NATIONAL BUREAU OF STANDARDS REPORT

5459

FACILITIES AVAILABLE AT THE NATIONAL BUREAU OF STANDARDS FOR STRUCTURAL DETERMINATION OF ASPHALTS

bу

Shigeru Ishihara



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by

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Jointly Sponsored by
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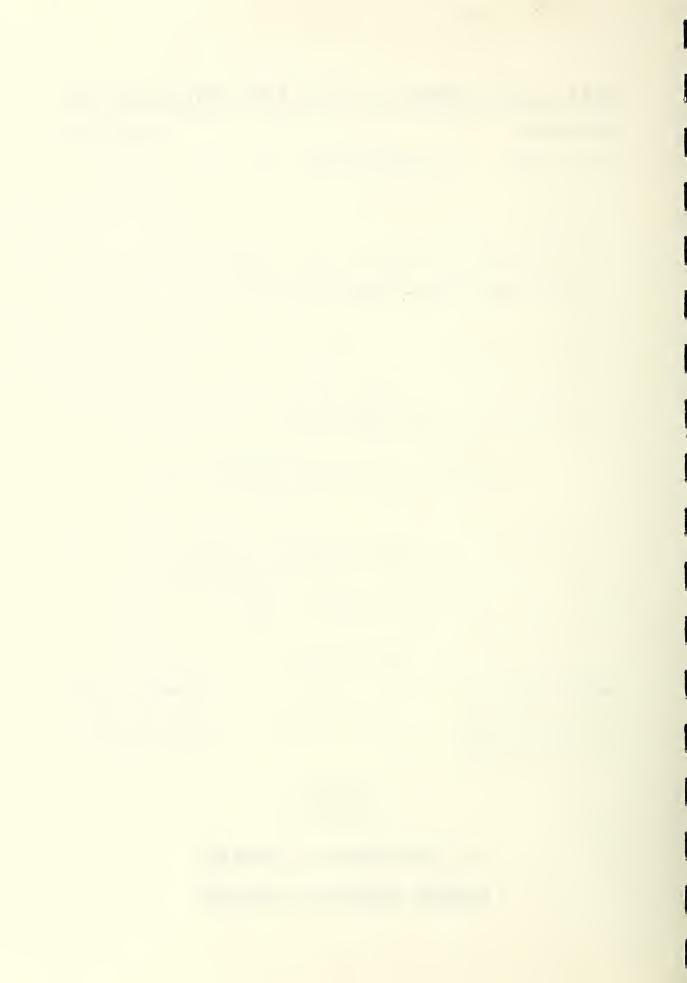
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INTRODUCTION

This report is presented to the members of the Asphalt Roofing Industry Bureau in answer to the request made in the committee meeting of June 1957. A survey of the methods and equipment used in the determination of the physical structure present in systems composed of organic compounds has been conducted and herein are brief descriptions of methods and instruments which may be applicable for study with asphalts.

The first five sections are the areas of study where the survey showed promise of positive evaluation in measuring the structure of asphalt in entirety. Sections 6, 7, and 9 are brief descriptions of techniques that have been employed on pure compounds and further developments for complex mixtures are desirable. Section 8, on use of the electron microscope, has been mentioned, but at the present time there is no full-time electron microscopist on the Bureau staff.

Many methods of structural determination of organic compounds were not included because the techniques developed to date were not applicable for exploratory work with the entire asphalt. Among these methods are: (1) nuclear magnetic resonance; (2) paramagnetic resonance; (3) direct measurement of the dynamic bulk modulus; (4) far-infrared analysis, and (5) mass spectrophotometric analysis.

1. THERMAL EXPANSION STUDY

Thermal expansion of high molecular weight organic compounds exhibits features quite different from the usual behavior of simple organic compounds. The volume-temperature curve is characterized by the presence of a temperature, or a range of temperatures, which separates the curve into two portions corresponding to markedly different coefficients of thermal expansion (1).1

 $[\]frac{1}{2}$ Numbers in parentheses refer to references at the end of this paper.



second-order transition temperature. As the material is gradually heated past this temperature, there appears to be a phase change, without apparent latent heat, resulting in larger values of the coefficient of thermal expansion and in changes in physical properties. The material softens and acquires rubber-like elastic properties(2). On further heating the material becomes completely amorphous or liquid if, meanwhile, it does not decompose.

L. A. Wood and N. Bekkedahl have made studies on unvulcanized rubber and found that this property was time dependent. When amorphous rubber was maintained at any constant temperature between -50°C and +20°C, changes in volume were observed. The rate of change of the volume vs. temperature curve was somewhat like a probability curve with a maximum rate near -25°C for unvulcanized rubber (3).

The volume change at constant temperature was attributed to crystallization of rubber and had been substantiated by x-ray diffraction (4), heat capacity (5), light absorption (6), birefringence (7), and hardness measurements (6). At temperatures below -50°C the tendency of the molecules to orient themselves is restricted by high viscosity; above 20°C this tendency is opposed by increased molecular motion.

J. D. Hoffman of the Dielectric Section at the National Bureau of Standards and others have observed similar behavior in thermal expansion in materials such as polyethylene and Kel-F2/, and have shown crystal-like structures associated with these compounds.

At times volume-change measurements are more sensitive than x-ray diffraction for detecting structural changes. X-ray diffraction methods often require crystal-like particles with more than 50 angstroms path length for detection. In the work with structure formation in Kel-F, J. D. Hoffman was able to detect low degrees of crystallinity where x-ray diffraction patterns showed nothing.

^{2/} Polychlorotrifluoroethylene.



Many time-dependent properties of coating-grade asphalts strongly indicate that some structural formation occurs during storage; for example, changes in softening point, degrees of blister formation, penetration, bulk viscosity (8), elongation with constant load, etc., have been observed.

If the thermal expansion study of asphalt gives us positive results, i.e., a volume-temperature-time relationship, information from the data will give us our insight on the proper approach for methods of studying structure in asphalt.

The amount of time required for this study is dependent on the time required for the change in physical properties. Data on viscosity measurement indicated that up to 30 weeks standing at room temperature are required for some road asphalts before viscosities reach a steady value (8). In one coating-grade asphalt, 400 hours elapsed before elongation at constant load approached a steady value (10). A year's time is a reasonable period for this work, since specimens must be subjected to various temperatures for various lengths of time. Once test specimens are prepared, the work required to gather data would be a matter of a few minutes each day.

The estimated cost of balance, controlled temperature bath, and density accessories is \$1,200 (1957).

2. ELASTIC MODULI DETERMINATION BY SONIC METHOD

The bulk, shear, and Young's moduli of a material have always been important properties to be considered by structural engineers and architects. These elastic moduli result from measuring deformation under compressional, shear, and linear stresses. Sonic methods for determination of elastic moduli have been used for 18 years and have certain advantages over static methods. These dynamic moduli are obtained with minute alternating stresses below the elastic limit of the material and do not give rise to complex "creep" effects or elastic hysteresis. Also, these minute stresses in no way affect the specimen; hence, repeated tests can be made on the same specimen.

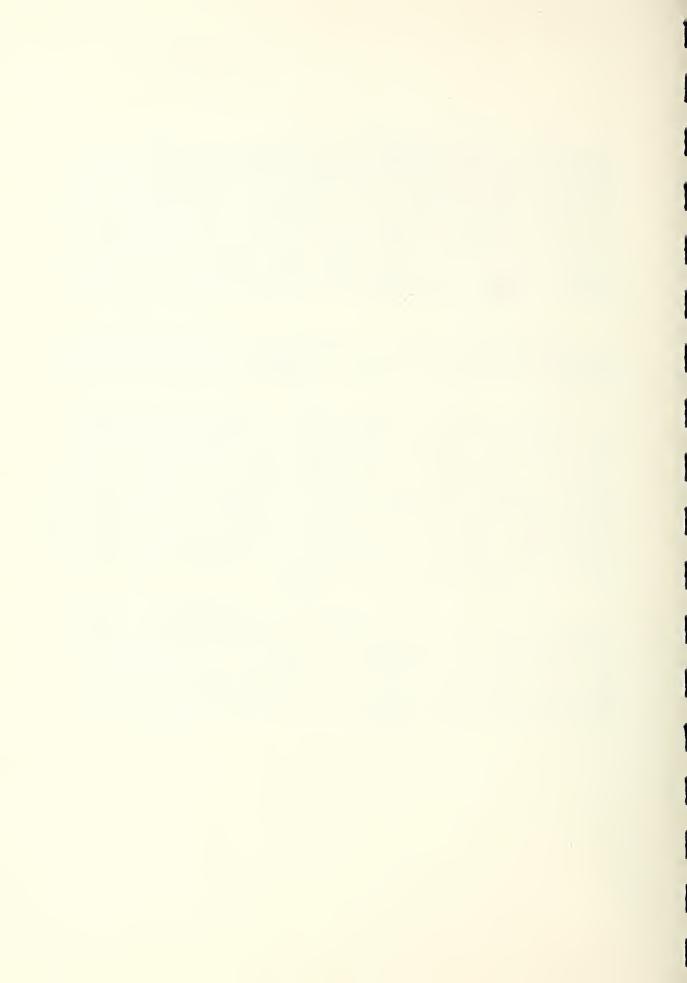


The velocity of sound in a medium is related to the density and elastic moduli of the material. It is also related to the fundamental resonating frequency of sound waves and dimensions of the material. The apparatus used for this study measures the fundamental resonant frequency to determine elastic moduli of a substance. Briefly, vibrations are induced in the specimen by an electromagnetic driver. At resonance the reinforced amplitude of vibration is detected by a crystal pickup and, together with an audio signal of the same frequency, produces a Lissajou pattern on an oscilloscope.

Sam Spinner, et. al., determined Young's modulus of glasses and concrete (9). In concrete, correlation between resonant frequency and both the compressional and flexural strength of the material was found.

Sonic equipment is available at the National Bureau of Standards for exploratory work in asphalts in the Glass and Concreting Materials Sections. Although this method is useful in studies of glass and concrete at room temperature, non-rigid and viscoelastic materials such as coatinggrade asphalts may require different ambient conditions because dimensional stability of the specimen is an important property for accurate and informative measurements. On the other hand, this study may lead to interesting and empirical relationships between apparent elastic moduli and various physical characteristics of asphalt.

Allowing two months to develop a procedure for determining elastic moduli of asphalts, a minimum of one year is estimated for comprehensive work on the project. If the fragments of information gotten in the developmental state show definite promise, equipment should be bought. The cost, which includes oscilloscope, amplifier, signal generator, crystal pickup and frequency counter will be about \$2000.00 (1957 price).



3. DIFFERENTIAL THERMAL ANALYSIS

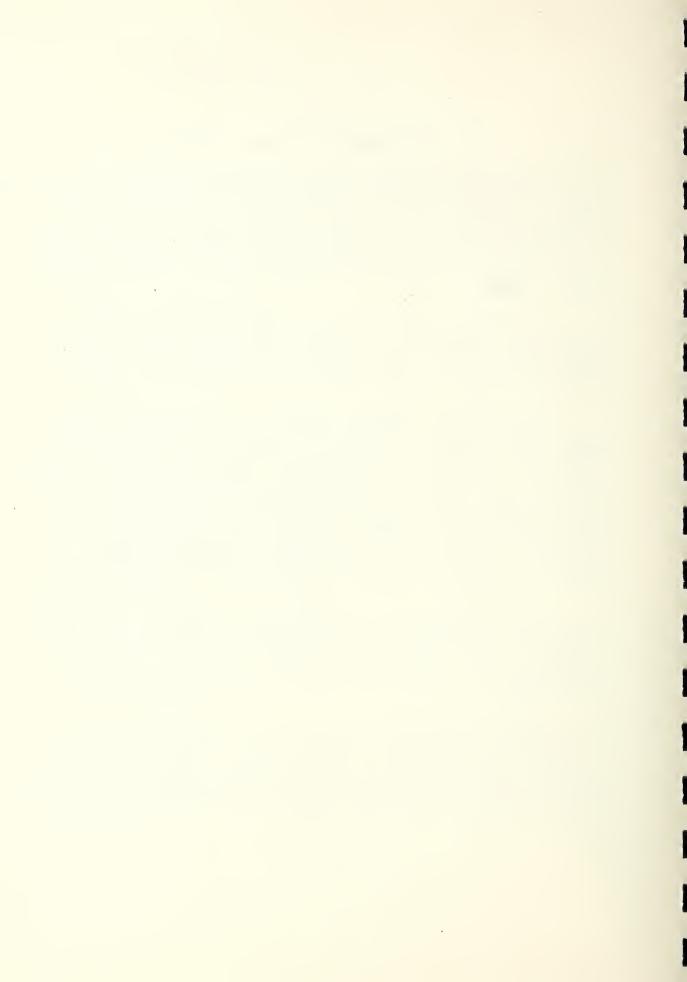
In all substances, changes in phase and chemical reactions are accompanied by energy changes which generally manifest themselves as heat energy. Some produce heat and are called exothermic; some require heat for the change and are called endothermic. The phase change which applies to asphalt is the transition from "structured" to "liquid" state. Differential thermal analyzer is the apparatus used for detecting phase changes.

The method of Differential Thermal Analysis (DTA) has reached the prominence of other techniques such as x-ray diffraction, microscopes, etc. as a helpful research and control tool (11). This impetus is attributed to rapid advances in the field of electronics.

A differential thermal analyzer is composed of three major components: a sample holder, a controlled source of heat, and a device for the measurement of heat generated or absorbed. Two substances, one inert material, such as aluminum oxide or Nujol, and the other, the specimen, are heated at a constant rate in a well-designed furnace. When the specimen undergoes a phase change, the analyzer detects the heat generated or absorbed by measuring the thermocouple voltages induced by the temperature difference between the sample and the reference. A difference of one microvolt can be amplified electronically and recorded automatically.

H. E. Kissinger, E. S. Newman and others of the Constitution and Microstructure and Concreting Materials Sections of the National Bureau of Standards have made studies on numerous products such as plasters, alloys, metals, crystals, and glass (12). Others have made studies on greases and soaps (13).

With the advice and assistance of H. E. Kissinger, a few exploratory runs were made on two asphalts, Midcontinent-200 and California-200. Differences were observed in the differential heating and cooling curves of the two asphalts. The California-200 heated erratically while Midcontinent-200 heated uniformly when Nujol was used as a reference material.



The asphalts were cast into glass vials containing a chromel-alumel thermocouple. Allowing sufficient time for cooling, differential heating and cooling curves were made with Nujol as the inert reference. Results indicated that 3 or 4 months time of intensive investigation in technique would be necessary before reliable and reproducible results could be made for evaluation.

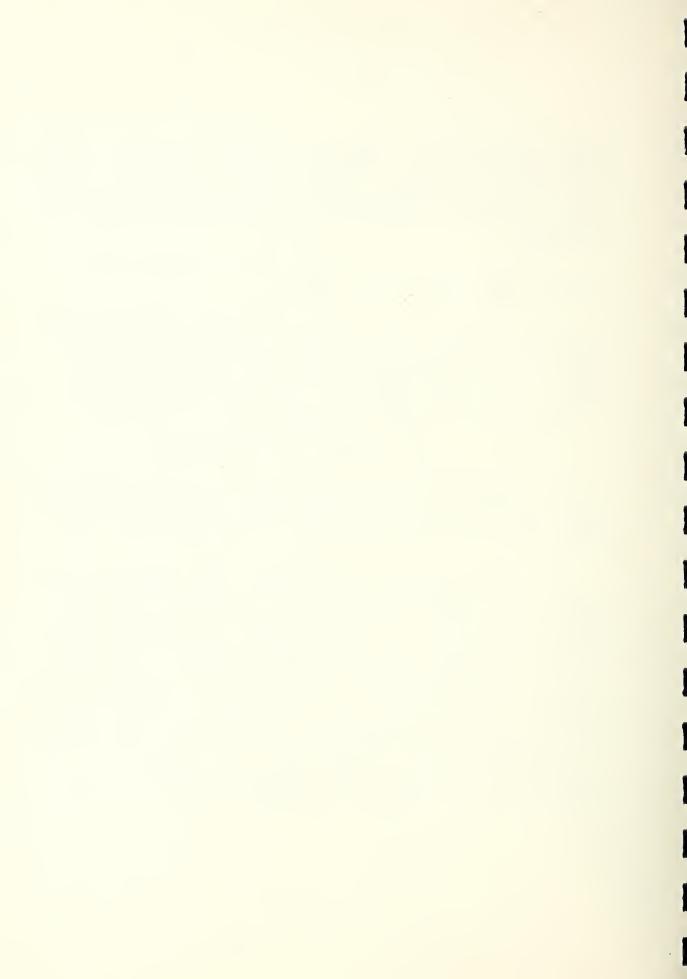
The DTA is available commercially at prices ranging from \$2,000 to \$6,000 (1957 price).

Evaluation in this work is necessarily empirical unless each component contributing toward temperature changes is studied separately. Manifested in the measurements are specific heats, latent heat, chemical reaction, heats of vaporization, conductivity, and various factors such as sample size, sample geometry, rate of heating, noise level of amplifiers, stability of potentiometer, etc. (16).

4. X-RAY SHADOW MICROSCOPE STUDY

The x-ray microscope study applied to asphalt provides a direct means of "seeing" structure formation if the structure size is large enough and if the structure has x-ray absorption coefficients different from those of the bulk of the asphalt.

The General Electric shadow microscope operates on the principle of using a point source of x-rays and casting an x-ray shadow of the specimen on a fluorescent screen or camera. The x-ray microscope has the advantage of viewing thru material opaque to visible light. Also the specimen is not subjected to electron beams or vacuum. This cannot be done with electron microscopes. In metallurgy, it is used for identification of inclusions, study of grain boundary precipitations, structure study of various layers of electroplated coatings, etc. In chemistry, it is used for structure studies of organic oils and coatings, studies of film changes due to "weathering", and penetration of dyes and adhesives. Magnifications up to 1500 diameters are possible. This study can be informative and can be made in conjunction with a thermal expansion study.



The Metal Physics Section at the National Bureau of Standards has recently installed a General Electric x-ray microscope. It will be used for studies in metals and alloys. With asphalts, thin films can be etched on the surfaces with ethyl ether or naphtha to observe structures (17).

Three month's time should be sufficient to develop a technique for observing structures with this method.

The actual cost of services for the use of the x-ray microscope has not been quoted, because the time required by skilled technicians for this study is hard to determine. A reasonable figure would be around \$10.00 per photograph.

5. PARTICLE SIZE DETERMINATION BY THE METHOD OF LOW-ANGLE X-RAY SCATTERING

A low-angle x-ray scattering apparatus is being developed by A. S. Posner of the Dental Research Section. In principle, the scattering of x-rays is dependent on the size and shape of the particles. The smaller the lattice spacing, the larger is the angle of x-ray scattering. For larger spacing, such as those of higher molecular weight biological compounds, the angle of x-ray scattering is of the order of 2 minutes (18). The magnitudes of these scattered angles in low-angle x-ray scattering studies is too small to be measured by conventional x-ray diffraction methods.

The intensity spectrum of the scattered x-rays is mathematically treated to give us information on the size and shape of the physical structures present in solids or solutions. By this method structures of different varieties of carbon and age hardening of alloys have been studied (18).

The instrument is designed to measure particle sizes of 10 A to 1000 A. The instrument is expected to be completed by Fall 1957.

Unless a few samples of asphalts are subjected to low-angle x-ray analysis, no evaluation can be made which will tell whether this method is applicable or not. It has been made



clear that the cost of extended use of this technique, which requires highly skilled technicians for correct interpretation, would include considerable overhead. The actual cost has not been quoted.

6. MOLECULAR WEIGHT DISTRIBUTION STUDY

In this study, substances in dilute solution are placed in an ultracentrifuge at 65,000 rpm. The materials in solution will distribute themselves along the radius of rotation according to their particle weight. Optical means, such as light transmission and index of refraction are used for measuring the extent of sedimentation. For a monodisperse substance, a sharp boundary is found in the solution-solvent system, while a diffuse boundary indicates the presence of a polydisperse substance. The molecular weight or particle weight can be determined by the rate of shift of the boundary and the distribution curve of the molecular weight can be found from its diffuseness (14).

In a complex system such as asphalt where countless numbers of compounds are present, a proper procedure for measuring "structure" is difficult to foresee.

In order to apply this technique to structural determination of whole asphalt, it must first be established that "structured" asphalt is not adversely affected when it is dispersed in a solvent.

S. G. Weissberg, who has done work on plasma volume expanders and polystyrene, recommends study of a more simplified fraction of asphalt, rather than the total asphalt, to greatly reduce the task in establishing a proper procedure and to improve the chances of getting correct and positive results.

The time and cost required in this study are very difficult to estimate. Many variables such as viscosities, concentration, solvent interaction, particle shape, stability of "structured" asphalt in dispersion, etc., must be considered and accounted for. The equipment necessary is subject to these variables.



This study should be considered a long-term project even in the developmental state, because the method is tedious and time-consuming. Furthermore, development in technique has not reached a point where accurate and reliable results are obtainable with any assurance.

7. DIELECTRIC STUDY

A compound is said to have dielectric properties when the center of negative charges does not coincide with the center of positive charges. The extent of displacement of the charges is manifested in the dielectric constant and measured almost entirely by the ratio of capacity of a condensor with and without the medium. The dielectric constants of almost all petroleum products are in the range of values 2.0 to 2.9 (15). Many factors cause changes in the dielectric constant, one of which is a structural formation.

Information on dielectric changes would be desirable in any study of changes in physical structure. Unfortunately, the precision of the measurement on relatively non-polar materials is limited and small changes are difficult to detect and reproduce. Dimensional stability is another desirable feature in accurate dielectric studies.

Literature study and consultation with experts indicated that considerable development in technique and instrumentation is needed to interpret the results obtained for such a complex and non-polar material as asphalt.

Both on a theoretical and practical basis, dielectric studies should be considered a long-term project. From a theoretical standpoint, the opinion is that there would be very little change in dielectric properties of asphalts. Mathematical relationships between dielectric constant and index of refraction, polarization, etc., indicate that the effect of the change in the index of refraction on the dielectric constant of asphalt would be so overwhelming that it would mask the changes contributed by structures.

Estimate of cost and time involved cannot be determined unless a few exploratory runs are made on asphalts.



8. ELECTRON MICROSCOPE STUDY

When a particle to be observed under a microscope is of the order of the wave length of the light waves used for observation, considerable loss in resolution and intensity (due to diffraction and reflection) of the image is experienced. In the electron microscope a beam of electrons is focused on the sample and either the scattered rays or the passing beam of electrons is focused on the image screen. Magnification up to 50,000 diameters or higher is possible with this instrument. It is with this instrument that larger molecules have been first observed.

There are two objectionable features when asphalt is observed with this instrument. The specimen must be stable to electron bombardment and thin sections of asphalts at very high vacuum must be used. If the material is unstable under these conditions, considerable work in the development of technique and in the maintenance of the expensive instrument is to be expected.

Estimate of cost and time involved cannot be determined unless a few exploratory runs are made on asphalt.

9. METHOD OF INFRARED ANALYSIS

Infrared absorption spectra have long been recognized as a convenient means for studying the structure of organic molecules. The interpretations of the spectra are based on the energy interactions of the molecule and the infrared radiation. This radiation gives rise to vibrations of the constituent atoms and molecular rotations. For simple or highly symmetrical molecules, the determination of the normal modes of vibration (in effect, the normal modes of vibrations are the varieties of vibration possible without affecting the position of the center of mass of the molecules) and the calculation of the absorption frequency are relatively simple and straightforward. For more complicated organic compounds the computations become increasingly difficult because with each additional atom the normal modes of vibration are increased by three. However, some useful information can be gotten by empirically comparing the spectra with those obtained for simpler compounds with known structure.



The study of structural formation is best accomplished by observing the changes in the absorption spectrum of either thin films or KBr pelleted samples of powdered asphalt. The interpretation of these changes must be substantiated by controlled and comparative methods.

The Building Technology Division will have the use of an infrared spectrophotometer in the near future. This spectrophotometer is a dual-beam, automatic-recording type in the range 2-15 microns.

Three month's time should be sufficient to develop a technique for this study. If the results are positive, this study may develop into a new method of measuring different types of structural formation in asphalts.

The cost of equipment will be merely the cost of accessories such as KBr powder, specimen die, NaCl cells, etc., which are necessary for almost all infrared study. The cost will be about \$500.00.



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