The identification and determination of plagioclase felspars by the X-ray powder method.

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1. Introduction.

THERE appear to be few published data correlating X-ray powder patterns of chemically analysed plagioclase felspars with composition. Tuttle and Bowen (1950) have examined the powder patterns of low-temperature and high-temperature modifications and find that pure albite from Amelia, Virginia, when heated for ten days at 1050° C., gives a powder pattern identical with that of synthetic albite. They have not given, however, complete data over the whole range from An_0Ab_{100} to $An_{100}Ab_0$. Claisse (1950) selected six reflections from the powder patterns of eight plagioclases of composition extending over the complete range, and plotted line separations against composition determined optically, but did not distinguish between high- or low-temperature forms.

More recently, Gay (1953) has examined the single-crystal diffraction patterns of a number of plagioclases in the range $An_{70}Ab_{30}$–$An_{100}Ab_0$, and has correlated the nature of each pattern with the composition and thermal history of the specimen. Gay's work has provided a convenient method for distinguishing between high- and low- temperature forms of anorthite; he has found that pure anorthites which have been heated naturally give the same type of pattern as a synthetic anorthite crystallized at a temperature of the order of 1500° C.; further, when this synthetic anorthite is 'annealed' at 1100° C. for 72 hours the pattern obtained is of the type given by pure anorthites of low-temperature origin.

In the present work are examined powder patterns of chemically analysed plagioclases of presumably low-temperature origin, and of synthetic materials probably having high-temperature structures. The aim of this paper is twofold: (i) to provide X-ray powder data for the identification of plagioclase felspars when admixed with associated

minerals, and (ii) to establish relationships enabling the composition and thermal modification of a felspar to be determined from certain line positions in the X-ray pattern.

2. Description of the plagioclase specimens.

Descriptions of the materials employed in this investigation are given in tables I and II. The specimens were supplied to the authors by Dr. W. H. Taylor, but had been presented to him by the following:

Prof. C. E. Tilley, Department of Mineralogy and Petrology, University of Cambridge.
Prof. R. C. Emmons, University of Wisconsin.
Prof. H. H. Hess, University of Princeton, New Jersey.
Prof. T. G. Sahama, Institute of Geology, Helsinki, Finland.
Prof. H. Kuno, Geological Institute, Tokyo University, Japan.

Table I. Details of natural plagioclases

Specimen no.	Source.	Ref.	Name and locality.	Mode of occurrence.	Composition (wt. %) from Chem. anal. Ab.	An.	Or.	R.I. An.
1.	Emmons	A (31)	Albite, Rutherford mica mine, Amelia, Va.	Pegmatite	98·0	0·4	1·6	0·0
2.	Emmons	A (4)	Oligoclase, Petrick quarry, Llano Co., Texas.	Granite	81·6	16·5	1·9	19·2
3.	Sahama	B (9)	Oligoclase, Hawk mica mine, N.C.	Crystals from pegmatite	74·4	22·1	3·5	20·8
4.	Emmons	A (28)	Andesine, Corundum Hill mine, Macon Co., N.C.	See ref. A (28)	68·2	31·1	0·7	27·4
5.	Emmons	A (8)	Andesine, Crestmore, Calif.	Granodiorite	60·2	38·0	2·0	34·8
6.	Emmons	A (10)	Labradorite, Shelby, N.C.	Hornblende-gabbro	47·3	51·6	1·1	46·5
7.	Emmons	A (17)	Labradorite, Grand Marais, Minnesota.	Anorthosite	32·7	64·5	2·8	62·0
8.	Emmons	A (18)	Labradorite, Chester Co., Pennsylvania.	Diabase	31·6	66·3	2·1	—
9.	Emmons	—	Bytownite, Crystal Bay, Minnesota.	Anorthosite	28·7	71·3	0·0	—
10.	Hess	B (4)	Bytownite, Rustenburg mines, Transvaal.	Norite	22·0	77·0	1·0	73·6
11.	Hess	B (3)	Bytownite, Stillwater, Montana.	Anorthosite	18·6	81·0	0·4	78·1
12.	Emmons	A (27)	Bytownite, Merrill, Wis.	Troctolite	16·7	81·5	1·8	77·9
13.	Hess	B (2)	Bytownite, Stillwater, Montana.	Anorthosite	13·2	86·5	0·3	85·0
14.	Tilley	B (1)	Anorthite, Grass Valley, California.	Olivine-norite	6·7	93·0	0·3	93·6
15.	Kuno	—	Anorthite, Hakoné volcano, Japan.	Allivalite, ejected block in volcanic tuff	1·4	98·3	0·3	—
16.	Tilley	—	Anorthite, Pasmeda Alp, Fassa Valley, Tirol.	Veins in limestone block	—	—	—	~100

References: A, Emmons (1953); B, Kracek and Neuvonen (1952).

TABLE II. Details of synthetic plagioclases.*

Specimen no.	Crystallization temperature.	Composition, wt. % An.
17	1090° C.	5
18	1090	10
19	1140	30
20	1160	40
21	1265	60
22	1310	70
23	1350	80
24	1385	90
25	1400	100
26	1520	100

* All these materials came from Dr. J. F. Schairer originally, but were obtained (along with specimens of natural materials) from the collections in the Department of Mineralogy and Petrology and in the Crystallographic Laboratory of the University of Cambridge.

Specimens 17 to 26 were synthesized by Dr. J. F. Schairer, Geophysical Laboratory, Washington, D.C., and obtained by Prof. Tilley.

The compositions have been determined from the chemical analyses on the basis of relative amounts of Na_2O, CaO, and K_2O, without any attempt to eliminate excess silica; compositions have also been calculated from refractive indices using the equations of Chayes (1952). Full chemical analyses and values of refractive indices appear in the references appended to table I. In the case of Emmons's specimens the composition obtained from refractive index measurements represents an average from determinations on three to six grains, the composition of the grains for any specimen differing by as much as 8 % anorthite. Since in no case is the orthoclase content excessive, a consideration of its effect on the X-ray data has been ignored.

All the synthetic materials with the exception of specimen 26 (100 % anorthite) crystallized in a finely divided form, sufficient to give continuous X-ray powder reflections. Specimen 26, however, was prepared at a later date and has been examined by Gay (1953) using a single-crystal technique.

Specimen 15 is of volcanic origin and is likely to be a high-temperature material, whilst the remaining natural specimens are probably low-temperature forms. The complete powder pattern of the former is not considered in this paper.

3. THE X-RAY DATA.

It is well known that the composition of a felspar can vary considerably throughout a particular specimen; this is well illustrated by the

spread of values that can occur when an attempt is made to determine the composition of the material by studying the optics of several grains. If X-ray diffraction patterns are to be linked with bulk chemical analyses, the specimen to be X-rayed should contain a representative selection of material, a condition that can be fulfilled by the powder method.

X-ray diffraction photographs were taken with filtered Co-$K\alpha$ radiation, using a 20 cm.-diameter semi-focusing camera of the type employed by Brindley and his co-workers (1946). In the experimental arrangement the powder is packed, without adhesive, in a shallow cavity ($4 \times 3 \times 0{\cdot}5$ mm.) in a flat glass plate. By varying the angle between the flat specimen surface and the incident beam, focusing in different regions of Bragg angle can be achieved. To eliminate systematic errors in calibration, the powder pattern of rock-salt was recorded on each felspar photograph; this method proved quite satisfactory as only a few calibration lines were added to the complicated felspar patterns. With this technique the position of a good quality reflection was reproducible to within 0·01° in θ. A few photographs have also been taken using an evacuable 19 cm. camera of the Bradley type. Although these had the advantage of low background, nevertheless the resolution was not sufficiently good for reflections occurring close together.

In tables III and IV are listed the powder patterns of natural and synthetic plagioclases. Reflections of spacing greater than about 2·5 kX have been measured and relative intensities estimated visually. For identification purposes, the patterns become too complex below 2·5 kX, but there is a useful characteristic group of three reflections in the range 1·76–1·84 kX. The latter are described as 4, 5, and 6 by Claisse (1950), and will be described in section 5.

Lattice spacings have been calculated for natural materials of compositions 0, 55, and 100 % anorthite using the parameters given by Cole, Sörum, and Taylor (1951), and for synthetic albite using data given by Donnay and Donnay (1952). It seems that, in general, all the observed patterns can be accounted for in terms of reflections for which $(h+k)$ is even, the indices referring in all cases to a c-axis $\sim$7 kX. These constitute the (a)-type reflections described by Gay (1953), and it is probable that Gay's (b)-, (c)-, and (d)-type reflections are not observed with the present technique (but see section 4e).

Certain reflections have been omitted from the pattern of specimen 2 since they were identified as quartz lines, and indeed the chemical analysis of this material indicated the presence of about 19 % free

TABLE III. X-ray powder patterns of

Albite 0 % An. (calc.)		1 Albite 0·4 % An.			2 Oligoclase 16·5 % An.		4 Andesine 31·1 % An.		5 Andesine 38·0 % An.		6 Labradorite 51·6 % An.		Labradorite 55 % An. (calc.)	
hkl.	*d* (kX).	*d* (kX).	Int.		*d* (kX).	Int.	*d* (kX).	Int.	*d* (kX).	Int.	*d* (kX).	Int.	*hkl.*	*d* (kX).
—	—	—	—		—	—	—	—	—	—	6·43	w	$1\bar{1}0$	6·42
001	6·38	6·38	m		6·37	m	6·40	wm	6·40	wm	—	—	—	—
020	6·36	—	—		—	—	—	—	—	—	—	—	020	6·40
—	—	—	—		—	—	—	—	—	—	—	—	001	6·35
$1\bar{1}0$	6·29	—	—		—	—	—	—	—	—	—	—	—	—
$11\bar{1}$	5·89	5·93	w		5·92	f	5·86	f	5·85	f	—	—	$11\bar{1}$	5·82
$\bar{1}11$	5·57	5·60	w		5·61	f	5·63	f	5·65	f	—	—	$\bar{1}11$	5·61
$0\bar{2}1$	4·65	—	—		—	—	4·68	f	4·67	f	4·67	f	$0\bar{2}1$	4·67
$20\bar{1}$	4·018	4·016	s		4·018	s	4·024	s	4·028	s	4·026	s	$20\bar{1}$	4·028
$1\bar{1}1$	3·847	3·843	wm		3·851	wm	3·863	wm	3·870	wm	3·878	wm	$1\bar{1}1$	3·881
—	—	—	—		—	—	—	—	—	—	—	—	—	—
111	3·771	3·767	ms		3·754	ms	3·745	ms	3·751	ms	3·749	ms	111	3·758
—	—	—	—		3·678	m	3·708	ms	3·715	m	3·723	ms	$1\bar{3}0$	3·729
130	3·676	—	—		—	—	—	—	—	—	—	—	130	3·645
131	3·658	3·660	s b		3·651	m	3·634	ms	3·640	ms	3·633	ms	$13\bar{1}$	3·624
$1\bar{3}0$	3·651	—	—		—	—	—	—	—	—	—	—	—	—
$11\bar{2}$	3·496	3·495	m		3·481	wm	3·469	wm	3·468	wm	3·464	wm	$11\bar{2}$	3·463
$22\bar{1}$	3·473	—	—		—	—	3·421	vw	3·430	vw	3·424	vw	$22\bar{1}$	3·431
$\bar{1}12$	3·371	3·364	wm		3·355	wm	3·356	m	3·359	m	3·352	m	$\bar{1}12$	3·354
—	—	—	—		—	—	—	—	—	—	—	—	—	—
—	—	—	—		—	—	—	—	—	—	—	—	$2\bar{2}0$	3·212
$20\bar{2}$	3·206	3·206	s		3·198	s	—	—	—	—	—	—	—	—
002	3·188	—	—		—	—	—	—	—	—	—	—	—	—
040	3·182	3·179	vvs		3·176	vvs	3·195	vvs	3·199	vvs	3·197	vvs	040	3·201
—	—	—	—		—	—	—	—	—	—	—	—	$20\bar{2}$	3·192
—	—	—	—		—	—	3·166	s	3·169	vs	3·165	vs	002	3·173
220	3·165	—	—		—	—	3·132	ms	3·134	ms	3·131	ms	220	3·140
$2\bar{2}0$	3·144	3·141	wm		3·142	wm	—	—	—	—	—	—	—	—
$13\bar{1}$	2·959	2·952	ms		2·975	m	2·993	m	2·999	m	3·003	m	$13\bar{1}$	3·005
$22\bar{2}$	2·947	—	—		—	—	—	—	—	—	—	—	—	—
$0\bar{2}2$	2·924	—	—		—	—	—	—	—	—	—	—	—	—
$04\bar{1}$	2·921	2·919	m		2·927	ms	2·929	ms b	2·928	ms b	2·933	ms	$04\bar{1}$	2·939
—	—	—	—		—	—	—	—	—	—	2·916	wm	$0\bar{2}2$	2·923
—	—	—	—		—	—	—	—	—	—	—	—	$22\bar{2}$	2·909
131	2·858	2·856	m		2·837	m	2·829	m	2·833	m	2·834	m	131	2·839
—	—	—	—		—	—	—	—	—	—	—	—	$\bar{3}11$	2·653
$31\bar{1}$	2·658	—	—		—	—	—	—	—	—	—	—	$31\bar{1}$	2·652
$\bar{1}32$	2·633	2·638	wm		2·642	wm	2·646	wm	2·647	wm	2·648	m	$\bar{1}32$	2·643
—	—	—	—		—	—	—	—	—	—	—	—	—	—
—	—	—	—		—	—	—	—	—	—	—	—	$22\bar{1}$	2·529
$\bar{3}11$	2·620	—	—		—	—	—	—	—	—	—	—	—	—
—	—	—	—		—	—	—	—	—	—	—	—	—	—
—	—	—	—		—	—	—	—	—	—	—	—	—	—
$24\bar{1}$	2·555	2·561	m		2·545	m	2·529	m	2·528	ms	2·525	ms	$24\bar{1}$	2·523
$31\bar{2}$	2·522	—	—		—	—	—	—	—	—	—	—	$31\bar{2}$	2·515
$1\bar{1}2$	2·508	2·509	vw		2·511	vw	2·515	vw	—	—	—	—	$1\bar{1}2$	2·515
$22\bar{1}$	2·491	—	—		—	—	—	—	—	—	—	—	—	—
$\bar{3}12$	2·474	—	—		—	—	—	—	—	—	—	—	$\bar{3}12$	2·489
—	—	—	—		—	—	—	—	—	—	—	—	—	—
$\bar{2}41$	2·443	2·443	wm		2·465	wm	2·487	wm	2·489	m	2·494	m	$\bar{2}41$	2·489

Scale of intensities: vvs very very strong (10), s strong (8), m medium (6), or weak (4), f faint (2); b broad line or two unresolved lines, ? exceptionally spotty, * masked by calibration reflection of rock-salt.

some natural plagioclase felspars.

7 Labradorite 64·5 % An.		9 Bytownite 71·3 % An.		10 Bytownite 77·0 % An.		12 Bytownite 81·5 % An.		14 Anorthite 93·0 % An.		Anorthite 100 % An. (calc.).	
d (kX).	Int.	*d* (kX).	Int.	*d* (kX).	Int.	*d* (kX).	Int.	*d* (kX).	Int.	*hkl.*	*d* (kX).
6·47	w	6·50	w	6·51	w	6·52	w	6·55	w	$1\bar{1}0$	6·51
—	—	—	—	—	—	—	—	—	—	—	—
—	—	—	—	—	—	—	—	—	—	020	6·41
—	—	—	—	—	—	—	—	—	—	001	6·35
—	—	—	—	—	—	—	—	—	—	—	—
—	—	—	—	—	—	—	—	—	—	$11\bar{1}$	5·77
—	—	—	—	—	—	—	—	—	—	$\bar{1}11$	5·64
4·67	w	4·69	wm	4·68	wm	4·68	wm	4·69	m	$02\bar{1}$	4·67
4·036	s	4·025	s	4·024	s	4·021	s	4·033	ms	$20\bar{1}$	4·030
3·885	wm	3·887	wm	3·880	wm	3·885	wm	3·911	wm	$1\bar{1}1$	3·908
—	—	—	—	—	—	—	—	3·770	ms	$1\bar{3}0$	3·775
3·747	s	3·745	s	3·740	s	3·743	s	3·740	wm	111	3·749
—	—	—	—	—	—	—	—	—	—	—	—
—	—	—	—	—	—	—	—	—	—	130	3·613
3·631	ms	3·615	ms	3·617	ms	3·609	ms	3·611	ms	$13\bar{1}$	3·596
—	—	—	—	—	—	—	—	—	—	—	—
3·463	wm	3·454	wm	3·452	wm	3·456	wm	3·452	wm	$11\bar{2}$	3·452
3·423	vw	3·401	w	3·404	w	3·404	vw	3·390	vw	$22\bar{1}$	3·396
3·359	m	3·346	m	3·350	m	3·347	m	3·354	m	$\bar{1}12$	3·356
3·254	wm?	3·245	wm?	3·250	wm?	—	—	—	—	—	—
3·222	s	3·222	m	3·222	wm	3·231	ms	3·249	s	$2\bar{2}0$	3·254
—	—	—	—	—	—	—	—	—	—	—	—
—	—	—	—	—	—	—	—	—	—	—	—
3·198	vvs	3·191	vvs	3·190	vvs	3·191	vvs	3·197	vvs	040	3·204
—	—	—	—	—	—	—	—	—	—	$20\bar{2}$	3·188
3·170	vs	3·161	s	3·159	s	3·160	s	3·164	s	002	3·173
3·131	ms	3·117	ms	3·116	m	3·115	ms	3·114	ms	220	3·116
—	—	—	—	—	—	—	—	—	—	—	—
3·013	wm	3·015	wm	3·011	wm	3·016	m	3·030	m	$13\bar{1}$	3·036
—	—	—	—	—	—	—	—	—	—	—	—
—	—	—	—	—	—	—	—	—	—	—	—
2·940	ms b	2·937	ms b	2·939	ms b	2·933	ms b	2·944	ms	$04\bar{1}$	2·946
—	—	—	—	2·916	wm	—	—	2·927	wm	$0\bar{2}2$	2·928
—	—	—	—	—	—	2·887	vw	2·880	wm	$22\bar{2}$	2·887
*	*m*	*	*m*	*	*m*	*	*m*	*	*m*	131	2·822
—	—	—	—	—	—	—	—	—	—	$\bar{3}11$	2·670
—	—	—	—	—	—	—	—	—	—	—	—
2·651	m	2·642	m	2·647	m	2·652	m	2·649	m	$\bar{1}32$	2·650
—	—	—	—	—	—	—	—	—	—	$31\bar{1}$	2·642
—	—	—	—	—	—	—	—	—	—	$2\bar{2}1$	2·554
—	—	—	—	—	—	—	—	—	—	—	—
—	—	—	—	—	—	—	—	—	—	$1\bar{1}2$	2·524
—	—	—	—	—	—	—	—	2·519	ms	$\bar{2}41$	2·520
2·522	ms	2·506	s	2·511	ms	2·513	ms	—	—	—	—
—	—	—	—	—	—	—	—	—	—	$31\bar{2}$	2·504
—	—	—	—	—	—	—	—	—	—	—	—
—	—	—	—	—	—	—	—	—	—	—	—
—	—	—	—	—	—	—	—	—	—	$\bar{3}12$	2·499
—	—	—	—	—	—	—	—	2·497	ms	$24\bar{1}$	2·496
2·504	m	—	—	—	—	—	—	—	—	—	—

TABLE IV. X-ray powder patterns of

High-albite (calc.).		17 5 % An.		18 10 % An.		19 30 % An.		20 40 % An.	
hkl.	d (kX).	*d* (kX).	Int.	*d* (kX).	Int.	*d* (kX).	Int.	*d* (kX).	Int.
$1\bar{1}0$	6·44	—	—	—	—	—	—	—	—
020	6·41	6·42	m	6·43	m	6·42	wm	6·47	wm
001	6·35	—	—	—	—	—	—	—	—
$11\bar{1}$	5·83	5·85	w	5·85	w	—	tr	—	—
$\bar{1}11$	5·64	5·64	w	5·66	w	—	tr	—	—
$0\bar{2}1$	4·68	4·69	vw	4·68	vw	4·68	m	4·67	wm
$20\bar{1}$	4·036	4·019	s	4·022	s	4·015	s	4·015	s
$1\bar{1}1$	3·881	3·866	wm	3·872	wm	3·871	wm	3·883	wm
$1\bar{3}0$	3·747	3·732	s	3·735	s	3·734	s	3·739	ms
111	3·740	—	—	—	—	3·673	vw	3·687	w
130	3·636	3·621	ms	3·623	ms	3·620	ms	3·621	m
$13\bar{1}$	3·628	—	—	—	—	—	—	—	—
$\bar{1}31$	3·489	—	—	—	—	—	—	—	—
$11\bar{2}$	3·476	3·457	w	3·463	w	3·458	w	3·458	wm
$22\bar{1}$	3·426	3·411	f	3·420	vw	3·405	vw	3·409	vw
$\bar{2}21$	3·405	—	—	—	—	—	—	—	—
$\bar{1}12$	3·364	3·354	m	3·357	m	3·352	m	3·360	m
$2\bar{2}0$	3·221	—	—	3·249	vw	3·250	vw	3·253	m
$20\bar{2}$	3·207	—	—	—	—	—	—	—	—
040	3·206	3·198	vvs	3·201	vvs	3·196	vvs	3·199	vvs
002	3·174	3·160	s	3·163	s	3·162	s	3·174	vs
220	3·124	3·115	ms	3·119	ms	3·118	ms	3·119	ms
$1\bar{3}1$	3·016	3·003	m	3·006	m	3·007	m	3·012	m
$04\bar{1}$	2·949	2·936	ms	2·941	ms	2·933	ms	2·932	ms b
$0\bar{2}2$	2·930	—	—	—	—	—	—	—	—
$22\bar{2}$	2·919	2·908	m	*	—	2·905	m	*	—
131	2·828	—	—	—	—	—	—	—	—
$\bar{1}32$	2·650	2·646	wm	2·646	wm	2·648	wm	2·648	wm
$31\bar{1}$	2·650	—	—	—	—	—	—	—	—
$2\bar{2}1$	2·532	—	—	—	—	—	—	—	—
$31\bar{2}$	2·523	—	—	—	—	—	—	—	—
$24\bar{1}$	2·521	—	—	—	—	—	—	—	—
$1\bar{1}2$	2·515	2·510	ms	2·508	ms	2·511	ms	2·509	ms
$\bar{2}41$	2·504	—	—	—	—	—	—	—	—

Scale of intensities: vvs very very strong (10), s strong (8), m medium (6), w weak (4), f faint (2), tr trace (not recorded); b broad line or two unresolved lines, ? exceptionally spotty, * masked by calibration reflection of rock-salt.

some synthetic plagioclase felspars

21 60 % An.		22 70 % An.		23 80 % An.		24 90 % An.		25 100 % An.	
d (kX).	Int.	*d* (kX).	Int.	*d* (kX).	Int.	*d* (kX).	Int.	*d* (kX).	Int.
—	—	—	—	—	—	—	—	—	—
6·48	wm	6·53	w	6·51	w	6·54	vw	6·48	vw
—	—	—	—	—	—	—	—	—	—
—	—	—	—	—	—	—	—	—	—
—	—	—	—	—	—	—	—	—	—
4·67	wm	4·69	wm	4·69	wm	4·69	m	4·70	m
4·021	s	4·026	s	4·015	s	4·032	s	4·039	s
3·892	wm	3·892	m	3·888	wm	3·913	wm	3·923	wm
3·746	s	3·747	s	3·743	s	3·763	s	3·774	s
3·678	w	3·672	vw	3·685	vw	3·690	vw	3·698	vw
3·621	ms	3·618	ms	3·605	ms	3·614	s	3·617	s
—	—	—	—	—	—	—	—	—	—
—	—	—	—	—	—	—	—	—	—
3·458	wm	3·454	wm	3·444	wm	3·454	m	3·458	wm
3·405	vw	3·405	w	3·396	w	3·405	w	3·406	w
—	—	—	—	—	—	—	—	—	—
3·350	m	3·352	m	3·343	m	3·354	m	3·360	m
3·250	wm	—	—	3·257	wm ?	3·249	s	3·260	s
3·220	wm	3·230	ms	3·233	ms	—	—	—	—
3·192	vvs	3·194	vvs	3·185	vvs	3·196	vvs	3·204	vvs
3·165	s	3·168	s	3·163	vs	3·177	vs	3·181	vs
3·121	m	3·120	ms	3·113	s	3·120	ms	3·124	ms
3·016	wm	3·020	m	3·018	m	3·033	m	3·044	m
2·935	ms b	2·941	ms	2·934	ms	2·942	s b	2·946	ms b
—	—	2·924	wm	2·917	wm	—	—	—	—
*	—	2·894	w	2·888	w	2·893	vw	2·894	vw
—	—	—	—	—	—	—	—	—	—
2·648	wm	2·644	wm	2·638	wm	2·637	wm	2·645	wm
—	—	—	—	—	—	—	—	—	—
—	—	—	—	—	—	—	—	—	—
—	—	—	—	—	—	—	—	—	—
—	—	—	—	—	—	2·507	ms	2·520	ms
2·509	ms	2·506	ms	2·499	ms	2·491	ms	2·495	ms
—	—	—	—	—	—	—	—	—	—

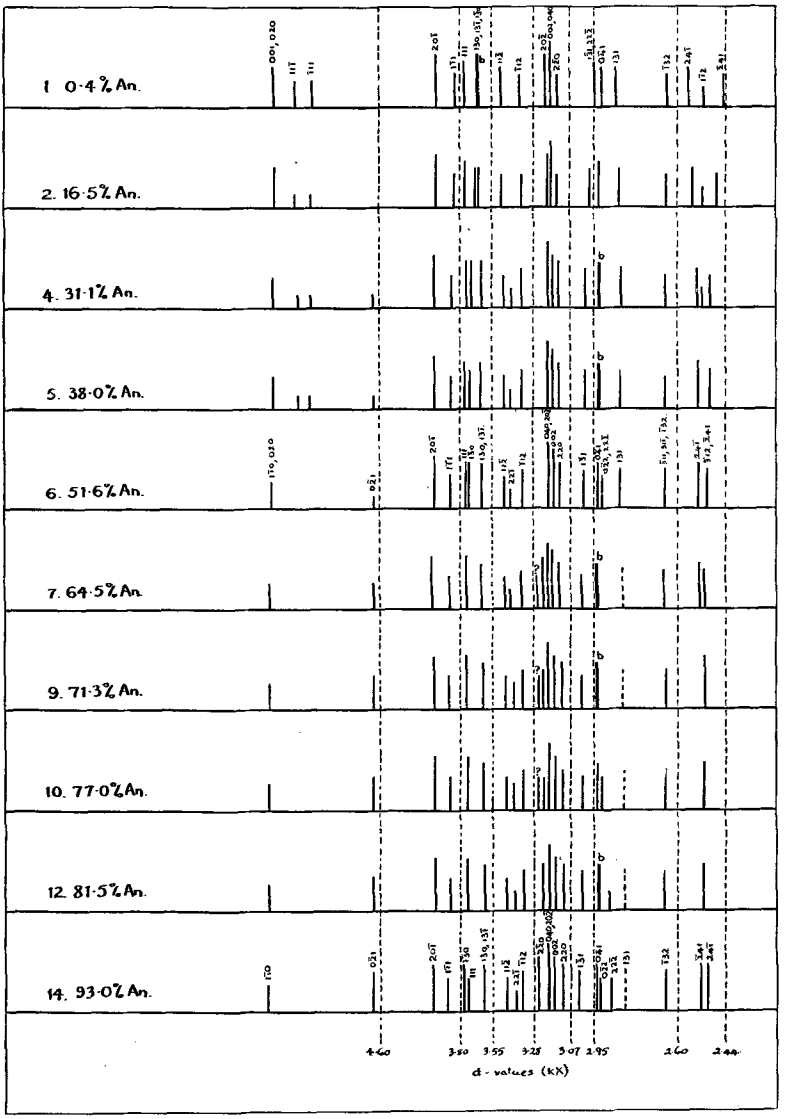

FIG. 1. Powder patterns of some natural plagioclases. Heights of lines represent relative intensities estimated visually. b, broad line or two unresolved lines; ?, exceptionally spotty; – – –, masked by calibration reflection of rock-salt.

FIG. 2. Powder patterns of some synthetic plagioclases. Heights of lines represent relative intensities estimated visually. b, broad line or two unresolved lines; ?, exceptionally spotty; – – –, masked by calibration reflection of rock-salt.

quartz. All other observed lines which have been recorded are interpreted as typical plagioclase reflections. A diagrammatic representation of the patterns is given in figs. 1 and 2.

4. Diagnostic features of the X-ray patterns.

A plagioclase can usually be identified, and at the same time distinguished from an alkali-felspar, by the occurrence of the following characteristics in an X-ray powder pattern:

(i) two very strong reflections of spacing in the range 3·16–3·21 kX;
(ii) a medium reflection of spacing 6·4–6·5 kX;
(iii) three medium to strong reflections with spacings 4·02–4·04, 3·73–3·77, and 3·61–3·66 kX respectively.

An observation of other plagioclase reflections (see data given in tables III and IV) ought to establish its presence conclusively.

A detailed inspection of a plagioclase pattern would reveal several useful features enabling the material to be assigned to an approximate composition range, and in some cases providing information regarding its thermal modification. The most diagnostic portions of the patterns are indicated in figs. 1 and 2, and a description of the reflections occurring in them is given below.

Changes occurring in the character of the patterns are particularly prominent at the albite end of the series. On the basis of the patterns which have been indexed, it is evident that the differences are due mainly to the variation that occurs in the reciprocal lattice parameter γ^* of low-temperature albite on increasing the anorthite content, or on heat-treatment.

(*a*) 2·44–2·60 kX.—Natural albite gives three reflections of intensities, medium, weak, and weak-medium in order of decreasing spacing, but the weakest of the group, the $1\bar{1}2$ reflection of albite, is not observed in the patterns of natural materials with more than 40 % anorthite. Although the intensities of the stronger reflections (indices $24\bar{1}$ and $\bar{2}41$ for albite) remain practically unchanged, their separation decreases with increasing anorthite content up to 65 % anorthite, there remaining but one unresolved reflection within the range $An_{70}Ab_{30}$–$An_{90}Ab_{10}$. The synthetic materials give only one corresponding reflection over the whole range An_0Ab_{100}–$An_{80}Ab_{20}$.

Both high- and low-temperature materials of composition $An_{90}Ab_{10}$–$An_{100}Ab_0$ give two medium-strong reflections whose separation increases with increasing anorthite content, being about 0·03 kX for pure low-

temperature anorthite and somewhat smaller for synthetic anorthite. The presence of these reflections is sufficiently diagnostic to identify anorthites with certainty.

It appears that the $24\bar{1}$ and $\bar{2}41$ reflections of low-temperature albite shift towards each other as the anorthite content increases, crossing somewhere in the range $An_{70}Ab_{30}$–$An_{80}Ab_{20}$ and being again resolved at $An_{90}Ab_{10}$. Although they are not resolved for specimen 13 (86·5 % anorthite), there is an unusually broad line in the X-ray pattern suggesting that their spacings have different values for this composition.

(*b*) 2·95–3·07 kX.—In this region all patterns have one reflection, with probable indices $1\bar{3}1$, whose spacing for natural materials varies from 2·95 at An_0Ab_{100} to 3·00 kX at $An_{40}Ab_{60}$, and from 3·01 at $An_{70}Ab_{30}$ to 3·03 kX at $An_{100}Ab_0$. The spacing of the $1\bar{3}1$ reflection for synthetic albite is about 3·00 kX, significantly greater than the corresponding value for low albite. For the remaining synthetic materials the spacing increases but slightly with anorthite content up to $An_{80}Ab_{20}$, changing from 3·02 to 3·04 kX in the range $An_{80}Ab_{20}$–$An_{100}Ab_0$.

Tuttle and Bowen (1950) pointed out the diagnostic value of the $1\bar{3}1$ spacing in distinguishing between high- and low-temperature albites. The present data indicate that a distinction can be made on this basis for materials containing up to 40 % anorthite.

(*c*) 3·07–3·28 kX.—This region is characterized by the presence of the two very strong plagioclase reflections. In the pattern of natural albite the weaker of these has the higher spacing with indices $20\bar{2}$, the other having possible indices 040 and 002; there is also a third weak-medium reflection with indices $2\bar{2}0$. A similar group of lines is found in the pattern of oligoclase, specimen 2.

In the patterns of the natural materials of higher anorthite content, and in those of all the synthetics, there is a medium-strong reflection, with possible indices 220, corresponding in position to the $2\bar{2}0$ line of natural albite, but the stronger of the two very strong reflections is now of higher spacing and there occur one or two additional reflections of even greater spacing. These differences are probably due to changes in the 220, $2\bar{2}0$, and 040 spacings.

(*d*) 3·55–3·80 kX.—The strong, rather broad line given in this region by albite probably consists of three overlapping reflections with indices 130, $13\bar{1}$, and $1\bar{3}0$. In the pattern of specimen 6 (51·6 % anorthite), the 130 and $13\bar{1}$ reflections again coincide, but the $1\bar{3}0$ reflection, of weak-medium strength, now lies quite close to the 111 line, whose spacing is not very different from that in albite. For materials of

intermediate composition a line, apparently corresponding to the $1\bar{3}0$ reflection, has a spacing which increases with increasing anorthite content. This variation might be useful in determining the approximate composition of a low-temperature plagioclase of anorthite content less than 50 %.

In the pattern of natural anorthite, specimen 14, the 130 and $13\bar{1}$ reflections again coincide, but the $1\bar{3}0$ reflection is now of greater spacing and intensity than the 111.

The synthetic albites, specimens 17 and 18, give 111 and 130 reflections whose separation is of the order found for natural albite, but the $1\bar{3}0$ spacing coincides with the 111. For materials of higher anorthite content a third intermediate and weaker reflection is also recorded.

(*e*) 4·60 kX and above.—This region of the pattern is not very useful for indicating composition or distinguishing between high- and low-temperature forms. The characteristic 6·4–6·5 kX reflection is of medium strength for albites and oligoclases, but decreases in intensity as anorthite content increases, and there is a similar change in the intensities of the $11\bar{1}$ and $\bar{1}11$ reflections which become too weak to be observed in patterns of labradorites, bytownites, and anorthites.

Interesting features are: (i) the appearance of the $0\bar{2}1$ reflection for materials of anorthite composition greater than 30 %; (ii) the appearance in the patterns of natural bytownites and anorthites of a few very faint reflections of spacings greater than 4·03 kX, which are not of the type $(h+k)$ even. These may possibly be (*b*)-type reflections (see Gay, 1953), and are not considered here, since the relatively high background intensities of the patterns do not permit accurate measurement.

In view of the importance of high-spacing reflections for identification purposes, it is intended to study this region taking careful precautions to minimize the background of the pattern.

5. Quantitative estimation of composition from X-ray data.

In the preceding section certain plagioclase reflections whose positions vary in a systematic manner with composition have been described. By recording such reflections very accurately, a plot of their angular positions against composition would probably furnish determinative curves for plagioclase analysis. It is unlikely that the accuracy required can be achieved by the usual photographic methods of recording powder patterns, since many systematic errors, some of which vary with the nature of the powder specimen, are likely to arise. This difficulty can

be overcome by measuring the angular separation of two adjacent reflections rather than the absolute position of one line; the measurement is then almost independent of even relatively large calibration errors and is accurately reproducible by most powder techniques. This method has been used by Claisse (1950) and by Tuttle and Bowen (1950) for the plagioclase felspars.

If any useful relationship is to be established, it is necessary to use two reflections whose separation is sensitive to small compositional changes over the whole range, or at least a large part of it, and which are well defined and set apart from other lines. Claisse chose two groups, each consisting of three reflections, which satisfied many of these conditions. Those of one group, named 1, 2, and 3 by Claisse and referred to in this paper as C, D, and E respectively, lie in the spacing range 2·60–3·10 kX. Those of the second group, Claisse's 4, 5, and 6 lines, have much lower spacings, and though they lie in a complex region of the pattern, nevertheless the group as a whole is prominent and easily recognized; the 4

TABLE V. Details of the reflections.

Line.	Spacing (kX units).	Intensity.	Probable indices.
A	3·11 –3·14	wm–ms	$2\bar{2}0$ or 220*
B	2·95 –3·03	wm–ms	$1\bar{3}1$
C	2·63 –2·65	wm–m	$\bar{1}32$
D	2·50 –2·56	m-s	$24\bar{1}$
E	2·44 –2·52	wm–s	$\bar{2}41$
F	1·825–1·833	m–ms	—
G	1·764–1·785	ms	—

* $2\bar{2}0$ for low-temperature albite.

TABLE VI. *Angular separations ($\Delta 2\theta^\circ$ for Cu-Ka radiation) of the A–B, C–D, C–E, and F–G reflections.*

Natural plagioclases						Synthetic plagioclases					
No.	An. (wt. %).	A–B.	C–D.	C–E.	F–G.	No.	An. (wt. %).	A–B.	C–D.	C–E.	F–G.
1	0·4	1·85	1·06	2·81	1·20	17	5	1·09	1·90	—	1·54
2	16·5	1·63	1·34	2·51	1·39	18	10	1·10	1·91	—	1·54
3	22·1	1·51	1·44	2·38	1·50	19	30	1·07	1·91	—	1·58
4	31·1	1·35	1·61	2·22	1·53	20	40	1·04	1·93	—	1·68
5	38·0	1·31	1·63	2·21	1·61	21	60	1·02	1·93	—	1·84
6	51·6	1·25	1·69	2·14	1·71	22	70	0·97	1·93	—	1·91
7	64·5	1·14	1·79	2·06	1·84	23	80	0·92	1·94	—	2·01
8	66·3	1·09	1·80	2·03	1·85	24	90	0·84	2·05	1·82	2·06
9	71·3	0·99	1·90		1·87	26	100	0·77	2·11	1·75	2·12
10	77·0	1·01	1·89		1·91	(heated)					
11	81·0	0·96	1·92		1·91	26	100	0·77	2·15	1·77	2·02
12	81·5	0·96	1·92		1·92						
13	86·5	0·88	1·92		1·94						
14	93·0	0·81	2·13	1·80	1·95						
16 (volcanic)	~100	0·79	2·19	1·76	1·98						
15	98·3	0·76	2·14	1·78	2·03						

and 6 lines only are dealt with here, being referred to as F and G respectively. The precise descriptions of these reflections as they appear in the patterns of the natural materials are given in table V, together with another group of two, named A and B, which are also used.

By plotting against composition the angular separations, $\Delta 2\theta°$, of the reflections in each of his two groups, Claisse obtained six linear relationships, each of which showed a break in the vicinity of $An_{30}Ab_{70}$–$An_{40}Ab_{60}$. The curves for the C–D–E combination were steeper than the others, but apparently suffered a disadvantage in that reflections D and E crossed each other at about $An_{80}Ab_{20}$, leading to some ambiguity in interpretation in this region. The F–G combination was free from this limitation, and the slope of the curve was sufficiently great to enable a reasonable estimate of composition to be made. By making use of all the curves the author claimed that, in general, it should be possible to estimate composition to 1 %.

Several objections can be raised to Claisse's work: (i) only eight plagioclases were used to cover the whole composition range, four of these lying within the limits An_0Ab_{100}–$An_{30}Ab_{70}$; (ii) all the materials were apparently assumed to be of low-temperature form, though the anorthite specimen came from a volcanic locality; (iii) all the analyses were based on optical measurements and compositions were claimed to be accurate to 1 %. Further, it is not clear from Claisse's paper whether or not each composition was determined on more than one grain. This is important, since presumably the powder specimens consisted of material representative of many grains. In the present work none of these difficulties arise, and an attempt has been made to describe in some detail the nature of the reflections employed.

The reflections A and B have been discussed in sections 4(*c*) and (*b*) respectively, and D and E in section 4(*a*), together with the corresponding reflections which appear in the patterns of the synthetic materials. The C reflection, with indices $\bar{1}32$ for natural albite, is similar in position and intensity in all the patterns. All these lines are sharp and can be readily recorded, the only complication occurring in the overlap of D and E in the patterns of natural materials of composition about $An_{80}Ab_{20}$.

Microphotometer traces of reflections F and G are shown in fig. 3, and from these it can be seen that the G peak is quite sharp for all specimens. While the F peak is reasonably resolved from neighbouring reflections for all the natural specimens, difficulties arise in its interpretation in the patterns of some of the synthetic materials. Specimens 20

and 21 show a diffuseness of the F peak which is certainly due to their poor crystallinity. In specimen 23 the peak is only just resolved from a neighbouring reflection of slightly larger spacing, and although specimen 24 apparently shows only one line in this region, the better crystallized specimen 26 quite definitely shows two. For this reason the pattern of the latter has been used to obtain the positions of the F and G reflections for synthetic anorthite.

Table VI contains the angular separations of the various combinations of reflections; the values, which are given for Cu-$K\alpha$ radiation to facilitate comparison with Claisse's data, are accurate to within $\pm 0{\cdot}03°$. Figs. 4, 5, and 6 show the curves obtained by plotting angular separations against composition determined chemically.

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 probably has the same, or similar, indices over the whole composition range. Although the break at $An_{30}Ab_{70}$ is very marked in each case, that at the calcic end appears to be more complex, and cannot be represented by the intersection of two straight lines. In the C–D and C–E curves the situation is further complicated by the failure to resolve the D and E reflections over an appreciable range of composition, and only tentative dotted lines can be used to represent the relationships from $An_{65}Ab_{35}$ to $An_{100}Ab_0$.

Corresponding curves for the synthetics show only one discontinuity at about $An_{75}Ab_{25}$. This is in harmony with the conception of a complete solid solution for high-temperature materials from An_0Ab_{100} to $An_{70}Ab_{30}$, which is suggested by the single-crystal diffraction work of Gay (private communication), although the early work of Bowen (1913) on melting phenomena indicated solid solution over the whole range from albite to anorthite.

The F–G curves for natural and synthetic materials, fig. 6, show slight discontinuities which cannot, in every case, be explained in terms of the structural scheme for the plagioclases, but since the F and G reflections occur in a more complex region of the pattern, it is possible that each does not have the same indices for all compositions.

By making use of selected portions of all the curves it should be possible to obtain reasonable analyses from X-ray data. The accuracy of an analysis so obtained will depend not only on the thermal history of the specimen but also on its composition.

(*a*) An_0Ab_{100}–$An_{30}Ab_{70}$.—The F–G separation enables a plagioclase

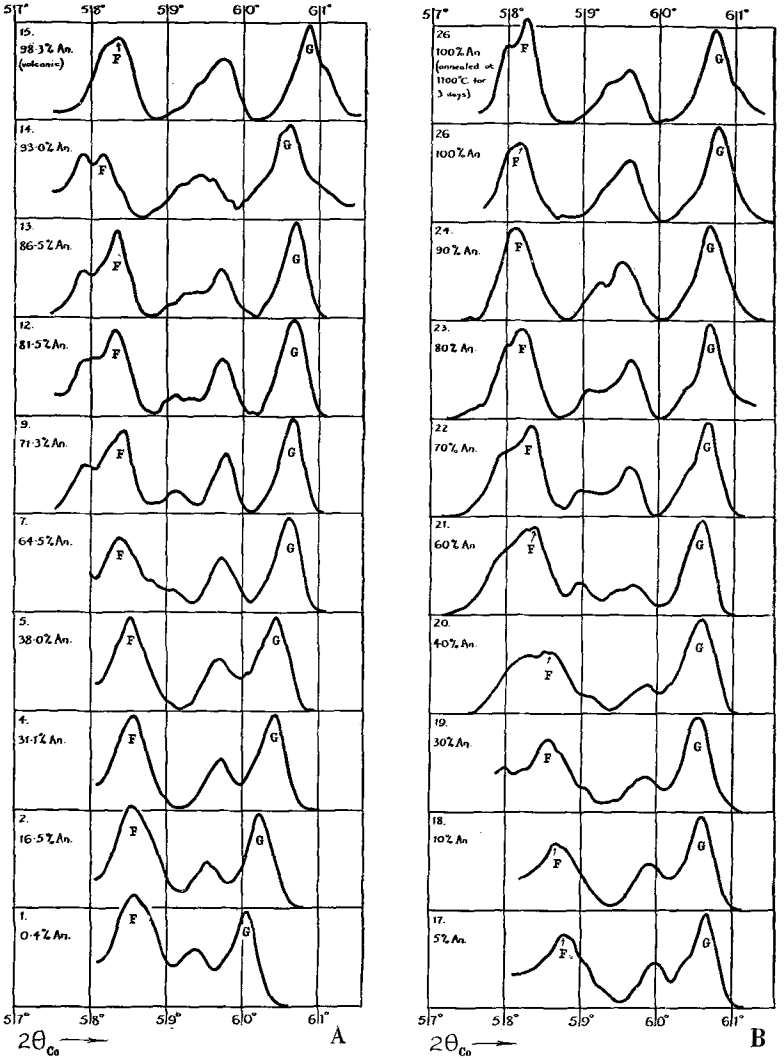

FIG. 3. Microphotometer traces of F and G reflections plotted for Co-$K\alpha$ radiation. G peak reduced to standard height. The 'annealed' no. 26 is discussed in section 5(*c*).

A, Natural plagioclases. B, Synthetic plagioclases.

to be assigned to this composition range. If the material is known to be of low-temperature form, the A–B, C–D, and C–E separations permit an accurate analysis to be made, since they are very sensitive to changes of composition. Although a high-temperature form can readily be recognized, there is no means of estimating its composition.

(*b*) $An_{30}Ab_{70}$–$An_{70}Ab_{30}$.—Using the A–B, C–D, and C–E curves, high- and low-modifications can be distinguished for compositions between $An_{30}Ab_{70}$ and $An_{65}Ab_{35}$, since a fairly good analysis can be obtained for both from the F–G curve.

At $An_{70}Ab_{30}$ there is practically no distinction between the characteristics of natural and synthetic materials; this was also found to be the case for specimen 9 when heated to 1250° C. for 48 hours.

(*c*) $An_{70}Ab_{30}$–$An_{100}Ab_{0}$.—An estimate of the composition can be made using the A–B curve, but there seems to be no precise way of distinguishing between high- and low-temperature forms. The F–G curve might provide this information for compositions between $An_{90}Ab_{10}$ and $An_{100}Ab_{0}$, although not so conclusively as Gay's single-crystal method.

Two specimens are of interest in this connexion: (i) the synthetic anorthite 26, when 'annealed' at 1100° C. for three days, gives a well-resolved F reflection and an F–G separation of 2·02°, a value nearly falling on the natural curve; (ii) specimen 15, of volcanic origin, gives a separation of 2·03°, higher than the value expected of a low-temperature material of the same composition. The latter specimen thus appears to be partially inverted by this criterion.

According to the present data, the anorthite specimen employed by Claisse (1950) is clearly a high-temperature form. If this interpretation is correct, and it seems possible judging by the locality of this specimen, it is not surprising that Claisse failed to find discontinuities in his curves at $An_{70}Ab_{30}$.

6. Concluding remarks.

The identification of a plagioclase felspar is simplified by the fact that the general nature of the X-ray pattern does not vary considerably with either composition or thermal history. At the same time the pattern is sufficiently diagnostic to enable a fairly accurate analysis to be made over a wide range of composition. Further, a distinction can be made between high- and low-temperature modifications particularly in the range $An_{0}Ab_{100}$–$An_{40}Ab_{60}$, readily for $An_{40}Ab_{60}$–$An_{65}Ab_{35}$, and with some difficulty for $An_{90}Ab_{10}$–$An_{100}Ab_{0}$. For this purpose, the X-ray powder

method does not appear to be much superior to that based on optical data, according to a recent survey of the latter by Reynolds (1952).

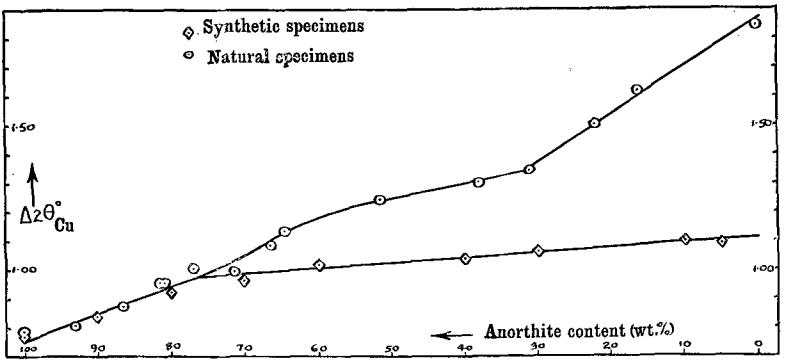

FIG. 4. Angular separation (calculated for Cu-$K\alpha$ radiation) of A–B reflections plotted against plagioclase composition.

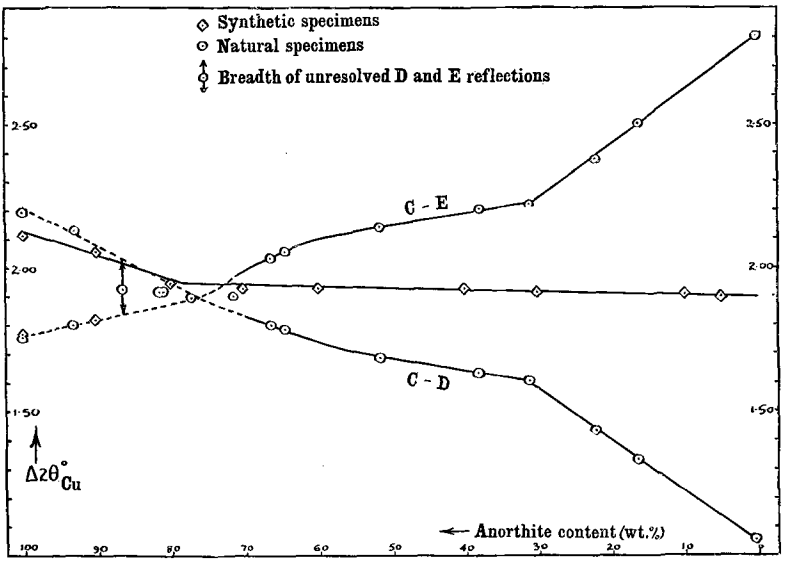

FIG. 5. Angular separation (calculated for Cu-$K\alpha$ radiation) of C–D–E reflections plotted against plagioclase composition.

The composition curves, figs. 4, 5, and 6, and their use in plagioclase analysis, cannot be taken as definitely established until their behaviour

has been studied in relation to heat-treated specimens. In this connexion complications are likely to arise from the possible occurrence of partially inverted materials. Further experiments are now in progress to examine these details.

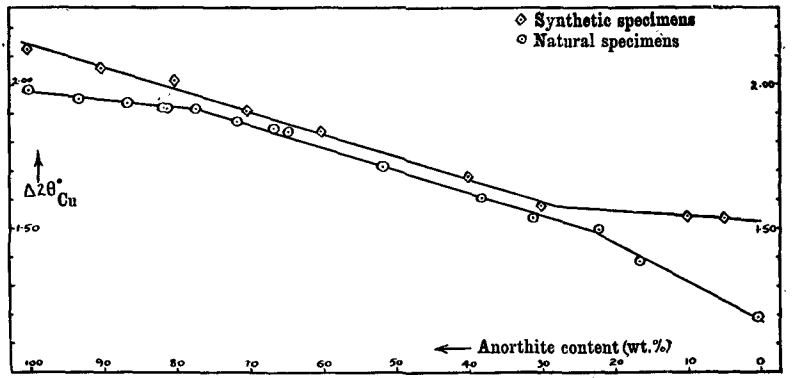

FIG. 6. Angular separation (calculated for Cu-$K\alpha$ radiation) of F–G reflections plotted against plagioclase composition.

The data presented in this paper show just how much can be done with the usual powder techniques employing photographic recording. More reliable data could no doubt be obtained by the modern Geiger counter spectrometer, but such a refinement does not appear to be necessary for the general problem of plagioclase identification.

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