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ZONED PLAGIOCLASES FROM ELBOW LAKE, MANITOBA
I. COMPARATIVE MICROPROBE AND OPTICAL ANALYSIS

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ABSTRACT

Nineteen zoned plagioclase crystals from a quartz-diorite stock at Elbow Lake, Manitoba have been examined by microprobe and U-stage, and the chemical compositions derived by the two methods for the several zones in each crystal. The principal observations and conclusions are: (1) Probe scans of the same zones on either side of the centre of several crystals yielded generally symmetrical compositional patterns; these results are taken as evidence for the reproducibility of the results. (2) The probe reveals within some zones that appear optically homogeneous, sharp compositional changes that are interpreted to be submicroscopic zonelets. (3) The probe reveals within other optically homogeneous zones smooth compositional changes that are interpreted as gradual chemical variations within one optical zone. (4) Certain zones characterized by wavy extinction are shown to have variable probe composition curves, as well as yielding generally poor agreement between the mean probe and the optical compositions. The authors conclude that optically derived compositions (and structural states) on such zones may be unreliable. (5) Regarding the compositions derived by the two methods, for 18 out of the 34 individual zones in the 18 crystals, the agreement is regarded as "poor", and this is attributed to one or more of: the presence of finely disseminated alteration products, the presence of coarse inclusions, the existence within one optical zone of submicroscopic zonelets or twin lamellae, or irregular topography of the upper surface of the probe sample. The results suggest that the compositions (and structural states) derived by only optical methods for the individual zones in many zoned plagioclases may be unreliable. (6) Either the probe or the optical results taken independently reveals that the zoning patterns from one crystal to another are distinctly different, and no scheme of zoning pervades all or most of the 18 crystals. This suggests a complex petrogenetic history for this stock.

INTRODUCTION

This paper is the first of a series of two or three that will present the results of a detailed examination by universal stage and electron microprobe of a large number of zoned plagioclase feldspar crystals in a small quartz-diorite stock at Elbow Lake, Manitoba. The purposes of this study were first, to compare for many of the individual zones the chemical composition derived by optical means with that derived by the microprobe, and second, from the optically observed zoning and twinning characteristics and from the microprobe results, to try to gain insights the genesis of this stock. A preliminary account of this work was given at the 1969 Annual Meeting of the Geological Society of America (Subbarao, Ferguson & Turnock 1969).

The Elbow Lake stock, which is about eight miles in diameter, had previously been sampled and described by Quaraishi (1967). He notes the occurrence of the zoned plagioclase crystals, gives evidence for an igneous origin of the stock, and presents a map of the sample locations which includes those samples used by the present authors.

In our examination, detailed optical observations were carried out on the U-stage on the several individual zones in each of about 200 different plagioclase crystals, and chemical compositions were derived for each of the zones. Of these crystals, 18 were chosen for probe analysis. The results of this comparative optical and probe examination of these 18 plagioclases are described in the present paper; the succeeding paper(s) will deal with the zoning and twinning in the large number of crystals examined only optically, and with the implications of the zoning and twinning for the genesis of this stock.

In this study, the experimental work on both the microscope and the probe was carried out by the first author (K.V.S.) ; the other two authors participated closely in the organizing of the project initially, in the interpretation of the results, and in the preparation of the published papers.

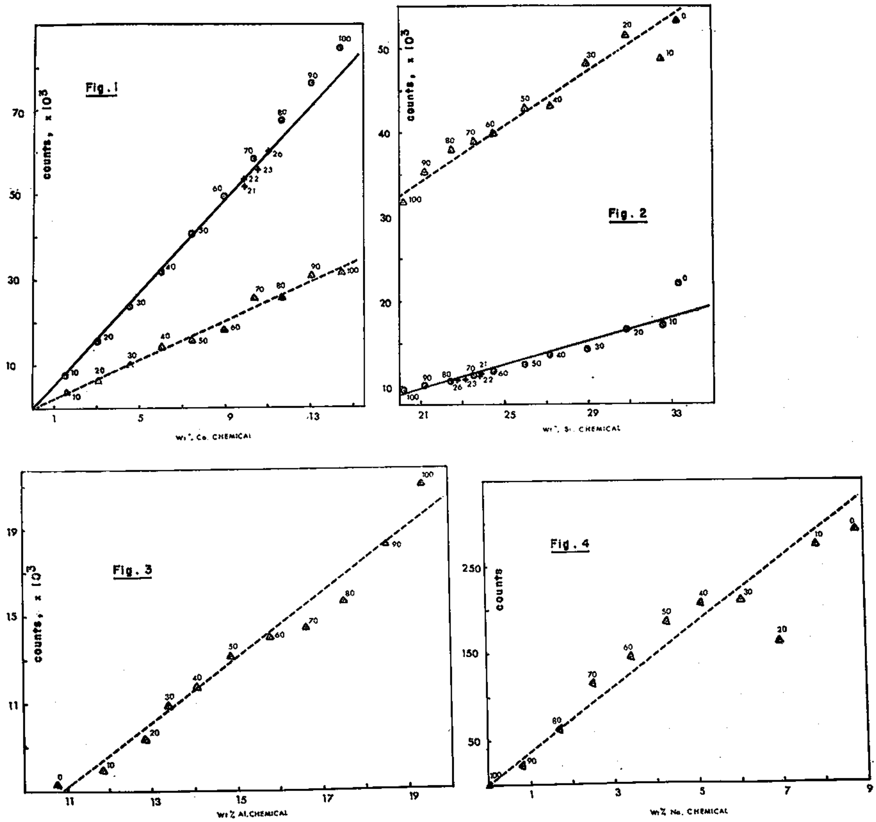
Concerning similar studies on plagioclases, Christophe-Michel-Levy & Goni (1964) compared probe and optical determinations for only one zoned crystal. Evans (1964) and Harme & Siivola (1966) compared compositions as determined by both methods, on four and one crystal respectively, but these comparisons were only between crystals from the same rocks, and not exactly the same crystals.

EXPERIMENTAL DETAILS

The optical measurements were carried out on a Fedorow 4-axis Universal Stage in accordance with the methods of Reinhard (1931) and Nikitin (1936), and compositions were derived from the recent high and low temperature curves of Burri *et al.* (1967). For extremely narrow zones, the Rittman zonal method (1929) was employed to determine the composition. In the case of doubtful results (twinned grains), the twin axis was constructed after the methods of Berek (1924) and Nikitin (1936). The errors in the derived compositions are estimated to be ± 2 mol% An, except for cloudy zones for which the estimated errors are ± 4 mol% An.

As part of the optical determinations, the structural state of each zone was also derived from the curves of Burri *et al.* (1967). The structural state is variable from low to high. This complex situation will be described in another paper.

The microprobe analyses were carried out on a Philips AMR/3 electron microprobe at the Whiteshell Nuclear Research Establishment, Pinawa, Manitoba. Sample preparation was similar to that of Smith (1965, p. 859). An electron beam of approximately 5 to 10 μm diameter was normally used with gun voltages ranging from 20 to 30 kv and with a sample current of 0.14 μa for all except the Na analyses for which a current of 0.05 μa was used. The errors in the derived compositions are estimated at about ± 5 mol% An.



Figs. 1 to 4. Calibration curves of microprobe counts (in 20 seconds) versus weight percent of the element in the standard samples used for the plagioclase analyses for particular runs. Fig. 1. Calcium. Fig. 2. Silicon. Fig. 3. Aluminum. Fig. 4. Sodium.

STANDARD SAMPLES AND CALIBRATION CURVES

Procedure

At the outset of this investigation, continuous scans for the four elements Na, Ca, Si and Al were made across several of the zoned plagioclases which had previously been examined optically. These scans produced erratic results and little correlation with the optically derived compositions. It was soon apparent that these erratic scan results were due mainly to inclusions, clouding and pits in the plagioclases, and that, rather than scanning, it would be necessary to take counts at individual spots which could be seen optically to be free of inclusions and clouding.

As standards for these plagioclase analyses, eleven synthetic glasses and four analyzed natural plagioclases were obtained from Dr. D. H. Lindsley and Dr. R. C. Emmons respectively. It was soon found, as described below, that some of the synthetic glasses are chemically inhomogeneous, and so in order to get a representative composition for each standards, five spots were selected at random and counts taken five times, each for 20 seconds. The mean values of the 25 measured counts on each standard glass sample were used to construct calibration curves for Ca, Si, Al and Na (Figs. 1 to 4 respectively). The scatter of the observed points about the best straight line through them compares favorably with that observed by Ribbe & Smith (1966) for their corresponding plots.

After the calibration curves had been constructed for all four of these elements, it was found that time would not permit analyzing the unknown samples for all four elements, so they were anlyzed (simultaneously) for only the two heavier elements Ca and Si. For each run on an unknown, new counts were taken for Ca and Si on four or five samples of the standards to derive a curve for that particular run. The compositions (in mole percent An) reported here (in Figs. 6 to 23) were derived from the Ca counts. The compositions derived from the Si counts appeared to be less reliable than those derived from Ca ; the compositions derived from the Si counts varied generally from those derived from the Ca counts by 5-10% An with the Si-derived compositions generally yielding a lower An composition. We cannot say whether this observation has significance for plagioclase compositions.

Inhomogeneity of the standard samples

In order to assess the inhomogeneity of the synthetic glasses, scans for Ca and Si were made across five of the samples for which the counts at different spots had been observed as most variable. Because the glasses appeared optically to be free of inclusions and clouding (in contrast with

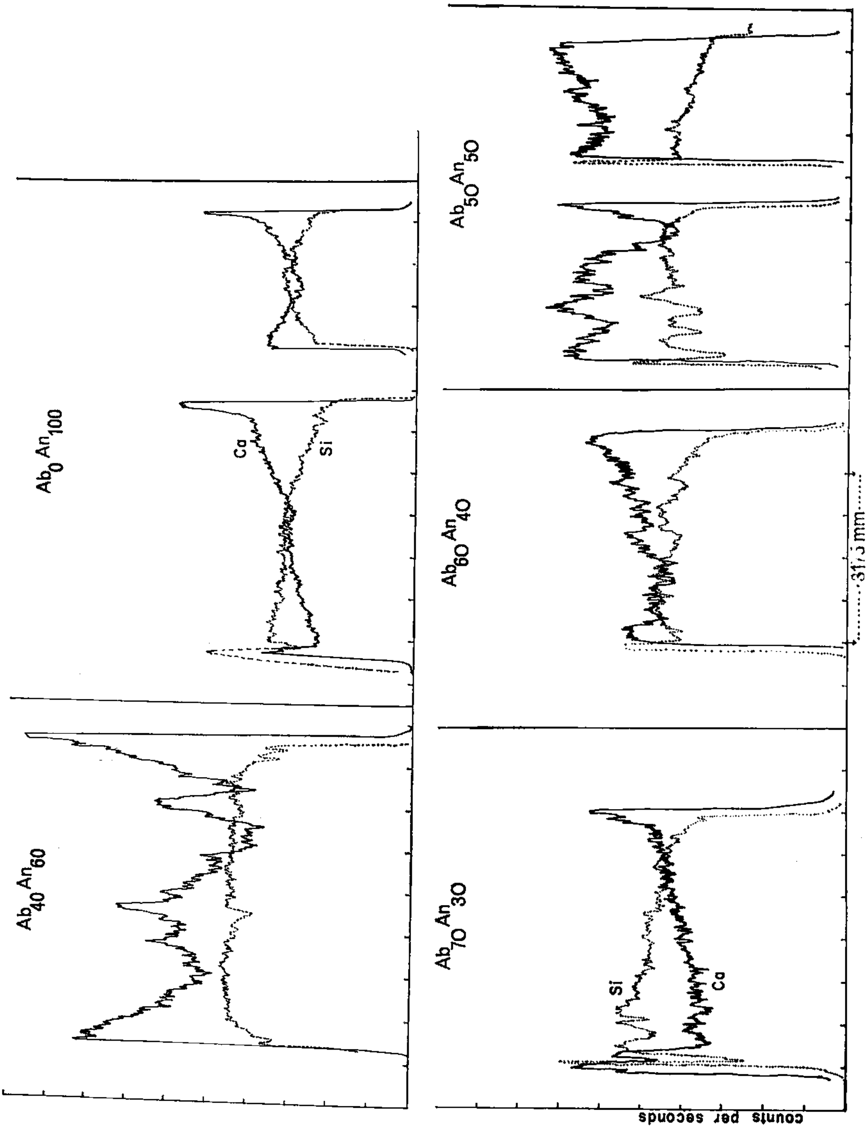


FIG. 5. X-ray scans for Ca and Si across five of Lindsley's synthetic plagioclase glasses.

the natural plagioclases), scanning was considered a valid method for determining the variations in composition. The scans for Ca and Si of the five glasses are shown in Fig. 5, where the curves for the two elements reveal the inhomogeneity of these glasses (and they show a general antipathetic relationship as one would expect). It was because of this inhomogeneity of the standard glasses that the procedure of taking five counts at each of five different spots on a given standard was adopted.

The only correction applied to the count readings was for background. Evans (1964), Boyd (1966) and Dahl (1969) have shown that where the unknown sample has nearly the same composition as the standards, and where only light elements are involved, corrections for absorption and for secondary fluorescence are negligible.

RESULTS

The compositions, expressed in terms of mole % An, obtained by both the optical and probe method on all 18 crystals are shown in Figs. 6 to 23. Table 1 is a summary of some of the observations (columns 3 to 8) and of the conclusions (columns 9 to 12) drawn from the results. Inevitably a large element of personal judgement was involved in deciding (a) what constitutes a boundary between major zones (in general, one showing corrosion characteristics, column 4), (b) the nature of the clouding (column 6), (c) whether the probe results indicated submicroscopic zoning (column 8), (d) the nature of the zoning pattern derived from the probe (column 9), (e) the quality of the zoning pattern correlation (column 10), and (f) the quality of the An% correlation (column 11). In general decisions (a) and (b) were made only by the first author (K.V.S.), whereas the other decisions were made jointly by all three authors.

These interpretations are as follows :

(1) For several of the crystals (Figs. 11, 15, 16 and 17), it was possible to scan by probe the same zones on either side of the centre of the crystal, and these yielded compositional patterns that are generally symmetrical about the centre. We regard this as evidence for the reproducibility of the probe results.

(2) The probe reveals within some zones that appear optically homogeneous, sharp compositional changes that we interpret to be submicroscopic zonelets (Table 1, column 8). This interpretation is based on analogy with the optical zoning, and implies the 3-dimensional extent of these compositional changes. This interpretation is supported by the symmetrical repetition across the centre of a symmetrical crystal, of these sharp com-

TABLE 1. SUMMARY OF CHARACTERISTICS AND COMMENTS ON RESULTS OF ZONED PLAGIOCLASES

In the table, OT = Optical Traverse, PT = Probe Traverse

		Optical Observations												
(1)	(2)	(3)	(4)	(5)	(6)	(7)	(8)	(9)		(10)	(11)	(12)		(13)
Fig. No.	Sample No. (Quaraishi, 1967)	Type of Zoning S=Simple C=Complex	Number of Major Zones	Major Zone Designation	Clouding C=Coarse FS=Fine Sericitized	Narrow Zones Present?	Submicroscopic Zonelets Revealed by Probe?	Zoning Pattern Derived from Optics*	Probe*	Zoning Pattern Correlation between OT & PT G=Good S=Satisfactory P=Poor	An% Correlation between OT & PT G=Good (<5% An) S=Satisfactory (5-10%) P=Poor (>10%)	If Poor An% Correlation Which Gives Higher An%?	Are Two Parallel?	Comments
6	Q81-1	C	2	A	FS	No	Yes	N	ON	S	S	-	-	-
				B	FS	No	Yes	N	ON	S	S	-	-	
7	Q81-4	C	1		C	Yes	Yes	ONE	OR	G	S	-	-	Very complex crystal
8	A37-1	C	3	A	C		Yes	R	OR	S	S	-	-	-
				B	FS	No	No	OR	ON	P	S	-	-	
				C	FS		Yes	N	ON	G	S	-	-	
9	R103-1	C	3	A	FS	-	No	ON	ON	G	G	-	-	-
				B	FS	Yes	No	OE	OE	G	G	-	-	
				C	-	-	Yes	-	OR	-	P	P	No	
10	A30-1	C	4	A	FS	Yes	Yes	OR	OR	G	P	P	Yes	-
				B	FS	-	Yes	R	ON	P	P	P	Yes	
				C	-	Yes	No	OR	OR	G	P	P	Yes	
				D	-	-	No	ON	ON	G	P	P	Yes	
11	A18-4	S	1	-	(1)	No	No	ON	ON	G	P	(2)	No	-
12	A18-5	S	1	I$^{(3)}$	C, FS	No	No	ON	ON	G	P	P	Yes	Wavy extinction
				II$^{(3)}$	C, FS	No	Yes	R	(4)	(4)	S	-	(4)	
13	A18-6	S	1	-	(1)	No	Yes	ON	ON	S	P	PT	Yes	Central zone shows wavy extinction
14	A18-7	S	1	-	-	No	Yes	ON	ON	S	P	OT	Yes	"

15	A17-5	S	1	-	-	No	No	ON	N	S	S	-	-	"
16	A17-6	S	1	-	-	No	Yes	N	ON	S	S	-	-	"
17	A17-1	S	1	-	C	No	No	N	N	G	S	-	-	-
18	Q79-4	S	1	-	-	No	No	ON	N	S	S	-	-	Central zone shows wavy extinction
19	Q79-1	C	2	IA(3) IB(3) I'(3)	C C -	Yes Yes No	No No No	ON OR -	OE ON -	P P -	P P G	P O, P -	Yes No -	-
20	A19-4	S	1	I(3) I'(3)	- (1)	No No	Yes Yes	N N	ON ON	S P	P P	O O, P	Yes No	Wavy extinction in the core of xl I
21	A19-1	S	1	-	FS(1)	No	Yes	N	ON	S	G	-	-	Core shows wavy extinction
22	E31-3	C	3	A B C	C FS FS	No Yes No	No No Yes	- ON N	OR N OR	- S P	P G P	P - O, P	Yes - No	Wavy extinction in broad zone
23	E31-1	C	2	A B	C -	No No	No No	R OE	OR N	P P	P P	O O	Yes No	-

* Symbols: E = Even, N = Normal, O = Oscillatory, R = Reversed

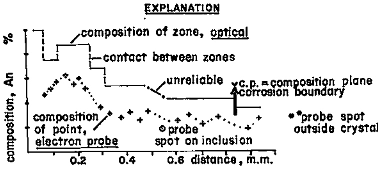
(1) Core partly clouded.

(2) Central region OT; marginal region PT

(3) Symbols I and I' indicate two individuals in twin relationship; I and II indicate two individuals in synneusis relationship

(4) Position of outer boundary uncertain

FIGS. 6 to 23. Photomicrographs of plagioclase crystals with probe traverse marked as line XY and some optically homogeneous zones marked as A, B, etc., and graphs of compositions as determined by both probe and optical methods. In some of the microphotographs, the traverse lines terminate in dark areas which are scratches in the slide (for aid in location).



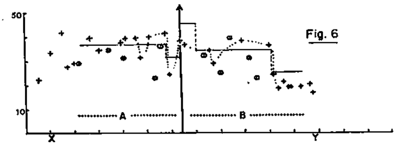
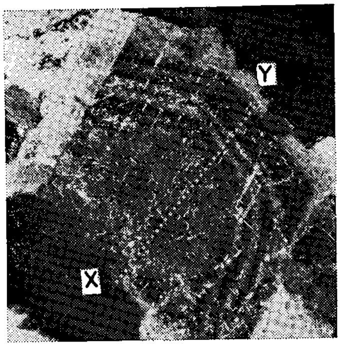


Fig. 6



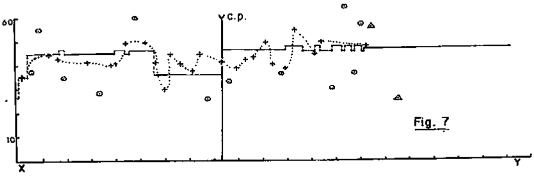
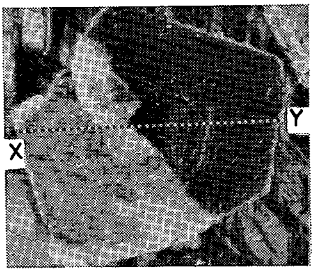


Fig. 7



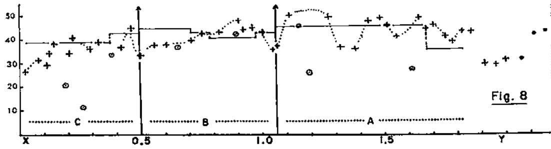
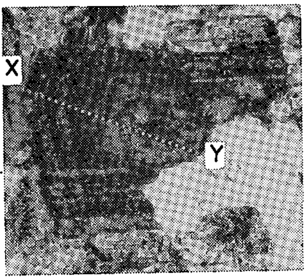


Fig. 8



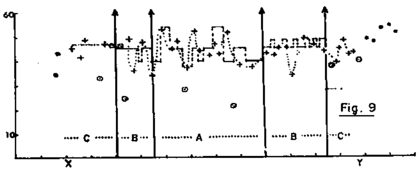
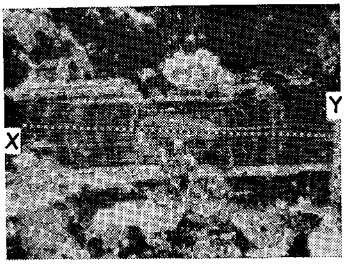
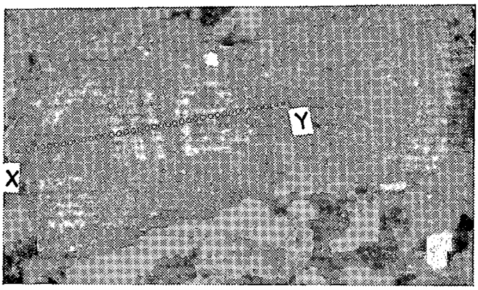


Fig. 9





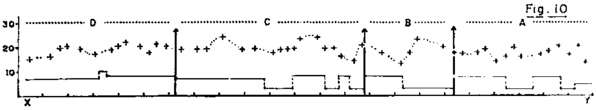
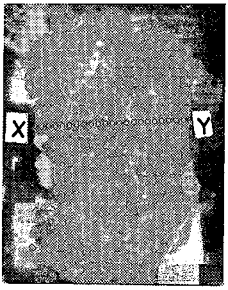
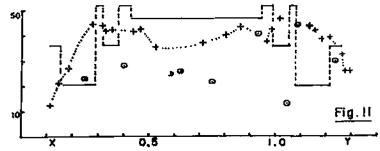


Fig. 10





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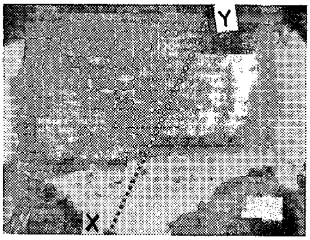


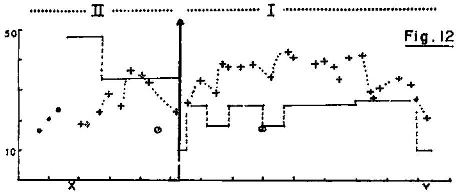
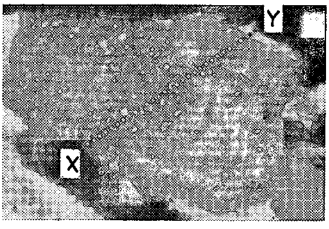
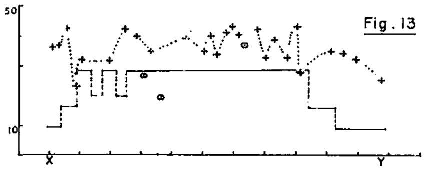


Fig. 12





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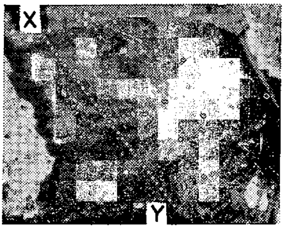


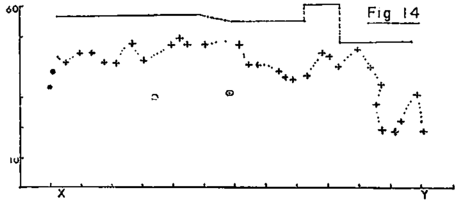


Fig 14

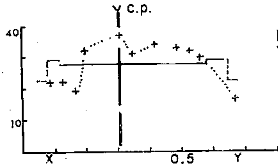
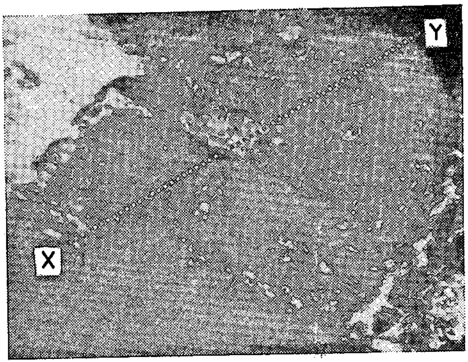


Fig. 15



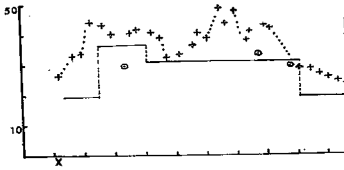
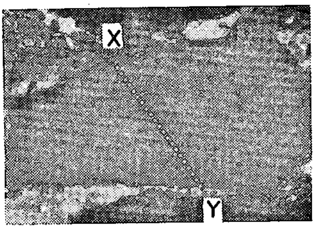
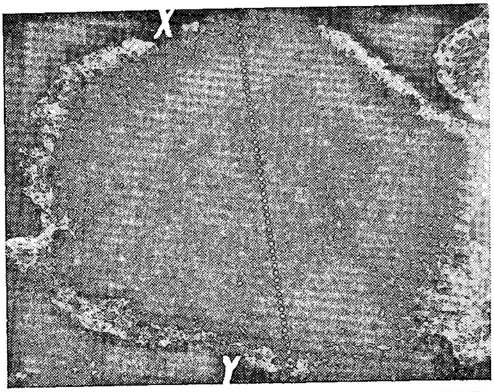
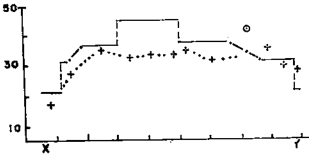


Fig. 16







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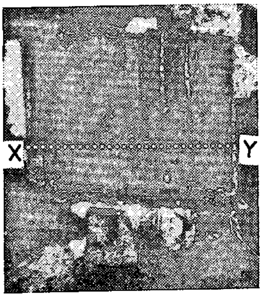


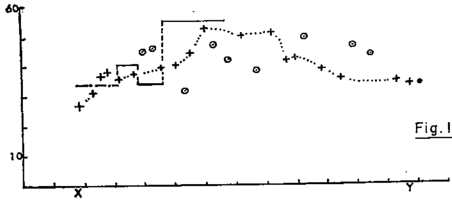


Fig. 18

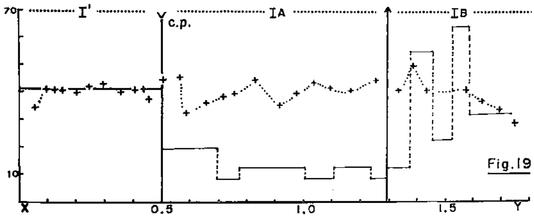
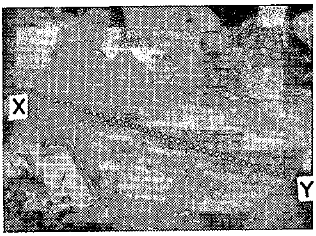


Fig. 19



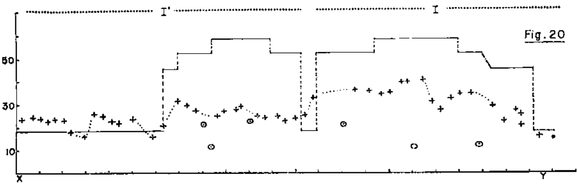
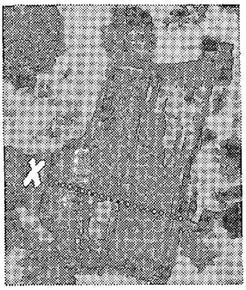


Fig. 20



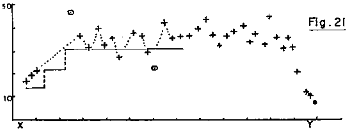
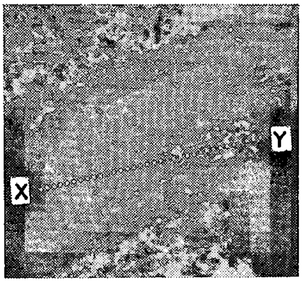


Fig. 21



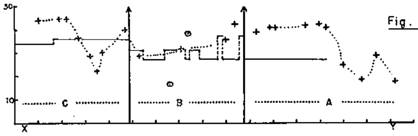


Fig. 22

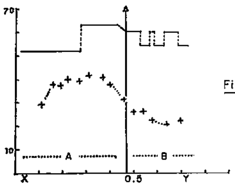
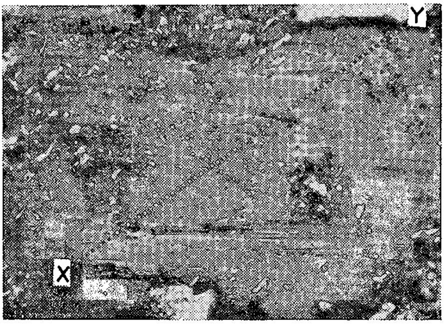


Fig. 23



positional changes on a fine scale as shown well in Fig. 21, and less well in Figs. 6, 9, 12 I, 13, 16, 20 I, 20 I'.

(3) In contrast with the sharp compositional changes shown by the probe traverses to exist in some of the optically single zones, certain other optical zones show smooth compositional changes in the probe traverses

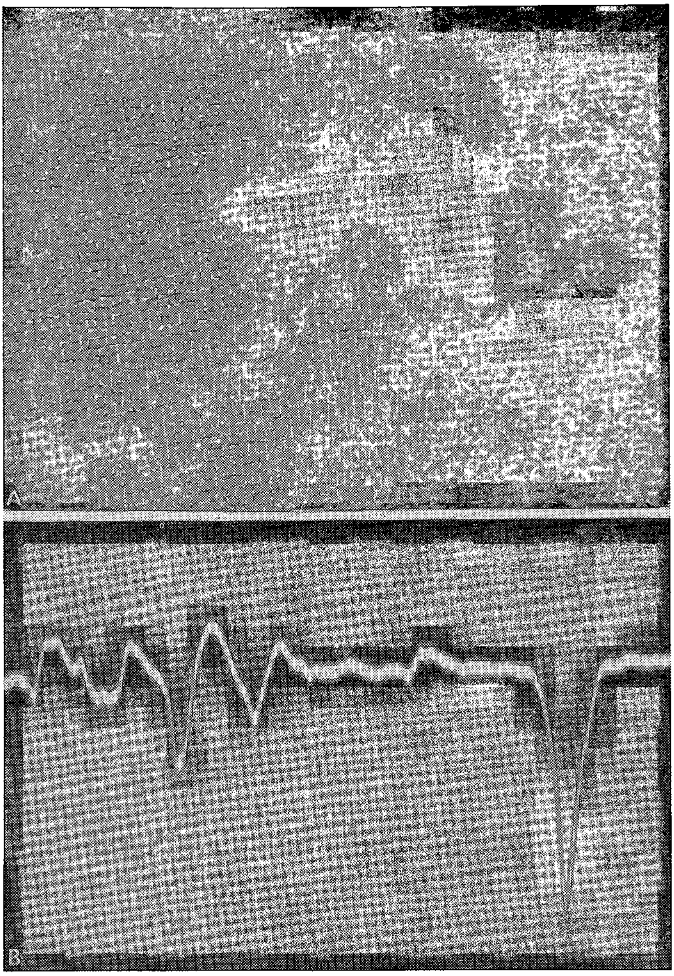


FIG. 24. Microprobe records of portions of sample No. A19-4. (a) Ca *x*-ray scan. The dark (Ca-free) and the bright (Ca-rich) areas are inclusions. (b) Backscatter line scan showing the topography along a line in an area near that shown in (a). The irregularities reveal pits in the surface of the sample. Width of field 250 μm.

(Figs. 11, 12, 15, 16, 18, 19, 21 and 23). We interpret these gradual compositional changes observed in the probe traverses to represent gradual chemical variations, as distinct from chemically different zonelets, within each such zone.

(4) The optical and probe curves for certain zones characterized by wavy extinction (Table 1, column 13), show that all such zones are characterized by either sharply or smoothly varying probe composition curves. Furthermore, most of these zones give generally poor agreement between the optically derived compositions and their mean probe compositions. This latter observation leads to the conclusion that composition (and presumably also structural states) derived by optical means for plagioclases showing wavy extinction should be treated with caution.

(5) Regarding the An% correlation between the optical and the mean probe results for a given zone, column (11) in Table 1 shows that, out of 34 individual zones (in the 18 crystals) 5 are designated as having a "Good" correlation by our criterion (difference <5% An), 11 a "Satisfactory" correlation (difference 5-10% An), and 18 a "Poor" correlation (difference >10% An). We attribute the "Poor" correlation in the zones so designated to one or more of the following : (a) The presence of finely disseminated alteration products ("FS" = Fine Sericitized, column 6) which could affect either or both of the optical and probe results. However, as columns (6) and (11) show in Table 1, not all zones showing a "Poor" An% correlation show "FS" clouding, and alternatively, not all "FS" zones show a "P" correlation. (b) The presence of coarse inclusions ("C" in column 6) which could affect the optical but not the probe determination (because it was possible to avoid such inclusions with the electron beam ; *e.g.* Fig. 24a) ; however, again Table 1 shows that there is no simple relationship between Coarse Clouding "C" and the poor compositional agreement "P" (column 11). (c) The existence within one optical zone of either (i) submicroscopic zonelets or (ii) submicroscopic twin lamellae, for which the mean optical effect may not correspond to the mean chemical composition of all the submicroscopic zonelets of twins. (d) The topography of the specimen in relation to the probe beam, which could introduce appreciable error into the probe results. Figure 24b shows an example of topography. Whatever the reasons for the poor agreement in over half of our observed zones between the chemical compositions derived by the two methods, we regard it as an important observation that these differences do exist. Our observations suggest that perhaps the compositions (and structural states) derived by only optical methods for the individual zones in many zoned plagioclases, especially complex ones with clouding, are not reliable, and that one cannot in general conclude from

the appearance of zones which ones are likely to yield reliable compositions from the optics.

(6) Either the probe or the optical results taken independently reveals that the zoning patterns from one crystal to another are distinctly different, even for two crystals in the same thin section. No scheme of zoning appears to pervade all or most of the 18 crystals, and this suggests a complex petrogenetic history for this stock. The petrogenesis of the stock is to be considered by two of us (K.V.S. and A.C.T.) in a separate paper not part of this series, on the basis of the optical observations on all 200 or so of the plagioclase crystals examined optically as well as on the probe and optical results reported here.

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