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THE SALISBURY ADAMELLITE PLUTON, NORTH CAROLINA

By

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ABSTRACT

The adamellite pluton southeast of Salisbury, North Carolina was mapped, sampled systematically on a one mile grid, and two thin sections from each sample were examined and point-counted. Since its initial emplacement the pluton has undergone one period of regional metamorphism and a later period of minor kinetic metamorphism which created a series of shear zones within the pluton. The regional metamorphism left recognizable relict igneous textures and minerals in the pluton. It also converted the plagioclase to albite and is the probable cause of the pink colored albite in parts of the pluton.

INTRODUCTION

Location

A body of granitic rock crops out two miles southeast of Salisbury in Rowan County, North Carolina (Figure 1) and trends southwestward for a distance of eight miles. It has a fairly constant outcrop width of about two miles and an areal extent of approximately 18 square miles. In the present report this granitic body will be called the Salisbury Pluton. The rock comprising this pluton has been classified as adamellite and named albite adamellite.

Previous Work

LeGrand (1954) published a reconnaissance geologic map of Rowan County as part of a ground water study of the area. He mapped the rocks as masses of homogeneous gabbro and granite interspersed in a diorite-granite injection complex. Chayes (1956) used samples from Salisbury in his study on the statistical reliability of modal analysis. Chayes (1952) also published a paper in which he ascribed perthite in these rocks to exsolution from an originally homogeneous microcline during the period in which a cataclastic texture was developed.

Snyder (1963) made a gravity survey of Rowan County. His Bouguer anomaly map shows two small negative anomalies over the Salisbury Pluton. One anomaly is at Granite Quarry and the other is

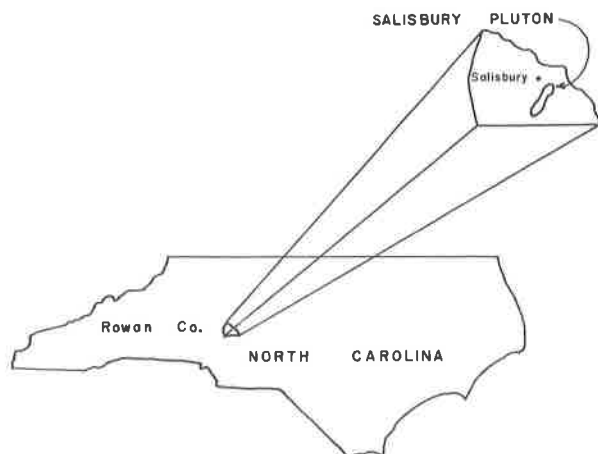


Figure 1. Index map.

about 5 miles south of Granite Quarry. Snyder thinks these two anomalies indicate the deepseated nature of the pluton.

Watson and Laney (1906) and Council (1954) published articles on the granites of North Carolina. Both these papers included descriptions of the rocks and comments on the economic aspects of several individual quarries in the Salisbury area.

Methods

Field work was done at intervals during the summer and fall of 1964 and spring of 1965. A one-half mile grid was constructed on the Rockwell and Salisbury 7.5 minute topographic sheets and samples were collected as near as possible to the center of each square. Modal analysis was restricted to samples collected on a one mile grid in order to reduce the number of thin sections required. Sample localities utilized for modal analysis are shown on Figures 3, 10, 11, and 12.

No exposed contacts were found in the field, however, the adamellite and the country rock yield distinctly different soil types upon weathering. This difference made mapping possible in areas of saprolite. Contacts shown on the map (Figure 2) are all approximate but are presumed to be within a few feet of the actual contact.

Thin sections were stained for both potash feldspar and plagioclase using the technique described by Bailey and Stevens (1960). Anorthite content of the plagioclase was determined by measuring rho of cleavage flakes in immersion oils of known index and utilizing a curve by Tsuboi (1923).

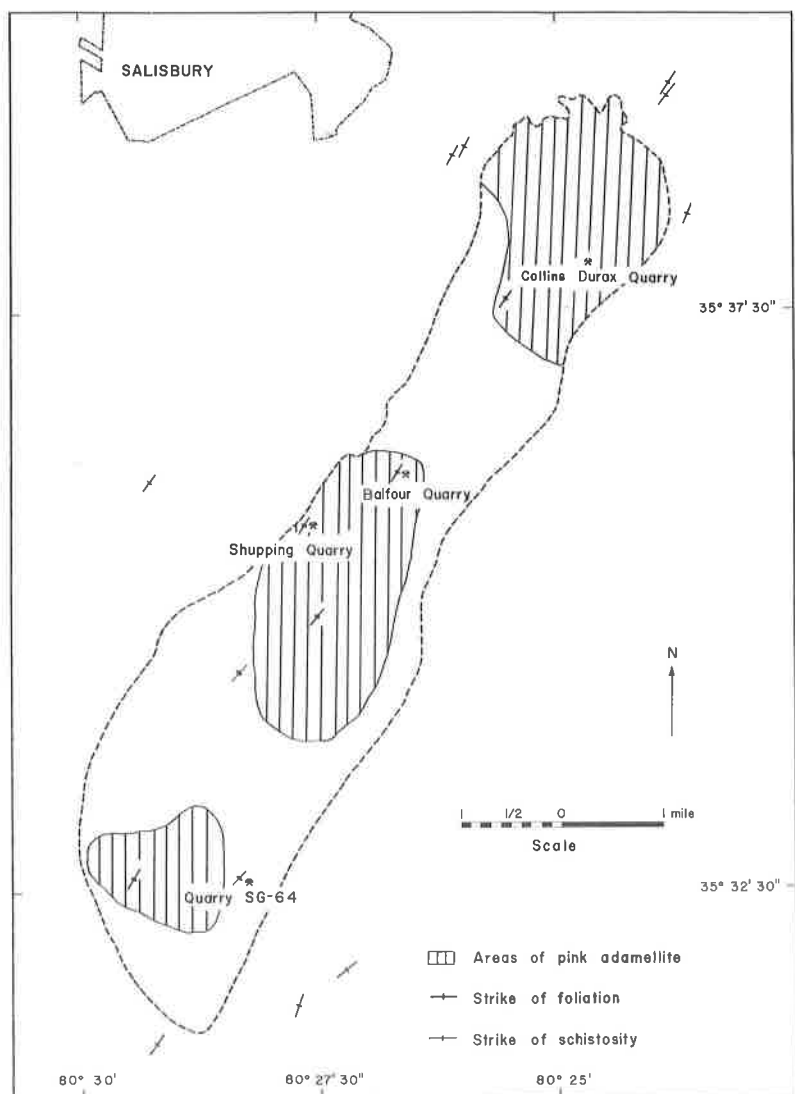


Figure 2. Geologic map.

Acknowledgements

The writer would like to thank Virgil I. Mann for his encouragement and constructive criticism during the course of active research. Expenses incurred by field work and thin section preparation were defrayed by the Division of Mineral Resources of the North Carolina Department of Conservation and Development. C. R. Deadwyler of the Harris Granite Quarries Company kindly furnished three chemical analyses.

GEOLOGY

Regional Geology

The Piedmont Province of the Southeastern United States consists of consecutive belts of metamorphic and igneous rocks, elongated in a northeast-southwest direction. King (1955) and previous workers gave these belts the following names from east to west: the Carolina Slate belt, the Charlotte belt, the Kings Mountain belt, the Inner Piedmont belt, and the Brevard belt. The Salisbury Pluton is part of the Charlotte belt.

McCauley (1961) in South Carolina, as well as Bell and Overstreet (1959) and Kesler (1944) in North Carolina, have been able to distinguish three to five episodes of intrusive activity in the Charlotte belt. Most of the younger plutons, such as the syenite ring dike in Cabarrus County, North Carolina (Morgan and Mann, 1964), and some of the gabbros in Newberry County, South Carolina (McCauley, 1961), show cross cutting relationships to the general northeast-southwest trend of the Charlotte belt rocks.

Field Relations of the Salisbury Pluton

The Salisbury Pluton crops out in an elongate, discontinuous ridge extending from a point two miles east of Salisbury to near the Rowan-Cabarrus county line about nine miles south of Salisbury (Figure 2). Large boulder outcrops are common and the flanks of the ridges are generally marked by expanses of bare rock exposed at the surfaces; however, most of the area is covered by a thin veneer of saprolite. Nearly every area of bare rock and most of the boulder outcrops have been opened by small quarrying operations in the past. At present only six large quarries are being actively worked.

The country rocks consist of low grade, regionally metamorphosed sedimentary rocks which have been intruded by small masses of diorite and granite. Metasedimentary rocks include quartzite, phyllite, sericite schist, biotite schist, and chlorite schist. They usually contain small quartz stringers and an occasional cross-cutting diabase dike. This entire complex is cut by numerous, and occasionally closely spaced, joint planes. Schistosity in these country rocks shows a rather consistent strike of $N32^{\circ}E$. Therefore, the pluton exhibits an overall concordant relationship with the surrounding Charlotte belt rocks.

Outcrops immediately surrounding the granitic mass are covered by a deep red saprolite containing small quartz veinlets. This saprolite occupies a zone varying between 200 and 400 feet in width. No fresh rock was found here. The zone gives way at its periphery to the typical metasedimentary-injection complex described above.

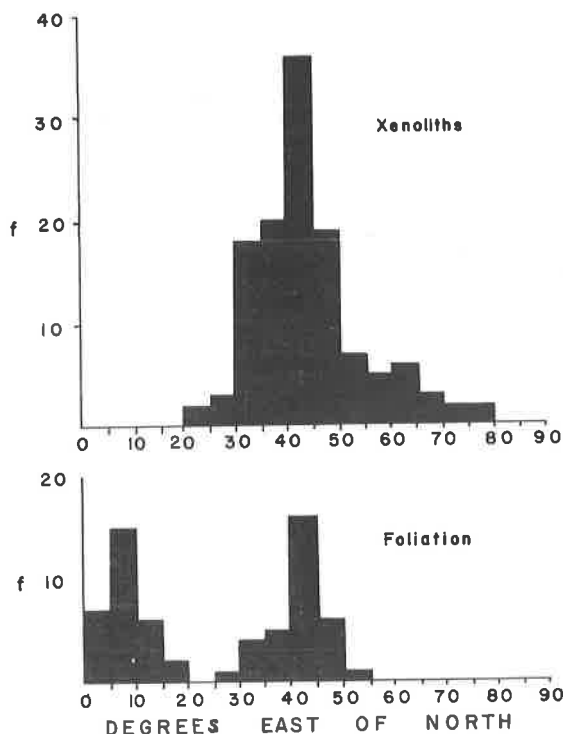


Figure 3. Histograms showing relation between strike of foliation in the adamellite and strike of long axes of xenoliths.

Within the pluton the adamellite exhibits a distinct foliation in most outcrops. In some outcrops it is apparent that this foliation strikes at an angle to the strike of the long axis of the xenoliths (Figure 4). Readings from several quarries were plotted and showed two distinct directions of foliation rather than a range of directions as had been anticipated. In order to substantiate this finding the present author studied quarry SG-64 (Figure 2) intensively. The strikes of the long axis of 128 xenoliths were measured and 33 readings of the strike of the foliation were also taken. Results of this study are shown in Figure 3. The histogram of readings on the strike of foliation shows two distinct peaks. The first peak, the mode of which is 5-10 degrees east of north, can be correlated in the quarry with a shear zone which imparts a distinct gnissoid appearance to the rock. The second peak, the mode of which is 40-45 degrees east of north, coincides with the peak of the xenolith histogram and is imparted to the rock by aligned quartz aggregates.

Xenoliths are common in a few quarries and entirely absent in



Figure 4. Foliation in the adamellite is parallel to the pencil and at an angle with the xenolith train. Quarry SG-64.

others. Most are rather small and spindle shaped with sharp contacts and smooth outlines (Figure 4). The Balfour quarry contains several large xenoliths, most of which have jagged or rectangular outlines. The rock in these large xenoliths is a slightly schistose argillite, and it has the same general appearance as that making up most of the small xenoliths. Nothing resembling an autolith was observed in any of the quarries.

No aplites or lamprophyres were observed in any of the quarries. All primary cavities or planes of weakness in the pluton are filled with what appears in hand specimen to be pure quartz. In quarry SG-64 this quartz, which occurs as small pods or long stringers, is pegmatitic. It contains large crystals of garnet and feldspar.

Most of the quarries showed systems of widely spaced joints. The joint surfaces are coated with either smoky quartz, epidote, or manganese oxides. Only a few joint surfaces show slickensides.

PETROGRAPHY

Hand Specimen Description

The Salisbury albite adamellite is massive to foliated, and is usually gray in color, but it has both pink and nearly white varieties. It is a holocrystalline, even grained, granular rock. Potash feldspar is always milky white in color and is apparently subhedral. In the pink varieties plagioclase has a uniform light pink color and appears euhedral. In most pink and gray varieties the quartz is light gray, although

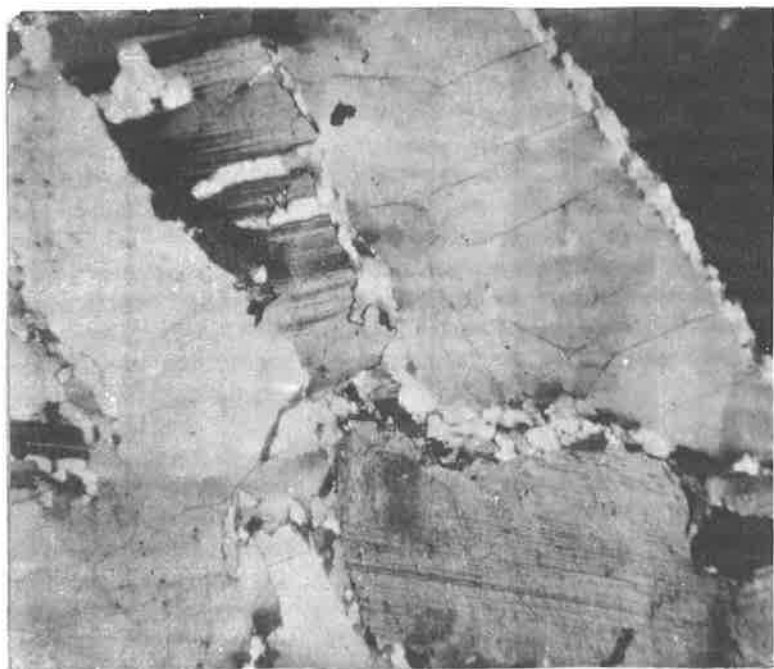


Figure 5. Mortar texture, crossed nicols X 44.

in some samples it is smoky. In the white rocks all three major constituents, quartz, potash feldspar, and plagioclase are milky white giving the rock a uniform white color, which is relieved only by occasional specks of yellow garnet and green chlorite. The accessories, including micas and opaque minerals, never make up more than 7 percent of the rock.

Thin Section Description of Textures

The adamellite has three distinct textural varieties. In the present report these three textural varieties have been given the following names: (1) mylonitic, (2) cataclastic, and (3) mortar. All three textures are the result of varying degrees of metamorphism. Rocks with mylonitic texture are found near shear zones. Large areas in both the northeast and southeast parts of the pluton contain rocks with a mortar texture. The remainder of the rocks possess a cataclastic texture.

No thin section showed the classic hypidiomorphic-granular texture, but the mortar texture appears to be a relict hypidiomorphic-granular texture with recrystallization of part of the quartz (Figure 5). At some localities kinetic metamorphism had a commanding influence on the generation of texture. The rock is lineated with bands of very small, flat, oriented crystals of quartz between larger feldspar

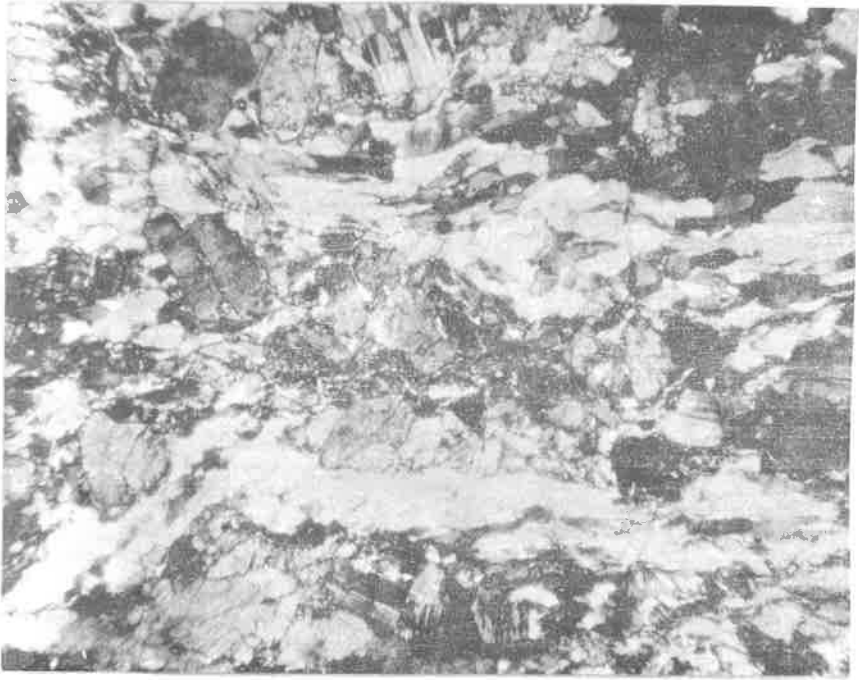


Figure 6. Mylonitic texture, crossed nicols X 44.

crystals. This texture can best be described as mylonitic (Figure 6), although all such rocks contain patches of relatively undisturbed feldspar or quartz.

The most widespread texture found in the pluton lies between these extremes. The best descriptive term for it is cataclastic (Figure 7). Grain size, amount of recrystallized quartz, and extent of deformation suffered by the feldspars are all intermediate between the mylonitic rocks and those having a relict igneous texture.

Thin Section Description of Minerals

General Statement. Examination of 67 thin sections of the adamellite reveals an almost constant mineral content. Essential minerals are microcline, plagioclase, and quartz. These three constitute at least 93 percent and usually 98 percent of the rock. Accessories include biotite, muscovite, calcite, garnet, chlorite, epidote, sphene, monazite(?), opaque minerals, an isotropic mineral that is probably fluorite, and an unidentified mineral with properties close to those of staurolite. In addition, some of the sections reveal a dark brown to opaque material ramifying along cracks between and within the other minerals. Field relations show this material to be a weathering product, probably manganese oxide.

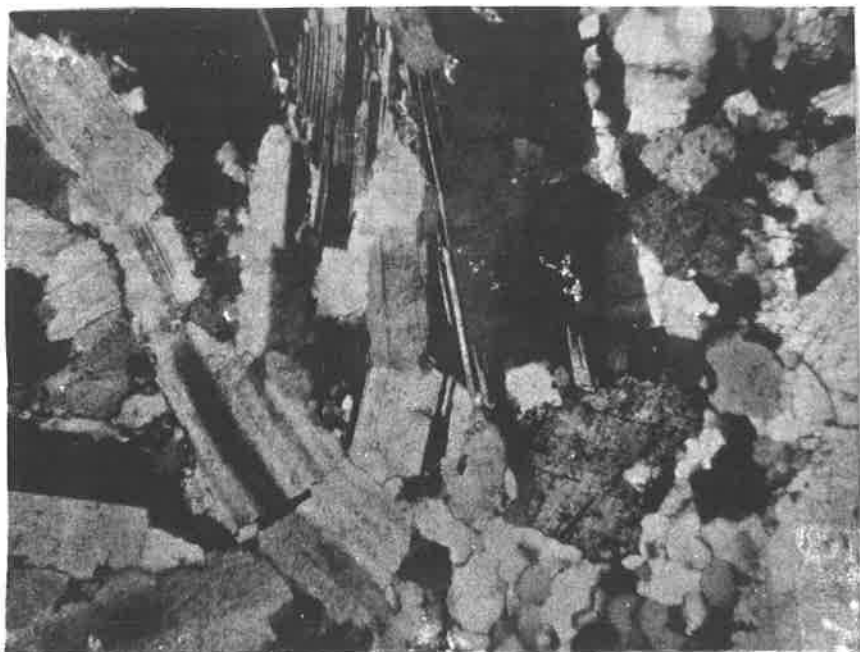


Figure 7. Cataclastic texture, crossed nicols X 44.

Quartz. There are two generations of quartz in the adamellite. Both have been observed in nearly every thin section studied. Type I is highly undulatory and occurs in large grains, with an average size of 3mm and a maximum of 6mm in longest dimension. It is interstitial to the feldspars in rocks with a mortar texture. Type II quartz occurs as an interlocking mosaic of fine grains bordering the larger minerals or in linear bands in specimens which show a mylonitic texture. In some cases it shows a slightly undulatory extinction but usually none at all. Type II quartz varies in size from 0.05 to 0.25 mm and averages 0.1 mm in its longest dimension. In the mylonitic rocks this fine grained quartz is oriented and may have formed later than the typical type II quartz.

Plagioclase. Plagioclase occurs as polysynthetically twinned laths averaging 3 mm in longest dimension. It is the only essential mineral possessing nearly euhedral crystalline outlines. The crystals show evidence of stress even in the least deformed parts of the pluton. They have slight undulations or pits in the crystalline outline or a fringe of either type II quartz or fibrous muscovite surrounding the crystal. In some thin sections the plagioclase laths are bent, resulting in extreme undulatory extinction, or cracked and having the crack filled with type II quartz or calcite. In others the laths have been broken into several pieces and these smaller remnants have been either separated

and rotated or crushed together. Despite this evidence of cataclastic action, plagioclase showed more resistance to deformation than either quartz or microcline.

No zoned plagioclase crystals were observed in the sections studied. Nearly every plagioclase lath showed polysynthetic albite twinning. Probable carlsbad and pericline twins were also observed in a few sections.

Most of the plagioclase contains inclusions of small discrete grains of muscovite with an average length of 0.03 mm. There are also specks of chlorite and small patches of calcite within the plagioclase. In some cases these inclusions are randomly arranged but in others they show one of two distinct patterns. In one type the mica flakes are arranged in two sets of parallel rows. These two sets make an angle of approximately 80° with each other and seem to be parallel to possible crystal faces. A second pattern consists of lines of inclusions parallel to the outline of the crystal. This pattern may indicate former zoning in the plagioclase. Very little sericite was observed in the sections studied. Under high magnifications the plagioclase in some sections is seen to contain inclusions of extremely fine grained opaque material. Sections showing this material can be roughly correlated with hand samples containing pink plagioclase. It is therefore assumed that this material imparts the pink color to the plagioclase.

Anorthite content of the plagioclase ranges from 1 to 11 percent (Table 1) and has an average of 3 percent anorthite. Thus the plagioclase is albite.

Potassium Feldspar. Potash feldspar occurs as anhedral grains with longest dimensions ranging from 0.2 mm to 0.5 mm. In general it is free from inclusions and clear under uncrossed nicols. A good portion of the potassium feldspar shows the typical grid twin pattern of microcline.

Potash feldspar also occurs, together with albite, in two types of perthite. Type I is "flame" perthite. This perthite is the type Chayes (1952) thinks was formed when the rock was granulated.

Type II perthite is of the patch variety usually attributed to replacement (Deer, Howie, and Zussman, 1963, p. 69). Replacement textural relationships in all stages from incipient attack, seen as a narrow zone of microcline around a plagioclase crystal (Figure 8), to the development of patch perthite (Figure 9) have been observed. In all cases the plagioclase is well twinned and boundary relationships are curves. The microcline has evidently replaced the albite. The author is well aware of the controversial nature of this statement. However, the relationships between microcline and albite, as exhibited in Figures 8 and 9, is universal in the Salisbury Pluton and permits no other interpretation. Also, since metasomatic replacement of plagioclase by microcline has been reported by others, among them Battey (1955), Robertson (1959), and Schermerhorn (1956), there seems to be no reason for summarily rejecting this interpretation.

Table 1. Modal Data in Volume Percent and Anorthite Content of Plagioclase.

Sample	Plagioclase	K-feldspar	Quartz	Color Index	An-content
SG-1	43.38	29.85	24.83	1.94	4
SG-4	41.14	24.45	32.34	2.07	1
SG-7	50.57	28.26	20.04	.64	7
SG-9	45.93	23.73	27.61	2.73	2
SG-15	34.77	27.20	35.82	2.21	5
SG-17	42.86	26.27	29.34	1.53	5
SG-19	37.71	26.69	33.89	1.71	7
SG-21	34.24	25.81	38.29	1.66	1
SG-23	37.22	26.93	33.01	2.84	3
SG-27	42.81	27.40	27.84	1.95	7
SG-28	38.57	24.09	33.94	3.40	4
SG-31	31.99	27.97	38.26	1.78	8
SG-33	36.37	29.80	30.84	2.99	2
SG-35	42.74	24.97	31.11	1.18	1
SG-40	44.52	28.02	25.70	1.76	5
SG-42	39.40	26.56	26.86	7.18	7
SG-50	43.35	24.31	30.94	1.40	1
SG-52	39.98	29.06	29.74	1.22	1
SG-59	41.57	26.16	29.40	2.87	10
SG-61	40.86	24.84	33.02	1.28	11
SG-63	40.74	27.41	30.79	1.06	9
SG-70	46.09	25.47	26.46	1.98	3
SG-72	34.59	27.61	30.59	7.21	5
SG-75	45.37	27.08	26.29	1.26	3
SG-79	41.27	24.21	32.99	1.53	4
SG-80	40.07	28.19	30.29	1.45	1

Accessory Minerals. Accessory minerals fall into two groups: those that formed before granulation of the rock, and those which formed during or after this period. Included in the first group are sphene, chlorite, biotite, monazite(?), and most of the opaque minerals. Calcite, muscovite, fluorite, epidote, garnet, the staurolite-like mineral, and some of the biotite all seem to have been formed at the same time or after the type II quartz; hereafter these minerals will be referred to as metamorphic minerals.

Muscovite occurs both as small flakes included in plagioclase and as fibrous aggregates interspersed with type II quartz and surrounding the essential minerals. The fibrous muscovite is common in those rocks with mylonitic texture and seems to have crystallized at the same time this texture developed. Biotite occurs as rather large flakes that are usually altered in part or, completely to chlorite. Garnet occurs

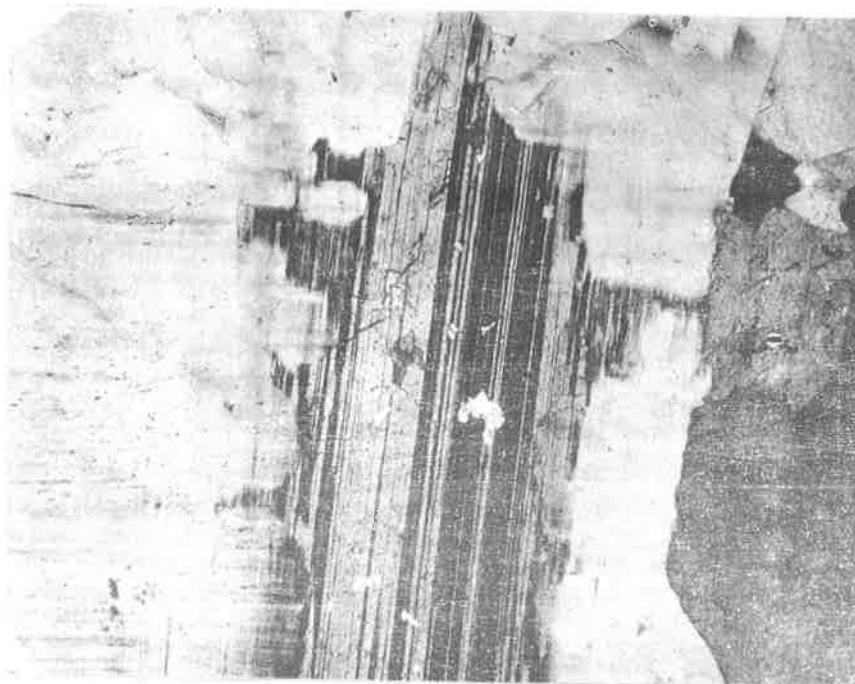


Figure 8. Apparent replacement of plagioclase by microcline, crossed nicols X 126.

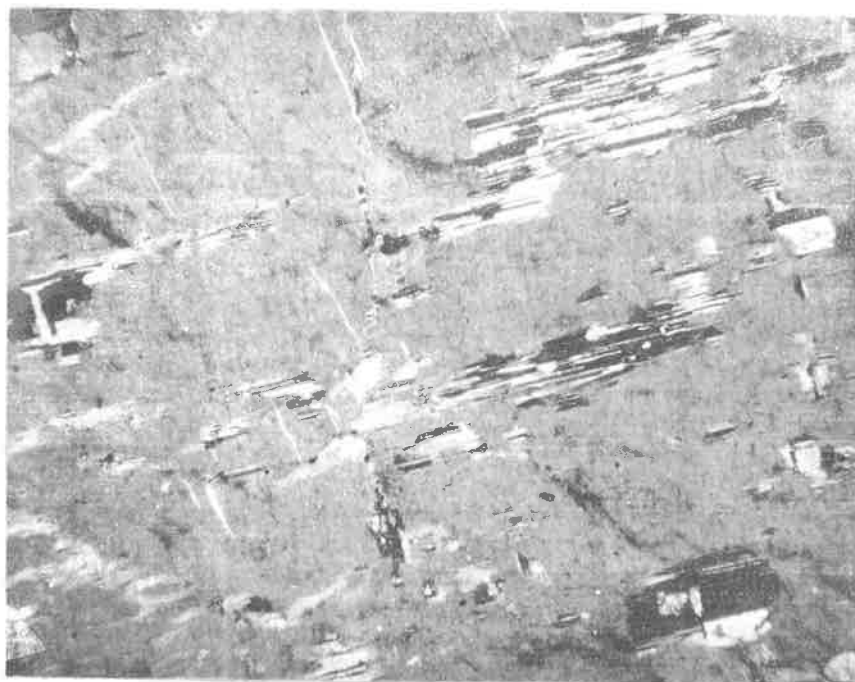


Figure 9. Patch perthite, crossed nicols X 44.

mostly in small euhedral crystals. Most of the grains are very fine and occur in patches along with other metamorphic minerals. Sphene is usually observed in typical euhedral, wedge-shaped crystals, and in one section it appears granulated. However, it is also found as small grains closely associated with metamorphic minerals. There is a possibility that two generations of sphene exist. Calcite and fluorite were the last minerals to form. They never exhibit a euhedral outline, and are found filling cracks in the essential minerals or as a matrix in a group of other accessory minerals. Part of the opaque minerals seem to have been formed before and part after granulation. One euhedral crystal of pyrite which probably formed during metamorphism was observed in hand specimen. Other opaque minerals, as seen in thin section, appear to have been crushed. One grain seems to have been crushed and partially replaced by garnet. Epidote occurs along joint planes and as small crystals associated with other late minerals.

MODAL ANALYSIS

Two thin sections from each of 26 samples were stained to facilitate identification of potassium feldspar and plagioclase. A measurement area of 750 mm² of each thin section was utilized for point counting. With an IC of 36 and using two sections this gives an analytical error of 2.45 percent of less (Chayes 1956). All albite was recorded as plagioclase. Plagioclase and microcline, where these two occurred together as perthite, were recorded separately.

Results obtained from the point count are presented in Table 1. These data are in volume percent of the three essential minerals plus color index. All accessory minerals are included in the color index. Plagioclase varies between 50.57 and 31.99 percent with an average of 40.70 percent. Potassium feldspar varies from 29.85 to 23.73 percent and averages 26.64 percent. Quartz varies from 38.29 to 20.04 percent and averages 30.39 percent.

These four variables were plotted on the base map and contoured (Figures 10, 11, and 12). Color index has an average value of 2.27 percent of the rock. Except for two anomalous high values of 7.21 and 7.18 percent, color index figures are very low and show no significant trends, therefore a contour map of color index variations is not included in this report. The two high values are the result of unusually high amounts of epidote and muscovite in these two samples. The volume percent of potassium feldspar, although it varies more than the color index, shows significantly less variation than either quartz or plagioclase. The potassium feldspar values are contoured on an interval of 1 percent while both plagioclase and quartz are contoured with an interval of 5 percent. The microcline map shows a low closure in the southern portion of the pluton (Figure 12). The northern half is somewhat uninformative. Both the plagioclase and quartz contour maps show closures

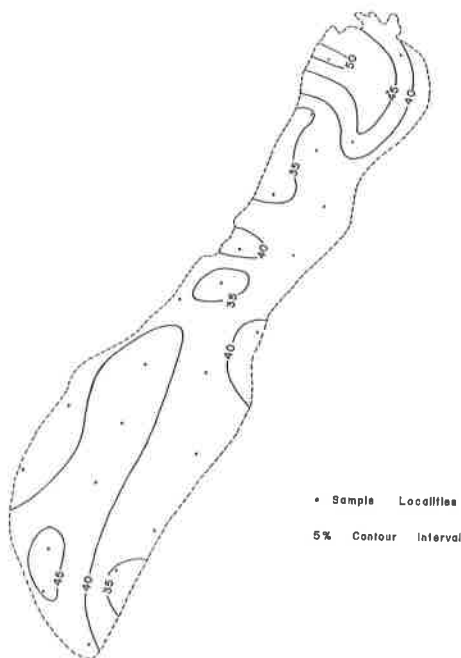


Figure 10. Contour map of plagioclase variations. Shows results of point count in volume percent.

in the southern portion of the pluton and a suggestion of one about half way up the northwest border (Figures 10 and 11).

CLASSIFICATION

An average modal content of the Salisbury rocks shows the following: plagioclase 40.70, potassium feldspar 26.64, quartz 30.39, and color index 2.27 percent. The plagioclase is albite, and if this is considered an alkali feldspar the rock would be classified as an alkali granite. Most recent textbooks advocate such an approach to the problem. Thus, it would seem permissible to classify the rock as an alkali granite. A perusal of the literature, however, proves this to be quite unsatisfactory. The rock would fall into Class I, Order I, Family 7 of Johannsen's classification and he reports no known examples of such a rock (Johannsen, 1932, V. II, p. 312). It would also fall into Class IA of Chayes' classification (1957), and he reports no examples of such a rock. If the albite is considered a plagioclase, instead of being included along with microcline in the alkali feldspars, a way off the horns of the dilemma is found. Figure 13 shows the Salisbury modes plotted on a triangular diagram. The albite is plotted as plagioclase

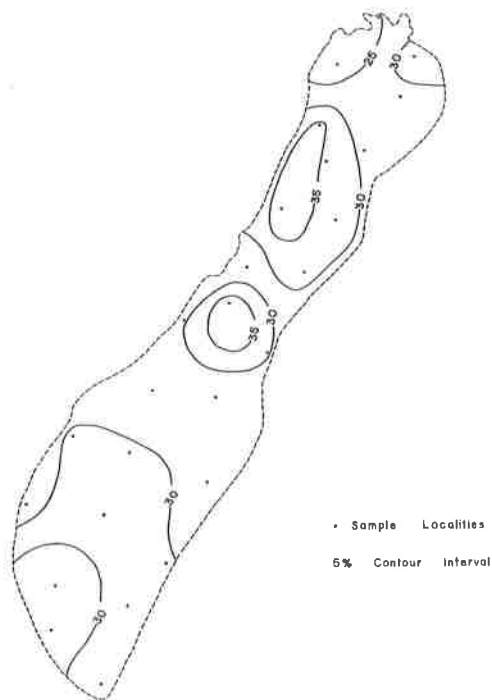


Figure 11. Contour map of quartz variations. Shows results of point count in volume percent.

and Chayes' (1957) division lines are superimposed. All the Salisbury modes fall into class II for which Chayes has proposed the name adamellite. The rock would also be classified as adamellite using any of the classifications found in modern textbooks and considering the albite as plagioclase.

The available chemical analyses (Table 2) yield more conflicting evidence. Figure 14 shows a comparison of the oxide percentages from Salisbury analysis #3 with Nockolds' (Nockolds, 1954) average adamellite and average peralkaline granite. The low iron content of the Salisbury sample is due to analyst technique. As can be seen, the Salisbury rock is significantly higher in sodium and lower in calcium than the average adamellite. The shape of the curve is nearer that of peralkaline granite than to adamellite. This anomalous chemical composition is probably the result of changes wrought by metamorphism of a rock whose original composition was near that of the average adamellite and is not here considered profound enough to change the rock's classification.

The best name for the rock would seem to be albite adamellite. This name could be confidently applied to most samples from Salisbury. There are certain parts of the pluton, however, in which regional

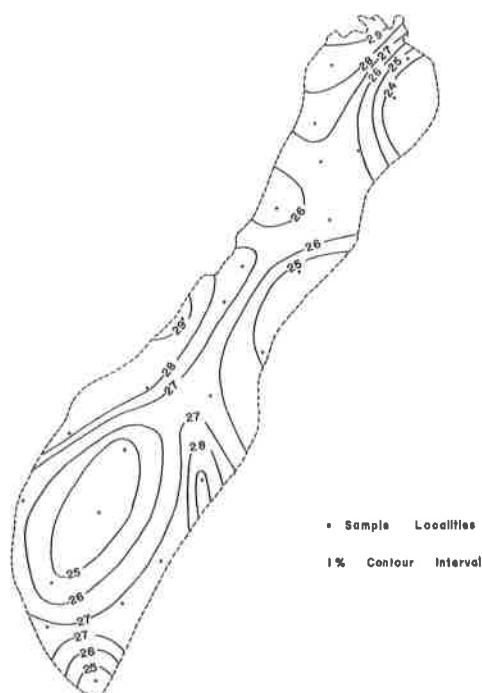


Figure 12. Contour map of potash feldspar variations. Shows results of point count in volume percent.

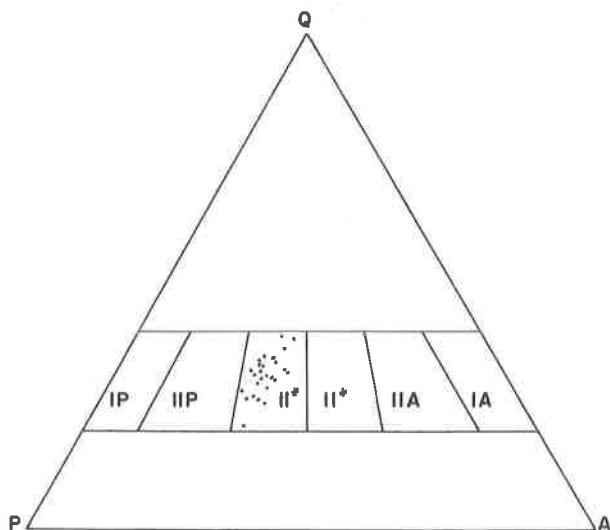


Figure 13. Modes plotted on a triangular diagram with Chayes' (1957) division lines superimposed. Class II* is adammellite.

Table 2. Chemical and Normative Composition of Adamellite.

Sample*	1	2	3
SiO ₂	77.00	76.50	72.80
Al ₂ O ₃	12.70	13.90	16.30
Fe ₂ O ₃	0.23	0.32	1.44
FeO			
MgO	0.69	0.58	0.15
CaO	0.48	0.48	0.83
Na ₂ O	4.16	3.78	5.50
K ₂ O	4.07	3.69	2.69
CO ₂			
TiO ₂	0.07	0.09	
P ₂ O ₅	0.02	0.02	
MnO	0.13	0.18	
SO ₃	0.07	0.05	
H ₂ O	0.09	0.17	0.10
Total	99.71	99.76	99.81
Norms			
Q	35.22	38.51	28.51
Or	24.05	21.82	15.92
Ab	35.19	31.99	46.52
An	2.28	2.31	4.12
C	.61	2.83	2.83
Hy	1.72	1.45	.37
Il	.09	.17	
Py	.10	.07	
Ap	.03	.03	
Hm		.05	1.44

*Sample No. 1 is pink adamellite from Balfour Quarry. Sample No. 2 is grey adamellite from Balfour Quarry. Both of these analyses were done by E. L. Conwell and Company. Sample No. 3 is pink adamellite from Collins-Durax Quarry, the analyst is unknown.

metamorphism has imposed a distinct foliation on the rock. Upon examining such an arbitrarily selected sample most petrologists would probably call it a gneissic granite. The majority of the rock appears to have a granitic texture in hand sample. Under the microscope this is resolved into a mortar or cataclastic texture which only in a few selected samples becomes mylonitic. Therefore the rock comprising the

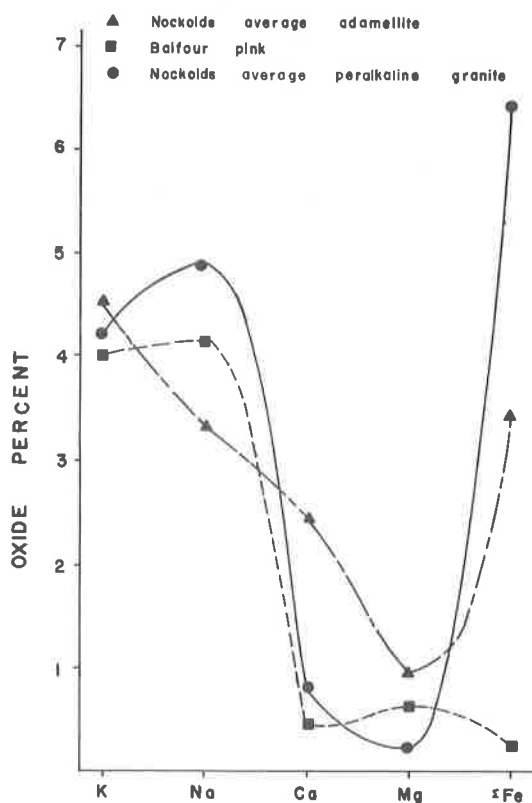


Figure 14. Variation diagram, based on oxide percentages, for Nockolds' average adamellite and average peralkaline granite plus analysis of Balfour Pink.

Salisbury pluton should be classified as an adamellite and given the name albite adamellite.

DISCUSSION

Depth of Emplacement

Buddington (1959) has shown that field relations yield useful information concerning the depth of emplacement of intrusive igneous rocks. Field relations of the Salisbury pluton are instructive in this respect if one keeps in mind the fact that the body has undergone subsequent metamorphism which masked some of the criteria and makes interpretation of others uncertain.

The Salisbury adamellite crops out over an area of slightly less than 18 square miles and is surrounded by a metasedimentary-injection complex. The body as a whole is parallel to the regional strike and only at its northern and southern ends does it have discordant relationships with the country rock (Figure 2). If it is true that regional metamorphism occurred some time after initial intrusion, then it is possible that the pluton was entirely discordant at the time of emplacement.

The adamellite is surrounded by a zone of contact metamorphosed sediments which are revealed only as a deep red saprolite. Large xenoliths are common in the Balfour quarry and one extensive roof pendent was observed. Fine grained rocks indicative of a chill zone were found at the northern and southern extremities of the body. There is no evidence of foliation due to flow within the pluton. Other negative evidence consists of the absence of a zone of migmatite separating the pluton from its country rock and the absence of aplite or pegmatite dikes in the area. All of the above points to emplacement in the epizone.

Differentiation

Contour maps of modal quartz (Figure 11) and plagioclase (Figure 10) show at least four closures, any or all of which could be interpreted as centers of differentiation. These trends can be explained by postulating differentiation as a result of fractional crystallization. Given the size and shape of the pluton it seems unlikely that as many as four centers of differentiation could be achieved. Gravity data (Snyder, 1963, p. 38) shows two closures. These two closures may indicate two centers of injection. If as field evidence seems to indicate, the present level of erosion is reasonably close to the former roof of the intrusion; and if, as the gravity data seem to indicate, the intrusion was a two-pronged affair; then one would reasonably expect two centers of differentiation. Why, then, do the data show four or more closures instead of two? The obvious answer and possibly the correct one is that the sampling technique was inadequate. The pluton should be sampled following the original plan of a 1/2 square mile grid, and more data, including specific gravity, should be obtained from each sample. If these data were obtained and subjected to trend surface analysis, it is probable that the picture which emerged would be less complicated.

The potash feldspar abundance contour map shows one low closure in the southern half of the pluton but is more indistinct in the northern portion (Figure 12). The center of this low corresponds with a plagioclase low and a quartz high. This relationship is not what one would expect under normal conditions of fractional crystallization. Perhaps this trend and some of the complexities in the plagioclase and quartz maps can be attributed to the superimposed metamorphism. The experimental work of P. M. Orville indicates that alkalis may be rather mobile during metamorphism. In his conclusion Orville (1963, p. 235) makes the following statements:

"These experimental studies suggest that alkali metasomatism may take place as a natural consequence of exchange reactions in a rock mass of heterogeneous composition that may also be under the influence of temperature or pressure gradients. . . . During regional metamorphism the vapor phase probably plays an essentially passive role and serves as a medium for diffusion of ions in response to concentration gradients established by reactions between crystalline phases and ions in solution."

The fact that part of the Salisbury pluton has a relict hypidiomorphic-granular texture and part has a cataclastic texture points to at least an energy gradient and possible thermal gradients produced by regional metamorphism. Finally, plagioclase-microcline boundary relationships in thin section indicate some replacement of plagioclase by microcline. Thus the distribution of potash feldspar within the pluton may be the result of regional metamorphism rather than of fractional crystallization.

In conclusion it can only be stated that the data indicate differentiation by means of crystal fractionation, but that the present study is somewhat inconclusive because of insufficient sampling density and late metamorphism.

Metamorphism

General Statement. Apparently the Salisbury albite adamellite pluton was originally emplaced as a magma by means of forceful injection accompanied by some stoping and crystallized as a normal two feldspar adamellite. The pluton underwent regional metamorphism and later kinetic metamorphism some time after its initial emplacement.

Evidence Supporting Magmatic Origin. The following lines of evidence indicates that the pluton originated as an igneous intrusion:

1. The train of xenoliths shown in Figure 4 was apparently controlled by currents in a melt. It seems impossible to explain the off-center position exhibited by one of the xenoliths unless movement in a magma is postulated. This xenolith seems to have been pushed aside by the one behind while both were caught in a current within a melt. Plastic flow would be unable to produce such a phenomena and it seems unlikely that metasomatic replacement could either.
2. Field evidence indicates that the intrusion produced a zone of contact metamorphism surrounding the pluton and a probable chill zone at the contact. Both these phenomena are incompatible with an origin by granitization.
3. Gravity data indicates that the pluton extends to considerable depth.
4. The relict hypidiomorphic-granular texture in parts of the pluton suggests an igneous origin for the rock. However,

this does not rule out granitization.

5. If the modal contour maps indicate valid differentiation trends, they constitute evidence for origin of the pluton as a granitic magma.
6. There is no zone of migmatite around the pluton and the country rocks belong to the greenschist metamorphic facies. Most rock bodies known to have been formed by granitization are surrounded first by migmatites and then metamorphic rocks of a higher facies.

Evidence Supporting Regional Metamorphism. The following evidence indicates that the pluton was subjected to low grade regional metamorphism following its emplacement and subsequent crystallization.

1. The mineral assemblage is characteristic of low-rank metamorphism. All of the second generation minerals; quartz, fibrous muscovite, garnet, and epidote are characteristically metamorphic. The two generations of quartz strongly suggest formation both before and after metamorphism. Albite may also be a low grade metamorphic mineral.
2. The relict hypidiomorphic-granular texture indicates metamorphism of an igneous rock. The fact that such a relict texture has been preserved indicates that equilibrium under metamorphic conditions was never reached.
3. Foliation within the pluton is parallel to the foliation of the country rock. The principal component of this foliation is recrystallized quartz which formed during metamorphism.
4. Exsolution perthite in the rock has been attributed to deformation by Chayes (1952).
5. In thin section the rock reveals predominantly metamorphic characteristics. The dominant texture is cataclastic. No zoned feldspars were observed. There is no sericite or other evidence of late hydrothermal action such as chessboard fabric in albite. There is no myrmekite. The feldspars do not exhibit the late rim albite so common in undisturbed igneous rocks. Many of the expected accessory minerals such as apatite are absent. The only fine-grained intergrowths of minerals consist of either (1) calcite, quartz, opaques, and muscovite, or (2) quartz, garnet, and muscovite; both of which are metamorphic assemblages.
6. The entire pluton exhibits signs of granulation. Every thin section studied contained some type II quartz. This would seem to indicate that the metamorphism was a regional phenomenon rather than resulting from local shear zones.
7. The pink color of the albite is probably the result of metamorphism. There is no direct evidence for this, however, in most magmatic granites the potash feldspar rather than the plagioclase is pink.

8. One of the sections contains plagioclase with inclusions arranged in a manner suggesting former oscillatory zoning in the mineral.
9. Garnet locally exhibits a granoblastic habit.
10. Garnet can be seen to have replaced opaques; this is a metamorphic mineral pseudomorphic after an igneous one.

Evidence Supporting Late Kinetic Metamorphism. The following evidence indicates late kinetic metamorphism concentrated along shear zones and occurring either at the end of regional metamorphism or perhaps during a distinctly later time period.

1. The Balfour Quarry contains a strongly fractured zone about 100 feet wide and striking northeast-southwest.
2. Several of the thin sections show a mylonitic texture resulting in lineation in thin section and a gneissic appearance in hand sample.
3. The minerals making up the mylonitic portion consist of fibrous muscovite, epidote, quartz, and calcite. The quartz is very fine grained, has a long, narrow shape, and the long axes of the grains are aligned. It is distinct from the more common type II quartz which has a slightly larger grain size and is nearly equidimensional.
4. Foliation in the pluton, which is parallel to a shear zone, cuts across xenoliths (Figure 4) and makes an angle with the foliation imposed by regional metamorphism.

It is possible that this evidence merely indicates extremes of regional metamorphism. However, the fact that there are two distinct directions of foliation (Figure 3) seems to indicate late kinetic metamorphism following an earlier period of regional metamorphism.

Hypothetical Geological History of the Pluton

The Salisbury albite adamellite pluton was apparently emplaced by means of forceful injection of a granitic melt into the lower epizonal portion of the earth's crust. Intrusion was accompanied by some stopping, limited contact metamorphism of the bedded sedimentary rocks which comprised the country rock, and development of a chilled margin during initial crystallization. Assimilation of nearly pure quartz sandstone could have been the major cause of the unusually high silica content shown by the chemical analyses of rock from the Balfour Quarry. The body probably crystallized as a normal two feldspar adamellite. During crystallization some differentiation occurred, probably about two centers; although there is also a possibility that the injection was originally a two pronged affair, perhaps two subsequent pulses from the subjacent magma reservoir. Initial emplacement probably took place in early Paleozoic time. The above statement is based on the hypothesis that the pluton has undergone two periods of metamorphism and the fact that three orogenic episodes have been reported in the area (Long,

Kulp, and Eckelmann 1959; Overstreet, Bell, Rose, and Stern, 1961). Therefore, the pluton must have been emplaced during the first episode and affected by the two later ones.

Regional metamorphism of the area probably took place in middle Paleozoic time. During this period the surrounding sedimentary rocks were changed into metamorphic rocks of the greenschist facies and a northeast-southwest foliation was developed in both the pluton and its surrounding rocks. Changes within the pluton as it attempted to adjust to the new pressure-temperature conditions were extremely complicated. Complete adjustment in this new environment may not have been achieved, yet a whole series of mineralogical and textural changes did occur.

The plagioclase component of the adamellite, probably oligoclase-andesine, was converted to albite during this period of metamorphism. Since the work of Bowen (Tuttle and Bowen, 1958) it has been generally agreed that it would be highly improbable to find albite which had crystallized from a granitic melt. In most granitic rocks albite is a minor component and can be ascribed to either exsolution from alkali feldspars or deuteric alteration. Where albite is a major component it has been interpreted as resulting from either assimilation of high soda sediments by a magma or massive hydrothermal alteration by fluids from a subjacent source. Field relations indicate only minor assimilation by the Salisbury magma, and the adamellite contains none of the textural criteria of hydrothermal alteration such as myrmekite and albite with a chessboard texture. Since these two possibilities can be ruled out the albite must have formed during metamorphism. Albite is the normal plagioclase expected in metamorphic rocks of the greenschist facies. In thin section the plagioclase laths are seen to be bent, fractured, broken and partially reoriented, or even slightly granulated; all seemingly the result of strain in the solid state. In short, the plagioclase along with the type I quartz exhibit signs of deformation in the solid; neither of them reached a liquid state. Therefore formation of the albite must have occurred in a solid state. It consisted primarily of diffusion of the calcium ions away from the feldspar lattice. There is some evidence in the form of late fine-grained intergrowths that fluid phases were active during this time. There may have acted as an avenue of escape for the calcium ions once they left the feldspar lattice. Other changes which occurred in the plagioclase concomitantly with escape of the calcium ions include obliteration of oscillatory zoning by the development of secondary polysynthetic albite twins. The albite twinning in these rocks seems to conform with Vance's criteria for secondary growth. According to Vance (1961, p. 1108) some of the characteristics of secondary glide twinning in plagioclase are:

"These lamellae often terminate at cracks within the crystal and fail to match in either number, width, or relative distribution across these cracks... secondary lamellae are genetically related to an evident bending or twisting of the crystal such that

the lamellae are localized and most broadly developed in the areas of greatest strain while they pinch out or are only scantily developed in the unstrained or more weakly deformed parts of the crystal.... These glide lamellae usually taper to very long, fine points which commonly are bent..."

Both Vance, and Emmons and Mann (1953) have shown that the development of such secondary polysynthetic twinning resulted in the obliteration of oscillatory zoning in plagioclase feldspar. Most of the muscovite inclusions found in the albite must have formed at this time. It is also quite probable that the plagioclase acquired its pink color during metamorphism. The color is imparted to the feldspar by very fine opaque inclusions. They probably represent iron exsolved from the plagioclase during metamorphism and oxidized to hematite.

Potash feldspar seems to have been relatively mobile during metamorphism. Thin sections show a replacement relationship between microcline and albite and it is possible that this replacement took place as reaction between two essentially solid phases at this time. Modal data show trends in the potash feldspar content contrary to those expected during differentiation. This anomaly could be explained by postulating mobility of potash feldspar during metamorphism.

Quartz was affected by metamorphism more than any other essential mineral. Every relict quartz grain shows extreme undulatory extinction. In places only small portions of the quartz have been recrystallized, resulting in deeply sutured contacts between large quartz grains and a few grains of fine-grained type II quartz along the boundaries of the relict quartz crystals. In other sections the quartz appears to have completely recrystallized. The type II quartz is believed to have been mobile during metamorphism. Field evidence indicates that the quartz filled every available opening in the pluton.

The last event to have any significant affect on the pluton was minor kinetic metamorphism. This could have happened either near the close of the period of regional metamorphism or at a distinctly later date. The rock affected, developed a mylonitic texture. Type II quartz became oriented; a reaction which took place essentially in a solid. Some of the plagioclase was granulated and fibrous muscovite and epidote formed at this time. The obvious foliation near the shear zones resulted from alignment of these minerals.

Intrusion of small bodies of gabbro, diorite, and granite into the metasedimentary rocks took place either during the close of regional metamorphism or at a later time. Examination of one thin section from a gabbro just south of the pluton shows no signs of regional metamorphism.

During Triassic time a series of diabase dikes invaded the area. Some of these were found near the edge of the pluton but none were found within it.

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OUTCROP OF THE YORKTOWN FORMATION (UPPER MIOCENE)

IN ONSLOW BAY, NORTH CAROLINA

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ABSTRACT

The Yorktown Formation (upper Miocene) occurs at a depth of 13 feet below the surface in a boring on Frying Pan Shoals, North Carolina. Dredge samples from Onslow Bay and Long Bay, North Carolina, were compared with samples from the boring on Frying Pan Shoals in order to locate possible outcrops of the Yorktown Formation in these areas.

Samples from Onslow Bay are mostly well-sorted quartz sands containing much calcareous shell material and small amounts of phosphorite. One sample contains unworn Miocene shark teeth, is nearly identical lithologically and texturally to samples of the phosphoritic Zone 1 of the Yorktown in the boring on Frying Pan Shoals, and indicates the presence of a small outcrop of the Yorktown.

Four other samples from Onslow Bay contain large phosphorite pebbles and have sand-size fractions lithologically similar to outcrops of the Yorktown Formation (Zone 1) of the North Carolina Coastal Plain. The locations of these samples may be near other small Yorktown outcrops.

Nearly all samples from Long Bay are non-phosphoritic. The presence of phosphorite in sediments in Onslow Bay and its almost total absence in Long Bay is in accordance with seismic structural data indicating that Miocene sediments should crop out in Onslow Bay but do not in Long Bay because of erosion or overlap by Pliocene sediments.

A cross-section of the Miocene under the floor of Onslow Bay, constructed on the basis of a compilation of structural and drilling data, shows that a 10-15 mile-wide, NE-SW band of the phosphoritic Zone 1 of the Yorktown Formation lies beneath a discontinuous veneer of Recent or relict Pleistocene sediments in Onslow Bay. Most of the phosphorite in surface sediments of Onslow Bay was probably derived from sporadic small outcrops of the Yorktown Formation (Zone 1) in the bay rather than from Miocene outcrops on land.

INTRODUCTION

Phosphorite is not an uncommon component of Continental Shelf sediments off the Southeastern United States, especially off Georgia (Pevear and Pilkey, 1966) and in Onslow Bay, North Carolina (Luternauer and Pilkey, in press). The origin of the phosphorite is a source of speculation although the consensus has been that it was eroded from Miocene and other Tertiary formations of the Coastal Plain by rivers during Pleistocene time (Gorsline, 1963, p. 431-432; Pevear and Pilkey, 1966, p. 855).

In the process of examining cuttings from a boring on Frying Pan Shoals, off Cape Fear, North Carolina (Figure 1), the top of the Yorktown Formation (upper Miocene) was found at a depth of 13 feet underneath a shell limestone of the Pamlico Formation (late Pleistocene). Water depth was 46 feet at the location of the boring.

The discovery of Miocene sediments so near the surface led to the speculation that phosphoritic Miocene sediments may crop out on the floor of Onslow Bay. Reworking of these outcrops could be the source of phosphorite in the sediments of Onslow Bay rather than erosion of formations cropping out on the Coastal Plain. A comparison of dredge samples from Onslow Bay and Long Bay (Figure 1) with samples of the Yorktown Formation from the boring on Frying Pan Shoals has been undertaken in order to test this hypothesis.

Stetson (1938), Gorsline (1963), Uchupi (1963), Pilkey (1963; 1964), Pevear and Pilkey (1966) and Luternauer and Pilkey (in press), have reported on aspects of sedimentation of the Continental Shelf off the Atlantic Coast of the Southeastern United States. These studies show that most of the shelf off the Carolinas is covered by relict (Pleistocene), well-sorted quartz sand containing much calcareous shell material and in Onslow Bay containing small percentages of phosphorite grains. Contemporary deposition is probably represented by a near-shore band of clean, well-sorted quartz sand 10-15 miles wide whereas the Continental Shelf is 50-70 miles wide in the area studied.

Acknowledgments

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T. J. Conomos provided valuable criticism on the manuscript.

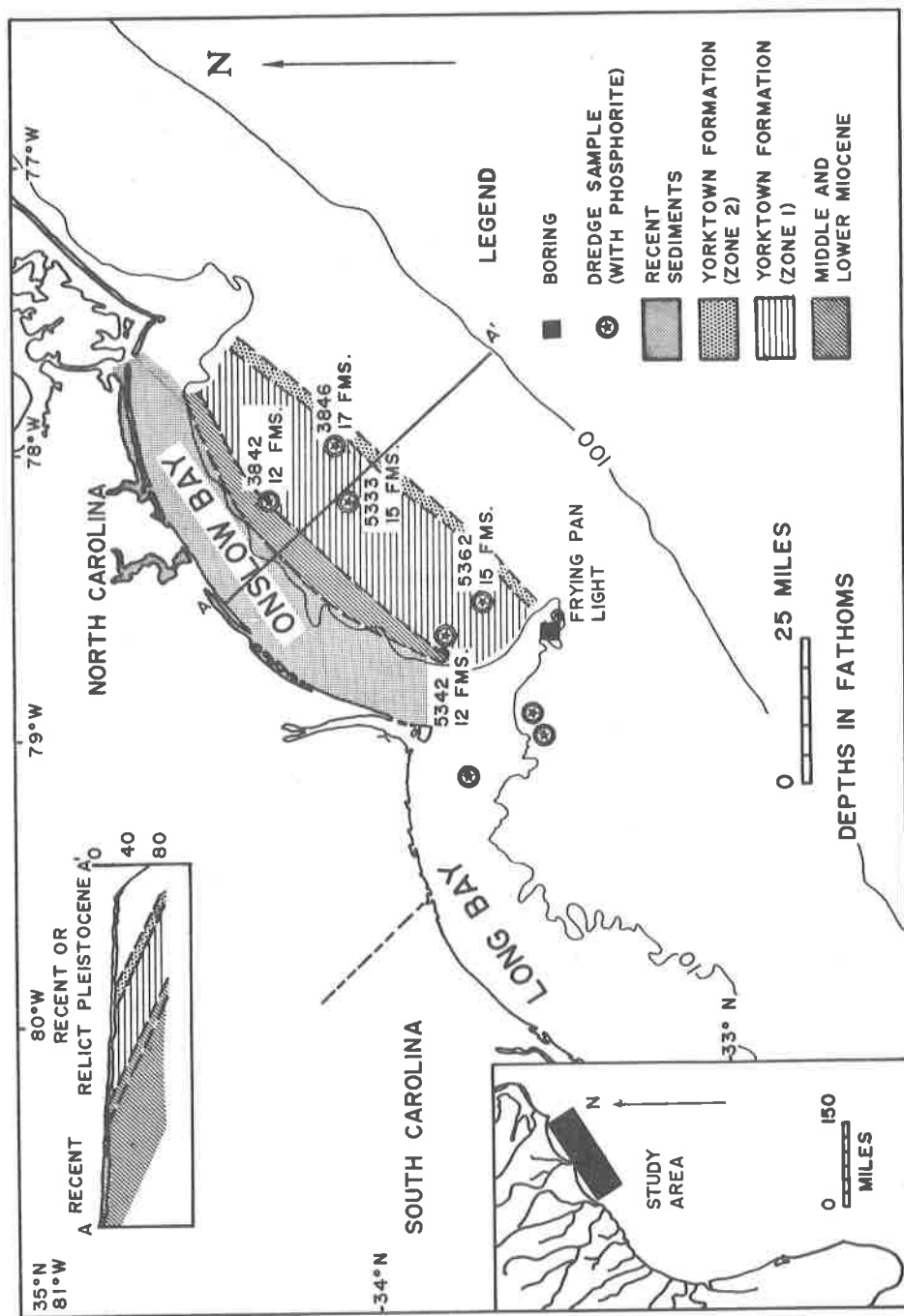


Figure 1. Locations of dredge and boring samples showing idealized geologic contacts. Inset: structure section A-A' based on seismic and drilling data.

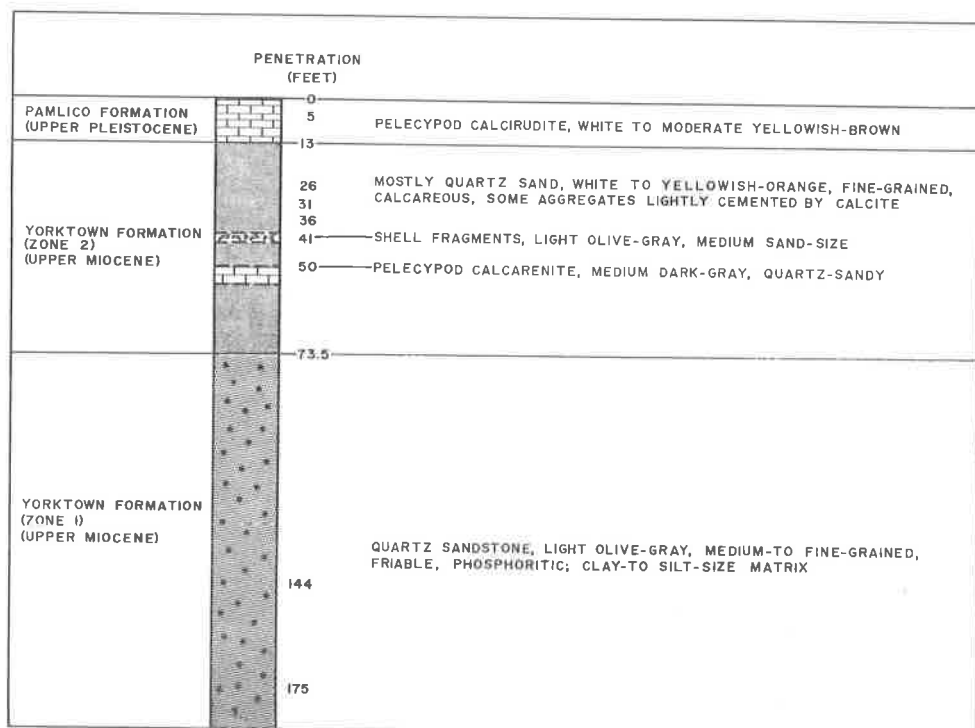


Figure 2. Stratigraphic column and summary lithologic description of samples of boring on Frying Pan Shoals off Cape Fear, North Carolina. Sample depths (in feet) from top of boring (not water surface).

METHODS

Twelve samples of a boring, made in 1962, on Frying Pan Shoals, by McClelland Engineers, Inc., under contracts to the U. S. Coast Guard, and 75 bucket dredge samples collected in 1965-66 from Onslow Bay and Long Bay (Figure 1) by O. H. Pilkey were examined under a binocular microscope. The dredge samples were compared with samples of the Yorktown Formation penetrated in the boring on Frying Pan Shoals (Figure 2) and with descriptions of the Tertiary formations of the southeastern Atlantic Coastal Plain (Richards, 1950; Swain, 1951; Malde, 1959; Darby and Hoyt, 1964; and Rooney and Kerr, 1964).

Sample color was determined by comparison with the GSA Rock-color Chart. Grain sizes were estimated by comparisons of samples with prepared slides containing standard size classes (according to Wentworth, 1922). Phosphorite was distinguished from other minerals by the standard spot test and the amount of phosphorite (by volume) was estimated by use of comparison charts (Terry and Chillingar, 1955).

RESULTS

The samples from the borings near Frying Pan Shoals Light are lithologically similar to the descriptions of Richards (1950, p. 22-27; 39-40) for the Yorktown Formation (upper Miocene) and the Pamlico Formation (late Pleistocene) of the North Carolina Coastal Plain (Figure 2).

Most of the samples from Onslow Bay are well-sorted, fine- to coarse-grained quartz sands containing appreciable amounts of white, brown, and gray calcareous shell fragments and gray, rounded, calcareous detrital particles with some (1-5 percent) amber to black, very fine- to medium-sand size phosphorite grains. Five samples (3842, 3846, 5333, 5342, and 5362), however, differ markedly from most samples in Onslow Bay (Figure 1). Sample 5342 consists of a mixture of small (1-2 cm), friable aggregates and loose, sand-size grains. The aggregates are light olive-gray, very fine to fine, subangular to subrounded, moderately well-sorted, quartz sand, in a matrix of clay and silt, containing 1-2 percent of very fine to fine, black phosphorite grains and a few Foraminifera. The loose material consists of disaggregated constituents of the aggregates; many coarse, subangular, quartz sand grains; calcareous shell fragments; several black phosphorite pebbles, and relatively unworn specimens of shark teeth identified as Carcharodon auriculatus, Carcharhinus (=Prionodon) egertoni, and Hemipristis serra (Dunkle, 1967, personal communication). The aggregates in this sample are indistinguishable from the aggregates in the cuttings of the Yorktown Formation penetrated at a depth of 144 feet by the boring on Frying Pan Shoals (Figure 2).

Samples 3842, 3846, 5333, and 5362 are poorly-sorted mixtures of light olive-gray to brownish-gray, fine to coarse, subangular to subrounded, quartz sand, fine sand- to pebble-size black phosphorite grains, and many calcareous shell fragments up to 2 cm long. The fine sand-size fractions of these samples are lithologically quite similar to the Frying Pan Shoals borings of the Yorktown Formation (Zone 1), except that the silt- or clay-size material has been winnowed out. The gross characteristics of the samples appear to be lithologically similar to descriptions of the Yorktown Formation (Zone 1) of the North Carolina Coastal Plain (Richards, 1950, p. 23-25).

Samples from Long Bay contain very little to no identifiable phosphorite. Very small amounts of phosphorite (less than 1 percent), were found in three samples (Figure 1) near Frying Pan Shoals and apparently were transported by bottom currents from nearby phosphorite-bearing sediments in Onslow Bay.

Samples from Long Bay show no significant lithologic similarities to samples from the borings in the Yorktown Formation on Frying Pan Shoals or to descriptions of North Carolina Coastal Plain sediments.

DISCUSSION

Seismic and drilling studies indicate that Miocene sediments underlie the Continental Shelf at shallow depth between North Carolina and Florida (Swain, 1951; Woolard, Bonini, and Meyer, 1957; Antoine and Henry, 1965; and JOIDES, 1965). Offshore seismic structural profiles across the Cape Fear Arch show that Miocene sediments may crop out in Onslow Bay but probably are covered by Pliocene sediments or have been removed by erosion in Long Bay (Woolard, *et al*, 1957, Figure 7 and 8).

Phosphorite is most notably abundant in the Miocene sediments (Pevear and Pilkey, 1966, p. 855) although it occurs in strata in many of the Tertiary formations of the southeastern Atlantic Coastal Plain (Mansfield, 1940; Richards, 1950; Malde, 1959; Adams, Groot, and Hiller, 1961; Herrick, 1961; and Rooney and Kerr, 1965). This, together with the structural evidence that the Miocene may crop out in Onslow Bay only and the lack of Miocene outcrops on the Coastal Plain adjacent to Long Bay (Pevear and Pilkey, 1966, Figure 4) can explain the occurrence of phosphorite in Onslow Bay and its almost total absence in Long Bay.

The small aggregates of clayey to silty, slightly phosphoritic, fine quartz sand of sample 5342 (Figure 1) are nearly identical lithologically to the sample of the Yorktown Formation at the 144-foot level of the boring on Frying Pan Shoals. Relatively unworn shark teeth, found in sample 5342, are of Miocene age and have been found in several Miocene formations of the Atlantic Coastal Plain, including the Calvert, St. Marys, Choptank, and Yorktown (Dunkle, 1967, personal communication). The lithologic characteristics of sample 5342 and its included Miocene shark teeth indicate that this sample was collected from very near an outcrop of the Yorktown Formation (Zone 1). The outcrop represented by sample 5342 is probably very small, as samples dredged from locations as close as 1.5 miles from this sample show no similarities to the boring samples or to descriptions of Miocene Coastal Plain sediments. According to Cleary (1967), a thin discontinuous veneer of sediment covers lithified bedrock in Onslow Bay.

The presence of phosphorite pebbles in samples 3842, 3846, 5333, and 5362 indicates that the locations of these samples may be near other small outcrops of the Yorktown Formation (Zone 1).

The boring on Frying Pan Shoals does not penetrate to the bottom of the phosphoritic Zone 1 of the Yorktown Formation (Figure 2). The exact thickness of Zone 1 also is not known on the North Carolina Coastal Plain, although a thickness of 150 feet has been reported (Richards, 1950, p. 23). Utilization of structural and drilling data (Swain, 1951, p. 9-10; Woolard, *et al*, 1957, Figures 7-11; this report Figure 2) permits construction of a structure section of the Yorktown Formation in Onslow Bay (Figure 1). This together with the location of Sample 5342 (Figure 1) indicates that Zone 1 of the Yorktown may be as

much as 250 feet thick in the Onslow Bay area. An idealized "outcrop pattern" of the phosphorite-bearing Zone 1 has been drawn on the basis of these data (Figure 1).

The idealized "outcrop pattern" shows the area in which the Miocene is either exposed or covered by a veneer of Recent or relict sediments. The dredge samples indicate that the actual area of outcrop is small. Actual outcrops are probably limited to areas contiguous to the numbered sample localities although other outcrops probably exist.

The areal extent of the possible outcrop pattern of Zone 1 of the Yorktown in Onslow Bay indicates that it may be the origin of a major portion of the phosphorite of surface sediments in this area. Probably only a small proportion of the phosphorite in Onslow Bay was eroded from outcrops on the Coastal Plain by rivers during Pleistocene time.

CONCLUSIONS

(1) One dredge sample collected in Onslow Bay, North Carolina represents a small outcrop of the upper Miocene Yorktown Formation (Zone 1).

(2) Four other dredge samples in Onslow Bay were collected near other small outcrops of the Yorktown Formation (Zone 1).

(3) A 10-15 mile-wide band of the phosphoritic Zone 1 of the Yorktown Formation on the floor of Onslow Bay is covered by a discontinuous veneer of Recent or relict Pleistocene sediments. Sporadic outcrops of Zone 1 have probably contributed most of the phosphorite of sediments in this area.

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THE EFFECT OF MINOR CHANGES OF EMISSION WAVELENGTH IN QUANTITATIVE X-RAY ANALYSIS

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ABSTRACT

A small difference in the x-ray emission wavelength of a sample and a comparison standard may seriously affect the analytical accuracy. This is demonstrated theoretically and experimentally for changes of the order of $\pm 0.0020 \text{ \AA}$. An experimental method is suggested for compensating wavelength differences of less than 0.0030 \AA .

* * * * *

In x-ray fluorescent and electron microprobe chemical analysis, it is necessary to compare the x-ray intensity of an element in a sample under consideration to the intensity emitted from a standard material of known composition. In most cases an intensity ratio of the sample relative to the standard is used. This normalizes the determinations and avoids the problem of minor instrumental variations. The best standard is generally one with a physical and chemical composition similar to that of the sample. However, it is shown below that even in this procedure, poor analytical accuracy may be obtained.

In certain mineral systems it is impossible to obtain mineral standards for electron probe microanalysis, because of extensive solid solution, exsolution, and common disequilibrium crystal growth. This results in zoning on a fine scale and/or chemically heterogeneity, e. g., in transition metal arsenides. It is sometimes not possible to produce solid grains synthetically which are large or solid enough for electron probe microanalysis. This is generally true in sulphide systems, although glasses are extremely useful in silicate and oxide systems. It may be necessary to use a pure metal as the comparison standard. This procedure often gives poor analytical accuracy, primarily due to differences between mass absorption coefficients in standard and unknown.

It is known that the absolute wavelength of an element for an x-ray emission wavelength varies with coordination number (Koffman and Moll, 1966). It is also known that the emission wavelength of an element in an ionic crystal lattice is slightly different from the x-ray

emission of pure metal (Theisen, 1966). The wavelength of an element in chemical combination can be measured relative to the pure metal and reported as a peak shift or difference of wavelength, and the exact values of the wavelength need not be known. Theisen (1966) states that peaks shifts of this nature cannot be detected between metals and the same element in either metallic or covalent bonded states. However, peak shifts of the order of $+ 0.002 \text{ \AA}$ have been measured in transitional metal arsenide minerals (Radcliffe, 1966) using a new technique and shown to affect seriously the analytical accuracy.

It is possible to demonstrate theoretically the effect of minor peak shifts. Figure 1 shows the peak apices of a sample containing 33 percent of an element relative to a standard metal, for three different peak shifts. The intensities over the peak apices are assumed to adhere to a normal frequency distribution and given a standard deviation value of $+ 0.002 \text{ \AA}$. With this standard deviation, the morphology of the peaks are very similar to those obtained experimentally when they are plotted on the same scale. Both the sample and the standard emission peaks were plotted with the same standard deviation, as the peak widths are controlled by the x-ray optical system and both of the x-ray emissions occur essentially over the same angular interval.

Of the three situations illustrated in Figure 1, A represents no peak shift and it can be seen that the same intensity ratio is obtained anywhere close to the peak maxima. However, B shows a minor peak shift relative to the standard of $+ 0.005 \text{ \AA}$. In this case a varying intensity ratio is obtained at different points close to the peak, and in C, the situation for a peak shift of $- 0.0010 \text{ \AA}$.

In Figure 1 is shown that there is a linear variation of the intensity ratio close to the peak maxima for minor peak shifts and the slope of this line is dependent on the amount of the peak shift relative to the standard. It is not possible during analysis to reproduce any one peak position to within a minimum of $+ 0.0005 \text{ \AA}$ due to the gear slap of the spectrometers and the difficulty of finding the exact peak maxima at this fine scale. Thus in case C an accuracy error of at least 3 percent would be obtained. If, during analysis, an intensity ratio was obtained using the sample emission peak one time, and the standard metal emission peak second time, a minimum accuracy error of 6 percent is obtained. It is thus possible to understand why electron probe analysis and to a lesser extent x-ray fluorescent analysis in mineral systems may only total 90-95 percent and are irreproducible, even with the best correction factors.

This measuring problem can be overcome by taking a series of intensity ratios at symmetrically different wavelengths before, after, and in between the two peaks. The mean of these results is the correct answer as long as a symmetrical series of readings are used and the peak shifts are small enough (less than 0.003 \AA) so that a linear array exists. The method of taking these readings is to advance carefully the spectrometer in small intervals over the two peaks, in the same

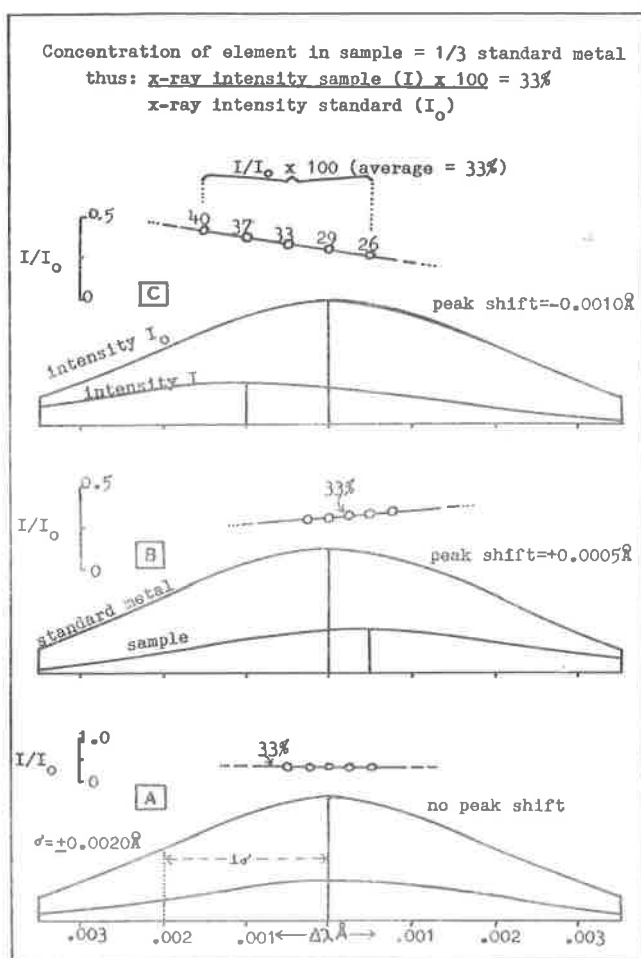


Figure 1. The effect of minor peak shifts on intensity ratio.

positive direction without reversing the spectrometer gears, and measure the intensity ratio of sample and standard at each interval. This procedure is quite rapid and reproducible if the standard and unknown are in the same mount.

During a preliminary electron probe analysis by Radcliffe (1966), sample 1B16 from Mt. Wright Range, Quebec, was used as a test sample. It was used because it proved to be unusually homogeneous. A statistically satisfactory number of readings (50 of each) was obtained for K α emissions by continuously re-measuring the Co, Fe, and As contents in sequence over the period of a few days. Pearson's coefficient (Mills, 1955) was used as a measure of the spread of the apparent

weight percents:

$$\text{Pearson's coefficient (P)} = \frac{\sum[(\Delta X)^2]}{N} / \bar{X}$$

where \bar{X} = mean intensity ratio, X = difference from the mean, and N = number of measurements. The following results were obtained:

	P%	Co	Fe	As
Peak Shift ($\times 10^{-4}$ Å)	=	3.8	1.7	1.6
	=	+17	0	-2

The analytical precision of Co is much poorer than that of Fe and As, and these results may be compared with the peak shifts. Thus Fe and As have the same relative negligible peak shift and the same effective analytical precision, whereas the peak shift for Co is +0.0017 Å. However, after Co was measured in the manner described above for compensating the effects of minor peak shifts, the analytical precision was reduced to 1.5 percent which is comparable to that of Fe and As.

In order to ascertain the effect of variable compositions on peak shifts, Co, Fe, Ni, and As, were measured in 11 samples (Table 1). Co, Ni, and As, are most affected and Fe least. There is also a tendency for an increase (positive) rather than a decrease (negative) of the x-ray emission wavelength for transition metals in covalently bonded arsenide atomic structures compared to the same metal in the metallic state. The apparent randomness of values is significant, since even if a material of similar physical and chemical composition is used as a comparison standard, a poor analytical accuracy could be still obtained. This is true especially for sulphides using sulphide standards. Koffman and Moll (1966) demonstrated little variation of the silicon emission spectra in some common silicates, and this effect may not be significant in ionic compounds.

Table 1. Peak Shifts and Mole Percent Compositions of Natural Transition Metal Arsenides $\Delta \lambda = Y \times 10^{-4}$ Å.

Catalog #	Co(Y)	Fe(Y)	Ni(Y)	As(Y)	Co	Fe	Ni
1B16	17	0	--	-2	32.6	67.4	--
1B30	3	4	--	4	15.4	84.6	--
1B31	4	2	--	-2	72.4	27.6	--
1B33	25	0	8	-8	7.6	0.4	92.0
1B34	6	11	--	12	64.2	35.8	--
1B42	23	0	0	5	62.9	31.4	5.7
1B55	-8	-17	-26	14	60.0	33.4	6.6
1B64	0	--	2	17	3.2	--	96.8
1B65	--	-2	--	0	--	100.0	--
1B72	22	0	--	14	94.0	6.0	--
1C07	-37	--	-19	7	0.7	--	99.3

The reason for the apparent randomness of the peak shifts with variable compositions is peculiar as the type and concentration of an element in an atomic lattice should have some influence on the change of x-ray emission wavelength. One can introduce a variety of theories to explain this discrepancy but a tenable hypothesis could be based on crystallographic orientation. Transitional metal diarsenides are characterized by the orthorhombic marcasite crystal structure involving an approximate 50 percent compression of the metal-arsenic octahedra parallel to the c crystallographic axis. According to Hulliger and Mooser (1965) such structures are characterized by "Jahn Teller" distortions in which the metal-arsenic electronic configuration is quite asymmetric.

In this explanation it is possible to envisage differential bonding energies of the electrons with varying crystallographic orientation. Thus the necessary x-ray excitation energy also varies with crystallographic orientation. In this manner the mean statistical emission wavelength could be slightly variable, depending on the mutual orientation of the crystallographic lattice and the impinging electron beam.

Whatever the explanation, it is a complex interaction of the excitation source, excitation depth in the sample, and mean bonding energies of the electron clouds surrounding the nuclei of the sample under consideration. This whole effect was demonstrated by microprobe studies on the easily absorbed AsL α 1 emission line using a K.A.P. diffracting crystal. Thus the relative value of the peak shifts vary in a regular manner with an increasing acceleration voltage of the exciting electron beam:

KV	Peak Shift (\AA)
20	+0.0010
15	-0.0001
10	-0.0005

These measurements were made on the same sample at approximately, but not exactly, the same point. This is an unusual phenomenon and as yet no fully satisfactory explanation is forthcoming.

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VIRGINIA METAMICT MINERALS:

X-RAY STUDY OF FERGUSONITE

by

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ABSTRACT

Fergusonite occurrences in Amelia, Amherst, and Bedford Counties, Virginia, are briefly described. Although the mineral is metamict, it can be recrystallized by heating in air. At temperatures approaching 700°C a tetragonal phase forms which has $\underline{a} = 5.16 \text{ \AA}$ and $\underline{c} = 10.95 \text{ \AA}$. Above 700°C a monoclinic phase begins to develop. A pure monoclinic phase, with $\underline{a} = 5.08 \text{ \AA}$, $\underline{b} = 10.97 \text{ \AA}$, $\underline{c} = 5.33 \text{ \AA}$, and $\beta = 94^{\circ} 30'$, was formed at 1000°C after heating one hour. Indexed X-ray powder data are given for both phases. A comprehensive survey of earlier studies of fergusonite was made in order to clarify the numerous discrepancies in X-ray data which exist in the literature.

INTRODUCTION

Recent X-ray studies of heat-treated metamict minerals which occur in Virginia have verified two known occurrences of fergusonite, and have shown two additional localities for the mineral. The aim of this paper is to present reliable X-ray powder data for Virginia fergusonite, and to summarize the occurrences of the mineral. A comprehensive literature survey of X-ray studies of fergusonite is also given in order to clarify the discrepancies in data which exist in published reports.

Acknowledgments

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HISTORY OF X-RAY STUDIES OF FERGUSONITE

Although X-ray powder data for heat-treated metamict fergusonite were published as early as four decades ago by Barth (1926),

there has been much confusion since that time about the crystallography of the mineral. Barth (1926) considered fergusonite to be tetragonal, and indexed his data accordingly. Berman (1952) reported the mineral to be dimorphous depending upon the temperature at which it is recrystallized. At about 400° C Berman (1952) obtained a pattern which he indexed with orthorhombic symmetry. Later he showed this to be a tetragonal phase (Berman, 1955). At temperatures from 850° C to 1050° C he obtained a pattern identical to the Barth (1926) structure, which was considered to be a second tetragonal phase. Pellas (1954) who studied a nonmetamict fergusonite from Japan obtained a tetragonal pattern from the unheated material as well as nearly an identical tetragonal pattern for materials heated to 1000° C. He found no indication of two phases for the mineral. Ferguson (1957) from a study of a fergusonite crystal synthesized at approximately 2100° C reported a monoclinic symmetry and concluded the earlier determinations that the mineral is tetragonal were incorrect. Komkov (1957), in studying a non-metamict fergusonite, showed the material is dimorphous. He published indexed powder data for a low-temperature tetragonal structure, and also indexed powder data for a higher temperature monoclinic structure. On heating the change from tetragonal to monoclinic started at about 750° C and was complete at about 900° C. Nearly identical results were reported by Kitadai (1961) who published partially indexed tetragonal data for the low temperature phase but evidently did not recognize the monoclinic nature of the other phase. Komkov (1959a) also studied various synthetic rare earth analogs of monoclinic fergusonite, and reported variations in cell size and shape as a function of the rare earth element present. In a similar way Rooksby and White (1963) studied the synthetic tetragonal and monoclinic rare earth analogs of fergusonite and reported the temperatures of transformation for these from the tetragonal to monoclinic forms. The structures of the tetragonal and monoclinic phases were worked out by Komkov (1959b). Gorzhevskaya and others (1963) found natural nonmetamict monoclinic fergusonite, and called the natural tetragonal form α -fergusonite, and the natural monoclinic form β -fergusonite.

Knowing that both tetragonal and monoclinic fergusonite structures exist, it is now possible to see that Barth's (1926) "tetragonal" data are in reality identical to the true monoclinic data. Ferguson (1957) attempted to explain Barth's misinterpretation of the data by relating the geometry of the monoclinic cell to Barth's incorrect cell. The "tetragonal" data of Berman (1955) for material heated to about 1200° C is also monoclinic with a few tetragonal lines. His data for samples heated at about 700° C are for the tetragonal phase as he stated. The tetragonal data reported by Pellas (1954) are for the true tetragonal phase. It is strange, however, that two of his samples heated to 1000° C should yield only the tetragonal powder data without any monoclinic lines.

In addition to the studies mentioned above, other writers have given unindexed powder data for fergusonite. Watters and others (1961) listed interplanar spacings which match monoclinic data. Soboleva and Pudovkina (1961, p. 322) gave values which represent tetragonal and monoclinic patterns. Petrova and others (1963) also gave patterns for both phases.

Numerous differential thermal analyses of fergusonite, including those of Kerr and Holland (1951), Ukai (1955), Kurath (1957), Soboleva and Pudovkina (1961, p. 322), and Petrova and others (1963), have shown a distinct exothermic peak between about 600° C and 750° C which probably is a function of the change from one phase to the other. Variations between samples probably are due to different degrees of metamictization of the materials as well as differences in compositions.

X-RAY DATA FOR VIRGINIA FERGUSONITE

X-ray powder photographs of all unheated Virginia samples showed no reflections, a condition typical for amorphous metamict substances. Samples heated in air showed either tetragonal or monoclinic symmetry or mixtures of both. Portions of a specimen from Little Friar Mountain, Amherst County (V3035d), were heated in air an hour each at different temperatures. The following results were obtained: 200° C, amorphous; 400° C, two tetragonal fergusonite lines, very weak; 600° C, several tetragonal fergusonite lines, faint and broad; 700° C, tetragonal fergusonite lines, sharp and distinct; 800° C, tetragonal and monoclinic fergusonite; 900° C, monoclinic fergusonite with two very weak tetragonal fergusonite lines; 1000° C, monoclinic fergusonite pattern. Samples from each of the other localities, heated at different temperatures for one hour gave nearly identical results. Samples from the Rutherford pegmatite, Amelia County (V3016d), heated at 600° C for 6 hours, showed a few very diffuse monoclinic lines in the tetragonal pattern; another heated at 900° C for 8 hours showed evidence of minor tetragonal lines in a monoclinic pattern. These observations compare well with the literature summarized above concerning the temperature at which the monoclinic phase forms, especially with the results of Komkov (1957), Berman (1952, 1955), and Kitadai (1961).

Indexed X-ray data for the tetragonal structure, obtained from Virginia specimens, are given in Table 1. These data represent averaged values derived from the measurements of five nearly identical films made in two cameras (11.46 cm diameter, CuK α radiation). All possible calculated interplanar spacings allowed by the 14₁/a space group (Komkov, 1959b) are reported through 1.37 Å. The cell size used is $a = 5.16$ Å, $c = 10.95$ Å, $c/a = 2.122$. The values in Table 1 correspond well with indexed values reported by Komkov (1957) for nonmetamict material, and by Kitadai (1961). The latter one, however, has

Table 1. X-ray Powder Data for Tetragonal Phase of Virginia Fergu-
sonite. Filtered Copper Radiation.

hkl	d (calc.) Å	d (meas.) Å	I (obs.)
101	4.67		
112	3.04	3.05	vvv
103	2.98		
004	2.74	2.74	m
200	2.58	2.58	m+
202	2.33		
211	2.26		
114	2.19	2.19	vvw
105	2.02		
213	1.95		
204	1.88	1.88	s
220	1.82		
222	1.73		
211	1.70		
116	1.63	1.64	w
215	1.59		
312	1.57	1.57	w
213	1.56		
224	1.52	1.52	vvw-
107	1.50		
206	1.49		
321	1.42		
314	1.41		
008	1.37	1.37	vvw
		1.29	vvw
		1.21	vw
		1.09	vvw-
		1.05	vvw

different indices because he used a c value of about half the one given here.

The monoclinic phase, of the Virginia material, is represented by the indexed X-ray data in Table 2. Measurements of five nearly identical films made in two cameras (11.46 cm diameter, CuK α radiation) were averaged. All possible calculated interplanar spacings allowed by the 12 space group (Komkov, 1959b) are reported through 1.77 Å. The cell used has a = 5.08 Å, b = 10.97 Å, c = 5.33 Å,

Table 2. X-ray Powder Data for Monoclinic Phase of Virginia Fergu-
sonite. Filtered Copper Radiation.

hkl	d (calc.) Å	d (meas.) Å	I (obs.)
020	5.49	5.51	vvw
011	4.78	4.75	vvw
110	4.60		
$\bar{1}01$	3.82		
101	3.53		
$\bar{1}21$	3.13	3.13	vs+
031	3.01		
121	2.97	2.97	vs
130	2.96		
040	2.74	2.74	m
002	2.66	2.65	mw
200	2.53	2.53	w
022	2.39		
$\bar{1}12$	2.37		
$\bar{2}11$	2.31		
220	2.30		
112, $\bar{1}41$	2.23	2.22	vvw
211	2.18		
141	2.17	2.17	w
051, $\bar{1}32$	2.03}	2.02	vw
150	2.01}		
$\bar{2}31$	1.98		
132	1.93		
$\bar{2}02$, 042	1.91	1.91	ms
231	1.90		
240	1.86	1.86	m
060	1.83	1.82	vvw
$\bar{2}22$	1.80		
202	1.77	1.76	mw
		1.65	mw
		1.63	mw
		1.57	mw
		1.51	w
		1.48	vw
		1.37	vw
		1.32	vvw
		1.29	vvw

$a:b:c = 0.463:1:0.486$, and $\beta = 94^\circ 30'$. The writer used the orientation of Komkov (1957) and Gorzhevskaya and others (1963), who described natural monoclinic fergusonite, rather than that of Ferguson (1957) and Rooksby and White (1963) who studied synthetic materials. The data of Table 2 closely match indexed values reported by Komkov (1957) and Ferguson (1957).

VIRGINIA FERGUSONITE OCCURRENCES

Fergusonite from the four localities described here was verified by X-ray studies of heat-treated samples. In each case the material was metamict before treatment. Although fergusonite from Little Friar Mountain, Amherst County (Mallet, 1877), and the Rutherford pegmatite, Amelia County (Hidden, 1891), has been known many years, the other localities were discovered during the course of this study.

Amelia County, Champion pegmatite. Fergusonite was found at the Champion pegmatite (Mitchell, 1965) in close association with a rhabdophane pseudomorph after a mineral with the shape of a blade, 2 mm by 7 mm and about 5 cm long. Several euhedral fergusonite crystals, about 1 cm long and 2 mm across, touch the blade at intervals along its length in a somewhat oblique manner, resembling branches. The similarity of this relationship and the one described below for the Rutherford pegmatite fergusonite is very striking. Isolated fergusonite prisms up to 1 cm long and 1 mm across also have been found embedded in feldspar cleavages. The Champion fergusonite is black and has a very bright submetallic luster. The mineralogy and geology of the pegmatite have been described in detail by Lemke and others (1952, p. 114) and Brown (1962, p. 66).

Amelia County Rutherford pegmatite. Fergusonite at the Rutherford pegmatite was first described by Hidden (1891) who found it closely associated with allanite. He observed a few crystals, up to 2 cm long, implanted at right angles upon an allanite blade. Both minerals were in a feldspar matrix. The fergusonite was in the form of square prisms terminated with poorly formed pyramidal faces. The crystals had dull surfaces, but when freshly fractured showed a brilliant resinous luster. The writer has observed a nearly identical specimen in which black to brownish black fergusonite crystals less than 1 cm long and about 2 mm wide are attached nearly perpendicular to an altered blade, presumably originally allanite. In another specimen, single, poorly shaped crystals over 1 cm long and up to 5 mm across are embedded in feldspar. Pegau (1928) and Glass (1935) have described the numerous rare minerals which occur in the pegmatite. Both of these writers erroneously referred to Fontaine (1883) for the original description of fergusonite at Amelia, but the mineral is not mentioned in his paper. The geology of the deposit is described by Lemke and others (1952, p. 121) and Brown (1962, p. 74).

Amherst County, Little Friar Mountain near Alhambra. Even though fergusonite was found ninety years ago in the allanite-rich pegmatite on the northwestern slope of Little Friar Mountain, it was not properly identified until much later. It was originally named sipylite by Mallet (1877) who considered it to be a new mineral. The mineral was described by Mallet (1877) and analyzed by Brown (1877). A subsequent analysis was done by Delafontaine (1878). Apparently, because of the infancy of the knowledge of rare earth chemistry at that time, these analyses are not entirely correct. At a much later date Goldschmidt and Thomassen (1924) showed by new analyses that the sipylite is fergusonite. According to Mallet (1877) the mineral is relatively rare in the deposit where it is found embedded or adherent to the outside of masses of allanite and magnetite. The largest single piece he observed weighed 40 grams. The fergusonite occurred as brownish black masses with poor rare faces. Later Mallet (1881) described one crystal, 1.5 cm long, bounded only by a tetragonal bipyramid whose angles he noticed were similar to fergusonite (he did not know his sipylite was fergusonite). He also reported pyramidal cleavage. Pegau (1932, p. 99) described crystals from the deposit, over 1 cm long and about 6 mm in diameter, prismatic in habit and terminated with a bipyramid. A recent examination of his specimen (V3585) has shown these are actually zircon, known to occur with the fergusonite. Specimens of the Little Friar Mountain fergusonite in Lewis Brooks Museum (V701, V3035d, V3036d, V2993d) are identical to those described by Mallet (1877, 1881). The subhedral crystals (one mass 2.5 cm by 3 cm) are brownish black, somewhat duller in luster than the Amelia County fergusonite, and are intimately associated with allanite and blue-gray quartz. The geology of the deposit, which is now deeply weathered, is described by Pegau (1932, p. 96).

Bedford County, Wheatly pegmatite near Moneta. Although fergusonite was mentioned as occurring in the Wheatley pegmatite in an earlier paper (Mitchell, 1966), it was not described. Apparently the mineral is very rare in the deposit. The specimen (V3055d) identified is a black prism, less than 5 mm long and about 1 mm thick. It is peripherally altered, and is embedded in microcline which also contains a well-formed spessartine garnet crystal. Other minerals found in the deposit are listed by Mitchell (1966); and the more common minerals as well as the geology are described by Griffiths and others (1953, p. 186).

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