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# Insoluble Residues of the Lower Mississippian Limestones of the Madison Group

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Montana School of Mines Butte, Montana

INSOLUBLE RESIDUES OF THE LOWER MISSISSIPPIAN
LIMESTONES OF THE MADISON GROUP

A Report
Submitted to the Department of Geology
in Partial Fulfillment of the
Requirements for the Degree of
Bachelor of Science in Geological Engineering

by

Fred G. Lines

May, 1942

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#### INTRODUCTION

The correlation of non-fossiliferous drill samples is one of the difficult problems that is encountered in sub-surface stratigraphy. In order to truly correlate a formation, it must have some distinctive features and have an areal persistence of these features. These requirements are probably met best by limestones. These are common rocks in subsurface work, and are found in sediments of all geologic ages excepting perhaps the Archeozoic. The usual means of identification is by faunal or lithologic characteristics. Commonly though the limestone will show no characteristics in these features. Insoluble residues have been found to be valuable for this type of problem.

The composition of limestone is primarily calcium carbonate, usually with some magnesium present in the form of dolomite. Besides these there are many insoluble minerals that are present in variable, but usually relatively small amounts; after acid digestion these minerals are left as a residue. In this paper the insoluble residues that will be mentioned will refer to that residue that is left after a limestone has been dissolved in warm hydrochloric acid.

The problem involved in this report is the correlation by insoluble residues of Lower Mississippian limestones of the Madison group. A preliminary investigation was made on the heavy mineral fraction of these residues, with a view towards correlation and also to determine the character of the nearby land masses. A little work was also done with spectroscopy; spectrograms being made of the heavy mineral fractions of the insoluble residues and of the original limestone. It was thought that correlation of limestones on the presence or absence of certain uncommon elements might be accomplished with the aid of spectroscopy.

## Previous Work Done

It had long been recognized that limestones might be correlated by insoluble residues. In 1925 the first practical use was made of this principle by McQueen, who used them while working on limestones in Missouri, in connection with drilling for sub-surface water. Since this time numerous workers have further proved the practicability of this method of correlation, and it has become a common tool to petroleum geologists.

Most of the work done was in the Mid Western states of the United States, and it was not until 1938,

<sup>1</sup> McQueen, H. S.; Insoluble Residue as a Guide in Strati-Studies; Missouri Bur. Geol. and Mines, Bien. Rept. 56, Append. 1, 1931.

that any appreciable amount of insoluble work was done in this part of the country. At that time, Ralph H. Hamblin and L. L. Sloss commenced to work the Upper Paleozoic formations of Montana, with particular reference to the Madison group. This report is in part a continuation of this work.

There has been very little, if any, work done on the heavy mineral fractions of the insoluble residue.

Milner mentions the heavy minerals of limestone, but says nothing about correlation by this method.

In respect to spectrographic correlation, as far as this writer knows, there has been nothing done with limestones. Some work of this sort has been done on lava flows with satisfactory results.

# Value of Insoluble Residues

Insoluble residues have been chiefly used as a tool for limestone correlation. Its application here is practicle and evident. McQueen<sup>2</sup> in his paper mentions that "Insoluble residues have a wide application in strictly scientific studies. They show the character of the rocks and the different constituents, and thus indicate the character of the sediments that were deposited, the possible source of the materials and the conditions under which they were laid down. In the

<sup>1</sup>Milner, H. B., Sedimentary Petrography

<sup>2</sup>See foot-note on page 2

method of preparation, the presence of organic matter can be easily detected. This determination is especially important in the field of Petroleum Geology."

# Acknowledgments

The writer wishes to acknowledge his indebtedness to Dr. L. L. Sloss, of the Geology Department of
the Montana School of Mines, for the assistance given
on the problem, to Dr. Eugene S. Perry, also of the
Geology Department, for his instruction on thesis
writing and finally to Dr. S. R. B. Cooke, of the
Mineral Dressing Department, for running the spectrograms mentioned in this report.

. 5

#### GEOLOGY

This paper is primarily concerned with the Madison limestones of Montanal. These limestones are an enormously widespread group, that were deposited in the central part of the Cordilleran geosyncline and in the epicontinental seas that covered the continent to the east. The name Madison was originally given to the Montana occurrences, but these rocks can be followed into all the surrounding states and into Canada. The limits of this group or of similar limestones with different names, is from Colorado to Central Alberta. To the east and west these sediments are concealed beneath overlying strata, but it is possible that they join with similar limestones of Kinderhook and Osage age in the Mississippi Valley.

# Stratigraphic Relations

The following stratigraphic column is given to show the relationship of the Madison with older and younger formations, as is found in South Boulder Canyon, Montana.

Jurassic

Ellis

Permian

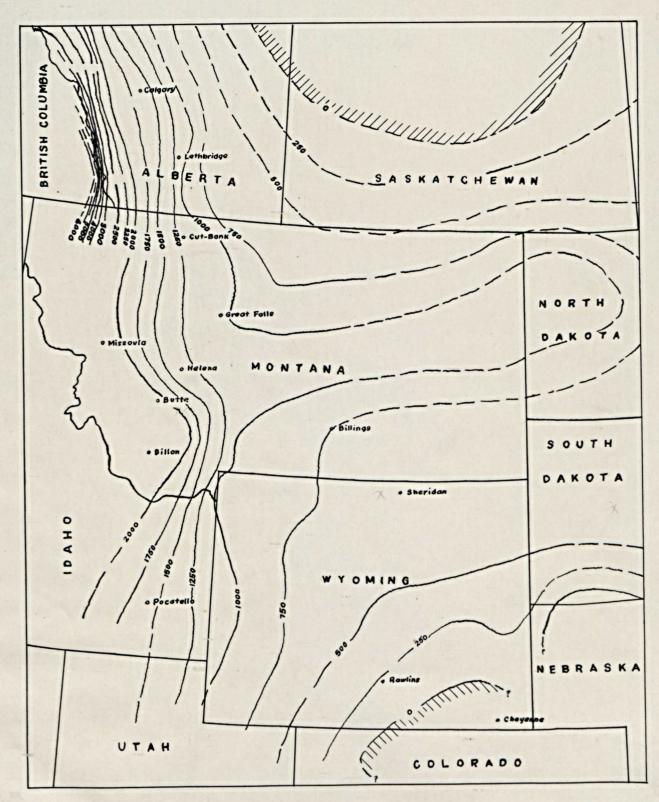
Phosphoria

Pennsylvanian

Quadrant

Amsden

For further information on the Madison group see: Sloss, L. L., and Hamblin, R. H.: Stratigraphy and Insoluble Residues of Madison Group (Mississippian) of Montana. A.A.P.G. Bull., vol. 26, no. 3, March, 1942.



ISOPACH MAP OF THE MADISON GROUP

Mission Canyon fm.	Lodgepole fm.	Three Forks sh.	



The Madison formation as it outcrops in the South Boulder Canyon.

Mission Canyon

Mississippian

Madison grp.

Lodgepole

Devonian

Three Forks shale

Jefferson dolomite

This above relationship does not hold over the entire area. The Madison rests on the Upper Devonian (Three Forks) over most of Montana, Southern Alberta and Wyoming. To the south it rests on progressively older beds, until it lies on Pre-Cambrian rocks in the Laramie Mountains of southern Wyoming. The Big Snowy group in central Montana, and the Sacajewea formation in Wyoming, lie conformably above the Madison. Over most of the remainder of the area the Amsden formation lies above the Madison, while to the north in northern Montana and in Canada the Ellis is the next higher formation.

# Divisions of the Madison

In Montana the name Madison was first given to the lower Mississippian limestones of the state by Peale 1. It was soon recognized that this thick section could be easily subdivided into two or three subdivisions. This has resulted in the following classification:

Group

Formation

Member

Mission Canyon

Madison

Woodhurst

Lodgepole

Paine

lpeale, A. C.; The Paleozoic Section in the Vicinity of Three Forks, Montana; Bull. U.S.G.S. 110, 1893.

The name Madison, which was first applied to the Lower Mississippian limestones found in central Montana, has spread, so that it is now given to similar limestones in Alberta, northern Wyoming, and in the western part of South Dakota as far east as the Black Hills.

## Lithology

On outcrops the Madison limestones are quite distinctive in appearance. In general the Mission Canyon weathers into massive blocky cliffs, often in turreted shapes. The limestone is dense, fine-grained, light grey, poorly bedded and massive. In the upper part, solution breccias are common, while in the middle, chert partings are numerous. As a whole the formation is quite fossiliferous, corals, brachiopods, crinoids and sponge spicules being common.

The Lodgepole formation is a thin bedded, dense limestone, variable in color. Characteristically it has beds an inch to three feet thick, separated by shale partings and has a persistent basal black shale bed. The Woodhurst is a massive limestone, commonly dark gray, fine grained with beds usually from two to six feet thick, separated by thin calcareous shale beds. The Paine has thinner beds and thicker shale partings,

which give this member a bacon rind appearance on a weathered surface.

In sub-surface work, it has been found that the Madison is often difficult to identify or sub-divide. First the lithology ceases to become distinctive from the many other limestones, and fossils cannot be reliably used for the identification of units of the group. "There are intirely too many localities which yield no fossils or where the fossils are so tightly bound in the matrix as to make recognition impossible."1

## Madison History

Late Paleozoic time found this part of the continent in a very dormant state. Epierec seas and the lack of mountains were the rule. The Cordilleran trough, lying north and south, crossed this part of the country through Idaho, eastern Washington, and western Montana with a shallow basin area running east from the trough over Montana, Wyoming, Alberta, and southern Saskatchewan.

This basin was the principle feature of Madison history in this part of the country. It was bounded on the north by a positive area in Saskatchewan, and on the south and southeast by positive areas in Colorado

<sup>1</sup>See foot-note on page 5.

and Nebraska. At its greatest extent the sea, which filled this area, probably spread as far as the Missispi Valley.

The historical sequence of events is given by Sloss as follows:

- 1. Local withdrawal of Devonian sea and marked changes in sedimentation and fauna.
- 2. Deposition of organic shale transition beds in shallow isolated basins.
- 3. Invasion of Paine sea from west into shallow east-west trough, through central Montana.

  Deposition of Paine beds in sea, subject to periodic influxes of clastic matter from west.

  No deposition in southeast over an area also lacking Silurian and Devonian strata.
- 4. Further spread of sea north and south and uniform deposition of Woodhurst beds. Clastic
  material diminishes and indirect stratification
  active with concentration of crinoid and
  brachiopod fragments in thick beds.
- 5. Farthest spread of sea and deposition of Mission Canyon limestone. Complete absence of sand and silt except in southeast margin of basin.
- 6. Withdrawal of sea. Exposure to subaerial erosion and solution active during part of middle and late Valmeyer time.

<sup>1</sup>see foot-note on page 5.

- 7. Transgression of late Valmeyer and early
  Chester seas. Deposition of Big Snowy sediments in central Montana, Sacajewea in northern
  Wyoming. Collapse and filling of caverns and
  channels. Continued erosion in area of MontanaWyoming border. Stripping of Madison from
  northern flanks on Uinta Front Range element
  (Wyo-Col) and Cambridge arch (Neb).
- 8. Deposition of later Paleozoics (and Triassics in south).
- 9. Early Mesozoic erosion north of present Big
  Snowy and Belt mountains, Montana, to form karst
  topography on the Madison where exposed. Madison completely removed in northern Alberta and
  northern Saskatchewan.
- 10. Deposition in Jurassic of France, Ellis, and Sundance.

# Economic Significance

The limestones of the Madison group are relatively important economically, chiefly as a reservoir rock for oil. In many widely separated localities, oil ang gas are being produced from this formation, the present important active fields are Turner Valley (Alta), Kevin-Sunburst, Pondera, (Mont), Garland and Frannie (Wyo).

The location of these pools seem to be governed more by porosity and permeability than by structure. So far all the oil has come from the Mission Canyon, from the top 10 to 100 feet below the erosion contact with the next succeding formation; the Lodgepole does not appear to be so susceptible to the factors which locate the Mission Canyon oil. These factors are chiefly solution action, that took place during the erosion of the top of the Mission Canyon in Pre-Jurassic exposure, and to dolomitization, where the calcite crystals have been replaced by dolomite crystals with a resulting reduction of space. Since both these factors are widespread, it may be said that the potentialities of oil in the Madison are considerable, and thus the practical value of an investigation of this type.

Besides oil, the Madison limestone is used for cement manufacture, and for industrial and agricultural lime.

# Areas Sectioned

The only area sectioned by this writer was at South Boulder Canyon, Montana. This area lies about 17 miles southeast of Whitehall, Montana. The canyon, cutting across the strike of the beds, has exposed the complete stratigraphic column for this part of the state. The Madison is about 2000 feet thick and occurs as a

Perry, Eugene S.; Natural Gas in Montana, Montana School of Mines Bulll, Memoir No. 3, Dec., 1937.

massive cliff about half way up the section. It can easily be divided into the Mission Canyon and Lodgepole formations.

Of the other areas sectioned, O. A. Seager of the Carter Oil Company took samples at Spearfish Canyon, in the north central Black Hills of South Dakota, at Warren Quarry, in Carbon county, Montana, at LaPrel Creek, in Converse county, Wyoming. The writer here wishes to thank Mr. Seager for the use of these samples. The samples from Cutbank were taken from Regan Tribal Well, No. 15.

Hamblin had previously sectioned and run insoluble residue analyses on areas at Lodgepole Creek in the Little Rockies, at Swimming Woman Canyon in the Big Snowy Mountains, at Monarch in the Little Belt Mountains, at Logan, Montana, at Rock Creek in the Madison Range near Jackass Creek, and in the Gallatin Range at West Fork.

<sup>1</sup> Hamblin, Ralph H.; Stratigraphy and Insoluble Residues at the Upper Paleozoic Formations of Montana; Masters Thesis, Montana School of Mines, 1938.

#### PROCEDURE

#### Insoluble Residues

The procedure for running insoluble residues varies slightly, but the following outline is the general method. For this work samples were taken at measured intervals along the outcrops. Since unweathered rock is the most desireable, the weathered surface was broken off and the relatively fresh rock beneath was taken. Actually all outcrop samples of this sort are weathered to a certain extent, but usually not enough to influence the results.

The best interval for taking samples is 5 feet, most of the samples were taken at this interval, but those taken at South Boulder were from 10 to 15 feet apart.

The next step in the procedure was crushing, the purpose of which was to increase the surface area of the rock and thus speed up the time it took to completely digest the sample.

After some experimenting the following procedure of crushing was found to be the quickest and most efficient. The samples were run through a Tyler Gyratory Crusher. This ground the rock to about a half inch maximum. Care had to be taken to clean out the crusher and after each sample the crushed sample was then screened to plus 20 mesh, and was ready for

either crushed to too small a size, which destroyed many features of the samples and produced too many fines, or were too hard to clean cut after each crush. Hand bucking was tried, but proved to take too long.

#### Treatment

The crushed samples were dissolved in C. P. hydrochloric acid, diluted with two parts of water.

The amount taken was measured out volumetrically, as this is much quicker and still accurate enough, since the main interest was not quantitative, but qualitative.



Fig. 1. Crushed limestone being treated with acid over the hot plate.

A 30 cc sample was measured out and put into a 200 cc glass. The acid was added to this in small amounts, as the effervescence was usually vigorous. The glasses were then put on the hot plate, and as soon as effervescence ceased more acid would be added, until the glass was filled. The solution was then decanted and more acid added.

when the limestone had been completely digested, the solution and slimes were decanted and the residues were thoroughly washed to remove the last traces of acid and the soluble salts. The easiest method of drying the residues was to leave them in the glasses, and when dry to put them into labeled vials.

# Inspection of Residues

The residues were inspected under a low power binocular microscope which had magnigications of 6.4, 26.6, and 43.5. Illumination was obtained either with a spot lamp or with three 100 watt bulbs that were mounted in a semicircular position on the underside of a platform, whose height was slightly less than that of the eyepiece of the instrument. This latter device proved to be very satisfactory, as this arrangement of lights prevented any shadows while using the instruments.

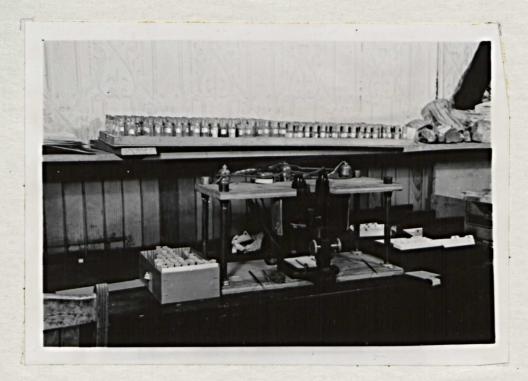


Fig. 2. Binoculars used for the inspection of samples. Note the stage used for illumination.

# Heavy Mineral Separation

A heavy mineral separation was made of the insoluble residues of the Madison limestone that had been sectioned at Monarch in the Little Belt Mountains, Montana. This process involves removing those minerals that have a relatively low specific gravity. To do this a float and sink method is used. This is based on the premise that when an object is put into a liquid it will float, if its specific gravity is less than that of the liquid, and sink if its specific gravity is greater than that of the liquid. By using heavy liquids heavier

fractions can be floated. Of the many heavy liquids available ethyl tetrabromide or tetrabromethane was used. It has a density of 2.964 at 20°C. Since it is quite expensive care had to be taken so as to lose as little as possible. The washing liquid used for



Fig. 3. The apparatus used for heavy mineral separation.

removing the heavy liquid from samples was carbon tetrachloride.

Separatory funnels were used to separate the various fractions, and were found to be satisfactory, though for this work, where speed is essential there

are numerous other methods that can be used. Funnels and flasks completed the equipment.

The separatory funnel was about one quarter filled with the heavy liquid. The sample was then poured in, shaken up and washed down from around the side of the funnel. After allowing time for settling the bottom half of the liquid containing the heavy fraction was poured out into a filter paper. This filter paper retained the sink fraction of the sample, and after the tetrabromide had all gone through, the filter was removed to another funnel and washed thoroughly free of all the heavy liquid.

Four solutions of carbon tetrachloride were used, the last of which was almost pure, and the first being relatively saturated with ethyl tetrabromide.

When the washing fluid became saturated it was set aside for evaporation, and in this way practically none of the heavy liquid was lost.

The float fraction was treated in the same manner as the sink. Both of these, when cleaned, were dried on a hot plate, and then put into vials.

To inspect the heavy minerals the same apparatus was used as was used with the whole residues.

For the non-opaque minerals, petrographic analyses were

mortar used was cleaned between samples by grinding quartz in it.

A small cut of the sample was put in a carbon anode, and an arc was started between the above anode and another carbon cathode. Under the intense heat of the arc the elements in the material are excited. In this excited state each element gives off a characteristic series of spectrum lines. These spectra are directed into the spectograph, are split by a diffraction grating and a certain range are photographed.

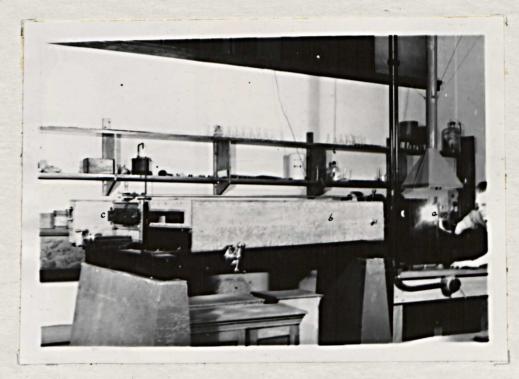


Fig. 4. The spectrograph in operation.

a.-the arc being generated.

b.-box in which the spectrum is diffracted.

c.-camera, on which the film inside this the spectrum lines are being impressed.

first run, in order to learn the diagnostic appearance of the various types. For example, one common medium brown mineral was identified as rutile, another light brown mineral was found to be titanite. Thus whenever similar minerals were found under the binoculars they were so named. This method might not have been exactly accurate, especially in the case of the colorless transparent minerals, but time could not be taken to identify every grain that was found. With the opaques, comparisons were made with known minerals, though a few could be identified because of their magnetic properties.

The following table was used for indentifying the minerals:

Opaque Minerals

Dark gray-silver gray. Magnetic ----- Magnetite
Steel gray. Sways under magnet ----- Ilmenite
Metallic black, brown on thin edge ---- Chromite
Brown, rust to reddish ----- Limonite
Black, red and brown in parts, associated

with above ---- Goethite

Brass yellow ----- Pyrite or Marcasite

Note: Probably most of this type of mineral is marcasite, but there was no way of making certainon the small amounts found, so hereafter in this report all such minerals will be called pyrite.

White or gray, usually with pits on the surface,

Sways to magnet ----- Leucoxene

Note: An ilmenite core accounts for the magnetism.

Non-Opaque Minerals

Usually violet tinted, frequently as octohedrons or as triangular plates. Isotropic ----- Fluorite Irregular, often fractured, commonly pale pink grains, optically isotropic ---- Garnet Colorless, glassy subangular irregular grains- Topaz Colorless, often in prismatic crystals, showing evidence of being of secondary origin ----- Barite Colorless to varicolored, tetragonal crystals with pyramidal terminations ---- Zircon Dark brown -----Rutile Light brown ----- Titanite Brown to red, prismatic and pyramidal grains -- Cassiterite Apple green, generally rounded elongated, prismatic, Blue to black, a few show vertical striations, some are flat, platelike and well rounded --- Tourmaline

# Spectroscopy

Since only a few samples were analysed by this method, the procedure will not be explained in any detail, but only very simply.

The samples were first crushed to about 400 mesh. Care had to be taken here in order to see that there was no contamination of the material. Thus the

and analysed. Note that on each film there are five spectograms, this is done with stepped down slots, which allow only part of the film to be exposed at one time. With each series, one iron spectogram is run. This is to establish iron lines, the length of which are known, and from these measurements can be made to unknown limes and their length found. Actually the quickest way of finding elements present in the spectogram, is by superimposing the unknown over a spectogram of a known element.

## Photographs

The photo-micrographs that were made were taken on a Reflex-Korelle camera. A binocular microscope was used for the magnification of the residues, this being found preferable, since it gave better depth perception, and had easily manipulated objectives that were mounted on a revolving stage. In taking the photographs, one ocular of the binoculars and the camera lense were removed. A metal tube was screwed into the camera and a tube slid into the microscope. This set-up resulted in magnifications of 6, 15.6, and 40.8 diameters. A spot lamp was used for illumination, and a black background was obtained with black paper. The films were exposed for 1/5 to 1/25 seconds, depending upon the brightness of the subject.

#### RESULTS

## Insoluble Residues

The commonest contituent of the insoluble residues inspected is silica. This occurs in the form of chert, fossil fragments, crystalline quartz, and sand grains. Accessory minerals occur in minor amounts throughout the limestone, while argillaceous material is predominant in certain zones, and is characteristic of the Paine member.

#### Types

Fossils. The fossils and fossil fragments that were found represented many phyla; for the most part they were the result of silica replacement of the original fossil shell. The fragments were so badly broken that complete identification was impossible. The Woodhurst residues are particularly abundant in fossil fragments.

Brachiopods probably form the commonest of the fossil fragments. The striated shells stood out distinctively in the residue, and the curved part of the shell around the umbo was often found.

crinoids are also very common in certain zones. The circular doughnut-like crinoid stem plates are very noticeable. The basal and radial plates are probably just as common as the stem plates, but they are not so distinctive.

Bryozoans do not seem to be as common as

Hamblin reported. This may, in part, be due to the author's unfamiliarity with these fossils, but probably was mostly due to the fact that the Paine member which carries many of the bryozoas in some localities was a predominantly argillaceous material and nothing else was noticed. In two Mission Canyon samples inspected, bryozoans were found in abundance.

Among the other fossils found were sponge spicules of many types and brachiopod spines. Chert is a common constituent of the residues. Much of it occurs as thin flakes that were apparently deposited secondarily along the bedding planes. The casts found are of two different types, Casts siliceous and argillaceous. The siliceous casts were formed by silica filling the limestone's interstitial pores, which in many cases were caused by dolomitization. These are called dolocasts, and vary from a fine latticelike framework of quartz to a porous lump of quartz. The argillaceous casts were due to clastic deposition of fine material along with the limestone ( e.g. calcareous limestone), when this limestone portion was dissolved away it left behind soft porous argillaceous lumps.

Ooloids This name is given to small round particles, usually siliceous and clearly not sand grains. They were found individually and sometimes cemented together

<sup>1</sup> see foot-note on page 12.

in clusters. The origin of these ooloids is probably due to silica replacement.

Granular Quartz This occurs in many forms, ranging from milky drusy masses, to honeycomb structures, to doubly terminated quartz crystals.

Sand Grains Well to sub-rounded sand grains are found throughout the section. These grains are quite small and are liable to pass unnoticed, except in a minute residue.

Accessory Minerals As with the sand grains, the accessory minerals are really only noticable where the residues are minute. With the heavy mineral separations that were run the author found that the accessory minerals were in relatively the same abundance throughout the Madison.

Ferruginous Material This material consists of pyrite, limonite, hematite, and glauconite. The latter two did not appear to be very common. Limonite is particularly abundant in certain zones, occuring usually as porous lumps.

#### General Statement

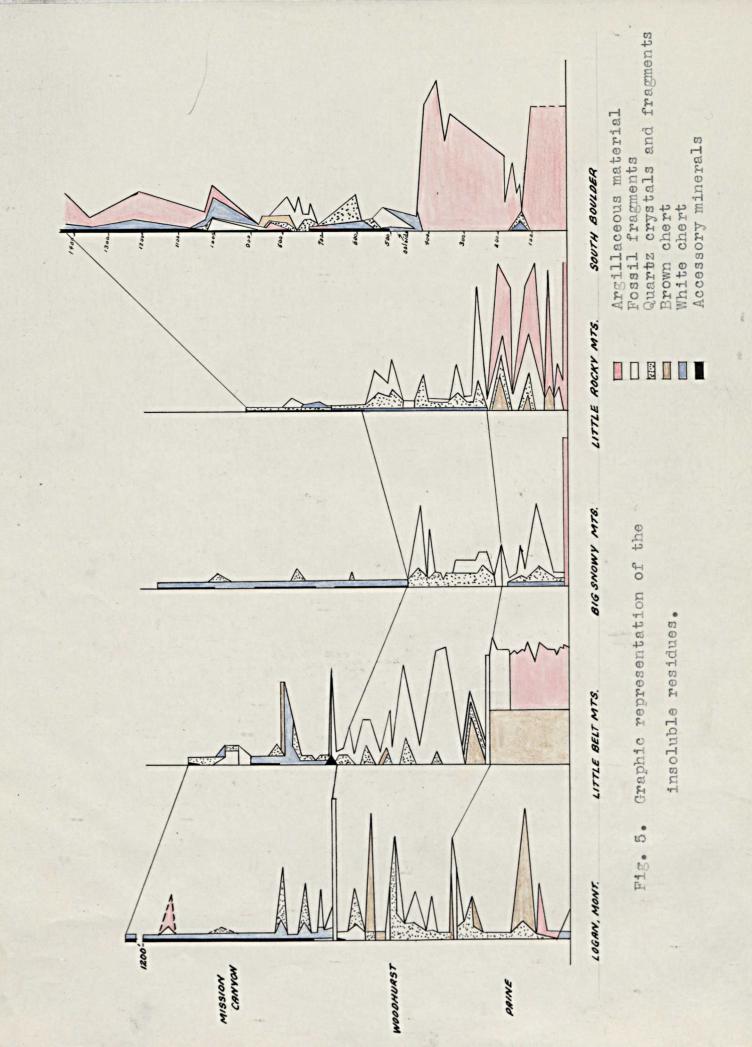
It was the opinion of Hamblin in his report that the Madison could be differentiated on the basis of the insoluble residues. The work done by this writer further confirms this statement. It was found

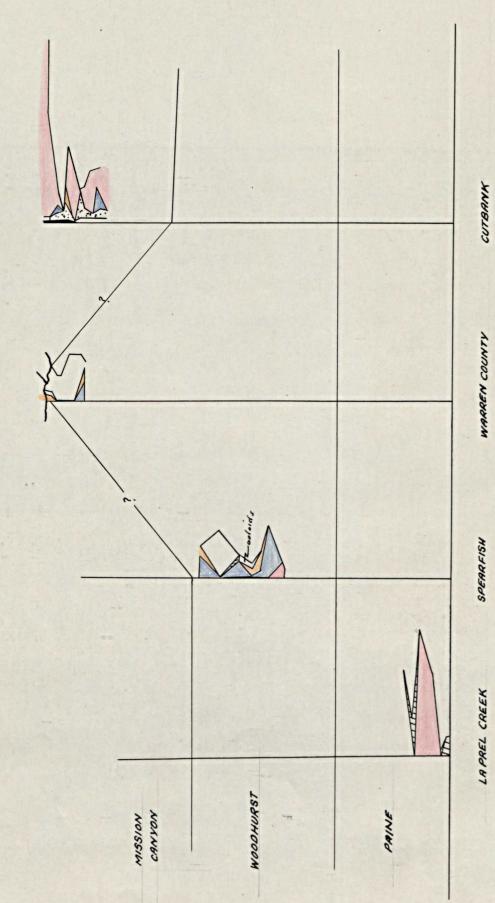
that the three divisions of the Madison, the Mission Canyon, Woodhurst, and Paine, could be recognized in the residues. This holds true over relatively widely separated localities. In general this is as fine a differentiation as can be made. Over any one small area, correlations could probably be made on very much smaller divisions. Work on other limestones has shown that this is usually possible, since minor variations in the residues will be consistent over this small area.

Mission Canyon These residues are minute (1 - 2%)
by volume of the original material. White chert in
thin flakes predominates; and sand grains and accessory minerals are relatively abundant. Fossil fragments in small amounts are commonly found.

Woodhurst The distinctive feature of these residues
is the large amount of silicified fossil fragments
that are found, and in certain zones coloids and
sand grains are plentiful.

Paine In those sections inspected, the residues were predominantly bulky lumps of argillaceous casts. The large amounts of this material obscured any other features. Other investigators report mottled tan and gray chert and silicified bryozoans as also being characteristic.





Graphic representation of the insoluble residues. F18. 6.

> LIMONITE

D010CAST

### Areas Sectioned

The accompanying chart of the results shows the type of residues found in the various sections.

South Boulder conforms with the general features of the residues.

The Paine here is very heavy in argillaceous material, and the top of the Mission Canyon also shows argillaceous residues.

Warren Quarry residues have a noteable abundance of fossil fragments, including good bryozoans.

Spearfish Canyon residues are also fossiliferous. Two samples showed excellent fine dolocasts.

Cutbank residues had an abundance of argillaceous material, with pyrite and bitumen.

La Prel Creek residues did not conform with the general Madison residues. Some were yellow argillaceous material and some were all limonite.

# Heavy Mineral Separation

Heavy mineral separations were made on the insoluble residues that had been obtained from the Madison section in the Little Belt Mountains. Only one section was examined by this method, and so there is no general statement that can be given as to correlation of the Madison on this basis. It was noted, though, that there are definite differences in the

type of heavy minerals that are found through the Madison section.

Differences That Were Noted in the Heavy Mineral Fraction.

Mission Limonite predominates; leucoxene is common, canyon but ilmenite is noteably rare. Barite is more abundant in this group than in any other.

Woodhurst The amount of heavy mineral is relatively
227-370' small, pyrite is practically absent and
limonite is not very abundant. White
colorless mineral grains are common and
there is a little rutile, titanite, and
fluorite.

Paine Pyrite predominates, with little or no

0-227' limonite. Ilmenite and leucoxene are both common. Among the minerals found are magnetite, rutile, titanite, and apatite.

The table of results that follows is included in order to show the occurrence of the heavy minerals and the basis for the statement above. The relative abundance of the different minerals is shown by dashes. Where Pred. is found this means that this mineral predominates.

# HEAVY MINERALS.

Others Black Opaque Minerals			Tourn.	7	Apatite Chromite	•				Tourm.			Apatite	1	Tourm.	Tourm.	1	
Barite	•	-	1	1	1			1	1		1	1				1	1	
Ilmenite																1		
Leucoxene	-	1		1							ı					1		ı
Pyrite								ı							1		1	
Limonite	-	-		-	-	1	Pred.		1	1	1	!	-		!	1	1	Pred.
Feet from base of Madison	868 - 873	852 - 868	813 - 823	778 - 798	738 - 763	713 - 723	693 - 703	673 - 683	653 - 663	633 - 643	613 - 623	583 - 593	557 - 563	536 - 552	525 - 536	513 - 525	507 - 513	487 - 492

Others Black Opaque Minerals			Tourm.	Tourm. gray		brown	fluorite		1	.Tourm.	fluorite Chromite ?	•		1	111111111111111111111111111111111111111	fluorite		fluorite -
Barite		ı				1	1		1	1		]						1
Ilmenite			•						1	1			1				1	
Leucoxene				1			ı		1			1			1	•		
Pyrite																	1	
Limonite	Pred.		1		1	1	1	-			1			•		1		
Feet from base of Madison	477 - 487	464 - 469	444 - 454	425 - 435	413 418	400 - 405	390 - 395	380 - 385	370 - 380	340 - 350	327 - 330	307 - 317	297 - 307	287 - 297	280 - 287	267 - 280	257 - 267	247 - 257

Others Black Opaque Minerals	Apatite	fluorite	1 0 0 1 0 0 0 0 0 0 0 0 0 0 0 0 0 0 0 0	apatite	rutile	apt. beryl -		rutile	rutile	magnetite	apatite	titanite	apatite mutile	Tourm. Magnetite				Tourm.
Barite		1	1		1		1									1		1
Ilmenite		1	1	1	•	1			1			1		1				
Leucoxene	-	:		1	1	1	1	1										
Pyrite		-	ı	-	1	1	-		-	1	1	1	1	-	Pred.	Pred.	Pred.	1
Limonite	1				•											1		
Feet from base of Madison	237 - 247	227 - 237	217 - 227	207 - 217	202 - 207	197 - 202	192 - 197	182 - 192	177 - 182	172 - 177	142 - 152	122 - 142	115 - 122	105 - 115	95 - 105	45 - 55	30 - 45	20 - 30

tion can be devided into those of secondary and those of primary origin. Those of primary origin were clastics that were deposited with the limestone; they were derived from the erosion of nearby land masses, and so reflect the character of these sources of erosion.

They represent only the most stable of minerals, since it takes a stable mineral to withstand the long period of weathering to which these mineral grains are subjected before deposition. Among their other characteristics the grains are small and angular.

From the results it can be seen that there was a change in type of minerals that were being deposited during Madison time. Thus the source of material was being changed.

The minerals of secondary origin are pyrite, limonite, barite, and leucoxene. The pyrite and barite are from secondary deposition, and the limonite and leucoxene are alteration products. The limonite is a result of the alteration of pyrite, note that the two seldom occur together and where they do the pyrite generally shows some alteration. If further work shows that the relative limonite and pyrite concentrations found in this paper are not local, then probably the pyrite alteration is a product of pre-Jurassic erosion. The leucoxene that occurs is an alteration

product of ilmenite. It was found to be usually weakly magnetic due to the presence of an ilmenite core.

Readers may wonder why no glauconite was found amongst the heavy minerals. The answer to this may be that glauconite has a tendency to decompose in hydrochloric acid, and so any glauconite that was present may have been lost during acid digestion.

### Conclusions

Before any positive facts can be said about correlation on the basis of the heavy mineral fraction of the residue a great deal more work must be done. The first requisite for correlation has been found, namely a vertical differentiation through the section. The next step will be to prove that there is a lateral consistency in these variations.

# Spectroscopy Results

As with the heavy minerals an insignificant amount of work was done on this subject. But evern if it only introduces the idea of limestone correlation by spectroscopy, it will justify its being included in this report.

Madison All Madison samples showed a marked similarity, the differences cannot really be seen on the print. By magnifying the negative against a white lighted background differences in the rarer elements can be seen, for example, copper, titanium, and vanadium.

Jefferson This is quite distinctive from the Madison, aside from the increase in magnesium that would be expected, since the Jefferson formation is a dolomite.

Amsden As with the Jefferson the spectrogram of these rocks was quite distinctive.

### Madison

Calcium -----very heavy

Aluminum -----strong

Magnesium ------quite strong

Silica ------strong

Strontium ------present

Barium -------present, rather stronger than would be expected.

Titanium --------present

Copper present -------present

Vanadium -----------present

Positively not present are the following:

Cadmium, cobalt, chromium, tin, nickel, zinc, antimony, mercury, and zircon.

The strength of the silica, barium, aluminum, copper, and titanium lines vary. This may be a means

of subdividing the formation into small divisions.

Note that there were other lines that were questionable, and so were not positively identified. Thus there is a possibility that other elements are present. With a limestone the calcium is so strong that even the weak lines show up, and are apt to be confusing.

# Conclusions

Before anything definite can be said concern-

ing limestone correlation by means of spectroscopy, a great deal of work must be done. The results that were obtained showed that the idea has promise. For example it was encouraging to note the consistency of the Madison spectrograms and the differences of the Jefferson and Amsden. This author hopes to be able to continue this investigation, for if something definite could be proved it would be of inestimable value to the petroleum industry.

## Bibliography

- Hamblin, Ralph H.;
  Stratigraphy and Insoluble Residues in the Upper Paleozoic Formations of Montana; Masters Thesis,
  Montana School of Mines, 1938
- Treland, H. A.:
  Use of Insoluble Residues for Correlation in Oklahoma; Bull., A.A.P.G., vol. 20, no. 8, pp. 1086 1121, 1936
- McQueen, H. S. Insoluble Residue as a Guide in Stratigraphic Studies; Missouri Bur. Geol. and Mines, Bien. Rept. 56, Append. 1, 1931
- Milner, H. B. Sedimentary Petrography
- Peale, A. C.
  The Paleozoic Section in the Vicinity of Three
  Forks, Montana; Bull. U.S.G.S. 110, 1893
- Perry, Eugene S. Natural Gas in Montana; Montana School of Mines Bull., Memoir No. 3, Dec., 1937
- Sloss, L. L. and Hamblin, Ralph H.
  Stratigraphy and Insoluble Residues of Madison
  Group (Mississippian) of Montana; A.A.P.G.
  Bull., vol. 26, no. 3, March, 1942

Fig. 1. Crinoid plates.

Fig. 2. Echinoid spines.

Fig. 3. Bryozoans.

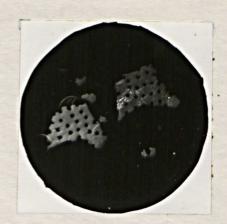
Fig. 4. Bryozoans and brachiopod spines.



Pig. 1.



Fig. 2.



F1g. 3.



Fig. 4.

- Fig. 1. Typical fossil material from Warren Quarry.

  Note the brachiopod fragment, crinoid plates,

  dolocasts, spine, coloids and other material.
- Fig. 2. Fossil material from Spearfish Canyon.

Fig. 3. Dolocasts, note the very fine white one in about the center of the field.



Fig. 1.



Fig. 2.



Fig. 3.

- Fig. 1. Coloids from Spearfish Canyon.
- Fig. 2. Ooloids and rounded sand grains (clear) form
  Spearfish Canyon.
- Fig. 3. Arigillaceous casts from the Lodgepole at South
  Boulder Canyon.
- Fig. 4. Limonite casts from La Prel Creek.





F1g. 2





Fig. 4

a & D Spectrograms; -

a Jefferson sample. b are Madison samples
is the iron spectrum
is an Amsden sample
is a Jefferson sample 000

From these results the similarity of the Madison samples can be seen while the differences between the Madison, Jefferson and Amsden are

quite apperent.