Petrographic Examination of the Effects of Oxidation/Dehydration on a Sub-bituminous, Cretaceous, New Mexico Coal

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ABSTRACT

Coal cores that have been stored for a period of some years probably undergo some amount of oxidation/dehydration. Oxidation even to a very slight extent, severely reduces the caloric value and petrochemical use of the coal. Petrographic studies of these effects have only recently been put into use. This oxidation presents itself in the form of microcracks and micropores. Decreases in the amount of microcracks and micropores towards the center of the drillcore might be an indication of post-coring oxidation. An attempt was also made to establish a relationship between the cracks and pseudovitrinite because it was believed that one might contribute to the distribution of the other.

Extensive petrographic examination of the polished sections has revealed that, there apparently is no progressive decrease in oxidation/dehydration towards the center of the core. The extremely variable amounts of cracks and pseudovitrinite seen indicate that there apparently is no relationship between these two. Vitrinite reflectance measurements also did not indicate that the dehydration cracks had served as a pathway for oxidation. Analyses seeking a relationship between cleats and maceral content on one hand and oxidation/dehydration on the other have so far proven to be futile. Comparison of the data obtained in this study with the maceral analysis conducted recently yielded the same results. The reflectance also was almost the same. Long periods of improper storage therefore do not appear to affect the petrography of a coal.
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INTRODUCTION

The low temperature oxidation of coals does not cause any significant changes in their reflectivity (Chandra 1966). However, even very slight oxidation, adversely affects the coking properties of all coals (Gray, Rhoades and King 1976). The low temperature oxidation, even if active over a period of ten years does not affect the reflectivity of the coal (Chandra 1966). Oxidation of this type, however, produces characteristic physical structures such as, microcracks and micropores in the coal that are easily identifiable. Similar behavior in coals was reported earlier by M. and R. Teichmuller (1950), Nakayanagi (1956), Noel (1958) and Jacob (1964).

Until recently most New Mexico coals were studied using proximate and ultimate analysis, and by measuring their BTU values. Petrographic studies of coals were for the most part confined to eastern and European coals. Also most of the studies done, involved, either artificially induced laboratory controlled conditions of oxidation or were performed on coals from stockpiles in which one encounters fairly elevated temperatures. The coals under study were suspected of having undergone oxidation and dehydration under conditions of atmospheric temperature and pressure. The purpose of this study therefore was to determine the effects that periods of long storage might have had on coal
cores that were stored under less than ideal conditions. By less than ideal conditions for coal storage I mean, storage of the coal in boxes but not sealed in plastic. These boxes in turn were stored in a tin shed with no air-conditioning. This lack of air-conditioning caused summer temperatures in the shed to rise well above 115 degrees Farenheit, while winter temperatures were typically below freezing.

It was the opinion of the author that any petrographic changes brought about by oxidation and/or dehydration would increase or decrease as we went from the periphery towards the center of the drillcore. Decreases in the amounts of microcracks and micropores towards the center might be an indication of post coring oxidation.

GEOLOGIC LOCATION

The coal under study was derived from the Upper Cretaceous, Bisti, Fruitland area. This seam was selected for it's particular thickness and apparent cleanliness. The seam was sampled at five intervals that were progressively deeper down the borehole. The exact location of the samples was selected at places where there were obvious changes in macrolithotypotypes in the coal. This was done to ensure that petrographic variations arising due to the changing coal macrolithotypotypes would come under scrutiny.
The Bisti field is located in San Juan county, New Mexico, at the southern margin of the San Juan Basin (Fig 1). It is between 30 and 55 miles south of Farmington, in a band that parallels the Chaco River. The Bisti is one of the strippable areas, all of which lie along the margins of the basin. This particular area of strippable coal contains 1,870 million tons of subbituminous, high ash, low sulfur coal below less than 250 feet of overburden. The sulfur content averages 0.7% and the overburden is principally shale and soft sandstone. These factors make this field relatively attractive from an economic standpoint. (Shomaker, Beaumont and Kottlowski 1971).

The coals are derived from the Upper Cretaceous Fruitland Formation which consists of more than 6000 feet of sediments and lies conformably on top of the Pictured Cliff Sandstones. (Fassett and Hinds 1977). The Fruitland is composed of a series of trangressive and regressive beds that make up a series of highly lenticular nonmarine claystones, silty and sandy shales and soft cross bedded sandstones with coal. The fluviatile, lagoonal and nearshore marine origin of the Fruitland Formation resulted in great variations in short distances, laterally and vertically. (Fassett and Hinds 1977). Most of the minable coal lies in the lower fifth of the formation. Their principal orientation is northwest to southeast.
Figure 1. General index map showing the Fruitland and Menefee Formation coal fields in the San Juan Basin.
The Fruitland Formation is overlain conformably by the Kirtland Shale which is also of late Cretaceous age. It represents deposition landward from the coal forming swamps of the Fruitland Formation. Due to the fact that different geologists have picked the contact between the Fruitland and Kirtland very differently, it is somewhat ambiguous. It is generally placed at the highest coal or carbonaceous shale. (Fassett and Hinds 1977).
Figure 2. Outline map of the Bisti Field. The tiny black square is T22N, R10W in which hole E-15 is located.
In the area of study, T22N, R10W (Fig. 2), almost the entire township is underlain by the Fruitland Formation. One main and consistent bed of coal is present. (Shomaker, Beaumont and Kottlowski 1971). The thickness of the bed varies between 14 and 30 feet and is approximately 23 feet in the area of study. This is known as the "main bed" and it directly overlies the Pictured Cliff Sandstone. It is from this bed that the petrographic samples were taken.

**SAMPLING**

The petrographic samples were taken from five locations that were progressively deeper down the drillhole. The depths of the five samples taken are:

- 304.35' - 304.46' E15-D1
- 305.80' - 305.95' E15-D2
- 308.00' - 308.20' E15-D3
- 310.50' - 310.62' E15-D4
- 311.80' - 311.94' E15-D5

As mentioned earlier, the exact locations of the above five samples were dictated by changes observed in the coal macrolithotypes in the seam. For instance, E15-D2 was taken at that particular site because the macrolithotype changed to primarily durainitic.
Figure 3. Column of drill hole E-15 showing the locations of the coal seams and the partings. Also, the various locations at which the seam was sampled are indicated.
Similarly, E15-D3 was sampled at that site because it was principally inertinitic in composition. A detailed description of the coal macrolithotypes is given in APPENDIX I.

Figure 3 represents the drillcore E-15 as logged by the author. The region representing coal is solid black and has not been differentiated into vitrinite, durain and fusain. This is because according to M. and Th. Mackowsky (in E. Stach 1982), a particular macrolithotype has to be greater than 10 millimeters in thickness for it to be differentiated from the seam and be called by its own name. If not, everything is lumped together as clairain. None of the bands seen were greater than 10 millimeters in thickness. The durainitic and inertinitic regions mentioned earlier consisted of bands that individually were less than 10 millimeters in thickness, but, as an aggregate were greater than 10 millimeters.

Recovery of the coal from the drillcore was very good indeed. There has been almost no loss of core particularly at the junction between the roof and floor rock and the coal. Regretably however, coring was done only where there were shows of coal. Consequently, changes that might have occurred in the partings have gone undetected.
SAMPLE PREPARATION

The locations of the five samples mentioned earlier are indicated in Fig. 3. The samples are designated E15-D1, E15-D2,... and so on. E-15 refers to the drillhole. 'D' stands for the disc, and the greater the value of the number following 'D', the deeper down the drillhole is the location of the sample.

The discs thus removed from the core were then impregnated with epoxy. This was done to prevent the core from crumbling when it was sawed into sections. Once the discs had set in the epoxy, they were each sawed into four sections that were parallel to the vertical axis of the core. These sections were cut progressively deeper into the core, (Figs. 4 and 5) and were designated A, B, C, D. 'A' being the outermost and 'D' being the innermost section. The sampling was done in this manner because, it was the belief of the author that, any oxidation/dehydration that might have taken place would probably decrease as one went from the periphery of the core towards the center.
SIDE VIEW OF A COAL DISC SHOWING POSITIONS OF THE FOUR
SECTIONS CUT WITH RESPECT TO THE PERIPHERY

Figure 4. This figure serves to illustrate the manner in which the discs derived from the core were cut to produce the four polished sections.
TOP VIEW OF DISC SHOWING THE FOUR SLICES CUT FOR
POLISHED SECTIONS FROM THE PERIPHERY INWARD

Figure 5. This figure aims to illustrate the same point as Figure 4, it however is a view from above.
The actual distances of the sections from the circumference of the core are as follows:

A is about one tenth of an inch from the circumference.
This obviously is the smallest polished surface.
B is one-half inch from the circumference.
C is one inch from the circumference.
D is the central diameter.

The size of the polished surface therefore, is greater as one gets closer to the center of the core. The polished surface after the first cut was a maximum of one-sixteenth of an inch away from the actual desired position.

Thus from the five discs taken from the core, a total of twenty approximately rectangular sections were derived (Fig. 6). Each section was then set in a block of plastic and subjected to several stages of grinding followed by several stages of polishing until a flat, reflective polished surface was attained. The preparation of these samples was done at The Pennsylvania State University.
TOP VIEW OF A POLISHED SURFACE EMBEDDED IN PLASTIC

Figure 6. This figure shows the completed polished section embedded in plastic.
The following is hand specimen description of the coal macrolithotypes seen in the five discs that were removed from the core for the petrographic samples.

**E15-D1**

The luster of the coal in this the first disc was intermediate. The disc was composed of fine to medium alternating bands of vitrain and durain with vitrain being the dominant lithotype. Accessory amounts of resain were seen along some of the cleats which were principally concoidal in pattern.

**E15-D2**

For the second disc, the luster was dull. Alternating bands of durain and vitrain were present, with durain being the dominant lithotype. No adventitious ash was seen and the cleat pattern was irregular to blocky. At the very bottom of the disc the coal is principally bright with some resain.

**E15-D3**

This disc being composed mainly of semifusinite, had an intermediate bright luster. The bands were of medium (almost thick) width and were alterations of inertinitic material and durain. The cleats were irregular in pattern and the ash present did not occupy any preferred location.
EL5-D4
This disc had an intermediate luster. The bands that composed it were thin to medium in width and were alternations of vitrain and durain. The cleat pattern was blocky and no adventitious ash was visible.

EL5-D5
This the last of the five discs had an intermediate dull luster. The bands were of medium width and were alternations of vitrain and durain. Adventitious ash seen comprised approximately 2% of the volume of the disc. Some resin was seen and it did not appear to occupy any preferred location. The cleat pattern was irregular to blocky.
ANALYTICAL METHODS

The petrographic analysis of the polished sections was accomplished by doing both, a detailed maceral analysis and by measuring the vitrinite reflectance of each section. Although according to Chandra (1966), the reflectance of coal does not undergo significant change as a result of minor oxidation, the author was of the belief that, if measured accurately enough, the minor changes that may have occurred would become apparent. This was based on the fact that, the aromaticity of oxidized vitrinite is higher than that of fresh vitrinite (Goodarzi, and Murchison, 1976). In any case, measuring the vitrinite reflectance proves to be a very accurate tool for determining the rank of coals.

MACERAL ANALYSIS

Maceral analysis on polished sections of coal can be performed according to two different systems.

1) The rigid system

2) The natural system

(M. and Th. Mackowsky 1977, in Stach 1982) In the rigid system, a series of polished sections are analysed at regular intervals. In the natural system, the series of polished sections are subdivided on the basis of different lithotypes as exhibited by the macropetrographic seam section.
The natural system obviously is more flexible and is the system used in this study. It also happens to be the system that is preferred by a majority of petrographers nowadays because, it excludes the possibility of splitting up bands of vitrain and durain.

The microscope used for the study was a Nikon Labophot with a 40X nonimmersion objective. The specimen holder on the automatic point counter being too small to hold the polished section, a modified mechanical stage was used.

A total of 500 points were counted on each polished surface. The point to point and line to line distance selected was 1.0 mm. Ideally a point to point and line to line distance of 0.5 mm should be used (M. and Th. Mackowsky, 1977, in E. Stach 1982). However considering the size of the area to be studied (25 mm X 25 mm), and the fact that doubling the number of points counted would increase the accuracy of the analysis by a mere 0.5% (Stach 1982), it was decided that 500 counts would adequately serve the purposes of the study. The total area of the polished section is larger than 25 mm X 25 mm, but, the author was interested in studying only the same unit area of the core going from the periphery towards the center, that is from section A to section D.
The counting was begun from the upper left hand corner of the section. The author then proceeded to go to the right across the section, one millimeter at a time for a total of 25 points. Having reached the right hand extremity of the designated counting area, the section was moved up by 1 mm and counting resumed 1 mm at a time towards the left extremity, (Fig. 7). This procedure was repeated until a total of 500 points had been counted.
TOP VIEW OF A POLISHED SURFACE SHOWING PATH

FOLLOWED FOR POINT COUNTING

500 COUNTS PER POLISHED SECTION

POINT TO POINT DISTANCE 1.00 mm

LINE TO LINE DISTANCE 1.00 mm

Figure 7. The point to point and line to line distance used in this study are as specified in E. Stach 1982.
The various macerals and mineral matter that would be recognized are listed below:

Vitrinite Group -
  Vitriinite A - basic pure vitrinite
  Vitriinite B - perhydrous vitrinite
  Vitriinite C - pseudovitrinite

Exinite Group -
  Sporinite
  Cutinite
  Resinite
  Alginite
  Suberinite
  Liptodetrinite

Inertinite Group -
  Semifusinite
  Fusinite
  Macrinite
  Micrinite
  Sclerotinite
  Inertodetrinite

Mineral Matter -
  Clay
  Pyrite
Quartz

Cracks - Microcracks only

The detailed tabulated results of the maceral analyses are presented in Appendix II. In reduced form, the results have been presented as graphs in the section entitled RESULTS.
REFLECTANCE MEASUREMENTS

The rank of a coal is characteristic of the stage reached by it in the course of the transformation process which began with decayed vegetable matter and the final stage that is represented by graphite, (M. and Th. Mackowsky 1977, in Stach 1982). Since determination of coking power depends primarily on the accurate determination of the rank of the coal, reflectance measurements have always been fairly important. For this study the one beam method as proposed by McCartney and Hofer (1955) was used. In this method, the reflectance is measured using a photomultiplier placed upon the microscope. In the one beam method two successive reflectance measurements are compared. The first reading is taken off of a standard and the second off of the coal. The reflectance of the vitrinite can then be calculated using the formula:

\[ R_v = R_s \times \frac{A_v}{A_s} \]

where

- \( R_v \) = reflectance of vitrinite expressed in % R
- \( R_s \) = reflectance of the standard substance in % R
- \( A_v \) = photometer reading derived from vitrinite
- \( A_s \) = photometer reading derived from the standard

For each polished surface a total of 200 reflectance measurements were made. In order to maintain a check on the
long-term constancy of the system, a standard reading was taken at the start and then after every 25 readings. The glass standard used had a reflectance of 0.506%, and the oil used has a refractive index of 1.5150 ± or −0.0002. The microscope used was a Leitz, fitted with a 50X oil immersion objective. A Hamamatsu R 928–20020 side on photomultiplier tube was used. This was hooked up to a Schoeffel M460 photometer. The specimen was moved from left to right and right to left in the same manner as was done for the maceral analysis, (Fig. 7). Obviously readings were taken from vitrinite grains only. The readings from the photometer were used to calculate the reflectance using the expression given above.

The results of the reflectance readings are presented in the form of histograms in Appendix III. Also on the histograms are given the mean, standard deviation and range of reflectance values for each sample studied. The reflectance values calculated are classified in groups, each group consisting of a reflectance range of 0.05%R. These are termed half V-steps, a V-step being equal to 0.10%R, (M. and Th. Mackowsky 1977, in Stach 1982). A summarized version of the results is presented in TABLE I (page 48), in the section entitled RESULTS.

One other minor study was performed using reflectance measurements. This was done in an attempt to
prove the author's theory that, any oxidation and/or dehydration that may have occurred would have done so along cracks and weaknesses in the coal. If the cracks had indeed served as a pathway along which oxidation and/or dehydration had taken place then, progressive changes in the reflectance might be seen going outward from the crack. This was based on the study of Goodarzi and Murchinson (1976), that found that the aromaticity and hence the reflectance of an oxidized coal is higher than that of the fresh coal. Similar observations were made by Alpern and Maume (1969), during their studies on coal stockpiles. Actually, one could also have the development of dark bands next to the crack getting progressively lighter away from it. This would occur if the oxidation was very strong and one had the formation of humic acids.

This study was performed by taking reflectance readings exclusively on the brighter vitrinite that seemed to be associated with the cracks. Readings were taken in four parallel lines that were spaced progressively further away from the crack. Twenty five readings were taken along each line at 1 mm spacing. The mean reflectance for each of the four lines was calculated and the four were then compared to see if there was any progressive change outward from the crack.
(26)

ANALYSIS OF CRACK RELATED DATA

In the earlier petrographic studies attempts were made to relate all observations directly to the position of the polished section in the core. However, other factors, independent of the position of the polished section in the core could very well be controlling the oxidation/dehydration seen in the core. Two factors that come to mind are:-

1) The original cracks (not dehydration) that are present in the coal, most likely as a result of the cleats.

2) The amount of inertinite.

The original cracks, the distribution of which obstensibly is governed by the cleat pattern in the coal, were not counted as part of the maceral analysis. They were assumed not to have contributed to the dehydration of the coal in any way. The dehydration cracks were counted as it was assumed that their concentrations would have some relationship with the drying of the core from the periphery inwards. These two types of cracks can be told apart quite easily. The original cracks follow the cleat pattern of the coal and as a result, are primarily blocky or concoidal in form. The dehydration cracks, besides being much finer, are also dendritic in form and radiate out from the original
cracks.

It was later assumed that two kinds of cracks probably bore a relationship to one another since one of the ways in which cleats form in coal is by dehydration (Ting 1977). The original cracks probably contributed to and governed the distribution of the dehydration cracks. An attempt was therefore made to try and establish a relationship between the two and then relate this to the position of the polished section in the core. Since the original cracks had not been counted in the maceral analysis, they were quantified by measuring their lengths as seen under a low power microscope. Their total length was initially measured by running a piece of string along the length of each recognizable crack. The lengths measured as such, were checked and corrected using a mileage gauge for road maps. Establishing the relationship between the two types of cracks was then attempted by dividing the number of dehydration cracks by the total length of the original (cleats) cracks. The ratio thus obtained was then plotted against the position of the polished section in the core. These plots are included in the results section of this thesis.

Inertinite having a porous structure was also thought to be a contributor to the dehydration of the coal. It was believed that the porous structure of inertinite
would allow for greater loss of moisture from the core resulting in a greater number of dehydration cracks. The inertinite having already been counted as part of the maceral analysis did not need to be counted again. Establishing the relationship between it and the cracks was similarly done by dividing the number of counts for cracks by the number of counts for inertinite. This ratio was in turn plotted against the position of the polished section in the core. The plots are presented in the result section that follows.

One further and final study was attempted using the data obtained on the cracks. It was believed that, any original major cracks running across the polished section would act just like an outer surface of the core i.e. the periphery. It is through these outer surfaces that the core is believed to have dehydrated. If there is a large crack running through the core, then, the core has this crack in addition to it's periphery through which to dehydrate. Because the petrographic study was restricted to a unit area in the center of the polished section, a crack running through section 'D' for example, would have a greater effect on the dehydration of that part of the core than would the periphery, simply because it is nearer than the periphery. The distance of the center of the polished section from the nearest outside surface, be it a crack or the periphery, might bear some relation to the amount of dehydration that
the coal has undergone in that particular section. If there are no cracks running across the polished section then, the nearest outer surface to the center of the core would be the periphery itself. Sections that have an original crack running through them would have their centers closer to an outside surface and would dehydrate faster and consequently would have a greater number of dehydration microcracks.

The shortest distance to an outside surface for each of the four polished sections A, B, C and D in the five discs is shown in Fig. 8. The lengths indicated are the shortest distance to an outside surface assuming that there are no cracks running across the polished section. In the actual polished sections there are original cracks present and therefore the distances to the nearest outer surfaces in most cases will be much shorter. Once these distances had been reported, an attempt was made to establish a relationship between the dehydration cracks and the distance to the nearest outer surface. It was hoped that this study would help explain why there apparently is an erratic instead of progressive change in the degree of oxidation/dehydration toward the center of the core.
Figure 8. The distances to the nearest outside surfaces shown here are the distances to the periphery of the core. These therefore are the distances measured assuming that there are no original (cleats) cracks at closer distances to the center of the polished sections.
RESULTS AND DISCUSSION

The actual results of most of the work done are presented in the form of tables in appendixes at the end of this thesis. Presented here in this section are only plots which are reductions of all the data in the appendixes.

The first figure presented in this section (Fig. 9) shows the five plot symbols used throughout this first part of the thesis. Each disc has been represented by one plot symbol and the same symbol represents the four polished sections of one and only one disc wherever it appears in this thesis. For example, all the polished sections from D1 have been represented by triangles on all the plots that are forthcoming.
Figure 9. This is the key to the symbols used throughout this thesis. For all the figures that follow the symbols used are the same and represent one and only one disc. For example, The X represents D3 throughout.
The first plot (Fig. 10), is that of percent cracks (of 500 counts), versus position of the polished section in the core. The cracks plotted here are the microcracks seen which as pointed out earlier, are the results of oxidation and dehydration. Progressive changes in the abundance of microcracks as one goes from the periphery of the core toward the center would probably be an indication of post-coring oxidation. The author expected the abundance of microcracks to decrease from the periphery toward the center as a result of oxidation that set in from the periphery and moved in as a converging front.
Figure 10. The only thing of note in this figure is the fact that there is no particular trend seen as one goes from the periphery of the core towards its center. There is no systematic increase or decrease in the number of cracks.
Looking at the graph in Figure 10 one notes that the trends seen are not quite what the author had expected. There is no progressive change in the amount of microcracks going from the periphery of the core toward the center. The actual trends seen are as follows:

**El5-D1** - The number of cracks increases very slightly from polished section A to polished section C and then drops sharply to a value slightly less than that in A when we get to section D.

**El5-D2** - The number of cracks increases sharply from polished section A to polished section C and then levels off between C and D.

**El5-D3** - Polished sections A and B exhibit the same concentrations of microcracks, this number increases from B to C and then levels off between C and D.

**El5-D5** - In this disc the number of microcracks decreases sharply from polished section A to polished section C and then increases slightly from C to D.

**El5-D5** - The number of microcracks first increases between A and B, then decreases between B and
C and levels off between C and D.

From the figure it is obvious that there is no progressive change in the number of cracks going from the periphery of the core inwards. The microcracks seen on each polished section owe their concentrations to some phenomena other than an oxidation front that moved in from the periphery of the core. This new and unknown agent or phenomena caused the evidence of oxidation in the core to be randomly distributed. This agent therefore has itself to be randomly distributed or reach down into the core from the surface. This would account for the number of microcracks, which are a direct reflection of the amount of oxidation, increasing and decreasing at random.
The second plot, (Figure 11), is that of percent Vitринite C (pseudovitrinite) versus position of the polished section in the core. It is known that Vitринite C or pseudovitrinite is an oxidation product. The author was of the belief that it was a product of post-coring oxidation. If such is the case then, the amount of Vitринite C should decrease as one goes from the periphery of the core toward the center in the same manner as was expected of the microcracks.
Figure 11. Believing Pseudovitrinite (vitrinite C) to be a product of post-coring oxidation, I had expected to see decreases in its concentration going from the periphery of the core toward the center. The figure above proves that my theory does not work. There is no progressive decrease in the vitrinite C concentration toward the center of the core.
Looking at the figure however, one again finds that the trends seen are not quite what the author had expected them to be. The amount of Vitrinite C in each set of four polished sections seems to change at random, sometimes increasing while at others decreasing. There appears to be no systematic or progressive change as one goes from the periphery inwards. The trends seen in each individual disc are as follows:

**E15-D1** - The amount of Vitrinite C decreases from polished sections A to B, it then decreases sharply from B to C and then increases sharply from C to D.

**E15-D2** - This the second disc also shows a gradual decrease from polished sections A to B, the amount of Vitrinite C then falls to zero in section C and then climbs sharply to polished section D.

**E15-D3** - The amount of Vitrinite C seems to stay fairly steady. It decreases slightly from polished section A to B, then increases slightly to C and stays almost static from C to D.

**E15-D4** - There is a sharp increase in the amount
of Vitrinite C from polished section A to B, a small decrease from B to C and then a fairly sharp increase from C to D.

EL5-D5 – In disc 5 the amount of Vitrinite C increases slightly from A to B. It then increases sharply from B to C and decreases sharply from C to D.

There therefore is no progressive change in the amount of Vitrinite C be it an increase or decrease from the periphery of the core inwards. The distribution of the Vitrinite C must therefore be governed by some agent other than an oxidation front moving inwards from the periphery of the core.
After examining the previous two plots it was suspected that maybe there was some agent other than an oxidation front that was governing the distribution of both the microcracks and the Vitrinite C. The first possibility that comes to mind is, maybe one of them is directly or dependent on the other or the two are interdependent. If such is the case then, a plot of one against the other probably should produce a straight line.
Figure 12. From this figure it is evident that the expected direct relationship between microcracks and the pseudovitrinite does not exist and the points from a single polished section plotting close to each other is simply a result of the dominant coal lithotype being the same for all the polished sections in a particular disc.
Upon examining figure 12 the author found that the expected linear relationship between microcracks and Vitrinite C did not exist. Increases or decreases in the amount of microcracks with Vitrinite C and vice versa do not take place.

Looking at the individual discs the author noticed the following:

**E15-D1** - The four points representing the polished sections from D1 plot in a relatively small almost closed area. The expected linear relationship is nowhere present.

**E15-D2** - The four points representing D2 seem to plot in a more open array than those in D1. However this is caused by the fact that polished section C does not have any Vitrinite C. The remaining points from D2 plot fairly close to each other.

**E15-D3** - The same results are observed in this the third disc. There is no linear relationship, but, again the four points representing this disc plot close to each other.

**E15-D4** - The four points representing the polished sections in this disc are
a more open arrangement akin to those in D2. Again however, only one point seems to be at a little bit of a distance from the other three.

EI5–D5 - The four points from this disc follow the general trend of plotting close to each other.

Again the authors expectations have not been met. There is no direct relationship between the microcracks and the Vitrinite C. The proximity of the points to each other can be explained simply as being due to the influence of the dominant coal macrolithotype. The macrolithotypes being different for each set of four points resulted in the points plotting in a relatively small though not isolated area.
Now, according to Alpern and Maume (1969), and Goodarzi and Murchison (1976), the reflectance of an oxidised coal is higher than that of the corresponding fresh coal. After measuring the reflectance of the Vitrinite in each polished section, the mean maximum reflectance was plotted against the position of the polished section in the core. As was evidenced with the microcracks, there appears to be no progressive change in the reflectance as one goes from the periphery of the core toward the center. However the microcracks being a definite indication of oxidation, the author decided to plot the mean maximum reflectance against the amount of microcracks seen. He was hoping to be able to detect any possible relationship that there might exist between the two. This plot represented by figure 13, appears on the following page.
Figure 13. It was hoped that there would be some relationship between the amount of microcracks and the mean reflectance. According to Alpern and Maume (1969), the aromaticity of a coal and consequently 't's reflectance should increase with oxidation. From the figure it is evident that no relationship exist between the two parameters.
<table>
<thead>
<tr>
<th>SAMPLE NUMBER</th>
<th>MEAN MAX. REFLECTANCE %R</th>
<th>STANDARD DEV s</th>
</tr>
</thead>
<tbody>
<tr>
<td>E15-D1-A</td>
<td>0.39</td>
<td>0.07</td>
</tr>
<tr>
<td>B</td>
<td>0.42</td>
<td>0.07</td>
</tr>
<tr>
<td>C</td>
<td>0.32</td>
<td>0.12</td>
</tr>
<tr>
<td>D</td>
<td>0.35</td>
<td>0.05</td>
</tr>
<tr>
<td>E15-D2-A</td>
<td>0.37</td>
<td>0.06</td>
</tr>
<tr>
<td>B</td>
<td>0.40</td>
<td>0.07</td>
</tr>
<tr>
<td>C</td>
<td>0.36</td>
<td>0.06</td>
</tr>
<tr>
<td>D</td>
<td>0.34</td>
<td>0.06</td>
</tr>
<tr>
<td>E15-D3-A</td>
<td>0.37</td>
<td>0.06</td>
</tr>
<tr>
<td>B</td>
<td>0.36</td>
<td>0.07</td>
</tr>
<tr>
<td>C</td>
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<td>0.06</td>
</tr>
<tr>
<td>D</td>
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<td>0.78</td>
</tr>
<tr>
<td>E15-D4-A</td>
<td>0.33</td>
<td>0.07</td>
</tr>
<tr>
<td>B</td>
<td>0.34</td>
<td>0.07</td>
</tr>
<tr>
<td>C</td>
<td>0.38</td>
<td>0.13</td>
</tr>
<tr>
<td>D</td>
<td>0.32</td>
<td>0.06</td>
</tr>
<tr>
<td>E15-D5-A</td>
<td>0.33</td>
<td>0.06</td>
</tr>
<tr>
<td>B</td>
<td>0.33</td>
<td>0.07</td>
</tr>
<tr>
<td>C</td>
<td>0.36</td>
<td>0.07</td>
</tr>
<tr>
<td>D</td>
<td>0.31</td>
<td>0.06</td>
</tr>
</tbody>
</table>
As explained in the section on analytical methods, two hundred readings of reflectance were taken for each polished section. These were then plotted up as histograms which in a majority of cases showed that we had a normal distribution of reflectance values. The histograms are included in Appendix III. The mean values of reflectance that appear on each histogram are the same values used in the next figure and that appear in Table I.

Upon close examination of figure 13, it was found that there apparently is no direct relationship between mean reflectance and the number of microcracks. However, the author did notice that, similar to figure 12, the points in this figure that represent a single disc all seem to plot in a relatively small almost closed area. These almost closed areas the author speaks of are smaller in figure 13 than they are in figure 12. Again, the explanation that he offers for this phenomena is that, the behavior of the points plotted is governed by the coal macrolithotype in a particular disc. Thus, for example, in the case of D3 whose four polished sections are represented by the X's, the macrolithotype throughout the disc is inertinitic and this causes the four points to plot relatively close to each other in a somewhat closed fashion.
When plots of mean reflectance versus microcrack abundance did not show any direct relationship between the two, the author examined the changes taking place in the reflectance as one went from the crack itself, outward into the surrounding coal. A decrease in the reflectance away from the crack would indicate that the crack had served a pathway for the oxidising agent. The reflectance however did not decrease systematically away from the crack. The microcracks therefore are not contributing to the oxidation though they most definitely are a result of the same.

Thus far the author has taken into account all the factors that are known to account for oxidation or at least are what he thought to be indicators of oxidation. These factors are:

1) Microcracks
2) Vitrinite C (pseudovitrinite)
3) Reflectance
4) Position of the polished section

None of these showed any progressive change in the degree of oxidation as one went from the periphery of the core toward the center. Changes in the degree of oxidation seen were for the most part random and therefore an explanation was sought for this randomness. This resulted in the "Crack Analysis" that follows immediately. The results of this analysis are presented in Table II. The data in the table includes the total length of the "original cracks" 'L'
(cleat fractures), the number of microcracks 'X', the number of counts of inertinite 'I', the ratio of microcracks to total length of the original cracks X/L, the ratio of microcracks to inertinite X/I and finally, the distance to the nearest outer surface or original crack (cleat) P'.
### TABLE II

<table>
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<tr>
<th>SAMPLE</th>
<th>L</th>
<th>X</th>
<th>I</th>
<th>X/L</th>
<th>X/I</th>
<th>P'</th>
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<tbody>
<tr>
<td>E15-D1-A</td>
<td>16.55&quot;</td>
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<td>57</td>
<td>1.32</td>
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<td>0.10&quot;</td>
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<tr>
<td>B</td>
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<td>1.47</td>
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<td>0.15&quot;</td>
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<td>11</td>
<td>2.11</td>
<td>3.82</td>
<td>0.55&quot;</td>
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<tr>
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<td>0.10&quot;</td>
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<tr>
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<td>1.41</td>
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<td>0.15&quot;</td>
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<tr>
<td>C</td>
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<td>66</td>
<td>2.43</td>
<td>0.38</td>
<td>0.50&quot;</td>
</tr>
<tr>
<td>D</td>
<td>11.95&quot;</td>
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<td>63</td>
<td>1.51</td>
<td>0.29</td>
<td>0.55&quot;</td>
</tr>
<tr>
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<td>220</td>
<td>3.24</td>
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<td>B</td>
<td>26.65&quot;</td>
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<td>213</td>
<td>2.55</td>
<td>0.32</td>
<td>0.35&quot;</td>
</tr>
<tr>
<td>C</td>
<td>23.40&quot;</td>
<td>74</td>
<td>170</td>
<td>3.16</td>
<td>0.44</td>
<td>0.40&quot;</td>
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<td>D</td>
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<td>144</td>
<td>3.51</td>
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<td>0.00&quot;</td>
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<tr>
<td>E15-D4-A</td>
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<td>74</td>
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<td>0.77</td>
<td>0.10&quot;</td>
</tr>
<tr>
<td>B</td>
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<td>64</td>
<td>2.90</td>
<td>0.63</td>
<td>0.20&quot;</td>
</tr>
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<td>1.87</td>
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<td>0.15&quot;</td>
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<td>56</td>
<td>1.91</td>
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<td>0.20&quot;</td>
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<td>E15-D5-A</td>
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<td>0.05&quot;</td>
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<td>0.10&quot;</td>
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<td>17.90&quot;</td>
<td>13</td>
<td>64</td>
<td>0.73</td>
<td>0.20</td>
<td>0.20&quot;</td>
</tr>
</tbody>
</table>

Where:

- **L** = Total measured length of original cracks (cleats)
- **X** = Number of microcracks
- **I** = Number of inertinite counts
- **X/L** = Microcracks per unit original crack length
- **X/I** = Microcracks per inertinite count
- **P'** = Distance to nearest outer surface
Figure 14. Notice that no trend exists either from the periphery of the core to the center or vice versa.
In figure 14, the ratio of microcracks to original crack length has been plotted against the position of the polished section in the core. This was done in the hope that the ratios would change with position in the core. Plotting the microcrack abundance against the original crack length did not yield any conclusions. The plot shown in figure 14 was made using data only from E15-D2. This was done because D2 best approximates the behavior of the other discs collectively.

Looking at figure 14 one finds that the ratio drops from polished section A to polished section B. It then climbs from B to C and finally drops again to D. The numbers appearing on the figure next to the points plotted are those of inertinite counts. Notice that the fluctuations in the ratio bear no relationship to the inertinite concentrations or to the position of the polished section in the core.

Since the author assumed there to be a relationship between inertinite concentration and the degree of oxidation/dehydration because of the porous structure of inertinite, the next plot, figure 15, is that of ratio of microcracks to counts of inertinite versus position in the core. Here again only the behavior exhibited by disc D-2 is shown.
Figure 15. This is almost the same as figure 14. There is no apparent trend going from the outside in or vice versa.
Similar to figure 14, figure 15 shows a drop in the ratio from polished section A to polished section B, then an increase to C and again a decrease to D. There apparently is no relationship and position in the core or between the ratio and the corresponding total length of the original (starting) cracks 'L', which is written below each plotted point on the figure.

Having failed thus far to discover any relationship between oxidation and the various other parameters, the author did one last test. This took into consideration the distance of the center of each polished section from the nearest cleat fracture, also known as original crack or the distance to the periphery of the core whichever was closer. The cleat fracture it was believed would serve to expose the interior of the core to conditions that were similar to those experienced by the periphery. It was hoped that this would help explain why some parts of the interior of the core exhibited similar degrees of oxidation as the exterior. Therefore the closer a polished section to an outer surface or original crack (cleat), the greater should be the degree of its oxidation and consequently the greater the number of microcracks seen in that particular polished section.

The shortest distance to the nearest real outer surface (distance to periphery) for each polished surface is shown in figure 7. The measured distances to the nearest
outer surfaces be they the periphery or a cleat fracture are listed in Table II and denoted as P'. This value P' was then plotted against the percent microcracks as shown in figure 16 on the next page.
Figure 16. The expected relation between the distance to the nearest starting surface and the degree of oxidation is not seen. Apparently being close to a cleat or outer surface is not as good a control on oxidation as the primary coal lithotype in the disc.
It was hoped that there would be a direct linear relationship between the two quantities plotted in the figure. If such was the case then, it would be simple enough to explain the random distributions of the evidences of oxidation seen. The reasoning behind this being, the closer the cleat or outside surface to a particular polished section, the greater should be the degree of oxidation evidenced in that particular section.

Looking at figure 16 however, one notes that there apparently is no direct linear relationship between the distance to the nearest outside surface and the degree of oxidation seen. The points plot quite randomly and the degree of oxidation does not increase or decrease predictably. The only trend seen that probably is worth mentioning is that the four points representing the four polished sections from a particular disc seem to roughly plot in a straight vertical line with the exception of those representing D4. Again the explanation for this is that the coal macrolithotype being uniform over the entire disc, it apparently has some control over the distribution of the points.

This having failed, a plot of percent inertinite versus percent microcracks was made. As explained earlier, inertinite by virtue of it's porous nature would probably contribute to the oxidation/dehydration of the coal. If this
were the case, the amount of dehydration microcracks should increase with the amount of inertinite. This plot is shown in figure 17 and appears on the next page.
Figure 17. The porous nature of inertinite does seem to have some control on the degree of oxidation that a coal may be subjected to. The inertinite does not take part in the oxidation itself.
Upon examining figure 17 we note that maybe there does exist some relationship between the amounts of inertinite and the and microcracks. This is evident especially in the cases of D3 and D4 (X's and trees). D5, represented by the asterices, does not exhibit any relationship while D1 and D2 (triangles and boxes show some relationship though they are not quite as clearcut as in the case of D4 and D5. It should be noted that, the inertinite cannot contribute to the oxidation in any other way other than serving as a porous medium through which the oxidation can affect the coal easier. Also, it is known that inertinite is more brittle than either vitrinite or exinite and therefore it is only natural to have a greater number of cracks where ever one has inertinite.
SUMMARY AND CONCLUSIONS

Taking each of the tests separately I find that:

1) Though oxidation is evident from the presence of the microcracks, it does not change progressively with distance into the core as was expected.

2) The concentration of the pseudovitrinite seen also does not change with distance from the periphery of the core.

3) The reflectance of the vitrinite also does not exhibit the expected progressive change in it's value.

4) There apparently is no relationship between the microcracks and the reflectance.

5) There also is no relationship between the pseudovitrinite and the microcracks. It is now believed that pseudovitrinite is not evidence of post-coring oxidation, but rather is a product of early oxidation that probably took place during or soon after the peat stage. This is consistent with the findings of Benedict, Berry et al. (1968).

6) The extent of cleat fracturing does not appear to have any control on the distribution of the microcracks. The ratio of microcracks to 'L' does not show any relationship with position in the core.

7) The same observations made in 6) above cannot be made when 'L' is substituted by 'I', the amount of inertinite in the sample.
8) The proximity of a polished section to an outer surface does not appear to have any control on the degree of oxidation of that particular polished section.

9) Repolishing of the polished section to obtain a fresh unoxidized surface at the end of the study did not change the maceral content, reflectance or other petrographic characteristics of the coal in question.

10) Comparisons of the petrographic results obtained in this study and those from the NMERDI project that used only fresh coals, proved that the basic petrographic characteristics of the coal had not changed appreciably at all.

In conclusion therefore:-

Conditions of poor storage for coal cores do not seem to affect the petrography of the coal. The maceral content and reflectance essentially remain the same. The coal may therefore be evaluated quite accurately using petrographic techniques even after long periods of improper storage. It is important to note, however, that the burning properties of the coal will have changed markedly. Also of some importance to note is the fact that, inertinite inspite of it's own inert nature may contribute to the oxidation of the coal wherever present.
(64)

REFERENCES


APPENDIX I

This appendix contains the detailed hand specimen descriptions of all the coal seen in hole E-15. All classifications of bands and luster are according to Schopf (1950).
APPENDIX I

DETAILED DESCRIPTION OF COAL MACROLITHOTYPES

E 15

Interval - 301.00' - 319.00'

Sub-Interval - 301.00' - 301.75'

Luster - Intermediate

Bands - Medium to thick. Alternating

bands of vitrain and durain. Some resin present. Ash is principally CaCO₃.

Cleats - Irregular.

Sub-Interval - 301.75' - 303.20'

Luster - Intermediate bright.

Bands - Fine to thick. Alternating

bands of bright and dull vitrain.

Resin only present in small quantities.

Ash content decreasing.

Cleats - Very irregular.

Sub-Interval - 303.20' - 303.82'

Luster - Intermediate bright

Bands - Fine to thick. Again only

alternating bright and dull vitrain.

Resin and ash not present anymore.

Cleats - blocky.

Sub-Interval - 303.82' - 304.00'

Luster - Dull

Bands - Fine to medium. Alternating

durain and vitrain. Ash and resin now begin
to reappear.

Cleats - Irregular.

Sub-Interval- 304.00' - 304.90'

Luster - Intermediate
Bands - Fine to medium. Alternating vitrain and durain. Resin seen in a few places along cleats.
Cleats - Concoidal.

Sub-Interval- 304.90' - 305.60'

Luster - Intermediate bright
Bands - Medium to thick. Alternations of vitrain and durain. Some ash also present along laminations.
Cleats - Blocky.

Sub-Interval- 305.60' - 306.00'

Luster - Dull
Cleats - Irregular

Sub-Interval- 306.00' - 307.10'

Luster - Bright
Cleats - Concoidal.

Sub-Interval- 307.10' - 307.38'
Luster - Dull
Cleats - Irregular.

Sub-Interval - 307.38' - 309.05'
Luster - Intermediate bright.
Bands - Medium. Alternating inertinitic material and durain. Trace amounts of ash.
Cleats - Irregular to blocky.

Sub-Interval - 309.05' - 311.00'
Luster - Intermediate dull
Cleats - Blocky.

Sub-Interval - 311.00' - 311.80'
Luster - Intermediate
Cleats - Irregular.

Sub-Interval - 311.80' - 314.80'
Luster - Intermediate dull.
Cleats - Irregular to concoidal.

Sub-Interval - 314.80' - 315.90'
Luster - Intermediate

Bands - Medium. Alternating bright and dull vitrain. Some durain present. Trace amounts of ash.

Cleats - Irregular.

Sub-Interval - 315.90' - 319.00'
Luster - Intermediate bright

Bands - Medium. Alternating dull and bright vitrain. Resain present as irregular blebs.

No ash present.

Cleats - Irregular to blocky.

Sub-Interval - 319.00' - 319.40'
Carbonaceous Shale.

Sub-Interval - 319.40' - 320.40'
Luster - Intermediate dull.

Bands - Medium. Alternating vitrain and durain. Accessory amounts of ash.

Cleats - Blocky.

Sub-Interval - 320.40' - 322.12'
Shale

Sub-Interval - 322.12' - 323.00'
Luster - Intermediate

Bands - Medium. Alternating vitrain and durain. Resain present as blebs.

Cleats - Blocky.

Sub-Interval - 323.00' - 323.50'
Luster - Dull
Cleats - Irregular.

Sub-Interval: 323.50' - 324.05'
Luster - Intermediate
Bands - Medium. Alternating vitrain and durain. Thin strengers of claycy material seen
Cleats - Blocky.

Sub-Interval: 324.05' - 325.90'
Shale

Sub-Interval: 325.90' - 326.60'
Luster - Intermediate
Cleats - Blocky.
APPENDIX II

This appendix contains the results of the detailed maceral analysis that was performed on the twenty polished sections.
APPENDIX II

VITRINE GROUP

<table>
<thead>
<tr>
<th>SAMPLE NUMBER</th>
<th>VITRINE A</th>
<th>VITRINE B</th>
<th>VITRINE C</th>
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<td>50</td>
</tr>
<tr>
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<td>C</td>
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APPENDIX II (continued)

INERTINITE GROUP

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<th>FUSINITE</th>
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<th>MICRINITE</th>
<th>SCLEROTINITE</th>
<th>INERTODETRINITE</th>
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APPENDIX II (continued)

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### Appendix II (continued)

**Mineral Matter & Microcracks**

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APPENDIX III

This appendix contains the histograms of the reflectance data. One histogram was plotted up for each polished section off of which measurements were taken. Note that for the most part they exhibit normal distributions and are unimodal.
E15-12-C

Mean = 0.365399
Standard Dev. = 0.068399
Range = 0.273228

Reflectance Classes

reflectance classes
EIS-D1-0.

MEDN = 0.30589
STANDARD DEV. = 0.202466
RANGE = 0.30589

FREQUENCY

0.00 0.05 0.10 0.15 0.20 0.25 0.30 0.35 0.40 0.45 0.50 0.55 0.60 0.65 0.70 0.75 0.80 0.85 0.90 0.95 1.00

REFLECTANCE CLASSES
E15-DH-A

Mean = 0.034627
Standard Dev. = 0.20529
Range = 0.420670

Reflectance Classes

0.00 0.05 0.10 0.15 0.20 0.25 0.30 0.35 0.40 0.45 0.50 0.55 0.60 0.65 0.70 0.75 0.80 0.85 0.90 1.00
E15-D4-C.

Mean = 0.283629
Standard Dev. = 0.130679
Range = 0.543214

Reflectance Classes

0.00 0.05 0.10 0.15 0.20 0.25 0.30 0.35 0.40 0.45 0.50 0.55 0.60 0.65 0.70 0.75 0.80 0.85 0.90 0.95 1.00
Histogram showing frequency distribution of reflectance classes.

- **Mean**: 0.32373
- **Standard Dev.**: 0.267528
- **Range**: 0.244525

Reflectance Classes:

- 0.00
- 0.05
- 0.10
- 0.15
- 0.20
- 0.25
- 0.30
- 0.35
- 0.40
- 0.45
- 0.50
- 0.55
- 0.60
- 0.65
- 0.70
- 0.75
- 0.80
- 0.85
- 0.90
- 0.95
- 1.00
EIS-DS-A1

Mean = 0.330256
Standard Dev. = 0.085167
Range = 0.325763

Frequency Classes:
0.00 0.25 0.50 0.75 1.00
E15-05-0.

MEAN = 0.215041

STANDARD DEV. = 0.106490

RANGE = 0.202500

FREQUENCY CLASSES

0.00 0.05 0.10 0.15 0.20 0.25 0.30 0.35 0.40 0.45 0.50 0.55 0.60 0.65 0.70 0.75 0.80 0.85 0.90 0.95 1.00