

EVALUATION OF ASPHALT PROPERTIES AND THEIR  
RELATIONSHIP TO PAVEMENT PERFORMANCE

INTERIM REPORT:  
LITERATURE REVIEW AND DEVELOPMENT OF  
COMPOSITION ANALYSIS METHOD

FEBRUARY, 1986

G. Thenoux  
Graduate Research Assistant  
Oregon State University

C.A. Bell  
Assistant Professor of Civil Engineering  
Oregon State University

J.E. Wilson  
Assistant Engineer of Materials  
Oregon State Highway Division

D. Eakin  
Group Leader--Paint and Petroleum Products  
Oregon State Highway Division

and

M. Schroeder  
Paint and Petroleum Group  
Oregon State Highway Division

prepared for

Oregon Department of Transportation

in cooperation with

U.S. Department of Transportation

Federal Highway Administration

1. Report No. FHWA-OR-RD-86-C2		2. Government Accession No. ---		3. Recipient's Catalog No. ---	
4. Title and Subtitle INTERIM REPORT: EVALUATION OF ASPHALT PROPERTIES AND THEIR RELATIONSHIP TO PAVEMENT PERFORMANCE- LITERATURE REVIEW AND DEVELOPMENT OF COMPOSITION ANALYSIS METHOD				5. Report Date February 1986	
				6. Performing Organization Code ---	
7. Author(s) Guillermo Thenoux, et al.				8. Performing Organization Report No. TE-86-4	
9. Performing Organization Name and Address Oregon State University Department of Civil Engineering Corvallis, OR 97331-2302				10. Work Unit No. (TRAIS) ---	
				11. Contract or Grant No. HP&R 084-5165	
12. Sponsoring Agency Name and Address Oregon Department of Transportation Materials and Research Section Salem, OR 97310				13. Type of Report and Period Covered Interim Jan. 1984-Dec. 1985	
				14. Sponsoring Agency Code ---	
15. Supplementary Notes					
16. Abstract  <p>This interim report presents the findings of the literature review and information regarding selection, implementation, and development of a chemical composition procedure for asphalt cements.</p> <p>The chemical composition analysis selected was the Corbett-Swarbrick method, standardized as ASTM D4124. This procedure will be used in a comprehensive study for which eight highway projects throughout Oregon State were selected. This report outlines the program study which involves the study of highway performance, laboratory aging procedures, and methods of asphalt extraction from cores.</p> <p>During implementation of the Corbett-Swarbrick method, a number of refinements were made to improve its efficiency. These are presented in a comprehensive users manual as an appendix of this report.</p>					
17. Key Words Asphalt, pavement life, chemistry			18. Distribution Statement No Restrictions		
19. Security Classif. (of this report)		20. Security Classif. (of this page)		21. No. of Pages	22. Price

#### ACKNOWLEDGEMENTS

The work presented in this report was conducted as part of a Highway Planning and Research (HP&R) Project funded through the U.S. Department of Transportation Federal Highway Administration (FHWA), and Oregon Department of Transportation (ODOT).

The authors are grateful for the support of Glenn Boyle, Bituminous Mix Design Group Leader and other staff in the Materials Section of Oregon State Highway Division. We are also grateful to the Department of Civil Engineering, Oregon State University (OSU) for provision of laboratory facilities. We are indebted to Laurie Campbell and Peggy Offutt of OSU's Engineering Experiment Station for their expert typing and translation of our English.

#### DISCLAIMER

The opinions expressed in this report are those of the authors and not necessarily those of FHWA or ODOT.

## TABLE OF CONTENTS

	<u>Page</u>
1.0 INTRODUCTION.....	1
1.1 Problem Statement.....	1
1.2 Objectives.....	1
1.3 Purpose.....	3
1.4 Organization of the Report.....	4
2.0 EVALUATION OF CHEMICAL COMPOSITION SEPARATION TECHNIQUES.....	5
2.1 Introduction.....	5
2.2 Chemical Composition of Asphalt: State-of-the-Art Summary..	6
2.3 Test Methods for Evaluating Asphalt Chemical Composition: Review.....	10
2.3.1 Introduction.....	10
2.3.2 Corbett-Swarbrick Method.....	11
2.3.3 Rostler and Sternberg Method.....	13
2.3.4 The Clay-Gel Method.....	13
2.3.5 High Pressure Gel Permeation Chromatography - HPGPC..	14
2.3.6 Asphaltene Settling Test.....	16
2.3.7 Heithaus Solubility Parameters.....	16
2.3.8 Other Procedures.....	17
2.4 Chemical Procedure Selected.....	17
3.0 LABORATORY WORK PROGRAM AND DESCRIPTION.....	25
3.1 Phase I.....	25
3.2 Phase II.....	25
4.0 IMPLEMENTATION OF CORBETT-SWARBRICK METHOD AND DISCUSSION.....	31
4.1 Procedure Study and Adoption.....	31
4.2 Modifications and Recommendations to the Test.....	32
4.2.1 Asphalt Sample Preparation.....	32
4.2.2 Asphaltene Fraction Precipitation.....	33
4.2.3 Filtering.....	33
4.2.4 Solvent Evaporation from Asphaltene Fraction.....	33
4.2.5 Column and Alumina.....	34

	<u>Page</u>
4.2.6 Fraction Concentration.....	35
4.2.7 General Problems.....	36
5.0 CONCLUSIONS.....	41
6.0 RECOMMENDATIONS.....	42
7.0 REFERENCES.....	43

APPENDICES

- A TEST INSTRUCTION MANUAL FOR SEPARATION OF ASPHALT INTO FOUR FRACTIONS BY THE CORBETT-SWARBRICK PROCEDURE, ASTM D4124-82, METHOD A (1000 mm COLUMN)
- B EQUIPMENT, MATERIALS, CHEMICALS, AND PRICE LIST FOR ASTM D4124-82, METHOD A
- C TEST INSTRUCTION MANUAL FOR SEPARATION OF ASPHALT INTO FOUR FRACTIONS BY THE CORBETT-SWARBRICK PROCEDURE, ASTM D4124-82 METHOD B (500 mm COLUMN)
- D EQUIPMENT, MATERIALS, CHEMICALS, AND PRICE LIST FOR ASTM D4124-82, METHOD B
- E TEST METHODS AND BIBLIOGRAPHY OF LITERATURE DESCRIBING STUDIES OF ASPHALT COMPOSITION OR ASPHALT CHEMISTRY
- F COMPUTER INVENTORY OF ASPHALT PROPERTIES

## 1.0 INTRODUCTION

### 1.1 Problem Statement

Asphaltic materials used in road construction are specified and controlled by their physical properties. Various procedures have been employed to describe asphalt chemical composition and a number of researchers have attempted to relate composition to asphalt flow behavior (physical properties) and performance of asphalt paving materials.

### 1.2 Objectives

The overall objectives of this research are:

- a) Review existing chemical procedures that are being used presently by researchers and/or highway agencies in the USA.
- b) Select one (or two) chemical procedures so that the following desirable requisites can be met:
  - Potential to relate chemical composition to pavement performance,
  - Ability to identify those fractions that are currently accepted by the asphalt chemical industry,
  - Nondestructive procedure, so that recovered fractions could be used for further analysis,
  - Appropriate for routine tests, and
  - Relatively inexpensive.
- c) Perform a series of experimental tests with the selected chemical procedure(s) so that any improvements or modifications are made before the application of the method in a programmed research.

- d) Along with the Oregon State Highway Division (OSHD), use the selected method in a comprehensive research study. The research initially involves the selection of eight projects ranging from five to twelve years old. Original and recovered asphalt will be tested for physical and chemical properties.

The chemical composition of asphalt, as obtained by the selected procedure, will be analyzed for potential correlation with the performance of the eight projects selected. Results will also be used to assess the current specification (physical) tests.

- e) The chemical procedure used to analyze the eight proposed projects will also serve to complement two parallel studies.

- The first study involves the use of two different methods for artificially aging asphalt. Three projects are going to be selected for this study. The chemical procedure chosen will serve to compare the chemical changes occurring during natural aging as compared to chemical changes due to artificial aging. This could be used in further recommendations for appropriate laboratory aging procedures.
- The second study relates to the extraction procedures used to separate asphalt from a mixture. The Rotovapor technique, which has recently been reported as a desirable recovery method (45), will be investigated and compared to the current Oregon State Highway Division procedure (Abson

recovery method). Each procedure could use high or low temperatures for recovery. Thus, the actual study involves the assessment of four different approaches for the extraction of asphalt from core samples.

- f) Develop a convenient computer-based method for recording, retrieving, and comparing the results of specifications compliance test and chemical composition test, and to evaluate the resulting properties.

Throughout the entire project, the results of all regular physical and chemical tests for asphalts will be monitored and processed with the computer software. During the development of the computer software, the inventory of properties will be updated to include all currently available asphalt properties, and a procedure for convenient processing of data will be established. The information related to the computer software is going to be given entirely in Appendix F.

### 1.3 Purpose

The purpose of this interim report is to present the findings of the literature review, and information regarding development and implementation of the procedure used in this study to determine the chemical composition of asphalt cements. Preliminary data resulting from the use of selected procedures will be included. The development of database of asphalt properties is also included.

The final report for this study will concentrate on presentation and evaluation of data collected from this study.



#### 1.4 Organization of the Report

The work of the present research is presented in the following six chapters and six appendices. The six appendices represent independent and self-explanatory documents that can be used separately from the main body of the report.

Chapter 2, the literature review, concentrates on two main aspects of asphalt chemistry. These are the state-of-the-art and evaluation of the most common chemical procedures used today. Chapter 2 is supplemented with the information given in Appendix E.

Chapters 3 and 4 are related to the laboratory work program of the research and its implementation. Chapter 4 is accompanied by Appendices A, B, C, and D which correspond to the instructional procedure of the methods selected for chemical analysis.

Chapters 5 and 6 give conclusions and recommendations, respectively, of the work accomplished before December 1985.

Appendix F corresponds to the computer software and inventory of asphalt properties.

## 2.0 EVALUATION OF CHEMICAL COMPOSITION SEPARATION TECHNIQUES

### 2.1 Introduction

Asphaltic materials used in road construction are generally specified and controlled by their physical properties. The majority of highway agencies utilize asphalt specifications similar to those standardized by the American Association of State Highway Officials (AASHTO M-26) and the American Society for Testing Materials (ASTM D-3381/D-946).

Asphalt may come from straight reduction or some other refinery process, and is often obtained from a blend of different crudes. Compliance with specifications based primarily on physical properties is not a guarantee for having an identical product. Differences in the quality of asphalts from different sources (different chemical composition) and relationships between composition and performance properties have long been recognized, as evidenced by the many publications on the subject, a few of which are cited (1-13).

The most desirable property of an asphalt, assuming it meets initial performance requirements, is its resistance to change during in-service use (durability). Durability may be assessed by changes in physical properties of the asphalt, which in turn, are related directly to chemical composition. Changes in physical properties are evidenced by changes in the flow behavior (rheological properties), such as viscosity or penetration.

The hardening phenomenon is common for all types of asphalt, regardless of their initial physical properties. Nevertheless, different asphalts from different sources do differ in their hardening susceptibility, in fact, asphalts meeting the same specifications often produce pavements with widely differing performance and serviceability (4,9). This is attributable to the specifications allowing a range of physical properties, but primarily to

asphalt chemical composition. However, it should also be noted that construction and climatic variables often have a greater influence on pavement performance.

Physical and chemical properties of asphalts influence most of the properties of mixtures of asphalt and aggregate. In particular, the following mixture properties are affected by such asphalt property changes:

- a) Temperature susceptibility,
- b) Workability (and therefore, compaction),
- c) Stripping of asphalt from the aggregate,
- d) Deformation and cracking susceptibility, and
- e) Long- and short-term durability due to asphalt aging.

From the above discussion, it can be inferred that an understanding of asphalt chemical composition is fundamental to understand the factors that control asphalt physical properties during storage, mixing, transport, or in-service use.

This report is intended to review the state-of-the-art of asphalt chemistry as related to the physical properties of asphalt, as well as to outline the different fractionation techniques available to characterize asphalt materials.

## 2.2 Chemical Composition of Asphalt: State-of-the-Art Summary

Asphalt is not composed of a single chemical species, but rather a complex mixture of organic molecules that vary widely in structure, composition, reactivity, and affinity.

Elemental analysis of several representative petroleum asphalts are shown in Table 2.1. Although asphalt molecules are composed predominantly of carbon and hydrogen, most molecules contain one or more of the so-called heteroatoms,

nitrogen, sulfur, and oxygen, together with trace amounts of metals, principally vanadium and nickel. As seen in the table, the heteroatoms, although a minor component compared to the percent of carbon and hydrogen molecules, can vary in concentration over a wide range, depending on the source of the asphalt. Because the heteroatoms often impart functionality and polarity to the molecules, their presence may make a disproportionately large contribution to the differences in physical properties among asphalt from different sources.

Elemental analyses reveal little information about how the atoms are incorporated in the molecules or what molecular structures are present. Molecular type and structure information are necessary for a fundamental understanding of how composition affects physical properties. However, because the number of molecules in asphalts with different chemical structures and reactivities is extremely large, chemists have not seriously considered attempts to separate and identify all the different molecules in asphalt.

Early researchers (14,15) represented asphalt cement as a colloidal dispersion of high molecular weight hydrocarbons called "asphaltenes" in a dispersion medium called "maltenes" or "petrolenes." The latter two terms are used interchangeably by many authors, but, as Richardson (16) and Rostler (17) suggested, they should be used to distinguish between high and low boiling fractions of asphaltic bitumen, respectively.

The asphaltene fraction is the most complex component and generally contains those molecules with the highest polarity and tendency to interact and associate. This fraction is insoluble (or precipitated) when the asphalt is dissolved in a nonpolar solvent such as pentane, hexane, or heptane. The part of the asphalt that is dissolved and remains in solution is called "maltenes" (or "petrolenes").

The maltenes contain both the oils and resins. The resins are said to be the agents that disperse the asphaltenes throughout the oils to provide a homogeneous liquid. Each of the three fractions (asphaltenes, oils, and resins) are composed of many different chemical compounds, and the "mix" in each fraction will vary greatly with the source of the petroleum from which the asphalt has been extracted and with the method by which it has been obtained.

The relative amounts and characteristics of asphaltenes, oils, and resins in a given asphalt have an important effect on its physical properties. It is known that asphalts exhibit properties that deviate from those of a true solution and thus, may be considered a colloidal system. The asphaltene fraction is considered to be insoluble in the oil fraction, however, they exist in asphalt as a fine or a coarse dispersion due to the presence of the resins fraction. The ability or lack of ability of the resinous components to keep these highly associated asphalt components dispersed in the oil phase is referred to as their "peptizing" ability, and this largely determines the "gel" or non-Newtonian flow characteristics of the asphalt (14,15).

The generally accepted subdivision of asphalt into a relatively sharp separation of asphaltene and maltenes (petrolenes) is the base for many fractionation procedures. On the basis of the literature (18-23), it can be concluded that asphaltenes are not a chemically unique fraction of asphalt cement, but are dependent on the chemical composition of asphalt and the types of solvent used to precipitate asphaltenes (24). It can also be concluded that the asphaltene fraction, if it exists as such in asphalt cement, is a dispersed phase with varying degrees of association. Further, the degree of association depends on the polarity of the asphaltenes and the power of the maltene fraction to dissolve the asphaltenes (10,25,26,27).

Although the asphaltene-maltene model was useful to explain the flow properties of an asphalt cement from a single source (viscosity is inversely related to asphaltene content) it has been found that not all available methods for compositional analysis are alone, adequate in terms of explaining the flow characteristics of all asphalts from different sources (28-29).

Peterson (30) has taken a new approach in which the concept of molecular interaction is used to explain the relationship between chemical composition and flow properties. The principles of Peterson's approach could be summarized as follows:

The wide spectrum of molecular types in asphalt, all interacting together, gives asphalt its unique properties and makes it appear as homogeneous material. However, on a molecular level, asphalt is undoubtedly heterogeneous with a delicate balance existing among strongly associated or agglomerated components and dispersing or solubilizing components. Because the physical properties of asphalt are controlled by the interaction of the molecules from which it is composed, an understanding of these interactions should provide the basis for understanding its physical behavior, and thus, its durability.

The molecular interaction approach identifies and characterizes the various types of chemical and structural features of the asphalt molecules. The model defines those molecules of different composition but having similar features as having the same "functionality" and that they will produce similar effects on physical properties of asphalt.

Many asphalt molecules have diverse types of chemical functionality, which frustrate chemical fractionation procedures since the whole molecules must be moved into a given fraction.

Because of the molecular interaction approach, most of the available fractionation techniques should be used in conjunction with some other method, in which compatibility and, if possible, the molecular structure could be assessed.

The relation of asphalt chemistry to physical properties has been reviewed recently by Peterson (30) and Halstead (31). Peterson attempts to explain the relationship between the chemical composition and flow properties using a new approach of molecular interaction. Halstead presents a synthesis of information concerning the relation of chemical composition to asphalt properties and performance as a highway material.

The importance of the two papers cited above can be explained as follows. A few years ago (1981-1982) there was still wide disagreement among prominent researchers in the asphalt industry as to how to study and explain asphalt chemistry behavior. Peterson and Halstead have managed, to a certain extent, to bring most of the ideas together and have explained the cause of some disagreement among researchers.

## 2.3 Test Methods for Evaluating Asphalt Chemical Composition: Review

### 2.3.1 Introduction

The potential role of chemistry for evaluating asphaltic paving materials is identified in the following areas:

- a) Evaluation of fresh asphaltic materials.
- b) Evaluation of aged asphaltic materials.
- c) Supplementing physical properties.

- d) Evaluation of pavement performance.
- e) Material specifications.
- f) Design considerations (blending for particular consistency).

So far, no available method for chemical characterization has been widely accepted to be the perfect tool in achieving all the above desirable requirements.

Varieties of procedures have been employed in attempts to describe asphalt composition so that the flow behavior and performance of asphalt paving materials can be predicted. While it is not possible to review all of them, the following section is intended to describe those techniques most commonly used.

#### Chemical Composition Techniques

##### 2.3.2 Corbett-Swarbrick Method (10)

Currently accepted as ASTM D4124, this is essentially a selective adsorption-desorption column chromatography technique, as shown in Figure 2.1.

The asphaltenes are first separated based on their insolubility in a nonpolar paraffinic solvent (n-heptane). This removes the most polar and least soluble asphalt components so that further separation is possible of the remaining fraction known as petrolene (maltenes). The remaining petrolene fraction is then adsorbed on a chromatographic column (alumina is used as the adsorbent phase) and sequentially desorbed with solvents of increasing polarity. Three fractions are obtained from the petrolenes: saturates, naphthene aromatics, and polar aromatics.

This method is being implemented and routinely used at the present time by the authors for the research program.



From the literature review and the present experience of the authors, several important aspects of the test may be summarized:

- a) The fractionation scheme separates asphalt into less complex and more homogeneous fractions. The "generic fractions" obtained are in themselves still complex mixtures of molecular groups and not well-defined chemical species.
- b) The asphaltene fraction in this method differs from all the asphaltene fractions given by most other methods because the precipitating solvent is changed from n-pentane to n-heptane.
- c) The method itself has been used in several research projects (10,32,33) presenting one important advantage, that is, the method is considered to be "nondestructive" and further separations or analysis can be done over the remaining fractions. Thus, the method is open to further improvements or refinements, so it may be adaptable to some of the future techniques not yet available.
- d) The method is lengthy. This is also true for most of the chemical compositional analyses available for asphalt materials. A short version of the method has been presented to ASTM committee recently but it has not yet been published. Basically, the size of the column has been reduced and the filtering process simplified.
- e) The method as described by the current ASTM D4142 is considered to be insufficiently explained. This can lead to poor reproducibility of the results from one laboratory to another.

Both the original version and the short version of the test were implemented by the authors. A detailed procedure for each version was prepared. The implementation of the Corbett procedure is described in Chapter 4.

### 2.3.3 Rostler and Sternberg Method (34)

ASTM method D2006 discontinued in 1976. This technique is illustrated in Figure 2.2. The procedure entails the removal of asphaltenes with reagent grade n-pentane. After separation of the asphaltenes, the remaining components are sequentially separated into fractions based on their reactivity with sulfuric acid of increasing acid strengths (decreasing degree of hydration).

Although the Rostler fraction scheme has been used by many materials laboratories, and correlations with field performance attempted (39,40), it has generally not been accepted as an accurate predictor of field performance. Nevertheless, the method is still used at the present time mainly because the data available on chemical composition of past experience was obtained by using this standard procedure.

The Rostler-Sternberg method has been criticized because of extraneous acid conditions imparted to the maltene fractions. This is a destructive step and the method does not enable the recovery of the altered fractions for further analysis.

Another criticism to the test is related to the hazardous working environment.

### 2.3.4 The Clay-Gel Method (ASTM D2007)

This procedure was developed to replace the Rostler-Sternberg method. This technique is illustrated in Figure 2.3. Asphaltene is first separated by

using n-pentane. The remaining fraction is then added to a pair of columns which are assembled in series, the upper column being packed with attapulgus clay and the lower column with activated silica gel plus a layer of clay on top.

The sample is allowed to percolate using n-pentane as the eluting solvent. The saturated fraction is collected first and the columns are separated. The lower silica gel column is stripped with a polar solvent to yield the aromatic fraction and the upper clay column is stripped with a solvent of increased polarity to yield the polar fraction.

Results from the clay-gel technique of separation have been used for characterization of both virgin and aged asphalts and recycling agents (35). The method is used extensively at the New Mexico Engineering Research Institute at the present time (35,36,37), basically for the same purpose (recycling studies).

This method is claimed to be simple, relatively inexpensive, free of hazardous concentrated acid, and yields repeatable results. Also, the data obtained from this procedure is almost identical to that from the Rostler-Sternberg analysis (36).

The clay-gel compositional analysis as described by the present ASTM may be considered a relatively new procedure and although there are a number of researchers that have made use of it, the method is still being subjected to minor improvements by the original developers.

#### 2.3.5 High Pressure Gel Permeation Chromatography - HPGPC (ASTM D3593)

This technique separates asphalts according to molecular size. Jennings (38) has done a great deal of work using HPGPC on asphalts. A typical chromatogram from Jennings' work is shown in Figure 2.4. The elution time is along the abscissa and relative amounts along the ordinate.

The small peak at time zero is the internal standard. Large molecular size (LMS) is eluted first, then medium molecular size (MMS), and, finally, the small molecular size (SMS). Five criteria are examined when comparing asphalts:

- a) Elution time of the largest molecules;
- b) Height of the curve in LMS region denotes which asphalt has a higher LMS content;
- c) Elution time of the curve maximum denotes the asphalt which has a higher molecular size fraction by the relative time to occurrence;
- d) Height of curve maximum relative to height of LMS and SMS regions denote which asphalt has higher proportion of LMS and SMS components relative to MMS; and
- e) Height of the curve in SMS region denotes which asphalt has more SMS components.

Jennings (38) has determined that HPGPC is useful for a variety of purposes listed below.

- a) HPGPC and asphaltene analysis has been shown to define characteristics of an asphalt which is satisfactory with respect to durability.
- b) HPGPC is useful in the characterization of asphalts from different refineries.
- c) HPGPC has shown that the LMS region of the chromatogram is not only composed of asphaltenes but also other constituents and that asphaltene percentages alone do not provide enough data by which to judge asphalt quality and performance.

- d) Batch or drum dryer plant processing cause a 20 to 80 percent increase in LMS material as shown by HPGPC.

#### 2.3.6. Asphaltene Settling Test

This test, described by Plancher (39), consisted of dissolving 2 gm of asphalt in 50 ml of n-hexane by stirring 24 hours at 20°C. The asphalt/hexane solution is transferred to a 50-ml graduated cylinder. The asphalt settling time (T) is defined as the time, in minutes, required for the asphaltene meniscus to descend to the 25-ml level. A typical asphaltene settling curve is shown in Figure 2.5.

The Laramie Energy Technology Center, where the test was developed, has suggested that the test will be useful as a means to rapidly evaluate asphalt durability and to determine the compatibility of asphalt with softening agents which have been proposed for use in recycled asphalt mixtures.

The test was critically evaluated by Kennedy, et al., (40) who have concluded that test precision is a problem. Further, Kennedy, et al., suggested several modifications to the test.

#### 2.3.7 Heithaus Solubility Parameters (41)

In the Heithaus procedure asphalt cement is first dissolved in toluene at various concentrations. The asphalt-toluene solutions are then titrated with heptane until the asphaltenes precipitate from the solution. The end point is determined as in the Oliensus spot test (42) and by microscopic examination. The flocculation ratio is defined as the ratio of toluene to toluene plus heptane at flocculation. The deflection ratio is defined as the ratio of the volume of toluene plus heptane to the weight of the asphalts at flocculation. These ratios are then used to define the degree of peptization of the

asphalt or "state of peptization" which is an attempt to measure the inter-compatibility of the components in asphalt -- how good a dispersant are the maltenes for the asphaltenes -- and how readily are the asphaltenes dispersed.

#### 2.3.8 Other Procedures

J.L. Goodrich (43) has prepared a review of test methods and a bibliography of literature describing studies of asphalt composition or asphalt chemistry. This review is presented in Appendix E in its original format. Appendix E constitutes a separate document of the present interim report and contains its own list of references.

#### 2.4 Chemical Procedure Selected

Based on the preliminary literature review, economic consideration, and from the experience of the Oregon State Highway Division (OSHD), the Corbett-Swarbrick procedure (ASTM D4124) was initially chosen for the experimental study.

The Rostler procedure (formerly ASTM D2006) has also been considered to be used in the present research. The equipment and glassware are available at Oregon State Highway Division (OSHD). Results from the Rostler analysis are going to be used as supplementary information. Due to the fact that ASTM D2006 has been discontinued, this procedure was not selected for definitive adoption.

The Clay-Gel Method (ASTM D2007), being essentially an adsorption chromatography, the same as the Corbett-Swarbrick procedure, was considered as an alternative choice for ASTM D4142.

The High Pressure Gel Permeation Chromatography (HP-GPC) technique, although an ASTM standard (ASTM D3593), is considered to have limited appli-

cations. The asphalt fractionation on "large," "medium," and "small" molecular size is still controversial and the final proportion of the three molecular sizes may be affected by many factors not controlled by the present standard procedure (43).

The Asphaltene Settling Test, according to Kennedy and Lin (40), needs additional study if the test method is to have practical value. Kennedy and Lin (40), who did extensive research to evaluate the test, concluded that precision was the main problem of the test and suggested several modifications to it.

Heithaus Solubility Parameters (41) is not a fractionation technique, but the test could be considered in future studies for supplementary information on asphalt chemistry behavior.

Table 2.1. Elemental Analysis of Representative Petroleum Asphalts.\*

Code Source	Mexican Blend	Arkansas-Louisiana	Boscan	California
Carbon, %	83.77	85.78	82.90	86.77
Hydrogen, %	9.91	10.19	10.45	10.93
Nitrogen, %	0.28	0.26	0.78	1.10
Sulfur, %	5.25	3.41	5.43	0.99
Oxygen, %	0.77	0.36	0.29	0.20
Vanadium, ppm	180	7	1380	4
Nickel, ppm	22	0.4	109	6

\*After Reference (30)



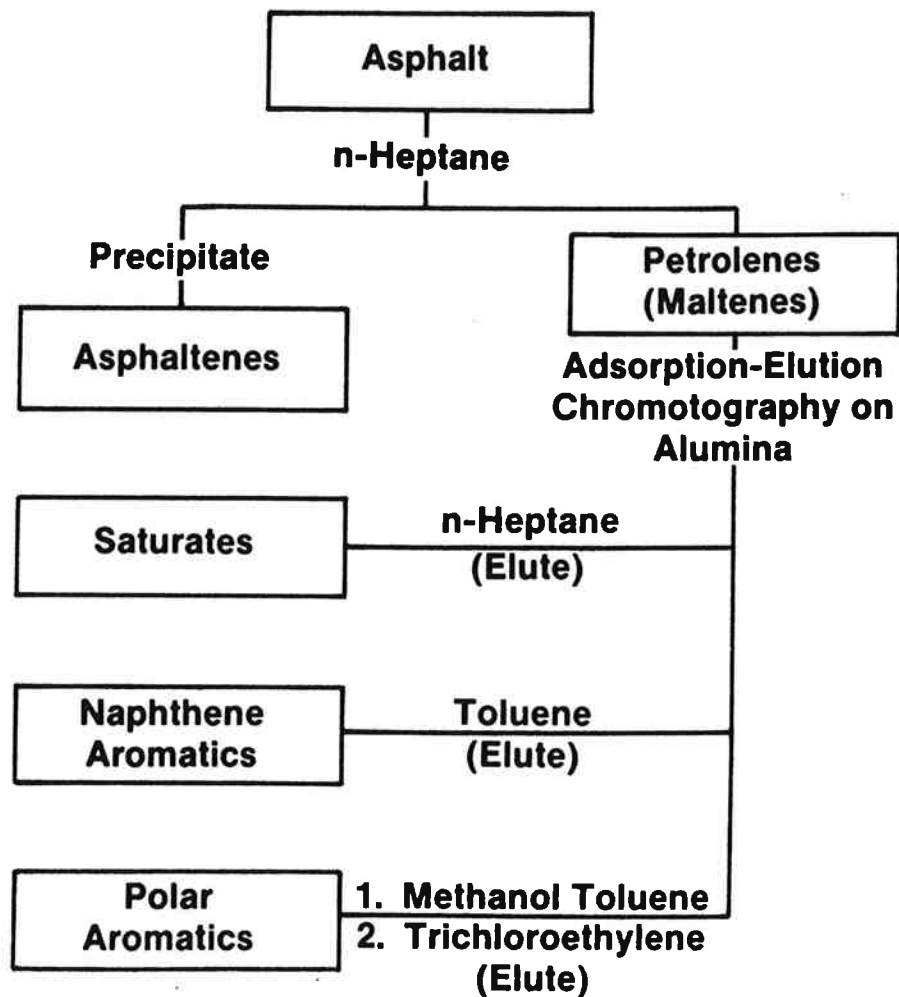


Figure 2.1. : Adsorption/Desorption Chromatography  
 Corbett-Swarbrick  
 ASTM D4124  
 (after reference 41)

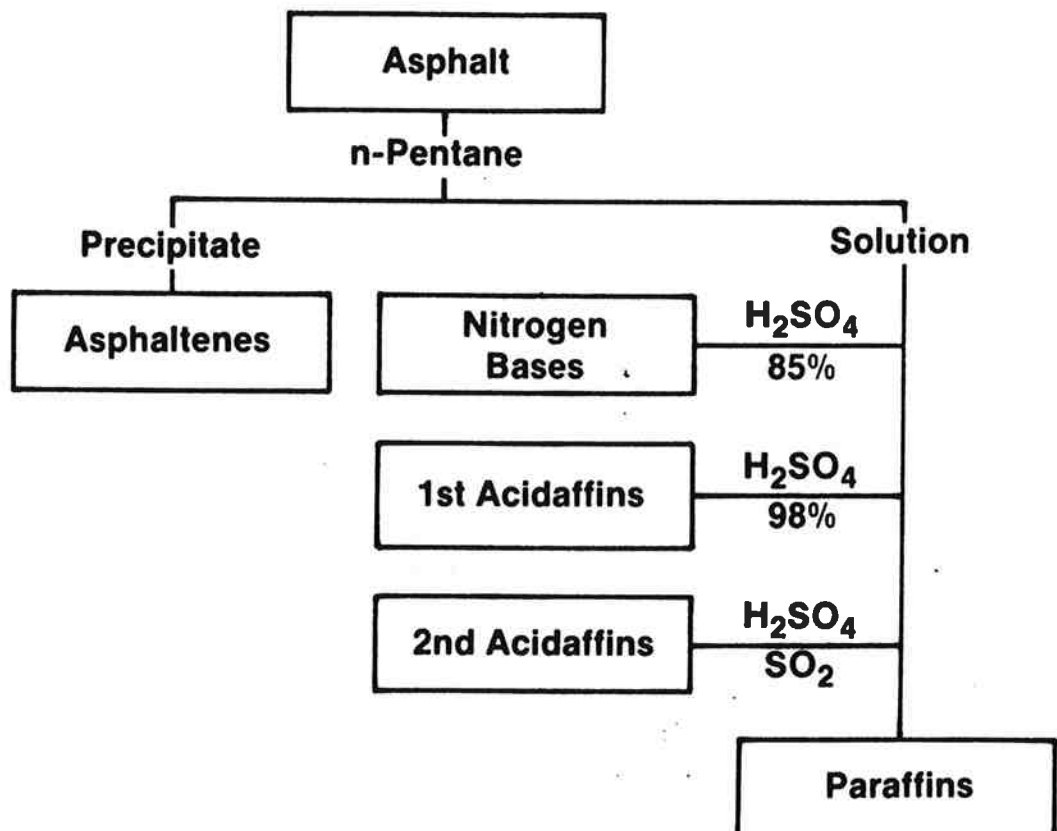


Figure 2.2. : Chemical Precipitation  
 Rostler-Sternberg  
 ASTM D2006  
 (after reference 41)

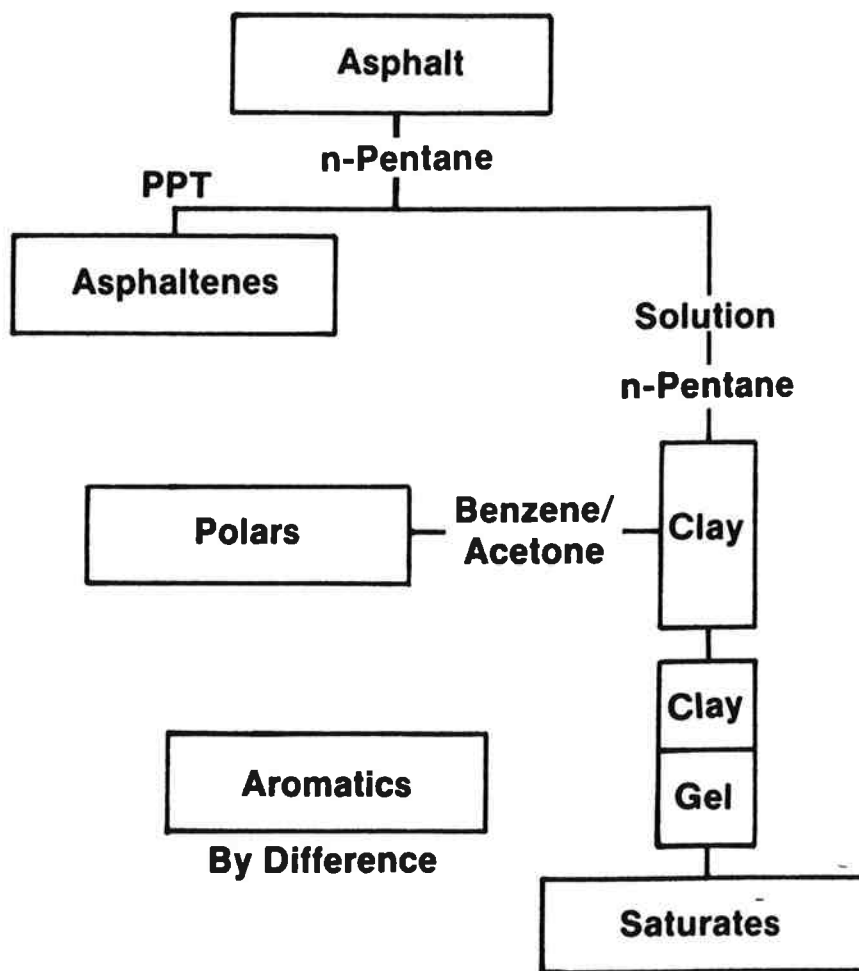


Figure 2.3. : Adsorption/Desorption Chromatography  
 "Clay-Gel"  
 ASTM D2007  
 (after reference 41)

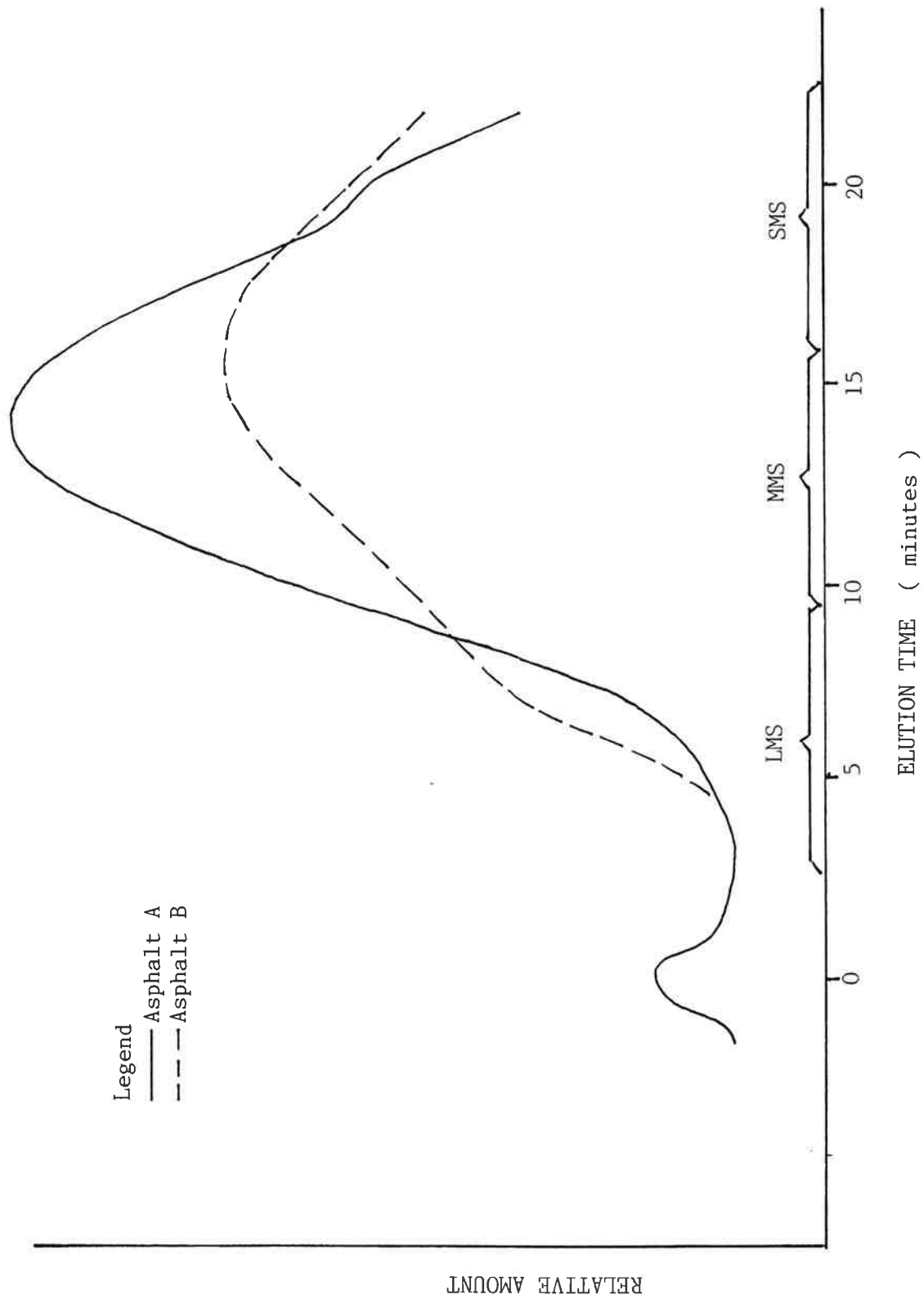


Figure 2.4. : Typical Chromatograms of Whole Asphalts.  
(After reference 38)

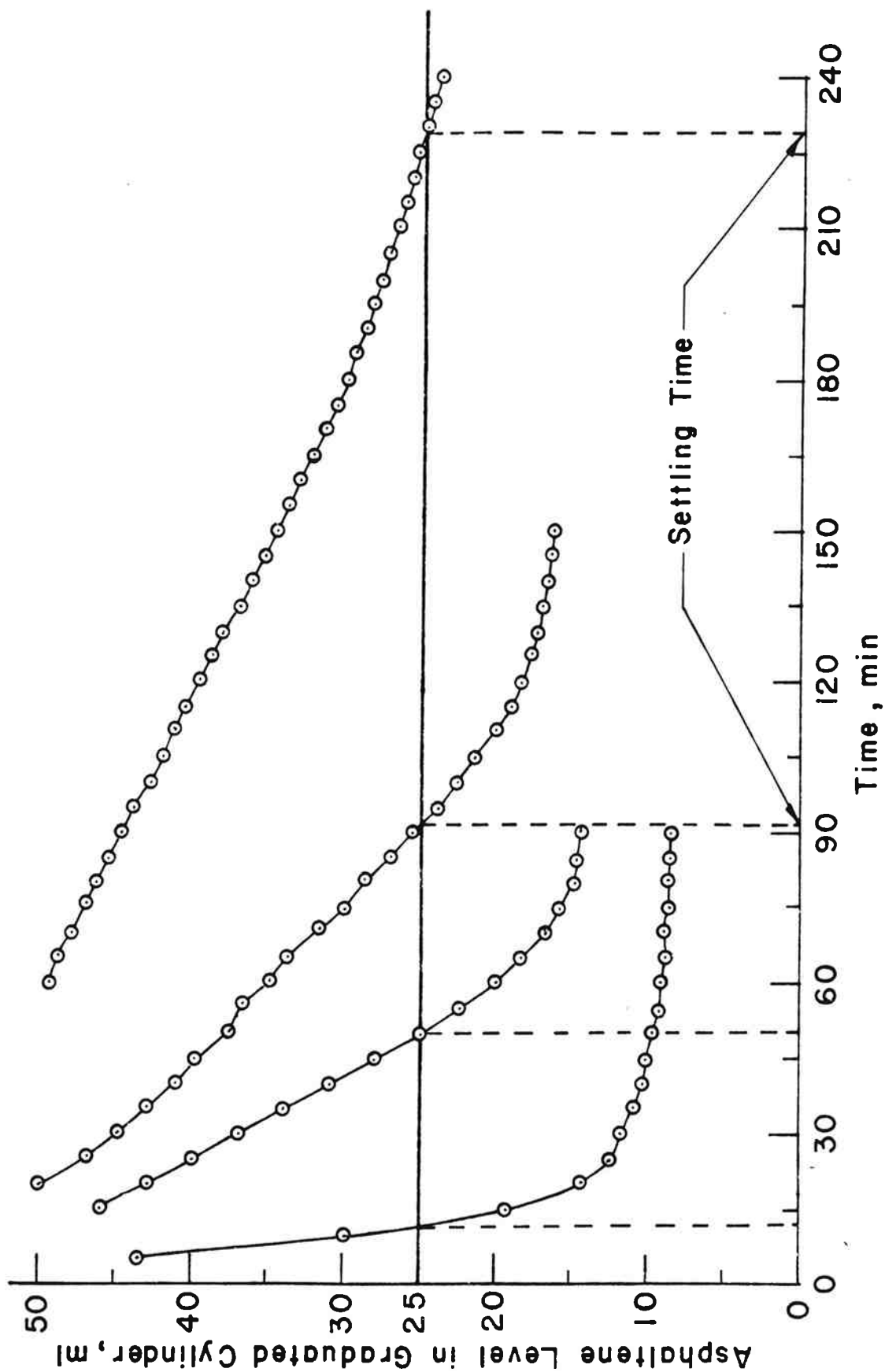


Figure 2.5. : Typical Asphaltene Settling Curves.  
(After reference 40)

### 3.0 LABORATORY WORK PROGRAM AND DESCRIPTION

The laboratory work was divided into two distinctive parts, namely:

Phase I: Implementation of the chemical procedure selected.

Objectives b) and c).

Phase II: Investigation of asphalt performance, aging laboratory techniques, and asphalt extraction techniques by using data obtained from physical test and the chemical procedure implemented on Phase I, Objectives d) and e).

#### 3.1 Phase I

The various steps needed to implement the procedure selected for chemical analysis are given in Chapter 4.

#### 3.2 Phase II

Various projects (throughout) Oregon have been examined in previous studies (42,43,44,45) for which asphalt and mixture properties are known. These projects, together with five new projects, were selected to represent a range of performance. Project ages range from five to twelve years. Original asphalt samples are available for all eight projects. Description and location of all eight projects being studied are given in Table 3.1 and Figure 3.1, respectively.

Original and recovered asphalt will be tested for physical and chemical properties according to the summary program shown in Figure 3.1.

Core samples were obtained at each site and were cut into two pieces so that the study could distinguish between the section of the pavement exposed to the surface and the one that was protected from detrimental environmental conditions (e.g., sunlight, oxygen, oil, and other contaminating agents).

To investigate asphalt performance, aging procedures, and asphalt extraction techniques, chemical composition analysis will be run in duplicate for each asphalt sample using the Corbett-Swarbrick chromatographic technique method B. Physical properties to be obtained on OSHD and OSU laboratories are:

- a) Penetration at 25°C and 4°C,
- b) Absolute viscosity at 60°C,
- c) Kinematic viscosity at 135°C,
- d) Ductility at 25°C and 7.2°C, and
- e) Fraass test.

The performance study is done on all projects by comparing physical and chemical changes of original samples, extracted asphalt from cores (Abson Recovery, Modified AASHTO Designation T164-70 and T170-55), and artificially aged asphalt (Rolling Thin Film Oven, ASTM D2872).

The aging procedures study involves evaluation of the effects of the Rolling Thin Film Oven Test (RTFO, ASTM D2872) and use of the Pressure Oxygen Bomb (POB) for oxidative aging. The POB has been implemented at Oregon State University by Kim and Bell (49). Three projects were considered for the aging study (Projects 3, 5, and 7 as described in Table 3.1).

The extraction techniques considered for the study were:

- Industrial Asphalt Modified Abson Recovery, OSHD Test Method 314-83 (Modified AASHTO Designation T164-70 and T170-55)
- Vacuum Extractor, OSH Test Method 309-85
- Industrial Asphalt Modified Abson Recovery plus Rotovapor
- Vacuum Extractor plus Rotovapor

The last two methods given above are presently being developed at Oregon State Highway Division.

The same three projects selected for aging study were selected for the extraction procedures study.



Table 3.1. Project Locations.

Project No.	Year	Name	Highway No.	Location
1	1980	•Grande Ronde-Wallace Bridge	St 18	•East Grande Ronde
2	1980	•Pacific Hwy-Dayton Jct-Lafayette	St 18	•Dayton-Lafayette Jct.
3	1974	•Idylwood Street	City Street	•Idylwood-Lane Oak Intersection
4	1976	•Elk River-Port Orford	US 101	•North Port Orford
5	1980	•Plainview Rd-Deschutes River	US 20	•West Bend
6	1981	•Klamath Falls	US 97	•Klamath Falls-Green Spring Jct.
7	1973	•Arnold Ice Caves-Horse Ridge	US 20	•East Bend
8	1974	•S. Fork Malheur	St 78	•East New Princeton

#	CODE	YEAR	NAME
1	9076	1980	GRANDE RONDE
2	9013	1980	DAYTON JCT.
3	CITY	1974	IDLYWOOD ST.
4	8325	1976	ELK RIVER
5	9028	1980	PLAINVIEW
6	9229	1981	N. KLAMATH FALLS
7	7789	1973	ARNOLD ICE CAVES
8	7995	1974	S. FORK MALHEUR

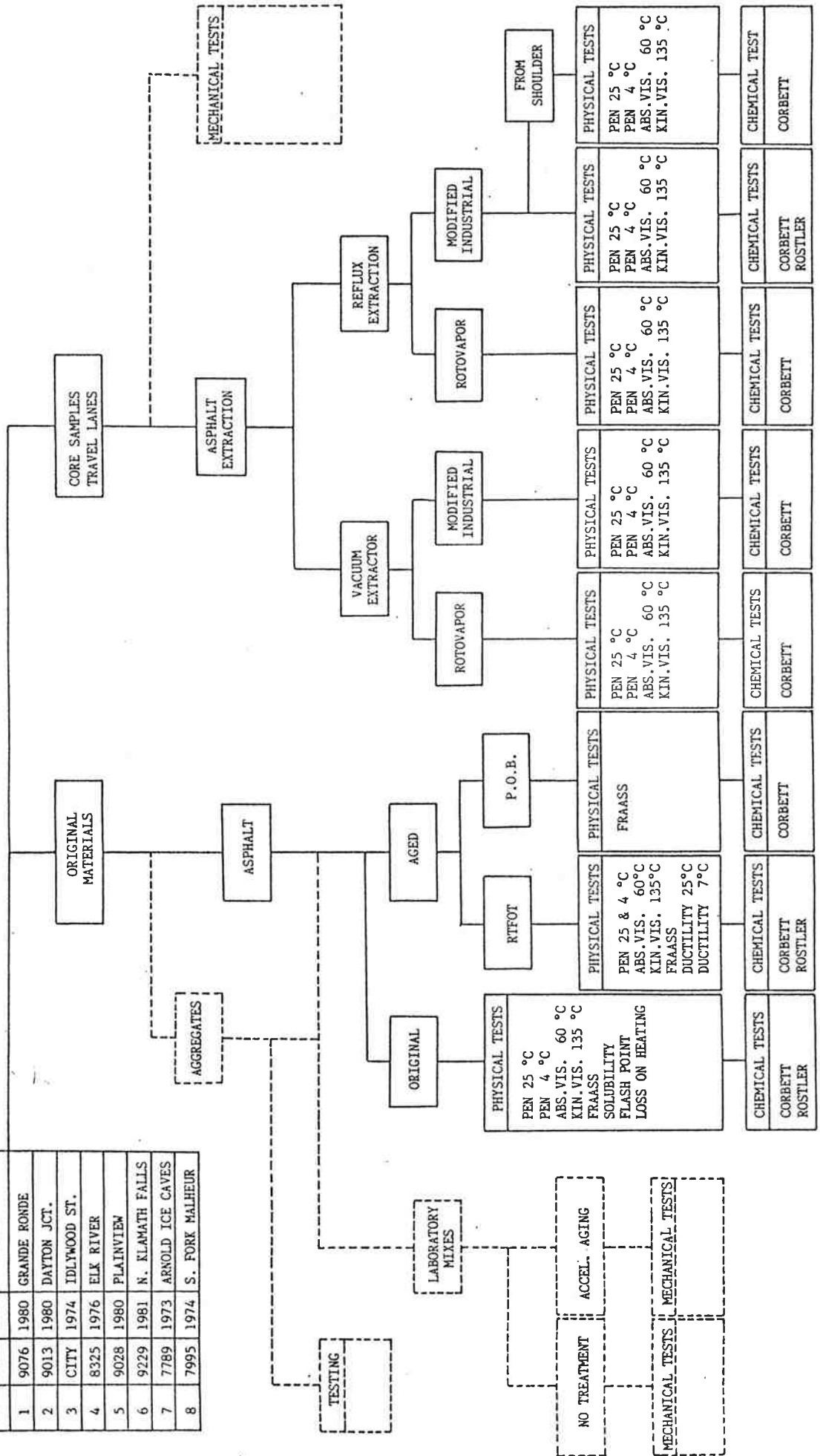


Figure 3.1. Laboratory Work Program

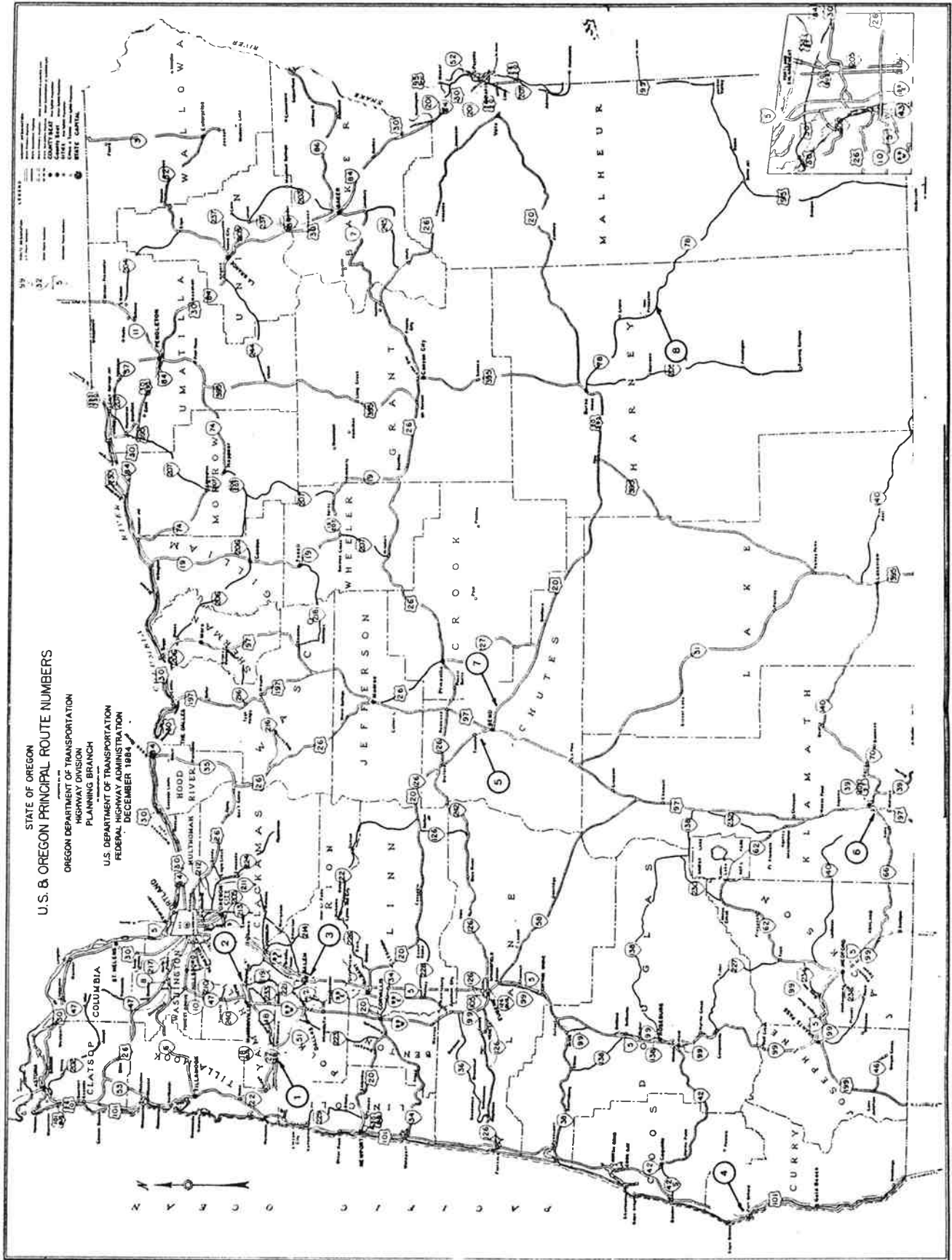


Figure 3.2. Project Locations in Oregon

#### 4.0 IMPLEMENTATION OF CORBETT-SWARBRICK METHOD AND DISCUSSION

##### 4.1 Procedure Study and Adoption

This task includes the various steps needed to implement the procedure selected for the chemical analysis of road asphalt.

Work was started with the ASTM D4124 method in the summer of 1984. A considerable number of tests were done to select the appropriate instrumentation and glassware required and also to select the best procedures for precipitation, filtration, elution, and concentration of the fractions. A detailed report was prepared with the recommended instruction to perform the test. The report also included an inventory of glass wear and instrumentation together with a present-cost analysis. These are given in Appendices A and B, respectively.

Following the initial experience, the test was also set up in the laboratories of the Oregon State Highway Division (OSHD). Both laboratories (OSU and OSHD) were involved in parallel testing work in spring 1985 so that repeatability could be assessed.

With the repetition of many tests, it was found that, even with improvements made during the present research to expedite the procedure, the test was still lengthy (2 days work per test), expensive to run, and relatively hazardous due to the large amounts of solvent handled. Further, following the ASTM procedure in both laboratories (OSHD and OSU) reproducibility of the results was poor.

The standard test ASTM D-4124-82 has been submitted for revision to ASTM Committee D04.47. The revised procedure involves considerable modifications to the existing standard. Although this new procedure (ASTM D4124-Method B) is not yet a standard, it was decided to adopt it instead of the current standard procedure (Method A) originally described in ASTM D4124-82.

Laboratory work was started to replace ASTM D4124, Method A with the new proposed procedure, Method B. Method B could be described as a small-scale version of the current ASTM D-4124.

After provision of the required glassware and instrumentation, a number of experimental tests were performed and a new instructional manual prepared, together with an inventory of glasswear, instrumentation, and material cost per test. These are given in Appendices C and D, respectively.

This new instructional procedure developed at OSU was submitted to the ASTM committee chairman at his request as it included various modifications and improvements to the proposed Method B.

#### 4.2 Modifications and Recommendations to the Test

Several problems arose during the implementation of the procedure. Since Method-B (500 mm column) was the procedure finally adopted, the present section will discuss the problems referring to the small column. Nevertheless, Method A which uses a 1000 mm column is essentially a large-scale test compared with Method B which uses a 500 mm column. Thus, the problems could be considered to be similar in both cases.

##### 4.2.1 Asphalt, Sample Preparation

Heating the asphalt repeatedly or for long periods during preparation causes an increase of the amount of asphaltene fraction (up to 4% or more in some cases).

To achieve the right weight of sample to be tested without excess heating, it is recommended to warm up the asphalt enough so that the sample could be stirred in its container. Following this, the asphalt sample should be poured onto wax paper and left to cool at room temperature (approximately

22°C). After this, the material could be easily cut to the exact weight desired and placed in the stirring flask with no further heating.

#### 4.2.2 Asphaltene Fraction, Precipitation

The standard procedure recommends the warming of the asphalt in the flask before pouring the precipitating solvent (n-heptane). Also, the standard specified that during the stirring process, n-heptane should be kept at a temperature near its boiling point (approximately 90°C). None of the heating application is recommended by the author due to the direct effect that this has on the final asphaltene portion.

The n-heptane (precipitating agent) should not be interchanged with any other solvent (e.g., pentane or hexane). It has been shown (50) that the amount of precipitated asphaltene does vary considerably among the different solvents used in the various standards.

#### 4.2.3 Filtering

The filtration procedure described in the instruction manual prepared at OSU (Appendix C) does not correspond to the one given in the proposed standard. The method given in Appendix C is considered to be cheaper and more rapidly performed. The yield of asphaltene fraction is the same as the one obtained by using the standard procedure.

#### 4.2.4 Solvent Evaporation from Asphaltene Fraction

The standard procedure calls for the use of oven temperatures of 104°C to evaporate the solvent from the recovered asphaltene fraction.

This is not recommended in the present instruction manual. It has been observed that asphaltenes have very unstable weight if heated at higher temperatures (above 60°C) or heated for longer periods. The asphaltene frac-

tion should be allowed to dry at room temperature (20 to 25°C) for 24 hours or at maximum temperature of 60°C for 30 minutes.

#### 4.2.5 Column and Alumina

Special problems were encountered during the present research with the alumina used as the adsorption-desorption material in the chromatographic column.

Alumina is specified in the standard as: "F-20 chromatographic grade calcined at 413°C for 16 hours." The standard suggests that alumina under the above specifications could be obtained directly from the manufacturer. Nevertheless, it was found that the 'quality' of the alumina will vary among manufacturers and within the production lots of one manufacturer.

Table 4.1 shows results of a number of tests performed with one asphalt but with alumina samples of two manufacturers (SIGMA Chemica Co. and MCB Manufacturing Chemists, Inc.). Further, SIGMA alumina samples were obtained from three different lot productions. The results shown in Table 4.1 indicate that the alumina as it is received from the supplier does not comply with the specifications and has different adsorptive capacity.

The explanation to the above problem, which caused considerable delay in the testing program, was that the adsorptive activity of alumina is a function of moisture content, size, and surface area (51,52,53). The size and surface area is controlled basically by the selection of an 80-200 mesh alumina. The moisture content is controlled by calcining the material at 413°C for 16 hours.

Although alumina has been calcined before being packed in sealed bottles, the packing procedure probably has not been carried out under vacuum conditions. Thus, during transportation and storage, the material can adsorb various amounts of water.

The solution to this problem was to retreat the alumina (413°C for 16 hours and store in vacuum dessicator) according to the specifications given in the standard. Unfortunately, the standard was not clear to specify the above treatment as essential. This may have caused other researchers to have the same problem as it was found later by the authors through personal contacts.

The alumina from Sigma Co. and MCB, Inc., was retreated and tested using another asphalt. The results are shown in Table 4.2 and appears that there are no major variations among the different sources of alumina used if they are recalcined before the test.

#### 4.2.6 Fraction Concentration

Two methods have been used in the concentration of the maltene fraction and the three subsequent fractions obtained through the chromatographic column.

One of the methods uses the set-up shown in Appendix C, Figure C.4. the other method being used at OSHD (Oregon State Highway Division) is a rotary evaporator (Rotovapor used as commercial name).

The Rotovapor (which is a standard commercial apparatus) has several advantages when compared with the traditional methods. Some of the advantages are:

- a) Materials are concentrated in shorter time.
- b) Less chance to overheat the concentrate solution.
- c) When the solution is composed of several solvents of different boiling points and specific gravity there is no danger of explosion.

The above advantages need to be explained in more detail. The boiling point and specific gravity of the solvent used in the Corbett procedure are



given in Table 4.3. The solution of the last fraction (Polar-Aromatics) is composed by three solvents (Toluene, Methanol and Trichloroethylene) since the heaviest solvent has a lower boiling point than Toluene this causes the build-up of gases within the solution and subsequent minor or major explosions. The rotational effect obtained with a rotovapor causes continuous mixing of the solution, breaking its surface tension and letting the gases escape uniformly.

Using the traditional concentration method this could be avoided by the use of "boiling stones" or the introduction of air bubbles. The use of boiling stones was discarded because this upsets the net weight of the concentrated fraction. Air bubbles were used but their effect is limited up to a certain concentration of the solution.

In general the advantage of continuous rotation of the solution is that it permits the gases to escape more easily especially when the solution reaches a higher viscosity. This also speeds up the concentration procedure without overheating the mixture.

#### 4.2.7 General Problems

Most of the problems related to the test procedure arise with the interpretation of the standard. Most of these problems are discussed in Appendix C which is the latest version of a complete and detailed procedure to perform the standard column using the 500 mm column.

One problem that arises with the interpretation of the standard is the way in which the "Eluant Feed Solvent Table" is presented (Appendix C, Table C.1)

The table suggests that the cut point between Saturates and N-A is after collecting the 35 mL Toluene eluant, while our experience shows that there is little or no Toluene in the first fraction solution.

For the second and third fraction, the table suggests that the N-A are contained within the Methanol/Toluene eluant. The addition of this last combined eluant immediately creates the black ring where the second cut has to be made. Thus, there is no Methanol in the N-A fraction.

The description of the fractions collected are given in the standard. These include: the saturates--desorbed by n-Heptane and the N-A--desorbed by Toluene. But according to the table in the standard, saturates are also desorbed by Toluene and N-A are also desorbed by Methanol which is not true.

Table 4.1. Comparison of Alumina Obtained from Different Manufacturers and Different Lot Productions.

Fractions	Sigma Chemical Co.			MCB Manufacturing Chemist Inc.
	<u>Lot Number</u>			
	034F-0321	114F-0108	055F-0805	
Asphaltenes	15.21%	15.53%	15.67%	15.08%
Saturates	23.42%	15.84%	10.15%	12.77%
N-Aromatics	45.10%	44.02%	38.21%	40.91%
P-Aromatics	15.61%	22.85%	35.08%	29.91%
TOTAL	99.34%	98.24%	99.11%	98.67%
AVERAGE OF:	6 tests	3 tests	2 tests	1 test

Table 4.2. Comparison of Alumina Obtained from Different Manufacturers Before and After Retreatment.

Fraction	Sigma Chemical Co.		MCB Manufacturing Chemist Inc.	
	No Retreatment	With Retreatment	No Retreatment	With Retreatment
Asphaltenes	13.83%	13.73%	13.47%	13.72%
Saturates	16.76%	11.35%	14.14%	11.26%
N-Aromatics	44.42%	24.65%	36.29%	24.24%
P-Aromatics	23.01%	49.30%	36.05%	49.07%
TOTAL	98.02%	99.03%	99.95%	98.29%
AVERAGE OF:	2 Tests	2 Tests	1 Test	1 Test

Table 4.3. Solvents Used in the Corbett Procedure and Its Physical Properties.

Solvent	Boiling Point (°C)	Specific Gravity
n-Heptane	98.0	.67
Toluene	111.0	.87
Methanol	65.0	.79
Trichloroethylene	87.0	1.46

## 5.0 CONCLUSIONS

Several conclusions may be drawn from the research program based on the data collected up to this point.

- 1) Asphalt chemistry is not well understood. This is because crude oil is derived from living organisms and its composition is complex.
- 2) The application of chemical analysis in some specific areas of asphalt technology (aging, production control, recycling and others) is most valuable for pursuing improvements on asphalt performance.
- 3) There are many chemical techniques used to fractionate asphalt but no single procedure has proved to be satisfactory in correlating asphalt composition to asphalt performance.
- 4) The many chemical procedures available do not correlate with each other and there is no standard terminology to refer to asphalt components.
- 5) The selection of the Corbett-Swarbrick procedure in the present research does not imply that it is the best procedure available. Rather, it was deemed most suitable for this study.

## 6.0 RECOMMENDATIONS

Major recommendations from the present research will be given in the final report after completion of the test program given in Chapter 3.

Recommendations so far are related to the implementation of the Corbett-Swarbrick procedure. These are:

- 1) The introduction of a small column to the Corbett procedure is an important step in simplifying the test.
- 2) Heat should not be used during sample preparation, asphaltene precipitation and solvent evaporation from asphaltene.
- 3) A new method is proposed to speed up the filtering process phase one.
- 4) Alumina must be recalced and vacuum stored before running a test.
- 5) Rotovapor is the best option for fraction concentration.
- 6) Vacuum could be applied when collecting last fraction (Polar-aromatics) from the column to speed up the test.
- 7) The Corbett procedure was preferred due to some particular advantages (given in Chapter 2) that suit the objectives of the present research. The method is considered to be "nondestructive" and further separations or analysis can be done over the remaining fractions. The method is open to further improvements or refinements, so it may be adaptable to some of the future techniques not yet available.
- 8) The standard procedure (ASTM D4124) is difficult to interpret. A more detailed laboratory procedure should be used (as the one given in Appendix C, for example) so that the same mistakes are not made by different operators.

7.0 REFERENCES

1. F.N. Hveem, Quality Tests for Asphalts--A Progress Report. Proceedings, Association of Asphalt Paving Technologists, Vol. 15, 1943, pp. 111-152.
2. W.P. Van Oort, "Durability of Asphalt," Industrial and Engineering Chemistry, Vol. 48, 1956, pp. 1196-1201.
3. P.C. Blokker and H. van Hoorn, "Durability of Bitumen in Theory and Practice," Fifth World Petroleum Congress, Section VI, 1959, pp. 417-432.
4. F.N. Hveem, E. Zube, and J. Skog, "Progress Report on the Zaca-Wigmore Experimental Asphalt Test Project," ASTM Special Technical Publication No. 277, 1959, pp. 1-45.
5. F.S. Rostler and R.M. White, "Influence of Chemical Composition of Asphalts on Performance, Particularly Durability," American Society for Testing Materials, ASTM Special Technical Publication No. 277, 1959, pp. 64-88.
6. F.S. Rostler and R.M. White, "Composition and Changes in Composition of Highway Asphalts, 85-100 Penetration Grade," Proceedings, Association of Asphalt Paving Technologists, Vol. 31, 1962, pp. 35-89.
7. S.H. Greenfeld and J.R. Wright, "Four Methods for Predicting the Durability of Roofing Asphalts," Materials and Research Standards, Vol. 2, 1962, pp. 738-745.
8. C.D. Smith, C.C. Scheutz, and R.S. Hodgson, "Relationship Between Chemical Structures and Weatherability of Coating Asphalts as Shown by Infrared Adsorption Spectroscopy," Industrial and Engineering Chemistry, Product Research and Development, Vol. 5, 1966, pp. 153-161.



9. T.C. Davis and J.C. Petersen, "An Inverse GLC Study of Asphalts Used in the Zaca-Wigmore Experimental Test Road," Proceedings, Association of Asphalt Paving Technologists, Vol. 36, 1967, pp. 1-15.
10. L.W. Corbett, "Composition of Asphalt Based on Generic Fractionation Using Solvent Deasphalting, Elution-Adsorption Chromatography, and Densimetric Characterization," Analytical Chemistry, Vol. 41, 1969, pp. 576-579.
11. J. Knotnerus, "Bitumen Durability--Measurement by Oxygen Adsorption," Industrial and Engineering Chemistry, Product Research and Development, Vol. 11, 1972, pp. 411-422.
12. R.L. Griffin, W.C. Simpson, and T.K. Miles, "Influence of Composition of Paving Asphalts on Viscosity, Viscosity-Temperature Susceptibility, and Durability," Journal of Chemical and Engineering Data, Vol. 4, 1959, pp. 349-354.
13. R.J. Peters, "Compositional Considerations of Asphalt for Durability Improvement," Transportation Research Board Record No. 544, 1975, pp. 46-55.
14. E.B. Barth, "Asphalt Science and Technology," Gordon and Breach, 1962.
15. J.P. Pfeiffer and R. Saal, "Asphaltic Bitumen as Colloid Systems," Journal Phys. Chem., Vol. 44, 1940, pp. 139.
16. C. Richardson, "The Modern Asphalt Pavement," 2nd Ed., Wiley, N.Y., 1914, pp. 115-126.
17. F.S. Rostler, "Fractional Composition: Analytical and Functional Significance, Bituminous Materials," 2, Inter Science Pub., 1965, pp. 151-222.

18. H. Eilers, "The Colloidal Structure of Asphalt," *Journal of Physical and Colloidal Chem.*, Vol. 53, 1949, pp. 1195.
19. H. Reerink, "Size and Shape of Asphaltic Particles in Relation to High Temperature Viscosity," *I&EC Prod. Res. and Dev.*, Vol. 12, 1973, p. 81.
20. K.H. Altgelt and O.H. Harle, "The Effect of Asphaltenes on Asphalt Viscosity," *I&EC Prod. Res. and Dev.*, Vol. 14, 1975, p. 240.
21. S.W. Ferris, E.P. Black, and J.B. Clelland, "Aromatic Structure in Asphalt Fracture," *I&EC Prod. Res. Dev.*, Vol. 6, 1967, p. 126.
22. H. Kim and R.B. Long, "Characterization of Heavy Residuum by Small Angle X-Ray Scattering Technique," *Ind. Eng. Chem. Fundam.*, Vol. 17, 1979, p. 60.
23. M.M. Boduszynski, J.F. Mackay, and D.R. Latham, "Asphaltenes, Where are you?," *Proc. AAPT*, Vol. 49, 1980, p. 123.
24. J.P. Girdler and R. Saal, "Asphaltic Bitumen as Colloid Systems," *Journal Phys. Chem.*, Vol. 44, 1940, p. 139.
25. J.C. Petersen, "Relationships Between Asphalt Chemical Composition and Performance-Related Properties," Presented at the Annual Meeting of the Asphalt Emulsion Manufacturers Assoc., Las Vegas, March 1982.
26. R.S. Winniford, "The Evidence for Association of Asphaltener in Dilute Solutions," *Div. of Petrol. Chem., Chem. Soc. Preprint*, Vol. L-3, 1962, p. 149.
27. J.J. Heithaus, "Measurement and Significance of Asphaltene Peptization," *Div. of Petrol. Chem., Chem. Soc. Preprints*, Vol. 5-4, 1960, p. 423.
28. D.A. Anderson and E.L. Dukatz, Jr., "Relation between Asphalt Flow Properties and Asphalt Composition," *Procc. AAPT*, Vol. 53, 1984, p.

29. D.A. Anderson, E.L. Dukatz, and J.L. Rosenberger, "Properties of Asphalt Cement and Asphaltic Concrete," Procc. AAPT, Vol. 52, 1983, p.
30. J.C. Peterson, "Chemical Composition of Asphalt as Related to Asphalt Durability - State of the Art," Transportation Research Board, 63rd Annual Meeting, 1984.
31. W.J. Halstead, "Relation of Asphalt Chemistry to Physical Properties and Specification," To be published on Procc. AAPT, 1985.
32. L.W. Corbett, Relationship Between Composition and Physical Properties of Asphalt, Procc. AAPT, Vol. 39, 1970, pp. 342-346.
33. L.W. Corbett and R.E. Merz, "Asphalt Binder Hardening in the Michigan Test Road After 18 years of Service," TRB 554, pp. 27-34.
34. F.S. Rostler and R.M. White, "Influence of Chemical Composition of Asphalts on Performance, Particularly Durability," ASTM STP No. 277, 1959, pp. 63-88.
35. B.M. Kiggundu, B.J. Nusser, D.E. Newcomb, and D.M. Zallen, "Correlation Between Physical and Chemical Properties in Recycled-Asphalt Binder Studies," Presented to the 21st Paving and Transportation Conference, The University of New Mexico, January 9-11, 1984.
36. R.D. Pavlovich, B.J. Nusser-Humphrey, B.M. Kiggundu, D.M. Zallen, and J.A. Murfee, "Asphalt Fractional Composition: Correlation Between Rostler and Clay Gel Components," Presented to Transportation Research Board Annual Meeting, Committee A2D01, January 1985.
37. B.M. Kiggundu, Et al., "The Potential Role of Chemistry for Evaluating Asphaltic Paving Materials," Presented at the Twentieth Paving and Transportation Conference, University of New Mexico, January 1983.

38. P.W. Jennings, "High Pressure Liquid Chromatography as a Method of Measuring Asphalt Composition," Montana Dept. of Highways, HRR-7930 and FWA, March 1980.
39. H. Plancher, A.J. Hoiberg, S.C. Suhaka, and J.C. Petersen, "A Settling Test to Evaluate the Relative Degree of Dispersion of Asphaltenes," Procc. AAPT, Vol. 48, 1979, pp. 351-374.
40. T.W. Kennedy and Chee-Chong Lin, "An Evaluation of the Asphaltene Settling Test," Research Report 253-2, Center for Transportation Research, The University of Texas at Austin, July 1981.
41. J.J. Heithaus, "Measurement and Significance of Asphaltene Peptization," Preprints Div. of Petroleum Chemistry, American Chemical Society, Vol. 5, No. 4-A, 1960, pp. A23-A38.
42. G.L. Oliensis, "The Oliensis Spot Test as a Quality Test," Procc. AAPT, Vol. 26, 1957, pp. 82-125.
43. J.L. Goodrich, J.E. Goodrich, and W.J. Kari, "Asphalt Composition Tests: Their Application and Relation to Field Performance," Presented at 65th Annual Meeting of the Transportation Research Board, January 1986.
44. J.E. Wilson and R.G. Hicks, "Evaluation of Construction and Short-Term Performance Problems for Asphalt Pavements in Oregon," Procc. AAPT, Vol. 48, 1979.
45. P. Puangchit, R.G. Hicks, J.E. Wilson, and C.A. Bell, "Impact of Variation in Material Properties on Asphalt Pavement Life," Final Report on Development of Rational Pay Adjustment Factors, FHWA-OR-82-3, FHWA, May 1982.

46. "Effect of Moisture and Aging on Asphalt Pavement Life," Joint HPR Study Between OSHD and OSU, in progress.
47. R.G. Hicks, J.E. Wilson, and G.E. Boyle, "Identification and Quantification of the Extent of Asphalt Stripping in Flexible Pavements in Oregon - Phase I," FHWA-OR-83-3, March 1983.
48. H.L. Davis, "AR Grading System Subcommittee to Investigate ASTM D1856-75, Abson Recovery Test Method," Progress Report, Eighteenth Pacific Coast Conference on Asphalt Specifications, May 1983.
49. O.K. Kim and C.A. Bell, "Effect of Moisture and Aging on Asphalt Pavement Life, Part 2: Effect of Aging," Draft Report prepared for Oregon Department of Transportation in cooperation with U.S. Department of Transportation FHWA, January 1986.
50. P.B. Girdler, "Constitution of Asphaltenes and Related Studies," Proc. AAPT, Vol. 33, 1965, pp. 45-79.
51. S. Hala, M. Kuras and M. Popl, "Analysis of Complex Hydrocarbon Mixtures," Volumen XIII, Elsevier Scientific Publishing Company, 1981, pp. 116-120.
52. R.L. Pecsok, L.D. Shield, T. Cairns and I.G. McWilliam, "Modern Methods of Chemical Analysis," Second Edition, John Wiley and Sons, 1976, pp. 56.
53. F.A. Cotton and G. Wilkinson, "Advance Inorganic Chemistry," Fourth Edition, John Wiley and Sons, 1979, pp. 329-330.

A P P E N D I X   A

TEST INSTRUCTION MANUAL  
FOR  
SEPARATION OF ASPHALT INTO FOUR FRACTIONS  
BY THE  
CORBETT-SWARBRICK PROCEDURE  
ASTM D4124-82, METHOD A  
(1000 mm COLUMN)

Prepared by

G. Thenoux

and

C.A. Bell

Civil Engineering Department  
Oregon State University  
Corvallis, OR 97331

For

Oregon State Highway Division  
Salem, OR 97310

September 1984

### A.1. SCOPE

The following instructions were prepared in order to give a more detailed description of the test procedure described in ASTM D4124-82.

The test procedure covers the separation of petroleum asphalt into four fractions:

- Asphaltenes
- Saturates
- Naphthene-Aromatics                      Petrolenes
- Polar-Aromatics

as defined in ASTM D4124 and shown in Figure A.11.

The procedure as described here has been slightly modified from the standard.

### A.2. SUMMARY OF THE METHOD

The sample containing the four defined fractions is first separated into n-heptane-insoluble (asphaltenes) and in n-heptane-soluble (petrolenes) components. Petrolenes are then further fractionated into saturates, naphthene aromatic, and polar aromatic in a glass chromatographic column.

### A.3. EQUIPMENT, MATERIALS, AND CHEMICALS

The required equipment, materials, and chemicals are given in Appendix B, which includes the cost and descriptions of the necessary equipment and glasswear to set the test. Also, the amount of chemical required to perform one test is given, together with cost per test.



#### A.4. SAMPLING AND SAMPLE PREPARATION

Bulk samples taken in accordance with ASTM method D-140 shall be representative and free of foreign substance. Samples for testing can be transferred by chilling or by heating to a maximum of 110°C (200°F).

Another simple method involves using a warm spoon. The correct amount of asphalt is placed on waxed paper and weighed. The sample can then be easily transferred to the Erlenmeyer flask.

#### A.5. PROCEDURE

##### A.5.1 Separation of Asphaltenes and Petrolenes

###### A.5.1.1 Preparation

- Weigh 11 to 13 g of asphalt to the nearest .01 g.
- Prepare a 2-L Erlenmeyer flask filled with approximately 1100 mL n-heptane. (n-heptane solvent should be in the ratio of 100 mL of solvent to 1 g of asphalt). A wide-mouth Erlenmeyer flask is preferred.
- Place asphalt sample into the 2-L Erlenmeyer flask.

###### A.5.1.2 Stirring

- Install an air-powered stirrer assembly into the 2-L Erlenmeyer flask. The stirring rod should rotate rotate inside a foil-covered rubber stopper that is used to seal the Erlenmeyer flask to reduce the evaporation of n-heptane.
- Place the flask in a water bath and maintain the solvent temperature near its boiling point (use approximately 80 to 90°C). See Figures A.1 and A.6 for complete assembly.

- Stir the contents of the flask at a moderate rate until there is no visual evidence of undispersed asphalt. Continue stirring for an additional hour for straight reduced asphalts, but for air-blown or chemically-modified asphalts the digestion time should be extended a further 1.5 hours. In general, 4 to 5 hours of total stirring is recommended.
- Remove the stirring assembly and rinse the stirring rod as it is removed from the flask with n-heptane (use a squeeze bottle). It is recommended to remove the stirring rod just before going to the next step (filtration) so a final stirring can be done to keep all the asphaltenes in suspension.

#### A.5.1.3 Filtration

- The filtration process is done in two different phases.

##### First Phase:

- Take the 430 x 123 extraction thimble and place it in the specially designed glass funnel (Figure 2). Place the glass funnel with the extraction thimble in a 2-L suction flask as shown in Figures A.2 and A.7. To increase the rate of filtration in this first phase, 5 to 6 holes can be made with a needle to the bottom of the extraction thimble.
- Wet the extraction thimble with n-heptane and apply suction to the flask. Transfer **all** the contents of the Erlenmeyer flask directly into the glass funnel fitted with the extraction thimble using a Pyrex funnel of 100 mm diameter and with a 95 mm stem (see Figure 4). During filtration the level of the solution should be kept as low as possible when passing through

the extraction thimble (no higher than 40 mm). Using additional solvent (n-heptane), repeatedly wash the extraction thimble until filtrate becomes colorless.

Second Phase:

- Set up a 12.5 cm diameter Buchner funnel appropriately fitted with a tared filter paper (Whatman #3, qualitative) in a new 2-L suction flask. Set up a 1-L separatory funnel suspended about 25 mm above the center of the filter paper. See Figures A.3 and A.8.
- Transfer filtrate content directly into the separatory funnel. Wet the filter paper in the Buchner funnel with n-heptane from a squeeze bottle and apply sufficient suction to the flask to secure the filter paper firmly to the funnel surface before beginning the filtration step. Add petrolene solution from the separatory funnel at a closely controlled rate and in such a manner that all the filtering takes place in the center of the paper. The filter paper should be wetted periodically from the squeeze bottle to ensure a tight seal with the funnel surface. Continue washing the asphaltene cake until the filtrate is colorless. Take care that no insolubles creep over the edges of the filter paper.
- Test the filtrate for insolubles by placing a drop of the filtrate on a clean filter paper. Repeat the second filtration if a ring appears.

#### A.5.1.4 Solvent Evaporation

- Transfer both the extraction thimble and the filter paper to a 16 cm evaporating dish and dry in an oven until a constant mass is achieved (use temperature less than 80°C). Record the net mass of asphaltenes recovered and stored, if desired.

#### A.5.1.5 Concentration of Petrolenes

- Concentrate the petrolene solution to about 500 mL. If a rotovapor is not available, heptane can be removed by using the setup shown in Figures A.4 and A.9.
- A heating mantle, a 1000 mL round-bottom flask, a condenser, and a 600 mL Erlenmeyer flask used as a receiver are set up as shown in Figure A.7. The sample is heated above its boiling point and at the same time nitrogen bubbles are introduced directly to the petrolene solution to break the surface tension and speed the concentration process.

By applying vacuum at the same time, through the receiver, the heating temperature can be reduced and the concentration process speeded up. This is not recommended since there is a great risk of sucking part of the sample through the condenser at the end of the process. Nevertheless, it may be required to use vacuum when concentrating the saturates due to the high boiling point of toluene, which is the solvent required to elute one of the fractions from the column.

## A.5.2 Separation of Petrolene Concentrate into Three Defined Fractions

### A.5.2.1 Column Preparation

- Set up the chromatographic column with appurtenances as shown in Figures A.5 and A.10.
- Place a concave filter pad, cut from the bottom of an extraction thimble (330 x 94), in the bottom of the column. Next, the column is partially filled with n-heptane. A loose plug of glass wool is tamped down into the bottom of the column with a long glass or steel rod until all trapped air is forced out as bubbles. Care should be taken not to plug the bottom of the column totally by tamping the glass wool too hard. A smaller layer of clean sand is formed on top of the glass wool by pouring sand into the column. The column is tapped to level the surface of the sand. Any sand adhering to the side of the column should be washed down with solvent. The final height of glass wool and sand should be 50 mm (approximately 25 mm each).

### A.5.2.2 Depositing the Adsorbent

- The dry pack method is preferred.
- In the dry pack method, the column is filled with solvent and allowed to drain slowly. The dry adsorbent (calcined F-20 alumina) is added, a little at a time, from a beaker, while the column is tapped constantly and gently on the side with a pencil fitted with a rubber stopper. When the height of the solvent has the desired length (680 cm + 10 mm, ~ 450 g) no more adsorbent is added. The solvent should be left draining, with continuous tapping of the column, until it is 5 cm (approximately) over the top of the

alumina. The top of the alumina should be perfectly level and the column should be perfectly vertical.

- Place a glass wool plug on top of the alumina bed, to protect it from being disturbed.

#### A.5.2.3 Applying the Sample to the Column

- The sample is added using a pipette. The bottom end of the pipette should be touching the walls of the column and a little below the surface of the solvent which is about 50 mm above the alumina bed.
- Transfer of the petrolene concentrate to the column should be done using the minimum amount of n-heptane.
- When all the sample has been added, the 50 mm layer of liquid is drained into the column until the top surface of the column just begins to dry. A layer of 15 cm of n-heptane is added using the pipette, with care not to disturb the surface.
- Slowly drain the column until the level of n-heptane goes to 5 cm above the alumina bed.

#### A.5.2.4 Elution

- Start addition of eluants with 200 mL of n-heptane (see Table A.1) from the equal-pressure funnel at a drip rate of about 5 mL/min; simultaneously collect the eluate at a rate of  $5 \pm 1$  mL/min in the receiving funnel; and subsequently drain eluate into tared containers, in accordance with the schedule in Table A.1.
- **CAUTION:** at no time should the liquid level in the column be allowed to drop below the top of the alumina bed.

- Start adding the new eluant when the last is 2 cm above the alumina bed. Follow the separation schedule given in Table A.1.

#### A.5.2.5 Cut Point

- Cutting the first fraction (saturates from naphthene-aromatics) requires close attention since a clear ring is not shown on the column. The cutting point should be done by looking at the distilled saturates which should have a different color from the naphthene aromatics. The first one is white, while the second one is yellow.
- The difference between the naphthene-aromatic and the polar-aromatic fractions is more obvious. A distinct black ring will show clearly the cut point for the naphthene aromatics.
- The final fraction (polar-aromatics) is almost black. The residual material in the column (end of the final fraction) is the remaining solvent after the polar aromatics. This should be essentially colorless.

#### A.5.2.6 Recovery of the Three Fractions

- Use the same concentration procedure described in A.5.1.5.
- Record the net mass of each fraction.

#### A.5.3 Calculations and Report

- Calculate the weight percent of each fraction as the mass percent of the original sample.
- Report percentages to the nearest 0.1%.

Table A.1. Separation Schedule

Column Feed Volumes		Fractions Received in Tared Containers	
Eluant Solvent	mL	Eluant Fraction	mL <sup>A</sup>
n-Heptane	200	Saturates (S)	300
Toluene	100		
Toluene	300	Naphthene-Aromatics (N-A)	600
Methanol/Toluene 50/50	300		
Trichloroethylene	600	Polar-Aromatics (P-A)	600 + hold-up
Column hold-up	-		

<sup>A</sup>Approximate eluant volumes since cut points may be adjusted (10.3.3) and hold-up can vary.

Methanol/Toluene 50/50	300	Naphthene-aromatics (N-A)	600 + hold-up
------------------------	-----	---------------------------	---------------



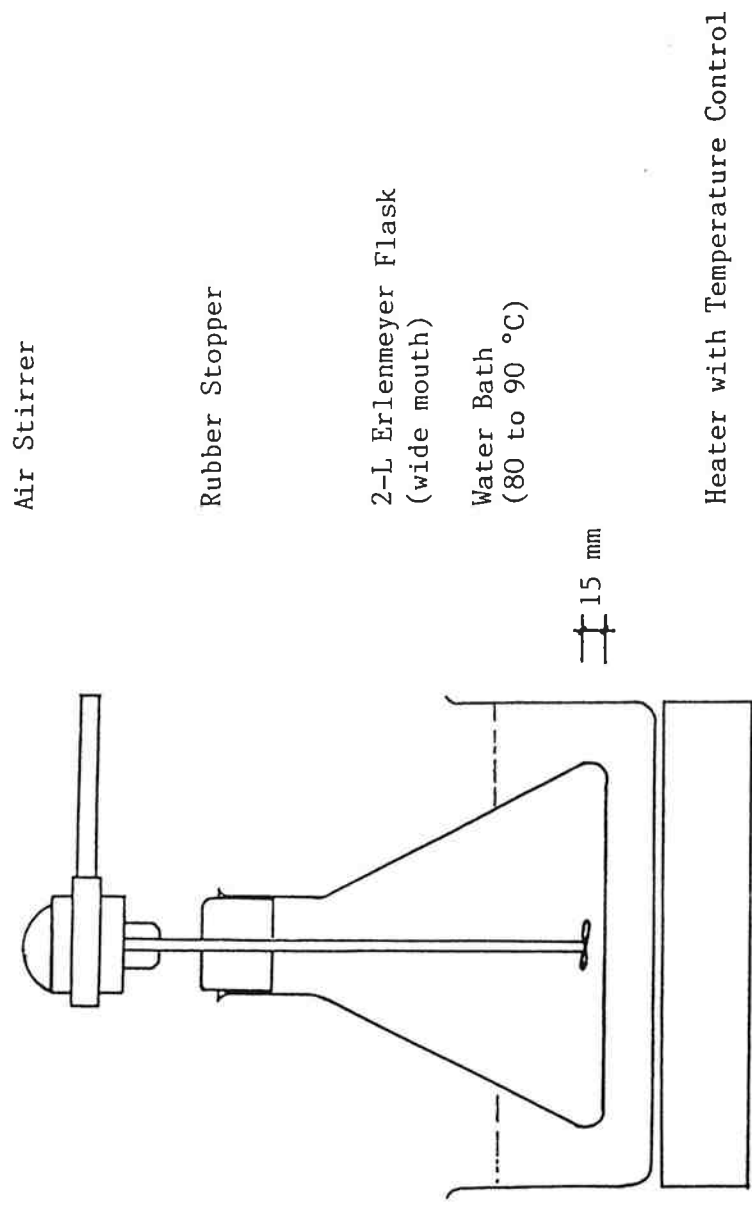
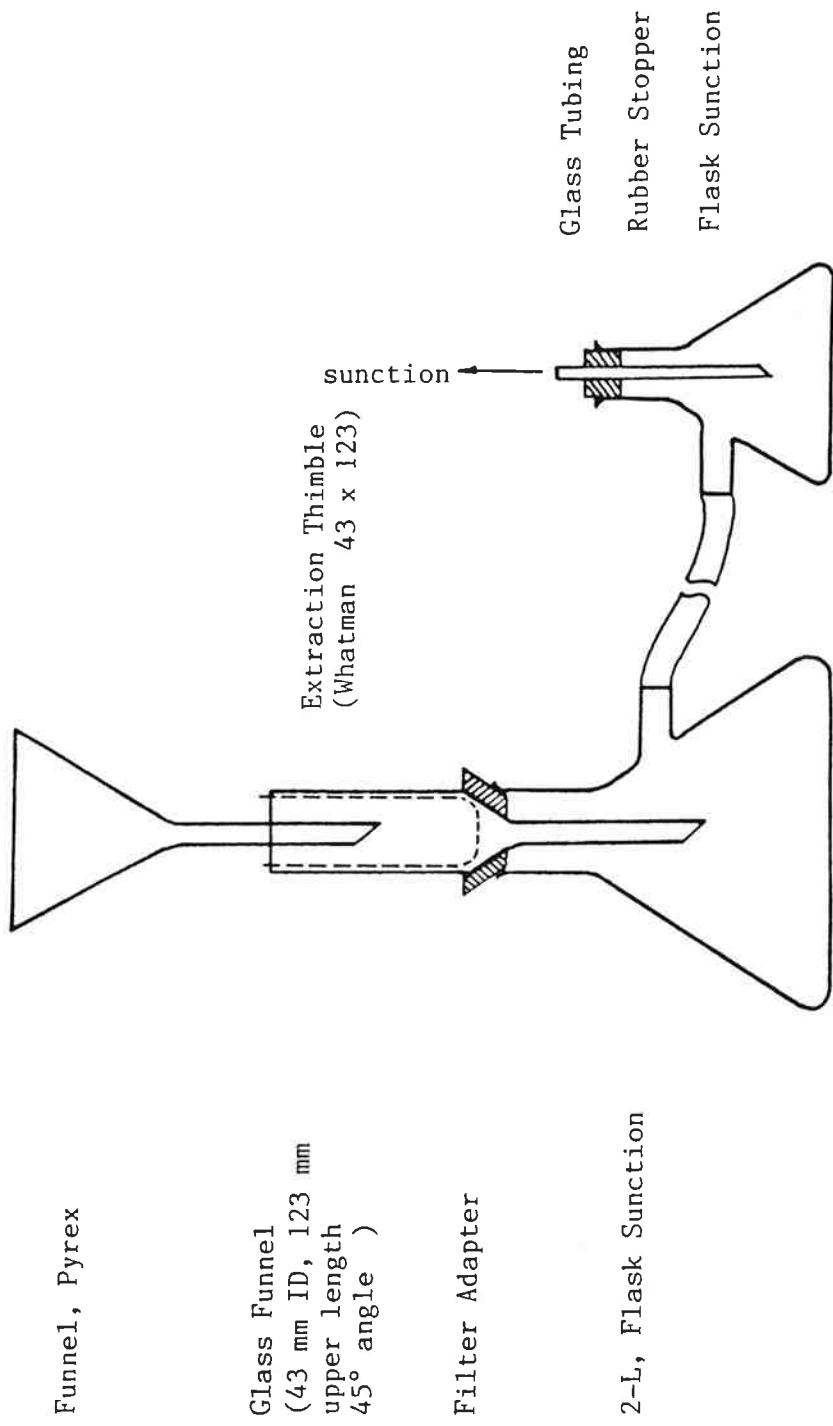


Figure A.1 : Stirring Process



Funnel, Pyrex

Glass Funnel  
(43 mm ID, 123 mm  
upper length  
45° angle )

Filter Adapter

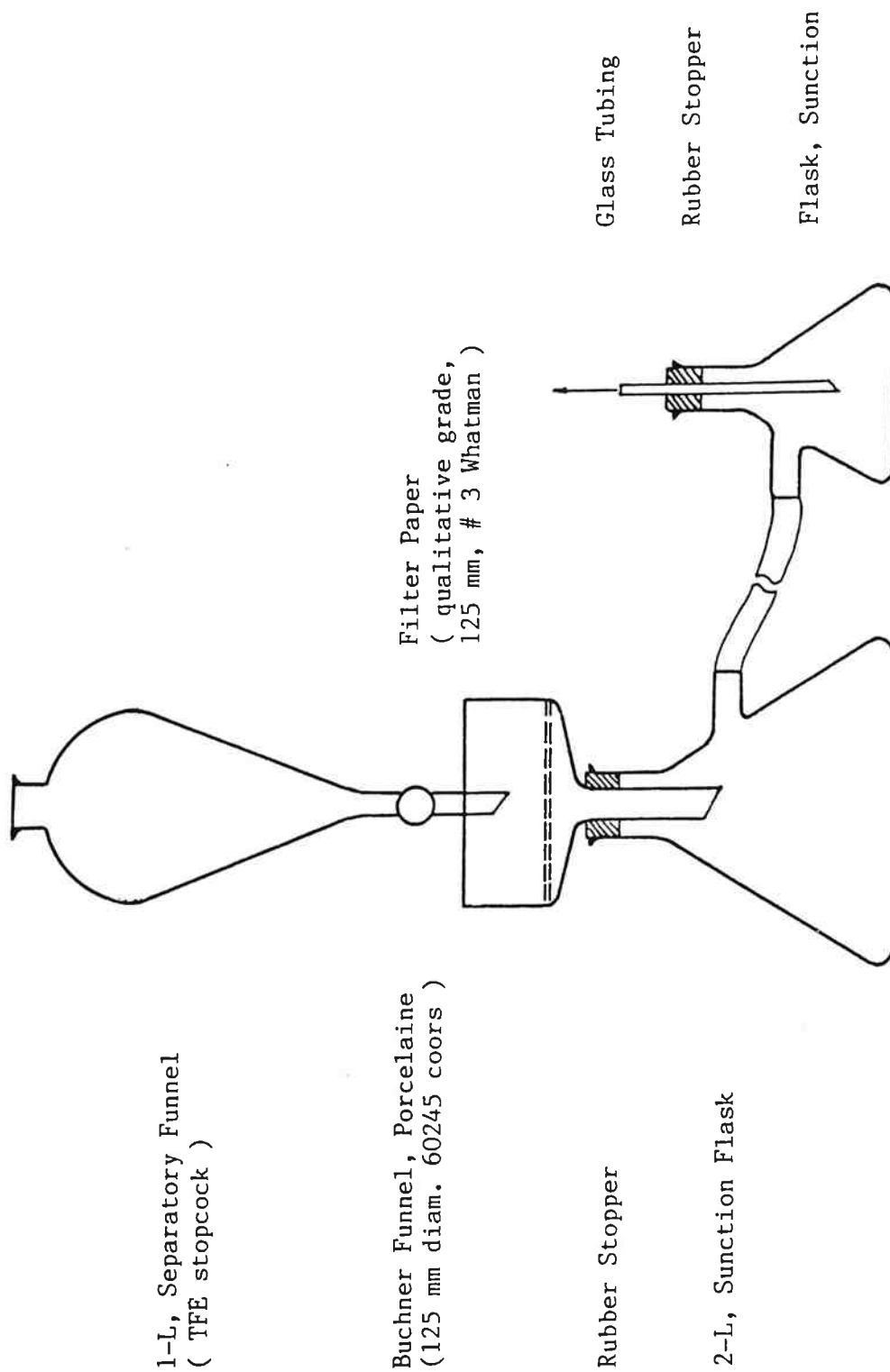
2-L, Flask Sunction

Glass Tubing  
Rubber Stopper  
Flask Sunction

Extraction Thimble  
(Whatman 43 x 123)

sunction

Figure A.2 : Filtration, First Phase



1-L, Separatory Funnel  
( TFE stopcock )

Buchner Funnel, Porcelaine  
(125 mm diam. 60245 coors )

Rubber Stopper

2-L, Sunction Flask

Filter Paper  
( qualitative grade,  
125 mm, # 3 Whatman )

Glass Tubing

Rubber Stopper

Flask, Sunction

Figure A.3: Filtration, Second Phase

Note : Use 24/40  
Standard Taper  
Joints

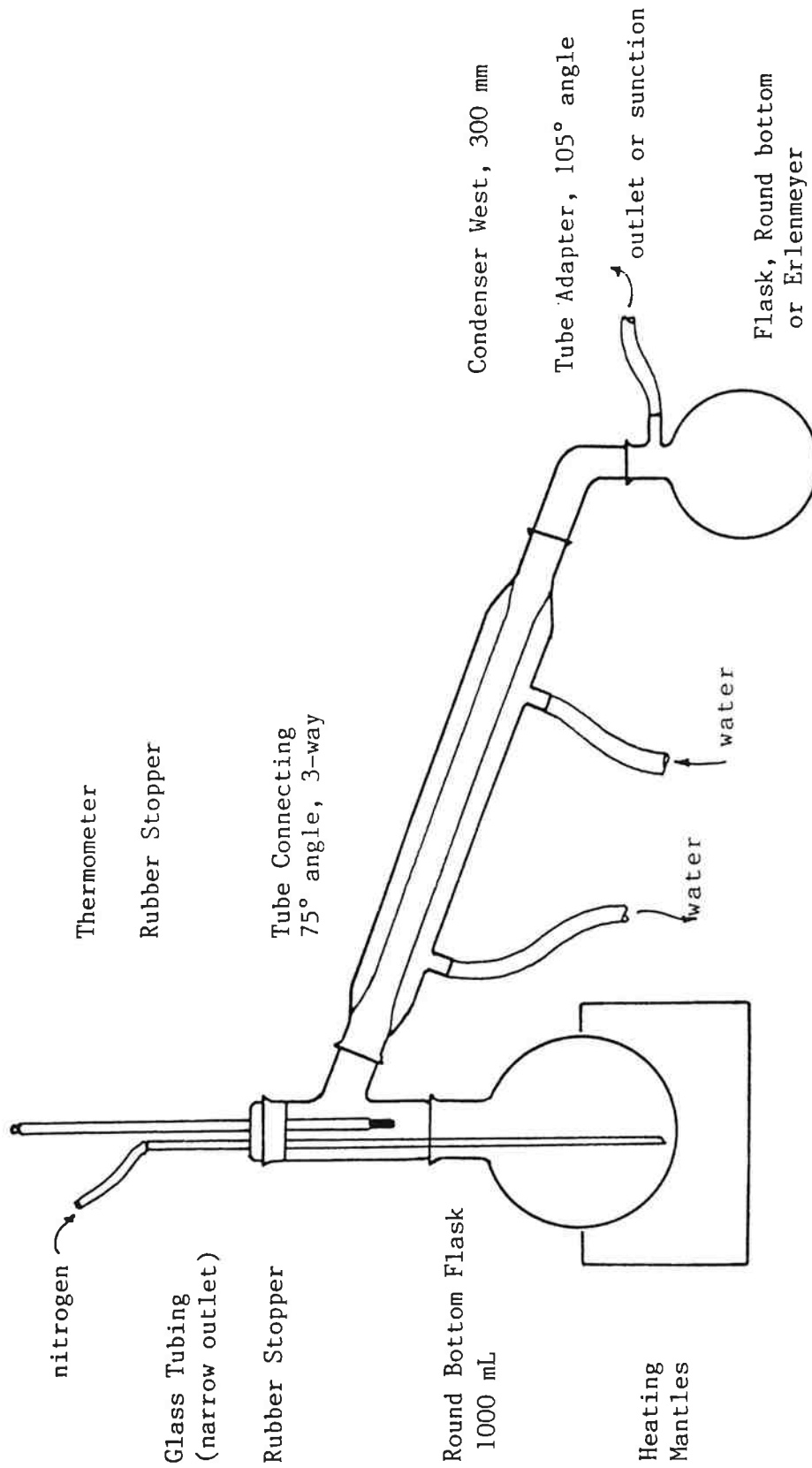


Figure A.4 : Fraction Concentration

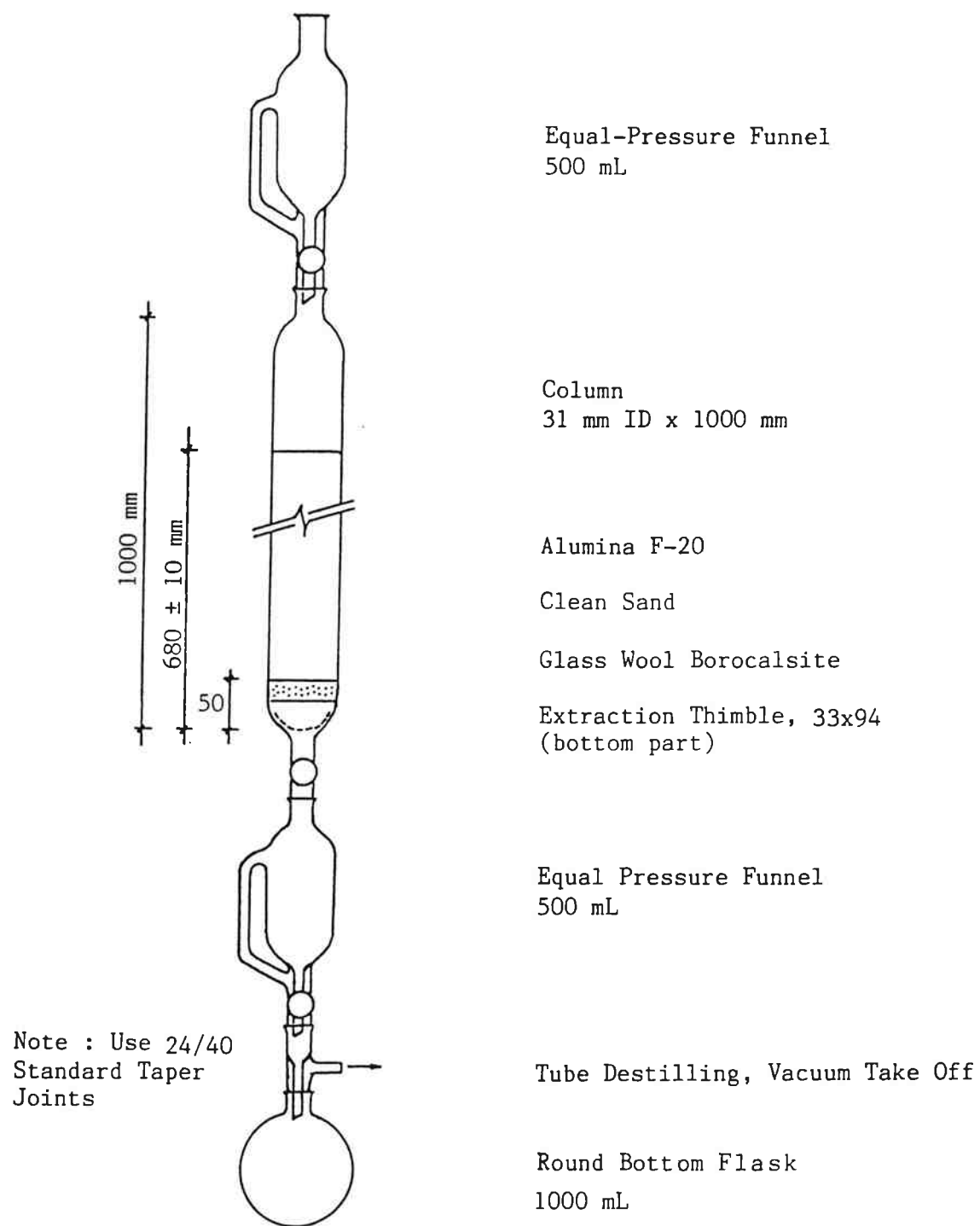


Figure A.5 : Chromatographic Column

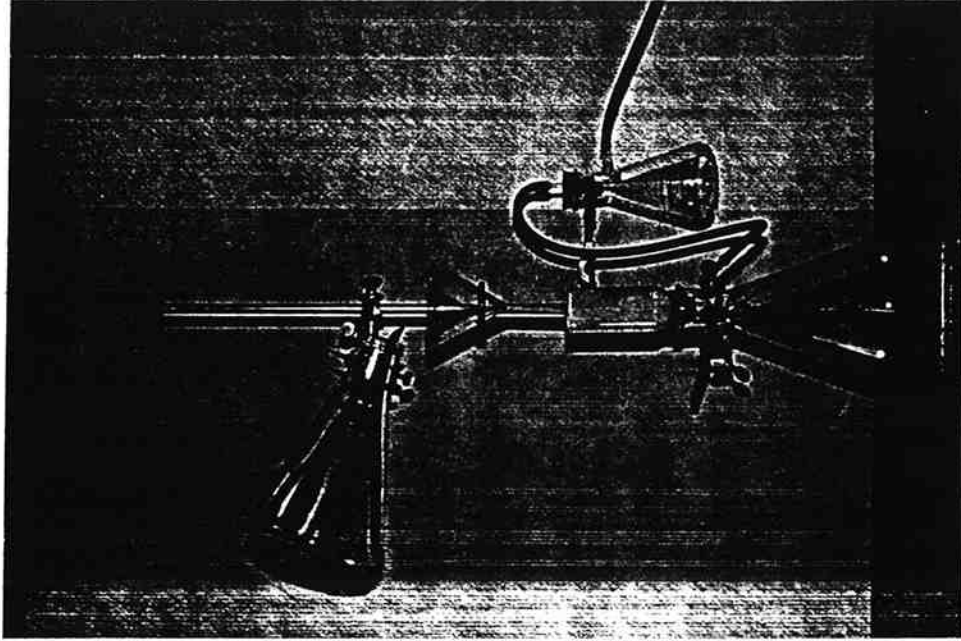


Figure A.7: Filtration, First Phase

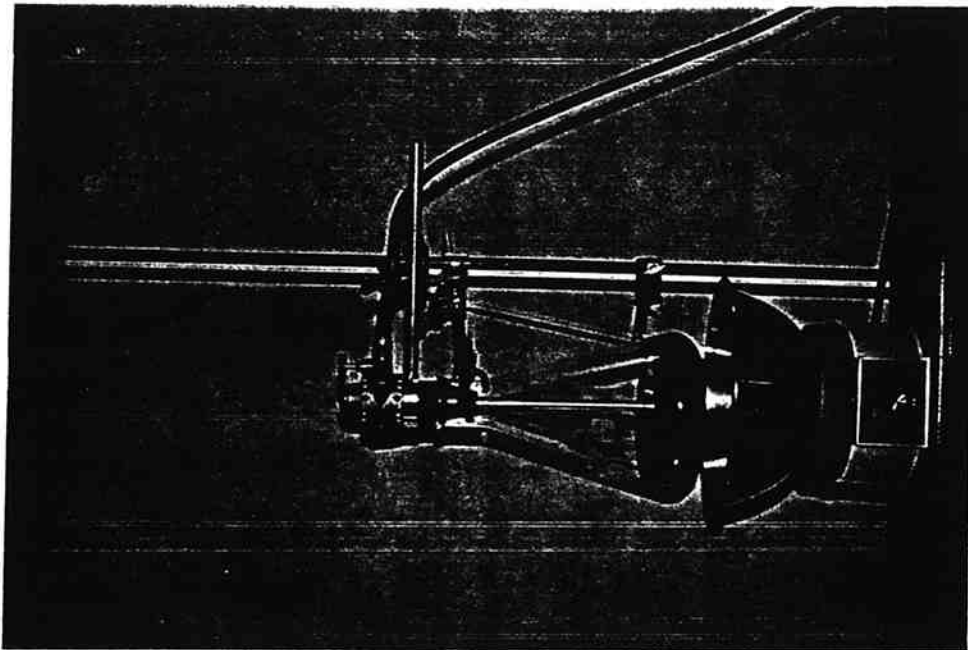


Figure A.6: Stirring Process

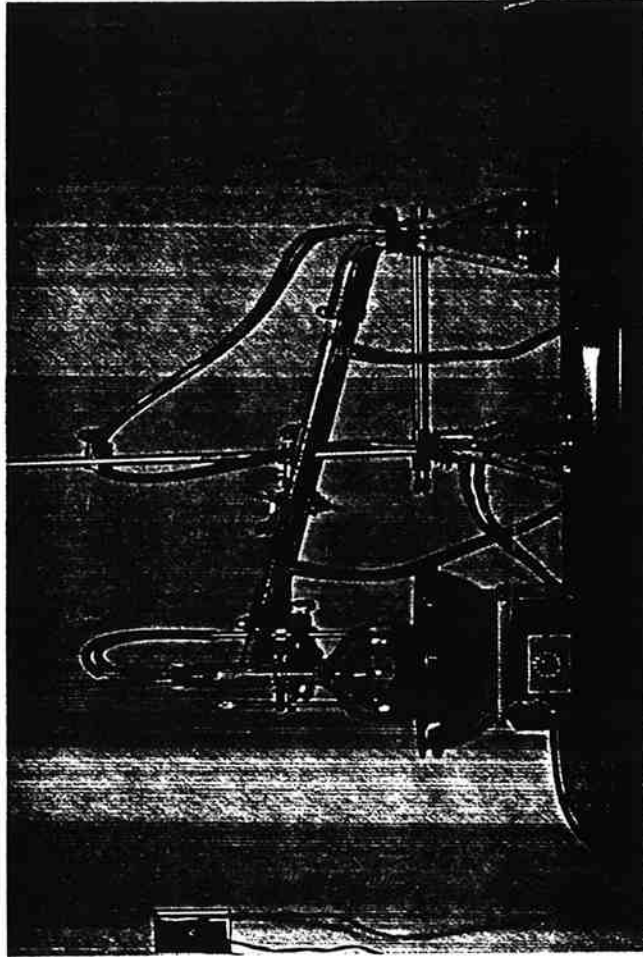


Figure A.9: Fraction Concentration

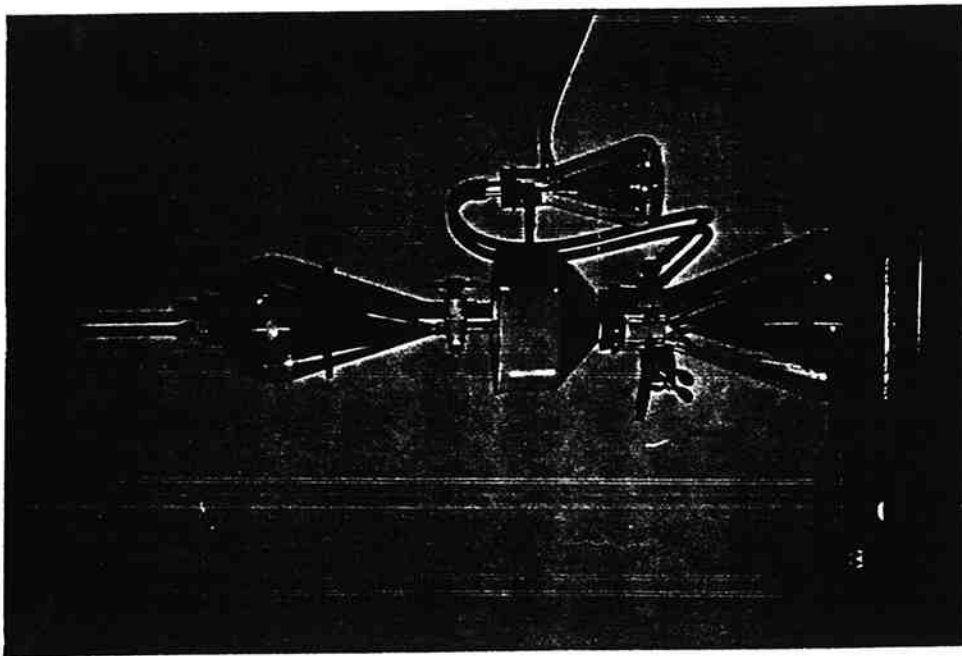


Figure A.8: Filtration, Second Phase

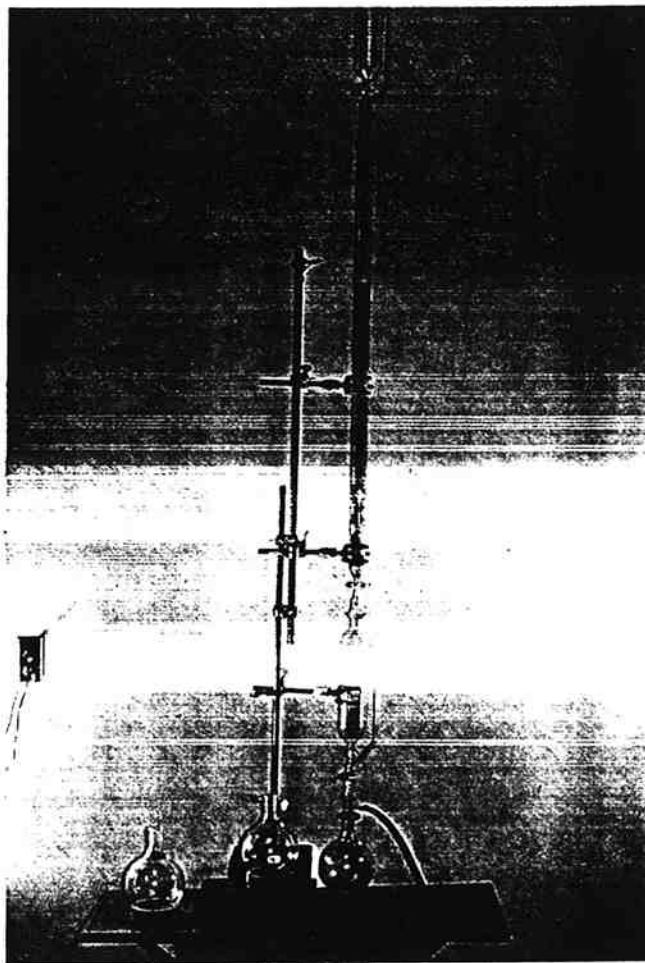


Figure A.10 : Chromatographic Column



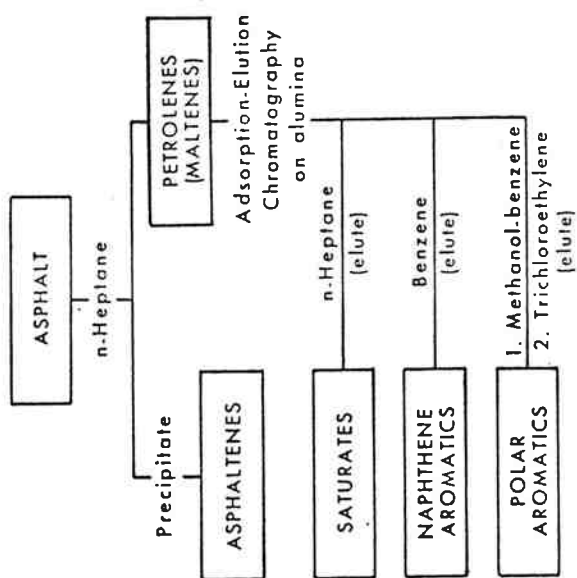


Figure A.11: Flow Diagram

APPENDIX B

## APPENDIX B

Equipment, Materials, Chemicals, and Price List for  
ASTM D4124-82  
Standard Test Method for Separation of Asphalt into Four Fractions

November 1984  
Oregon State University

prepared by

Guillermo Thenoux

### B.1 Introduction

A detailed list of equipment and materials required to perform ASTM D4124-82 has been prepared. The list of materials given in Tables A.1 through A.4 considers all the basic instruments required to perform the standard test according to the instruction procedure given in Appendix A.

The prices given are those corresponding to summer 1984.

### 3.2 Price List

Tables B.1 through B.3 give the general price list for all equipment and instruments required, divided as follows:

Table B.1: Glassware

Table B.2: Basic Instruments

Table B.3: General Instruments

Price Lists include instrument specifications, unit price, quantity required (for one set ASTM D4124), total cost and place of purchase.

Table B.4 contains chemicals and other materials required to perform one test. The price list contains the same as the above tables.

3.3 Remarks

a) There are other materials that are required that are not listed that depend in the way the instruments are going to be set up. These are:

- \* stands
- \* clamps
- \* ring clamps
- \* rubber tubes for vacuum, pressure, water, and nitrogen

b) The following facilities should be available:

- \* fume cover
- \* vacuum
- \* air pressure
- \* nitrogen source
- \* balance (minimum 500g with a precision of 0.01g)
- \* oven

B.4. Summary\*

a) Equipment	
Glassware	\$591.50
Basic Instruments	300.60
General Instruments	<u>55.18</u>
	\$947.28
b) Chemicals and materials per test	
	\$ 32.45

---

\*Prices, August 1984.

Table B.1. Glassware

Item No.	Description	Unit	Qty	Unit Price	Total	Purchased From	Remarks
1	Erlenmeyer Flask, Pyrex with Scale, 2000 mL, Wide Mouth	#	1	6.0	6.0	OSU	
2	Erlenmeyer Flask, Pyrex with Scale, 1000 mL	#	4	3.0	12.0	OSU	
3	Flask Filter, Pyrex with Scale, 2000 mL	#	2	22.0	44.0	OSU	
4	Erlenmeyer Flask, Pyrex with Scale, 500 mL	#	5	1.5	7.5	OSU	
5	Beaker, Griffin with Scale, 1000 mL	#	2	3.0	6.0	OSU	
6	Beaker, Griffin with Scale, 600 mL	#	4	1.5	6.0	OSU	
7	Round Bottom Flask, 1000 mL	#	4	15.0	30.0	OSU	
8	Buchner Funnel, Porcelain, 125 mm, 60245 Coors.	#	1	38.0	38.0	VWR	
9	Funnel, Pyrex, 60° Angle, 100 mm diam., 95 mm Stem	#	3	3.0	9.0	OSU	
10	Glass Funnel to accommodate 43 mm I.D. x 123 mm Extraction Thimble. 45° Bottom Angle, 50 mm Stem	#	1	20.0	20.0	OSU	Manufactured at OSU
11	Tube Distilling, Vacuum Take Off, 24/40	#	1	27.0	27.0	OSU	
12	Tube Adapter 105° Angle, 24/40	#	1	7.0	7.0	OSU	
13	Tube connecting 75° Angle (Downward) 3-way, 24/40	#	1	21.0	21.0	OSU	
14	Evaporating dish, procelain, 160 mm diam.	#	1	16.0	16.0	VWR	
15	Funnel, Separatory, 1000 mL, TFE Fluorocarbon Stopcock	#	1	55.0	55.0	OSU	
16	Pipet, ~ 15 mm diam., ~ 300 mm length	#	1	10.0	10.0	OSU	
17	Pipet, ~ 3 mm diam., ~ 200 mm length	#	1	0.5	0.5	OSU	
18	Column Chromatography, Whit 2 Funnels Pressure Equalizing of 500 mL	#	1	250.0	250.0	OSU	Manufactured at OSU according to standard spec.
19	Condenser West, 300 mm, 24/40	#	1	26.5	26.5	OSU	
				Total	591.5		

Table B.2. Basic Instruments

Item No.	Description	Unit	Qty	Unit Price	Total	Purchased From	Remarks
1	Stirrer, Air Driven, (Arrow Type A)	#	1	104.0	104.0	A.H. Thomas Co.	#8583-J10
2	Heating Mantles with Controller Electro Thermal to fit 1000 mL Round Bottom Flask	#	1	130.0	130.0	VWR	#33749-743
3	Hot Plate, 750 watt, 120 volts	#	1	66.6	66.6	VWR	#33922-054
				Total	300.6		

Table B.3 Materials and General Instruments

Item No.	Description	Unit	Qty	Unit Price	Total	Purchased From	Remarks
1	Cork Ring, 110x60 mm	#	4	1.82	7.28	OSU	
2	Poly Joint Clamp #24	#	4	0.20	0.80	OSU	
3	Rubber Stopper #10	#	4	0.90	3.60	OSU	
4	Rubber Stopper #9	#	4	0.72	2.88	OSU	
5	Rubber Stopper #7	#	6	0.37	2.22	OSU	
6	Filter Adapters (Set of 6)	#	1	15.0	15.0	VWR	
7	Polyethylene Wash Bottle, 500 mL	#	2	2.0	4.0	OSU	
8	Sauce Pan for Water Bath (Should contain a 2000 mL Erlenmeyer Flask)	#	1	4.0	4.0	Fred Meyer	
9	Tongs (Straight Forceps) 5 in.	#	1	5.0	5.0	OSU	
10	Steel Rod, Approximately 10 mm diam. x 1.5 m length	#	1	2.0	2.0	OSU	
11	Mercury Thermometer, up to +150°C	#	2	4.2	8.4	OSU	
				<b>Total</b>	<b>55.18</b>		

Table B.4. Chemicals and Other Materials to be Used Per Test

Item No.	Description	Unit	Qty	Unit Price	Total	Purchased From	Remarks
1	N-Heptane, 99 Min.Mol%, Pure Grade	gal	~ 1	22.0	22.0	OSU	Normally not in stock
2	Methanol, Anhydrous Reagent Grade	pint	.3	3.0	.9	OSU	
3	Toluene, Reagent Grade	gal	.15	12.0	1.8	OSU	
4	Trichloroethylene, BP 86.5 to 87.5°C	gal	.16	26.0	4.0	OSU	
5	Alumina, F-20, Chromatographic Grade calcined at 77.5°F/16 hrs	k	.5	20.0	10.0	Sigma Chemical Co.	
6	Extraction Thimble, 33x94 (Whatman)	#	1	1.2	1.2	OSU	
7	Extraction Thimble, 43x123 (Whatman)	#	1	1.8	1.8	OSU	
8	Glass Wool Borocalsite	lb	.01	26.0	.2	OSU	
9	Filter Paper, Qualitative Grade, 125 mm #3 Whatman (Pack of 100)	Pack	-	6.45	.1	OSU	
10	Sand, very clean	-	-	-	-	-	-
11	Wax Paper	-	-	-	-	-	Small quantity per test
12	Aluminum Foil	-	-	-	-	-	Small quantity per test
Total					32.45		



APPENDIX C

TEST INSTRUCTION MANUAL  
FOR  
SEPARATION OF ASPHALT INTO FOUR FRACTIONS  
BY THE  
CORBETT-SWARBRICK PROCEDURE  
ASTM D4124-82, METHOD B\*  
(500 mm COLUMN)

Prepared by

G. Thenoux

and

C.A. Bell

Civil Engineering Department  
Oregon State University  
Corvallis, OR 97331

For

Oregon Highway Division  
Salem, OR 97310

September 1985

---

\*Method "B", currently being revised by ASTM Committee D04.47.

### C.1. SCOPE

The following instructions were prepared in order to give a more detailed description of the proposed Corbett-Swarbrick test method ('short procedure') for separation of asphalt into four fractions. This method (Method B) is based on the standard test procedure described in ASTM D4124-82 (Method A), and is currently being revised by the ASTM Committee D04.47.

The test procedure described here introduces some modifications and recommendations to the present proposed Method B (ASTM D4124-82 short procedure) in view of the experience gained by the authors.

### C.2. SUMMARY OF THE METHOD

The test procedure covers the separation of petroleum asphalt into four fractions:

- Asphaltenes
- Saturates
- Naphthene-Aromatics                      Petrolenes
- Polar-Aromatics

as defined in ASTM D4124 and reproduced here in paragraph C.3.

The sample containing the four defined fractions is first separated into n-heptane-insoluble (asphaltenes) and in n-heptane-soluble (petrolenes) components. Petrolenes are then further fractionated into saturates, naphthene aromatics, and polar aromatics in a glass chromatographic column.

### C.3. DESCRIPTION OF TERMS AS IN ASTM D4124-82

- Asphaltenes or n-heptane insolubles - matter that can be separated from asphalt following digesting of the asphalt in n-heptane under the specified conditions in this method.
- Petrolenes - the n-heptane-soluble matter recovered following separation of the asphaltenes from the digested mixture under the specified conditions in this method.
- Saturates - material that, on percolation in an n-heptane eluant, is not absorbed on calcined F-20 alumina absorbent under the conditions specified.
- Naphthene aromatics - material that is adsorbed on calcined F-20 alumina in the presence of n-heptane, and desorbed by toluene, after removal of the saturates under the conditions specified.
- Polar aromatics - material desorbed from calcined F-20 alumina absorbent, after the saturates and naphthene aromatics have been removed, using methanol, toluene, and trichloroethylene eluants under the conditions specified.

### C.4. REAGENTS AND ADSORBENT

- Alumina, F-20 chromatographic grade, calcined at 413°C (775°F) for 16 hours and stored in an evacuated desiccator or airtight bottle.

**Note 1:** Although the standards suggest that alumina under the above specifications could be obtained from some manufacturers, it is necessary and recommended to retreat the alumina in a muffle furnace according to the required ASTM specifications.

- n-Heptane, 99 minimum mol % (pure grade).

**Note 2:** n-Heptane should be totally free of moisture. Pretreatment of n-heptane with 5A molecular sieves or by refluxing over calcium hydride may be necessary to remove residual moisture in the solvent.

- Methanol, anhydrous, reagent grade.
- Toluene, reagent grade.
- Trichloroethylene, boiling point 86.5 to 87.5°C.

### C.5 EQUIPMENT, MATERIALS, AND CHEMICALS

The required equipment, materials, and chemicals are given in Appendix D, which includes a description of the necessary equipment and glassware to set the test. Also, the amounts of chemicals required to perform one test is given, together with cost per test.

### C.6. SAMPLING AND SAMPLE PREPARATION

Bulk samples taken in accordance with ASTM method D-140 shall be representative and free of foreign substance. Samples for testing can be transferred by chilling or by heating to a maximum of 110°C (200°F).

Another simple method involves the use of waxed paper. Warm asphalt samples at 70 to 80°C (160 to 180°F) and transfer the approximate amount to a (2x2 in.) waxed paper. Let the sample cool down to room temperature in a desiccator. If sample is not going to be used immediately after cooling, it is recommended that it be covered with another piece of waxed paper.

The reason for recommending the use of a waxed paper is because the sample can be easily cut to the desired weight, after cooling, and can be easily transferred to the Erlenmeyer flask for precipitation, without further treatment.

C.7. PROCEDUREC.7.1 Separation of Asphaltenes and PetrolenesC.7.1.1 Preparation

- Weigh to the nearest 0.001 g and place into a 600 mL Erlenmeyer flask (wide mouth) the required quantity of asphalt so that it yields about 2 g of petrolenes as indicated by the following equation:

$$\text{Sample weight, g} = \frac{200}{100 - \% \text{ asphaltenes}}$$

- For paving asphalt, this would be 2.3 to 2.5 g of asphalt. For airblown asphalt, for recovered asphalt (road aged), or artificially aged asphalt, the amount of asphalt required could go from 2.3 g up to 2.9 or 3.0 g.
- Place in flask asphalt sample. (Prefer to place sample to the side of flask so that this does not interfere with the rotating movement of the stirring rod - see Figure C.1.). Add n-heptane solvent in the ratio of 100 mL solvent per 1 g of sample.
- The standard procedure recommends that the Erlenmeyer flask should be warmed gently to disperse the asphalt sample over the bottom and lower sides of the flask before adding the n-heptane. This step is not recommended by the author. By warming the flask before and during the stirring process, it has been observed that the amount of asphaltenes sticking to the glass will considerably increase. The asphaltenes sticking to the glass are not removable with h-heptane.

**Note 3:** All weighings are to be done with the materials at room temperature.

#### C.7.1.2. Stirring

- Install an air powered stirrer assembly into the 2-L Erlenmeyer flask. See Figure C.1.
- The standard procedure uses a steam bath to maintain solvent temperature near its boiling point ( $\sim 98^{\circ}\text{C}$  or  $208^{\circ}\text{F}$ ). For the same reasons quoted above (C.7.1.1), the authors recommend working at room temperature. No differences have been noticed in the total amount of precipitated asphaltenes when using a steambath or not.
- Stir the contents of the flask for 45 minutes at a moderate rate.
- Remove the stirring assembly and rinse the stirring rod as it is removed from the flask with n-heptane (use a squeeze bottle). Removing the stirring rod is recommended just before going to the next step (filtration) so a final stirring can be done to keep all the asphaltenes in suspension.

#### C.7.1.3. Filtration

**Note 4:** The filtration procedure described here does not correspond to the one given in the proposed standard (Method B). The results of the method described here are cheaper and more rapidly performed. Also, it has been observed that it yields the same proportion of asphaltenes.

- The proposed procedure considers the use of at least two filtration phases. The first phase is intended to collect the bulk of the precipitated asphaltenes immediately after the stirring process so that chances for asphaltenes to stick to the glass are reduced. At the same time, there is no need for waiting 24 hours for the settlement of the asphaltenes. The second phase follows exactly the filtration procedure described in the original standard ASTM 4124-82.

#### First Phase

- Set up an 83 mm Buchner funnel with an "open glass cylinder" of the same internal diameter of funnel and a 125 mm filter paper (Whatman #1, qualitative), see Figure C.2.a.

**Note 5:** Weigh open glass cylinder with filter paper before setting up the above filter system.

- Place funnel with appurtenances in a 1-L suction flask as shown in Figure C.2.b. Wet filter paper and apply suction to the flask.
- Immediately after the stirring process, the petrolene solution is transferred to a 600 mL beaker. (If the materials are not transferred immediately after stirring, stir the sample again before transferring to the beaker).
- With the asphaltene in suspension, transfer all the petrolene solution from the 600 mL beaker to the filtration flask at a moderate rate. With one hand, press down the "open glass cylinder" to the funnel so that petrolenes are sucked faster. Using additional solvent (n-heptane), repeatedly wash the filter paper until filtrate becomes colorless.



**Note 6:** The advantage of using this procedure is that the bulk of the asphaltenes can be collected immediately after stirring, in a relatively short time without clogging the filtering setup.

The time required for an asphalt with approximately 12% asphaltenes is usually 10 to 15 minutes.

- Remove "open glass cylinder" together with the filter paper and transfer to a Petri dish (100 mm diam.).

### Second Phase

- Set up a 12.5 cm diameter Buchner funnel appropriately fitted with a tared filter paper (Whatman #3, qualitative) in a new 1-L suction flask. Set up a 1-L separatory funnel suspended about 25 mm above the center of the filter paper. See Figure C.3.
- Transfer filtrate content directly into the separatory funnel. Wet the filter paper in the Buchner funnel with n-heptane from a squeeze bottle and apply sufficient suction to the flask to secure the filter paper firmly to the funnel surface before beginning the filtration step. Add petrolene solution from the separatory funnel at a closely controlled rate and in such a manner that all the filtering takes place in the center of the paper. The filter paper should be wetted periodically from the squeeze bottle to ensure a tight seal with the funnel surface. Continue washing the asphaltene cake until the filtrate is colorless. Take care that no insolubles creep over the edges of the filter paper.
- Test the filtrate for insolubles by placing a drop of the filtrate on a clean filter paper. Repeat the second filtration if a ring appears.
- Transfer filter paper to a 16 cm evaporating dish.

#### C.7.1.4 Solvent Evaporation

- Dry both filter papers at 60°C (50°F) for a time no longer than 30 minutes (see Note 8). Allow the materials to cool to room temperature before weighing.
- Record net mass of asphaltenes.

**Note 7:** For the first filtration phase, do not separate filter paper from the open glass cylinder. These should be weighed together, before and after filtration for recording the net mass of asphaltenes.

**Note 8:** It has been observed that asphaltenes have very unstable weight if heated at higher temperatures or longer periods of time.

#### C.7.1.5 Concentration of Petrolenes

- Concentrate the petrolene solution to about 100 to 50 mL. If a rotovapor is not available, heptane can be removed by using the setup shown in Figures C.4.
- A heating mantle, a 1000 mL round-bottom flask, a condenser, and a 600 mL Erlenmeyer flask used as a receiver are set up as shown in Figure C.4. The sample is heated above its boiling point and at the same time nitrogen bubbles are introduced directly to the petrolene solution to break the surface tension and speed the concentration process.
- Transfer concentrate solution to a 400 mL graduated beaker. The petrolene solution should be further concentrated by placing the beaker in a heating plate at temperature not greater than 80°C (75°F).

- Remove beaker from heating plate when the petrolene solution has concentrated to 25 mL, as shown by the graduating marks in the beaker. Sufficient fluidity should remain in the petrolene concentrate to permit easy transfer of concentrate to chromatographic column.

## C.7.2 Separation of Petrolene Concentrate into Three Defined Fractions

### C.7.2.1 Column Preparation

- Set up the chromatographic column with appurtenances as shown in Figures C.5.
- Place a concave filter pad, cut from the bottom of an extraction thimble (26 x 60 mm), in the bottom of the column. Next, the column is partially filled with n-heptane. A loose plug of glass wool is tamped down into the bottom of the column with a long glass or steel rod until all trapped air is forced out as bubbles. Care should be taken not to plug the bottom of the column totally by tamping the glass wool too hard. A smaller layer of clean sand is formed on top of the glass wool by pouring sand into the column. The column is tapped to level the surface of the sand. Any sand adhering to the side of the column should be washed down with solvent. The final height of glass wool and sand should be 40 mm (approximately 25 mm glass wool, 15 mm sand).

**Note 9:** The sand plug should have the following characteristics to act as a filter:

1. 100% retained on #200 sieve.
2. 10-20% retained on #50 sieve.
3. 100% passing #8 sieve.

#### C.7.2.2 Depositing the Adsorbent

- The dry pack method is preferred. See Reference C1.
- In the dry pack method, the column is filled with solvent and allowed to drain slowly. The dry adsorbent (calcined F-20 alumina) is added, a little at a time, from a beaker, while the column is tapped constantly and gently on the side with a pencil fitted with a rubber stopper. When the height of the alumina has the desired length ( $400 \pm 10$  mm,  $\sim 180$  g) no more adsorbent is added. The solvent should be left draining, with continuous tapping of the column, until it is 5 mm (approximately) over the top of the alumina. The top of the alumina should be perfectly level and the column should be perfectly vertical.
- Add 10 mm of sand on top of the alumina bed, to protect it from being disturbed.

### C.7.2.3 Applying the Sample to the Column

- Place a glass funnel on top of chromatographic column and let the funnel stem touch the side wall of the column. Gently transfer with the beaker, the petrolene concentrate to the column so that the solution drains down the side of the column wall. Use the minimum amount of solvent (n-heptane) to clean beaker and funnel.
- When all the sample has been added, the layer of solution is drained into the column until the top surface of the column just begins to dry. A layer of 10 mm of n-heptane is added using the pipette, with care not to disturb the surface.
- Slowly drain the column until the level of n-heptane goes to 5 mm above the sand bed.

### C.7.2.4 Elution

- Start addition of eluants with 65 mL of n-heptane (see Table C.1) from the equal-pressure funnel at a drip rate of about 5 mL/min; simultaneously collect the eluate at a rate of  $5 \pm 1$  mL/min in the receiving funnel; and subsequently drain eluate into tared containers, in accordance with the schedule in Table C.1.
- **CAUTION:** at no time should the liquid level in the column be allowed to drop below the top of the alumina bed.
- Start adding the new eluant when the last is 2 cm above the alumina bed. Follow the separation schedule given in Table C.1.

#### C.7.2.5 Cut Point

- Cutting the first fraction (saturates from naphthene-aromatics) requires close attention since a clear ring is not shown on the column. The cutting point should be done by looking at the glass wool color which will turn from white (saturates) to yellow (naphthene-aromatics).
- The difference between naphthene-aromatic and the polar-aromatic fractions is more obvious. A distinct black ring will show clearly the cut point for the naphthene-aromatics.
- After collecting the second fraction (naphthene-aromatics), load column with the last solvent, open widely valves "A" and "B", and close valve "C" (Figure C.5). Apply continuous vacuum to the column until trichloroethylene reaches the top of alumina bed. Stop vacuum, drain equalizer pressure funnel "F" and fill equalizer pressure funnel "E" with 250 mL n-heptane.
- Applied continuous vacuum for the second time until solvent reaches the top of alumina bed. Check if the solvent draining to funnel B is coming out colorless; if not, 100 mL of n-heptane should be passed through the column again by repeating the above procedure.

**Note 10:** Do not let alumina dry off since it is easier to clean the column when alumina is in a slurry state.

**Note 11:** The n-heptane used to clean any remaining polar aromatics ("hold-up") in the column, could be that recovered during the concentration of the petrolenes.

#### C.7.2.5 Recovery of the Three Fractions

- If a rotovapor is not available, the fractions could be concentrated using the setup shown in Figure C.4.
- If using setup shown in Figure C.4., it is recommended to concentrate fractions to approximately 100 mL and then transfer to a tared 250 mL beaker.
- Continue concentration of solutions by placing beakers in a hot plate at 80°C until all the solvent has evaporated.

**Note 12:** By using the above procedure, when a rotovapor is not available, it is possible to eliminate all the solvent without reaching very high temperatures in the heating mantles. At the same time, the cleaning of the round bottom flask is much easier.

- Record the net mass of each fraction. Materials should be at room temperature.

#### C.7.3 Calculations and Report

- Calculate the weight percent of each fraction as the mass percent of the original sample.
- Report percentages to the nearest 0.1%.

#### C.7.4 Cleaning Column

- Do not drain column solvent. Solvent should cover top of alumina at all times.
- Place column horizontally and remove stop-cock. Apply moderate air pressure from the bottom of column. Press the opening left by the stop-cock with fingers.

- The alumina should come out with a slurry consistency.
- Collect the alumina in a tray and discard. It is not economical to clean and reactivate.

**Note 13:** The bottom of the extraction thimble could be used several times.



Table C.1. Column Feed Volumes

Eluant Solvent	mL	Fraction Received	Approximate mL
n-Heptane	65	Saturates	100
Toluene	35		
Toluene	100	Naphthene-Aromatics	200
Methanol/Toluene (50/50)	100		
Trichloroethylene	200	Polar-Aromatics	200 + Cleaning Solvent

\*For cleaning solvent, n-Heptane could be used if vacuum is applied.

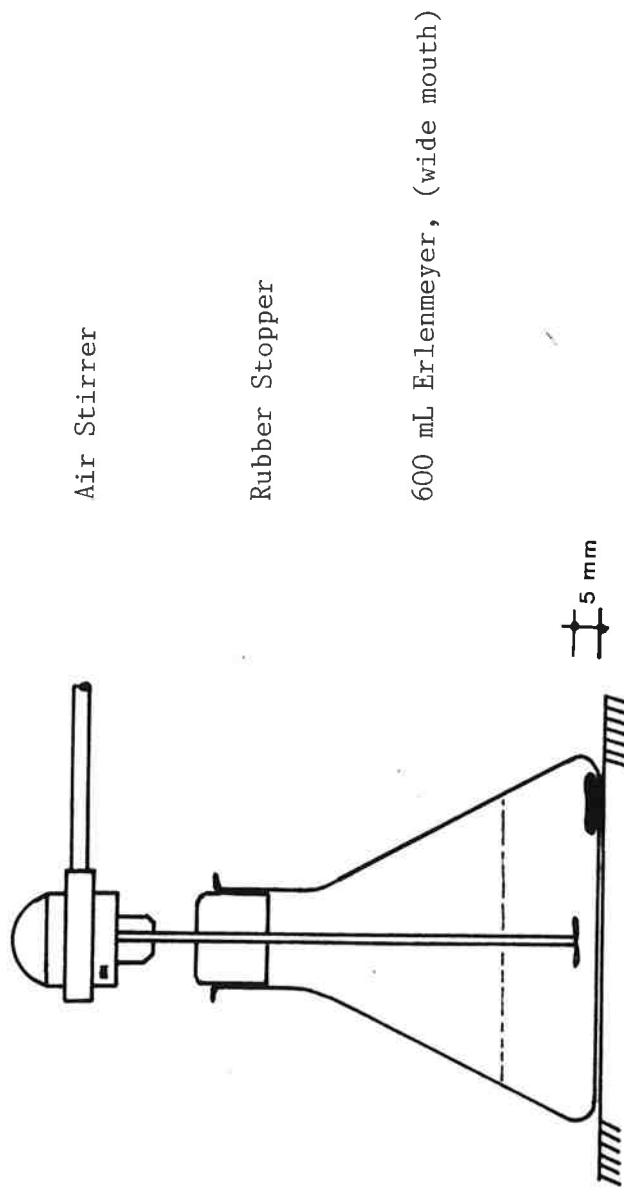


FIGURE C.1. : STIRRING PROCESS

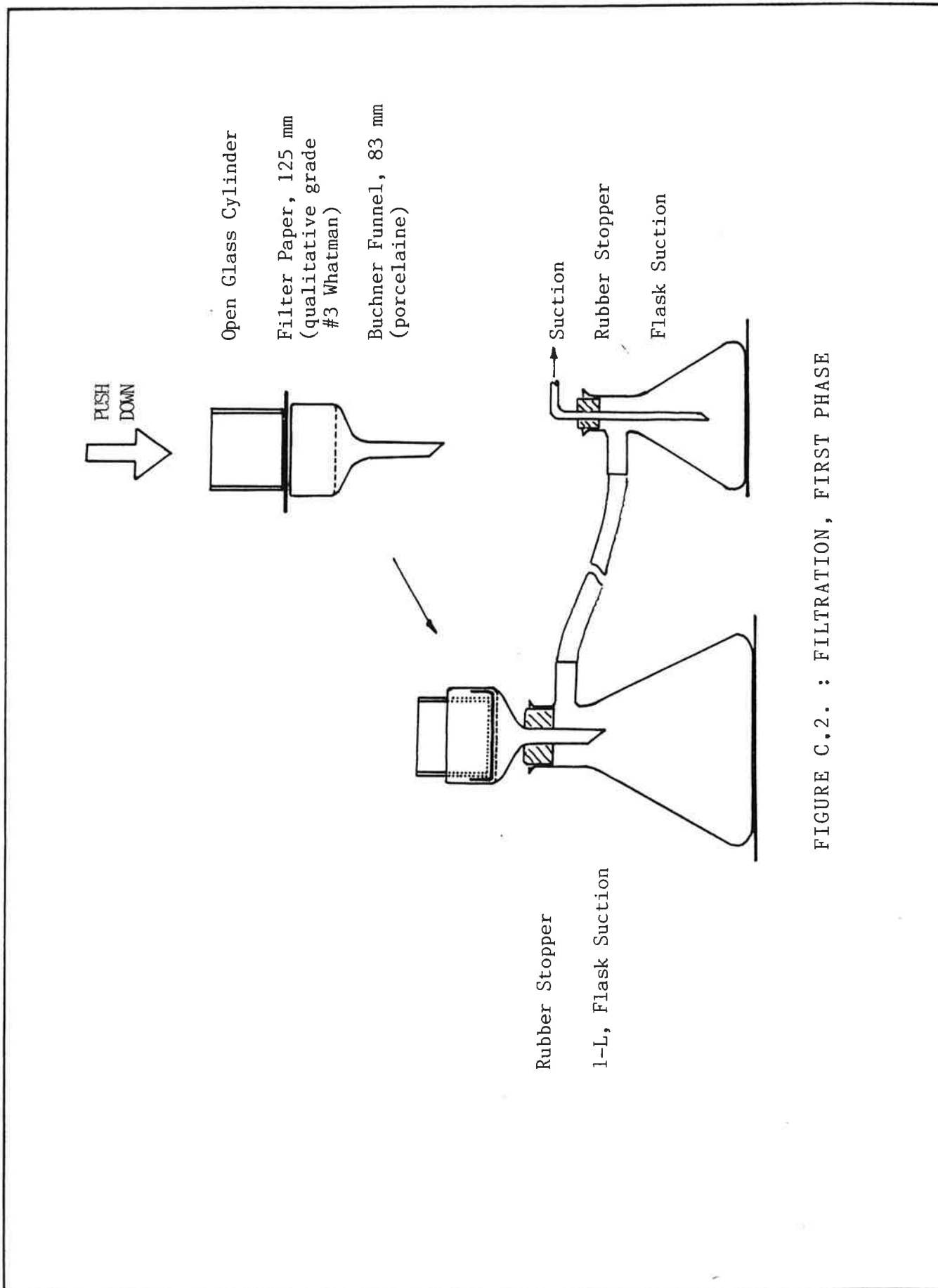


FIGURE C.2. : FILTRATION, FIRST PHASE

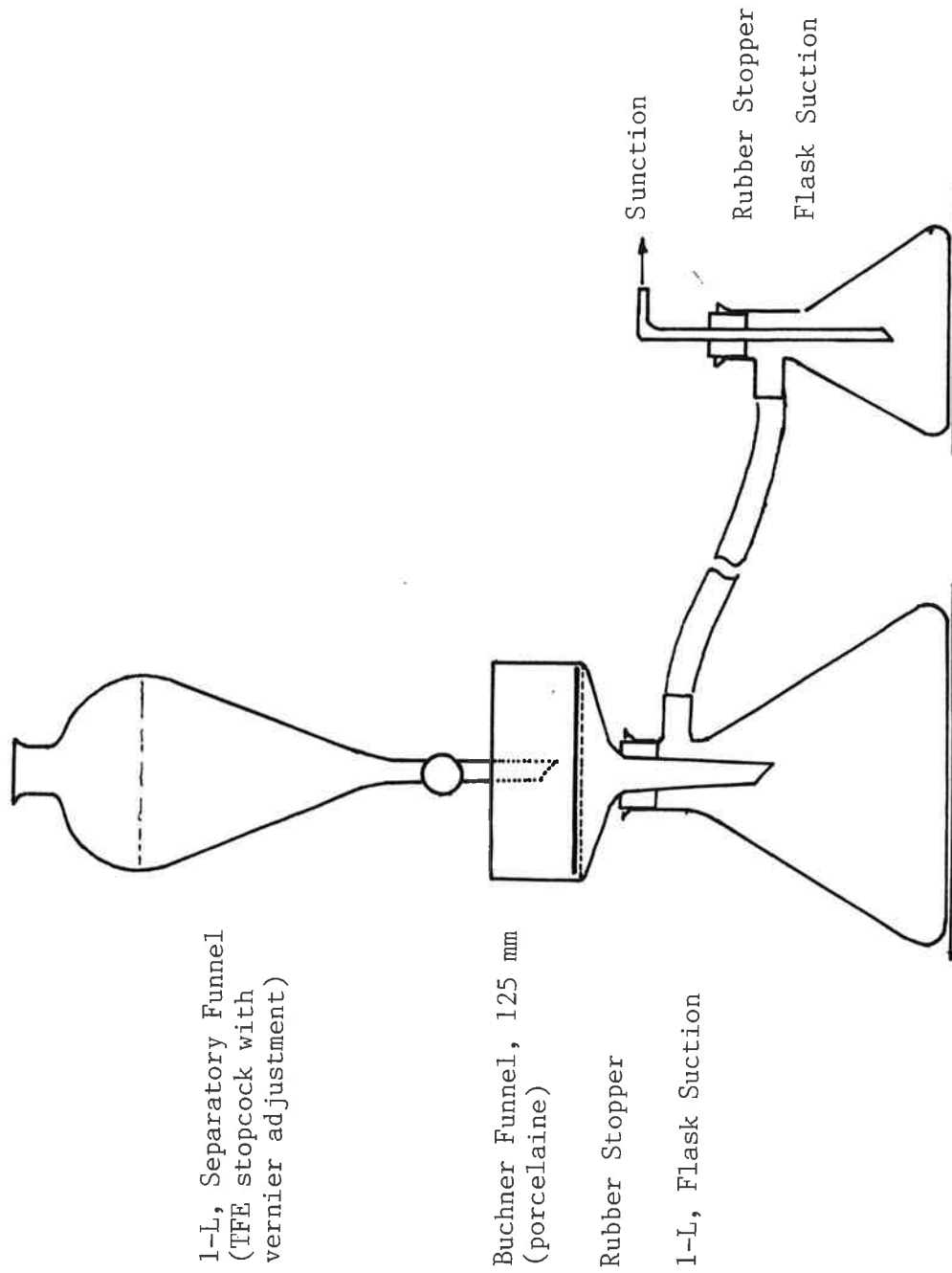


FIGURE C.3. : FILTRATION, SECOND PHASE

Note : Use 24/40  
Standard Taper  
Joints

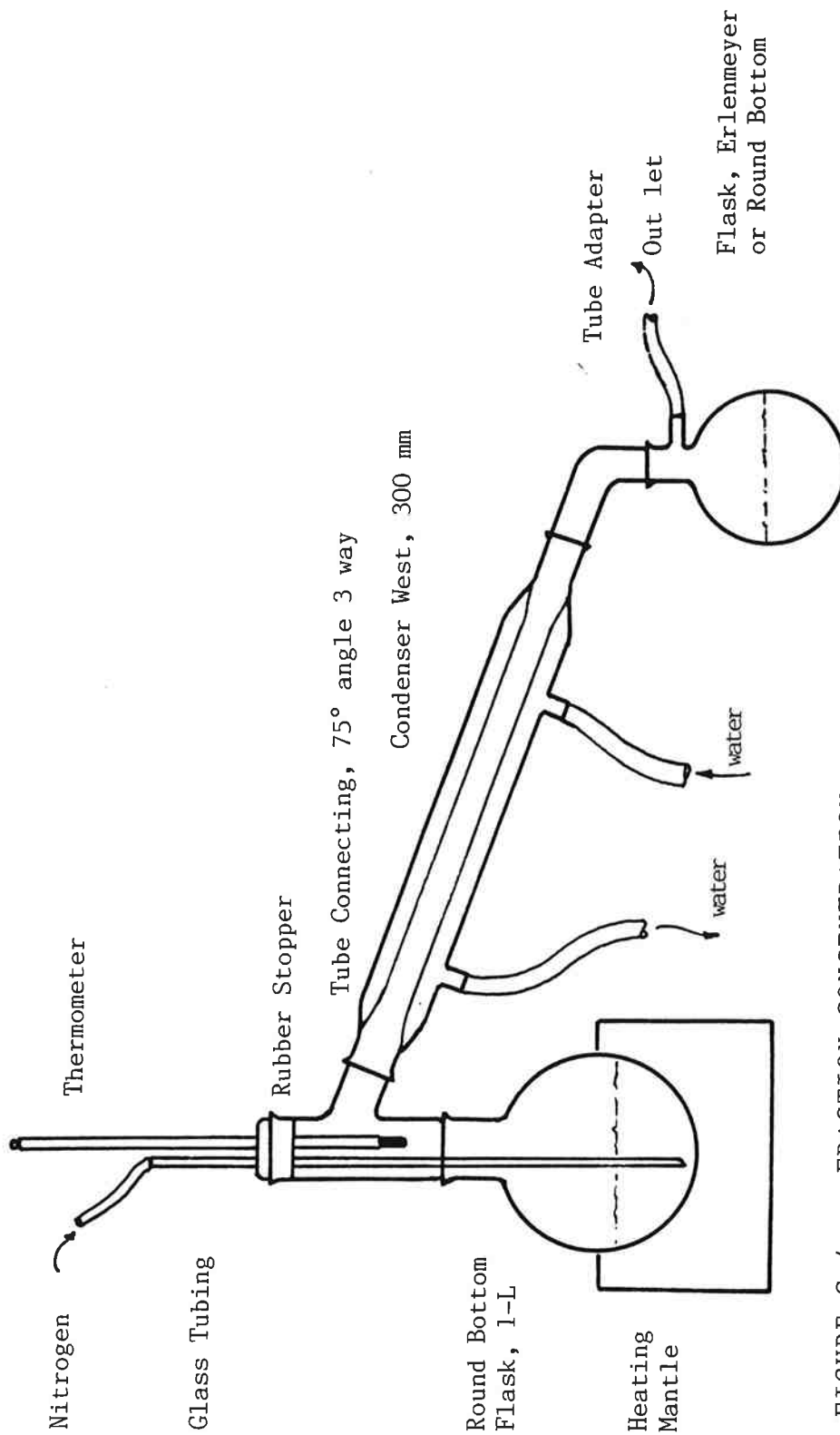


FIGURE C.4. : FRACTION CONCENTRATION  
(Alternative Method)

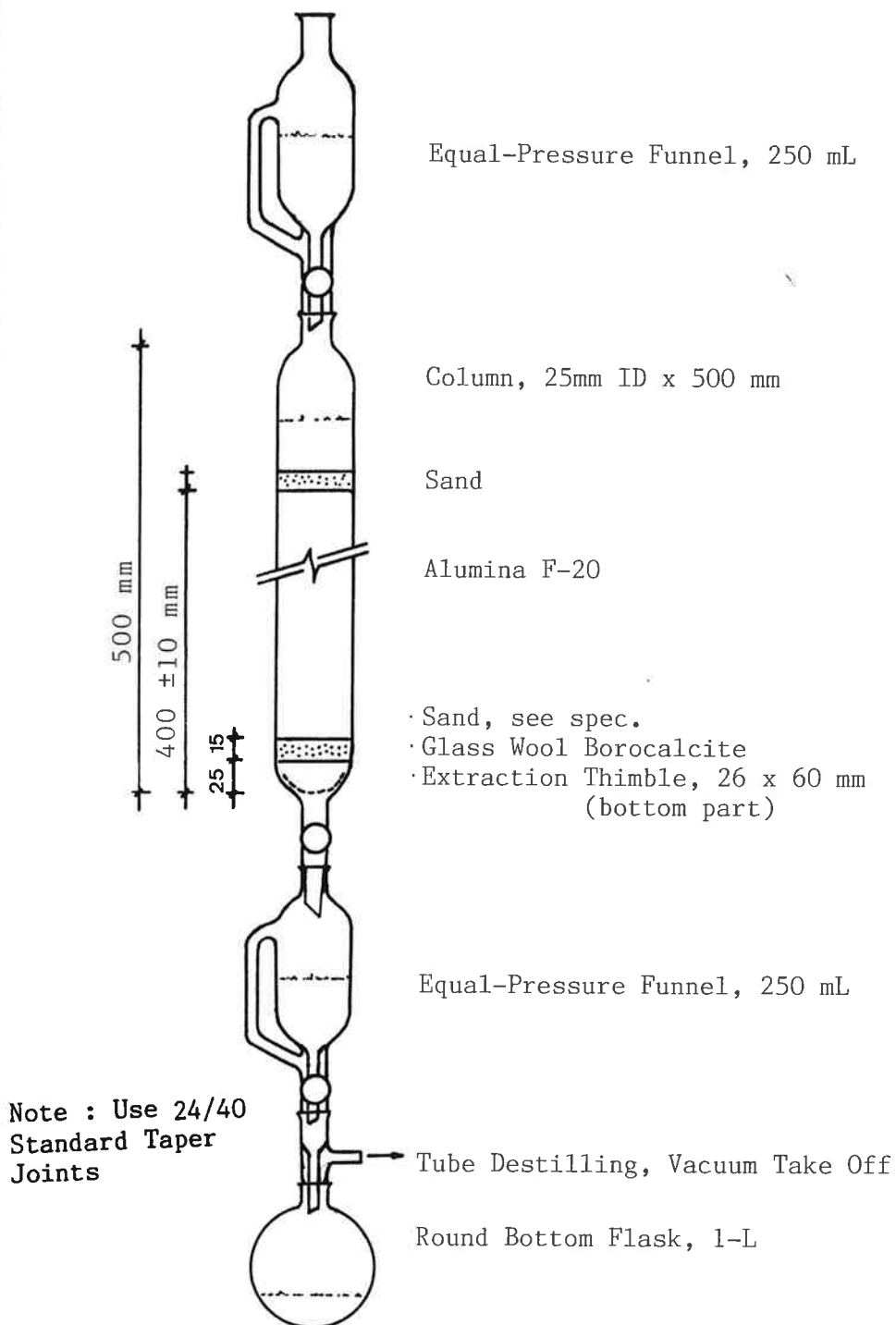


FIGURE C.5. : CHROMATOGRAPHIC COLUMN

APPENDIX D

## APPENDIX D

## PRICE LISTING FOR EQUIPMENT, MATERIALS, AND CHEMICALS

## REQUIRED TO PERFORM ASTM D4124

## (METHOD B, SHORT PROCEDURE)

## 500 mm COLUMN

D.1 INTRODUCTION

The list of equipment and materials required to perform ASTM D4124 Method B have been listed in Table D.1 through D.3. The test procedure is described in Appendix C, and does not correpond to the exact version given in the proposed standard procedure.

D.2 PRICE LISTING

Tables D.1 through D.3 give the list of materials in the following order:

Table D.1: Glassware

Table D.2: Basic Instruments

Table D.3: Chemicals per Test.

D.3 REMARKS

There are other materials that are required that are not listed that depend in the way the instruments are going to be set up. These are:

- \* stands
- \* clamps
- \* ring clamps
- \* rubber tubes for vacuum, pressure, water and nitrogen
- \* cork rings (6)
- \* rubber stoppers



- \* polyethylene wash bottles (2)
- \* steel rod or wood rod 800 mm length
- \* tongs
- \* desiccator

The following facilities should be available in the laboratory:

- \* fume cover
- \* vacuum
- \* air pressure
- \* nitrogen source
- \* balance (min 500 g  $\pm$  0.01 g)
- \* oven
- \* furnaces (415°C).

Table D.1. Glassware

Item No.	Description	Unit	Qty	Unit Price	Total	Purchased From	Remarks
• Stirring:							
1	Erlenmeyer Flask, Pyrex with Scale, 600 mL, Wide Mouth	#	1	8.0	8.0	OSU	Wide mouth custom made to stirrer size.
2	Beaker, Griffin with Scale, 600 mL	#	1	2.0	2.0	OSU	
• Filtering:							
3	Flask Filter, Pyrex with Scale, 1000 mL	#	1	24.0	24.0	OSU	
4	Flask Filter, Pyrex, 500 mL	#	1	12.0	12.0	OSU	
5	Funnel, Separatory, 1000 mL, TFE Stopcock with Vernier Adjustment	#	1	60.0	60.0	OSU	
6	Buchner Funnel, Porcelain, 125 mm 60245 Coors	#	1	38.0	38.0	VWR	
7	Buchner Funnel, Porcelain, 100 mm 60243 Coors	#	1	21.0	21.0	VWR	
8	Glass Cylinder, 60 mm height, I.D. to fit Buchner Funnel 100 mm	#	1	5.0	5.0	OSU	Custom made.
9	Funnel, Short Stem, Pyrex, 60° 125 mm	#	1	10.0	10.0	OSU	
10	Evaporating Dish, Porcelain, 160 mm diam.	#	2	16.0	32.0	OSU	
• Column Chromatography:							
11	Column, 500 mm height, ID 25 mm, 24/40 joints	#	1	85.0	85.0	OSU	Custom made
12	Funnel, Separatory, 250 mL, TFE Fluorocarbon Stopcock	#	2	50.0	100.0	OSU	
13	Tube distilling, Vacuum Take Off 24/40	#	1	28.0	28.0	OSU	
14	Poly Joint Clamp #24	#	2	3.0	6.0	OSU	
15	Round Bottom Flask, 1000 mL, 24/40 Standard Joint	#	3	16.0	48.0	OSU	
• Fraction Concentration:							
Note: If Rotovapor is not available the following glassware is needed.							
16	Round Bottom Flask, 1000 mL, 24/40 Standard Joint	#	2	16.0	32.0	OSU	
17	Condenser West, 300 mm, 24/40	#	1	30.0	30.0	OSU	
18	Tube, Connecting, 3 way, 75°, 24/40	#	1	29.0	29.0	VWR	
19	Tube, Connecting, Distilling Vacuum 105°, 24/40	#	1	50.0	50.0	VWR	
20	Thermometer, min 150°C	#	1	9.0	9.0	VWR	
21	Poly Joint Clamp #24	#	4	3.0	12.0	VWR	
					Total	641.0	

Table D.2. Basic Instruments

Item No.	Description	Unit	Qty	Unit Price	Total	Purchased From	Remarks
1	Stirrer, Air Driven, (Arrow Type A)	#	1	104.0	104.0	A.H. Thomas Co.	#8583-J10
2	Heating Mantles with Controller Electro Thermal to fit 1000 mL Round Bottom Flask	#	1	130.0	130.0	VWR	#33749-743
3	Hot Plate, 750 watt, 120 volts	#	1	66.6	66.6	VWR	#33922-054
4	Rotovapor	#	1	2000.0	2000.0	VWR	Complete with five, 1 liter flasks.

Table D.3. Chemicals and Other Materials to be Used Per Test

Item No.	Description	Unit	Qty	Unit Price	Total	Purchased From	Remarks
1	N-Heptane, 99 Min.Mol%, Pure Grade	l	~ 0.80	7.0	5.60	OSU	
2	Methanol, Anhydrous Reagent Grade	l	0.05	15.0	0.75	VWR	
3	Toluene, Reagent Grade	l	0.20	8.0	1.60	VWR	
4	Trichloroethylene, BP 86.5 to 87.5°C	l	0.20	7.50	1.50	VWR	
5	Alumina, F-20, Chromatographic Grade	k	0.18	20.0	3.60	VWR	
6	Extraction Thimble, 33x94 (Whatman)	#	1	1.2	1.2	VWR	
7	Filter Papers #3 and #1	#	2	-	0.20	VWR	
8	Glass Wool Borocalsite	-	-	-	0.10	-	
				Total	14.55		

APPENDIX E

APPENDIX E

TEST METHODS AND BIBLIOGRAPHY OF LITERATURE DESCRIBING  
STUDIES OF ASPHALT COMPOSITION OR ASPHALT CHEMISTRY

---

As in reference (43) by J.L. Goodrich, 1986.

## TESTS USED TO ANALYZE THE COMPOSITION OF ASPHALT:

- |      |  |   |
|------|--|---|
| I.   | SOLVENT PRECIPITATION  | fraction<br>names   |
|      | a. Solvent precipitation   |   |
|      | 1. Traxler & Schwyer [108b]  | n-butanol asphaltics<br>cyclics<br>paraffinics  |
| II.  | CHEMICAL PRECIPITATION   | fraction<br>names   |
|      | a. Solvent precipitation +<br>chemical precipitation   |   |
|      | 1. Rostler & Sternberg [108a,42]<br>[ASTM D2006]   | asphaltenes (n-pentane insolubles)<br>n-pentane solubles<br>nitrogen bases<br>1st acidifins<br>2nd acidifins<br>paraffins                   |
| III. | LIQUID CHROMATOGRAPHY —adsorption [77]   | fraction<br>names   |
|      | a. Solvent precipitation +<br>adsorption chromatography                                      |   |
|      | 1. Corbett [102,94]<br>(column packing: alumina)   | asphaltenes (n-hexane insolubles)<br>n-hexane petrolenes<br>polar aromatics<br>naphthene aromatics<br>saturates                             |
|      | 2. ASTM D4124<br>(column packing: alumina)   | asphaltenes (n-heptane insolubles)<br>n-heptane petrolenes<br>polar aromatics<br>naphthene aromatics<br>saturates                           |
|      | 3. ASTM D2007 "Clay-Gel"<br>[ASTM D2007]<br>(column packing: attapulgis<br>clay, silica gel) | asphaltenes (n-pentane insolubles)<br>polar compounds<br>aromatics<br>saturates   |
|      | b. HPLC [high pressure LC]   |   |
|      | 1. Hattingh [32]<br>(column packing: silica gel)   | Hattingh separated asphalt into ten solvent-<br>defined fractions.  |
| IV.  | LIQUID CHROMATOGRAPHY — ion exchange [77]  |   |
|      | a. Anion-exchange chromatography   |   |
|      | 1. Boduszynski [70,82,84]  | separated the acidic components [containing<br>carboxylic acids, phenols, pyrroles, 2-quin-<br>olones] into four solvent-defined fractions. |
|      | 2. McKey [73]  |   |
|      | b. Cationic-exchange chromatography  |   |
|      | 1. Boduszynski [70,82,84]  | separated the basic components [containing  |

## 2. McKay [73]

pyridines, amides and pyrroles] into four solvent-defined fractions.

## V. LIQUID CHROMATOGRAPHY — coordination

## a. Ferric chloride coordination chromatography

1. Boduszynski [70,82,84]
2. McKay [73]

separated the neutral Lewis bases from acid and base-free asphalt [see ion-exchange LC]. IR analysis showed that amides and pyrroles were the dominant polar groups in this fraction.

## VI. THIN-LAYER CHROMATOGRAPHY (TLC)

## a. Thin-layer chromatography (TLC)

1. Masek [105a]
2. Altgelt [77]
3. Poirier [106a]
4. Ray [106b]

TLC has been used to detect the presence of benzo-[a]pyrene in distillate residues. High resolution HPLC has substantially replaced the two-dimensional TLC techniques. Poirier and Ray report using TLC "chromarods", quartz rods with a sintered layer containing silica gel or alumina as the adsorption medium. The "chromarods" are scanned by the flame of a flame ionization detector. The method has been compared to a D2007 separation [106b].

## VII. GAS-LIQUID CHROMATOGRAPHY (GLC)

## a. Gas chromatography (GC)

1. Altgelt [77]
2. Puzinauskas [?]
3. Botvin'eva [55]
4. Asphalt Institute [66a]

Very useful for analysing low-to-medium boiling range hydrocarbons. GC was used to analyze the hydrocarbon emissions from asphalt hot mixes. GC can be used to identify the "light ends" from a vacuum distillation of asphalt. GC coupled with mass spectroscopy is a powerful analytical tool for identifying compounds at very low concentrations.

## b. Inverse gas-liquid chromatography (IGLC)

1. Robertson [92]
2. Davis & Petersen [9]
3. Dorrence & Petersen [10]

Used asphalt as the stationary phase (coated on Teflon media) and various solvents as the mobile phase). IGLC tests on Zaca-Wigmore asphalts correlated well with road performance.

## VIII. SIZE EXCLUSION CHROMATOGRAPHY (SEC) [77]

## a. Gel permeation chromatography (GPC)

1. Altgelt [80,79,78]
2. Long [72]
3. Dickie & Yen [71a]
4. Marvillet [51]
5. Winniford [75]

separates compound based on molecular aggregate size—largest size is eluted from the Styragel column first. Time defined fractions are collected. Used to estimate molecular mass based on the retention time of polystyrene standards.

## b. High pressure GPC (HP-GPC)

1. Bynum [85]
2. Jennings [87-91]

HP-GPC chromatograms result in an apparent molecular aggregate size profile of the sample.



3. Hattingh [32]
4. Plummer [97]
5. Boduszynski [84,69]

Winniford cautions about asphaltene associations [aggregations] indilute solvent solutions. Molecular masses within an asphalt as measured by GPC is 300 to 100,000 daltons. Jennings believes that road cracking is related to the "LMS" fraction determined from the GPC chromatogram. Plummer finds that the cracking is not related to a large/small imbalance; rather, cracking is related to the mean molecular size. Both Jennings and Plummer find that the cracked roads have harder asphalts (penetration/ductility).

#### IX. VAPOR PRESSURE OSMOMETRY (VPO)

- a. VPO (i.e., ASTM D3592)
  1. Boduszynski [69]
  2. Ferris [57]
  3. Speight [66,74]
  4. Long [72]
  5. Marvillet [51]

Used to estimate molecular weight by measuring the heat of absorption of solvent vapor into a solution of the asphalt in the same solvent. Ave. molecular mass of D4124 asphalt fractions by VPO: saturates = 1000, asphaltenes = 4000.

#### X. MASS SPECTROMETRY (MS) [101a]

- a. low resolution MS
  1. Clerc [55a]
- b. high resolution MS
  1. Dickie & Yen [71a]
  2. Gallegos [58]
  3. Asphalt Institute [66a]
- c. field ionization MS (FIMS)
  1. Boduszynski [69,70,81]

Mass spectrometry is used to determine the mass of individual molecules. In a mass spectrometer asphalt is bombarded with electrons and forms molecular ions which are accelerated and deflected in a magnetic field. The detection of the ion will give rise to a peak in the spectrum at the mass of the molecular weight. The FIMS allows resolution of high molecular masses, (ave. molecular mass of D4124 asphalt fractions: saturates = 1000, asphaltenes = 900)

#### XI. ELECTROPHOTOMETRIC SPECTROSCOPY

- a. Infrared spectroscopy and differential IR spectroscopy
  1. Dorrence [10]
  2. Petersen [64,63,106]
  3. Barbour [8]
  4. Martin [28]
  5. Boduszynski [70]
  6. Plancher [39]
  7. Speight [74]
  8. Asphalt Institute [66a]
- b. Ultraviolet spectroscopy
  1. Botvin'eva [55]
  2. Corbett [102]
  3. Asphalt Institute [66a]

Petersen, et.al. have developed methods for quantitatively measuring the types of polar compounds in asphalt which absorb in the carbonyl region (1850 to 1640  $\text{cm}^{-1}$ ). Measurement of ketones, 2-quinolone, carboxylic acids and dicarboxylic anhydrides allowed estimation of the mechanism of asphalt oxidation and viscosity increase. Sulfoxides were measured at 1030  $\text{cm}^{-1}$ . Speight used IR to indicate hydrogen bonding in asphalt solutions.

Botvin'eva studied saturated hydrocarbons (n-, iso- and cyclo paraffins). Adsorption at 270,286, and 258  $\mu$ -microns was used to detect 1,2 and 3 ring aromatics in the saturate and naphthene aromatic fractions [102].

- XII. NUCLEAR MAGNETIC RESONANCE (NMR) SPECTROSCOPY**
- a. NMR Spectroscopy (1H & 13C)**
1. Couper [102a]
  2. Plummer [97]
  3. Corbett [102,34]
  4. Paukku [62]
  5. Ferris [57]
  6. Altgelt [78,79,54]
- Both proton and carbon-13 NMR have been used to estimate saturate/aromatic carbon ratios. Boduszynski cautions against proton NMR when analysing asphalt fractions [70a]. Paukku reports determining the relative distributions of methyl, methylene and methyne groups, saturated groups in alpha position with respect to the aromatic ring and aromatic structures by integration of the PNMR bands in the regions of 0.5-1, 1-2, 2-5, and 6-8.5 ppm, respectively.
- XIII. ELECTRON SPIN RESONANCE (ESR) SPECTROSCOPY**
- a. ESR Spectroscopy**
1. Ferris [57]
  2. Yen [67]
  3. Corbett [102]
- Measures the abundance of free radical sites. Ferris found a correlation between nitrogen content and ESR peaks.
- XIV. SPECTROCHEMICAL ANALYSIS**
- a. X-ray fluorescence spectroscopy
  - b. Neutron activation analysis
  - c. X-ray diffraction
    1. Ferris [57]
    2. Yen [67]
- determination of heavy metal content. provides elemental analysis. Used to estimate the structure of asphaltenes.
- XV. ELEMENTAL ANALYSIS**
- a. Elemental analysis**
1. Marvillet [51]
- A variety of techniques are used to determine the elemental analysis of asphalt. Elements most often measured include hydrogen, carbon, nitrogen, sulfur and oxygen. The analysis usually involves pyrolysis (combustion or reduction), purification of the resultant gasses, and detection by IR, coulometric titration, chemiluminescence, etc.
- XVI. DISTILLATION FRACTIONATION**
- a. Thin-film, high vacuum distillation
  - b. Thermogravimetric analysis
- permits obtaining samples of narrow distillation cuts for further analysis. obtains a distillation profile on a milligram-scale sample...effluent can be in-line detected/analysed.
- XVII. WAX CONTENT**
- a. Methylene chloride**
1. Evans [48]
- Wax has the opposite effect of asphaltenes on asphalt rheology...wax decreases the PVN [77,140] making asphalt more temperature susceptible.

## XVIII. PHOTOCHEMICAL REACTIONS OF ASPHALT

## a. Traxler procedure (UV box)

1. Traxler [43]
2. Predoshl [30]

Thin films of asphalt (5 microns) showed an increase in viscosity correlated to RTFC viscosity increases. Thick films (20 microns) showed little differences between asphalts of differing composition (D2007).

## XIX. ACID NUMBER

## a. Neutralization Number (ASTM D664)

1. Marvillet [51]

May have an effect on asphalt emulsification.

## XX. INTERNAL DISPERSION STABILITY

## a. Heithaus parameter, P

1. Heithaus [98]
2. Petersen [37]

The Heithaus "state of peptization" (P) is an attempt to measure the intercompatibility of the components in asphalt...how good a dispersant are the maltenes for the asphaltenes (Po)?...and how readily are the asphaltenes dispersed (Pa)?  $P = Pa/(1-Pa)$ . Peterson commented that the "P" of the Zaca-Wigmore asphalts correlated with performance better than did the Rostler ratio.

## b. Oliensis Spot Test

1. Oliensis [99a,99b]
2. Heithaus [99]

This test was designed to identify the presence of cracked asphalt...resulting in a positive spot. Considered by Heithaus, in 1959, to be no longer a useful test: a 24 hour test did correlate with road performance.

## c. Asphaltene settling test

1. Plancher [100]

In this test a 2gm sample of asphalt is digested in 50 ml of n-hexane for 24 hrs. The settlement of the asphaltenes in an undisturbed cylinder is then observed. Settling time is defined by when the asphaltenes have settled to half the solution height. Test precision is a problem.

## XXI. TITRIMETRIC/GRAVIMETRIC ANALYSIS

1. Cheronis [55b]

A wealth of analytical techniques is available for the analysis of asphalt. Although many of the "wet" chemistry techniques have been replaced with instrumental analysis, still others survive: i.e. acid number (by titration), salt or mineral filler content (by ash), water content (Karl Fischer titration).

## BIBLIOGRAPHY

## MICHIGAN TEST ROADS — 1954

- 1 Corbett, L.W., "Asphalt Composition Analysis, Its Significance and Use", presented to ASTM Subcommittee D04.47, Philadelphia, PA, June 1979.
- 2 Corbett, L.W. and Schwyer, H.E., "Composition and Rheology Considerations in Age Hardening of Bitumen", Proc. of AAPT, Vol.50 (1981), pp. 573-581.
- 3 Corbett, L.W. and Merz, R.E., "Asphalt Binder Hardening in the Michigan Test Road After 18 Years of Service", TRB Record 544, 1975, pp. 27-34.
- 4 Parr, W.K. and Serafin, P.J., "Four Year Test Trends on the Michigan Bituminous Experimental Road", Proc. of AAPT, Vol. 28 (1959), pp. 295-315.
- 5 Parr, W.K., Serafin, P.J. and Humphries, T., "Michigan State Highway Experimental Bituminous Construction Project", Proc. of AAPT, Vol.24 (1955), pp. 125-171.

## TEXAS TEST ROADS — 1954-1959

- 6 Galloway, B.M., "Factors Relating Chemical Composition and Rheological Properties of Paving Asphalts with Durability", Proc. of AAPT, Vol.28 (1959), pp. 280-294.
- 7 Traxler, R.N., "Relation Between Hardening and Composition of Asphalt", Preprints Div. of Petroleum Chemistry, American Chemical Society, Vol.5, No.4-A (1960), pp. A-71 to A-77.

## ZACA-WIGMORE TEST ROAD — 1954-1964

- 8 Barbour and Petersen, "Molecular Interactions of Asphalt: An Infrared Study of the Hydrogen-Bonding Basicity of Asphalt," Analytical Chemistry, Vol. 46 No. 2, February 1974, pp.273-277.
- 9 Davis, T.C. and Petersen, J.C., "An Inverse GLC Study of Asphalts Used in the Zaca-Wigmore Experimental Road Test," Proc. of AAPT, Vol 36 (1967), pp. 1-10.
- 10 Dorrence, Barbour and Petersen, "Direct Evidence of Ketones in Oxidized Asphalts", Analytical Chemistry, Vol. 46 No. 14, December 1974, pp. 2242-2244.
- 11 Hveem, F.N., Zube, E. and Skog, J., "Progress Report on the Zaca-Wigmore Experimental Asphalt Test Project", ASTM STP No. 277, 1960, pp. 3-46.
- 12 Petersen, J.C., "Chemical Composition of Asphalt as Related to Asphalt Durability—State of the Art," TRB 1984 preprint, 42 pp.
- 13 Santucci, L.E. and Schmidt, R.J., "The Effect of Asphalt Properties on the Fatigue Resistance of Asphalt Paving Mixtures", Proc. of AAPT, (1969), pp. 65-97.
- 14 Schmidt, R.J. and Santucci, L.E., "The Effect of Asphalt Properties the Fatigue Cracking of Asphalt Concrete on the Zaca-Wigmore Test Project", Proc. of AAPT (1969), pp. 39-60.
- 15 Simpson, W.C., Griffin, R.L. and Miles, T.K., "Correlation of the Microfilm Durability Test with Field Hardening Observed in the Zaca-Wigmore Experimental Project", ASTM STP No. 277, 1960, pp. 52-63.
- 16 Skog, J.B., "Effect of Changes in Asphalt Properties on Pavement Performance: Zaca-Wigmore Test Road," Proc. of AAPT, Vol. 50 (1981), pp. 538-545.
- 17 Skog, J., "Results of Cooperative Test Series on Asphalts from the Zaca-Wigmore Experimental Project", ASTM STP No. 277, 1960, pp. 46-51.
- 18 Zube, E. and Skog, J., "Final Report on the Zaca-Wigmore Asphalt Test Road", Proc. of AAPT, Vol.38 (1969), pp. 1-38.

## PENNSYLVANIA TEST ROADS — 1961-1976

- 20 Kandhal, P.S. and Koehler, W.C., "Significant Studies on Asphalt Durability: Pennsylvania Experience", Pennsylvania Department of Transportation presented at the Symposium on Asphalt Durability at the TRB Meeting, January 1984.
- 19 Kandhal, P.S., Mellott, D.B. and Basso, H.R., "Durability Study of Viscosity Graded AC-20 Asphalts in Pennsylvania", Pennsylvania Department of Transportation, April 1984.
- 21 Kandhal, P.S., "Low-Temperature Ductility in Relation to Pavement Performance" in Low-Temperature Properties of Bituminous Materials and Compacted Bituminous Paving Mixtures, ASTM STP 628, 1977, pp. 95-106.

## EUROPEAN TEST ROADS — 1963-1964

- 22 Chipperfield, E.H., et.al., "Asphalt Characteristics in Relation to Road Performance", Proc. of AAPT, Vol.39 [1970], pp. 575-613.
- 22a Chipperfield, E.H. and Welsh, T.R., "Studies on the Relationship between the Properties of Road Bitumens and Their Service Performance", Proc. of AAPT, Vol.36 [1967], pp. 421-488.

## TEXAS TEST ROADS [1963-1966]

- 85 Bynum, D. and Traxler, R.N., "Gal Permeation Chromatography Data on Asphalts Before and After Service in Pavements", Proc. of AAPT, Vol.39 [1970], pp. 683-702.

## CALIFORNIA TEST ROADS — 1964-1973

- 23 Kemp, G.R., "Asphalt Durability Tests and Their Relationship to Field Hardening", ASTM STP 532, 1973, pp. 100-122.
- 24 Schmidt, R.J., "Laboratory Measurement of the Durability of Paving Asphalts", ASTM STP 532, 1973, pp. 79-99.
- 25 Skog, J., "'Setting' and Durability Studies on Paving Grade Asphalts", Proc. of AAPT, Vol. 36, pp. 387-420.

## AUSTRALIAN TEST ROADS — 1966-1976

- 26 Dickinson, E.J., "The Hardening of Middle East Petroleum Asphalts in Pavement Surfacing", Proc. of AAPT, Vol. 49 [1980], pp. 30-64.
- 27 Haley, G.A., "Changes in Chemical Composition of a Kuwait Short Residue During Air Blowing," Analytical Chemistry, Vol. 47, No. 14, Dec. 1975, pp. 2432-2437.
- 28 Martin, C.L. and Bowering, R.H., "The Durability of Various Asphalts in Pavement Seals in South Eastern Australia", Proc. of AAPT, Vol. 50 [1981], pp. 417-441.

## CALIFORNIA ASPHALT DURABILITY STUDY — 1974-1980

- 29 Kemp, G.R. and Predoehl, N.H., "A Comparison of Field and Laboratory Environments on Asphalt Durability", Proc. of AAPT, Vol. 50 [1981], pp. 492-537.
- 30 Predoehl, N.H. and Kemp, G.R., "An Investigation of the Effectiveness of Asphalt Durability Tests - Initial Phase", FHWA-CA-TL-78-26, California DOT, August 1978.
- 31 Santucci, L.E., Goodrich, J.E., Sundberg, J.E., "The Effect of Crude Source and Additives on the Long-Term Oven Aging of Paving Asphalts", Proc. of AAPT, Vol. 50 [1981], pp. 560-571.

## MONTANA STUDY

- 87 Jennings, P.W., Pribanic, J.A.S. and Dawson, K.R., "Use of HPLC to Det-

- ermine the Effects of Various Additives and Fillers on the Characteristics of Asphalt" in a paper prepared for the Montana Department of Highways and U.S. DOT Federal Highway Admin., June 1982, 24 pp.
- 88 Jennings, P.W., Pribanic, J.A.S., Dawson, K.R. and Bricca, C.E., "Use of HPLC and NMR Spectroscopy to Characterize Asphaltic Materials", Preprints Div. of Petrol. Chem., American Chemical Society, Vol. 26, No. 4, Sept. 1981, pp. 915-922.
- 90 Jannings, P.W., et. al., "Chemical Composition of Commercial Asphalt Cement as Determined by High Pressure Liquid Chromatography", FHWA Report No. MT-7929, Department of Chemistry, Montana State University, Bozeman, Montana, December 1977.
- 89 Jennings, P.W., Pribanic, J., et.al., "High Pressure Liquid Chromatography as a Method of Measuring Asphalt Composition", FHWA Report No. MTD0H-7930, Department of Chemistry, Montana State University, Bozeman, Montana, March 1980.
- 91 Montana State Department of Highways special provision, "Requirements for the Gel Permeation Mode (HP-GPC) of Asphalt Cement", Nov. 14, 1983.

## SOUTH AFRICA — 1984

- 32 Hattingh, M.M., "The Fractionation of Asphalt," paper prepared for AAPT meeting, January 1984.

## COMPOSITION and ASPHALT DURABILITY

- 33 Corbett, L.W. and Schweyer, H.E., "Composition and Rheology Considerations in Age Hardening of Bitumen", Proc. of AAPT, Vol.50 [1981], pp. 571-582.
- 34 Corbett, L.W. and Swarbrick, R.E., "Compositional Analysis Used to Explore Asphalt Hardening", Proc. of AAPT, Vol.29 [1960], pp. 104-114.
- 34a Davis, T.C. and Petersen, J.C., "Inverse GLC—An Extension of the Technique to The Study of Oxidation of Asphalts", Highway Research Record No. 134, Highway Research Board, National Academy of Sciences [1966], pp. 1-7.
- 34b Gotolski, Ciesielski, Smith and Kofelt, "Study of Physical Factors Affecting the Durability of Asphalt Pavements", Highway Research Record No. 231, Highway Research Board, National Academy of Sciences [1968], pp. 1-23.
- 34c Greenfield, S.H., "Chemical Changes Occurring During the Weathering of Two Coating-Grade Asphalts", Preprints of the Div. of Petrol. Chem., American Chemical Society, Vol.5, No.4 [1960], pp. A-89 to A-106.
- 35 Knowles, E.C., McCoy, F.C., et.al., "Relation of Asphalt Composition to its Durability in Service", Preprints Div. of Petroleum Chem., American Chemical Society, Vol.3 No.2 [1958], pp. A-29-37.
- 36 Peters, R.J., "Compositional Considerations of Asphalt for Durability Improvement", TRB Record 544, 1975, pp. 46-55.
- 37 Petersen, J.C., "Chemical Composition of Asphalt as Related to Asphalt Durability—State of the Art", paper presented at TRB, January 1984.
- 38 Petersen, J.C., "Relationships Between Asphalt Chemical Composition and Performance-Related Properties", paper presented at AEMA, March 1982.
- 39 Plancher, H., Green, E.L. and Petersen, J.C., "Reduction of Oxidative Hardening of Asphalts by Treatment with Hydrated Lime—A Mechanistic Study", Proc. of AAPT, Vol.45 [1976], pp. 1-24.
- 40 Rostler, F. and Rostler, K.S., "Basic Considerations in Asphalt Research Pertaining to Durability", Proc. of AAPT, Vol. 50 [1981], pp. 582-621.
- 41 Rostler, F.S. and White, R.M., "Composition and Changes in Composition of Highway Asphalts, 85-100 Penetration Grade", Proc. of AAPT, No.31 [1962], pp. 35-89.
- 42 Rostler, F.S. and White, R.M., "Influence of Chemical Composition of

- Asphalts on Performance, Particularly Durability" in ASTM STP No. 277, [1959], pp. 64-88.
- 43 Traxler, R.N., "Durability of Asphalt Cements", Proc. of AAPT, Vol.32 [1963], pp. 44-63.
- 43a Welborn, J.Y., "The Fact and Fallacy of Asphalt Hardening", Proc. Canadian Technical Asphalt Association, paper presented November 1969.

## COMPOSITION and ASPHALT RHEOLOGY

- 44 Anderson, D.A. and Dukatz, E.L., "Relationship Between Asphalt Flow Properties and Asphalt Composition", presented at AAPT, April 1984.
- 45 Anderson, D.A. and Dukatz, E.L., "Asphalt Properties and Composition: 1950-1980", Proc. of AAPT, Vol.49 [1980], pp. 1-29.
- 46 Bransky, D.W., et.al., "Effect of Composition on Properties of Paving Asphalt", presented at Div. of Petroleum Chem., American Chemical Society, Vol.3, No.2 [1958], pp. A5-A12.
- 46a Button, J.W., Epps, J.A., Little, D.W. and Gallaway, B.M., "Influence of Asphalt Temperature Susceptibility on Pavement Construction and Performance", Texas Transportation Institute, Nov. 1983, rev. June 1984.
- 47 Corbett, L.W., "Relationship between Composition and Physical Properties of Asphalt", Proc. of AAPT, Vol.39 [1970], pp. 481-491.
- 48 Evans, J.V., et.al., "Contributions of Components to Asphalt Consistency", [resented to Div. of Petroleum Chem., American Chemical Society, Vol.16, No.1 [1971], pp. D5-D17.
- 49 Griffin, R.L., et.al., "Influence of Composition of Paving Asphalt on Viscosity, Viscosity-Temperature Susceptibility, and Durability", presented at Div. of Petroleum Chem., American Chemical Society, Vol.3, No.2 [1958], pp. A13-A27.
- 49a Hughes, E.C. and Hardman, H.F., "Some Correlations of Asphalt Composition with Physical Properties", Proc. of AAPT, Vol. 20 [1951], pp. 1-22.
- 50 Kennedy, T.W., Cummings, L.O and White, T.D., "Changing Asphalt Through Creation of Metal Complexes", Proc. of AAPT, Vol.50 [1981], pp. 442-468.
- 50a Lamb, C.W. and Couper, J.R., "Relationship of Flow Properties to Chemical Composition for Paving Asphalts", Highway Research Record No. 231, Highway Research Board, National Academy of Sciences, pp. 24-33.
- 51 Marvillet, J., "Influence of Asphalt Composition on Its Rheological Behavior", Proc. of AAPT, Vol.44 [1975], pp. 416-443.
- 52 Simpson, W.C., Griffin, R.L., et.al., "Relationship of Asphalt Properties to Chemical Constitution", J. Chem. Eng., Vol.6, No.3, [July 1961], pp. 426-429.
- 52a Tingle, E.D. and Green, E.H., "Changes in Bitumen Composition Causing Changes in Viscosity", Highway Research Record No. 134, Highway Research Board, National Academy of Sciences, pp. 51-62.
- 53 Welborn, J.Y., "Physical Properties as Related to Asphalt Durability: State-of-the-Art", presented to TRB, January 1984.

## COMPOSITION: MOLECULAR STRUCTURE &amp; CHEMICAL IDENTIFICATION

- 54 Altgelt, K.H., "Integrated Structural Analysis. A Method for the Determination of Average Structural Parameters of Petroleum Heavy Ends", Anal. Chem., Vol.42, October 1970, pp. 1330-1339.
- 55 Botvin'eva, A.M., et.al., "Composition of Paraffinic and Naphthenic Hydrocarbons in Petroleum Asphalts", Khimiya i Tekhnologiya, No.4 [1982], pp. 32-34.
- 55a Cherone, N.D. and Ma, T.S., Organic Functional Group Analysis by Micro and Semimicro Methods, Interscience, John Wiley & Sons, New York, 1964.
- 55b Clerc, R.J. and O'Neal, M.J., "The Mass Spectrometric Analysis of

- Asphalt - A Preliminary Investigation" in Symposium on Fundamental Nature of Asphalt, Div. of Petroleum Chem., American Chemical Society, New York, Vol.5, No. 4-A, September 1960.
- 56 Dorrance, S.M., Barbour, F.A. and Petersen, J.C., "Direct Evidence of Ketones in Oxidized Asphalts", *Anal. Chem.*, Vol.46, No.14, December 1974, pp. 2242-2244.
- 57 Ferris, S.W., et.al., "Aromatic Structure in Asphalt Fractions", *I&EC Prod. R&D*, Vol.6, No.2, June 1967, pp. 127-132.
- 58 Gallegos, E.J., "High Resolution Mass Spectrometry of Asphalt Fractions", *Proc. 7th World Petroleum Congress*, Vol.4 (1967), pp. 249-260.
- 59 Hall, G and Herron, S.P., "Size Characterization of Petroleum Asphaltenes and Maltenes", presented Div. of Petroleum Chemistry, American Chemical Society, Vol.24, No.4 (1979), pp. 924-934.
- 60 Labout, J.W.A., "Constitution of Asphaltic Bitumen" in *The Properties of Asphaltic Bitumen* (Ed. J.Ph. Pfeiffer), Elsevier, New York (1950), pp. 13-48.
- 61 Labout, J.W.A., "Chemical Reactivity of Asphaltic Bitumen" in *The Properties of Asphaltic Bitumen* (Ed. J.Ph. Pfeiffer), Elsevier, New York (1950), pp. 109-120.
- 61a Durisson, G. Albracht, P. and Rohmer, M., "Microbial Origin of Fossil Fuels", *Scientific American*, Vol.250, No.8 (Aug. 1984), pp. 44-51.
- 62 Paukku, A.N., et.al., "Investigation of Chemical Composition of Polar Components in the Resinous-Asphaltene Substances of Petroleum", *Neftekhim*, Vol.21, No.4 (1981), pp. 625-629.
- 63 Petersen, J.C. and Plancher, H., "Quantitative Determination of Carboxylic Acids and Their Salts and Anhydrides in Asphalts by Selective Chemical Reactions and Differential Infrared Spectrometry", *Anal. Chem.*, May 1981, pp. 786-789.
- 64 Petersen, J.C., Barbour, F.A. and Dorrance, S.M., "Identification of Dicarboxylic Anhydrides in Oxidized Asphalts", *Anal. Chem.*, Vol.47, No.1, January 1975, pp. 107-111.
- 65 Plancher, H., Dorrance, S.M. and Petersen, J.C., "Identification of Chemical Types in Asphalts Strongly Adsorbed at the Asphalt-Aggregate Interface and Their Relative Displacement by Water", *Proc. of AAPT*, Vol.46 (1977), pp. 151-175.
- 66 Speight, J.G., et.al., "Some Observations on the Molecular 'Nature' of Petroleum Asphaltenes", presented Div. of Petrol. Chem., American Chemical Society, Vol.24, No.4 (1979), pp. 910-923.
- 66a The Asphalt Institute, Asphalt Hot-Mix Emission Study, Research Report 75-1 (RR-75-1), College Park, Maryland, March 1975.
- 67 Yan, T.F. and Dickie, J.P., "The Compactness of the Aromatic Systems in Petroleum Asphaltics", presented at Div. of Petroleum Chem., American Chemical Society, Vol.11, No.3 (1966), pp. 49-54.

## COMPOSITION OF ASPHALTENES

- 68 Altgelt, K.H., Harle, O.L., "The Effect of Asphaltenes on Asphalt Viscosity", *Ind. Eng. Chem., Prod. Res. Dev.*, Vol.14, No.4 (1975) pp. 240-246.
- 69 Boduszynski, M.M., "Asphaltenes in Petroleum Asphalts, Composition and Formation" in *Chemistry of Asphaltenes* (Ed. J.W. Bunger, N.C. Li) *Advances in Chemistry Series 195*, Chap.7, American Chemical Society, Washington D.C., 1981.
- 70 Boduszynski, M.M., McKay, J.F. and Latham, D.R., "Asphaltenes, Where Are You" in *Proc. of AAPT*, Vol. 49 (1980), pp. 123-143.
- 70a personal conversations with M.M. Boduszynski, July/August 1984.
- 71 Boduszynski, M.M., "Asphaltenes in Petroleum Asphalts—Composition and



- Formation", presented Div. of Petrol. Chem., American Chemical Society, Vol.24, No.4 [1979], pp. 935-948.
- 71a Dickie, J.P. and Yen, T.F., Analytical Chem., Vol.39, No. 14 [1967], pp. 1847.
- 72 Long, R.B., "The Concept of Asphaltene", presented Div. of Petrol. Chem., American Chemical Society, Vol.24, No.4 [1979], pp. 891-900.
- 73 McKay, J.F., Amend, P.J., et.al., "Petroleum Asphaltene—Chemistry and Composition", presented at Div. of Petroleum Chem., American Chemical Society, Vol.22, No.2 [1977], pp.708-715.
- 74 Speight, J.G., "The Structure of Petroleum Asphaltene—Current Concepts", Alberta Research Council, Information Series 81, 1978, 28 pps.
- 75 Winniford, R.S., "The Evidence for Association of Asphaltene in Dilute Solutions", Preprints Div. of Petroleum Chemistry, American Chemical Society, Vol.5, No.4-A [1960], pp. A-11 to A-22.
- 76 Yen, T.F., "Structural Difference Between Petroleum and Coal-Derived Asphaltene", presented Div. of Petroleum Chem., American Chemical Society, Vol.24, No.4 [1979], pp. 901-908.

#### COMPOSITION BY LIQUID CHROMATOGRAPHY (LC, HPLC, GPC, HP-GPC, IGLC)

- 77 Altgelt, K.H. and Gouw, T.H., "Chromatography of Heavy Petroleum Fractions" in Advances in Chromatography [Ed. J.C. Giddings, et.al.], New York, Marcel Dekker, Inc., 1975, Vol. 13., pp. 71-175.
- 78 Altgelt, K.H., "GPC in Structural Research of Asphalt", Bitumen, Teere, Asphalte, Pecher, Vol.21 [1970], pp. 475-.
- 79 Altgelt, K.H. and Hirsch, E., "GPC Separation and Integrated Structural Analysis of Petroleum Heavy Ends", Separation Science 5[6] 855 [1970].
- 80 Altgelt, K.H., "Fractionation of Asphaltene by Gel Permeation Chromatography", J. of App. Polymer Science, Vol.9 [1965], pp. 3389-3393.
- 81 Boduszynski, M.M., "Relationship between solvent-derived and compound-class fractions in coal-derived distillates and vacuum still bottoms", Fuel, Vol. 63, 1984, pp. 93-100.
- 82 Boduszynski, M.M., "Investigations on Romanshkiño asphalt bitumen. 1. Use of ion-exchange and adsorption chromatography for the separation of maltene", Fuel, Vol. 56, 1977, pp. 145-148.
- 83 Boduszynski, M.M., "Investigations on Romanshkiño asphalt bitumen. 2. Study of maltene fractions using inverse gas-liquid chromatography", Fuel, Vol. 56, 1977, pp. 149-152.
- 84 Boduszynski, M.M., "Investigations on Romanshkiño asphalt bitumen. 3. Fractionation of asphaltene using ion-exchange chromatography", Fuel, Vol. 56, 1977, pp. 432-436.
- 85 Bynum, D. and Traxler, R.N., "Gel Permeation Chromatography Data on Asphalts Before and After Service in Pavements", Proc. of AAPT, Vol.39 [1970], pp. 683-702.
- 86 Determann, H., Gel Chromatography, New York, Springer-Verlag New York, 1980.
- 87 Jennings, P.W., Pribanic, J.A.S. and Dawson, K.R., "Use of HPLC to Determine the Effects of Various Additives and Fillers on the Characteristics of Asphalt" in a paper prepared for the Montana Department of Highways and U.S. DOT Federal Highway Admin., June 1982, 24 pp.
- 88 Jennings, P.W., Pribanic, J.A.S., Dawson, K.R. and Bricca, C.E., "Use of HPLC and NMR Spectroscopy to Characterize Asphaltic Materials", Preprints Div. of Petrol. Chem., American Chemical Society, Vol. 26, No. 4, Sept. 1981, pp. 915-922.
- 90 Jennings, P.W., et. al., "Chemical Composition of Commercial Asphalt Cement as Determined by High Pressure Liquid Chromatography", FHWA Report No. MT-7929, Department of Chemistry, Montana State University,

- Bozeman, Montana, December 1977.
- 89 Jennings, P.W., Pribanic, J., et.al., "High Pressure Liquid Chromatography as a Method of Measuring Asphalt Composition", FHWA Report No. MTDOH-7930, Department of Chemistry, Montana State University, Bozeman, Montana, March 1980.
- 81 Montana State Department of Highways special provision, "Requirements for the Gel Permeation Mode (HP-GPC) of Asphalt Cement", Nov. 14, 1983.
- 82 Robertson, J.C. and Moore, J.R., "A Correlation of the Studies of Inverse GLC of Paving Asphalts with Paired Comparison Performance Results", Proc. of AAPT, Vol.40 (1971), pp. 438-462.
- 93 Snyder, L.R. and Kirkland, J.J., Introduction to Modern Liquid Chromatography, Second Edition, New York, John Wiley & Sons, Inc., 1979.

## COMPOSITION and REFINING

- 94 Corbett, L.W., "Refinery Processing of Asphalt Cement", paper presented TRB, January 1984.
- 95 Corbett, L.W., "Dumbbell Mix for Better Asphalt", Hydrocarbon Processing, Vol.58, April 1979, pp. 173-177.
- 96 Corbett, L.W., et.al., "Differences in Distillation and Solvent Asphalt Residue", Ind.Eng.Chem. Prod.Res.Dev., Vol.17, No.4 (1978), pp. 342-346.
- 97 Plummer, M.A. and Zimmerman, C.C., "Asphalt Quality and Yield Predictions from Crude Oil Analyses", presented at AAPT, April 1984.

## ASPHALT DISPERSION STABILITY

- 98 Heithaus, J.J., "Measurement and Significance of Asphaltene Peptization", Preprints Div. of Petroleum Chemistry, American Chemical Society, Vol.5, No. 4-A (1960), pp. A-23 to A-38.
- 99 Heithaus, J.J. and Fink, D.F., "An Examination of the Significance of the Oliensis Spot Test", Proc. of AAPT, Vol.28 (1959), pp. 353-371.
- 99a Oliensis, G.L., "The Oliensis Spot Test as a Quality Test", Proc. of AAPT, Vol.26 (1957), pp. 82-125.
- 99b Oliensis, G.L., "A Qualitative Test for Determining the Degree of Heterogeneity of Asphalts", Proc. of ASTM, Vol.33 (1933), pp. 715-728.
- 100 Plancher, H., Hoiberg, A.J., Suhaka, S.C. and Petersen, J.C., "A Settling Test to Evaluate the Relative Degree of Dispersion of Asphaltenes", Proc. of AAPT, Vol.48 (1979), pp. 351-374.
- 101 White, R.M., Mitten, W.R. and Skog, J.B., "Fractional Components of Asphalt—Compatibility and Interchangeability of Fractions Produced from Different Asphalts", Proc. of AAPT, Vol.39 (1970), pp. 492-531.

## COMPOSITION TESTS

- 101a Biemann, K., Mass Spectrometry - Organic Chemical Applications, McGraw-Hill, New York, 1962.
- 102 Corbett, L.W. and Swarbrick, R.E., "Clues to Asphalt Composition", Proc. AAPT, Vol.27 (1958), pp. 107-121.
- 102a Couper, J.R., "Asphalt", Anal. Chem., Vol.55, No.5, April 1983, pp. 257R-262R.
- 103 Fryazinov, V.V., et.al., "The Interrelationship Between the Density, the Composition, and the Properties of Bitumens", Khimiya i tekhnologiya topliv i masel, No. 9 (1979), pp. 41-44.
- 104 Halstead, W.J., Rostler, F.S. and White, R.M., "Properties of Highway Asphalts—Part III, Influence of Chemical Composition", Proc. of AAPT, Vol.35 (1966), pp. 91-138.
- 105 Kozlova, G.G., et.al., "Rapid Method for Determination of Group Composition of Petroleum Asphalts", Khimiya i Tekhnologiya, No.2 (1981)

- pp. 47-48.
- 105a Masak, V., Zentralbl. Arbeitsmed. Arbeitsschutz Prophyl., Vol.29, No.9 [1979], pp. 241-244.
- 106 Petersen, J.C. and Plancher, H., "Quantitative Method Using Differential Infrared Spectrometry for the Determination of Compound Types Absorbing in the Carbonyl Region in Asphalts", Anal. Chem, Vol.47 No.1, January 1975, pp. 112-117.
- 106a Poirier, M. and Albert, E.G., "Rapid Method for the Determination of Maltheane and Asphaltene Content in Bitumen, Heavy Oils, and Synthetic Fuels by Pyrolysis TLC", Journal of Chromatographic Science, Vol.21, July 1983, pp. 331-333.
- 106b Ray, J.E., Oliver, K.M. and Wainwright, J.C., "The Application of the Iatroscan TLC Technique to the Analysis of Fossil Fuels", in Petroanalysis '81 [Ed. G.B. Crump], Advances in Analytical Chemistry in the Petroleum Industry 1975-1982, Chapter 33, John Wiley & Sons, New York, 1982.
- 107 Rostler, F.S., Rostler, K.S., et.al., "Fingerprinting of Highway Asphalts", Proc. of AAPT, Vol.41 [1972], pp. 665-715.
- 108 Rostler, F.S., "Fractional Composition: Analytical and Functional Significance" in Bituminous Materials: Asphalts, Tars, and Pitches, Vol.2, Part 1 [Ed. A.J. Hoiberg], Interscience, New York [1965], pp. 151-223.
- 108a Rostler, F.S. and Starnberg, H.W., "Compounding Rubber with Petroleum Products", Industrial and Engineering Chemistry, Vol.41, March 1949, pp. 598-608.
- 108b Traxler, R.N. and Schweyer, H.T., Proc. of ASTM, Vol.36,II, 554 [1936]

## HOT-MIX RECYCLING

- 109 Davidson, D.D., Canessa, W. and Escobar, S.J., "Recycling of Sub-standard or Deteriorated Asphalt Pavements—A Guideline for Design Procedures", Proc. of AAPT, Vol.48 [1977], pp. 496-526.
- 110 Escobar, S.J. and Davidson, D.D., "Role of Recycling Agents in the Restoration of Aged Asphalt Cements", Proc. AAPT, Vol.48 [1979], pp. 375-402.
- 111 Gannon, C.R., et.al., "Recycling Conventional and Rubberized Bituminous Concrete Pavements Using Recycling Agents and Virgin Asphalt as Modifiers", Proc. of AAPT, Vol.49 [1980], pp. 95-123.
- 112 Kari, W.J., et.al., "Prototype Specifications for Recycling Agents Used in Hot-Mix Recycling", Proc. of AAPT, Vol.49 [1980], pp. 177-199.
- 113 Kari, W.J., et.al., "Hot Mix Recycling of Asphalt Pavements", Proc. of AAPT, Vol.48 [1979], pp. 192-220.
- 114 Newcomb, D.E., Nusser, B.J., Kiggundu, B.M., et.al., "A Laboratory Study of the Effects of Recycling Modifiers on Aged Asphalt Cement", paper presented at TRB, 1984.
- 115 Thompson, E.A., "Chemical Aspects of Asphalt Pavement Rejuvenation", Proc. 26th Ann. Conf., CTAA, Vol.26 [1981], pp. 84-96.

## HISTORICAL

- 116 From comments made by Dr. N.W. McLeod, ASTM Subcommittee D4.46, Kansas City, Missouri, June 23, 1983.
- 117 Krchma, L.C. and Gagle, D.W., "Manufacture and Distribution of Asphalt", Proc. of AAPT, Vol.43A [1974], pp. 25-88.
- 118 Baskin, C.M., "Asphalt, Its Chemistry, Significance of Source and Effect of Modern Processes on Present Day Specifications", Proc. of AAPT, Vol.4 [1932], pp. 71-92.
- 119 Mack, C., "Physico-Chemical Aspect of Asphalts", Proc. of AAPT, Vol.5, [1933], pp. 40-53.

APPENDIX F

## APPENDIX F

## COMPUTER INVENTORY OF ASPHALT PROPERTIES

The following pages present computer output which synthesizes asphalt cement test data obtained by the asphalt laboratory at Oregon State Highway Division during a four-year period (1981 to 1985). This data represents all "complete" asphalt tests carried out during the period represented. The Oregon State University CYBER Computer was used to develop this output.

Development of a microcomputer based inventory of asphalt properties is underway, but not sufficiently developed to include in this report. The details of the microcomputer inventory will be presented in the final report for this research study.

\*\*\*\*\*  
\*  
\* TABLES OF ASPHALT PROPERTIES COLLECTED \*  
\* BY THE OREGON STATE HIGHWAYS DIVISION \*  
\*  
\*\*\*\*\*

PROPERTIES GROUPED BY:

- BRAND
- SPEC
- GRADE
- DATE

SPEC/GRD LEGEND

IN TABLE:	MEANS:
AR X	AR X000
ARW X	AR X000 W
FOR AC SPECS:	
AC 25	AC 2.5
OTHERWISE READ AS IS.	
FOR PENETRATION GRADES VALUE	
SHOWN IS UPPER LIMIT:	
PEN 150	120-150

TABLE OF ASPHALT PROPERTIES COLLECTED  
 BY THE OREGON STATE HIGHWAYS DIVISION  
 (GROUPED BY SPECIFICATION/GRADE/DATE)

PAGE 1

BRAND: ARIZONA

DATE	LAB#	SPEC/GRD	FLASH	SOLUB	PEN (DEG F) (77)	PEN (39) (39)	PEN RATIO	LOSS	KINEMATIC VISCOSITY	ABSOLUTE VISCOSITY	RESIDUE PEN %ORIG	DUCTILITY (77)	DUCTILITY (45)
7/31/84	9888	ARM 4	515	99.99	82	20	24	1.04	258 / 406	1228 / 3099	42	51	100 0

TABLE OF ASPHALT PROPERTIES COLLECTED  
 BY THE OREGON STATE HIGHWAYS DIVISION  
 (GROUPED BY SPECIFICATION/GRADE/DATE)

PAGE 2

BRAND: BOSCAN

DATE	LAB#	SPEC/GRD	FLASH	SOLUB	PEN (DEG F) (77)	PEN (39) (39)	PEN RATIO	LOSS	KINEMATIC VISCOSITY	ABSOLUTE VISCOSITY	RESIDUE PEN %ORIG	DUCTILITY (77)	DUCTILITY (45)
2/23/83	1183	AC 20	490	99.99	95	36	38	.79	499 / 911	2123 / 7193	50	53	100 38

TABLE OF ASPHALT PROPERTIES COLLECTED  
 BY THE OREGON STATE HIGHWAYS DIVISION  
 (GROUPED BY SPECIFICATION/GRADE/DATE)

BRAND: CENEX

DATE	LAB#	SPEC/GRD	FLASH	SOLUB	PEN (DEG F)		PEN RATIO		LOSS	KINEMATIC VISCOSITY		ABSOLUTE VISCOSITY		RESIDUE PEN %ORIG		DUCTILITY (77) (45)		
10/ 5/82	12970	AC 10	600	99.90	90	28	31	.57	286	/	438	1139	/	3587	44	49	100	12
6/30/82	6843	PEN 100	510	99.99	92	31	34	.49	263	/	449	1125	/	3606	47	51	100	14
7/29/82	8573	PEN 100	505	99.98	88	20	23	.68	224	/	417	1181	/	3782	41	47	100	18
MEAN:					90.00	25.50	28.50	.59	243.50/	433.00	1153.00/	3694.00	44.00	49.00				
ST DEV:					2.83	7.78	7.78	.13	27.58/	22.63	39.60/	124.45	4.24	2.83				



TABLE OF ASPHALT PROPERTIES COLLECTED  
BY THE OREGON STATE HIGHWAYS DIVISION  
(GROUPED BY SPECIFICATION/GRADE/DATE)

BRAND: CHEVRON

DATE	LAB#	SPEC/GRD	FLASH	SOLUB	PEN (DEG F)		PEN	LOSS	KINEMATIC		ABSOLUTE		RESIDUE		DUCTILITY		
					(77)	(39)	RATIO		VISCOSITY	VISCOSITY	VISCOSITY	PEN	%ORIG	(77)	(45)		
12/15/82	16922	AC 20	475	99.99	93	32	34	1.05	495 / 917	2055 / 7981	48	52	100	0			
6/15/83	6789	AC 20	555	99.94	65	15	23	.83	335 / 559	2091 / 6158	25	54	100	10			
7/21/83	9304	AC 20	470	99.95	67	24	36	.85	319 / 510	1191 / 5305	37	55	100	18			
7/21/83	9303	AC 20	465	99.99	70	19	27	.88	295 / 457	1701 / 4446	39	56	100	16			
8/26/83	10486	AC 20	465	99.96	68	14	21	.74	305 / 475	1788 / 4729	37	54	100	19			
8/26/83	10487	AC 20	455	99.98	67	14	21	.67	311 / 480	1829 / 4721	37	55	100	20			
8/31/83	11579	AC 20	505	99.94	64	18	28	.66	340 / 528	2043 / 5507	36	56	100	16			
9/ 1/83	12177	AC 20	515	99.97	62	13	21	.42	330 / 506	2056 / 5587	34	55	100	14			
9/15/83	12679	AC 20	520	99.99	63	13	21	.93	330 / 532	2036 / 5884	34	54	100	20			
9/26/83	13506	AC 20	530	99.94	62	14	23	.65	337 / 526	2112 / 5427	36	58	100	12			
9/27/83	13962	AC 20	515	99.99	62	12	19	.64	350 / 528	2270 / 5536	40	65	100	8			
10/10/83	15197	AC 20	520	99.91	58	13	22	.71	364 / 571	2303 / 6301	30	52	100	10			
MEAN:					64.36	15.36	23.82	.73	328.73/	515.64	1947.27/	5418.27	35.00	55.82			
ST DEV:					3.44	3.59	4.85	.14	20.11/	34.80	312.94/	592.71	4.27	3.40			
3/13/84	4422	AC 10	505	99.86	76	23	30	1.18	319 / 524	1626 / 5403	40	53	100	52			
2/ 2/84	1213	AC 20	510	99.98	51	12	24	.53	418 / 661	3037 / 8274	30	59	100	13			
3/15/84	3334	AC 20	510	99.96	67	24	36	.66	353 / 552	2003 / 5425	37	55	100	11			
6/20/84	7247	AC 20	525	99.94	57	16	28	.55	386 / 529	2026 / 5017	33	52	100	9			
6/20/84	7249	AC 20	530	99.98	58	16	28	1.00	336 / 524	2017 / 4909	33	57	100	12			
6/21/84	7248	AC 20	465	99.99	58	17	29	.59	352 / 544	2001 / 5089	33	57	100	12			
7/ 2/84	7931	AC 20	490	99.99	68	15	45	.83	394 / 545	1184 / 5833	36	53	100	7			
7/ 3/84	8041	AC 20	510	99.99	66	23	35	.72	379 / 612	2050 / 6193	35	53	100	11			
7/ 9/84	8346	AC 20	490	99.51	66	17	26	.79	418 / 670	2207 / 6590	35	53	100	17			
7/10/84	8352	AC 20	480	99.99	63	15	24	.78	412 / 683	2320 / 6333	34	54	100	16			
7/24/84	9152	AC 20	480	99.99	62	20	32	.67	422 / 668	2319 / 6322	34	55	100	13			
7/25/84	9314	AC 20	545	99.94	65	16	25	.86	411 / 704	2225 / 6249	36	55	100	13			
7/26/84	9316	AC 20	495	99.99	63	19	30	.86	407 / 674	2185 / 6386	36	57	100	15			
7/26/84	9572	AC 20	490	99.99	68	20	29	.97	391 / 637	1981 / 6032	37	54	100	15			
7/26/84	9315	AC 20	500	99.99	64	21	33	.95	418 / 674	2167 / 6733	32	50	100	14			
MEAN:					62.57	17.93	30.29	.77	392.64/	619.79	2123.00/	6098.93	34.36	54.57			
ST DEV:					4.94	3.36	5.69	.15	28.25/	66.34	381.60/	865.06	2.02	2.38			
3/30/81	3680	AR 2	560	99.78	118	25	22	.45	241 / 330	743 / 1501	66	59	100	65			
10/22/81	15320	AR 2	440	99.98	117	25	21	.71	212 / 343	690 / 1771	58	50	100	25			
MEAN:					117.50	25.00	21.50	.58	226.50/	336.50	716.50/	1636.00	62.00	54.50			
ST DEV:					.71	.00	.71	.18	20.51/	9.19	37.48/	190.92	5.66	6.36			
1/12/82	16776	AR 2	510	99.98	112	26	23	.64	230 / 351	701 / 1815	57	51	0	0			
4/ 6/84	4526	AR 2	490	99.99	82	14	17	.76	209 / 296	962 / 2028	48	59	100	32			
6/11/84	6638	AR 2	460	99.99	85	20	24	.79	211 / 310	948 / 2179	45	53	100	24			
6/12/84	7011	AR 2	465	99.96	81	18	22	.81	214 / 300	995 / 2167	46	57	100	28			

\*\*\*\*\* SEE NEXT PAGE \*\*\*\*\*

TABLE OF ASPHALT PROPERTIES COLLECTED  
BY THE OREGON STATE HIGHWAYS DIVISION  
(GROUPED BY SPECIFICATION/GRADE/DATE)

BRAND: CHEVRON

DATE	LAB#	SPEC/GRD	FLASH	SOLUB	PEN (DEG F) (77) (39)	PEN RATIO	LOSS	KINEMATIC VISCOSITY	ABSOLUTE VISCOSITY	RESIDUE PEN	DUCTILITY (77) (45)					
6/12/84	7008	AR	2	470	99.94	84	23	27	.82	213 / 294	978 / 2009	46	55	100	26	
6/19/84	7016	AR	2	460	99.99	86	21	24	.81	199 / 308	985 / 2156	48	56	100	28	
6/19/84	7072	AR	2	470	99.94	83	10	12	.95	208 / 298	937 / 2174	44	53	100	19	
7/10/84	8349	AR	2	500	99.99	83	15	18	.74	204 / 296	974 / 2047	49	59	100	47	
7/18/84	8521	AR	2	565	99.99	86	18	21	1.00	204 / 304	953 / 2164	46	53	100	100	
MEAN:					83.75	17.38	20.63	.84	207.75/	300.75	966.50/	2115.50	46.50	55.63		
ST DEV:					1.83	4.21	4.78	.09	5.12/	5.95	19.85/	73.47	1.69	2.56		
4/ 6/84	4527	AR	4	560	99.99	41	7	17	.14	286 / 370	2186 / 3777	28	68	100	11	
6/11/84	7007	AR	4	560	99.99	60	13	22	.59	277 / 389	1551 / 3430	34	57	100	11	
7/ 3/84	8040	AR	4	525	99.98	59	17	29	.85	290 / 418	1775 / 3837	34	58	100	13	
MEAN:					53.33	12.33	22.67	.53	284.33/	392.33	1837.33/	3681.33	32.00	61.00		
ST DEV:					10.69	5.03	6.03	.36	6.66/	24.17	322.06/	219.72	3.46	6.08		
10/29/81	16148	AR	8	0	.00	54	0	0	.96	396 / 701	2425 / 8449	26	48	0	0	
4/22/82	3722	ARW	2	585	99.86	113	24	21	.70	224 / 325	725 / 1714	62	55	100	2	
6/15/82	6223	ARW	2	515	99.93	158	56	35	1.17	218 / 383	580 / 2025	72	46	100	31	
7/13/82	7606	ARW	2	530	99.88	170	50	35	.93	222 / 388	584 / 2203	77	45	100	57	
7/22/82	8342	ARW	2	535	99.96	168	50	30	1.13	180 / 401	629 / 2333	76	45	100	64	
8/20/82	10437	ARW	2	485	99.95	144	43	30	1.18	219 / 360	676 / 2096	70	49	100	100	
10/21/82	14228	ARW	2	500	99.96	125	28	22	1.22	204 / 332	750 / 2107	58	46	100	100	
11/12/82	14846	ARW	2	485	99.93	132	39	20	1.24	197 / 311	709 / 1850	66	50	100	100	
MEAN:					144.29	41.43	27.57	1.08	209.14/	357.14	664.71/	2046.86	68.71	48.00		
ST DEV:					22.04	11.91	6.50	.20	16.25/	34.99	68.27/	209.09	7.09	3.65		
3/18/83	2457	ARW	2	540	99.93	128	36	28	1.29	202 / 322	705 / 1997	61	48	100	100	
5/26/83	4860	ARW	2	545	99.96	156	42	27	1.64	178 / 295	557 / 1746	72	46	100	100	
5/26/83	4883	ARW	2	490	99.95	118	29	25	.91	200 / 292	746 / 1311	64	54	100	100	
7/13/83	7702	ARW	2	525	99.92	113	34	30	1.07	215 / 324	833 / 2123	61	54	100	100	
7/20/83	9300	ARW	2	475	99.99	116	32	28	.86	207 / 309	800 / 1830	65	56	100	100	
10/20/83	16788	ARW	2	520	99.91	103	25	24	.65	224 / 348	916 / 2241	58	56	100	10	
12/13/83	19865	ARW	2	530	99.81	103	20	19	.73	228 / 342	944 / 2179	58	56	100	10	
MEAN:					119.57	31.14	25.86	1.02	207.71/	318.86	785.86/	1918.14	62.71	52.86		
ST DEV:					18.28	7.27	3.63	.35	16.86/	21.64	132.21/	323.13	4.89	4.14		
4/26/84	4572	ARW	2	555	99.95	95	19	20	.83	240 / 362	940 / 2434	51	54	100	100	
6/12/84	7010	ARW	2	565	99.96	99	30	30	.91	218 / 293	857 / 1784	61	62	100	100	
6/22/84	7155	ARW	2	495	99.99	97	24	25	.65	227 / 295	887 / 1788	65	67	100	88	
6/27/84	7256	ARW	2	540	99.83	106	23	22	.95	204 / 263	749 / 1553	62	58	100	100	
MEAN:					99.25	24.00	24.25	.84	222.25/	303.25	858.25/	1889.75	59.75	60.25		
ST DEV:					4.79	4.55	4.35	.13	15.15/	41.81	80.51/	379.10	6.08	5.56		
3/25/81	3497	ARW	4	530	99.91	100	28	28	.83	381 / 616	1379 / 4038	54	54	100	30	

\*\*\*\*\* SEE NEXT PAGE \*\*\*\*\*

TABLE OF ASPHALT PROPERTIES COLLECTED  
BY THE OREGON STATE HIGHWAYS DIVISION  
(GROUPED BY SPECIFICATION/GRADE/DATE)

BRAND: CHEVRON

DATE	LAB#	SPEC/GRD	FLASH	SOLUB	PEN (DEG F) (77) (39)	PEN RATIO	LOSS	KINEMATIC VISCOSITY	ABSOLUTE VISCOSITY	RESIDUE PEN XORIG	DUCTILITY (77) (45)			
3/26/81	3585	ARW 4	520	99.97	116 39	34	1.08	393 / 651	1298 / 4168	56 48	100 40			
3/26/81	3632	ARW 4	475	99.99	123 43	35	1.12	396 / 685	1247 / 4496	62 50	100 38			
3/31/81	1480	ARW 4	480	99.87	110 37	34	1.12	396 / 697	1400 / 4734	56 51	100 40			
4/ 9/81	3919	ARW 4	510	99.95	123 39	32	1.27	396 / 718	1297 / 4775	58 47	100 48			
4/14/81	4270	ARW 4	595	99.95	71 22	31	.47	400 / 579	1751 / 4444	40 56	100 15			
4/30/81	4677	ARW 4	600	99.62	86 25	29	.79	360 / 562	1416 / 3935	44 51	100 19			
5/11/81	5446	ARW 4	575	99.94	82 25	30	.59	360 / 536	1471 / 3724	48 59	100 29			
6/ 2/81	6834	ARW 4	590	99.95	82 25	30	.63	358 / 527	1405 / 3564	46 56	100 22			
6/ 2/81	7718	ARW 4	600	99.99	79 21	27	.56	355 / 542	1434 / 3780	45 57	0 0			
6/ 2/81	7016	ARW 4	560	99.98	81 23	28	.48	355 / 522	1398 / 3340	48 59	100 0			
7/ 1/81	8398	ARW 4	555	99.97	82 25	27	.85	349 / 564	1279 / 3818	48 52	0 0			
7/31/81	9253	ARW 4	555	99.99	82 26	32	.44	354 / 501	1403 / 3279	47 57	100 36			
8/ 4/81	9322	ARW 4	565	99.99	77 21	27	.62	348 / 498	1427 / 3477	46 60	100 31			
8/ 4/81	9323	ARW 4	560	99.97	87 27	31	.68	337 / 515	1315 / 3324	43 49	100 23			
8/ 4/81	10344	ARW 4	560	99.98	81 28	35	.72	318 / 498	1307 / 3870	41 51	100 19			
8/ 6/81	10703	ARW 4	580	99.95	83 25	30	.58	327 / 498	1284 / 3409	45 54	100 25			
8/ 6/81	10709	ARW 4	575	99.96	85 25	29	.66	294 / 458	1162 / 3118	45 53	100 22			
9/ 8/81	12367	ARW 4	590	99.21	86 22	26	.89	260 / 396	1138 / 3151	42 49	100 19			
9/ 8/81	11632	ARW 4	545	99.77	82 28	34	.93	283 / 433	1144 / 3099	43 52	100 15			
9/ 8/81	12609	ARW 4	555	99.85	85 23	30	.89	276 / 458	1132 / 3262	43 52	100 21			
9/ 8/81	11796	ARW 4	540	99.54	87 22	25	.89	301 / 448	1211 / 3838	41 47	100 13			
9/ 8/81	12377	ARW 4	555	99.92	79 23	29	.98	301 / 418	1167 / 2856	45 57	100 21			
9/ 8/81	12172	ARW 4	570	99.58	83 24	29	.94	282 / 441	1157 / 3656	42 51	100 16			
9/ 8/81	13285	ARW 4	545	99.95	75 20	27	1.03	260 / 485	1146 / 3079	40 53	100 19			
9/ 8/81	12570	ARW 4	550	99.89	84 23	27	.86	308 / 457	1120 / 3081	46 55	100 20			
9/ 8/81	12827	ARW 4	555	99.92	77 19	25	.94	275 / 412	1155 / 3024	39 51	100 20			
9/30/81	14042	ARW 4	555	99.96	75 17	23	1.12	264 / 426	1225 / 3633	34 45	100 13			
9/30/81	14110	ARW 4	530	99.96	75 17	23	.99	263 / 437	1281 / 3550	38 52	100 14			
10/ 9/81	14536	ARW 4	535	99.98	73 16	22	.50	262 / 397	1284 / 3409	37 51	100 16			
10/14/81	14957	ARW 4	530	99.95	66 15	23	.89	272 / 432	1400 / 3707	35 53	100 13			
10/21/81	15163	ARW 4	550	99.97	67 11	16	.96	275 / 456	1436 / 4513	32 48	100 11			
10/21/81	15113	ARW 4	520	99.98	66 13	20	.92	284 / 440	1494 / 3880	35 53	100 12			
10/22/81	15303	ARW 4	530	99.97	74 16	22	1.00	279 / 451	1384 / 3941	39 53	100 12			
10/28/81	15321	ARW 4	535	99.98	67 16	24	.87	269 / 443	1383 / 3491	36 54	100 16			
10/29/81	15610	ARW 4	545	99.98	74 16	22	.86	274 / 448	1315 / 3508	39 53	100 16			
10/29/81	15378	ARW 4	500	99.98	65 13	23	1.13	224 / 414	1429 / 4036	31 48	100 12			
10/29/81	15864	ARW 4	515	99.95	75 17	23	1.31	272 / 468	1517 / 4072	31 41	100 17			
10/29/81	15919	ARW 4	510	99.97	75 18	24	1.01	270 / 454	1505 / 3824	31 41	100 16			
10/92/81	15429	ARW 4	500	99.95	71 15	21	.95	280 / 396	1371 / 3647	34 48	100 12			
MEAN:					82.28	22.70	27.18	.86	312.78/	494.43	1326.68/	3688.75	42.63	51.78
ST DEV:					14.09	7.28	4.52	.22	49.05/	84.09	136.00/	475.33	7.52	4.32
3/16/82	2372	ARW 4	505	99.96	79 20	25	1.08	292 / 477	1438 / 4254	39 49	100 15			
4/20/82	3705	ARW 4	520	99.91	73 16	22	1.25	270 / 458	1423 / 4449	36 49	100 10			
4/20/82	3703	ARW 4	565	99.85	76 20	26	1.31	261 / 438	1379 / 4317	35 46	100 9			
4/20/82	3704	ARW 4	515	99.78	78 18	23	1.41	260 / 438	1343 / 3915	36 46	100 10			
4/21/82	3707	ARW 4	500	99.78	0 63	0	4.58	163 / 405	374 / 3202	44 0	100 11			
4/21/82	3706	ARW 4	510	99.69	74 17	23	1.26	264 / 445	1388 / 4150	37 50	100 10			
4/22/82	3723	ARW 4	495	99.91	94 22	23	1.06	272 / 443	1194 / 3451	45 48	100 14			

\*\*\*\*\* SEE NEXT PAGE \*\*\*\*\*

TABLE OF ASPHALT PROPERTIES COLLECTED  
BY THE OREGON STATE HIGHWAYS DIVISION  
(GROUPED BY SPECIFICATION/GRADE/DATE)

BRAND: CHEVRON

DATE	LAB#	SPEC/GRD	FLASH	SOLUB	PEN (DEG F) (77) (39)	PEN RATIO	LOSS	KINEMATIC VISCOSITY	ABSOLUTE VISCOSITY	RESIDUE PEN %ORIG	DUCTILITY (77) (45)			
5/20/82	4931	ARW 4	535	99.98	94 37	39	1.10	271 / 457	1175 / 3644	47 50	100 0			
5/26/82	5093	ARW 4	505	99.90	98 24	24	1.15	270 / 460	1136 / 3504	48 49	100 16			
5/26/82	5094	ARW 4	515	99.98	98 24	24	1.16	273 / 445	1148 / 3412	47 48	100 17			
6/ 2/82	5373	ARW 4	525	99.99	96 26	27	1.11	268 / 433	1143 / 3517	47 49	100 13			
6/15/82	6170	ARW 4	520	99.75	80 29	36	.99	277 / 450	1429 / 3985	41 51	100 2			
6/17/82	6440	ARW 4	530	99.88	70 20	29	1.31	267 / 500	1448 / 5367	33 47	100 13			
6/28/82	6903	ARW 4	525	99.92	58 24	27	1.37	271 / 455	1279 / 4014	41 47	100 10			
6/30/82	6968	ARW 4	560	99.99	83 23	28	1.13	290 / 460	1356 / 3901	41 49	100 11			
7/ 1/82	7330	ARW 4	555	99.97	85 27	32	.90	275 / 440	1300 / 3677	41 48	100 13			
7/ 6/82	7407	ARW 4	550	99.98	85 23	27	1.24	276 / 467	1333 / 4112	41 48	100 12			
7/15/82	8059	ARW 4	510	99.97	81 21	25	1.04	273 / 439	1332 / 3799	42 49	100 18			
7/15/82	7946	ARW 4	515	99.75	85 22	26	1.22	285 / 472	1376 / 4490	39 46	100 13			
7/19/82	8091	ARW 4	515	99.99	85 30	35	1.19	268 / 452	1302 / 3952	40 47	100 20			
7/29/82	8835	ARW 4	525	99.95	79 16	20	1.08	243 / 428	1404 / 3912	40 51	100 38			
7/29/82	8780	ARW 4	515	99.91	80 17	21	1.14	238 / 414	1350 / 3712	40 50	100 40			
8/ 4/82	9005	ARW 4	480	99.98	86 23	27	1.21	278 / 438	1289 / 3798	43 50	100 44			
8/ 4/82	8926	ARW 4	500	99.99	83 22	26	1.09	269 / 433	1337 / 3772	41 49	100 40			
8/10/82	9534	ARW 4	505	99.93	85 26	31	1.14	281 / 439	1305 / 3856	36 42	100 46			
8/10/82	9357	ARW 4	505	99.94	77 24	31	1.12	261 / 418	1352 / 3978	39 51	100 38			
8/12/82	9724	ARW 4	545	99.94	82 20	24	1.28	258 / 424	1303 / 3793	41 50	100 0			
8/17/82	9927	ARW 4	465	99.97	76 22	29	1.11	268 / 383	1452 / 3772	40 53	100 0			
8/17/82	9935	ARW 4	590	99.99	82 22	27	.90	272 / 370	1327 / 3691	41 50	100 0			
8/17/82	9936	ARW 4	500	99.95	81 21	26	1.08	266 / 358	1319 / 3633	40 49	100 0			
8/18/82	10051	ARW 4	490	99.92	84 20	24	.91	236 / 364	1242 / 2957	46 55	100 0			
8/18/82	9980	ARW 4	495	99.92	87 19	22	1.05	240 / 374	1189 / 3237	44 51	100 0			
8/20/82	10135	ARW 4	500	99.94	90 23	26	.98	248 / 406	1178 / 3631	46 51	100 100			
8/20/82	10436	ARW 4	505	99.96	84 22	26	.78	251 / 371	1306 / 3096	46 55	100 100			
9/ 3/82	11450	ARW 4	525	99.95	77 15	19	.87	247 / 440	1288 / 3222	41 53	100 100			
9/ 3/82	11503	ARW 4	520	99.96	81 15	19	1.01	240 / 337	1204 / 3322	42 52	100 100			
9/ 7/82	11566	ARW 4	520	99.96	84 28	33	.96	250 / 372	1247 / 3246	42 50	100 88			
9/ 7/82	11565	ARW 4	525	99.98	82 28	34	.98	242 / 373	1247 / 3137	43 52	100 100			
9/24/82	12159	ARW 4	480	99.96	95 35	37	1.21	224 / 304	1071 / 2836	46 48	100 47			
9/27/82	12513	ARW 4	485	99.99	91 32	35	1.07	227 / 351	1097 / 3015	45 50	100 56			
9/27/82	12548	ARW 4	475	99.96	101 39	39	1.48	216 / 304	998 / 2930	46 0	100 42			
10/15/82	13171	ARW 4	485	99.94	89 30	34	1.25	227 / 363	1126 / 3119	44 49	100 26			
11/ 8/82	14264	ARW 4	500	99.91	80 20	25	1.13	242 / 278	1306 / 3414	40 50	100 32			
11/10/82	14851	ARW 4	475	99.99	80 26	32	1.03	242 / 378	1318 / 3496	41 51	100 30			
11/10/82	14244	ARW 4	515	99.98	83 28	34	1.08	230 / 362	1237 / 3274	42 51	100 24			
11/15/82	14245	ARW 4	510	99.95	85 23	27	1.06	229 / 359	1193 / 3188	42 49	100 31			
11/24/82	16088	ARW 4	495	99.99	79 23	29	1.16	244 / 343	1856 / 3695	40 51	100 26			
11/24/82	16089	ARW 4	480	99.98	77 27	35	1.16	244 / 404	1366 / 3702	40 52	100 28			
12/ 1/82	16496	ARW 4	485	99.97	80 30	38	1.06	242 / 380	1247 / 3435	41 51	100 30			
MEAN:					81.47	24.53	27.57	1.19	255.12/	408.20	1274.49/	3652.67	41.59	47.59
ST DEV:					14.16	7.76	6.67	.51	23.05/	50.11	186.06/	472.50	3.38	10.18
2/16/83	626	ARW 4	485	99.98	89 17	19	1.34	263 / 439	1283 / 4688	42 47	100 48			
2/16/83	686	ARW 4	570	99.99	78 22	28	.98	249 / 398	1363 / 3822	37 47	100 45			
3/16/83	2189	ARW 4	520	99.94	85 27	32	1.35	274 / 441	1366 / 4090	43 51	100 41			
3/18/83	2458	ARW 4	515	99.98	88 22	26	1.38	269 / 456	1319 / 4205	40 47	100 40			

\*\*\*\*\* SEE NEXT PAGE \*\*\*\*\*

TABLE OF ASPHALT PROPERTIES COLLECTED  
BY THE OREGON STATE HIGHWAYS DIVISION  
(GROUPED BY SPECIFICATION/GRADE/DATE)

BRAND: CHEVRON

DATE	LAB#	SPEC/GRD	FLASH	SOLUB	PEN (DEG F) (77) (39)	PEN RATIO	LOSS	KINEMATIC VISCOSITY	ABSOLUTE VISCOSITY	RESIDUE PEN %ORIG	DUCTILITY (77) (45)
4/26/83	2902	ARW 4	510	99.97	91 21	23	1.30	227 / 359	1100 / 3024	46 51	100 51
5/10/83	4500	ARW 4	480	99.99	84 24	29	1.46	244 / 380	1273 / 3564	42 50	100 15
5/11/83	4603	ARW 4	485	99.99	93 21	23	1.21	235 / 364	1143 / 3017	46 50	100 16
5/11/83	4603	ARW 4	485	99.99	93 21	23	1.21	235 / 364	1143 / 3017	46 50	100 10
6/ 1/83	5854	ARW 4	495	99.99	80 24	30	.99	243 / 385	1343 / 3912	38 48	100 28
6/ 1/83	5119	ARW 4	505	99.97	81 27	33	1.13	297 / 393	1321 / 3734	40 49	100 30
6/ 9/83	6212	ARW 4	495	99.98	84 23	27	1.08	240 / 373	1271 / 3408	42 50	100 16
6/ 9/83	6433	ARW 4	490	99.96	78 19	24	.86	255 / 381	1372 / 3489	41 53	100 14
6/15/83	6800	ARW 4	505	99.94	78 18	23	.78	253 / 369	1252 / 3169	43 53	100 19
7/ 7/83	7686	ARW 4	510	99.96	81 19	23	.93	247 / 360	1144 / 3003	43 53	100 30
7/13/83	7701	ARW 4	515	99.89	82 30	37	.83	236 / 347	1202 / 2912	44 54	100 23
7/14/83	8332	ARW 4	495	99.95	80 17	21	1.10	233 / 340	1182 / 2755	46 58	100 36
7/14/83	7966	ARW 4	505	99.92	79 18	23	.82	241 / 355	1191 / 3089	41 52	100 17
7/21/83	8920	ARW 4	545	99.98	80 18	23	.78	235 / 342	1266 / 2895	45 56	100 47
7/28/83	9574	ARW 4	495	99.99	78 19	24	.69	237 / 350	1306 / 2870	45 58	100 40
7/28/83	9675	ARW 4	515	99.99	77 20	26	.66	239 / 353	1297 / 2929	44 57	100 40
8/11/83	10068	ARW 4	505	99.98	76 22	29	.65	239 / 362	1269 / 3182	41 54	100 51
8/15/83	10145	ARW 4	505	99.90	76 22	29	.69	239 / 355	1257 / 3021	46 61	100 33
8/28/83	10875	ARW 4	500	99.98	78 18	23	.70	238 / 358	1292 / 2834	43 55	100 48
8/30/83	10646	ARW 4	500	99.95	77 23	30	.82	237 / 367	1252 / 3042	43 56	100 51
8/31/83	11199	ARW 4	516	99.98	78 19	24	.54	232 / 325	1224 / 2608	47 60	100 56
9/ 1/83	12364	ARW 4	510	99.85	75 13	17	.60	239 / 337	1224 / 2859	44 59	100 40
9/ 1/83	11725	ARW 4	500	99.99	76 11	14	.69	235 / 359	1259 / 2769	43 57	100 48
9/14/83	12600	ARW 4	520	99.86	75 14	19	.63	242 / 356	1295 / 3074	42 56	100 52
9/14/83	12655	ARW 4	515	99.84	78 14	18	.64	235 / 340	1231 / 2717	46 59	100 60
9/15/83	12716	ARW 4	510	99.96	76 16	21	.63	244 / 351	1332 / 2933	43 57	100 44
9/22/83	13265	ARW 4	495	99.93	81 19	23	.64	258 / 397	1392 / 3272	45 56	100 47
9/22/83	13263	ARW 4	530	99.98	77 20	26	.67	237 / 345	1284 / 2873	43 56	100 38
9/22/83	13264	ARW 4	550	99.96	74 16	22	.60	240 / 355	1318 / 3034	42 57	100 33
9/26/83	13606	ARW 4	515	99.98	74 15	20	.59	246 / 357	1389 / 3098	44 59	100 50
9/26/83	13505	ARW 4	505	99.96	77 14	18	.55	239 / 347	1284 / 2780	45 58	100 52
9/29/83	14434	ARW 4	515	99.97	76 17	22	.58	243 / 350	1316 / 2988	42 55	100 39
9/29/83	14171	ARW 4	485	99.99	77 17	22	.76	235 / 345	1276 / 2964	41 53	100 30
10/ 3/83	14846	ARW 4	0	99.99	72 16	22	.61	241 / 345	1341 / 2897	44 61	100 52
10/ 3/83	14763	ARW 4	490	99.98	73 17	23	.66	244 / 356	1333 / 3055	42 58	100 49
10/ 3/83	15222	ARW 4	500	99.97	74 17	23	.67	254 / 372	1364 / 3150	43 58	100 42
10/10/83	15223	ARW 4	510	99.92	79 16	20	.73	255 / 369	1402 / 3176	40 51	100 0
10/11/83	15526	ARW 4	520	99.86	75 13	17	.77	243 / 375	1334 / 3394	41 55	100 22
10/11/83	15516	ARW 4	515	99.84	74 13	18	.82	243 / 376	1355 / 3415	41 55	100 20
10/13/83	15826	ARW 4	510	99.96	79 15	29	.63	252 / 374	1360 / 3154	40 53	100 23
10/13/83	15681	ARW 4	520	99.95	75 20	27	.72	252 / 372	1340 / 3227	41 55	100 28
10/19/83	16567	ARW 4	510	99.99	73 16	22	.57	239 / 367	1366 / 3233	42 58	100 30
10/19/83	16553	ARW 4	515	99.97	75 15	20	.54	240 / 351	1452 / 3012	42 57	100 26
10/20/83	16583	ARW 4	505	99.98	75 15	20	.64	247 / 369	1303 / 3127	42 56	100 20
10/20/83	16573	ARW 4	520	99.92	75 15	20	.61	250 / 366	1345 / 3203	42 56	100 33
11/ 1/83	17147	ARW 4	545	99.96	70 17	24	.80	246 / 364	1405 / 3293	40 57	100 15
11/ 1/83	17510	ARW 4	540	99.96	70 17	24	.60	264 / 372	1435 / 3277	40 57	100 15
11/ 2/83	17511	ARW 4	565	99.97	68 19	28	.77	248 / 376	1487 / 3486	39 57	100 14
11/ 2/83	17691	ARW 4	0	99.95	69 19	28	.56	245 / 368	1366 / 3211	41 59	100 15
11/ 2/83	17516	ARW 4	520	99.97	71 20	28	.58	253 / 371	1417 / 3238	40 56	100 14
11/12/83	15276	ARW 4	490	99.92	80 26	32	1.09	244 / 385	1315 / 3582	41 51	100 32

\*\*\*\*\* SEE NEXT PAGE \*\*\*\*\*

TABLE OF ASPHALT PROPERTIES COLLECTED  
BY THE OREGON STATE HIGHWAYS DIVISION  
(GROUPED BY SPECIFICATION/GRADE/DATE)

BRAND: CHEVRON

DATE	LAB#	SPEC/GRD	FLASH	SOLUB	PEN (DEG F) (77) (39)	PEN RATIO	LOSS	KINEMATIC VISCOSITY	ABSOLUTE VISCOSITY	RESIDUE PEN %ORIG	DUCTILITY (77) (45)			
11/17/83	18004	ARW 4	525	99.98	72 18	25	.68	254 / 373	1405 / 3235	40 56	100 16			
11/17/83	17972	ARW 4	495	99.98	75 17	23	.71	249 / 367	1333 / 3207	41 55	100 18			
11/22/83	19126	ARW 4	510	99.99	67 16	24	.63	252 / 375	1487 / 3378	40 60	100 10			
12/14/83	20220	ARW 4	545	99.97	70 18	26	.61	262 / 382	1505 / 3365	41 59	100 30			
12/19/83	20536	ARW 4	520	99.99	69 17	25	.81	269 / 398	1573 / 3803	38 55	100 27			
MEAN:					77.33	18.48	24.03	.79	246.27/	367.97	1317.57/	3212.63	42.25	54.95
ST DEV:					5.68	3.78	4.39	.24	11.91/	23.70	91.74/	383.00	2.24	3.61
3/ 2/84	2605	ARW 4	495	99.96	85 20	24	.93	236 / 366	1182 / 3100	43 51	100 40			
3/10/84	3333	ARW 4	485	99.96	83 16	19	.89	236 / 360	1212 / 3043	46 55	100 49			
5/30/84	6400	ARW 4	0	99.99	78 14	18	.86	277 / 409	1477 / 3534	34 44	100 10			
5/30/84	6712	ARW 4	0	99.99	66 16	24	.84	265 / 391	1226 / 2976	42 64	100 19			
6/11/84	6637	ARW 4	540	99.99	75 26	35	.79	280 / 397	1286 / 3033	42 56	100 13			
6/12/84	7012	ARW 4	515	99.98	76 17	22	.68	268 / 373	1322 / 2721	42 55	100 16			
6/12/84	7009	ARW 4	525	99.96	81 25	31	.90	258 / 407	1201 / 3051	43 53	100 15			
6/19/84	7246	ARW 4	480	99.99	80 21	26	.85	261 / 388	1242 / 2881	45 56	100 14			
6/19/84	7144	ARW 4	480	99.99	78 21	27	.87	278 / 389	1345 / 2774	47 60	100 20			
6/21/84	7391	ARW 4	495	99.95	78 25	32	.82	278 / 400	1256 / 3016	44 56	100 21			
6/21/84	7143	ARW 4	510	99.99	77 24	31	.69	275 / 392	1346 / 2848	45 58	100 19			
6/21/84	7015	ARW 4	585	99.99	78 25	32	.71	275 / 382	1295 / 2789	45 58	100 17			
6/22/84	7106	ARW 4	555	99.99	79 23	29	.89	271 / 407	1280 / 3108	42 53	100 20			
6/27/84	7180	ARW 4	510	99.84	72 16	22	.68	268 / 424	1371 / 3421	41 57	100 29			
7/ 2/84	8065	ARW 4	485	99.99	78 15	52	.97	264 / 398	2001 / 3263	43 55	100 19			
7/ 2/84	7534	ARW 4	490	99.99	79 15	13	.93	272 / 405	1260 / 3052	42 53	100 17			
7/ 2/84	7930	ARW 4	495	99.99	82 15	55	.96	270 / 406	1177 / 3136	45 55	100 23			
7/ 3/84	8255	ARW 4	485	99.99	79 25	32	1.09	256 / 422	1246 / 3527	40 51	100 22			
7/ 9/84	8347	ARW 4	510	99.49	81 23	28	.91	255 / 403	1242 / 3080	42 52	100 11			
7/ 9/84	8348	ARW 4	510	99.53	81 25	31	1.07	266 / 413	1151 / 3155	41 51	100 5			
7/10/84	8350	ARW 4	480	99.99	81 16	20	1.15	262 / 396	1158 / 3073	40 49	100 17			
7/11/84	8518	ARW 4	480	99.99	88 17	19	.94	284 / 387	1065 / 2700	47 53	100 65			
7/16/84	8679	ARW 4	520	99.99	87 23	26	.96	259 / 392	1120 / 2831	45 51	100 63			
7/16/84	8407	ARW 4	510	99.95	88 25	28	1.07	262 / 413	1162 / 3034	40 45	100 58			
7/18/84	9211	ARW 4	535	99.99	86 17	20	1.14	264 / 428	1269 / 3390	42 49	100 38			
7/18/84	8520	ARW 4	580	99.99	88 20	23	1.04	254 / 387	1067 / 2600	49 56	100 100			
7/19/84	8523	ARW 4	565	99.99	83 16	19	1.13	256 / 385	1085 / 2723	42 51	100 60			
7/24/84	9175	ARW 4	470	99.99	84 24	29	1.02	254 / 394	1068 / 2870	41 49	100 47			
7/24/84	9272	ARW 4	480	99.99	81 24	30	.86	264 / 398	1138 / 3241	44 54	100 100			
7/25/84	9313	ARW 4	485	99.94	86 19	22	1.18	253 / 400	1064 / 2870	43 50	100 60			
7/25/84	9592	ARW 4	490	99.99	81 13	16	1.39	268 / 392	1224 / 2809	37 46	100 0			
7/31/84	9743	ARW 4	505	99.95	80 20	25	.87	271 / 380	1149 / 2946	44 55	100 0			
7/31/84	9581	ARW 4	520	99.95	78 21	27	.98	278 / 415	1241 / 3255	40 51	100 0			
MEAN:					80.52	20.06	26.88	.94	264.79/	396.94	1240.24/	3025.76	42.67	53.09
ST DEV:					4.71	4.08	8.69	.16	11.27/	15.54	167.59/	236.38	2.90	4.22
4/ 3/81	3681	ARW 8	555	99.99	77 27	35	.88	488 / 846	2167 / 7140	42 55	100 14			
10/28/81	15322	ARW 8	505	99.97	52 13	25	.94	390 / 691	2507 / 7329	23 44	100 8			
MEAN:					64.50	20.00	30.00	.91	439.00/	768.50	2337.00/	7234.50	32.50	49.50
ST DEV:					17.68	9.90	7.07	.04	69.30/	109.60	240.42/	133.64	13.44	7.78
5/ 3/82	4216	ARW 8	550	99.85	68 21	31	1.04	355 / 609	1920 / 5991	34 50	100 10			

\*\*\*\*\* SEE NEXT PAGE \*\*\*\*\*

TABLE OF ASPHALT PROPERTIES COLLECTED  
BY THE OREGON STATE HIGHWAYS DIVISION  
(GROUPED BY SPECIFICATION/GRADE/DATE)

BRAND: CHEVRON

DATE	LAB#	SPEC/GRD	FLASH	SOLUB	PEN (DEG F) (77)	PEN (39)	PEN RATIO	LOSS	KINEMATIC VISCOSITY	ABSOLUTE VISCOSITY	RESIDUE PEN	DUCTILITY #ORIG (77)	DUCTILITY (45)
9/24/82	12158	ARW 8	520	99.94	51	21	37	.90	432 / 693	2582 / 8374	30	53	100 19
					MEAN:	59.50	21.00	34.00	.97	393.50/ 651.00	2251.00/ 7182.50	32.00	51.50
					ST DEV:	12.02	.00	4.24	.10	54.45/ 59.40	468.10/ 1685.04	2.83	2.12
3/25/83	2459	ARW 8	510	99.94	60	19	32	1.14	424 / 734	2746 / 9527	30	50	100 9
5/ 3/83	4198	ARW 8	510	99.95	61	19	31	1.34	390 / 758	2634 / 10163	30	49	100 10
					MEAN:	60.50	19.00	31.50	1.24	407.00/ 746.00	2690.00/ 9845.00	30.00	49.50
					ST DEV:	.71	.00	.71	.14	24.04/ 16.97	79.20/ 449.72	.00	.71

TABLE OF ASPHALT PROPERTIES COLLECTED  
BY THE OREGON STATE HIGHWAYS DIVISION  
(GROUPED BY SPECIFICATION/GRADE/DATE)

BRAND: DOUGLAS

DATE	LAB#	SPEC/GRD	FLASH	SOLUB	PEN (DEG F) (77) (39)	PEN RATIO	LOSS	KINEMATIC VISCOSITY	ABSOLUTE VISCOSITY	RESIDUE PEN %ORIG	DUCTILITY (77) (45)			
3/23/81	2979	ARW 4	555	99.98	105 35	33	.99	390 / 660	1375 / 4541	54 51	100 29			
5/13/81	5890	ARW 4	555	99.95	90 24	27	.56	351 / 525	1392 / 3260	51 57	100 28			
8/ 7/81	10342	ARW 4	550	99.96	85 24	28	.67	355 / 531	1419 / 3668	46 54	100 31			
8/ 7/81	10343	ARW 4	555	99.94	86 25	29	.71	304 / 500	1271 / 3575	45 52	100 32			
8/ 7/81	10947	ARW 4	555	99.97	84 23	27	.61	290 / 462	1210 / 3184	45 54	100 27			
8/11/81	1262	ARW 4	550	99.91	86 24	28	.96	299 / 467	1223 / 3750	43 50	100 34			
8/14/81	11588	ARW 4	555	99.94	81 30	37	.85	288 / 441	1159 / 3173	42 52	100 18			
8/14/81	11090	ARW 4	555	99.96	85 31	36	.81	291 / 470	1202 / 3559	47 49	100 18			
9/ 8/81	12176	ARW 4	540	99.27	81 22	27	.94	295 / 452	1238 / 3742	41 51	100 13			
9/ 8/81	13540	ARW 4	540	99.93	72 20	27	.96	277 / 423	1160 / 3492	40 54	100 14			
9/ 8/81	13347	ARW 4	520	99.83	74 21	28	.92	284 / 459	1212 / 3808	38 51	100 15			
10/ 9/81	14746	ARW 4	500	99.96	75 17	23	.96	268 / 438	1201 / 3231	38 51	100 15			
10/14/81	14823	ARW 4	505	99.93	72 19	26	1.01	256 / 414	1283 / 3585	35 49	100 12			
MEAN:					82.77	24.23	28.92	.84	303.69/	480.15	1257.31/	3582.15	43.46	51.92
ST DEV:					8.92	5.09	4.01	.16	38.36/	64.64	86.69/	364.38	5.35	2.29
5/17/82	4675	ARW 4	505	99.90	109 34	31	1.02	313 / 529	1159 / 3432	56 51	100 23			
6/ 1/82	5262	ARW 4	505	99.99	87 21	24	.95	275 / 435	1310 / 3566	44 48	100 11			
7/13/82	7897	ARW 4	560	99.92	74 24	32	1.08	299 / 490	1472 / 4466	38 51	100 13			
8/30/82	11042	ARW 4	490	99.98	97 26	27	1.71	253 / 432	1130 / 3729	41 42	100 72			
MEAN:					91.75	26.25	28.50	1.19	285.00/	471.50	1267.75/	3798.25	44.75	48.00
ST DEV:					14.86	5.56	3.70	.35	26.48/	46.69	157.38/	461.43	7.89	4.24
5/17/82	4676	ARW 8	560	99.93	65 22	34	.51	545 / 886	2898 / 8210	37 57	100 10			



TABLE OF ASPHALT PROPERTIES COLLECTED  
 BY THE OREGON STATE HIGHWAYS DIVISION  
 (GROUPED BY SPECIFICATION/GRADE/DATE)

BRAND: HUNTWAY

DATE	LAB#	SPEC/GRD	FLASH	SOLUB	PEN (DEG F) (77) (39)	PEN RATIO	LOSS	KINEMATIC VISCOSITY	ABSOLUTE VISCOSITY	RESIDUE PEN %OR16	DUCTILITY (77) (45)
12/ 7/82	16511	AR 2	600	99.99	94 20	21	.54	203 / 288	920 / 1905	58 62	100 100

TABLE OF ASPHALT PROPERTIES COLLECTED  
 BY THE OREGON STATE HIGHWAYS DIVISION  
 (GROUPED BY SPECIFICATION/GRADE/DATE)

BRAND: HUSKY

DATE	LAB#	SPEC/GRD	FLASH	SOLUB	PEN (DEG F)		PEN RATIO	LOSS	KINEMATIC VISCOSITY		ABSOLUTE VISCOSITY		RESIDUE PEN %ORIG		DUCTILITY (77) (45)			
					(77)	(39)				/		/						
3/17/82	2129	AC 10	550	99.86	107	32	30	.11	291	/	409	1057	/	2326	64	60	100	22
3/17/82	2130	AC 20	550	99.93	71	21	30	.14	404	/	579	2012	/	4815	43	61	100	8
4/ 3/84	4324	AR 1	490	99.92	185	49	39	.62	171	/	277	357	/	932	97	52	100	100
4/ 3/84	4325	AR 2	460	99.91	175	43	41	.95	196	/	295	406	/	1183	85	49	100	41
10/28/81	15476	AR 4	505	99.99	64	17	27	.16	433	/	595	2149	/	4953	37	58	100	12
12/ 7/82	16512	AR 4	575	99.99	60	14	23	.41	288	/	427	1720	/	3971	37	62	100	15
4/ 4/84	4326	AR 4	505	99.96	79	18	23	.48	354	/	474	1384	/	3528	45	57	100	13
12/ 8/82	16513	AR 8	500	99.99	47	16	34	.25	438	/	701	2849	/	8325	27	57	100	10

TABLE OF ASPHALT PROPERTIES COLLECTED  
 BY THE OREGON STATE HIGHWAYS DIVISION  
 (GROUPED BY SPECIFICATION/GRADE/DATE)

BRAND: MCCALL

DATE	LAB#	SPEC/GRD	FLASH	SOLUB	PEN (DEG F)		PEN	LOSS	KINEMATIC		ABSOLUTE		RESIDUE		DUCTILITY				
					(77)	(39)	RATIO		VISCOSITY		VISCOSITY		PEN	*ORIG	(77)	(45)			
2/16/83	621	ARW	4	560	99.99	55	18	33	.71	251	/	368	1887	/	3777	29	53	100	40
5/11/83	4636	ARW	4	475	99.98	84	26	31	1.12	243	/	384	1312	/	3561	42	50	100	13
MEAN:					69.50	22.00	32.00	.92	247.00/	376.00	1599.50/	3669.00	35.50	51.50					
ST DEV:					20.51	5.66	1.41	.29	5.66/	11.31	406.59/	152.74	9.19	2.12					

TABLE OF ASPHALT PROPERTIES COLLECTED  
BY THE OREGON STATE HIGHWAYS DIVISION  
(GROUPED BY SPECIFICATION/GRADE/DATE)

BRAND: SHELL

DATE	LAB#	SPEC/GRD	FLASH	SOLUB	PEN (DEG F) (77) (39)	PEN RATIO	LOSS	KINEMATIC VISCOSITY	ABSOLUTE VISCOSITY	RESIDUE PEN %ORIG	DUCTILITY (77) (45)			
3/26/81	3440	AR	2	560	99.94	71 14	20	.27	221 / 310	1217 / 2389	44 62 100 40			
5/ 1/81	4677	AR	2	580	99.68	78 10	13	.21	212 / 282	1030 / 1861	49 63 100 70			
5/27/81	6235	AR	2	590	99.98	84 24	20	.15	179 / 238	937 / 1789	54 64 100 100			
MEAN:					77.67	16.00	17.67	.21	204.00/	276.67	1061.33/	2013.00	49.00	63.00
ST DEV:					6.51	7.21	4.04	.06	22.11/	36.30	142.61/	327.61	5.00	1.00
3/ 9/82	2050	AR	2	470	99.98	83 18	22	.41	185 / 249	1041 / 2041	49 59 100 56			
3/23/82	2575	AR	2	475	99.98	80 18	23	.33	197 / 255	1095 / 2063	49 61 100 55			
8/ 3/82	8974	AR	2	575	99.99	75 16	21	.42	201 / 261	1168 / 2230	47 63 100 100			
MEAN:					79.33	17.33	22.00	.39	194.33/	255.00	1101.33/	2111.33	48.33	61.00
ST DEV:					4.04	1.15	1.00	.05	8.33/	6.00	63.74/	103.36	1.15	2.00
4/19/83	2939	AR	2	565	99.92	52 11	21	.27	243 / 331	1905 / 3554	34 65 100 10			
3/25/81	3441	AR	4	575	99.90	47 13	28	.26	277 / 380	1973 / 3877	31 66 100 10			
5/ 1/81	4528	AR	4	590	99.72	45 6	13	.21	283 / 381	2008 / 3971	29 64 100 0			
8/ 6/81	10972	AR	4	515	99.95	51 17	33	.27	268 / 449	1810 / 3562	32 63 100 15			
8/11/81	11138	AR	4	515	99.93	50 17	34	.31	269 / 373	1783 / 3919	30 60 100 3			
MEAN:					48.25	13.25	27.00	.26	274.25/	395.75	1893.50/	3832.25	30.50	63.25
ST DEV:					2.75	5.19	9.70	.04	7.09/	35.68	113.45/	184.22	1.29	2.50
3/ 8/82	1759	AR	4	480	99.99	52 14	27	.22	269 / 303	1754 / 3305	36 69 100 16			
3/ 9/82	2051	AR	4	490	99.98	53 12	23	.41	243 / 367	1894 / 3990	31 58 100 6			
3/23/82	2576	AR	4	490	99.98	49 11	22	.40	258 / 361	2045 / 4394	30 61 100 8			
8/ 4/82	8925	AR	4	575	99.97	50 12	24	.31	247 / 338	1991 / 3951	31 62 100 84			
8/18/82	9984	AR	4	545	99.89	46 10	22	.34	261 / 352	3366 / 4250	29 63 100 0			
9/27/82	12373	AR	4	575	99.99	52 14	27	.15	225 / 387	1810 / 3501	33 64 100 19			
11/12/82	14652	AR	4	600	99.94	46 12	26	.22	255 / 341	2136 / 4061	29 63 100 14			
MEAN:					49.71	12.14	24.43	.29	251.14/	349.86	2142.29/	3921.71	31.29	62.86
ST DEV:					2.87	1.46	2.23	.10	14.40/	26.51	555.68/	390.10	2.50	3.34
6/ 9/83	6204	AR	4	525	99.97	92 30	33	.15	357 / 547	1658 / 3912	59 64 100 37			
3/25/81	3442	AR	8	595	99.88	35 10	29	.25	371 / 514	3163 / 6669	24 69 100 0			
8/11/81	11139	AR	8	520	99.95	31 8	26	.43	387 / 524	3724 / 7888	15 48 100 0			
MEAN:					33.00	9.00	27.50	.34	379.00/	519.00	3443.50/	7278.50	19.50	58.50
ST DEV:					2.83	1.41	2.12	.13	11.31/	7.07	396.69/	861.96	6.36	14.85
3/ 8/82	1760	AR	8	510	99.93	29 3	10	.16	332 / 472	3499 / 7266	20 69 100 5			
10/10/83	15332	ARW	2	535	99.88	112 26	23	1.23	216 / 314	891 / 2265	61 50 100 10			

\*\*\*\*\* SEE NEXT PAGE \*\*\*\*\*

TABLE OF ASPHALT PROPERTIES COLLECTED  
 BY THE OREGON STATE HIGHWAYS DIVISION  
 (GROUPED BY SPECIFICATION/GRADE/DATE)

BRAND: SHELL

DATE	LAB#	SPEC/GRD	FLASH	SOLOB	PEN (DEG F) (77)	PEN (39) (39)	PEN RATIO	LOSS	KINEMATIC VISCOSITY	ABSOLUTE VISCOSITY	RESIDUE PEN %ORIG	DUCTILITY (77)	DUCTILITY (45)
6/20/84	7014	ARW 4	485	99.97	80	25	31	.86	273 / 391	1477 / 3098	47 59	100	32

TABLE OF ASPHALT PROPERTIES COLLECTED  
BY THE OREGON STATE HIGHWAYS DIVISION  
(GROUPED BY SPECIFICATION/GRADE/DATE)

BRAND: SOUND

DATE	LAB#	SPEC/GRD	FLASH	SOLUB	PEN (DEG F)		PEN RATIO	LOSS	KINEMATIC VISCOSITY		ABSOLUTE VISCOSITY		RESIDUE PEN %ORIG		DUCTILITY (77) (45)				
3/16/83	1789	AR	2	500	99.92	138	68	49	.30	283	/	439	939	/	2418	78	57	100	100
11/15/82	15638	AR	4	515	99.96	92	35	38	.32	290	/	427	1355	/	3168	54	59	100	34
7/10/84	8517	ARW	2	445	99.95	133	34	26	1.61	203	/	334	762	/	2199	64	48	100	100
6/27/84	7392	ARW	4	530	99.99	158	42	27	.85	186	/	282	550	/	1297	87	55	100	100
7/ 5/84	8353	ARW	4	410	99.99	161	68	42	1.96	181	/	310	582	/	2136	64	39	100	100
7/11/84	8795	ARW	4	520	99.99	72	16	22	.36	271	/	374	1540	/	3294	44	61	100	65
7/11/84	8840	ARW	4	460	99.99	80	17	22	.91	272	/	407	1533	/	3497	45	56	100	65
MEAN:					117.75	35.75	28.25	1.02	227.50/	343.25	1051.25/	2556.00	60.00	52.75					
ST DEV:					48.33	24.64	9.46	.67	50.85/	57.35	560.48/	1031.44	20.22	9.54					

TABLE OF ASPHALT PROPERTIES COLLECTED  
BY THE OREGON STATE HIGHWAYS DIVISION  
(GROUPED BY SPECIFICATION/GRADE/DATE)

BRAND: UNION

DATE	LAB#	SPEC/GRD	FLASH	SOLUB	PEN (DEG F)		PEN RATIO	LOSS	KINEMATIC VISCOSITY		ABSOLUTE VISCOSITY		RESIDUE PEN %DRIG		DUCTILITY (77) (45)				
4/14/81	4237	AR	8	530	99.95	66	23	35	.80	629	/	1030	3324	/	11195	38	58	100	11
5/26/81	6202	AR	8	510	99.99	71	27	38	.81	532	/	943	2636	/	8776	37	52	100	11
7/31/81	9254	AR	8	510	99.97	65	25	38	.50	515	/	736	2571	/	7709	37	57	100	14
10/ 8/81	14264	AR	8	540	99.87	54	14	25	.93	408	/	670	2390	/	7327	29	51	100	10
MEAN:					64.00	22.25	34.00	.76	521.00/	844.75	2730.25/	8751.75	35.25	54.50					
ST DEV:					7.16	5.74	6.16	.18	90.54/	169.64	409.29/	1740.43	4.19	3.51					
5/ 6/82	4338	AR	8	600	99.99	58	17	29	.91	388	/	661	2374	/	7896	30	52	100	8
5/ 4/82	4249	ARW	2	570	99.56	103	25	24	.75	237	/	355	812	/	1977	55	53	100	13
4/14/81	4236	ARW	4	595	99.94	81	21	25	.64	352	/	542	1404	/	3528	47	55	100	30
4/30/81	4617	ARW	4	605	99.76	85	24	28	.66	363	/	574	1388	/	4007	45	53	100	19
5/26/81	6170	ARW	4	610	99.93	86	26	30	.75	368	/	575	1429	/	4035	46	53	100	24
5/26/81	6201	ARW	4	580	99.94	86	26	30	.72	364	/	577	1440	/	3778	46	53	100	21
6/ 2/81	7992	ARW	4	560	99.93	83	21	25	.59	354	/	524	1399	/	3436	45	54	0	0
8/ 3/81	9195	ARW	4	570	99.96	82	35	43	.58	361	/	561	1448	/	3759	47	57	100	34
8/ 5/81	9546	ARW	4	560	99.95	80	26	33	.45	350	/	525	1418	/	3572	45	56	100	26
8/ 5/81	10866	ARW	4	555	99.95	83	24	29	.83	298	/	453	1192	/	3453	45	57	100	22
10/ 8/81	14263	ARW	4	535	99.86	80	18	23	.85	280	/	457	1217	/	3215	43	54	100	17
MEAN:					82.89	24.56	29.56	.67	343.33/	532.00	1370.56/	3642.56	45.44	54.67					
ST DEV:					2.37	4.80	5.92	.13	31.69/	48.16	96.29/	273.46	1.24	1.66					
5/ 6/82	4337	ARW	4	500	99.97	81	23	28	1.13	285	/	465	1371	/	4023	42	52	100	12
8/15/83	10369	ARW	4	535	99.88	75	11	15	.65	241	/	342	1207	/	2928	44	59	100	31
8/15/83	10370	ARW	4	530	99.91	76	15	20	.72	243	/	354	1260	/	3029	44	58	100	28
8/30/83	11100	ARW	4	515	99.99	77	20	26	.66	222	/	345	1257	/	2866	44	57	100	10
MEAN:					76.00	15.33	20.33	.68	235.33/	347.00	1241.33/	2941.00	44.00	58.00					
ST DEV:					1.00	4.51	5.51	.04	11.59/	6.24	29.77/	82.27	.00	1.00					
7/ 5/84	8344	ARW	4	510	99.99	75	25	33	.83	271	/	419	1342	/	3480	38	50	100	13

TABLE OF ASPHALT PROPERTIES COLLECTED  
BY THE OREGON STATE HIGHWAYS DIVISION  
(GROUPED BY SPECIFICATION/GRADE/DATE)

BRAND: US OIL

DATE	LAB#	SPEC/GRD	FLASH	SOLUB	PEN (DEG F)		PEN	LOSS	KINEMATIC		ABSOLUTE		RESIDUE		DUCTILITY				
					(77)	(39)	RATIO		VISCOSITY		VISCOSITY		PEN	%ORIG	(77)	(45)			
6/12/84	7013	AR	4	540	99.96	93	28	30	.46	361	/	537	1412	/	3621	53	57	100	21
9/27/83	13814	ARW	4	510	99.97	93	21	23	.65	323	/	548	1500	/	4597	46	49	100	0
10/13/83	15926	ARW	4	525	99.92	89	21	24	.87	343	/	559	1422	/	4232	45	51	100	31
MEAN:					91.00	21.00	23.50	.76	333.00/	553.50	1461.00/	4414.50	45.50	50.00					
ST DEV:					2.83	.00	.71	.16	14.14/	7.78	55.15/	258.09	.71	1.41					
1/ 5/84	20756	ARW	4	545	99.93	97	30	31	1.03	378	/	635	1388	/	4681	47	48	100	0
3/26/84	3569	ARW	4	595	99.98	91	24	26	.77	362	/	540	1281	/	3418	47	52	100	58
7/19/84	8524	ARW	4	545	99.99	77	17	22	.44	370	/	528	1652	/	3740	47	61	100	37
MEAN:					88.33	23.67	26.33	.75	370.00/	567.67	1440.33/	3946.33	47.00	53.67					
ST DEV:					10.26	6.51	4.51	.30	8.00/	58.62	190.96/	656.29	.00	6.66					



TABLE OF ASPHALT PROPERTIES COLLECTED  
BY THE OREGON STATE HIGHWAYS DIVISION  
(GROUPED BY SPECIFICATION/GRADE/DATE)

BRAND: WITCO

DATE	LAB#	SPEC/GRD	FLASH	SOLUB	PEN (DEG F) (77) (39)	PEN RATIO	LOSS	KINEMATIC VISCOSITY	ABSOLUTE VISCOSITY	RESIDUE PEN %ORIG	DUCTILITY (77) (45)			
4/ 6/81	3743	AR 2	515	99.83	80 16	20	.81	198 / 296	1105 / 2179	47 59	100 100			
4/13/81	3906	AR 2	510	99.83	85 17	20	.66	201 / 255	1052 / 1876	53 62	100 100			
5/27/81	6169	AR 2	530	99.87	80 16	20	.56	199 / 257	1077 / 1830	55 69	100 100			
6/ 2/81	6666	AR 2	560	99.82	78 20	26	.52	211 / 272	1143 / 2104	51 65	100 100			
6/ 2/81	7401	AR 2	475	99.86	83 24	29	.41	196 / 352	1044 / 1974	56 67	0 0			
7/ 1/81	8653	AR 2	525	99.83	81 16	20	.54	197 / 258	1026 / 1886	52 64	100 100			
8/ 3/81	8656	AR 2	490	99.91	79 30	38	.56	197 / 262	1086 / 2038	53 67	100 100			
9/29/81	13768	AR 2	515	99.56	83 15	18	.60	204 / 272	1088 / 2119	49 59	100 100			
9/29/81	13759	AR 2	520	99.88	78 14	18	.75	210 / 273	1162 / 2178	48 62	100 100			
9/29/81	13778	AR 2	515	99.55	80 17	21	.55	194 / 262	1034 / 1840	53 66	100 100			
9/30/81	14081	AR 2	529	99.87	84 25	30	.54	202 / 265	1053 / 1920	53 63	100 100			
10/ 5/81	14312	AR 2	515	99.70	85 20	24	.51	203 / 259	1024 / 1751	52 61	100 100			
10/ 9/81	14512	AR 2	510	99.75	87 16	18	1.00	203 / 255	1007 / 1901	56 64	100 100			
MEAN:					81.77	18.92	23.23	.62	201.15/	272.15	1069.31/	1968.92	52.15	63.69
ST DEV:					2.92	4.73	6.02	.16	5.15/	26.45	46.77/	141.23	2.82	3.09
7/ 9/82	7444	AR 2	550	99.91	78 19	24	.49	215 / 270	1195 / 2053	52 67	100 100			
7/ 9/82	7443	AR 2	545	99.81	86 21	24	.45	195 / 242	1034 / 1744	59 69	100 62			
8/25/82	10750	AR 2	520	99.82	75 18	24	.42	208 / 263	1212 / 2103	53 71	100 100			
10/21/82	13994	AR 2	525	99.79	78 19	24	.48	203 / 293	1107 / 1941	50 64	100 100			
10/21/82	14153	AR 2	520	99.82	76 18	24	.40	204 / 291	759 / 2129	49 64	100 100			
11/10/82	14590	AR 2	600	99.81	75 25	33	.34	205 / 254	1188 / 1988	51 68	100 100			
MEAN:					78.00	20.00	25.50	.43	205.00/	268.83	1082.50/	1993.00	52.33	67.17
ST DEV:					4.15	2.68	3.67	.06	6.54/	20.25	172.14/	140.66	3.56	2.79
4/ 6/81	3744	AR 4	535	99.85	51 10	20	.53	267 / 342	1937 / 3487	30 59	100 2			
4/13/81	3907	AR 4	525	99.86	51 10	20	.44	259 / 341	1943 / 3538	33 65	100 15			
10/29/81	15380	AR 4	510	99.94	49 9	18	.22	256 / 345	1918 / 3364	31 63	100 3			
MEAN:					50.33	9.67	19.33	.40	260.67/	342.67	1932.67/	3463.00	31.33	62.33
ST DEV:					1.15	.58	1.15	.16	5.69/	2.08	13.05/	89.45	1.53	3.06
7/ 7/82	7120	AR 4	510	99.52	43 10	23	.36	298 / 388	2584 / 4541	29 67	100 2			
7/ 7/82	7134	AR 4	540	99.49	46 10	22	.08	275 / 333	2194 / 3541	33 72	100 2			
7/ 9/82	7442	AR 4	540	99.69	46 10	22	.20	280 / 359	2260 / 4071	31 67	100 2			
8/ 3/82	8872	AR 4	570	99.78	44 10	23	.25	287 / 503	2478 / 4525	29 66	100 42			
8/ 3/82	8874	AR 4	535	99.78	46 10	22	.24	281 / 479	2309 / 4142	30 65	100 44			
10/ 4/82	12681	AR 4	525	99.82	75 16	21	.40	206 / 262	1220 / 2167	49 65	100 100			
MEAN:					50.00	11.00	22.17	.26	271.17/	387.33	2174.17/	3831.17	33.50	67.00
ST DEV:					12.31	2.45	.75	.11	32.88/	90.82	489.21/	893.65	7.74	2.61
7/ 5/84	8345	AR 4	575	99.99	46 13	28	.00	313 / 402	2432 / 4388	30 65	100 8			
4/ 6/81	3745	AR 8	550	99.87	26 1	23	.42	392 / 535	4382 / 8718	18 69	100 1			

\*\*\*\*\* SEE NEXT PAGE \*\*\*\*\*

TABLE OF ASPHALT PROPERTIES COLLECTED  
 BY THE OREGON STATE HIGHWAYS DIVISION  
 (GROUPED BY SPECIFICATION/GRADE/DATE)

BRAND: WITCO

DATE	LAB#	SPEC/GRD	FLASH	SOLUB	PEN (DEG F) (77) (39)	PEN RATIO	LOSS	KINEMATIC VISCOSITY	ABSOLUTE VISCOSITY	RESIDUE PEN %ORIG	DUCTILITY (77) (45)
7/ 7/82	7141	AR 8	535	99.75	31 5	16	.32	359 / 469	3860 / 7171	20 65	100 1