

The powder patterns and lattice parameters of plagioclase feldspars. I. The soda-rich plagioclases.

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Summary.—Geiger-counter powder records of soda-rich plagioclases have been carefully measured and indexed. The lattice parameters of the synthetic specimens are almost independent of the lime content, the largest variation (that in β^*) amounting to only 0.2°. After strong heating the powder patterns of the natural specimens closely approached those of the synthetic specimens.

Eight out of the nine natural plagioclases gave lattice parameters which fall within experimental error on a continuous line. There is a fairly large variation in lattice parameters, especially for γ^* , which changes by 2°. As the lime content increases the lattice parameters of the natural plagioclases approach those for the high-temperature synthetic and heated natural specimens until at An_{50} the separation is only one-fifth of that at An_0 . The ninth specimen (from a dacite) gives parameters midway between the curves, thus exhibiting a state of partial inversion.

The partially heated natural plagioclases and the plagioclase from the dacite give parameters indistinguishable within experimental error from the parameters of unheated plagioclases with a higher lime content. There is, therefore, no reliable powder X-ray method for the determination of composition in the region An_0 – An_{50} . If the composition is known the powder method may be used for the determination of the thermal state. If the plagioclase can be judged to be in the low-temperature state from geological evidence the powder method can be used to estimate the composition with an accuracy of 2% An.

RESEARCH carried out by many workers in the last few decades culminated in the conclusive demonstration by Tuttle and Bowen (1950) that there are two structural series of soda-rich plagioclases. Careful measurements of the optic axial angle, $2V$, and of the difference between the Bragg angles $2\theta(1\bar{3}2)^2$ and $2\theta(1\bar{3}1)$ showed that natural soda-rich plagioclases from low-temperature³ environments differed

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² The detailed measurements made in the present work show that this is the (131) reflection. Throughout the remainder of the paper the correction will be made without further reference.

³ A low-temperature environment is taken to be one for which there has been ample opportunity for attainment of the state which is stable at low temperatures. This definition includes plutonic conditions.

considerably from their synthetic counterparts but could be converted into a similar state by prolonged heating just below the solidus. A natural plagioclase from a high-temperature environment, rhyolite, gave results close to those for the synthetic material. Tuttle and Bowen found that the differences between the two series decreased as the proportion of lime increased from An_0 to An_{50} , at which composition the differences were slight.

As plagioclase feldspars are anorthic and have six independent lattice parameters, the author hoped that detailed examination of accurate X-ray powder patterns would lead to a routine X-ray method for the determination of the two variables, composition and thermal state. In addition, careful measurements might yield information on the position of the boundaries between regions of different structure. In view of the successful use by Donnay and Donnay (1952) of the Geiger-counter focusing-diffractometer in the study of high-temperature alkali feldspars, this method was chosen in preference to the less accurate photographic powder method and the more tedious single-crystal method. The powerful resolution and high accuracy at low angles of the focusing-diffractometer permit the indexing of powder patterns and the determination of the lattice parameters for anorthic crystals.

Three studies of the powder patterns of plagioclase feldspars have so far been recorded. Claisse (1950) published photographic powder patterns of eight natural plagioclases together with determinative curves based on the positions of certain reflections on the photographic film. The effect of thermal state was not considered. Chayes and Robbins (1953) have measured certain angular relations from diffractometer records of albites and oligoclases. Goodyear and Duffin (1954) have recently examined the powder patterns of sixteen natural and ten synthetic specimens by photographic methods, with the following two objects:

(i) to provide X-ray powder data for the identification of plagioclase feldspars when admixed with associated minerals, and (ii) to establish relationships enabling the composition and thermal modification of a feldspar to be determined from certain line positions in the X-ray pattern.

Studies of soda-rich plagioclases by single-crystal techniques have been reported by Cole, Sörum, and Taylor (1951), Laves (1954), Gay and Smith (1955), and Gay (1956). Laves found that some natural albites and oligoclases are unmixed into two phases of composition near An_0 and An_{50} (known as unmixing of the peristerite type). This unmixing was confirmed by Gay and Smith, who found that the lattice angles α^* and γ^* of the two phases corresponded to compositions near An_3 and

An₂₃. Cole *et al.* found that in the region An₃₀-An₇₀ the X-ray rotation patterns showed weak subsidiary layer-lines in addition to the strong layer-lines which correspond to the 7 Å. albite-type structure. More detailed study by Gay has revealed that most natural specimens in the composition range An₂₀-An₇₀ exhibit these subsidiary layer-lines. Certain specimens from high-temperature environments revealed either no subsidiary layer-lines or very weak diffuse ones. None of the powder records obtained in the present study has shown the presence of the subsidiary reflections: this is not surprising, for measurements made on the single-crystal cameras showed that their intensities are small.

Two of the specimens used in the present investigation of the lattice parameters lie in the range An₃-An₂₀ for which unmixing of the peristerite type might be expected. Single-crystal photographs of specimen 81822, composition An_{17.9}, reveal no unmixing, in contrast to photographs of the other specimen, BM 1940,27, composition An_{11.2}, which do. The powder record of the latter specimen, however, does not give a clear

TABLE I. Origin and chemical composition of the plagioclase samples.

Label number.	Origin.	Ref.	Weight %			100 An An+Ab
			An.	Ab.	Or.	
Amelia	Amelia, Va., pegmatite.	1	0.0	98.2	1.8	0.0
BM 1940,27	Kenya, pegmatite.	2	10.9	86.5	2.6	11.2
81822	South Carolina, pegmatite.	1	17.7	81.0	1.3	17.9
103086	Hawk Mine, N.C., pegmatite.	1	22.1	74.4	3.5	22.9
97490	Little Rock Creek, Mitchel Co., N.C., pegmatite.	1	29.9	66.5	3.6	31.0
152 (6)	Spanish Peak, Calif., gneissoid granodiorite.	3	35.9	62.6	1.5	36.4
64 (7)	San Luis Obispo Co., Calif., dacite.	3	36.1	60.7	3.2	37.3
144 (8)	Crestmore, Calif., granodiorite.	3	38.0	60.0	2.0	38.8
24 (9)	Essex Co., N.Y., anorthosite.	3	49.0	47.2	3.8	50.9
	Synthetic; 5 days 800° C. and 1000 Kg./cm. ² H ₂ O.	4	0.0	100.0	0.0	0.0
	Synthetic; 70 days 1080° C.; 90 days 1100° C.; dry.	5	10.0	90.0	0.0	10.0
	Synthetic; 26 days 1150° C., 14 days 1140° C.; dry.	5	30.0	70.0	0.0	30.0
	Synthetic; dry.	5	40.0	60.0	0.0	40.0
	Synthetic; 5 days 1230° C., 11 days 1170° C.; dry.	5	50.0	50.0	0.0	50.0

1. Kracek and Neuvonen (1952).
2. Game (1949).
3. Emmons *et al.* (1953).
4. Bowen and Tuttle (1950).
5. J. F. Schairer, unpublished.

indication of the presence of two phases and may be indexed on the basis of a single phase. Examination of powder records of several other specimens in this composition range (listed in table VII) also reveals the difficulty of detecting unmixing. Only when the proportions of the two phases are roughly equal can the unmixing be detected with certainty in powder records of peristerites. It is doubtful if the lower resolution of the photographic powder methods permits the detection of peristeritic unmixing even when the proportions are nearly equal. Although the unmixing is not obvious in the records of BM 1940,27 it is likely that the lattice parameters deduced on the basis of a single phase do not give a true average of the two phases: but the error is probably no larger than the effect produced by 2% An. Precautions for minimizing this error are given in the final section of this paper.

TABLE II. Indexed powder patterns of Amelia albite. [Cu-K α radiation. Only those reflections with $2\theta_{\text{calc}}$ sufficiently close to observed values of 2θ to make a contribution possible are listed. The observed values of 2θ are the means of four observations.

Natural Amelia albite.				Heated Amelia albite.			
<i>I</i> .	$2\theta_{\text{obs.}}$	$2\theta_{\text{calc.}}$	(hkl) .	<i>I</i> .	$2\theta_{\text{obs.}}$	$2\theta_{\text{calc.}}$	(hkl) .
75	13.86	13.855	001	—	—	13.73	110
—	—	13.89	020	20	13.775	13.79	020
2	14.9	14.98	11 $\bar{1}$	30	13.93	13.94	001
4	15.85	15.86	$\bar{1}11$	3	15.17	15.185	11 $\bar{1}$
60	22.057	22.055	20 $\bar{1}$	3	15.64	15.68	$\bar{1}11$
20	23.071	23.07	$\bar{1}11$	2	18.92	18.955	02 $\bar{1}$
85	23.538	23.54	111	60	22.00	21.995	20 $\bar{1}$
70	24.155	24.155	130	40	22.915	22.905	$\bar{1}11$
50	24.306	24.27	13 $\bar{1}$	100	23.73	23.715	130
—	—	24.335	130	—	—	23.75	111
5	25.185	—	002 β	35	24.46	24.47	130
35	25.392	25.40	11 $\bar{2}$	—	—	24.54	13 $\bar{1}$
5	25.57	25.565	22 $\bar{1}$	5	25.35	—	002 β
20	26.418	26.415	$\bar{1}12$	15	25.63	25.635	11 $\bar{2}$
360	27.915	27.92	002	25	26.445	26.46	$\bar{1}12$
—	—	27.99	040	—	—	27.66	220
—	—	28.12	220	160	27.785	27.79	040
30	28.325	28.325	220	—	—	27.805	20 $\bar{2}$
30	30.148	30.145	131	360	28.095	28.095	002
—	—	30.225	22 $\bar{2}$	40	28.525	28.535	220
50	30.48	30.51	02 $\bar{2}$	25	29.62	29.62	131
—	—	30.54	04 $\bar{1}$	30	30.295	30.305	04 $\bar{1}$
20	31.210	31.23	131	35	30.54	30.52	02 $\bar{2}$
3	31.47	31.505	13 $\bar{2}$	5?	30.65	30.655	22 $\bar{2}$
3	32.11	32.105	022	35	31.61	31.595	131
α_1	—	—	—	α_1	—	—	—
—	—	32.155	041	12	33.74	33.745	$\bar{1}32$
15	33.946	33.94	$\bar{1}32$	—	—	33.78	31 $\bar{1}$

TABLE II (cont.)

Natural Amelia albite.				Heated Amelia albite.			
<i>I.</i>	2θobs.	2θcalc.	(<i>hkl</i>).	<i>I.</i>	2θobs.	2θcalc.	(<i>hkl</i>).
20	34.996	34.995	24 $\bar{1}$	—	—	35.57	31 $\bar{2}$
3	35.34	35.32	31 $\bar{2}$	20	35.62	35.595	24 $\bar{1}$
4	35.72	35.70	1 $\bar{1}$ 2	20	35.80	35.685	1 $\bar{1}$ 2
2	35.95	35.945	221	—	—	35.78	24 $\bar{1}$
2	36.5	36.495	221	—	—	35.835	312
7	36.757	36.76	241	3	36.655	36.64	112
5	36.95	36.955	15 $\bar{1}$	—	—	36.645	150
5	37.35	37.305	240	7	37.94	37.925	240
—	—	37.425	150	5	39.12	39.085	331
7	37.64	37.625	310	4	39.47	39.44	33 $\bar{1}$
10	38.78	38.78	33 $\bar{1}$	5	39.735	39.72	113
4	39.52	39.515	113	3	40.13	40.13	132
10	41.21	41.20	042	—	40.49	40.485	242
—	—	41.22	242	—	—	40.515	151
20	42.51	42.50	060	2	41.34	41.32	042
—	—	42.62	133	—	—	41.375	332
15	42.63	42.665	151	20	42.19	42.19	060
5	43.55	43.545	241	—	—	42.535	313
5	44.48	44.47	241	15	42.6	42.665	003
3	45.29	45.305	202	—	—	42.70	132
10	45.78	45.795	061	7	43.025	43.025	151
5	47.125	47.145	42 $\bar{1}$	3 ?	45.5	45.47	113
—	—	48.105	35 $\bar{1}$	5	45.62	45.605	311
20	48.115	48.11	222	—	—	46.735	243
8	49.17	49.19	40 $\bar{3}$	5	46.77	46.755	222
10	49.38	49.405	152	3	47.2	47.19	422
—	—	49.405	261	15	48.57	48.57	222
—	—	49.435	260	5	49.22	49.175	403
15	49.80	49.82	260	—	—	49.875	043
—	—	49.825	243	20	49.89	49.88	113
65	49.945	49.93	043	—	—	49.88	400
—	—	49.98	35 $\bar{1}$	5	50.1	50.11	260
—	—	49.985	062	35	50.91	50.915	113
20	50.54	50.55	113	25	51.46	51.455	204
30	51.115	51.12	204	—	—	—	—

Seven out of the nine natural plagioclases used in the determination of lattice parameters were converted into the high-temperature form by prolonged heating just under the solidus. A large sample was put into a furnace and heated for several days. A small amount was removed and a powder record obtained. The remainder was reheated. This cycle was continued until no further change occurred in the X-ray pattern, when it was assumed that the material was in the high-temperature form. Lack of material prevented the complete conversion of specimen BM 1940.27.

TABLE III. 2θ values for the soda-rich plagioclases.

Indices.	20I.	III.	130.	112.	040.	002.	220.	181.	131.	132.	24I.	000.	222.	113.	204.	
<i>Unheated</i>																
Amelia...	22-057	23-071	23-538	24-306	24-155	25-392	26-418	—	—	30-148	31-210	33-946	34-896	36-757	42-507	48-118
BM 1940,27	22-042	23-03	23-567	24-19	25-445	26-427	—	—	—	30-04	31-295	33-908	35-108	36-557	42-425	48-163
81822	22-013	22-981	23-586	24-078	24-248	25-486	26-428	—	—	29-941	31-346	33-876	35-202	36-42	42-363	48-235
103086	22-00	22-958	23-60	23-988	24-275	25-508	26-455	—	—	29-838	31-363	33-853	35-370	36-223	42-31	48-245
97490	21-983	22-918	23-612	23-922	24-302	25-542	26-435	—	—	29-782	31-377	33-799	35-319	36-125	42-285	48-32
152	22-01	22-925	23-655	23-88	24-36	25-565	26-458	27-84	28-032	28-365	29-73	31-465	33-815	35-43	42-275	48-385
64	21-99	22-865	—	—	24-39	25-635	26-455	27-80	28-04	28-44	29-65	31-49	33-76	—	42-22	48-42
144	22-015	22-895	—	—	24-375	25-575	26-45	27-815	28-035	28-37	29-71	31-46	33-81	—	42-24	48-37
24	21-967	22-862	—	—	24-369	25-644	26-435	27-80	28-037	28-374	29-649	31-462	33-772	—	42-235	48-354
<i>Heated</i>																
Amelia...	22-00	22-915	—	—	24-46	25-63	26-445	27-785	28-035	28-525	29-62	31-61	33-74	—	42-19	48-57
BM 1940,27; 12 days	22-02	22-97	—	—	24-33	25-52	26-42	27-84	28-005	28-315	29-81	31-435	33-82	—	42-305	48-40
BM 1940,27; 24 days	22-012	22-92	—	—	24-397	25-57	26-435	27-827	28-047	28-44	29-665	31-47	33-76	—	42-225	48-49
81822	21-998	22-863	—	—	24-44	25-61	26-44	27-773	28-053	28-483	29-60	31-54	33-735	—	42-18	48-49
103086	21-985	22-868	—	—	24-435	25-60	26-42	27-75	28-04	28-47	29-59	31-52	33-66	—	42-15	48-46
97490	21-99	22-855	—	—	24-427	25-632	26-45	27-775	28-045	28-465	29-58	31-492	33-69	—	42-16	48-45
152	21-988	22-868	—	—	24-460	25-635	26-47	27-797	28-057	28-477	29-587	31-535	33-75	—	42-18	48-46
64	21-988	22-848	—	—	24-435	25-635	26-465	27-782	28-027	28-442	29-562	31-525	33-72	—	42-185	48-47
<i>Synthetic</i>																
A ₉₀	22-005	22-885	—	—	24-465	25-65	26-48	27-80	28-085	28-53	29-59	31-62	33-75	—	42-19	48-54
Ad ₉₀	22-025	22-875	—	—	24-475	25-66	26-47	27-805	28-075	28-53	29-605	31-585	33-735	—	42-19	48-56
AB ₉₀	22-025	22-865	—	—	24-465	25-67	26-43	27-795	28-04	28-485	29-69	31-52	33-735	—	42-18	48-475
AB ₉₀	22-02	22-805	—	—	24-47	25-675	26-46	27-81	28-03	28-46	29-55	31-525	33-735	—	42-195	48-395

Experimental procedure for the determination of the lattice parameters.

X-ray powder records were taken on a Norelco high-angle diffractometer using either pressed mounts made by the method of Adams and Rowe (1954) or carefully made smear mounts. The pressed mounts gave slightly more intense peaks than the smear mounts, but their preparation was more tedious and required more material. The settings of the diffractometer were: time constant, 4 seconds; divergence and scatter slits, 1° ; receiving slit, 0.006 inches; scanning speed, 0.25 degree of 2θ per minute; chart scale, one degree per inch; filtered Cu- $K\alpha$ radiation.

Measurements of the plagioclase peaks obtained from both types of mount agreed within random experimental error only when the records were calibrated from the peaks given by the secondary standard, quartz, which was intermixed with the plagioclase in the mount. The quartz sample is number F2-16 (Tuttle and Keith, 1952) and the positions of the quartz reflections were taken to be those listed by Parrish (1953).

The measurements on the synthetic albite disagreed with those given by Donnay and Donnay (1952) who used the same sample and the same instrumental setting except for the width of the receiving slit, which was half that used here. The corrected values of 2θ were, on the average, about 0.025° higher than those given by Donnay and Donnay. In view of this discrepancy further measurements using both pressed and smear mounts were taken, but the discrepancy persisted. As the quartz and plagioclase were ground together in acetone for 5 minutes it seems certain that a homogeneous mixture was obtained and that the correction *relative to quartz* was valid. Donnay and Donnay calibrated the diffractometer by using a separate pressed mount of the secondary standard, silicon. There appear to be two possible explanations of the discrepancy: either the lattice parameters of quartz and silicon given by Parrish are not applicable to the samples used here, or the indirect method of calibration used by Donnay and Donnay was in error. In order to test the two explanations, photographic powder patterns of the silicon and the quartz were taken and measured according to the extrapolation procedure given by Nelson and Riley (1945), with the following results:

The lattice parameter of the silicon agrees within the experimental error of 1/20 000 with the value listed by Parrish and thence used by Donnay and Donnay.

The relative positions of the reflections of quartz and silicon obtained from a mixed powder specimen agree very closely with the values given by Parrish. Assuming the 2θ -values for the silicon (533) reflection to be identical with those given by Parrish, the adjacent quartz reflections (1453) and (2026) near $2\theta = 137^\circ$ are 0.02° in 2θ lower than those given by Parrish. Tuttle and Keith, however, found that for this particular quartz sample, F2-16, the (2354) quartz reflection near $2\theta = 154^\circ$ was 0.02° in 2θ higher than the value given by Parrish. The discrepancy of 0.02° in 2θ at these high angles corresponds to a discrepancy of about 1/15 000 in the lattice parameters and to 0.002° in 2θ for $2\theta \approx 30^\circ$.

From these measurements it seems reasonably certain that the positions of the quartz reflections assumed in the present work are accurate to about 1/15 000 and that an error in the indirect calibration method of Donnay and Donnay is the source of the discrepancy.

TABLE IV. Lattice parameters for soda-rich plagioclases.

Sample.	Heating conditions.	Wt. % An An+Ab	a Å.	b Å.	c Å.	α degrees.	β degrees.	γ degrees.	a* Å. ⁻¹	b* Å. ⁻¹	c* Å. ⁻¹	α* degrees.	β* degrees.	γ* degrees.
Amelia	natural	0-0	8-144	12-787	7-160	94-26	116-58	87-67	0-1373	0-07843	0-15648	86-40	63-51	90-48
BM 1940,27	natural	11-2	8-149	12-806	7-153	94-12	116-56	88-17	0-1372	0-07829	0-15662	86-31	63-50	89-99
81822	natural	17-9	8-152	12-821	7-139	93-99	116-46	88-58	0-1370	0-07819	0-15681	86-25	63-57	89-60
103086	natural	22-9	8-169	12-836	7-134	93-83	116-45	88-99	0-1367	0-07809	0-15688	86-23	63-56	89-22
97490	natural	31-0	8-171	12-846	7-129	93-75	116-44	89-25	0-1367	0-07803	0-15700	86-19	63-55	88-97
152	natural	36-4	8-160	12-848	7-121	93-72	116-36	89-52	0-1368	0-07802	0-15710	86-09	63-61	88-69
64	natural	37-3	8-166	12-864	7-112	93-51	116-30	89-98	0-1366	0-07792	0-15721	86-10	63-65	88-28
144	natural	38-8	8-164	12-857	7-118	93-69	116-34	89-59	0-1367	0-07796	0-15712	86-08	63-63	88-63
24	natural	50-9	8-180	12-859	7-112	93-52	116-27	89-89	0-1364	0-07794	0-15717	86-12	63-68	88-38
Amelia	23 days, 1060° C.	0-0	8-165	12-872	7-111	93-45	116-43	90-28	0-1368	0-07788	0-15743	86-01	63-50	87-97
BM 1940,27	12 days, 1060° C.	11-2	8-157	12-841	7-131	93-83	116-45	89-24	0-1369	0-07806	0-15698	86-10	63-54	88-94
BM 1940,27	24 days, 1060° C.	11-2	8-167	12-864	7-122	93-62	116-44	89-87	0-1368	0-07792	0-15720	86-03	63-51	88-35
81822	42 days, 1090° C.	17-9	8-169	12-877	7-112	93-48	116-35	90-20	0-1367	0-07780	0-15729	86-02	63-58	88-05
103086	42 days, 1090° C.	22-9	8-175	12-884	7-115	93-40	116-36	90-24	0-1366	0-07780	0-15723	86-08	63-57	88-04
97490	42 days, 1090° C.	31-0	8-174	12-880	7-112	93-34	116-30	90-29	0-1365	0-07782	0-15719	86-13	63-64	88-02
152	28 days, 1090° C.	36-4	8-169	12-874	7-109	93-46	116-29	90-23	0-1366	0-07787	0-15727	86-02	63-64	88-03
64	14 days, 1090° C.	37-3	8-172	12-876	7-112	93-44	116-29	90-28	0-1366	0-07785	0-15720	86-03	63-64	87-99
An ₀	hydrothermal	0-0	8-171	12-872	7-108	93-47	116-39	90-33	0-1367	0-07788	0-15743	85-96	63-53	87-90
An ₁₀	dry, synthetic	10-0	8-160	12-872	7-109	93-52	116-37	90-24	0-1369	0-07788	0-15740	85-95	63-56	87-99
An ₃₀	dry, synthetic	30-0	8-163	12-875	7-107	93-39	116-27	90-29	0-1367	0-07786	0-15728	86-07	63-66	88-00
An ₅₀	dry, synthetic	50-0	8-178	12-870	7-102	93-36	116-18	90-40	0-1363	0-07789	0-15727	86-06	63-74	87-90

Lattice parameter measurements recorded in the literature.

Kodarma†	natural	0	8-135	12-788	7-154	94-23	116-52	87-71	0-1374	0-07841	0-15652	86-42	63-57	90-43
24†	natural	50-9	8-156	12-773	7-170	94-32	116-67	87-62	0-1365	0-07792	0-15711	86-21	63-57	88-43
Amelia‡	natural	0	8-115	12-871	7-127	93-57	116-50	89-98	0-1370	0-07852	0-15635	86-37	63-43	90-50
Amelia‡	heated	0-0	8-172	12-884	7-123	93-65	116-45	90-19	0-1371	0-07798	0-15713	86-03	63-45	88-25
An ₅₀ §	hydrothermal	0-0	8-172	12-884	7-123	93-65	116-45	90-19	0-1368	0-07782	0-15721	85-83	63-48	87-97

† Cole, Sörum, and Taylor (1951), ‡ Laves (1952), § Donnay and Donnay (1952).

The values of 2θ for the plagioclase reflections, corrected by the quartz internal standard, were converted into Q-values by the tables of Donnay and Donnay (1951). The peaks were indexed with the aid of the accurate data given by Cole *et al.* (1951) and Donnay and Donnay (1952). Table II contains the data for the indexed patterns of low- and high-temperature albite. All other powder patterns obtained from soda-rich plagioclases may be indexed by interpolation, though some caution is necessary, especially for (hkl) and $(hk0)$ reflections, which interchange position as γ^* passes through 90° .

The relation between Q and the reciprocal lattice parameters is given by the equation

$$Q_{(hkl)} = 1/d_{(hkl)}^2 = h^2a^{*2} + k^2b^{*2} + l^2c^{*2} + 2klb^*c^*\cos\alpha^* \\ + 2lhc^*a^*\cos\beta^* + 2hk a^*b^*\cos\gamma^*.$$

Thus for each reflection (hkl) there is a linear equation connecting the observable quantity Q with the six functions of the lattice parameters by the known values of h , k , and l . Although only six values of Q are needed for a determination of the six lattice parameters, it is desirable to obtain as many values of Q as possible in order to increase the accuracy of determination.

It was soon found that trial-and-error adjustment of the lattice parameters was tedious and uncertain, and a systematic method was adopted that yields least-squares estimates of the variable quantities a^{*2} , $b^*c^*\cos\alpha^*$, &c. The method is based on the standard procedure for the solution of linear equations described by Fisher (1950). Since the method may be used for powder X-ray data of any soda-rich plagioclase and a variation of it may be used for any solid-solution series, a brief account will be given here.

The procedure for calculating the lattice functions a^{*2} , b^{*2} , c^{*2} , $2b^*c^*\cos\alpha^*$, $2c^*a^*\cos\beta^*$ and $2a^*b^*\cos\gamma^*$ of unheated specimen 152 is displayed in table V. The values of \bar{h}^2 , \bar{k}^2 , \bar{l}^2 , \bar{kl} , \bar{lh} , and \bar{hk} for the set of reflections $20\bar{1}$, $\bar{1}1\bar{1}$, $11\bar{1}$, $\bar{1}30$, 130 , $11\bar{2}$, $\bar{1}\bar{1}2$, $\bar{1}31$, $\bar{1}32$, $24\bar{1}$, $\bar{2}41$, 060 , 222 , 113 , and $20\bar{4}$ are listed as the array D , E , F , G , I , J . The numbers n , o , p , q , r , s are obtained by multiplying corresponding values of Q and D , E , F , G , I , J , and adding. Thus

$$10^5 n = 4 \times 6132 + 1 \times 6645 + \dots + 1 \times 30\ 971 + 4 \times 31\ 698; \\ 10^6 s = 0 \times 6132 + \bar{1} \times 6645 + \dots + 1 \times 30\ 971 + 0 \times 31\ 698.$$

The normal equations for this set of reflections, represented in matrix form, are:

$$\begin{vmatrix} 90 & 194 & 117 & 25 & -39 & 14 \\ 194 & 2234 & 121 & 73 & -47 & -10 \\ 117 & 121 & 408 & 67 & -111 & 13 \\ 25 & 73 & 67 & 121 & 13 & -47 \\ -39 & -47 & -111 & 13 & 117 & 25 \\ 14 & -10 & 13 & -47 & 25 & 194 \end{vmatrix} \begin{vmatrix} u \\ v \\ w \\ x \\ y \\ z \end{vmatrix} = \begin{vmatrix} n \\ o \\ p \\ q \\ r \\ s \end{vmatrix}$$

where u , v , w , x , y , z are the least-squares estimates of the functions a^{*2} , b^{*2} , c^{*2} ,

$2b^*c^*\cos\alpha^*$, $2c^*a^*\cos\beta^*$, $2a^*b^*\cos\gamma^*$. This equation may be transformed into the equation

$$\begin{pmatrix} u \\ v \\ w \\ x \\ y \\ z \end{pmatrix} = \begin{pmatrix} \text{Matrix} \\ A \end{pmatrix} \begin{pmatrix} n \\ o \\ p \\ q \\ r \\ s \end{pmatrix}$$

where the covariance matrix A is the inverse of the left-hand matrix in the set of normal equations. The numerical values of the covariance matrix A are listed in table VI. In table V the above set of equations is written out in full and the least-squares estimates u, v, w, x, y, z have been calculated from the values of n, o, p, q, r, s derived previously (R and C in table V refer to the row and column of matrix A).

The standard deviation, σ , of the individual measurements is estimated by $\hat{\sigma}$ where

$$\begin{aligned} (\hat{\sigma})^2 &= \sum (Q_{\text{obs}} - Q_{\text{calc}})^2 / N - 6 \\ &= \frac{\sum Q_{\text{obs}}^2 - nu - ov - pw - qx - ry - sz}{N - 6} \end{aligned}$$

where N is the number of observations, Q_{calc} is the value of $1/d^2$ calculated from the

TABLE V. Calculation form for obtaining reciprocal lattice functions from the observed Q values.

$hkl.$	$D.$	$E.$	$F.$	$G.$	$I.$	$J.$	$10^5 \times Q_{\text{obs.}}$	$10^5 n =$	$10^5 \times Q_{\text{calc.}}$
20 $\bar{1}$	4	0	1	0	$\bar{2}$	0	6132	$10^5 n = 5.05706$	6133
$\bar{1}\bar{1}$	1	1	1	$\bar{1}$	1	$\bar{1}$	6645	$10^5 o = 19.43690$	6642
111	1	1	1	1	1	1	7069	$10^5 p = 10.99405$	7074
$\bar{1}\bar{3}0$	1	9	0	0	$\bar{3}$	$\bar{3}$	7202	$10^5 q = 2.99321$	7203
130	1	9	0	0	0	3	7491	$10^5 r = -1.48679$	7495
$\bar{1}\bar{1}\bar{2}$	1	1	4	$\bar{2}$	$\bar{2}$	1	8237	$10^5 s = 1.01662$	8245
$\bar{1}\bar{1}\bar{2}$	1	1	4	2	$\bar{2}$	$\bar{1}$	8813	—	8817
$\bar{1}\bar{3}\bar{1}$	1	9	1	$\bar{3}$	1	$\bar{3}$	11075	—	11079
131	1	9	1	3	1	3	12371	—	12375
$\bar{1}\bar{3}\bar{2}$	1	9	4	6	$\bar{2}$	$\bar{3}$	14256	—	14258
24 $\bar{1}$	4	16	1	$\bar{4}$	$\bar{2}$	8	15606	—	15595
$\bar{2}\bar{4}\bar{1}$	4	16	1	4	$\bar{2}$	$\bar{8}$	16152	—	16151
060	0	36	0	0	0	0	21918	—	21914
222	4	4	4	4	4	4	28306	—	28298
113	1	1	9	3	3	1	30971	—	30975
204	4	0	16	0	$\bar{8}$	0	31698	—	31693

$u = a^{*2}$	$= nR1C1 + oR1C2 + pR1C3 + qR1C4 + rR1C5 + sR1C6 = 0.18721608$
$v = b^{*2}$	$= nR2C1 + oR2C2 + pR2C3 + qR2C4 + rR2C5 + sR2C6 = 0.06087481$
$w = c^{*2}$	$= nR3C1 + oR3C2 + pR3C3 + qR3C4 + rR3C5 + sR3C6 = 0.24678439$
$x = 2b^*c^*\cos\alpha^*$	$= nR4C1 + oR4C2 + pR4C3 + qR4C4 + rR4C5 + sR4C6 = 0.01670947$
$y = 2a^*c^*\cos\beta^*$	$= nR5C1 + oR5C2 + pR5C3 + qR5C4 + rR5C5 + sR5C6 = 0.19100256$
$z = 2a^*b^*\cos\gamma^*$	$= nR6C1 + oR6C2 + pR6C3 + qR6C4 + rR6C5 + sR6C6 = 0.00492788$

$$\sum Q_{\text{obs}}^2 - (nu + ov + pw + qx + ry + sz) = 4.271 \times 10^{-7}$$

$$\hat{\sigma}^2 = 4.271 \times 10^{-7} / (16 - 6) = 4.271 \times 10^{-8}$$

$\sigma(a^{*2})$	$= \sqrt{(4.271 \times 10^{-8} \times R1C1)} = 3.2 \times 10^{-5}$
$\sigma(b^{*2})$	$= \sqrt{(4.271 \times 10^{-8} \times R2C2)} = 1.6 \times 10^{-5}$
$\sigma(c^{*2})$	$= \sqrt{(4.271 \times 10^{-8} \times R3C3)} = 4.9 \times 10^{-6}$
$\sigma(2b^*c^*\cos\alpha^*)$	$= \sqrt{(4.271 \times 10^{-8} \times R4C4)} = 7.6 \times 10^{-6}$
$\sigma(2a^*c^*\cos\beta^*)$	$= \sqrt{(4.271 \times 10^{-8} \times R5C5)} = 8.1 \times 10^{-6}$
$\sigma(2a^*b^*\cos\gamma^*)$	$= \sqrt{(4.271 \times 10^{-8} \times R6C6)} = 5.5 \times 10^{-6}$

predicted lattice parameters. The standard deviations of the least-squares estimates of u , v , w , x , y , z are given by the product of σ and the square root of the element on the principal diagonal of the covariance matrix A corresponding to the coefficient. This procedure is shown in table V. The reciprocal and direct lattice parameters may be obtained from u , v , w , x , y , z by the calculation form given by Donnay and Donnay (1952).

TABLE VI. Values of the elements of the covariance matrices A' and A'' .

Row.	Column.	Matrix A' number.	Matrix A'' number.
1	1	+0.02347618450	+0.03238200385
1	2		
2	1	-0.00163406513	-0.00066591061
1	3		
3	1	-0.00482351590	-0.00650073022
1	4		
4	1	-0.00253461419	-0.00141082551
1	5		
5	1	+0.00341049343	+0.00407451374
1	6		
6	1	-0.00250871511	-0.01405724085
2	2	+0.00057733243	+0.00055631682
2	3		
3	2	+0.00031275979	+0.00005422679
2	4		
4	2	-0.00014515327	-0.00032088990
2	5		
5	2	-0.00002003478	-0.00017839258
2	6		
6	2	+0.00009413930	+0.00017044361
3	3	+0.00551545320	+0.00559225729
3	4		
4	3	-0.00324697742	-0.00326925275
3	5		
5	3	+0.00440162048	+0.00422479977
3	6		
6	3	-0.00135923980	+0.00125130378
4	4	+0.01305835264	+0.01522927428
4	5		
5	4	-0.00636993768	-0.00514882007
4	6		
6	4	+0.00437749891	-0.00035348307
5	5	+0.01542991344	+0.01649704278
5	6		
6	5	-0.00407372798	-0.00679057469
6	6	+0.00701710912	+0.02219432485

The most laborious part of the procedure is the calculation of the covariance matrix A from the normal equations. Considerable labour can be saved if the same set of reflections (hkl) are used for more than one powder pattern, since the same covariance matrix A can be used. Even though many reflections are not resolved over the whole composition range, it was possible to find two sets of reflections which between them covered the whole composition range and which contained sufficient

reflections for the attainment of a suitable precision. The first set has already been listed above and in table V, and its covariance matrix, A' , is listed in table VI. The second set is $20\bar{1}$, $1\bar{1}1$, 130 , $11\bar{2}$, $\bar{1}12$, 040 , 002 , 220 , $1\bar{3}1$, 131 , $\bar{1}32$, 060 , 222 , 113 , $20\bar{4}$. The covariance matrix A'' for this set of reflections is also listed in table VI.

I am indebted to Mr. J. M. Cameron for his assistance in devising this method and to the Applied Mathematics Division of the National Bureau of Standards for the calculation of the two inverse matrices.

In addition to the error caused by the random experimental error there are two other possible sources. The first arises from the calibration obtained from the quartz internal standard. The uncertainty in the measurements of the quartz reflections is about 0.01° in 2θ and results in a corresponding uncertainty in the measurements of the plagioclase reflections. This possible error has little effect on the values for the angles α^* , β^* , and γ^* , for the coefficients connecting these functions with the values of Q_{obs} are both positive and negative and tend to cancel the errors. For the reciprocal lengths a^* , b^* , and c^* , however, the coefficients are all positive, and an error in calibration gives rise to an error in the reciprocal lengths. The possible error of 0.01° in 2θ corresponds to a possible error of about $1/4000$ in a^* , b^* , and c^* . The other source of error arises from displacement of the centre of a peak by interference from adjacent reflections. This effect is difficult to evaluate but is largely included in the estimate of the random experimental error, for any interference from other reflections will tend to increase the differences between the observed and calculated values of Q .

The random errors for unheated specimen 152 correspond to standard deviations of approximately $1/1000$ in a^* , $1/8000$ in b^* , $1/10\ 000$ in c^* , and 0.01 – 0.02° in α^* , β^* , and γ^* . Similar values were found for the other natural plagioclases.¹ The accuracy of the parameters for the synthetic plagioclases is somewhat poorer.

Table III contains the 2θ values of the reflections used in the calculations, and table IV the lattice parameters obtained from the calculations and from the literature.

The significance of the lattice parameters.

The relations between the lattice parameters and the chemical composition are displayed graphically in figs. 1 and 2. The only serious

¹ For one specimen, unheated BM 1940,27, the 130 and $1\bar{3}0$ reflections were unresolved. In order to use the set of reflections A, estimated values of Q were inserted in place of the two unmeasurable values of Q . From the resulting functions of the lattice parameters revised values of Q were calculated and new estimates of the lattice parameters were obtained by insertion of the new values of Q into the calculation procedure.

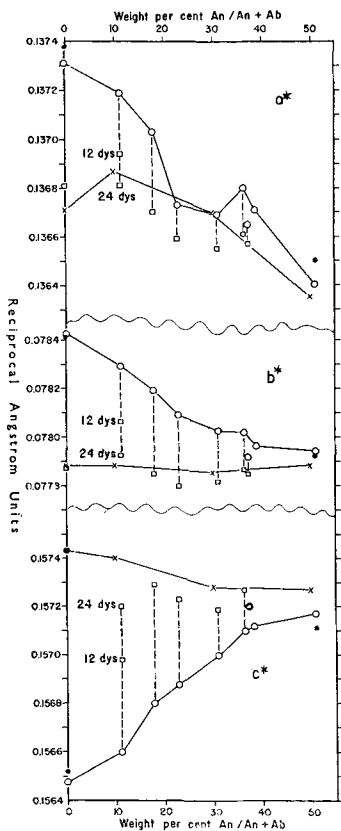


FIG. 1.

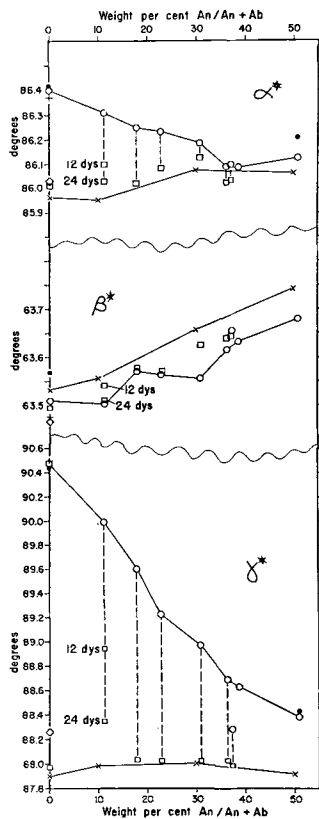


FIG. 2.

FIG. 1. The values of the reciprocal lattice parameters a^* , b^* , and c^* plotted against chemical composition. The ordinate is expressed in reciprocal Ångströms, the abscissa in weight % of the anorthite component. Notice the changes of scale for the ordinates. \circ natural, present work; \square heated natural, present work; \times synthetic, present work; \bullet natural, Cole *et al.*; \diamond heated natural, Laves. The captions 12 dys and 24 dys denote specimens heated for 12 and 24 days at 1060° C. The data are given in table IV.

FIG. 2. The values of the reciprocal lattice parameters α^* , β^* , and γ^* plotted against anorthite content. Notice the changes of scale for the ordinates. \circ natural, present work; \square heated natural, present work; \times synthetic, present work; \bullet natural, Cole *et al.*; \diamond heated natural, Laves. The data are given in table IV.

discrepancy between the values obtained by other workers and those in the present work is the value of γ^* for heated Amelia albite listed by Laves (1952); this specimen was probably only partly converted into

the high-temperature state. Otherwise the measurements obtained in this work agree within the possible errors with those obtained by other workers from single-crystal patterns.

Eight out of the nine natural plagioclases occur in low-temperature environments; that is, environments in which the plagioclase should have had ample opportunity to attain the form stable at low temperatures. The remaining one is from a dacite. In conformity with this division the lattice parameters of the eight low-temperature plagioclases lie, within experimental error, on continuous curves, while the parameters of the volcanic specimen are considerably displaced.

During strong heating followed by quenching, the lattice parameters of the natural plagioclases slowly changed. For six of the specimens the final state (or a good approximation to it) was established, and the resulting lattice parameters lie fairly closely on another set of continuous curves. One of the specimens, BM 1940,27, did not reach equilibrium but would probably have done so if further heating had been applied. The lattice parameters of the unheated volcanic specimen lie approximately midway between the two sets of curves, thus indicating a transitional thermal state for this specimen. Gay (1955) also finds evidence for a transitional state from the subsidiary reflections on single-crystal photographs. The existence of this transitional state suggests that the attainment of equilibrium is very slow; and in conformity with this, all synthetic soda-rich plagioclases so far produced are in the high-temperature state.¹

The lattice parameters of the synthetic plagioclases used in this study lie on a set of curves that are continuous within experimental error. One of the specimens was produced hydrothermally (An_0), the others (An_{10} , An_{30} , and An_{50}) were grown in the dry state.² Although the curves lie close to the data for the heated natural material there are deviations that are greater than the experimental error. It is thought that these deviations are caused by the solid solution of potash feldspar in the natural plagioclases. The chemical analyses of those natural specimens that were converted into the high-temperature state reveal the following amounts of potash-feldspar in solid solution: Amelia, 1.8 %; No. 81822,

¹ MacKenzie and Smith, however (Nov. 1955 meeting of the Geological Society of America), have recently produced synthetic albite with an X-ray powder pattern intermediate between high- and low-albite.

² A specimen of composition An_{40} gave a powder pattern that contained the lines of a plagioclase together with a line in the position of the strongest line of hexagonal anorthite (Davis and Tuttle, 1952). In view of the uncertainty in the composition of the plagioclase this specimen was disregarded.

1.3 %; No. 103086, 3.5 %; No. 97490, 3.6 %; No. 152(6), 1.5 %; and No. 64(7), 3.2 %. The lattice parameters of the three specimens with less than 2 % KAlSi_3O_8 lie closer to the values for the synthetic plagioclases than do the parameters for the other three specimens. Moreover, the deviations lie, on the average, in the same direction as the changes produced in the lattice parameters of the high-temperature alkali-feldspars by substitution of potash (Donnay and Donnay, 1952). The magnitudes of the deviations are only one-half to one-third of the changes produced in high-albite by an equivalent amount of potash, which suggests that, if the deviations are indeed caused by potash, the effect of potash content decreases with increasing lime content.

It is probable, therefore, that structurally there is only one series¹ of high-temperature soda-rich plagioclases. By slow cooling or growth at low temperatures a series of low-temperature soda-rich plagioclases is produced. Specimens may exist in all stages between these two extremes.

There is no measurable discontinuity in the lattice parameters of the high-temperature material, thus conforming with the evidence obtained by Bowen (1913) from melting phenomena, by Gay (1955) from single-crystal phenomena, and by Goodyear and Duffin (1954) from powder X-ray data, that complete solid-solution occurs just below the solidus in soda-rich plagioclases.

The lattice parameters of the high-temperature soda-rich plagioclases are almost independent of composition; the substitution of 50 % An results in the following changes: a^* 1/400, b^* zero, c^* 1/100, α^* 0.1°, β^* 0.2°, γ^* zero. These changes are so small that there is no reliable routine method for determining the composition of high-temperature soda-rich plagioclases from the angular relations displayed in X-ray photographs. The largest change, that in β^* , is only ten times the experimental error of 0.02°, and, moreover, the substitution of small amounts of potash feldspar in high-albite (Donnay and Donnay, 1952) produces changes comparable with those produced by much larger amounts of anorthite.

In contrast to the high-temperature plagioclases the natural low-temperature plagioclases show large changes in the lattice parameters. From An_0 to An_{50} the changes are as follows: a^* -1/150, b^* -1/150, c^* +1/200, α^* -0.3°, β^* +0.2°, γ^* -1.8°. The largest change is in γ^* , and this is the basis of all the methods proposed for the determination of composition of soda-rich plagioclases from powder records. It is

¹ Neglecting the very small differences between X-ray properties of synthetic albite produced hydrothermally and in the dry way (MacKenzie, 1952).

interesting to note that all the high-temperature plagioclases, together with those low-temperature plagioclases which are more calcic than An_{10} , have γ^* acute, whereas the low-temperature plagioclases from An_0 to An_{10} have an obtuse value for γ^* .

There is no evidence for a discontinuity or sharp change in gradient in the variation of lattice parameters with composition for the natural low-temperature specimens. Examination of the graphs for the more sensitive parameters γ^* , b^* , and c^* shows that the gradient of the curves becomes gradually less steep as the anorthite content increases. The curves tend to become asymptotic to the very flat curves for the high-temperature specimens. (The zigzag in a^* could be caused either by the comparatively large potash content of specimens 103086 and 97490 or by the experimental error of 1/1000.) Goodyear and Duffin (1954) report that the A-B, C-D and C-E curves for the natural materials each show prominent discontinuities reasonably close to the structural discontinuities at An_{30} , Ab_{70} and An_{70} , Ab_{30} proposed by Cole *et al.* (1951). These are significant as each of these reflections (A, B, C, D, and E) probably has the same, or similar, indices over the whole composition range.

The present observations are not in harmony with the findings of Goodyear and Duffin.

The determination of soda-rich plagioclases by X-ray powder photography.

One of the important developments of the past decade has been the increasing use of indirect physical methods in the determination of chemical composition, thus by-passing the more tedious methods of analytical chemistry. In some mineral groups (e.g. alkali feldspars and nephelines) it has been shown that the ratio of the two dominant oxides (such as Na_2O and K_2O) can be determined by X-ray powder methods in a few minutes as a matter of routine. Similar methods for determining the composition and thermal state of plagioclases would be extremely useful.

Unfortunately the present work shows that there is no routine X-ray powder method available for the accurate determination of both the composition and the thermal state of soda-rich plagioclases. Five of the lattice parameters for the intermediate states given by the heated specimen BM 1940,27, composition An_{11} , are identical within experimental error with the parameters given by a low-temperature specimen of higher anorthite content. The specimen heated for 12 days has a^* , b^* , c^* , α^* , and γ^* indistinguishable from those given by a low-temperature plagioclase of composition An_{30} . The same five parameters for

BM 1940,27, heated 24 days, and for the specimen from a dacite, composition An_{37} , are identical within experimental error and correspond to a low-temperature plagioclase of composition greater than An_{50} (extrapolated from the curves). The remaining parameter, β^* , is different: the values of β^* for the plagioclase from the dacite and for BM 1940,27, heated 24 days, differ by 0.14° . Thus only β^* is sufficiently different to provide a distinction, and even here the difference is so small that the composition could not be determined with an accuracy higher than 5–10 % An: the determination of the thermal state would also be crude. In routine work the best that can be obtained from powder patterns alone is an upper limit to the anorthite content. Thus if γ^* was found to be 90.0° , then the anorthite content must be less than 11 %.

However, by combining powder X-ray data with petrological, optical, and chemical data much valuable information can readily be obtained. Of the two variables, chemical composition and thermal state, one can be determined by X-ray powder methods if the other is known. If the CaO/Na_2O ratio is known, the simplest way of determining the thermal state is by powder X-ray data. The other two methods for determining the thermal state—single-crystal X-ray or optical determinations—are much more tedious and not so suitable for routine application. The CaO/Na_2O ratio can either be determined by the standard chemical or physico-chemical methods or approximated by the measurement of refractive indices. If the plagioclase can be adjudged to be in the low-temperature state the CaO/Na_2O ratio can readily be determined from X-ray powder patterns. Specimens that are taken from large plutonic masses or that have been regionally metamorphosed should be in the low-temperature state. The thermal state of specimens taken from volcanic, hypabyssal, and small plutonic masses must be regarded as uncertain. Specimens that show schiller in the hand-specimen are probably in the low-temperature state. Unmixed albite-oligoclases may reveal their low-temperature state in well-resolved powder records so long as the proportions of the two phases are roughly equal. The presence of peristeritic unmixing of albite-oligoclases is readily seen in suitable single-crystal X-ray photographs, and so are the subsidiary layer-lines of intermediate specimens, but single-crystal methods are not so suitable for routine work, for plagioclases exhibit complex pseudo-symmetry and are anorthic.

The choice of X-ray reflections for the two determinative methods is complicated by the overlapping of otherwise suitable reflections, which is especially serious for $(hk0)$ and $(h\bar{k}0)$ reflections, which cross over as γ^*

passes through 90° , and by the presence of two separate phases in low-temperature albite-oligoclases. The latter imposes a contradictory condition on the choice of reflections. In order to obtain the highest accuracy one normally chooses the reflections that move the most; however, if the reflections for the two phases in the peristerite move a long way they may become partly resolved and produce a false measurement. For if the peaks are partly resolved and one phase is more abundant than the other it is likely that only the strong peak would be measured. For the highest accuracy in the peristerite region it is necessary to choose reflections which move about half the width of the peaks and to measure the centre of gravity of the peaks. Two sets of adjacent peaks satisfy this condition: (111) and $(\bar{1}\bar{1}\bar{1})$, which occur at $2\theta_{\text{Cu}} = 23.6 \pm 0.1^\circ$ and $23.0 \pm 0.1^\circ$, respectively; and $(\bar{1}\bar{3}2)$ and (131), which occur at $2\theta_{\text{Cu}} = 33.85 \pm 0.1$ and $31.4 \pm 0.15^\circ$, respectively. In the remaining composition range, the two most suitable reflections appear to be the ones used by Tuttle and Bowen—the (131) and $(\bar{1}\bar{3}\bar{1})$, which occur at $2\theta_{\text{Cu}} = 31.4 \pm 0.15^\circ$ and $29.9 \pm 0.25^\circ$. This pair of reflections is not suitable in the peristerite region, for the $(\bar{1}\bar{3}\bar{1})$ reflection is often unresolved from adjacent reflections and, moreover, moves too far. In the remainder of the plagioclase series both reflections are fully resolved. The $(2\bar{2}0)$, $(\bar{1}\bar{3}2)$, $(24\bar{1})$, and $(\bar{2}41)$ reflections have been used, first by Claisse and then by Goodyear and Duffin, but they are not suitable in the peristerite region, for they move too fast with composition and the $(2\bar{2}0)$ and (220) reflections cross over. Nor are the latter three reflections suitable in the region above An_{70} , for the $(24\bar{1})$ and $(\bar{2}41)$ reflections cross over. In the remaining region, An_{20} – An_{70} , they are very suitable for low-temperature specimens, but are not so suitable for high-temperature specimens, again because of the poor resolution between the $(24\bar{1})$ and $(\bar{2}41)$ reflections.

The following procedures are recommended for the two determinations:

Determination of the thermal state.

If the anorthite content lies between An_{20} and An_{40} , measure the angular separation 2θ (131)– 2θ ($\bar{1}\bar{3}\bar{1}$) and determine the thermal state from fig. 3, which is a graph of 2θ (131)– 2θ ($\bar{1}\bar{3}\bar{1}$) against composition for all the specimens used for the determination of lattice parameters together with some extra ones. If the anorthite content lies between An_0 and An_{20} , measure the separation 2θ ($\bar{1}\bar{3}2$)– 2θ (131) and compare with fig. 4, which shows the variation of 2θ ($\bar{1}\bar{3}2$)– 2θ (131) with chemical composition and thermal state. If the thermal state is found to be low-

temperature, measure $2\theta(111) - 2\theta(\bar{1}\bar{1}1)$ as a check and compare with fig. 5, which shows the variation of $2\theta(111) - 2\theta(\bar{1}\bar{1}1)$ with chemical composition for low-temperature albites and oligoclases. If the thermal state is found to be high-temperature or intermediate, measure $2\theta(131) - 2\theta(\bar{1}\bar{3}1)$ as a check and compare with fig. 3.

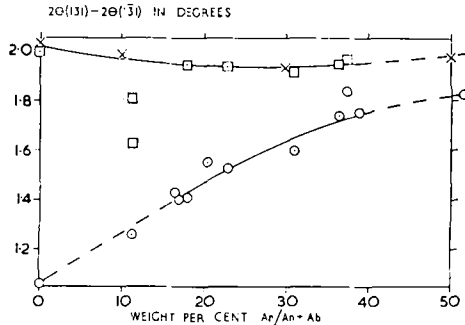


FIG. 3. The values of $2\theta(131) - 2\theta(\bar{1}\bar{3}1)$ plotted against chemical composition. \circ natural; \square heated natural; \times synthetic. The data are listed in table VII.

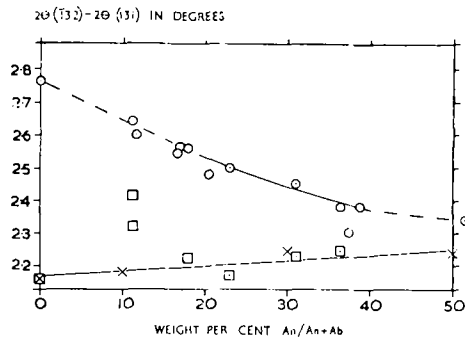


FIG. 4. The values of $2\theta(\bar{1}\bar{3}2) - 2\theta(131)$ plotted against chemical composition. \circ natural; \square heated natural; \times synthetic. The data are listed in table VII.

Determination of the composition of low-temperature specimens.

If the thermal state is adjudged to be low-temperature, measure $2\theta(131) - 2\theta(\bar{1}\bar{3}1)$ and compare with fig. 3. If the measurement indicates a composition lying between An_0 and An_{20} , measure either or both of

$2\theta (\bar{1}32) - 2\theta (131)$ and $2\theta (111) - 2\theta (1\bar{1}1)$ and compare with figs. 4 and 5. The resultant composition should be more reliable than the composition deduced from $2\theta (131) - 2\theta (1\bar{3}1)$.

The data for the construction of figs. 3, 4, and 5 are given in table VII.

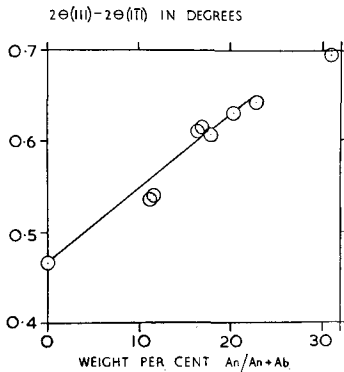


FIG. 5. The values of $2\theta (111) - 2\theta (1\bar{1}\bar{1})$ plotted against chemical composition for natural low-temperature specimens. The data are listed in table VII.

(131) and ($\bar{1}32$). Assuming optimum conditions, it is likely that the precision of measurement can reach 0.005 to 0.02° in 2θ for the expenditure of 10 to 20 minutes running-time of a diffractometer (Smith and Sahama, 1954). In photographic powder cameras the precision is likely to be lower. In addition to this error, the presence of potash or other oxides in the plagioclase may lead to uncertainty in the interpretation of the measurement. It seems likely that in the region $An_0 - An_{30}$ the composition of low-temperature specimens can be determined with an accuracy of 2% An and that a clear differentiation of the thermal states can be obtained.

In conclusion, there are a few minor points concerning the work of Goodyear and Duffin. The *F* and *G* reflections of Goodyear and Duffin have the following indices: *G*, (204); *F*, probably mainly (04 $\bar{3}$), but includes other reflections as well. Diffractometer records of reflection *F* often show a complex structure and it is doubtful whether it is suitable for measurement. In fig. 2 and table IV of Goodyear and Duffin's paper the reflection (131) is missing, although it is one of the strongest reflections near $2\theta = 31^\circ$. Probably it was masked by a rock-salt reflection.

The accuracy of determination of composition or thermal state is rather difficult to estimate. Unfortunately the plagioclase powder pattern is so complex that the peaks tend to be rather weak, the only prominent one being the composite peak at $2\theta_{Cu} = 28^\circ$ composed largely of the (002) and (040) reflections. It is advisable, therefore, to use only samples in which the plagioclase is the major component. The (131) and ($\bar{1}31$) reflections are among the strongest ones in the region around $2\theta = 30^\circ$. The ($\bar{1}32$) is rather weaker, and it may often be found preferable to use the pair (111) and ($1\bar{1}\bar{1}$) in preference to

TABLE VII. Data for figs. 3, 4, and 5. All values are for the estimated centre of gravity of the $\alpha_1 + \alpha_2$ doublet. An/(An + Ab) is given in weight %. The values of 2θ are in degrees, and the accuracy of the measurements lies between 0.005 and 0.02°.

Specimen	An (An + Ab)	$2\theta_{(111)} -$	$2\theta_{(131)} -$	$2\theta_{(\bar{1}32)} -$
		$2\theta_{(\bar{1}\bar{1}1)}$	$2\theta_{(\bar{1}\bar{3}1)}$	$2\theta_{(131)}$
Amelia unheated	0.0	0.467	1.062	2.766
Amelia heated	0.0	—	1.990	2.16
BM 1940,27 unheated	11.2	0.537	1.255	2.643
BM 1940,27; 12 days 1060° C.	11.2	—	1.625	2.415
BM 1940,27; 24 days 1060° C.	11.2	—	1.805	2.320
Emmons 10(2) unheated	11.6	0.540	not	2.630
			measurable	
Emmons 73(3) unheated	16.6	0.610	1.425	2.575
Emmons 156(4) unheated	16.9	0.615	1.390	2.595
81822 unheated	17.9	0.605	1.405	2.560
81822 heated	17.9	—	1.940	2.225
Emmons 94(5) unheated	20.4	0.630	1.550	2.510
103086 unheated	22.9	0.642	1.525	2.500
103086 heated	22.9	—	1.930	2.170
97490 unheated	31.0	0.694	1.595	2.462
97490 heated	31.0	—	1.912	2.228
Emmons 152(6) unheated	36.4	—	1.735	2.380
Emmons 152(6) heated	36.4	—	1.948	2.245
Emmons 64(7) unheated	37.3	—	1.840	2.300
Emmons 64(7) heated	37.3	—	1.963	2.225
Emmons 144(8) unheated	38.8	—	1.750	2.380
Emmons 24(9) unheated	50.9	—	1.813	2.340
Synthetic	0	—	2.030	2.160
Synthetic	10	—	1.980	2.180
Synthetic	30	—	1.930	2.245
Synthetic	50	—	1.975	2.240

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