MINERALOGICAL INVESTIGATION OF COAL MINE ROOF SHALES IN PART OF THE SOUTHERN APPALACHIAN COAL FIELD

by

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INTRODUCTION

Many coal mines in southwestern Virginia and West Virginia are plagued with excessive deterioration of roof shales. Shale flakes and sheets spall off the roofs at unpredictable intervals and create hazards in many coal mines (Pl.1). The failures are more frequent during the summer months when the incoming air is at a higher temperature than the roofs and walls of the mine. This difference in temperature causes the moisture of the air to condense on the cooler parts of the mine.

Prof. C.T.Holland, Department of Mining Engineering, Virginia Polytechnic Institute, has suggested that this type of roof failure may depend upon some mineralogical or chemical change caused by the increase in moisture on the rooms of the mine.

In addition to the weathering process, some roof shales are notoriously susceptible to failure in those places where ground-water seepage is prevalent. The failures caused by this type of situation at the entry of a mine are usually more predictable than those caused by weathering.

The purpose of this investigation was to determine if there were any significant differences in the mineral, chemical, or textural compositions of roof rocks which have failed and those which have not.

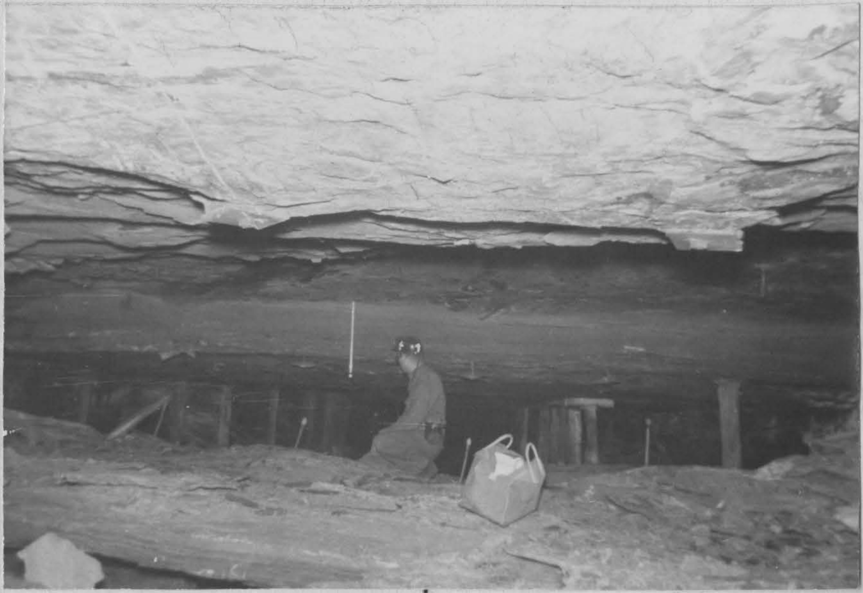
Petrographic study of thin sections cut perpendicular to the bedding were made in order to determine the mineral composition and grain size as well as to determine the fabric of the rocks. The clay mineral fractions of the shales were identified by x-ray diffraction patterns and differential thermal analyses, supplemented and confirmed by information derived from ethylene-glycol absorption data and partial chemical analyses.

Soluble salts within the samples were extracted by artificially induced efflorescence and also by leaching with distilled water. The products were identified by x-ray and wet chemical procedures.

Plate 1.- A. Two miners examine the roof shale near the heading at location 4.

B. A mining engineer measures 18 inches of roof section on which he is sitting. Note the roof bolts protruding from the collapsed section.

A.



B.

Plate 1.

PREVIOUS WORK

There are no published reports on the mineralogy of roof shales in the area studied. The General geology of the area has been studied in detail through the combined efforts of the Virginia Geological Survey and the U.S. Geological Survey (Eby, 1923; Giles, 1925; Harnsberger, 1919; Hinds, 1918; and Wentworth, 1922). Wanless (1946) described the stratigraphy of the region and contributed general descriptions of the rocks. The coal reserves were estimated by Brown and others (1952) of the U.S. Geological Survey.

The most intensive work to date on Pennsylvanian strata has been done at the Illinois Geological Survey. The petrology and clay content of the shales were reported by Grim (1935). He recognized kaolinite and chlorite as constituents of the shales by using established clay-mineral techniques (Bray and others, 1935; Ross and Kerr, 1931, p.55). Later, Grim (1937) defined and described the mineral illite. He then concluded that illite and kaolinite are the most abundant clay minerals of Pennsylvanian strata in Illinois. He also detected the presence of montmorillonite in one sample of shale of this age (Grim, 1937).

Baver (1948), Breston and Johnston (1945), Grim (1944), and Johnston (1945) have found that the type of cation that saturates a clay material strongly affects the physical properties of the clay. These workers have shown that cation fixation has been an important factor in controlling diagenetic processes in sediments.

Robbins (1937) has shown that a one degree Fahrenheit temperature change can cause a 0.0005 per cent expansion of a shale parallel to bedding and a 0.001 per cent expansion perpendicular to bedding. A sudden rise in temperature can cause an expansion of as much as one per cent of the thickness of the bed.

GENERAL FEATURES

Location.- The samples studied are from the coal fields of southwestern Virginia except for one sample from Mingo County, West Virginia (Pl.2). The southwest Virginia coal field is about 110 miles long and is as much as 30 miles wide. The field includes all of Buchanan and Dickenson counties, and those parts of Lee, Russell, Scott, Tazewell, and Wise counties which are in the Cumberland Plateau.

Topography and drainage.- The plateau is highly dissected with steep mountains and narrow valleys. Relief of 100 feet or more is common throughout the area. Powell Mountain, with an elevation of 4,162 feet, is the highest ridge in the area. Southern Wise County has the lowest elevation, 1,333 feet above sea level (Brown and others, 1952).

Big Sandy River drains the northern part of the region and Clinch River, also a tributary of Ohio River, drains the southern part. The northern part has a dendritic drainage pattern whereas the extreme southern part has structurally controlled drainage.

Climate.- The mean monthly temperature in the area ranges from 36°F. in January to 72°F. in July (Table 1). The most pronounced daily changes in temperature occur during the summer months when warm days and cool nights prevail. Precipitation averages 50 inches per year.

Plate 2.- Map of southwest Virginia showing counties and locations of mines studied.

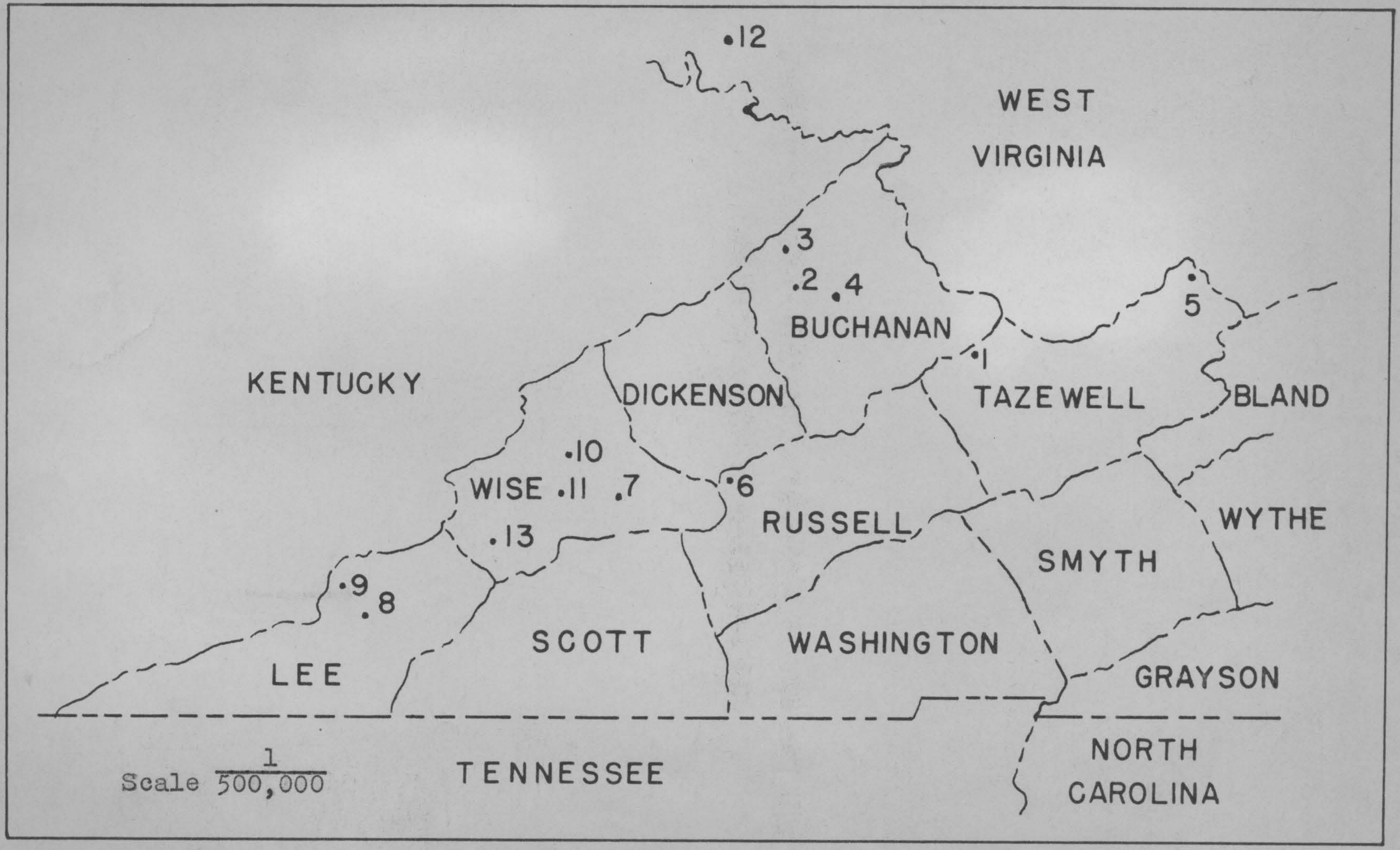

•12
WEST
VIRGINIA
.3
.2
•4
BUCHANAN
•5
•1
KENTUCKY
DICKENSON
TAZEWELL
BLAND
•10
WISE
.11
.7
•6
RUSSELL
WYTHE
.13
SMYTH
•9
.8
SCOTT
WASHINGTON
LEE
GRAYSON
Scale 1/500,000
TENNESSEE
NORTH
CAROLINA

Plate 2.

Table 1.- Average temperature and precipitation at Dante, Virginia.

Month	Average Temperature	Average Precipitation
Jan.	36.1	3.94
Feb.	38.8	3.60
Mar.	45.3	4.47
April	53.8	3.54
May	63.7	4.42
*June	69.2	4.72
*July	72.1	5.90
*Aug.	71.3	4.83
*Sept.	67.2	3.32
Oct.	55.9	2.95
Nov.	44.8	2.86
Dec.	38.0	3.59

Temperature is in degrees Fahrenheit and precipitation is in inches.

All averages are for the years 1891-1953, inclusive, except for the months marked *, which are for the years 1891-1949, inclusive.

The mine temperatures range between 54° F. and 60° F. Incoming ventilating air is rapidly cooled on contact with mine workings on the warm days in the summer. This causes a local saturated condition which results in condensation of the moisture of the air on the walls and roofs of the mines (Pl.3). The high rainfall of the area causes a constant seepage of water into the mines near the entries (Plates 4 and 5). This seepage is also found in the mines in places where the overburden is shallow, as beneath hollows.

Plate 3.- Moisture condensation limits in a plan view of a mine.

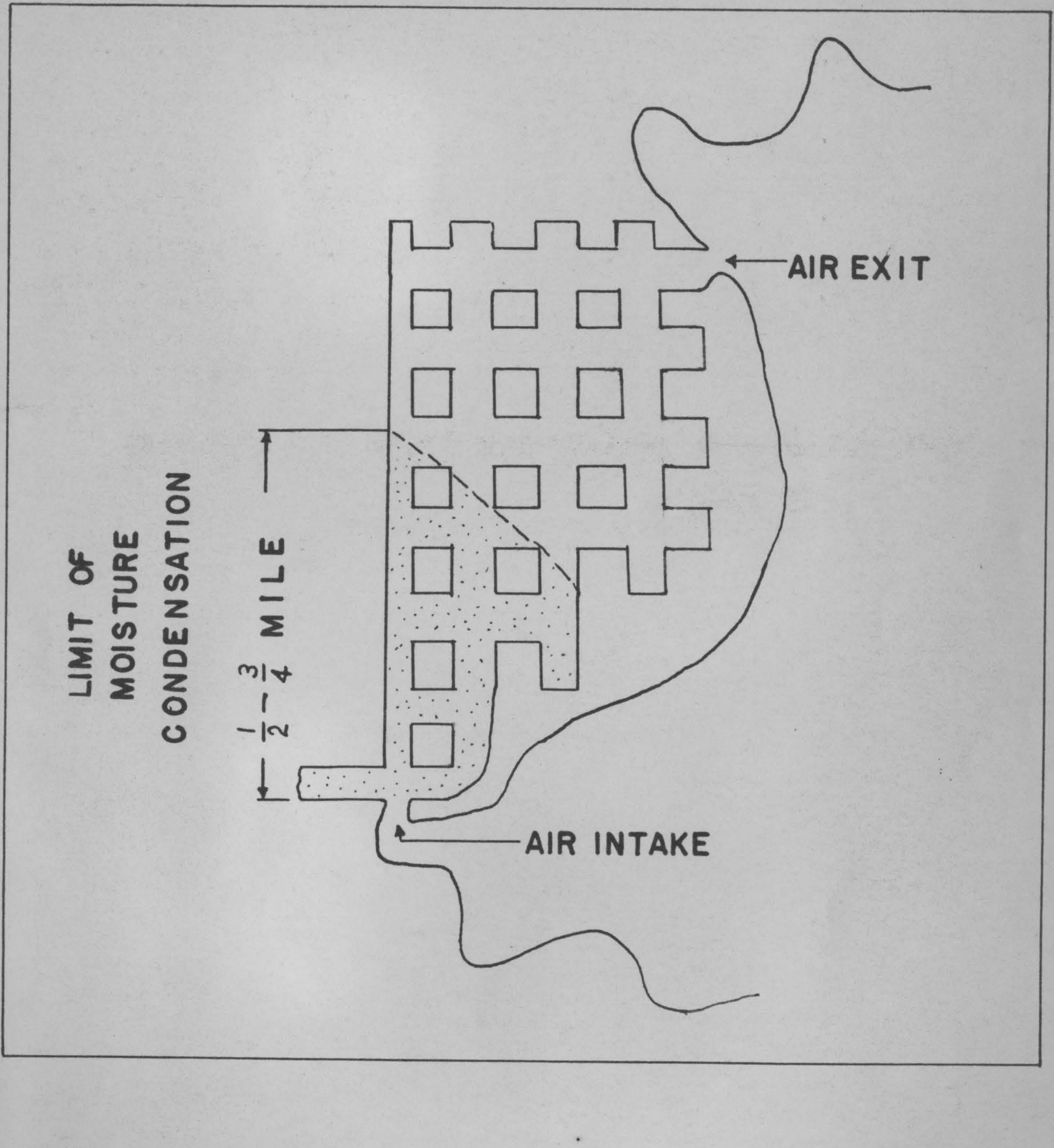
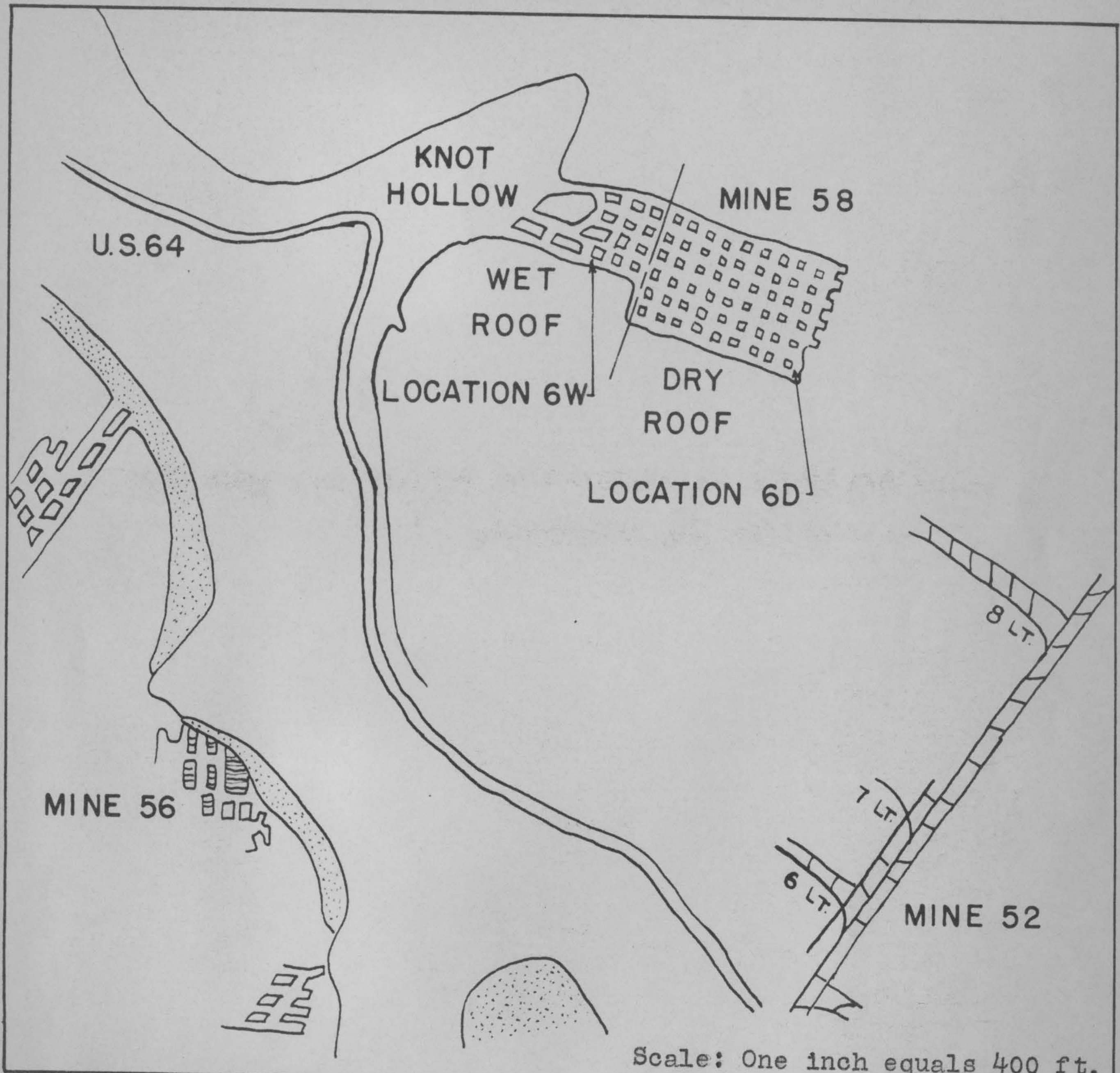

AIR EXIT
LIMIT OF
MOISTURE
CONDENSATION
$\frac{1}{2}$ - $\frac{3}{4}$ MILE
AIR INTAKE

Plate 3.

Plate 4.- Limits of ground-water seepage in a plan view of mine 58, location 6.



Scale: One inch equals 400 ft.

Plate 4.

Plate 5.- Ground-water seepage in a cross section view of mine 58, location 6.

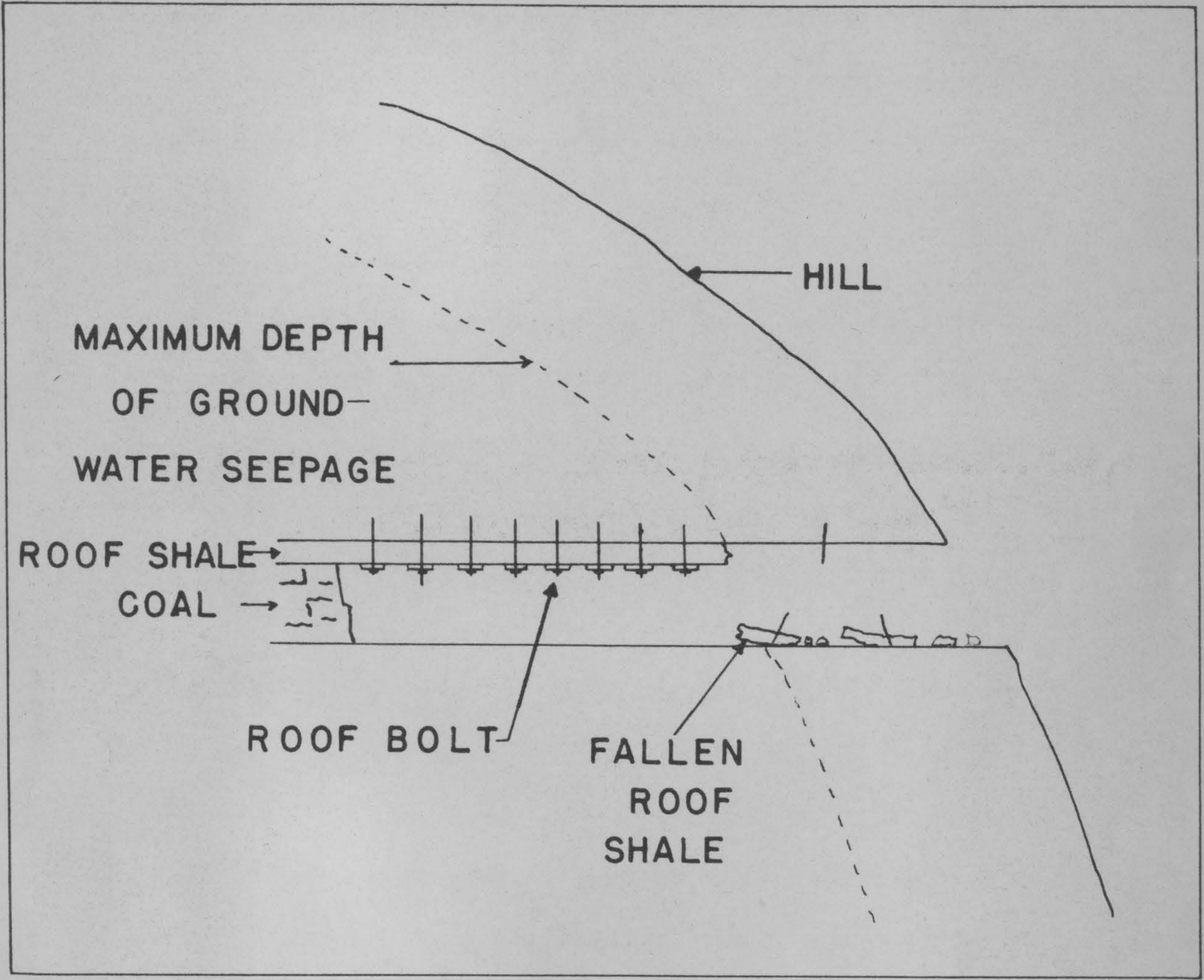

HILL
MAXIMUM DEPTH
OF GROUND-
WATER SEEPAGE
ROOF SHALE
COAL
ROOF BOLT
FALLEN
ROOF
SHALE

Plate 5.

INVESTIGATIONS OF COAL MINE ROOF SHALES

Mining methods.- The room and pillar method of mining is used in all the mines examined. The coal is undercut, blasted, and then loaded either by hand or machine. Transportation of the coal is by rail, conveyor belt, or battery-driven buggies. The roofs of most mines are supported by timbers, although roof bolts are used in some of the larger mines.

Rock sampled.- The rocks are part of a series of alternating sandstones, shales, coals, and intercalated conglomeratic strata. The oldest beds are exposed along the southern part of the region and the youngest are exposed to the north along the Kentucky border.

The Lee formation at the base of the Pottsville group and directly overlying Mississippian strata, is about 1500-1800 feet thick in the southeast part of the field near the Kentucky border. There is no noticeable change in thickness from northeast to southwest. The Pocahontas No.3 coal is the only seam sampled in this formation. It is found about 360 feet above the base of the Lee formation, generally with a massive sandstone above it. Locally there is a shale present which pinches out because of rolls in the roof.

The Norton formation overlies the Lee formation and is exposed near the Tazewell-Buchanan county border. This formation in Wise County is predominantly shale with a few sandstones found in the Guest River basin. Of the eleven coal seams in this formation, the Jewell Ridge or Raven seam is the third from the base, or about 400 feet from the top of the Lee formation. Rolls in the roof and floor cause pinchouts of the shale, which otherwise separates the coal from the overlying sandstone. The Lower Banner coal bed is about 800 feet above the Lee formation where sampled in Russell County. A fine-grained black shale overlies the coal seam, and this in turn is overlain by an arkosic sandstone. The Splash Dam seam is found between 90 and 170 feet above the Lower Banner coal bed, or a few feet above the sandstone which overlies the Upper Banner seam. This bed typically is overlain by a friable brown sandstone, with a shale several inches to a few feet thick between the coal and the sandstone. The Norton coal bed is found about 1450 feet above the base of the Norton formation. It is overlain in some places by the Gladeville sandstone, but elsewhere there is some shale between the two.

The Gladeville sandstone, which separates the Norton and Wise formations, is a hard gray quartzose sandstone about 100 feet thick. It is conglomeratic to the west and shaly to the north.

The Wise formation directly overlies the Gladeville sandstone and contains 21 coal beds in Lee County. It is 2300 feet thick in western Wise County, and 2070 feet thick near the south fork of the Pound River. About one-third of the formation is sandstone and most of the remainder is shale. The Blair coal, where sampled in Wise County, is between 90 and 120 feet above the Gladeville sandstone. It has a sandstone roof at the locality sampled. The Clintwood coal is between 70 and 95 feet above the Blair seam in Buchanan County. It has a thick shale above the coal bed. The Imboden coal lies about 650 feet above the Gladeville sandstone at the locality sampled in Wise County. About five feet of fine-grained shale overlie the coal there. The Red Jacket or Cedar Grove coal is about 700 feet above the Gladeville sandstone where sampled in Mingo County, West Virginia. A 20-foot thick shale member overlies the coal bed here. The "No.10" coal is about 2400 feet above the Gladeville sandstone in Lee County, which means it is near the top of the Wise formation which is 3000 feet thick in this area. Several feet of shale near the coal form a weaker roof than the sandstone which is sometimes present.

Structure.- Although the coal-bearing plateau region is underlain by an essentially flat lying series of beds, there are a number of small structural features present that create special mining problems. In most of the area the beds dip gently so that natural drainage can be obtained by having the mine entry on the side of a hill. Near Tacoma, where the beds dip steeply under the valley surface, constant pumping of ground water from the mine is necessary. Other features such as rolls and pinchouts of the roofs and minor faults create hazards to the miners. The most troublesome feature seems to be the unsuspected thinning of a roof shale. Some companies solve this problem by roof bolting.

Sampling procedure.- All samples were collected from roof rocks overlying coal seams of early Pennsylvanian age. Samples were listed in stratigraphic succession with the oldest seam at the bottom (Table 2). The sampling was done in the spring of 1954. Thirteen mines were sampled on the basis of their availability for study rather than on the quality of their roofs. Localities within the mines, however, were selected on the basis of roof conditions. Where possible, both good (G) and bad (B) roofs were sampled in each mine. A roof was called good if it did not spall after six months exposure in a mine. If a roof was considered weak throughout an entire mine, samples were taken in both recently exposed and in older, more weathered areas, indicated as A and B, respectively. Samples were broken from the roofs with a pick and stored in ice cream containers until analyzed. Sample localities are described in Table 3.

Petrographic examination.- Examinations of thin sections cut perpendicular to the bedding of the shales were made in order to determine possible relations

Table 2.- <u>Compilation of seams and roof quality</u>.

Formation	Coal bed	Sample	Quality
Wise	"No. 10"	9	Good
Do.	"No. 10"	8	Do.
Do.	Red Jacket	12A	Bad
Do.	do.	12B	Do.
Do.	Imboden	13A	Do.
Do.	do.	13B	Do.
Do.	Clintwood	3	Do.
Do.	Blair	10A	Do.
Do.	do.	10B	Do.
Norton	Norton	11G	Good
Do.	do.	11B	Bad
Do.	Splash Dam	2A	Do.
Do.	do.	2B	Do.
Do.	Lower Banner	6D	Good
Do.	do.	6W	Bad
Do.	Raven	7	Good
Do.	do.	1	Do.
Lee	Pocahontas No.3	5	Bad

Coal beds are listed with the oldest bed at the bottom.

Table 3.- Sample locations in the mines

Sample	Mine	Sample locations	Method of roof support
1	-	300 feet from entry, M.C. tunnel	Roof bolts
2	Charles	Third east, 13 left	Roof bolts
3	Kentucky	Along 29 west, three right	Roof bolts
4	C.W.R.	Main heading, two south	Timbers
5	Junior	Along D-8, 13 right	Timbers
6D	No. 58	No. 6 heading, sixth break	Timbers, bolts
6W	No. 58	No. 5 heading, second break	Timbers, bolts
7	Tacoma	Main drift, five left	Timbers
8	Pennington Gap	No. 3 heading, 29 right	Timbers
9	Bonny Blue	Main airway, four right	Timbers
10A	Dale Branch No. 1	600 feet in main portal	Timbers
10B	Dale Branch No. 1	Portal sample, main heading	-
11G	Dorchester	No. 1 west, No. 1 south, 29 right	Roof bolts
11B	Dorchester	No. 1 west, No. 1 south, 29 left	Roof bolts
12A	Key Mountain	No. 4 entry, three right	Roof bolts
12B	Key Mountain	Portal sample, main heading	Roof bolts
13A	Crossbrook	No. 2 entry, 11 left	Roof bolts
13B	Crossbrook	Portal sample, main heading	-

between the mineral composition , texture, and strength of the roof rocks. Estimates of the quantities of the constituents were made and prominent textural features noted. It was found that masses of organic material obscured the other minerals, especially the clay minerals (Grim, 1934, 1955). Clay minerals were not distinguished optically for this reason.

Differential thermal procedure.- It was necessary to separate the shales into different size fractions before analyzing them for the clay and non-clay materials present (Bray, 1937). The procedure used was similar to that described by Jackson and others (1949). The minus 2 micron fractions were separated and analyzed with differential thermal equipment.

A circular sample holder 1.5 inches in diameter and 0.375 inch thick was used. It had one central 1/4 inch hole and four symmetrically placed 1/4 inch holes halfway between the center and the edge of the block. Platinum-platinum 10 per cent rhodium thermocouples were held in the center of each hole by porcelain insulators 1/16 inch in diameter and with two holes for the wires. The sample holders and thermocouples were designed and built by Dr. C.I.Rich of the Virginia Agricultural Experiment Station.

The sample was packed firmly in one hole of the sample holder, and calcined alundum was packed firmly in the other three holes. The furnace was heated up at the rate of 12.5° C. per minute, and exothermic and endothermic changes were recorded on an x-y recorder between 100°C. and 1000°C. The recording setup was the same as that described by Kerr and Kulp (1949), except that a single point unit was used instead of a six point unit.

X-ray procedure.- In order to achieve uniformity among the clay samples extracted from the shales, aliquots of clay suspensions were saturated with calcium acetate and potassium chloride, and solvated with glycerol. Calcium-saturation gave the most intense reflections for the clay minerals. Potassium-saturation was effected in order to determine if vermiculite was present. Glycerol-solvation was performed in order to ascertain if montmorillonite was present (MacEwan, 1944). Slides with calcium acetate-saturated clay were heated at 500°C. and 600°C. for two hours and subjected to x-ray diffraction after each heating.

The procedure utilized involved an aliquot of suspension containing 50 mgm of clay and five ml of 1 N saturating solution or two drops of glycerol (about 50 mgm). This suspension was mixed, centrifuged, and poured on a glass slide to dry. This treatment resulted in a preferred orientation for the clay minerals with their basal planes parallel to the slide. This orientation gave the most intense reflections for the clays (Nagelschmidt, 1941).

The silt size materials were packed into a depression in a plastic slide and flattened with a spatula before running on the x-ray machine.

The General Electric XRD-3 diffraction unit No.1 SPG spectrogoniometer arranged for recording diffraction effects from a flat sample was used. Copper Kα radiation with a nickel filter 0.0007 inch thick and Cobalt Kα radiation with an iron filter weighing 0.0003 gm per sq. cm were used. The geiger tube voltage was 40 KV and the amperage was 15 ma. A beam slit of one degree with medium resolution of the Soller slit, a four degree target angle, and a scanning speed of two degrees per minute were used.

Partial chemical analyses.- Semimicrochemical silicate analyses were used to obtain the potash and iron contents of some of the clay samples (Corey and Jackson, 1953). A modification of this technique using a polarograph was developed by Dr.C.I.Rich of the Virginia Agricultural Experiment Station for the iron analyses. The free iron oxides were segregated according to procedures outlined by Aguilera and Jackson (1953), and also run on the polarograph.

Soluble salt technique.- The presence of soluble salts in the shales was first detected in some samples that had been stored for several months. White crystals had efflorsced along several seams of the shale (Pl.5). An attempt was made to grow these salts artificially by placing the shales upright in shallow dishes containing about 0.5 inch of distilled water (Pl.6). Apparently water traversed the bedding planes by capillary action, dissolved the salts on these planes, and precipitated them on the surface of the shale as the water evaporated. The mineral compositions of the effloresced salts were investigated by x-ray diffraction methods, by spectrographic methods, and by petrographic methods. In addition, wet chemical analyses were made of the salts. After it was learned that only a few of the shale samples effloresced their salts in sufficient quantities for analysis, it was learned that the salts could be leached out of pulverized shale with distilled water. Buchner funnels were loaded with 20 gm of ground shale, attached to an Erlenmeyer flask which was attached to suction apparatus, and distilled water passed over the shale. Two 12-hour periods of leaching were employed. During the intervening nine hours, no suction was applied to the funnel, but the sample was kept immersed in water to keep the shales from drying out.

A nephelometer designed and built by Dr.J.W.Murray of the Department of Chemistry of Virginia Polytechnic

Plate 6.- A roof shale with effloresced salts.

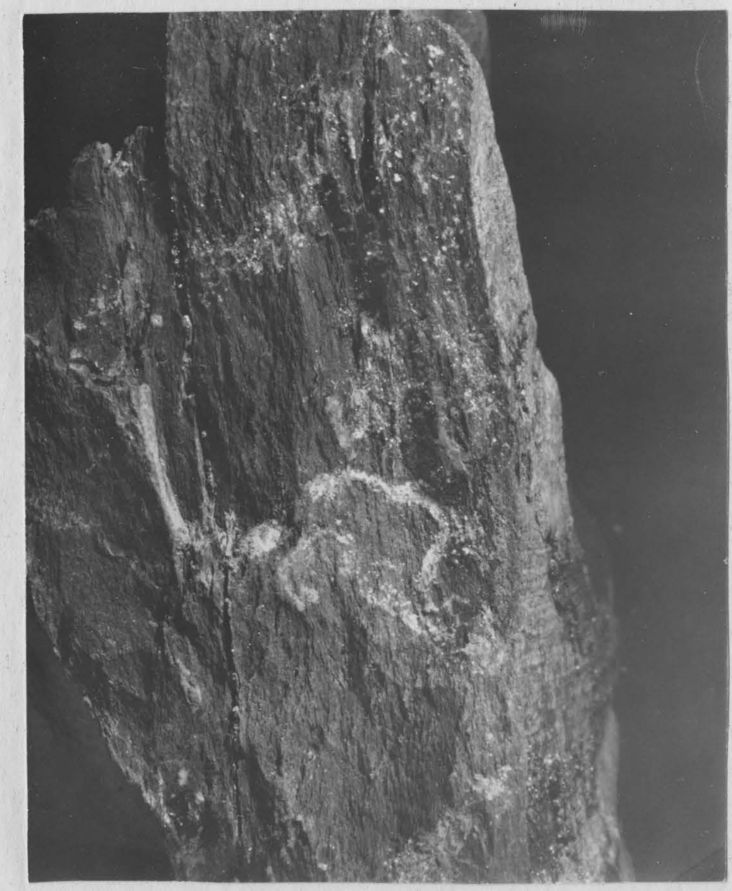


Plate 6.

Plate 7.- Artificial efflorescent arrangement.

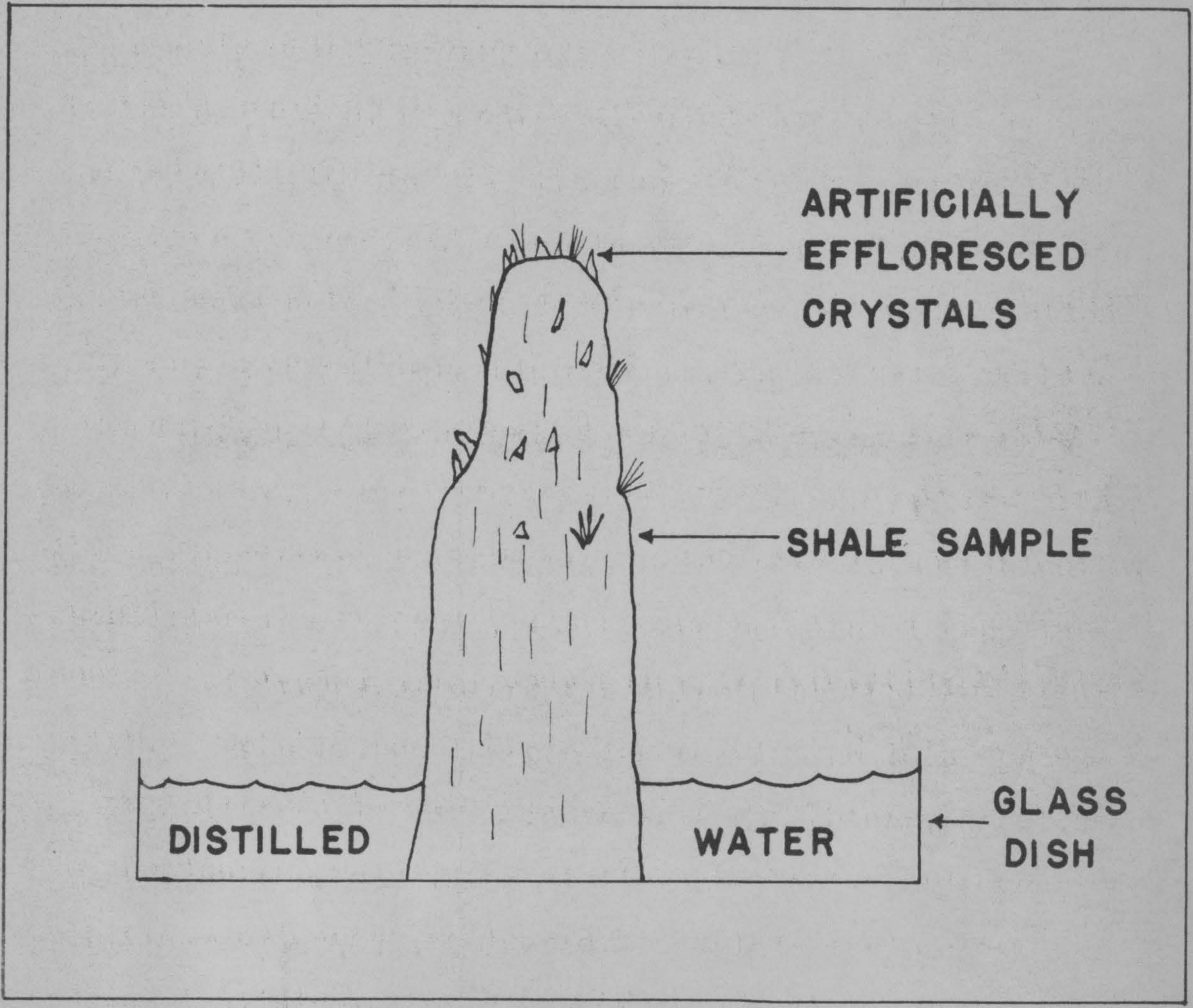

ARTIFICIALLY EFFLORESCED CRYSTALS
SHALE SAMPLE
DISTILLED
WATER
GLASS DISH

Plate 7.

Institute was used to determine the chloride and sulphate contents of the dissolved salts.

For determination of chloride ions, 0.1 ml of concentrated HNO_3 was added to a 10 ml flask and mixed with 1-2 ml of the concentrate containing the dissolved salts. Then 1 ml of 10 per cent $AgNO_3$ was added, and the flask made up to volume. This solution was shaken well, poured into test tubes, and tested on the nephelometer.

Test solutions for sulphates were prepared by adding 1-2 ml of the unknown solution to a 10 ml flask, adding 2 ml of a buffer solution containing NaCl and HCl, and making the flask up to volume. After pouring the resulting solution into a beaker, an excess of $BaCl_2$ of 20-30 mesh size was added. The mixture was stirred, poured into test tubes, and tested on the nephelometer.

A second attempt to leach the salts from the same ground shales of samples 2 and 3 was made in order to determine the efficiency of the earlier leaching. It was found that only eight per cent of the amount first extracted could be obtained on a second run. This was considered to be insufficient to justify a second leaching for all the shales.

Mechanical analyses.- No special separation of the size fractions was attempted other than that performed for sample preparation for differential thermal analyses.

Using this procedure, it was learned that the amount of clay size material extractable could be increased greatly by slight additional grinding of the larger size material. It was found on petrographic examination that a large percentage of the material present was in the size fraction just larger than the clay size fraction. This explained the ease with which the clay size fraction could be increased, and also made it evident that no really accurate size determination could be made.

RESULTS OF INVESTIGATION

Petrography.- Most of the roof rocks studied in thin sections are gray to black siltstones or shales. A few specimens are very fine-grained sandstones. Slickensides are visible on specimen 7 from Tacoma, Virginia. Carbonaceous material is distributed irregularly as thin lenses in all of the rocks.

The mineral composition and texture of each sample is presented in Table 4. Quartz and feldspar are more common in the coarser grained rocks. The shape of these mineral particles is equidimensional and subrounded in contrast to the platy micas. The fabric is a mixture of that which was formed at the time of deposition and that which is the result of post-depositional processes. Probably most of the platy minerals were aligned during deposition, in contrast to the illite and other diagenetic minerals which developed with their long axes parallel to the bedding.

Wavy extinction is visible in all minerals of the coarser than 0.25 mm size fractions, except for a few of the chlorite and illite grains. Minerals less than 0.001 mm in maximum dimension are indistinguishable because of the masking effect of the opaque iron oxides and carbonaceous material present.

The coarse grains may be the products of clastic

Table 4.- Mineral composition and texture of thin sections.

Sample	Mica	Chlorite	Clay	Organic	Quartz	Feldspar	Texture
1	10	5	15	15	20	35	C-a
2	20	5	20	10	15	30	C-i
3	55	5	30	5	5	-	M-i
4	15	5	30	15	10	25	M-i
5	30	5	40	10	10	5	C-i
6D	40	5	20	5	10	20	C-i
6W	40	5	20	5	10	20	C-i
*7	5	5	10	20	10	50	C-a
8	20	4	23	15	15	23	C-a
9	10	3	15	20	15	37	C-a
10A	40	20	25	5	10	-	C-i
10B	40	1	40	9	10	-	C-i
*12A	5	2	20	33	30	10	C-a
12B	5	2	20	33	30	10	C-a
13A	35	2	5	3	10	45	C-a
13B	60	2	20	5	10	8	C-i

* - less than one per cent pyrite is present

C - detrital minerals predominate

M - detrital and diagenetic minerals present

a - sand sizes predominate

i - silt sizes predominate

All numbers are estimated percentages of minerals present.

deposition whereas many of the fine grains may be the alteration products of clays which were deposited contemporaneously with the coarser fractions. No differences in the composition of the strong and the weak roof rocks were noted, but it was evident that the coarser textured rocks were stronger than the finer textured rocks.

Differential thermal analyses.- The curves obtained for the minus two micron size fractions are shown in Figures 1-6. Some of the most intense reactions are obtained from small amounts of intensely reactive minerals (Grim and Rowland, 1942). The position and intensity of the peaks is also affected by the size and degree of crystallinity of the minerals (Grim, 1948). Grim (1948) also states that the starting temperature is more characteristic than the peak and conclusion of a reaction.

Table 5. gives the writers interpretation of the curves obtained. The organic matter present has the greatest effect on the peaks (Grim and Rowland, 1944). It typically bives broad exothermic effects between 200°C. and 650°C. On the curves shown (Figures 1-6), this is an upward movement of the curve. Illite has endothermic peaks between 100°C. and 250°C., 500°C. and 625°C., and between 850°C. and 925°C. Kaolinite has a strong endothermic reaction between 550°C. and 650°C., and a sharp exothermic peak between 960°C. and 990°C. The presence

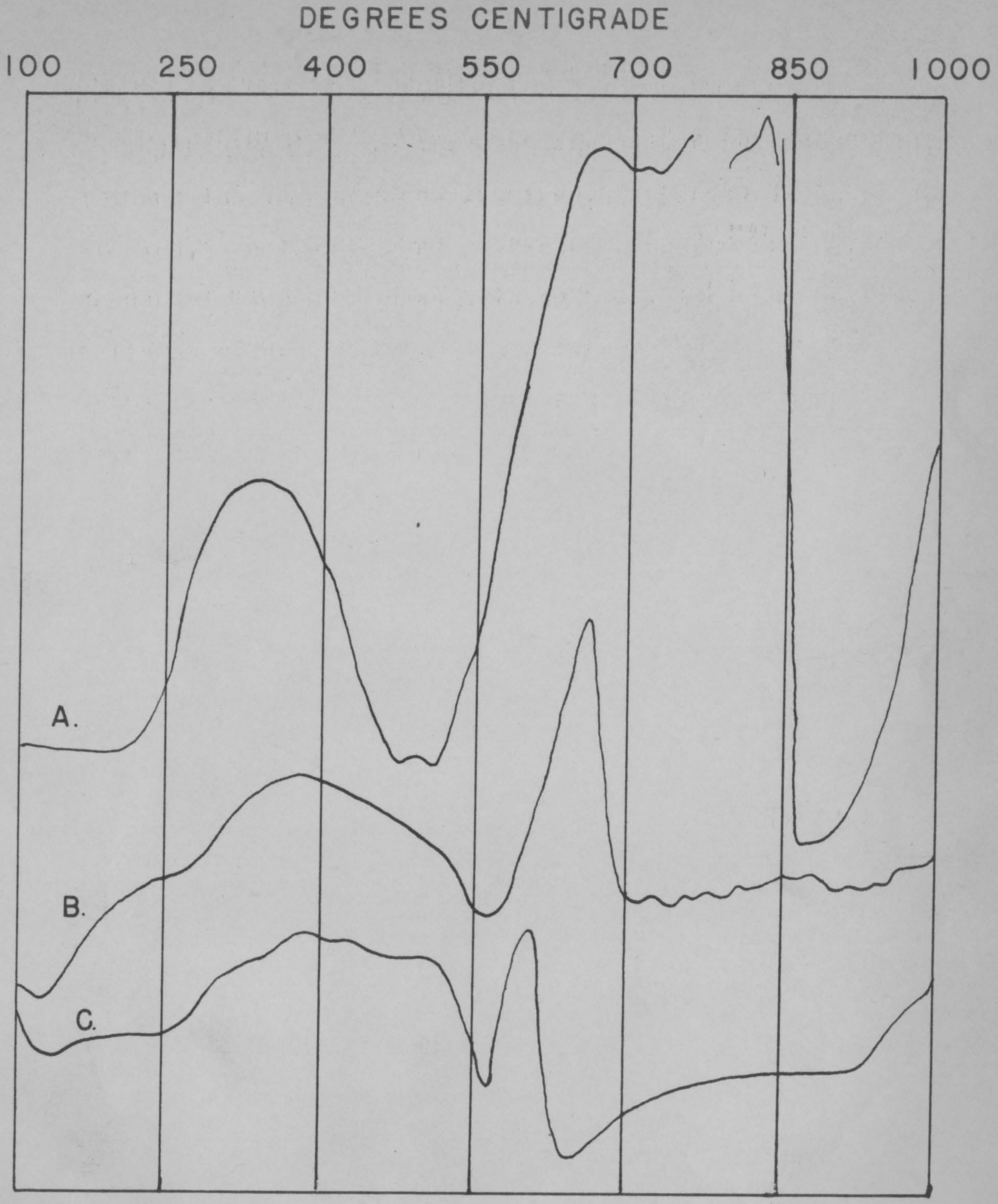


FIG. 1.-DIFFERENTIAL THERMAL CURVES, MINUS 2 u SIZE, FOR A.-RAVEN SEAM, LOCATION 7; B.-RAVEN SEAM, LOCATION 1; AND C.-POCAHONTAS SEAM, LOCATION 5.

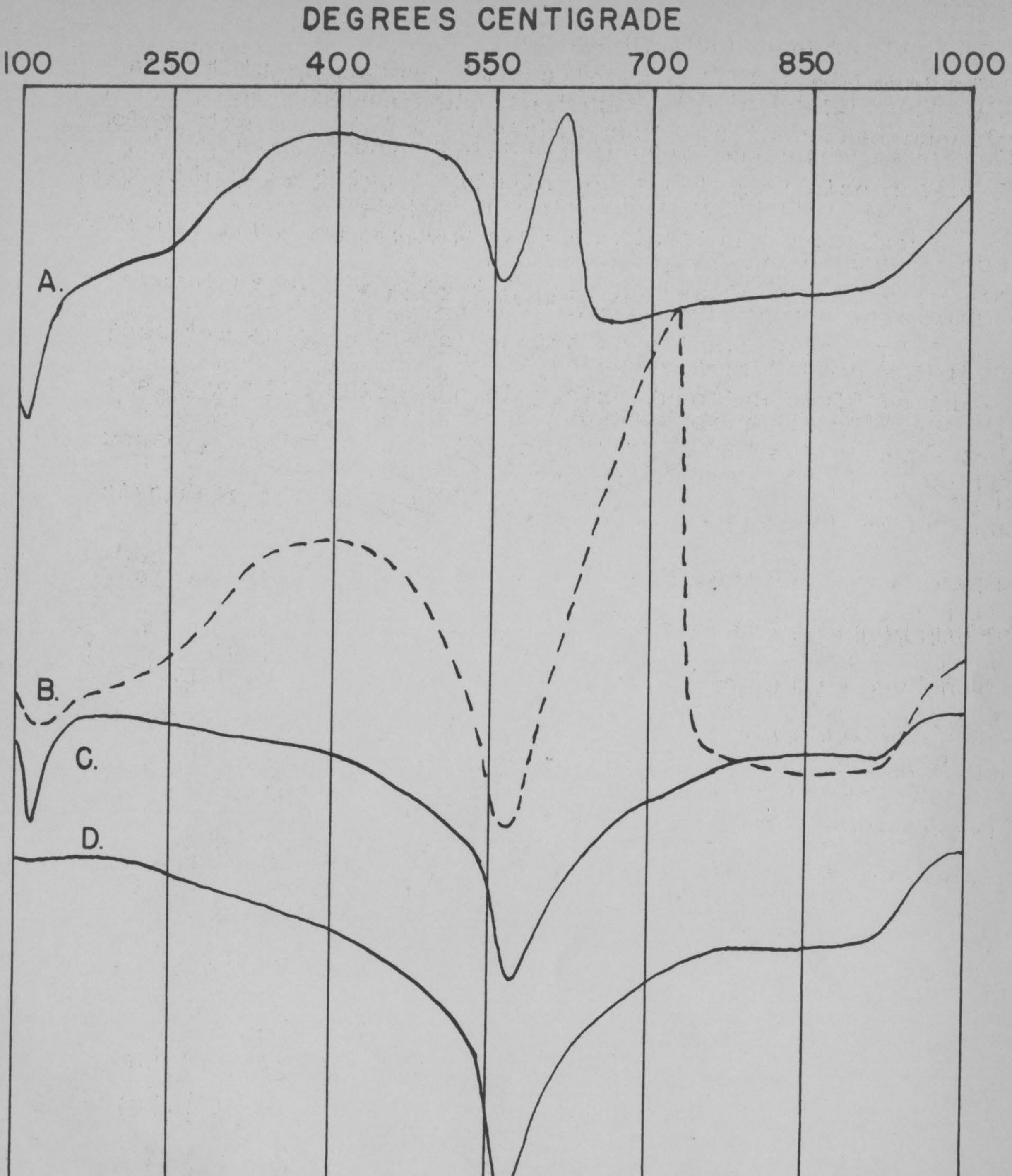


FIG. 2.-DIFFERENTIAL THERMAL CURVES, MINUS 2 u SIZE, FOR SPLASH DAM SEAM, A.-LOCATION 2 2B, B.-LOCATION 2A; AND LOWER BANNER SEAM, C.-LOCATION 6D, D.-LOCATION 6W.

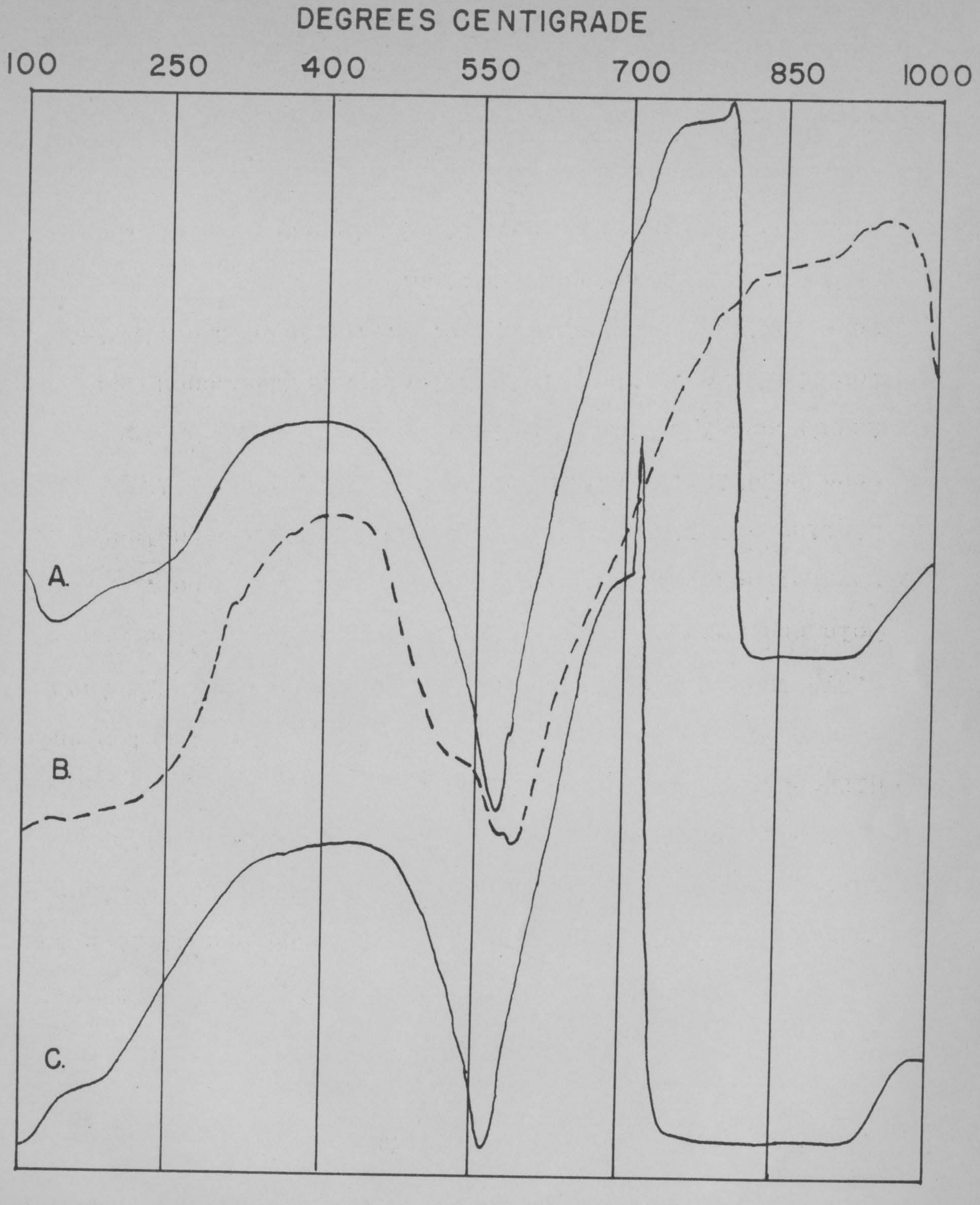


FIG. 3.- DIFFERENTIAL THERMAL CURVES, MINUS 2 u SIZE, FOR A.-NORTON SEAM, LOCATION 11B; B.- NORTON SEAM, LOCATION 11G; C.-SPLASH DAM SEAM, LOCATION 4.

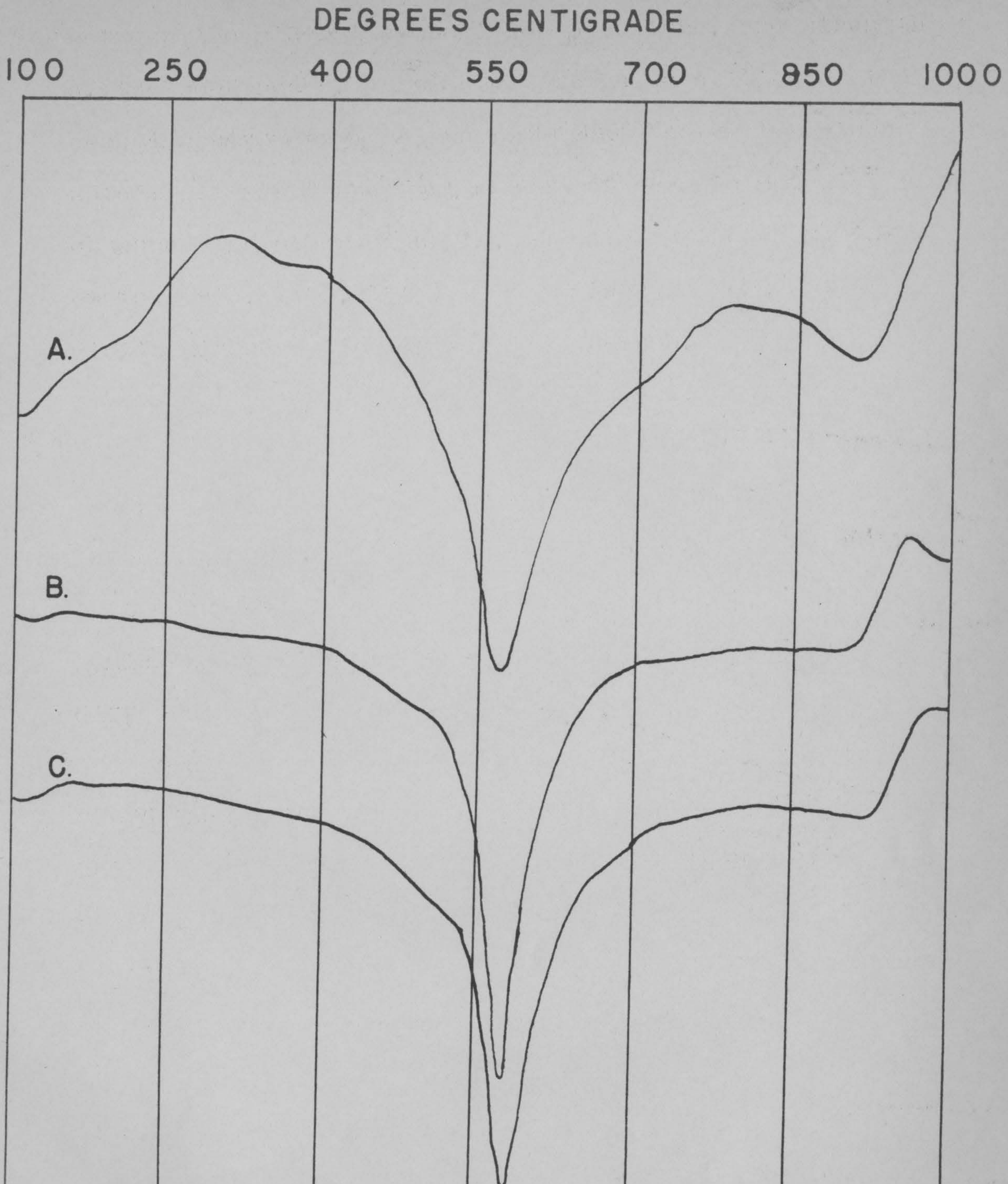


FIG.4.- DIFFERENTIAL THERMAL CURVES, MINUS 2 u SIZE, FOR A.-CLINTWOOD SEAM, LOCATION 3; B.-BLAIR SEAM, LOCATION 10B; AND C.-BLAIR SEAM, LOCATION 10A.

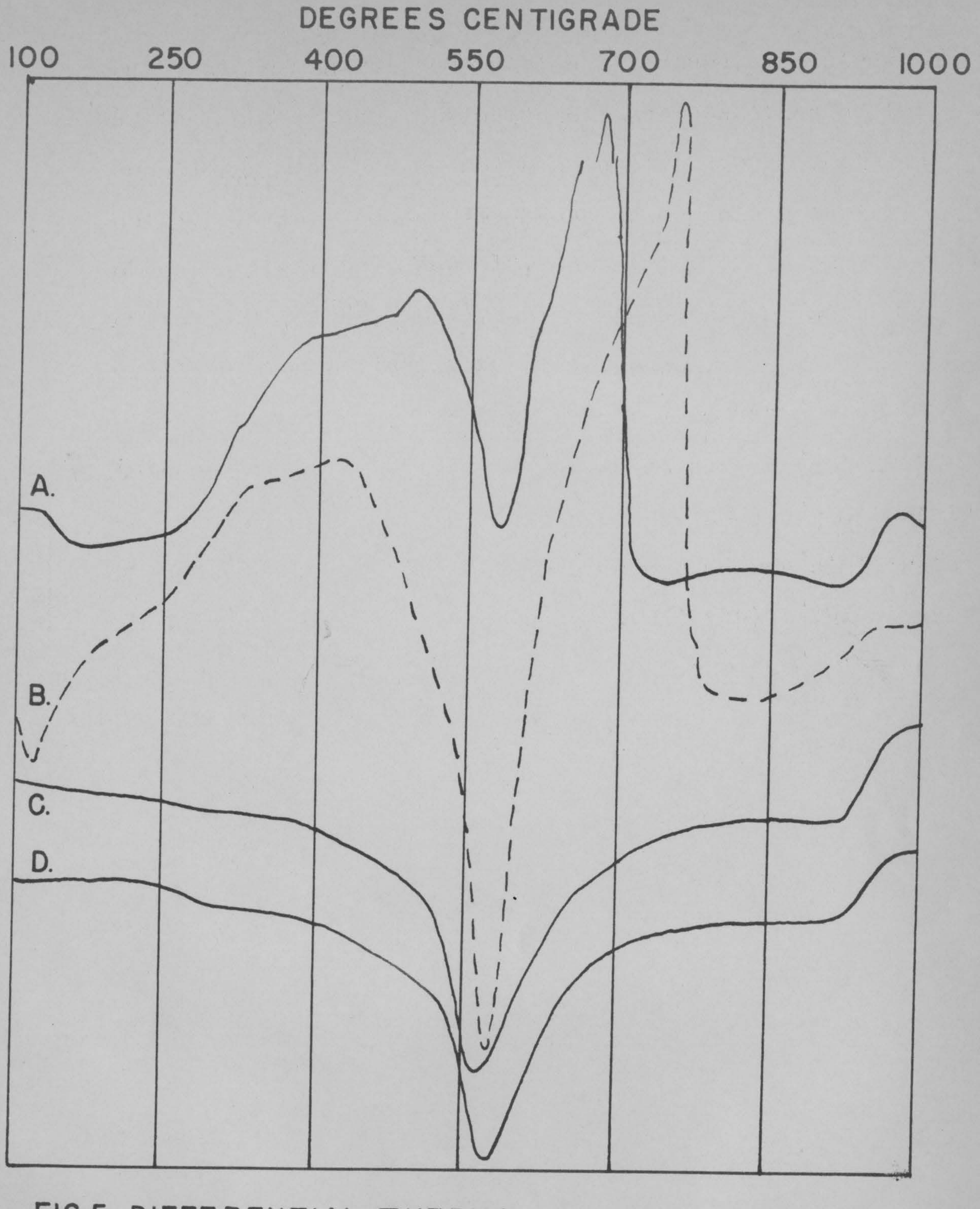


FIG. 5. DIFFERENTIAL THERMAL CURVES, MINUS 2 u SIZE, FOR RED JACKET SEAM, A.-LOCATION 12B, B.-LOCATION 12A.; AND IMBODEN SEAM, C.-LOCATION 13B, D.-LOCATION 13A.

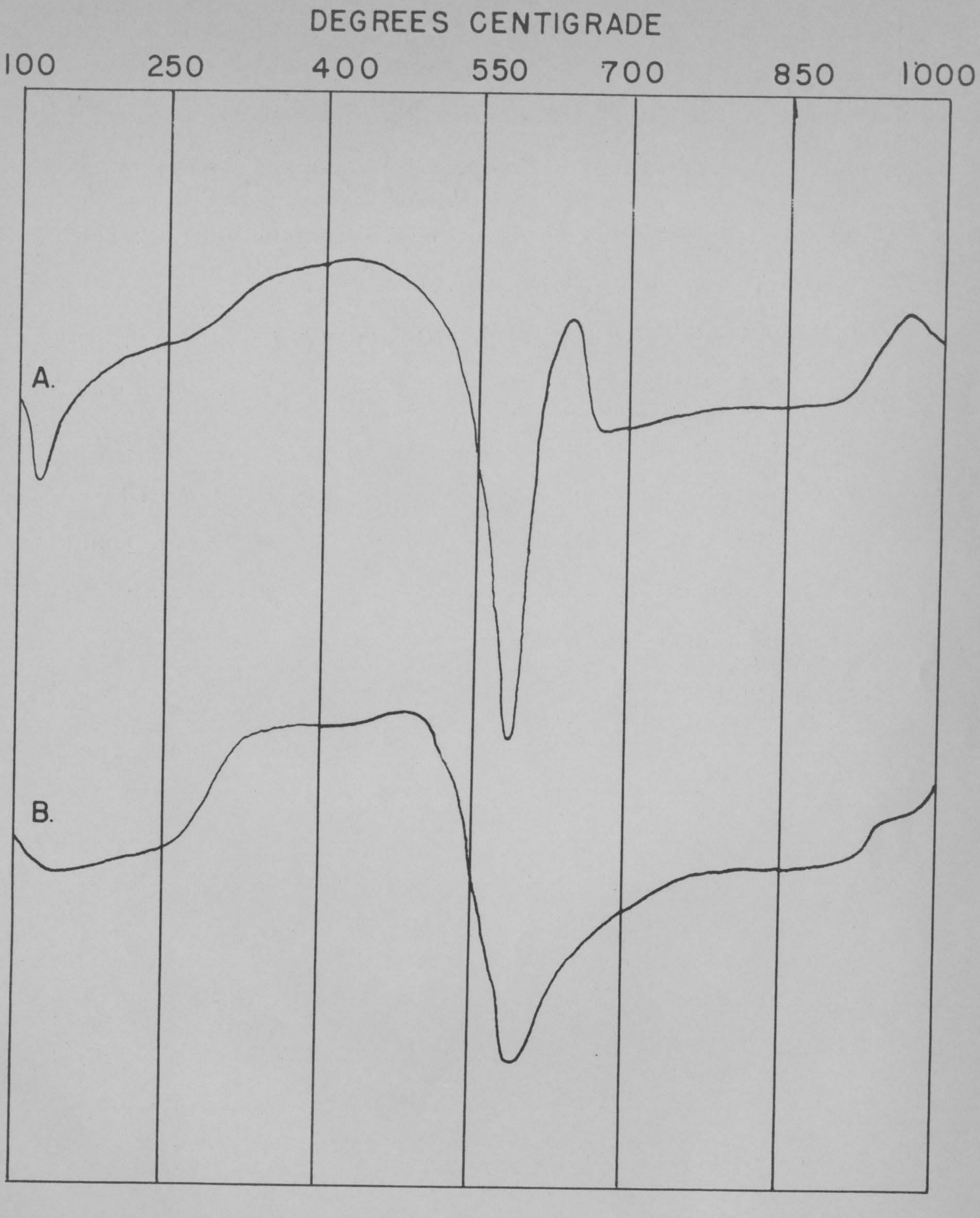


FIG. 6.- DIFFERENTIAL THERMAL CURVES, MINUS 2u SIZE, FOR A.-NO. 10 SEAM, LOCATION 9, AND B-NO. 10 SEAM, LOCATION 8.

Table 5.- Estimated percentages of minerals present, from differential thermal curves.

	Percentage Composition			
Sample	Kaolinite	Illite	Organic Matter	Chlorite
1	45	50	5	-
2A	43	50	7	-
2B	40	55	5	-
3	38	60	2	-
4	25	65	10	-
5	38	60	2	-
6D	40	60	-	-
6W	40	60	-	-
7	-	70	10	20
*8	40	55	5	-
9	45	50	5	-
10A	43	55	2	-
10B	43	55	2	-
11G	30	50	10	-
11B	30	50	10	-
12A	43	50	7	-
12B	47	50	3	-
13A	38	60	2	-
13B	38	60	2	-

* - about two per cent pyrite is present.

All numbers are estimated percentages of <2μ size.

of quartz reduces the first kaolinite peak, and the presence of illite reduces the second kaolinite peak. Quantitative estimates of the minerals present are made by comparing the sample curves with known mixtures (Grim, 1948).

The analyses reveal the presence of kaolinite, illite, and minor amounts of organic matter, as constituents of all the shales, and pyrite in one of the shales studied.

X-ray diffraction patterns.- X-ray diffraction patterns are recognized as the most useful of all available procedures for identifying clay minerals (Brindley, 1951). However, quantitative evaluation of mixtures of clay minerals according to the intensity of their peaks is subject ot error because of differential absorption effects.(Carl, 1948).

The minus two micron size fraction of all the shales studied show large amounts of illite and kaolinite, and lesser amounts of quartz, chlorite, and iron oxides (Figures 7-14). When well formed, kaolinite, illite, and chlorite have characteristic peaks at 7.2, 10.0, and 14.4 Angstroms, respectively (Brindley, 1955). These are the first order spacings and are the strongest and most characteristic for these minerals.

In order to determine the presence of chlorite, it is necessary to examine the patterns of the glycerol-

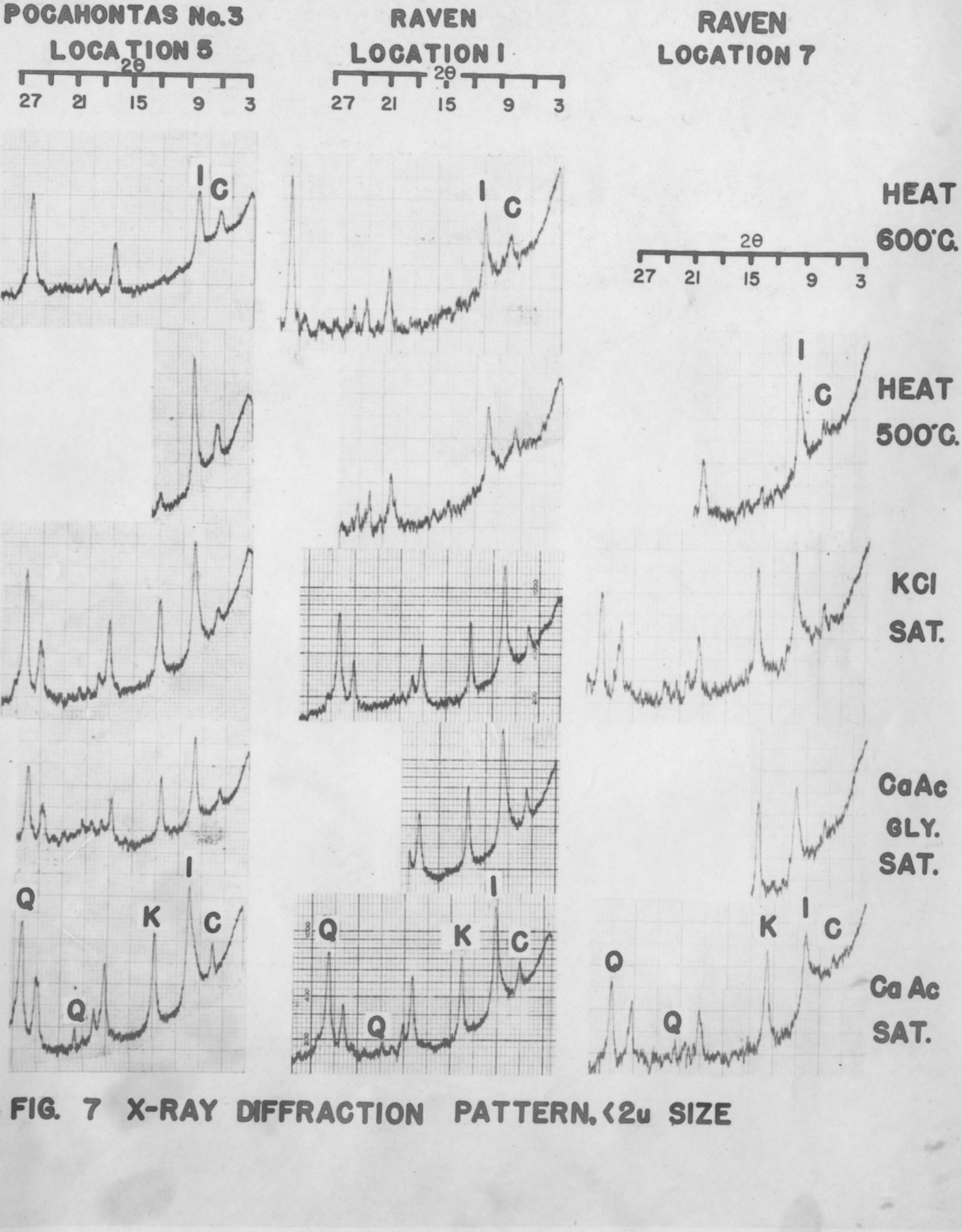


FIG. 7 X-RAY DIFFRACTION PATTERN, <2u SIZE

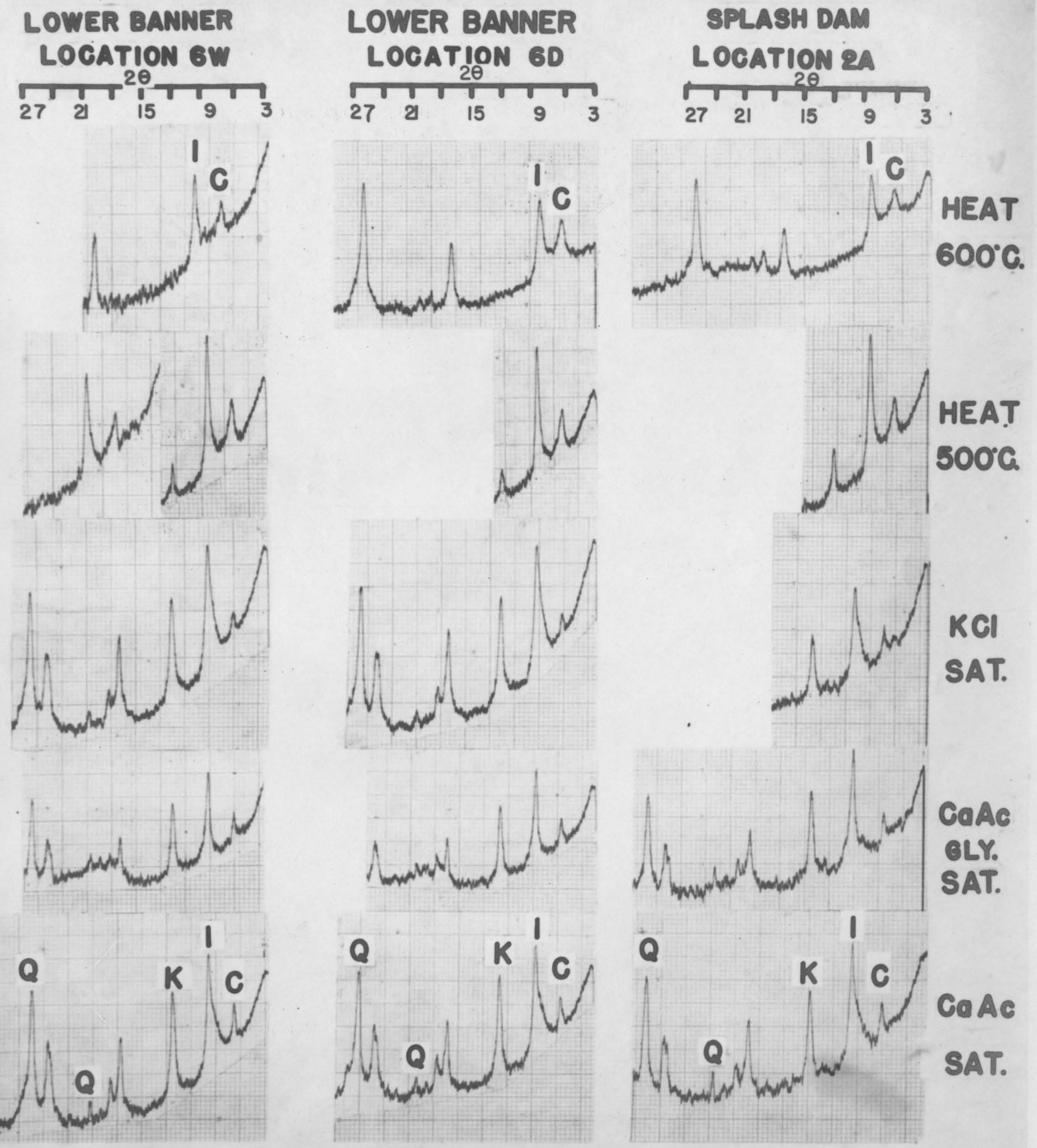
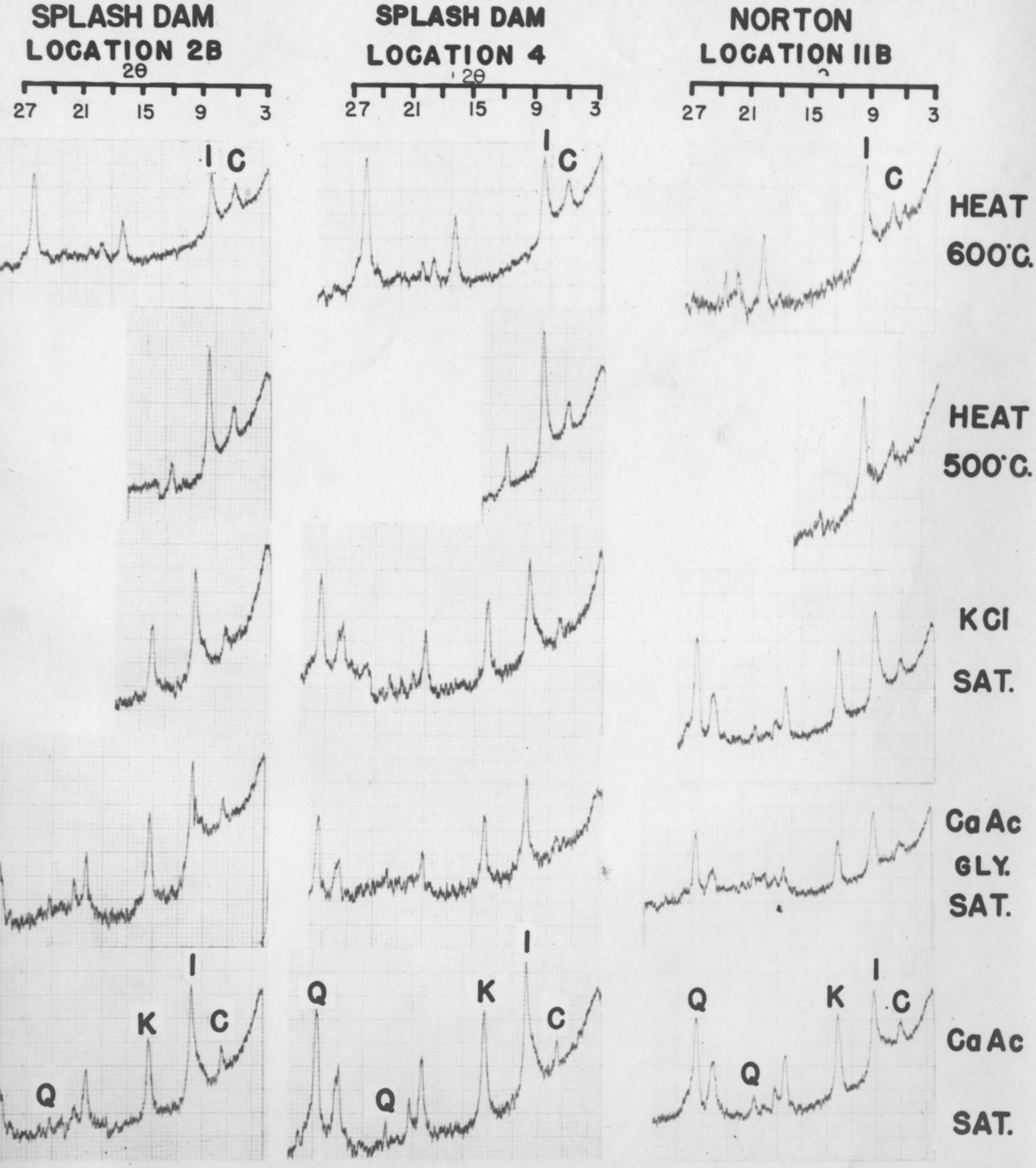


FIG. 8 X-RAY DIFFRACTION PATTERN, <2u SIZE



IG. 9 X-RAY DIFFRACTION PATTERN, <2u SIZE

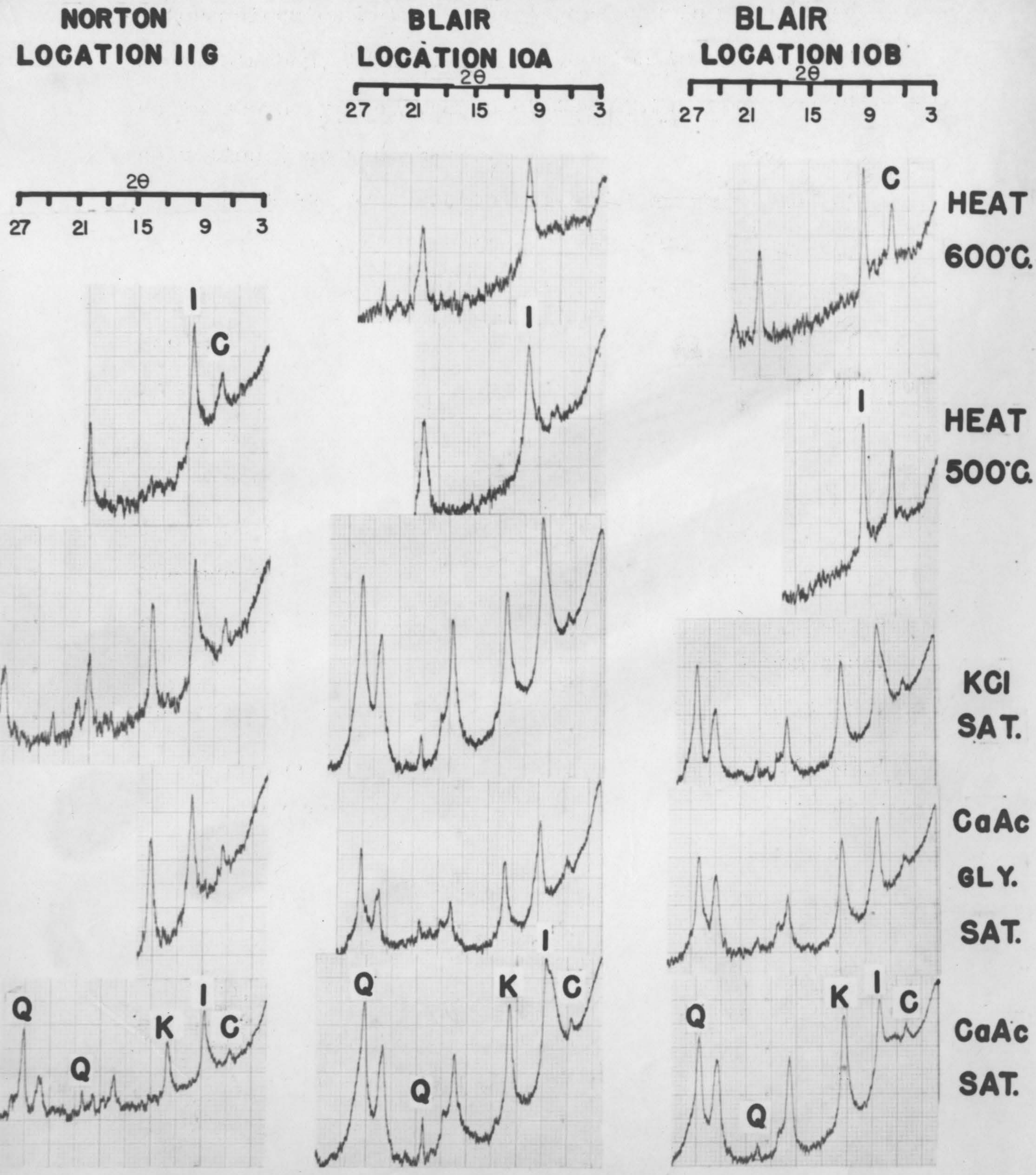
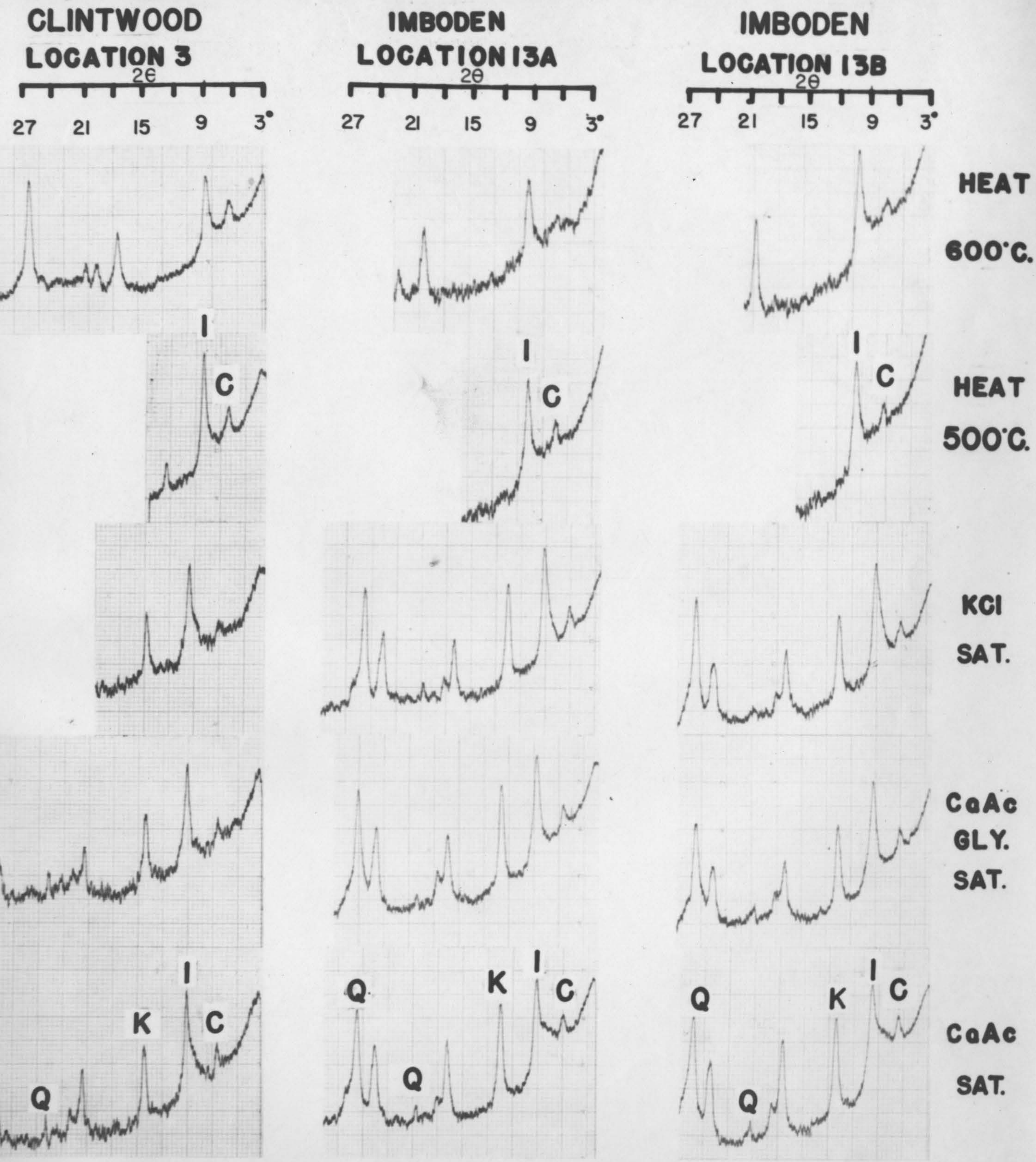


FIG. 10 X-RAY DIFFRACTION PATTERN. <2u SIZE



IG. 11 X-RAY DIFFRACTION PATTERN, <2u SIZE

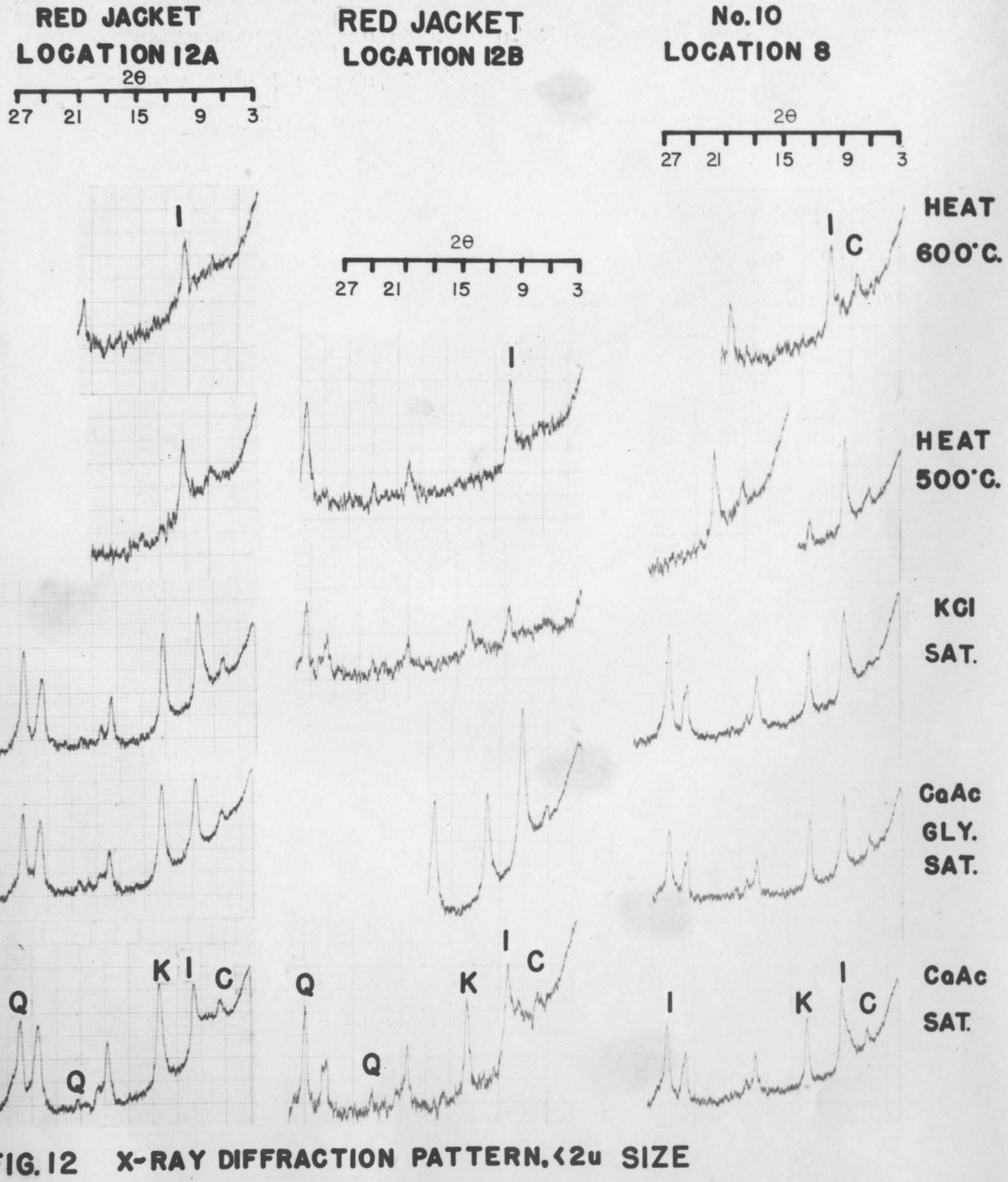


FIG. 12 X-RAY DIFFRACTION PATTERN, <2u SIZE

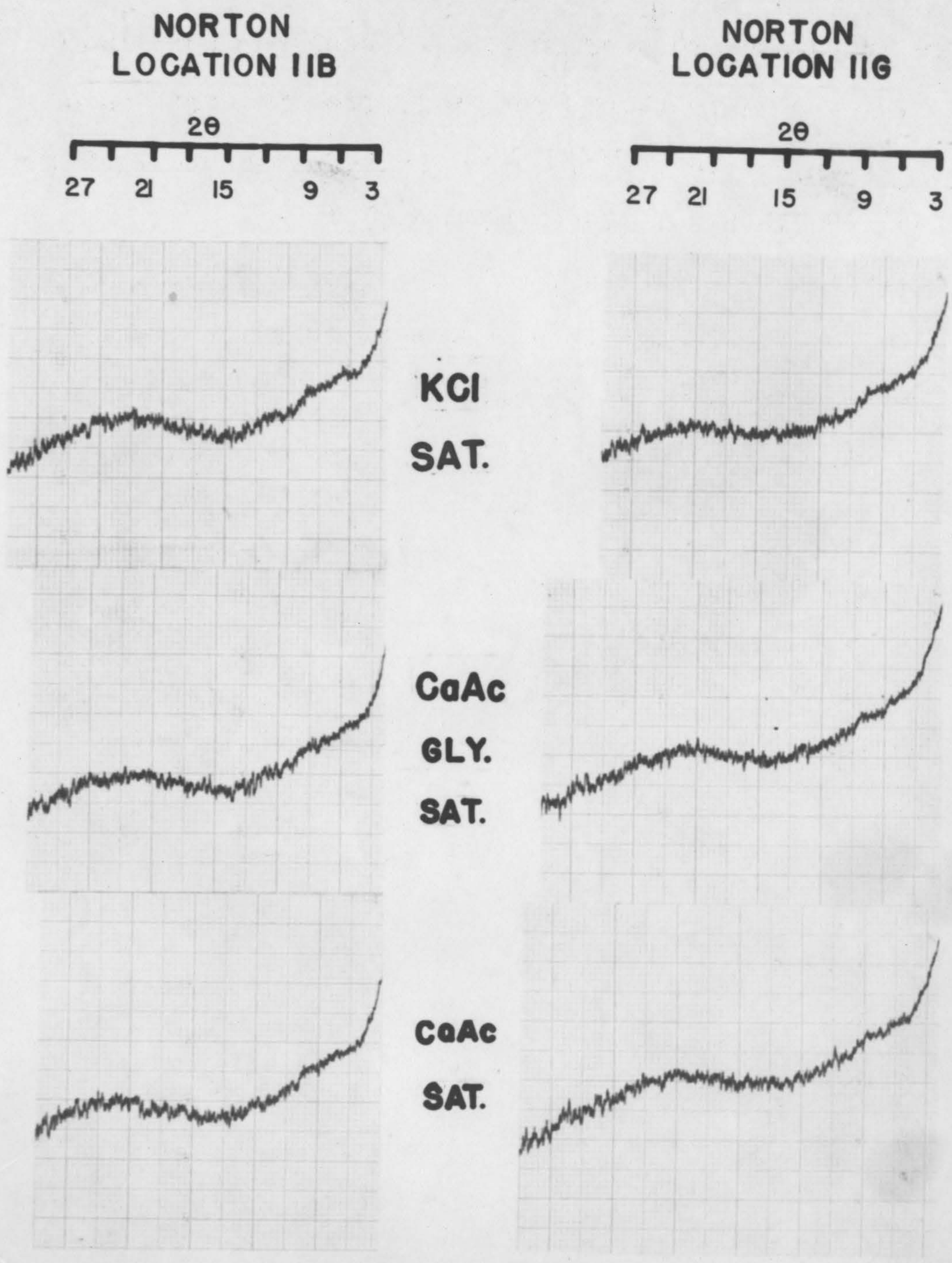


FIG. 13 X-RAY DIFFRACTION PATTERN, <.08u SIZE

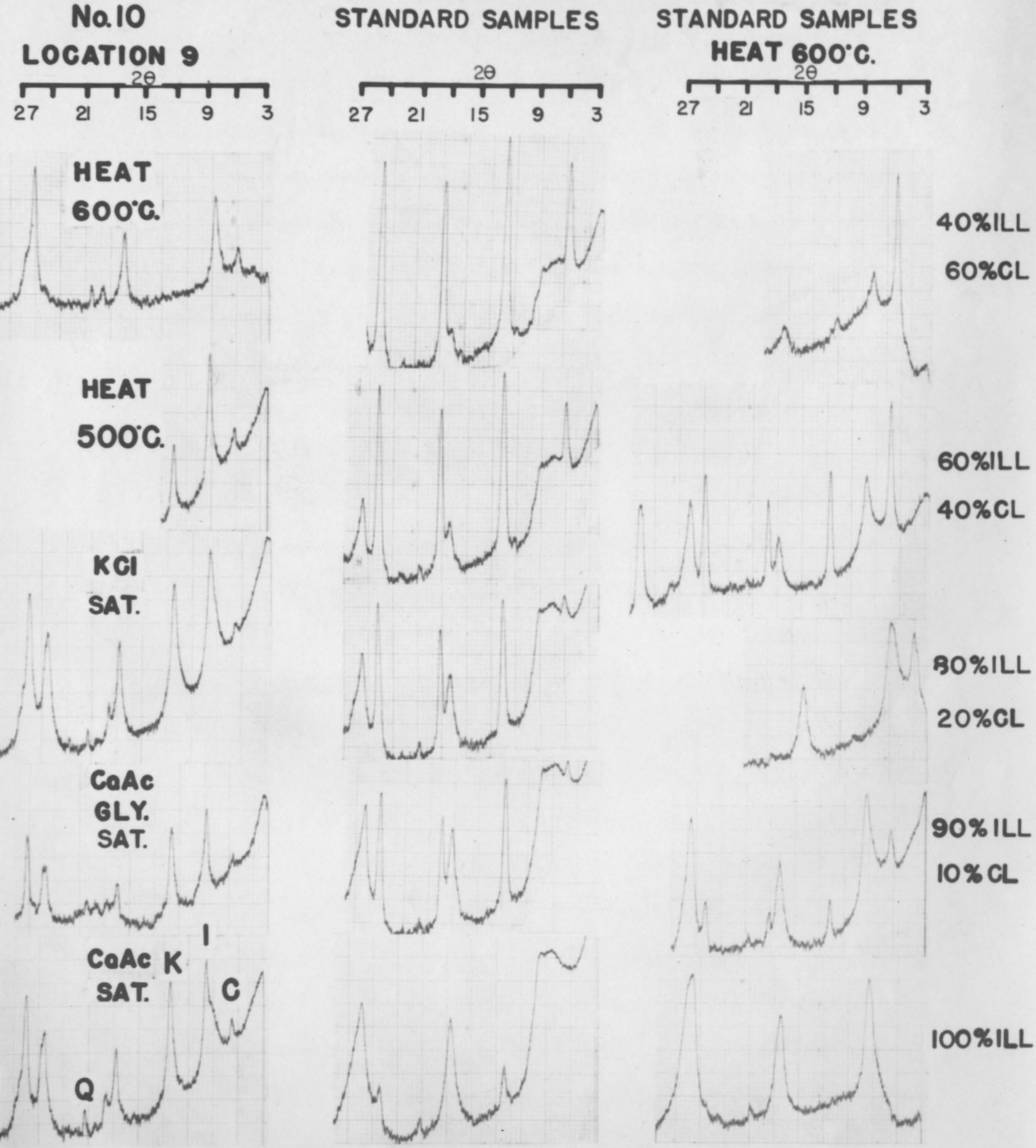


FIG. 14 X-RAY DIFFRACTION PATTERN, < 2u SIZE

solvated and potassium-saturated clays. If the 14.4 A. peak represents montmorillonite in the pattern of the calcium-saturated clay, it would shift to 17.7 A. on glycerol solvation. The same peak collapses on potassium-saturation if it is the result of the presence of vermiculite. Only if it is caused by the presence of chlorite does it remain at 14.4 A. (Bray, 1937; Jackson and others, 1949). A confirmation for the absence of vermiculite is heat treatment at 500°C. for two hours. The 14.4 A. peak remains even after such heating if the mineral present is chlorite. The first and third order peaks of the chlorites are weaker than the second and fourth orders, thus indicating that the chlorite is iron-rich (Brindley, 1951). The chlorite peaks may be displaced from their normal 14.4 A, 7.2 A., 4.67 A., and 3.52 A. values because of irregular substitution of iron for magnesium in the lattice (Grim, 1953)

Illite reflections are found at 10.0 A., 5.0 A., and 3.34 A. units. The 10.0 A. peak may be asymmetrical with a sharp edge at the lower values of the Angstrom units (corresponding to a higher 2 θ value), and a gradually increasing edge at the higher Angstrom values. The broad edge of the 10.0 A. peak is eliminated on heating the the slide above 540°C. It is believed that the broad edge of the peak is caused by a partial conversion of the illite to montmorillonite with an interpolation of water molecules between the layers causing the broad edge (Stephen, 1952). The conversion is not complete, as is shown by the absence of the 17.7 A. peak on glycerol-solvation (MacEwan, 1944).

Kaolinite is suspected when there are strong peaks at 7.2 A. and 3.52 A. units. The first peak reduces on heating the clay to 600° C. which proves the presence of kaolinite. It does not completely disappear because it represents in part the second order of chlorite (Ross and Kerr, 1931).

Quartz peaks are found at 4.26 A. and 3.35 A. Only the 4.26 A. peak can be used for quantitative determinations because the 3.35 A. peak is superimposed on the third order of illite (Figure 16).

The x-ray patterns of the minus 0.08-micron size fraction of samples 11G and 11B shoe no well defined peaks (Figure 13). This is interpretated to mean that only broken fragments of the larger, better crystallized minerals are present in this size fraction.

The 50-micron size fractions contain illite, chlorite, kaolinite, quartz, iron oxides, microcline, calcic plagioclase, and iron sulphides (Figure 15).

Partial chemical analyses.- The potash content of the clays is assumed to belong to the illite present. Pure illite is assumed to contain six per cent potash. The

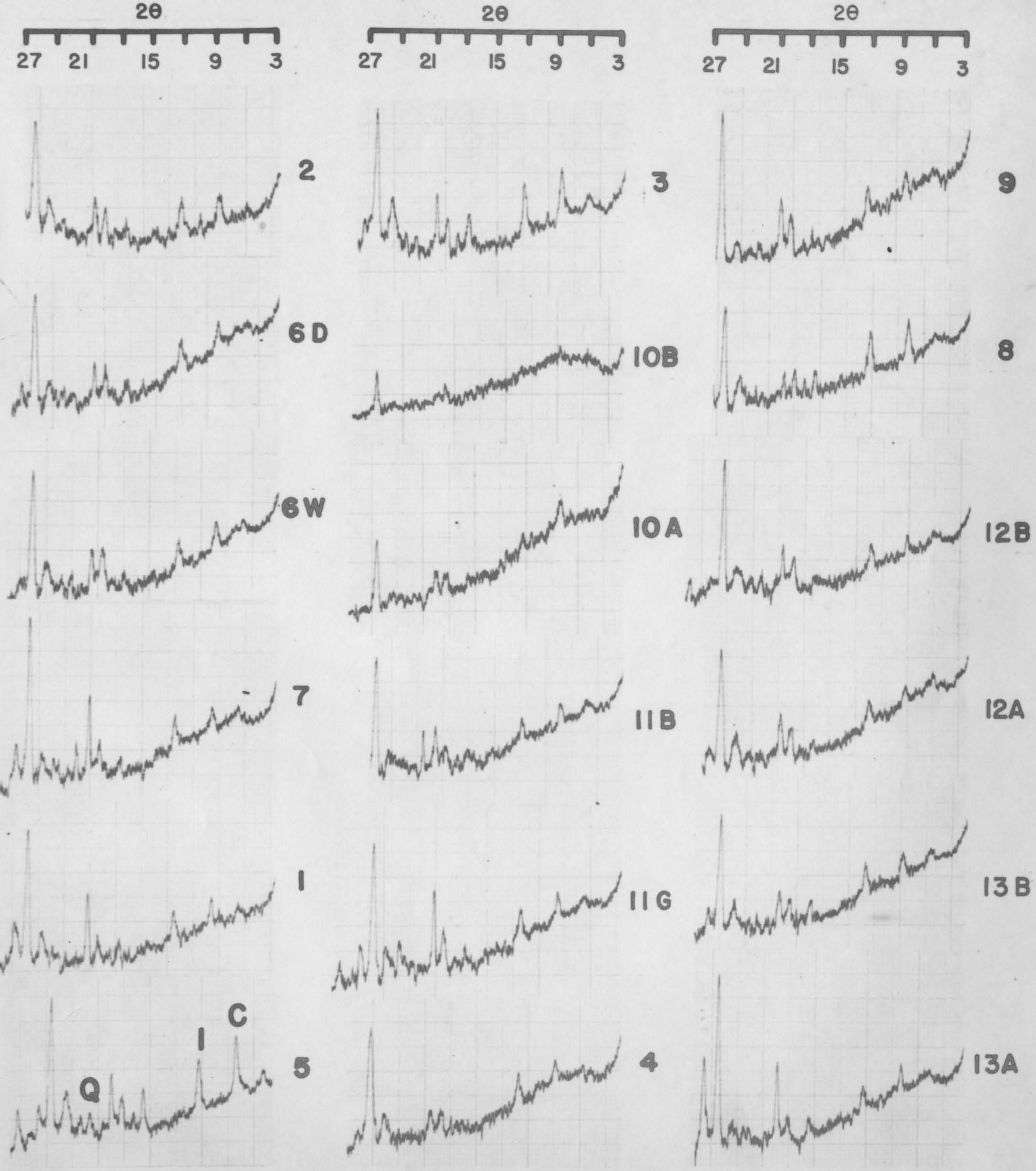


FIG. 15 X-RAY DIFFRACTION PATTERN, >50u SIZE

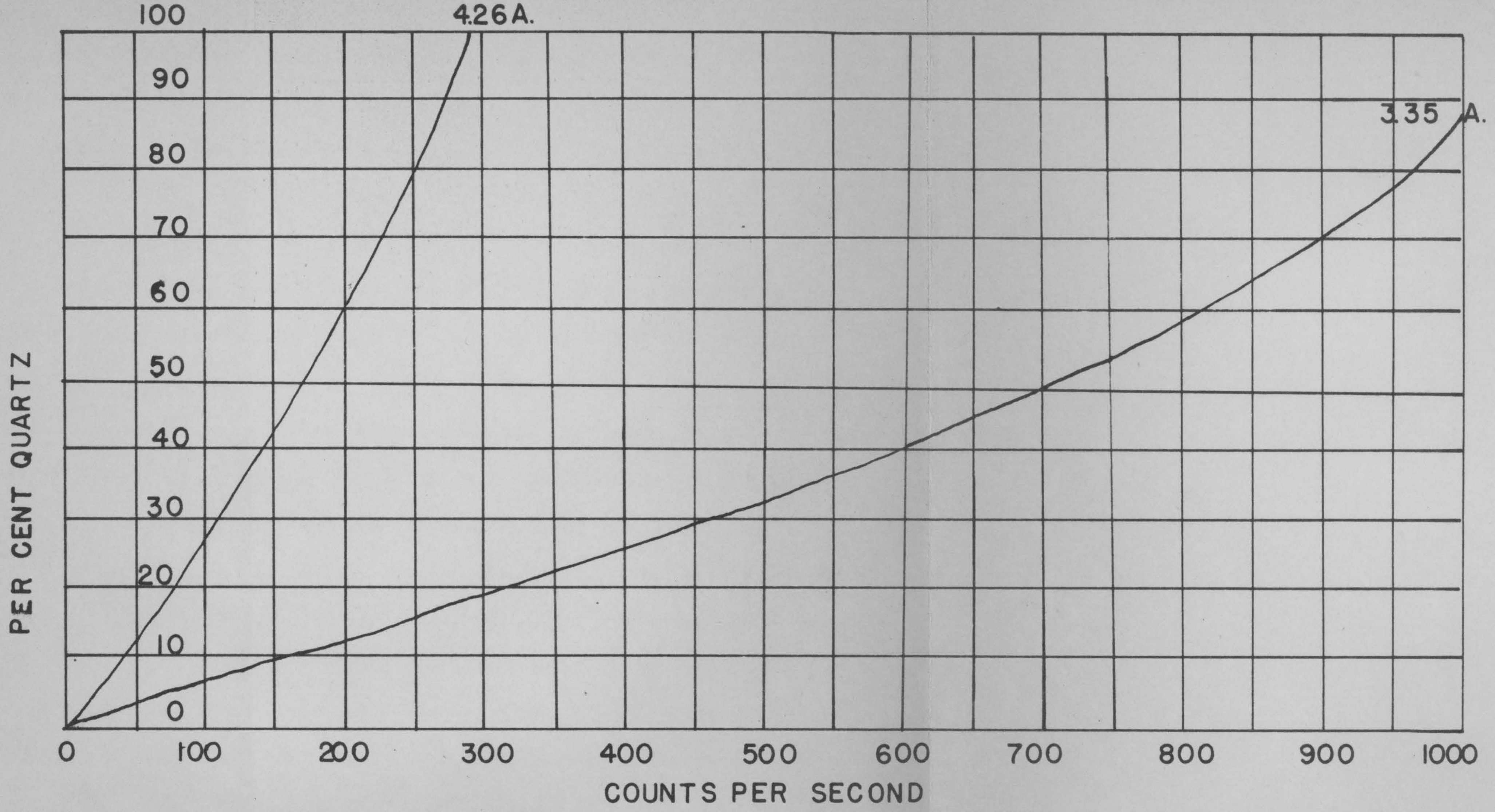


FIG. 16.- QUARTZ CALIBRATION CURVE WITH 50mgm. PER SLIDE OF 2-.08u SIZE. (BASED ON INFORMATION FURNISHED BY DR. C. I. RICH, DEPARTMENT OF AGRONOMY, VIRGINIA POLYTECHNIC INSTITUTE).

amount of illite present is computed from the ratio of the per cent potash present to six per cent potash (Table 6).

The chlorite, iron sulphides, and iron oxides all contribute to the total iron content of the shales. The free iron oxides are believed to be formed for the most part from the iron sulphides. The free iron oxides from the wet part of the mine sampled (6W) are twice as concentrated as those from the dry part of the same mine (6D). Iron sulphides have about half the volume of their equivalent weight in oxidation products. This factor may be of importance in causing roof failures in those mines which are continuously wet because of ground-water seepage.

Soluble salt analyses.- The x-ray and wet chemical analyses show the presence of Halite (NaCl), Chloromagnesite (MgCl), Thenardite (Na SO), and several forms of MgSO (The anhydrite-MgSO , the hexahydrite-MgSO 6H O, and the tetrahydrite forms have been identified by x-ray analyses). The ions extracted from 20 gm of ground shale are shown in Table 7. The fastest weathering roof shales have about four times as much MgSO in them as do the other shales. Graham (1954) States that these salts may cause spalling of a shale if they crystallize within it, they may cause blistering of paints applied as protective coatings on the roof. He also states that there is little correlation between the amount

Table 6.- Partial chemical analyses of clays extracted from roof shale samples.

Sample	K_2O	Illite	Total Fe_2O_3	Free Fe_2O_3
1	3.03	61.6	-	-
2	2.73	45.5	-	-
3	4.39	73.2	-	-
4	3.67	61.2	-	-
5	3.70	61.6	-	-
6D	3.33	55.5	7.29	2.72
6W	3.26	54.3	5.01	4.49
7	3.18	53.0	-	-
8	3.91	65.2	-	-
9	2.62	43.7	-	-
10A	3.24	54.0	5.13	-
10B	3.36	56.0	-	-
11G	2.90	48.3	2.46	-
11B	2.50	41.7	3.20	-
12A	2.82	47.0	7.17	-
12B	2.42	40.4	5.40	-
13A	2.56	42.7	-	-
13B	3.66	61.0	-	-

All numbers are percentages by weight of the constituents.

Table 7.- Soluble salt content extracted from shales by leaching with distilled water.

Sample	Na^+	Mg^{2+}	SO_4^{2-}	Cl^-
1	.0060	.0083	.610	.310
2	.0129	.0031	.469	.140
3	.0069	.0028	~~1~~.625	.012
4	.0058	.0009	.408	.010
5	.0043	.0150	.074	.088
6D	.0751	.0032	.328	.045
6W	.0230	.0008	.407	.060
7	.0251	.0003	.000	.069
8	.0052	.0025	.625	.030
9	.0360	.0003	.624	.087
10A	.0043	.0325	2.430	.231
10B	.0020	.0070	.313	.019
11G	.0052	.0013	.950	.010
11B	.0042	.0007	.407	.016
12A	.0776	.0020	.920	.197
12B	.0028	.0130	.344	.025
13A	.0210	.0013	.156	.003
13B	.0026	.0004	.625	.002

All values are percentages by weight of the constituents.

of salt that can be leached and that which effloresces.

Taber(1917) has shown that salt crystals can grow in the direction of an opposing force because of the molecular forces associated with the separation of solids from liquids. It has been shown that crystals can grow in limited areas so long as they are supplied with supersaturated solution (Graham, 1954).

The writer believes that the salts extracted were originally deposited with the sediment or else they were carried to the position found from some overlying saline deposit. Not only does the mineral composition suggest the sediments were deposited in a brackish environment, but also the composition of the salts extracted indicates that the source of the salts is connate sea water. Illites are believed to be formed in marine or brackish water conditions (Grim, 1953). If iron salts had been extracted, it would have been easier to explain the salts as being derived from the oxidation of sulphides found in the shale.

The writer believes these salts contribute to some roof failures. The magnesium and sodium sulphates found are efflorescent minerals, which would explain why there is spalling of the shales only when there is much water on the roofs. The growth of the salts by the incorporation of water in their crystal structure is believed to be strong enough to force layers of shale apart.

Mechanical analyses.- The percentage of clay size material present in the shales is given in Table 8. The amount of clay present which was estimated in the petrographic study of the thin sections is also given in order to show the relationship of the two methods of analysis. The writer believes a refinement of the mechanical separation could be made, and a more complete extraction of the clay sizes then be obtained. However, he does not believe that any major difference in the mineral composition or the percentages of the minerals present would be achieved by such efforts.

Table 8.- <u>Clay content of shales from mechanical analyses</u>.[*]

Sample	Clay Extracted	Clay Present
1	5.6	15
2A	5.85	20
2B	7.05	20
3	5.0	30
4	3.76	30
5	12.0	40
6D	12.7	20
6W	15.7	20
7	12.0	10
8	8.9	23
9	10.95	15
10A	21.8	25
10B	13.7	40
11G	13.2	-
11B	15.1	-
12A	9.5	20
12B	10.1	20
13A	7.5	5
13B	12.9	20

[*] Clay content is expressed as per cent by weight of ground shale. The clay present is based on a petrographic estimate.

SUMMARY

Petrographic study reveals the presence of mica, clay, quartz, feldspar, organic matter, pyrite, and chalcopyrite in the roof shales sampled. Fewer failures occur in those roofs with the majority of the grains larger than 0.25 mm than in finer grained roof rocks.

X-ray diffraction patterns, differential thermal, and chemical analyses show that most of the clay fraction of the roof shales is illite and kaolinite. Minor amounts of chlorite, quartz, and iron oxides are also present. These minerals do not swell appreciably on contact with water, and therefore they are not believed to contribute significantly to the spalling of roof shales.

Chemical analyses of the free iron oxides in one mine shows that there is about twice as much iron oxide in the part of the mine which is continuously wet as in the part which is dry. It is believed that most of these oxides are formed from iron sulphides. The expansion in volume that occurs during oxidation is believed to cause some of the roof failures.

Sodium chloride and magnesium sulphates are present in the roof shales in forms that can be leached out readily at room temperature with distilled water. These salts are present in greater quantity in weak roof shales than in the strong shales. Some of the salts can be induced to effloresce on the surface of the shales. This same efflorescence is believed to occur along constricted bedding planes in the roof shales and thus cause roof failures.

No one factor is the cause of all roof failures which have occurred in the area studied. This means that remedial treatment must be designed for each particular mine in order to be effective.

RECOMMENDATIONS FOR FUTURE WORK

The writer believes that before any roof treatment, either preventative or remedial, is attempted in a mine, the cause of the failures in that particular mine must be determined.

The writer believes that the quantity of water that penetrates a roof is a significant factor in roof failures, and that it should be studied to see what amount of water passage is necessary before failure occurs. This study should be supplemented by a study of the composition of the ground water before and after it enters a mine. The study of the iron sulphides and iron oxides in the roofs of mines with this water condition should tell which part of the mine weathers the most.

In those mines whose roof failures appear to be caused by the action of condensed moisture on the roof shales, analyses should be made in order to determine the nature and quantity of soluble salts present. Then experiments with barium and water-base paints should be made in order to see if their application to roofs reduces failures. These tests should be run on freshly exposed and on long exposed roof shales. Laboratory tests should be attempted using reagents that cause the salts to be non-efflorescent. Tests might be devised with the goal of finding a binding agent for the salts and clays in the roofs.

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