Sulfur Extended Asphalt Laboratory Investigation — Mixture Characterization

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SULFUR EXTENDED ASPHALT LABORATORY INVESTIGATION - MIXTURE CHARACTERIZATION

by

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16. Abstract

This report provides a summary of a laboratory study on sulfur extended asphalt paving mixtures. The following major features were included in the study:

1. Design of laboratory experiment which included the use of mixtures which contained various amounts of sulfur, two viscosity levels of asphalt cement, and two types of aggregate (basalt and granite).
2. Evaluation of mixture design methods (Hveem and Marshall).
3. Determination of optimum binder contents.
4. Evaluation of mixture durability and aging characteristics.
5. Development of revised mixture design criteria.

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CHAPTER I

INTRODUCTION

INITIAL SEA PAVING IN WASHINGTON

The first field experimental work performed in the State of Washington related to replacing part of the asphalt in hot-mix paving with sulfur was accomplished in a WSDOT sponsored project entitled "Sulfur Extended Asphalt Binder Evaluation". This project was a cooperative effort involving WSDOT, the University of Washington (UW), Washington State University (WSU), the Federal Highway Administration (FHWA), the Sulphur Development Institute of Canada (SUDIC) and the Asphalt Paving Association of Washington. The study involved an initial laboratory mixture design phase followed by construction and evaluation of test pavements at two sites near Pullman, Washington. The work was initiated during May 1979 and will continue through the evaluation phase until summer 1982.

That study is significant for a number of reasons but at least two are particularly notable. First, it was the first SEA paving project constructed in the state thus providing experience with the unique features of this material. Second, pavement test sections were constructed at the WSU Test Track and on a nearby state highway (SR 270) at Pullman. The operation of the WSU Test Track allowed for accelerated loading of the pavement sections. This resulted in a field determination of how the SEA paving mixtures performed relative to conventional asphalt concrete (Class B graded aggregate was used for all mixtures).

The result of the analysis performed on the test track data indicates that a 30/70 SEA (30 percent added sulfur and 70 percent asphalt by weight) paving mixture is approximately equivalent to conventional asphalt concrete with regard to fatigue.

To address the issue of long-term durability for the Pullman test mixtures, full-scale pavement sections were constructed on SR 270. These sections were constructed as overlays 1.8 in. (4.6 cm.) thick. The exposure to Eastern Washington climatic cycles and highway traffic are providing insight into how the test mixtures deteriorate with time. The winter traffic in the Pullman area has a high percentage of vehicles with studded tires. After three winters of exposure, the SEA sections are experiencing higher amounts of surface aggregate loss in the wheel paths than the conventional asphalt concrete. The cause of this occurrence is due in part to the mechanical abrasion of studded tires.

Thus, from a structural viewpoint, the initial examination of SEA paving mixtures appeared promising or at least these mixtures were comparable to conventional asphalt concrete. From a durability viewpoint, this initial examination revealed that the SEA mixtures used at Pullman may be slightly inferior to the conventional asphalt paved at the same time.

Given the preceding background information, the primary objectives of this research study are:

1. Further evaluate the applicability and desirability of using SEA paving mixtures in the State of Washington.
2. Develop design criteria which will improve the utilization of SEA mixtures.
3. Assess the availability of sulfur in the State of Washington.

The last of the above objectives (availability of sulfur) is addressed in another project report.

In order to achieve the above objectives, the study contained the following basic steps (stated in approximate order of accomplishment):

1. Literature search
2. Design laboratory experimentation
3. Evaluate mixture design methods
4. Determine optimum mixture designs
5. Evaluate mixture durability
6. Develop conclusions and recommendations

Each of Steps 2-5 required various test methods and/or analyses which are reported primarily in Chapter II.

SEA MIXTURE DESIGN CRITERIA

The design of conventional asphalt concrete mixtures is a function of two primary variables - strength and durability. Strength is directly a function of such items as mixture density, aggregate and type and amount of binder. Durability is more difficult to simulate in the laboratory but is at least in part a function of the binder and aggregate quality, amount of air voids, etc.

Several methods are available to adequately evaluate the strength of conventional asphalt concrete. The most widely used are the Hveem and Marshall methods. Since SEA mixtures have somewhat differing characteristics during the mixing and compaction process (generally a lower viscosity), the question becomes "Are conventional mixture design methods adequate for designing SEA mixtures?" There have been various attempts to formulate an answer. The study has been used to examine this question.

For the Marshall mixture design procedure the following items are normally considered in selecting a final binder content:

1. Marshall stability
2. Flow
3. Percent voids in the total mixture
4. Percent voids filled with binder
5. Unit weight or density

For conventional mixtures, evaluation of these separate criteria are adequate but for SEA mixtures this may not always be the case. An example is Marshall stability. As shown in Table 1 for "heavy traffic" a minimum Marshall stability of 750 lbs (340 kg.) is required. This is a meaningful criterion for conventional mixtures but SEA mixtures tend to have significantly higher values, thus effectively reducing or eliminating the benefit of this criterion. This problem increases as the sulfur-asphalt ratio increases. Thus, one goal in the study has been to develop more realistic criteria for the two commonly used mixture design procedures.

Another question which has been examined is the concept of designing SEA mixtures to maximize (or minimize) physical material properties (current practice) or to design these mixtures on the basis of "equal strength". Since an SEA binder is normally more viscous at ambient temperature than asphalt cement, an SEA mixture which is designed to have strength equal to that of an "acceptable" conventional asphalt concrete is expected to require a lesser volume of binder. This concept has not been adequately examined in previous SEA studies. If reduced volumes of binders are appropriate for SEA mixtures, the potential asphalt savings will be increased. A possible problem with the equal strength concept is that a reduced binder volume content which demonstrates adequate strength may also produce a mixture which has poor durability.

Another issue which is not thoroughly understood is how well the sulfur blends with the asphalt and what process achieves the best blending. For example, SUDIC recommends the use of the Pronk in-line blender to achieve an intimate mixture of the liquid sulfur and asphalt. This is based on their belief that the Pronk blender produces the best SEA binder for the smallest cost. The U.S. Bureau of Mines has shown that direct addition of the sulfur and asphalt into the pugmill followed by thorough mixing with the aggregate is adequate. This approach appears to be the least expensive to use if it can be shown that it produces SEA binders comparable to the other processes.

SOLUBILITY OF SULFUR IN ASPHALT

While a considerable amount of research has been performed on methods of preparing the sulfur-asphalt binder used in SEA pavements, very little research has been undertaken to define the role sulfur plays in SEA mixtures and how sulfur is related to the physical and mechanical properties

Table 1. Marshall Design Criteria [After Ref. 8]

Traffic Category No. of Compaction Blows Each End of Specimen	Heavy 75		Medium 50		Light 35	
Test Property	Min.	Max.	Min.	Max.	Min.	Max.
Stability, all mixtures, (lb)	750	-	500	-	500	-
Flow, all mixtures, (0.01 in.)	8	16	8	18	8	20
Percent air voids						
Surfacing or Leveling	3	5	3	5	3	5
Base	3	8	3	8	3	8

1 N = 2.248 lbf

1 mm = 0.039 in.

of the pavements [1]. The purpose of this section is to examine the information currently available on the solubility of sulfur in SEA binders. This information will provide insight into the experimental data collected during this study and hence the effect of sulfur on the behavior of an SEA mixture.

Sulfur exists in many allotropic forms which differ in their physical and chemical properties [2]. The principal allotropics are orthorhombic (S_α), monoclinic (S_β), and polymeric sulfur (S_χ). S_α and S_β are crystalline materials consisting of sulfur rings. S_χ consists of chains of up to 10^6 sulfur atoms.

Sulfur crystallizes as a monoclinic polymorph which on cooling to room temperature reverts to an orthorhombic form [3]. The reversion rate has been measured and is shown in Figure 1. S_α is the only allotropic form of sulfur that is stable under ordinary conditions of temperature and pressure.

A valid and important question is, "How much sulfur can be "dissolved" in asphalt"? In other words, how much sulfur can be used as an asphalt extender.

The chemical reaction between the sulfur component and the hydrocarbon of the asphalt is known to occur with the basic chemistry akin to vulcanization [4]. When sulfur and asphalt are heated and combined in an emulsion, three distinct types of reactions can occur:

1. The sulfur can react chemically with the asphalt and result in dehydrogenation.
2. The sulfur can be dissolved in the asphalt.
3. Sulfur in the crystalline form can remain in suspension in the asphalt [5].

The mode of reaction which results in the dissolved sulfur is considered to be sulfurization through attack of the double bonds in the asphalt cement [6].

Sulfur can be dissolved in asphalt at temperatures below 300°F (149°C) The dissolving mechanism is believed to be separate from the chemical dehydrogenation reactions [5]. At temperatures above about 305°F (152°C), dehydrogenation occurs and hydrogen sulfide gas is formed, which results in significant changes in the rheological properties of the asphalt. The sulfur that does not chemically combine with the asphalt may be dissolved in true solution, dispersed as a colloid, or appear in the asphaltic mixture as crystalline sulfur. In this form, it can undergo allotropic interconversion, both at the molecular and macroscopic levels [4].

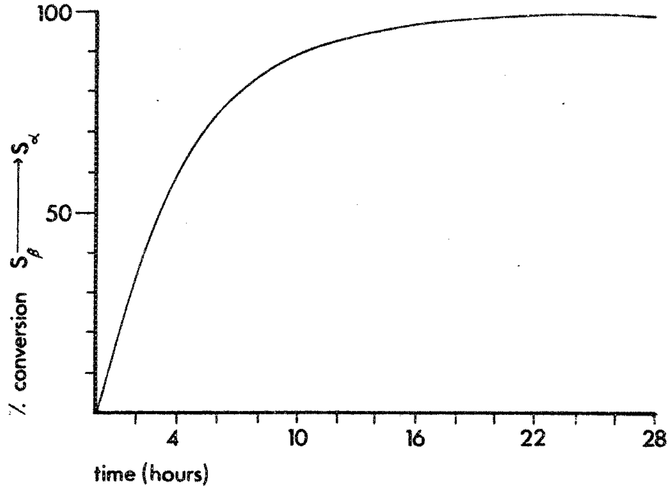

Figure 1. Reversion Rate of Monoclinic Sulfur to Orthorhombic Sulfur at Ambient Temperature [3].

The relative amounts of dissolved sulfur in the sulfur-asphalt emulsions have been determined by use of differential thermal analysis (DTA) equipment. DTA equipment operates on the principle that thermal energy is absorbed (endothermal reaction) or evolved (exothermal reaction) during a physical or chemical change as a sample of material is heated [5]. A composite typical data trace is shown in Figure 2. The area under the portion of the thermogram at the melting point was used in calculating the amount of crystalline sulfur. The monoclinc sulfur (S_β) will revert to orthorhombic sulfur (S_α), but at least 20 hours is required for this reaction to occur completely. The relative amount of dissolved and crystalline sulfur for typical SEA blends is presented graphically in Figure 3. Similar results have been obtained by use of the differential scanning calorimetry (DSC) [6].

Tests conducted by Garrigues [7] indicate that solubilities of sulfur in asphalt cement were 14 and 18 percent by weight at mixing temperatures of 302°F (150°C) and 356°F (180°C), respectively. Kennepohl reports that up to 19 percent by weight can be dissolved [6]. Pickett, et al, [5] reported measured solubilities (using DTA equipment) ranging from a low of 10 percent to a high of about 23 percent by weight of binder.

Matrecon, Inc., of Oakland, California was retained by the study team to measure various characteristics of the SEA binders produced for the Pullman test pavements. By use of gel permeation chromatography (GPC) and differential scanning calorimetry (DSC), the dissolved sulfur was calculated based upon the difference between the free sulfur content (obtained by GPC) and the crystalline sulfur content (obtained by DSC). The results indicate that approximately 10 to 14 percent of the sulfur was dissolved (original binder was a 30/70 SEA with AR-4000W Husky asphalt cement).

McBee [1] used a different approach in determining the solubility of sulfur in asphalt cement. Diffusion couples were made by laying opposing layers of elemental sulfur and asphalt in a glass vial. The diffusion occurred at various temperatures and time periods. Count rates for sulfur K_α X-rays were measured as a function of distance from the sulfur-asphalt interface. The count rates from the diffusion couple can be converted to sulfur concentrations which in turn can be used to calculate the solubility of elemental sulfur in asphalt. The solubility trend so derived as a function of temperature is shown in Figure 4.

This solubility data indicates a decrease in sulfur solubility with decreasing temperature. A low solubility at ambient temperatures of approximately 4 percent is indicated. The result is large amounts of crystalline sulfur existing in the paving mixture generally as "needle-like" structures. The driving force for nucleation and growth of the sulfur needles is believed to be provided by the solubility differences at

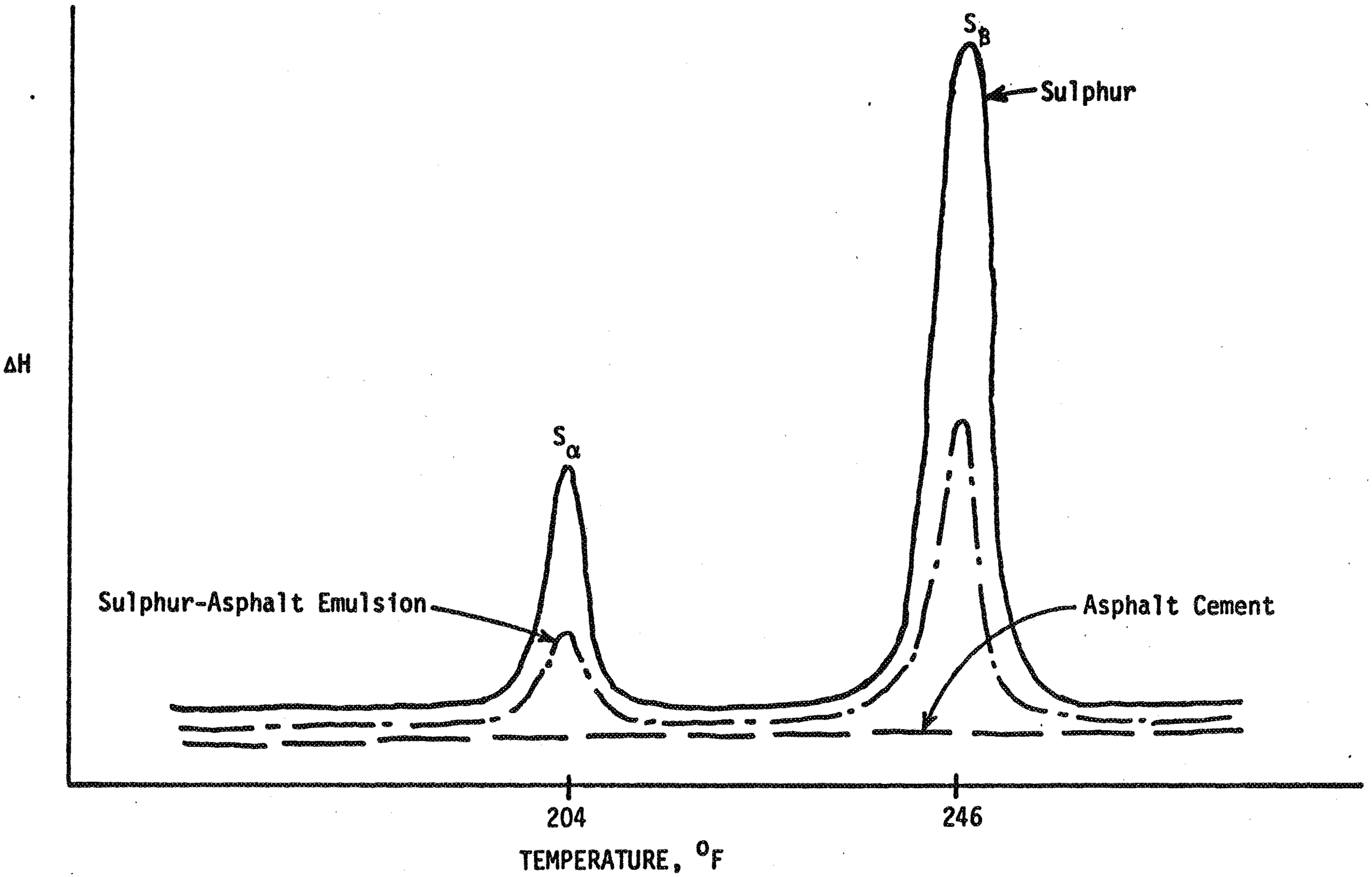

Figure 2. Typical Composite DTA Trace to Determine Solubility of Sulfur in Sulfur-Asphalt Emulsions [5].

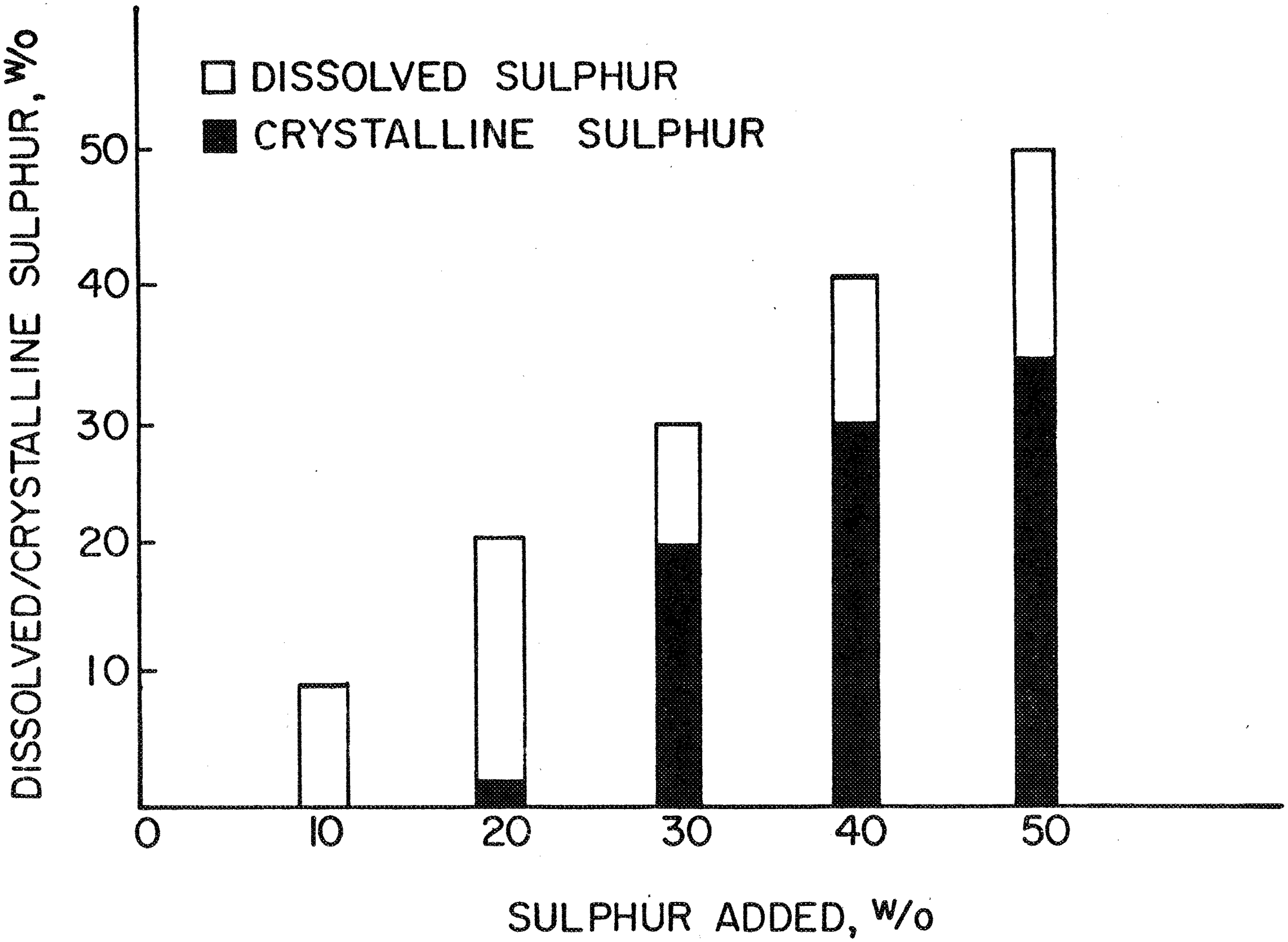

Figure 3. Dissolved and Crystalline Sulfur Determined by DTA [5].

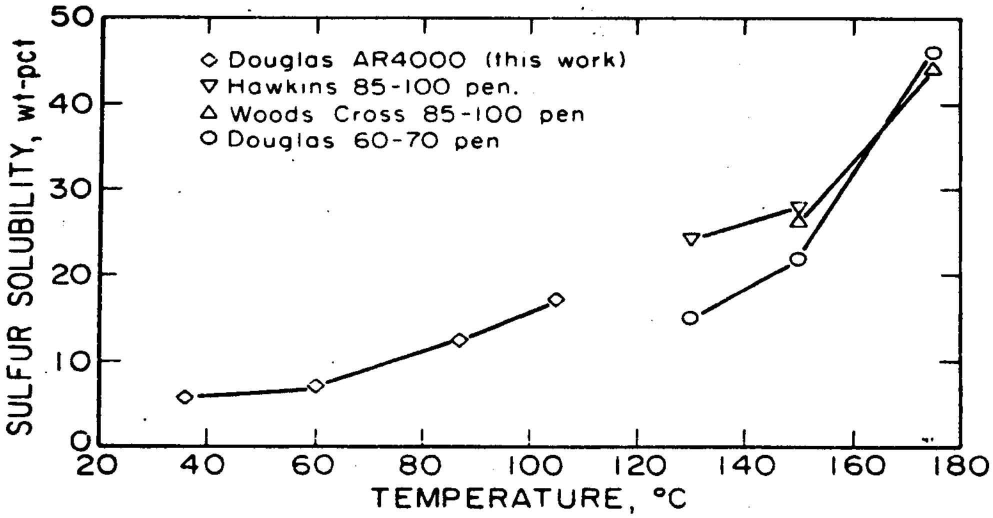

Figure 4. Solubility of Sulfur Measured in Several Asphalts [1]

mixing and ambient temperatures [1]. The solubility data could be underestimated because the amount of "dissolved" sulfur could be more than that of diffused due to the constraints of surface energy and other interface factors.

Available sulfur solubility data [14, 15] for coal-tar oil, olive oil, and linseed oil indicate that the solubility of sulfur in these oils decreases with decreasing temperatures. The following further illustrates this trend:

	Dissolved Sulfur (%)			
Temperature °F (°C)	Asphalt Cement(1)	Coal Tar Oil(2,3)	Olive Oil(2,4)	Linseed Oil (2)
59 (15)	-	6.0	2.3	0.4
122 (50)	7	10.0	9.0	1.2
194 (90)	12	44.8	21.5	2.6
266 (130)	15-25	-	43.0	5.0
320 (160)	33	-	-	10.0

(1) After Ref. 1
(2) After Ref. 14
(3) Specific gravity = 1.01 and boiling point = 410-572°F (210-300°C) for base coal-tar oil
(4) Specific gravity = 0.885 for olive oil

If we examine the temperature range of 122 to 266°F (50 to 130°C) (a range representing the temperature of a pavement on a warm, sunny day up to normal SEA mixing temperatures), the solubility of sulfur in three of the listed oils (including the asphalt cement) decreases by about a factor of 4 to 5 for the 144°F (80°C) decrease in temperature. The data further indicates that this factor increases to a range of about 10 to 20 for a temperature drop from 266 to 59°F (130 to 15°C) (olive and linseed oils only). Thus, if a similar trend holds for asphalt cements, the solubility of sulfur at modest ambient temperatures (say 59°C (15°C)) might be as low as 2 to 6 percent. However, this solubility percentage is speculative at this time.

Thus, it is not clear how much sulfur is actually "dissolved" in a typical SEA binder at ambient temperatures. The lowest potential solubility appears to be about 4 percent. However, at binder mixing temperatures, the solubility of sulfur in asphalt appears to be about 18 percent. The end result is that the larger the amount of added sulfur to an asphalt the larger the amount of sulfur not in solution and hence existing in a crystalline state. The mechanism of how sulfur exists in a paving mixture will dictate in part how the paving mixture will perform with regard to fatigue and durability.

CHAPTER II

LABORATORY EVALUATION OF SEA MIXTURES

The following items were required to properly evaluate the SEA mixtures examined in this study:

1. Design laboratory experiment.
2. Evaluate mixture design methods.
3. Determine optimum mixture designs.
4. Evaluate mixture durability.
5. Develop appropriate mixture design criteria.

The approach and results relating to the above items will be addressed in the sections contained in this chapter.

EXPERIMENTAL DESIGN

The experimental design included the following major variables:

1. SEA ratios:
    - (a) 0/100 (conventional asphalt concrete)
    - (b) 20/80 (20 percent added sulfur, 80 percent asphalt cement by weight)
    - (c) 30/70 (30 percent added sulfur, 70 percent asphalt cement by weight)
    - (d) 40/60 (40 percent added sulfur, 60 percent asphalt cement by weight)
    - (e) 50/50 (50 percent added sulfur, 50 percent asphalt cement by weight).
2. Aggregates:
    - (a) Eastern Washington crushed basalt
    - (b) Western Washington crushed gravel
3. Asphalt cements:
    - (a) Chevron AR-4000W
    - (b) Chevron AR-2000

4. Mixture compaction methods:

    (a) Kneading compaction (Hveem)
    (b) Marshall compaction

The above mixture variables represent a wide range of mixture conditions for the types of paving materials commonly used throughout the State of Washington.

The types of tests conducted on the laboratory prepared binders and mixtures included:

1. Binder tests:

    (a) Viscosity (Sliding Plate @ 77°F (25°C))
    (b) Penetration @ 77°F (25°C) @ 100 g., 5 sec, ASTM D5
    (c) Scanning electron microscope with photographic and X-ray scans
    (d) Determination of "natural" sulfur content in asphalt cements

2. Mixture tests:

    (a) Kneading compaction (WSDOT Test Method 701)

        (i) Stabilometer value (WSDOT Test Method 703)
        (ii) Bulk specific gravity (WSDOT Test Method 704)
        (iii) Maximum specific gravity (WSDOT Test Method 705)
        (iv) Marshall stability and flow (ASTM D1559)

    (b) Marshall compaction (ASTM D1559)

        (i) Marshall stability and flow (ASTM D1559)
        (ii) Bulk specific gravity (WSDOT Test Method 704)
        (iii) Maximum specific gravity (WSDOT Test Method 705)

    (c) Tests common to all compacted mixtures

        (i) Resilient Modulus (ASTM "Indirect Tensile Test Method for Resilient Modulus of Bituminous Mixtures")
        (ii) Conditioning tests - details provided in section on mixture durability and Appendix G
        (iii) Scanning electron microscope with photographic and X-ray scans

INDIVIDUAL MATERIAL CHARACTERISTICS

AGGREGATES

The Eastern Washington crushed basalt was obtained from a quarry operated by United Paving, Inc. (WSDOT Designation QS-P-95) located near Pullman, Washington. The selection of this aggregate source was primarily due to its use for the SEA experimental pavements constructed in Pullman in 1979. Previously conducted tests on this aggregate revealed the following bulk specific gravities and percent moisture absorption [9]:

1. Bulk specific gravity:

    (a) Coarse (ASTM C127): 2.75
    (b) Fine (ASTM C128): 2.75

2. Percent absorption:

    (a) Coarse (ASTM C127): 2.14
    (b) Fine (ASTM C128): 2.09

The percent asphalt absorption as measured by WSDOT [9] was 1.3 percent.

The Western Washington crushed gravel was obtained from the gravel pit operated by Lone Star Industries (WSDOT Designation PS-B-1) located near Steilacoom, Washington (approximately 50 mi. (80.5 km) south of Seattle on the eastern shore of Puget Sound). This aggregate can be broadly classified as a glacial gravel (crushed granite). Previously conducted tests reveal the following bulk specific gravities and percent moisture absorption [11]:

1. Bulk specific gravity:

    (a) Coarse: 2.64
    (b) Fine: 2.43

2. Percent absorption:

    (a) Coarse: 1.12
    (b) Fine: 5.49

The Los Angeles abrasion for this aggregate has been previously shown to be relatively low - approximately 15 percent [11].

The aggregate gradation used for both aggregates was in accordance with the WSDOT Class B specification (Table 2).

ASPHALT CEMENTS

Both asphalt cements used in the study were produced by Chevron U.S.A. The AR-4000W was obtained from the Chevron, Richmond Beach facility (Seattle) and the AR-2000 was shipped to UW from the Chevron, Portland, Oregon facility. Initially, it was planned to use both Chevron and Husky asphalts of the same viscosity grading. However, it became apparent that it would be of greater value to use a range of asphalt cement viscosities; hence, the use of the AR-4000 and 2000 grades.

Characterization tests on the original asphalt cements are as follows:

1. Viscosity (sliding plate) @ 77°F (25°C)

    (a) AR-4000: 1.3×10^6 poises
    (b) AR-2000: 0.72×10^6 poises

2. Penetration @ 77°F (25°C) @ 100 g., 5 sec.

    (a) AR-4000: 89
    (b) AR-2000: 151

3. Total sulfur content in original asphalt cement (by weight)*

    (a) AR-4000: 2.34%
    (b) AR-2000: 4.34%

Measurements of viscosity (sliding plate) and penetration were made with time for the SEA binder combinations studied. This data is summarized in Appendix H. Initially, the binder viscosity was lower and the penetration was higher than the original asphalt cements for increasing amounts of added sulfur. However, with time, the trend that evolved was that the binders containing added sulfur exhibited higher viscosities and lower penetration values than the original AR-4000 and 2000 asphalt cements.

SULFUR

The sulfur used was an 80-mesh ground sulfur from the Montana Sulfur and Chemical Company, Billings, Montana. The sulfur was not tested for purity due to the attested purity stated by the producer.

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*Tests conducted by Matrecon, Inc., Oakland, California during Jan.-Mar., 1982.

Table 2. Aggregate Gradation Used in Laboratory Mixtures (WSDOT Class B)

Sieve Size	Actual Cumulative Percent Passing	WSDOT Specification Limits [10]
5/8"	100	100
1/2"	95 (100)*	90-100
3/8"	82	75-90
1/4"	65	55-75
No. 10	40	32-48
No. 40	18	11-24
No. 80	10	6-15
No. 200	5	3-7

*Pullman Aggregate: 100% passed 1/2" sieve

MIXTURE DESIGN

DETERMINATION OF OPTIMUM BINDER CONTENTS

For approximately the last ten years, numerous SEA mix designs have been performed by various agencies and investigators. Much of this knowledge is summarized by McBee, et al [12] which contains guidelines for the design and construction of SEA paving mixtures. A brief summary of those mix design guidelines is as follows:

1. Determine the optimum binder content for a conventional mix, using either the Hveem or Marshall method.
2. Select an SEA ratio (i.e., 20/80, 30/70, etc.) and calculate the equivalent volume replacement of binder (in weight-percent) by the following equation:

$$\text{SEA (equal volume equivalent) wt-pct} = \frac{10000\ AR}{10000R - 100P_s(R-1) + AP_s(R-1)}$$

where:

A = weight percent asphalt cement in conventional design

R = sulfur substitution ratio = $\frac{G_s}{G_a}$

P_s = weight percent sulfur in the SEA binder

G_a = specific gravity of asphalt at ambient temperature

G_s = specific gravity of sulfur at ambient temperature

(Note: sulfur is about twice as dense as asphalt cement, therefore a given weight of sulfur has about 1/2 the volume of the same weight of asphalt cement).

3. Using the values obtained in Steps 1 and 2 as minimum and maximum values, respectively, choose intermediate points to evaluate.
4. Use either the Hveem or Marshall methods to prepare samples for testing to determine the optimum binder content.

In general, these guidelines were followed during the early mix design phase of this study. It soon became apparent that the optimum binder contents obtained in Step 4 were essentially the same as those calculated by use of the equivalent volume formula (Step 2) for a sulfur substitution ratio of slightly less than 2.0. Therefore, throughout the remainder of the study, the optimum binder contents were those which provided an equal volume of binder. The optimum binder contents are summarized in Table 3 with specific mix design data contained in

Table 3. Summary of Optimum Binder Contents for Marshall and Hveem Mix Designs

Aggregate	Asphalt*	Compaction Technique	Optimum Binder Contents			
			0/100 SEA	20/80 SEA	30/70 SEA	40/60 SEA
Western Washington Crushed Gravel	AR-4000	Marshall	6.0	6.4	6.8	7.2
	AR-2000		5.8	6.4	6.6	7.1
	AR-4000	Hveem (Kneading)	6.0	6.5	6.9	7.3
	AR-2000		5.8	6.4	6.7	7.1
Eastern Washington Crushed Basalt	AR-4000	Marshall	5.5	6.0	6.4	6.7
	AR-2000		5.5	6.1	6.4	6.8
	AR-4000	Hveem (Kneading)	5.5	6.0	6.4	6.7
	AR-2000		5.5	6.1	6.4	6.8

*Sp. Gr. Chevron AR-4000 = 1.023
Sp. Gr. Chevron AR-2000 = 1.017

Appendix D. It should be noted that this finding is supported by the results of the laboratory mix design phase of the Pullman SEA study as reported in Reference 9.

The initial mix designs were tested for stability (Marshall or Hveem) two days after compaction. This was later changed to 14 days to help account for the time dependent strength gain of SEA mixtures. These differences can be observed in the detailed mix design data contained in Appendix D.

After analyzing the various mixtures prepared for this study including the SEM data, the concept of reducing the volume of binder in an SEA mixture to produce a mix with strength approximately equal to conventional asphalt concrete does not appear desirable. While the strength of the SEA paving mixture may be adequate, the durability probably will be poor. The decreased durability can be attributed to one or more of the following:

1. Excessive amount of air voids
2. Thinner coating of binder
3. Reduced effectiveness of binder in the presence of water due to added sulfur.

These factors will be further illustrated in the section on moisture conditioning.

The evaluation of the two mixture design methods (Hveem and Marshall) with respect to the preparation of SEA mixtures is further described in the sections which follow. Overall, as one might expect, the optimum binder contents obtained for both methods are essentially the same for a range of mixture combinations (Table 3).

MIXTURE DURABILITY

INTRODUCTION

One of the most important features of this study was the investigation of SEA mixture aging and other environmentally induced mixture deterioration. The reason for this emphasis area is at least twofold. First, the Pullman test pavements (SR 270 sections) have shown that the SEA paved sections experienced greater amounts of surface aggregate loss than did the conventional asphalt concrete sections. Even though the differences were not great, the matter required further examination. Second, a review of various construction materials which contained added sulfur generally revealed susceptibility to moisture and/or freeze-thaw damage.

In general, pre-study data indicated that it is advisable to thoroughly investigate SEA mixture durability prior to construction of large-scale SEA projects in Washington. The preferable approach would be to observe field mixture changes (or performance) with time for test sites with various environmental conditions. However, the time and expense associated with such research is probably not necessary at this time. Thus, accelerated laboratory tests were required to achieve the full evaluation of the SEA mixtures.

The conditioning tests selected for use in the study included the following:

1. Accelerated aging conditioning @ 140°F (60°C),
2. Wet freeze-thaw conditioning ("Lottman" procedure).

The "aging" conditioning was conducted in order to study the physiochemical aging phenomenon and its effect on mixture resilient modulus properties. This in effect accelerated the sulfur recrystallization process thus providing estimates of long-term mixture stiffness values.

The wet freeze-thaw conditioning was patterned after the procedures developed by Lottman at the University of Idaho [3] and recently revised (refer to Appendix G). The objective of this conditioning process was to determine the mechanical properties of a given mixture due to the combined effects of moisture and freeze-thaw cycling. This information was then compared to the same properties obtained from pre-conditioning testing. The resulting ratios can be used as a relative index of environmental susceptibility.

Both the aging and moisture conditioning procedures were initiated 14 days after the sample mixtures were compacted; hence, the initial mixture stiffnesses shown were obtained 14 days after sample preparation.

AGING CONDITIONING

The general trend for all samples subjected to the aging conditioning procedure was an increase in resilient modulus with time. These relationships are shown in Figures 5 through 12. However, as shown in Table 4, the 20/80 SEA mixtures had the highest percent of strength gain (186%) followed by the 30/70, 0/100 and 40/60 SEA mixtures, respectively (the 50/50 SEA mixtures were not considered as the number of samples tested was small).

In reviewing the information available on the solubility of sulfur in asphalt, an explanation for the greater increase in stiffness for the 20/80 mixtures can be found. As was stated earlier, the solubility of sulfur in asphalt appears to be about 18 to 20 percent at mixing temperatures and 4 to 10 percent at ambient temperatures. Also, sulfur that is not in solution crystallizes to form "needle-like" structures and it is these structures which are thought to be the cause of increased stiffness in SEA mixtures. Therefore, it is reasonable to expect that

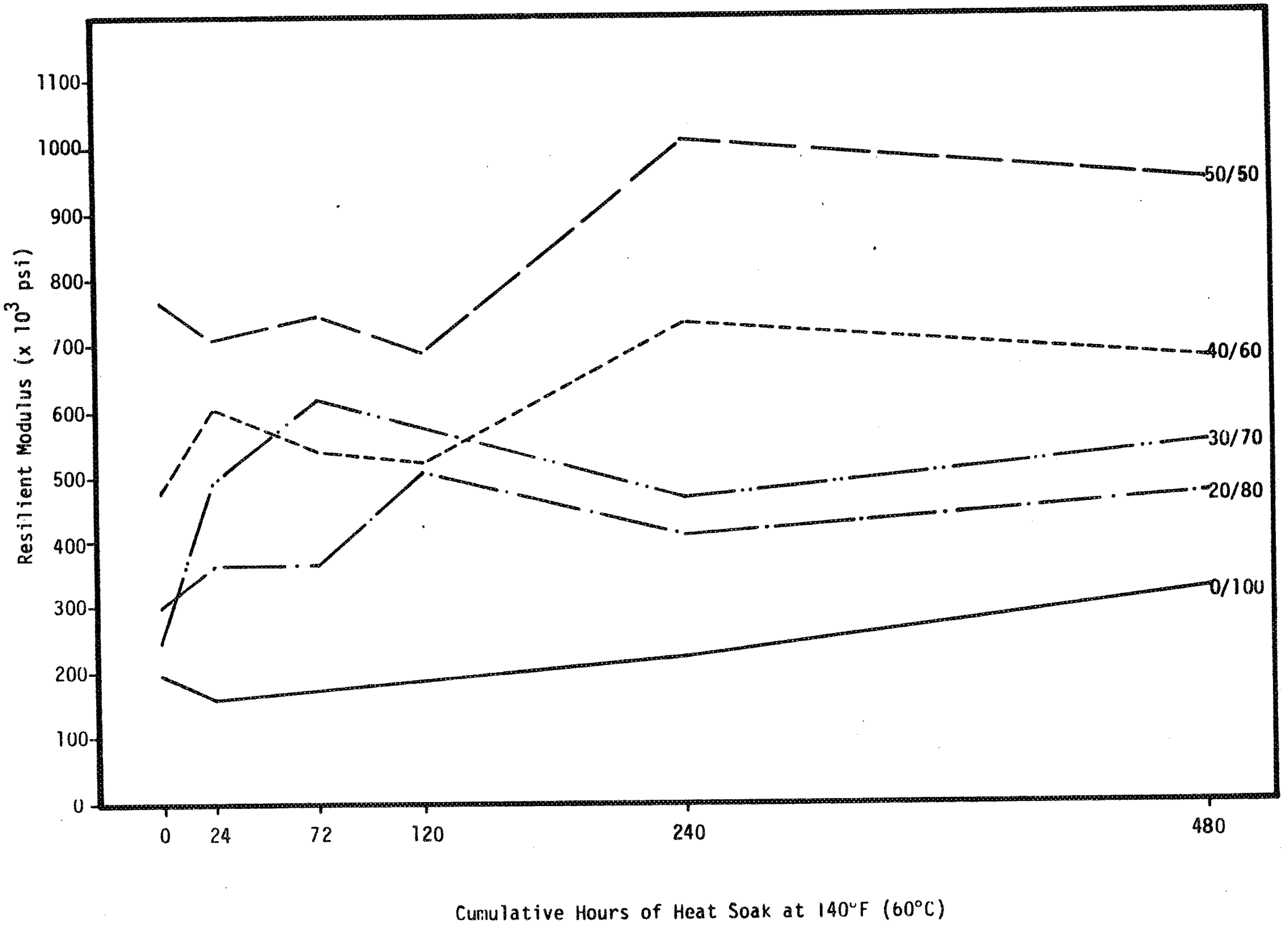


Figure 5. Resilient Modulus After Heat Soaking (AR-4000, crushed gravel, Marshall compaction, test temp: 77°F (25°C))

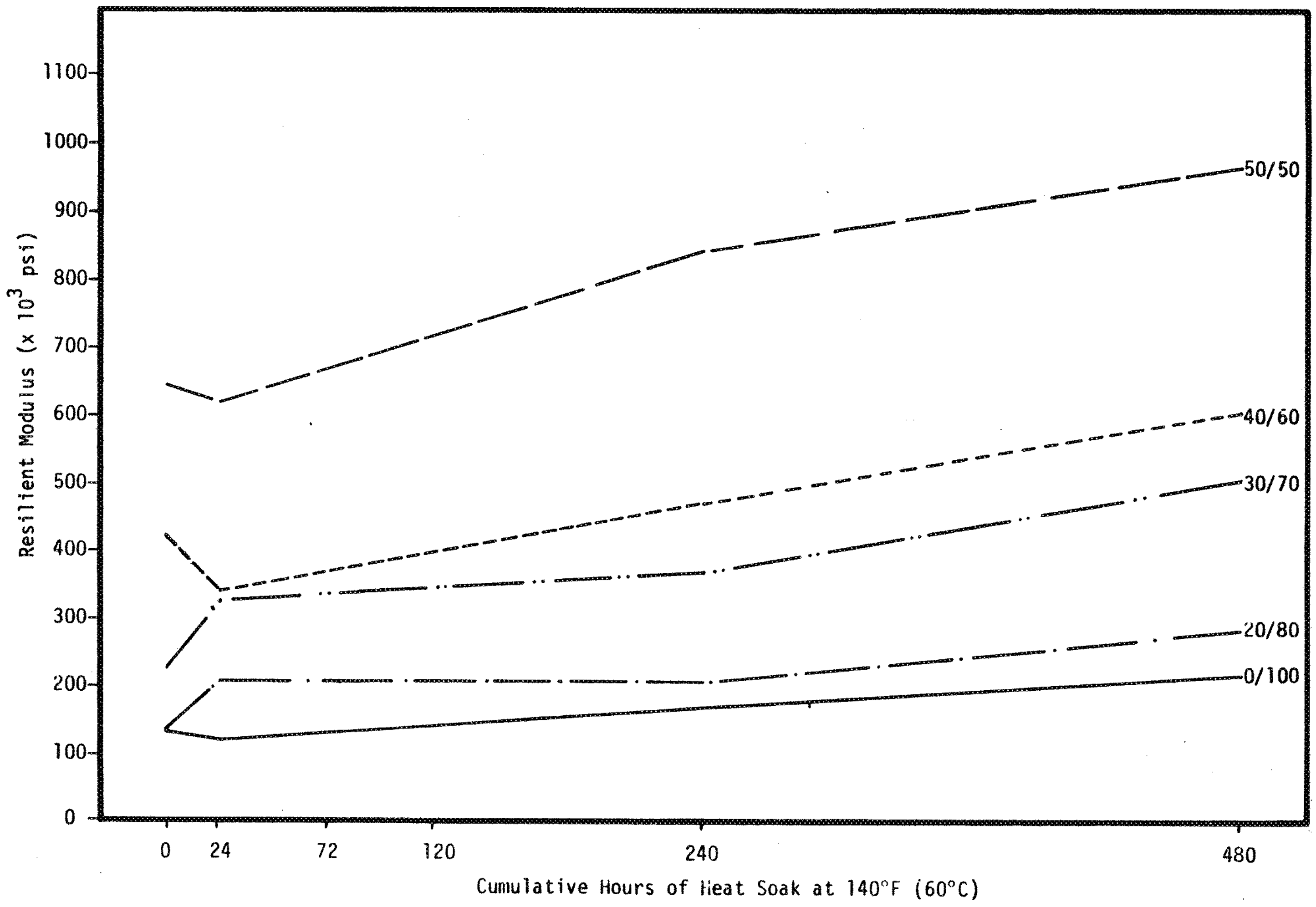


Figure 6. Resilient Modulus After Heat Soaking (AR-2000, crushed gravel, Marshall compaction, test temp: 77°F (25°C))

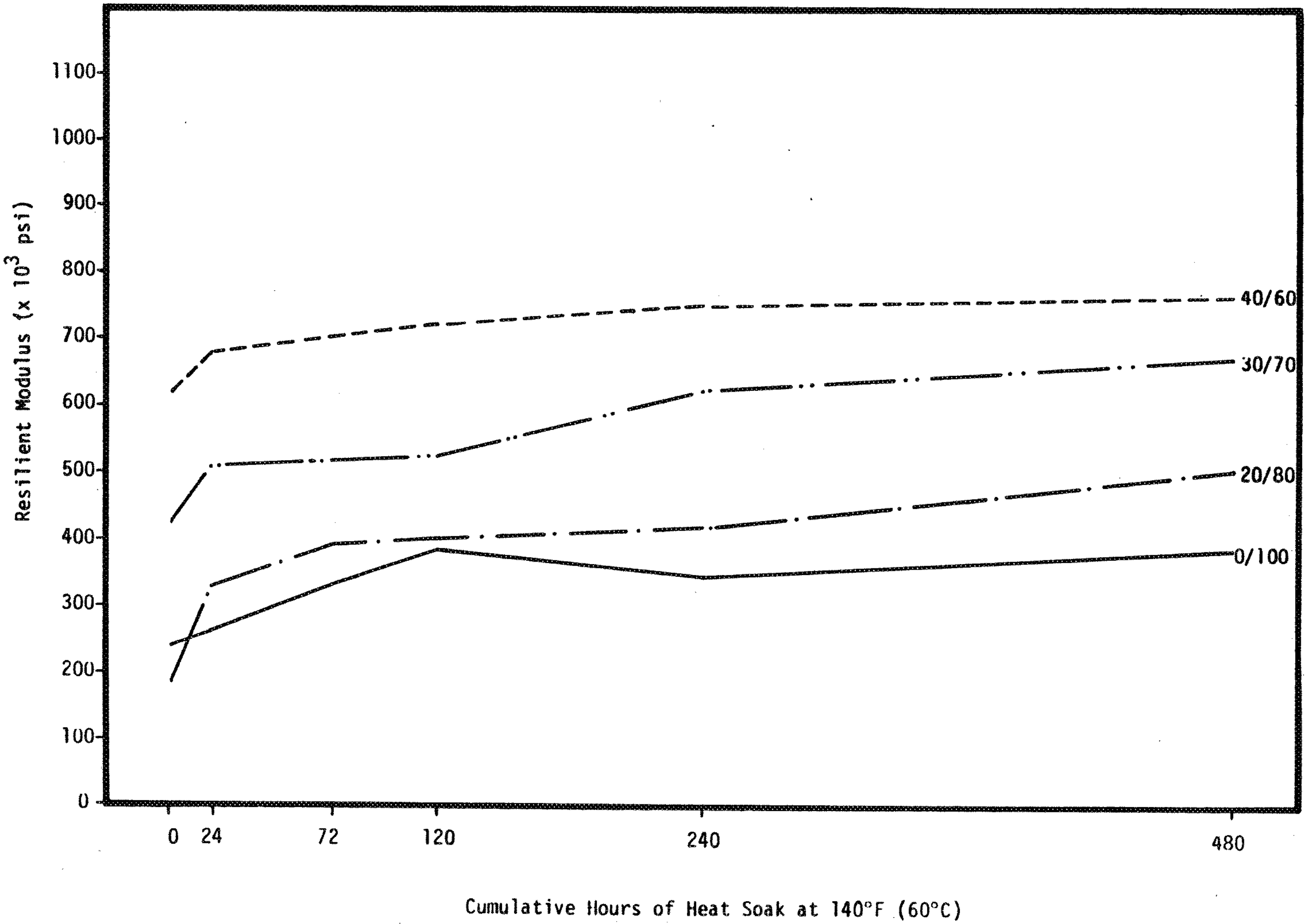


Figure 7. Resilient Modulus After Heat Soaking (AR-4000, crushed basalt, Marshall compaction, test temp: 77°F (25°C))

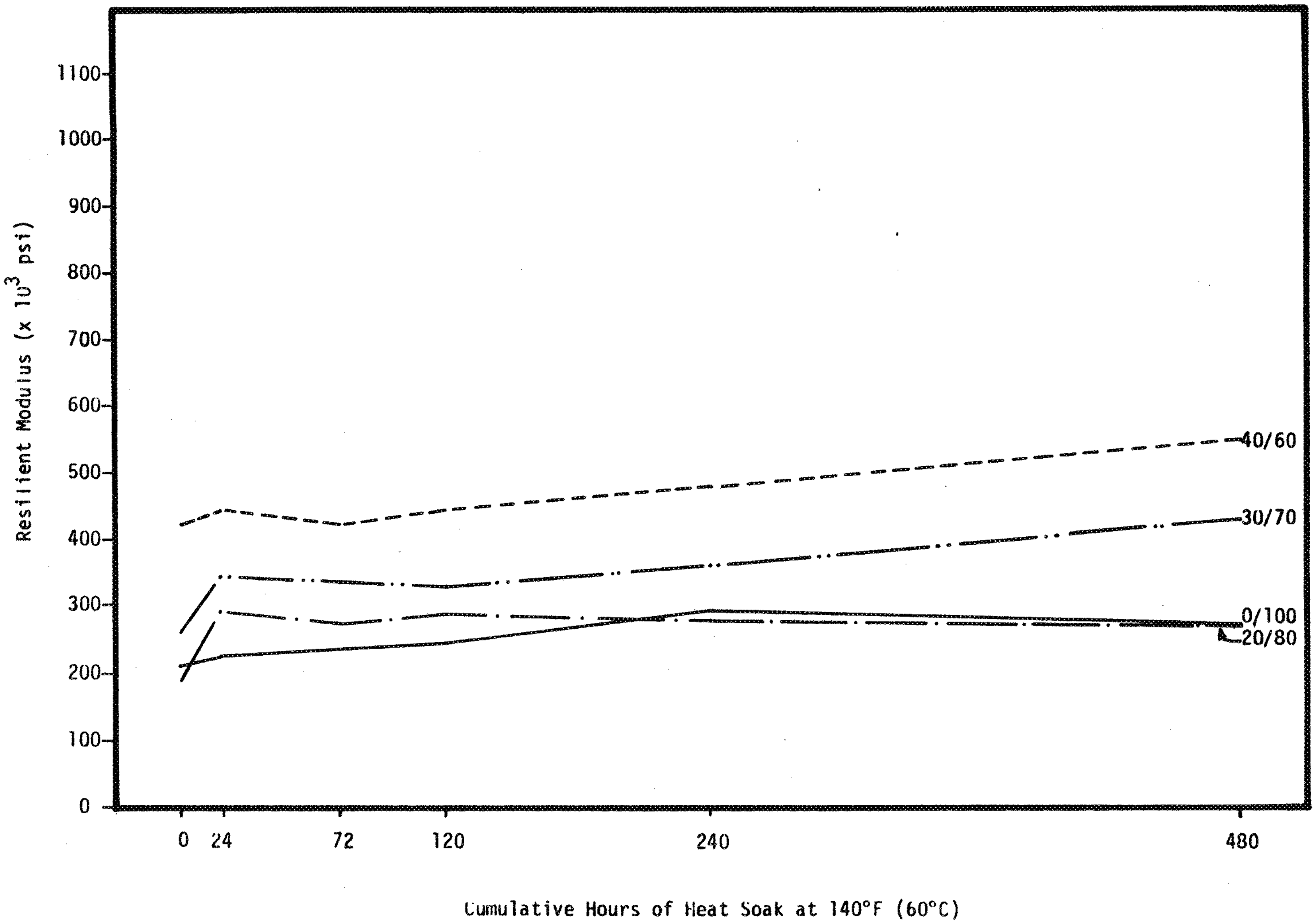


Figure 8. Resilient Modulus After Heat Soaking (AR-2000, crushed basalt, Marshall compaction, test temp: 77°F (25°C))

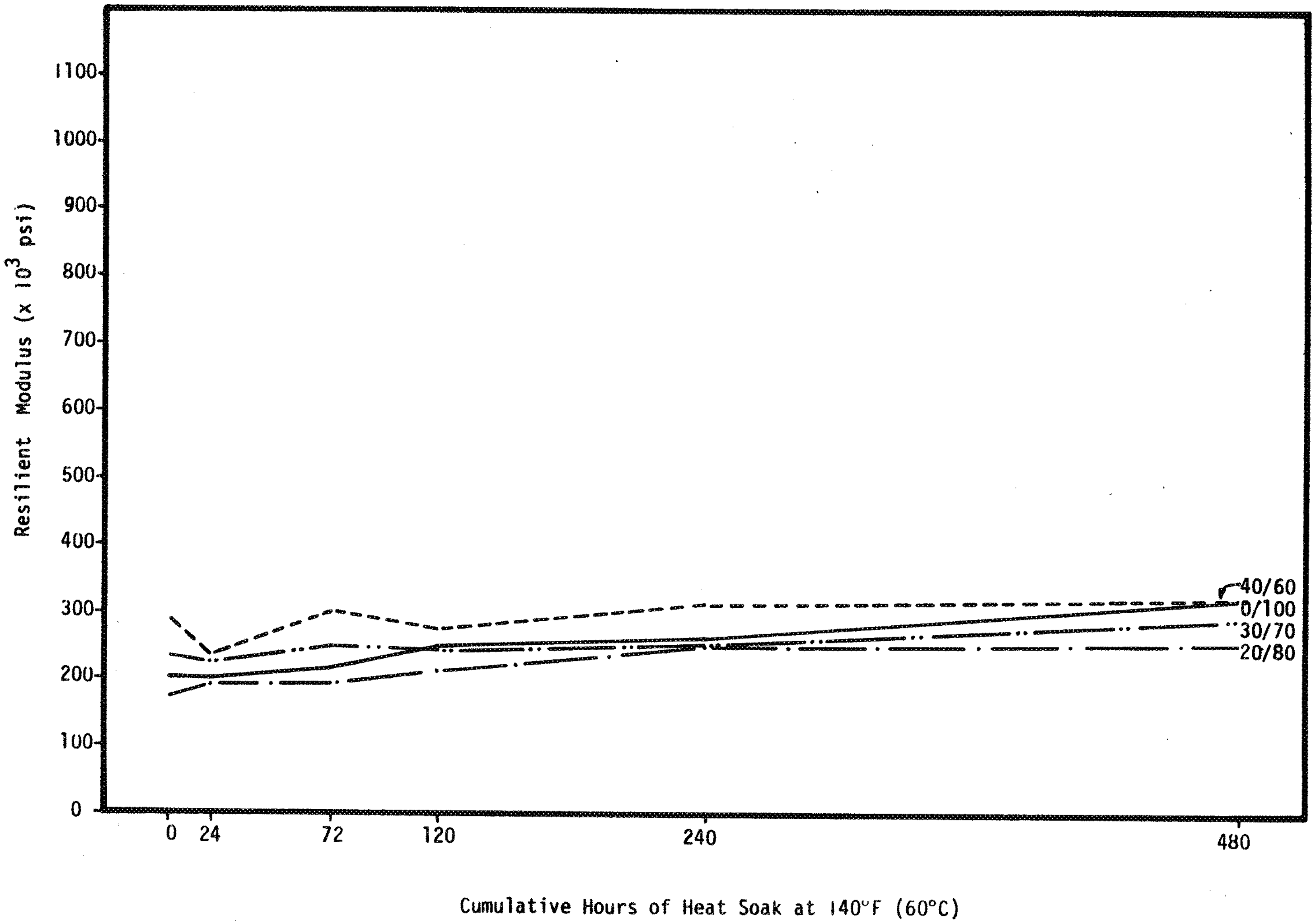


Figure 9. Resilient Modulus After Heat Soaking (AR-4000, crushed gravel, kneading compaction, test temp: 77°F (25°C))

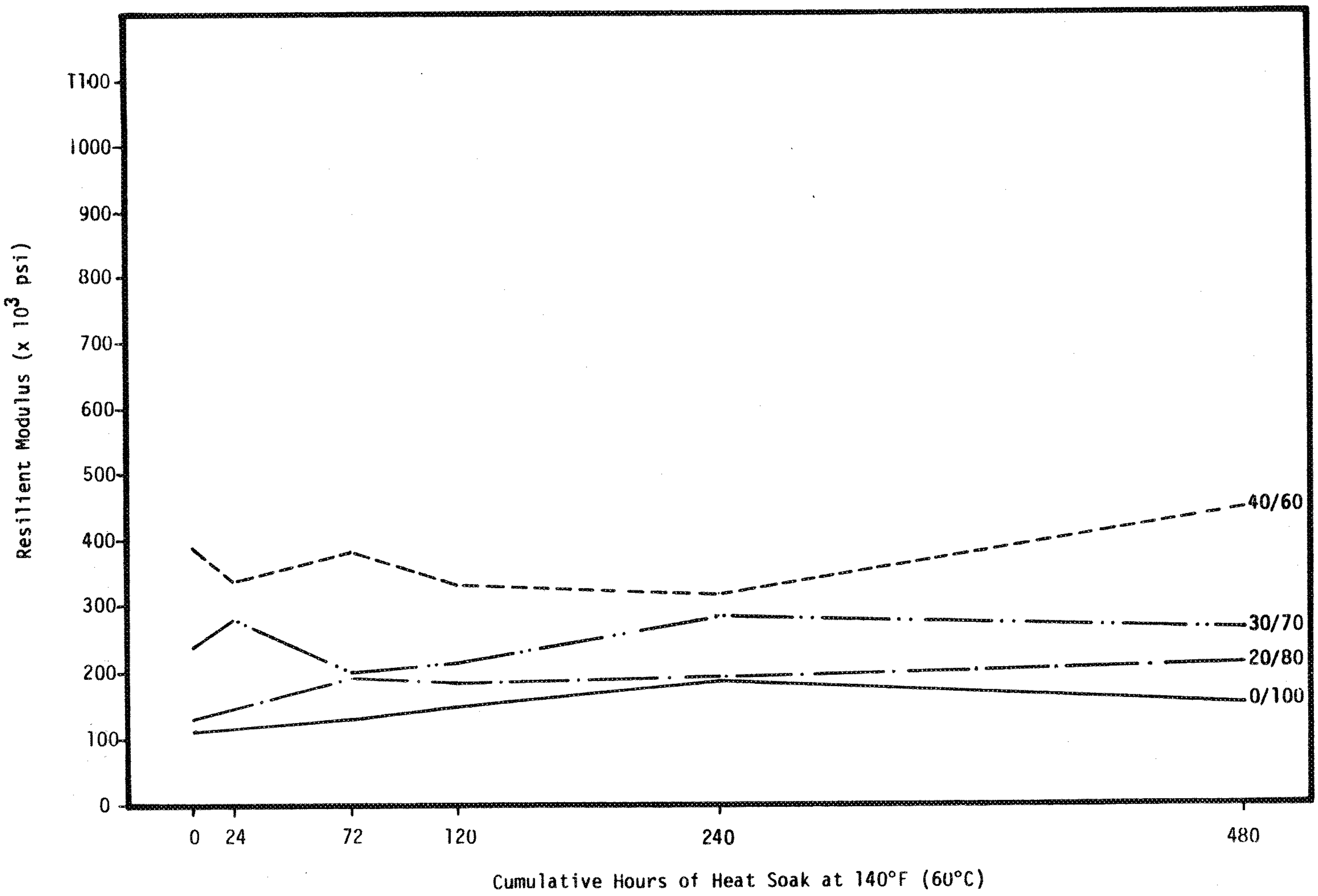


Figure 10. Resilient Modulus After Heat Soaking (AR-2000, crushed gravel, kneading compaction, test temp: 77°F (25°C))

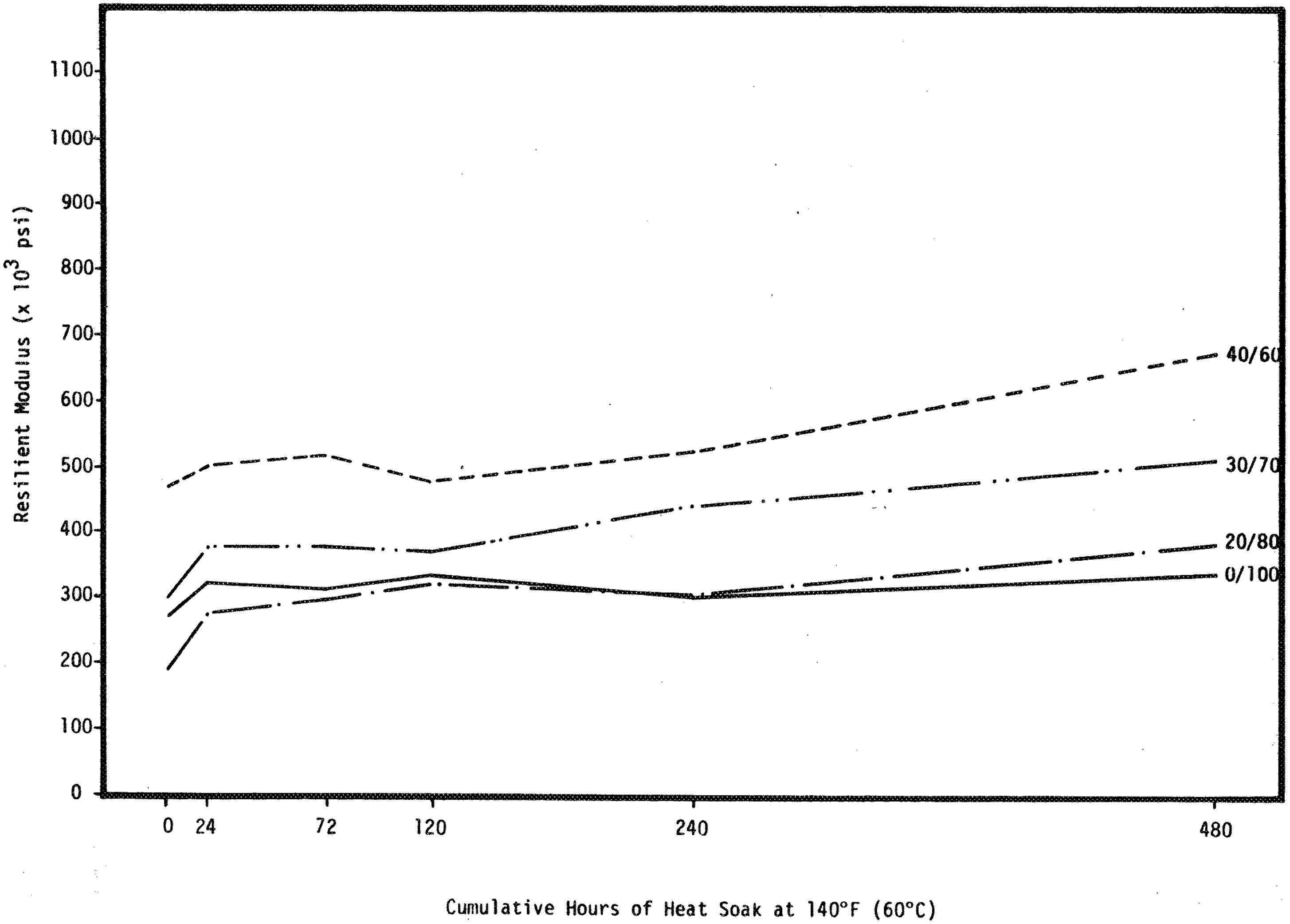


Figure 11. Resilient Modulus After Heat Soaking (AR-4000, crushed basalt, kneading compaction, test temp: 77°F (25°C))

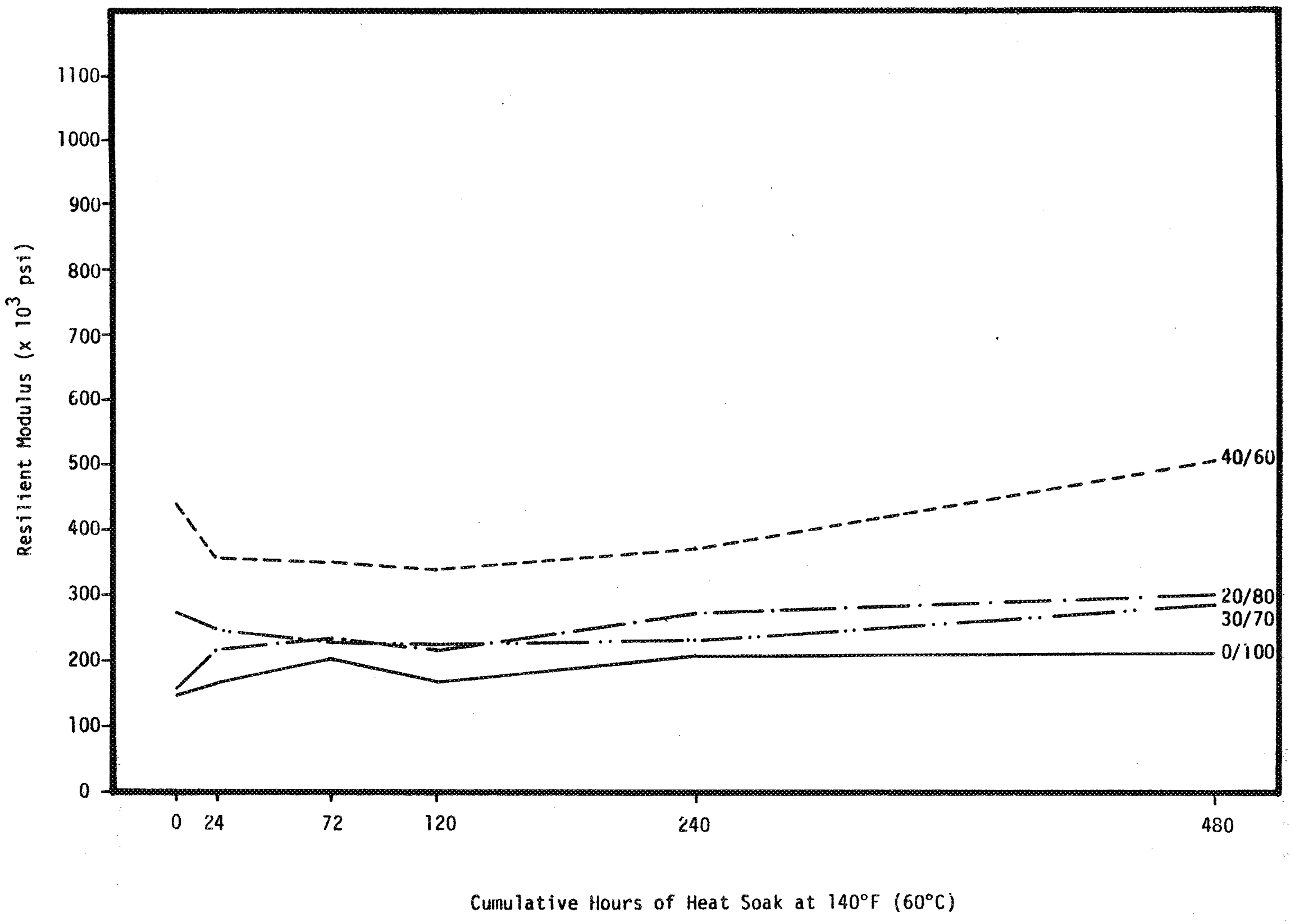


Figure 12. Resilient Modulus After Heat Soaking (AR-2000, crushed basalt, kneading compaction, test temp: 77°F (25°C))

Table 4. Percent Stiffness Gained During Aging for SEA Mixtures

SEA Ratio / Sample Type	Percent Strength Gained				
	0/100	20/80	30/70	40/60	50/50
Marshall Compaction, Crushed Gravel, AR-4000	165	157	219	143	124
Marshall Compaction, Crushed Gravel, AR-2000	167	210	226	144	150
Marshall Compaction, Crushed Basalt, AR-4000	162	274	158	124	-
Marshall Compaction, Crushed Basalt, AR-2000	130	144	166	132	-
Kneading Compaction, Crushed Gravel, AR-4000	162	148	124	114	-
Kneading Compaction, Crushed Gravel, AR-2000	134	165	112	116	-
Kneading Compaction, Crushed Basalt, AR-4000	126	200	171	144	-
Kneading Compaction, Crushed Basalt, AR-2000	146	190	105	116	-
Average	149	186	160	129	137

since approximately all of the sulfur in the 20/80 mixtures is in solution at the time of mixing, the initial stiffnesses will be approximately the same as the 0/100 (conventional) mixtures. And, it is reasonable to assume that the increase in stiffness with age as the sulfur comes out of solution and forms crystals will be greater than that expected due to normal asphalt cement stiffening.

This was indeed the case as shown by the fact that the 20/80 mixtures gained more stiffness than the 0/100 mixtures. It would also be expected that the initial stiffnesses (measured 14 days after compaction) for the 30/70 and 40/60 mixtures would be higher than those for the 0/100 and 20/80 mixtures since a greater amount of sulfur was initially out of solution in the mixtures. This was found to be the case and the average initial stiffnesses were as follows:

0/100	190 ksi
20/80	184 ksi
30/70	277 ksi
40/60	388 ksi

Further analysis of the data shows that in general, for all SEA ratios, the Marshall compacted samples experienced greater gains in stiffness than did the samples prepared using kneading compaction (refer to Table 4 and Appendix E). This can in part be attributed to the fact that the Marshall compacted samples had slightly higher air voids than the kneading compacted samples for equal amounts of binder. From the SEM work, it was apparent that sulfur crystals were more abundent in the voids than in the binder itself, thus the more voids, the more crystals and the greater the potential mixture stiffness. Further, air void distribution and size probably influenced the results; however, such measurements were not made during the study.

The mixes produced with AR-4000 asphalt cement resulted in higher resilient moduli than those produced using AR-2000 asphalt, as would be expected. The type of aggregate had a modest effect on the results.

Overall, it is apparent that significant gains in mixture stiffness will occur with time. Heat soaking for time periods as short as 24 hours will assist in obtaining an improved estimate of the near-term mixture stiffness.

MOISTURE CONDITIONING

In general, it was found that mixture moisture susceptibility increases when increasing amounts of sulfur are added to the binder (i.e., increasing SEA ratio). The test results are presented numerically in

Table 5 and graphically in Figures 13 to 17. Moisture susceptibility was determined by use of the conditioning procedure described in Appendix G.

The primary anomoly in the data was the 20/80 SEA mixtures, which had, on the average, the same percent strength retained (as measured by resilient modulus) as the 0/100 SEA mixtures. This phenomenon can be explained based on the concept of solubility of sulfur in asphalt cement. As stated in the section on aging conditioning, the amount of sulfur in the 20/80 mixtures is near the solubility saturation point. Since there is no initial "free" sulfur available to crystallize, and thus increase mixture stiffness, it appears that the 20/80 SEA mixtures perform in a similar way to the 0/100 SEA mixtures. If, however, the 20/80 SEA mixtures are aged so that part of the sulfur in solution has time to crystallize, it would be expected this mix would show greater moisture susceptibility. To illustrate this, for all SEA ratios, the resilient modulus values from the aging samples, after 120 hours of conditioning, were used as the "before" resilient moduli, and along with the actual "after" mixture conditioning resilient moduli values, the percent strength retained was calculated. These results are summarized in Figure 17. It can be seen that the 20/80 mixtures showed slightly greater moisture susceptibility than the 0/100 mixtures using this method (however, the same samples were not used for before and after testing). The apparent relationship for the mixtures examined between percent strength retained and SEA ratio appears to be approximately linear up to a 40/60 SEA ratio with increasing moisture susceptibility for increasing SEA ratio.

After scanning numerous sample mixtures with the SEM, it became apparent that the sulfur crystals formed in the samples prior to moisture conditioning were often destroyed or broken during the conditioning process (refer to Figures C9, C10, C11 and C12 in Appendix C). Since strength (more specifically stiffness) seems to be imparted to the SEA samples through establishment of a network of sulfur crystals in the voids and binder (refer to Figures C35, C36 and C37 in Appendix C), it is likely that this strength will decrease when that network is damaged by the combination of moisture and freeze-thawing. It was also observed that once the sulfur crystals had been damaged or destroyed new crystals could form if crystal growth conditions were favorable (i.e., if sulfur is available and/or voids exist). The phenomenon of crystal growth is exemplified in Figure C38 (Appendix C), and the gain in strength is shown in Figure 18. The results shown in Figure 18 were obtained by taking a set of samples that had been previously subjected to the full moisture conditioning procedure and then about six months later were vacuum saturated and soaked (thus only part of the full conditioning procedure was accomplished, i.e., no freeze-thaw cycling). These results

Table 5. Percent Strength Retained After Moisture Conditioning for SEA Mixtures

Sample Type \ SEA Ratio	Percent Strength Retained				
	0/100	20/80	30/70	40/60	50/50
Marshall Compaction, Crushed Gravel, AR-4000	0.79	0.66	0.89	0.51	0.24
Marshall Compaction, Crushed Gravel, AR-2000	0.61	0.82	0.54	0.37	0.23
Marshall Compaction, Crushed Basalt, AR-4000	0.93	1.97	0.87(1)	0.68(1)	-
Marshall Compaction, Crushed Basalt, AR-2000	0.62	0.66	0.54	0.41	-
Kneading Compaction, Crushed Gravel, AR-4000	0.92	0.99	0.84	0.91	-
Kneading Compaction, Crushed Gravel, AR-2000	1.23(1)	0.80(1)	0.67	0.52	-
Kneading Compaction, Crushed Basalt, AR-4000	0.72	0.94	0.68	0.51	-
Kneading Compaction, Crushed Basalt, AR-2000	0.79	0.92	0.62	0.44	-
Average	0.83	.96(.82)(2)	.70	.49	0.24

(1) Values based on results of second Lottman moisture conditioning cycle.
(2) Excluding the 1.97 value.

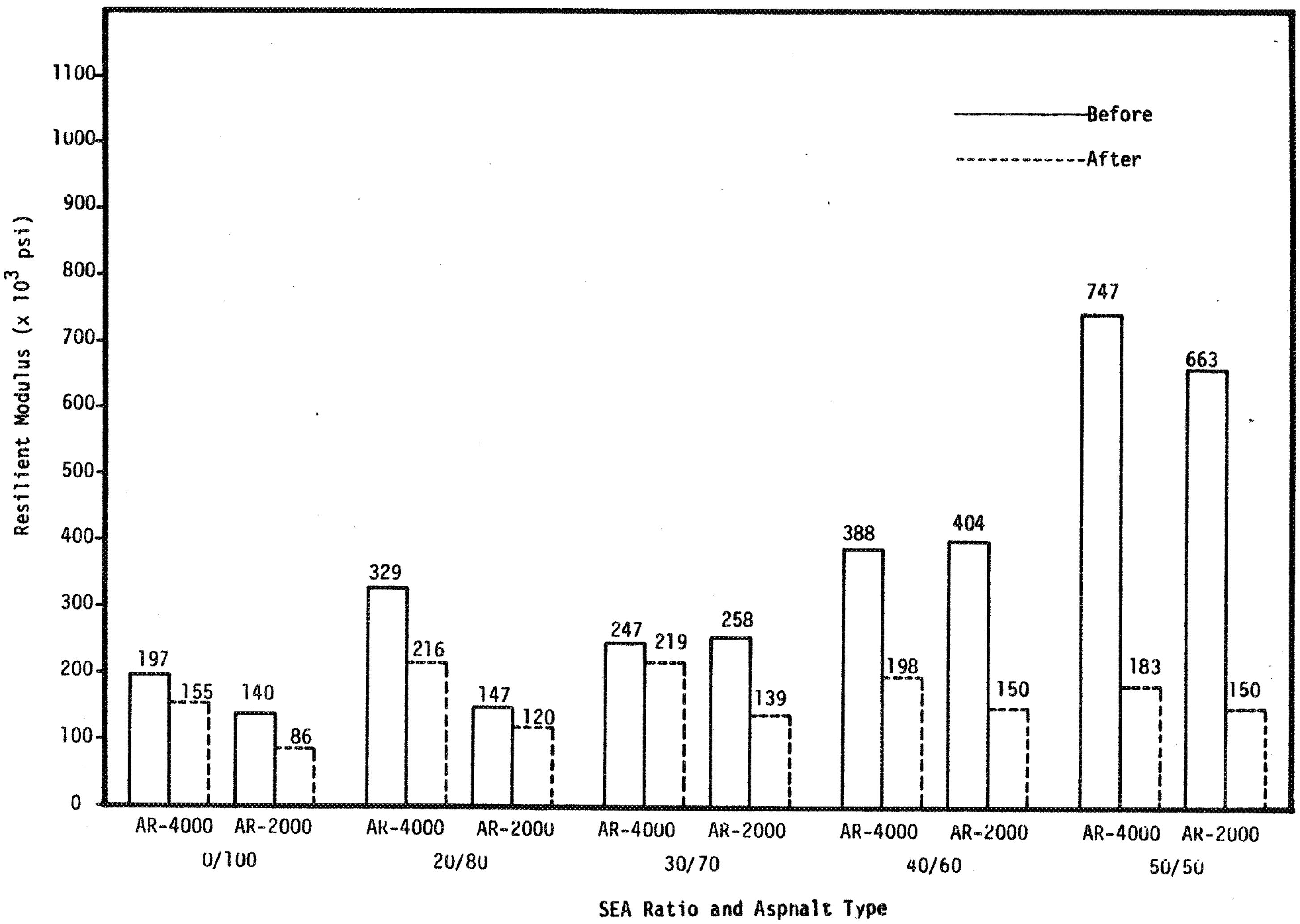
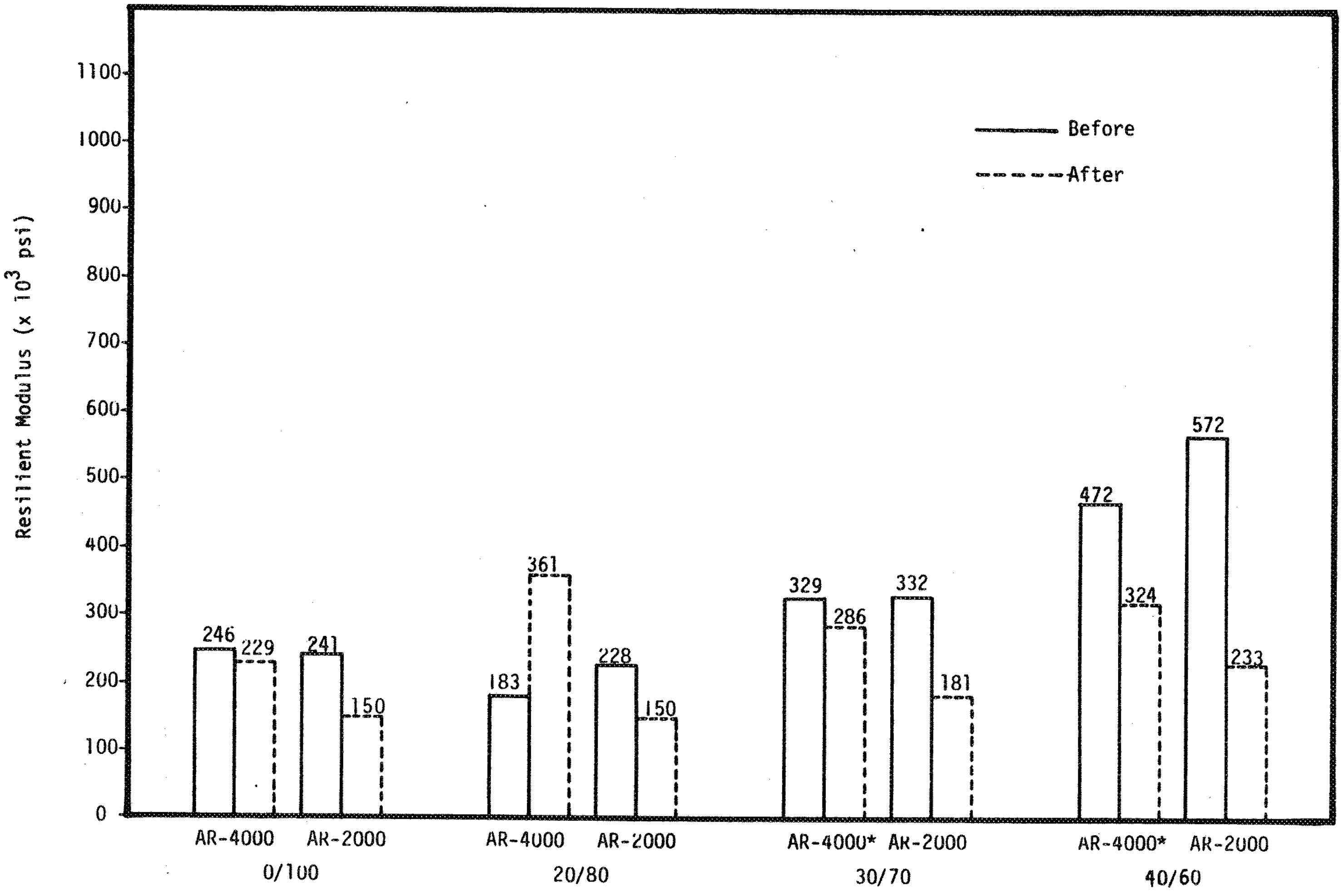
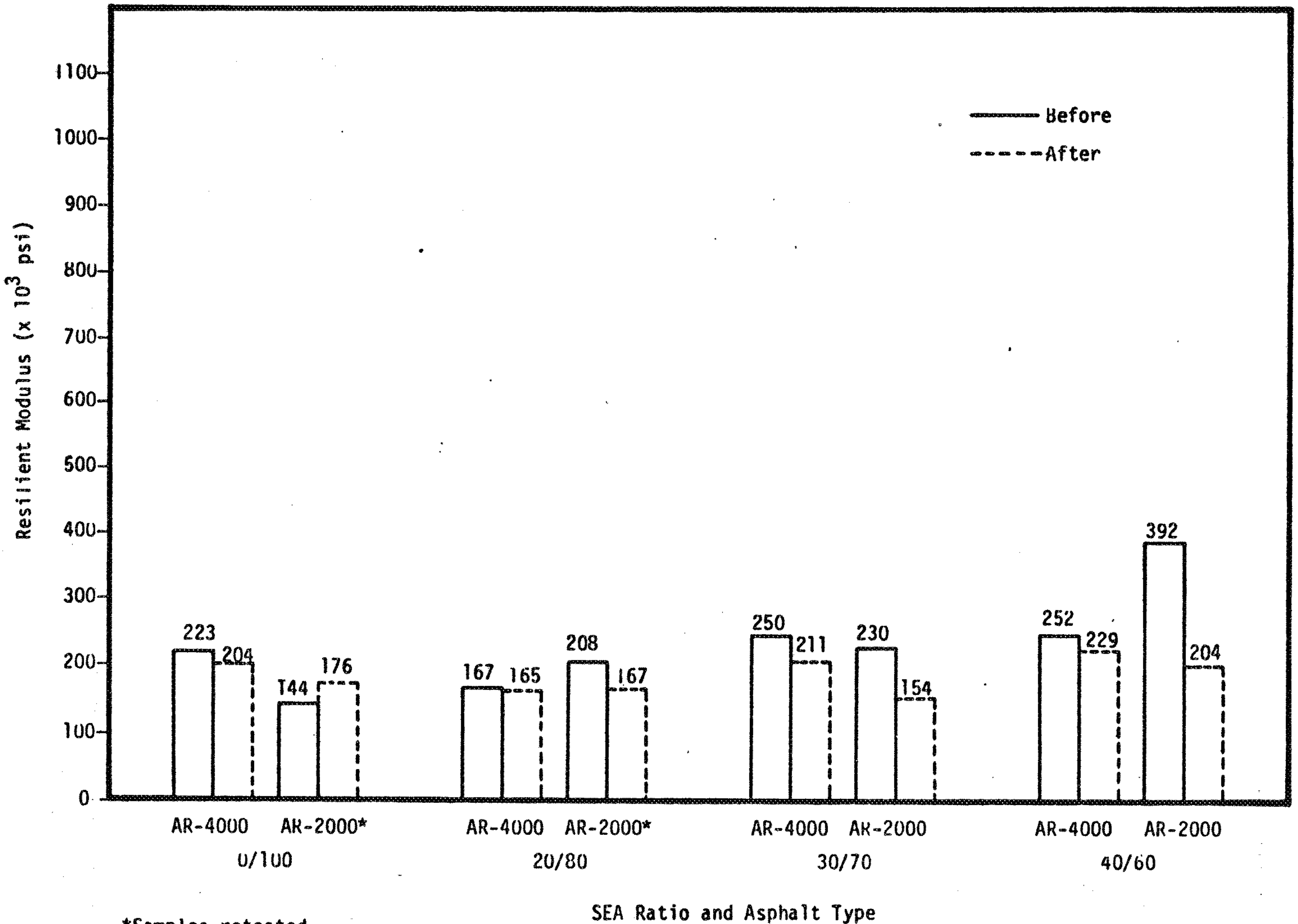


Figure 13. Moisture Susceptibility as Measured by the Change in Resilient Modulus (crushed gravel, Marshall compaction, test temp: 77°F (25°C))



*Samples retested

Figure 14. Moisture Susceptibility as Measured by the Change in Resilient Modulus (crushed basalt, Marshall compaction, test temp: 77°F (25°C))



*Samples retested

Figure 15. Moisture Susceptibility as Measured by the Change, in Resilient Modulus (crushed gravel, kneading compaction test temp: 77°F (25°C))

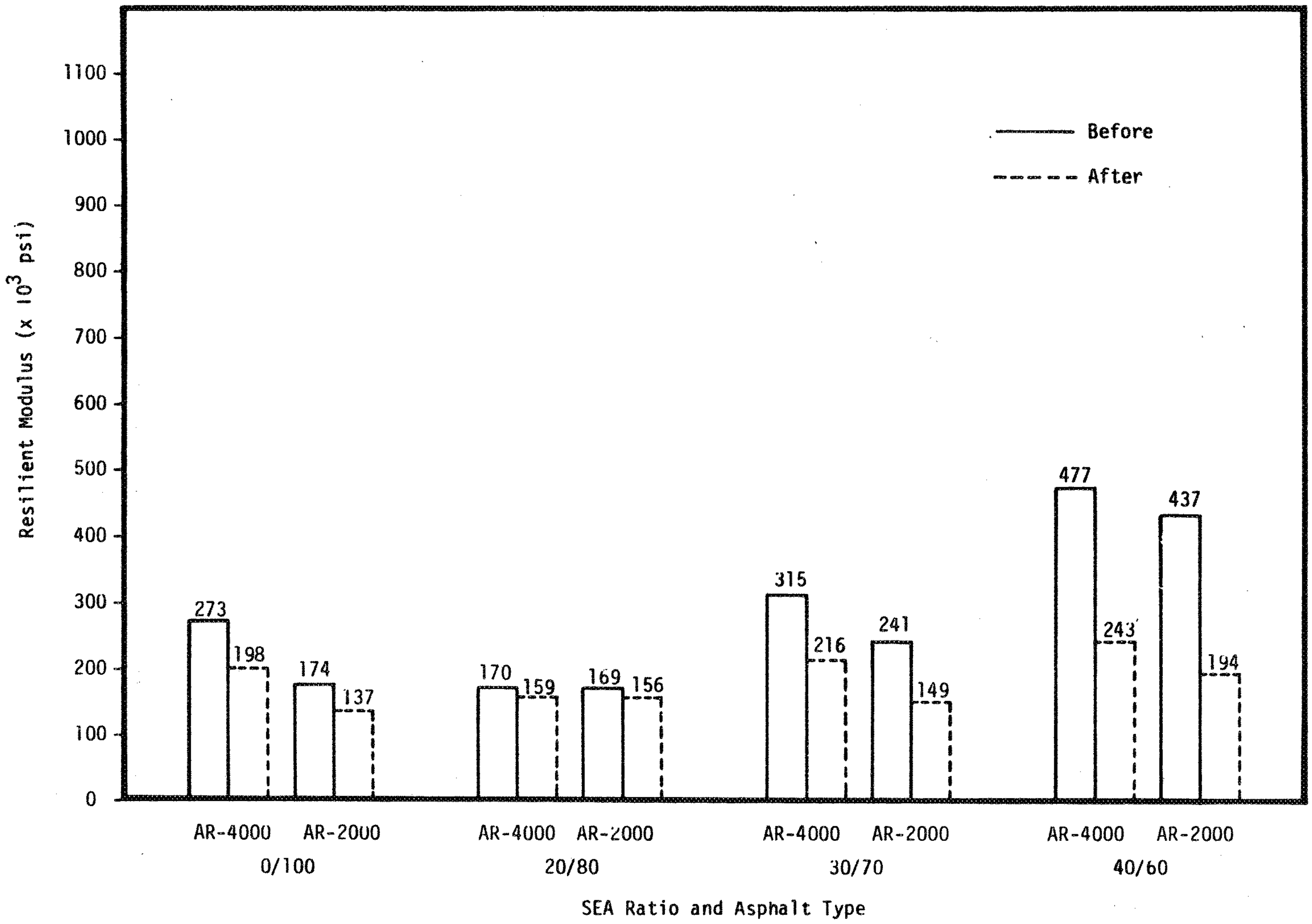


Figure 16. Moisture Susceptibility as Measured by the Change in Resilient Modulus (crushed basalt, kneading compaction, test temp: 77°F (25°C))

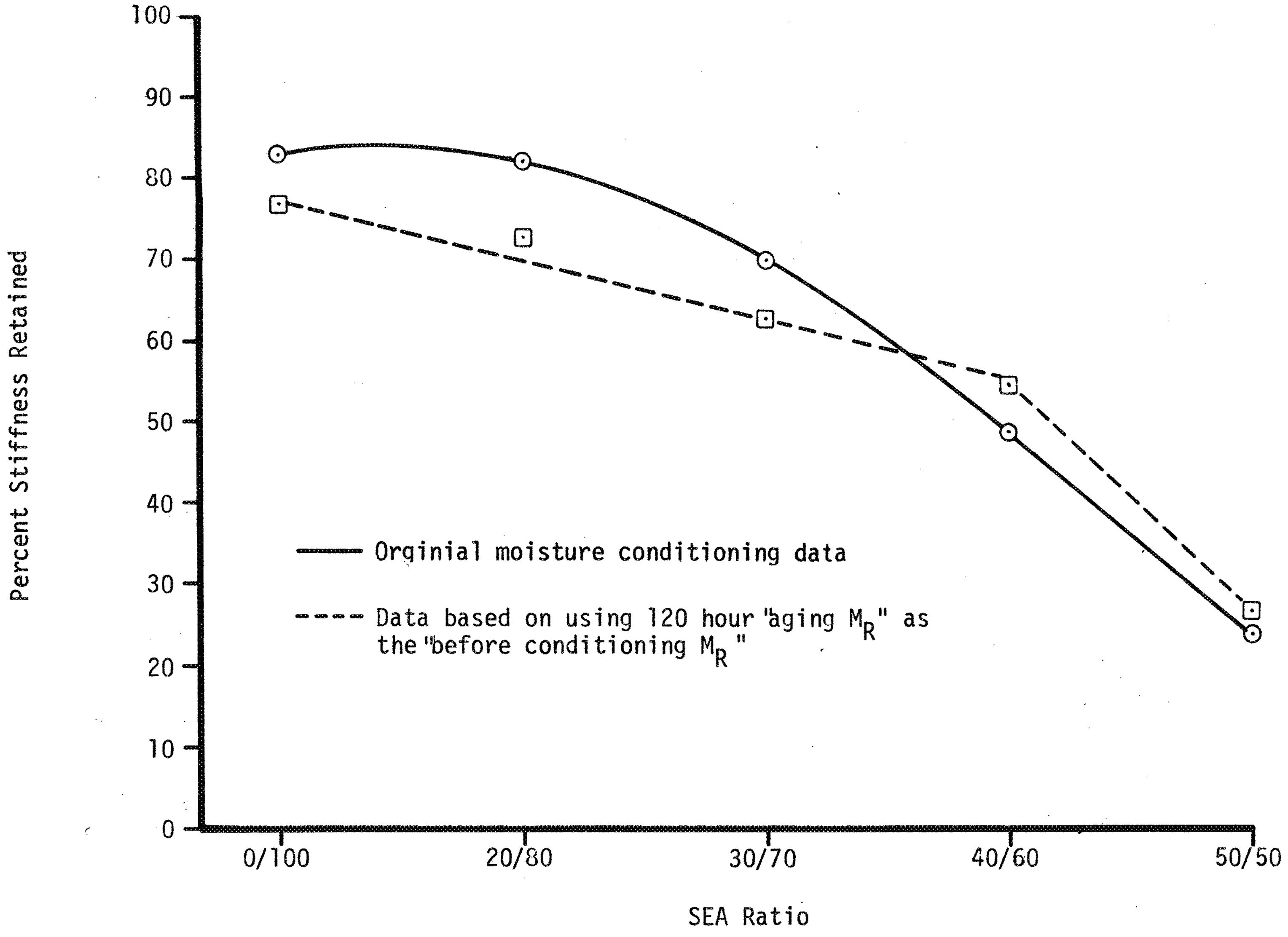


Figure 17. Percent Strength Retained as a Function of SEA Ratio

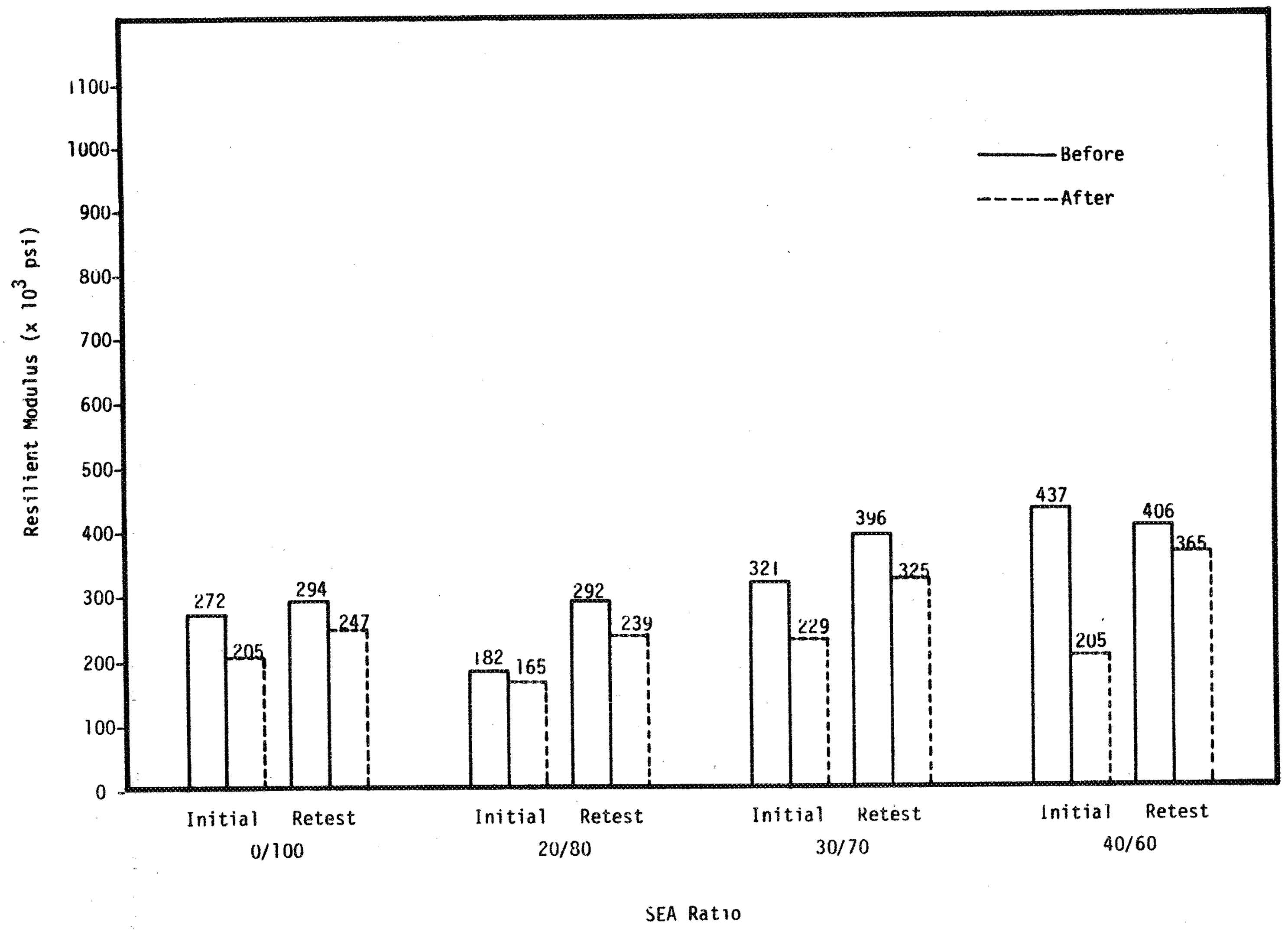


Figure 18. Comparison of Initial and Partial Retest Results of Moisture Conditioning Procedure (AR-4000, crushed basalt, kneading compaction, test temp: 77°F (25°C))

show that a substantial gain in stiffness is possible by reducing the moisture content for all test mixtures. This illustrates the fact that moisture and freeze-thaw cycling can significantly change mixture stiffness but some "recovery" can occur. Further, the relatively small reductions in stiffness of the SEA mixtures due only to vacuum saturation and soaking shows that moisture alone did not necessarily reduce stiffness significantly for the evaluated mixtures but that the combination of moisture and freeze-thaw cycling did.

In most cases, the Marshall compacted samples exhibited a greater stiffness loss than the kneading compacted samples. This is probably due to the higher air voids in the Marshall samples. Sulfur crystals form most readily in void spaces and because moisture susceptibility is at least in part a function of crystal destruction, it is likely that the Marshall compacted samples would be the most susceptible in this study.

It appears that samples made with AR-4000 asphalt cement resulted in a lower strength loss than those made with AR-2000 asphalt cement. Type of aggregate had little effect on the results.

ANTI-STRIP EVALUATION

To determine the effects of adding an anti-strip binder additive on the strength characteristics of samples before and after moisture conditioning, mixtures were prepared using two percentages (by total weight of binder) of Pave Bond Special anti-strip additive.

The two percentages used were one-half and one. SEA ratios of 0/100, 30/70 and 40/60 were used, as were the crushed basalt aggregate and both the Chevron AR-2000 and AR-4000 asphalt cements. The binder contents of each mixture were the optimum contents determined previously in the study. All samples were prepared using the kneading compaction method.

The samples were conditioned using the full moisture conditioning procedure followed throughout the testing program with the results presented in Table 6. Also presented in the same table are the results of tests for samples containing no anti-strip. The results are presented graphically in Figures 19 and 20.

The general trend observed for samples containing the anti-strip additive agreed with those without anti-strip, namely that moisture susceptibility appears to increase with increasing sulfur content in the binder. For both asphalts at the 0/100 SEA ratio the addition of anti-strip had no appreciable effect on the percent of strength (stiffness) retained. The 30/70 SEA ratio results showed that the AR-4000 asphalt cement with the two percentages of anti-strip increased retained strength

Table 6. Percent Retained Strength for SEA Mixtures with and without Anti-strip Additive

Binder Type and Amount of Anti-strip*	0/100 SEA	30/70 SEA	40/60 SEA
	% Strength Retained	% Strength Retained	% Strength Retained
AR 4000-1/2%	61.3	**94.5	58.9
AR 2000-1/2%	62.9	54.6	45.1
AR 4000-1%	75.0	84.5	52.7
AR 2000-1%	77.6	58.2	49.4
AR 4000-0%	72.0	68.0	51.0
AR 2000-0%	79.0	62.0	44.0

*Anti-strip: Pave Bond Special

**Two values > 100%

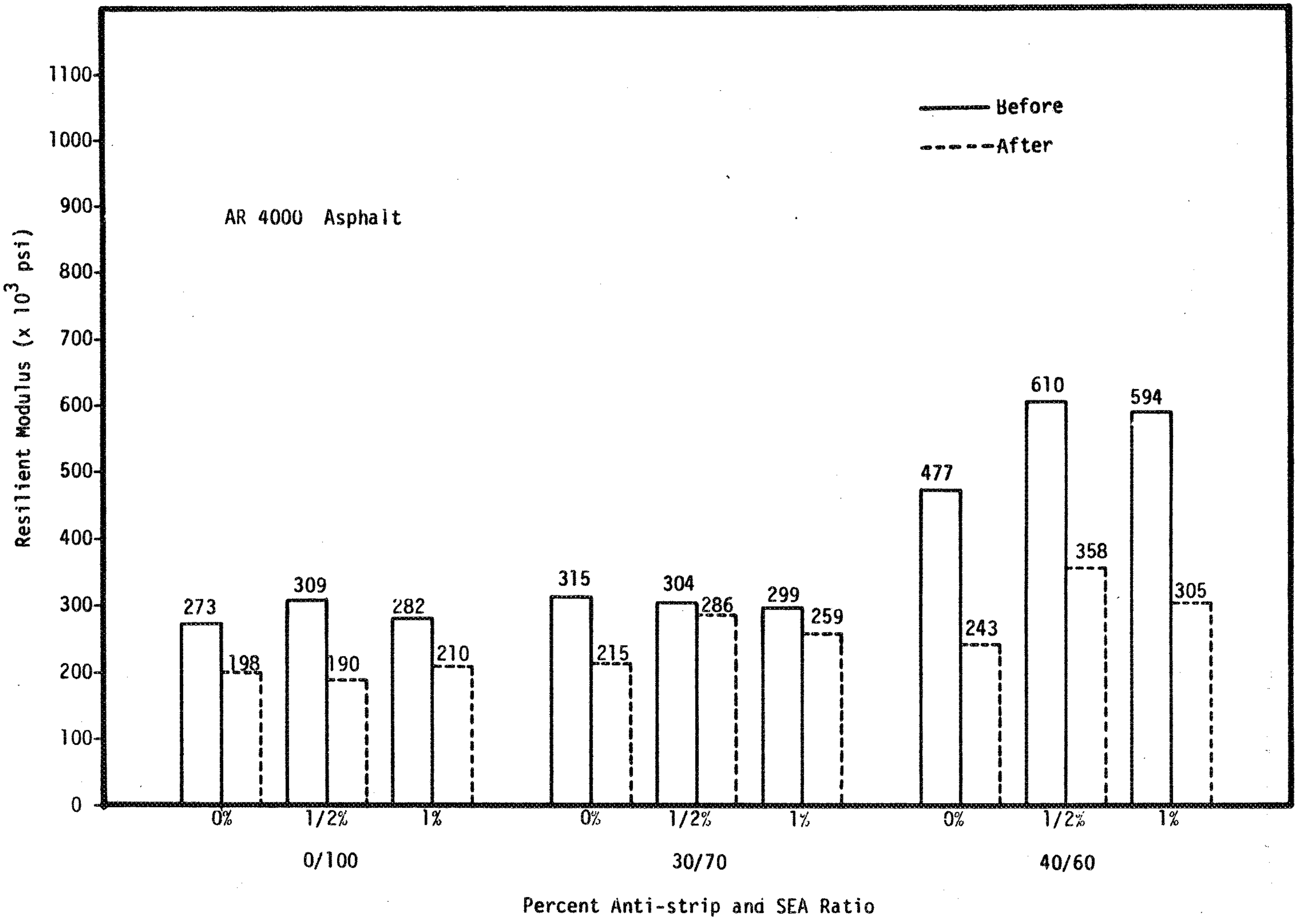


Figure 19. Moisture Susceptibility as Measured by the Change in Resilient Modulus for Different Percents of Anti-strip (AR-4000, crushed basalt, kneading compaction, test temp: 77°F (25°C))

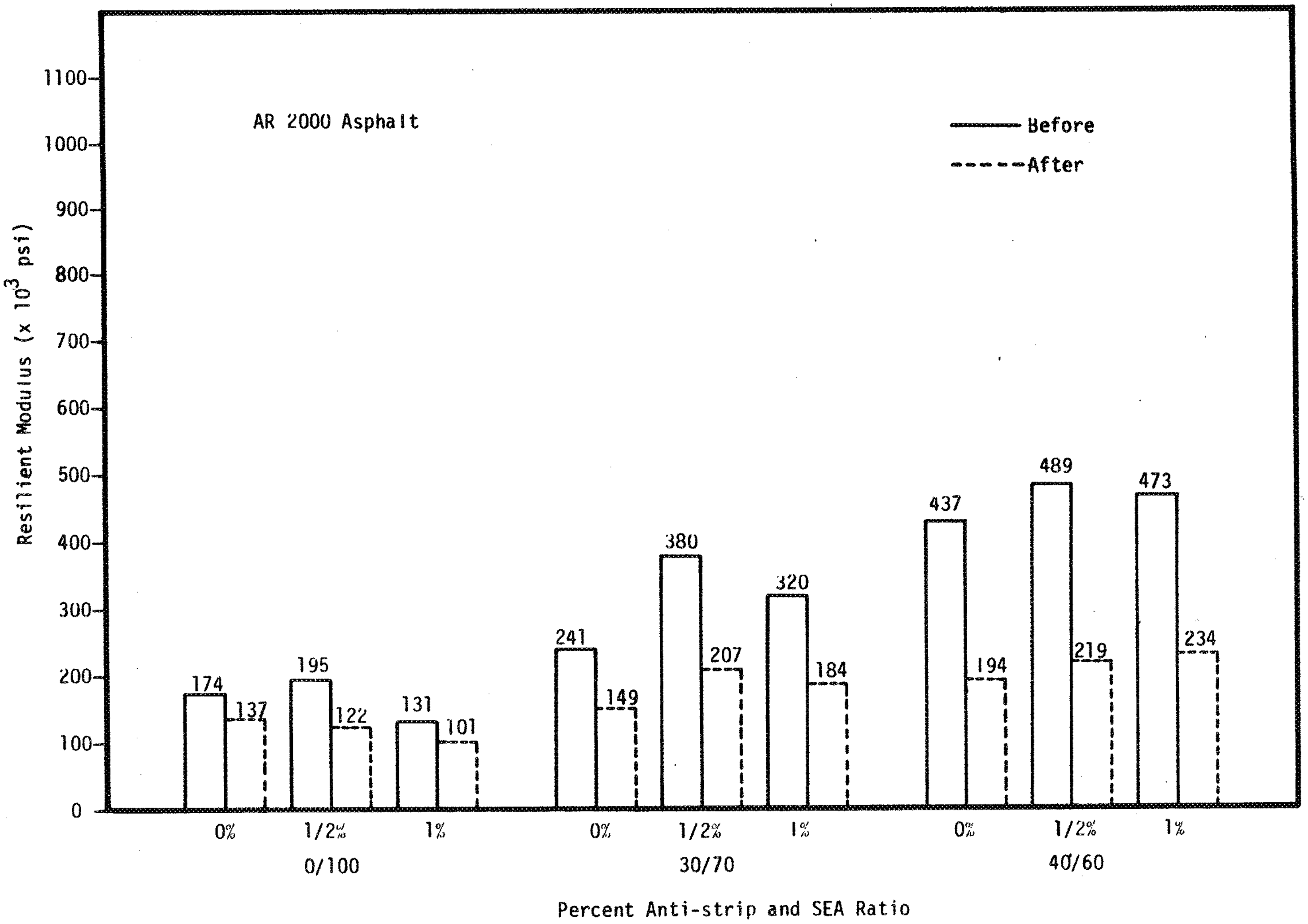


Figure 20. Moisture Susceptibility as Measured by the Change in Resilient Modulus for Different Percents of Anti-strip (AR-2000, crushed basalt, kneading compaction, test temp: 77°F (25°C))

over similar samples with no anti-strip; however, the AR-2000 asphalt cement with anti-strip showed no clear improvement. Both the AR-4000 and AR-2000 samples with anti-strip retained more strength than the samples with no anti-strip for the 40/60 SEA ratio. It should be noted that in none of the cases studied was the difference in strength retained between samples with or without anti-strip considered substantial.

CHAPTER III

CONCLUSIONS AND RECOMMENDATIONS

CONCLUSIONS

The following conclusions are appropriate:

1. The lower limit solubility of sulfur in asphalt cement at ambient conditions appears to range between 4 to 10 percent (p. 9).
2. A significant amount of the added sulfur in SEA mixtures appears to exist as orthorhombic crystals (p. 31 and Appendix C).
3. A minimum SEA binder content equivalent to the volume of an "optimum" amount of asphalt cement appears appropriate (p. 19, Figure 17 (p. 37)).
4. Both the Marshall and Hveem mixture design methods provide essentially the same optimum binder contents (by weight) for the mixture conditions evaluated (Table 3 (p. 18)).
5. The "equal strength" mixture design approach for SEA mixtures does not appear to be viable (p. 19).
6. A minimum time of 14 to 21 days at ambient conditions between mixture compaction and subsequent testing provides for more realistic strength and stiffness test data. Further, subsequent heat soaking (aging conditioning) of these laboratory prepared mixtures at a temperature of 140°F (60°C) for 120 hours provides more consistent estimates of the near-term mixture stiffness (Figure 5-11 (p. 21-28)).
7. Increasing amounts of added sulfur results in increased mixture stiffness (p. 30).
8. Increased amounts of air voids in mixtures appear to increase the observable sulfur crystals (p. 30).
9. The ultimate mixture stiffnesses obtained for the Marshall compacted samples were generally higher and more variable than similar mixture combinations prepared using kneading compaction (p. 30).
10. Increasing amounts of added sulfur generally result in increased mixture stiffness loss following the moisture and freeze-thaw conditioning process developed by Lottman. Significant sulfur crystal damage is possible due to the combined effects of moisture and freeze-thaw conditions. However, the stiffness loss is not necessarily permanent. Some stiffness recovery can occur after drying of the mixture (p. 30, Figure 17 (p. 37)).
11. The anti-strip additive evaluated in the study did not alter the preceding conclusion (p. 39).

RECOMMENDATIONS

1. A minimum SEA binder content equivalent to the volume of an optimum amount of asphalt cement should be used in SEA mixtures.

2. SEA mixtures should have lower than normal air void contents in order to minimize full crystalline sulfur growth.
3. The time dependency of SEA mixtures must be recognized in the mix design process in order to use design criteria properly.
4. Future SEA mixture designs should be evaluated in the laboratory by use of the moisture conditioning procedure as developed by Lottman.
5. Based on the results of this and the Pullman studies, SEA surface course mixtures are not recommended in areas which experience the combined effects of wet freeze-thaw cycles and significant amounts of studded or chained tire wear. However, SEA mixtures should be effective in all climate and traffic areas as a structural base.

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APPENDIX A

DEFINITIONS

DEFINITIONS
(After Ref. A1)

ALLOTROY: The assumption by an element or other substance of two or more different forms or structures which are most frequently stable in different temperature ranges.

AMORPHOUS: Pertaining to a solid which is noncrystalline, having neither definite form nor structure.

CRYSTAL: A homogeneous solid made up of an element, chemical compound or isomorphous mixture throughout which the atoms or molecules are arranged in a regularly repeating pattern.

CRYSTALLINE: Of, pertaining to, resembling, or composed of crystals.

DEHYDROGENATION: Removal of hydrogen from a compound.

MONOCLINIC SYSTEM: One of the six crystal systems characterized by a single, two-fold symmetry axis or a single symmetry plane.

ORTHORHOMBIC LATTICE: A crystal lattice in which the three axes of a unit cell are mutually perpendicular and no two have the same length.

POLYMER: Substance made of large molecules formed by the union of simple molecules (monomers).

POLYMORPH: A crystal form of a polymorphic material.

POLYMORPHISM: The property of a chemical substance crystallizing into two or more forms having different structures, such as diamond and graphite.

SULFUR (Chem): A nonmetallic element, symbol S, atomic number 16, atomic weight 32.064, existing in crystalline or amorphous form.

SULFUR (Mineral): A yellow orthorhombic mineral occurring in crystals, masses, or layers, and existing in several allotropic forms.

VULCANIZATION: A chemical reaction of sulfur with rubber or plastic to cause cross-linking of the polymer chains; it increases strength and resiliency of the polymer.

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APPENDIX B

BRIEF DESCRIPTION OF CRYSTAL SYSTEMS

BRIEF DESCRIPTION OF CRYSTAL SYSTEMS

INTRODUCTION

To enable a better understanding of how sulfur exists in paving mixtures, a brief description of crystal systems is warranted. This is due to the fact that a large amount of the added sulfur in such mixes exists as orthorhombic sulfur.

The three basic states for all matter are as a gas, liquid or solid (B1). In the gaseous state, molecules or atoms are in constant and random motion. A gas, of course, assumes the shape of its container. In the liquid state, the random molecular motion is much more restricted and a liquid has a higher viscosity than a gas. In the solid state, molecular motion is confined and solids may be crystalline or amorphous.

A crystalline solid has a regular or repeated arrangement of molecules or atoms in a fixed or rigid pattern (lattice). A crystalline substance usually exhibits an anisotropic state whereby mechanical, electrical, magnetic and optical properties can vary according to the direction in which they are measured. An amorphous solid has a random arrangement of molecules or atoms and normally exhibits isotropic properties (properties the same in all directions of measurement).

CRYSTAL SYMMETRY

Geometric shapes can be used to help classify crystals. The three elements of symmetry are:

1. Symmetry about a point
2. Symmetry about a line (or axis)
3. Symmetry about a plane

If a crystal is rotated through 360° about an axis, it is returned to its original position. If the crystal appears to have returned to its original position more than once during a complete rotation, the chosen axis is an "axis of symmetry". If the crystal is rotated 180° before coming into coincidence with its original position, the axis is one of "two-fold symmetry". Three-fold, four-fold, etc., symmetries are possible for some types of crystals.

CRYSTAL SYSTEMS

There are 32 possible combinations of the three previously described elements of symmetry. These 32 combinations have been grouped into seven systems as follows (B1).

1. Regular
2. Tetragonal
3. Orthorhombic
4. Monclinic
5. Triclinic
6. Trigonal
7. Hexagonal

The first six of the above systems can be described with reference to three axes denoted x, y and z. The z-axis is vertical, x-axis from front to back and the y-axis from right to left (Figure B1). The angle between the y and z-axes is denoted as "α", between the x and z-axes by "β" and between the x and y-axes by "γ". Descriptions of the seven crystal systems are contained in Table B1.

REFERENCE B

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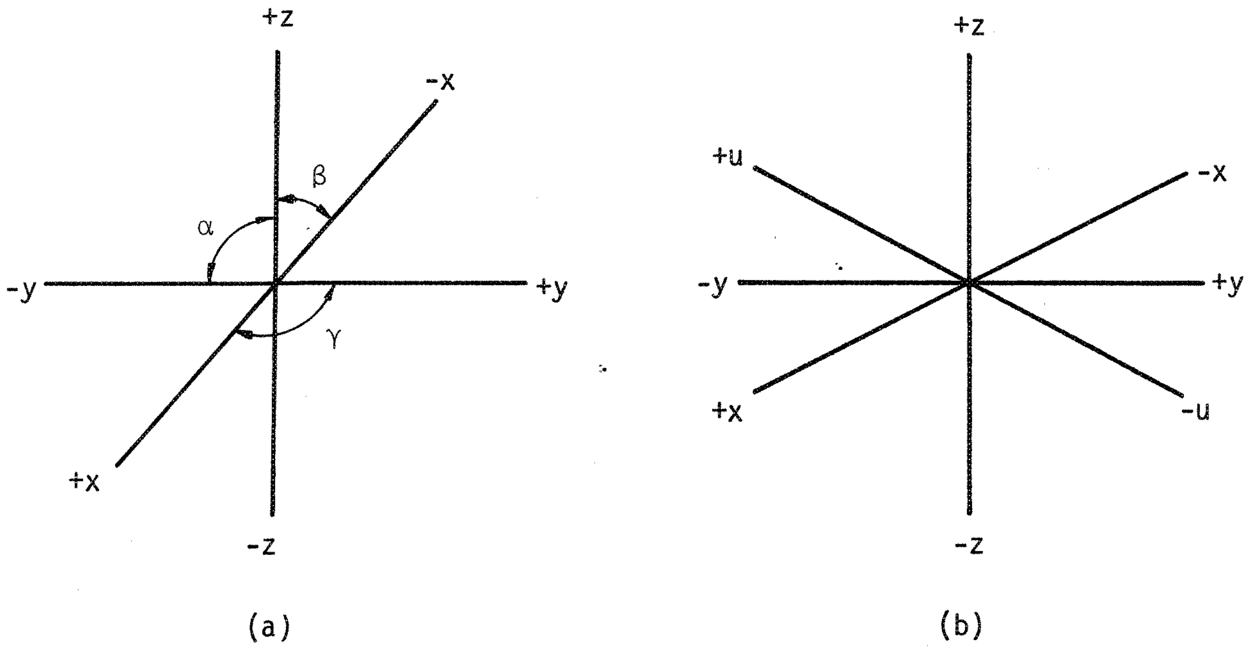

Figure B1. Axes for Describing the Seven Crystal Systems (After Ref. B1)

Table B1. Descriptions of the Seven Crystal Systems (After Ref. B1)

System	Other Names	Angles Between Axles	Relative Length of Axles	Examples
1. Regular	Cubic Octahedral Isometric	$\alpha = \beta = \gamma = 90°$	$x = y = z$	Sodium Chloride Potassium Chloride Diamond
2. Tetragonal	Pyramidal Quadratic	$\alpha = \beta = \gamma = 90°$	$x = y \neq z$	Zircon Nickel Sulphate
3. Orthorbombic	Rhombic Prismatic Isoclinic	$\alpha = \beta = \gamma = 90°$	$x \neq y \neq z$	Silver Nitrate Iodine α-Sulfur (S_α)
4. Monoclinic	Oblique Monosymmetric Clinorhombic	$\alpha = \beta = 90° \neq \gamma$	$x \neq y \neq z$	Potassium Chlorate Sucrose β-Sulfur (S_β)
5. Triclinic	Asymmetric	$\alpha \neq \beta \neq \gamma \neq 90°$	$x \neq y \neq z$	Copper Sulphate
6. Trigonal	Rhombohedral	$\alpha = \beta = \gamma \neq 90°$	$x = y = z$	Sodium Nitrate Ruby Sapphire
7. Hexagonal	None	z axis is perpendicular to the x, y and u axes which are inclined at 60° to each other.	$x = y = u \neq z$	Graphite Water (ice)

APPENDIX C

SCANNING ELECTRON MICROSCOPE PHOTOGRAPHS OF SEA MIXTURES

SCANNING ELECTRON MICROSCOPE (SEM) PHOTOGRAPHS OF SEA MIXTURES

The photographs contained in this appendix were obtained with a Cambridge scanning electron microscope (model MKIIA). Associated with this instrument is an EDAX X-ray analysis system. This attachment provided for sample scans which were used to determine qualitatively the location of sulfur.

Most of the mixture combinations examined in this study were sampled for SEM study. The following index is provided for the figures contained in this appendix:

Figures	SEA Ratio	Asphalt Cement Type	Aggregate Type	Compaction Method
C1-C8	30/70	AR-2000	Crushed Gravel	Kneading
C9-C19	30/70	AR-4000	Crushed Gravel	Kneading
C20-C27	20/80	AR-4000	Crushed Basalt	Marshall
C28-C34	30/70	AR-2000	Crushed Basalt	Marshall
C35-C40	30/70	AR-4000	Crushed Basalt	Marshall
C41-C46	40/60	AR-4000	Crushed Basalt	Marshall
C47-C54	30/70	AR-2000	Crushed Basalt	Kneading
C55-C58	30/70	AR-4000	Crushed Basalt	Kneading

The primary emphasis was placed on an examination of the 30/70 SEA mixtures. This was done because this mixture was felt to represent a "near optimum" SEA mixture condition.

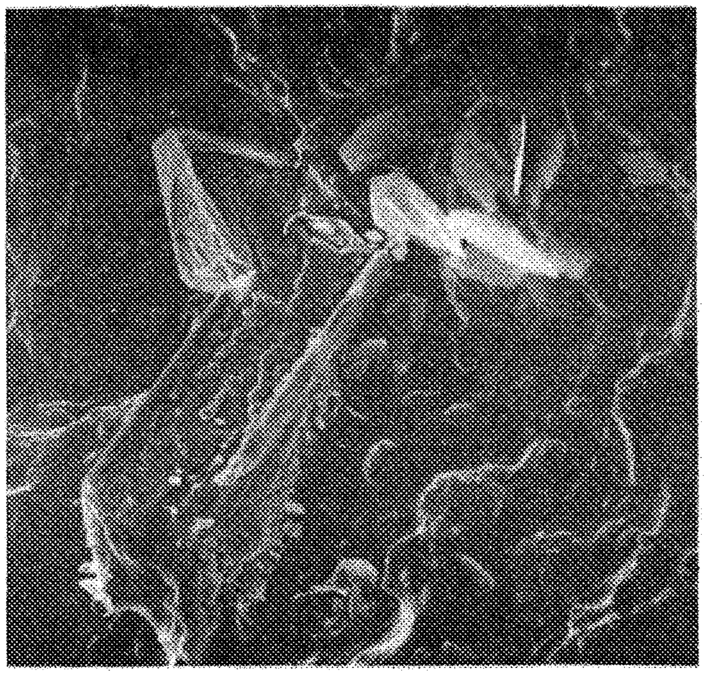

Figure C1. SEA 30/70 Mixture (AR-2000, crushed gravel, kneading compaction) After Heat Soaking (320x)

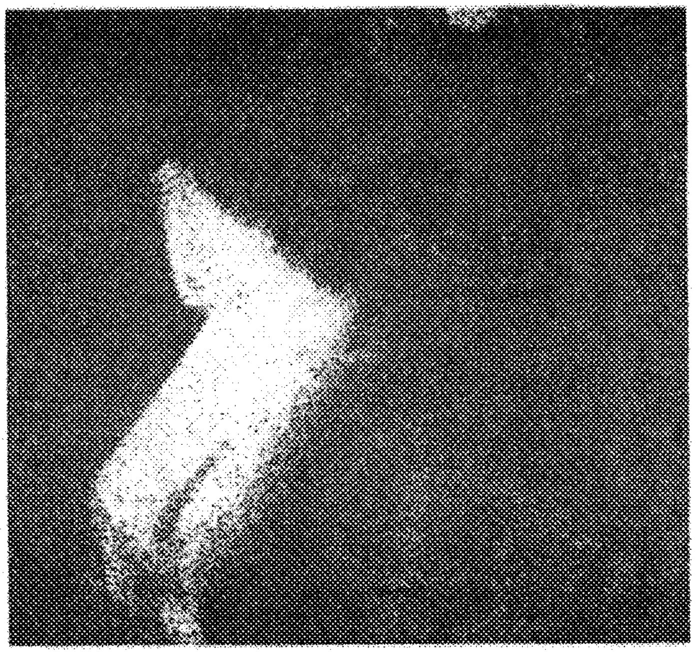

Figure C2. X-Ray Scan for Sulfur (Sample, location and magnification same as in Figure C1)

Figure C3. SEA 30/70 Mixture (AR-2000, crushed gravel, kneading compaction) After Heat Soaking (518x)

Figure C4. SEA 30/70 Mixture (AR-2000, crushed gravel, kneading compaction) After Heat Soaking (522x)

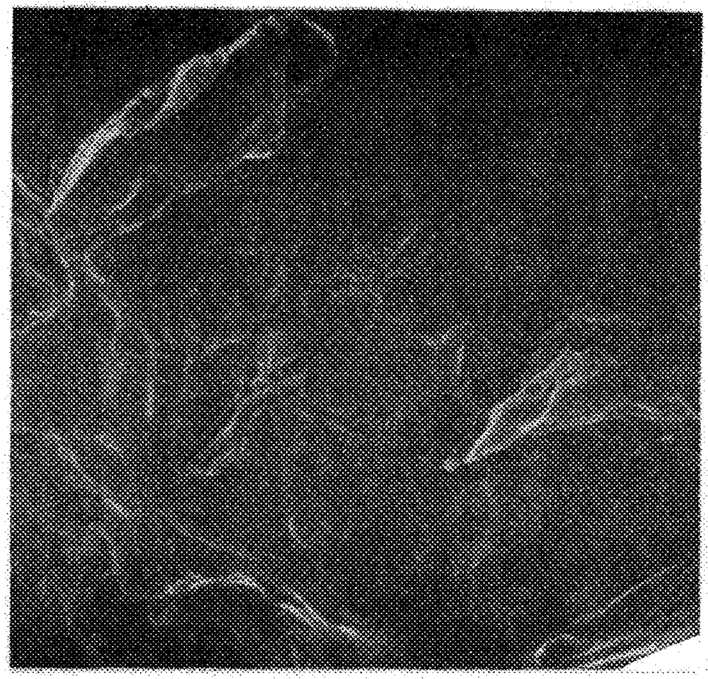

Figure C5. SEA 30/70 Mixture (AR-2000, crushed gravel, kneading compaction) After Lottman Conditioning (473x)

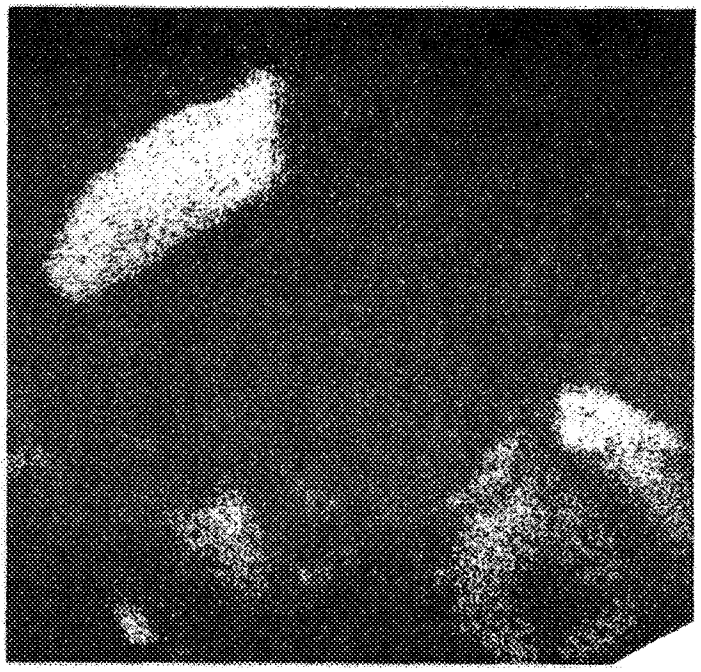

Figure C6. X-Ray Scan for Sulfur (Sample, location and magnification same as Figure C5)

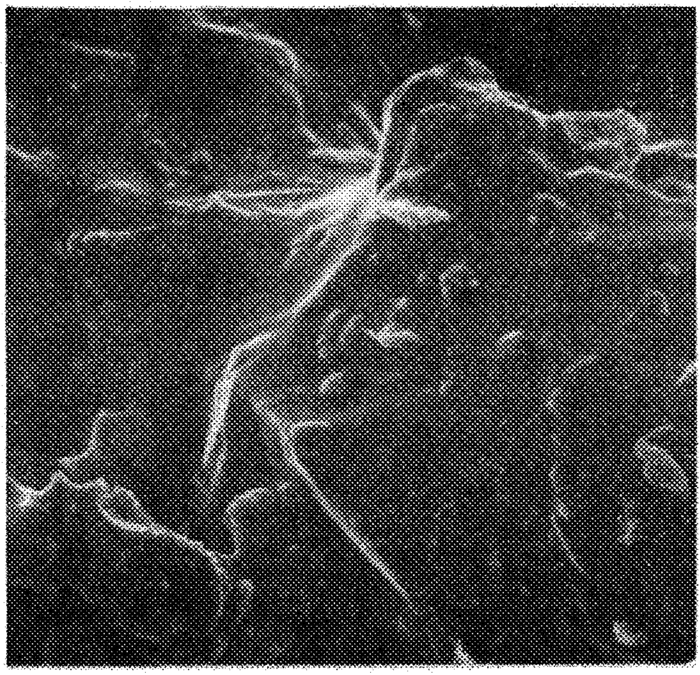

Figure C7. SEA 30/70 Mixture (AR-2000, crushed gravel, kneading compaction) After Lottman Conditioning (532x)

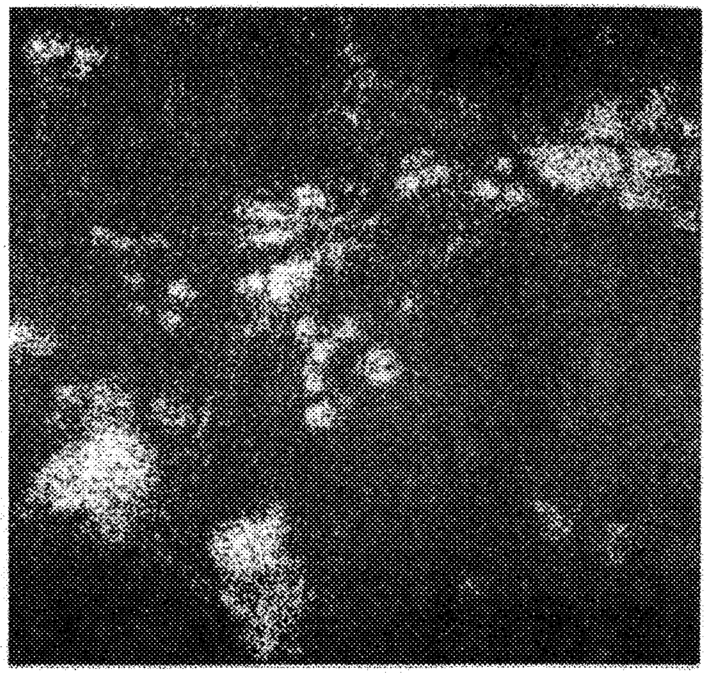

Figure C8. X-Ray Scan for Sulfur (Sample, location and magnification same as in Figure C7)

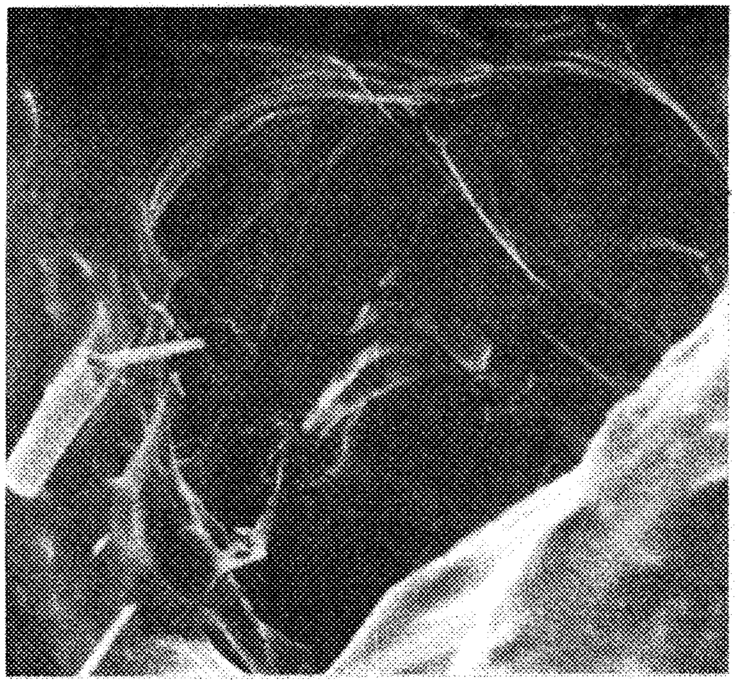

Figure C9. SEA 30/70 Mixture (AR-4000, crushed gravel, kneading compaction) After Heat Soaking (410x)

Figure C10. Same Sample and Location as Shown in Figure C9 After Five Freeze-Thaw Cycles (Temperature range: -5°C to 25°C)

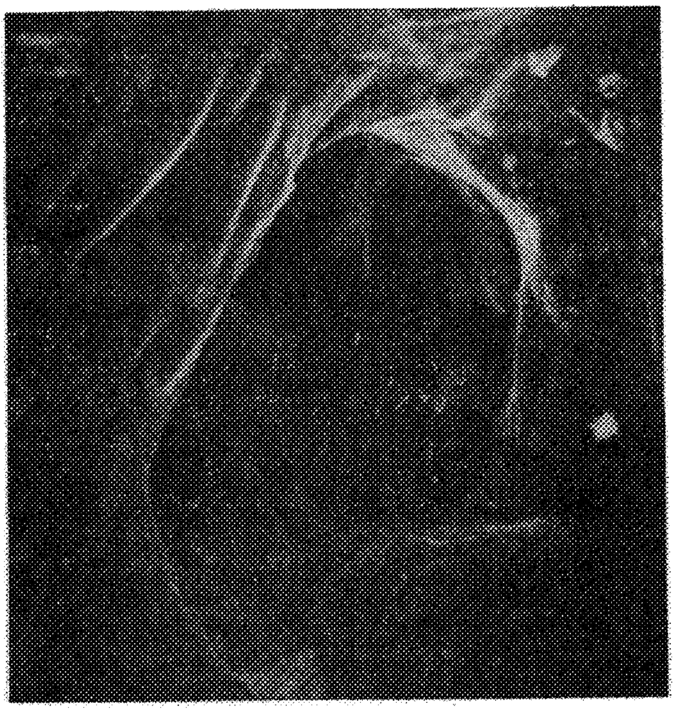

Figure C11. Same Sample and Location as Shown in Figure C9 After Five Wet Freeze-Thaw Cycles (Temperature range: -5°C to 25°C)(400x). Note: void distorted due to cycling and angle of view which is different than that shown in Figures C9 and C10

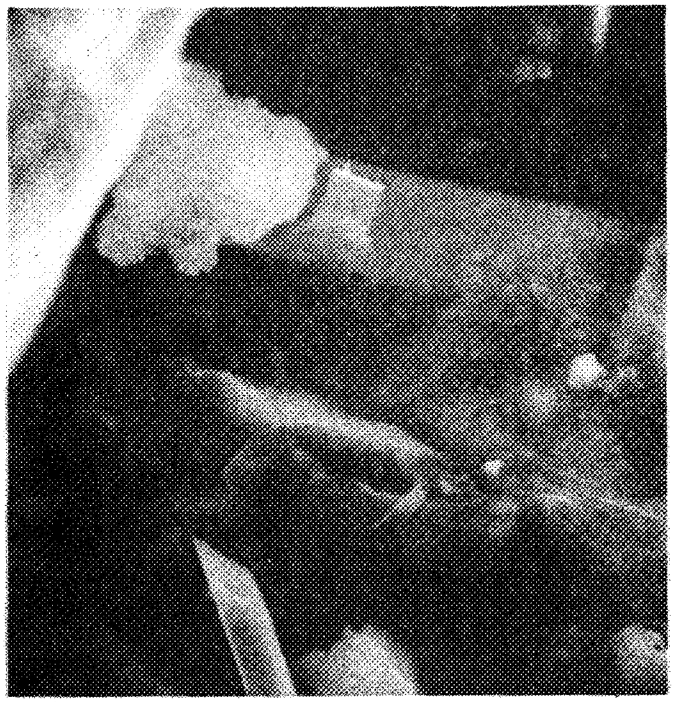

Figure C12. Close-up of Void Originally Shown in Figure C11 (1170x)

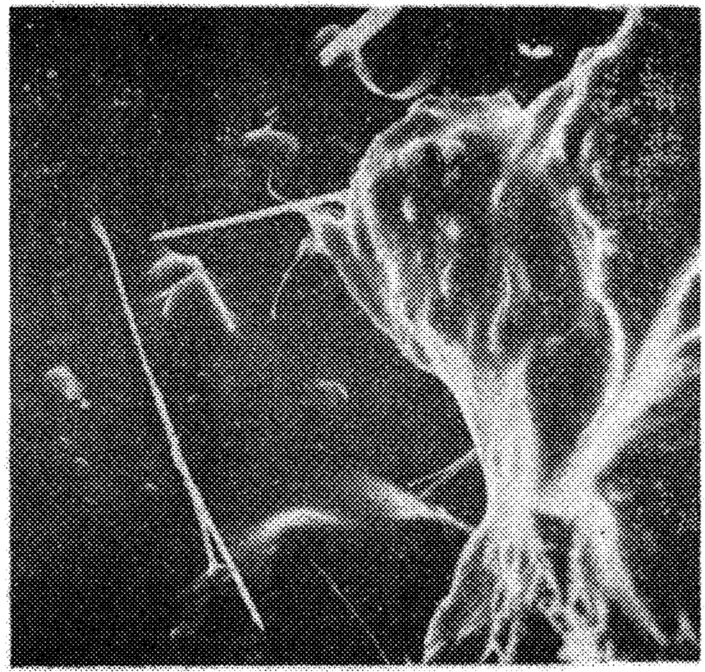

Figure C13. SEA 30/70 Mixture (AR-4000, crushed gravel, kneading compaction) After Heat Soaking (530x)

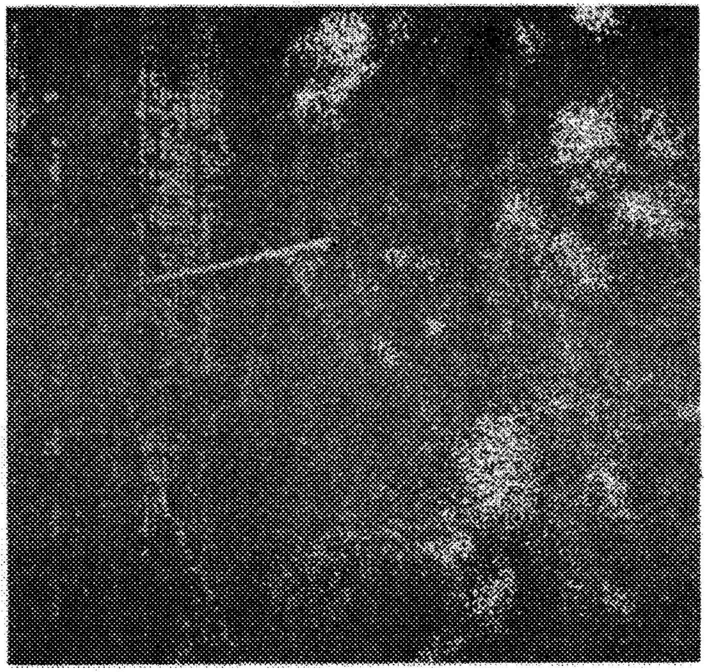

Figure C14. X-Ray Scan for Sulfur (Sample, location and magnification same as Figure C13)

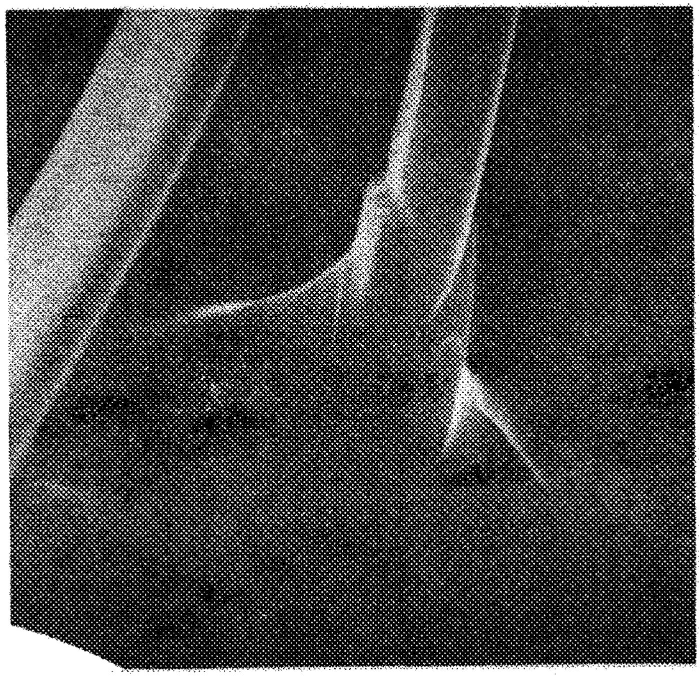

Figure C15. Close-up of Bottom of Sulfur Originally Shown in Figure C13 (10,600x)

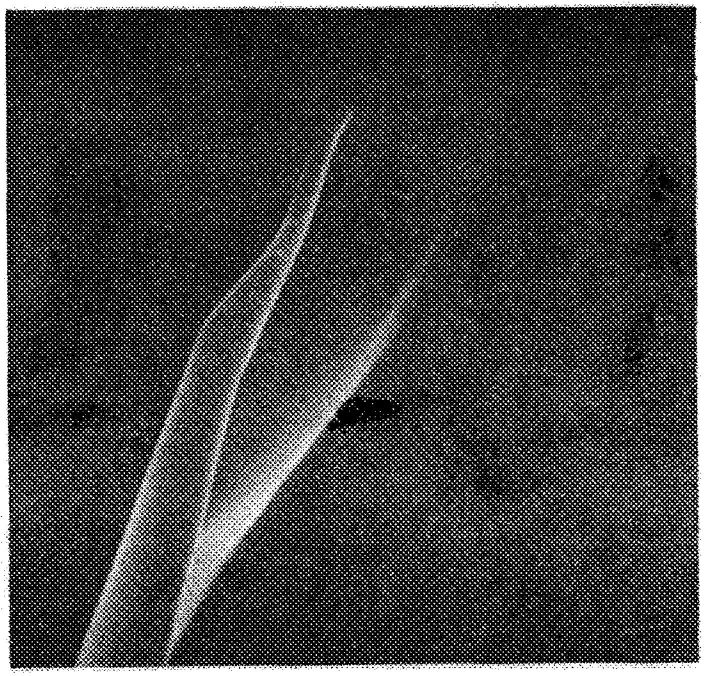

Figure C16. Close-up of Top Sulfur Originally Shown in Figure C13 (10,600x)

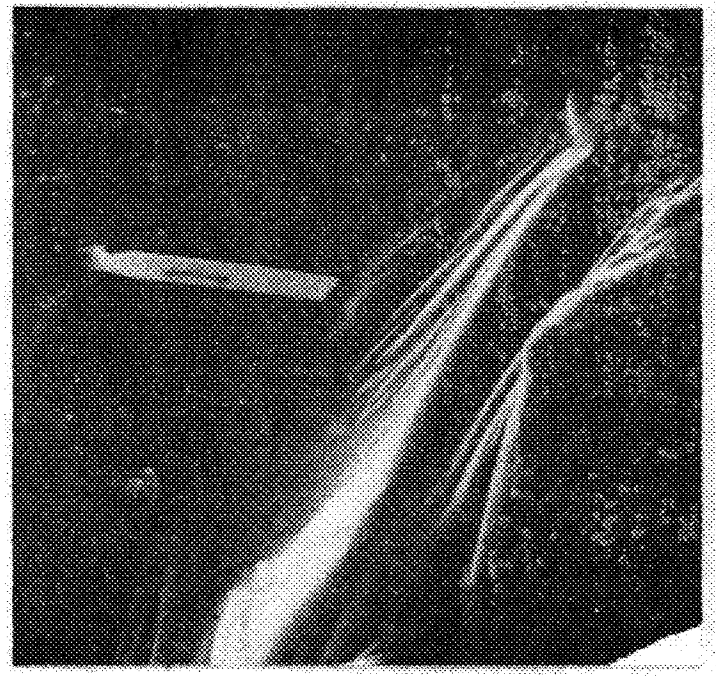

Figure C17. SEA 30/70 Mixture (AR-4000, crushed gravel, kneading compaction) After Lottman Conditioning (555X)

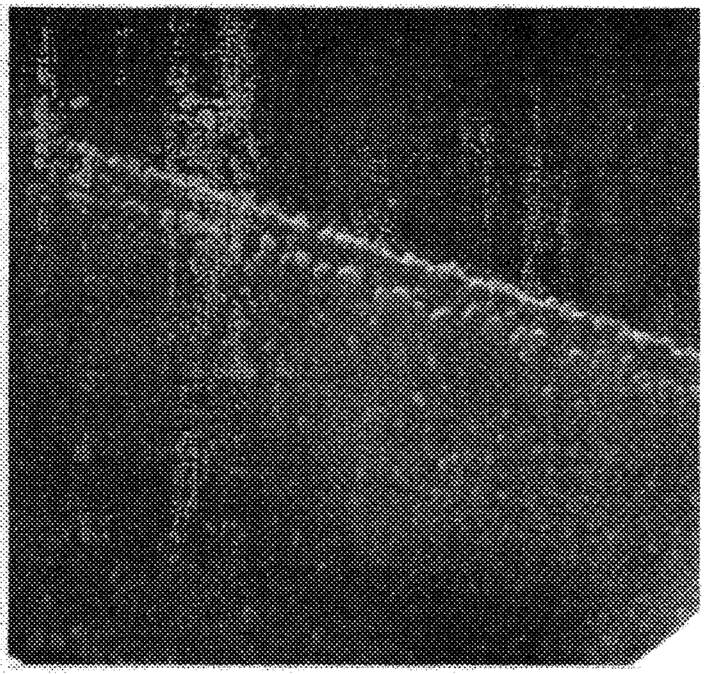

Figure C 18. Close-up of Darkened Area on Sulfur Originally Shown in Figure C17 (11,400x)

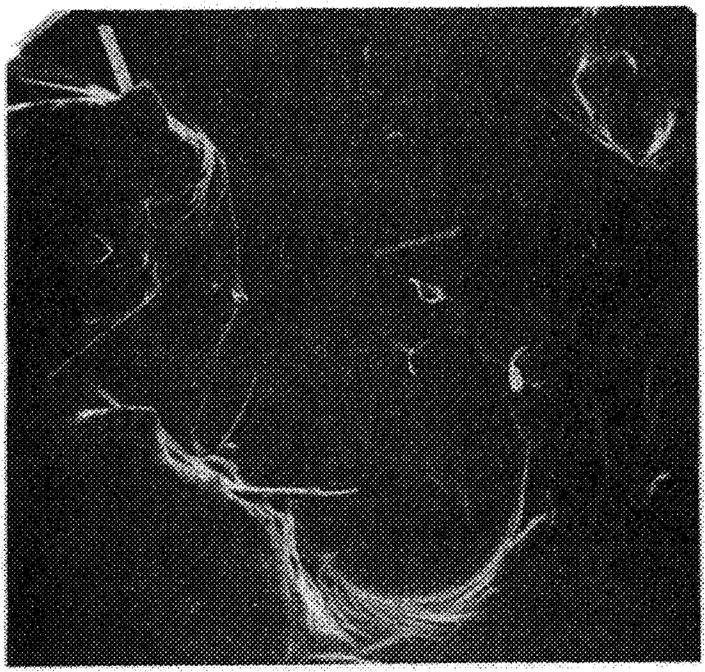

Figure C19. SEA 30/70 Mixture (AR-4000, crushed gravel, kneading compaction) After Lottman Conditioning (217x)

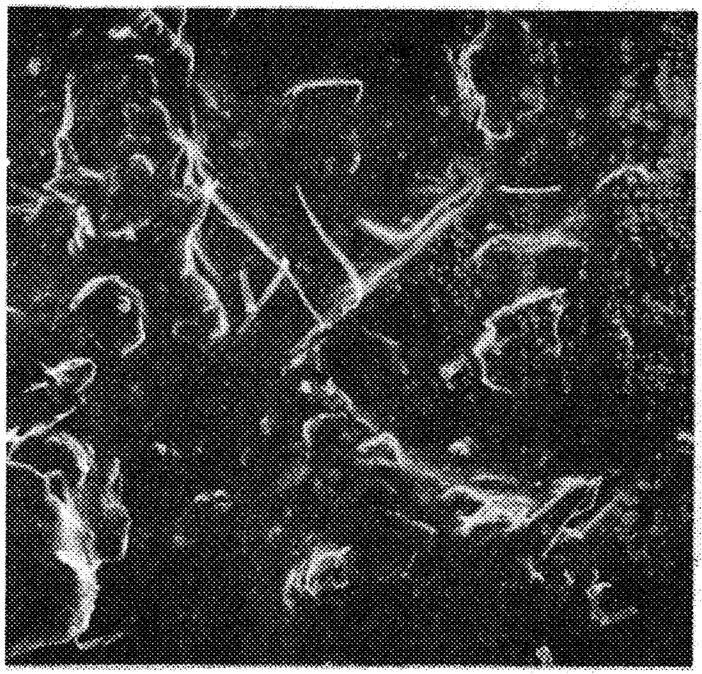

Figure C20. SEA 20/80 Mixture (AR-4000, crushed basalt, Marshall compaction) After Heat Soaking (224x)

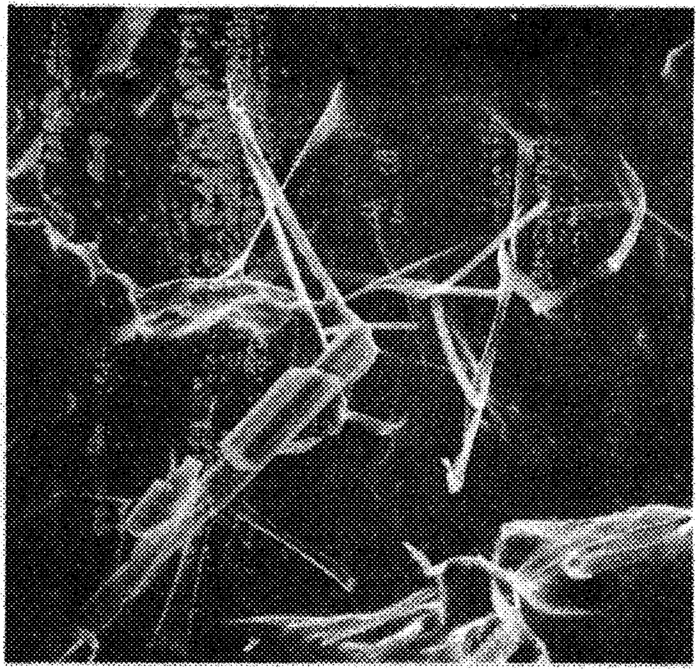

Figure C21. SEA 20/80 Mixture (AR-4000, crushed basalt, Marshall compaction) After Heat Soaking (530x)

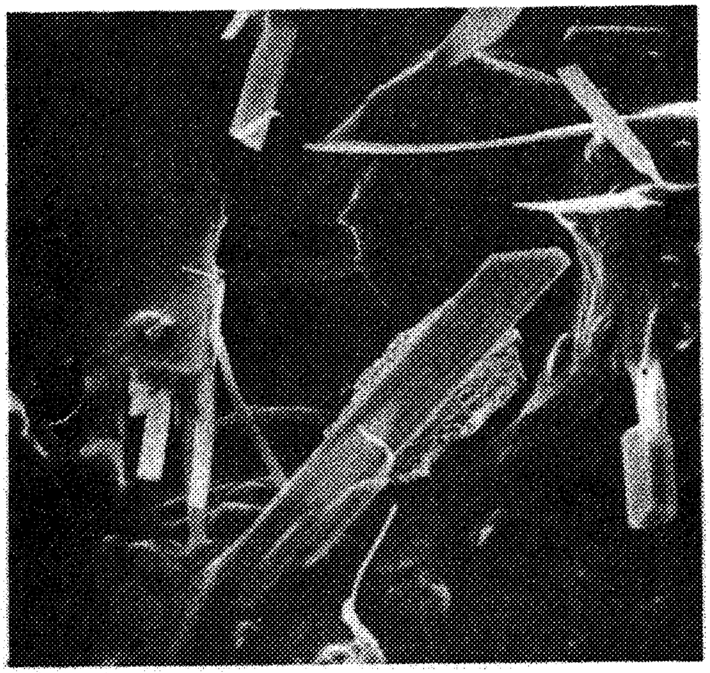

Figure C22. SEA 20/80 Mixture (AR-4000, crushed basalt, Marshall compaction) After Heat Soaking (515x)

Figure C23. SEA 20/80 Mixture (AR-4000, crushed basalt, Marshall compaction) After Heat Soaking (211x)

Figure C24. X-Ray Scan for Sulfur (Sample, location and magnification same as Figure C23)

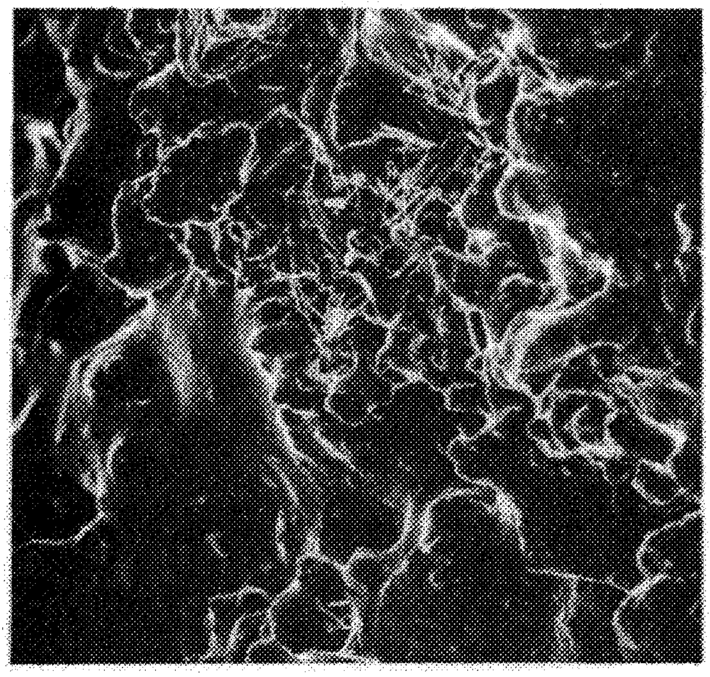

Figure C25. SEA 20/80 Mixture (AR-4000, crushed basalt, Marshall compaction) After Lottman conditioning (110x). Note: essentially no crystalline sulfur was observed in the voids for most of this sample

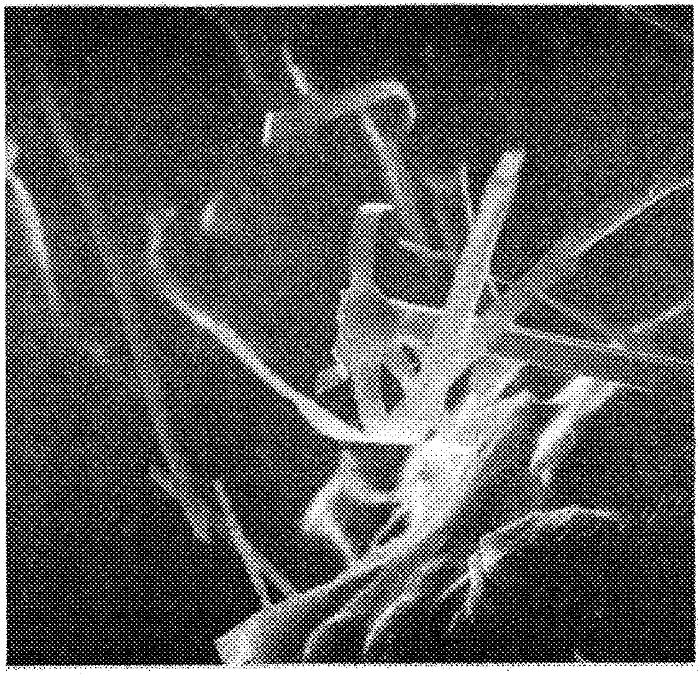

Figure C26. Close-up of Sulfur Originally Shown in Figure C25 (1100x)

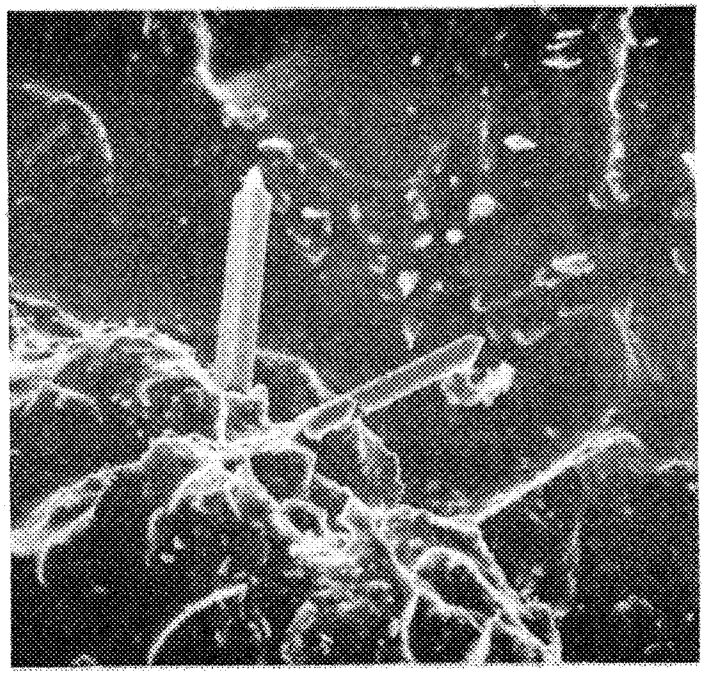

Figure C27. SEA 20/80 Mixture (AR-4000, crushed basalt, Marshall compaction) After Lottman Conditioning (310x)

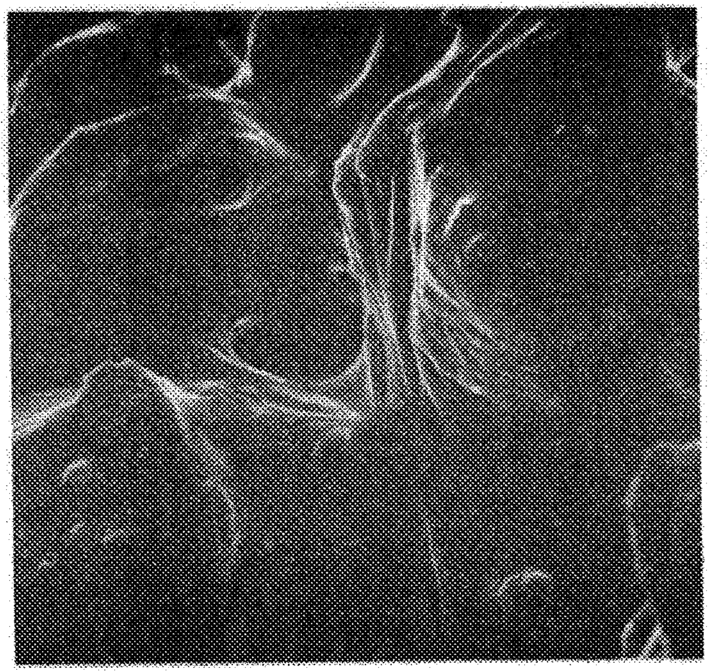

Figure C28. SEA 30/70 Mixture (AR-2000, crushed basalt, Marshall compaction) After Heat Soaking (585x)

Figure C29. X-Ray Scan for Sulfur (Sample, location and magnification same as Figure C28)

Figure C30. SEA 30/70 Mixture (AR-2000, crushed basalt, Marshall compaction) After Heat Soaking (192x). Note: oval shaped rupture in binder film contains sulfur as determined with EDAX.

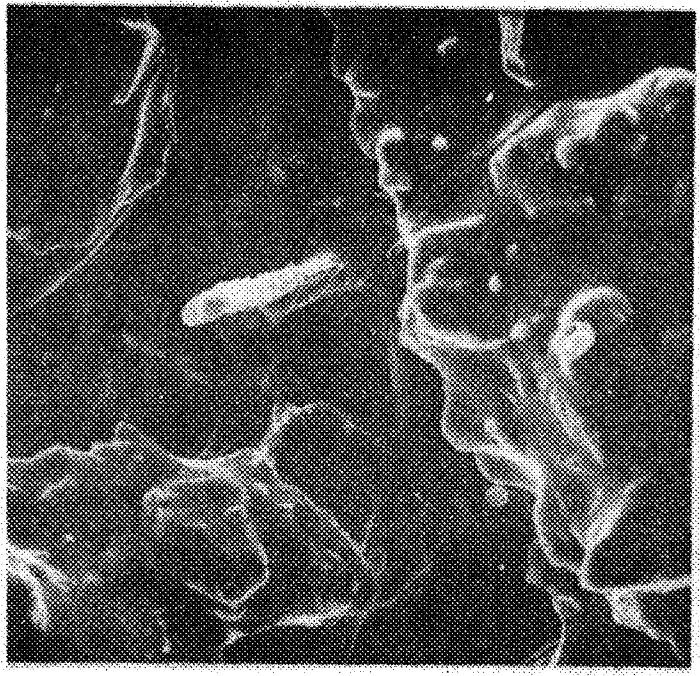

Figure C31. SEA 30/70 Mixture (AR-2000, crushed basalt, Marshall compaction) After Lottman Conditioning (560x)

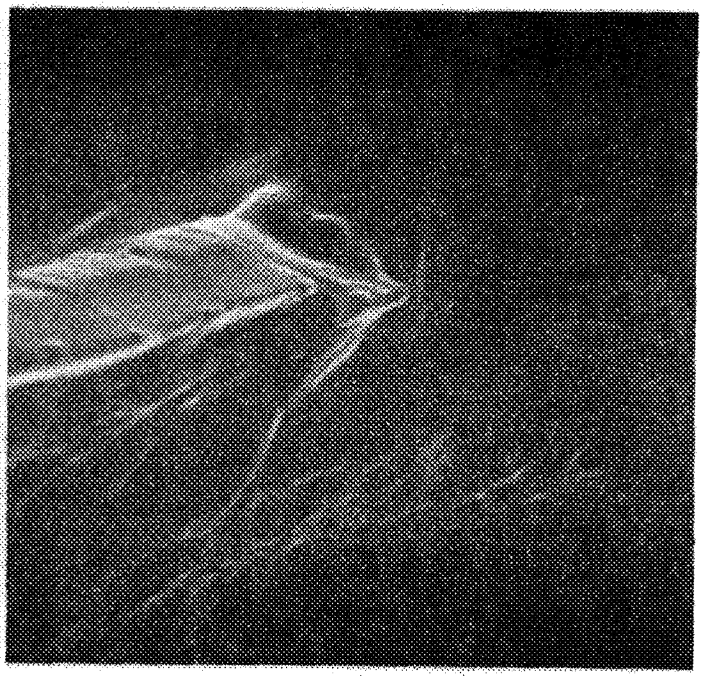

Figure C32. Close-up of Base of Sulfur Originally Shown in Figure C31 (5,600x)

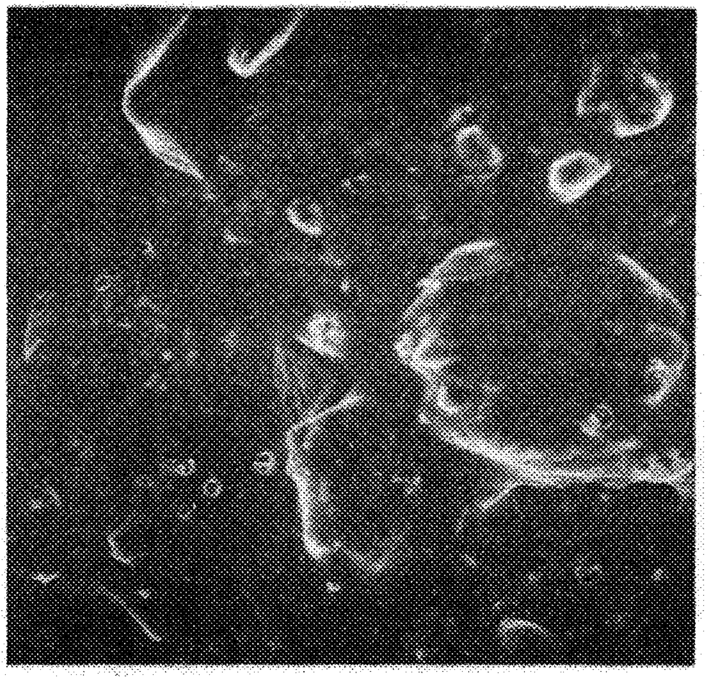

Figure C33. SEA 30/70 Mixture (AR-2000, crushed basalt, Marshall compaction) After Lottman Conditioning (510x)

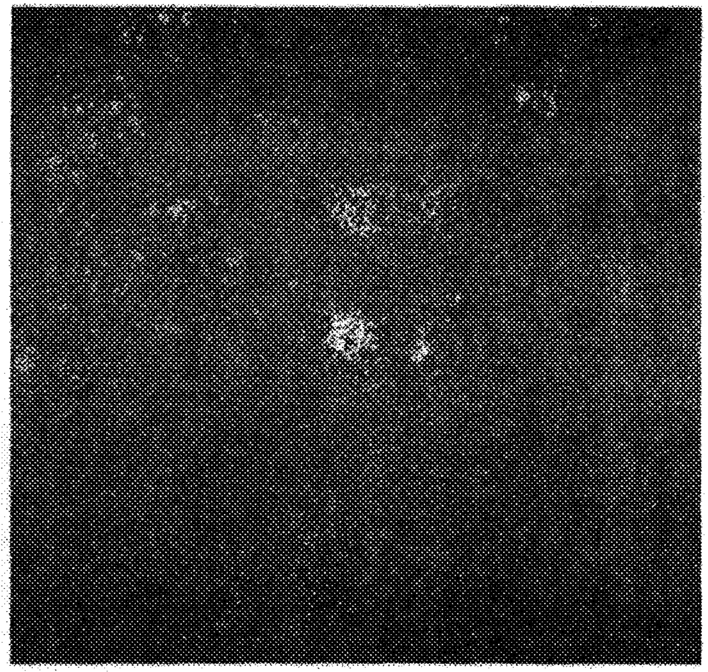

Figure C34. X-Ray Scan for Sulfur (Sample, location and magnification same as Figure C33)

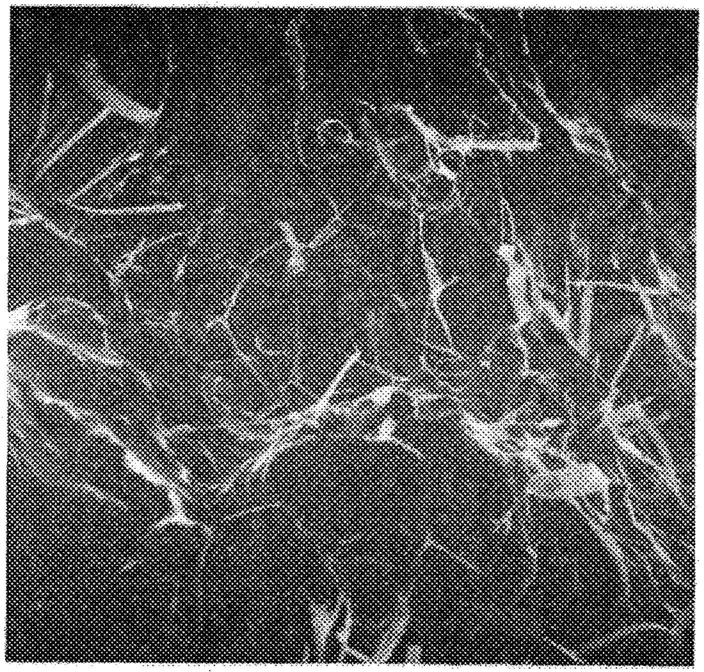

Figure C35. SEA 30/70 Mixture (AR-4000, crushed basalt, Marshall compaction) After Heat Soaking (216x)

C36. SEA 30/70 Mixture (AR-4000, crushed basalt, Marshall compaction) After Heat Soaking (1,040x).

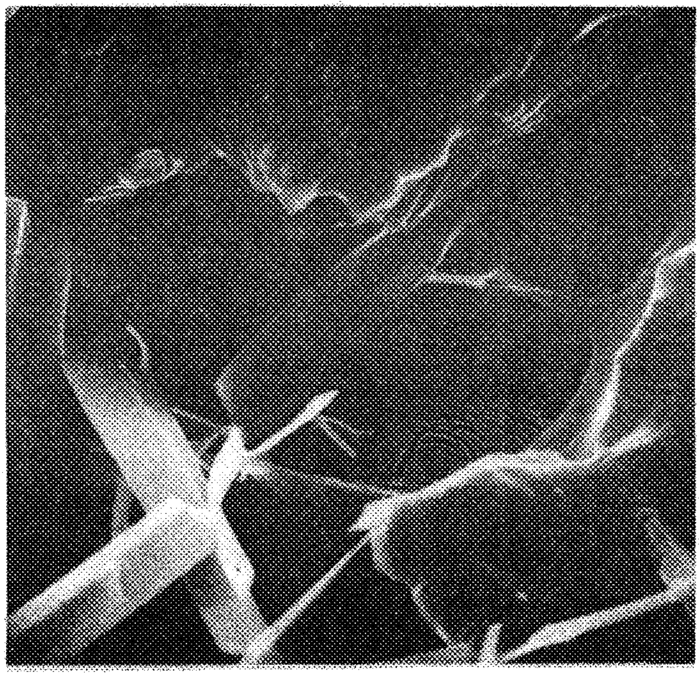

Figure C37. SEA 30/70 Mixture (AR-4000, crushed basalt, Marshall compaction) After Heat Soaking (1,060x).

Figure C38. SEA 30/70 Mixture (AR-4000, crushed basalt, Marshall compaction) After Heat Soaking (100x). Note: it is probable that cracks are due to sample preparation for SEM. Large sulfur crystals formed within three days following sample preparation.

Figure C39. SEA 30/70 Mixture (AR-4000, crushed basalt, Marshall compaction) After Lottman Conditioning (511x). Note: major features in photo are sulfur

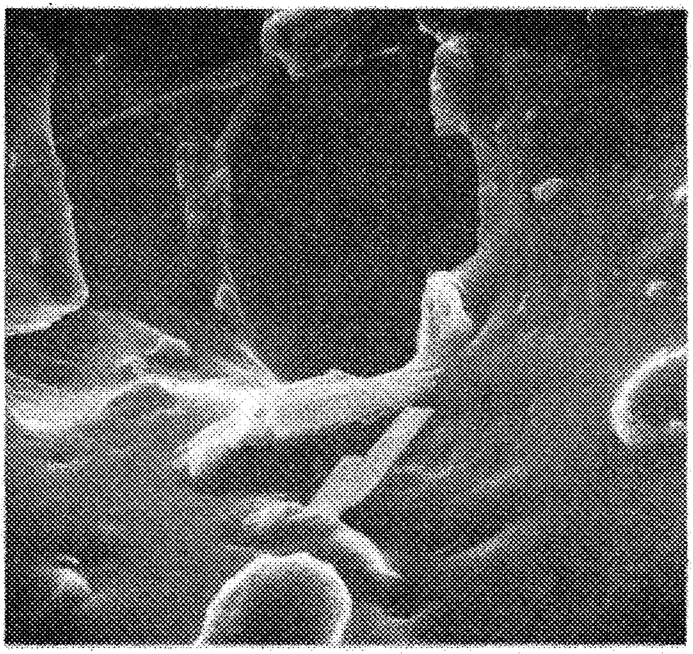

Figure C40. SEA 30/70 Mixture (AR-4000, crushed basalt, Marshall compaction) After Lottman Conditioning (1,095x)

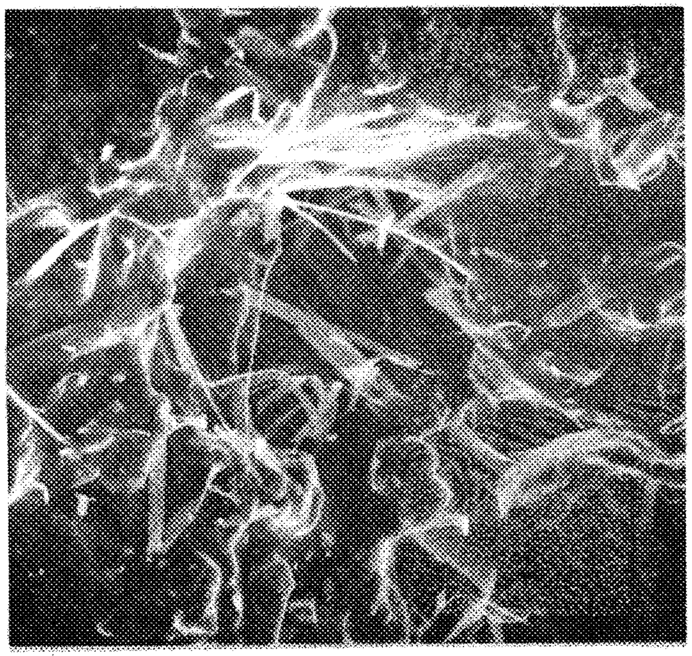

Figure C41. SEA 40/60 Mixture (AR-4000, crushed basalt, Marshall compaction) After Heat Soaking (325x)

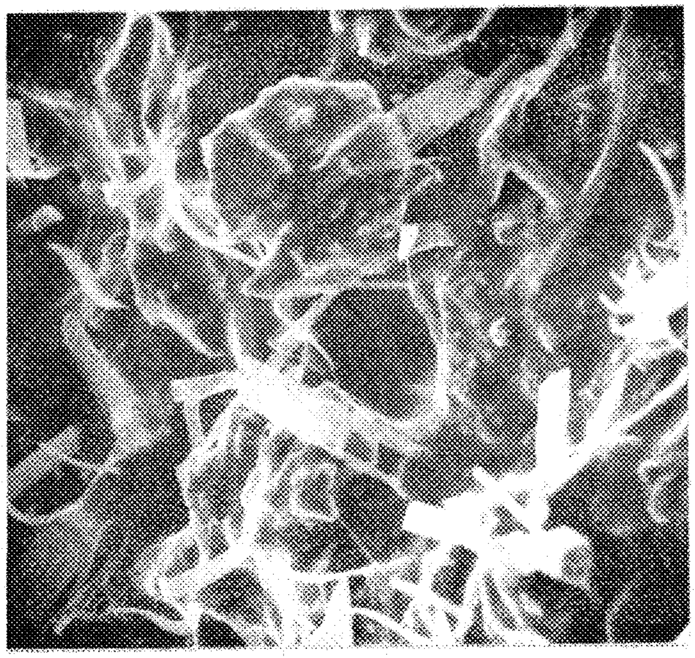

Figure C42. SEA 40/60 Mixture (AR-4000, crushed basalt, Marshall compaction) After Heat Soaking (495x)

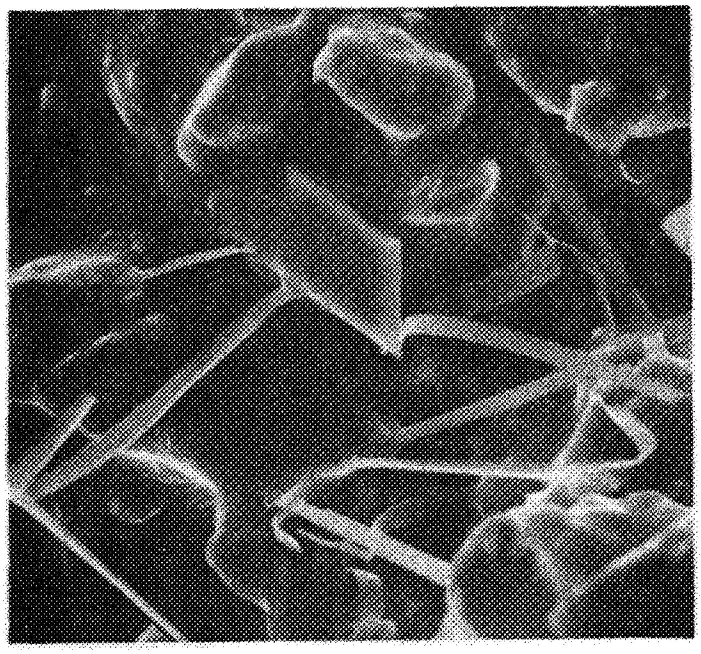

Figure C43. SEA 40/60 Mixture (AR-4000, crushed basalt, Marshall compaction) After Heat Soaking (1010x)

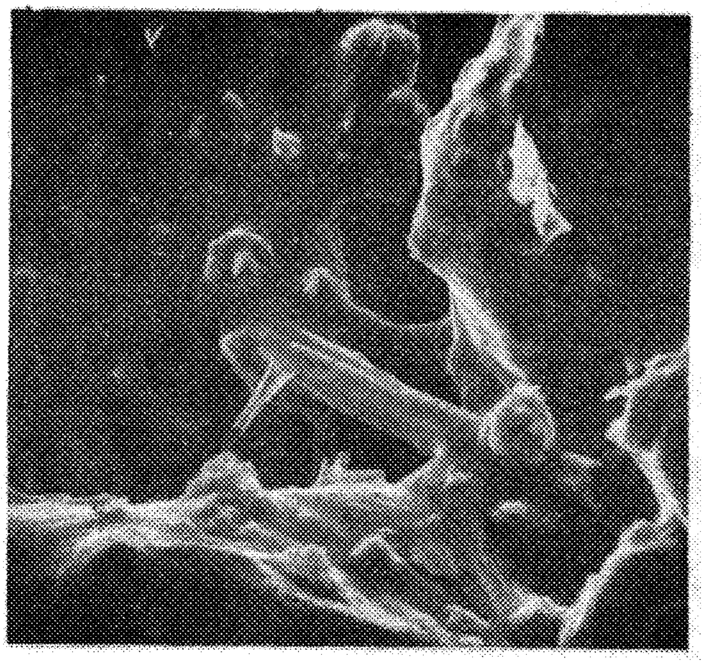

Figure C44. SEA 40/60 Mixture (AR-4000, crushed basalt, Marshall compaction) After Lottman Conditioning (535x)

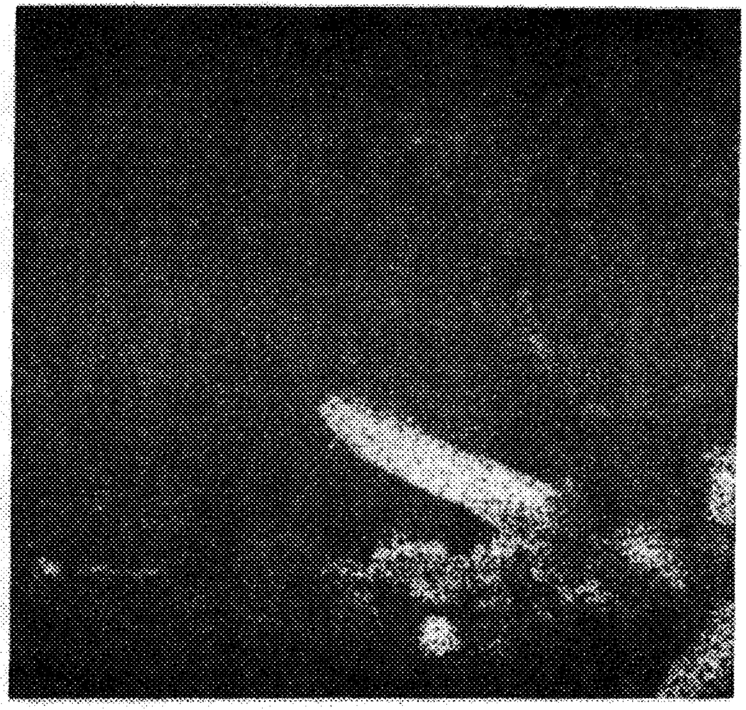

Figure C45. X-Ray Scan for Sulfur (Sample, location and magnification same as Figure C44)

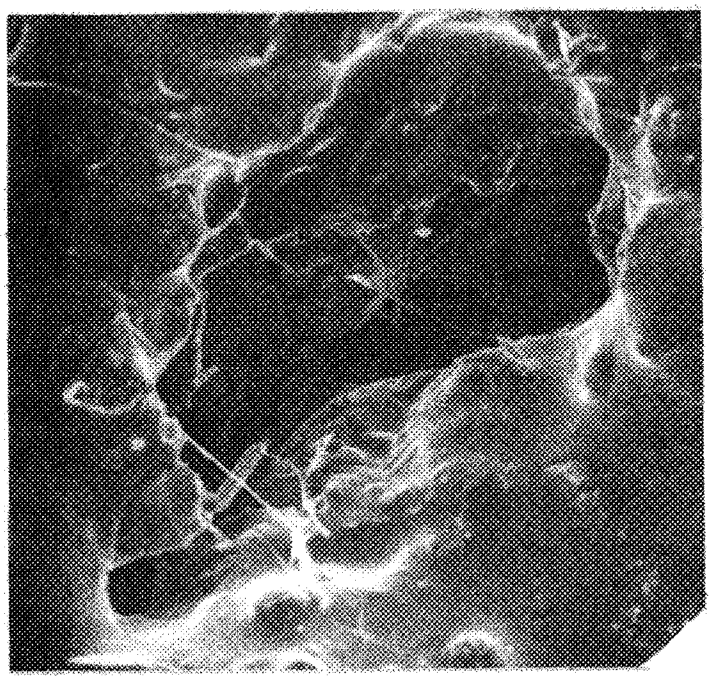

Figure C46. SEA 40/60 Mixture (AR-4000, crushed basalt, Marshall compaction) After Lottman Conditioning (208x). Note: this mixture void is not typical for the scanned sample in that few voids contained sulfur crystals

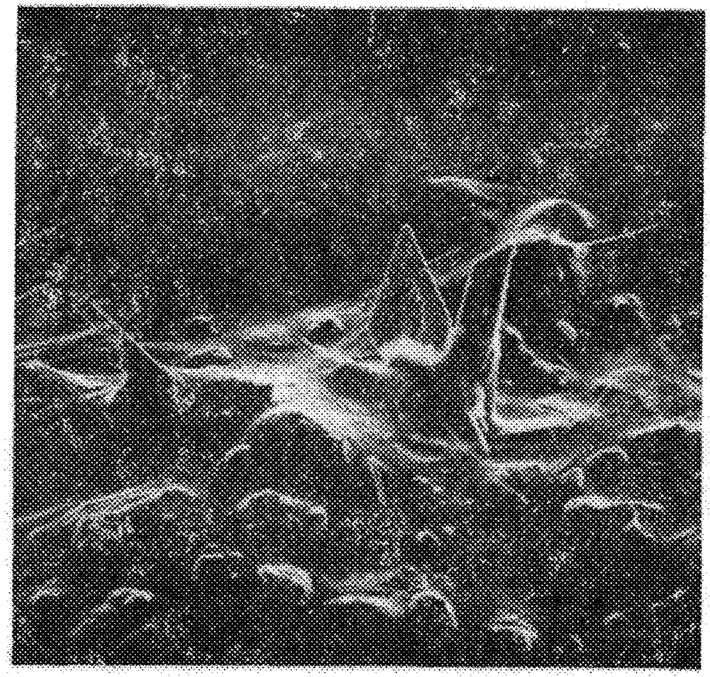

Figure C47. SEA 30/70 Mixture (AR-2000, crushed basalt, kneading compaction) After Heat Soaking (198x)

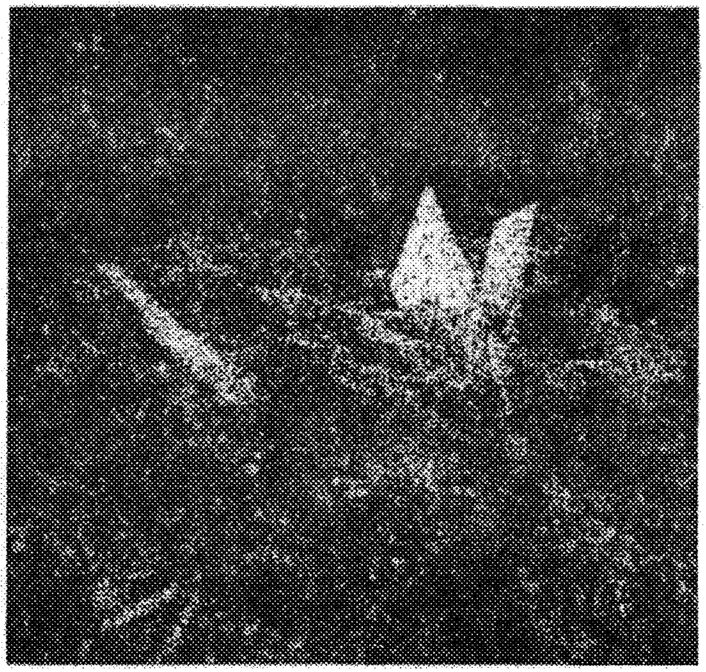

Figure C48. X-Ray Scan for Sulfur (Sample, location and magnification same as in Figure C47)

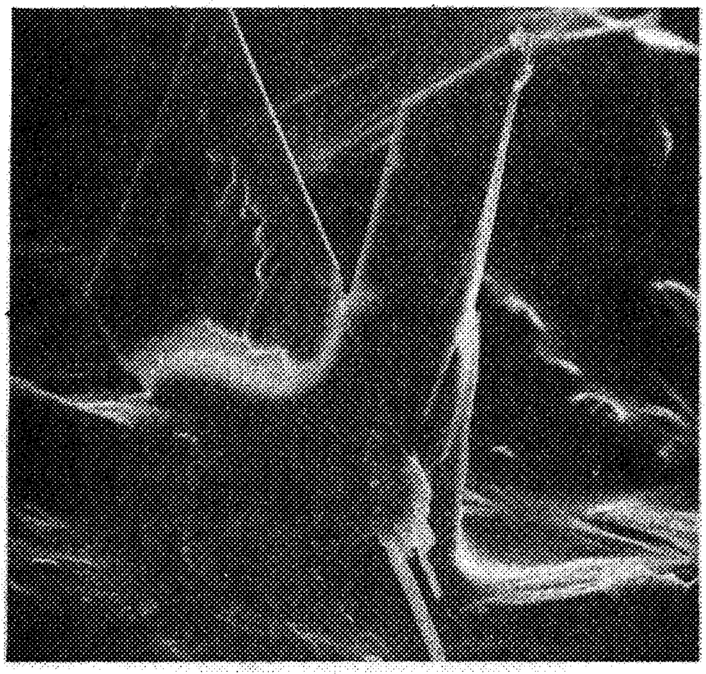

Figure C49. Close-up of Sulfur Originally Shown in Figure C47 (595x)

Figure C50. SEA 30/70 Mixture (AR-2000, crushed basalt, kneading compaction) After Heat Soaking (640x). Note: sulfur has ruptured the binder film

Figure C51. X-Ray Scan for Sulfur (Sample, location and magnification same as Figure C50). Note: low sulfur count in lower right of photo due to sample geometry

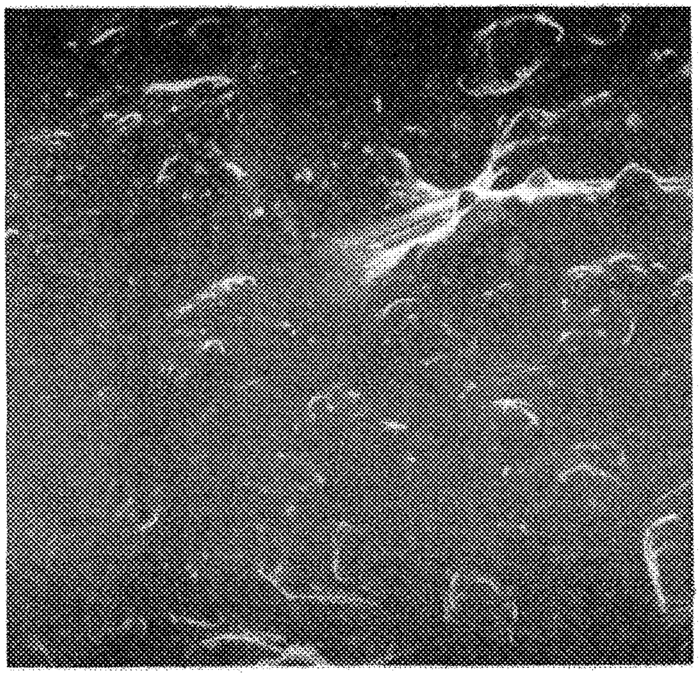

Figure C52. SEA 30/70 Mixture (AR-2000, crushed basalt, kneading compaction) After Lottman Conditioning (222x)

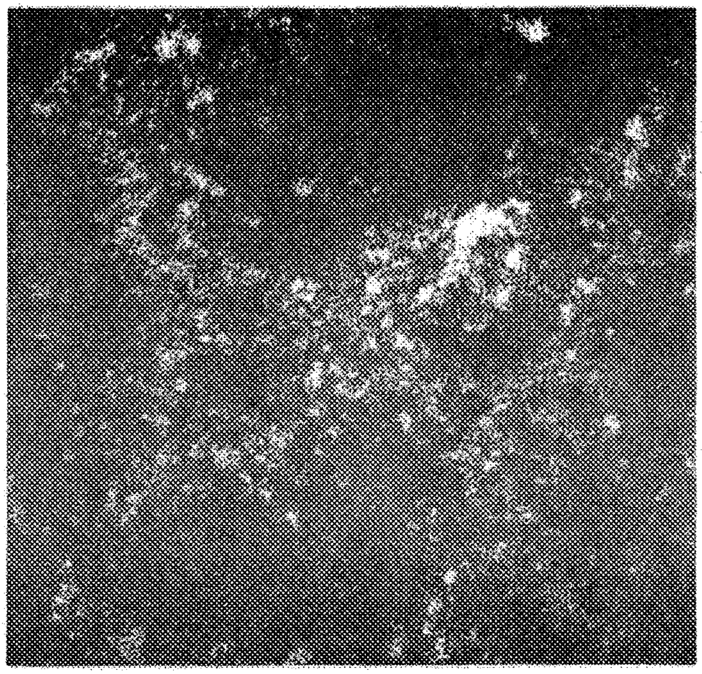

Figure C53. X-Ray Scan for Sulfur (Sample, location and magnification same as Figure C52). Note: dark areas primarily due to sample geometry

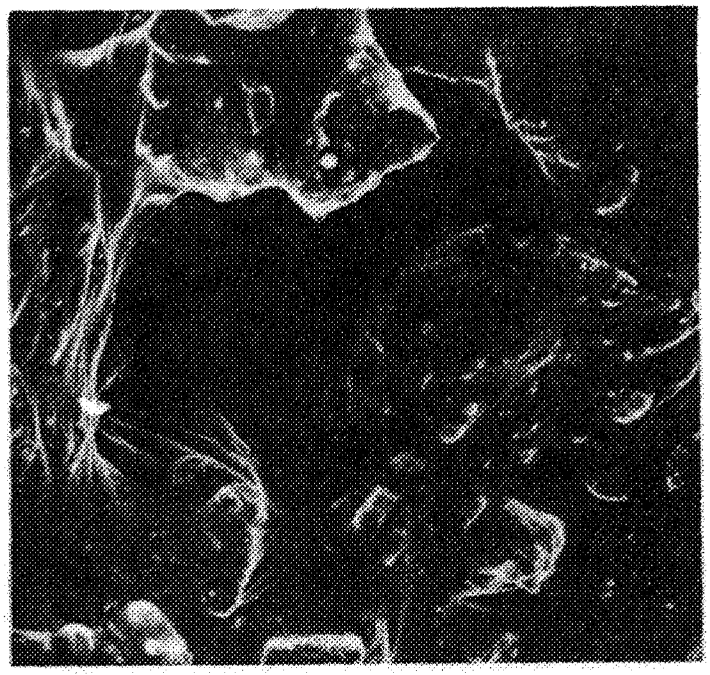

Figure C54. SEA 30/70 Mixture (AR-2000, crushed basalt, kneading compaction) After Lottman Conditioning (232x)

Figure C55. SEA 30/70 Mixture (AR-4000, crushed basalt, kneading compaction) After Heat Soaking (210x)

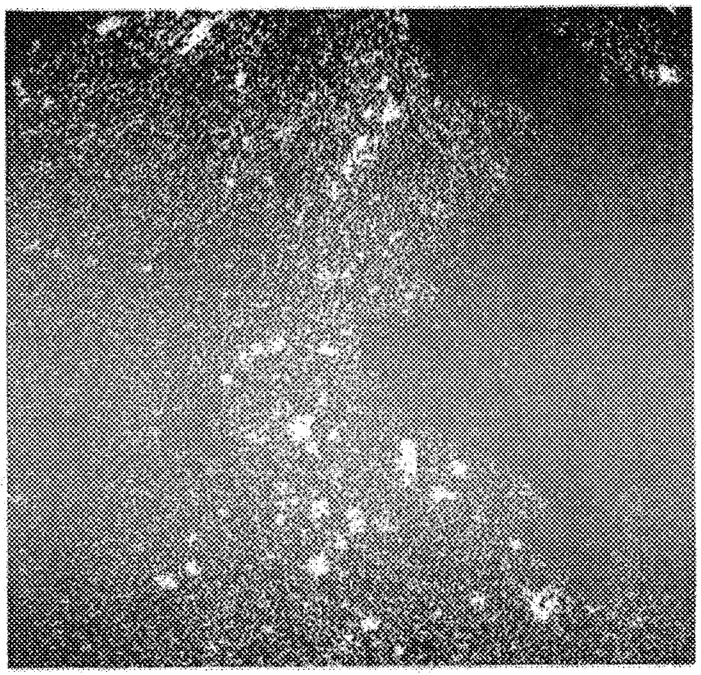

Figure C56. X-Ray Scan for Sulfur (Sample, location and magnification same as Figure C55). Note: dark area in right portion of photo due to sample geometry

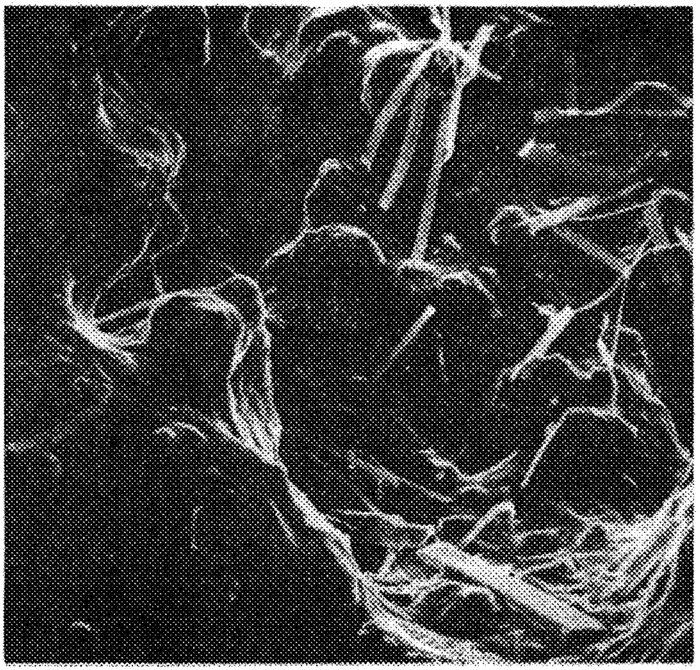

Figure C57. SEA 30/70 Mixture (AR-4000, crushed basalt, kneading compaction) After Heat Soaking (216x).

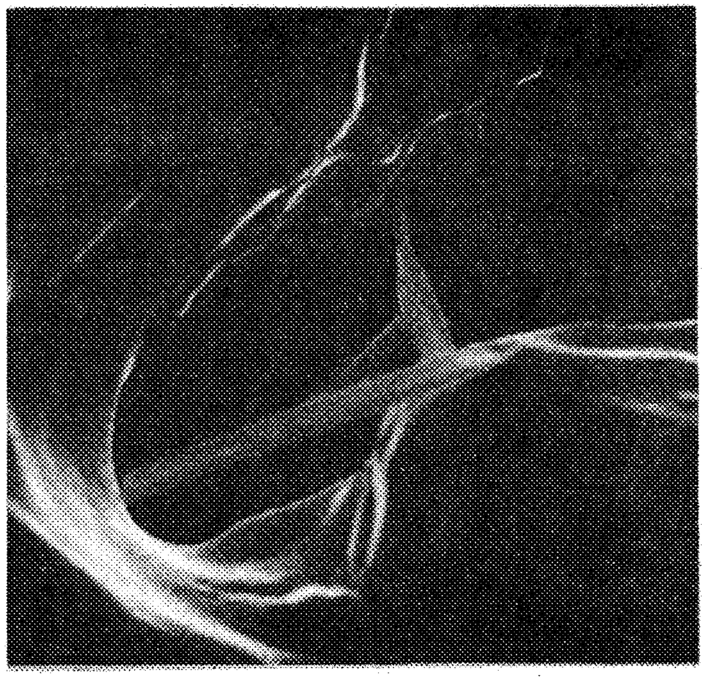

Figure C58. Close-up of Sulfur Originally Shown in Figure C57 (1,095x)

APPENDIX D

MIXTURE DESIGN DATA

MIXTURE DESIGN DATA

Tables D1-D15 contained in this appendix are used to document the initial mixture design data for the various mixture combinations studied. Early in the study, the decision was made to use equivalent binder volumes for all SEA mixtures. An index for the initial mixture design data is as follows:

Table	SEA Ratio	Asphalt Cement Type	Aggregate Type	Compaction Method
D1	0/100	AR-4000	Crushed Gravel	Marshall
D2	20/80	AR-4000	Crushed Gravel	Marshall
D3	30/70	AR-4000	Crushed Gravel	Marshall
D4	40/60	AR-4000	Crushed Gravel	Marshall
D5	50/50	AR-4000	Crushed Gravel	Marshall
D6	0/100	AR-2000	Crushed Gravel	Marshall
D7	0/100	AR-4000	Crushed Basalt	Marshall
D8	0/100	AR-2000	Crushed Basalt	Marshall
D9	0/100	AR-4000	Crushed Gravel	Kneading
D10	20/80	AR-4000	Crushed Gravel	Kneading
D11	30/70	AR-4000	Crushed Gravel	Kneading
D12	40/60	AR-4000	Crushed Gravel	Kneading
D13	0/100	AR-2000	Crushed Gravel	Kneading
D14	0/100	AR-4000	Crushed Basalt	Kneading
D15	0/100	AR-2000	Crushed Basalt	Kneading

Tables D16-D23 are used to provide summaries of the optimum binder contents selected for each SEA ratio and mixture combination.

Table D1. Marshall Mix Design Data Summary
(0/100 Mixture - Conventional Class B)

Aggregate: Crushed Gravel
Asphalt: AR-4000
SEA Ratio: 0/100

Binder Content (% by wt.)	Bulk Sp. Gr.	Unit Wt. (pcf)	Max Sp. Gr.	Marshall		VMA	Air Voids (%)
				Stability* (lb)	Flow		
4.5	2.288	142.8	2.537	2212	8.5	16.5	9.8
5.0	2.327	145.2	2.517	2423	8.3	15.6	7.6
5.5	2.345	146.3	2.498	2405	9.5	15.4	6.1
6.0	2.370	147.9	2.479	2413	11.1	14.9	4.4
6.5	2.392	149.3	2.461	2317	11.3	14.6	2.8
7.0	2.392	149.3	2.442	2210	12.7	15.0	2.0

*Time after compaction: 2 days

Table D2. Marshall Mix Design Data Summary (20/80 SEA Binder)

Aggregate: Crushed Gravel
Asphalt: AR-4000
SEA Ratio: 20/80

Binder Content	Bulk Sp. Gr.	Unit Wt.	Max. Sp. Gr.	Marshall		VMA	Air Voids
				Stability*	Flow		
4.5	2.300	143.5	2.523	1911	8.1	16.1	8.8
5.0	2.324	145.0	2.506	2126	8.4	15.7	7.3
5.5	2.331	145.5	2.490	2271	8.7	15.9	6.4
6.0	2.360	147.3	2.473	2443	10.8	15.3	4.6
6.5	2.375	148.2	2.457	2157	8.5	15.2	3.3
7.0	2.381	148.6	2.441	2040	10.4	15.4	2.5

*Time after compaction: 2 days

Table D3. Marshall Mix Design Data Summary (30/70 SEA Binder)

Aggregate: Crushed Gravel
Asphalt: AR-4000
SEA Ratio: 30/70

Binder Content	Bulk Sp. Gr.	Unit Wt.	Max. Sp. Gr.	Marshall		VMA	Air Voids
				Stability*	Flow		
5.0	2.305	143.8	2.561	1985	7.7	16.4	10.0
5.5	2.323	145.0	2.545	2078	8.3	16.2	8.7
6.0	2.338	145.9	2.530	1804	8.4	16.1	7.6
6.5	2.355	147.0	2.514	2037	8.4	15.9	6.3
7.0	2.384	148.8	2.499	2009	8.7	15.3	4.6
7.5	2.393	149.3	2.484	2102	9.5	15.5	3.7

*Time after compaction: 2 days

Table D4. Marshall Mix Design Data
Summary (40/60 SEA Binder)

Aggregate: Crushed Gravel
Asphalt: AR-4000
SEA Ratio: 40/60

Binder Content	Bulk Sp. Gr.	Unit Wt.	Max. Sp. Gr.	Marshall		VMA	Air Voids
				Stability*	Flow		
6.5	2.340	146.0	2.524	1896	8.3	16.4	7.3
7.0	2.373	148.1	2.511	1937	9.3	15.7	5.5
7.5	2.393	149.3	2.497	2078	9.4	15.5	4.2

*Time after compaction: 2 days

Table D5. Marshall Mix Design Data Summary (50/50 SEA Binder)

Aggregate: Crushed Gravel
Asphalt: AR-4000
SEA Ratio: 50/50

Binder Content	Bulk Sp. Gr.	Unit Wt.	Max. Sp. Gr.	Marshall		VMA	Air Voids
				Stability*	Flow		
7.0	2.373	148.0	2.544	3487	10.5	15.7	6.7
7.5	2.379	148.4	2.532	3225	9.5	15.9	6.0
8.0	2.387	148.9	2.520	3044	9.5	16.1	5.3

*Time after compaction: 2 days

Table D6. Marshall Mix Design Data Summary
(0/100 Mixture - Conventional Class B)

Aggregate: Crushed Gravel
Asphalt: AR-2000
SEA Ratio: 0/100

Binder Content	Bulk Sp. Gr.	Unit Wt.	Max. Sp. Gr.	Marshall		VMA	Air Voids
				Stability*	Flow		
4.5	2.331	145.4	2.533	2294	8.6	15.0	8.0
5.0	2.339	145.9	2.513	2121	8.4	15.1	6.9
5.5	2.375	148.2	2.494	2275	9.3	14.3	4.8
6.0	2.395	149.4	2.475	2290	9.2	14.0	3.2
6.5	2.407	150.2	2.456	2308	10.7	14.0	2.0
7.0	2.408	150.3	2.438	2294	11.9	14.5	1.2

*Time after compaction: 2 days

Table D7. Marshall Mix Design Data Summary
(0/100 Mixture - Conventional Class B)

Aggregate: Crushed Basalt
Asphalt: AR-4000
SEA Ratio: 0/100

Binder Content (% by wt.)	Bulk Sp. Gr.	Unit Wt. (pcf)	Max. Sp. Gr.	Marshall		VMA (%)	Air Voids (%)
				Stability* (lb)	Flow		
4.9	2.503	156.2	2.672	3195	8.7	12.0	6.4
5.2	2.530	157.9	2.659	2963	9.6	11.2	4.9
5.5	2.560	159.7	2.646	3217	11.3	10.5	3.3
5.8	2.557	159.6	2.633	3062	12.9	10.8	2.8
6.1	2.556	159.5	2.619	2867	11.4	11.0	2.3

*Time after compaction: 2 days

Table D8. Marshall Mix Design Data Summary (0/100 Mixture - Conventional Class B)

Aggregate: Crushed Basalt
Asphalt: AR-2000
SEA Ratio: 0/100

Binder Content (% by wt.)	Bulk Sp. Gr.	Unit Wt. (pcf)	Max. Sp. Gr.	Marshall		VMA (%)	Air Voids (%)
				Stability* (lb)	Flow		
4.7	2.496	155.8	2.706	2602	8.1	11.9	7.0
5.0	2.500	156.0	2.643	2678	7.7	12.1	6.1
5.3	2.532	158.0	2.660	2814	8.5	11.2	4.4
5.6	2.547	158.9	2.628	2811	9.3	11.0	3.4
5.9	2.548	159.0	2.601	2746	9.5	11.3	2.9

*Time after compaction: 2 days

Table D9. Hveem Mix Design Data Summary (0/100 Mixture - Conventional Class B)

Aggregate: Crushed Gravel
Asphalt: AR-4000
SEA Ratio: 0/100

Binder Content (% by wt.)	Bulk Sp. Gr.	Unit Wt. (pcf)	Max. Sp. Gr.	Hveem Stability	Marshall		VMA (%)	Air Voids (%)
					Stability* (lb)	Flow		
4.5	2.340	146.0	2.525	38	2616	13.4	14.6	6.9
5.0	2.343	146.2	2.503	38	2530	14.0	15.1	6.0
5.5	2.363	147.2	2.476	37	2586	14.1	14.5	4.4
6.0	2.373	147.9	2.457	39	2816	15.4	14.5	3.7
6.5	2.396	149.8	2.471	34	2603	15.9	14.3	1.6

Marshall values obtained after Hveem stability measurement

*Time after compaction: 2 days

Table D10. Hveem Mix Design Data Summary
(20/80 SEA Binder)

Aggregate: Crushed Gravel
Asphalt: AR-4000
SEA Ratio: 20/80

Binder Content (% by wt.)	Bulk Sp. Gr.	Unit Wt. (pcf)	Max. Sp. Gr.	Hveem Stability	Marshall		VMA (%)	Air Voids (%)
					Stability* (lb)	Flow		
5.5	2.366	147.7	2.474	38	1942	13.4	14.5	5.8
6.0	2.406	150.2	2.495	36	2276	15.1	13.8	3.9
6.5	2.413	150.4	2.481	33	2370	15.1	13.9	2.8

Marshall values obtained after Hveem stability measurement

*Time after compaction: 2 days

Table D11. Hveem Mix Design Data Summary
(30/70 SEA Binder)

Aggregate: Crushed Gravel
Asphalt: AR-4000
SEA Ratio: 30/70

Binder Content (% by wt.)	Bulk Sp. Gr.	Unit Wt. (pcf)	Max. Sp. Gr.	Hveem Stability*	Marshall		VMA (%)	Air Voids (%)
					Stability (lb)	Flow		
6.0	2.383	148.7	2.504	38	2283	12.8	14.5	4.9
6.5	2.417	151.0	2.488	35	2747	13.8	13.6	2.7
7.0	2.433	151.0	2.474	36	2737	14.7	14.0	2.2

Marshall values obtained after Hveem stability measurement

*Time after compaction: 2 days

Table D12. Hveem Mix Design Data Summary
(40/60 SEA Binder)

Aggregate: Crushed Gravel
Asphalt: AR-4000
SEA Ratio: 40/60

Binder Content (% by wt.)	Bulk Sp. Gr.	Unit Wt. (pcf)	Max. Sp. Gr.	Hveem Stability*	Marshall		VMA (%)	Air Voids (%)
					Stability (lb)	Flow		
6.0	2.396	149.6	2.501	48	2316	13.3	13.8	4.4
6.5	2.433	151.8	2.488	45	2989	14.3	13.2	2.3
7.0	2.423	151.2	2.475	43	2737	14.3	14.0	2.2

Marshall values obtained after Hveem stability measurement

*Time after compaction: 2 days

Table D13. Hveem Mix Design Data Summary (0/100 Mixture - Conventional Class B)

Aggregate: Crushed Gravel
Asphalt: AR-2000
SEA Ratio: 0/100

Binder Content (% by wt.)	Bulk Sp. Gr.	Unit Wt. (pcf)	Max. Sp. Gr.	Hveem Stability*	Marshall		VMA (%)	Air Voids (%)
					Stability (lb)	Flow		
4.9	2.320	144.8	2.521	38	2087	12.0	15.7	8.6
5.2	2.343	146.2	2.510	40	2345	14.6	15.3	6.8
5.5	2.377	148.3	2.481	37	2485	13.6	14.1	4.8
5.8	2.396	149.6	2.490	35	2504	12.7	13.6	3.5
6.1	2.410	150.4	2.481	31	2597	13.8	13.6	3.4

Marshall values obtained after Hveem stability measurement

*Time after compaction: 2 days

Table D14. Hveem Mix Design Data Summary
(0/100 Mixture - Conventional Class B)

Aggregate: Crushed Basalt
Asphalt: AR-4000
SEA Ratio: 0/100

Binder Content (% by wt.)	Bulk Sp. Gr.	Unit Wt. (pcf)	Max. Sp. Gr.	Hveem Stability*	Marshall		VMA (%)	Air Voids (%)
					Stability (lb)	Flow		
4.5	2.438	152.1	2.686	45	2148	11.1	13.8	9.2
5.0	2.479	154.7	2.664	42	2646	12.0	12.8	6.9
5.5	2.520	157.3	2.641	40	2737	14.3	11.9	4.5
6.0	2.525	157.6	2.620	38	2840	14.1	11.9	3.6
6.5	2.558	159.6	2.598	24	3139	20.6	11.4	1.5

Marshall values obtained after Hveem stability measurement

*Time after compaction: 2 days

Table D15. Hveem Mix Design Data Summary (0/100 Mixture - Conventional Class B)

Aggregate: Crushed Basalt
Asphalt: AR-2000
SEA Ratio: 0/100

Binder Content (% by wt.)	Bulk Sp. Gr.	Unit Wt. (pcf)	Max. Sp. Gr.	Hveem Stability*	Marshall		VMA (%)	Air Voids (%)
					Stability (lb)	Flow		
4.5	2.442	152.4	2.732	37	2150	11.5	13.7	9.8
5.1	2.482	154.8	2.684	40	2628	10.8	12.8	7.3
5.5	2.485	155.0	2.640	39	2260	10.5	13.1	5.3
6.0	2.540	158.5	2.631	36	2888	13.1	11.6	3.7
6.5	2.551	159.2	2.594	30	2788	15.2	11.7	2.5

Marshall values obtained after Hveem stability measurement

*Time after compaction: 2 days

Table D16. Summary of Optimum Binder Contents for Marshall Mix Design (14 days after compaction)

Aggregate: Crushed Gravel
Asphalt: AR-4000

SEA Ratio	Binder Content (%)*	Bulk Sp. Gr.	Marshall	
			Stability (lb)	Flow
0/100	6.0	2.389	2173	9.2
20/80	6.4	2.390	2301	9.3
30/70	6.8	2.395	2754	9.1
40/60	7.2	2.415	3478	8.7
50/50	7.7	2.405	4441	8.6

*SEA binder contents based on equal volume of 0/100 mixture.

Table D17. Summary of Optimum Binder Contents for Marshall Mix Design (14 days after compaction)

Aggregate: Crushed Gravel
Asphalt: AR-2000

SEA Ratio	Binder Content (%)*	Bulk Sp. Gr.	Marshall	
			Stability (lb)	Flow
0/100	5.8	2.373	2073	8.9
20/80	6.4	2.396	2394	8.3
30/70	6.6	2.400	2883	8.7
40/60	7.1	2.387	3666	8.9
50/50	7.4	2.383	4130	8.1

*SEA binder contents based on equal volume of 0/100 mixture.

Table D18. Summary of Optimum Binder Contents for Marshall Mix Design (14 days after compaction)

Aggregate: Crushed Basalt
Asphalt: AR-4000

SEA Ratio	Binder Content (%)	Bulk Sp. Gr.	Marshall	
			Stability (lb)	Flow
0/100	5.5	2.576	3353	11.0
20/80	6.0	2.566	3221	8.5
30/70	6.4	2.545	3918	8.9
40/60	6.7	2.569	5736	8.5

Table D19. Summary of Optimum Binder Contents for Marshall Mix Design (14 days after compaction)

Aggregate: Crushed Basalt

Asphalt: AR-2000

SEA Ratio	Binder Content (%)	Bulk Sp. Gr.	Marshall	
			Stability (lb)	Flow
0/100	5.5	2.555	2831	9.4
20/80	6.1	2.558	3063	8.4
30/70	6.4	2.567	3252	11.4
40/60	6.8	2.578	3156	11.1

Table D20. Summary of Optimum Binder Contents for Hveem Mix Design (14 days after compaction)

Aggregate: Crushed Gravel
Asphalt: AR-4000

SEA Ratio	Binder Content (%)	Bulk Sp. Gr.	Hveem Stability	Marshall*	
				Stability (lb)	Flow
0/100	6.0	2.370	33	2743	13.7
20/80	6.5	2.399	32	2787	13.4
30/70	6.9	2.464	34	3257	16.2
40/60	7.3	2.439	30	3376	16.2

*Marshall values obtained after Hveem stability measurement

Table D21. Summary of Optimum Binder Contents for Hveem Mix Design (14 days after compaction)

Aggregate: Crushed Gravel
Asphalt: AR-2000

SEA Ratio	Binder Content (%)	Bulk Sp. Gr.	Hveem Stability	Marshall*	
				Stability (lb)	Flow
0/100	5.8	2.380	36	2420	16.0
20/80	6.4	2.410	37	3163	15.6
30/70	6.7	2.446	39	2681	12.0
40/60	7.1	2.458	45	3287	10.4

*Marshall values obtained after Hveem stability measurement

Table D22. Summary of Optimum Binder Contents for Hveem Mix Design (14 days after compaction)

Aggregate: Crushed Basalt
Asphalt: AR-4000

SEA Ratio	Binder Content (%)	Bulk Sp. Gr.	Hveem Stability	Marshall*	
				Stability (lb)	Flow
0/100	5.5	2.511	39	2649	14.5
20/80	6.0	2.497	38	2319	12.2
30/70	6.4	2.519	39	2743	12.0
40/60	6.7	2.524	43	3484	10.2

*Marshall values obtained after Hveem stability measurement

Table D23. Summary of Optimum Binder Contents for Hveem Mix Design (14 days after compaction)

Aggregate: Crushed Basalt

Asphalt: AR-2000

SEA Ratio	Binder Content (%)	Bulk Sp. Gr.	Hveem Stability	Marshall*	
				Stability (lb)	Flow
0/100	5.5	2.525	34	2365	14.3
20/80	6.1	2.541	39	2653	14.5
30/70	6.4	2.530	40	2700	13.1
40/60	6.8	2.542	38	3473	11.3

*Marshall values obtained after Hveem stability measurement

APPENDIX E

AGING AND MOISTURE CONDITIONING DATA

Table E1. Resilient Modulus After Heat Soaking for Marshall Compacted Samples

Aggregate: Crushed Gravel

Asphalt: Chevron AR-4000

Cumulative Heat Soak Time (hrs)*	Resilient Modulus (x 10^3 psi) @ 25°C				
	0/100 SEA Ratio	20/80 SEA Ratio	30/70 SEA Ratio	40/60 SEA Ratio	50/50 SEA Ratio
0**	200.3	302.4	252.5	474.2	765.4
24	163.8	364.2	490.3	599.7	707.9
72	-	366.7	617.0	538.6	744.1
120	-	506.3	425.3	521.3	687.6
240	227.4	409.8	446.6	731.0	1011.5
480	331.2	474.1	553.3	680.4	951.2

*Heat soak temperature = 60°C

**Initial test: 14 days after compaction

Table E2. Resilient Modulus After Heat Soaking for Marshall Compacted Samples

Aggregate: Crushed Gravel
Asphalt: Chevron AR-2000

Cumulative Heat Soak Time (hrs)*	Resilient Modulus (x 10^3 psi) @ 25°C				
	0/100 SEA Ratio	20/80 SEA Ratio	30/70 SEA Ratio	40/60 SEA Ratio	50/50 SEA Ratio
0**	129.6	134.5	223.9	421.2	643.9
24	119.7	204.6	321.0	339.5	618.9
72	-	-	-	-	-
120	-	-	-	-	-
240	167.9	205.0	367.7	470.8	843.6
480	216.8	283.0	507.2	606.3	969.5

*Heat soak temperature = 60°C

**Initial test: 14 days after compaction

Table E3. Resilient Modulus After Heat Soaking for Marshall Compacted Samples

Aggregate: Crushed Basalt

Asphalt: Chevron AR-4000

Cumulative Heat Soak Time (hrs)*	Resilient Modulus (x 10^3 psi) @ 25°C			
	0/100 SEA	20/80 SEA	30/70 SEA	40/60 SEA
0**	240.6	185.7	425.8	620.7
24	262.3	329.2	507.7	680.7
72	332.3	393.4	-	-
120	386.2	401.4	522.2	726.6
240	345.7	421.5	625.7	756.1
480	389.2	508.9	675.1	770.3

*Heat soak temperature = 60°C

**Initial test: 14 days after compaction

Table E4. Resilient Modulus After Heat Soaking for Marshall Compacted Samples

Aggregate: Crushed Basalt

Asphalt: Chevron AR-2000

Cumulative Heat Soak Time (hrs)*	Resilient Modulus (x 10^3 psi) @ 25°C			
	0/100 SEA	20/80 SEA	30/70 SEA	40/60 SEA
0**	213.3	190.3	261.0	422.0
24	224.9	293.9	345.3	444.8
72	237.9	274.3	337.1	423.8
120	246.3	289.6	332.9	443.2
240	293.1	280.8	363.8	481.8
480	277.3	274.1	433.9	555.6

*Heat soak temperature = 60°C

**Initial test: 14 days after compaction

Table E5. Resilient Modulus After Heat Soaking for Kneading Compacted Samples

Aggregate: Crushed Gravel

Asphalt: Chevron AR-4000

Cumulative Heat Soak Time (hrs)*	Resilient Modulus (x 10^3 psi) @ 25°C			
	0/100 SEA	20/80 SEA	30/70 SEA	40/60 SEA
0**	201.7	173.9	236.5	287.6
24	200.0	193.1	224.9	237.8
72	215.9	194.8	250.3	300.5
120	249.1	212.3	245.5	275.1
240	262.4	249.5	251.6	293.7
480	327.4	257.5	293.0	326.9

*Heat soak temperature = 60°C

**Initial test: 14 days after compaction

Table E6. Resilient Modulus After Heat Soaking for Kneading Compacted Samples

Aggregate: Crushed Gravel

Asphalt: Chevron AR-2000

Cumulative Heat Soak Time (hrs)*	Resilient Modulus (x 10^3 psi) @ 25°C			
	0/100 SEA	20/80 SEA	30/70 SEA	40/60 SEA
0**	116.4	131.3	240.3	387.6
24	-	-	279.2	340.7
72	131.0	194.2	202.6	383.2
120	148.4	183.2	216.8	332.6
240	186.1	193.2	284.5	319.3
480	156.4	216.6	268.6	448.9

*Heat soak temperature = 60°C

**Initial test: 14 days after compaction

Table E7. Resilient Modulus After Heat Soaking for Kneading Compacted Samples

Aggregate: Crushed Basalt

Asphalt: Chevron AR-4000

Cumulative Heat Soak Time (hrs)*	Resilient Modulus (x 10^3 psi) @ 25°C			
	0/100 SEA	20/80 SEA	30/70 SEA	40/60 SEA
0**	272.3	193.3	301.1	470.6
24	325.9	275.4	378.4	498.2
72	316.3	298.0	380.4	516.9
120	336.1	321.4	371.3	477.1
240	303.7	306.6	443.1	526.5
480	342.3	386.6	515.6	678.0

*Heat soak temperature = 60°C

**Initial test: 14 days after compaction

Table E8. Resilient Modulus After Heat Soaking for Kneading Compacted Samples

Aggregate: Crushed Basalt

Asphalt: Chevron AR-2000

Cumulative Heat Soak Time (hrs)*	Resilient Modulus (x 10^3 psi) @ 25°C			
	0/100 SEA	20/80 SEA	30/70 SEA	40/60 SEA
0**	148.7	158.6	273.9	440.5
24	163.7	216.0	247.9	359.9
72	202.2	233.1	230.5	351.3
120	169.6	215.1	222.5	340.3
240	209.5	271.0	231.8	374.1
480	216.9	302.1	286.9	510.6

*Heat soak temperature = 60°C

**Initial test: 14 days after compaction

Table E9. Resilient Modulus Before and After Moisture Conditioning for Marshall Compacted Samples

Aggregate: Crushed Gravel

Asphalt: Chevron AR-4000

Conditioning Status	Resilient Modulus (x 10^3 psi) @ 25°C				
	0/100 SEA Ratio	20/80 SEA Ratio	30/70 SEA Ratio	40/60 SEA Ratio	50/50 SEA Ratio
Before Moisture Conditioning *	196.8	323.9	246.9	388.0	747.2
After Moisture Conditioning	154.8	216.3	218.9	197.9	183.0
M_R Ratio (After ÷ Before)	.79	0.66	0.89	0.51	0.24

*Before resilient modulus testing: 14 days after compaction

Table E10. Resilient Modulus Before and After Moisture Conditioning for Marshall Compacted Samples

Aggregate: Crushed Gravel
Asphalt: Chevron AR-2000

Conditioning Status	Resilient Modulus (x 10^3 psi) @ 25°C				
	0/100 SEA Ratio	20/80 SEA Ratio	30/70 SEA Ratio	40/60 SEA Ratio	50/50 SEA Ratio
Before Moisture Conditioning*	140.0	146.6	257.8	403.7	662.7
After Moisture Conditioning	85.5	119.8	139.1	149.6	150.0
M_R Ratio (After ÷ Before)	0.61	0.82	0.54	0.37	0.23

*Before resilient modulus testing: 14 days after compaction

Table E11. Resilient Modulus Before and After Moisture Conditioning for Marshall Compacted Samples

Aggregate: Crushed Basalt

Asphalt: Chevron AR-4000

Conditioning Status	Resilient Modulus (x 10^3 psi) @ 25°C			
	0/100 SEA Ratio	20/80 SEA Ratio	30/70 SEA Ratio	40/60 SEA Ratio
Before Moisture Conditioning**	246.1	182.8	329.4*	472.5*
After Moisture Conditioning	229.4	360.9	286.4*	323.6*
M_R Ratio (After ÷ Before)	0.93	1.97	0.87	0.68

*Samples retested

**"Before" resilient modulus testing: 14 days after compaction

Table E12. Resilient Modulus Before and After Moisture Conditioning for Marshall Compacted Samples

Aggregate: Crushed Basalt

Asphalt: Chevron AR-2000

Conditioning Status	Resilient Modulus (x 10^3 psi) @ 25°C			
	0/100 SEA Ratio	20/80 SEA Ratio	30/70 SEA Ratio	40/60 SEA Ratio
Before Moisture Conditioning*	241.0	227.8	332.1	571.6
After Moisture Conditioning	149.9	150.4	180.9	232.9
M_R Ratio (After ÷ Before)	0.62	0.66	0.54	0.41

*"Before" resilient modulus testing: 14 days after compaction

Table E13. Resilient Modulus Before and After Moisture Conditioning for Kneading Compacted Samples

Aggregate: Crushed Gravel

Asphalt: Chevron AR-4000

Conditioning Status	Resilient Modulus (x 10^3 psi) @ 25°C			
	0/100 SEA Ratio	20/80 SEA Ratio	30/70 SEA Ratio	40/60 SEA Ratio
Before Moisture Conditioning*	223.2	167.1	250.2	252.2
After Moisture Conditioning	204.4	164.6	211.3	228.7
M_R Ratio (After ÷ Before)	0.92	0.99	0.84	0.91

*"Before" resilient modulus testing: 14 days after compaction

Table E14. Resilient Modulus Before and After Moisture Conditioning for Kneading Compacted Samples

Aggregate: Crushed Gravel

Asphalt: Chevron AR-2000

Conditioning Status	Resilient Modulus (x 10^3 psi) @ 25°C			
	0/100 SEA Ratio	20/80 SEA Ratio	30/70 SEA Ratio	40/60 SEA Ratio
Before Moisture Conditioning**	143.8*	208.3*	229.6	391.8
After Moisture Conditioning	176.4*	167.4*	153.7	203.8
M_R Ratio (After ÷ Before)	1.23	0.80	0.67	0.52

*Samples retested

**"Before" resilient modulus testing: 14 days after compaction

Table E15. Resilient Modulus Before and After Moisture Conditioning for Kneading Compacted Samples

Aggregate: Crushed Basalt

Asphalt: Chevron AR-4000

Conditioning Status	Resilient Modulus (x 10^3 psi) @ 25°C			
	0/100 SEA Ratio	20/80 SEA Ratio	30/70 SEA Ratio	40/60 SEA Ratio
Before Moisture Conditioning*	272.7	169.9	314.9	477.3
After Moisture Conditioning	198.3	158.9	215.7	242.7
M_R Ratio (After ÷ Before)	0.72	0.94	0.68	0.51

*"Before" resilient modulus testing: 14 days after compaction

Table E16. Resilient Modulus Before and After Moisture Conditioning for Kneading Compacted Samples

Aggregate: Crushed Basalt

Asphalt: Chevron AR-2000

Conditioning Status	Resilient Modulus (x 10^3 psi) @ 25°C			
	0/100 SEA Ratio	20/80 SEA Ratio	30/70 SEA Ratio	40/60 SEA Ratio
Before Moisture Conditioning*	174.1	168.9	241.3	436.6
After Moisture Conditioning	136.7	156.0	148.9	193.7
M_R Ratio (After ÷ Before)	0.79	0.92	0.62	0.44

*"Before" resilient modulus testing: 14 days after compaction

APPENDIX F

AGING AND MOISTURE CONDITIONING PROCEDURES

AGING CONDITIONING PROCEDURE

The following procedure was used in the study:

1. Place laboratory prepared samples in environmental cabinet at 77°F (25°C) for 3 - 4 hours.
2. Determine resilient modulus.
3. Place samples in oven at 140°F (60°C) for 24 hours.
4. Remove samples from oven and place in environmental cabinet at 77°F (25°C) overnight.
5. Determine resilient modulus.
6. Place samples in oven at 140°F (60°C) for 48 hours.
7. Remove samples from oven and place in environmental cabinet at 77°F (25°C) overnight.
8. Determine resilient modulus.
9. Place samples in oven at 140°F (60°C) for 48 hours.
10. Remove samples from oven and place in environmental cabinet at 77°F (60°C) overnight.
11. Determine resilient modulus.
12. Place samples in oven at 140°F (60°C) for 120 hours.
13. Remove samples from oven and place in environmental cabinet at 77°F (60°C) overnight.
14. Determine resilient modulus.
15. Place samples in oven at 140°F (60°C) for 240 hours (total cumulative hours of heat soak is 480).
16. Remove samples from oven and place in environmental cabinet at 77°F (25°C) overnight.
17. Determine resilient modulus.

MOISTURE CONDITIONING PROCEDURE

The following procedure was obtained from B. Lottman (University of Idaho) during 1981:

1. Prepare Specimens

   Prepare standard lab compacted specimens. Let specimens sit in lab for 14 days prior to starting testing.

2. Stabilize specimens at 77°F (25°C) for 3 - 4 hours and determine resilient modulus.

3. Vacuum Saturation and Soak

   Vacuum saturate the samples at 25 in. Hg for 15 minutes. Soak in distilled water for 30 minutes.

4. Prepare Specimen for Freezing

   Tightly wrap specimen in plastic wrap. Seal with freezer tape (sample is still wet from Step 2 at this point). Place wrapped specimen in plastic bag containing 10 ml of water. Seal bag.

5. Freeze Specimen

   Place specimen (as prepared in Step 3) in freezer. Freeze specimen for 15 hours at a temperature of 0 to 10°F (-18 to -12°C).

6. Heat Soak

   Remove specimen from freezer and place into a water bath at 140°F (60°C) for 2 to 3 minutes. This will thaw the plastic wrap and the surface of the specimen, thus allowing removal of the plastic wrap. Following removal of plastic wrap, place specimen back into the 140°F (60°C) water bath for 24 hours. For this 24 hour period, the specimen has no coating or container on or around it. Each specimen (while in water bath) should rest on a flat metal plate or disk for support. This insures that no deformation or distortion of the specimen occurs in the water bath.

7. Resilient Modulus Testing

   To prepare the specimen for resilient modulus determination, place into a water bath set at the appropriate testing temperature (say 41°F (5°C), 77°F (25°C), or 104°F (40°C). Let specimen remain in the water bath for at least 3 hours. After this soaking period, remove the specimen and quickly test.

8. General Comments by B. Lottman

(a) This revised testing procedure should be approximately equivalent to the original procedure for vac-sat + 12 freeze-thaw cycles. Should approximate field mixtures after 3 to 7 years of service.

(b) Lottman emphasized the importance of visually examining the specimens following completion of all physical tests. This is best accomplished by failing specimen in splitting tension and examining the interior of the failed sample. He stated that the SEM can be of help at this point. His recommended splitting tension test utilizes a test temperature of 55°F (13°C) and a deformation rate of 0.65 in./minute (1.65 cm/minute).

(c) The potential for stripping of the binder-aggregate system as measured by this revised procedure approximates the earlier developed procedure. Therefore, the ratios of M_R (after conditioning) ÷ M_R (before conditioning) still apply as previously reported. For example, if a specimen exhibits a $M_{R_A}/M_{R_B} = 0.30$, then this mixture exhibits a high potential for "stripping".

(d) The motivation for the procedure change is to simplify the equipment required to conduct the freeze - thaw cycling as originally proposed.

APPENDIX G

AIR VOIDS

AIR VOIDS

Table G1 is a summary of the air voids of samples prepared during the mix design phase of this study.

Tables G2 through G4 show summaries of the air voids of samples prepared during the testing phase. Three samples of each mixture type were subjected to aging conditioning, three to moisture conditioning, and three to Hveem stabilometer and/or Marshall stability. Air voids were determined for one sample from each of these groups.

Table G5 is a summary of the air voids of the anti-strip samples.

Table G1. Summary of Air Voids for Marshall Compacted Samples, 0/100 SEA Ratio

Mixture Type	Binder Content (% by wt.)													
	4.5	4.7	4.9	5.0	5.2	5.3	5.5	5.6	5.8	5.9	6.0	6.1	6.5	7.0
Crushed gravel, AR-4000	9.8			7.6			6.1				4.4		2.8	2.0
Crushed gravel, AR-2000	8.0			6.9			4.8				3.2		2.0	1.2
Crushed basalt, AR-4000			6.4		4.9		3.3		2.8			2.3		
Crushed basalt, AR-2000		7.0		6.1		4.4		3.4		2.9				

Table G2. Air Voids for Marshall Compacted Samples, Crushed Basalt Aggregate

SEA Ratio / Asphalt Type	0/100	20/80	30/70	40/60
AR-4000	2.0 (A) 3.0 (D) 1.9 (H)	3.6 (H) 4.6 (D) 2.9 (A)	4.0 (H) 4.5 (D) 4.2 (A)	4.0 (H) 3.4 (D) 3.0 (A)
AR-2000	3.0 (A) 3.1 (H) 3.9 (D)	4.9 (D) 3.4 (H) 3.9 (A)	4.7 (D) 3.5 (A) 3.1 (H)	2.8 (H) 4.2 (D) 3.6 (A)

(A): aging conditioning samples
(D): moisture conditioning samples
(H): Hveem stabilometer and/or Marshall stability samples

Table G3. Air Voids for Kneading Compacted Samples, Crushed Gravel Aggregate

SEA Ratio Asphalt Type	0/100	20/80	30/70	40/60
AR-4000	4.1 (A)	3.8 (A)	3.4 (D)	0.7 (A)
	3.3 (H)	2.9 (H)	3.3 (H)	1.8 (D)
	2.7 (D)	2.1 (D)	1.9 (A)	1.6 (H)
AR-2000	5.1 (H)	2.6 (H)	3.0 (D)	1.0 (A)
	3.9 (A)	3.9 (D)	1.5 (A)	1.0 (H)
	4.8 (D)	2.7 (A)	1.4 (H)	2.5 (A)
			2.0 (A)	2.2 (D)

(A): aging conditioning samples
(D): moisture conditioning samples
(H): Hveem stabilometer and/or Marshall stability samples

Table G4. Air Voids for Kneading Compacted Samples, Crushed Basalt Aggregate

SEA Ratio / Asphalt Type	0/100	20/80	30/70	40/60
AR-4000	5.1 (H) 5.7 (A) 5.9 (D)	5.5 (A) 5.8 (H) 6.5 (D)	4.4 (H) 4.1 (D) 3.4 (A)	7.3 (D) 5.8 (H) 4.9 (A)
AR-2000	3.7 (H) 3.2 (A) 2.7 (D)	3.6 (A) 5.6 (D) 4.3 (H)	4.4 (D) 5.9 (H) 5.4 (A)	5.3 (H) 5.2 (A) 5.0 (D)

(A): aging conditioning samples
(D): moisture conditioning samples
(H): Hveem stabilometer and/or Marshall stability samples

Table G5. Air Voids for Anti-strip Samples, Kneading Compaction, Crushed Basalt Aggregate

SEA Ratio	0/100		30/70		40/60	
Percent Anti-Strip / Asphalt Type	1/2	1	1/2	1	1/2	1
AR-4000	3.8	3.8	4.5	4.1	4.1	5.4
AR-2000	6.0	2.2	3.8	3.3	4.6	4.3

APPENDIX H

VISCOSITY AND PENETRATION CHARACTERIZATION OF SEA BINDERS

VISCOSITY AND PENETRATION

CHARACTERIZATION OF SEA BINDERS

SAMPLE PREPARATION

Sulfur and the asphalt cement were maintained separately at a temperature of 284°F (140°C). Then they were proportioned in one pint cans to yield the desired SEA binder ratio with each batch weighing 400 grams. Following proportioning, the sulfur and asphalt cement were mixed on a hot plate for 5 minutes using a standard mixer at 1,000 r.p.m. The mixing can was insulated to minimize the temperature drop during the mixing period.

Following mixing, penetration and sliding plate samples were prepared for subsequent testing. An infrared lamp heating unit was used to heat the plates for viscosity testing. The day the specimens were prepared was considered as day 0. The penetration and viscosity samples were aged at room temperature.

Figures H1 and H2 show the change in penetration with time for the two asphalt cement grades used and the different SEA binder ratios. The penetration of the 50/50 SEA binder could not be measured due to excessive stiffness and shrinkage cavities.

Addition of sulfur to asphalt cement initially increases the penetration making the binder softer than the original asphalt. But with time the penetration starts to drop for all SEA binder ratios. The rate and value of this drop depends on the SEA ratio. The more sulfur there is in the binder, the higher the drop rate in penetration. It is also observed that the penetration value levels off for each SEA ratio and is decreased with the increase in sulfur percentage in the binder. The data also indicates that there are not large differences in these leveling off penetration values for SEA binders with the same base asphalt cement.

Figures H1 and H2 conclusively show the influence of age on the SEA binders when in bulk (i.e. penetration tins). In paving mixes, the binder is a thin film covering the aggregates. For this reason, the sliding plate microviscometer was also used to evaluate the influence of age on the SEA binders. Figures H3 and H4 show the influence of age and SEA ratio on the viscosities of the different binders for the two asphalt cement grades used. From the available data, the following can be postulated:

1. Addition of sulfur to asphalt cement reduces the viscosity initially but further addition of sulfur increases the viscosity.

2. The observed trend is that with time the viscosities of the different SEA binders will exceed that of the original asphalt cement.

3. The data for SEA binders with an SEA ratio of 50/50 are questionable due to large amounts of crystalline sulfur on the sliding plates.

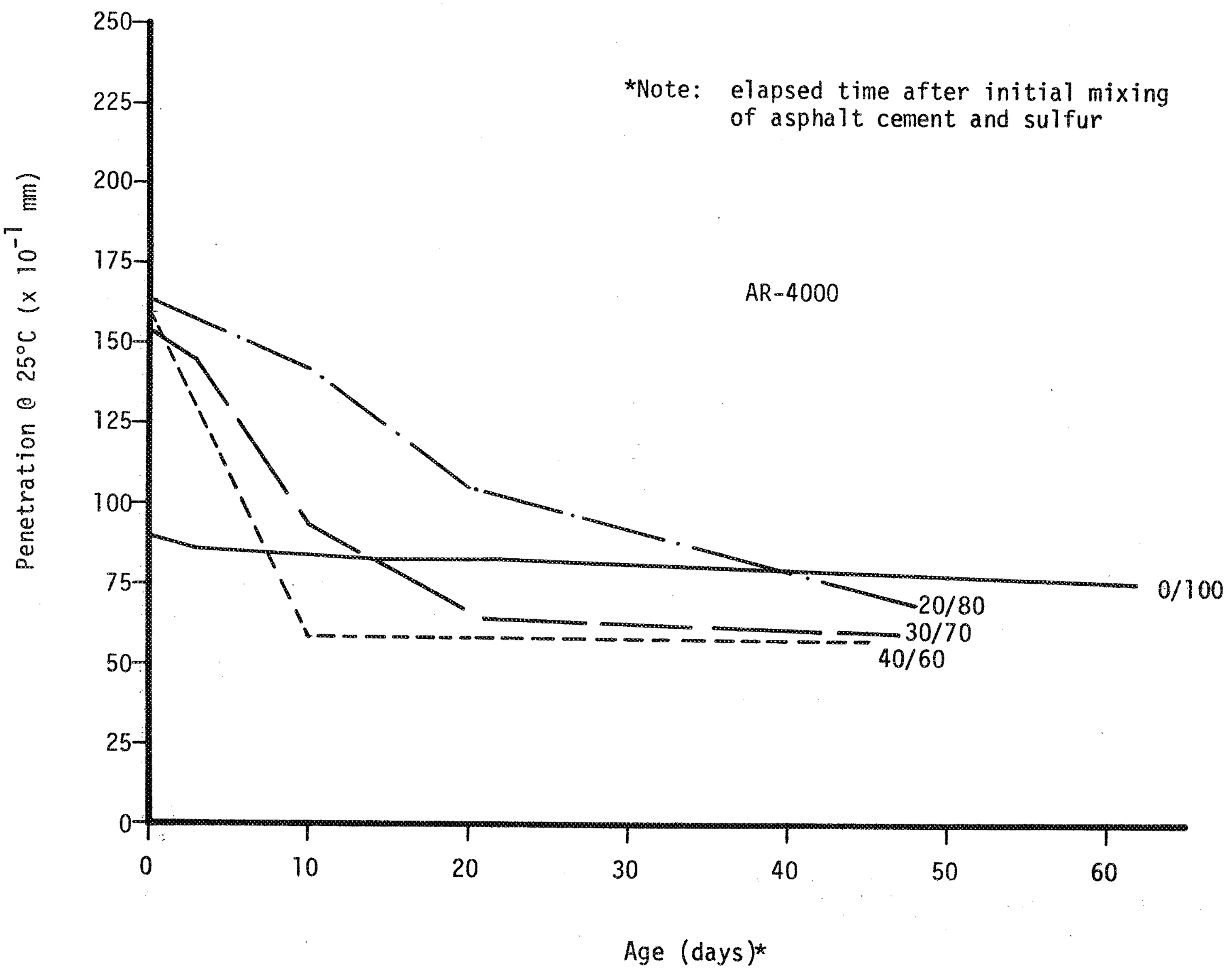

Figure H1. Penetration vs. Age for SEA Binders

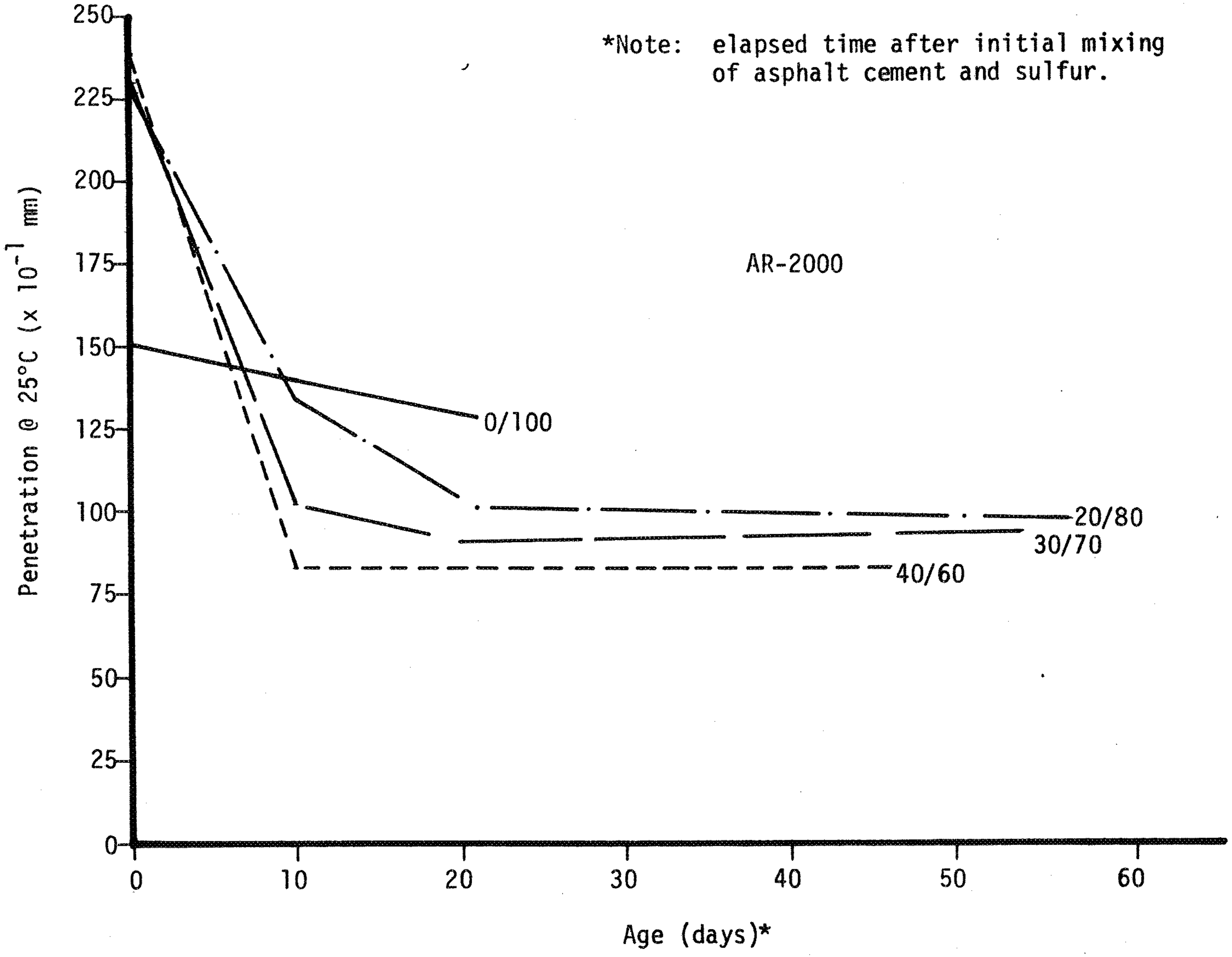

Figure H2. Penetration vs. Age for SEA Binders

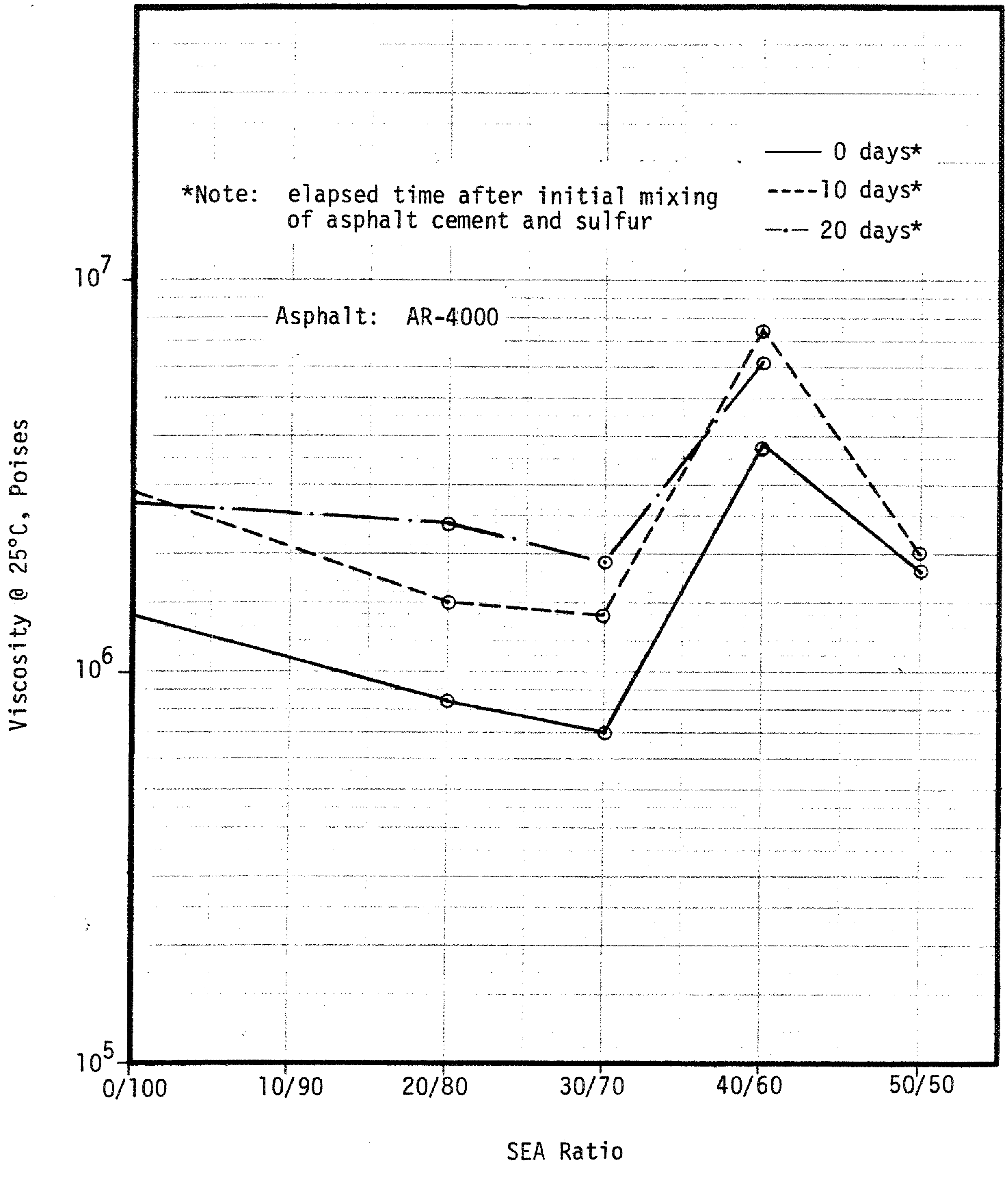

Figure H3. Relationship Between Viscosity and SEA Ratio with Time (AR-4000)

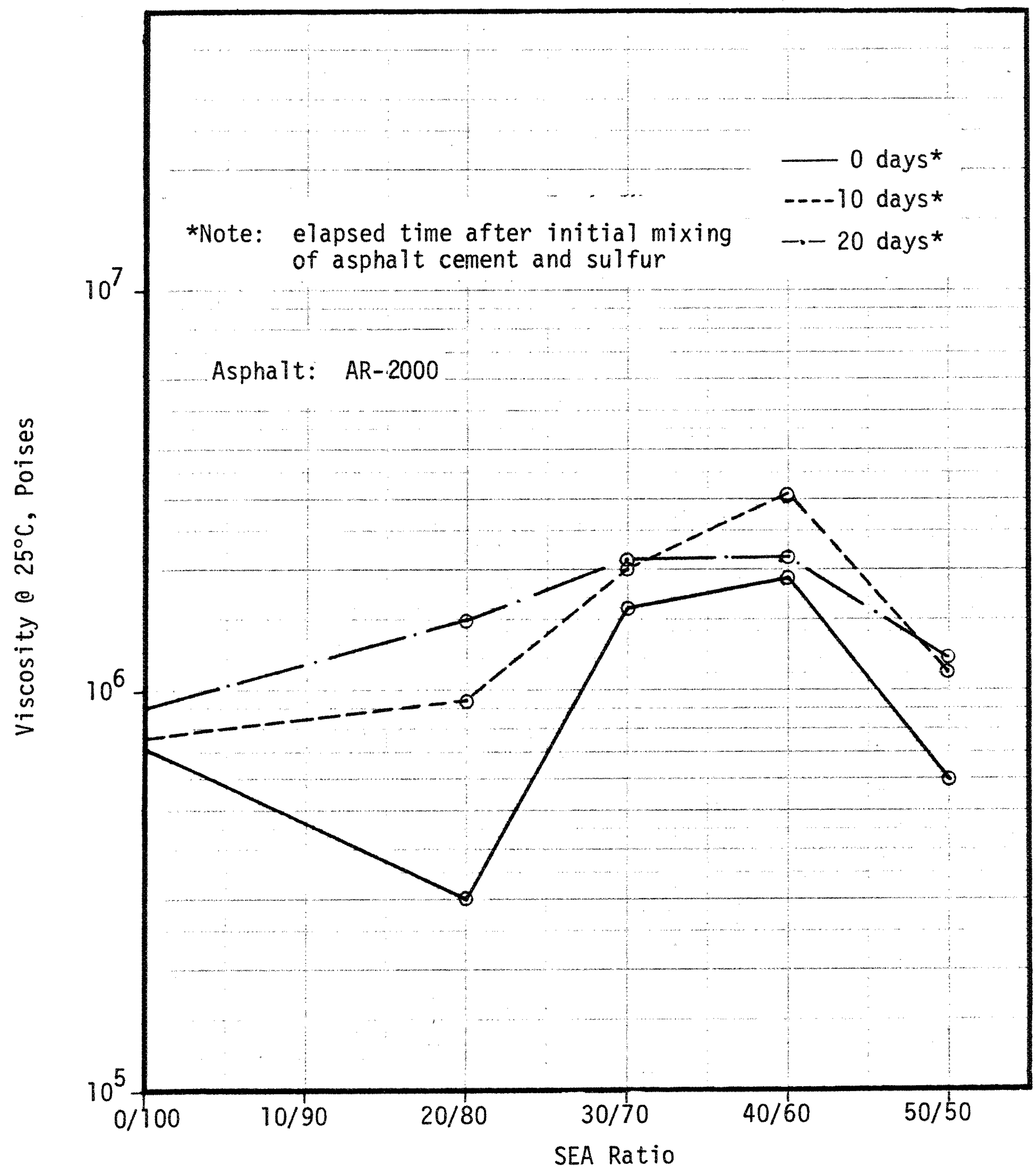

Figure H4. Relationship Between Viscosity and SEA Ratio with Time (AR-2000)