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## **Crystal Chemistry, Unit Cell Dimensions, and Structural State of Labradorite Megacrysts from Sonora, Mexico**

By *James T. Gutmann* (Middletown, Conn., U.S.A.)\* and  
*Robert F. Martin* (Montreal, Quebec, Canada)\*\*)

With 2 tables in the text

### **Abstract**

Twelve complete chemical analyses of single crystals of labradorite are presented together with refined unit cell parameters for each. The crystals are gem-quality megacrysts collected from flows and pyroclastic units of hawaiite in the Pinacate volcanic field, Sonora, Mexico. They span a compositional range of 7 mol% anorthite but are very similar to one another with respect to minor element content. An hypothesis of possible departure of these crystals from stoichiometry toward excess silica is considered and rejected at this time. The X-ray diffraction data indicate a significant range in structural state. The relative degrees of ordering implied by these data evidently reflect the thermal histories of the volcanic specimens and indicate that even crystals from mafic pyroclastic units can attain considerable Si-Al order.

### **INTRODUCTION**

The Pinacate volcanic field is located in northwestern Sonora, Mexico, east of the northern end of the Gulf of California. The field contains numerous alkali basalt and hawaiite flows of late Pleistocene to Holocene age and is characterized by an abundance of basaltic pyroclastic deposits, including more than 300 cinder cones. The lavas typically are porphyritic, with phenocrysts of labradorite, olivine, augite, and magnetite. These crystals exhibit a seriate size distribution ranging from tiny microphenocrysts continuously upward to megacrysts which, in the case of labradorite, attain as much as 10 cm in length.

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Thin sections of these large labradorite crystals demonstrate that most are remarkably homogeneous in composition. With the exception of exceedingly thin rims, which may be slightly more sodic or calcic than cores, the bulk of each crystal either is free of optically detectable zonation or is zoned only through a very narrow compositional range, generally less than 1% An. Many of the megacrysts are of gem quality. Accordingly, these large feldspars afford the mineralogist an opportunity to gather various chemical and physical data not only from the same crystal but also with the assurance that all samples from that crystal are essentially identical in composition.

A more complete understanding of the plagioclase feldspars and of the myriad factors that influence their structures and cell dimensions will result from the establishment of a large body of data representing carefully and completely characterized samples throughout the compositional range. Such data from volcanic labradorites are available but sparse. Electron microprobe analyses fail to differentiate between FeO and Fe<sub>2</sub>O<sub>3</sub>. We here present complete chemical analyses and refined unit cell parameters for twelve crystals of labradorite ranging in composition from An<sub>57</sub> to An<sub>64</sub>.

The volcanic units from which these crystals were collected are located at or near Crater Elegante, a large collapse depression in the eastern part of the lava field. These units are noted in Table 1 and are fully described by GUTMANN (1972; in press). Six of the samples were loose megacrysts within cinder cones and six were collected from flows. Two crystals were studied from each volcanic unit. Each crystal consists of colorless to pale citrine, gem-quality feldspar with vitreous luster, conchoidal fracture, and relatively difficult cleavages. Most of the crystals are simply and sparsely twinned, in general according to the albite law. The analyzed crystals were 3 to 5 cm in maximum dimension, except that specimen PE-X19 was a composite sample of three smaller, unzoned grains with identical refractive indices, from a polygranular mass of feldspar and oikocrystic olivine. Crystal forms ranged from euhedral to subhedral; shapes were elongate parallel to *a*, equant, or tabular parallel to (010). Many of the associated phenocrysts are skeletal, and some of the labradorite megacrysts in Pinacate lavas contain tubular fluid inclusions of primary origin, indicating that water-rich fluid was exsolving from the magmas as the crystals grew (GUTMANN, 1974). Evidently many of these phenocrysts formed rapidly and at relatively shallow depth, although the homogeneity of the feldspar megacrysts indicates sensibly constant magmatic conditions during their growth.

The samples were prepared for analysis by careful handpicking of crushed transparent grains under a binocular microscope. Examination of crystal fragments at magnifications up to 800× revealed no minute inclusions; the constancy of ferrous iron concentration among the samples (Table 1) further suggests freedom of the analyzed powders from contamination with either basalt

Table 1. *Chemical Analyses of Labradorite Megacrysts*

	PS-X1	PS-X4	PE-X2	PE-57	PE-X12	PE-X13	PE-X14	PE-X15	PE-X16	PE-X17	PE-X18	PE-X19
SiO <sub>2</sub>	52.8%	52.8	53.5	52.42	53.5	53.5	53.5	53.5	54.0	53.8	53.0	53.0
Al <sub>2</sub> O <sub>3</sub>	29.1	28.8	29.4	29.7	28.4	28.6	29.1	29.4	28.6	29.2	29.2	29.6
Fe <sub>2</sub> O <sub>3</sub>	0.34	0.42	0.39	0.36	0.38	0.36	0.48*	0.38	0.52*	0.40	0.32	0.46
TiO <sub>2</sub>	0.08	0.08	0.09	0.09	0.08	0.08	0.08	0.08	0.10	0.10	0.09	0.10
CaO	12.8	12.2	12.3	12.65	11.9	12.4	12.1	12.0	11.4	11.6	12.4	12.3
FeO	0.13	0.13	0.13	0.13	0.13	0.13	**)	0.13	**)	0.13	0.19	0.08
SrO	0.110	0.105	0.100	0.105	0.094	0.094	0.100	0.096	0.105	0.105	0.105	0.100
MgO	0.07	0.08	0.07	0.08	0.10	0.10	0.09	0.10	0.04	0.05	0.09	0.09
Na <sub>2</sub> O	3.96	3.90	3.96	4.07	3.96	4.13	4.21	4.26	4.59	4.33	3.86	4.01
K <sub>2</sub> O	0.25	0.23	0.24	0.21	0.21	0.22	0.25	0.26	0.33	0.28	0.24	0.25
Total	99.64	98.75	100.18	99.82	98.75	99.61	99.91	100.21	99.69	100.00	99.50	99.99
Z	3.981	3.994	3.998	3.990	3.994	3.980	3.992	3.997	3.994	4.004	3.992	4.000
X	1.005	0.976	0.972	1.004	0.964	0.998	0.989	0.986	0.991	0.972	0.979	0.979
Ab	34.74	35.42	35.59	35.64	36.34	36.40	37.34	37.76	40.61	38.91	34.71	35.86
"An"	63.84	63.20	62.98	63.15	62.38	62.33	61.20	60.74	57.46	59.45	63.86	62.68
Or	1.44	1.38	1.43	1.22	1.28	1.28	1.45	1.51	1.92	1.64	1.43	1.47
Source Units	Cinders of Salva- terra Cinder Cone	Upper Brown Cin- ders, Crater Elegante	Gray Cinders, Crater Elegante	Flow Ib <sub>1</sub> , Crater Elegante	Flow Ib <sub>3</sub> , Crater Elegante	The Wedge, Crater Elegante						

Analyses performed by NORMAN H. SUHR as described in text. Figures for Ab, An, and Or in mol %. BaO and MnO < 0.01% in all samples. "An" reflects content of all X cations except Na and K, as described in text.

\*) Total Iron. \*\*) Not determined, but estimated to be 0.13% for purposes of recalculation (c.f., the other analyses).

glass or coexisting mineral phases. The plagioclases were fused in a lithium metaborate flux, and then dissolved. Silica and lime were determined by atomic absorption as described by MEDLIN et al. (1969), the alkalis by flame photometry, ferrous iron by titration, and all other constituents by direct-reading emission spectrometry of the solution, as described by SUHR and INGAMELLS (1966). However, for sample PE-57, silica and alumina were determined gravimetrically and lime by titration in order to check the accuracy of determinations by the above techniques. BaO and MnO are less than 0.01% in all samples. Analyses and calculated site occupancies are presented in Table 1.

A portion of the powder submitted for analysis was mixed with a spinel internal standard ( $a = 8.0833 \text{ \AA}$ ) and X-rayed at room temperature either with a Guinier-de Wolff camera (samples PE-X2, PS-X4, PE-X12, and PE-X13) or a Guinier-Hägg focusing powder camera (Cu  $K_{\alpha_1}$  radiation,  $\lambda = 1.54050 \text{ \AA}$ ). The corrected  $2\theta$  values were uniquely indexed and used as input for the cell-parameter refinement program of APPLEMAN and EVANS (1973). The resulting unit cell parameters are presented in Table 2.

## DISCUSSION

The analyses presented in Table 1 show that the megacrysts contain relatively constant amounts of ferric iron, titanium, ferrous iron, strontium, magnesium, and potassium. Two crystals, PE-X16 and PE-X17, are somewhat poorer in calcium than the other ten and contain slightly more sodium and potassium. These two plagioclases also are relatively poor in magnesium, which suggests that Mg is positively correlated with mol % An, as noted by SMITH (1974, p. 107). The pale citrine color of many of these large labradorites presumably reflects their high ferric iron contents: the yellow color of ferri-ferrous orthoclase has been linked to the optical absorption spectrum of tetrahedrally bonded ferric iron by FAYE (1969). It should be noted that the relatively high contents of ferric iron, magnesium, and titanium in these crystals evidently increase their refractive indices substantially: anorthite contents estimated via refractive indices in (001) cleavage flakes are in excess of analytical values by 2–5% An, and the discrepancies vary directly with the total amounts of these minor elements present in the crystals.

The chemical data have been recast to balance 16.000 negative charges, i.e., the structural formula is taken as  $X Z_{4.000} O_{8.000}$ . The X site is occupied primarily by Ca, Na, and K, with minor amounts of  $Fe^{2+}$ , Mg, and Sr. The site occupied by Ti in feldspars is not known. HARTMAN (1969) emphasizes the low likelihood of Ti occurring in tetrahedral sites in silicates. Ti has been pro-

visionally assigned (as  $Ti^{4+}$ ) to the X site in recalculation of these analyses. The Z site is filled by Si, Al, and  $Fe^{3+}$ ; these cations generate more than 14 positive charges out of 16 per formula unit. Thus, close agreement is found between expected and calculated occupancy of the Z site (Table 1).

The charge deficiencies apparent in the X site of most of the plagioclases in Table 1 probably reflect experimental error in the analysis of Si and Al. Analysis of PE-57 performed at the same time as and by the same methods used for the other samples indicated the presence of somewhat more  $SiO_2$  (1.1 wt.%) and less  $Al_2O_3$  (0.6 wt.%) than do the gravimetric analyses of these constituents in the same powder. The excess of silica and deficiency of alumina in the instrumental analysis of this sample resulted in the appearance of 2.3% deficiency in the X site together with departure from stoichiometry in a direction generally toward  $SiO_2$ . Decreasing Si by 1 to 2% of the amount reported, considered the precision of the method used (N. H. SUHR, priv. comm., 1975), would yield calculated X occupancies in these crystals much closer to 1.000. The gravimetric data indicate that PE-57 is essentially stoichiometric. Probably the other feldspars are as well.

The possibility of real, slight departures from stoichiometry toward the "end member"  $\square Si_4O_8$  must also be considered. Evidence of metastable non-stoichiometry involving Si-for-Al substitution and vacancies in the X site has been found in synthetic anorthite (BRUNO and FACCHINELLI, 1974) and Sr-feldspar (GRUNDY and ITO, 1974). Anomalies in lunar plagioclase compositions apparently may also be due in large part to such a scheme of substitution (BRUNO and FACCHINELLI, in press). These latter authors emphasize that Ca per 8 oxygens,  $Ca/(Na + Ca + K)$ , and  $Si/Al$  are independent representations of composition in all plagioclases that show non-stoichiometry toward  $\square Si_4O_8$ . They conclude that  $\gamma$  depends primarily on the Si/Al ratio; their sodium-free persilicic anorthites define a pattern of decreasing  $\gamma$  with increasing Si/Al ratio which is subparallel to the trend of decreasing  $\gamma$  with increasing Si/Al in the calcic portion of NISSEN's (1969) series of "low" plagioclases (c.f., BRUNO and FACCHINELLI, 1974, Fig. 4). Thus, the presence of  $\square Si_4O_8$  in the Pinacate labradorites would be expected to decrease  $\gamma$  in these crystals.

Accordingly, a plot was prepared to show the variation of  $\gamma$  in the Pinacate specimens (from values predicted by  $\gamma$  of the synthetic plagioclases of KROLL, 1971) with apparent departure from stoichiometry toward excess silica (estimated by the method of GUTMANN, 1972). The correlation appears absent. The  $\gamma$  angles of the Pinacate megacrysts all are less than those expected in disordered pure plagioclases of identical An content. They range from values just below the curve for synthetic materials to values displaced as much as 36% down from this curve toward that defined by natural plagioclases ascribed to low structural states (BAMBAUER et al., 1967). These departures presumably reflect chiefly Si-Al ordering and minor element content of the Pinacate

samples and correspond reasonably well with degrees of Si-Al ordering inferred in other ways (see below). No corroborative evidence is known to support the hypothesis, suggested by some of the chemical data, of presence of  $\square$   $\text{Si}_4\text{O}_8$  in solid solution in these feldspars; this hypothesis therefore is rejected at this time. However, if such departures from stoichiometry are metastable, as BRUNO and FACCHINELLI (1974) indicate, natural terrestrial examples might most reasonably be expected to occur in volcanic environments and/or as a result of rapid growth.

The relative uniformity of the minor element contents (Table 1) permits internal comparison of the X-ray diffraction data and interpretations in terms of two main variables: anorthite content and degree of Si-Al order. Despite the narrow compositional range (7% An), scatter of the data is so reduced by this uniformity that the megacrysts clearly display the expected increase in  $\gamma$  and decrease in  $\alpha$ ,  $\beta$ , and  $c$  with increasing anorthite content, as documented in higher-purity plagioclases.

Structural states of the megacrysts were estimated in several ways. Absolute measures of the extent of ordering probably cannot yet be obtained with confidence from the available data owing to various interpretive uncertainties, including the poorly known effects of minor substituents in the crystal structures. However, RIBBE (1972) has defined a linear relationship between the  $2\theta(131) - 2\theta(1\bar{3}1)$  structural indicator of SMITH and YODER (1956) and Al distribution among tetrahedral sites in plagioclase of any given composition. BAMBAUER et al. (1967) provided curves showing the variation in this indicator for synthetic and low potassium natural ordered plagioclases. Hence their determinative curves should offer a means of estimating precisely the *relative* structural states within this group of labradorites. Calculated values of  $2\theta(131) - 2\theta(1\bar{3}1)$ , designated  $\psi$ , are provided in Table 2 together with the percentages of displacement of the data points from the "plagioclase (high)" curve toward the "plagioclase (low)" curve of BAMBAUER et al. (1967, Fig. 5). The writers wish to emphasize that these percentage figures do not in any sense represent absolute measures of ordering. Indeed, certain difficulties become apparent when various empirical functions of Si-Al order are compared, as will be noted below. The percentage figures merely provide a convenient means of expressing *relative* degrees of Si-Al order within this suite of megacrysts, as judged from compositional and  $\psi$  data.

The percentages indicate variation among the megacrysts ranging from 27% to 53% of displacement from the disordered plagioclase curve toward the curve defined by low-K natural plagioclase of maximum inferred order. To some extent, these displacements are to be expected inasmuch as the disordered plagioclase curve of BAMBAUER et al. was defined using synthetic plagioclases devoid of Or; the addition of Or would reduce the 131 indicator (SMITH, 1974, Fig. 7-48). However, the relative displacements span a con-



Table 2. Unit Cell Dimensions of Labradorite Megacrysts\*)

Sample Number	a (Å)	b (Å)	c (Å)	$\alpha$	$\beta$	$\gamma$	V (Å <sup>3</sup> )	Number of lines used	Standard error unit wt. obs.	$\psi$ ( $2\theta_{131}$ — $2\theta_{\bar{1}\bar{3}1}$ )	% Displacement toward "low" curve**)
PS-X1	8.1713 ±0.0007	12.8766 ±0.0012	7.1011 ±0.0005	93° 26.7' ±0.4	116° 05.3' ±0.4	90° 27.7' ±0.4	669.34 ±0.06	47	0.009	2.050	42
PS-X4	8.1762 ±0.0006	12.8733 ±0.0009	7.1046 ±0.0005	93° 27.0' ±0.5	116° 06.2' ±0.3	90° 26.0' ±0.4	669.82 ±0.06	46	0.007	2.038	45
PE-X2	8.1757 ±0.0008	12.8748 ±0.0010	7.1040 ±0.0006	93° 26.3' ±0.5	116° 02.2' ±0.5	90° 29.8' ±0.5	670.15 ±0.07	33	0.007	2.062	28
PE-57	8.1751 ±0.0010	12.8802 ±0.0015	7.1015 ±0.0009	93° 27.7' ±0.6	116° 06.2' ±0.5	90° 26.6' ±0.6	669.78 ±0.09	40	0.012	2.049	36
PE-X12	8.1740 ±0.0011	12.8740 ±0.0018	7.1048 ±0.0010	93° 27.5' ±0.9	116° 06.7' ±0.8	90° 22.9' ±0.9	669.66 ±0.12	33	0.013	2.017	53
PE-X13	8.1712 ±0.0009	12.8739 ±0.0014	7.1039 ±0.0008	93° 26.3' ±0.8	116° 06.0' ±0.6	90° 24.9' ±0.7	669.41 ±0.09	35	0.011	2.024	50
PE-X14	8.1762 ±0.0008	12.8808 ±0.0010	7.1049 ±0.0006	93° 28.4' ±0.5	116° 06.4' ±0.4	90° 25.6' ±0.5	670.19 ±0.07	40	0.009	2.047	27
PE-X15	8.1750 ±0.0010	12.8730 ±0.0011	7.1049 ±0.0007	93° 27.8' ±0.6	116° 06.8' ±0.6	90° 24.3' ±0.6	669.68 ±0.09	32	0.010	2.032	34
PE-X16	8.1739 ±0.0008	12.8719 ±0.0016	7.1059 ±0.0007	93° 29.1' ±0.7	116° 08.4' ±0.5	90° 19.5' ±0.7	669.48 ±0.10	25	0.009	2.005	33
PE-X17	8.1759 ±0.0011	12.8794 ±0.0017	7.1054 ±0.0009	93° 27.8' ±0.8	116° 07.8' ±0.7	90° 23.5' ±0.7	670.05 ±0.11	35	0.013	2.026	30
PE-X18	8.1751 ±0.0013	12.8712 ±0.0027	7.1030 ±0.0011	93° 24.2' ±1.1	116° 04.4' ±0.9	90° 29.9' ±1.1	669.65 ±0.16	25	0.014	2.046	45
PE-X19	8.1760 ±0.0014	12.8715 ±0.0025	7.1043 ±0.0011	93° 26.4' ±1.2	116° 06.1' ±0.8	90° 26.6' ±1.2	669.69 ±0.15	30	0.015	2.040	40

\*) Refinements based on a 7 Å repeat of the c cell edge. \*\*) See text for explanation.



siderable range. Although precision of the diffraction data permits errors of up to 7% in each figure for relative displacement, the extent to which the two megacrysts from each volcanic source unit show similar and distinctive degrees of displacement from the high plagioclase curve suggests that the range of displacements is largely real.

Other methods of estimating structural state include the peak separations  $2\theta(131) + 2\theta(220) - 4\theta(1\bar{3}1)$  and  $2\theta(1\bar{1}1) - 2\theta(\bar{2}01)$ , Cu  $K_\alpha$  radiation; these indicators are  $I$  and  $B$ , respectively, of SMITH and GAY (1958). The  $B$  parameter does not reveal appreciable variation in structural state among the crystals, all but one of which plot in a narrow band intermediate between the curves for plutonic and heated natural plagioclases (SMITH and GAY, 1958, Fig. 2); one sample (PE-X2) plots close to the curve for synthetic feldspars. The  $I$  parameter more clearly reveals variation in structural state in the megacrysts, which plot along the curve for disordered feldspars and range down one-third of the way toward that for plutonic samples (SMITH and GAY, 1958, Fig. 1). As would be expected, the *relative* degrees of ordering inferred from the  $I$  indicator for these twelve crystals agree well with those inferred from  $\psi$ . However, it is worthy of note that these three parameters of Si-Al order evidently are *not* mutually consistent in displaying the absolute positions of the points relative to the curves for synthetic and plutonic materials. Absolute estimates of the degree of Si-Al ordering clearly cannot now be derived accurately from all three of these parameters, if indeed from any one of them, irrespective of the complications posed by minor element contents.

The structural state of plagioclase also has a marked effect on the orientation of the optical indicatrix. The compositions and structural states of the six megacrysts from pyroclastic rocks were estimated in thin sections of albite-twinned crystals by the four-axis Turner method, utilizing the determinative curves of SLEMMONS (1962). The indicated compositions agree well with the analytical values, although the former tend to be less rich in anorthite by 1 to 2%. The data points plot in positions ranging downward from the curve for volcanic plagioclase half way to that for plutonic plagioclase. Positions of the feldspars relative to one another between these curves are identical to their relative positions judged from the 131 indicator.

Unlike other diagrams discussed thus far, a plot of  $\beta^*$  versus  $\gamma^*$  (SMITH, 1974, Fig. 7-44) holds promise in separating the two independent variables composition and structural state. Unfortunately, intermediate plagioclases cannot be interpreted unambiguously on such a diagram because variation curves for ordered and disordered series cross at a high angle in the region of interest. If the isopleths are assumed to be straight lines between the two series and the effect of Or content is ignored, the megacrysts appear to consist of  $An_{66}$  to  $An_{69}$  having intermediate Si-Al order, or  $An_{45}$  to  $An_{50}$  having essentially no Si-Al order. However, if the isopleths are not straight

lines, the pertinent isopleths could pass through the cluster of data points, and the data would reflect various degrees of Si-Al order, as inferred above.

In summary, data derived from both X-ray diffraction and optical studies suggest similar and significant variations in structural state among the volcanic megacrysts. The variations evidently cannot be ascribed to the effects of minor elements, although the 131 structural indicator in samples PE-X16 and PE-X17 may be relatively depressed by the higher potassium contents of these crystals. However, the variations in structural state apparently do reflect the thermal histories of these specimens with considerable sensitivity.

The four samples from lava flows (PE-X14 through 17) exhibit  $\psi$  displacements toward the curve for plutonic plagioclase ranging from 27% to 34%. The samples were collected approximately 2 m below the tops of the flows. Samples PE-X2 and PE-57 (28% and 36% displacement, respectively) were ejected in cinder eruptions and must have cooled rapidly in the air but were then buried beneath several meters of nonwelded scoriaceous bombs and lapilli, the latter sample more deeply (8 m) than the former (2 m). Samples PS-X1 and PS-X4 (42% and 45% displacement) also were collected from a cinder cone but were more deeply buried (probably ca. 10–15 m); their  $\psi$  displacements are not significantly different from those of samples PE-X18 and PE-X19 (45% and 40%), which were collected from the marginal parts of a shallow intrusion within cinders about 8–10 m below the surface of a cinder cone. Samples PE-X12 and PE-X13 show the greatest departures toward the curve for plutonic plagioclase (53% and 50%). They were collected from loose cinders buried 32 m stratigraphically beneath the surface of the same cone.

Although the cooling histories of these crystals cannot be inferred in detail from field observations, it would appear that the relative displacements of their 131 indicators toward the curve for plutonic plagioclase probably do reflect these histories in a general way. If indeed the displacements do provide accurate measures of the relative degrees of Si-Al order in this group of compositionally uniform labradorites, it is evident solely from the range of the displacements that plagioclase crystals even within mafic pyroclastic units may become significantly ordered owing to prolonged cooling beneath a warm, insulating blanket of basaltic cinders.

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