2	81.1
2	3	189.6	192.9
4	3	35.9	40.1
6	3	117.5	-118.4
8	3	36.2	-50.3
10	3	80.2	79.3
12	3*	25.1	38.2
14	3	59.0	-56.9
1	4	75.0	80.0
3	4	45.3	37.5
5	4	79.6	-79.3
7	4	122.2	-118.3
9	4	108.7	-107.6
11	4	52.9	46.6
13	4*	26.7	8.5
2	5	33.5	-39.5
4	5	128.1	123.9
6	5	62.1	66.6
8	5	120.9	-118.2
10	5	36.1	-33.9
12	5	59.4	55.5
14	5*	28.3	10.3
1	6	99.1	106.2
3	6	119.3	123.0
5	6	51.6	-48.9
7	6	168.6	-171.1
9	6*	23.1	-26.7
11	6	63.3	62.2
13	6	68.4	-70.6
2	7	95.7	-97.5
4	7	35.6	32.3
6	7	74.7	82.2
8	7	71.2	-29.4
10	7	24.9	-20.1
12	7	27.0	-27.6
1	8	109.3	-112.5
3	8	65.8	61.8
5	8	131.6	133.7
7	8	22.9	40.1
9	8	28.2	32.3
11	8	26.7	-30.1
13	8	28.9	-48.0
2	9	21.1	28.4
4	9	180.8	-188.8
6	9	23.1	13.6
8	9	82.4	82.6
10	9	26.5	-41.7
12	9	28.5	4.9
1	10	183.3	-188.7
3	10	22.6	33.5
5	10	108.3	114.5
7	10	85.9	90.6
9	10	26.5	-4.6
11	10	28.3	-36.0
2	11	48.2	56.1
4	11	24.2	44.0
6	11	25.3	-51.4
8	11	26.7	-55.0
10	11	28.3	25.0
1	12	24.6	22.2
3	12	25.1	-43.9
5	12	60.8	-65.9
7	12	27.0	-5.2
9	12	28.5	-5.1
2	13	26.1	36.1
4	13	26.7	34.8
6	13	27.6	-23.4
8	13	28.9	9.9
1	14	125.4	125.0
3	14	92.0	-95.3
2	15	64.0	-61.8

Column group 4

ℓ = 4 (continued)

h	k	$|F_o|$	$|F_c|$
6	0	44.1	-45.7
8	0	143.9	-146.3
10	0*	22.4	32.7
12	0*	24.9	37.7
14	0*	27.5	19.4
1	1	38.6	42.7
3	1	177.4	176.1
5	1	35.6	-32.7
7	1	189.1	-189.1
9	1*	21.3	-30.3
11	1	92.9	94.1
13	1*	26.2	12.2
15	1*	28.8	-26.9
2	2	14.2	15.3
4	2	34.6	23.5
6	2	15.1	21.5
8	2	112.3	112.8
10	2	140.9	-145.4
12	2*	25.1	-22.0
14	2*	27.7	39.5
1	3	91.0	90.0
3	3	104.4	-105.8
5	3*	17.5	-2.0
7	3	154.6	154.8
9	3	22.0	8.2
11	3	139.8	-141.0
13	3*	26.6	-17.4
2	4*	15.9	-29.2
4	4	140.1	-147.9
6	4	51.5	52.5
8	4	93.8	98.5
10	4	80.4	-79.3
12	4*	25.7	-3.9
14	4*	28.2	-0.9
1	5	86.5	-89.4
3	5*	17.5	-26.6
5	5	112.4	111.6
7	5	54.3	53.4
9	5	46.9	-47.2
11	5*	25.0	-0.9
13	5*	27.4	5.9
2	6	85.4	-80.0
4	6*	19.1	6.3
6	6	102.0	104.0
8	6	62.7	-63.5
10	6	24.5	-27.0
12	6*	26.7	18.2
1	7	11.2	22.6
3	7	107.7	114.8
5	7	58.4	-61.2
7	7	88.8	-92.4
9	7*	24.2	62.6
11	7	78.7	71.7
13	7*	28.5	-33.2
2	8	28.9	26.6
4	8	114.1	109.2
6	8*	22.4	-21.7
8	8	39.0	-35.3
10	8	25.9	14.9
12	8*	28.0	-11.3
1	9*	21.3	-17.2
3	9	83.7	-88.4
5	9*	22.8	-16.3
7	9	49.4	40.4
9	9*	25.8	2.7
11	9*	27.8	-19.3
2	10	125.4	130.5
4	10	38.6	-47.7
6	10	74.2	-82.1
8	10	80.3	81.4
10	10*	27.7	42.4
1	11	54.5	-51.5
3	11	53.3	-45.3
5	11	25.0	23.0
7	11	26.2	6.1
9	11*	27.8	-10.4
1	12*	25.1	-1.8
4	12*	66.7	-73.7
6	12	70.5	71.4
1	12*	28.0	3.2
1	13	48.0	52.2
3	13*	26.6	58.0
5	13*	27.4	-11.3
7	13*	28.5	-30.1
2	14	47.8	-46.5
4	14	77.8	72.1
1	15*	28.8	30.7

ℓ = 5

h	k	$|F_o|$	$|F_c|$
1	0	14.4	-918
3	0	118.0	-115.6
5	0*	17.4	-0.1
7	0	43.0	47.4
9	0	72.9	72.5
11	0	80.0	-84.6
13	0	41.8	-40.8
2	1	85.6	-79.6
4	1	29.7	31.0
6	1*	18.5	-1.8
8	1	20.7	-16.1
10	1*	23.0	-26.6
12	1	46.3	-39.4
14	1*	27.9	27.0
1	2	53.5	66.0
3	2	102.5	-95.6
5	2	48.7	43.7
7	2	19.8	25.4
9	2*	22.0	-36.5
11	2	42.6	-40.9
13	2*	26.8	42.1
2	3	22.6	-9.8
4	3*	17.4	21.6
6	3	39.2	-35.4
8	3	58.8	-52.9

Column group 5

ℓ = 5 (continued)

h	k	$|F_o|$	$|F_c|$
10	3	90.1	-87.7
12	3*	25.8	-14.9
14	3	75.2	70.8
1	4	58.1	50.8
3	4	56.5	64.7
5	4	4.5	-4.3
7	4*	20.7	-11.6
9	4*	22.8	-26.2
11	4	72.3	68.2
13	4*	27.3	-40.6
2	5	51.6	44.9
4	5	91.3	-96.1
6	5*	20.4	-1.2
8	5	101.8	101.8
10	5*	24.4	-1.8
12	5*	26.6	-35.7
1	6	48.9	-49.5
3	6	41.6	50.2
5	6*	20.4	16.0
7	6	22.0	-29.6
9	6*	23.9	40.9
11	6	47.4	38.2
13	6	66.5	-62.7
2	7*	19.8	-41.2
4	7*	20.7	-42.7
6	7*	22.0	43.3
8	7	149.9	150.4
10	7*	25.6	43.1
12	7	38.9	-52.9
1	8*	20.7	-39.9
3	8*	21.2	-4.1
5	8	37.3	30.8
7	8	27.0	-32.3
9	8*	25.4	-24.5
11	8*	27.3	4.3
2	9*	22.0	-23.1
4	9	81.7	82.5
6	9*	23.9	-26.3
8	9*	25.4	-48.0
10	9*	27.2	41.5
1	10	115.5	-121.5
3	10	72.5	78.9
5	10	53.0	52.5
7	10	46.3	-45.3
9	10	60.6	-52.5
2	11*	24.4	19.1
4	11*	25.0	48.3
6	11	26.0	-70.8
8	11	92.3	-93.3
1	12*	25.4	41.5
3	12*	25.8	-50.0
5	12*	26.6	-22.6
7	12	91.9	88.6
2	13*	26.8	-7.1
4	13	70.1	-67.8
6	13*	28.3	34.5
1	14	58.1	61.6
3	14*	28.3	-40.0

ℓ = 6

h	k	$|F_o|$	$|F_c|$
2	0	154.3	154.6
4	0	136.0	138.7
6	0	94.8	-97.8
8	0	121.9	-123.4
10	0*	23.5	35.0
12	0*	25.9	14.3
14	0*	28.3	-37.8
1	1*	19.4	17.8
3	1	53.1	57.3
5	1	50.4	-51.6
7	1	105.7	-109.7
9	1	44.9	50.4
11	1	24.7	22.4
13	1*	27.1	-23.4
2	2*	16.6	0.6
4	2	85.7	-87.5
6	2	113.7	113.5
8	2	172.3	166.9
10	2	74.6	-75.3
12	2	127.9	-127.6
14	2*	28.5	16.7
1	3	71.5	-69.3
3	3	164.7	164.0
5	3	25.0	-7.1
7	3	93.6	-99.3
9	3*	22.9	-16.6
11	3*	25.1	14.2
13	3*	27.5	-6.4
2	4	95.0	-93.0
4	4	102.3	106.0
6	4*	20.5	47.3
8	4	46.4	-49.7
10	4*	24.4	34.0
12	4*	24.6	-46.8
1	5	44.0	39.8
3	5	119.3	-122.7
5	5	48.7	55.4
7	5	97.7	100.4
9	5*	23.9	-19.8
11	5*	26.0	-41.8
13	5*	28.2	21.2
2	6	46.6	48.4
4	6*	20.5	-18.9
6	6	110.3	-110.1
8	6*	23.5	-9.0
10	6	79.3	75.0
12	6*	27.6	23.5
3	7	19.8	23.5
5	7	253.8	-257.6
7	7	144.4	147.3
9	7*	60.7	-57.4
11	7*	27.1	11.2

Column group 6

ℓ = 6 (continued)

h	k	$|F_o|$	$|F_c|$
2	8	107.1	-109.0
4	8	108.9	-113.0
6	8	57.8	57.7
8	8*	25.0	34.3
10	8	65.2	-65.4
12	8*	28.8	-12.9
1	9	82.2	-89.1
3	9*	22.9	11.6
5	9*	23.9	37.3
7	9*	25.1	-40.6
9	9*	26.7	-25.1
11	9	66.3	69.5
2	10*	23.7	35.7
4	10*	24.4	37.9
6	10*	25.5	-31.2
8	10*	26.8	-26.9
10	10*	28.5	20.6
1	11*	24.7	17.2
3	11	66.9	66.1
5	11	66.5	-69.2
7	11*	27.1	-32.9
9	11	55.5	54.9
2	12	110.1	115.3
4	12*	26.6	43.7
6	12*	27.6	-34.9
8	12*	28.8	-12.8
1	13*	27.1	23.5
3	13*	27.5	3.1
5	13*	28.2	-30.3
2	14*	28.5	-50.5

ℓ = 7

h	k	$|F_o|$	$|F_c|$
1	0	67.7	76.0
3	0	61.9	63.8
5	0	93.6	-98.4
7	0	108.0	-111.8
9	0*	23.1	-17.2
11	0	62.2	62.2
13	0*	27.6	-46.8
2	1	32.5	33.9
4	1	38.7	36.2
6	1	46.4	42.9
8	1	67.0	56.6
10	1*	24.2	-9.1
12	1*	26.5	-28.6
14	1*	28.9	-21.9
1	2	93.0	94.7
3	2	157.6	154.9
5	2	67.9	-76.1
7	2	117.2	-108.2
9	2*	23.3	-9.4
11	2	63.1	56.9
13	2*	27.8	7.2
2	3*	160.3	-161.1
4	3	89.4	-86.9
6	3	140.6	135.0
8	3	158.7	159.4
10	3*	24.7	-12.0
12	3*	26.9	-45.1
1	4	165.3	-168.0
3	4	45.6	-43.3
5	4	122.3	123.3
7	4	60.6	60.5
9	4*	25.0	11.1
11	4*	26.1	-17.4
13	4*	28.3	27.6
2	5	27.4	-22.3
4	5	90.4	-94.9
6	5	21.9	-45.3
8	5	43.1	40.8
10	5*	25.5	7.6
12	5*	27.6	14.4
1	6	31.2	-37.1
3	6	47.5	-56.2
5	6	57.1	-50.7
7	6	107.5	101.3
9	6	78.0	77.5
11	6	65.9	-64.2
2	7	115.5	116.2
4	7*	22.1	-19.9
6	7	158.4	-159.3
8	7*	24.9	-55.7
10	7*	26.7	13.6
12	7	28.7	28.7
1	8	203.3	209.2
3	8	73.2	-84.0
5	8	128.1	-132.6
7	8*	24.9	21.3
9	8*	26.5	-28.9
11	8*	28.3	-1.9
2	9	23.3	27.7
4	9	116.1	118.8
6	9*	25.1	-16.2
8	9	26.5	-44.0
10	9*	28.2	18.4
1	10	24.2	40.3
3	10	24.7	28.7
5	10	25.5	16.1
7	10	98.0	-98.8
9	10*	28.2	-39.6
2	11	70.7	-73.3
4	11*	26.1	30.4
6	11	111.6	119.6
8	11	54.1	57.4
1	12*	26.5	-34.8
3	12	26.9	14.4
5	12*	27.6	18.5
7	12	28.7	25.9
2	13*	27.8	-38.9
4	13*	28.3	-14.4
1	14	76.2	-75.9

Column group 7

ℓ = 8

h	k	$|F_o|$	$|F_c|$
0	0	236.1	236.3
2	0	115.4	-116.5
4	0	100.5	-105.8
6	0	59.1	59.6
8	0*	22.9	19.8
10	0*	24.9	-41.9
12	0*	27.1	22.1
1	1	37.2	-37.9
3	1	147.1	-142.4
5	1	30.8	24.7
7	1	171.6	171.0
9	1	44.5	49.8
11	1	85.6	-81.9
13	1*	28.3	-25.6
2	2*	19.0	-20.6
4	2	95.2	-93.3
6	2	44.0	-45.7
8	2	48.0	-54.6
10	2	80.4	88.0
12	2*	27.3	-7.7
1	3	72.5	-69.9
3	3	85.8	83.3
5	3*	21.0	-7.3
7	3	127.6	-130.2
9	3*	24.4	4.0
11	3	139.9	137.9
13	3*	28.6	22.0
2	4	63.9	66.4
4	4	77.6	73.3
6	4	96.7	-94.3
8	4	66.5	-61.0
10	4	91.6	104.6
12	4*	27.8	-2.4
1	5	81.7	83.6
3	5*	21.0	39.5
5	5	87.0	-91.2
7	5	88.6	-83.4
9	5*	25.2	20.3
11	5*	27.3	14.7
2	6	102.9	105.4
4	6	40.9	41.6
6	6	53.1	-51.3
8	6	49.2	56.3
10	6*	26.7	-4.3
12	6*	28.7	-40.6
1	7*	25.0	-10.0
3	7	98.1	-93.1
5	7	48.2	54.3
7	7	89.2	82.8
9	7	64.9	-63.8
11	7	87.8	-88.9
2	8	64.6	-74.6
4	8*	23.8	-11.8
6	8	90.2	93.7
8	8	26.3	-13.1
10	8	62.6	-62.0
1	9*	23.9	-1.7
3	9	69.3	77.7
5	9	25.2	27.5
7	9*	26.4	-7.9
9	9*	27.9	-2.9
2	10	85.7	-92.0
4	10*	26.7	-16.7
6	10*	26.7	3.3
8	10*	28.0	-32.9
1	11	51.4	48.8
3	11*	25.0	26.4
5	11*	27.2	-30.7
7	11*	28.3	-1.1
2	12*	27.3	-13.0
4	12*	27.8	-3.1
6	12	65.3	-59.3
1	13*	28.3	-37.8
3	13*	28.6	-54.0

ℓ = 9

h	k	$|F_o|$	$|F_c|$
1	0	33.9	-33.8
3	0*	20.3	-0.3
5	0*	21.4	-1.9
7	0	50.1	56.1
9	0	56.3	60.6
11	0	98.2	99.4
2	1	76.6	75.0
4	1	78.4	-79.5
6	1	29.4	33.2
8	1	43.0	33.1
10	1	62.6	-63.6
12	1*	27.8	15.8
1	2	168.2	-167.2
3	2	56.5	-37.2
5	2*	21.7	-22.4
7	2	23.2	-10.9
9	2*	24.9	29.4
11	2	71.7	67.3
2	3	120.6	-120.3
4	3	56.3	56.1
6	3	90.0	96.3
8	3	49.6	-50.2
10	3*	26.1	3.7
12	3*	28.2	27.0
1	4*	20.9	-6.4
3	4*	21.4	-28.1
5	4*	21.9	-24.7
7	4	71.9	-74.4
9	4	69.3	-65.4
11	4	62.4	-56.7
2	5*	21.7	44.2
4	5*	22.2	-11.1
6	5	50.7	50.1
8	5*	25.1	-26.2
10	5	73.7	77.4

TABLE 1, Continued

h	k	$\lvert F_o \rvert$	$\lvert F_c \rvert$	h	k	$\lvert F_o \rvert$	$\lvert F_c \rvert$	h	k	$\lvert F_o \rvert$	$\lvert F_c \rvert$	h	k	$\lvert F_o \rvert$	$\lvert F_c \rvert$	h	k	$\lvert F_o \rvert$	$\lvert F_c \rvert$	h	k	$\lvert F_o \rvert$	$\lvert F_c \rvert$	h	k	$\lvert F_o \rvert$	$\lvert F_c \rvert$
1	6	143.2	147.4	7	1	86.0	90.1	1	9*	25.7	12.8	2	5*	23.9	19.1	6	2	32.5	35.2	2	1*	24.9	-6.6	2	2*	26.3	-15.1
3	6	20.9	25.0	9	1*	25.7	-3.2	3	9*	26.1	-43.4	4	5*	24.5	-0.0	8	2*	26.9	4.0	4	1	55.5	60.9	4	2*	26.9	15.9
5	6*	26.3	-37.8	11	1*	27.6	-33.7	5	9	113.9	-109.0	6	5	40.8	-41.2	10	2*	28.6	-50.0	6	1*	26.5	-12.1	6	2*	27.8	-38.8
7	6*	24.9	-2.8					7	9*	27.9	-43.3	8	5*	26.9	8.7					8	1*	27.8	5.0	1	3	53.0	-52.0
9	6*	26.5	-35.1	2	2	47.9	61.0	2	10*	26.7	-14.7	10	5	63.5	57.7	1	3*	24.0	7.4					3	3*	26.8	34.5
11	6	79.3	-69.1	4	2*	22.2	39.2	4	10*	27.3	3.4					3	3	53.1	-44.7	1	2	52.0	49.1	5	3*	27.5	-31.1
				6	2*	23.4	-39.6	6	10*	28.2	28.6	1	6	67.1	-67.4	5	3*	25.2	-9.2	3	2*	25.3	-18.0	7	3*	28.6	-4.3
2	7*	23.2	21.9	8	2	50.0	-58.3					3	6*	24.7	10.3	7	3*	26.4	35.9	5	2*	26.1	-6.2				
4	7*	23.8	-19.9	10	2*	26.7	17.2	1	11*	27.6	59.8	5	6	55.9	47.5	9	3*	27.9	-12.7	7	2*	27.3	7.7	2	4*	26.9	-4.5
6	7*	24.9	3.6	12	2*	28.7	33.2	3	11*	27.9	-23.6	7	6	68.0	-62.1					9	2*	28.7	19.6	4	4*	27.4	-15.0
8	7	37.8	-51.3					5	11*	28.7	0.9	9	6*	28.2	-21.1	2	4	46.0	-54.0					6	4*	28.3	-9.6
10	7*	28.0	-32.0	1	3	74.1	77.0	2	12*	28.7	-41.3					4	4*	25.1	-4.5	2	3*	25.3	2.1	1	5	38.4	33.2
				3	3*	22.1	-25.3					2	7	49.9	-56.5	6	4	92.8	104.3	4	3*	25.9	-53.7	3	5	93.0	-93.3
1	8*	23.8	38.1	5	3	86.4	100.3	$\ell = 11$				4	7*	25.8	-19.8	8	4*	27.5	-24.7	6	3*	26.9	-9.2	5	5*	28.3	-32.9
3	8*	24.3	7.7	7	3*	24.4	-24.3					6	7	102.4	101.4					8	3	98.8	93.5				
5	8*	25.1	13.6	9	3	95.4	-94.1	1	0	63.4	-69.1	8	7*	28.0	25.3	1	5*	24.8	2.5					2	6*	27.8	-22.8
7	8	81.3	87.4	11	3*	27.9	19.3	3	0	42.7	40.9					3	5*	25.2	-20.3	1	4	78.3	74.9	4	6*	28.3	7.5
9	8	49.1	51.3					5	0	72.7	69.7	1	8	126.9	-123.8	5	5*	26.0	-4.0	3	4*	25.9	30.9	1	7*	28.3	-4.0
				2	4*	22.2	8.1	7	0*	24.9	-15.6	3	8	50.2	49.7	7	5*	27.2	24.9	5	4*	26.7	-73.2	3	7*	28.6	-51.1
2	9*	24.9	-13.5	4	4*	22.9	-33.4	9	0	64.0	-65.5	5	8*	26.9	45.5	9	5*	28.6	-4.3	7	4*	27.8	17.3				
4	9*	25.5	20.5	6	4*	24.1	17.4	11	0	67.1	-62.6	7	8	80.9	-68.8									$\ell = 15$			
6	9*	26.5	25.5	8	4*	25.6	14.8					2	9*	26.7	-8.4	2	6	93.8	-96.7	2	5*	26.1	-35.1				
8	9*	27.8	-26.4	10	4*	27.3	-28.1	2	1*	22.5	30.2	4	9*	27.3	10.8	4	6	64.1	-60.3	4	5	53.5	-49.7	1	0*	27.2	17.8
1	10*	25.7	-21.8					4	1*	23.2	15.7	6	9*	28.2	33.1	6	6*	27.1	34.9	6	5*	27.6	22.7	3	0*	27.6	-13.7
3	10*	26.1	-49.5	1	5*	25.0	-16.0	6	1*	24.3	10.4					8	6	23.2	-24.8	1	6	71.4	-71.7	5	0*	28.3	-19.9
5	10*	26.9	14.7	3	5	232.4	239.1	8	1	38.9	-30.5	1	10*	27.5	27.9					3	6*	26.9	4.8	2	1	58.1	-51.1
7	10*	28.0	46.6	5	5	39.7	40.5	10	1*	27.5	-40.8	3	10*	27.9	-0.3	1	7*	26.0	-4.9	5	6.	75.2	71.9	4	1*	28.0	-3.6
				7	5*	25.2	-36.6					5	10*	28.6	-29.4	3	7*	26.4	34.0	7	6*	28.7	-11.1	6	1*	28.9	21.3
2	11*	26.9	14.7	9	5*	25.0	26.3	1	2	64.9	61.5	2	11	53.4	60.9	5	7*	27.2	6.5								
4	11	87.4	-94.0	11	5	50.0	-47.6	3	2*	22.9	-11.9					7	7*	28.3	-19.0	2	7*	27.3	11.6	1	2*	27.4	-51.1
6	11*	28.4	15.0					5	2*	23.9	11.7	$\ell = 12$								4	7*	27.8	-9.9	3	2*	27.8	-46.1
1	12*	27.8	-18.0	2	6*	23.4	-23.7	7	2	80.3	70.9					2	8*	26.9	58.1	6	7*	28.7	-39.2	5	2*	28.5	-7.9
3	12*	28.2	2.0	4	6*	24.1	-5.5	9	2*	26.7	7.7	0	0*	23.4	-5.0	4	8*	27.5	-35.2	1	8	86.5	-89.4	2	3*	27.8	34.5
				6	6*	25.1	19.0	11	2	78.5	-82.6	2	0	91.0	95.0	6	8	102.9	-102.9	3	8	17.2	24.9				
$\ell = 10$				8	6*	26.5	13.0					4	0*	24.3	-11.1	1	9*	27.6	-1.7	2	9*	28.7	22.5	1	4	62.6	-55.1
				10	6*	28.2	-5.8	2	3*	22.9	-23.2	6	0	63.5	-68.5	3	9*	27.9	-6.8					3	4*	28.3	24.9
2	0	43.0	-43.7					4	3	91.5	86.9	8	0*	26.7	28.5	5	9*	28.6	8.3	$\ell = 14$				2	5*	28.5	0.2
4	0*	22.0	-32.0	1	7	79.6	-85.7	6	3*	24.7	-12.9	10	0*	28.4	43.6	2	10	61.7	66.2					1	6	74.3	70.9
6	0*	23.2	19.5	3	7*	24.6	39.0	8	3	127.3	-114.3									2	0*	26.1	-7.9				
8	0	46.5	44.3	5	7*	25.2	-34.3	10	3*	27.9	-0.4	1	1*	23.5	8.3	$\ell = 13$				4	0*	26.7	-16.7	$\ell = 16$			
10	0*	26.5	-14.3	7	7*	26.4	19.7					3	1*	24.0	35.4					6	0*	27.6	10.6				
12	0*	28.6	-11.1	9	7	64.9	61.7	1	4	84.9	81.6	5	1*	24.8	-10.7	1	0*	24.7	3.1	8	0*	28.9	-15.5	0	0*	28.4	-26.7
								3	4	28.0	-29.4	7	1	54.6	-33.9	3	0*	25.1	-21.8					2	0	64.1	-59.8
1	1	215.2	216.5	2	8*	23.9	46.5	5	4*	24.5	-31.4	9	1*	27.6	1.3	5	0*	25.9	40.0	1	1	108.0	-15.7	1	1*	28.5	-3.9
3	1	204.5	-210.7	4	8*	25.6	40.6	7	4*	25.8	44.9					7	0*	27.1	18.3	3	1	93.9	101.4	3	1	28.9	31.2
5	1	184.9	-186.9	6	8*	26.5	-30.6	9	4*	27.3	38.2	2	2*	23.8	21.7	9	0	60.5	-61.1	5	1	77.6	72.5	2	2*	28.8	-15.4
				8	8*	27.8	-19.8					4	2	100.6	102.7					7	1*	28.3	-55.4	1	3	64.4	57.5

TABLE 2. Final Atomic Coordinates, Anisotropic Temperature Factors ($\times 10^4$)

Atom	x	y	z	$B_H(\mathring{A}^2)$	β_{11}	β_{22}	β_{33}	β_{12}	β_{13}	β_{23}
T(1)	0.0579(1)	0.3964(1)	0.1666(1)	0.90	15(1)	10(1)	13(1)	-1(1)	-1(1)	-2(1)
	0.0878(2)	*0.375*	*0.1622(2)*	*2.7*						
T(2)	0.1676(1)	0.6115(1)	0.1283(1)	0.89	12(1)	14(1)	11(1)	-2(1)	-1(1)	3(1)
	0.1622	*0.5878*	*0.125*							
T(3)	0.3924(1)	0.6418(1)	0.0860(1)	0.97	13(1)	16(1)	12(1)	1(1)	0(1)	-2(1)
	0.375	*0.6622*	*0.0878*							
O(1)	0.1318(4)	0.3131(4)	0.1100(4)	2.37	42(4)	25(3)	33(3)	0(3)	6(3)	-7(3)
	0.1322(10)	*0.2800(7)*	*0.1033(10)*	*7.3*						
O(2)	0.0921(4)	0.5107(3)	0.1303(4)	2.32	39(3)	24(3)	36(3)	-12(3)	1(3)	2(3)
	0.1467	*0.4700*	*0.1178*							
O(3)	0.1453(4)	0.6798(4)	0.2275(3)	1.82	33(3)	30(3)	16(3)	-6(3)	0(2)	-1(2)
	0.1033	*0.6322*	*0.2200*							
O(4)	0.1333(4)	0.6841(4)	0.0354(3)	1.88	29(3)	33(3)	18(3)	15(3)	5(2)	7(2)
	0.1178	*0.6467*	*0.0300*							
O(5)	0.2900(3)	0.5772(3)	0.1205(4)	1.99	23(3)	25(3)	35(3)	7(2)	-5(3)	2(3)
	0.2800	*0.6033*	*0.1322*							
O(6)	0.4826(3)	0.6174(4)	0.1667(3)	1.87	27(3)	26(3)	26(3)	-7(2)	0(2)	5(3)
	0.4700	*0.6178*	*0.1467*							
K	0.3660(2)	0.3645(2)	0.1147(2)	4.21	39(1)	46(1)	90(2)	-6(1)	0(1)	-15(1)
	0.375	*0.375*	*0.125*	*15.4*						

For comparison, atomic parameters of cubic leucite *(Peacor, 1968)* are written in italics below each corresponding tetragonal position. B_H-values are equivalent temperature factors after *Hamilton (1959)*. T's host 0.68 Si and 0.32 Al. K hosts 0.94 K, 0.05 Na and 0.01 Ca.

Standard deviations are indicated in parentheses in terms of last significant figures.

The anisotropic temperature factor has the form: $exp\ (-h^2\beta_{11} -k^2\beta_{22} -l^2\beta_{33} -2hk\beta_{12} -2hl\beta_{13} -2kl\beta_{23})$

TABLE 3. Dimensions and Orientations of Thermal Ellipsoids

Atom	Rms vib.	U^a	U^b	U^c	Rms vib.	U^a	U^b	U^c	Rms vib.	U^a	U^b	U^c
T(1)	0.09(1)	100 (9)	159 (9)	108 (9)	0.11(1)	22(50)	105(17)	74(54)	0.12(1)	70(56)	76(18)	156(37)
T(2)	0.09(1)	72(22)	48 (7)	133(15)	0.10(1)	29(16)	86(17)	62(19)	0.12(1)	69 (8)	138 (7)	124 (7)
T(3)	0.10(1)	95(45)	122(13)	148 (9)	0.11(1)	11(24)	101(27)	89(40)	0.12(1)	100(13)	146(10)	58(10)
O(1)	0.13(1)	82 (8)	150(10)	118 (9)	0.18(1)	127(15)	117(10)	49(14)	0.20(1)	142(15)	79 (9)	126(13)
O(2)	0.12(1)	119 (5)	150 (5)	83 (7)	0.19(1)	88(25)	83(16)	7(10)	0.20(1)	151 (5)	61 (6)	92(29)
O(3)	0.12(1)	92(11)	97(14)	173(16)	0.15(1)	54(13)	37(13)	97(17)	0.18(1)	143(13)	54(13)	92 (8)
O(4)	0.11(1)	57(33)	132(12)	59(47)	0.12(1)	124(32)	80(34)	36(43)	0.21(1)	129 (5)	136 (4)	107 (5)
O(5)	0.12(1)	139 (8)	52 (9)	103 (6)	0.16(1)	55(10)	38 (9)	77(16)	0.19(1)	72(11)	87(14)	162(13)
O(6)	0.13(1)	124(12)	136 (8)	66(12)	0.16(1)	48(18)	95(16)	42(19)	0.18(1)	60(16)	134 (8)	122(18)
K	0.17(1)	140 (6)	128 (5)	99 (2)	0.20(1)	130 (6)	43 (5)	77 (2)	0.30(1)	91 (1)	74 (1)	164 (1)

Principal vibrations (root-mean-square) are in Å; angles between crystallographic axes (a, b, c) and principal axes (U) of vibration ellipsoids are in degrees.
Standard deviations are indicated in parentheses in terms of the last significant figures.

the existence of merohedric twinning, which would superimpose the *hkl* diffraction of the first individual with that for *khl* of the second, and vice-versa. The relative weight percent of the two individuals was calculated as follows: 20 diffractions featuring relevant $|F_{obs} - F_{calc}|$ differences were selected. Let us call x the fraction of the first individual, $[1 - x]$ being the fraction of the second one: x was calculated so as to minimize the sum

$$\sum |F_{obs}^2(hkl) - \{xF_{calc}^2(hkl) + [1 - x]F_{calc}^2(khl)\}|$$

This minimum was obtained with $x = 0.82$. In the fol-

lowing step the solution of the system:

$$\begin{cases} 0.82F_{obs}^2(hkl)_{corr} + 0.18F_{obs}^2(khl)_{corr} = F_{obs}^2(hkl) \\ 0.18F_{obs}^2(hkl)_{corr} + 0.82F_{obs}^2(khl)_{corr} = F_{obs}^2(khl) \end{cases}$$

gave $F_{obs}(hkl)_{corr}$ and $F_{obs}(khl)_{corr}$ which were used in further steps of structure refinement.

Two more least-squares cycles with anisotropic temperature factors yielded $R = 0.046$ using the 511 observed reflections. Refinement including all reflections converged in the same number of cycles to $R = 0.152$. The assignment of different proportions

TABLE 4. Interatomic Distances and Angles

T(1)-tetrahedron		T(2)-tetrahedron		T(3)-tetrahedron		K-polyhedron	
T(1 I)-O(1 I)	1.652(6)Å	T(2 I)-O(2 I)	1.648(5)Å	T(3 I)-O(3IV)	1.644(5)Å	K(I)-O(1 I)	3.138(7)Å
-O(1 II)	1.637(6)	-O(3 I)	1.657(5)	-O(5 I)	1.654(6)	-O(2 VI)	3.036(7)
-O(2 I)	1.640(5)	-O(4 I)	1.653(5)	-O(6 I)	1.651(5)	-O(3VII)	2.986(7)
-O(4III)	1.645(5)	-O(5 I)	1.668(5)	-O(6 V)	1.676(5)	-O(4VII)	2.966(6)
						-O(5 I)	2.956(7)
mean	1.643	mean	1.656	mean	1.656	-O(6 IV)	3.004(7)
						mean	3.014
O(1 I)-O(1 II)	2.736(8)	O(2 I)-O(3 I)	2.677(7)	O(3IV)-O(5 I)	2.710(7)	K(I)-O(1III)	3.506(7)
-O(2 I)	2.652(8)	-O(4 I)	2.672(7)	-O(6 I)	2.698(7)	-O(3 IV)	3.759(7)
-O(4III)	2.721(7)	-O(5 I)	2.736(8)	-O(6 V)	2.705(7)	-O(3 VI)	3.724(7)
O(1II)-O(2 I)	2.644(8)	O(3 I)-O(4 I)	2.646(7)	O(5 I)-O(6 I)	2.652(7)	-O(4 VI)	3.634(7)
-O(4III)	2.617(7)	-O(5 I)	2.748(7)	-O(6 V)	2.728(8)	-O(5 IV)	3.617(7)
O(2 I)-O(4III)	2.729(7)	O(4 I)-O(5 I)	2.745(7)	O(6 I)-O(6 V)	2.728(8)	-O(6 I)	3.714(7)
						mean	3.659
O(1 I)-T(1 I)-O(1 II)	112.6(3)°	O(2 I)-T(2 I)-O(3 I)	108.2(3)°	O(3IV)-T(3 I)-O(5 I)	110.5(3)°	T(1 I)-O(1 I)-T(1 VI)	144.3(3)°
-O(2 I)	107.4(3)	-O(4 I)	108.1(3)	-O(6 I)	109.9(3)	-O(2 I)-T(2 I)	154.0(4)
-O(4III)	111.2(3)	-O(5 I)	111.2(3)	-O(6 V)	109.1(2)	T(2 I)-O(3 I)-T(3III)	129.7(3)
O(1II)-T(1 I)-O(2 I)	107.6(3)	O(3 I)-T(2 I)-O(4 I)	106.2(2)	O(5 I)-T(3 I)-O(6 I)	106.7(2)	-O(4 I)-T(1 IV)	140.8(3)
-O(4III)	105.7(3)	-O(5 I)	111.5(3)	-O(6 V)	110.5(2)	-O(5 I)-T(3 I)	131.2(3)
O(2 I)-T(1 I)-O(4III)	112.3(3)	O(4 I)-T(2 I)-O(5 I)	111.5(3)	O(6 I)-T(3 I)-O(6 V)	110.1(3)	T(3 I)-O(6 I)-T(3VII)	130.4(3)

Roman numerals refer to the following positions:

	x	y	z	III	3/4-y	1/4+x	1/4+z	V	3/4+y-1	1/4-x+1	1/4-z	VII	1/4-y+1	1/4+x	1/4-z
II	1/4-y	1/4+x	1/4-z	IV	3/4+y-1	3/4-x	3/4+z-1	VI	3/4+y-1	1/4-x	1/4-z				

Standard deviations are indicated in parentheses in terms of last significant figures.

relating the two individuals of the twin did not yield better R values.

The observed and calculated structure factors are compared in Table 1. Final atomic parameters and anisotropic thermal parameters are given in Table 2. Table 3 gives the thermal ellipsoid data. Bond distances and angles are listed in Table 4.

A comparison of the final atomic parameters with those deduced from the cubic structure adapted to the tetragonal system (Table 2), or with those proposed by Wyart (1940), shows differences larger than 1 Å. This explains the failure of least-squares refinement using these as starting coordinates.

Discussion of the structure

The tetrahedral framework of tetragonal leucite is topologically identical to that of the cubic phase

(Peacor, 1968); for the description of this framework see Taylor (1930). No (Si,Al) ordering has been detected, as foreseen by Peacor (1968) on the basis of the rapidity of the high-low inversion. The average $T(2)$–O and $T(3)$–O distances are in perfect agreement with the expected value (1.656 A) according to Jones (1968) for Al:(Si+Al) = 0.32. The average $T(1)$–O distance of 1.634 Å (0.013 shorter) would be proper for Al:(Si+Al) = 0.25. None of the framework atoms shows a marked anisotropic thermal vibration. The kind of displacements the framework undergoes when changing from the cubic to the tetragonal structure is shown in Figure 1. The changes pertain mainly to the shape of the cavities occupied by K atoms. To explain this point, let us consider the average K–O distances in feldspars, which range from 2.93 to 3.00 Å. The average value for 12-
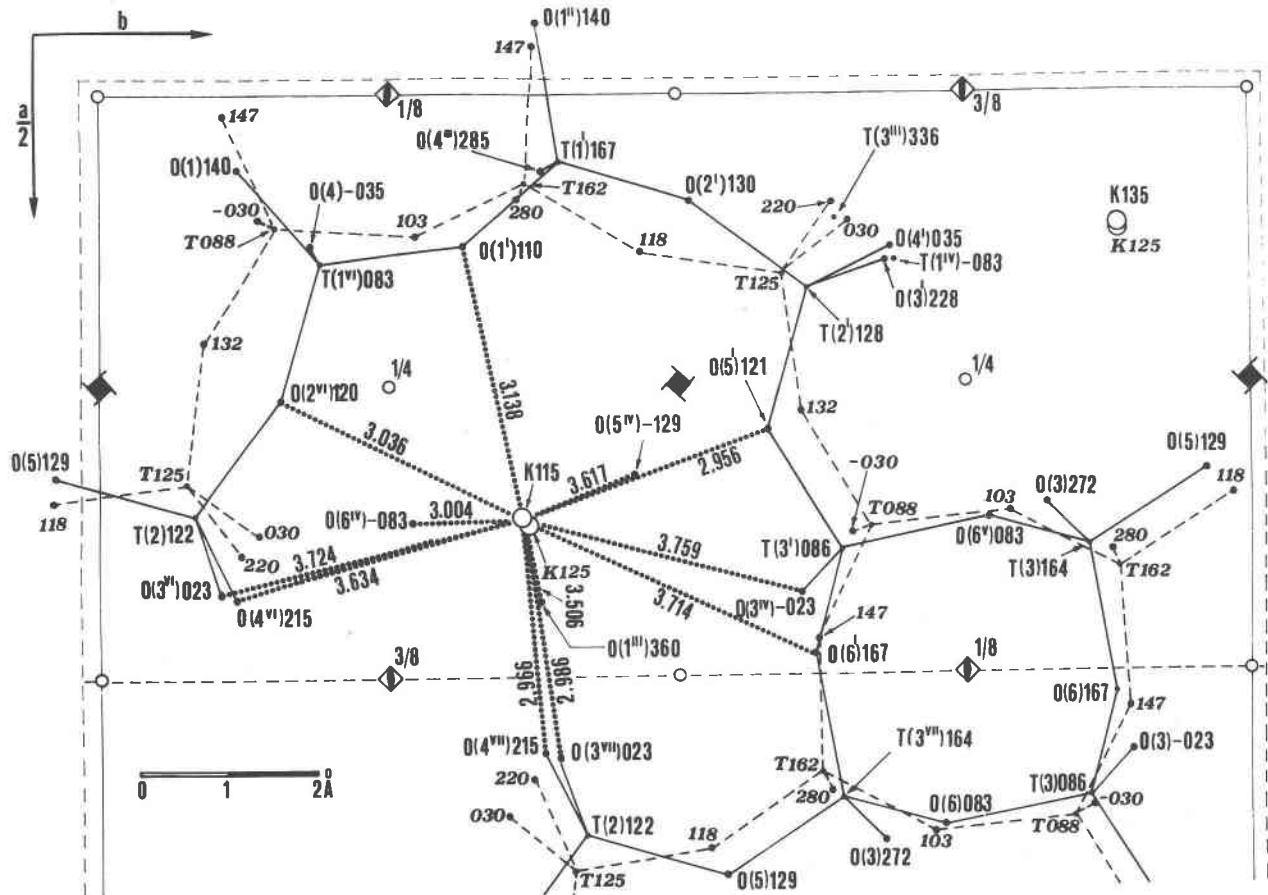
FIG. 1. The lower part (from z = 0 to z = 1/4) of the unit cell of leucite projected along c. Both the tetragonal and the cubic structures are shown. The tetragonal is shown as full lines, and the positions of the corresponding atoms are given in Roman letters (K, T, O) and three digit numbers which give the height over the projection plane as permillage of c. The cubic framework is shown as dashed lines, and the positions of the corresponding atoms are given in italic letters (K, T, no letter for oxygens) and numbers with the same meaning as before. The symmetry elements and the dotted lines (K–O bonds) refer to the tetragonal structure. The origin of the cubic cell is shifted by a/2 with respect to the tetragonal cell.

TABLE 5.   Some Bond Distances in Minerals with the Leucite Framework.

Mineral	Bond	Expected bond distances	Six shortest distances in the cubic structure	Average of the six shortest distances in the non-cubic structure
Leucite	K–O	2.98 Å (1)	3.35 Å (Peacor, 1968)	3.01 Å (this work)
Analcime	O(H_2O)–O	2.60–3.04 (2)	3.41 (Ferraris et al., 1972)	?
Pollucite	Cs–O	3.26 (1)	3.39 (Beger, 1969)	–

(1)   Highest possible average values (12-fold coordination) (Shannon and Prewitt, 1969)
(2)   Hamilton and Ibers, 1968

coordination is 2.98 Å (Table 5). In cubic leucite (Peacor, 1968) each K atom is coordinated to 12 oxygens, with 6 oxygens at 3.35 A and 6 at 3.54 A; these are proper values at high temperature, where the cubic phase is stable, but they are high for room temperature. In tetragonal leucite the K cavity is compressed, so that each atom is coordinated to 6 oxygens at ~3.0 Å, with 6 other oxygens at 3.5 to 3.8 Å. The latter distances are too long to be considered bond distances. The thermal vibrations of the K atoms are remarkably anisotropic.

Possible implications for structures with leucite framework

Analcime, which is isostructural with leucite, also exhibits a polymorphic inversion from a high-temperature form with space group $Ia3d$ to a low-temperature form of unknown symmetry (see, for instance, Coombs, 1955). The change of lattice parameters is less marked than in leucite, but in the powder pattern some peak-broadening occurs and, in one case, peak-splitting. At least four lines are present which do not conform to the condition "hhl: $2h + l = 4n$" of the space group $Ia3d$. Their spacings are 6.88, 3.24, 2.36, and 2.29 Å. The first was indexed as 200 by Coombs (1955), the second is either 411 or 330, the third is 530 or 433, the fourth is 600 or 442. Reflections 200, 411, 433, 600, and 442 are all compatible with the space group of low-leucite $I4_1/a$, and so we may advance the hypothesis that the low-temperature symmetry is the same for both analcime and leucite, and that the cubic structure is displaced in the same way for both minerals during the high-low inversion. On the other hand the chemical analogy is not as strong because, in analcime, water molecules occupy the equivalent K sites of leucite and Na enters sites that are vacant in leucite.

With the aim of giving a better explanation of the similarities between the high-low inversions of leucite and analcime, we have collected some data in Table 5 concerning these two minerals and iso-structural pollucite. A first glance allows us to conclude that in the cubic structures the cavity is too large for K and H_2O, but not for Cs; thus at lower temperature a tetragonal structure is more stable when the cavity is occupied by K (leucite) or by H_2O (analcime), but no evidence has been given so far of the existence of a non-cubic structure for pollucite, Cs[AlSi$_2$O$_6$], and in our opinion the cubic phase is the only one possible for this compound.

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