**EVALUATION OF ASPHALT-AGGREGATE
DISTRIBUTION IN BITUMINOUS MIXES
BY AUTORADIOGRAPHY**

APRIL 1962

NO. 11

**Joint
Highway
Research
Project**

by
D. R. LAMB

**PURDUE UNIVERSITY
LAFAYETTE INDIANA**

Final Report

EVALUATION OF ASPHALT-AGGREGATE DISTRIBUTION
IN BITUMINOUS MIXES BY AUTORADIOGRAPHY

TO: K. B. Woods, Director
Joint Highway Research Project

April 11, 1962

FROM: H. L. Michael, Associate Director
Joint Highway Research Project

File: 2-10-2
Project: C-36-24B

Attached is a final report entitled "Evaluation of Asphalt-Aggregate Distribution in Bituminous Mixes by Autoradiography". The report has been authored by Mr. Donald Lamb, Research Fellow in the School of Civil Engineering, who performed the research reported for a Ph.D. thesis. Mr. Lamb received his Ph.D. degree from Purdue in January 1962. Mr. Lamb was a National Science Foundation Faculty Fellow and the only expense to the Project of this study was for equipment, supplies and extra labor.

The report contains a review of a number of published articles and books on isotope utilization and phase relationships of bituminous mixes. The report also contains sections on radioactivity and autoradiography as well as a report of research conducted on the fundamentals of phase relationship in bituminous mixes.

This research was conducted with the excellent cooperation and assistance of the Biomechanics Department of the School of Pharmacy. Professor J. E. Christain, Head of that Department, and Professor W. H. Goetz of our staff served as co-directors of this research.

The report is presented for the record.

Respectfully submitted,

Harold L. Michael, Secretary

HLM:kmc

Attachment

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Donald Lamb
Research Fellow

Joint Highway Research Project
File No: 2-10-2
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ABSTRACT

Lamb, Donald R. Ph. D., Purdue University, January, 1962. Evaluation of Asphalt-Aggregate Distribution in Bituminous Mixes by Autoradiography. Major Professor: William H. Goetz.

A laboratory study was performed to see if the radioactive tracer technique could be used in the evaluation of asphalt-aggregate distribution in bituminous mixtures. The measuring device used was the autoradiograph. Sample preparation and different types of photographic emulsions were investigated and compared. The effect of changes in viscosity of asphalt and changes in mixing time was also investigated.

Sulfur-35 in elemental form dissolved in benzene was used to tag the asphalt. A radioactive mineral, Wyoming urananite, was used as an aggregate. These were the only radioactive materials incorporated into the bituminous mixes. Other naturally radioactive aggregates were investigated but were discarded.

Sample preparation was important in obtaining good autoradiographs. A plane surface was needed so that intimate contact with the film emulsion could be obtained over the entire surface of the sample. The sample was cut on a

diamond saw, machine polished with a belt sander and given a final hand polishing to create the desired plane surface.

A series of photographic emulsions was investigated. Emulsions that were sensitive gave poor definition and those that were insensitive gave excellent definition. Because the X-ray film proved sensitive and yet gave good definition it was used for most of the autoradiographs made during this research. The Kodak NTB Autoradiographic Stripping film gave excellent definition but was quite insensitive, and because of its cost should be used only when excellence of definition is needed beyond that of the X-ray film.

Film thicknesses can be measured by the use of autoradiographs and can be delineated on aggregate particles not embedded in the asphalt-fine aggregate matrix. The evaluation of film thickness was investigated by coating small cylinders of pyrex glass, bakelite and limestone with different film thicknesses obtained by varying the viscosity of the asphalt.

A series of mixes was prepared using different ratios of coarse to fine aggregate. In mixes where the coarse aggregate was predominate, relatively open gradations, the fines formed a mastic with the asphalt and coated the coarse aggregate. As the gradation became more dense the coarse particles had no discernible film. Also in this series of tests the Wyoming urananite was used as the radioactive material. When selected-size fractions of the radio-

active aggregate were mixed into the bituminous mixture, very even distribution throughout the mix resulted in each case. If there was any affinity of the asphalt for any one particular size range it did not appear during this investigation.

When the aggregate was degraded in the compaction process, the liquid asphalt was forced into the resulting breaks. This was shown in the autoradiographs on aggregates which had colorations ranging from white to black. Thus when the aggregate and the asphalt have the same colorations the autoradiographs will show this phenomena.

The effect on asphalt-aggregate distribution of viscosity and mixing time was investigated. The open mixes showed asphalt films on the aggregate pieces which lost their identity as the mixes became more dense. The observed films became thicker as the asphalt became more viscous. Tests using variable mixing time showed that even though there was not 100 percent coverage of the aggregate after mixing, the autoradiographs indicated that complete coverage was obtained after compaction. This study shows that autoradiographic technique is a useful tool with which to study asphalt-aggregate distribution in bituminous mixes. Using this technique and naturally occurring radioactive aggregates the asphalt does not show any affinity for a particular size. It also shows

that asphalt films lose their identity in densely graded mixtures and complete coverage of the aggregate by the asphalt occurs during the compaction process.

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INTRODUCTION

With the advent of atomic power during World War II an important research tool, the radioactive isotope came into being. Since that time extensive utilization of this tool has been made by many people in science and industry. It has been stated that the range of usefulness of radioactive isotopes as tools of research is unsurpassed by any other tool at our disposal with the possible exception of the microscope and the analytical balance. The utility of this research tool has been recognized in most areas of the sciences and applied sciences. In fields of pharmaceutical sciences, biological sciences, medicine, and some of the areas of engineering, this tool has had widespread use for quite a number of years.

In Civil Engineering, however, the use of radio isotopes as a research tool has not been used to its utmost advantage. In the area of bituminous mixtures the use of radio isotopes in basic research has not found particular favor. The determination of asphalt content in a bituminous mixture

and the use of an isotope to determine the amount of stripping of asphalt from aggregate are the only bituminous areas in which isotopes have been used in basic research. It was the object of this research to apply radioactive isotope techniques to the investigation of some of the fundamentals of phase relationships in bituminous mixes.

The development, then, of this dissertation begins with a review of some of the published articles and books in the area of isotope utilization and phase relationships in bituminous mixes. This is followed by a section on radioactivity which is important to the understanding of this research. A brief description of autoradiography is then followed by an account of the materials and procedures used in the research phase of this thesis. The results of the research are presented and followed by a discussion of them. The conclusions based on the results of the research and a short recommendation for further investigations conclude the body of this work.

REVIEW OF LITERATURE

In the general area of medicine, since World War II, a tremendous amount of research has been carried on using the by-products of the atomic pile. The pharmaceutical and biological scientists have used tracer techniques to follow chemicals made from isotopes through the body and have noted especially the points of high concentrations (16, 20, 26, 31)*. Researchers in the area have studied growth and other metabolic changes of life using the above tracer technique (12, 14, 25). Engineers have been slower to use radio-isotopes in their fields, but some work has been done. In the area of mechanical engineering radio-isotopes have been used to study bearing failures and other general topics which include lubrication and physical degradation of machinery (4). Industrial organizations have used these new tools to control mixing efficiency and to gauge thicknesses. In the civil engineering field uses of radio-isotopes include determination of underground leaks, the measurements of soil density and moisture content, asphalt content in a bituminous mixture, fluid flow in filter beds and sewage mixing efficiency in stream flow.

* Numbers in parenthesis refer to selected references.

Black and Kerwick (11) sealed a radioactive source in a float weighted so that it had the same density as the fluid in the pipe system. The float was then placed in the system which was filled with liquid and sealed. A current within the sealed system could only occur where leaks were present and the float would be attracted to these areas. The position of the float could be determined with the use of a detector. A gamma emitter was needed so that the rays would penetrate the material covering the pipe.

A group at Cornell University headed by Belcher (5,6,7) working under contract for the Civil Aeronautics Administration was the first to use isotopes to study soil density and soil moisture content. This study utilized the Compton effect of gamma rays. The more dense the material the greater was the backscatter to the counter tube and hence a higher reading was recorded.

In the soil moisture content the effect of hydrogen nuclei in slowing down fast neutrons to produce slow or thermal neutrons was employed. The greater number of slow neutrons which returned to the counting tube the higher the moisture content.

Others in the United States and Canada (8,9,10,19,22,24) used the backscatter technique or the absorption of gamma rays to determine soil density, and placed the soil sample between the fast neutron source and the counting tube to determine soil moisture content. A commercial device using these principles is now on the market.

Lamb (23) working at the University of Wyoming, recognizing the fact that asphalt is composed of a large number of hydrocarbons, developed a method using a radium-beryllium source for fast neutrons, in the determination of the asphalt content of bituminous mixes.

Gzowski (1) of the Atlantic Refining Company used radioactive isotopes for measuring bituminous stripping. He applied radioactive calcium, in the form of calcium chloride, to the aggregate surface before the aggregate was coated with the bituminous material. Then the radiation intensity of the immersion water was used as a measure of the aggregate area accessible in the water exposure. The radioactive tracer technique was criticized because the aggregate surface was definitely changed by the application of the calcium chloride. The measuring counter used in this research was the scintillation counter.

In the area of sanitary engineering, radioactive isotopes have been used to evaluate the effectiveness of sand filter beds (27). The mixing efficiency of sewage effluent in streams has been measured by sampling the water at several places downstream from where the tagged sewage was placed in the stream (28).

In regard to asphalt distribution during the mixing of a bituminous mix, Ward and Warden (30), using the "Ross Count Method" found that 100% "mixing efficiency" was obtained in 40 seconds of mixing. This probably applies

only to the type of mixes that they used. They also separated the mix on a No. 4 screen and found a higher percentage of asphalt with fine fraction. They recommended that this be investigated further.

Covault (15) also has studied mixing efficiency using a radioactive isotope in his method of measurement.

Film thickness considerations have long been used as an approach to the selection of asphalt content in design procedures. The film thickness would seem to vary with size in the aggregate gradation from thick films on coarse fractions to thin films on fine fractions according to Ward and Warden (30). This is not born out by Rex in his discussion of a paper by Campen et al (13) in which he states "...as far as film thickness is concerned, there are no data in the literature which give a method for measuring film thicknesses on aggregate or which show how the asphalt distributes itself in compacted mixtures on the various sizes of aggregate particles, or if the thickness is the same at the contact area as it is on the rest of the area." and also "..., by realizing that an asphaltic concrete mixture does not, even in uncompacted condition, consist of discrete particles of aggregate each separately and uniformly coated with asphalt, but that on the contrary it consists of larger particles of aggregate coated with mastic composed of asphalt and particles of aggregate of smaller size".

It therefore seems that there is indeed a real need for the further investigation of the relationship between the aggregate and asphalt in a bituminous mix.

RADIOCATIVITY

Historical Review

When Wilhelm Conrad Roentgen discovered X-rays in 1895 he stimulated the imagination of physicists in many countries. Henry Becquerel, in France, observed that X-ray tubes fluoresced brilliantly when emitting X-rays, and wondered if substances which fluoresced under sunlight might also emit invisible radiations. Fortunately among the first materials he tested were some uranium compounds, and in 1896 he announced that these did in fact give off radiations, and furthermore that fluorescence had nothing to do with the phenomenon. Madam Curie took up the study of these substances, choosing the words "radioactive" and "radioactivity" to describe the process. She investigated systematically the known elements and compounds and found that all compounds of uranium and thorium possessed this property, and other substances did not. She found also that some of the natural ores of uranium were much more active than the element itself, and so concluded that they must contain unknown and very radioactive elements. In 1898 Madam Curie and her husband announced the discovery of not one, but two radioactive elements, which they named polonium and radium.

The radiations emitted by these radioactive substances, in common with X-rays, were found to be capable of darkening photographic plates and of discharging electrically charged bodies. It was early observed that most of these rays were nonpenetrating and could be stopped by a sheet of paper. A large part of those passing through the paper could traverse a few millimeters of wood or paper, but not more. A very small part of the original beam was very penetrating, traversing considerable thicknesses of metal.

At first the rays were thought to be all alike, differing only in penetrating power, and little was known of their nature. However, extensive researches by Becquerel, Pierre Curie, and Villard showed that there were three quite distinct types of rays. This was demonstrated by a very simple experiment as shown in Figure 1. When a beam of moving, electrically charged particles is passed between the poles of a magnet, their paths are bent into circles whose radii depend on the strength of the field, the magnitude of the charge, and the kinetic energy of the particles. The direction of the deflection depends on the sign of the charge. In Figure 1, the magnetic field is perpendicular to the page, with the north pole above and the south pole below. A very unpenetrating radiation is deflected clockwise, and the paths are circles of rather large radius, indicating that these are positively charged particles and rather heavy. The moderately penetrating radiation is deflected in the opposite

Figure 1. The Deviation of Alpha and Beta Rays in a Magnetic Field

direction and the paths are much more strongly bent. These are therefore lighter, negatively charged particles. The heavier component was called the alpha (α) radiation, or more properly, particles; the lighter, beta (β) radiation, or particles. The undeflected component obviously did not consist of charged particles; its real nature was demonstrated by Villard, who showed that these rays were identical with those emitted by the X-ray tube. They were called gamma (γ) rays.

Further studies showed that the alpha particles have a mass about four times that of the hydrogen atom, and two unit positive charges. They are really nuclei of helium atoms, stripped of their orbital electrons and traveling with speeds of eight to fifteen thousand miles per second. The beta particles have a mass of about $1/1835$ that of the hydrogen atom, a single negative charge, and travel at a speed almost up to that of light. They are actually electrons. The gamma rays are electromagnetic radiations of the same sort as X-rays.

Isotopes

The chemical properties of an element are determined entirely by the number of electrons in the planetary systems of its individual atom, given by the atomic number. All the atoms present in a sample of any one element must have the same atomic number, but not necessarily the same weight. All the atoms will have the same number of nuclear protons but

the number of neutrons in the nuclei may vary. These atoms, differing in the number of neutrons in their nuclei, are termed isotopes of the element. The term denotes elements of different atomic weight occupying the same position in the periodic table. Isotopes of any particular element have identical chemical reactions, and, excluding for the moment the question of radioactivity, they differ only in their atomic weights. Practically all the known elements occur normally as mixtures of two or more isotopes.

Atomic Disintegration

It had been found that radioactivity was a property of the atoms of the radioactive elements. For a given quantity of such an element, the radiation was always the same, regardless of the chemical composition of the compound under study. Therefore it appeared that the actual atoms must be breaking down in some manner to eject material particles. The radioactive elements which were discovered at first were all very high in the atomic table. Their nuclei obviously contained large numbers of particles and apparently were too complicated to be completely stable. It is now known that every element with an atomic number greater than 83, or mass number greater than 209 possesses the property of spontaneous emission of radiation, or is radioactive. These nuclei contain in a very small volume, large numbers of charged and uncharged particles, all in motion and hence exert electric,

magnetic, gravitational and internuclear forces upon one another. It is not surprising that at some time a configuration is produced which is unstable, so that the nucleus breaks down. This breakdown or disintegration is not however, complete destruction. It always consists of the expulsion of a relatively small particle accompanied by energy. The remaining nuclear components then rearrange themselves and settle down as an atom of a different element. The removal of an alpha particle decreases nuclear charge by two units and mass by four. The removal of a negative beta particle increases the positive nuclear charge by one unit. The mass is essentially unchanged.

Artificial Radioactivity

In 1919 Rutherford demonstrated the disruption of stable nitrogen nuclei by bombarding them with natural alpha particles, with the production of hydrogen and a heavy isotope of oxygen. This was not an induced radioactivity; the instant the bombardment stopped the production of new particles stopped. However, in 1934 Curie and Jolliots discovered that when they bombarded aluminum with alpha particles, a radiation appeared which continued after the removal of the source and which died away in the same manner as radiations from known radioactive substances. They had actually produced a radioactive isotope of phosphorus by introducing the extra mass and charge into the aluminium nucleus. Since

that time artificially produced radioactive isotopes of every stable element have been prepared.

Atoms of any particular radio-element always disintegrate in the same way. It is apparently not possible, for example, for radium sometimes to omit an alpha particle and sometimes a beta particle. There are few instances in which an isotope appears to do this; in these cases a fixed percentage of the material disintegrating always goes by each path. This consistency of type of radiation emitted is one specific characteristic of radioactivity.

The other specific characteristic is consistency of rate of disintegration. The amount of any radioactive element is always gradually decreasing; this gradual decrease is the result of many sudden disintegrations of individual atoms. In any measureable quantity of an element there is always an enormous number of atoms. During any instant, a relatively small fraction of these achieve instability and disintegrate. For every radioactive element, a fixed percentage of all the atoms present disintegrate per unit of time. There is no way of knowing which atoms will disintegrate in a given interval, but statistically it is possible to know how many will change. Actual rates vary enormously among various radioactive elements; in some cases only a small fraction of one percent of all the atoms decay or disintegrate in a century, in others a high percentage is transmitted per second.

Mathematical Expressions

The following section will present the mathematical concepts of the disintegration law and isotope half life as well as units of radioactive quantity.

Disintegration Law

Decay or disintegration of atoms can be described mathematically. If N represents the number of radioactive atoms present at any one instant, $-\frac{dN}{dt}$ represents the decrease in this number during a very short interval of time. This decrease is a fixed percentage of all the atoms present. Hence

$$-\frac{dN}{dt} = \lambda N,$$

where the decay, or the fraction transformed per unit time, is constant if the time unit is chosen short enough so that only a small fraction of the total number of atoms disintegrate in that interval. Lambda (λ) is then the fraction per second or per day, etc. This differential equation can be integrated,

$$N_t = N_0 e^{-\lambda t}$$

N_t is the number of atoms remaining from an initial number N_0 after a period of time t ; λ is the decay constant for the unit of time in terms of which the interval t is expressed; e is the base of natural logarithms, 2.71828. This is the expression of an exponential decay law. This decay constant is the characteristic of the nuclear species and cannot be changed by any means known at present. Great variations in

temperature, pressure, chemical state, magnetic, electric and gravitational fields have been completely without effect.

Half Life

Instead of using the decay constant it is possible to express the rate of radioactive transformation by specifying the period during which half of all the atoms initially present will disintegrate. This half period or half life can be obtained from a knowledge of the decay constant. In the preceding equation, N_t at the end of a half period is $\frac{1}{2}N_0$. The half period is indicated by T . The equation then becomes

$$\frac{1}{2}N_0 = N_0 e^{-\lambda T}$$

Solving this equation by natural logarithms,

$$\ln \frac{1}{2} = -\lambda T,$$

$$\text{or } \lambda T = \ln 2,$$

$$\text{whence } T = \frac{0.693}{\lambda}$$

Thus the decay constant and the half period or half life bear a fixed relation to each other; if one is found by any means the other then can be calculated. A portion of the decay curve for sulfur-35 that was used in the experimental part of this study is shown in Figure 2. Note the half life or time for 50% remaining is about 87.1 days.

Units of Radioactive Quantity

The unit of quantity of radioactive material is the Curie (c). This is the quantity in which 3.7×10^{10} atoms disintegrate per second. The millicurie (mc) and the

Figure 2. Decay Curve - Sulfur-35

microcurie (μc) are respectively the thousandth and millionth part of a Curie, or the quantities in which 3.7×10^7 and 3.7×10^4 atoms disintegrate per second. Obviously there is no simple relationship between Curies and total number of atoms or weight of material. A nuclide with a short half period or half life will not require such a large reservoir of the element to supply atoms at the needed disintegration rate as one with a long half life. However, either the number of atoms or the weight per Curie can be readily calculated when the half period and the atomic weight are known.

Measurement of Radioactivity

There are five basic devices for detection of radiation. These detectors are ionization chambers, proportional counters, Geiger-Mueller counters, scintillation counters, and photographic emulsions. Since the measurement of radioactivity depends entirely on the phenomena occurring when radiation passes through matter, a brief description of the five types of detectors seems to be in order.

Ionization Chamber

When any charged particle traveling with a sufficiently high velocity passes through a gas, electrostatic interaction will occur between the particle and the orbital electrons of any gaseous molecules with which it comes into

sufficiently close proximity. As a result of this interaction a number of molecules of the gas along the path of the particles will be split into ion pairs, ie, a negatively charged electron and a heavily charged positive ion comprising the remainder of the molecule. If the gas which is being ionized is confined between two oppositely charged electrodes the ion pairs will separate and drift toward the electrodes of opposite sign, ie, the electrons to the anode and the positive ions to the cathode. Owing to the difference in mass between the electrons and the positive ions the latter will travel toward the cathode at a very much lower velocity than that of the electrons toward the anode. The arrival of the electrons at the anode will result in a drop in its potential, which can be measured in a variety of ways and from this fall in anode potential the amount of ionizing radiation present can thus be determined.

A gas ionization chamber is in essence simply a vessel containing two electrodes mounted in some good insulating material. If the vessel is made of a conducting material it may itself form the cathode and have a single anode mounted within it.

Proportional Counter

As explained above when a gas ionizes in a chamber the number of particles collected on the anode reflect a change in the voltage of the anode. Thus in a proportional counter

the height of the individual voltage pulses produced is proportional to the specific ionizing power of the radiation traversing the chamber. Proportional counters may therefore be used to record pulses produced by one type of radiation while ignoring those produced by another type, or even those produced by radiation of the same type but markedly different in energy. This may be particularly valuable in investigations where it is necessary to determine one radioactive isotope in the presence of others, e.g. in radioactivation analyses.

Geiger-Mueller Counter

The Geiger-Mueller counter is generally the most sensitive type of radiation detecting instrument, and the one most applicable to a wide variety of different types of measurement. A wide range of Geiger-Mueller counter tubes (G-M tubes) and association recording equipment are available commercially.

The characteristic which distinguishes the G-M tube from other types of radiation measurement instruments is that, owing to the high degree of gas-amplification (with a factor as high as 10^8) occurring in the tube due to the production of secondary, tertiary and quaternary ion pairs by the accelerated electrons, a single ionizing radiation entering or produced in the tube gives rise to large pulses. These pulses are independent of the amount of ionization originally produced by the radiation and require little or

no further amplification before they will operate the recorder. A picture of an open window Geiger-Mueller tube and the associated scaler is shown in Figure 3. This equipment was used in the research connected with this dissertation.

Scintillation Counter

When the radiation is absorbed in certain liquid or solid materials which possess the property of luminescence (and now known collectively as phosphors) part of the energy dissipated in molecular excitation and ionization is re-emitted as visible or ultra-violet light. A scintillation counter is an instrument designed for the observation or recording of these light flashes. In earlier instruments the scintillations produced on a zinc sulphide screen were observed visually, but in the modern scintillation counters the scintillations produced in the phosphor are observed and converted into voltage pulses by a photomultiplier. These pulses may then be used to operate a suitable registering device in the same manner as those obtained from a Geiger-Mueller counter.

Photographic Emulsions

The blackening of a photographic plate by X-rays and by other ionizing radiations is at least as familiar to the reader as luminescence although the processes involved are far from simple. The essential difference between the two mechanisms is that in a scintillation counter changes due to

Figure 3. Open End Counting Tube and Scaler

absorption of radiation are fully reversible; after the emission of a light pulse the original state of the crystal is restored while the phenomena which occur in photographic emulsions are more permanent chemical changes. For this reason, a photographic emulsion is of no use when the frequency of radioactive disintegrations have to be detected. But it is a simple and convenient device to measure the accumulated amount of radiation, for instance, the dose. It is, of course, also of unique value in the determination of the spatial distribution of radiation sources.

The term used for this type of detection is radiography or autoradiography and the results of the process are referred to as autoradiographs or autoradiograms. In this instance the author has used the words autoradiography and autoradiographs as a convention throughout this dissertation. A more detailed section on autoradiography is presented as an introduction to the experimental work of this investigation.

In summary some of the general properties of the different types of radiation detectors are as follows:

Photographic emulsion can be used only to measure directly the total dose, in addition to their particular properties of localizing radiation sources by image formation.

Ionizing chambers can be used to measure both dose rate and total dose.

Neither of the two allows the observation of single radiation bursts during the disintegration of the radioactive isotope.

Proportional counters give electrical pulses when they are traversed by radiation bursts. The height of the electrical pulse is proportional to the ionization occurring during such bursts.

Geiger-Mueller counters give electrical pulses when ionizing events occur in the sensitive volume in a similar manner as the proportional counter, but the pulse height is independent of the ionization intensity of the initial radiation bursts.

Scintillation counters which are made of solids or liquids absorb more radiation energy than the devices containing gas; they have the highest sensitivity for penetrating radiation such as Gamma or X-rays. The light intensity of each scintillation depends on the energy of radiation.

In addition to the five main types of radiation detectors there are many auxiliary instruments used for monitoring of laboratory and personnel as well as specific devices that are used or made up of two or more components of the detectors discussed.

AUTORADIOGRAPHY

The autoradiographic technique is presented in more detail than the general treatment of the previous section since it was the device used to the greatest extent in this work. As stated before, Henri Becquerel was the first autoradiographer. Since his first experiment many people have contributed to the field.

Photographic emulsions consist of fine grains of silver halide in an emulsion. When a radio particle hits these "grains" it reduces them to metallic silver. This metallic silver works as a catalyst in the reduction of silver halide in the near vicinity during the development process. Therefore if a photographic emulsion was subjected to radioactivity for a sufficient period of time (exposed in the dark of course), and subsequently developed, darkening of the film would be noted wherever the rays from the radioactive source impinged upon it.

There are basically two types of autoradiography; they are contrast autoradiography and track autoradiography. In the contrast type of autoradiography, areas in contact with the radioactive material are darkened and thus the general shape of the darkened areas gives the distribution of the

radioactive material. In track autoradiography, as the particles pass through the emulsion they leave behind definite tracks of blackened grains, which may be distinguished under a high-powered microscope. Alpha particles have short straight, well defined tracks but β particles produce more erratic tracks owing to their smaller mass.

Sensitivity

The sensitivity of the emulsion is determined in part by the concentration of the silver halide, but it is also influenced by the thickness of the emulsion film and the size of the grains. The larger the grains the more sensitive it is. Thus X-ray film is very sensitive, some of the regular photographic emulsions have medium sensitivity, and stripping film has low sensitivity.

For good contrast autoradiography and satisfactory darkening it has been estimated that it is necessary for 10^6 to 10^8 electrons to strike each square centimeter of X-ray film. Therefore a rough estimate of exposure time required may often be made by monitoring the section with a thin end-window counter; if 1 cm^2 section gives 100 cpm, 10^6 counts would be collected in about 7 days, provided the isotope has a long half life. This is the minimum likely to produce satisfactory results, and trial and error will provide the best time of exposure.

Since over exposure leads to loss of definition, it is always important to expose several specimens simultaneously

and examine the trials at various time intervals to obtain the best results.

Specific activities of one-hundredth to one-thousandth of those given above may be satisfactory for track radiography, since in this case individual particles are counted, in much the same way as with a G-M tube.

Definition

The definition of a contrast autoradiograph may be defined as the minimum distance apart which two point sources must be in order for them to be distinguished from each other in the developed film. Naturally, the more sensitive large grained films have lower resolving powers and produce autoradiographs of poorer definition than small grained films. The major factors affecting the definition of an autoradiograph are, however, the nature of the radiation and the experimental technique employed for making the exposures.

If a point source of radioactive material is placed on the surface of the photographic film, it will produce a circle of blackening around itself of a radius equal to the maximum range of the ejected particles in the emulsion. Therefore, the range of the particles obviously will be a factor in determining whether the adjacent point sources are capable of being resolved. It follows, therefore, that other things being equal, the sharpest definition will be obtained with the softest radiation, i.e. radiation with the lowest

particle energy. Soft Beta emitting isotopes such as carbon 14, calcium 45, and sulfur-35 give very good resolution, but as the energy of the beta particle emission increases the autoradiograph becomes more diffused. Unfortunately, the advantage of the high resolution obtainable in autoradiographs by the use of low energy containing specimens is to some extent counteracted by the fact that the very low energy of the radiation frequently necessitates the use of rather long exposure times to obtain recognizable films.

Background Fog

Just as cosmic radiation and external radioactive substances will produce a background count rate with instruments used for measuring radioactivity, so will external causes produce a background fog on autoradiographs. The fogging of the plate naturally will be less if the emulsions used are fresh, but it will increase with increase in exposure time. This is a particular disadvantage in a contrast autoradiograph. In track autoradiography this is not quite so serious a drawback since tracks due to external sources can usually be distinguished from those due to radioactive particles in the specimen section by the fact that these particles will normally be the point of origin of more than one track. As a sideline to this background fog it might also be possible that, with the increasing use of radioactive isotopes in this "atomic age", small amounts of carbon 14, not sufficient to

cause any health hazard but sufficient to increase the background fog in autoradiography, may become incorporated in the cowhide from which the gelatin used in the emulsion is prepared. The degree of background fogging of the plate can be controlled to some extent by using particular developers. In this particular research project the contact autoradiography was used and contrast autoradiographs were made.

MATERIALS AND PROCEDURES

This section deals with the materials that were used in this research and the procedures that were followed in each phase of the investigation. The subdivision of this section is based on these two facets of the work.

Materials

Mineral aggregates, bituminous material, radioactive materials, and photographic emulsions are the materials that were used in this investigation. They are discussed in the order stated.

Mineral Aggregates

A crushed limestone donated by the Ohio and Indiana Stone Company of Greencastle, Indiana was the primary mineral aggregate used in this research. This Mississippian limestone was quarried from the St. Genevieve formation. Special aggregates used in two phases of the work were Lafayette gravel, a valley train deposit of the Wabash River laid down during the Wisconsin glacial period, and Belgium black marble. The marble was purchased from a commercial firm and no geologic data for it were available.

Two other aggregates were used for this research, both of which contained naturally occurring radioactive minerals.

The first one incorporated into a mix was a New Hampshire granite sent by Dr. J. Harold Zoller, Durham, New Hampshire. This material was Concord granite from the New Hampshire magma series and was of late Devonian(?) age. The second radioactive aggregate was Wyoming urananite. This rock was found and sent by Kenneth G. Lamb, Rawlins, Wyoming and was a lime cemented sandstone from the Tertiary Wind River formation in southcentral Wyoming.

Bituminous Material

The bituminous binder that was used was a 60-70 penetration grade asphalt cement. This material was furnished by the Texas Company of Port Neches, Texas, and its test properties are shown in Table 1.

Radioactive Materials

The unstable isotope of sulfur, sulfur-35, was chosen as the radioactive substance for this research because it had three properties that were compatible. The first was a low beta emission energy which is very good for contrast autoradiography. The second property was the relatively short half life; if a piece of equipment could not be decontaminated it need not be discarded, but held for ten half lives to reduce the activity to a tolerable level. The third consideration was the fact that it could be purchased as elemental sulfur in benzene. This made its incorporation into the asphalt cement an easy matter.

TABLE 1

Test Properties of Asphalt Cement

Texaco 65 Paving Cement No. R-2885-B

Specific Gravity @77°F	1.036
Softening Point, R&B, °F	124
Ductility @77°F, cm.	200+
Penetration @77°F	66
Penetration @32°F	17
Loss, 50/5/325°F, %	0.01
Penetration of Residue @77°F, % of original	89
Flash, COC, °F	595
Soluble in CCl ₄ , %	99.84
Oliensis Spot Test	Negative

The isotope was purchased from Oak Ridge National Laboratories. The energy of the emitted beta particle from sulfur-35 is 0.167 Mev. and the half life of this isotope is 87.1 days.

The two aggregates used that had naturally-occurring activity were the New Hampshire granite and the Wyoming urananite. The granite was a primary alpha emitter with some associated gamma emissions. The emulations from the Wyoming urananite were alpha, beta and gamma.

Photographic Emulsions

The X-ray film used was EK Medical X-ray film, Blue Brand. Some regular photographic emulsions were also used. Eastman Royal Pan is of medium resolving power and relatively fine grained. Kodak Contrast Ortho was used not only for autoradiography but also for the reflected light pictures. This has a very high resolving power and is an extremely fine grained emulsion.

A Dupont LTSX Pan film was also used. This is an extremely fast film and fairly fine grained. A regular autoradiographic film was purchased and used. This was the Kodak Autoradiographic Stripping Film Type NTB (emulsion number FW-1826-1) and is a very fine grained emulsion and a very slow film. All the photographic emulsions were processed according to the directions given for each one by the manufacturer as to processing requirements such as chemicals, time in developer and fixer, etc.

Laboratory Procedures

The order of presentation in this section does not necessarily follow chronological steps used during the research phase of this work. In most cases the grouping was made so that continuity in presentation could be achieved. The divisions discussed are as follows: isotope-asphalt mixing, film thickness, sample preparation, autoradiography and counting specific activity.

Radioactive Material Utilization

The method of handling radioactive isotopes is different than handling ordinary chemicals. The first consideration in handling radioactive material is to eliminate the possibility of contamination. Since the quantities of the isotope to be used were very small, the micro-pipette had to be used. Because of the health hazard, the mouth cannot be used for suction. Neither can work be done without the use of rubber gloves on the hands. A small syringe was attached to the top of the micro-pipette by a short piece of rubber tubing. The plunger of the syringe was brought up from the bottom of the syringe before placing the end of the pipette into the radioactive solution. The plunger was placed in position before pipetting was accomplished so that all the fluid in the pipette could be expelled without working the plunger in and out.

After receiving the sulfur-35 in benzene, a pre-calculated amount of the nonradioactive isotope of sulfur dissolved in benzene was added to the purchased mixture as a carrier for the radioactive sulfur. This was done so that a uniform distribution of the sulfur would be made in the asphalt. The amount of benzene and carrier that were added was calculated so that 500 λ of final mixture of benzene, carrier and sulfur-35 would have one μ c of activity as of the day of preparation.

The 500 λ of mixture was then added to 50 grams of the asphalt cement the temperature of which was 275°F and mixed by hand with a tongue depressor until it was too viscous to mix any longer. It was reheated to 275°F, mixed slightly again and then placed in the mixing bowl with the aggregate.

The micropipettes could be and were decontaminated, but the wooden tongue depressors that were used for mixing the isotope and asphalt, as well as the container, were thrown away.

The naturally occurring radioactive rocks were received in large pieces from the donor. These were broken up with a hammer so that the resulting fragments could be placed in a jaw crusher. The crusher reduced the size so that in the screening operation all sizes were obtained. During the crushing and screening operation the work area was monitored with an air monitoring device to make sure that air contamination did not occur. After the crushing-screening

operation was finished the laboratory was completely checked by the Health Physicist for any residual contamination.

The portions of radioactive rock that were separated by the screening operation were placed in containers so that no contamination occurred. From these containers portions were weighed out for incorporation into bituminous concrete samples. Vigilance was maintained to keep any contamination from taking place. The samples were mixed by hand in the same bowl and all the equipment was monitored at the end of one day's operation. Any contaminated equipment was decontaminated at the end of the research and checked by the Health Physicist before it was returned to regular laboratory use.

When incorporating the radioactive aggregate into the bituminous concrete specimens the asphalt cement was not tagged and in some cases only one fraction of the total aggregate gradation was radioactive.

Film Thickness

In the study of film thicknesses a group of specimens were dipped into the tagged asphalt and then encased in a plaster of paris cylinder (four inches in diameter and four inches high) so that they could be held in a chuck for cutting on the saw.

The different types of materials that were used for this study were limestone, pyrex glass, bakelite and Teflon. The

glass, bakelite and Teflon came in one-half inch rods. Individual pieces used were cut one-half inch long. The limestone pieces that were used were cylinders one-half inch in diameter and one-half inch high and prisms one-half inch on each side.

The change of film thickness was achieved by two methods of procedure: by changing the temperature of asphalt and specimens, and by mixing a heavy oil with the asphalt. The temperature range of the asphalt was from 225°F to 275°F. Combinations of these temperatures for the tagged asphalt and the specimens gave different film thicknesses. The change of viscosity with the addition of the oil did not work well as the temperature method because the changed asphalt would not set up, ran off the specimens, penetrated into the plaster of paris, or was dragged across the face of the cut made by the saw blade.

The saw used to cut all samples for this research was a regular masonry saw equipped with a diamond blade, over which a hood was placed. A suction fan was placed on top of this so that a negative pressure could be maintained on the hood at all times. Ports for rubber gloves used in handling the materials or specimens inside of the box were made and the rubber gloves installed. The hood and the saw are shown in Figure 4.

The Teflon was discarded after the first series of tests because it was found that the diamond saw would not cut it.

Figure 4. Diamond Saw and Hood

Sample Preparation

The asphalt-aggregate samples were hand mixed and were compacted using the Marshall equipment. These samples were cut and then the cut surface was ground and polished to a plane surface. This was necessary so that complete contact of the sample surface with the film would result when the autoradiographs were being made.

Sample Cutting

Several methods of cutting the samples were tried before one was finally adopted. The first method involved cutting the sample at room temperature. This proved unsatisfactory because of the tendency to drag some of the asphalt across the surface of the aggregate and influence the distribution of asphalt as read on the autoradiograph.

The next method attempted was the impingement of CO_2 on the surface of the specimen as the saw cut in an attempt to freeze the material immediately forward of the cutting blade. It was anticipated that this procedure would absorb the heat and prevent the movement of the asphalt across the surface of the aggregate as the blade moved through the specimen. This, however, did not prove satisfactory because the velocity of the CO_2 was so high as to degrade the specimen, knocking out not only small pieces of aggregate but also large pieces of aggregate ruining the specimen.

The final sawing technique selected was the freezing of

the specimens in the freezing compartment of a refrigerator for 48 hours at 10°F and then cutting the specimen as rapidly as possible after removal.

Sample Saturation. In one part of this study samples were made up of aggregates of only one size. It was very difficult to cut some of these samples on the saw because the asphalt was not strong enough to hold the one-size pieces of coarse aggregate in place. However, when the size of aggregate was below the No. 8 sieve size (No. 8 to No. 16 on down) the voids were fairly well filled with asphalt and cutting was found to be suitable.

A series of tests was made to see if there was some way to impregnate the one-size coarse-aggregate samples to facilitate the cutting. The first method used was to try to impregnate the sample with Carbowax 4000. However, the Carbowax was solid at room temperature and needed to be heated to 150 degrees Fahrenheit before it would melt and impregnate the sample. On heating the sample to this temperature the asphalt readjusted itself on the aggregate and some of it settled to the bottom of the specimen, thus changing the asphalt-aggregate distribution that was initially present when the specimen was made.

The next method used was to vacuum saturate the sample using water. This saturated sample was then frozen and cut in the frozen condition. This did not work out as well as was anticipated because the water used to lubricate the saw

melted the ice in advance of the saw blade. The next trial was to vacuum saturate the sample with a thin slurry of plaster of paris, let the plaster of paris set up, and then cut the sample after the plaster of paris had reached a final set. This procedure did not work because the plaster of paris was not a strong enough cement.

The final method used was to vacuum saturate the asphalt aggregate sample with a slurry of type III cement. After curing in water for three days the sample was cut. This method worked very well. Not only did the slurry make the sample into an integral unit when set, but it also was fluid enough initially to completely saturate the sample. Even the relatively small voids that were present in the sample were filled. Upon cutting the saturated sample a relatively smooth surface was achieved and spalling at the specimen edges did not occur.

Sample Polishing. It is important to have the sample as close to a plane surface as possible in order to improve the resolution of the autoradiographs. The first attempt at the preparation of a plane surface was to polish the cut surface using abrasive on a plate glass table. This did not work too well because of the length of time required to prepare the sample. An aluminium oxide paper was next used, resulting in a substantial saving of time. The next method tried was to obtain initial smoothness with a belt sander

using a No. 120 grit paper and then to polish this surface with a No. 240 grit paper. This procedure proved very satisfactory. However, further improvements were made.

The method finally evolved was to sand initially on No. 100 grit paper by hand, use the belt sander with No. 120 grit paper, followed by belt sanding with No. 240 grit, and then to provide final polishing by hand using No. 320 grit aluminium oxide paper. This gave the best surface that was obtained in this research work and was followed for all the final phases of the autoradiographic work. The above mentioned polishing was done at room temperature.

The polishing procedure finally used seemed to prevent the asphalt in the specimen from sticking to the photographic plate as it had in some of the initial autoradiographs that were made.

Autoradiography

The first step in the autoradiographic technique was to cut a one centimeter square opening in a piece of 1/16 inch lead plate large enough to cover the specimen. This plate was then placed over the specimen allowing the counts to be made from a one square centimeter area of the specimen as was described in the section on autoradiography. About five-three minute counts over the surface of the specimen were averaged and the time for contact with the film was determined.

Using this as the minimum contact time for the film a series of film exposures was made. Beginning with the minimum time, each exposure time thereafter was longer. The best autoradiograph was then correlated with average counting time, as explained. The time in contact with the film was varied as well as the processing time in order to see how the contact time and processing time were related to the resolution of the final autoradiograph. Using one millicurie of radioisotope in its first half life period per 50 gm. of asphalt 48 hours contact time, and the normal processing time for the type of film used gave the best definition.

In using the natural radioactive materials determining the time for best resolution was strictly a trial and error procedure. The radioactive New Hampshire granite was not usable because only one of the constituents of the granite was radioactive and only points of high intensity were shown (alpha particles). However, in the Wyoming urananite the radioactive mineral was very evenly distributed throughout the rock and very good definition was found after incorporating the ground rock into asphalt concrete specimens. Even the Wyoming urananite aggregate gave small points of very high intensity showing the places of alpha emission. However, these were minor and did not interfere with the reading of the autoradiographs.

The relative position of the specimen with regard to the photographic emulsion was studied before the final procedure was evolved. The first attempt to produce an

autoradiograph was to place the specimen directly on the emulsion in an X-ray cossette. In this trial the asphalt adhered to the photographic film. The same procedure was followed and a piece of Mylar* film was placed between the specimen and the emulsion. This method proved satisfactory. However, because of the size of the X-ray cossette only thin specimens could be used.

In order to use specimens of greater thicknesses on the film a light-free box with a removable top was built. A one-fourth inch thick sponge rubber mat was placed in the bottom of the box. The photographic film was placed on the rubber mat and a piece of Mylar film was used to cover the film surface. The cut face of the specimen was then placed on the Mylar film. If the specimen was not heavy enough to insure good contact a weight was placed on the specimen. However, after the polishing technique was standardized the Mylar film was no longer needed.

Counting Specific Activity

Some of the specimens that were made were counted for the specific activity at the surface. These were the specimens that were made using the Greencastle limestone and the tagged asphalt. An open window counter* shown in conjunction with the scaler in Figure 3 was used for this counting. This

*Mylar is an extremely thin plastic film.

*Manufactured by Packard Instrument Co., LaGrange, Ill.

flow window counter is used primarily for beta counting. Sensitivity for low energy radiation is slightly less than for a windowless flow counter, but greater than the normally sealed counters using the thinnest possible mica windows. In this counter gas flowed through the counting tube during counting because the window was so thin that it would not hold static gas pressure. A 90% argon and 10% methane gas mixture was used as the counting gas. The data on this part of the research are given in Appendix B.

RESULTS

In this section all of the results of the laboratory investigation are given in the form of typical autoradiographs. These are supplemented with reflected-light photographs as well as some photomicrographs. The presentation of results is subdivided into sections on autoradiographic definition and asphalt-aggregate distribution. This section on presentation of results is followed by a section in which they are discussed.

Autoradiographic Definition

The first autoradiographs made were those of the asphalt-isotope mixture. Based on the number of particles striking the film surface, the specimens were left on the film for 24, 48, and 72 hours. The autoradiographs were then studied for uniformity of mixing. Figure 5 shows the 48-hour autoradiograph and the uniformity that was obtained.

A digression is in order at this point to explain how the autoradiographs and the other photographs are handled in this presentation. Due to the fact that the asphalt, generally speaking, was radioactive, the film actually showed a positive picture. Thus when viewing the film the darkened

Figure 5. Autoradiograph of Asphalt-Sulfur-35 Mix

area represents the asphalt and the light area the aggregate. (An original autoradiograph is included in a packet inside the back cover).

In contrast to the autoradiograph the film in a reflected light picture is a negative. To keep as much definition as possible in all pictures a contact print was made in all cases whether autoradiograph or reflected light photography was used. This made the prints of the autoradiographs actually negatives and those of the regular photographs positives. The reader must school himself to this difference so that the figures in this thesis are to be meaningful to him.

Film Thickness Studies

A series of film thickness studies was made to see if these films would be defined by the autoradiographs. The first method of changing the film thickness was to cut back the radioactive asphalt. This method was unsatisfactory. When the asphalt was cut back far enough to produce a thin film it would not stay on the aggregate pieces during the curing of the asphalt. The asphalt would also invade the plaster of paris mold, used to encase the cylinders, and bleed from the surface of the cut specimen if the specimen were processed before the asphalt hardened. The above work was done at room temperature.

The next procedure to change the film thickness was to vary the temperature of the radioactive asphalt and the

temperature of the cylinders of limestone, pyrex glass, bakelite and Teflon used for aggregate. The cylinders of pyrex glass, bakelite, and Teflon were cut from one-half inch rods. The diameter of the limestone cylinders was one-half inch. When the asphalt and cylinders reached the prescribed temperature, the cylinders were hand dipped in the asphalt and allowed to cool to room temperature before being encased in the plaster of paris mold. The autoradiograph was then made from the cut surface of each specimen. The Teflon specimens were discarded because the diamond saw would not cut them.

The temperatures that were used, to heat both the asphaltic cement and the cylinders, were 173° , 210° , 150° , 300° , and 325° Fahrenheit. The three remaining materials acted the same and showed that with an increase in temperature there was a decrease in film thickness. It was noted that there was an increase in film thickness if a cylinder was at room temperature when dipped rather than at the temperature of the asphaltic cement.

The series of films in Figure 6, using glass cylinders, show the progressively thinner film thicknesses. The first sample is at 170°F and the last sample is at 325°F . Figure 7 shows the comparison between the hot and cold cylinders and the asphaltic cement temperature of 300°F . The hot cylinder was at 300°F and the cold cylinder was at room temperature.

Asphalt and Glass Cylinder
at 173°F

Asphalt and Glass Cylinder
at 210°F

Asphalt and Glass Cylinder
at 250°F

Autoradiographs of Film Thicknesses on Glass
Cylinders Resulting from Changes of Temperature
of the Asphalt and Glass Cylinders.

Asphalt and Glass Cylinder
at 300°F

Asphalt and Glass Cylinder
at 325°F

Figure 6. (Continued) Autoradiographs of Film Thicknesses on Glass Cylinders Resulting from Changes of Temperature of the Asphalt and Glass Cylinders.

Hot Glass Cylinder
(300° F)

Cold Glass Cylinder
(Room Temperature)

Figure 7. Autoradiographs Showing the Comparison Between ^o
Hot and Cold Cylinders—Asphalt Temperature 300 F.

The viscosities and approximate film thickness for the temperatures cited above are given in Table 2.

Effect of Specimen Preparation

The first specimens that were prepared by cutting on the diamond saw gave very poor autoradiographic definition because the surface was not plane. Figure 8 shows one of the early autoradiographs for an asphalt-aggregate specimen made from one-sized limestone aggregate. Note the areas where the shading covers the aggregate indicating that the aggregate was not in contact with the emulsion and the water that was trapped between the sample and a Mylar film. Water spots on the film, when not protected by the Mylar film, are shown in Figure 9, an autoradiograph for an asphalt-aggregate specimen made from three-quarter inch limestone.

After spending more time perfecting the cutting of the specimen a more uniform surface was obtained from using the saw. This is shown in Figure 10, an autoradiograph for an asphalt-aggregate specimen made from three-eighths inch limestone. Notice that only the actual voids in the specimen have any variation in tone change indicating that the beta rays had lost energy passing through the air in the void. Wherever the asphalt-aggregate interface was cut a very clean line can be seen. Also note, however, that some asphalt has been deposited on the face of the aggregate by the saw blade. This of course, detracts from the definition of the

TABLE 2

Temperature Viscosity—Film
Thickness Relationships

Temperature Degrees Fahrenheit	Viscosity Saybolt-Furol Seconds	Approximate Film Thickness, mm
173	5200	1.5
200	2300	1.2
250	520	0.8
300	115	0.4
325	56	0.3

Figure 8. Initial Autoradiograph Showing Very Poor Definition

Figure 9. Autoradiograph Showing Effects of Water on the Film During Exposure

Figure 10. Autoradiograph of Improved Definition but with the Asphalt Deposited on the Aggregate Face by the Saw

autoradiograph. This specimen was allowed to dry in the air so that the water spots do not appear.

Definition produced by the saw cut alone was greatly improved by wet polishing on glass using a polishing compound. The polishing compound removed any asphalt that had been deposited in the cut surface of the aggregate by the saw blade (as shown in Figure 10). It also made a plane surface so that intimate contact with the film was obtained over the entire surface of the specimen. An example of this series of tests is given in Figure 11, an early autoradiograph, for an asphalt-aggregate specimen made from the same limestone as shown in Figure 10. The voids in the sample still show as a tonal change in the autoradiograph, but are now distinguishable from poor sample-film contact. However, though the definition is much better than without polishing the edges of the sample are ragged and good definition is lost in the peripheral area. This loss is due to some extent to the beta particles hitting the photographic film at an angle of less than 90° .

The method of changing the face of the cut specimen to a plane surface by dry sanding with a belt sander (as described in the section on sample polishing) gave the best definition as shown in Figure 12. This figure shows an autoradiograph for an asphalt-aggregate specimen made from graded limestone. A smooth even tonal quality to the

Figure 11. Improved Autoradiographic Definition by Wet Polishing

Figure 12. Autoradiographic Definition Using Dry Polishing Technique

autoradiograph with each piece of aggregate well defined shows excellent definition. Also to be noted here is that a piece of the limestone aggregate had some natural radioactivity. In the autoradiograph the radioactive limestone appears to have absorbed some asphalt but upon closer examination of the aggregate in the specimen this supposition proved untrue.

Effect of Film Type

The preceding discussion has been describing only the definition due to the physical changes of the sample and all comparisons were made using the X-ray photographic film. This portion of the text presents the definition results obtained from the use of different types of photographic film.

The EK Medical X-ray film, Blue Brand, was the standard film used throughout this research for the autoradiography. Figures 5 through 12 were obtained with this film. As was previously stated in the section under autoradiography it is a sensitive film with lower resolving power than some fine grained films. Since the definition required in this research was of macro size, the X-ray film was well suited as far as resolving power and definition were concerned.

However, if micro work in this same area would be required, the resolving power of the X-ray film would go down with the resultant loss of definition. Both the Eastman

Royal Pan and the Kodak Contrast Ortho are excellent films for regular photograph, but neither one produced a readable autoradiograph. Even using exposure times up to 20 times the contact time for the X-ray film they did not produce autoradiographs that were useful.

The Dupont LTSX Pan film produced satisfactory autoradiographs. The only size of film that was used was 35 mm and a strip autoradiograph using it is shown in Figure 13. Figure 13 shows a strip autoradiograph for an asphalt-aggregate specimen made from graded limestone. This shows excellent definition. Since sensitivity was about half that of the X-ray film, twice as much contact time was needed to produce this autoradiograph.

Kodak Autoradiographic Stripping film is not a very sensitive film because of the fine grained emulsion used. However, it gave excellent definition. The contact time was exceedingly long. Figure 14 shows an autoradiograph made with this type of film using a contact time of 18 days. The specimen used to make the autoradiograph in Figure 12 was made from graded limestone. Since this is a special emulsion only made when specifically ordered, it is very expensive. For the sheet film in size 4 by 5 inches this autoradiographic film is about 45 times as expensive as the X-ray film. Therefore selection of this film must be on the basis of value assigned to the additional definition obtainable.

Figure 13. A Strip Autoradiograph Using Dupont LTSX Pan 35mm Strip Film

Figure 14. An Autoradiograph Made On Kodak NTB Stripping Film

Examination for Definition

In order to investigate the definition obtained in the autoradiographs in more detail than can be done with them in the natural size, an overhead projector was used to project the autoradiograph image on a screen. An increase of 20 diameters was very convenient for the room and screen size. Since it would be difficult to put this into a thesis, a series of photomicrographs was made. These were made in the School of Metallurgical Engineering, Purdue University, Dr. P. G. Winchell supervising.

A recognizable position was chosen on the polished surface of a specimen and a photomicrograph was made of this area. Then the autoradiograph was placed under the microscope and the same area was again photographed. Care was taken so that the same area was covered in each instance. The two photomicrographs could then be compared as to definition. Figure 15 shows an autoradiograph for an asphalt-aggregate specimen using a graded limestone showing the area chosen for the photomicrograph. The small circle outlines this area. Figure 16 is the photomicrograph of the face of the sample, and Figure 17 is the photomicrograph of the autoradiograph. Due to the fact that both photomicrographs were taken from positives, negatives were made and positive prints for the two figures were produced.

In comparing the two photomicrographs notice that there is more detail shown from the print of the autoradiograph

Figure 15. Autoradiograph Showing Position of Photomicrographs (Example 1.)

Figure 16. Photomicrograph of Specimen Face — Position Shown in Figure 13 (x 20)

Figure 17. Photomicrograph of Autoradiograph — Position Shown in Figure 13 (x 20)

than from the one of the specimen. Also notice that the emulsion grains show in the photograph from the autoradiograph because the picture was made from the film.

A similar comparison for a different asphaltic concrete sample is shown in Figures 18, 19 and 20. There are two scratches on the negative of Figure 20 that show as dark lines. The same specimen was used for the autoradiographs shown in Figures 14 and 18.

Compaction in the Marshall apparatus produced some degradation of the coarse aggregate. This degradation occurred when the coarse material was not cushioned by the matrix. The hot bituminous material was then forced into many of the fractures caused by the rupturing of the coarse aggregate. When the autoradiograph was made from a section through one of these asphalt filled fractures the breaking of the aggregate was recorded by the film.

A regular photograph of the sample face also revealed the impregnated fissure when light colored aggregate was used. However, when a dark aggregate was used, Belgium black marble, only the autoradiograph gave the true picture of the asphalt distribution. Figure 21 is the autoradiograph from a graded limestone-aggregate specimen with Figure 22 the companion photograph of the same specimen. Figure 23 is the autoradiograph from the Belgium black marble aggregate specimen and Figure 24, is the photograph of the same specimen surface.

Figure 18. Autoradiograph Showing Position of Photomicrographs (Example 2)

Figure 19. Photomicrograph of Specimen Face — Position Shown in Figure 15 (x 20)

Figure 20. Photomicrograph of Autoradiograph — Position Shown in Figure 15 (x 20)

Figure 21. Autoradiograph of a Limestone-Aggregate Specimen

Figure 22. Photograph of Specimen Shown in Figure 21.

Figure 23. Autoradiograph of a Belgium-Black-Aggregate Specimen

Figure 24. Photograph of Specimen Shown in Figure 23

It is also important to note that the photographs show how the stone was broken by the diamond saw while the autoradiographs do not show these imperfections.

Asphalt-Aggregate Distribution

The study of asphalt-aggregate distribution was divided into three phases. One phase used specimens with a range of gradation from coarse aggregate only at one extreme to fine aggregate only at the other extreme and combinations of the two in between. The second phase utilized radioactive aggregate to see how this was distributed. The third phase investigated the effect of asphalt viscosity and mixing time on asphalt-aggregate distribution.

Effect of Gradation

To determine the influence of aggregate gradation on asphalt-aggregate distribution, a fixed gradation of coarse aggregate and a fixed gradation of fine aggregate were blended in various proportions. The coarse fraction chosen had 80 percent of this fraction between the $3/4$ and $1/2$ inch sieves and 20 percent between the $1/2$ and $3/8$ inch sieves. A series of mixes was then made to see how much asphalt would adhere to the coarse material. Mixing temperature for mixes in this part of the research was 275°F .

Mixes were made with from 1 to 6 percent asphalt by weight of the coarse aggregate. A Marshall sample was made

and the mixing bowl was weighed back to see how much asphalt was actually on the aggregate. This procedure determined that the coarse aggregate retained about 4 percent asphalt by weight of aggregate.

The gradation chosen for the fine aggregate is shown in Table 3. The amount of asphalt that this gradation would carry was found by making Marshall specimens using 5 percent asphalt by weight of the aggregate and increasing percentages by increments of 1 percent until 9 percent was reached. The specimens were then evaluated by the normal Marshall test and the optimum percentage chosen from this procedure. The asphalt content chosen for the fine aggregate was 6 percent.

Eleven mixes were then made beginning with 100 percent coarse aggregate and zero percent fine aggregate. The mix composition (90 percent coarse aggregate and 10 percent fine aggregate) was changed by increments of 10 percent until the mix contained zero percent coarse aggregate and 100 percent fine aggregate. The asphalt content was determined on the basis of the percent of coarse and fine aggregate used. In the 50-50 mix the total asphalt content was 50 percent of the 4 percent asphalt content of the coarse aggregate and 50 percent of the 6 percent asphalt content of the fine aggregate.

The autoradiographs of the complete series beginning with the coarse aggregate only and ending with the fine aggregate only are presented in Figures 25 through 35. In the coarse aggregate series the area that is crosshatched is

TABLE 3

Gradation of Fine Aggregate

Sieve Sizes	Percent Between
<hr/>	
No. 6 to No. 8	5
No. 8 to No. 16	30
No. 16 to No. 30	26
No. 30 to No. 50	26
No. 50 to No. 100	10
No. 100 to No. 200	2
Passing No. 200	1

Figure 25. Autoradiograph of Specimen Containing All Coarse Aggregate

Figure 26. Autoradiograph of Specimen With 90 Percent Coarse Aggregate and 10 Percent Fine Aggregate

Figure 27. Autoradiograph of Specimen With 80 Percent Coarse Aggregate and 20 Percent Fine Aggregate

Figure 28. Autoradiograph of Specimen With 70 Percent Coarse Aggregate and 30 Percent Fine Aggregate

Figure 29. Autoradiograph of Specimen With 60 Percent Coarse Aggregate and 40 Percent Fine Aggregate

Figure 30. Autoradiograph of Specimen With 50 Percent of Both Coarse and Fine Aggregate

Figure 31. Autoradiograph of Specimen With 40 Percent Coarse Aggregate and 60 Percent Fine Aggregate

Figure 32. Autoradiograph of Specimen With 30 Percent Coarse Aggregate and 70 Percent Fine Aggregate

Figure 33. Autoradiograph of Specimen With 20 Percent Coarse Aggregate and 80 Percent Fine Aggregate

Figure 34. Autoradiograph of Specimen With 10 Percent Coarse Aggregate and 90 Percent Fine Aggregate

Figure 35. Autoradiograph of Specimen Containing All Fine Aggregate

the cement slurry that was used to bind the specimens together for the cutting and polishing process. Only the specimens from 100 percent coarse aggregate through 70 percent coarse aggregate needed to be saturated with the cement slurry in order to be cut. All the rest of the mixes formed a stable specimen.

Use of Radioactive Aggregate

One phase of this investigation used radioactive Wyoming urananite aggregate to study asphalt-aggregate distribution. When the Wyoming urananite was used as the entire aggregate there was so much activity that the autoradiographs were very low in definition. This can be seen in Figure 36. The amount of Wyoming urananite incorporated into the specimen was then changed so that only one size fraction at a time was used to see how this size was distributed throughout the specimen. The first step was to make a specimen using only untagged asphalt and filler (Wyoming urananite). The amounts of asphalt and filler used were the same throughout the mixes that are to follow. A two inch specimen was made using 80 grams of filler and 60 grams of asphalt. This specimen was encased in a four inch plaster of paris cylinder, cut and polished. The autoradiograph of this specimen is shown in Figure 37. Notice the texture of the autoradiograph indicating even distribution of the filler material throughout the asphalt.

Figure 36. . Autoradiograph of Specimen With the Aggregate
of Wyoming Urananite

Figure 37. Autoradiograph of Asphalt and Wyoming Urananite Filler

A series of specimens was made using the following aggregate weights; fraction between the 1/2 and 3/8 inch sieves, 460 grams; fraction between the No. 16 and No. 30 sieves, 460 grams; filler fraction (as above) 80 grams; and the asphalt portion (as above) 60 grams. This made the asphalt content 6 percent by weight of the aggregate. The first specimens were made with the Wyoming urananite as filler and with Greencastle limestone for the other two fractions. The second group was made with the No. 16 to No. 30 size fraction of Wyoming urananite and the other two fractions of the limestone. Due to the fact that the emphasis here was on aggregate distribution in the smaller sizes no samples were made using the urananite for the coarse fraction. The autoradiograph of a specimen where the filler was the urananite and the balance of the aggregate was limestone is shown in Figure 38. Figure 39 shows the autoradiograph for a specimen in which the No. 16 to No. 30 size was radioactive. The points of excessive lightness are the places where the alpha particles impinged upon the film.

Since the aggregate used in this research was principally crushed stone, two specimens were made using a gravel. The autoradiograph for one of these, shown only for comparison with the crushed stone, is presented in Figure 40. The mix used consisted of 50 percent coarse aggregate and 50 percent fine aggregate with maximum size of the coarse aggregate 1/2 inch rather than 3/4 of an inch.

Figure 38. Autoradiograph of Specimen Containing Wyoming Urananite Filler

Figure 39. Autoradiograph of Specimen Containing Wyoming Uraninite Between the No. 16 and No. 30 Sieves

Figure 40. Autoradiograph of a Specimen Made with Gravel

Effect of Mixing Time and Asphalt Viscosity

A group of specimens was made to see if the autoradiographic technique could be employed to study changes in asphalt-aggregate distribution that occur when mixing time and viscosity of the asphalt are varied. For this part of the research a modified Hobart mixer was used for all the mixing as opposed to the hand mixing that was used for all the preceding mixes.

From the coarse aggregate-fine aggregate mixtures that were used in the portion of this study involving the effect of aggregate gradation, two combinations were chosen for this phase, the 80 percent coarse aggregate and the 20 percent fine aggregate mix for one, and the 30 percent coarse aggregate and 70 percent fine aggregate for the other. In the autoradiographs for this portion of the work, the cross-hatching in the 80-20 mix as before, shows the hardened Portland cement slurry used to help in the cutting and polishing phases of specimen preparation.

Mixing times of 15, 30, 60, and 90 seconds were used in varying the mixing time. The aggregate appeared to be completely coated at the end of the 60 seconds of mixing. However, a 90 second mixing time was used to insure complete coverage. The autoradiographs of this series of tests are shown in Figures 40 through 48. The mixing temperature for this portion of the research was 275°F.

Figure 41. Autoradiograph of the 30 Percent Coarse Aggregate — Sample Mixed for 15 Seconds

Figure 42. Autoradiograph of the 80 Percent Coarse Aggregate — Sample Mixed for 15 Seconds

Figure 43. Autoradiograph of 30 Percent Coarse Aggregate Sample Mixed for 30 Seconds

Figure 44. Autoradiograph of 80 Percent Coarse Aggregate -
Sample Mixed for 30 Seconds

Figure 45. Autoradiograph of 30 Percent Coarse Aggregate -
Sample Mixed for 60 Seconds

Figure 46. Autoradiograph of 80 Percent Coarse Aggregate -
Sample Mixed for 60 Seconds

Figure 47. Autoradiograph of 30 Percent Coarse Aggregate —
Sample Mixed for 90 Seconds

Figure 48. Autoradiograph of 80 Percent Coarse Aggregate -
Sample Mixed for 90 Seconds

A comparison was made between mixes made by the Hobart mixer and those made by hand. The coarse mixture (80-20) was used and hand mixed until all the pieces were coated. The autoradiograph for the specimen made from the hand mixed sample is shown in Figure 49 for comparison.

Another group of specimens was made using the same aggregate distribution as the above (80-20 and 30-70) and the 90 second mixing time. The variation here was asphalt viscosity produced by varying the mixing temperature. The temperatures used were 225°, 250° and 275°F. An attempt was made at 200°F, but the asphalt was too viscous to mix with the aggregate. The autoradiographs of this series are shown in Figures 50 through 55.

Figure 49. Autoradiograph of 80 Percent Coarse Aggregate -
Sample Mixed by Hand

Figure 50. Autoradiograph of 30 Percent Coarse Aggregate -
Sample Mixed at 225°F for 90 Seconds

Figure 51. Autoradiograph of 80 Percent Coarse Aggregate -
Sample Mixed at 225°F for 90 Seconds

Figure 52. Autoradiograph of 30 Percent Coarse Aggregate —
Sample Mixed at 250°F for 90 Seconds

Figure 53. Autoradiograph of 80 Percent Coarse Aggregate —
Sample Mixed at 250°F for 90 Seconds

Figure 54. Autoradiograph of 30 Percent Coarse Aggregate -
Sample Mixed at 275°F for 90 Seconds

Figure 55. Autoradiograph of 80 Percent Coarse Aggregate -
Sample Mixed at 275°F for 90 Seconds

DISCUSSION OF RESULTS

A discussion of the results given in the preceding section is presented separately to avoid confusion which might occur by introducing the data and discussing it simultaneously. This discussion is divided into two parts; autoradiographic technique and asphalt-aggregate distribution.

Autoradiographic Technique

A considerable amount of effort went into the developing of a technique for obtaining good definition in the autoradiographs. The first reaction when viewing the autoradiograph is that normal photography will show as much detail as the autoradiograph. However, closer examination of the two shows that broken aggregate is visible in the autoradiographs only when radioactive asphalt migrates into the break. The natural photographs show all aggregate pieces that were broken or scarred during the compaction and cutting processes. The crevices due to cutting were so deep that the polishing did not remove the irregularities. This comparison can readily be seen in Figure 21 (the autoradiograph), and Figure 22 (the regular photograph). Both pictures are made from

the same specimen face. Thus the autoradiographic technique offers a means by which fracture due to mixing and compacting, and a fracture due to aggregate breakage under load, or from cutting or some other treatment, might be distinguished.

A better comparison can be made where other than a light-colored aggregate is used. When a dark aggregate is used, such as a trap rock, the definition of the autoradiograph does not change, whereas the normal photography will lose definition until it is almost indistinguishable. This comparison can be seen in the two companion pictures (Figure 23 and Figure 24) when the Belgium black marble was used as the aggregate. In the autoradiograph (Figure 23) the broken aggregate with the asphalt forced into the crack is plainly visible, but in the photograph (Figure 24), this cannot be seen with much clarity. Of course there are all shades of comparison between these two and probably there are situations where the color and surface texture of the asphalt and aggregate very closely approach each other and make differentiation by ordinary photography even more difficult.

The next comparison made is that between the photomicrograph of the sample face and the photomicrograph of the autoradiograph. In Figure 16 the photomicrograph of the sample face is presented and in Figure 17 the photomicrograph of the autoradiograph is shown. The grain of the original autoradiograph is visible in the photomicrograph,

but even with this deterrent better resolution is shown than in the regular photomicrograph.

The larger pieces of aggregate are about equally distinguishable in Figures 16 and 17. However, as the size of the aggregate decreases the definition is lost, first in Figure 16. The very small aggregate particles, the minus No. 100 and No. 200, can be measured in Figure 17.

With respect to the effect of different types of film emulsions, the X-ray film does not show as much definition as the NTB film does, but the sensitivity of the X-ray film is greater than the NTB film. As was brought out in an earlier portion of this thesis the larger the emulsion grain size the greater the sensitivity and the poorer the definition. The X-ray emulsion has a fairly large grain size as compared to the NTB emulsion. However, the X-ray film has a grain size that works well as far as sensitivity is concerned and yet has good definition. The other films used were not as adaptable as the two discussed above although the Dupont emulsion could be used in sheet film.

Considering the fact that it was the least expensive of all films used, the X-ray film proved to be the best all around autoradiographic film. If in some particular cases an added amount of definition was needed a few pieces of NTB sheet film could be used. The X-ray film was used for the volume work until such time as specific high resolution was

needed to show or give emphasis to a particular spot on a specimen. Then the use of the NTB film was warranted. In addition to costing about 45 times as much the NTB film takes at least 10 times the exposure time as does the X-ray film.

Asphaltic preference of different minerals was also shown by use of the autoradiograph. In Figure 32 the largest piece of aggregate on the specimen face is a piece of limestone and chert. Under magnification it is seen that there are discontinuities along the asphalt-chert interface. Figure 56 is a photograph of this same specimen face showing the same relationship as well as the different colors of the two mineral types. However, this photograph does not contain the detail shown in Figure 32. Thus the autoradiographs should be useful in studying the asphalt preference of different types of minerals.

In the film thickness part of the research, the thickness of the film was altered by the change in viscosity of the asphalt brought about by a variation in temperature. The changes of the film thickness are shown in Figures 6 and 7. The lower the temperature the greater the thickness of the film produced when the temperature of the asphalt and the solid are identical. The film thickness was also increased when the asphalt was at a given temperature and the glass or bakelite pieces were at room temperature.

Figure 56. Photograph of a Sample with a Piece of Aggregate That is Part Limestone and Part Chert

Asphalt-Aggregate Distribution

The asphalt-aggregate distribution studies considered the effect of aggregate gradation, use of radioactive aggregates, and effect of asphalt viscosity and mixing time variables. These all seem to be closely interrelated as far as this research is concerned.

In the aggregate gradation phase of the research two aggregate fractions were mixed with the percentage of asphalt that gave maximum stability in the Marshall test (fine aggregate), or the amount of asphalt the aggregate could hold at the particular mixing temperature (coarse aggregate). This gave about the optimum asphalt content for each fraction. If the asphalt had any preference for one particular fraction in the mixture it would concentrate around this fraction and be noticed in the autoradiographs. However this preference for a particular fraction was not observed.

In Figure 25 through 35 a series of mixes is shown beginning with the 100 percent coarse aggregate. (The apparent fines in Figure 25 are due to the aggregate degradation during mixing and compacting).

As the fines are added to the mix there does not seem to be any change of the film thickness of the asphalt around the coarse pieces. In Figure 25 the outline of each piece by the light line shows this film thickness. (Again the cross-hatched area is the Portland cement slurry that was

used to hold the sample together during the cutting and polishing process.) In each of the following figures (Figures 26 through 35) as the percentage of fines increases the fines mix with the asphalt as a mortar and then coat the coarse pieces. By the time 40 percent of the total mix is fine aggregate the coarse fraction is dispersed in the mortar of the fine aggregate and the asphaltic cement.

The coating on the coarse pieces is not a film of asphaltic cement alone, but a matrix of the mortar consisting of asphalt with the fine fractions embedded in it. In Figure 34 this is quite pronounced due to this mix having only 10 percent of the coarse fraction in it. In this figure only four coarse pieces can be seen, each embedded in the matrix of the fine aggregate-asphaltic cement mortar.

It is also noted that as the fine aggregate is added to the coarse fraction the fines always adhere to the coarse fraction or appear in a concentration in the interstitial part of the coarse fraction. This adherence to the coarse fraction is part of the so-called film thickness of the coarse aggregate. As was indicated before it is composed of the asphalt and the finer parts of the fine aggregate. When there are some of the coarser fractions of the fine aggregate present, they tend to form a mortar and fill the interstitial part of the coarse-aggregate mix.

The specimens made which contained radioactive aggregate as only one fraction of the aggregate gradation showed an

even asphalt-aggregate distribution. When the material passing the No. 200 sieve was radioactive it combined with the asphalt in much the same way as did the sulfur-35. If this mortar had any preference for an aggregate fraction it would show in an autoradiograph as a light area at the surface of the aggregate size it preferred. This did not occur with either the radioactive aggregate or the tagged asphalt. Therefore when the filler material combines with the asphalt, the mortar thus formed acts very much like the asphalt itself when placed in a mix. In Figure 38 the minus 200 material was radioactive and the other two fractions were limestone. In this autoradiograph there is no concentration any place (the light spots are due to alpha particles and are not to be confused with a high concentration of the beta particles). The uniformity of the autoradiograph shows no concentration, but does show even distribution of the radioactive material throughout the specimen cross section.

In Figure 39, the No. 16 to No. 30 fraction was the radioactive aggregate and the other two fractions were limestone. Again there is an even distribution of the radioactivity. Some of the coarse aggregate pieces, shown in Figures 38 and 39, do not have a sharp line of demarcation because the energy level of the beta particles from the aggregate is higher than those from the sulfur-35. An even distribution of radioactive aggregate is shown in the three

autoradiographs (Figures 37, 38 and 39) made from specimens that were made with one fraction of the radioactive aggregate.

Mixing time and asphalt viscosity were varied to see if the autoradiographic technique would reveal differences in asphalt-aggregate distribution resulting from these variations. In Figures 41 and 42 a 15 second mixing time was used. After this mixing period most of the coarse pieces still were not completely coated. However, after they were compacted there was evidence that almost complete coating was accomplished. In Figure 41 the 30 percent coarse aggregate specimen shows that only at points of coarse aggregate contact does a coating seem to be lacking. In Figure 42 wherever the coarse aggregate was not coated, but was in contact with some other coated piece, or matrix, a coating exists. If no contact was made the aggregate remained uncoated. (See large piece at right hand side of autoradiograph, where there is no change between the aggregate piece and the Portland cement slurry). In all the rest of the 30 percent coarse aggregate autoradiographs, the coarse aggregate is coated (see Figures 43, 45, and 47).

In Figure 44 (mixing time 30 seconds) some contact points between the coarse particles are not coated but generally speaking all the pieces are coated. In Figure 46 (60 seconds mixing time) all the pieces are coated, and most

of the contact points are coated. The only place where coating does not occur is where aggregate degradation occurred. These are filled if there was some of the matrix close enough to them for the hydraulic action of the compaction to force the asphalt into the fracture. In Figure 48 all pieces are coated except a few contact points. The contact points are not coated because the high pressures at these points of contact forced the asphalt away during the compacting process. In Figure 49 even more coverage is shown than in the machine mixed samples, but in this case the sample was mixed by hand until all the pieces were coated. Notice also that in the hand mixed sample more of the coverage is of the matrix type.

In Figure 45 the circled part of the autoradiograph is a hook on a large piece of the coarse aggregate. The compactive effort of the Marshall hammer has forced the matrix into this hook showing the extent of the hydraulic action produced by compaction. This coupled with the above information indicates that in a dense graded-mix, even though there is not complete coverage at the end of the mixing operation, coverage is obtained when the material is compacted.

The asphalt viscosity variations were achieved by varying mixing temperature. In Figure 50 (225°F) there are some balls of fines and asphalt that were not broken down during

the mixing operation. When the temperature was increased to 250°F only one very small ball of this material could be found (Figure 52). When the temperature reached 275°F none of this material was left unmixed.

Autoradiographs of mixes that incorporated a change of viscosity as the variable are shown in Figures 51, 53 and 55. These autoradiographs show that as the asphalt became more viscous a thicker film of matrix covered the coarse aggregate pieces.

SUMMARY AND CONCLUSIONS

In this section of the thesis the results obtained are summarized and the conclusions that were drawn from these results are presented. The statements of the summary and the conclusions apply to asphalt-aggregate mixtures specifically.

Summary of Results

The following points summarize the results that were obtained from the research phase of this study. They are organized to place emphasis on certain groupings.

1. Sulfur-35, elemental sulfur in benzene, can be mixed with heated asphalt so that it is evenly dispersed. A carrier for the isotope should be used for best results.
2. When the tagged asphalt was used in making asphaltic concrete, autoradiographs could be made from sections of a compacted specimen.
3. A diamond saw can be used to satisfactorily cut the asphaltic concrete samples. It was found that the technique works best if water is used as the coolant and the sample temperature is about 10°F.
4. A galvanized iron cover and exhaust fan for the saw were designed to control radioactive contamination during specimen cutting.

5. A plane surface was obtained on the cut face of the asphaltic concrete specimen by dry grinding with a belt sander. Hand polishing with aluminum oxide paper then produced a surface of satisfactory smoothness.

6. Excellent autoradiographs were obtained when the procedure as finally developed in this study was followed.

7. Autoradiographs could be made of asphalt-aggregate mixtures using radioactive aggregate.

8. Good definition was achieved in autoradiographs where the only radioactive material is one fraction of the mineral aggregate.

9. The mixing of radioactive filler material and asphalt gave as even a dispersion as was obtained with the elemental sulfur-35 in benzene.

10. Photomicrographs can be made from an autoradiograph.

11. Asphaltic intrusion into very small cracks can be read from an autoradiograph irrespective of the type or color of the aggregate used.

12. In some cases differences in asphalt film thickness can be measured using the autoradiographic technique.

13. Differences can be seen in the autoradiographs from which it might be possible to determine the variation in adhesion of asphalt to different mineral types.

14. Differences in asphalt-aggregate distribution resulting from variations in mixing time can be observed using isotope tracers.

15. Differences in asphalt-aggregate distribution resulting from variations in asphaltic viscosity can be observed using the autoradiographs.

16. Autoradiographs like photographs retain the size, shape and orientation, at a given time, of the asphalt-aggregate distribution of a given sample.

17. Autoradiographs can be made on different types of film emulsions.

Conclusions

The results obtained from the analysis of the test data appear to justify the following conclusions. However, it should be realized that in general the conclusions are only applicable to the particular asphaltic mixtures and sample preparations that were used in this research.

The autoradiographic technique is very adaptable to the study of the asphalt-aggregate distribution in an asphaltic mixture. Good definition can be obtained using X-ray film emulsions, but better definition can be achieved by the use of the NTB autoradiographic stripping film emulsions. The use of a weak beta emitter gives better definition than the use of a highly energetic beta emitter.

Autoradiographs can be made using many different types of film emulsions. The data collected during the research phase of this investigation proved that the X-ray film worked very well for the multitude of autoradiographs made because

of its high sensitivity and its good definition. The NTB film gave better definition, but was less sensitive and more expensive, and should be used only if the extra definition is needed.

Autoradiographs give more detail than regular photographs where the aggregate is dark and it is difficult to distinguish between the color of the asphalt and the aggregate. The autoradiograph will not show the imperfections of the aggregate particles that are caused by the cutting of the sample. Polishing of the sample face will remove some of these imperfections as well as any asphalt that has been deposited on the aggregate during the cutting process, and the aggregate will remain clear in the autoradiograph. Imperfections that occur during the mixing and compaction operation will show when the asphalt has intruded into the crack. Thus the autoradiograph indicates the asphalt-aggregate distribution more precisely than the photograph when the aggregate-asphalt colors are very similar.

Film thickness can be observed in open bituminous mixtures and single pieces of aggregate when hand dipped. However, when the gradation gives a relative dense mixture specific film thickness cannot be observed. In this dense condition the fines and the asphalt form a matrix and the coarse pieces are dispersed in the matrix. Therefore, in this dense condition the mixture acts very much like Portland

cement concrete in its ground mass to phenocryst relationship. The mixture also resembles Portland cement concrete in appearance of the binder-aggregate distribution. In dense-graded mixtures, no films as such are formed on any particular size of the aggregate.

Even though complete coverage might not be achieved at the conclusion of mixing, in a graded mixture any portion of the aggregate not covered will be covered during the compaction process. In open mixes this will not occur because at points of high contact pressures between points of the aggregate, the asphalt will be forced away from the aggregate. Thus in speaking of mixing efficiencies of bituminous mixes it is important to separate open and graded mixes.

RECOMMENDATIONS FOR FURTHER RESEARCH

The autoradiographic technique offers a very versatile tool for the investigation of some of the fundamentals of bituminous mixtures. The use of isotopes opens a vast area of undetermined scope in the field of bituminous research, in fact, in the whole field of Civil Engineering. There are numerous investigations that could be made to further the basic understanding of bituminous mixtures. The following are but a few of the many facets that could be construed as future research projects.

1. The use of the tracer technique employing radio-isotopes could be further expanded than is done in this thesis. A follow up on further methods and uses would indeed add to the versatility of this tool. It would also aid in understanding some of the basic fundamentals of asphaltic concretes and bituminous mixtures.

2. A combination of the autoradiographic approach and specific activity determination could be explored to see if a correlation could be made so that the autoradiograph could be more analytic.

3. The work on mixing efficiency should be expanded so that a comparison of "as mixed" and "as compacted" conditions

could be made. If the coverage could be made in compacting the mixture the "as mixed" coverage is not important.

4. A follow up on research previously done on asphalt content in bituminous mix in the field would be an excellent application of the radioactive technique. Added to this should be in place density.

5. A further extension of the work on film thickness could be made to see where the film thickness criteria can be applied and where the matrix or ground mass criteria is applicable.

6. Some field work could be done using a radio-isotope or radioactive aggregate to see if there is much vertical displacement of the aggregate during rutting and shoving.

7. A study of adhesion of asphalt to different mineral types could be made to study stripping characteristics of each mineral before any water or water vapor tests were made. Also specimen cross sections could be studied while the stripping test was in progress.

8. A study of aggregate break-down in laboratory testing and aggregate degradation in service could be made utilizing radioactive tracers.

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APPENDIX A

RADIOLOGICAL CONTROL

Laboratory Procedure

The precautions for safe procedure in the laboratory may be summarized as follows:

1. Do not smoke, eat, or use lipstick in the laboratory.
2. All protective clothing should be kept in the laboratory.
3. Where prescribed by the use of certain radioactive materials a film badge or other approved personnel monitoring device should be worn in the laboratory at all times. The keeping of records of personnel radiation doses should be done by a central agency in charge and not by each individual.
4. Do not use the mouth for pipetting and do not put the pipette in contact with other laboratory equipment.
5. Work over absorbent paper so that if slight losses occur they will be caught by the paper.
6. Plan the work carefully to minimize any danger of spilling radioactive material.
7. Meter all radioactive samples and determine the safe working distance or shielding procedure before beginning work.

8. Avoid handling samples in such a way that any radioactive material can be transferred to the hands or other parts of the body.

9. Wear rubber gloves when using samples that could contaminate the hands while working.

10. Always keep samples covered, to prevent counter contamination never place samples in a counter unless permission is secured.

11. Meter the working area at the beginning of each working period and again at the end. The health officer will give the permissible levels.

12. If a spill occurs report immediately to the health officer and begin taking the requisite steps for cleaning it up according to the prescribed radiological control manual.

13. All waste disposal will follow that section on waste disposal in the radiological control manual.

14. Remember to respect radioactive materials as any caustic or harmful chemical.

APPENDIX B

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SPECIFIC ACTIVITY COUNTING

The equipment used to count specific activity was described in the portion of this thesis entitled "Materials and Procedures" and is shown in Figure 3. The specimens used were cut into three pieces and each face was polished in the prescribed manner. The open-window counter was then placed on the specimen face in such a manner that five different counting positions were obtained. In each position three one minute counts were recorded. Therefore, for each average shown in Table 4 there were three one minute counts per position, five positions per face and four faces per specimen, on a total of sixty counts per average.

The first data were obtained using a counter with a window opening diameter of 0.98 inches. When the data were analyzed there was no significant difference between different gradations of aggregate. In the G-3 series a reversal of counts in asphalt percentage between specimen G-3-5 and G-3-7 occurred.

The next data were obtained using a counter with a window opening diameter of 1.14 inches. The counting process was repeated. Again there was no significant change in the data recorded.

It was hoped that by using this more analytical technique the effectiveness of the autoradiographic information would be augmented. If more time were allotted to this technique it is possible that a method could be perfected, and a relationship between counts, percentage of asphalt and gradation might be established.

In Table 4, the specimen number, viz,

G-1-3 means:

G = Graded aggregate distribution,

1 = Gradation (coarse, medium and fine).

and 3 = Asphalt (percent by weight of aggregate).

TABLE 4

Specific Activity Counting Data

Specimen Number	Average Counts per Second	
	Counting Diameter 0.98 inches	Counting Diameter 1.14 inches
G-1-3	95	186
G-1-5	147	292
G-1-7	157	317
G-1-9	186	365
G-2-3	111	213
G-2-5	133	264
G-2-7	140	267
G-2-9	161	310
G-3-3	114	215
G-3-5	164	326
G-3-7	149	289
G-3-9	182	353

VITA

VITA

Donald Roy Lamb was born on May 6, 1923 in Yuma, Colorado. He went to grade and high school in Casper, Wyoming.

He entered Hastings College, Hastings, Nebraska in 1941. During the years 1943-1945 he served in the 8th. Air Force as a navigator. He returned to Hastings College in the fall of 1945 and finished in August 1946, receiving a Bachelor of Arts Degree.

In the fall of 1946 he was Superintendent of Schools at Dalton, Nebraska. During the years 1947-1949 he served as coach and classroom teacher in the high school of Madrid Consolidated Schools, Madrid, Nebraska.

He entered the University of Wyoming in 1949 receiving his Bachelor of Science in General Engineering, June 1951. He was Supply Instructor of Civil Engineering for the next two years and received his Masters Degree in Civil Engineering in March 1953. He was then made an Instructor of Civil Engineering in the fall of 1953 and received his Degree of Civil Engineer in 1958 from the University of Wyoming. He received a National Science Foundation Faculty Fellowship for 15 months beginning June 1960, and a sabbatical leave

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He is a member of Sigma Tau, Tau Beta Pi, Phi Kappa Phi, Sigma Xi, Omicron Delta Kappa, the American Society of Civil Engineers, the American Society for Engineering Education, National Society for Professional Engineers, Wyoming Engineering Society and is a registered Professional Engineer in the State of Wyoming. He has also served in many capacities in his home community on religious, charitable, educational and service groups or committees.

The titles of his theses for Master of Science in Civil Engineering, and Civil Engineer are as follows:

1. "A Thermal Study of Wyoming Aggregate", 1953.
2. "A Study of the Application of Nuclear Particles to Some Properties of Engineering Materials", 1958.

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1. "Concrete Aggregate Problems in Wyoming", (with H. S. Sweet), 8th Annual Field Conference Guidebook, Wyoming Geological Association, 1953.
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