

Zeitschrift: Schweizerische mineralogische und petrographische Mitteilungen = Bulletin suisse de minéralogie et pétrographie
Band: 47 (1967)
Heft: 1: Feldspäte

Artikel: Diagrams for the determination of plagioclases using x-ray powder methods. Part III of laboratory investigations on plagioclases
Autor: Bambauer, H.U. / Corlett, M. / Eberhard, E.
DOI: <https://doi.org/10.5169/seals-36956>

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Diagrams for the Determination of Plagioclases using X-ray Powder Methods

(Part III of Laboratory Investigations on Plagioclases)

By *H. U. Bambauer* (Münster)*, *M. Corlett*, *E. Eberhard* and
K. Viswanathan (Zürich)**)

With 7 figures in the text

Zusammenfassung

Neue Diagramme zur Plagioklasbestimmung mit Hilfe der Liniendifferenzen $\Delta(\theta)_1 = 2\theta_{131} - 2\theta_{1\bar{3}1}$ und $\Delta(\theta)_2 = 2\theta_{241} - 2\theta_{2\bar{4}1}$ (Cu K α_1) im Pulverdiagramm werden mitgeteilt. Zugrunde liegen Messungen an ca. 300 synthetischen und mit der Mikrosonde analysierten natürlichen Proben. Die erhaltenen Bestimmungskurven zeigen charakteristische Diskontinuitäten. Schon Or-Gehalte > 1—2 Mol-% in der Plagioklasmatrix können einen merklichen Einfluss auf die Liniendifferenzen, insbesondere $\Delta(\theta)_1$, ausüben. Die gezeigten Diagramme erlauben relative Aussagen über den strukturellen Zustand verschiedener Plagioklasproben.

Abstract

New diagrams are presented for the determination of plagioclases with the help of the line-differences $\Delta(\theta)_1 = 2\theta_{131} - 2\theta_{1\bar{3}1}$ and $\Delta(\theta)_2 = 2\theta_{241} - 2\theta_{2\bar{4}1}$ (Cu K α_1) found in powder photographs. This work is based on about 300 natural and synthetic specimens; natural specimens were analysed with the electron microprobe analyser. The determinative diagrams obtained show characteristic discontinuities. Or-contents as low as 1—2 mol % can exercise a noticeable influence on the line-differences, particularly $\Delta(\theta)_1$. These diagrams permit relative statements on the structural state of plagioclases.

*) Institut für Mineralogie und Petrologie der Universität Münster, D-44 Münster, Hüfferstrasse 1.

**) Institut für Kristallographie und Petrographie der Eidg. Techn. Hochschule, CH-8006 Zürich, Sonneggstrasse 5.

I. INTRODUCTION

The position and intensity of the X-ray diffraction lines of plagioclase show characteristic variations depending upon anorthite content and structural state. From the diagrams given by GOODYEAR and DUFFIN (1955), SMITH (1956), SMITH and YODER (1956), SLEMMONS (1962), it is clear that the powder method is (with reservations) a suitable means for the determination of plagioclases. Though the data presented by the different authors are more or less similar, they do show noticeable and sometimes even considerable discrepancies. Therefore we considered it necessary to construct new and more accurate diagrams for the determination of plagioclases. Preliminary results have been published (BAMBAUER et al., 1965).

By "structural state" we mean the type of distribution of Al and Si in the $(\text{Al}, \text{Si})\text{O}_4$ -tetrahedra of the feldspar structure. Furthermore "type of distribution" shall on the basis of present knowledge only be taken to mean that Al and Si tend toward an ordered (disordered) distribution with falling (rising) temperature. In this usage "type of distribution" means only "degree of order"; atomic site distributions are not given and cannot be given, since refined structure determinations are not at present being carried out or planned. This lack of crystal-structure data is not important for the following investigation and discussion.

It should be kept in mind that in addition to the degree of order present, this degree of order may be "stable" or "unstable". The principle of this distinction has been discussed by LAVES (1960a, b) for the relatively simple alkali feldspar system. Indeed, the "stable" phases are perhaps only approached in nature and are available for research only as more or less "unstable" phases. To the names of plagioclases with stable ordering of Al and Si are added the suffixes high, intermediate and low (with reference to temperature). They correspond to the stable states of least, intermediate and best ordering which are possible for a given composition. Independently of these stability considerations, BAMBAUER (1966) proposed the collective names D-plagioclase (D = disordered), O-plagioclase (O = ordered) and O/D-plagioclase. These plagioclases would have properties respectively indicative of disordered, ordered and intermediate Al, Si distributions.

Few details of the different states which may be attained by plagioclases are known. Likewise little has been done to distinguish between the stable and unstable states. However, a general correlation between the lattice constants and d-spacings on the one hand and Al, Si states

of order on the other is now known. Of the lattice constants γ^* shows the strongest variation, not only with An-content but also with degree of order (BROWN, 1960). In powder patterns the line-differences $\Delta(\theta)_1 = 2\theta_{131} - 2\theta_{\bar{1}\bar{3}\bar{1}}$ and $\Delta(\theta)_2 = 2\theta_{\bar{2}41} - 2\theta_{2\bar{4}\bar{1}}$ [range of 2θ (Cu K α) = $21^\circ - 37^\circ$] show the strongest relative movement, when only lines with sufficient intensities throughout the plagioclase series are considered. [Since $\Delta(\theta)_2$ -values pass through zero, the algebraic difference must be used (cf. Fig. 1).] In addition, the influence of other cations (e. g. K-content) must apparently be taken into account. Therefore it may be assumed that the state of order/disorder (chemical composition remaining constant) will be indicated with greatest sensitivity by the values of the above mentioned line-differences in the powder photographs. The members of the following chemical series should be regarded as extreme states under equilibrium conditions:

a) Monalbite (as analbite at room temperatures) — plagioclase (high) — anorthite (body-centred). This series comprises the feldspars with stable states of highest possible Al, Si disorder. They are summarised here under the simple term “plagioclase (high)”. The characteristic feature of their powder diagrams is that for a given chemical composition they possess a maximum value for $\Delta(\theta)_1$ and a minimum algebraic value of $\Delta(\theta)_2$.

b) Albite (low) — anorthite (primitive). This series, “plagioclase (low)”, is interrupted by the peristerite gap. It is assumed that this group possesses the highest Al, Si-ordering which can be attained under natural conditions. However, it has often been questioned (most recently by LAVES et al., 1965), whether the chemically intermediate members of this group are indeed stable. This group possesses a minimum $\Delta(\theta)_1$ -value and a maximum algebraic $\Delta(\theta)_2$ -value for a given chemical composition.

Recent summaries of plagioclase data are given by DEER et al. (1963) and BAMBAUER (1966).

II. EXPERIMENTAL

a) *Material investigated*

Attempts have been made to synthesise the series “plagioclase (high)”. Plagioclase glasses with chemical compositions at 10 mol-% An intervals were devitrified by tempering just below the respective solidus-temperatures. As is well known, devitrification in the case of An-rich compositions

takes from a few minutes to hours. In the case of Ab-rich plagioclases, a few weeks are required for complete devitrification. The crystals formed are extremely fine and spherulitic. There is still some doubt about their complete homogeneity. In the case of pure soda-feldspar, Na-tungstate was used as a mineralisator (H. J. SCHEEL, personal communication), and also made possible the formation of larger crystals. Further tempering after complete devitrification did not produce any noticeable change in the powder patterns; it was therefore assumed that complete equilibrium was at least closely approached.

There are of course difficulties in the preparation of "plagioclase (low)" because of the extremely low diffusion rates of Al and Si (EBERHARD, 1967). Therefore 300 natural plagioclases were selected which, because of their occurrences (in plutonic rocks, metamorphic rocks, pegmatites and alpine fissures), could be expected to approach closely the structural state "plagioclase (low)". Reference may be made to CORLETT and EBERHARD (1967) for further details about the individual specimens.

b) Chemical analysis

Chemical analyses were carried out on the above mentioned natural plagioclases. Flame photometric determination of Na and K, and titrimetric determination of Ca, for 30 specimens were obtained through the courtesy of Mr. P. Thommen. However only the bulk analysis is obtained by these methods. If the plagioclase is, for example, antiperthitic, the true An-content is then underestimated. Therefore all the 300 specimens were quantitatively analysed with an ARL-electron microprobe analyser for Ca and K (some specimens with high An-content were also analysed for Na); only those individual analyses were used for calculating the plagioclase composition which were free of the influence of exsolved impurities. The reproducibility of the Ca-values in the range An_{15-100} is within $\pm 1\%$ (variation range of the average values); in the case of 0.8% K, it lies within $\pm 10\%$. Further the specimens were examined for inclusions of foreign minerals. For further details regarding accuracy, method . . . etc., the reader is referred to CORLETT and RIBBE (1967).

c) X-ray methods

Powder photographs were taken of all the specimens in an AEG-Guinier camera with $Cu K\alpha_1$ radiation (silicon with $a_0 = 5.4305 \text{ \AA}$ was used as a standard). The line-differences $\Delta(\theta)_1$ and $\Delta(\theta)_2$ in every film were measured independently by at least three of the authors (accuracy

Fig. 1. X-ray diffractometer patterns of analbite and some members of the series "plagioclase (low)":

$$2\theta (\text{Cu K}\alpha) = 20^\circ - 40^\circ.$$

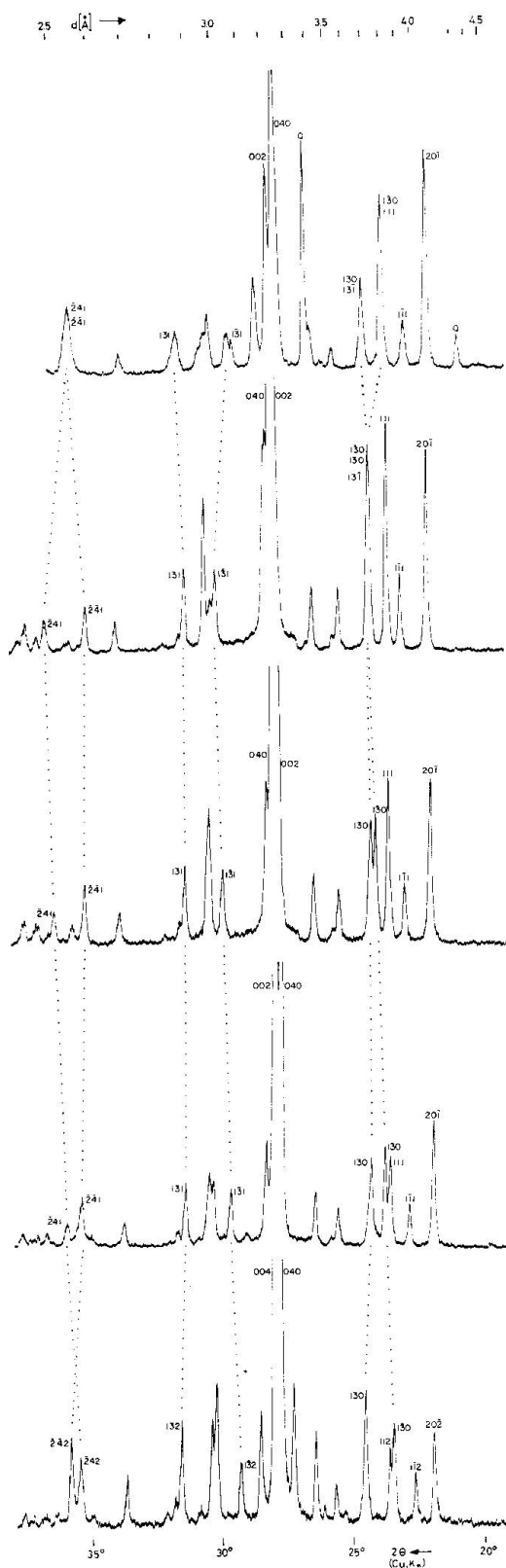
a) Synthetic analbite.

b) Albite (low).

c) Oligoclase (low); ca. An_{17} .

d) Labradorite (low); ca. An_{45} .

e) Anorthite; ca. An_{95} .



of measurement 2×10^{-3} mm). The position of these line-pairs can be seen in the diffractometer patterns in Fig. 1. For specimens which showed sharp lines, the discrepancies in the measurements made by the different authors were usually not greater than $\pm 1 \times 10^{-2}$ mm ($\approx \frac{1}{2}'$ of 4θ).

III. RESULTS

1. If we compare roughly the $\Delta(\theta)_1$ values with the data found in the literature [$\Delta(\theta)_2$ values have not to our knowledge been published] we may safely assume that most of the specimens investigated here have attained or closely approached the state of "plagioclase (low)".

2. On the basis of the flame photometric determinations made largely on An-poor and chemically intermediate plagioclases, it is observed that the bulk K-content of the morphological unit was usually within 0—4 mol-% Or, with a maximum of 8 mol-% Or.

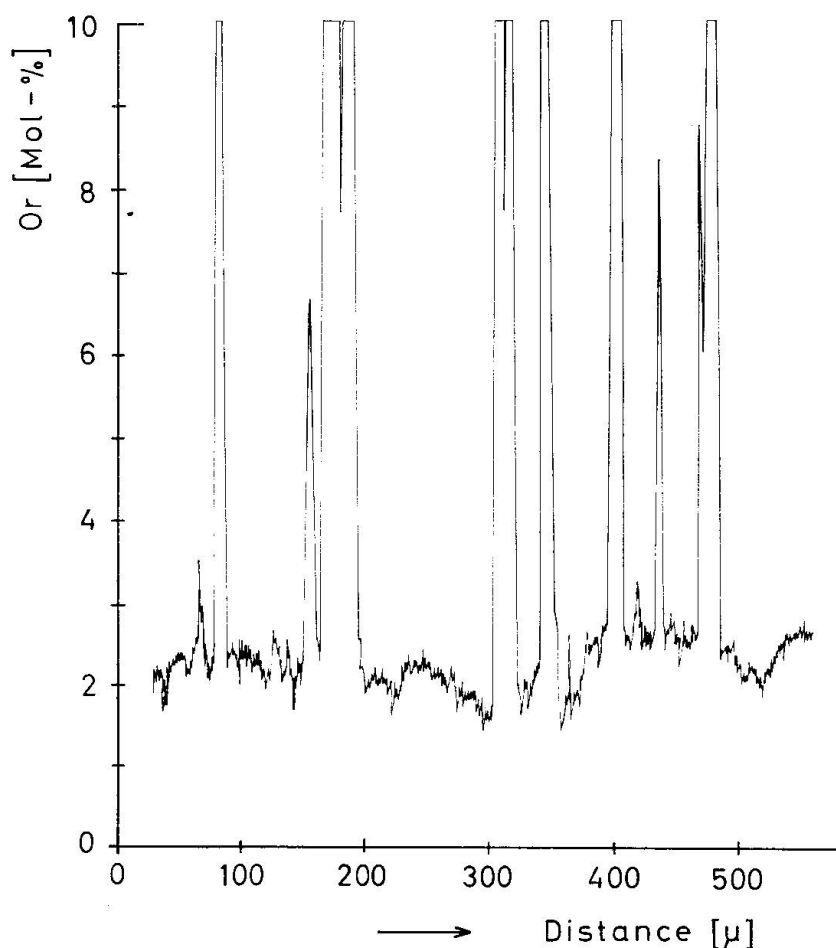


Fig. 2. Irregular K-distribution in a plagioclase.

3. Electron microprobe analysis shows that many specimens are inhomogeneous with reference to their An-content. This could often be confirmed optically (slight zonal structure). Such specimens usually show more or less diffuse and broad lines in powder pictures; in this respect, the line-pair $\overline{241}/\overline{241}$ is much more sensitive than the pair $131/\overline{131}$. In a few cases, chemically homogeneous substances were found to show broad lines and vice versa; in such cases the Al, Si-ordering seems to play a role.

4. Further it was found with the electron microprobe analyser that almost all the specimens are micro- and/or cryptoantiperthitic. In this respect two extreme types of inhomogeneity may be distinguished:

a) The K-content varies irregularly from place to place within a specimen, with or without bigger uniform lamellae (Fig. 2). This may correspond to an irregular distribution of numerous, small, closely-spaced bodies of K-feldspar.

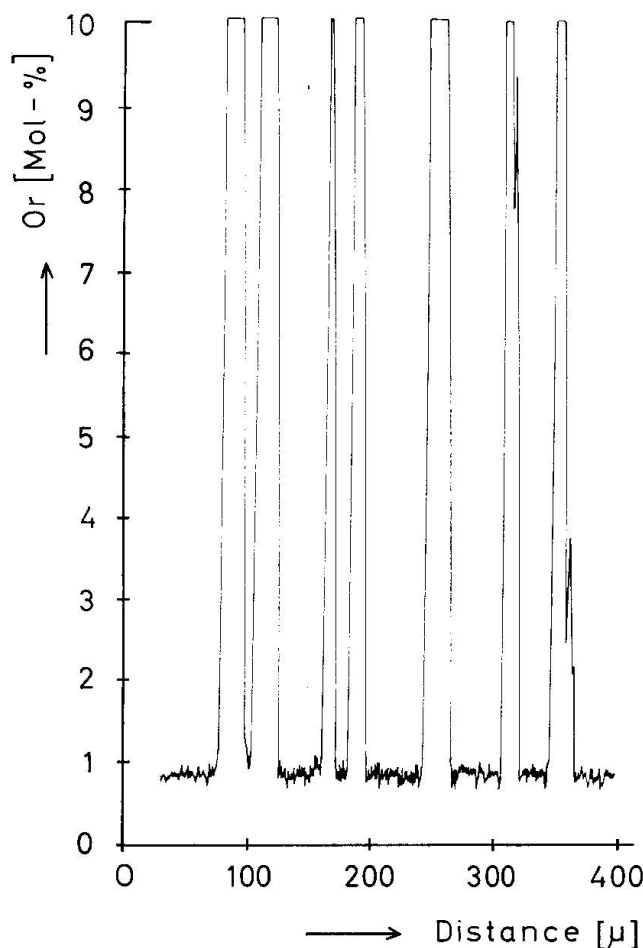


Fig. 3. Regular K-distribution in a plagioclase.

b) There are bigger areas of uniform, usually low, K-content alternating with small patches of considerably higher K-content (Fig. 3). This corresponds to a uniform plagioclase matrix, in which lie independent bodies of K-feldspar with well-defined boundaries. However, even with the resolving capacity of the electron microprobe analyser (minimum

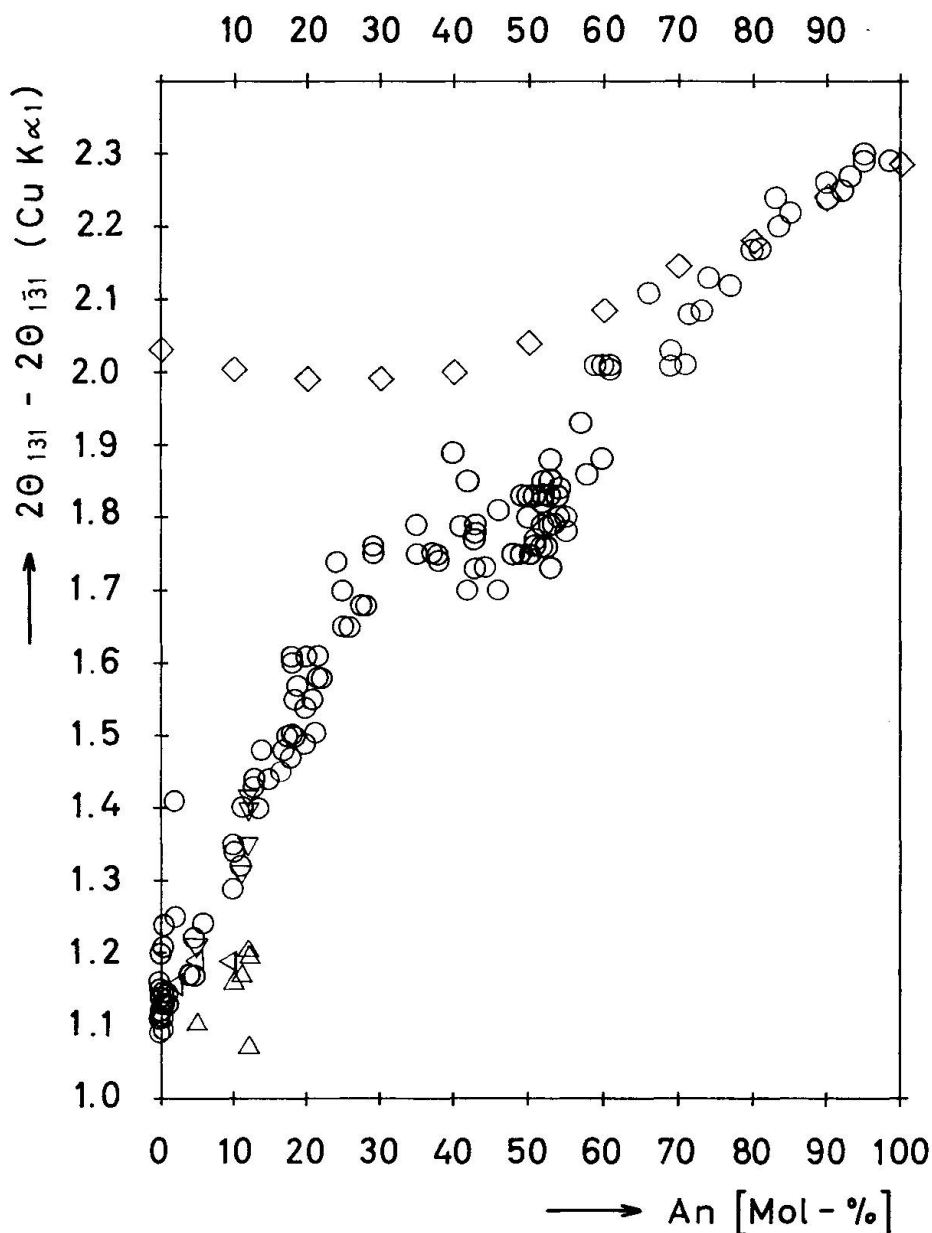


Fig. 4. $\Delta(\theta)_1 = 2\theta_{131} - 2\theta_{\bar{1}31}$ (Cu $K\alpha_1$ radiation) vs An content for ca. 190 plagioclases. \diamond synthetic specimens; \circ natural specimens (only one plagioclase phase); peristerites: \triangle Ab-rich component, ∇ An-rich component, \triangleleft proven peristerites (see text), only one component found.

width of the electron beam $1\ \mu$), it could not be ascertained whether the potassium lies in the matrix *isomorphously*, or whether it corresponds to a very regular fluctuation in the sense of a "quasihomogeneous" (LAVES, 1952) antiperthitic unmixing.

5. The potassium content of the homogeneous plagioclase matrix lying between the antiperthitic K-feldspars is usually $< 0.5\text{--}0.8$ mol-% Or. Exceptions are the plagioclases with intermediate An-contents, which can contain up to 4% Or. These plagioclases lie in the range An_{20} to An_{55-60} and most of them show schiller (schillering "labradorites").

All the Or-contents mentioned in the text, unless otherwise stated, correspond to the content of the matrix. For further details, see CORLETT and RIBBE (1967).

6. On comparing the $\Delta(\theta)$ values with the degree of homogeneity of the antiperthites (corresponding to point 4) the following tendencies are recognised: the nearer the Si, Al-ordering of a specimen to that of "plagioclase (low)", the more similar is the antiperthite formation to Fig. 3, in the sense that it contains independent patches of K-feldspar and a homogeneous plagioclase matrix. Moreover, the Or-content of the matrix seems to be dependent on the An-content (and naturally also on the K-content) of the system. See CORLETT and RIBBE (1967).

The chemical and the X-ray data of all the investigated specimens have been summarised by CORLETT and EBERHARD (1967) in tabular form.

At a second stage of selection, the following natural specimens were excluded: a) those for which there was the danger of coincidence of lines due to impurities, b) those showing noticeable chemical inhomogeneity (e. g. zoning) and c) those which showed broad line-pairs $131/1\bar{3}1$ and $241/2\bar{4}1$. After this selection, only 180 specimens remained. Their $\Delta(\theta)_1$ values are plotted against their An-content as in Fig. 4. In this figure, there are also plots of synthetic plagioclases. Those peristerites which could be unequivocally identified are specially marked. Fig. 4 appears roughly similar to many of the diagrams in the literature. However the diagram in this form appeared to us unsatisfactory as the basis of a determinative diagram for natural plagioclases.

IV. DETERMINATIVE DIAGRAMS

In a third stage of selection all of the natural specimens used for the construction of Fig. 4 which did not meet the following criteria were discarded:

- a) only one plagioclase phase present;
- b) homogeneous An-content, within the limits of analytical error,
- c) K-content homogeneous throughout the plagioclase matrix, and less than 0.5—0.8 mol-% Or in that matrix;

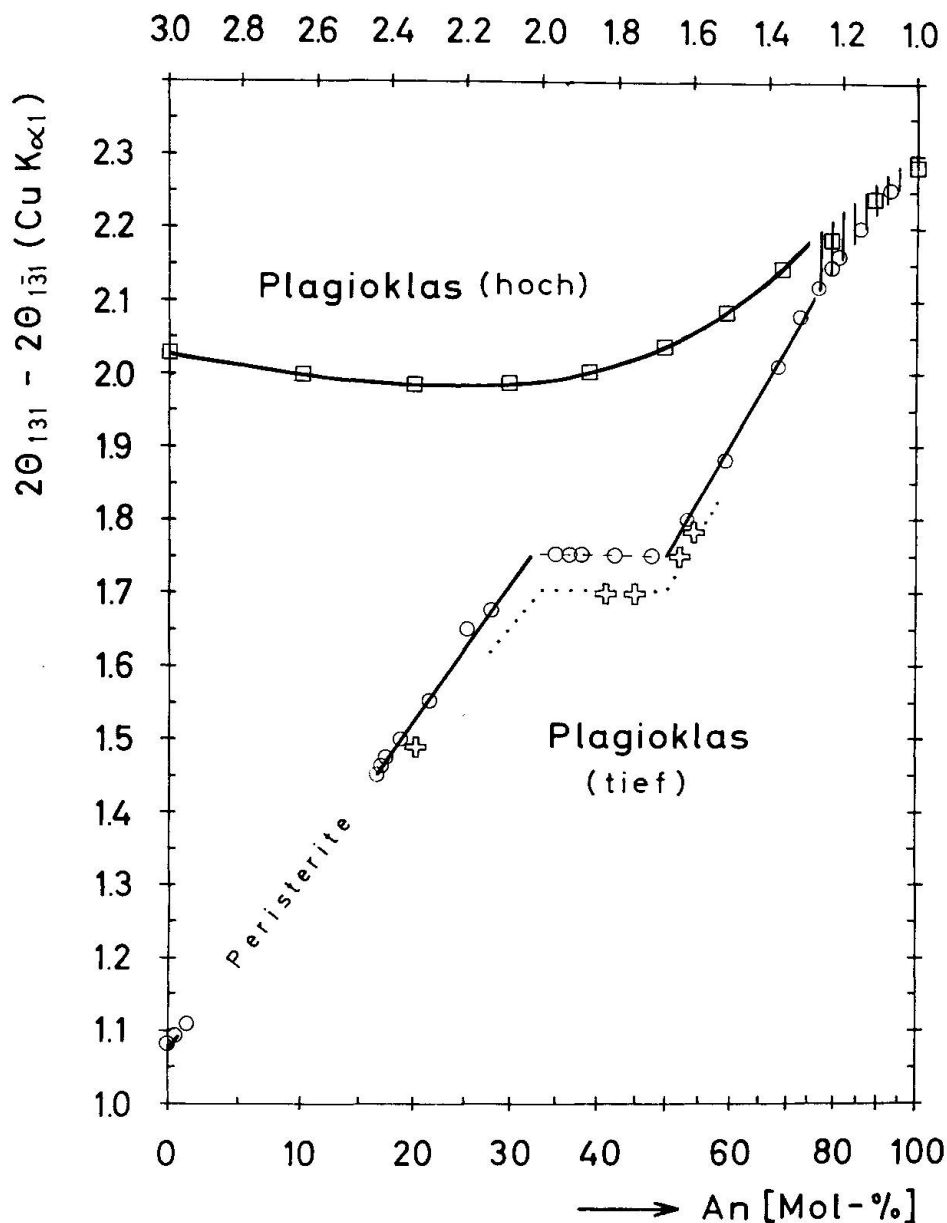


Fig. 5. Boundary curves for the variation of $\Delta(\theta)_1 = 2\theta_{131} - 2\theta_{1\bar{3}1}$ (Cu $K\alpha_1$ radiation) and Si/Al ratio (An content also shown) for plagioclases with $Or_{<0.5-0.8}$. The dotted boundary curve is for "plagioclase (low)" with ca. An_{40-60} and Or_4 . \circ , "plagioclase (low)", $Or_{<0.5-0.8}$; \square , synthetic "plagioclase (high)"; \oplus , "plagioclase (low)", $Or_{ca. 4}$.

- d) minimum value of $\Delta(\theta)_1$ and maximum value of $\Delta(\theta)_2$ for any given An content.

Furthermore, chemically intermediate plagioclases with ca. 3—4 mol-% Or (those referred to as exceptions in section III. 5) were not discarded if they fulfilled condition d) above. In the light of present knowledge it

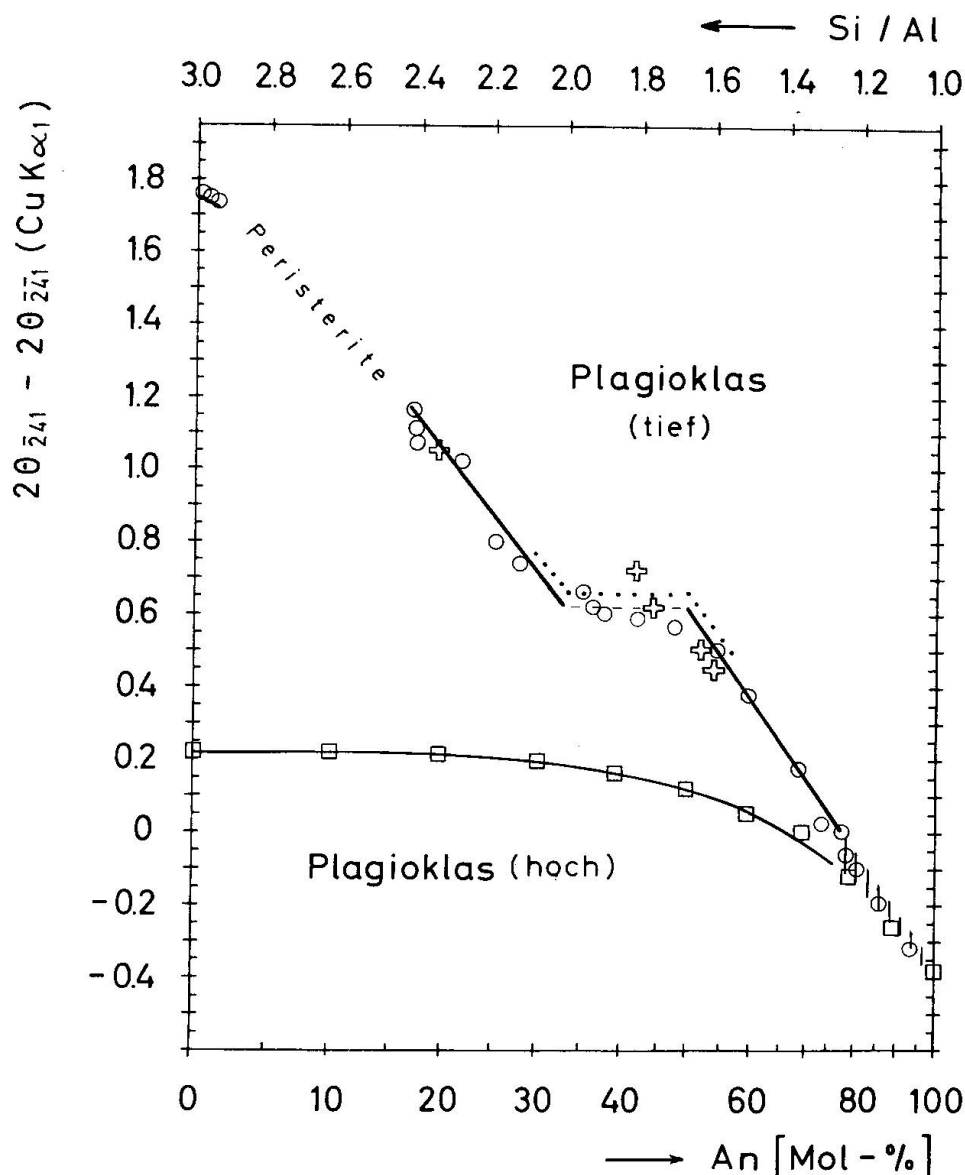


Fig. 6. Boundary curves for the variation of $\Delta(\theta)_2 = 2\theta_{241} - 2\theta_{2\bar{4}1}$ (Cu $K\alpha_1$ radiation) and Si/Al ratio (An content also shown) for plagioclases with $Or < 0.5-0.8$. The dotted boundary curve is for "plagioclase (low)" with ca. An_{40-60} and Or_4 . \circ , "plagioclase (low)", $Or < 0.5-0.8$; \square , synthetic "plagioclase (high)"; \oplus , "plagioclase (low)", $Or_{ca. 4}$.

may be assumed that the 32 specimens remaining after this rigorous selection approach the series "plagioclase (low)" in the closest possible way.

Since BROWN (1960) has shown that γ^* bears a linear relation to the Si/Al ratio (rather than to the An content), this ratio was taken as the basis for both diagrams (Figs. 5 and 6). The lines shown are boundary curves that are drawn through the currently-known most extreme values observed in the field between "plagioclase (high)", and "plagioclase (low)". The $\Delta(\theta)_1$ diagram (Fig. 5) is better suited to routine work, since the lines $131/1\bar{3}1$ are stronger than $241/2\bar{4}1$ and in practice never coincide with other plagioclase lines (see BAMBAUER et al., 1967). One can recognize the relatively greater variation of $\Delta(\theta)_2$ in the shortened ordinate scale of Fig. 6. Considering for the moment only plagioclases with less than 0.5–0.8 mol-% Or in the matrix, the diagrams have the following characteristics in common:

1. The boundary curve for "plagioclase (high)" is continuous to at least Si/Al = 1.25 (An₇₆). At about An_{76–80} there may be a discontinuity.
2. The boundary curve for "plagioclase (low)" shows the following sequence:

- a) Si/Al = 3.0–2.97 (An_{0–1.3}): linear increase in $\Delta(\theta)_1$ and decrease in $\Delta(\theta)_2$. This represents the stability region of albite (low)¹.
- b) Si/Al = 2.97–2.45 (An_{1.3–16}): the peristerite gap. It is commonly believed that no homogeneous members of the "plagioclase (low)" series occur in this region. This has in general been confirmed by us (see below).
- c) Si/Al = 2.45–2.02 (An_{16–33}): linear increase in $\Delta(\theta)_1$ and decrease in $\Delta(\theta)_2$.
- d) Si/Al = 2.02–1.67 (An_{33–50}): the continuation is not certain; it is probably horizontal (see below).
- e) Si/Al = 1.67 to at least 1.25 (An_{50–76}): linear increase in $\Delta(\theta)_1$ and decrease in $\Delta(\theta)_2$.
- f) Si/Al = 1.25–1.0 (An_{76–100}): there is a discontinuity around An_{70–80}. An unequivocal distinction of "plagioclase (high)" and "plagioclase (low)" is perhaps not possible in this region using $\Delta(\theta)$ values. In addition to the Si, Al-order/disorder processes the so-called "distributive transformation" (GOLDSMITH and LAVES, 1955; BROWN et al., 1963) also occurs here.

¹) The boundary value An_{1.3} is based on a microprobe analysis of the Ab-rich phase of a peristerite.

In both diagrams an attempt was made to draw boundary curves for chemically intermediate "plagioclase (low)" containing 4 mol-% Or. The position of these line segments was derived from measured and calculated data (see BAMBAUER et al. 1967). These curves may perhaps be capable of improvement in details (the agreement between measured and calculated values is good for $\Delta(\theta)_1$, but poor for $\Delta(\theta)_2$), but it is nevertheless apparent that not only the An-content but also the isomorphously incorporated Or-content of a plagioclase must be known in order to analyse its structural state on the basis of the $\Delta(\theta)$ values. For this purpose $\Delta(\theta)_1$ is more sensitive than $\Delta(\theta)_2$.

The slopes of the linear sections of the boundary curves for "plagioclase (low)" vary; they are not parallel to each other. Further, no segment projects on to either the low albite or the anorthite values. It was not possible to establish whether the segment An₁₆₋₃₃ extends past An₃₃ into the "plagioclase field"; that is, whether for a certain distance it runs approximately parallel to the section An₅₀₋₇₆. The end point An₅₀ and the possible end point An₃₃ may be joined by a horizontal line in the $\Delta(\theta)_1$ diagram; in the $\Delta(\theta)_2$ diagram and in the lattice constants (cf. BAMBAUER et al., 1967) it is difficult to define the "plagioclase (low)" boundary in this region. In any case a remarkable, possibly ambiguous, scatter of the values in the region Si/Al=2.0—1.67 is to be observed, which at present cannot be explained. It would be interesting to examine a chemical series of "plagioclases (low)" with a common growth environment, the basic members of which extend past An₃₃.

It has already been noted that $\Delta(\theta)_1$ is more sensitive to the Or-content of the matrix than $\Delta(\theta)_2$. It should therefore be possible to make some estimate of the Or-content by a comparison of these two values. In Fig. 7 $\Delta(\theta)_1$ is plotted against $\Delta(\theta)_2$ for the series "plagioclase (low)" with Or_{<0.5-0.8}. A knowledge of the chemical composition is not required for the construction of this diagram. Like Figs. 5 and 6, it shows linear sections and discontinuities. It is astonishing that the majority of points for structurally intermediate plagioclases (from Figs. 5 and 6) with low Or-contents also fall on these straight lines. In any case the calibration with respect to An-content is different for different structural states; for example, analbite lies on the curve, not at the point for low albite, but rather more toward the middle. The following types of specimens deviate from this curve:

1. Specimens with a higher Or content; the values lie to the left of the curve for Or_{<0.5-0.8}. The curve for Or \approx 4 mol-% is shown by the dotted line. It is thus possible to estimate the Or content of the matrix.

2. Some O/D-plagioclases from Fig. 5; their points lie clearly to the right of the curve. It may be assumed that this nonconformity has a special structural reason. If a stable series "plagioclase (low)" really exists from An_{16-100} , it seems possible that points representing stable Al, Si distributions lie on the curve, while unstable configurations lie beside it. An adequate explanation has not yet been found.

It is usually difficult or indeed impossible to examine both the compo-

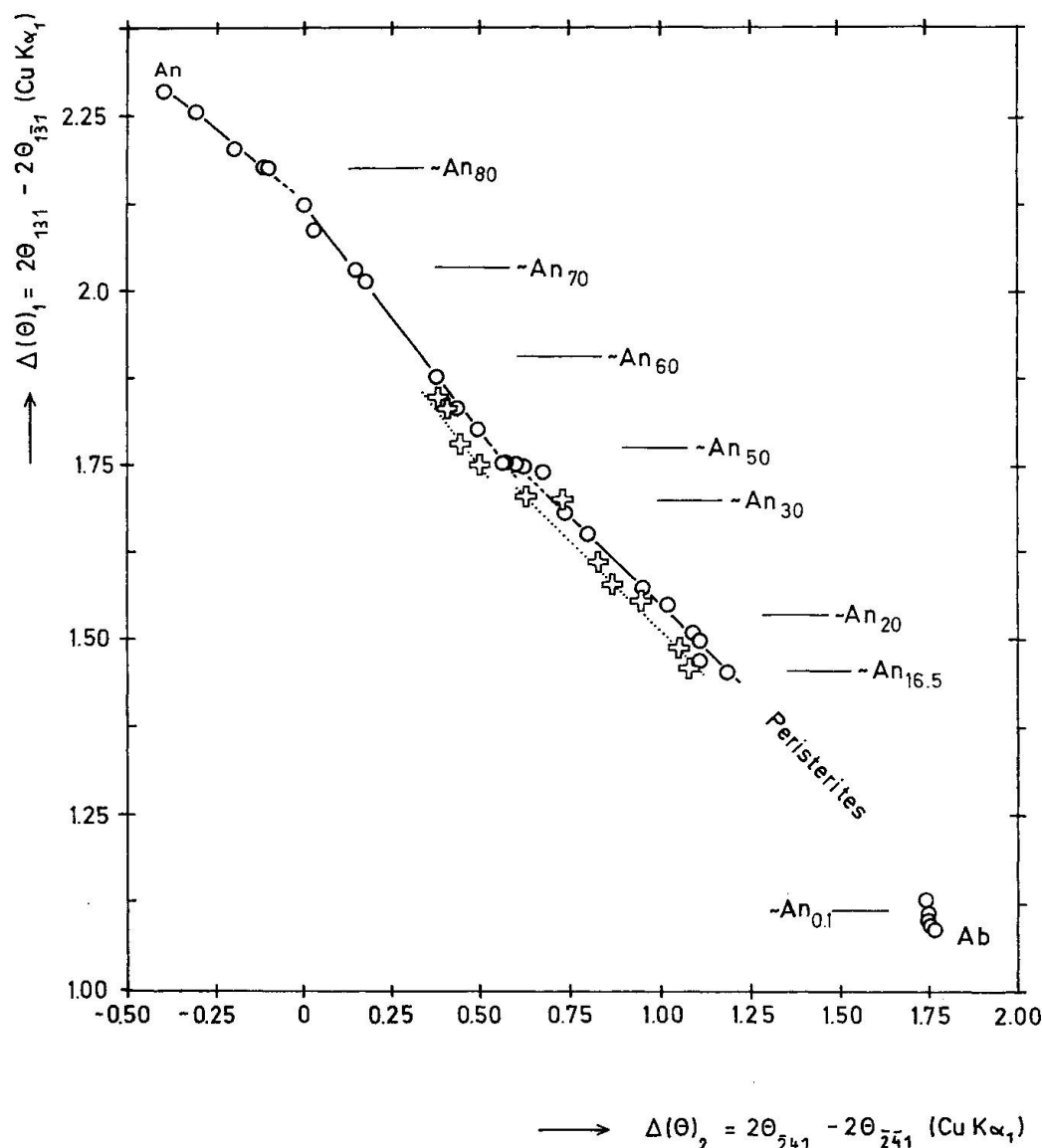


Fig. 7. Relation between $\Delta(\theta)_1$ and $\Delta(\theta)_2$ (cf. Figs. 5 and 6) for 0- and 0/D-plagioclases. The An-calibration is given for 0-plagioclases. \circ : 0-plagioclase with $Or_{<0.5-0.8}$; \oplus : 0- and 0/D-plagioclases with Or_{2-5} . The boundary curve for $Or_{\sim 4}$ is shown by the dotted line.

nents of peristerites sufficiently by means of powder methods. Often even the identification of a peristerite is difficult. For a well-resolvable peristerite one finds two diffraction patterns, representing albite (low) and an oligoclase (low). $131/1\bar{3}1$ values of peristerites are shown in Fig. 4 by triangles whose vertices face each other (at the respective bulk An-content of the peristerite). In most cases, however, the lines of one or both components are broad and diffuse. In proven peristerites²⁾ with low An-contents the An-rich component often could not be identified, apparently because of too low an intensity and too great a diffuseness of its lines. Such specimens then have in Fig. 5 an ordinate value corresponding to about low albite, but because of the higher bulk An-content the points fall beneath the estimated extension of the boundary curve in the peristerite region. That is, they fall completely outside of the plagioclase field in Fig. 5. In addition it was sometimes observed that in Fig. 5 the $\Delta(\theta)_1$ values of specimens with a peristerite chemical composition can fall by chance on this "plagioclase (low)" curve interpolated between $An_{1.6-16}$. However, this was never the case for the $\Delta(\theta)_2$ values in Fig. 6. An interpretation of this is not given here; a limitation of the powder method can nevertheless be recognised.

Small volume percentages of isolated inclusions of foreign minerals and/or antiperthitic K-feldspar do not affect the X-ray data of the plagioclase. It is obvious, however, that considerable errors can occur in the results, depending on whether one compares these data with a) the true composition of the plagioclase matrix, or b) a bulk chemical analysis.

V. CONCLUSIONS

If one assumes that the boundary curves in Figs. 5 and 6 correspond to the two *stable* series "plagioclase (high)" and "plagioclase (low)" defined in the introduction, then the following observations can be made:

1. Only those plagioclases whose values fall on the boundary curves appropriate to their Or-content are clearly defined as *stable* members of these series.

2. Plagioclases whose corresponding values lie within the field bounded by the curves "high" and "low" exist in one of the possible structurally intermediate states. In this case there is still no completely positive method of determining whether the state is stable or unstable. That is,

²⁾ Identified by means of single-crystal X-ray methods or by electron microscopy.

within the plagioclase field an infinite number of possibilities of ambiguity is theoretically possible with given data (LAVES, 1960a). It is therefore practicable to use the following terms, referred to in the introduction, to describe the structural state of such specimens (that is, of the majority of all plagioclases):

- a) D-plagioclase, if the value lies near the "plagioclase (high)" curve.
- b) O-plagioclase, if the corresponding value lies near the "plagioclase (low)" curve.
- c) O/D-plagioclase, if the value lies in the intermediate region of the field.

3. Or-contents of more than about 1 mol-% in the plagioclase matrix should not be ignored when conclusions are drawn on the basis of $\Delta(\theta)$ values. As a rule, however, the Or-content is notably less than the bulk Or-content of the morphological unit.

4. A quantitatively restrictive subdivision into grades of order (e. g. "intermediacy index" of SLEMMONS, 1962) is to be avoided a) because absolutely no quantitative relation between the $\Delta(\theta)$ values and the actual Al, Si distribution is known and b) because of the difficulties mentioned in the introduction of distinguishing between stable and unstable Al, Si distributions (see also point 2).

Provided that the above mentioned analytical and theoretical limitations are observed, relative statements on the structural state of different plagioclases may be made with the help of the diagrams here described.

Acknowledgements

This research was supported in part by the "Schweizerische Nationalfonds zur Förderung der wissenschaftlichen Forschung". All calculations were made at the computer centre of the ETH.

We are grateful to Professor F. Laves for many discussions and for his generous support of this project. Also we wish to thank Mr. P. Thommen for the wet chemical analyses, Miss H. Wulf for programming, Mr. H. Kroll for transferring the data to punch cards and Mr. H. J. Scheel for a feldspar synthesis, as well as the many donors of research material and all the helpful members of the institutes in Zürich and Münster.

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