

A METHOD OF DIFFERENTIATING CARBONATE AND  
SILICATE FACIES OF THE NEGAUNEE IRON FORMATION  
BY SPECTROCHEMICAL ANALYSIS

Thesis for the Degree of M. S.

MICHIGAN STATE UNIVERSITY

THOMAS D. WAGGONER

1967

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## ABSTRACT

### A METHOD OF DIFFERENTIATING CARBONATE AND SILICATE FACIES OF THE NEGAUNEE IRON FORMATION BY SPECTROCHEMICAL ANALYSIS

by Thomas D. Waggoner

A spectrochemical method of differentiating a predominantly magnetite-chert-carbonate facies from a magnetite-chert-silicate facies has been developed and successfully applied to the primary magnetite-chert deposits of the Negaunee iron-formation.

Interpretation of the spectrochemical data depends on a point distribution representing the elements: magnesium, manganese, aluminum and iron. Manganese ions substitute readily for cations in the complex iron carbonates and only to a very minor extent in the iron silicates; the amount of manganese present can be used to approximate the quantity of carbonate present. A plot of manganese values versus magnesium values shows a trend of both elements which indicate higher concentrations of carbonate and possibly silicate. To evaluate the amount of silicate present when both Mg and Mn values are high, a plot of magnesium values versus aluminum is considered.

Aluminum occurs exclusively in the iron silicates which are products of a similar chemical environment to those of carbonates and magnetite. The comparative analysis becomes vague when detrital feldspar contributes to the aluminum values.

A plot of magnesium against iron may indicate the abundance of magnetite, but caution must be used in considering the type plot shown due to the extensive iron values present in all the minerals except chert.

Silica cannot be used to correlate facies types due to its erratic occurrence which is governed by stability fields not sufficiently clear at the time of the present study.

Phosphorous has little value in making an accurate correlation due to its dependence on a pH condition and not on the other elements or their stability fields.

Spectrochemical technique can be utilized in differentiating soft carbonate ores from the harder silicate types in iron ore blending and beneficiation.

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FACIES OF THE NEGAUNEE IRON FORMATION  
BY SPECTROCHEMICAL ANALYSIS**

by

**Thomas D. Waggoner**

**A THESIS**

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Michigan State University  
in partial fulfillment of the requirements  
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**1967**

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1. The first part of the text discusses the importance of maintaining accurate records of all transactions.

2. This is followed by a detailed explanation of the accounting cycle.

3. The text then describes the various methods used to record transactions, including journalizing and posting.

4. It also covers the process of balancing the books and preparing financial statements.

5. The final part of the text discusses the importance of internal controls and the role of the auditor.

6. The text concludes with a summary of the key points discussed throughout the chapter.

7. The text also includes several examples and exercises to help illustrate the concepts discussed.

8. The text is written in a clear and concise style, making it easy to understand and follow.

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## INTRODUCTION

Magnetic ores of the Marquette Range can be divided into two general types: magnetite-chert-carbonate and magnetite-chert silicate. Nomenclature may be misleading when discussing carbonate or silicate facies and in interpreting the spectrochemical data. Both the carbonate and silicate minerals are minor constituents of the predominantly magnetite-chert rock and only the quantity relationship of one to the other defines the facies type. The name 'silicate' ore is based on the relative abundance of iron silicate minerals, but in actuality the silicate facies usually contain almost twice as much carbonate as does the defined carbonate facies. Thus the carbonate facies contains little or no iron silicate minerals while the silicate facies contains from one to five percent iron silicate minerals but may contain abundant carbonate.

In a discussion of element distribution in the iron-formation, the mineralogical composition and the general environment under which the mineral assemblage was deposited are considered. Since the mineralogical composition dictates a definite element distribution, an attempt will be made through the use of spectrochemical analysis to differentiate between the two major ore types. The advantage to the proposed method is one of speed and reproducibility. This can be achieved once the procedure is standardized.

## GEOLOGY

The Negaunee iron-formation is part of a sequence of Animikan (Huronian) meta-sediments which form a western pitching synclorium. The iron-formation is middle Animikan in age and is underlain by the Siamo slates and graywacke. Further discussion of the complete stratigraphy and lithologies are covered in work done by Van Hise (1897), Twenhofel (1952) and Hase (1957).

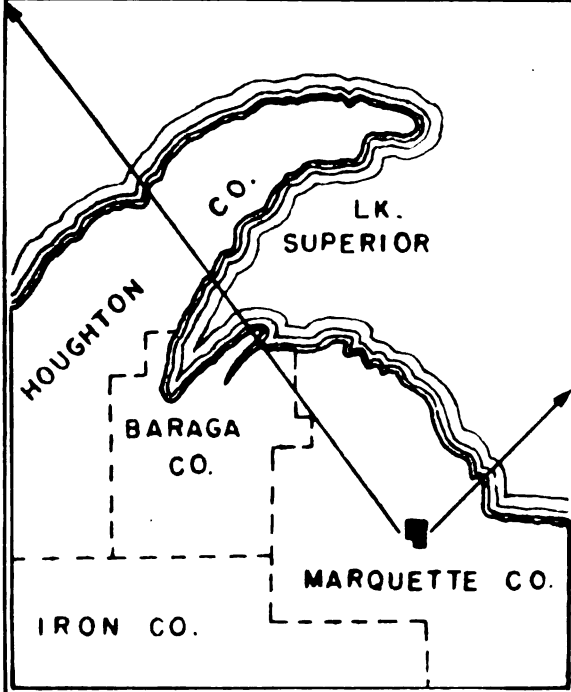
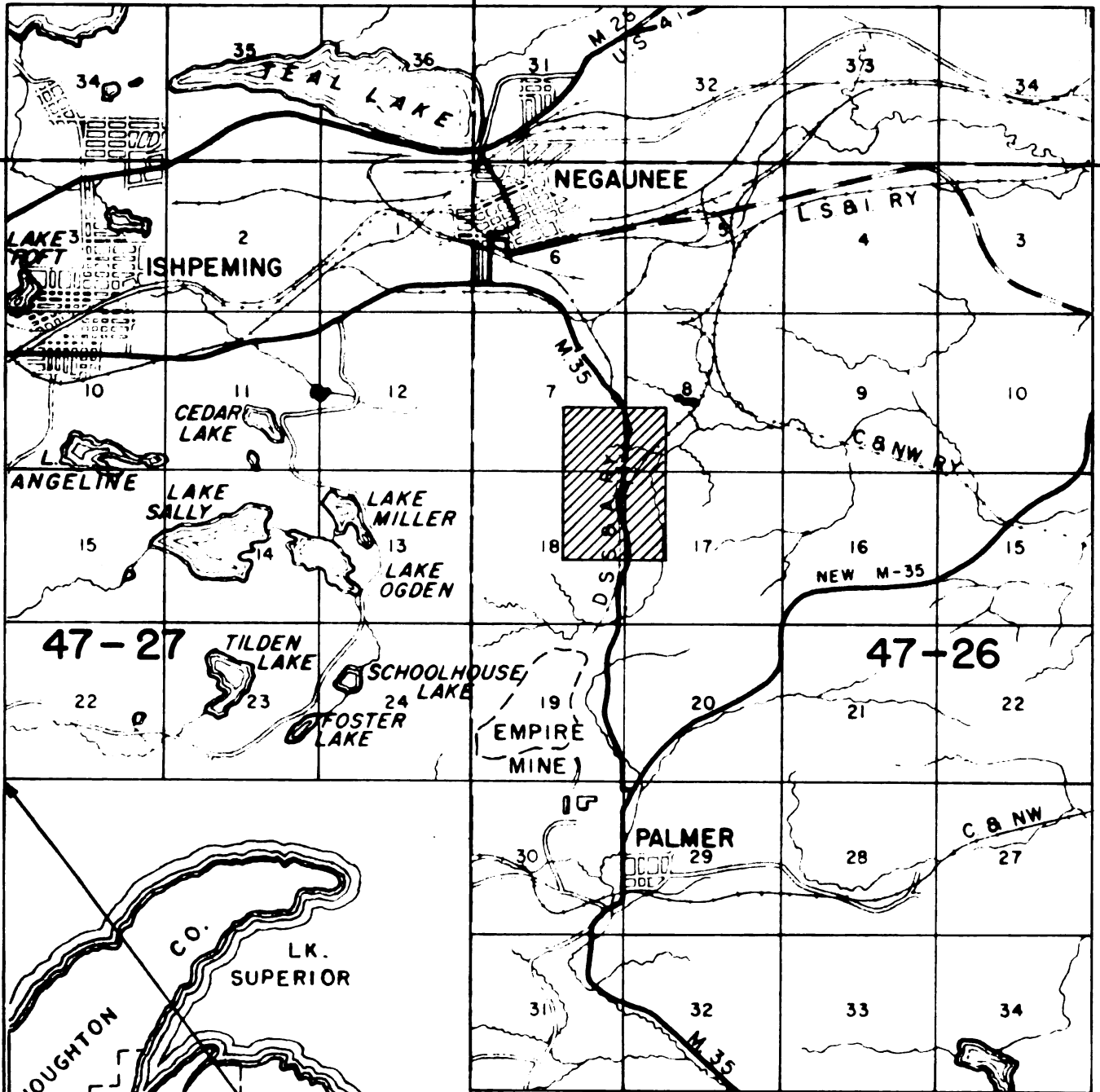
The iron formation has a gross composition of chert, magnetite, carbonate and silicate and can be divided into two distinct zones (Adler, 1935). The upper secondarily oxidized portion reflects mainly a chert-hematite-magnetite sedimentary facies. The lower portion, where unoxidized, reflects mainly a chert-magnetite-carbonate-silicate sedimentary facies which exhibits large individual quantity variations for any random portion.

Abundant magnetite-carbonate-silicate facies occur in the eastern end of the Marquette geosyncline in the lower portion. Secondary oxidation is confined to fracture patterns adjacent to dikes.

The geosyncline has undergone various stages of metamorphism. The western portion of the geosyncline has been highly metamorphosed (staurolite facies) perhaps causing ion migration during the formation of specularite, grunerite and stilpnomelane. The eastern portion shows the lowest metamorphic rank (greenschist facies--quartz-albite-muscovite-chlorite subfacies). It is logical to assume that the least ion migration has occurred in the eastern portion and that the primary element concentration has not been notably disturbed.

R. 27 W. R. 26 W.

T. 47 N. T. 46 N.



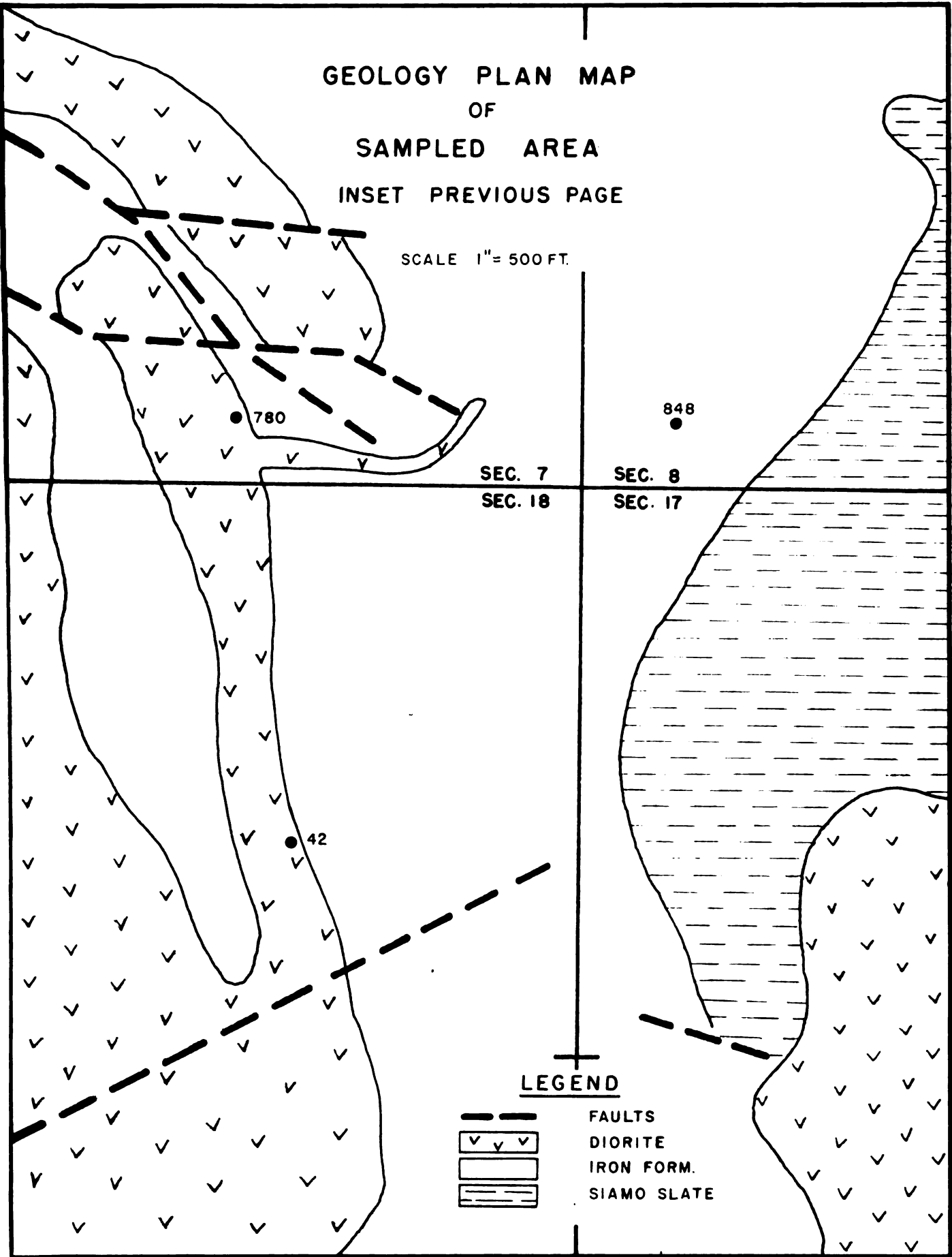
**LOCATION PLAN MAP  
OF  
SAMPLED AREA  
MARQUETTE COUNTY, MICHIGAN**

SCALE 1" = 1 MILE





Selection of the drill locations for core sampling was based on three factors: the presence of carbonate-silicate facies, the extent of alteration-oxidation and the degree of metamorphism. Samples of core were taken from three drill holes which penetrated the lower portion of the Negaunee iron-formation. The drill holes are located in section 7, 8, and 18, T. 47 N, R. 26 W, Marquette County, Michigan (page 5). The drill angle, depth, lithologies and corresponding specimen number are given in appendix A.

GEOLOGY PLAN MAP  
OF  
SAMPLED AREA  
INSET PREVIOUS PAGE

SCALE 1" = 500 FT.



LEGEND

-  FAULTS
-  DIORITE
-  IRON FORM.
-  SIAMO SLATE

## MINERALOGY

The portion of the Negaunee iron-formation studied contains the following minerals in the decreasing order of their abundance: chert, magnetite, iron carbonate, minnesotaite, hematite, stilpnomelane, chlorite, biotite, clastic quartz and feldspar, apatite, glaucophane and tourmaline. Almost 98 per cent of the formation consists of chert, magnetite, hematite, carbonate and iron silicates in various proportions. Detrital zones are prevalent near the top of the unoxidized lower portion of the formation. Quartz, feldspar and biotite are usually restricted to these sediments and are not important in the consideration of the facies studied. Minor graphite and pyrite occur in certain contacts between the clastic and chemical facies. Their occurrence is minor and need not be considered here.

Chert ( $\text{SiO}_2$ ) In the present study the term chert refers to a fine grained chemically precipitated form of quartz. It is associated with magnetite, iron carbonate and iron silicates in various proportions. In thin section (Plate I) it appears as white, yellow or bluish-white grains; the abnormal color is due to the thick sections.

Magnetite ( $\text{Fe}_3\text{O}_4$ ) The mineral form is commonly euhedral (octahedron) and the size varies from a few microns to a few millimeters (Plate III). Identification is made from its crystalline shape and opacity. Generally, the magnetite is associated with chert and to a minor extent occurs in the chert-carbonate and/or chert-silicate laminae. Most of the magnetite is believed to be recrystallized which is indicated by widespread replacement



## 1. Introduction

The first part of the document discusses the importance of maintaining accurate records.

It is essential to ensure that all data is properly documented and stored.

This section outlines the various methods used to collect and analyze the data.

The results of the study are presented in the following sections.

The data shows a clear trend in the behavior of the system.

These findings are consistent with previous research in the field.

Conclusion

The study has shown that the proposed method is effective in solving the problem.

Further research is needed to explore the limitations of the current approach.

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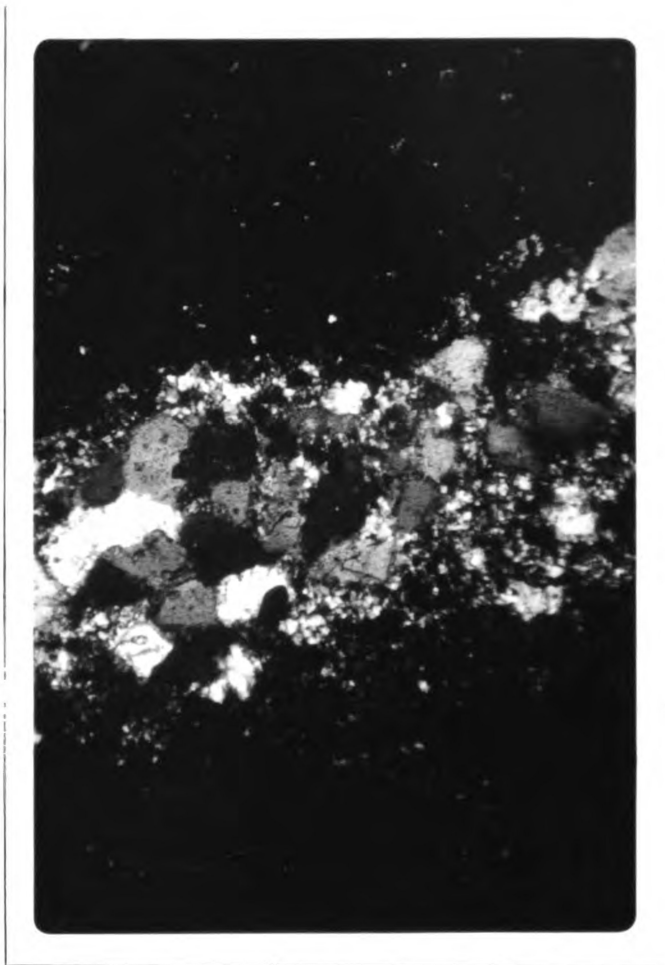
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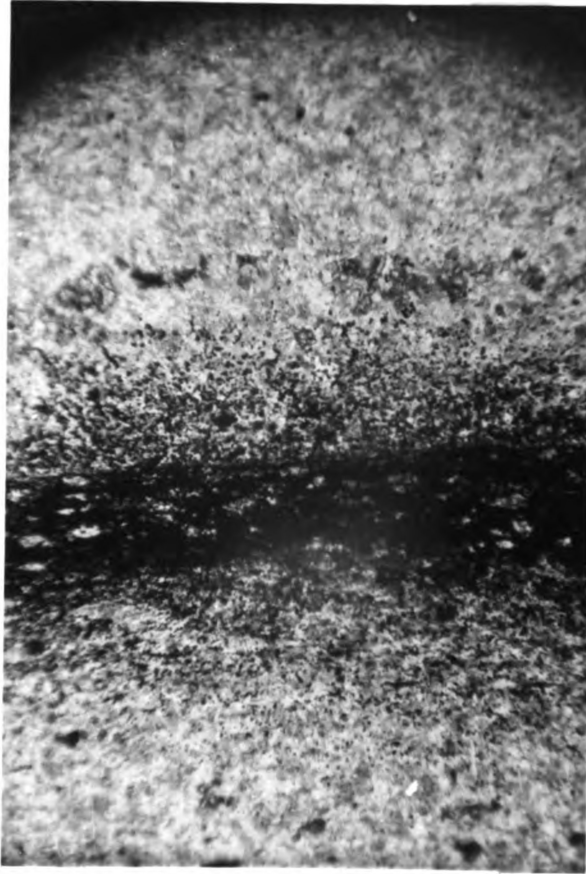
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**PLATE I**

Sample B-1a, cross nicols. Secondary quartz band showing the outlines of chert and primary clastic quartz (white and blue). The green material is chlorite. The black material consists of magnetite and hematite. Three second exposure with Ectachrome X.

All pictures were taken with an Exacta Single Reflex camera specifically adapted to a Leitz-Wetzlar Polarizing Microscope. All shots were made at 200x magnification. Photographs were taken with Kodak products.



**PLATE II**

**Sample A-4, plane polarized light.  
Gradation of magnetite facies into chert-  
carbonate-silicate facies. Two second ex-  
posure with Panatomic X.**

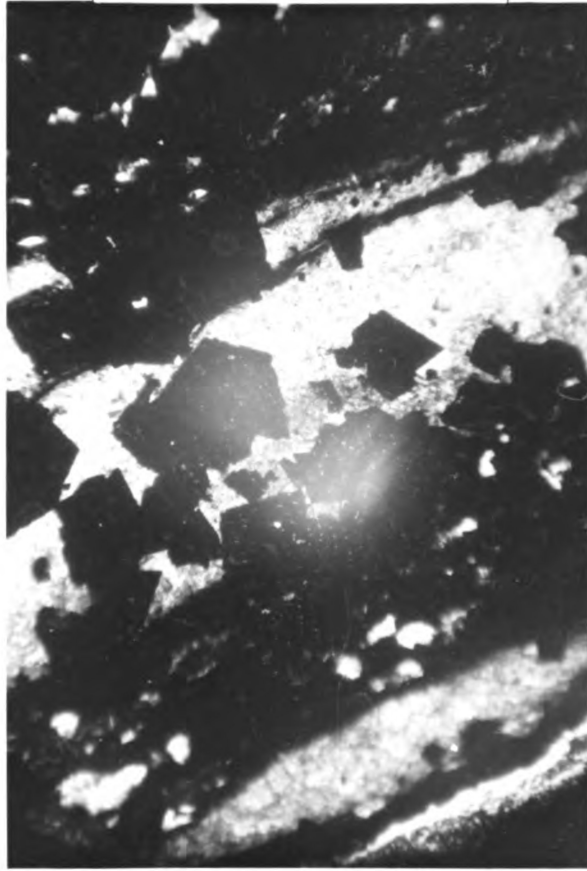
of chert by magnetite. The quantity of magnetite varies between 20 and 40 per cent by weight in any random sample.

Hematite ( $\text{Fe}_2\text{O}_3$ ) Occasional hematite was observed along bedding planes and fractures. In shallow portions of hole B, hematite occurs as martite, a pseudomorph after magnetite. In areas of secondary oxidation fine flakes and red stains indicate ground water activity. In these areas the carbonate and silicates have been oxidized and elements have been transported in and out. Under such conditions the facies analysis is not representative of primary conditions, and core samples containing traces of oxidation were not used.

Iron Carbonate ( $\text{FeMgMnCa}$ ) $\text{CO}_3$  Identification was based on optical properties and in some cases the obvious rhombohedral form. The color varies from clear to light green to slightly brownish. The mineral is usually observed in distinct laminae or associated with chert and minor silicates. The actual chemical composition is variable ( $\text{Fe}_{.64-.76}$   $\text{Mg}_{.25-.20}$   $\text{Mn}_{.10-.03}$   $\text{Ca}_{.01}$ ) $\text{CO}_3$  (Clarke); other variations are always possible. Recent work on carbonate iron-formations indicate distinct zones of ankerite and siderite with minor cation variation within a given carbonate (Trendal, 1966).

Stilpnomelane  $(\text{OH})_4(\text{KNaCa})_{0-1}(\text{FeMgAl})_{7-8}(\text{Si}_{18}\text{O}_{23-24}) \cdot 4\text{H}_2\text{O}$  The mineral was only tentatively identified due to the fact that it closely resembles biotite. Very few occurrences were noted in the area studied.

Minnesotaite  $(\text{OH})_{22}(\text{FeMg})_{22}(\text{Si}_3\text{OAlFe})_2\text{O}_{74}$  The predominate iron silicate is minnesotaite. In thin section it is not pleochroic but does show a distinct green color. Nearly parallel extinction and strong birefringence help distinguish it from chlorite which occurs in the clastic zones. Structurally it appears as radiating needles intergrown with the chert and carbonate (Plates V and VI).



**PLATE III**

**Sample A-21, plane polarized light.  
Secondary magnetite octahedra in a ground  
mass of chert-carbonate-silicate. Two sec-  
ond exposure with Panatomic X.**

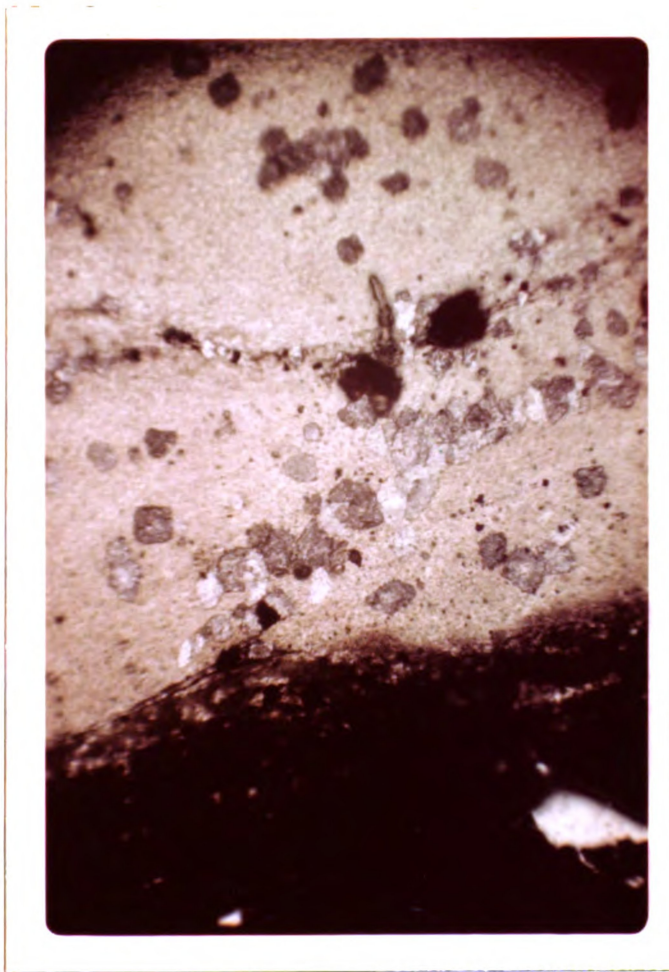


PLATE IV

Sample B-10, plane polarized light. Secondary  $(\text{FeMgMnCa})\text{CO}_3$  rhombohedra in a chert-carbonate-silicate band. Note only minor magnetite is found in the band but that it predominates both stratigraphically above and below.

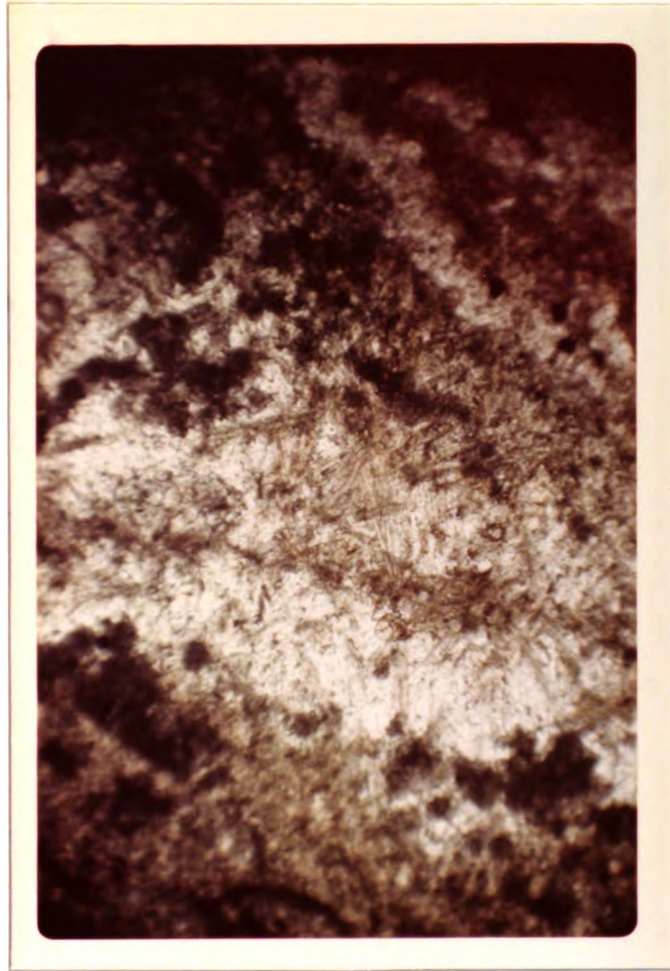


PLATE V

Sample A-19, plane polarized light. Minnesotaite has a slightly green color, which is intensified by the green color imparted to the photograph by the blue light filter used on the microscope. Radiating needles and negative sign help distinguish the mineral from similar iron silicates. Two second exposure with Ectachrome X.

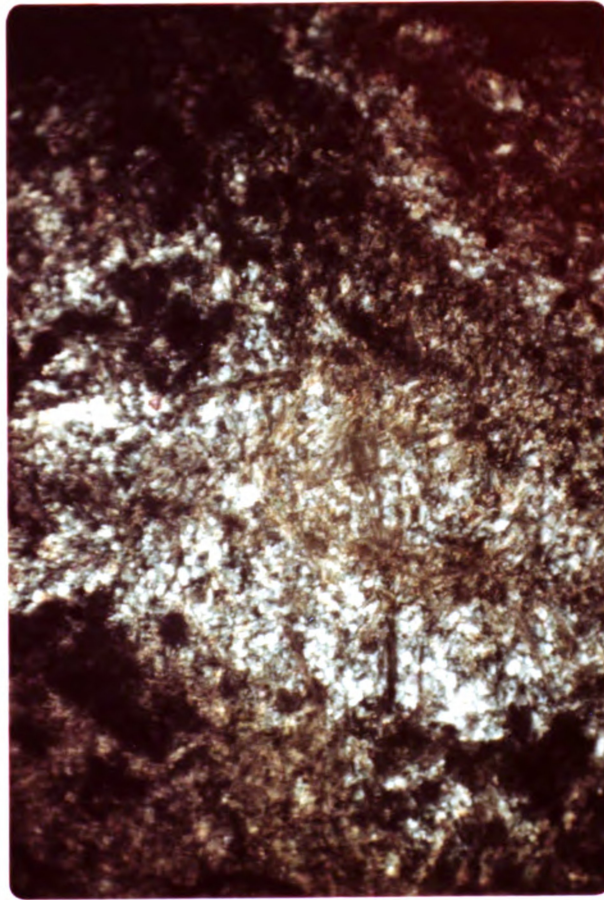


PLATE VI

Sample A-19, cross nicols. The blue, white and yellow grains are chert while the dark portions are a combination of chert-silicate-carbonate which have undergone surface oxidation. Four second exposure with Ectachrome X.



## ENVIRONMENTAL CONSIDERATIONS

To understand and interpret the element distribution, a discussion of the geochemistry and the general environment is presented.

Phase equilibrium conditions for  $\text{FeCO}_3$ ,  $\text{MgCO}_3$ ,  $\text{MnCO}_3$  and  $\text{Fe}_3\text{O}_4$  are discussed in works by Huber (1953, 1958) and Garrels (1952, 1960). No quantitative energy values are available for the iron silicates, but due to their natural association with carbonates, it is assumed that they are governed by similar types of thermodynamic considerations.

Under the same stability field of deposition, magnetite will form in preference to iron carbonates as long as sufficient oxygen is available. The explanation involves the lower and more stable energy state achieved by magnetite with the use of less energy (heat) under any given Eh condition.

Although hematitic portions of the formation have been avoided in the analysis procedure, it is helpful to discuss oxidation as it relates to the rate of alteration of the minerals in the carbonate-silicate facies. Entropy ( $\Delta s = \text{cal/deg. mole}$ ) can be defined as the energy state of a system which is determined by the division of the absorbed energy by the absolute temperature of formation.

$$\Delta s = \frac{dE}{T}$$

The following are entropy values at 25°C.:

$\text{FeCO}_3$	. . . .	22.2
$\text{MgCO}_3$	. . . .	15.1
$\text{MnCO}_3$	. . . .	20.5
complex iron silicate		< 35

Oxygenated circulating ground water oxidizes carbonates and silicates to hematite at a faster rate than magnetite is altered to martite. The entropy levels of the silicates and carbonates are lower so that energy input at any given temperature will almost always affect the carbonates and silicates first.

Silica precipitation is also covered by thermodynamic conditions, but they have no direct correlative value in the present study. Work done on the equilibrium conditions has given conflicting and inconclusive results. Krauskoff (1956) maintains that pH has absolutely no effect on silica precipitation in true solution from a pH of 0 to 9. However, Moore and Maynard (1929) indicate silica, carried as a colloid, is precipitated when transporting acidic solutions are neutralized in the presence of a carbonate. Krauskoff mentioned a precipitate of uncertain composition formed when a normal silica solution approached a pH of 7 from an acid solution.

Huber and Garrels (1953) have shown silica is transported currently by fresh water streams with pH values of 5 to 7 and an Eh of .25 to .30. The water has a high  $\text{CO}_2$  content which would generally increase for colder streams.

The chert and silicate all contribute to the intensity of the silica spectrum line. The results show that silica is of little value in facies differentiation.

The quantitative values for the carbonate cations have been previously stated and from them it is noted that iron and magnesium predominate. The quantity of iron present suggests that the principle carbonate is siderite ( $\text{FeCO}_3$ ) with minor magnesite ( $\text{MgCO}_3$ ) or an isomorphous series of the two near the iron rich end ( $\text{Fe}_{.70}\text{Mg}_{.30}\text{CO}_3$ ). Manganese, by

virtue of its similar ionic radius, may possibly replace either iron or magnesium in the carbonate series.

The chemical behavior of the dolomite group has been extensively studied by many authors so that only a brief pertinent consideration is necessary here. Only very minor quantities occur in the carbonate-silicate facies in the mineral form of ankerite  $\text{Ca}(\text{Mg}, \text{FeMn})(\text{CO}_3)_2$ . Calcium was concentrated in dolomite  $\text{CaMg}(\text{CO}_3)_2$  as a replacement halo around chert which was then replaced by magnetite. Dolomite replacement is not a major feature and was not present in any of the tested samples.

One important chemical criteria is indicated by the lack of calcium carbonate and that is the pH conditions were less than 7.8. The assumption here is that there is sufficient calcium but that conditions were not sufficiently alkaline to make  $\text{CaMg}(\text{CO}_3)_2$  stable. The remainder of the calcium is found in the sporadic spatite grains.

Iron will precipitate in both an oxidizing or reducing environment under alkali conditions, but only the ferrous compounds, carbonate and magnetite, will form under a reducing environment. The carbonate has a stability field which overlaps magnetite up to a possible pH of 8.5 (James, 1954). The carbonate will begin precipitation at a pH of 5.1 under a rapidly neutralizing condition and a highly dissolved  $\text{CO}_2$  content. At the stability field of 5.1 or higher a negative oxidation potential is essential.

One factor which controls the amount of Mn present in the carbonate facies is the chemical equilibrium conditions. While remaining in solution under oxidizing conditions, manganese begins precipitation at a pH of 7 (Krausakoff, 1951) and increases as pH increases. Manganese will precipitate at a faster rate as the pH rises but will always follow the

ferrous compounds. Manganese carbonates are much more soluble than the iron carbonates; thus more susceptible to resolubility. Temperature has no effect on the Fe-Mn interrelationship within the ranges expected in natural environments.

The facies are not in complete chemical equilibrium as noted by the associated mineral assemblages. During precipitation or shortly after the diagenetic process had begun, limited ion migration and substitution definitely occurred. A second factor now must be explained to account for the extent of manganese substitution.

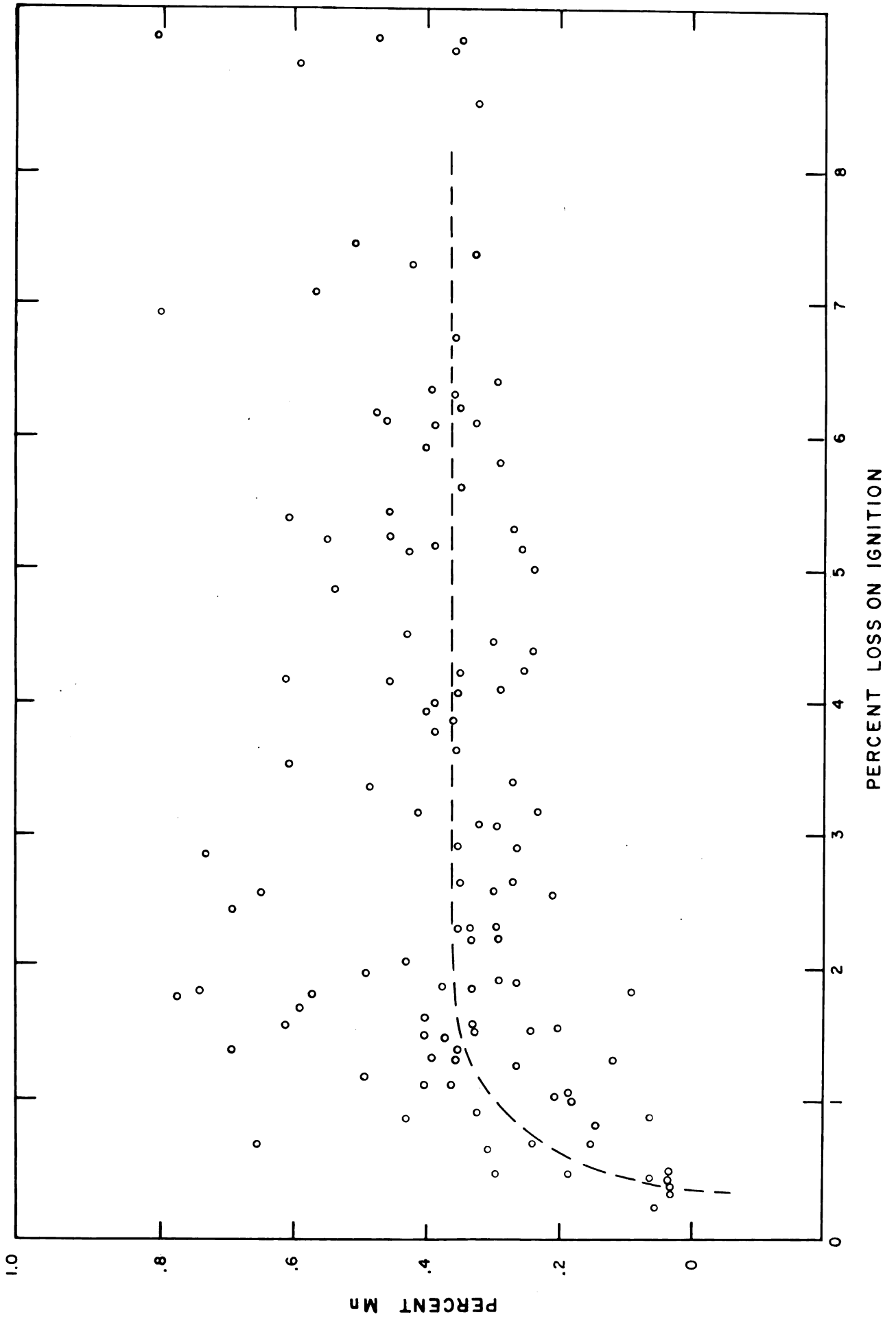
The quantity of Mn cations present and the degree of precipitation to be expected under the environmental conditions have to be considered. Seventy samples similar to the facies studied were tested for manganese by wet chemical methods and loss on ignition (L.O.I.) tests. The L.O.I. test is explained in Appendix C.

A plot of the per cent manganese versus L.O.I. (Figure I) gives a point distribution approximated by a horizontal line which becomes tangent to the abscissa for low manganese and low L.O.I. values.

The inference drawn from the distribution is that the quantity of manganese in the facies is a function of manganese ion availability during deposition and not necessarily dependent on the pH condition which controls the  $\text{CO}_3$  radical. It can be stated that ion substitution is a function of manganese availability and to a lesser degree the amount of ferrous iron which will form stable carbonates.

Magnesium carbonate precipitation is only dependent on the  $\text{CO}_3$  radical. Its ionic size allows free substitution for iron in the isomorphous carbonate series. The amount of magnesium substitution is probably related to the ion availability under any given physical equilibrium

FIGURE I



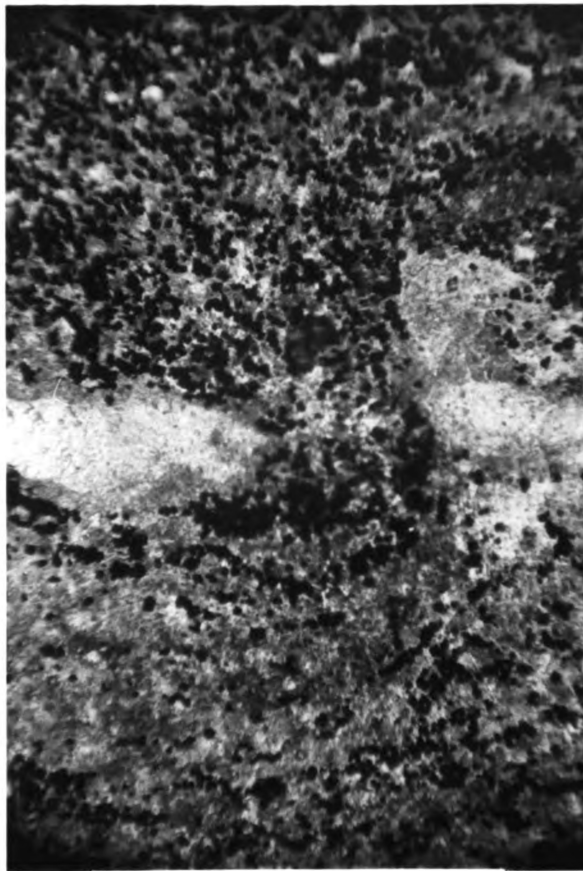
condition. Magnesium also substitutes on a limited scale for  $Fe^{++}$  in the complex silicates.

Phosphorous occurs in the form of apatite. Due to the lack of associated clastics most of the phosphorous can be considered a product of precipitation. If precipitated, it would be in the form of tricalcium phosphate  $Ca_3(PO_4)_2$ . Oxidation potentials have no effect on either calcium or the phosphate radical. However, the phosphate radical is very dependent on pH conditions. Ideally, a pH of 7 to 7.5 is conducive to precipitation (Krumbein, 1952). The quantity precipitated is directly related to the amount of  $(PO_4)$  in solution and represents a normal level to be expected in either saline or fresh water.

Aluminum is present in the complex silicates, possibly as a chemical precipitate, but the equilibrium conditions and stability fields are obscure. Its presence can be utilized to distinguish the silicates, and its excesses can show the presence of fine detrital clays.

A chemical environment can be postulated from the brief stability and equilibrium data previously presented. A pH range from 5.1 to a maximum of 7.8 is possible from stability field criteria and would reflect a reducing environment with a possible Eh range between 0 and -.2.

From the sedimentary sequence both above and below the iron-formation, it has been suggested that the area was part of a geosyncline involved in the various stages of tectonic events (Hase, 1957, Van Hise, 1897). A closed or restricted basin would allow inflowing acid streams with low clastic loads to enter the basin forming brackish waters. The idea of a fresh water deposit (Hough, 1958) is possible, but the sequence of sedimentation containing algal structures in dolomite suggest at least partial brackishness.



#### PLATE VII

Sample C-11, plane polarized light. A fine crystalline carbonate and chert band (white) ruptured allowing the magnetite-chert-carbonate above to flow downward. A disturbance, possibly current or wave action, caused movement in the partially solidified facies resulting in the observed structure. Although the formation does exhibit a low metamorphic rank, the structure is believed to be primary and not the result of plastic flow. There is no continuation of the phenomena stratigraphically above or below the band. Two and one-half second exposure with Panatomic X.

The almost structureless character of the formation indicates a deposition zone below wave base. Certain phenomena (Plate VII) indicate that currents or occasional disturbances did disrupt the rhythmic layering. The depth consideration plus the ideal  $MnCO_3$  and  $SiO_2$  precipitation range suggests a more alkaline environment around a pH of 6.9 to 7.4. The depth does not necessarily reflect a distance from the ion source as there are distinct coarse clastic zones which possibly indicate a nearness to source.

Although the environmental considerations are brief, they are necessary for a fuller understanding of the spectrochemical results. The problems of biological influence, state of ion transportation and source of ions have been avoided due to the inherent difficulties of each subject and the remote influence they have on the direct interpretation of the results.



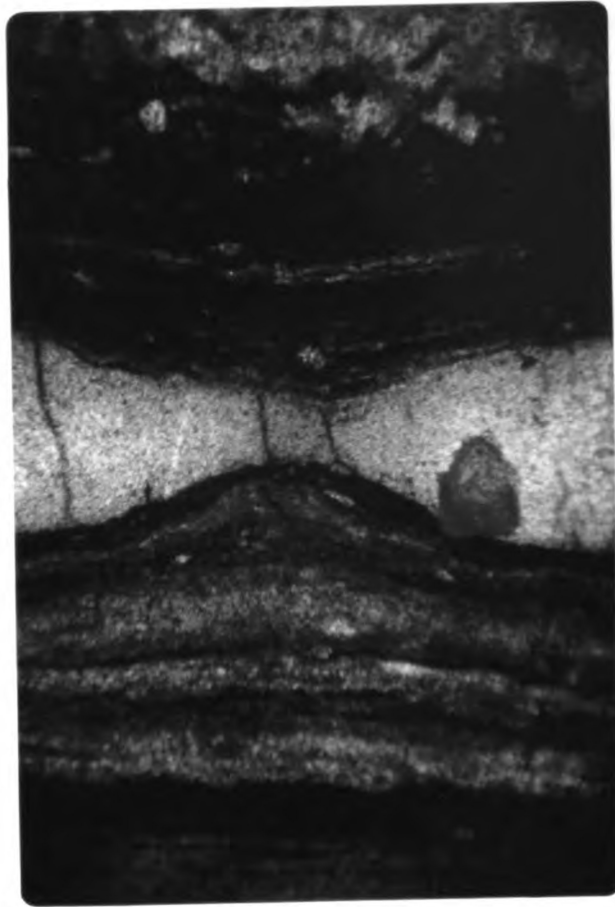


PLATE VIII

Sample B-16, plane polarized light. Bleb chert caused by the cohesion force in the colloidal silica which pulled a lens of the material into a bubble. The underlying material was depressed by the weight while the overlying laminae were draped over the bleb and thinned on the top. Three second exposure with Ectachrome X.



PLATE IX

Sample B-16, plane polarized light. The chert in this photograph shows total separation (bleb) which resulted in depression under the thickened portion. This is probably a colloidal phenomena and does not result from a physical force after diagenesis. Two second exposure with Ectachrome X.

## PROCEDURE

Each core sample was broken by a hammer blow. The chips from the inner portion of the core were chosen because the outer surface was considered contaminated by the drill bit and hammer blows. The chips were ground in an agate mortar and pestle to approximately -500 mesh. The equipment was washed with alcohol between each sample to prevent contamination.

From each ground sample a .2 gram quantity was weighed out on a piece of cigarette paper and added to .2 grams of pure carbon and .02 grams of internal standard ( $\text{SrCO}_3$ ). The graphite was added to stabilize the arcing (homogeneity) by providing better conductivity in each sample.

The internal standard used in spectrochemical analysis has four basic purposes. It is used to correct the emission errors originating from the source; to compensate for poor photographic techniques; to correct for inconsistent time settings due to mechanical malfunctions and to compensate for weight errors in the amount of each sample burned.

The most important factor considered in the choice of an internal standard is the quantity of that particular element in the unknown sample and the variability of this quantity from one sample to another. For these reasons strontium carbonate was chosen for the internal standard.

Strontium may substitute for calcium in carbonates. In work carried out by Vinogradov (1956) it was found that while Pre-Cambrian Ca/Sr ratios averaged fifteen times higher than Paleozoic ratios (6500 versus 350), relatively pure Pre-Cambrian carbonates had less than 40 ppm strontium

present. In the carbonate-silicate rocks studied, there is very little calcium present (<100 ppm) so that the strontium content would be so low that variations would not appreciably change the strontium line intensities from the internal standard. Two samples were run for strontium but showed only traces of the element.

A second criteria considered in the choice of  $\text{SrCO}_3$  as an internal standard was its ionization potential (2.3EV) which is slightly lower than those of the elements sought.

A third criteria was that  $\text{SrCO}_3$  has a molecular structure which is not unlike the majority of the compounds in the samples. The mean identity of molecular configurations reduces the complex molecular excitation response which often interferes with element spectra lines.

A Baush and Lomb grating spectrograph (15,000 lines to the inch) was used in the experimental procedure. A low voltage DC continuous arc was sufficient to produce a spectrum. The instrument settings used were:

250 volts

0 ohms (above that of the normal system)

360 microhenries inductance

30 microfarads capacitance

2400 Å - 3400 Å wave length span

A one and one-half second pre-exposure burn was followed by a three second exposure for each sample. The pre-exposure burn was used to obtain a uniform heat; thus cutting down the low spectra emission caused by slow ignition starts. Secondly, maximum conductivity which is essential to achieve maximum excitation is not always reached at the beginning of each arc. Finally, poor surface distribution of the elements due to the settling at the top of the electrode chamber is eliminated in a pre-arc burn.

Being unable to make a direct quantitative approach, an alternate analysis procedure was initiated with success. The premise that the photographic spectral line density is directly proportional to the quantity of the element in the sample, allows a direct quantity relationship between different elements or the same element in different samples when four variables are adjusted. These variables mentioned earlier may be compensated for with reference to the internal standard.

A densitometer was used to examine the spectra and record the density of a chosen spectral line for each of the elements sought. The recorded graph of each line density was measured for amplitude from a constant lower division on the graph paper and width at that lower division point.

By taking the total rectangular area under the curve, a close correlative value is obtained for intensity of the spectra emission. Internal line intensities are then equated for each spectrum, and element line intensities are adjusted up or down by a factor.

The procedure which was used to develop the photographic plates was kept as uniform as conditions permitted in order to allow accurate correlation between plates. Each exposed plate was agitated at one minute intervals in a solution of Kodak Dextal (700 grams per one liter of water) for eight minutes. From the developer the plate was taken and placed in a stop solution composed of three per cent hydrochloric acid and water for thirty seconds. The plate was then placed in a hypo (fix) solution ( $\text{Na}_2\text{S}_2\text{O}_3$ ) for five minutes. Finally, the plate was allowed to dry.

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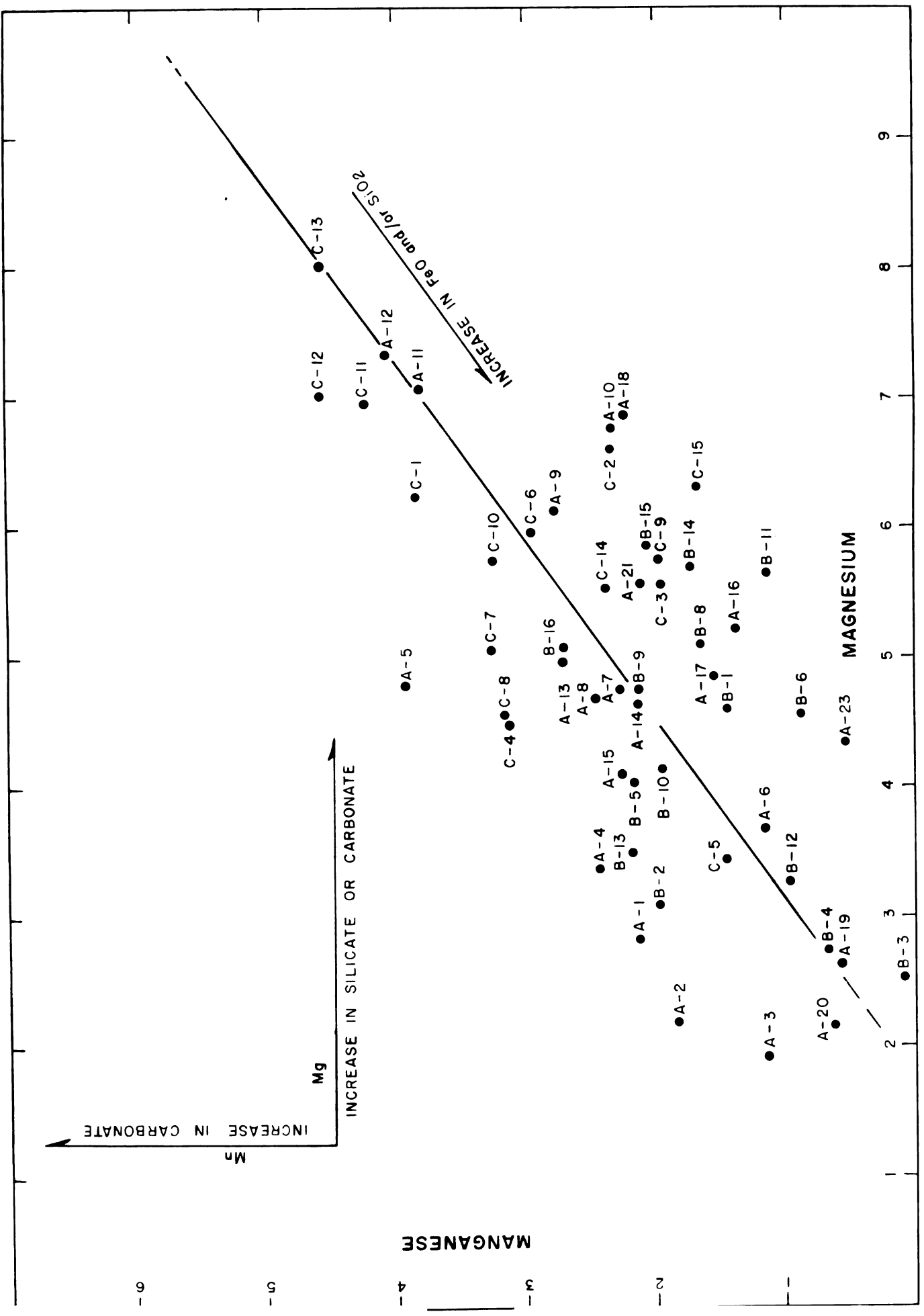
## INTERPRETATION

Manganese variations are considered a reflection of the relative amount of complex carbonate present based on substitution principles previously described. When the manganese values increase, the magnesium would also be expected to increase. The trend is shown by the inclined line which represents the point average as established by the least square method (Figure II). The significance of the point deviation above or below the line must be evaluated by using a second graph showing the relation of aluminum to magnesium. If the aluminum present is only a component of the iron silicates, the point will reflect the relative amount of silicate present. The apparent trend of Mg/Al values in Figure III would be expected with an increase in silicates containing both elements. When both point relations in Figures II and III are above the average, it would indicate a high per cent of carbonate and silicate minerals present.

High manganese values show carbonate to be present in concentrations between 10-20 per cent and low values show concentrations of less than 10 per cent. Exact per cents cannot be applied due to the lack of standardization and the variable substitution of manganese in the carbonates.

Silicate values range from zero to a maximum of five per cent. A zone (Figure III) was chosen to indicate the transition from a predominantly carbonate facies to one containing high proportions of silicate minerals along with various concentrations of carbonate. The

FIGURE II



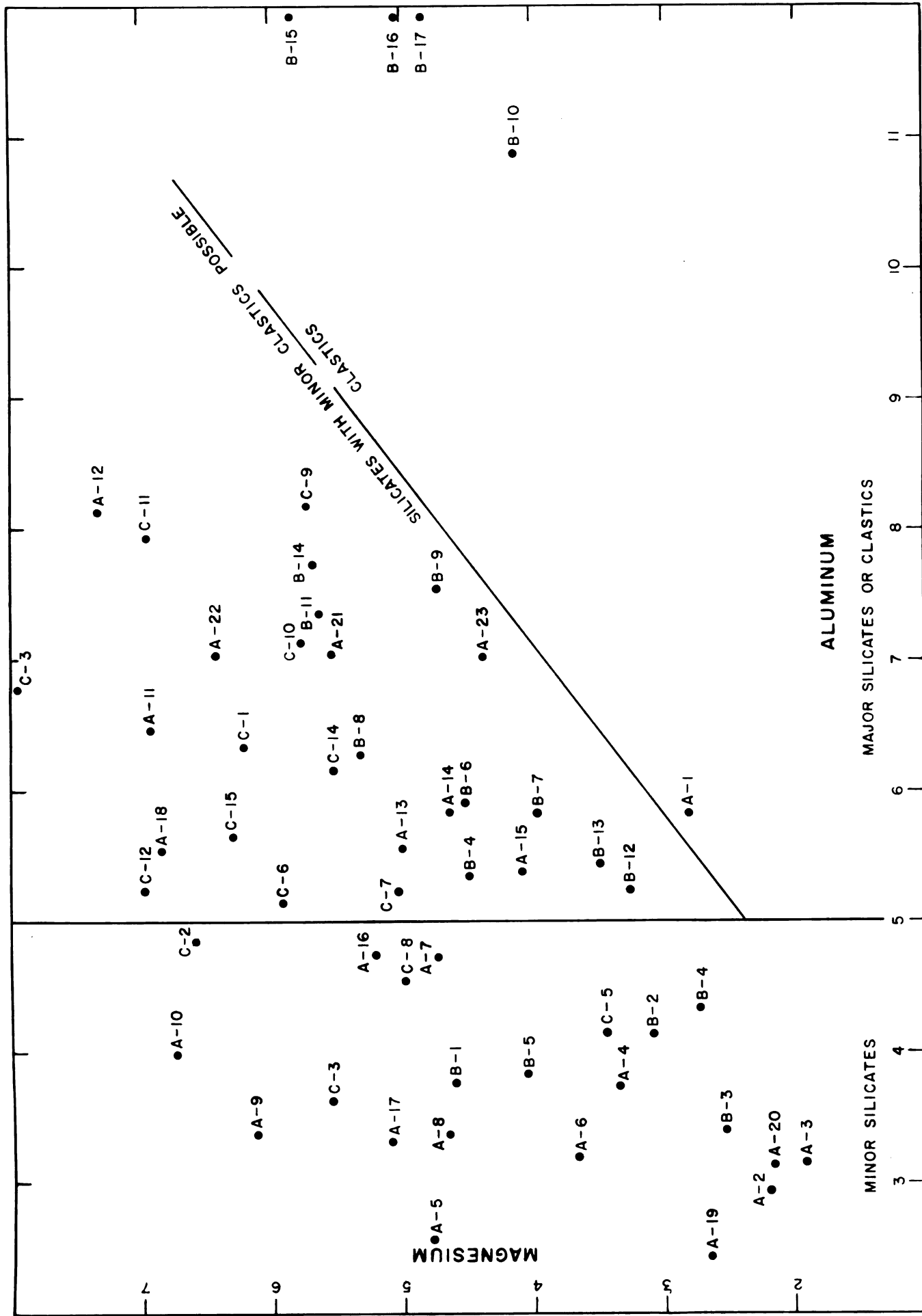


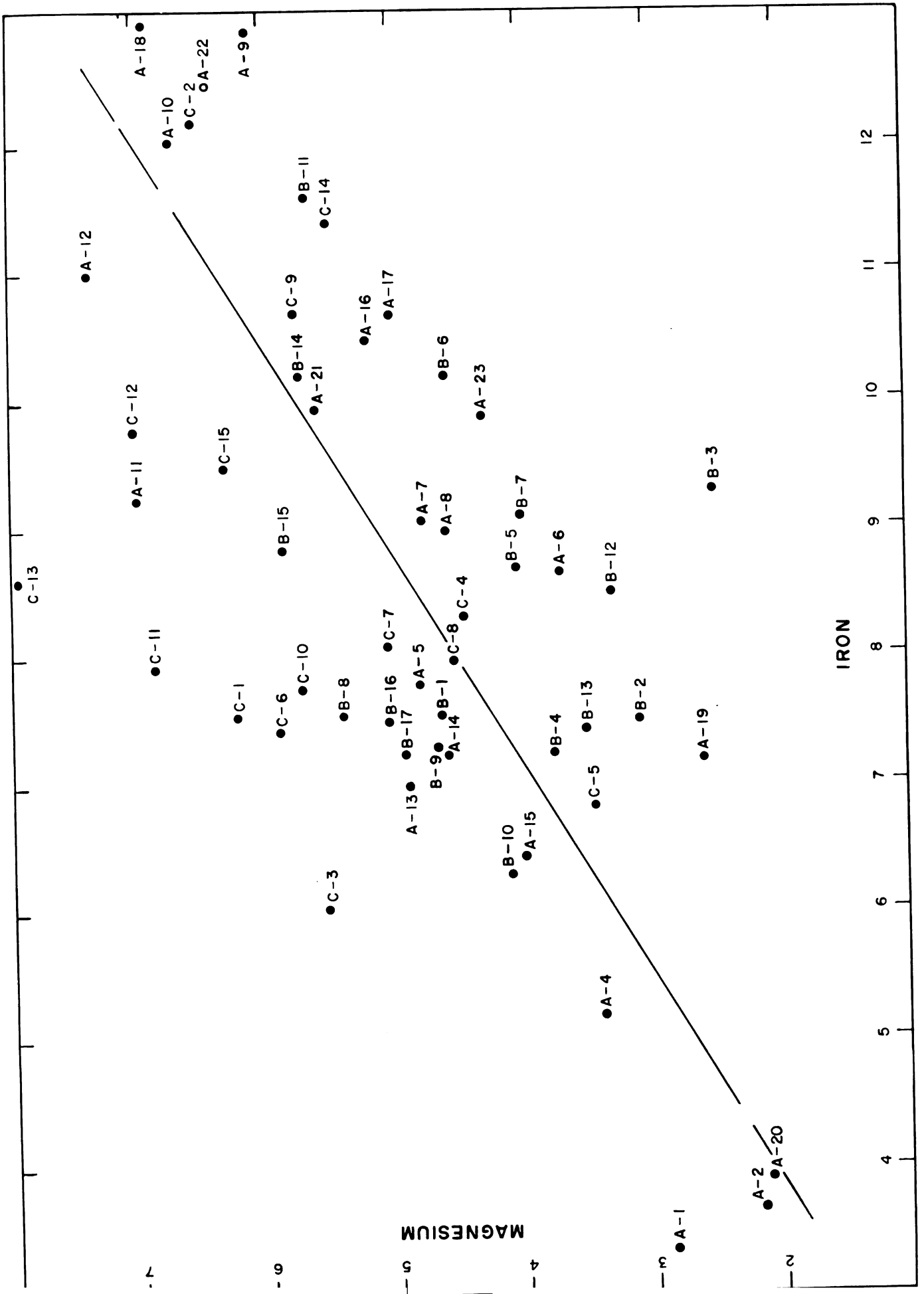
transition point was chosen from drill logs of those holes used in the sampling. The major facies types from the logs were used to approximate the maximum number of aluminum points falling into the respective facies as defined. Individual samples will not always fall into the gross classification due to possible mineral variation within any zone.

In Figure II the points designated A-10 and A-18 have similar concentrations of manganese and magnesium, but distinctly different aluminum values (Figure III) indicate possible clastics in sample A-18. Similarly, by comparing A-11 which contains additional manganese with A-18, there is a noted increase in both silicate and carbonate minerals. Sample A-11 would be classified as a silicate. High aluminum with average magnesium indicates the presence of clastics, usually quartz and feldspar grains (sample B-10, 15, 16, 17-- Figure III).

A plot of magnesium against iron (Figure IV) shows a similar point trend which would naturally follow since iron is also present in ever increasing amounts as Mn and Mg increase. Although the implication of high iron and low magnesium could indicate an increase in the iron oxide, this would not always hold true due to the inherent variable substitution of iron in the carbonate and silicates (i.e. high iron-low magnesium carbonate).

As a test, facies determinations were made for each point from the graphs and plotted on drill logs (Appendix A) at the proper footages. A good correlation exists between macroscopic logging and the spectrochemical results. There is also good stratigraphic correlation of facies along strike of the basal Negaunee iron-formation as evidenced by holes A and B which penetrated the slate footwall.





## CONCLUSIONS

A basic spectrochemical procedure has been established to produce relative element values which can be used to evaluate the facies for any given portion of the primary Negaunee iron-formation. Interpretation is based on the distribution and relation of the elements magnesium, manganese, aluminum and iron.

The method affords a rapid and reliable tool to form ratios that can be plotted in graph form which in turn can be used to approximate the quantity of carbonate or silicate present. The standardized spectrochemical procedure has time advantages over polished section studies, loss on ignition tests and grindability studies which can also be used to differentiate the two facies types.

The bases for ion concentration in the facies can be related to both physical and chemical environmental conditions. A distinct pH zone of 6.9 to 7.4 and an Eh of possibly 0 to -2 has been indicated by the elements present, the concentrations and the compounds formed. From the stability fields governed by the above conditions, a reliable interpretation of both element occurrence and concentration can be made from the spectrochemical data.

Manganese substitutes for iron and magnesium in the complex carbonate and can be used to approximate the quantity of carbonate within the limits of the possible manganese (.1 - .01% of the cations) substitution in the mineral structure. The manganese substitution is controlled by ion availability.

Both plots of manganese and aluminum against magnesium show a trend of mutual increase or decrease. The behavior is anticipated due to the presence of magnesium in both the carbonate and the silicate, the quantity being distinctly higher in the carbonate due to greater substitution and the greater per cent of carbonate present.

Silicate values can be approximated by the aluminum values when they have not been increased by the presence of detrital feldspar.

High iron values in conjunction with low Mn and Mg values may show the presence of iron oxides, but caution must be exercised in accepting the apparent analysis due to the variable iron concentration in both the carbonate and silicates. Similarly, low Mg and Mn values with lower Fe show a predominance of chert. Indications are that the silica and phosphorous values obtained by the spectrochemical method do not help evaluate the facies types.

Further experimentation may possibly resolve the problems of making a set of workable standards in a carbonate matrix so that direct quantitative results could be obtained. A study of trace elements in the same facies may also help differentiate between the two types. It would seem that a detailed trace element study would depend on knowledge of the relations of these elements in the particular facies and the reason for their distribution. Based on equilibrium studies, these reasons are not completely understood at the present.

In recent years metallurgical advances have permitted active mining concerns to open new low grade magnetite properties. Since magnetite-chert-silicate ore is twice as hard as magnetite-chert-carbonate ore, it is important to define the two types prior to profitable beneficiation. The spectrochemical method could be utilized, when standardized, to predetermine the carbonate-silicate ore types.



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## APPENDIX A

Each sample used in the spectrochemical procedure is listed in the following tables. The number is followed by the footage interval sampled, the soluble iron for that ten foot interval from which the sample was taken, and the classification based on the spectrochemical data. A cross section of the diamond drill hole is shown with the gross classification as established at the time of drilling by a company geologist and an abbreviated spectrographic classification at the proper footages.

### LEGEND

c .... carbonate  
s .... silicate  
cs .... carbonate-silicate  
/c .... clastics

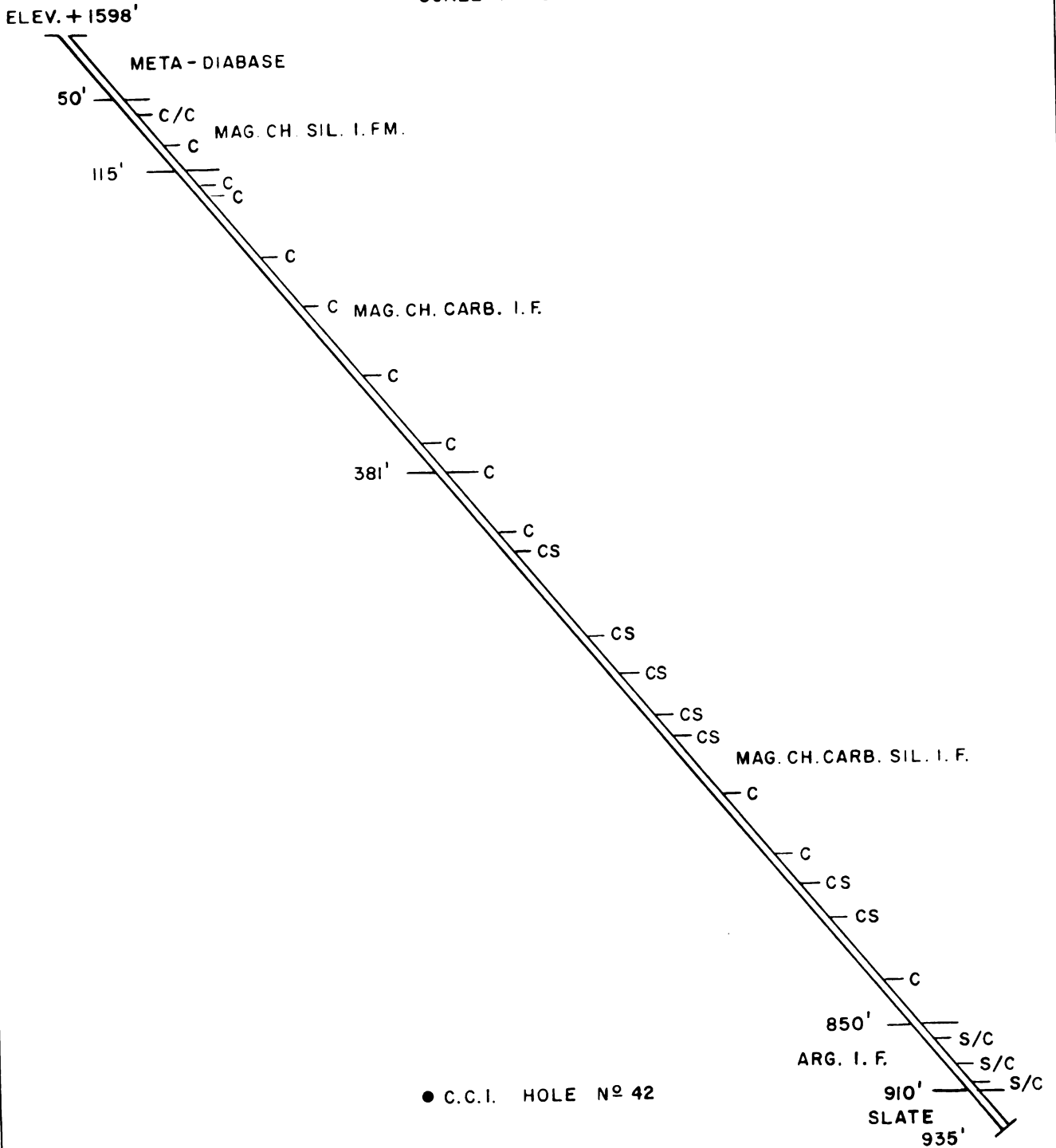
<u>SAMPLE A*</u>	<u>FOOTAGE SAMPLED</u>	<u>% SOL. FE FOR 10' INTERVAL</u>	<u>SPECTROCHEMICAL CLASSIFICATION</u>
A 1	68	24.3	c/c
A 2	98	30.6	c
A 3	127	30.1	c
A 4	134	29.0	c
A 5	190	31.0	c
A 6	238	31.2	c
A 7	297	34.9	c
A 8	358	33.8	c
A 9	380	29.4	c
A 10	431	31.6	c
A 11	494	31.2	cs
A 12	523	32.0	cs
A 13	552	30.3	cs
A 14	586	29.4	cs
A 15	604	29.3	cs
A 16	658	34.3	c
A 17	700	36.5	c
A 18	734	34.9	cs
A 19	766	33.7	c
A 20	819	31.5	c
A 21	863	31.3	s/c
A 22	886	28.5	s/c
A 23	901	----	s/c
			<u>slate</u>

\*Core donated by the Cleveland-Cliffs Iron Company.

# CROSS SECTION HOLE "A"

INCLINATION -45°, BEARING DUE EAST

SCALE 1"=100 FT.



<u>SAMPLE B*</u>	<u>FOOTAGE SAMPLED</u>	<u>% SOL. FE FOR 10' INTERVAL</u>	<u>SPECTROCHEMICAL CLASSIFICATION</u>
B 1	164	40.6	c
B 2	228	32.9	c
B 3	248	35.5	c
B 4	273	34.5	c
B 5	307	33.2	c
B 6	318	35.0	s
B 7	337	30.8	s
B 8	358	32.6	s
B 9	384	34.8	cs
B 10	402	32.8	cs
B 11	422	32.8	s
B 12	463	40.3	s
B 13	512	35.5	cs
B 14	542	31.7	cs
B 15	554	26.6	cs
B 16	566	22.3	cs/c
B 17	576	10.6	cs/c
			<u>slate</u>

\*Core donated by the Jones & Laughlin Steel Corporation.

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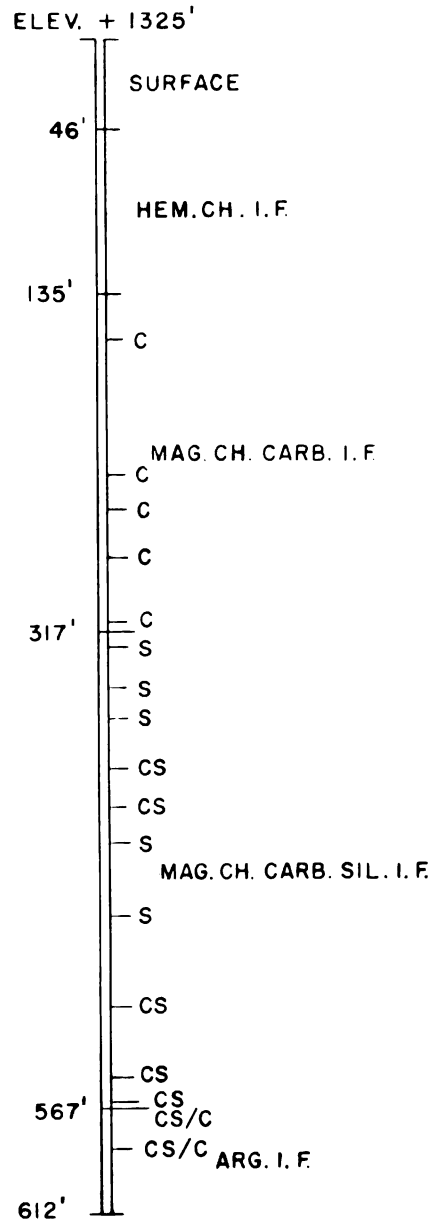
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# CROSS SECTION HOLE "B"

INCLINATION -90°

SCALE 1"=100 FT.



● J. & L. HOLE N<sup>o</sup> 848



<u>SAMPLE C*</u>	<u>FOOTAGE SAMPLED</u>	<u>% SOL. FE FOR 10' INTERVAL</u>	<u>SPECTROCHEMICAL CLASSIFICATION</u>
C 1	145	----	CS
C 2	214	26.9	C
C 3	224	26.5	C
C 4	247	26.5	CS
C 5	277	27.3	C
C 6	281	25.6	CS
C 7	301	28.5	CS
C 8	366	31.3	C
C 9	399	33.2	CS
C 10	459	34.9	CS
C 11	529	36.3	CS
C 12	579	37.6	CS
C 13	639	37.6	CS
C 14	696	33.6	CS
C 15	739	33.0	CS

**\*Core donated by the Jones & Laughlin Steel Corporation.**

1. The first part of the document discusses the importance of maintaining accurate records of all transactions.

2. It also highlights the need for regular audits to ensure compliance with applicable laws and regulations.

3. Furthermore, the document emphasizes the role of transparency in building trust with stakeholders.

4. Finally, it concludes by stating that a strong internal control system is essential for the long-term success of any organization.

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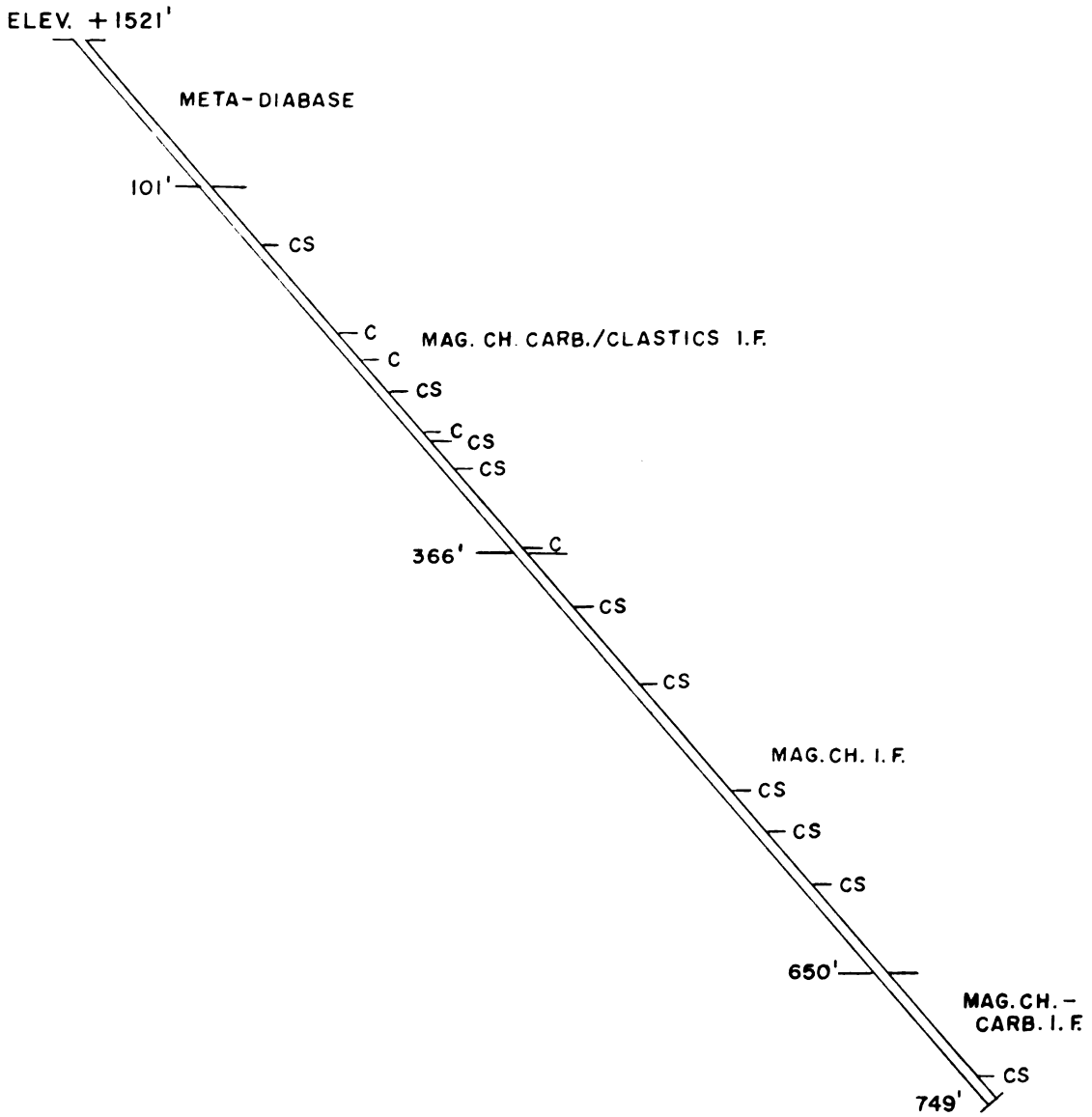
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# CROSS SECTION HOLE "C"

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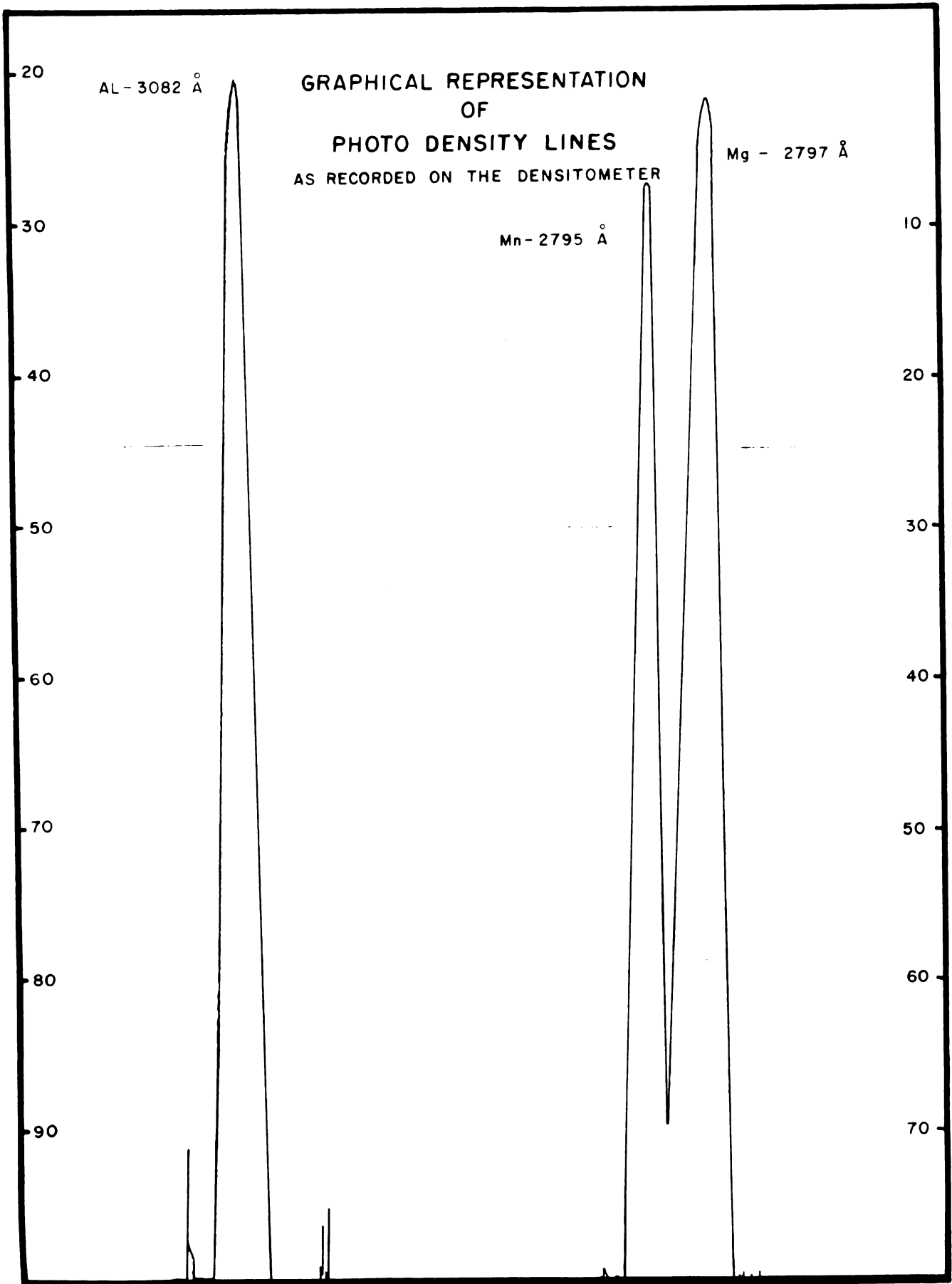


● J. & L. HOLE N° 780

## APPENDIX B

The following charts represent the measured data obtained from the densitometer graphs. Measurement of amplitude was taken from a constant base line and width values were taken between the two graph lines which intersect the base line. The measurements were made in centimeters so that the area is in square centimeters. The type of scale is unimportant as the only significant factor is the use of a standardized scale for all data.

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D A T A H O L E B

<u>SAMPLE</u> B	<u>CORRECTION FACTOR</u>	<u>STRONTIUM 3381 A</u>		<u>IRON 3021 A</u>		<u>ALUMINUM 3082 A</u>	
		<u>VOLUME</u>	<u>CORRECTED VALUE</u>	<u>VOLUME</u>	<u>CORRECTED VALUE</u>	<u>VOLUME</u>	<u>CORRECTED VALUE</u>
1	1.06	7.56	8.00	7.10	7.54	3.56	3.78
2	.94	8.48	"	7.98	7.50	4.43	4.16
3	1.44	5.55	"	6.46	9.30	2.36	3.40
4	.97	8.28	"	7.51	7.29	4.47	4.34
5	1.21	6.61	"	7.18	8.70	3.19	3.86
6	1.57	5.08	"	6.45	10.20	3.77	5.92
7	1.36	5.88	"	6.67	9.10	4.28	5.82
8	.97	8.24	"	7.77	7.55	6.50	6.30
9	.85	9.40	"	8.58	7.29	8.90	7.56
10	.72	11.10	"	8.80	6.33	15.20	10.90
11	1.68	4.76	"	6.94	11.60	4.38	7.35
12	.97	8.26	"	8.80	8.54	5.42	5.25
13	.83	9.60	"	9.00	7.47	6.56	5.44
14	1.29	6.21	"	7.88	10.20	5.94	7.66
15	.97	8.25	"	9.02	8.75	18.10	17.50
16	.96	8.31	"	7.81	7.51	12.70	12.20
17	1.00	8.01	"	7.25	7.25	18.96	18.96

D A T A   H O L E   B

<u>SAMPLE</u> <u>B</u>	<u>CORRECTION FACTOR</u>	<u>MAGNESIUM 2797 A</u>		<u>MANGANESE 2795 A</u>	
		<u>VOLUME</u>	<u>CORRECTED VALUE</u>	<u>VOLUME</u>	<u>CORRECTED VALUE</u>
1	1.06	4.36	4.62	1.38	1.46
2	.94	3.30	3.10	2.12	1.99
3	1.44	1.74	2.51	.05	.07
4	.97	2.82	2.73	.70	.68
5	1.21	3.32	4.02	1.80	2.18
6	1.57	2.91	4.57	.57	.89
7	1.36	2.94	4.00	.57	.77
8	.97	5.55	5.38	1.79	1.73
9	.85	5.57	4.74	2.48	2.11
10	.72	5.65	4.07	2.75	1.98
11	1.68	3.36	5.65	.68	1.14
12	.97	3.39	3.29	.99	.96
13	.83	4.22	3.50	2.62	2.18
14	1.29	4.43	5.71	1.34	1.73
15	.97	6.02	5.84	2.15	2.08
16	.96	5.30	5.08	2.81	2.70
17	1.00	4.88	4.88	1.58	1.58



D A T A   H O L E   B

<u>SAMPLE</u> <u>B</u>	<u>CORRECTION FACTOR</u>	<u>SILICON 3881 A</u>		<u>PHOSPHOROUS 2553 A</u>	
		<u>VOLUME</u>	<u>CORRECTED VALUE</u>	<u>VOLUME</u>	<u>CORRECTED VALUE</u>
1	1.06	5.95	6.31	7.86	8.34
2	.94	6.26	5.88	7.24	6.80
3	1.44	4.15	5.98	2.28	3.28
4	.97	5.92	5.74	6.48	6.28
5	1.21	4.78	5.79	3.26	3.95
6	1.57	4.05	6.36	1.26	1.98
7	1.36	5.26	7.16	1.07	1.46
8	.97	5.44	5.27	3.69	3.58
9	.85	6.18	5.25	6.00	5.10
10	.72	6.86	4.94	7.38	5.31
11	1.68	4.17	7.01	1.18	1.97
12	.97	6.87	6.66	7.78	7.55
13	.83	5.51	5.47	8.90	7.39
14	1.29	5.01	6.45	2.54	3.28
15	.97	6.94	6.64	6.08	5.90
16	.96	8.33	8.00	5.87	5.63
17	1.00	9.15	9.15	4.00	4.00

D A T A   H O L E   C

SAMPLE C	CORRECTION FACTOR	STRONTIUM 3381 A		IRON 3021 A		ALUMINUM 3082 A	
		VOLUME	CORRECTED VALUE	VOLUME	CORRECTED VALUE	VOLUME	CORRECTED VALUE
1	.93	8.66	8.00	8.13	7.55	6.81	6.34
2	1.76	4.56	"	6.95	12.20	2.75	4.84
3	.84	9.55	"	7.25	6.09	4.31	3.62
4	.97	8.28	"	8.60	8.35	5.54	5.37
5	.97	8.25	"	7.05	6.85	4.24	4.11
6	.92	8.72	"	8.08	7.43	5.58	5.14
7	.93	8.60	"	8.72	8.10	5.67	5.27
8	.99	8.10	"	8.05	7.97	5.22	5.16
9	1.37	5.86	"	7.86	10.70	5.98	8.20
10	.93	8.64	"	8.38	7.79	7.69	7.15
11	.86	9.27	"	9.20	7.91	9.21	7.94
12	1.04	7.66	"	9.40	9.78	5.03	5.23
13	1.02	7.84	"	8.46	8.64	6.55	6.68
14	1.52	5.26	"	7.54	11.40	4.06	6.17
15	1.43	5.59	"	6.65	9.51	3.97	5.67

D A T A H O L E C

<u>SAMPLE</u> <u>C</u>	<u>CORRECTION FACTOR</u>	<u>MAGNESIUM 2797 Å</u> <u>VOLUME CORRECTED VALUE</u>	<u>MANGANESE 2795 Å</u> <u>VOLUME CORRECTED VALUE</u>
1	.93	6.71	4.14
2	1.76	3.72	1.32
3	.84	6.62	2.33
4.	.97	4.64	3.23
5	.97	3.57	1.53
6	.92	6.44	3.24
7	.93	5.42	3.53
8	.99	4.62	3.20
9	1.37	4.64	1.45
10	.93	6.17	3.50
11	.86	8.05	4.90
12	1.04	6.72	4.40
13	1.02	7.91	4.48
14	1.52	3.62	1.51
15	1.43	4.42	1.18
			3.85
			2.32
			1.96
			3.13
			1.48
			2.98
			3.28
			3.17
			1.99
			3.25
			4.22
			4.58
			4.57
			2.39
			1.69

D A T A   H O L E   C

<u>SAMPLE</u> <u>C</u>	<u>CORRECTION FACTOR</u>	<u>SILICON 2881 Å</u> <u>VOLUME CORRECTED VALUE</u>	<u>PHOSPHOROUS 2553 Å</u> <u>VOLUME CORRECTED VALUE</u>
1	.93	7.08	8.18
2	1.76	6.79	2.62
3	.84	5.67	4.22
4	.97	6.72	6.76
5	.97	8.05	1.46
6	.92	5.48	4.28
7	.93	5.58	8.05
8	.99	10.69	4.82
9	1.37	7.71	4.02
10	.93	6.34	9.73
11	.86	5.89	11.00
12	1.04	8.89	11.10
13	1.02	6.16	6.64
14	1.52	7.00	1.56
15	1.43	5.44	1.90

D A T A H O L E A

<u>SAMPLE</u> <u>A</u>	<u>STRONTIUM 3381 A</u>		<u>IRON 3021 A</u>		<u>ALUMINUM 3082 A</u>	
	<u>CORRECTION FACTOR</u>	<u>VOLUME CORRECTED VALUE</u>	<u>VOLUME CORRECTED VALUE</u>	<u>VOLUME CORRECTED VALUE</u>	<u>VOLUME CORRECTED VALUE</u>	<u>VOLUME CORRECTED VALUE</u>
1	.38	21.10	8.00	9.41	3.58	15.30
2	.36	22.30	"	10.30	3.71	5.45
3	.36	22.10	"	-----	-----	8.85
4	.57	14.20	"	9.12	5.19	6.57
5	.79	10.20	"	9.94	7.85	3.24
6	1.03	7.80	"	8.47	8.64	3.11
7	1.10	7.25	"	8.20	9.03	4.30
8	1.17	6.84	"	7.68	9.00	2.88
9	1.86	4.31	"	7.51	14.00	1.82
10	1.78	4.50	"	6.87	12.20	2.26
11	.99	8.07	"	9.50	9.40	6.51
12	1.18	6.79	"	9.36	11.00	6.88
13	.76	10.50	"	9.31	7.08	7.34
14	.78	10.30	"	9.30	7.25	7.49
15	.64	12.50	"	10.40	6.66	8.48
16	1.43	5.61	"	7.32	10.50	3.31
17	1.43	5.61	"	7.47	10.70	2.32
18	1.51	5.31	"	8.60	13.00	3.65
19	.82	9.82	"	9.09	7.45	2.99
20	.46	17.60	"	8.51	3.87	6.80
21	.97	8.27	"	10.10	9.97	7.30
22	1.64	4.87	"	7.64	12.50	4.30
23	1.21	6.22	"	8.20	9.90	5.80

D A T A   H O L E   A

SAMPLE A	CORRECTION FACTOR	MAGNESIUM 2797 Å		MANGANESE 2795 Å	
		VOLUME	CORRECTED VALUE	VOLUME	CORRECTED VALUE
1	.38	7.47	2.84	5.56	2.11
2	.36	6.08	2.19	5.15	1.85
3	.36	5.37	1.93	3.18	1.14
4	.57	5.91	3.37	4.24	2.42
5	.79	6.08	4.80	4.97	3.93
6	1.03	3.58	3.69	1.11	1.14
7	1.10	4.31	4.74	2.08	2.29
8	1.17	4.01	4.69	2.13	2.49
9	1.86	3.29	6.12	1.49	2.77
10	1.78	3.81	6.78	1.33	2.37
11	.99	7.16	7.09	3.86	3.82
12	1.18	6.24	7.36	3.45	4.07
13	.76	6.55	4.98	3.58	2.72
14	.78	5.95	4.64	2.85	2.22
15	.64	6.41	4.10	3.55	2.77
16	1.43	3.65	5.22	.97	1.39
17	1.43	3.57	5.10	1.17	1.67
18	1.51	4.55	6.86	1.50	2.26
19	.82	3.25	2.66	.69	.57
20	.46	4.65	2.14	1.40	.64
21	.97	5.76	5.58	2.18	2.11
22	1.64	3.92	6.43	.02	.03
23	1.21	3.58	4.33	.45	.54

D A T A H O L E A

<u>SAMPLE</u> <u>A</u>	<u>CORRECTION FACTOR</u>	<u>SILICON 2881 A</u> <u>VOLUME CORRECTED VALUE</u>	<u>PHOSPHOROUS 2553 A</u> <u>VOLUME CORRECTED VALUE</u>
1	.38	10.40	10.40
2	.36	9.16	8.65
3	.36	9.03	6.94
4	.57	8.54	6.73
5	.79	6.40	5.63
6	1.03	4.04	2.83
7	1.10	6.06	3.11
8	1.17	9.74	.63
9	1.86	5.68	.69
10	1.78	4.47	----
11	.99	5.98	4.54
12	1.18	7.72	3.61
13	.76	8.01	4.06
14	.78	9.35	3.97
15	.64	8.05	3.92
16	1.43	4.72	.20
17	1.43	4.18	.14
18	1.51	5.51	2.96
19	.82	3.75	4.16
20	.42	5.62	2.35
21	.97	6.82	6.65
22	1.64	4.05	.03
23	1.21	6.91	1.77
			2.14

## APPENDIX C

Loss on ignition tests determine the amount of volatile material in any given rock. When applied to carbonate-silicate facies, it is possible to determine the amount of carbonate present by the per cent of CO<sub>2</sub> driven off.

A weighed sample is placed in a platinum crucible and heated for two hours at 105°C. When the moisture has been evaporated the sample is weighed to determine the loss. The crucible is then placed in an oven for one hour at 1000°C. after which the remaining sample is weighed. The end weight subtracted from the original weight minus the moisture determines the per cent of CO<sub>2</sub> that was volatilized.



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