

AN ABSTRACT OF THE THESIS OF

Thomas Szymoniak for the degree of Master of Science in Civil Engineering presented on October 16th, 1986.

Title: Development of A Standard Accelerated Weathering Test for Aggregates Using Dimethyl Sulfoxide (DMSO)

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Some basaltic rocks common in Oregon when incorporated into a pavement structure as the base course aggregate or in the surfacing layer degrade and disintegrate owing to the minerals contained within the rock and the environment in which it is placed. To simulate the chemical degradation of basaltic rocks an accelerated weathering test using Dimethyl Sulfoxide (DMSO) was developed. The objective of the research reported herein was to: 1) study the interaction of DMSO with clay standards, 2) develop a reliable method of determining clay type and content in basalt rocks, 3) study the parameters influencing the current test and develop a standard procedure, and 4) determine acceptable weight loss limits for untreated basalt aggregates after immersion in DMSO.

By investigating the electro-static interaction between clay standards and DMSO it was found that the DMSO molecule has a preference for cations and it is able to donate hydrogen ions. Both factors allow DMSO to penetrate into a rock matrix seeking out the cations held either to clay minerals or zeolites. After a thorough study of the

parameters influencing the current procedure, such as container geometry, aggregate particle size, sample weight, and immersion time, a standard test was developed. The recommended procedure consisted of immersing a 1000g sample of aggregate in the size range between 2.38 mm (#8) and 4.76 mm (#4) for a period of five (5) days.

At the end of the immersion time the aggregate is re-screened over the 2.38 (#8) sieve and the percent weight loss is calculated. The acceptable weight loss limit for the DMSO Accelerated Weathering Test was established by correlating the test results to those obtained through petrographic analysis. The petrographic analysis consisted of determining the percentages of deleterious secondary minerals and their textural distribution within the rock matrix. With these two parameters a Secondary Mineral Rating for each quarry was calculated and compared to the DMSO test results. In addition, it was found that DMSO may overreact if the minerals analcime and calcite are present in the quarry rock. A second indirect test, the Clay Index, was presented as a simple method to determine the likelihood of DMSO to overreact, since it was found to be insensitive to the type of minerals present in the rock.

Development of a Standard Accelerated Weathering Test
for Aggregates Using Dimethyl Sulfoxide (DMSO)

by

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PREFACE

The thesis presented in the following pages is a compilation of three articles written for separate publication. Chapters 2 through 4 were written to stand alone, so some repetition may be noted. Citations in the text refer to references at the end of each chapter. A comprehensive bibliography is given at the end of the text in which all of the cited references are collected. In chapters 3, Jim Wilson and Neal Walker are listed as co-authors because of their help in creating and sharing their experiences with aggregate degradation tests. Dr. Vinson served as my consultant, guide and most importantly, as an editor in preparing the manuscripts.

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DEVELOPMENT OF A STANDARD ACCELERATED WEATHERING TEST FOR AGGREGATES USING DIMETHYL SULFOXIDE (DMSO)

1.0 INTRODUCTION

Currently large sums of money are being spent for reconstruction and improvement of roads, streets and highways throughout the United States. This work has lead to a depletion of good quality durable aggregates and the increased use of marginal aggregates. Marginal aggregates can perform satisfactorily in service, but the physical properties of the rock and the environment in which it is placed must be clearly understood. The physical properties of a rock are typically assessed through standard laboratory tests in which the aggregate produced from a quarry must meet the criteria established by the governing agency. The laboratory tests currently used by the Oregon Department of Transportation (ODOT) tend to reflect the mechanical durability of a rock, which results in the actual physical breakdown, generally associated with the action of equipment during construction and the operation of traffic throughout the life of the pavement. However, chemical weathering of a rock, caused by the environment in which the aggregate is placed and resulting in the disintegration of mineral components of the rock, is not reflected by these tests. In fact, many types of basaltic rock in Oregon generally have good mechanical durability properties and will usually pass the standard aggregate durability specifications. Field experience indicates that aggregates produced from these basalt rock sources degrade owing to the chemical weathering of the minerals present in the rock matrix. Specifically, the basalts contain swelling clays,

such as smectites, which in the presence of water expand and lead to the eventual failure and breakdown of the aggregate.

As a result of the inability of the standard tests to predict the durability of basalt aggregates, the Materials Section of the Federal Highway Administration (FHWA) Western Direct Federal District (WDFD) developed an immersion test in Dimethyl Sulfoxide (DMSO) to assess the chemical degradation potential of an aggregate source. The applicability of the DMSO test to predict the degradation characteristics of a rock and the meaning of the index parameter determined is not well understood. In recognition of this situation, a research study was undertaken and reported herein.

Chapter 2 reviews the formation of clay minerals in basaltic rock and correlates the clay mineral type and content determined from petrographic techniques to those obtained from indirect test such as the DMSO Accelerated Weathering Test. Chapter 3 reports the results of a study of the parameters affecting the DMSO procedure used by the FHWA and presents a standard testing procedure. Chapter 4 incorporates the results discussed in the preceding two chapters and presents acceptable weight losses for the DMSO Accelerated Weathering test. Chapter 4 also presents guidelines to determine if the DMSO solution has overreacted with certain minerals contained within the rock matrix. Where possible, the results obtained in the study are compared to those reported by others in the literature.

2.0 DETERMINING CLAY MINERALS IN BASALT AGGREGATES

by Tom Szymoniak¹, A.M. ASCE and Ted S. Vinson, M. ASCE

ABSTRACT: Eight basaltic rock quarries were sampled to compare clay type and content determined by direct methods to those obtained from indirect methods. The direct methods consisted of petrographic and X-ray diffraction analyses of representative samples of rock taken from the quarries. The petrographic analysis was used to determine the clay content; the type of clay mineral was identified by X-ray diffraction. Three indirect methods were employed to compare clay type and content: 1) the DMSO Accelerated Weathering Test, 2) the Clay Index Test, and 3) Atterberg Limits. Based on the test results, the DMSO Accelerated Weathering Test and the Clay Index test were found to be valid indicators of clay content in basalt aggregates. Correlation equations to predict the clay content given the results from these tests are presented. The weight loss for aggregates immersed in DMSO was also noted to be affected to a great degree by the type and distribution of certain minerals contained within the rock matrix. The Clay Index Test results were not found to be affected by minerals type and distribution, but were influenced by the initial pulverized aggregate particle size employed in the test. The third indirect test, Atterberg Limits, did not reflect clay content owing to the inability to pulverize the aggregate to sizes smaller than the clay size.

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2.1 INTRODUCTION

The continued use of aggregates in the construction of roads, buildings, and other civil works projects has lead to a depletion of high quality materials in many areas of the United States. This has resulted in the need to utilize marginal aggregate sources, which require more detailed and intensive study prior to their usage since aggregate performance is directly related to the quality of the parent rock. Through an understanding of the rock properties that lead to the deterioration of aggregates, steps can be taken during the design phase to reduce the likelihood or eliminate post construction failures, thereby saving the cost of importing high quality aggregates and substantially reducing the long term cost of a civil works project.

One attribute of roadway aggregates that has been found to be detrimental to pavement structure performance is swelling clays contained within the rock matrix. Specifically, swelling clays have been identified as being deleterious since in the presence of water they expand, causing the rock to disintegrate, and ultimately failing the pavement structure. Swelling clays are typically found as weathering products of igneous rocks, which are one of the most common rock types utilized in the Pacific Northwest. In fact, basalts (such as marine basalt) generally have good mechanical durability properties and will usually pass standard aggregate specifications. However, when marine basalts are incorporated into a pavement structure as the base course aggregate or in the asphaltic concrete mix, field experience suggests that the rocks degrade due to the presence of the swelling clays.

Objective and Scope of Work

In recognition of the need to determine clay mineralogy and content in basalt aggregate sources, a research study was undertaken to evaluate the direct and indirect methods used to determine clay mineral type and content in aggregates. The study consisted of field sampling eight quarries and determining the clay mineral type and content through the use of petrographic and X-ray diffraction analyses, the DMSO Accelerated Weathering Test, the Clay Index Test and Atterberg Limits performed on pulverized rock samples. The results from this work are reported herein.

2.2 ROLE OF CLAY MINERALS ON AGGREGATE PERFORMANCE

Background. — Direct and indirect methods may be employed to determine clay mineral type and content. Direct methods consist of: 1) performing a petrographic analysis on stained rock thin sections to determine the clay content, and 2) employing X-ray diffraction methods on pulverized samples of the aggregate to identify the type of clay mineral. Indirect methods include: 1) accelerated weathering tests, such as immersion of the aggregates in Dimethyl Sulfoxide (DMSO) or ethylene glycol, 2) determination of the Atterberg Limits and, 3) determination of the Clay Index on pulverized aggregate samples. Indirect methods, however, must be correlated to direct methods and, therefore, it is appropriate to establish a reliable direct method in which the clay content and distribution can be determined. Before continuing with a discussion of the methods that are employed it is important to understand the mineralogy of igneous rocks, and the resulting formation of swelling clays due to chemical weathering.

Formation of Clays From Igneous Rocks. -- Igneous rocks are formed from the solidification of molten material in the Earth's crust. The minerals which are formed are dependent upon the composition of the original magma and the conditions of crystallization. Bowen (25) found that the crystallization of minerals followed two series, namely, a discontinuous ferromagnesian series and a continuous feldspar series. Bowen's reaction series and the resulting classification of igneous rocks is illustrated in Figure 2.1. The left branch of the figure represents the discontinuous ferromagnesian series. The minerals on this branch are dark in color and are commonly referred to as mafic. The right branch of the figure is the continuous feldspar series. The minerals are light-colored and often referred to as felsic. The classification of basalts, as shown in the figure, is a function of crystal size and the type of minerals present at the time crystallization occurs.

The felsic plutonic igneous rocks, such as granites, are coarsely crystalline due to slow cooling at great depths. Owing to the chemical composition at the time of cooling these rocks tend to be highly resistant to chemical weathering. Generally speaking felsic rocks have lower mechanical toughness compared to rocks cooled quickly and are more susceptible to mechanical degradation (i.e., physical breakdown of the rock occurring through the action of equipment during construction or the operation of traffic throughout the life of the pavement structure). The durability of felsic rocks is reflected

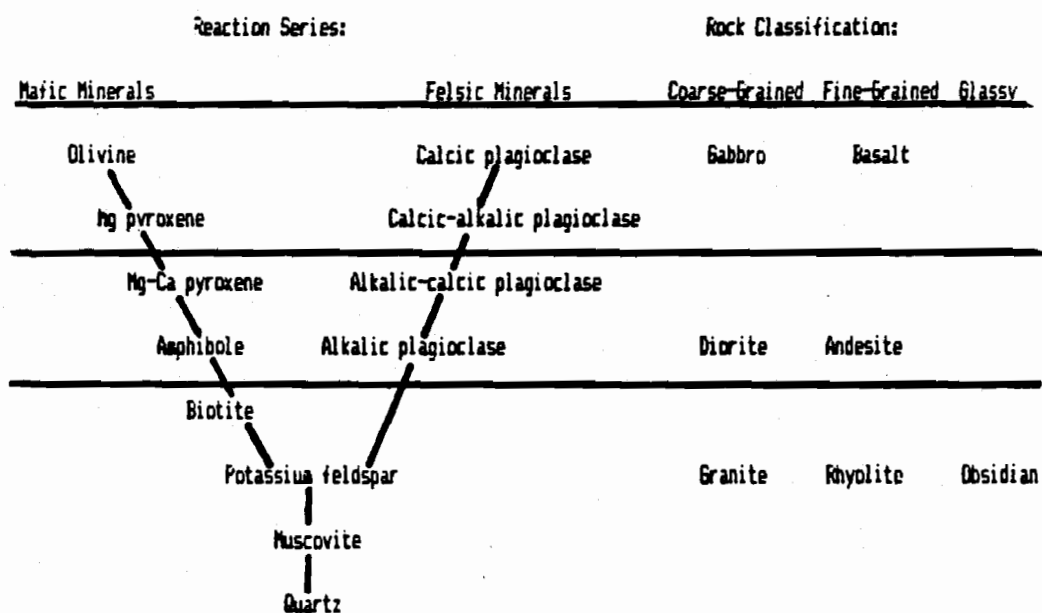


Figure 2.1. - Bowen's Reaction Series and the Classification of Igneous Rocks.

to a high degree by the results of mechanical degradation tests such as the Los Angeles Abrasion Test and the Washington Degradation Test.

If however, the rate of cooling is rapid and the magma is near the beginning of Bowen's reaction series, small crystal growth will occur and the minerals formed will be greatly affected by chemical weathering processes. Basalts are typical of this formation process. The mechanical durability for this type of rock is excellent due to good interlocking between the small angular crystals, but because of the mafic minerals present in the matrix, basalt aggregates may disintegrate in a short period of time due to chemical degradation.

The constituents that are found in igneous rocks can be divided into four groups: primary minerals, glass, deuteric, and secondary minerals. Primary minerals are the original constituents of a rock, which crystallize from the magma. Some of the primary minerals found in basalt include plagioclase, pyroxene (augite), amphibole (hornblende), and magnetite (20).

The second group is glass, which is formed from rapid cooling, or quenching of the molten rock. It has little internal ionic structure and varies widely in chemical composition (6). Glass tends to be highly susceptible to chemical weathering due to the lack of internal structure.

The other two groups, deuteric and secondary, are associated with the alteration of the primary minerals and glass. Deuteric alteration occurs during the crystallization process and results from the chemical reaction of hot water or gases with the minerals.

Secondary minerals are formed through the weathering of minerals under temperature, pressure, and/or chemical conditions at or near the earth's surface. Some secondary minerals that can be found in basalts include iron oxides, zeolites, and calcite (20).

Clay minerals are formed by deuteric alteration and chemical weathering of the primary minerals and glass. These processes result in the formation of the following types of minerals:

- 1) Smectite - a group of clay minerals produced through the alteration of plagioclase; these minerals are fine-grained, possess high swelling properties, and have high ion exchange capacities.
- 2) Iddingsite - formed by the deutric alteration of olivine; composed predominately of an interstratified layers of smectite and chlorite.
- 3) Palagonite - an alteration product caused by the oxidation and/or devitrification of basaltic glass; it can further alter to a smectite clay.

The alteration products which are present in basalt rock are a function of several factors. First, the environment that existed during the formation of the rock (i.e. the rate of cooling, temperature, and the amount of moisture) influences the later sensitivity of the rock to weather (6). The entrapment of volatiles at high temperatures for sustained periods of time can cause alteration of the interior mass and, hence, the inner rock mass may have a lower durability and a greater amount of alteration products. Second, the presence (or absence) of alteration products is related to chemical

weathering. Chemical weathering is typically limited to the external surface of a rock, where it is exposed to groundwater, and at the contact zone between different basalt flow units. Voids, contraction joints, and vesicles are areas which are most susceptible to chemical weathering.

Historical Prospective of the Problem. -- Failure of pavement structures as a result of the degradation of the aggregates used in construction has been a problem in highway engineering for many years. In 1916 Lord (11) prepared a report on the effects of mineral composition on the physical properties of rock. He compared 83 samples of mafic crystalline rock which did not slake in water with 48 samples which slaked. The non-slaking rocks contained 89 percent primary minerals, whereas those that slaked contained only 69 percent primary minerals. In 1955 Scott (13) determined that 0 to 20 percent secondary minerals in a fine-grained aggregate will have little effect on pavement performance; 20 to 35 percent will produce some failures and borderline performance; and above 35 percent will almost certainly produce failures. In 1974 Van Atta and Ludowise (20) concluded that the durability of aggregates could not be determined solely by the percentages of weathering products found in the rocks. The durability was, however, related to certain combinations of secondary minerals present in the rock and the textural distribution of the swelling clays in the rock matrix. They also noted that an aggregate with a "stained" smectite clay content greater than 10 percent would result in poor pavement performance. Higgs (9) in an independent study conducted during 1976 found that as little as 11

percent of discrete montmorillonite was sufficient to cause basalts to degrade.

Higgs (9) also suggested that the quality of a rock could be further assessed by noting the stage in the weathering of volcanic glass. In his study, Higgs found that volcanic glass goes through four stages as it weathers into a montmorillonite clay. In the first stage the rock contains clear unaltered glass; palagonite is formed in the second stage due to the release of iron oxides; the third stage results in devitrification to montmorillonite and the palagonite develops a scaly spherulitic appearance; and the fourth stage leads to the formation of distinct discrete montmorillonite. He further states that concern for aggregate slaking begins in the third stage with the development of montmorillonite.

The different stages of weathering in aggregates and the presence or absence of clay minerals can best be assessed through the use of thin sections of rock. The results obtained are affected by the rock sample from which the thin section was cut, which may or may not reflect the aggregate produced from a given quarry. In this respect, it is imperative that petrographic analyses be conducted on representative rock samples taken from the quarry. Durability tests, however, employ larger sample sizes and provide for a better representation of the aggregate produced. Durability tests, like the DMSO Accelerated Weathering Test have been suggested as an indirect method to estimate the quantity of swelling clays present in the rock.

Development of Accelerated Weathering Tests. -- Standard durability tests using mechanical methods, such as the Los Angeles Abrasion Test (ASTM C131) and the Sodium Sulfate Soundness Test (ASTM C88), were developed in the early 1900's and have become a part of most state highway department's standards for aggregate specifications. West, et al (24) presented an excellent review of durability tests employed to evaluate the degradation of base course aggregates. In their research effort, 140 separate quarries, covering 12 states and representing the three major rock types, were sampled and tested. Determinations were made for 1) percent loss by the Los Angeles Abrasion Test, 2) freeze-thaw loss, 3) specific gravity, and 4) petrographic analysis including measurement of grain size, roundness, and interlock. As a result of the data collected, West et al (24) recommended that aggregates be divided into three groups, each having their own specific testing program. The groups are: 1) carbonates, clastic sedimentaries, and coarse-grained igneous and metamorphic rocks; 2) basalt and basalt like aggregates; and 3) heterogeneous gravels. It was noted that petrographic analysis, in which deleterious materials such as volcanic glass and palagonite are determined, was the most reliable method to predict the potential of basalt to chemically degrade. It was further noted that for basalts there was no clear relationship between the Los Angeles Abrasion loss and field performance, and the results for the Sodium Sulfate Soundness test were not reproducible to a high degree of confidence.

In recognition of the inability of standard durability tests to reflect field performance for basalt rocks, several agencies in the

Pacific Northwest developed accelerated weathering tests to simulate the chemical degradation of an aggregate. In the late 1960's the Army Corps of Engineers proposed the use of an ethylene glycol degradation test to assess the quality of a basaltic rock. Ethylene glycol has been used in X-ray diffraction analysis as a tool in the identification of swelling clays, since it causes the expansion and swelling of certain clay minerals. The X-ray diffraction pattern of swelling clays increases from the normal basal spacing of approximately 14A to 17A after treatment in a saturated ethylene glycol environment (16). The swelling pressure created by the expansion of the clay structure in the rock matrix results in the deterioration and breakdown of the aggregate.

The original procedure proposed by the Corps used a ten rock sample with aggregate in the size range of 9.5mm (3/8in.) to 25.4mm (1in.). The rocks were selected from a representative sample of the aggregate source and were soaked in ethylene glycol for a period of 10 days, at which time the number of rocks observed to crack, spall, or disintegrate were recorded (23). This value was reported as an index value to relate the quality of the aggregate source; if four or more rocks cracked, spalled, or disintegrated the quarry was deemed unsuitable.

In the mid-1970's this test was modified by the Federal Highway Administration (FHWA) Region 10 Materials Section by substituting DMSO for ethylene glycol. The ten rock procedure was the same up until 1978, when a further modification was adopted. The new procedure was made similar to the Sodium Sulfate Soundness Test in that a

weighted loss was reported for the aggregate sample. Szymoniak, et al (17) found the weighted loss reported by the 1978 procedure was affected by many factors including the aggregate particle size, sample size, and immersion time. They recommended a standard DMSO test procedure using 1000g of aggregate in the size range of 4.8mm (#4) to 2.4mm(#8), and immersed in a 100 percent solution of DMSO for a period of 5 days. Considering the weight loss results from the DMSO Accelerated Weathering test for eight quarries with respect to aggregate performance criteria established through other means, Szymoniak et al(18) concluded that for crushed base aggregates the acceptable loss limit should be 22 percent.

Two other accelerated weathering tests were proposed in 1982 by Fielding and Maccarrone (7). The two methods involve either five cycles of boiling in a reflux apparatus for a total of 40 hours using ethylene glycol, or pressurization for three hours at 232°C and 1300 kPa in a pressure vessel using an aqueous solution of 80 percent (by volume) of ethylene glycol. One cycle in the boiling method consisted of heating the samples to 197 °C during the day and then allowing the samples to cool overnight.

Fielding and Maccarrone termed the result of the test the Accelerated Soundness Index. The index is obtained at the conclusion of the test, and indicates the percentage of the total mass retained on a 6.7mm sieve. Through a comparison of the test results to secondary mineral content, total surface area and field performance, sound rock had an index value greater than 94, and unsound rock had values less than 90.

Close examination of the Accelerated Soundness Index procedure by Cole et al (4) established that the difference in clay content between sound and unsound rock was 1 percent. They concluded that the limits established for the test to assess rock quality would be more stringent than those based on secondary mineral contents and, therefore, concluded that the test was too severe.

2.3 METHODS OF STUDY

Two tasks were undertaken to investigate the methods employed to establish clay mineral type and content in roadway aggregates. The first task dealt with determining the clay mineral type and content by direct methods for eight basalt quarries in Oregon. The investigation included determination of the percentage of primary, secondary and clay minerals in thin sections of rock, and identification of the type of clay through the use of X-ray diffraction analysis. The second task was to correlate the indirect methods (e.g., DMSO Accelerated Weathering Test, Atterberg Limits, and the Clay Index test) to the direct methods, and to make an assessment of the suitability of the indirect methods to establish clay mineral type and content. The location of the eight quarries selected for the study are shown in Figure 2.2. The remainder of the paper discusses the direct and indirect methods employed to determine the clay mineral type and content for the aggregates considered, and presents the data collected to correlate these two methods.

Direct Methods

Petrographic Analysis. -- Petrographic analysis refers to the description

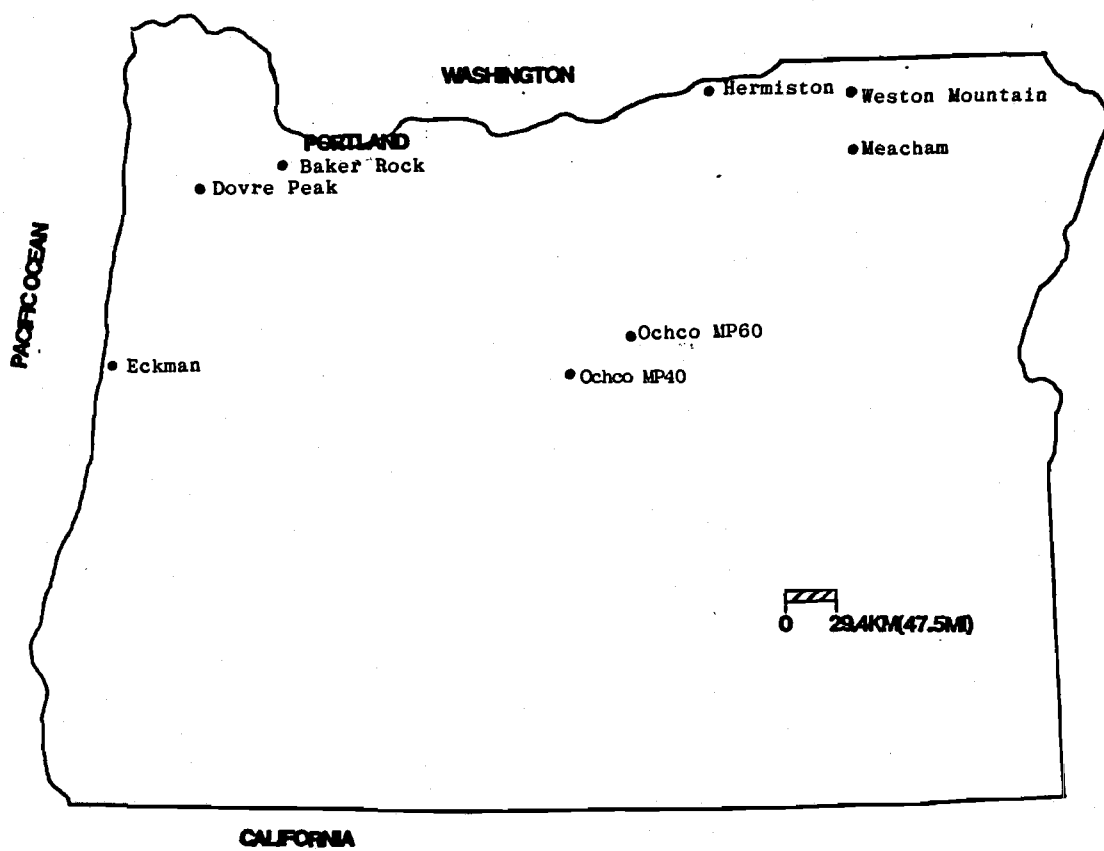


Figure 2.2. - Location Map of Quarries Sampled.

and classification of rocks. The analysis can be performed by megascopic, or microscopic examination. Under megascopic examination the gross mineralogy and weathering aspects are determined. Megascopic examination was used in this study to evaluate the fracturing and weathering occurring at the quarry.

Microscopic examination was conducted on thin sections cut from the parent rock. Thin sections are thinly ground rock slices (about 0.03mm thick) that are mounted on glass slides. Light transmitted through the thin sections placed under a polarizing microscope allows for a complete identification of the rock mineralogy, and measurement of the rock texture. Chayes (2) conducted a statistical analysis on thin section compositions. He showed that mineral composition can be determined by the point counting method and that for igneous rocks the mineral constituent is directly related to the crystal grain diameter in the thin section.

The point counting method is an extension of the relationship between mineral type and grain diameter. It is a means of ascertaining the areal proportions of constituent minerals in a rock by superimposing a grid on a planar section through the rock. If the fabric is isotropic, a representative planar section will yield a valid indication of the volumetric proportions of minerals in the rock (1).

The pointing counting procedure utilized for this study is as follows:

- 1) place the thin section under a polarizing microscope, equipped with a counting stage to move the slide at regular intervals

across an imaginary grid;

- 2) adjust the grid size to correspond to the average grain size of the rock;
- 3) identify the mineral appearing directly below the crosshairs of the microscope;
- 4) tally the identification as one point, and maintain a separate count for each different mineral;
- 5) determine the percentage of each mineral in the rock by dividing the point count for this mineral by the total number of points counted.

The number of points counted in the modal analysis is a function of the abundance of the minerals of interest and of the accuracy desired. In this study, it was established that the desired accuracy would be achieved if over 500 points were counted in the thin sections.

The point counting method by itself is not sufficient to determine the clay mineralogy, and the amount or distribution of the clays in thin section. The clay mineral alone is very small and is not easily visible under an ordinary microscopes. To aid in the determination of the clay content and distribution in thin section staining techniques are conducted to augment the modal analysis. In addition, it is often necessary to conduct X-ray diffraction analysis on powdered aggregates to determine the type of clay.

Staining techniques were first applied to determine clay mineralogy in 1871 by Behrens (8). The reactions between organic compounds and clay minerals often produce a color change of the mineral. The color changes vary depending upon the type of clay mineral and its composition.

A staining test has the advantage of being simple and rapid to perform, and when coupled with the point counting method a percentage clay content may be determined. Szymoniak et al (19) recommended staining thin sections of rock with rhodamine-b dye. They concluded that the reaction between rhodamine-b and clay minerals was easily distinguishable from other constituents in the thin section, and that the stain was specific for smectite clays.

X-ray Diffraction Analysis. — X-ray diffraction analyses were conducted to aid in the identification of clay minerals found in the thin sections. The samples were prepared by soaking the minus 0.074mm (#200) pulverized aggregate in distilled water for 24 hours. Silts and sands were first separated from the sample by centrifuging for 5 minutes at 750 rpm. The suspension was then centrifuged again for 10 minutes at 6,000 rpm to remove the clays from suspension. After the clays had settled, the clear supernate was poured off, and the clay material was smeared on glass slides. The samples were allowed to air dry, and then were placed in either an ethylene glycol or 54% relative humidity dessicator and allowed to equilibrate to that environment. Identification of the clays followed the standard procedures established by the U.S. Geologic Survey (16).

Cole et al (5) have proposed a method to determine clay contents in basalt aggregates using the peak height for smectite clay taken from the X-ray diffraction analysis. The diffraction analysis is performed on powdered aggregate samples without separating the clays. Cole et al (5) found that the peak heights from the X-ray diffraction pattern for the air dried samples correlated to a high degree to the

clay contents determined from petrographic analysis.

There are several disadvantages in using X-ray diffraction patterns for quantitative estimates. One, the method assumes only one type of clay mineral is present in the basalt rock. The clay mineral is assumed to be a smectite, and the peak height is obtained from the diffraction pattern since it has a characteristic strong peak centered at 5-6° of 14A (CuK radiation) for a sample in the air dried state(4). If two type of clays are interstratified in the rock, the peak intensity for smectite may be masked by the peak for the other mineral. Johns et al (10) employed ethylene glycol and heat treatments to correct for interstratified clays, and they also noted that X-ray patterns smoothed after the treatments allowing for easier identification of peak heights. Second, the X-ray diffraction procedure must be standardized such that the powdered samples are prepared in the same manner. Poor sample preparation may result in reducing the orientation of the crystals which would reduce the peak height. Starkey et al (16) noted that poor crystallinity and weathering may also cause the peaks to broaden or reduce the intensity. Third, X-ray spectrometer equipment is not readily available for use by most agencies in the Pacific Northwest and in this respect the use of the peak heights for determination of clay contents would be impractical. Although the X-ray diffraction analysis is simple to perform and correlates well to direct methods, it was not adopted for this study due the disadvantages noted.

The direct methods, petrographic analysis and X-ray diffraction, require experienced personnel and equipment, and are the most accurate

means in assessing the clay mineral type and content of an aggregate source. Indirect methods can also be employed to determine the clay contents in order to save time, and limit petrographic analysis to borderline sources. One must, however, understand the limitations of the indirect tests and the comparison of the results obtained to the direct methods.

Indirect Test Methods

Three indirect methods were used to assess the clay contents for the quarries studied. The first indirect method performed was the DMSO Accelerated Weathering test using the procedure recommended by Szymoniak et al (17), previously noted. The second method was the determination of the Atterberg Limits (ASTM D4318), and the third method utilized was the Clay Index test.

Atterberg Limits. -- The Atterberg Limits, liquid and plastic limit, were obtained using pulverized aggregate passing the .074mm (#200) size. The purpose in performing the Atterberg Limits was to obtain the liquid limit of the powdered aggregate which could be related to the type and content of clay minerals present in the sample. However, it is well known that the relationship between the liquid limit and clay content is a function of the type of clay and its activity. The activity is computed by dividing the plasticity index by the percent by weight finer than .002 mm. Swelling clays typically have a high plasticity index and high activities. Thus, if it is assumed that only swelling clays are present in the pulverized aggregate, the liquid limits can be compared to clay contents. The liquid limits for the pulverized aggregate were compared to the

results obtained by Seed et al (14), who studied the relationship between clay content and liquid limit using different clay standards.

Clay Index. -- The Clay Index Test, is a methylene-blue titration procedure which results in the determination of the Clay Index for the pulverized aggregate. Both the clay mineral type and content influence the index value. Swelling clays tend to absorb more methylene blue and as such will be represented by the higher index values. Sameshima and Black (12) defined the Clay Index as the number of milliliters of 4.5gm/l methylene-blue solution absorbed by 1 gm of material passing the .074mm (#200) sieve. The index test was meant to be a simple method of assessing the quality and performance of an aggregate source.

The original procedure suggested by Sameshima and Black (12) called for the addition of hydrogen peroxide and sulfuric acid to the minus .074mm (#200) sample prior to the titration with methylene blue. The hydrogen peroxide was added to neutralize any organics and the addition of sulfuric acid replaces the exchangeable ions held on the clay surface with hydrogen ions to facilitate the absorption of methylene blue. The procedure as used in this study was modified by eliminating both chemical treatments since organics were not anticipated and the acid treatment destroys the structure of nontronite, an iron rich smectite clay (22). The endpoint of the titration, which is the value reported for the Clay Index, is obtained by placing a drop of the powdered aggregate-water sample on Whatman No. 1 filter paper. When the suspension is near the endpoint a light blue corona will appear around the mud grains; the mudgrains are stained dark blue.

The endpoint is reached, when after shaking the sample for one minute the blue halo does not disappear. A more complete description of the test procedure is given by Sameshima and Black (12).

2.4 DISCUSSION OF RESULTS

Table 2.1 presents the results of the petrographic analyses conducted for the eight quarries sampled for the study. Of the quarries sampled, five were selected for further testing using the standard durability tests employed by the Oregon Department of Transportation (ODOT). The results from the durability testing (Sodium Sulfate Soundness, Oregon Aggregate Degradation, Los Angeles Abrasion, and the DMSO Accelerated Weathering Test) are given in Table 2.2.

Geology of Quarries and Results of the Direct Methods Employed

Baker Rock Quarry. — The Baker Rock Quarry was divided into three separate units, each having different geologic characteristics. The first unit was the upper bench (B1), the second unit was the lower bench (B2), which is now a mid bench since a third unit (B3) was opened during the summer of 1985.

The rock in the upper bench is moderately to highly fractured, breaking into blocks ranging from fist size to the size of large boulders. The section sampled consisted of two flow units, each about 4.8m (15 ft.) thick, separated by a 30 to 90 mm (3 to 6 in.) layer of red paleosol. The rock has undergone considerable weathering as evidenced by the presence of limonite infilling around the vesicles,

Table 2.1 - Petrographic Analyses for Quarries Samples.

Quarry (1)	Plagioclase (2)	Olivine (3)	Pyroxene (4)	Magnetite/ Ilmenite (5)	Glass (6)	Smectite Iron Clay (7)	Oxides (8)	Sepiolite (9)
Baker Rock								
Unit #1 (B1)	42	3	-	8	1	31	2	13
Unit #2 (B2)	37	5	3	10	-	31	4	10
(unoxidized)								
Unit #2 (B2)	52	1	19	7	-	3	18	-
(oxidized)								
Unit #3 (B3)	38	1	3	7	-	28	12	11
Meacham (M)								
Quarry	36	-	15	11	-	21	18	-
Ochco MP40 (M4)								
Quarry	55	-	14	6	5	20	-	-
Ochco MP60 (M6)								
Mid-Top	54	-	9	7	-	30	-	-
* Dovre Peak (D)								
Upper Unit	39	-	22	4	6	3	25	-
Lower Unit	43	-	23	4	4	7	19	-
Crusher	18	6	17	9	-	39	11	-
Weston Mtn. (W)								
Quarry	36	-	19	11	-	21	17	-
**Eckman (E)								
Quarry	56	10	-	4	-	30	-	-
Hermiston (H)								
Quarry	41	5	4	11	-	12	25	2

* Reference 20

** Reference 3

Table 2.2 - Durability Summary for Quarries Utilized in Study.

QUARRY	Sodium Sulfate		Oregon Aggregate		Los Angeles	DMSO
	Soundness		Degradation		Abrasion	Accelerated
	% Loss		Sediment	% Passing		Weathering
	Coarse	Fine	ht.	0.84mm (#20)	%	%
	Aggregate	Aggregate	cm	Sieve	Wear	Loss
(1)	(2)	(3)	(4)	(5)	(6)	(7)
Baker Rock						
Upper Bench(B1)						
Random	15.1	13.5	8.6	16.4	18.5	49.0
Vesicular	9.9	23.4	7.4	21.1	21.6	20.9
Olivine	4.6	15.9	3.3	15.4	20.8	12.9
Lower Bench(B2)	4.8	13.8	2.5	16.6	17.6	17.7
Meacham(M)						
Quarry	1.7	7.7	-	-	-	8.6
Ochoco MP60(M6)						
Westside	9.2	6.0	8.6	18.7	15.8	18.7
Mid-Top	4.6	9.9	2.8	13.5	16.4	49.3
Ochoco MP40(M4)						
Quarry	3.8	10.8	3.1	15.7	20.8	16.4
Dovre Peak(D)						
Quarry	23.1	24.1	-	-	-	77.0

and the manganese oxide coating. Due to the variability of the rock in the upper bench the samples were split into three groups; random, olivine, and vesicular. In general, the rock in the upper bench had the highest clay content and the lowest durability of the quarries selected for the study.

The primary mineral content for the rocks in the upper bench ranged from 54 to 56 percent, and the secondary mineral content ranged from 44 to 48 percent. The smectite clay content varied between 26 to 38 percent, with the higher values being typical of the vesicular rocks. The size of the crystals, as measured by the groundmass size, ranged from 2-3mm, which indicates that rocks in the upper bench are porous. X-ray diffraction analysis identified only nontronite, an iron rich smectite clay.

The rock in the lower bench (B2) was unfractured, uniformly dense, and very fine-grained. Megascopic analysis at the time the quarry was sampled revealed two distinct weathering patterns, one unoxidized and the other oxidized. The oxidized section was exposed to local groundwater and, hence, some of the zones were fresh with little alteration and low clay contents. The primary and secondary mineral contents in this bench are 68 percent and 29 percent, respectively. Although the smectite clay content is low in the oxidized section (only 3 percent) the percentage of iron oxides from the weathering process are higher than those in the other sections of the quarry. The unoxidized sections have clay contents which are very similar to that of the upper bench. The results from the durability tests reflect the mixture of those two dissimilar materials.

The rock in the third unit (B3) is part of another flow, different from those in the other benches. It has also undergone some weathering due to the appearance of the manganese oxide coating the rock. The primary and secondary mineral contents for rock in the third unit are 48 percent and 52 percent, respectively. Thin section analysis showed a high percent of iron oxides and smectite clays comprising the secondary mineral content. Although the rock from this unit showed the highest degree of weathering, the results from the DMSO Accelerated Weathering test were low. The low results can be attributed to the small size of the groundmass and the lower permeability of the aggregate.

Meacham Quarry. -- Rock from the Meacham Quarry is highly fractured and jointed into crude columns, which tend to break into small blocks ranging from 25 to 120mm (1 to 4in). The fractured rock is very fine-grained and dense. Thin section analysis showed the rock to be comprised of 62 percent primary minerals and 38 percent secondary minerals and the size of the groundmass was in the range of 0.05 to 0.1mm (0.002 to 0.004in). X-ray diffraction identified an interstratified clay mixture of smectite and chlorite.

The deleterious, secondary mineral palagonite was identified in thin section. Its percentage was included in the iron oxide count, since it was difficult to distinguish between the two. The palagonite was in the third stage of weathering to a smectite clay, which accounts for the high clay content. Although rock from Meacham had a high clay content, it was the most durable of those tested using the standard mechanical tests, and had the lowest weight loss after

immersion in DMSO.

Ochco Highway Quarries- Milepost 40 and 60. -- The exposed rock for the Milepost 40 (M4) quarry is a fractured basalt with a dark gray to black color. The quarry face is approximately 15m (50 ft) thick, but fresh rock is only well exposed in the upper third. The rock is fine-grained, and many fractures are filled with chalcedony, a micro-crystalline variety of quartz. Minor amounts of calcite and iron oxides occur in the fractures as well. In thin section the rock had 74 percent primary minerals, 5 percent glass, and 20 percent smectite clays. The glass found in the thin section was in the second stage of development to a smectite clay.

The Milepost 60 (M6)quarry is the same age as the Milepost 40 quarry. The quarry face is about 12m (40 ft) thick and the rock is jointed into columns averaging approximately 30cm (1 ft) across. The columns are generally unfractured and seamed with veins of chalcedony and calcite. Along the westside of the quarry the weathering was more pronounced due to alteration and coating of a clay weathering rind but in thin section analysis the quarry had 70 percent primary and 30 percent secondary minerals. Also, under high magnification the plagioclase crystals were infilled by calcite. The infilling of calcite most likely contributed to the high DMSO weight loss determined for the Milepost 60 quarry. Both quarries have acceptable mechanical durability properties as shown in Table 2.2.

Dovre Peak Quarry. -- Dovre Peak quarry was studied by Van Atta and Ludowise in 1974 and 1976. A description of the quarry from their 1974 report (20) is as follows:

" Rock exposed in the Dovre Peak quarry face occurs in two different basaltic units. The lower unit consists of a coarse-grained aggregate of plagioclase feldspar and pyroxene with interstitial microcrystalline plagioclase feldspar set in a matrix of hydrated iron oxide and nontronite, or interstitial turbid brownish opal which has orange-brown nontronite in spherules or vermicular aggregates scattered through it. The upper unit is similar, except that instead of opal, prominent yellow palagonite fills vesicles or is interstitial between plagioclase and pyroxene crystals. The palagonite is partly altered to montmorillonite, which occurs as spherules at the margin of the glass."

In thin section they determined the quarry to have 65 percent primary and 28 percent secondary minerals and 6 percent glass in the upper unit. The lower unit had 70 percent primary and 30 percent secondary minerals with 4 percent glass. The samples which were employed for the durability tests originated from the crusher site, which as noted in the petrographic table contain a secondary mineral content of 50 percent and a smectite clay content of 39 percent. The high clay content for the samples is reflected by the poor durability measured by the standard mechanical tests and the DMSO Accelerated Weathering test.

Weston Mountain Quarry. — Rock in the Weston Mountain quarry is well fractured and the face is covered by rubble ranging in size from 5cm (2 in.) to approximately 40cm (1.5 ft.). A high degree of weathering has occurred throughout the rock mass due to fracturing, and as a result the rock is coated with a light tan to yellow alteration rind. The rock is fine-grained with vesicles from 3 to 10mm in size comprising about 10 to 15 percent of the rock volume. Thin section analysis determined the primary and secondary mineral content to be 66 percent, and 39 percent, respectively, with smectite clays comprising 21 percent of the total secondary mineral content. The

size of the groundmass in thin section was found to range from 1 to 2mm. Minor amounts of palagonite were also identified in the thin section to be in the third stage of weathering to a smectite clay. X-ray diffraction analysis showed that the only clay mineral present was nontronite.

Eckman Creek Quarry. — Rock from the Eckman Creek quarry is representative of a marine basalt, and was identified by Clemmons (2) as being a source of marginal aggregate on the Oregon Coast. The rock on the quarry face is unfractured, vesicular and porphyritic and crudely jointed into columns. Some of the vesicles are filled with a green alteration product, which was identified to be a smectite clay mineral by X-ray diffraction analysis. In thin section the primary mineral content was determined to be 70 percent, and the combination of smectite clays and glass comprise the remainder of the rock volume. The glass which was found in the thin section was largely altered to palagonite. The groundmass size was large, up to 4mm for some plagioclase laths. In addition to identifying the clay minerals, the X-ray diffraction also identified the presence of analcime, a sodium-aluminum silicate which can be classed with zeolites. Zeolites have high cation exchange capacities, similar in magnitude to swelling clays. Szymoniak et al (18) attributed the high DMSO weight losses to the chemical reaction occurring between the analcite mineral and DMSO.

Hermiston Quarry. — The Hermiston quarry is a homogeneous river run gravel source. Thin section analysis showed the source to have 61 percent primary and 39 percent secondary minerals. Interstitial

palagonite was found in the thin section and was identified to be in the third stage of development into a smectite clay. The X-ray diffractogram identified the presence of smectite clays and paragonite. Paragonite is very similar to mica.

Indirect Test Results

DMSO Accelerated Weathering Test. — The results from the DMSO Accelerated Weathering Test for the eight quarries studied are shown in Table 2.2. The DMSO losses were determined for the aggregates in the particle size range of 2.4mm (#8) to 4.8mm (#4). Figure 2.3 shows the relationship between clay contents determined from the stained thin sections to DMSO loss for the quarries. Regression analysis for the DMSO loss (DMSO) as a function of clay content (C) gave the following :

$$\text{DMSO} = .20133 * \exp[0.159 * (C)] \quad (1)$$

which has a correlation coefficient of 0.92. Equation 1 is illustrated in Figure 2.3. The results from the Eckman Creek Quarry and Milepost 60 quarries were neglected since it was concluded that DMSO was reacting with the analcime and calcite minerals found in the quarry rocks and creating the additional weight loss. In general, the correlation of the clay content to the DMSO loss appears quite good, which supports the use of the DMSO Accelerated Weathering test to indicate the clay content in basalt aggregates. The high correlation also suggests that DMSO and the rhodamine-b stain are reacting with

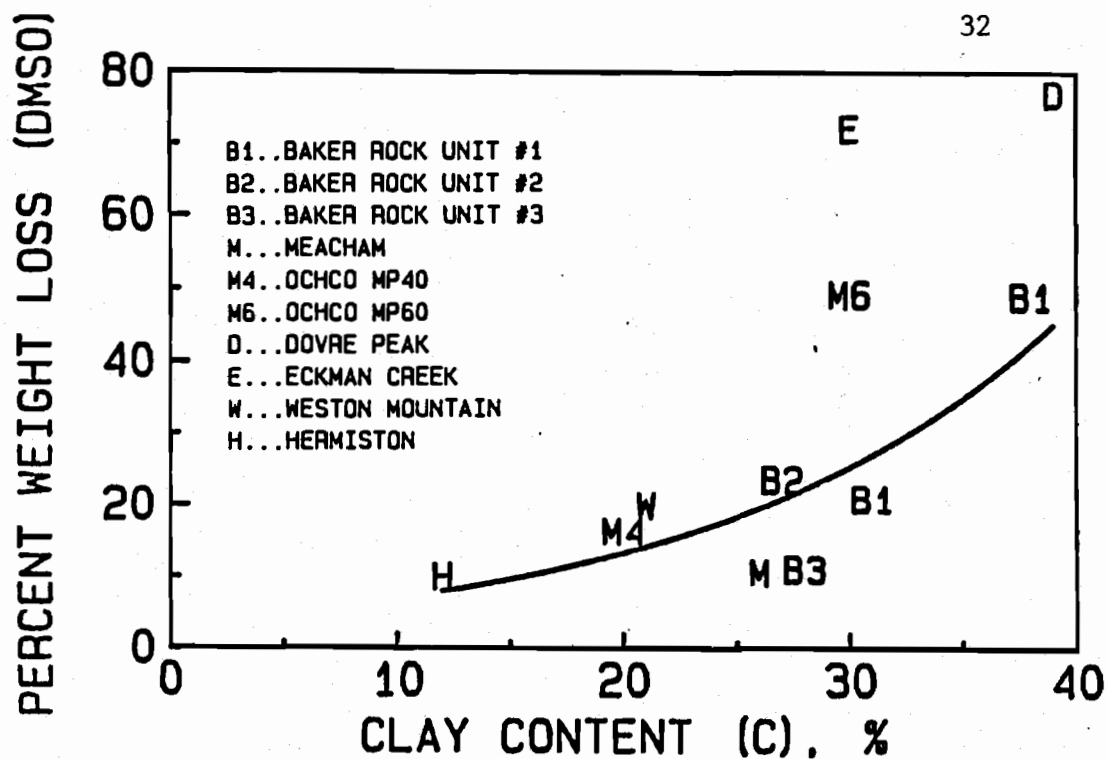


Figure 2.3 - Percent Loss by Weight from the DMSO Accelerated Weathering Test versus Clay Content Determined from Petrographic Analysis.

the same minerals, which are predominately swelling type clays. This fact verifies the use of DMSO to predict the presence of swelling clays. In addition, the DMSO loss tends to be highly dependent upon the textural distribution of the clay minerals and the size of the groundmass. (Recall Van Atta and Ludowise (20) concluded that the deleterious mineral contents alone were poor indicators of aggregate performance, and must be augmented with the textural distribution of the mineral.) In assessing this situation, Szymoniak et al (18) found that the secondary mineral rating, suggested by Cole and Sandy (5), which accounts for the type of deleterious mineral and its distribution in the clays, gave a excellent correlation to DMSO loss. However, it should be noted that if a quarry has a high DMSO loss (greater than 60 percent), this fact alone does not indicate that the rock has a high clay content and is unusable, as is the case for the Eckman Creek Quarry. If zeolites are present in the quarry rock the DMSO loss may be high, and as such a complete petrographic analysis should be conducted to determine the clay content.

Atterberg Limits. — The results from the Atterberg Limit Tests are shown in Table 2.3. Liquid limits for the quarries ranged from 27.3 to 22.5 percent, and only three quarries had measurable plasticity. The relationship between liquid limit versus clay content, from the stained thin sections is shown in Figure 2.4 Also shown are results obtained by Seed et al (14) for clay standards. The results for the quarries studied are much lower than those obtained by Seed et al for clay standards with activity levels typical of swelling clays. There are two possible explanations for the poor agreement.

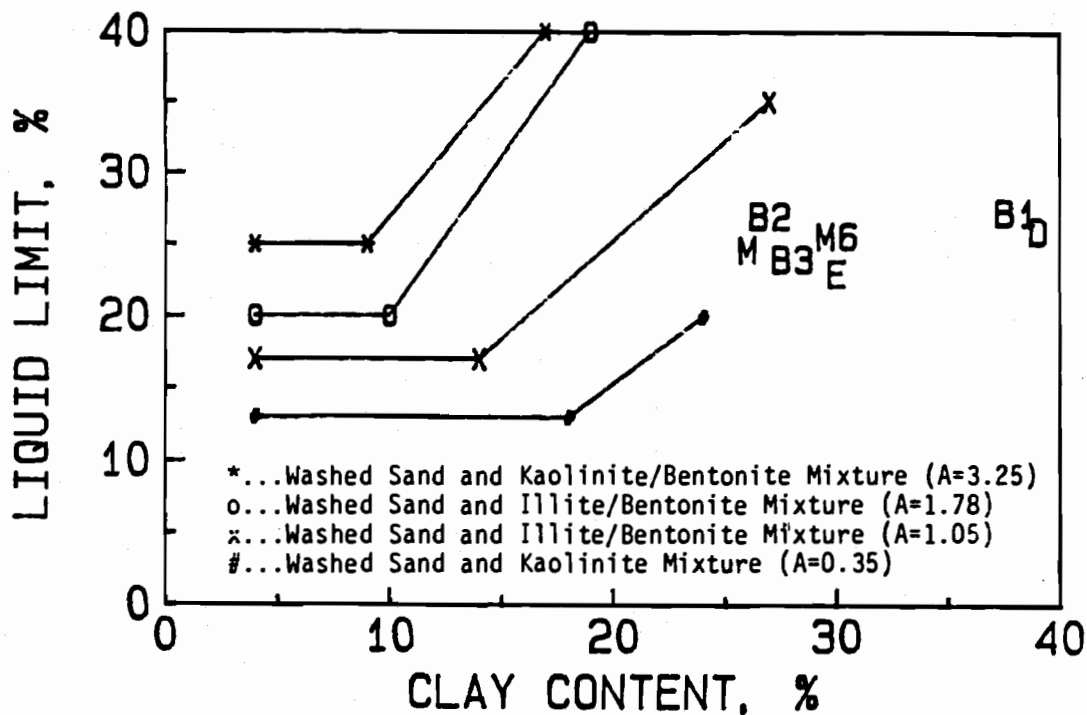
Table 2.3 - Atterberg Limit and Clay Index Summary.

Quarry	Liquid Limit	Plastic Limit	Plasticity Index	Clay Index MBA	Petrographic Hydrometer	
					Clay Content	Clay Content
Baker Rock Unit #1	27.3	25.8	1.5	4.8	38.0	22.1
Baker Rock Unit #2	27.0	N/P	-	1.3	27.0	8.2
Baker Rock Unit #3	24.0	N/P	-	3.3	28.0	10.4
Meacham	24.7	N/P	-	1.1	26.0	-
Milepost 40	-	-	-	1.5	20.0	10.4
Milepost 60	25.6	N/P	-	1.3	30.0	5.9
Dovre Peak	26.6	25.5	1.1	2.0	29.0	14.7
Weston	-	-	-	2.3	21.0	10.8
Eckman	23.0	21.5	1.5	3.2	30.0	12.4

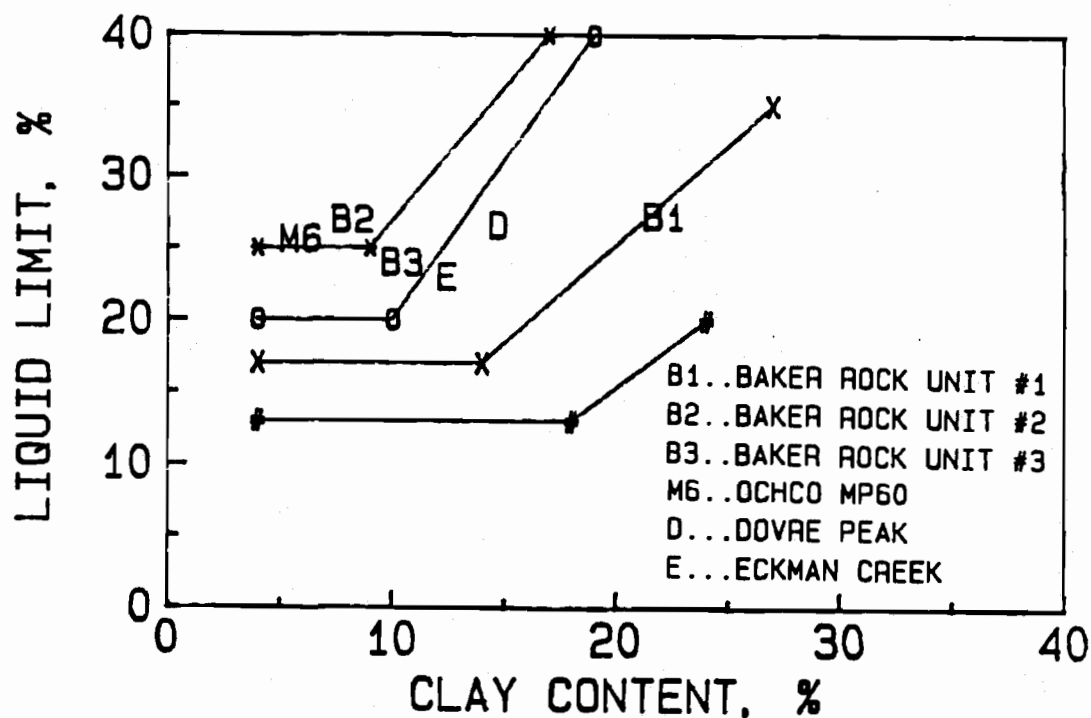
The first explanation is that in using the standard Casagrande device the pulverized aggregate liquefied, rather than experiencing a shear failure. Consequently, the values reported would be lower than the actual liquid limit. The British have adopted a cone penetrometer procedure to measure the liquid limit (BS 1377). The method employs an 80g cone penetrometer with a 30 degree angled tip; a penetration of 20mm into a 35mm by 55mm cup defines the liquid limit. A complete description of the cone penetrometer test procedure is given by Sherwood and Ryley (15).

The cone procedure eliminates several of the operator errors inherent with the Casagrande device and avoids problems associated with liquefaction. Liquid limits determined using the cone are also shown in Figure 2.4b. However, the values obtained using the cone are still lower than those obtained by Seed et al (14).

The second explanation is that clay contents measured in the thin section are based on volume whereas the clay contents reflected by the liquid limit tests results on pulverized aggregate are on weight. The two are, however, related by the specific gravities of the minerals contained in the rock. For example, the percentage of clay by weight can be calculated if one assumes that the rock contains, by volume, 56 percent plagioclase with a specific gravity of 2.7, 10 percent olivine with a specific gravity of 4.5, and 34 percent smectite with a specific gravity of 2.8. The calculation of the percent clay by weight is 33, which is in close agreement to the assumed percent clay by volume. This factor leads one to conclude that the poor agreement between the results obtained in the present



(a) - Liquid Limit versus Clay Content Determined from Petrographic Analysis.



(b) - Liquid Limit versus Clay Content Determined from Hydrometer Analyses.

Figure 2.4- Determination of Clay Contents from Liquid Limits.
 size.

study and those obtained by Seed et al are the consequence of not pulverizing the crushed aggregate to sizes smaller than the clay

The pulverized material used for the Atterberg Limits was produced from aggregate in the size range between 19-12.5mm (3/4-1/2 in.). Crushing the aggregate to this size would have broken the rock along planes of weakness, such as the clay seams. Consequently, the clay fraction would not have been present in the larger sizes, but would increase with decreasing particle sizes of aggregate used to produce the powder for the liquid limit test. In addition, it is unlikely that the rock was pulverized sufficiently to release the clay minerals from the rock matrix. To verify this hypothesis hydrometer analyses were conducted on several of the pulverized aggregate samples and the percentage of clay (sizes less than 0.075mm) was determined. The liquid limits are plotted in Figure 2.4b versus the clay contents determined by the hydrometer analyses. The data collected using the clay content from the hydrometer analysis corresponds to the results presented by Seed et al (14), indicating that the liquid limit of pulverized rock is a function of the aggregate particle size used and that the pulverization of the aggregate did not release the clays held within the matrix.

Clay Index. — The results from the Clay Index test are given in Table 2.3, and the values are plotted with respect to the clay content in Figure 2.5. A regression analysis for the relationship between clay content (C) determined from petrographic analysis and to the Clay Index (CI) gave the following:

$$CI = 0.321 * \exp [(0.0659 * (C))] \quad (2)$$

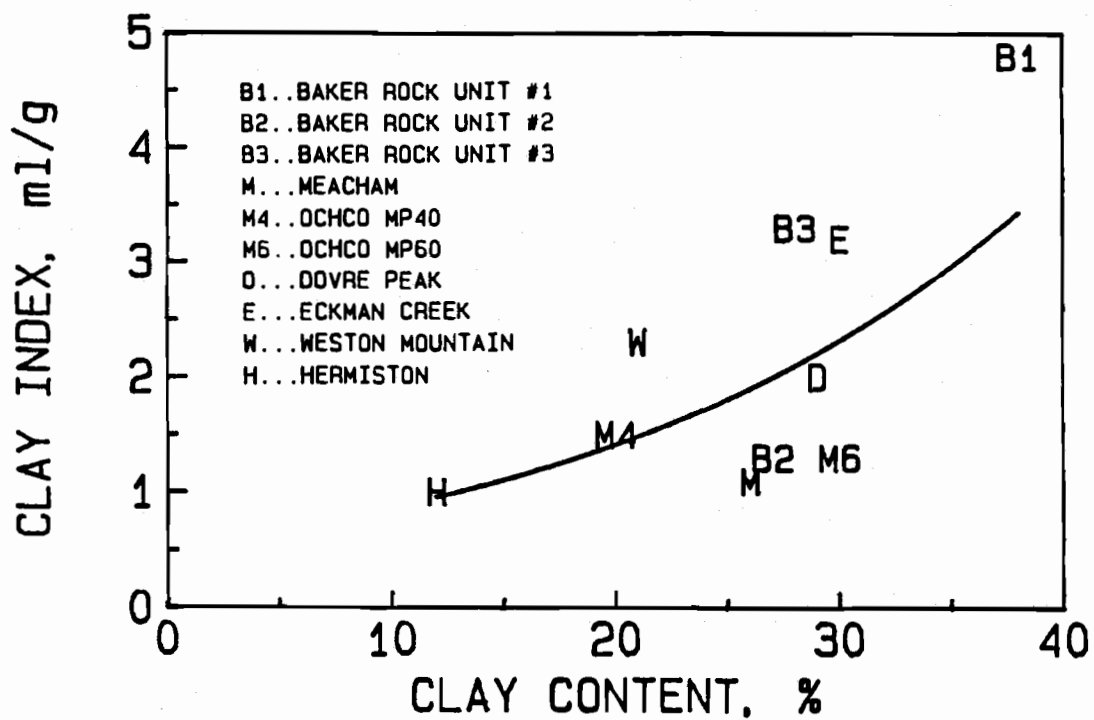


Figure 2.5. - Clay Index versus Clay Content Determined from Petrographic Analysis.

which has a correlation coefficient of 0.85. The good correlation between the two indicates that the Clay Index test is a valid indirect method to assess the clay content. Unlike the DMSO Accelerated Weathering Test, the Clay Index values do not reflect the presence of the analcime mineral found in the Eckman Creek quarry. Sameshima and Black (12) also showed indicated that pure zeolites do not absorb the methylene blue stain employed in the test.

The advantages in using the Clay Index Test over the other indirect tests considered in the study are associated with (1) the simple test procedure and (2) the time required to complete the test. It takes a minimum of six days to perform the DMSO test, whereas the Clay Index test can be completed in five minutes, after the aggregate has been pulverized.

Figure 2.6 presents the result from a study on the effect of the initial aggregate particle size pulverized to produce the powder for the Clay Index test. The investigation was conducted to determine if there was a optimal initial aggregate particle size that should be employed to yield the highest Clay Index value. In reviewing Figure 2.6, it is apparent that the Clay Index values maybe associated with the distribution of the clay in the rock. During crushing, the aggregates will break along planes of weakness, such as the clay seams. The larger particle sizes tend to have the same clay content, and nearly the same Clay Index. As the initial aggregate particles become smaller, the clay content decreases which acts to decreases the Clay Index value. Eventually the particle size becomes so small that the

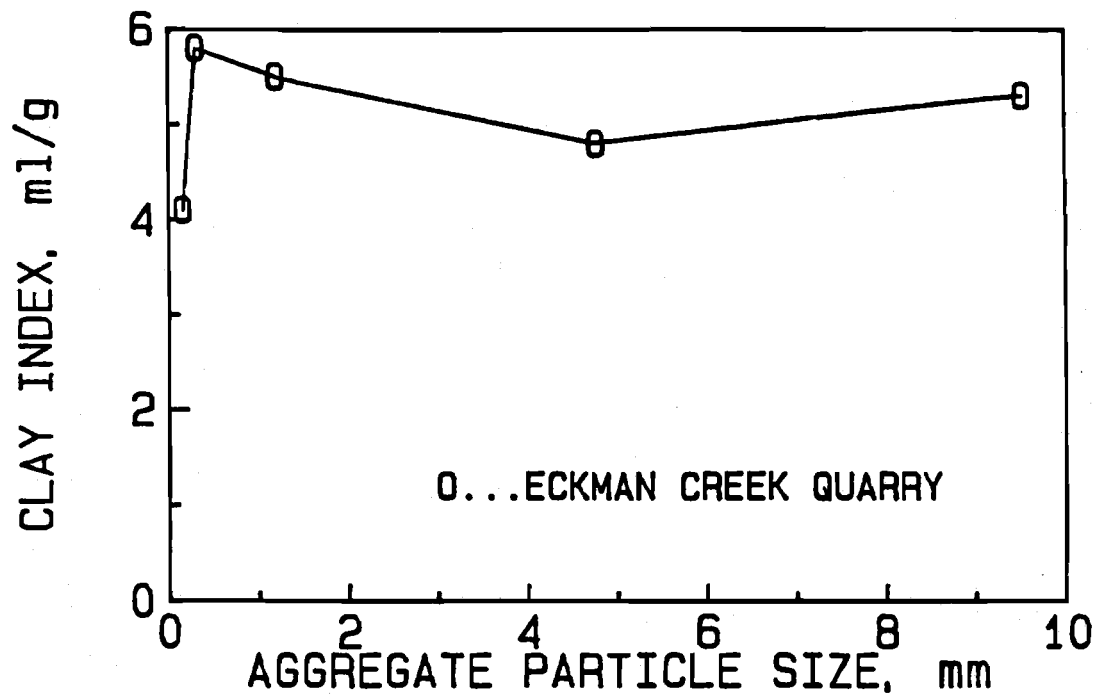


Figure 2.6. - Clay Index versus Initial Aggregate Particle Size Pulverized.

clays are removed by crushing. Generally, the results indicate the Clay Index is constant for particles sizes greater than 0.3mm and decreases below this value. This limited data set would suggest that the Clay Index could be performed on pulverized aggregate produced from the particle size range above 0.6mm (#30).

2.5 SUMMARY AND CONCLUSIONS

There are two general methods that can be employed to determine the clay mineralogy and content for roadway aggregates. Direct methods include petrographic and X-ray diffraction analysis. The petrographic analysis is conducted on thin sections of rock in which clay content is determined by a point counting procedure. To augment the point counting procedure (and reduce the need for X-ray diffraction analysis) a rhodamine - b stain which is specific for smectite clays can be utilized.

Indirect methods to determine the clay content include: an accelerated weathering test employing DMSO or ethylene glycol, Atterberg Limits, and a Clay Index Test which is a titration procedure using methylene blue dye. The results from the DMSO Accelerated Weathering Test employed in this study indicate that the loss determined reflects the type of clay and content. The results also indicate that the DMSO loss for aggregate samples is highly affected by the distribution of the clay and other minerals within the rock matrix. An equation to predict the clay content from the DMSO weight loss was presented.

The second indirect method, Atterberg Limits, resulted in a poor correlation between liquid limit and the clay content determined from

rock thin sections. The poor correlation is owing to the fact that the clay minerals in the rock matrix are not released during pulverization of the aggregate. This was verified by conducting hydrometer analyses on several samples of the pulverized aggregate. The correlation between liquid limit and clay content determined from the hydrometer analyses was very good compared to published correlations for clays with high activities

The third indirect test utilized was the Clay Index, which is defined as the number of milliliters of 4.5g/l of methylene blue dye adsorbed by 1g of minus .074mm (#200) pulverized aggregate. The results indicate that the Clay Index value provides an acceptable correlation to the clay content. An equation to predict the clay content from the Clay Index is presented. The data collected suggest that the Clay Index test should be performed on pulverized rock produced from aggregate particle sizes above 0.6mm (#30).

Practical Significance of Test Program

Both direct and indirect methods can be utilized to determine the clay contents of roadway aggregates. The direct methods, petrographic analysis and X-ray diffraction, were simplified by employing a rhodamine-b stain which was found to be specific for smectite clays.

Of the three indirect methods employed in this study, the results from the DMSO Accelerated Weathering and the Clay Index test were found to correlate to clay contents from the stained thin sections of rock. The advantages in using a reliable indirect test include the fact that large samples can be utilized which would better reflect

the aggregate produced from the quarry, and the procedures associated with the two indirect tests are not complex and can be preformed by field personnel. Of the two tests, the Clay Index test is simpler to perform and significantly reduces the time required to determine the clay mineral content in basalt aggregates.

2.6 ACKNOWLEDGEMENTS

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3.0 THE DIMETHYL SULFOXIDE (DMSO) ACCELERATED

WEATHERING TEST FOR AGGREGATES

by Tom Szymoniak, Ted S. Vinson, James E. Wilson and Neal Walker

ABSTRACT : A standard Accelerated Weathering Test using Dimethyl Sulfoxide (DMSO) was developed to simulate the chemical degradation of basaltic rock. The final development of the standard test involved a study of the interaction of DMSO with clay standards and an investigation of the test condition parameters affecting an existing DMSO test procedure. The weighted loss reported under the existing test procedure (similar to the Sodium Sulfate Soundness test) was affected by the aggregate particle size, sample weight, immersion time, and container geometry. In the recommended standard DMSO Accelerated Weathering test, aggregates in the particle size range of 2.4 mm to 4.8 mm are immersed in a container of DMSO for a period of five days. The weight loss determined by sieving the material on a 2.4mm (#8) sieve after five days immersion provides a measure of durability. Immersion of basaltic aggregates in DMSO results in a greater breakdown owing to the type and concentration and concentration of ions contained in the rock matrix. Specifically, it was found that DMSO reacts with the cations held to the swelling clay minerals in the aggregate. The results from Atterberg Limits tests indicate that DMSO substantially reduces the liquid limit for clays with high activities and increases it for clays with low activities. The changes in the liquid limits were influenced by the interaction of DMSO with cations held on the surface of the clay mineral and the ability of DMSO to donate hydrogen ions.

In selecting an aggregate source to insure satisfactory pavement performance both the physical properties of the rock and the environment in which it is placed must be considered. Physical properties include surface texture, size, shape, gradation, strength, durability, and mineralogical composition(1). Environmental factors include weather, traffic, and the availability of water. Degradation or disintegration of the aggregate in the pavement structure owing to the interaction of the rock properties with the environment is caused by mechanical and chemical weathering processes. Mechanical weathering processes which result in the actual physical breakdown of a rock are generally associated with the action of equipment during construction or the operation of traffic throughout the life of the pavement. Chemical weathering of a rock, caused by the environment in which the aggregate is placed, results in the disintegration of the mineral components of the rock.

The physical properties of a rock are typically assessed through standard laboratory tests in which the aggregate produced from a quarry must meet the criteria established by the governing agency. For example, the Oregon Department of Transportation (ODOT) uses the Los Angeles Abrasion test (ASTM C131), the Sodium Sulfate Soundness test (ASTM C88), and the Oregon Aggregate Degradation test (Oregon TM208) to assess the durability characteristics of an aggregate. These tests tend to reflect the mechanical degradation potential but do not simulate the chemical degradation common to many types of basaltic rocks in Oregon. In fact, the basalts generally have good mechanical durability properties, and will usually pass the standard aggregate

durability specifications. However, when some types of basalts are incorporated into a pavement structure as the base course aggregate or in the asphaltic concrete, field experience indicates that the aggregates degrade owing to chemical weathering of the minerals present in the rock. Specifically, the basalts may contain swelling clays, such as smectites, which, in the presence of water, expand and lead to the eventual failure of the pavement.

As a result of the inability of the standard tests to predict the durability of basalt aggregates the Materials Section of the Federal Highway Administration (FHWA) Western District Federal District (WDFD) developed an immersion test in Dimethyl Sulfoxide (DMSO) to assess the chemical degradation potential of an aggregate source. The applicability of the DMSO test to predict the degradation characteristics of a rock and the meaning of the index parameter determined is not well understood. In recognition of this situation, a research study was undertaken to: 1) identify the factors affecting the DMSO test procedure, and 2) develop a standardized test procedure. The results from this study are reported herein.

3.2 EVOLUTION OF THE DMSO ACCELERATED WEATHERING TEST

The use of an accelerated weathering test to simulate the chemical degradation of aggregates began in the late 1960's when the Army Corps of Engineers proposed an ethylene glycol immersion test to assess the quality of basaltic rock. Ethylene glycol has a history of use in X-ray diffraction analysis as an aid in the identification of swelling clays. The X-ray diffraction pattern of swelling clays increases from the normal basal spacing of approximately 14 Å to 17 Å

after treatment in a saturated ethylene glycol environment(2). Considering this fact it was reasoned that the swelling pressure created by the expansion of the clay structure in a rock matrix would result in the deterioration and breakdown of the rock and, therefore, immersion of aggregates in ethylene glycol would provide an ideal accelerated weathering test.

The original procedure proposed by the Corps utilized a ten rock sample of aggregate in the size range of 9.5mm (3/8in.) to 25.4mm (1in.). The ten rocks were selected from a representative sample of the aggregate source and were immersed in ethylene glycol for a period of ten days, at which time the number of rocks observed to crack, spall, or disintegrate were recorded (3). This value was reported as an index to relate the quality of the aggregate source; if four or more rocks cracked, spalled, or disintegrated the quarry was deemed unacceptable.

In the mid-1970's the ten rock test was modified by the FHWA Region 10 Materials Section by substituting DMSO for ethylene glycol. The substitution was based on a study of basalt aggregates conducted by Van Atta and Ludowise(4), in which they noted that DMSO caused an expansion of swelling clays from the normal basal spacing of 14 A to 18 A. The difference in the magnitude of expansion, when compared to ethylene glycol, suggested that DMSO immersion would lead to a more severe breakage of the aggregate. In comparative testing between ethylene glycol and DMSO, using the same aggregate source, immersion in the DMSO solution typically resulted in an additional breakdown of 2-4 rocks. This relationship is reflected by the

results presented in Figure 3.1 which indicates, for the fifty-seven quarries considered, that immersion in DMSO results in a greater number of the rocks cracking, spalling or disintegrating.

The ten rock procedure was modified in 1978 by incorporating a procedure which reported a weighted loss after a five day immersion period in DMSO. The procedure adopted was similar to the Sodium Sulfate Soundness test in which greater sample weights are utilized for increasing aggregate size fractions. Figure 3.2 presents a relationship between the ten rock test and the weighted loss test procedure. The test procedure as it presently exists consists of the following steps:

- 1) wash and oven dry the crushed aggregate to a constant mass at $110 \pm 5^{\circ}\text{C}$ ($230 \pm 9^{\circ}\text{F}$);
- 2) separate the aggregate into the sieve size ranges as given in Table 3.1 and weigh out the minimum amount of aggregate fractions noted;
- 3) immerse the samples in DMSO solution for a period not less than 112 hours nor more than 120 hours (the samples are covered and maintained at a temperature of $21 \pm 5^{\circ}\text{C}$ ($70 \pm 5^{\circ}\text{F}$));
- 4) remove the samples, rinse in tap water and dry to a constant mass at $110 \pm 5^{\circ}\text{C}$ ($230 \pm 9^{\circ}\text{F}$);
- 5) sieve the samples over the sizes indicated in Table 3.1 and calculate the percentage weight loss for each size fraction;
- 6) the reported weighted loss is calculated by multiplying

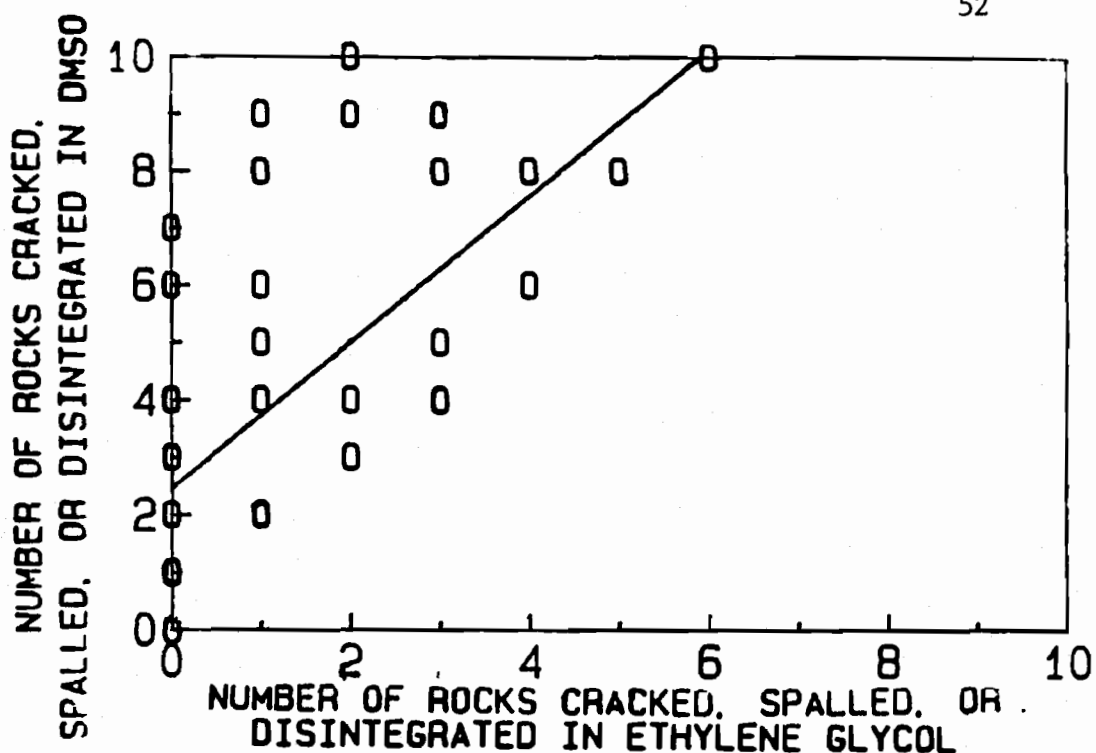


Figure 3.1. - Ten Rock Test Procedure Results for Immersion in DMSO to Immersion in Ethylene Glycol.

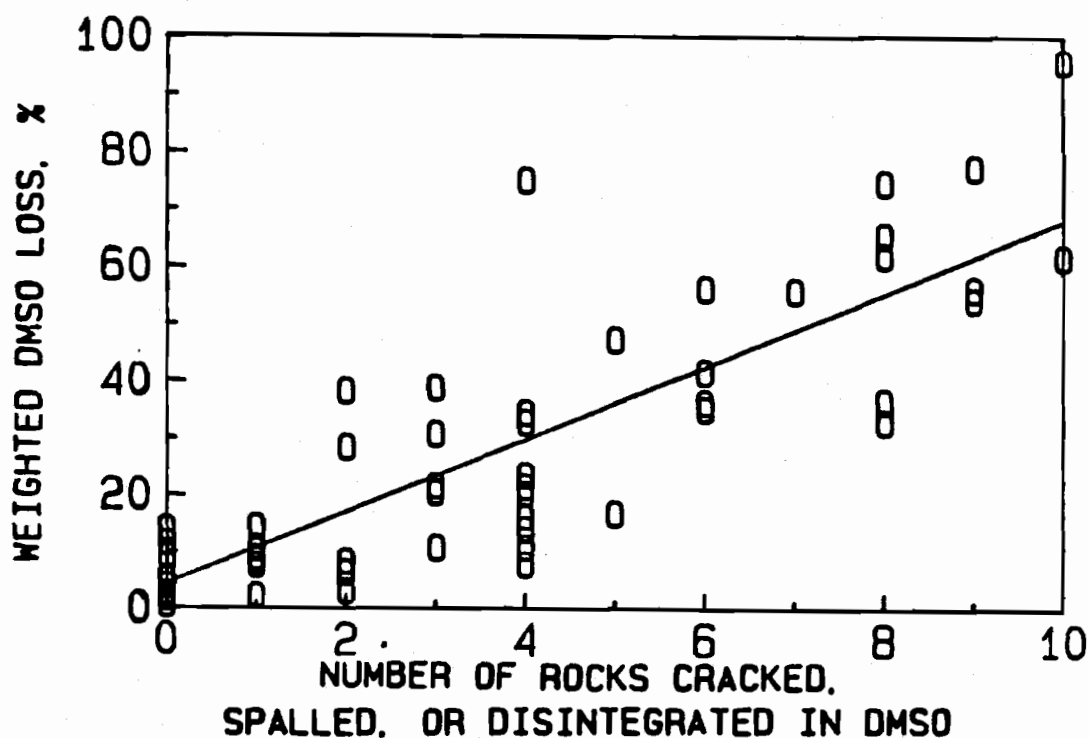


Figure 3.2. - Ten Rock Procedure Results versus Weighted Loss Results for Samples Immersed in DMSO.

Table 3.1 - Federal Highway Administration Western Direct
Federal Division - DMSO Immersion Test Requirements.

Aggregate Particle Size	Sample Weight	Sieve Size Used to Determine Loss
4.75 to 9.5mm	300 g	4.0mm (No. 5)
9.5 to 19.0mm	1000 g	8.0mm (5/16in.)
33% - 9.5 to 12.5mm	-	-
67% - 12.5 to 19.0mm	-	-
19.0 to 37.5mm	1500 g	16.0mm (5/8in.)
33% - 19.0 to 25.0mm	-	-
67% - 25.0 to 37.5mm	-	-
37.5 to 63mm	3000 g	31.5mm (1-1/4in.)
50% - 37.5 to 50mm	-	-
50% - 50 to 63mm	-	-

the percent loss following immersion in DMSO by the percentage of the same size fraction identified in the aggregate gradation specification.

The allowable weighted loss limit for this test procedure was initially set at 20 percent but was later lowered to 12 percent. The lower limit was established after observing failures of aggregates with losses between 12 and 20 percent. If a quarry rock had a weighted loss greater than 12 percent it was deemed unsuitable.

Before continuing with the discussion of the factors affecting the DMSO test, the chemical itself and its interaction with clay minerals must be understood. This discussion is presented in the following section.

3.3 DMSO - WONDER DRUG OF THE 1980's

DMSO has often been called the wonder drug of the 80's. It has reportedly been used to cure everything from the common headache to providing effective pain relief for arthritis (5). DMSO was first brought to the public's attention in the early 1960's because of its medical possibilities and has since been the subject of many articles in both the medical and scientific professions. DMSO is a versatile solvent currently being used industrially and in chemical laboratories as a medium for completing chemical reactions(6). It is a colorless, odorless (when pure), very hygroscopic, stable liquid. The chemical properties of DMSO are summarized in Table 3.2 along with comparable properties for both water and ethylene glycol.

DMSO is manufactured commercially by Crown Zellerbach Corporation,

Table 3.2. - Physical and Chemical Properties of DMSO, Water, and Ethylene GlycolA.

Property	WATER	DMSO	Ethylene Glycol
Chemical Composition	H ₂ O	(CH ₃) ₂ SO	(CH ₂ OH) ₂
Specific gravity	0.99823	1.1008	1.115
Molecular weight	18.0	78.13	62.07
Boiling point, deg C at 760mm	100.0	189.0	197.6
Freezing point, deg C	0.0	18.45	-13.0
Specific heat, cal/g	1.0	0.47	0.56
Mole heat, cal/mole	18.0	36.7	34.8
Mole volume, cc/mole	18.0	71.3	55.9
Heat capacity, cal/cc	1.0	0.517	0.720
Surface tension, dyne/cm	72.45	42.85	48.40
Refractive Index @ 20°C	1.33	1.48	1.43
Conductivity, ohm ⁻¹ cm ⁻¹ @ 20 °C	-	3.0 x10 ⁻⁸	1.07x10 ⁻⁸
Dielectric Constant, 10MHz @ 20°C	80.0	48.9	38.7
Dipole Moment, Debye Units	1.89	4.3	2.2
(Dielectric Constant)x(mole volume) ^{1/3}	209	204	148
Absolute Viscosity, centipoises @ 20°C	1.0	2.1	180.0

A References 7,8,9,

(one of the world's largest paper-processing companies) by reacting the black liquor formed from the kraft pulp process with molten sulfur to form dimethyl sulfide, CH_3SCH_3 . The dimethyl sulfide is then oxidized with nitrogen tetroxide to yield DMSO (6).

DMSO was first synthesized by the Russian chemist A. N. Saytzeff in 1866 (5) but it was not until the late 1950's that its medical possibilities were discovered by Herschler, a research chemist from Crown Zellerbach Corporation. Herschler(5) suspected that DMSO was penetrating the skin and getting directly into his system after inadvertently spilling it on his hands. (He noticed the penetrating power after developing an oyster-like taste in his mouth!) Recognizing the penetration ability, Herschler applied DMSO to a burned area on his hand and found that it prevented the burn sore from blistering. One of his co-workers then tried the chemical on a sprained ankle and found that it reduced the amount of swelling and healing time.

Despite intense research and the number of successful applications the chemical has been associated with over the years to indicate its possible medical value, DMSO has not been approved for general public usage. The Federal Drug Administration (FDA) banned the use of DMSO and the testing of the chemical on humans in 1965 after clinical studies on laboratory rabbits showed that their eye lenses became distorted upon repeated contact with DMSO (5). FDA later approved its use in 1974 for only one condition, a rare and painful bladder disease known as interstitial cystitis(3). Industrial use of the chemical has, however, continued since it is such an excellent solvent and penetrant.

DMSO has a remarkable ability to penetrate biological membranes without permanently altering or damaging the membrane structure(6). The greater penetrating power of DMSO as compared to ethylene glycol is a desirable feature in developing an accelerated weathering test since the degradation of the aggregate samples would occur in shorter periods of time.

Franz and Van Bruggen (10) proposed two mechanisms by which DMSO may improve passage through a barrier. The first mechanism is related to the osmotic pressure created by an electrical potential between two charged ions which provides for a non-selective increase in the permeability of the barrier; the second mechanism is due to the interaction between DMSO and ionic species on the opposite side of the membrane. An analogy which relates to these mechanisms would be the drainage of a saturated clay soil by electroosmosis in which the water contained in the soil migrates from the anode to the cathode. Under the influence of an electric potential, ions move toward an electrode of the opposite sign dragging the bonded water molecules. As a result of this, the permeability of the clay increases by a factor of 100 to 1000 times the untreated value (11).

DMSO is a very polar compound and due to the attachment of the sulfur molecule it is slightly anionic. The electric potential, which aids in increasing the permeability, is created by the attraction of the anionic DMSO compound with cations on the opposite side of the membrane. As the concentration of cations increases on the opposing side of a membrane so does the ability for the DMSO compound to penetrate owing to the attraction with the opposite charged ions.

Unlike water and ethylene glycol, DMSO does not have the ability to hydrogen bond and it is considered a hydrogen donator. By donating hydrogen to a reaction rather than bonding (as would be the case with water or ethylene glycol) the DMSO molecule can further penetrate into a structure. Hydrogen bonding would slow the progression by effectively creating a drag on the fluid as it crosses the membrane.

Dissolution, or solvation of molecules, is affected by: 1) the dipole moment of the solvent, 2) the amount of shielding of the dipole charges, and 3) the nature of the atoms carrying the dipole charges (12). The dipole moments of ethylene glycol and DMSO (3.9 and 2.2 Debye units, re. Table 2), are larger than the dipole moment of water but because of their lower dielectric constants their positive poles are not as accessible as that of water. Though the dielectric constant of DMSO is lower than water (48.9 at 20°C for the former and approximately 80 for the latter), the respective products of the dielectric constants and the cube roots of the mole volumes are approximately equal. Andrews et al (8) believed that the product of these two chemical properties reflected the capacity of the liquid to reduce the strength of an electric field. As shown in Table 2, the product is about the same for water and DMSO, while that for ethylene glycol is approximately 75 percent of the value of DMSO.

The second factor that is important for the solvation of a molecule is the amount of shielding of the dipoles. For the DMSO molecule the positive end of the dipole is not as accessible as in the case of water and ethylene glycol. This shielding is again due to the attachment of the sulfur molecule which makes the interaction

of the dipoles with anions considerably weaker than with cations(8). Dissolved anions and anionic surfaces are, therefore, relatively free from interaction with DMSO while cations become solvated.

As previously shown in Figure 3.1, rocks immersed in DMSO have a higher loss than those immersed in ethylene glycol solutions. The difference can be explained by considering their reactivity rates and different bonding capabilities. DMSO increases the chemical reaction rate by a factor of 10^4 above that of water because of its lower dielectric constant (12). The ability for DMSO to penetrate a rock by seeking out the cations that are held on the clay surface without dragging water molecules through hydrogen bonding also leads to the greater breakdown. It is also important to note that the data collected by Szymoniak and Vinson(13) and Miles (14) indicates that DMSO will not only react with the swelling clays but with other minerals such as zeolites and calcite.

3.4 LABORATORY TEST PROGRAM

In order to study the effects of DMSO on different rock types which contain degrading type minerals, it is first necessary to establish a standard test to allow results to be compared on the same basis. During the evolution of the existing DMSO test procedure the factors affecting the weighted loss were not considered to any great detail. Thus, under the present study the first step in the establishment of a standard DMSO Accelerated Weathering test was to investigate the influence of test condition parameters (e.g. particle size, sample size, immersion time and container geometry) on the test results obtained. In addition, the electrostatic interaction of DMSO with

clay standards was investigated in the laboratory testing program to provide an understanding of the mechanisms by which DMSO expands clay minerals.

The laboratory test program was divided into two segments. The first dealt with sampling six quarries in the State of Oregon and testing the aggregate produced using the existing DMSO weighted loss procedure as well as performing the standard ODOT durability tests. The existing DMSO procedure was used to study the factors affecting the weighted loss determined and aid in the development of a standard procedure. The second segment consisted of investigating the electrostatic interaction of DMSO with clay standards through the use of the Atterberg Limit Test.

In establishing any standard laboratory procedure it is important to understand both the test condition parameters of the test itself and the relationship of the test results to field performance. The performance of a pavement structure is a function of the quality of the aggregate, the environment, and construction practices. The performance, however, cannot be truly assessed until the pavement section has failed, and it is often difficult to separate the quality of the aggregate from other factors. Considering this situation, and due to the lack of understanding of the test condition parameters affecting the existing DMSO aggregate degradation test, no attempt was made to correlate the test results to field performance. However, the six quarries that were selected for this study have association with a performance problem such as a base course failure or an asphalt stripping failure.

Parameters Affecting the Existing Procedure.--- The test condition parameters of the DMSO test as it presently exists include the aggregate article size, sample weight, immersion time, and the geometry of the container. Three other factors which were held constant throughout the test program were the temperature of the samples during immersion, the concentration of DMSO and aggregate preparation prior to immersion. The temperature of the samples for the duration of a test was held constant at 21°C (70 °F). Higher temperatures lower the viscosity of the DMSO which would increase the penetration rate of the molecule into the rock matrix. Ease of penetration into the rock would result in higher degradation values.

The concentration of DMSO was held constant at 100 percent, which in turn establishes the viscosity at 2.0 centipoise (re. Table 2). If a mixture of water and DMSO had been used both the concentration and the viscosity would change which would affect the penetration rate and the reaction of DMSO with other molecules. Reducing the concentration of DMSO would result in lower degradation values. At a 70 percent concentration of DMSO in the mixture, the viscosity is 3.6 centipoise, which is greater than both pure water and DMSO. At concentrations above and below 70 percent the viscosity decreases. Recognizing this fact, any contamination of the concentrated DMSO by water during a test may result in substantially different weight losses. Thus, it is imperative that the concentration of DMSO be maintained at 100 percent during a test and that the sample container is tightly sealed.

The third factor which was held constant for the testing program

was aggregate preparation prior to immersion. Only air-dried the samples were immersed in DMSO. It is well known that oven drying soil samples which have high activity levels substantially reduces the liquid limit compared to air dried samples. The aggregates utilized for this study were fine-grained mafic igneous rocks and therefore, swelling clays were expected. Since oven drying may alter the clay minerals present in the rock, it is obviously prudent to use only air dried samples.

In the procedure utilized by the FHWA several different aggregate particle sizes are employed in order to determine a weighted loss. Since many different sizes are employed it would be advantageous to simplify the test by selecting one aggregate size range that could reflect the highest DMSO loss. In evaluating the effects of the aggregate particle size on the percent DMSO loss in the existing procedure the following size ranges were considered:

76 to 64mm (3.0 to 2.5in.), 38 to 19mm (1.5 to 0.75in.), 19 to 9.5mm (3/4 to 3/8in.), 9.5 to 4.8mm (3/8 to #4), 4.8 to 2.4mm (#4 to 8), 2.4 to 1.2mm (#8 to 16), 1.2 to 0.6 (#16 to 30), and 0.6 to 0.3mm (#16 to 30). The quarry rock was crushed using a mechanical jaw crusher and the various sizes were separated using standard sieves. The samples of the aggregate were weighed and combined in the proportions shown in Table 3.1, then immersed in a 100% solution of DMSO for a period of 5 days. The samples were contained in a plastic freezer container and covered with a lid. The containers were placed in a environmental cabinet where a constant temperature could be maintained throughout the test. The container dimensions were as follows: 18.5

cm (7.25 in.) high, a diameter of 14.6 cm (5.75 in.) at the top, and 13.0 cm (5.13 in.) at the bottom. The container had a total volume of 2750ml (93 oz.).

To evaluate the effect of sample weight used in the test the following weights were utilized: 2000g (4.4 lb.), 1000g (2.2 lb.), 500g (1.1 lb.) and 250g (0.6 lb.). The aggregate particle size was held constant throughout this portion of the study. The size employed was the fraction passing the 19mm (3/4 in.) and retained on a 9.5mm (3/8 in.) sieve, with approximately 67 percent of the total retained on the 12.7 (1/2 in.) sieve.

In addition to monitoring the effect of sample weight, the effect of immersion time was also noted. The weight loss was determined by wet screening the sample using DMSO on the next sieve size lower and then oven drying the material that passed through the sieves. The cumulative sum was compared to the final weight loss determined at the end of the test. This procedure differed from that used by Miles (14) in which samples were subjected to cycles of 18 hours of soaking and 6 hours of drying daily. The procedure as used in the present study was adopted after several trials showed that the final loss after oven drying agreed favorably with the cumulative sum. The samples were screened at approximate time intervals of 1, 2, 4, 5, 10, 20, 40, 90 days, for a total period of ranging from 90 to 120 days.

To study the effect of container geometry, samples at weights of 2000g (4.4 lb), 1000g(2.2 lb), 500g(1.1 lb.), and 250g(0.6 lb) were placed in a flat pan so that no aggregate particles were in contact with each other. The samples in the flat pans were screened at the time

intervals previously noted to study the effect of immersion time.

Atterberg Limit Tests. -- Atterberg Limit Tests (ASTM D4318) were performed to study the electrostatic interaction between swelling type clays and DMSO. The tests were conducted with a calcium montmorillonite clay standard from the Clay Mineralogy Society of America. There are four possible mechanisms by which water is absorbed on clay surfaces: 1) hydrogen bonding, 2) ion hydration, 3) attraction by osmosis, and 4) dipole attraction (15). At low water contents cation hydration is an important mechanism since the interaction between the held cations and the surface of the clays is intense. At high water contents, attraction by osmosis is important, due to the concentration of the cations and the water molecules diffusing towards the clay surface in an attempt to equalize the electrostatic charge (15). DMSO is an aprotic compound, i.e., it does not have the ability to form hydrogen bonds. Therefore, DMSO's interaction with clay surfaces is limited to the latter three mechanisms.

Quarry Geologic Description. -- The location of the six quarries sampled for this study are shown in Figure 3.3. Their durability properties using the standard tests employed by ODOT are summarized in Table 3. Following an initial evaluation of the durability characteristics, two aggregates were selected from the group to study the parameters affecting the existing DMSO test procedure. One aggregate chosen had a high DMSO loss while the other had a low DMSO loss.

The high DMSO loss material originated from the upper bench of Baker

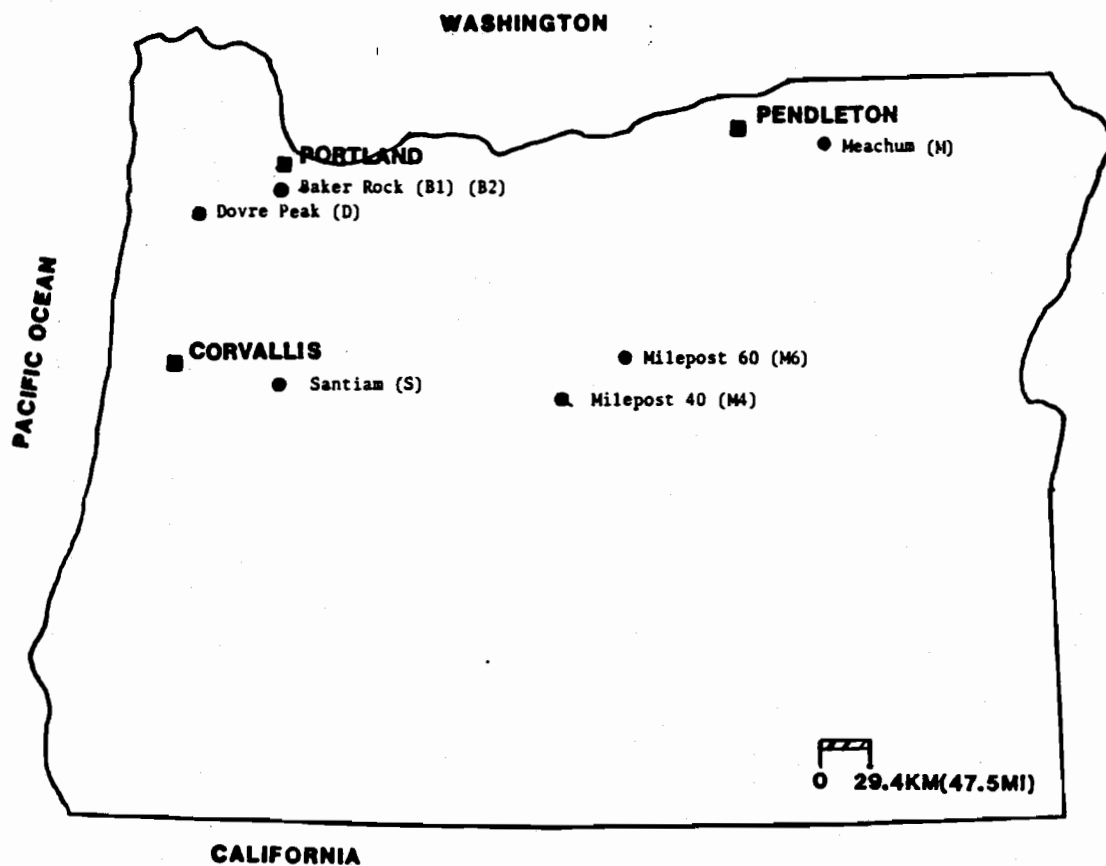


Figure 3.3. - Location of the Quarries Sampled for the DMSO Project.

Table 3.3. - Durability Summary for Quarries Utilized in Study.

	Sodium Sulfate		Oregon Aggregate		Los Angeles
	Soundness		Degradation		Abrasion
QUARRY	% Loss		Sediment	% Passing	
	Coarse	Fine	ht.	0.84mm (#20)	%
	Aggregate	Aggregate	cm	Sieve	Wear
Baker Rock					
Upper Bench(B1)					
Random	15.1	13.5	8.6	16.4	18.5
Vesicular	9.9	23.4	7.4	21.1	21.6
Olivene	4.6	15.9	3.3	15.4	20.8
Lower Bench(B2)	4.8	13.8	2.5	16.6	17.6
Meachum(M)					
Quarry	1.7	7.7	-	-	-
Ochoco MP60(M6)					
Westside	9.2	6.0	8.6	18.7	15.8
Mid-Top	4.6	9.9	2.8	13.5	16.4
Ochoco MP40(M4)	3.8	10.8			
Quarry			3.1	15.7	20.8
Santiam -Morse Pit(S)					
NE Corner	10.6	8.7	3.1	16.8	6.1
NW Corner	5.3	6.5	2.5	19.5	14.3
Dovre Peak(D)					
Quarry	23.1	24.1	-	-	-

Rock Quarry, located approximately ten miles south of Portland, Oregon. The quarry is privately owned and operated and along with other quarries in the same general area, provides the majority of the aggregate used in the south Portland area. The operator identified the rock on the upper bench as being the poorer quality material in the quarry. The rock in the upper bench is moderately to highly fractured, breaking into blocks ranging in size from cobbles to boulders. The section sampled consisted of two flow units, each about 48m (15 ft) thick, and separated by a 30 to 90mm (3 to 6 in.) layer of red paleosol. The rock at the bottom of the flow units is dense with less than 10 percent vesicles and increases to almost 50 percent near the top of the flow. The rock has undergone considerable weathering as evidenced by the presence of limonite infilling around the vesicles and the manganese oxide coating the rock.

Results of the petrographic analysis for the thin sections of rock taken from the quarries are shown in Table 3.4. The percent of primary and secondary minerals were determined by point counting using the procedure suggested by Best (16) after initially staining the thin section with a rhodamine-B dye to detect the smectite clay minerals. Szymoniak et al(17) found that the rhodamine-b was specific for smectite clays in rock thin sections. The upper bench has a smectite clay content ranging from 26 to 38 percent and a secondary mineral content between 41 to 54 percent. The average values of the mineral constituents are reflected in Table 3.4.

The low loss material originated from the Meacham Quarry, located 20 miles east of Pendleton, Oregon. The rock in the quarry

Table 3.4. - Petrographic Summary for the High and Low Loss Material.

Mineral	High Loss	Low Loss
	Upper Bench Baker Rock	Meachum
Plagioclase	42	36
Olivine	3	-
Pyroxene	1	15
Magnetite	8	11
Total Primary	54	62
Glass	1	-
Smectite	31	21
Iron Oxides	2	18
Green Alteration of Olivine	13	-
Total Secondary	46	38
# of Points Counted	645	605

is highly fractured and jointed into crude columns, which tend to break into small blocks ranging from 25 to 120mm (1 to 4in.). The fractured rock is very fine-grained and dense. Small vesicles (1 to 5mm(0.04 to 0.1in.)) comprise about 5 to 10 percent of the volume. As shown in Table 4, the Meacham quarry has a smectite clay content of 21 percent and a secondary mineral content of 38 percent. The majority of the secondary minerals present are palagonite; a weathering product of glass which eventually weathers into a smectite clay.

The difference in the DMSO losses between the two quarries is primarily a result of the groundmass size which influences the permeability. Rock from the Meacham quarry has a groundmass size of 0.05 to 0.1mm, while rock from the upper bench of Baker has a groundmass size of 2 to 3mm. The larger the size of the groundmass the greater the permeability of the rock matrix.

3.5 DISCUSSION OF RESULTS

The results of the Atterberg Limit tests are given in Table 2.5 for the Ca- Montmorillonite clay mineral mixed with water, ethylene glycol and DMSO, respectively. Also summarized in Table 2.5 are the results for DMSO mixed with two other clay types: kaolinite, and Na-bentonite, which were studied by Andrews et al (8).

Atterberg Limits are a simple method to assess the type and activity of a clay mineral. The liquid limit reflects the intensity of the net attractive forces between clay particles which, in turn, is related to the surface activity of the clay components (15). The liquid limit may be defined as the water content at which a soil has a shear strength of 2.0 to 2.5 kN/m² (40 to 50 psf.). The greater the

Table 3.5. - Atterberg Limits for Mixture of Clay Standards
with Water, Ethylene Glycol (EG) and DMSO.

Clay	Liquid	LL %	PL %	PI %	PI H ₂ O PI DMSO
Ca-Mont.	H ₂ O	118	37	81	-
	EG	88	71	17	-
	DMSO	92	n/p	n/p	-
Kaolinite ^A	H ₂ O	62	33	29	.53
	DMSO	105	50	55	-
Na-Bentonite ^A	H ₂ O	506	55	451	7.5
	DMSO	140	80	60	-

n/p - non-plastic

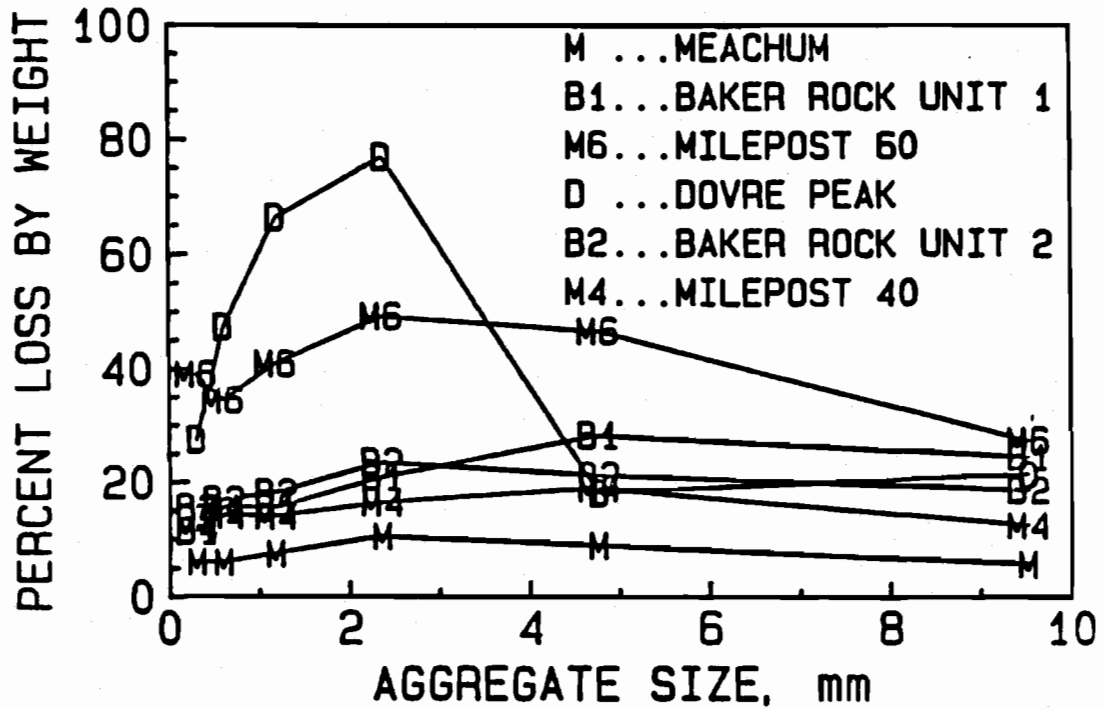
^A Reference 8

surfaces area and the intensity of the net attractive forces, (associated with highly active clays), the greater the spacing between particles and the higher water content at the liquid limit. The liquid limit values determined for clays mixed with DMSO and ethylene glycol instead of water are as expected: increased for kaolinite and substantially decreased for Na-bentonite and Ca-montmorillonite. The decrease in the liquid limit for mixtures of the highly active clays with DMSO or ethylene glycol is associated with their low dielectric constant, which increases Coulomb's force of attraction. The increase in Coulomb's attractive force increases the surface potential of the clay mineral and the concentration of the cations held near the surface. As the ion concentration near the clay surface increases, the thickness of the double layer is reduced, which in turn lowers the net attraction between clay particles. Thus, for the highly active clays the structure is more dispersed when mixed with DMSO (or ethylene glycol) as compared to mixing with water, which indicates that the held cations were solvated, thereby decreasing the net interparticle force and lowering the amount of liquid needed to maintain a shear strength of 2.5kN/m^2 .

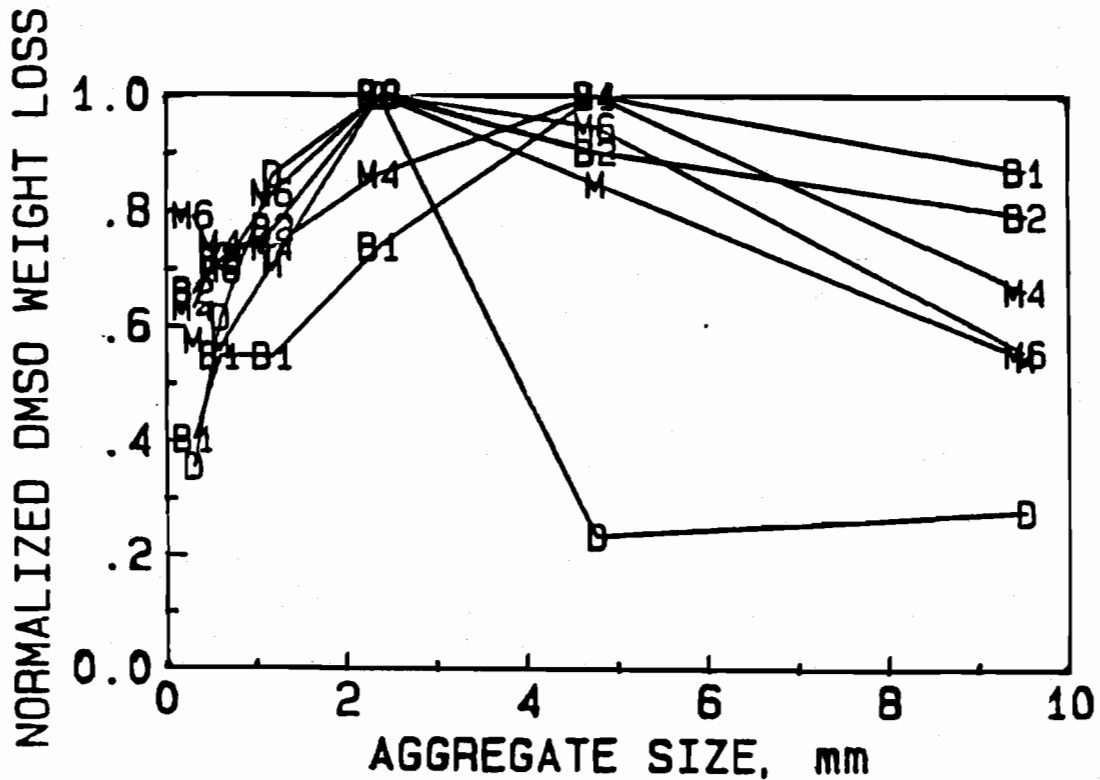
DMSO, however, had the opposite effect on kaolinite, and resulted in an increased interparticle force and an increase in the amount of liquid required to produce the shear strength at the liquid limit. Kaolinite is a 1:1 clay mineral, composed of alternating silica and octahedral sheets. The increase in the liquid limit was due to the reaction of the hydroxyl ion, held in the octahedral layer of the clay structure, with the anionic DMSO molecule. DMSO donates a hydrogen

molecule to the hydroxyl ion to produce water which is dehydrated upon oven drying. Also, as result of the attachment of the anionic DMSO molecule the net interparticle attraction force increases which raises the liquid limit of the material. Hiller (12) suggested that large bulky anions, like the hydroxyl ions, are solvated more by dipolar aprotic solvents like DMSO. Thus, for the kaolinite, DMSO bonds to the hydroxyl ion, thereby increasing the net repulsive force and forming water molecules which are dehydrated upon oven drying. Kaolinite was not found in any of the aggregate sampled, as expected, since it is thought to represent an end product in the geochemical reduction of the primary aluminosilicate minerals. Kaolinite may, however, be found in sedimentary or metamorphosed rocks.

Effect of Aggregate Particle Size. -- The effect of using different aggregate particle sizes in the DMSO test procedure for six quarries is shown in Figure 3.4a. Generally, it can be seen that for the high loss quarries, the effect of aggregate size is more important than for the low loss quarries. Also, it would appear that on the average the loss on the size range between 2.4mm (#8) and 4.8mm (#4) gives the highest percent loss. For this aggregate size range the textural distribution of the clays, or the planes of weakness, apparently the surface area increases the exposure of the aggregate to DMSO, but the increased surface area does not produce any additional loss since the planes of weakness have been removed by the crushing process. For particle sizes larger than 2.4mm, the surface area to volume is significantly lower, which reduces the area which DMSO can penetrate. In addition, these particle sizes still contain the planes of weakness and,



(a) - Percent Loss by Weight versus Aggregate Size.



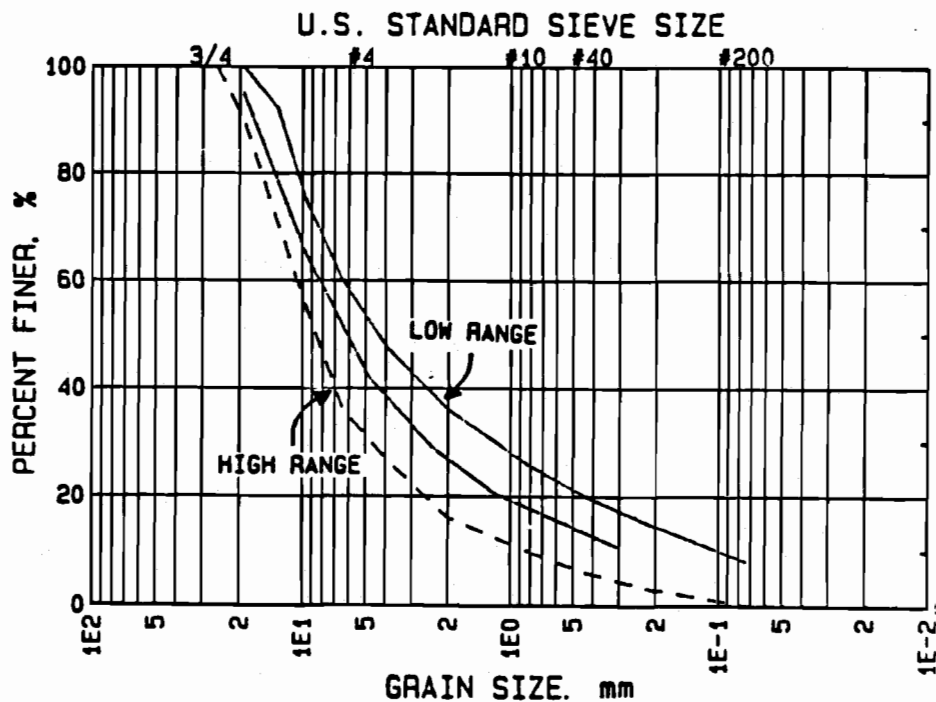
(b) - DMSO Loss Normalized to the Highest Loss versus Aggregate Size.

Figure 3.4. - Effect of the Size of Aggregate Used in the DMSO Immersion Test Procedure.

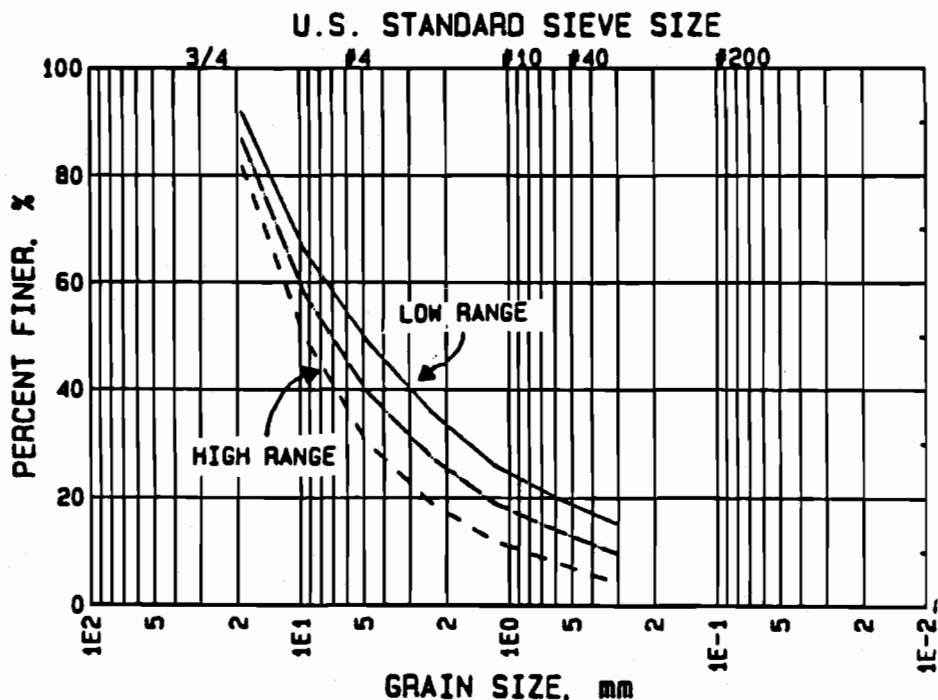
hence, the weight loss after immersion in DMSO tends to be constant as shown in Figure 3.4.

To further demonstrate that the aggregate in the size range of 2.4 to 4.8mm experiences the greatest DMSO loss, the losses in the various size ranges were normalized to the greatest loss reported for the quarry. Figure 3.4b shows that most of the quarries have the highest loss near the 2.4mm (#8) size and that above and below this size the loss decreases.

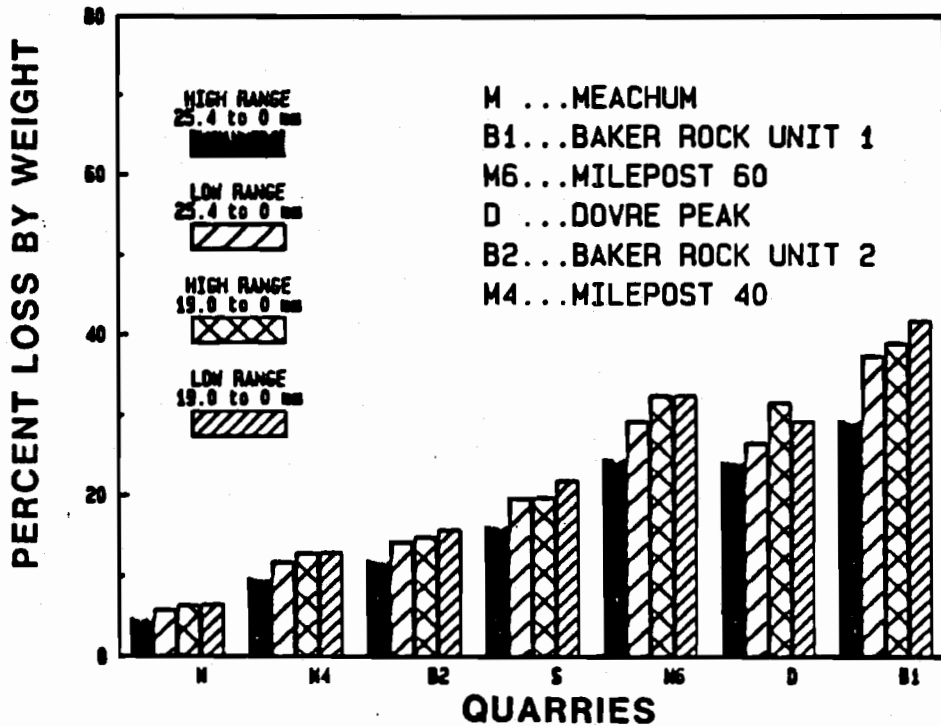
In the existing test procedure, a weighted DMSO loss is computed by multiplying the percent DMSO loss by the percent of the same size range of aggregate proposed for the field gradation. Thus, the weighted DMSO loss depends upon the gradation of the aggregate. The weighted DMSO loss was computed for the six quarries sampled for two typical aggregate specifications employed by ODOT, as illustrated in Figure 3.5. The figure defines the high, low and mid ranges used to calculate the weighted DMSO loss for the two aggregate gradations, 19 to 0 mm (3/4 to 0 in.) and 25.4 to 0 mm (1 to 0 in.). The ranges of the weighted DMSO losses for the selected quarries are shown in Figure 3.6a, using the low and high range from the gradation specifications for the 19.0 to 0 mm (3/4 to 0 in.) and the 25.4 to 0 mm (1.0 to 0 in.). From the results presented it is apparent that the effect of using different gradations is more pronounced in the high loss quarries, and that larger size aggregate gradation utilized (25.4 to 0 mm (1.0 to 0 in.)) results in a lower weighted loss. Figure 3.6b further illustrates the effect of using different gradations by comparing the average loss, the weighted loss for the two mid-range aggregate



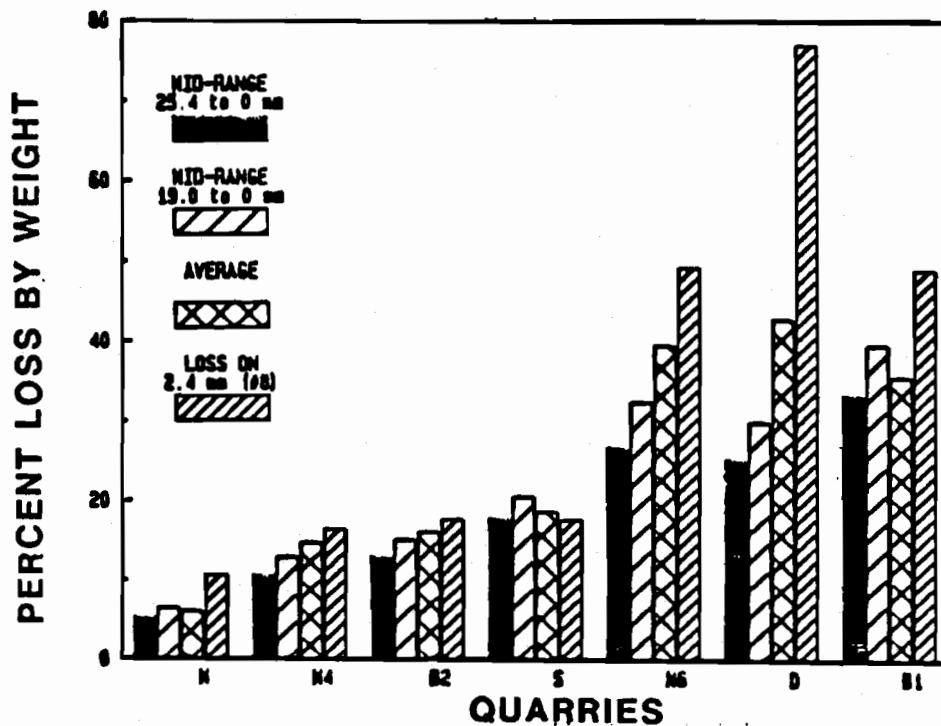
(a) - Gradation for the Aggregate Size 19.0 to 0mm (3/4 to 0in.).



(b) - Gradation for the Aggregate Size of 25.4 to 0mm (1 to 0in.).
 Figure 3.5. - Two Typical Aggregate Gradation Specifications Utilized by ODOT.



(a) - Comparison of the Weighted DMSO Loss for High and Low Aggregate Gradation Ranges.



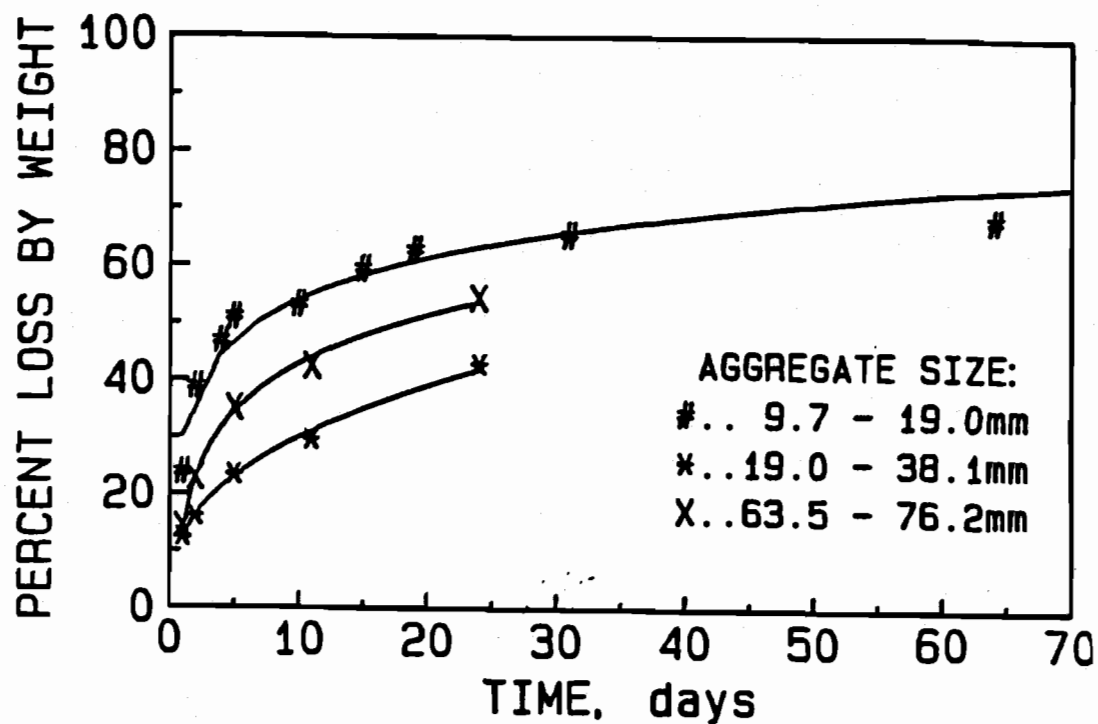
(b) - Comparison of the Average, the Weighted from the Mid-Range Aggregate Gradation and the 2.4mm (#8) Size.

Figure 3.6. - Comparison of the DMSO Losses Using Different Criteria for Two Aggregate Gradations Utilized by ODOT.

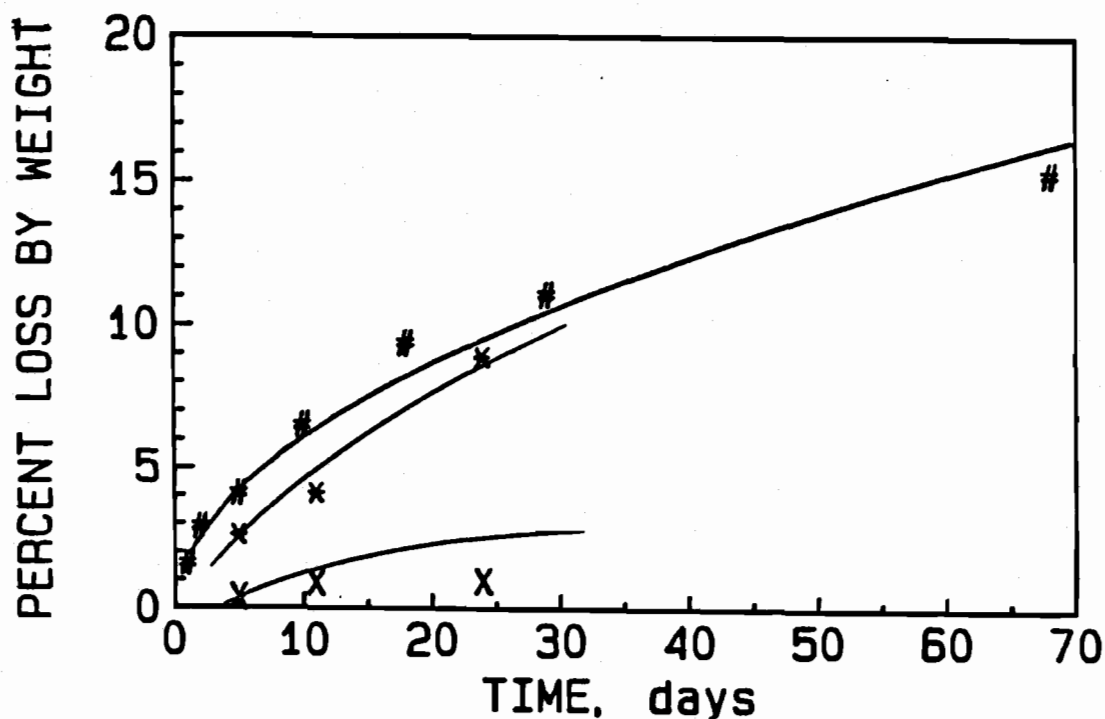
gradations, and the loss for the aggregate particle size between 2.4mm (#8) and 4.8mm(#4). The computed weighted DMSO loss for all of the quarries studied is lower than: 1) the average of the individual size ranges, and 2) the loss reported for the size range of 2.4mm (#8) to 4.8mm (#4). The results further support the use of one particle size range to reflect the highest loss after immersion in DMSO compared to the weighted loss or the average loss of all of the size ranges utilized.

Figure 3.7 presents the data collected from the study of the effect of immersion time in the DMSO solution for three aggregate particle sizes for the high and low loss material. The results further substantiate that the aggregate particle size has a significant effect on the loss after immersion in DMSO. The effect is more dramatic for the high loss material, where after five days of immersion, the loss can vary from 20 to 45 percent depending on the particle size used in the test. The trend indicated in the figure suggests that a smaller aggregate particle size be adopted for the standard test since it appears to achieve a higher loss in a shorter period of time.

Effect of Sample Weight. -- Figure 3.8 shows the effect of sample weight on the DMSO weight loss value. At the end of five days the loss can vary from 25 to 58 percent for the high loss material depending upon the sample weight. The low loss material was not sensitive to sample weight. For both the high and low loss material there is a difference in loss for a sample weight of 250 g compared to 2000g, however the difference between the 1000 and 2000g weight is small at the end of the five day immersion. This fact suggests



(a) - Percent Loss by Weight versus Time for the High Loss Material.

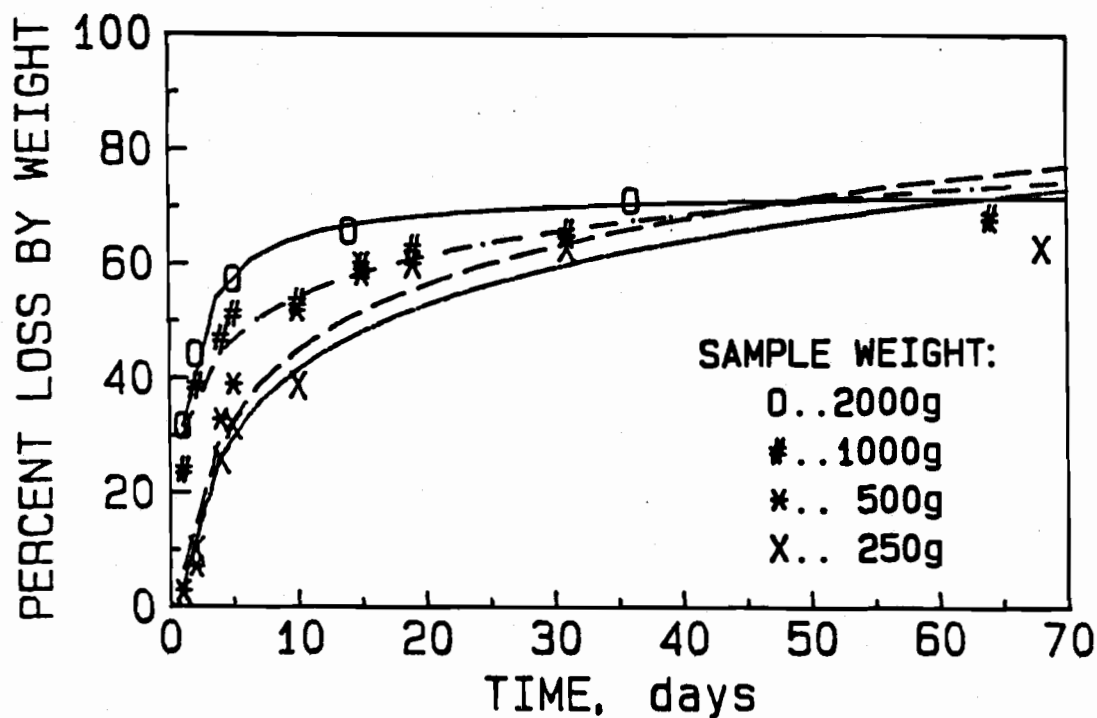


(b) - Percent loss by Weight versus Time for the Low Loss Material.
Figure 3.7 - Effects of Aggregate Particle Size Used in the DMSO test.

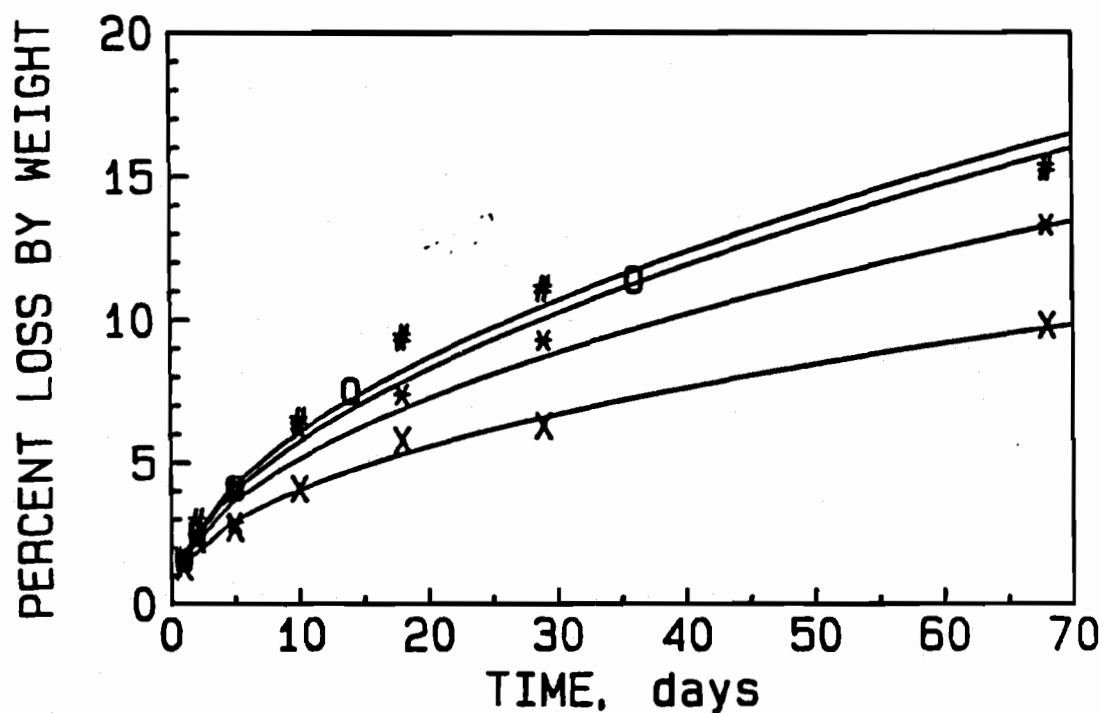
that a sample weight of 1000g can be adopted for the standard test.

Effect of Immersion Time. -- The effect of immersion time is also reflected in Figures 3.7 and 3.8. The results shown in both figures indicate that weight loss continues with time of immersion. After approximately seventy days the individual sample losses for the high loss material tend to approach the same limiting weight loss. At the end of five days immersion the weight loss has achieved 82 percent of the final limit, which suggests that if the rock is going to degrade, it will be apparent in the first five days. For the low loss material the weight loss increases at a decreasing rate and after five days of immersion the sample has only experienced 33 percent of the seventy day loss.

To further study the effect of immersion time and establish a standard time period, the rate of loss was calculated for the high and low loss material. The results of this study are shown in Figure 3.9. The data indicates that for the high loss material the rate of loss drops rapidly in the first ten days, after which the rate of loss substantially lowers. The pattern is similar for the low loss material, but to a much lesser degree. The data suggests that a 10-day immersion period should be adopted for the standard test. However, this length may be excessive since most of the degradation has taken place by the end of the fifth day. Recognizing that an acceptable weight loss criteria will be employed in conjunction with the standard test, it is believed that the degradation potential of the rock will be reflected after five days immersion in DMSO.



(a) - Percent Loss by Weight versus Time for the High Loss Material.



(b) - Percent Loss by Weight versus Time for the Low Loss Material.
 Figure 3.8. - Effects of Sample Weight on the DMSO Loss Using the Aggregate Size Range of 19.0-6.4mm.

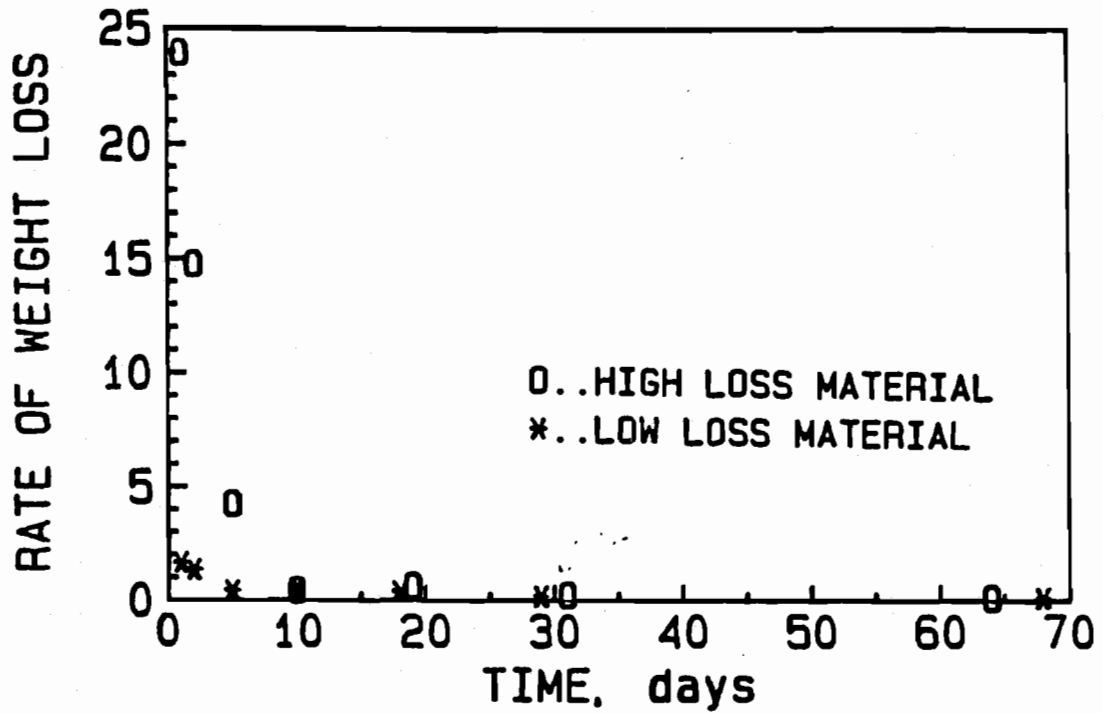


Figure 3.9. - Rate of Loss versus Time for the High and Low Loss Material Using a Sample Weight of 1000 g.

Effect of Container Geometry. - Two different types of containers were utilized to investigate the effect of container geometry. The first container was approximately cylindrical with dimensions previously noted and the second container was a flat pan. The dimensions of the flat pan were established so that no aggregate particles were in contact with one another. Figure 3.7a shows the results obtained for the round container and Figure 3.10 presents the weight loss data collected for the high loss material after immersion in the flat pan. The results from Figure 3.7a are superimposed on Figure 3.10 for comparison. Comparing the weight loss results for the two containers it may be noted that for all sample weights utilized the flat pan gave a higher loss. For the high loss material the effect of the container geometry results in a DMSO loss in the range of 25 to 50 percent for the round container compared to 45 to 65 percent in the flat pans. Although the weight loss was the greatest in the flat pans, the round container was adopted for the standard test since it is more suitable for covering, thereby reducing potential contamination and hydration of the DMSO solution.

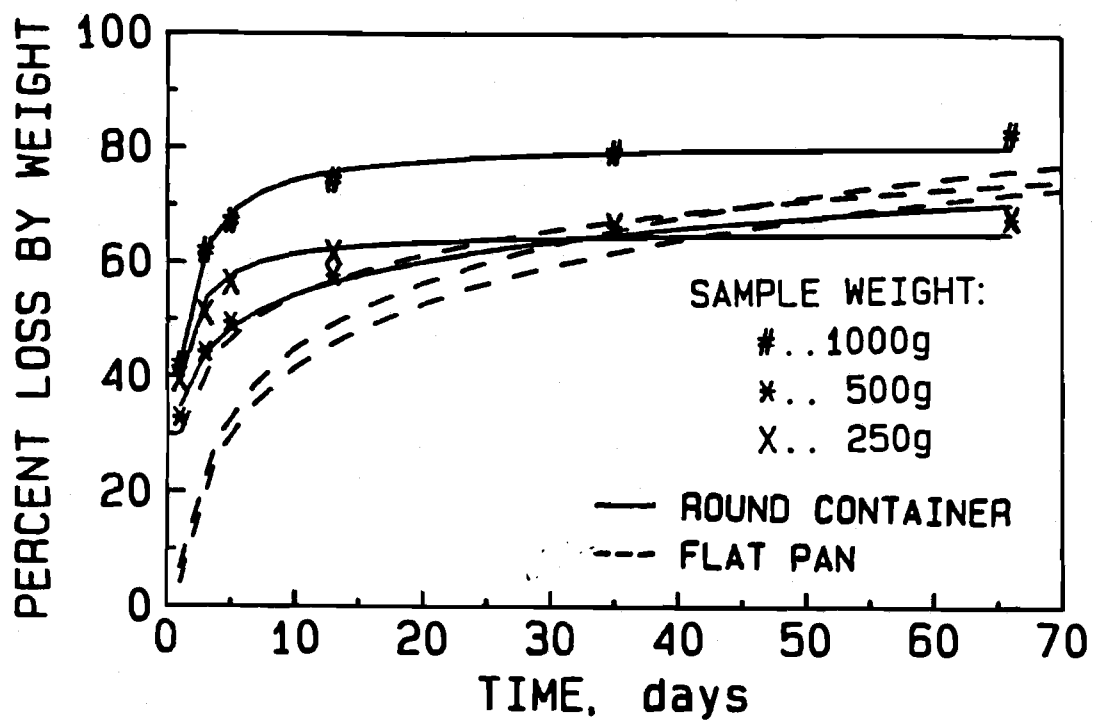


Figure 3.10.- Percent Loss by Weight versus Time for Samples Contained in a Flat Pan and in the Round Container.

3.6 RECOMMENDATIONS FOR A STANDARDIZED TESTING PROCEDURE

Based upon the results from this study the following DMSO Accelerated Weathering Test procedure is recommended:

- 1) Select a representative sample from the quarry,
- 2) Crush the material to sizes smaller than 37mm (1-1/2in.);
- 3) Separate the crushed aggregate by mechanical sieving so that particles in the size range of 2.4mm (#8) to 4.8mm (#4) are obtained; air dry the material;
- 4) Weigh 1000 ± 5 g of the material in the size range of 2.4mm(#8) to 4.8mm (#4), record the actual weight used to the nearest 0.1g; determine the moisture content of a representative air dried sample of the material to allow the initial dry weight to be determined;
- 5) Immerse the sample in a container containing a 100 percent solution of Industrial Grade DMSO. The container shall have equivalent dimensions of: 18.5 cm (7.25 in.) in height, a top diameter of 14.5 cm(5.75 in.) and a volume of 2750ml (93 oz.), The container must be capable of being sealed to reduce contamination and hydration. The aggregate in the container shall be covered with at least 25mm (1 in.) of solution. The temperature of the covered sample must be maintained at 21°C (70°F) $\pm 2^{\circ}\text{C}$ (5°F) for the duration of the test;
- 6) Remove the sample from the DMSO solution after a period of not less than 120 hrs or more than 128 hrs;
- 7) Rinse the samples in tap water using the 2.4mm (#8) sieve

to retain the unbroken pieces and oven dry the samples to a constant mass at 110 °C (230 °F);

- 8) Re-sieve the samples on the 2.4 mm (#8), and record the weight retained on the sieve;
- 9) Calculate the percent loss by weight;

Using this procedure Szymoniak et al (18) concluded that the limit for the DMSO loss for aggregates should be set at 22 percent. They also noted that DMSO may overreact and lead to a false indicator if zeolites and calcite are present in the rock.

3.7 SUMMARY AND CONCLUSIONS

DMSO is a powerful penetrant, and causes greater breakdown and disintegration of aggregates when compared to immersion of the aggregates in ethylene glycol or water. The use of DMSO in an accelerated weathering test for aggregates, simulating the chemical weathering of the mineral constituents of the rock, is desirable since the degradation potential will be reflected in a shorter period of time.

The penetration ability of DMSO, and thus the greater breakdown of the aggregates, is in part due to both the type and concentration of ions contained in the rock matrix. Since the DMSO compound is slightly anionic, it prefers and seeks out the cations that are held to clay minerals in the aggregate and, therefore, a greater loss or breakdown of an aggregate can be expected as the cation exchange capacity increases. Swelling clays, such as the clay mineral smectite, have high cation exchange capacities and, therefore, react with the DMSO compound.

Based on Atterberg Limit test results on clay standards it was

shown that DMSO substantially reduces the liquid limit of highly active clays. The reduction of the liquid limit owing to the lower dielectric constant of DMSO increases the attraction force between the clay surface and cations. Increasing the concentration of cations near the clay surface lowers the net interparticle attraction between clay particles which results in a reduction of the liquid limit. DMSO, however, has the opposite effect on kaolinite and results in an increased liquid limit. The increase in the liquid limit occurs owing to the anionic DMSO compound bonding to the hydroxyl ion by donating a hydrogen molecule to produce water which is dehydrated during oven drying. Understanding the interaction of DMSO with clay standards, and recognizing that basalts common in Oregon contain only swelling clays, immersion of the basaltic rock in DMSO should reflect the chemical degradation of basalt aggregates.

Based on the results of a test condition parameter study it was determined that the DMSO losses were influenced by the aggregate particle size, sample weight, container geometry, immersion time, and the aggregate gradation used to calculate a weighted average loss. The influence of the factors were found to be significantly different for aggregate material with a high DMSO loss, compared to aggregates with a low loss. For instance, depending upon the aggregate particle size used in the test the DMSO loss could range from as low as 22 percent to as high as 58 percent at the end of the five day immersion period.

From the results reported a procedure was recommended which utilized the crushed aggregate in the size range between 2.4mm to 4.8mm

(#8 to #4). The new procedure requires less material to be immersed and results in the greatest DMSO loss for the quarries considered in this study. Once the standardized test procedure is adopted the reliability of DMSO Accelerated Weathering Test should be further studied since it is apparent that DMSO is reacting with other minerals in addition to swelling type clays.

3.8 ACKNOWLEDGEMENTS

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4.0 RELIABILITY OF THE DIMETHYL SULFOXIDE (DMSO) ACCELERATED WEATHERING TEST TO PREDICT THE DEGRADATION OF AGGREGATES

by Tom Szymoniak, Ted S. Vinson and James E. Wilson

ABSTRACT: Dimethyl Sulfoxide (DMSO) has been proposed as an agent for use in an accelerated chemical weathering test owing to its ability to penetrate and dramatically expand the structure of swelling clay minerals in a rock matrix. In recognition of the need to establish the reliability of the DMSO Accelerated Weathering Test to predict the degradation potential of basaltic aggregates a research study was undertaken with the following objectives: 1) establish a correlation between DMSO Accelerated Weathering test results and the results of petrographic analysis which identify secondary minerals (e.g. swelling clays) that are known to lead to aggregate degradation, and 2) determine acceptable limits of the index parameter (i.e., percentage weight loss) obtained in the DMSO Accelerated Weathering Test. Based on laboratory test results from the aggregate samples obtained from eight rock quarries in Oregon, a correlation equation which relates the DMSO weight loss to results from petrographic analyses was established. The equation has a correlation coefficient of 0.99 indicating that an extremely strong relationship exists between DMSO weight loss and type, content, and textural distribution of minerals in the rock fabric. The correlation developed neglects the results from two quarries containing the minerals analcime and calcite. The reaction of DMSO with these minerals results in an erroneous indication of the presence of swelling clays. An acceptable weight loss limit for untreated aggregate utilizing the DMSO Accelerated Weathering Test

procedure was established at 22 percent. The limit is based upon correlations between the DMSO test results and quantitative measures of the type and content of deleterious minerals, and textural distribution of minerals in the rock fabric.

4.1 INTRODUCTION

In selecting an aggregate source to insure satisfactory performance in a pavement structure both the physical properties of the rock and the environment in which it is placed must be considered. The physical properties of an aggregate include surface texture, size, shape, gradation, strength, durability, and mineralogical composition (1). Environmental factors include weather, traffic, and the availability of water. Degradation, or disintegration of the aggregate in the pavement structure owing to the interaction of the rock properties with the environment is caused by mechanical and chemical weathering processes. Mechanical weathering processes result in the actual physical breakdown of a rock and are generally associated with the action of equipment during construction, the operation of traffic throughout the life of the pavement and freeze-thaw cycling. Chemical weathering of a rock, caused by the environment in which the aggregate is placed, results in the disintegration of the mineral components of the rocks.

The physical properties of a rock are typically assessed through standard laboratory tests in which the aggregates produced must meet the criteria established by the governing agency. For example, the Oregon Department of Transportation (ODOT) uses the Los Angeles Abrasion test (ASTM C131), the Sodium Sulfate Soundness test (ASTM C88), and

the Oregon Aggregate Degradation test (Oregon TM208) to assess the durability characteristics of an aggregate. These tests tend to reflect the mechanical degradation potential but do not simulate the chemical degradation common to some types of basaltic rocks in Oregon. In fact, the basalts generally have good mechanical durability properties and will usually pass the standard aggregate specifications. However, when some types of basalts are incorporated into a pavement structure as the base course aggregate or in the asphalt concrete, field experience suggests that the rocks degrade due to chemical weathering of the minerals present in the rock. Specifically, the basalts contain swelling clays, such as smectites, which, in the presence of water, expand and lead to the eventual failure of the pavement.

As a result of the inability of the standard aggregate tests to predict the durability of basalt aggregates the Materials Section of the Federal Highway Administration (FHWA) Western District Federal District (WDFD) developed an immersion test in Dimethyl Sulfoxide (DMSO) to assess the chemical degradation potential of an aggregate source. DMSO simulates the chemical degradation of an aggregate by reacting with the deleterious minerals (smectite clays) contained within the rock matrix.

OBJECTIVE AND SCOPE

DMSO has been used in aggregate durability testing since the late 1970's. However, the applicability of the DMSO test to reliably predict the degradation characteristics of aggregates and the meaning of the index parameter determined is not well understood. In

recognition of this situation a research study was undertaken to: 1) establish a correlation between DMSO Accelerated Weathering test results and the results of petrographic analyses which identify secondary minerals that are known to lead to aggregate degradation, and 2) determine the acceptable limits of the index parameter (i.e., percentage weight loss) obtained in the DMSO Accelerated Weathering Test. The results from this work are reported herein.

The paper is divided into four major areas associated with: 1) the evolution of the DMSO Accelerated Weathering Test, 2) a discussion concerning the mechanism by which DMSO penetrates and its interaction with clay minerals, 3) a review of the deleterious minerals found in road aggregates, and 4) a discussion and interpretation of the laboratory results obtained from the study of eight quarries in the state of Oregon.

4.2 EVOLUTION OF THE DMSO ACCELERATED WEATHERING TEST

Use of accelerated weathering tests to simulate the chemical degradation of aggregates began in the late 1960's when the Army Corps of Engineers proposed an ethylene glycol immersion test to assess the quality of basaltic rock. Ethylene glycol has a history of use in X-ray diffraction analysis as an aid in the identification of swelling clays. The X-ray diffraction pattern of swelling clays (i.e., smectites) increases from the normal basal spacing of approximately 14A to 17A after treatment in a saturated ethylene glycol environment (2). Considering this fact it was reasoned that the swelling pressure created by the expansion of the clay structure in a rock matrix would result

in the deterioration and breakdown of the rock and, therefore, immersion of aggregates in ethylene glycol would provide an ideal accelerated weathering test.

The original procedure proposed by the Corps utilized a ten rock sample of aggregate in the size range of 9.5mm (3/8 in.) to 25.4mm (1 in.). The rocks were selected from a representative sample of the aggregate source and were soaked in ethylene glycol for a period of ten days, at which time the number of rocks observed to crack, spall, or disintegrate were recorded (3). This value was reported as an index to relate the quality of the aggregate source; if four or more rocks cracked, spalled, or disintegrated the quarry was deemed unacceptable.

In the mid-1970's the ten rock test was modified by the FHWA Region 10 Materials Section by substituting DMSO for ethylene glycol. The substitution was based on a study by Van Atta and Ludowise (4), in which they noted that DMSO caused a greater expansion of swelling clay minerals as compared to ethylene glycol. The difference in magnitude between the two suggested that immersion of the aggregates in DMSO would lead to more severe breakage in a shorter period of time. In comparative testing using both chemicals on the same aggregate source, immersion in DMSO typically resulted in an additional 2-4 rocks breaking down (5). The ten rock procedure was later modified in 1978 by incorporating a procedure which reported a weighted loss after a five day immersion period in DMSO. The procedure adopted was similar to the Sodium Sulfate Soundness test in which greater sample weights are utilized for increasing aggregate size fractions. Szymoniak et al

(5) found that the weighted loss reported by this procedure was affected by many factors including the aggregate particle size, sample size, and immersion time. They recommend a standard testing procedure using 1000g of aggregate (in the size range of 2.4mm(#8) to 4.8mm (#4)) immersed in a standard container holding a 100 percent solution of DMSO for a period of 5 days.

The data collected by Szymoniak and Vinson (6) and Miles (7) indicated that DMSO reacts with minerals other than swelling clays, which may or may not be deleterious. Before continuing with a discussion of the deleterious minerals with which DMSO interacts, it is important to understand the properties of the chemical and it's interaction with clay minerals. This discussion is presented in the following sections.

4.3 CHEMICAL PROPERTIES OF DMSO

DMSO was first brought to the public's attention in the early 1960's owing to its potential applications in medicine. It has since been the subject of many articles in both the medical and scientific professions(8). Despite intensive research and the number of applications that the chemical has been associated with over the past decade indicating its medical value, DMSO has not been approved for general public usage. The Federal Drug Administration (FDA) banned the use of DMSO and testing of the chemical on humans in 1965, after clinical studies using laboratory rabbits showed that their eye lenses became distorted upon repeated contact with DMSO(8). The FDA later approved the use of DMSO in 1974 for only one condition, a rare and

painful bladder disease known as interstitial cystitis (3). However, industrial use of the chemical has continued since DMSO is an excellent solvent and penetrant.

The penetrating ability of DMSO across membranes is due to its interaction with ions on opposing sides of the membrane. Franz and Van Bruggen(9) proposed two mechanisms by which DMSO may improve passage through a barrier. The first mechanism is related to the osmotic pressure created by an electric potential between charged ions, which provides for a non-selective increase in the permeability of the barrier. The second mechanism is due to the interaction between DMSO and ionic species on the opposite side of the membrane. Szymoniak et al (5) suggested that the mechanisms for the penetration of DMSO could be compared to the increase in permeability of a saturated clay soil resulting from treatment by the electro-osmosis process. The electric potential, which aids in increasing the permeability of the membrane, is created by the attraction of the anionic DMSO compound to cations held on the opposite side of the membrane. DMSO, favors cations since it is slightly anionic owing to the attachment of the sulfur molecule. The attached sulfur molecule also shields the interaction of the dipoles with anions. Therefore, dissolved anions and anionic surfaces are relatively free from the interaction with DMSO while cations become solvated. Also, as the concentration of cations increase the ability for DMSO to penetrate across a membrane increases owing to the attraction of two unlike charges.

Besides interacting with cations DMSO acts as a hydrogen donator

to chemical reactions. Hiller (10) suggested that large bulky anions (i.e. the hydroxyl ion) are solvated by dipolar aprotic compounds like DMSO by donation of a hydrogen molecule. The ability of DMSO molecule to seek out cations and act as a hydrogen donator are the two mechanisms by which DMSO interacts with clay minerals. Swelling clays typically have high cation exchange capacities, consequently, DMSO will solvate the cations held on the mineral surfaces. Kaolinite, a non-swelling clay, is composed of hydroxyl ions on the exterior, which can also be solvated by the DMSO molecule.

4.4 INTERACTION OF DMSO WITH CLAY MINERALS

Szymoniak et al (5), and Andrews et al (11) employed Atterberg Limit tests to study the electrostatic interaction of DMSO with swelling and non-swelling clay minerals. Generally, they found that the liquid limit (i.e. the water content at which the soil has a shear strength of 2.0 to 2.5 kN/m² (12)) for Na-montmorillonite or Ca-montmorillonite, was substantially reduced when DMSO was substituted for water in the test. Szymoniak et al (5) explained the results by comparing the dielectric constants of the two fluids. Water has a dielectric constant of approximately 80 @ 20°C while DMSO has a value of 49. The dielectric constant influences the attraction of cations to the clay surface. Recalling that the electrostatic attraction force between two charged particles can be calculated using Coloumb's equation, and that the dielectric constant appears in the denominator, then the effect of lowering the dielectric constant is to increase the attractive force between charged particles. Specifically, when DMSO is mixed with a clay

mineral, cations are more easily attracted to the anionic clay surface. This increases the concentration of the cations held near the clay surface, and decreases the double layer thickness. As the cations are held much tighter to the clay surface the net interparticle attraction force between clay particles decreases. Therefore, lowering the dielectric constant decreases the quantity of fluid required to maintain the same shear strength at the liquid limit, since the liquid limit test is a measure of the net interparticle attraction force.

DMSO, however, had the opposite effect on kaolinite when substituted for water in the Atterberg Limit test and resulted in an increase in the liquid limit. The increase in the liquid limit was due to the reaction of the hydroxyl ion, held in the octahedral layer of the clay structure, with the anionic DMSO molecule. DMSO donates a hydrogen molecule to the hydroxyl ions to produce water which is dehydrated upon oven drying. Also, as a result of the attachment of the anionic DMSO molecule the net interparticle attraction force increases which raises the liquid limit of the soil.

In summary, the interaction of DMSO with clay minerals is a result of two processes. First, DMSO seeks out and solvates the cations which are held on the mineral surfaces. Secondly, DMSO dehydrates large bulky anions such as the hydroxyl ion by donating a hydrogen ion. Considering the results of the Atterberg Limit tests it may be noted that DMSO molecules react with both non-swelling and swelling clays. The non-swelling minerals which DMSO was found to react with may or may not cause the pavement structure to degrade and consequently, immersion

of the aggregates containing these minerals in DMSO may result in a false indication of field performance.

4.5 DELETERIOUS MINERALS IN ROAD AGGREGATES

A mineral is a substance with a definite chemical composition, an orderly structural arrangement, naturally occurring, and inorganic(13). Mineral constituents that are typically found in igneous rocks can be divided into four broad groups: primary minerals, glass, deuteric and secondary minerals. Primary minerals are the original constituents of a rock which crystallized from the magma. Some of the primary minerals found in basalts include plagioclase, pyroxene (augite), amphibole (hornblende), and magnetite(4).

The second group is glass, which is formed from the rapid cooling, or quenching of the molten rock. It has little internal ionic structure and varies widely in chemical composition (13). Glass tends to be highly susceptible to chemical weathering due to the lack of structure.

The other two groups, deuteric and secondary, are associated with the alteration of the primary minerals and glass. Deuteric alteration occurs during the crystallization process and results from the chemical reaction of hot water or gases with the minerals (14). Secondary minerals are formed through the weathering of minerals under temperature, pressure, and/or chemical conditions at, or near the earth's surface (13). Some secondary minerals that can be found in basalts include iron oxides, zeolites, and calcite (4).

Clay minerals are formed by the deuteric alteration and chemical weathering of the primary minerals and glass which results in the

formation of smectite clays and palagonite (14). Smectite clays are produced through the alteration of plagioclase, and have high swelling potential and high cation exchange capacities. Palagonite is an alteration product of caused by the oxidation and/or devitrification of basaltic glass; it can further alter to a smectite clay.

The actual content of these secondary minerals and clays are determined by petrographic analyses. For many years it was believed that the percentage of the secondary mineral content in a rock could be used to assess the degradation potential of an aggregate source. For instance, in 1955 Scott (15) determined that 0 to 20 percent secondary minerals in a fine aggregate will have little effect on pavement performance; 20 to 35 percent will produce some failures and borderline performances; and above 35 percent will almost certainly produce failures. However, in 1974 Van Atta and Ludowise (4) concluded that the durability of aggregates could not be determined solely by the percentages of weathering products found in the rocks. Durability was related to certain combinations of secondary minerals present in the rock and the textural distribution of these minerals in the rock matrix. Specifically they noted smectite clays and the mineral palagonite as being detrimental, and further indicated that an aggregate with a "stained" smectite clay content greater than 10 percent would result in poor pavement performance. Higgs (16) in an independent study conducted during 1976 found that as little as 11 percent of discrete montmorillonite was sufficient to cause basalts to degrade. He also suggested that the quality of a rock could be further assessed by noting

the stage in the weathering of volcanic glass. In his study Higgs found that volcanic glass goes through four stages as it weathers into smectite clay. In the first stage the rock contains clear unaltered glass; palagonite is formed in the second stage due to the release of iron oxides; the third stage results in devitrification of montmorillonite and the palagonite develops a scaly spherulitic appearance; and the fourth stage leads to the formation of distinct discrete montmorillonite. He further states that the concern for an aggregate's susceptibility to slake begins in the third stage with the development of montmorillonite.

Poor performance of aggregates in service is associated with the presence of swelling clay minerals (i.e. smectites), and the secondary mineral palagonite. Based upon the results of Van Atta and Ludowise(4), the use of a percent secondary mineral content or the percent clay content may not provide a good indicator of aggregate performance. These mineral contents should be augmented with knowledge of their textural distribution in the rock matrix. In recognition of this situation Cole and Sandy(17) proposed a secondary mineral rating system, which incorporates the type of deleterious mineral, and its' content and textural distribution within the rock fabric. The secondary mineral rating (RSM) of basaltic rocks is expressed by the following equation:

$$RSM = [(P \cdot M)] \cdot T_r \quad (1)$$

in which,

P = percentage of the secondary mineral present in the rock,

M = a numerical rating of the secondary minerals present from
least deleterious (2.0) to most deleterious (10.0),

T_r = a textural distribution factor.

Table 4.1 presents the numerical values for the mineral rating factor as suggested by Cole and Sandy. The secondary minerals in the table range from calcite (least deleterious) to smectite (most deleterious). Cole and Sandy further noted that zeolites may be more deleterious than indicated in Table 4.1. Table 4.2 shows the numerical values assigned for the textural rating factor which accounts for the distribution of the minerals within the rock matrix.

The RSM method to determine the potential degradation of an aggregate source requires that a thorough petrographic analysis be conducted, which is the most accurate method to evaluate the presence (or absence) of deleterious minerals in an aggregate source. However, petrographic analysis requires a trained petrologist, capable of identifying the primary and secondary minerals, and the use of a thin section of rock cut from the parent rock, which may or may not be representative of the aggregate produced. The rating procedure also requires a considerable amount of time to complete. Due to the disadvantages of the Secondary Mineral Rating system it is desirable to have a reliable accelerated weathering test, that would reflect the secondary mineral content and its textural distribution in the rock

Table 4.1. - Mineral Rating Factors (M)(after Cole and Sandy (17)).

Mineral Rating (M)		Mineral
Least Deleterious	2.0	Calcite, white micas (muscovite) and sericite)
	3.0	Kandites, chlorites, vermiculites, zeolites, hydrous micas (including illites), brown micas, (phlogopite and biotite)
	5.0	Swelling chlorite
	5.8	85% swelling chlorite: 15% smectite
	7.2	55% swelling chlorite: 45% smectite
	8.0	Iddingsite
Most Deleterious	10.0	Smectite

Table 4.2 - Textural Rating Factors (T) (after Cole and Sandy (17)).

	Textural Rating (T)	Texture (Occurrence of Secondary Mineral)
Least Deleterious	0.3	Partial alteration of phenocrysts (up to 50%)
	0.3	Incomplete vesicle filling (up to 50%)
	0.4	Complete alteration of phenocrysts (more than 50%) e.g. iddingsite after olivine
	0.5	Homogeneous scattered distribution in matrix
	0.6	Large irregular matrix patches (1 to 5 mm) (including filled vesicles)
	0.7	Irregular matrix patches minor interconnections
	1.0	Irregular partly connected patches in the matrix
Most Deleterious	2.0	Fine interconnected vein networks or patches (0 to 30 mm apart)

matrix. The DMSO Accelerated Weathering Test offers potential if the acceptable limits of weight loss are established.

4.6 METHODS OF STUDY

Two tasks were undertaken to establish the reliability of the DMSO Accelerated Weathering test and the acceptable weight loss. Under the first task the secondary mineral rating system, as previously discussed, was used to develop a correlation between the DMSO Accelerated Weathering test and the results from the rock thin section analysis. In developing the correlation between the DMSO loss and the Secondary Mineral Rating eight quarries were sampled in the state of Oregon. Figure 4.1 shows the location of the quarries. The quarries were selected to represent different sources of aggregate utilized throughout the state of Oregon. In addition to the DMSO test, the potential use of a methylene blue titration procedure, the Clay Index test (18), was evaluated. The Clay Index test was developed by Sameshima (18) to be a simple method of assessing the quality of an aggregate source.

The second task performed for the study was the determination of acceptable DMSO weight loss limit. This task was accomplished by considering performance criteria associated with the secondary mineral rating system. The secondary mineral rating system was used owing to the lack of good field data regarding the performance of aggregates produced from the quarries selected for the study.

Petrographic Analysis. -- A complete petrographic analysis was conducted for the eight quarries selected. The analysis consisted of the determination of the percentage of primary and secondary minerals

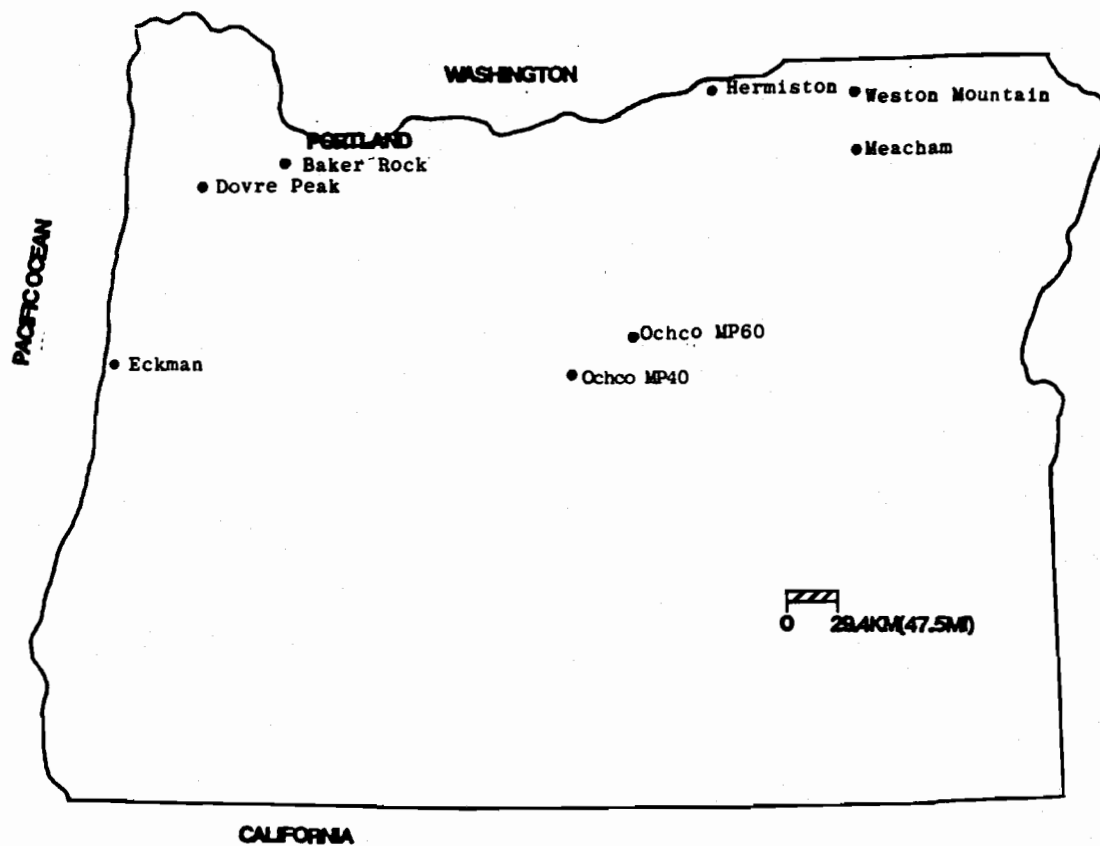


Figure 4.1. - Location of the Quarries Sampled for the DMSO Project.

staining the thin section with a rhodamine-b dye. Szymoniak et al (19) found rhodamine-b to be specific for smectite clays in thin sections of rock. A more detailed description of the procedure adopted for point counting and staining is given by Van Atta and Ludowise (13), Szymoniak and Vinson (6), and Szymoniak et al (19).

From the initial quarries sampled for the study, five were selected for additional testing purposes using the standard durability tests employed by ODOT. The results from the petrographic study are shown in Table 4.3; a summary of the durability test results for the five quarries is presented in Table 4.4. The following is a geologic description and a brief discussion concerning the performance of aggregates produced from the quarries.

Baker Rock Quarry.— The Baker Rock quarry was divided into three separate units, each having different geologic characteristics. The first unit was the upper bench (B1), the second unit was the lower bench (B2), which is now a mid bench since a third unit (B3) was opened during the summer of 1985. The operator of the quarry identified the rock on the upper bench as being the poorer quality material of the three units. The second unit was identified as being the good quality material and was the primary source of the aggregates used for the production of asphaltic concrete. The rock in the upper bench (B1) is moderately to highly fractured and weathered, breaking into blocks ranging in size from cobbles to large boulders. Due to the variability of the rock in the upper bench, the samples were split into three groups: random, olivine, and vesicular. In general, the

Table 4.3. - Petrographic Summary For Quarries Sampled.

Quarry	Plagioclase	Olivine	Pyroxene	Magnetite/ Ilmenite	Glass	Smectite Clay	Iron Oxides	Sepiolite
Baker Rock								
Unit #1 (B1)	42	3	-	8	1	31	2	13
Unit #2 (B2) (unoxidized)	37	5	3	10	-	31	4	10
Unit #2 (B2) (oxidized)	52	1	19	7	-	3	18	-
Unit #3 (B3)	38	1	3	7	-	28	12	11
Meacham (M) Quarry	36	-	15	11	-	21	18	-
Ochco MP40 (M4) Quarry	55	-	14	6	5	20	-	-
Ochco MP60 (M6) Mid-Top	54	-	9	7	-	30	-	-
* Dovre Peak (D) Upper Unit	39	-	22	4	6	3	25	-
Lower Unit	43	-	23	4	4	7	19	-
Crusher	18	6	17	9	-	39	11	-
Weston Mtn. (W) Quarry	36	-	19	11	-	21	17	-
**Eckman (E) Quarry	56	10	-	4	-	30	-	-

* Reference 22

** Reference 21

Table 4.4. - Durability Test Summary for Quarries Sampled.

QUARRY	Sodium Sulfate		Oregon Aggregate		Los Angeles
	Soundness		Degradation		Abrasion
	% Loss		Sediment	% Passing	
	Coarse	Fine	ht.	0.84mm (#20)	%
	Aggregate	Aggregate	cm	Sieve	Wear
Baker Rock					
Upper Bench(B1)					
Random	15.1	13.5	8.6	16.4	18.5
Vesicular	9.9	23.4	7.4	21.1	21.6
Olivine	4.6	15.9	3.3	15.4	20.8
Lower Bench(B2)	4.8	13.8	2.5	16.6	17.6
Meacham(M)					
Quarry	1.7	7.7	-	-	-
Ochoco MP60(M6)					
Westside	9.2	6.0	8.6	18.7	15.8
Mid-Top	4.6	9.9	2.8	13.5	16.4
Ochoco MP40(M4)	3.8	10.8			
Quarry			3.1	15.7	20.8
Santiam -Morse Pit(S)					
NE Corner	10.6	8.7	3.1	16.8	6.1
NW Corner	5.3	6.5	2.5	19.5	14.3
Dovre Peak(D)					
Quarry	23.1	24.1	-	-	-

rock in the upper bench had the highest clay content and the lowest durability of the quarries selected for the study. The thin section analysis showed a primary mineral content ranging from 53 to 56 percent, and a secondary mineral content ranging from 39 to 46 percent. The smectite clay content ranged from 26 to 38 percent.

The rock in the second unit (B2) was unfractured, uniformly dense, and very fine-grained. At the time the quarry was sampled two distinct weathering patterns were identified. One section had been oxidized and weathered by groundwater flow. As a result of this alteration process some of the zones are fresh with low clay contents. The unoxidized section has, however, undergone the same degree of weathering as in the upper unit and, therefore, it has clay contents which are very similar. The results from the durability tests reflect the mixture of these two dissimilar materials.

The rock in the third unit (B3) has also undergone chemical weathering due to the appearance of the manganese oxide coating. Thin section analysis revealed a higher percentage of iron oxides, and the size of the groundmass tended to be smaller than those identified for the other levels. As the size of the crystals become smaller the permeability of the rock decreases which then effects penetration of DMSO into the rock matrix.

Meacham Quarry. -- Rock from the Meacham (M) quarry is highly fractured and jointed into crude columns, which tend to break into small blocks. The fractured rock is very fine-grained and dense. Thin section analysis showed the rock to be comprised of 62 percent primary and 38

percent secondary minerals. The deleterious mineral palagonite was identified in the thin section and its' percentage was included in the iron oxide count, since it was difficult to distinguish between the two. The palagonite was in the third stage of weathering to a smectite clay, which most likely accounts for the high clay content determined. X-ray diffraction analysis identified an interstratified clay mixture of smectite and chlorite.

The Meacham quarry had the lowest weight loss after immersion in DMSO. The low loss after five days was attributed to the small size of the groundmass which effectively decreased the permeability of the rock. The quarry has been used extensively for paving projects along Interstate - 84. The most recent use was a recycling project, in which the new pavement bleed severely after construction and had to be re-surfaced the following year.

Ochoco Highway Quarries - Milepost 40 and 60. Rock in the Milepost 40 (M4) quarry is fine-grained and non-vesicular. Many of the fractures are filled with chalcedony, a micro-crystalline variety of quartz. Minor amounts of calcite and iron oxides occur in the fractures as well. In thin section, the rock had 74 percent primary minerals, 5 percent glass, and 20 percent smectite clays. The glass found in the thin section was still in the second stage of weathering to a smectite clay.

Rock in the Milepost 60 (M6) quarry is jointed into columns which are generally unfractured and seamed with veins of chalcedony and calcite. Weathering along the westside was thought to be more

pronounced due to the visible alteration and the coating of a clay weathering rind. In thin section analysis the rock on the westside had 70 percent primary and 30 percent secondary minerals. Under high magnification the plagioclase crystals showed replacement by calcite. Aggregate from the Milepost 60 quarry has performed satisfactorily when incorporated into an emulsified asphalt overlay. Since this was the first use of the quarry no other performance data is available.

Dovre Peak. — Dovre Peak quarry was originally studied by Van Atta and Ludowise in 1974 (4). Rock exposed on the quarry face occurs in two different basaltic units: an upper unit and a lower unit. In thin section Van Atta and Ludowise determined the upper unit to have 65 percent primary and 28 percent secondary minerals and 6 percent glass. The lower unit had 70 percent primary and 30 percent secondary minerals with 4 percent glass. The samples employed for the durability tests were obtained from the crusher site. Rock from this area has a secondary mineral content of 50 percent and a smectite clay content of 39 percent. The high clay content is reflected in the high DMSO loss and the low durability properties. Aggregate from the Dovre Peak quarry was first used in the base course layer and asphaltic concrete mix for a 22-mile timber access road in the Oregon Coast Range. Within one year after construction the pavement failed due to degradation of the aggregate in the base course. Aggregate from the quarry has since been used in a experimental test section for an open-graded emulsified asphaltic concrete. Ten years after being in service the performance of the pavement is comparable to that of a dense graded asphaltic

concrete pavement built with good quality aggregates(20). Aggregate from the Dovre Peak quarry had the highest DMSO loss of the quarries considered in the study.

Weston Mountain Quarry. -- Rock in the Weston Mountain quarry is fine-grained with small vesicles comprising part of the rock volume. It is also well fractured and weathered on exposed surfaces. Thin section analysis determined the primary and secondary mineral content to be 62 percent, and 39 percent, respectively, with smectite clays comprising one half of the total secondary mineral content. Minor amounts of palagonite were also identified in thin section to be in the third stage of development to a smectite clay.

Eckman Creek Quarry. -- Rock from the Eckman Creek quarry is representative of a marine basalt, and was identified by Clemmons (21) as being a source of marginal aggregate on the Oregon Coast. The rock on the quarry face is unfractured, vesicular and porphyritic and crudely jointed into columns. In thin section the primary mineral content was determined to be 70 percent, and the combination of smectite clays and glass comprise the remainder of the rock volume. The glass which was found in the thin section was largely altered to palagonite. In addition to identifying the clay minerals, the X-ray diffraction also identified the presence of analcime, a sodium-aluminum silicate which can be classed with zeolites. Zeolites have high cation exchange capacities, similar in magnitude to swelling clays. Rock from the Eckman Creek quarry had the second highest DMSO loss of the quarries considered, even though Clemmons (21) found it to have generally

acceptable mechanical durability properties.

Hermiston Quarry. — The Hermiston quarry is a homogeneous river run gravel source which has been used extensively for asphaltic concrete pavements on Interstate - 84. Thin section analysis showed the source to have 61 percent primary and 39 percent secondary minerals. Interstitial palagonite was found in the thin section and the mineral was in the third stage development to a smectite clay. The X-ray diffractogram identified the presence of smectite clays and paragonite.

Having identified the aggregate material used in the study, it is appropriate to present the test methods employed to aid in the establishment of the acceptable limits. Two procedures will be discussed: the DMSO Accelerated Weathering Test and the Clay Index Test.

DMSO Accelerated Weathering Test. -- The DMSO Accelerated Weathering Test involved crushing representative samples of rock material from the quarries and separating the crushed aggregate by mechanical sieving. The DMSO immersion test was conducted in accordance with the standard procedure given by Szymoniak et al (5). A 1000g sample of air-dried aggregates in the size range of 2.4mm (#8) to 4.8mm(#4) was immersed in a container of 100 percent concentration DMSO (Industrial Grade)for a period of five days in a 21°C (70°F) temperature controlled environment. At the end of the immersion period the samples were removed, rinsed in tap water, and oven dried to a constant mass at 110°C (230°F). The percent loss was determined after drying by re-sieving the sample on the 2.4mm (#8)sieve.

Clay Index. -- Sameshima and Black (18) defined the Clay Index (CI) as the number of milliliters of 4.5gm/l methylene-blue solution absorbed by 1 gm of material passing the .074mm(#200) sieve. The Clay Index Test was meant to be a simple method to assess the quality and performance of an aggregate source. In determining the quality of a road aggregate, Sameshima and Black suggested a Clay Index Grade ranging from 1 to 6, the lower values being typical of sound aggregate and the higher values representative of unsound rock. A Grade 1 would have a range of CI values from 0.1 to 1.0 and a Grade 6 would have a range from 5.0 to 6.0. A complete description of the test procedure is given by Szymoniak and Vinson (6) and Sameshima and Black(18).

4.7 DISCUSSION OF RESULTS

In reviewing the petrographic analysis and the performance history of the selected aggregate sources it was apparent that the acceptable limits for the DMSO Accelerated Weathering Test must be separated depending upon the use of the aggregate in the pavement structure. The limits can be separated into two categories depending upon whether the aggregate is treated or untreated. Monismith and Epps (1) concluded that if an aggregate is completely coated by a bituminous asphalt the potential for the aggregate to degrade is significantly reduced or eliminated. However, if the aggregate is not completely coated by the asphalt or there is poor adhesion with the aggregate, the asphalt may strip and lead to failure of the pavement. The DMSO loss limit for treated aggregate was not established in this study. However, preliminary test data suggests that DMSO Accelerated Weathering test

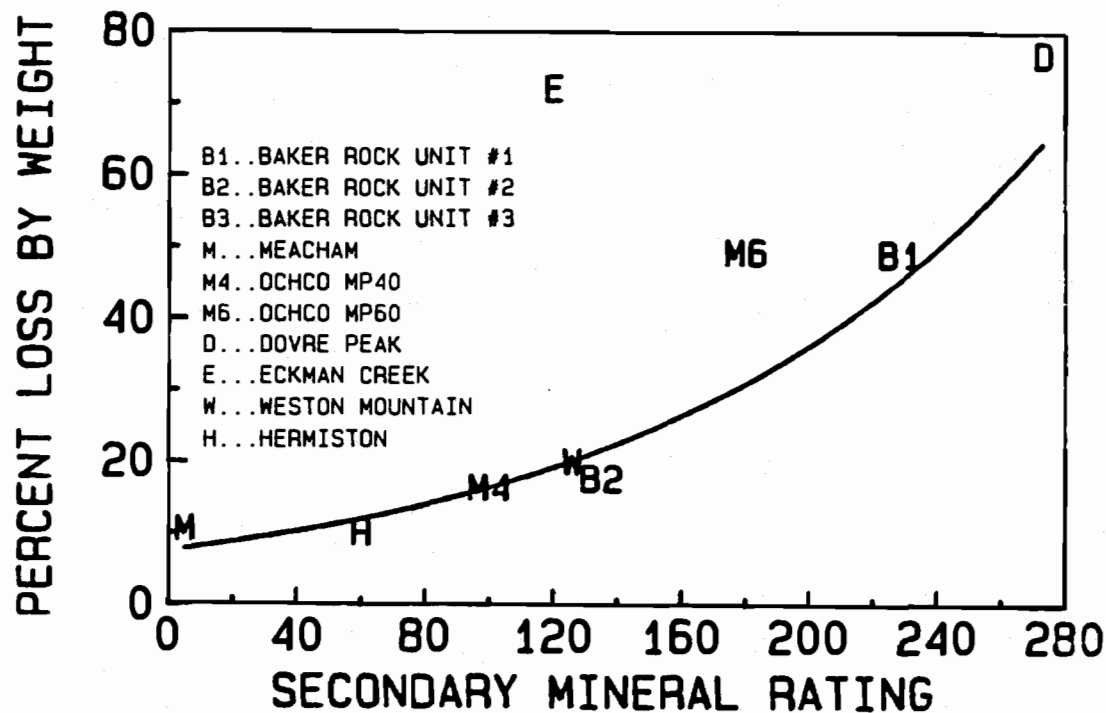
results may be a good indicator of a potential aggregate stripping failure. The remainder of this paper focuses on the development of criteria for untreated aggregate. The criteria includes acceptable limits for the DMSO Accelerated Weathering Test and the Clay Index Test.

Reliability of the DMSO Accelerated Weathering Test. — Figure 4.2 compares the results of the DMSO Accelerated Weathering test to the Secondary Mineral Rating. A regression analysis for the DMSO loss (DMSO) to the Secondary Mineral Rating (RSM) gave the following relationship:

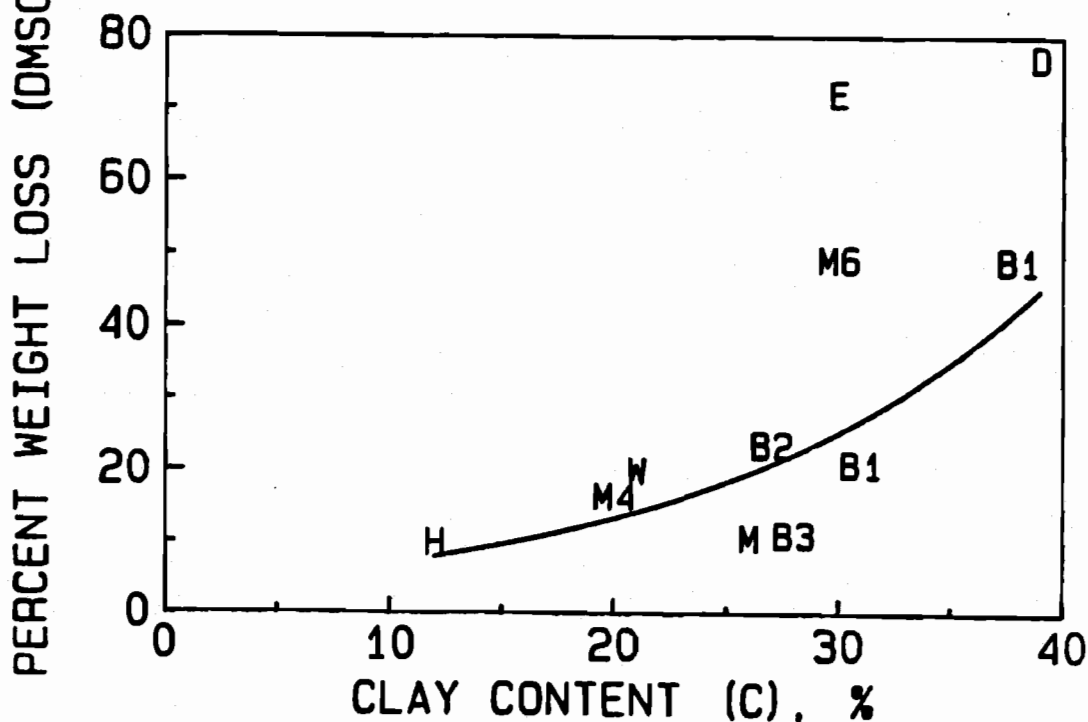
$$\text{DMSO} = 7.42 * \exp[0.0079 * (\text{RSM})] \quad (2)$$

which has a correlation coefficient of 0.99. Equation 2 is illustrated in Figure 4.2. The results from the Eckman Creek quarry and Milepost 60 are omitted from the correlation. Eckman Creek quarry contains the mineral analcime, and Milepost 60 contains calcite in microfractures. The analcime mineral is similar to a swelling clay in that it has a high cation exchange capacity; therefore, DMSO will seek out and solvate the cations which are held on the exterior of the mineral. The degradation of the Eckman Creek rocks immersed in DMSO occurred owing to the presence of analcime in the rock matrix and the size of the DMSO molecule which attaches to the mineral.

The calcite mineral found in the Milepost 60 quarry has a chemical formula of CaCO_3 (Ca^{+2} is an exchangeable ion(22)). It has been previously noted that the DMSO molecule can donate a hydrogen ion; this fact explains the interaction between DMSO and calcite. DMSO donates



(a) - Percent Weight Loss versus Secondary Mineral Rating.



(b) - Percent Weight Loss versus Clay Content determined from petrographic analysis.

Figure 4.2. - A Comparison of DMSO Accelerated Weathering Test and Petrographic Analysis Results.

a hydrogen ion to form HCO_3^- , which results in the solvation of the calcite mineral. Since calcite was found in microfractures the solvation of the mineral results in the partial disintegration of the rock.

The textural distribution of the analcime and calcite minerals in the rock matrix is also reflected by the DMSO test. The sensitivity to distribution is apparent since neither mineral was of sufficient quantity to be included in the petrographic analysis. This sensitivity is further illustrated in Figure 4.2b, which shows the relationship between DMSO losses and clay contents determined from petrographic analyses. The results suggest that the DMSO loss increases exponentially with increasing clay contents. The results also reflect the overreaction of DMSO with the two quarries containing analcime and calcite.

The importance of the minerals analcime and calcite in aggregates is not well understood. Van Atta and Ludowise (22) suggested that the analcime mineral forms in place of smectite clays. This fact would suggest that untreated aggregate containing analcime would perform satisfactorily in a pavement structure because of a lower clay content. However, reviewing the field performance of aggregates containing zeolites has not shown a definite trend since in one instance the performance was acceptable, and in another the performance was unacceptable(22).

Aggregates from the Milepost 60 quarry (containing calcite) have performed satisfactorily when used with an emulsified asphalt. The performance is most likely the result of the calcite mineral which makes

the aggregate basic, thereby promoting good adhesion characteristics with the acidic components of the asphalt. Other performance data concerning untreated aggregates from the quarry was unavailable, but it is likely that untreated aggregate would have failed owing to the high mineral rating factor. It is desirable to establish the acceptable limits of a standard test based upon field performance of aggregates. Unfortunately, it was not practical in this study to do so owing to the lack of good field data. Further, it is often very difficult to separate the effects of all possible contributing factors (e.g. construction materials, construction practices, traffic, and environment) after a pavement section has failed. Acknowledgement of this situation lead to the use of the secondary mineral rating system to establish the acceptable DMSO weight loss limits for untreated aggregate.

Cole and Sandy (17) recommended an RSM value of 140 as the limit between sound and unsound aggregate. The limit was based upon a correlation between the Secondary Mineral Rating and the Washington Durability test results. Cole and Sandy also reviewed aggregate field performance data and found that failures had occurred above this value while no failures were observed below the value. In reviewing Figure 4.2a it can be seen that only Dovre Peak, the upper bench of Baker Rock and the Milepost 60 quarry fall above the 140 limit. Only the untreated aggregate from Dovre Peak is known to have failed, but the upper bench was identified by the quarry operator as being poor quality material. Weston Mountain and the lower bench of Baker Rock quarry are near the

limit proposed by Cole and Sandy. Aggregate from the quarries has performed satisfactorily and the majority of the unacceptable laboratory results associated with the quarries are due to the production operation. Therefore, referring to Figure 4.2a it may be noted that an RSM of 140 corresponds to a DMSO weight loss of 22 percent.

ODOT does not use the Washington durability test, but employs the Oregon Aggregate Degradation test to indicate the durability of an aggregate source. Both tests are a means of assessing the mechanical durability of an aggregate source. Clemmons (21) and Vinson and Rogers (3) have shown that these tests do not correlate to DMSO test results, since the DMSO test simulates the chemical degradation potential.

The measurement of the degradation in the Oregon test is obtained by two methods: First, a sediment height is observed following the agitation of the aggregate sample by air, the sediment height is an indirect measure of the quantity of clay size particles that are produced; second, the weight loss after re-screening the aggregate over the 1.6mm (#20) sieve is determined to reflect the mechanical durability of the aggregate. Figure 4.3 shows the relationship between the DMSO loss and the sediment height (H). Regression analysis gave the following best relationship:

$$\text{DMSO} = 20.2 + 4.99 * (H) \quad (3)$$

which has a correlation coefficient of .79. The low correlation coefficient clearly suggests that the acceptable limits cannot be established through correlations with mechanical durability test results.

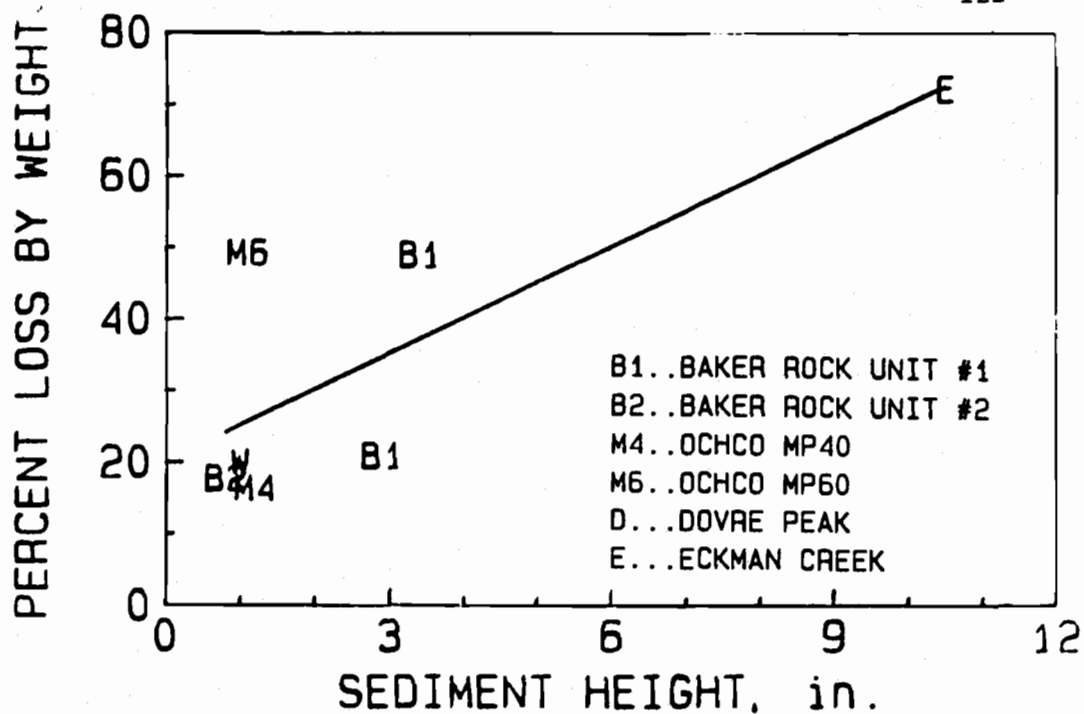


Figure 4.3. - Sediment Height determined from the Oregon Aggregate Degradation Test versus Percent DMSO Weight Loss.

Clay Index Test Results. — Figure 4.4 presents the relationship between the Clay Index (CI) and the Secondary Mineral Rating. A regression analysis for the Clay Index and Secondary Mineral Rating gave the following best fit relationship:

$$CI = 0.72 * \exp[0.0077 * (RSM)] \quad (4)$$

which has a correlation coefficient of 0.86. Equation 4 is illustrated in Figure 4.4. It may be noted by reviewing the figure that the Clay Index is apparently not sensitive to the presence of the minerals analcime and calcite. The scatter in the data is most likely due to: 1) the aggregate particle size used to produce the pulverized powder for the test, and 2) the subjective interpretation of the endpoint in the titration procedure.

In determining the acceptable Clay Index value a Secondary Mineral Rating limit of 140 was employed. With respect to Figure 4.4, the limit of 140 results in a corresponding Clay Index value of 2.2. The Clay Index value was established using the results from all of the quarries, since the Clay Index test was insensitive to the presence of analcime and calcite. The relationship between the Clay Index and DMSO loss is shown in Figure 5. A Clay Index value of 2.2 would correspond to a DMSO loss of 25 percent, which is reasonably close to the 22 percent limit previously established. The close agreement between the two further substantiates the use of an acceptable loss of 22 percent for the DMSO Accelerated Weathering Test. Further, based upon the results of this study for basalt aggregates in Oregon, the acceptable Clay Index limit for untreated aggregate should be 2.2.

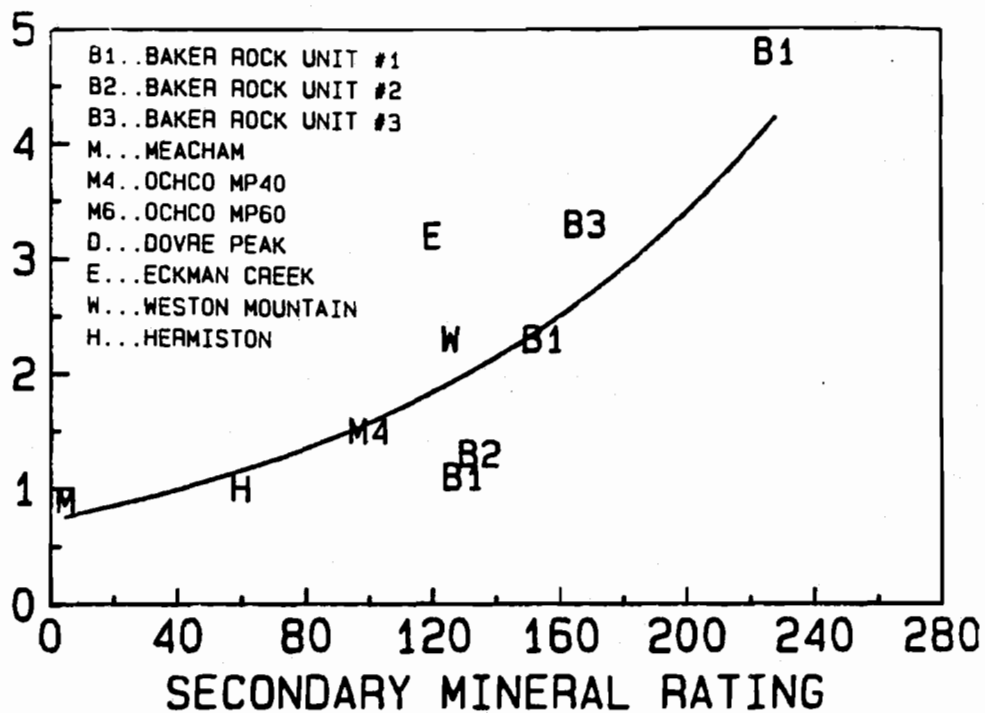


Figure 4.4. - Clay Index versus the Secondary Mineral Rating Factor.

The relationship between the Clay Index results and DMSO weight loss, presented in Figure 4.5, can also be used to determine if DMSO has overreacted with minerals other than swelling clays. In Figure 4.2 it was noted that DMSO will overreact if the minerals analcime or calcite are present in the rock matrix; in Figure 4.4 it was shown that the results from the Clay Index test were insensitive to the presence of either mineral. Thus, one can validate the DMSO test by comparing the weight loss and the Clay Index results to the relationship illustrated in Figure 4.5. The DMSO weight loss is valid if the intersection of the two test results falls reasonably close to the established relationship. However, if the test results deviate substantially from the established relationship a complete petrographic analysis should be conducted.

4.8 CONCLUSION AND SUMMARY

DMSO is a powerful penetrant, and causes greater breakdown and disintegration of aggregates when compared to immersion of the aggregates in ethylene glycol or water. Use of DMSO in an accelerated weathering test for aggregates, simulating the chemical weathering of the mineral constituents of the rock, is desirable since its degradation potential will be reflected in a shorter period of time.

The penetration ability of DMSO, and thus the greater breakdown of aggregates, is due in part to both the type and concentration of ions contained within the rock matrix. Through the use of Atterberg Limits and X-ray diffraction analysis it has been determined that DMSO reacts with swelling clays. Swelling clays are considered deleterious

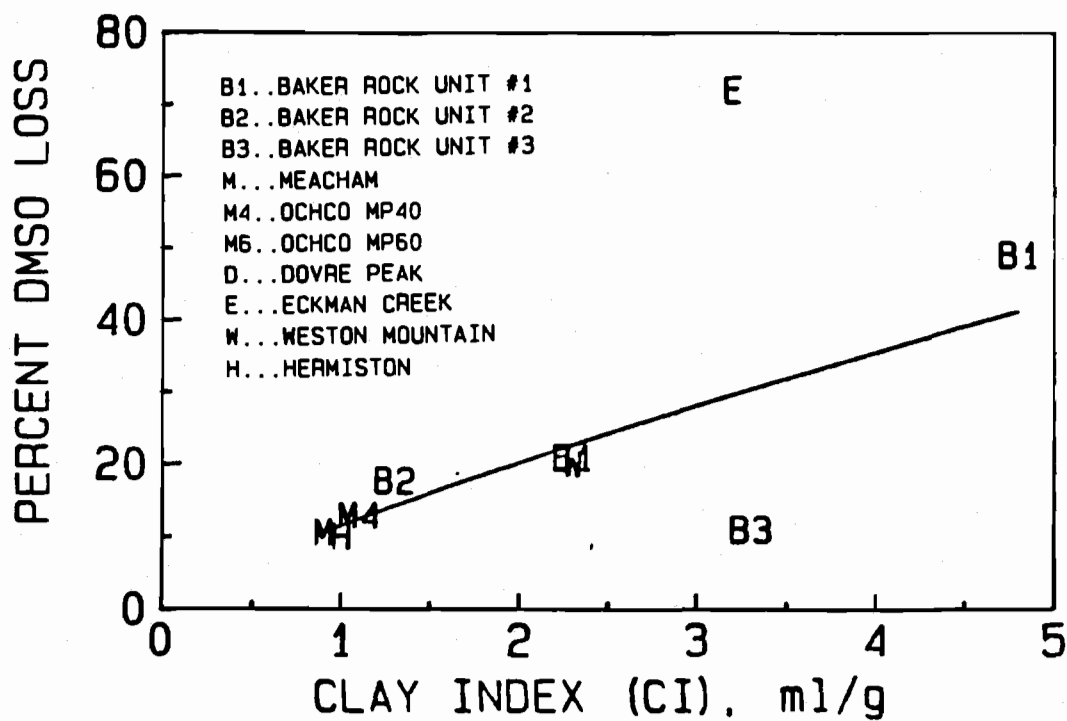


Figure 4.5 - Percent DMSO Weight Loss versus Clay Index.

minerals in basalt aggregates since in the presence of water they expand and lead to the disintegration of the aggregate. In this study it was found that DMSO overreacts with aggregates containing the secondary minerals analcime and calcite. The overreaction occurs owing to: 1) the solvation of cations held by the analcime mineral, which has a high cation exchange capacity similar to swelling clays, and 2) the ability for DMSO to donate a hydrogen ion to the calcite mineral to produce HCO_3^- .

Eight basalt quarries were sampled to establish acceptable DMSO weight loss limits; aggregate from two of the selected quarries contained the minerals analcime and calcite. The DMSO weight loss correlates to a high degree with the Secondary Mineral Rating determined from petrographic analysis, if the results from the two quarries which contained analcime and calcite are neglected. The Secondary Mineral Rating incorporates the type, content and textural distribution of deleterious minerals in the rock matrix.

Results obtained from the Clay Index Test did not reveal the overreaction with the analcime or calcite minerals reflected in the DMSO test. The Clay Index test is a much simpler test to perform, and the results concerning the suitability of an aggregate source can be achieved in a shorter period of time. The correlation between the Clay Index and Secondary Mineral Rating is good, but Clay Index may be influenced by: 1) the aggregate particle size used to produce the pulverized aggregate, and 2) the determination of the endpoint in the titration procedure. Acceptable limits for the DMSO Accelerated

Weathering Test and the Clay Index test were established through the use of the Secondary Mineral Rating. A Secondary Mineral Rating of 140 was used to establish the acceptable DMSO loss limits; this rating value was based upon observations of field performance and a correlation with Washington Durability test results. Based upon the investigation of eight quarries in Oregon the acceptable weight loss for the DMSO Accelerated Weathering test for untreated aggregates was established at 22 percent. This limit was confirmed through an analysis of the relationship between the Clay Index and the Secondary Mineral Rating. In addition, it was found that the relationship between the DMSO and Clay Index test results could be used to determine if DMSO had reacted with minerals other than swelling clays. If the results of the two tests deviated substantially from the established relationship a complete petrographic analysis should be conducted.

4.9 ACKNOWLEDGEMENTS

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5.0 CONCLUSION

5.1 SUMMARY

A testing procedure using Dimethyl Sulfoxide (DMSO) was developed to simulate the chemical weathering of basaltic rocks. The development revolved around the ability of the DMSO solution to react with swelling clays found in many types of basaltic rock. In addition, it was found that rocks taken from the quarries selected in this study, achieved the greatest weight loss in the particle size range between the #8 and #4 sieve after immersion in DMSO. Using the results from the standard procedure a acceptable weight loss was determined by correlating the losses to the Secondary Mineral Rating established by petrographic analyses

5.2 CONCLUSIONS

Based on the results of the study to develop an accelerated weathering test for aggregates using Dimethyl Sulfoxide the following may be concluded:

- 1) Clay mineral type and content are reflected in the aggregate weight loss after immersion in DMSO owing to the interaction of the clay mineral and DMSO.
- 2) The weight loss results after immersion are highly sensitive to the textural distribution of swelling clays and other minerals in the rock matrix.
- 3) The penetration ability of DMSO is in part due to both the

type and concentration of ions contained in the rock matrix.

- 4) Owing to the attachment of the sulfur molecule, DMSO is slightly anionic and thus prefers and seeks out cations.
- 5) DMSO substantially reduces the liquid limit of highly active clays by increasing the attractive force of cations to surface of the clay minerals, which in turn reduces the net interparticle attractive force between adjacent clay minerals
- 6) DMSO had the opposite effect on non-swelling clays such as kaolinite, and resulted in increasing the liquid limit. The increased liquid limit was associated with the interaction of DMSO and the hydroxyl ion attached to the exterior of the kaolin clay.
- 7) Aggregate weight losses after immersion in DMSO are influenced by aggregate particle size, sample weight, container geometry, and immersion time.
- 8) DMSO was found to overreact with rock containing the minerals analcime and calcite.

(a) it was suggested that the Clay Index Test results be used to determine if DMSO is likely to overreact.

- 9) Neglecting the quarries known to contain analcime and calcite the weight loss from the recommended DMSO Accelerated Weathering Test correlates to a high degree to clay content and the Secondary Mineral Rating.
- 10) The acceptable weight loss for the DMSO Accelerated Weathering Test was determined to be 22 percent for untreated aggregates.

5.3 RECOMMENDATIONS

Although valuable results were obtained in this research study, there are many areas that should be further examined. Therefore the following recommendations are made:

- 1) Adopt the testing procedure and correlate the results to field performance of untreated aggregates.
- 2) Investigate the possibility of DMSO to predict potential stripping of asphalt from aggregates.
- 3) Investigate the Clay Index testing for possible adoption as a field quality control test.

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