

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

by

Tom Szymoniak

Senior Engineer, Pavement Services Inc.

1630 SW Morrison

Portland, OR. 97205

(503)227-7630

Ted S. Vinson

Professor, Department of Civil Engineering

Oregon State University

Corvallis, OR 97333

(503)754-3494

and

Jim E. Wilson

Assistant Materials Engineer

Oregon State Highway Division

Salem, OR 97310

(503)378-2621

Submitted for Presentation  
to the Sixty-Sixth Annual Meeting of the  
Transportation Research Board

August 1986

Szymoniak, T., Vinson, T. S., and Wilson J. E.

ABSTRACT: The reliability of the Dimethyl Sulfoxide (DMSO) Accelerated Weathering Test to predict the degradation potential of untreated basaltic aggregates is evaluated. In the investigation two tasks were undertaken; 1) correlation of the tests results to petrographic analysis, and 2) establishment of acceptable weight loss limits after immersion of aggregates in DMSO. From the study of eight rock quarries in Oregon, it was found that the DMSO Accelerated Weathering Test results correlate well to a Secondary Mineral Rating. The Secondary Mineral Rating is based upon petrographic analyses, which accounts for the content and distribution of deleterious minerals in a rock. The correlation equation which relates the DMSO weight losses to petrographic analysis neglected the results from two quarries containing the minerals analcime and calcite. The reaction of aggregates containing these two minerals occurs owing to the mechanisms by which DMSO is able to penetrate into membranes. In regard to the potential for the DMSO to react with minerals other swelling clays, it was suggested that a Clay Index test be conducted since this index was found to be insensitive to the presence of analcime and calcite. The validity of the DMSO test for a particular quarry can be established by comparing the weight loss and the Clay Index to the relationship given in the paper. If the result deviates substantially from the indicated relationship a complete petrographic analysis may be required to determine the suitability of the aggregate. Data collected from this study indicate that an acceptable weight loss limit for untreated aggregate, utilizing the DMSO Accelerated Weathering Test procedure, should be in the range of 20 percent.

## 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 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.

#### 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. smectite) 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 (4). 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 (5), 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 (6).

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 (6) 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 recommended a testing procedure using 1000g of aggregate (in the size range of 2.4mm(#8) to 4.8mm (#4)) immersed in a container holding a 100 percent solution of DMSO for a period of 5 days.

The data collected by Szymoniak and Vinson (7) and Miles (8) 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 its interaction with clay minerals. This discussion is presented in the following sections.

#### INTERACTION OF DMSO WITH CLAY MINERALS

Szymoniak and Vinson (5), and Andrews et al (13) 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/m<sup>2</sup> (12)) for Na-montmorillonite or Ca-montmorillonite, was substantially reduced when DMSO was substituted for water in the test. Szymoniak and Vinson (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 Coulomb'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 octrahedral 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 to solvate 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.

#### DELETERIOUS MINERALS IN ROAD AGGREGATES

A mineral is a substance with a definite chemical composition, an orderly structural arrangement, naturally occurring, and inorganic(14). 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(5).

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 (14). 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 (15). Secondary minerals are formed through the weathering of minerals under temperature, pressure, and/or chemical conditions at, or near the earth's surface (14). Some secondary minerals that can be found in basalts include iron oxides, zeolites, and calcite (5).

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 (15). Smectite clays are produced through the alteration of plagioclase, and have high swelling potential and high cation exchange capacities. Palagonite is an alteration product caused by the oxidation and/or devitrification of basaltic glass; it further alters 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 (16) determined that 0 to 20% secondary minerals in a fine grained aggregate will have little effect on pavement performance; 20 to 35% will produce some failures and borderline performances; and above 35% will almost certainly produce failures. However, in 1974 Van Atta and Ludowise (5) 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 and their textural distribution of in a 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% would result in poor pavement performance. Higgs (3) in an independent study conducted during 1976 found that as little as 11% of discrete montmorillonite was sufficient to cause basalts to degrade.

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(5), 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:

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

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 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 1. Table 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, this method requires a trained petrologists, capable of identifying the primary and secondary minerals, and the use of 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 matrix. The DMSO Accelerated Weathering Test offers potential if the acceptable limits of weight loss are established.

#### 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 1 shows the location of the quarries. The quarries were selected to represent different sources of aggregate utilized throughout the state of Oregon and to study the rock types where the DMSO test may be a reliable indicator of field performance. 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 loss limits by considering the secondary mineral rating. The secondary mineral rating system was used in this study owing to the lack of good field data concerning the performance of the aggregates produced from the selected quarries. However, it should be noted that the quarries selected for this study have been associated with some sort of performance

problem. The majority of the field performance problems have been limited to asphalt stripping problems. Ideally, it would have been preferable to select quarries which experienced failures of untreated aggregates, but because ODOT primarily cement treats the aggregate base material this was not possible.

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 within a representative thin section of rock from the quarry. The percentage of smectite clay mineral was determined by a point count procedure after staining the thin section with a rhodamine-b dye. A more detailed description of the procedure adopted for point counting and staining is given by Van Atta and Ludowise (14), Szymoniak and Vinson (7), and Szymoniak et al (19,20).

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 3; and a summary of the durability test results for the five quarries are presented in Table 4. ODOT durability specification require that an untreated aggregate have a Sodium Sulfate Soundness loss not greater than 18 percent, an Oregon Aggregate Degradation value not greater than a sediment of 3.0 inches and 30 percent passing the 0.841 mm (#20) and a abrasion value not greater than 35 percent.

The following paragraphs provide a geologic description and a brief discussion concerning the performance of aggregates produced from the

quarries selected for the project.

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 upper unit has a primary mineral of ranging from 53 to 56 percent, a secondary mineral content of 39 to 46 percent and a clay content ranging from 26 to 38 percent. 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. This unit has a primary mineral content ranging from 55 to 79 percent, a secondary mineral content of 21 to 45 percent and a clay content ranging from 3 to 31 percent.

Meacham Quarry. -- 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. Untreated aggregate produced from the quarry has performed satisfactorily.

Ochco 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. Untreated aggregate produced from the quarry has performed well, but a seal coat had to be applied to the pavement surface due to the slight potholing and ravelling.

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. Because this was the first use of the quarry no other performance data was available.

Dovre Peak. -- 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 66 percent, and 39 percent respectively, with a smectite clays comprising one half of the total secondary mineral content. Minor amounts of palagonite were also identified in the thin section to be in the third stage of development to a smectite clay. Field performance of untreated aggregate from this quarry has been very good.

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. Untreated aggregate produced from this quarry is borderline, in terms of field performance, and varies depending upon where the material is taken from the quarry.

Hermiston Quarry. -- The Hermiston quarry is a homogeneous river run gravel (basalt) 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. Paragonite is very similar to a mica mineral.

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 test 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 quarry and separating the crushed aggregate by mechanical sieving. The

DMSO immersion test was conducted in accordance with the procedure given by Szymoniak et al (6). Air-dried aggregates in the size range of 2.4mm (#8) to 4.8mm(#4) were soaked in a container of 100 percent concentration of 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 the unsound rock. A Grade 1 would have a CI value range from 0.1 -1.0 and a Grade 6 would have a range of CI values between 5.0 and 6.0. A complete description of the test procedure is given by Szymoniak and Vinson (5) and Sameshima and Black(18).

#### 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 (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 the failure of pavement. The DMSO loss limit for treated aggregate is not established owing to the complex interaction between asphalt and aggregates. However, preliminary test data suggests that DMSO Accelerated Weathering test results may be a good indicator of an aggregates potential to strip. 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 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:

$$\text{DMSO} = 7.416e[0.0079262(\text{RSM})] \quad (2)$$

which has a correlation coefficient of 0.99. Equation 2 is illustrated in Figure 2a. 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 micro-fractures. 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 attached to the mineral.

The calcite mineral found in the Milepost 60 quarry has a chemical formula of  $\text{CaCO}_3$  ( $\text{Ca}^{+2}$  is an exchangeable ion(22)). It has been previously noted that the DMSO molecule can donate a hydrogen ions; this fact explains the interaction between DMSO and calcite. DMSO donates a hydrogen ion to form  $\text{HCO}_3^-$ , which then results in the solvation of the calcite mineral. Since calcite was found in micro-fractures 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 2b, which shows the relationship between DMSO losses and clay contents determined from petrographic analyses. The figure suggests that the DMSO loss increases exponentially with increasing clay contents. The figure also reflects 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 formed instead 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 limits for untreated aggregate.

Cole and Sandy (17) recommended an RSM value of 140 as the limit between the sound and unsound aggregate. The limit was based upon a correlations 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 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 a "poor" material. Weston Mountain, Eckman Creek and the second unit of Baker Rock quarry are near the limit proposed by Cole and Sandy. Aggregate from the Weston and Baker Rock quarries has performed satisfactorily and the majority of the unacceptable laboratory results associated with the quarries are due to the production operation. Eckman Creek quarry has been associated with both poor and good field performance. Thus, referring to Figure 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 3 shows the relationship between the DMSO loss and the sediment height (H). Regression analysis gave the following best relationship:

$$\text{DMSO} = 20.166 + 4.9874 (\text{H}) \quad (2)$$

which has a correlation coefficient of .79. The low correlation coefficient clearly suggests that the acceptable limits cannot be established by the mechanical durability tests.

Clay Index Test Results. -- Figure 4 and 5 presents the relationships between the Clay Index (CI) and the Secondary Mineral, and DMSO losses and the Clay Index. Regression analysis for the Clay Index and Secondary Mineral Rating gave the following best fit relationship:

$$\text{CI} = 0.7234 * \exp[ 0.007732 * (\text{RSM})] \quad (3)$$

which gave a correlation coefficient of 0.86. Equation 3 is illustrated in Figure 4. It may be noted 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, the limit of 140 results in a corresponding CI value of 2.2. The CI 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 CI and DMSO loss is shown in Figure 5. A CI 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.

Based upon the results of this study for basalt aggregates in Oregon, the acceptable Clay Index limit for untreated aggregate should be 2.2 ml of absorption of methylene blue per one gram of pulverized aggregate.

The relationship between the Clay Index results and the DMSO weight losses, presented in Figure 5, can also be used to determine if DMSO has overreacted with minerals other than swelling clays. In Figure 2 it was noted that DMSO will overreact if the minerals analcime or calcite are present in the rock matrix and in Figure 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 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.

#### CONCLUSION AND SUMMARY

DMSO is a powerful solvent, 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, in part, due 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 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  $\text{HCO}_3^-$ .

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 Mineral Rating was based upon observations of field performance and correlation with the Washington Durability test. Based upon the results from 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 utilized to determine if DMSO had reacted with minerals other than the swelling clays. If the results of the two tests deviated substantially from the established relationship a complete petrographic analysis should be conducted. Because the weight loss limit is based upon a limited data set, it is recommended that the DMSO test results be correlated to actual field data performance.

#### ACKNOWLEDGEMENTS

This study was funded by the Oregon Department of Transportation (ODOT). The support of both the Research Unit and the Materials Section within ODOT is gratefully acknowledged. The contributions of Neal Walker and with ODOT, and Robert Pintner and Isabelle Chavot with Oregon State University, are very much appreciated.

## REFERENCES

1. Monismith, C. L., and Epps, J. A., "Asphalt Paving Mixtures: Design, Construction, and Performance," Short Course Notes, Univ. of California, Berkeley, May 1984, pp. 30-50.
2. Starkey, H., Blackmon, P., and Hauff, P., The Routine Mineralogical Analysis of Clay-Bearing Samples, U.S. Geological Survey Bulletin 1563, U.S. Printing Office, Washington D.C., 1984.
3. Higgs, N., "Slaking Basalts," Bulletin of the Association of Engineering Geologists, Vol 13, NO. 2, Spring 1976, pp 151-162.
4. Vinson, T. S., and Rogers, F., "Reliability of DMSO (Dimethyl Sulfoxide) Method to Determine the Degradation Characteristics of Rock Other than Marine Basalt for Highway Construction," Federal Highway Administration, November 1983.
5. Van Atta, R. O., and Ludowise, H., "Microscopic and X-Ray Diffraction Examination of Basalt to Determine Factors Affecting Durability," Report NO. FHWA-RD-74-20, Federal Highway Administration, April 1974.
6. Szymoniak, T., Vinson, T. S., Wilson, J. E., and Walker, N., "The Dimethyl Sulfoxide (DMSO) Accelerated Weathering Test For Aggregates," Submitted for publication in ASTM Geotechnical Testing Journal, July 1986.
7. Szymoniak, T., and Vinson, T. S., "Determination of Clay Minerals in Road Aggregates," submitted for publication in Journal of Geotechnical Engineering, ASCE, July 1986.
8. Miles, D. K., "Accelerated Soundness Test for Aggregates: Final

- Report," Utah State Department of Highways, Materials and Tests Division, July 1972.
9. Tarhsis, B., DMSO: The True Story of a Remarkable Pain-Killing Drug, William Morrow and Company, New York, NY, 1981.
  10. Franz, T. J., and Van Bruggen, J. T., " A Possible Mechanism of Action of DMSO," New York Academy of Science, Annals No. 141, 1967, pp. 302-309.
  11. Hiller, F. W., "A Study of the Iodine-Formate Reaction in Dimethyl Sulfoxide - Water Mixtures," thesis presented to Oregon State University, Corvallis, OR, 1967, in partial fulfillment of the requirements for the degree of Doctor of Philosophy.
  12. Mitchell, J. K., Fundamentals of Soil Behavior, John Wiley and Sons Inc., New York, NY., 1976.
  13. Andrews, R. E., Gawarkiewicz, J. J., and Winterkorn, H. F.," Comparison of the Interaction of Three Clay Minerals With Water, Dimethyl Sulfoxide, and Dimethyl Formamide," Highway Research Record No. 209, Physicochemical Properties of Soils, 1967, pp. 66-78.
  14. Zoltai, T., and Stout, J., Mineralogy Concepts and Principles, Burgess Publishing Co., Minneapolis, Minn., 1984, pp. 34-70.
  15. Van Atta, R. O., and Ludowise, H., "Microscopic and X-Ray Examination of Rock Durability Testing," Report No. FHWA-RD-77-36, Federal Highway Administration, April 1974.
  16. Scott, L. E., "Secondary Minerals in Rock as a Cause of Pavement and Base Failure," Proceeding of the Thirty-Fourth Annual Meeting Highway Research Board, 1955, pp. 412-417.

17. Cole, W. F., and Sandy, M. J., "A Proposed Secondary Mineral Rating For Basalt Road Aggregate Durability," Australian Road Research, Vol.10, No.3, Sept, 1980, pp. 27-37.
18. Sameshima, T., and Black, P. M., "Clay Index - A Simple Method of Assessing the Quality and Performance of Roading Aggregates," in Proceedings New Zealand Road Symp., Vol A3, 1979, pp. 1-10.
19. Szymoniak, T., Chavoat, I., and Vinson, T. S., "A New Staining Procedure For Thin Sections of Rock," submitted for publication in Journal of Geotechnical Engineering, ASCE, August 1986.
20. Szymoniak, T., and Hicks, R. G., " Evaluation of Low Volume Road Surfacing Study Utilizing Marginal Aggregate - Nestucca River Road Study," Transportation Research Report 85-16, Oregon State Univ., Corvallis, OR, Sept. 1985.
21. Clemmons, G. H., "An Evaluation of Coastal Aggregates in Oregon," Transportation Research Report 79-5, Oregon State Univ., Corvallis, OR, June 1979.
22. Van Atta, R. O., and Ludowise, H., " A Study of Petrography and Degradation of Some Basaltic Aggregates, Nestucca River Area, Oregon,"  
Proceeding of the 14th Engineering Geology and Soils Engineering Symposium, Moscow, ID, April 1976.

Table 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 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)

Table 3. - Petrographic Summary for Quarries Sampled.

Quarry	Primary	Secondary	Smectite	
			Glass	Clay
Baker Rock				
Unit #1 (B1)	53	15	1	31
Unit #2 (B2)	55	14	-	31
(unoxidized)				
Unit #2 (B2)	79	18	-	3
(oxidized)				
Unit #3 (B3)	49	23	-	28
Meacham (M)				
Quarry	62	18	-	21
Ochco MP40 (M4)				
Quarry	75	-	5	20
Ochco MP60 (M6)				
Mid-Top	70	-	-	30
* Dovre Peak (D)				
U-per Unit	65	26	6	3
Lower Unit	70	19	4	7
Crusher	50	11	-	39
Weston Mtn. (W)				
Quarry	66	13	-	21
**Eckman (E)	70	-	-	30
Quarry				
Hermiston (H)				
Quarry	66	22	-	12

\* Reference 20

\*\* Reference 3

TABLE 4. - Durability Summary for Quarries Utilized in Study

QUARRY	Sodium Sulfate		Oregon Aggregate		Los Angeles
	Soundness		Degradation		Abrasion
	% Loss		Sediment	% Passing	
	Coarse Aggregate	Fine Aggregate	ht. in.	0.84mm (#20) Sieve	% Wear
Baker Rock					
Unit #1 (B1)					
Random	15.1	13.5	3.4	16.4	18.5
Vesicular	9.9	23.4	2.9	21.1	21.6
Olivine	4.6	15.9	1.3	15.4	20.8
Unit #2 (B2)	4.8	13.8	1.0	16.6	17.6
Meacham(M)					
Quarry	1.7	7.7	-	-	-
Ochoco MP60(M6)					
Westside	9.2	6.0	3.3	18.7	15.8
Mid-Top	4.6	9.9	1.1	13.5	16.4
Ochoco MP40(M4)	3.8	10.8			
Quarry			1.2	15.7	20.8
Dovre Peak(D)					
Quarry	23.1	24.1	-	-	-

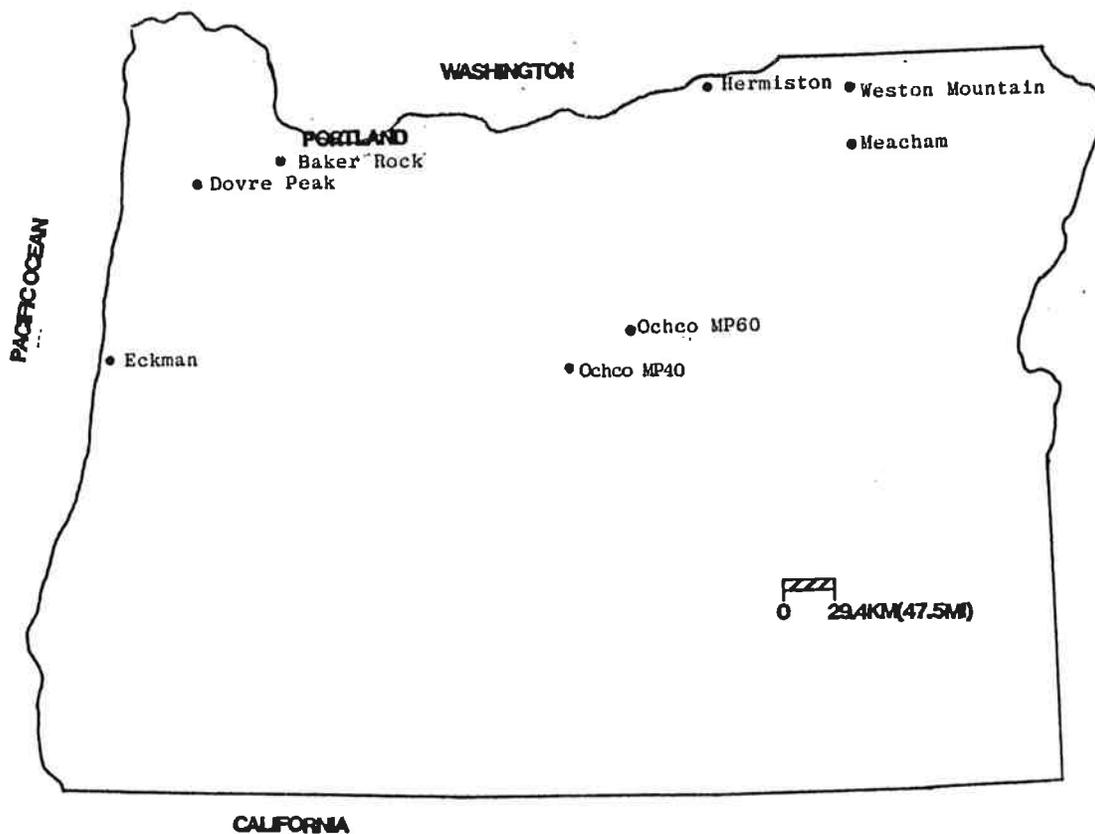
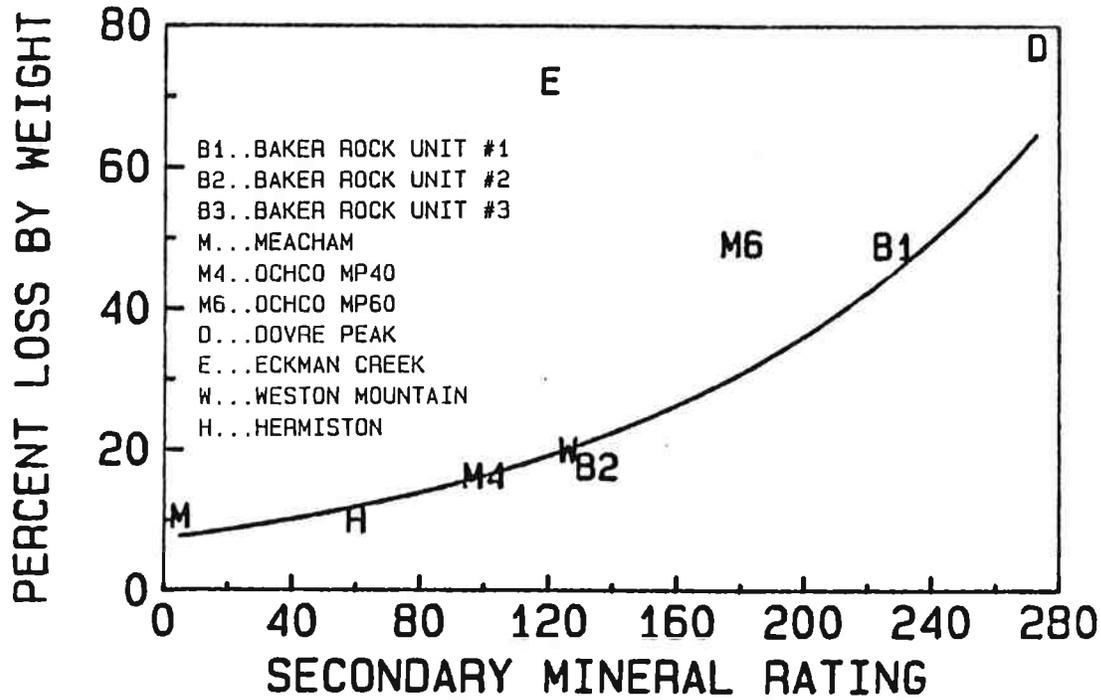
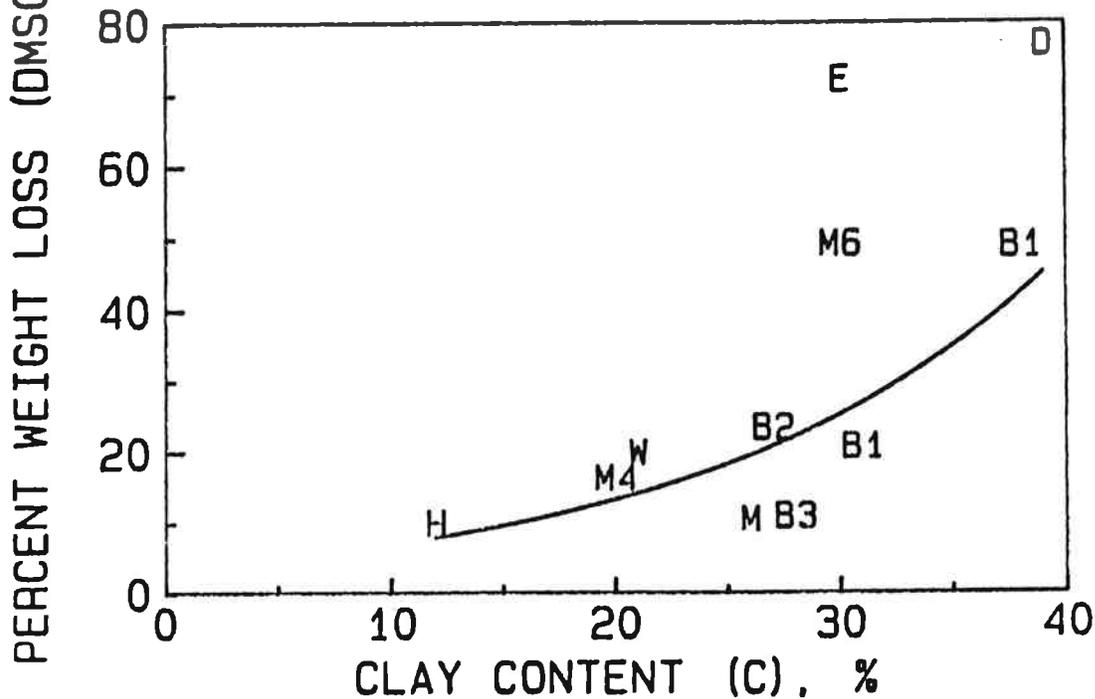


Figure 1. - Location of the Quarries Sampled for the DMSO Project.



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



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

Figure 2. - A Comparison of DMSO Accelerated Weathering Test and Petrographic Analysis Results.

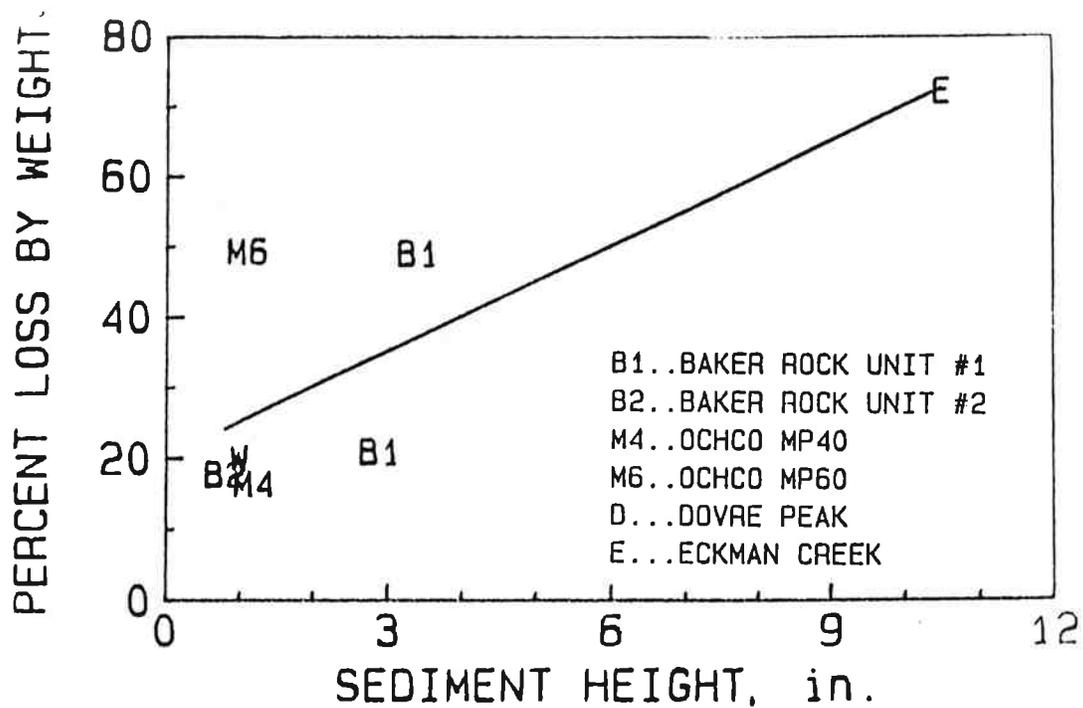


Figure 3. - Sediment Height determined from the Oregon Aggregate Degradation Test versus Percent DMSO Weight Loss

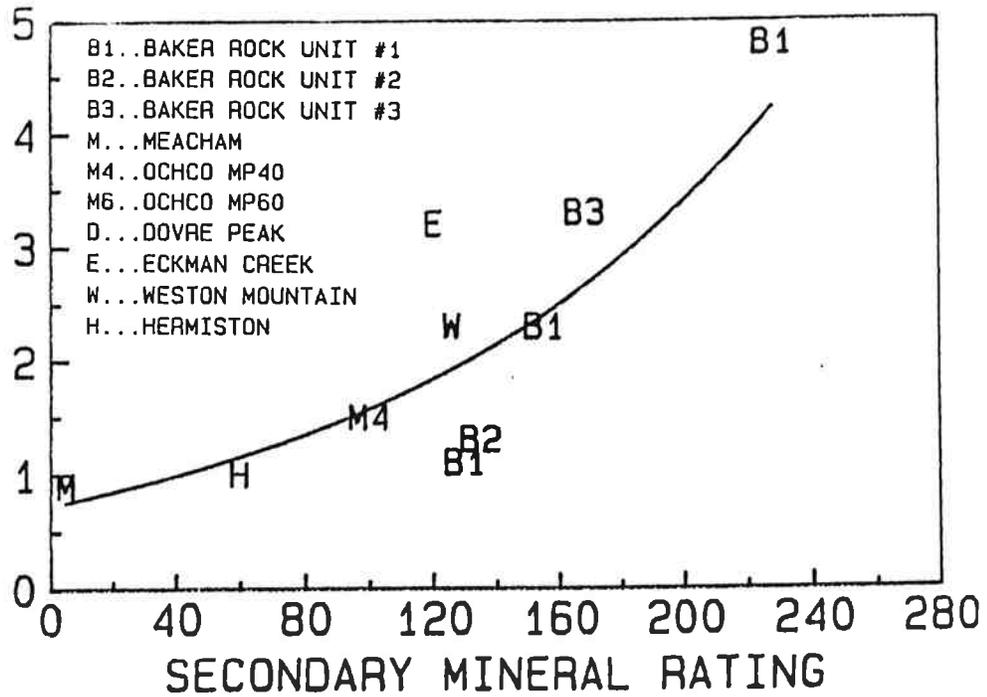


Figure 4. - Clay Index versus the Secondary Mineral Rating Factor.

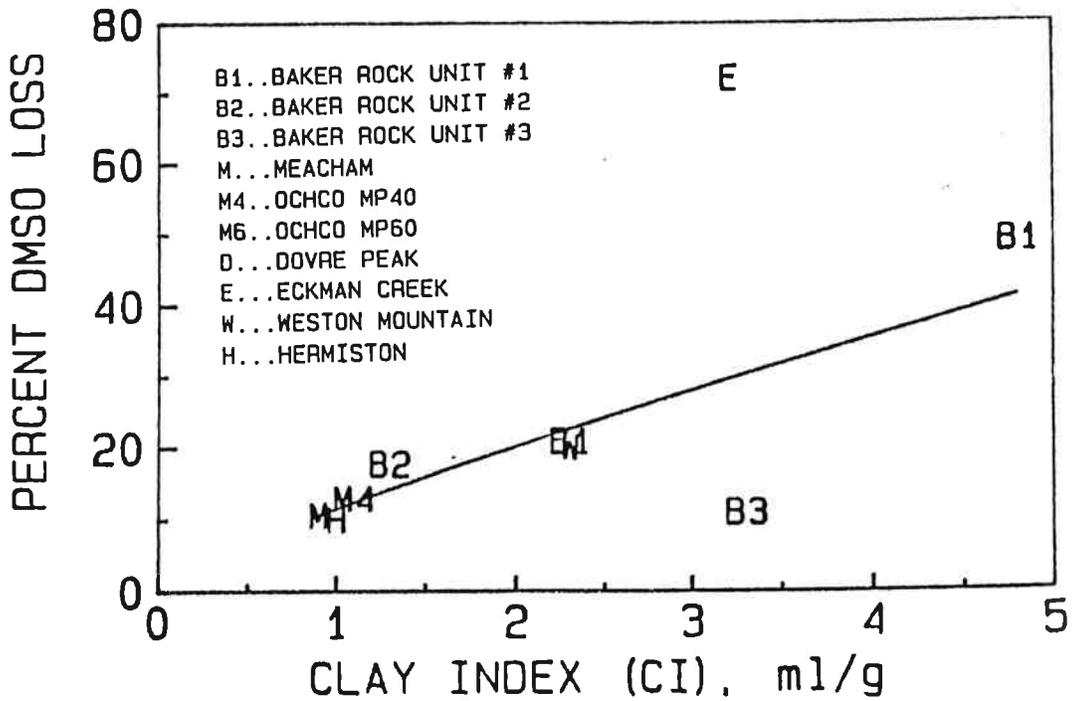


Figure 5. - Percent DMSO Weight Loss versus Clay Index.