

## AN ABSTRACT OF THE THESIS OF

Andrew J. Hoffman for the degree of Master of Science in Civil Engineering presented on  
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Title: Petroleum Refinery Residuals: A Recycling Feasibility Study.

*Redacted for Privacy*

Abstract Approved:

 James R. Lundy  \_\_\_\_\_

Large quantities of unused heavy oil and residual waste products from the petroleum refining industry were deposited in on-site landfills, pits, and lagoons, prior to the promulgation of regulations governing their disposal and technology development that would further utilize and/or recycle these materials.

Contacting petroleum products with sulfuric acid for clarifying and stabilizing purposes, adding lead oxide to certain crudes to reduce sulfur odors, and filtering products through a medium of Fuller's Earth has been customary practice within the industry for many years. "Acid tar," otherwise known as petroleum refinery residuals (PRR), is essentially a combination of one or more waste streams resulting from these processes.

Estimated gross quantities of PRR existing at an undetermined number of sites across the United States exceed 10 million cubic yards, of which, an estimated cost for remediation approaches one billion dollars. Contaminated sites must be remediated in order to be acceptable for land development and to meet regulatory and public scrutiny. Considering the volume of this waste currently existing in the United States, it is

imperative that feasible, cost effective, and realistic recycle and/or disposal options be defined and implemented.

This study evaluates the environmental implications, structural integrity, and economics of incorporating PRR into an asphalt concrete. Two PRR sources are evaluated for their potential use as a recycled material when incorporated into an asphalt concrete mixture: 1) A characteristically toxic and acidic PRR from Pennsylvania and 2) A non-hazardous acidic PRR from Oklahoma. Both sources maintained a sludge-like consistency following neutralization with lime (NRR). The Oklahoma source was further processed with lime to yield a mixture (TRR) with a consistency similar to that of a silty sand.

Hot and cold mix studies were conducted, utilizing gap and dense gradations with varying percentages of PRR. Mixtures excluding aggregate were also prepared using TRR and varying quantities of asphalt emulsion.

Hot-mix results produced favorable structural parameters, however, increased lead leachate and odor problems eliminated this option. Strength parameters for cold-mixed dense-graded specimens are within acceptable limits for use as a bituminous stabilized base.

Neutralized PRR from Oklahoma appear to be no more of an environmental threat than a typical asphalt concrete. However, water sensitivity of PRR-modified mixtures was found to be higher than non-modified mixtures.

Economically, this form of recycling is favored over incineration, when considering a characteristically hazardous PRR. However, other remedial alternatives may prove economically superior to recycling. Prudent means of remediation need to be considered on a site-by-site basis.

**PETROLEUM REFINERY RESIDUALS: A RECYCLING  
FEASIBILITY STUDY**

by

Andrew Joseph Hoffman

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# **PETROLEUM REFINERY RESIDUALS: A RECYCLING FEASIBILITY STUDY**

## ***1 INTRODUCTION***

Long before the drilling of the first oil well, petroleum was known to exist and was used in the United States exclusively for its illumination and medicinal purposes.<sup>1</sup> In Northwest Pennsylvania, it was initially collected by skimming from the surface of a small creek. During the month of August, 1859, near the town of Titusville, Pennsylvania, Edwin L. Drake demonstrated for the first time that there were vast quantities of petroleum that could be marketed commercially. This development was the beginning of a new era and may be the single most important event in the ensuing mechanical revolution.

Technology would render a market for mass production of various products derived from crude oil. However, lack of technology and a marketable product that exploited the heavy portion of crude oil hindered its utilization. Consequently, this product was deposited in on-site ponds and lagoons at many early refineries.

Clearly, new technologies can have a profound impact on society. They also impacted the environment, both positively and negatively. The challenge is to use technology in such a way that it does not lead simply to short-term advances in productivity at the expense of long-term resource viability. Recognizing that it is not technology alone that will lead to a sustainable future, but also progressive environmental



and economic policies, as well as changing human behavior that will determine whether the world achieves sustainable development.

### ***1.1 Purpose***

This report reviews possible solutions to address an environmental problem that has existed since the drilling of Drake's Well. Namely, a byproduct resulting from the petroleum refining practice of the past 120 years, called petroleum refinery residuals (PRR), otherwise known as acid tar. PRR is a concern to petroleum companies because of the incredible volume of material existing in their original disposal areas (i.e., pits and lagoons) and the cost of environmental restoration to these sites.

PRR is typically acidic, often contains significant amounts of lead, and is not structurally suitable for land development, as will be discussed in Chapter 2. Therefore, treatment of PRR, prior to transport or landfilling, is necessary. Table 1-1 establishes a conservative estimate of the costs involved for processing and landfilling PRR stores believed to exist in the United States (unit cost of landfilling based on recent project expenditures at a Superfund site near Tulsa Oklahoma).<sup>2</sup> The estimated gross quantity of PRR existing in the U.S. is 13,000,000 cubic yards (CY).<sup>3</sup> Multiplying this value by the estimated unit cost of \$75.63/CY<sup>2</sup> for processing reveals the estimated total cost of landfilling existing PRR stockpiles to \$983,190,000.

These figures establish the motivation behind this study. Namely, a solution that might consist of a treatment option that would render the PRR a useful and recyclable

product while remaining technologically and environmentally feasible. Any reduction in the unit cost of processing the PRR would result in direct savings to the property owner and translate to consumer savings as well. If a recycled product, such as a pavement layer, could be developed that utilized PRR, the effect would essentially be a transformation from what would have been a \$983,190,000 investment into an unusable landfill to a much needed element of the U.S. infrastructure.

**Table 1-1 Economics of Treatment and Landfilling PRR**

Activity	Estimated Quantity <sup>vv</sup>	Units	Unit Cost (\$)	Cost (\$)	Comments
Excavate and treat PRR <sup>v</sup>	13,000,000	CY	44.00	572,000,000	Cost est. based on actual costs of Oklahoma Project
Transporting cost	13,000,000	CY	7.50	97,500,000	Within 50 mile radius using a dump truck (\$0.15/CY/mi.)
Placement cost	13,000,000	CY	9.00	117,000,000	Grader/cat. compactor (estimate)
<b>Subtotal</b>				<b>786,500,000</b>	
General allowance	5%	-	3.03	39,325,000	
Waste considerations allowance (including QA/QC, health and safety, equip move/demove etc.)	20%	-	12.10	157,300,000	
<b>Estimated Total</b>	13,000,000	CY		<b>983,190,000</b>	

<sup>v</sup>End-product is treated PRR (TRR).

<sup>vv</sup>Estimated quantity based on initial estimate multiplied by ten (approximate number of other large oil companies in the U.S.).

The literature reviewed for this study revealed no reported occurrences or attempts to recycle PRR into any kind of product, as defined in Chapter 3. Remedial activities involving PRR contaminated sites have historically consisted of incineration, treatment and/or solidification of materials in situ or after excavation, and to a limited extent, bioremediation. All these technologies translate to exorbitant costs to responsible parties performing the remediation, and ultimately, to the consumer. A cost comparison table between incineration and recycling is developed in Chapter 5.

## ***1.2 Scope***

This report explores the possibility of recycling PRR into asphalt concrete by replacing portions of the asphalt cement and aggregates with PRR. Including PRR in pavement structures has several advantages over alternate disposal options. First, expenditures required for processing PRR could be offset by producing a salable product that could safely contribute to the nations infrastructure. Second, transportation industries, specifically road building, are the largest consumers of materials worldwide. This potentially creates a large market for PRR-modified materials. Third, transportation agencies are receptive, and in some cases, mandated to incorporate recycled materials and industrial byproducts in their road building materials (i.e., fly ash, and ground tire rubber). Therefore, the potential consumer is more receptive to PRR-modified products.<sup>4</sup>

In order to assess the viability of using PRR as an asphalt substitute and/or pavement mixture additive, samples from two sites were collected and tested: 1) an acidic

PRR from Pennsylvania that contained a total lead content of approximately 1 percent, and 2) an acidic PRR from Oklahoma that contained significant amounts of sulfur-based compounds, but minimal lead content. In addition, a field demonstration was conducted with the Oklahoma PRR where several cold mix asphalt emulsion designs, developed in the laboratory, were constructed and cured in the field.

Both PRR sources were tested in one or more of three combinations that had potential application in pavements. The first was a hot-mixed combination of aggregates, PRR, and asphalt cement heated to approximately 125C, 100C, and 125C, respectively. The second combination was cold-mixed using aggregates, PRR, and a cationic asphalt emulsion. All specimens prepared in this fashion were mixed with constituents at room temperature [approximately 22C (72°F)]. The third mix excluded aggregates and combined asphalt emulsion with a processed treated PRR (TRR), as will be defined in Chapter 3. The as-received TRR was mixed with emulsion at room temperature and immediately compacted.

Evaluating the structural and environmental suitability of the PRR-modified materials involved established testing procedures. Structural testing procedures included Marshall stability and flow, diametral resilient modulus, and evaluation of water sensitivity. Testing for the environmental suitability of PRR-modified materials included whole sample analysis, the Toxicity Characteristic Leachate Procedure (TCLP), and the environmental conditioning system [ECS, developed by the Strategic Highway Research Program (SHRP)].

The following chapter will define the primary constituents believed to comprise a typical PRR, discuss remedial activities historically implemented at PRR contaminated sites, evaluate pertinent regulatory information pertaining to the recyclability of PRR, describe a typical pavement structure, and discuss the necessary structural criteria that a PRR-modified material must meet for recycling to be considered as a viable option.

## **2 BACKGROUND**

### **2.1 *Petroleum Refinery Residuals (PRR)***

Characteristics of crude oil, and therefore PRR, vary and can often be predicted for a given geographical region. These variations consist of a broad range of physical properties, ranging from color to specific gravity. Regional site variations should be considered when evaluating treatment methods of PRR, for many of the crudes were likely refined near their source, due to transportation constraints during the mid to late 19th century. Synopses of crude oil characteristics from two regions within the United States<sup>5</sup> are as follows:

1. The Appalachian field, which includes crudes originating in New York, Pennsylvania, West Virginia, and Eastern Ohio and Kentucky, are typically paraffin based, have a high specific gravity, and are practically void of sulfur and asphalt. Oil products refined from Appalachian crudes are of the best found in the United States.
2. The Mid-continent field encompasses Oklahoma and Kansas. This field exhibits a wide range of characteristics that typically have a high sulfur content, low specific gravity, and are paraffin based with varying percentages of asphalt present.

### *2.1.1 Definition*

Petroleum refinery residuals (PRR) consist of the heaviest fraction of crude oils and residual waste constituents not utilized in products from the oil refining process of the late 19th and early 20th centuries. Prior to the advance of technologies that enabled further refining of these materials, wastes were typically disposed of on-site in landfills and lagoons, where mixing with native soil commonly occurred due to lack of confinement. As petroleum refining practices advanced (i.e., thermal cracking, acid recovery, etc.), the waste generated from each barrel of crude oil was gradually reduced. Due to the lack of advanced technology, the earlier sources of PRR contained a motley assortment of hydrocarbons, clay, and acidic solutions. The following materials combine to make up a typical PRR:

1. Acid tar or sludge—produced as a byproduct from a stabilization process where the untreated product stream was contacted with sulfuric acid, causing the polymerization of unstable compounds (i.e., olefins). This process improved product stability and color and decreased gum forming materials. When processing “sour crudes” (crudes with significant quantities of sulfur-based compounds), lead oxide was likely added in this stage of treatment as a sweetening process that removed sulfur odor by forming a lead sulfide.<sup>4</sup> The polymerized compounds, consumed acid, and, when present, insoluble lead sulfides then settled out and were removed as a sludge. This is the primary component existing in PRR materials.

2. Fuller's Earth—Certain clay minerals, notably palygorskite (Attapulgas clay originally found in Attapulgas, Georgia), sepiolite, and some smectites (bentonites), are used as a filter to improve color and stability of, or remove water from, petroleum products.<sup>6</sup> In the refining process, Attapulgas clay and calcium bentonites, as well as other clay minerals, are used to remove polar and heavy compounds that tend to cause gumming in petroleum products. Fuller's Earth minerals are a nonswelling form of clay that break down to a fine granular consistency after exposure to water. Excess water will often release hydrocarbons from the mineral structure in order to make preferential contact with water molecules.
3. Lubricating oils, cylinder oils, and gasoline fractions—The original refineries recovered illuminating oil only and discarded or burned the remainder of the crude (i.e., pre-1870's). The bottom of the barrel or the heaviest fraction was the last portion to be put to useful purposes. Depending on the refinery, the bottom fraction was discarded as late as the early 1900's.<sup>7</sup>

When left in their original disposal area, these materials render a site unsuitable for building and are an environmental hazard, due to acidity and possible high lead content,. PRR tend to have a very low shear strength (i.e., they tend to flow under their own weight), due to high moisture contents and composition of inherently weak materials. The original deposition of PRR (i.e., surface impoundments) require their removal or solidification prior to land development.



A typical PRR sampled in Pennsylvania consisted of approximately 27% water and 1.5% volatile organics. A toluene extraction revealed approximately 40.5% insoluble (i.e., soil, coke, etc.) and 31% soluble material, such as paraffin and/or other heavy organic constituents.<sup>a</sup>

### ***2.1.2 Prior Research Findings***

A previous study conducted on Pennsylvania PRR revealed the following pertinent information:<sup>8</sup>

- Flash Point per ASTM D93-79—PRR ignite at a temperature between 170C and 180C, after which, the material experienced significant volume and material character change (i.e., spattering, due to the presence of moisture);
- Neutralization (i.e., pH @ 7) of the PRR was accomplished by adding 8% hydrated lime;
- Moisture loss, due to heating, causes the material to loose adhesive qualities;
- Rheological properties of the PRR with respect to temperature and moisture variation are of concern; and
- PRR affinity for water indicates likely water sensitivity.

---

<sup>a</sup> Sample analysis conducted by PRI, Asphalt Technologies Inc., Tampa, Florida

Due to the inherent acidic nature of most PRR and possible high lead levels associated with refined sour (i.e., sulfur containing) crudes, processing of the PRR to mitigate these hazards is often necessary prior to, or in conjunction with, most treatment remedies. The following section outlines a variety of mitigation techniques historically used to remediate sites contaminated with PRR and discusses advantages and disadvantages associated with each method.

## ***2.2 Mitigation Techniques Historically Implemented***

The final disposition of PRR can vary depending on the chosen mitigation approach. Options include incineration, in situ treatment, off-site landfilling, on-site landfilling, or recycling.

### ***2.2.1 Incineration***

Incineration involves a fairly simple chemical process called combustion. When ignited, organic compounds (i.e., compounds that contain carbon, hydrogen, and sometimes oxygen) break down in an exothermic process to render carbon dioxide and water vapor. Incineration reduces the waste volume. However, if the material to be incinerated contains toxic components, it may still remain a threat to the environment if those components are not destroyed during incineration (i.e., expelled to the atmosphere or remain in ash residues).

Occasionally metals are complexed with an organic molecule (i.e., integrated into the organic structure—see Chapter 5) and therefore become entrapped in the post-combustion residues, such as ash and/or emissions to the atmosphere. This can complicate the incineration process by requiring the installation of air quality monitoring systems and exhaust stack scrubbers.<sup>9</sup> Disposal of the residual ash may also be regulated by the Resource Conservation and Recovery Act (RCRA). Therefore, wastes containing significant amounts of non-combustible contaminants may be better suited to other forms of treatment.

Many hazardous waste incineration companies prefer that the waste product be in a liquid form. The PRR evaluated in this study has a sludge-like consistency and would therefore require processing to attain a liquid state. The energy output of the PRR material considered in this study is from 10,000-12,000 Btu/lb.<sup>10</sup> The typical energy released by one pound of coal is approximately 13,000 Btu.<sup>11</sup> Therefore, coal and PRR have very similar combustible energy levels. However, combustion products resulting from PRR incineration, as previously mentioned, may be considered hazardous, and would therefore, still fall under RCRA regulation. As mentioned earlier, one PRR source evaluated was found to contain 40 percent (by weight of wet sample or 57 percent by weight of dry sample) insoluble material, such as coke and soil. Assuming this fraction would not be reduced by incineration, considerable waste handling would be required (i.e., landfill or recycle) after incineration.

### 2.2.2 *In Situ Treatment*

In situ literally means in place, and in the context of treatment systems means that the waste is not removed from the storage or disposal area to be processed. In situ treatment can be accomplished by



*Photo Courtesy of ENRECO, Inc.*

**Figure 2-1 Shearing Injector**

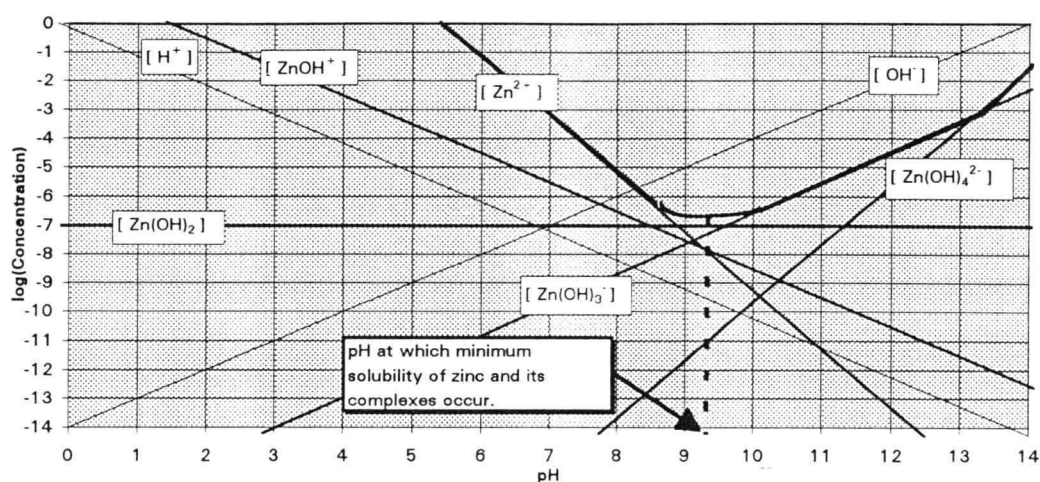
injecting neutralizing, stabilizing, biological and/or

solidifying agents into the sludge via a backhoe, auger, rotary tilling device, or shearing injector, as illustrated in Figure 2-1. Cutting blades attached to injectors fracture and loosen viscous sludge or soil while reagents flow into the furrows, and repetitive raking blends the components. Remedial action using this technology is useful for improving the appearance and stability of the PRR, coupled with neutralization of the sludge for an overall improvement to the environment. Certain PRR emit toxic sulfur dioxide gas and hydrogen sulfide gas after the injection of neutralizing agents such as lime or cement kiln dust. ENRECO Inc. has developed a method for mitigating these emissions by flooding a work area with a lime-water mixture, then slowly injecting the treating agent (lime in many cases) into the underlying sludge. The surface layer of lime and water then acts as a chemical scrubber to suppress emissions.<sup>12</sup>

The most common alkali material used for the chemical fixation and solidification (CFS) of sludge-like organic materials is lime. Quicklime ( $\text{CaO}$ ) and hydrated lime [ $\text{Ca}(\text{OH})_2$ ] are the two most common limes used in CFS work. Hydroxides released in lime-based systems form non-soluble complexes with metals at some optimum pH, thus causing precipitation of the metal.<sup>13</sup> What is meant by complexing and solubility is briefly explained below.

Metals exist in solution in forms other than simple ions or molecules, as they are commonly presented (i.e.,  $\text{Pb}^{++}$ ,  $\text{Ca}^{++}$ , etc.). Metal ions in solution are usually associated with water molecules in a definite arrangement. When the water molecules are replaced by other ions or molecules, the result is termed a metal complex. The chemical bonds involved are covalent rather than ionic, that is, electrons are shared between the bonded atoms, instead of being transferred. A metal complex may be inorganic or organic, an ion or a neutral molecule, and soluble or insoluble. Precipitation (i.e., changing to an insoluble state) occurs when the pH of a solution of dissolved metal ions is raised to some optimum level for a specific metal. The optimum pH is different for each metal, and often, for different valence states of a single metal. It may also vary for a specific metal ion with the presence of other species in solution.

As the pH is increased, the metal forms different complexes, some of which may be soluble. Thus, new species are formed, with combined concentrations accounting for all the metal in solution. Figure 2-2 illustrates the idea behind solubility and complexation. However, this is just an illustration. The actual phenomena occurring in the PRR-lime



**Figure 2-2** Log (concentration) vs. pH Diagram of a Typical Metal and its Hydroxide Complexes<sup>14</sup>

matrix may be entirely different, due to the complexity of the PRR chemical makeup, as discussed in Chapter 5.

Cement is also an alkali material suitable for acidic waste. Cement-based stabilization systems have a number of advantages. Technologies concerning handling, mixing, setting, and hardening of cement are well known. Cement is widely employed in the construction field, and, as a result, the material costs are relatively low and required equipment and skilled personnel are readily available. Dewatering of wet sludges is often not necessary as water is required for cement hydration. The only disadvantage of cement-based systems is the sensitivity of cement hydration to organic constituents in waste materials. Organic compounds may hinder the formation of the crystalline structure responsible for strength gain in concrete, impeding set time and decreasing

durability of the cement-based system.<sup>13</sup> Therefore, solidification using cement is not considered practical for use with organic-based wastes.

### ***2.2.3 Off-Site Treatment Facilities***

Off-site treatment facilities change the physical and/or chemical characteristics of a waste by degrading or destroying hazardous constituents, using any of a wide variety of physical, chemical, thermal, or biological methods. Transport of waste to off-site facilities for treatment and disposal is an option for sites not suitable for on-site in situ treatment, or not having sufficiently large volumes of PRR to justify the investment in an on-site facility. There are at least 50 commercially proven technologies for the recovery and treatment of hazardous waste.<sup>9</sup> However, the only technologies implemented on PRR-like material are solidification using lime-based products, incineration, and, to a limited extent, bioremediation. A hazardous waste facility may function with just one technology, or it may combine technologies in the event that it is serving a number of generators.

### ***2.2.4 On-Site Landfilling***

On-site landfilling involves the permanent emplacement of a treated waste on or below the land surface at the location of generation or storage. This option may be favorable at sites where the waste material is generated continuously, or has been generated in sufficient quantities to warrant placement of facilities and the development

of technologies for on-site disposal. The same treatment options would apply as those considered for off-site disposal.

### *2.2.5 Recycling Facilities*

Recycling facilities reclaim material as a salable product (typically solvents, oils, acids, or metals). Some recover energy values in waste, while others attempt to generate a marketable product for other industry uses (i.e., fly ash generated from the burning of coal has been incorporated into concrete mixtures for improved compressive strength).<sup>15</sup> On-site recycling is preferable because shipping hazardous waste off-site carries increased liability associated with waste handling and transport. In the literature reviewed, recycling of PRR was not found to be formerly implemented as a treatment option. This may in part be due to the lack of economic incentive, and therefore, reluctance of owners to implement a process whereby recyclable materials are separated from the PRR. The process would be complicated by the many constituents present in PRR. Chapter 5 offers a discussion on the economics of incineration versus recycling.

Treatment and/or disposal of any hazardous waste must be met with proper attention to governing environmental regulations. There are many regulations governing every aspect of waste generation, storage, and treatment. The following section will outline the process for determining if a PRR is hazardous, the waste classification, and the environmental regulations governing recycling and/or disposal.



## 2.3 *Environmental Considerations*

### 2.3.1 *Potential Hazardous Classifications of PRR*

A solid waste may be considered a hazardous waste because it is a listed hazardous waste or because it exhibits a characteristic that makes it hazardous. Listed hazardous wastes pertaining to petroleum refinery processes are as follows:<sup>16</sup>

- F037—Petroleum refinery primary oil/water/solids separation sludge—Any sludge generated from the gravitational separation of oil/water/solids during the storage or treatment of process wastewaters and oily cooling wastewaters;
- F038—Petroleum refinery secondary (emulsified) oil/water/solids separation sludge—Any sludge and/or float generated from the physical and/or chemical separation of oil/water/solids in process wastewaters and oily cooling wastewaters;
- K048—Dissolved air flotation (DAF);
- K049—Slop oil emulsion solids;
- K050—Heat exchanger cleaning sludge;
- K051—Separator sludge; and
- K052—Tank bottoms (leaded).

All of the above hazardous wastes are listed as toxic (hazard code "T").

If the waste is not listed, it still may be a hazardous waste based on the following characteristics:

1. Ignitability (D001)—a waste with a flash point lower than 60C (140°F);
2. Corrosivity (D002)—an aqueous waste with a pH less than or equal to 2.0 or greater than or equal to 12.5, or capable of corroding steel at a rate of more than 6.35 mm (0.250 inches) per year;
3. Reactivity (D003)—a waste that is explosive, reacts violently with water, or generates toxic gases when exposed to water or liquids that are moderately acidic or alkaline; and/or
4. Toxicity (D004-D043)—a waste for which the TCLP<sup>17</sup> extract contains a concentration of a specified contaminant above its regulatory threshold.

To determine whether a waste is a listed waste under the Resource Conservation and Recovery Act (RCRA) or the 1984 amendments to RCRA (the Hazardous and Solid Waste Amendments or HSWA), it is necessary to know the source of the waste. PRR that was placed in surface impoundments for disposal predate solid and hazardous waste management regulations and often the exact source is not known. If an affirmative determination cannot be made as to the exact source of the waste, then specific RCRA treatment requirements for a particular “listed” waste will not be applicable. However, RCRA requirements will be applicable if the response activity constitutes treatment, storage, or disposal. Disposal of hazardous waste, in particular, triggers a number of

significant requirements, including closure requirements and land disposal restrictions, which require treatment of wastes prior to land disposal.<sup>18</sup>

The waste materials considered in this study preexist current regulations and are likely some combination of the aforementioned listed wastes. Therefore, the waste can not be conveniently defined as one particular *listed waste* and would be classified hazardous only if it exhibits a “characteristic,” as defined in the following section.

### ***2.3.2 Overview of Applicable Regulations***

Land disposal restrictions (LDR) establish stringent standards prescribing how hazardous wastes must be treated before they can be disposed of in or on the land. Hazardous wastes have historically been assigned treatment standards, on a waste-code by waste-code basis. Under this method, it is likely that two different hazardous wastes would have two very different treatment standards for the same constituent(s). For example, consider two dissimilar materials that are considered hazardous based on varying levels of chromium. After being subjected to two dissimilar treatment methods, one waste exhibits higher leachable chromium than the other. However, both treatment methods establish the lowest “overall” concentration of leachable hazardous constituents historically achieved and are therefore selected as the best demonstrated available technology (BDAT) for each waste. The treatment standards for any other waste with the same classification then has to attain the levels achieved in the BDAT.

As of September 19, 1994, the EPA established the concept of the "universal treatment standard".<sup>19</sup> As a result, many of the treatment standards for listed wastes and certain characteristic wastes have been revised by replacing the existing limits with universal treatment standards (UTS). Depending on the physical state of a waste and how it is managed, it may have to be treated not only to address the constituent that makes the waste hazardous, but also to address any of 216 "underlying hazardous constituents." The UTS is essentially based on constituent concentrations in incinerator ash and residue. Therefore, treatment alternatives, other than incineration, able to attain UTS are extremely difficult, if not impossible.

Of particular concern to the PRR considered in this study, the U.S. Court of Appeals found it insufficient to simply remove or "deactivate" a waste characteristic (i.e., treat only the hazardous constituent in the waste) prior to land disposal. On September 25, 1992, the court ruled that wastes exhibiting the characteristics of ignitability and/or corrosivity must be treated, not only to remove the hazardous characteristic, but also to minimize the threat to human health and the environment, due to any hazardous constituents present in the waste (i.e., treat all underlying hazardous constituents to the UTS).<sup>20</sup>

As previously mentioned, prior research findings indicate the PRR not to be characteristic based on ignitability [i.e., it has a flash point greater than 60C (140°F)]. The characteristic of corrosivity is defined in Code of Federal Regulations (CFR) 40 261.22 as follows:

1. It is aqueous and has a pH less than or equal to 2 or greater than or equal to 12.5.
2. It is a liquid and corrodes steel at a rate greater than 6.35 mm (0.250 inch) per year at a test temperature of 55C (130°F).

Certain PRR samples tested had pH values less than or equal to 2, indicating possible classification as a corrosive waste (other samples tested had pH values greater than 2). The PRR was found to have a moisture content of approximately 28%. However, very little moisture was readily available (i.e., it could not be removed except through heating and evaporation).

A clarification on the definition of aqueous was requested from the RCRA Hot-Line.<sup>21</sup> A memo clarifying this definition is included in Appendix C, and states that “aqueous” implies a material that has greater than or equal to 20% free water (i.e., 20% water that is separable from other constituents by no means other than simply pouring it off). It is concluded that the PRR materials dealt with in this study does not meet the regulatory definition of an aqueous waste, and are therefore, not a characteristic waste based on corrosivity.

### ***2.3.3 EPA and the Recycling Option***

A “recycled material,” as defined by the EPA, means “a material that can be utilized in place of a raw or virgin material that consists of materials derived from post consumer waste which can be used in the manufacture of new products”.<sup>22</sup>

Recycled wastes are referred to by the EPA as “materials used in a manner that constitute disposal.” Recycled materials that are used in a manner that constitutes disposal are governed by 40 CFR 266 Subpart C. Additional regulations govern generators, operators, and transporters:

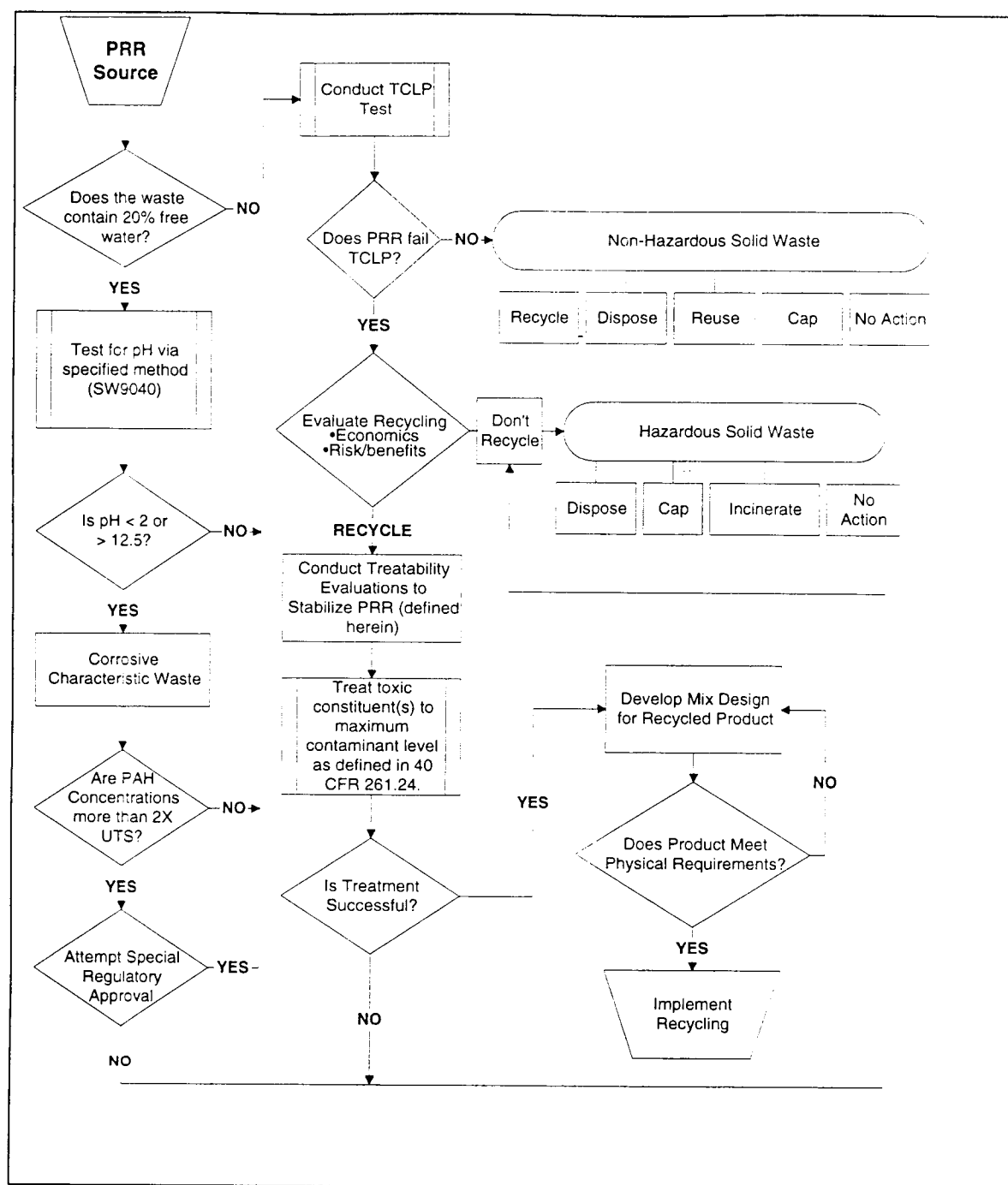
- Recyclable materials used in products produced for use by the general public must have undergone a chemical reaction to become physically inseparable and meet applicable treatment standards in 40 CFR 268 Subpart D.
- Generators and transporters of hazardous materials are subject to 40 CFR Parts 262 and 263.
- Owners and operators of facilities that store recyclable materials are regulated under applicable provisions of 40 CFR Parts 264, 265, 270, and 124.

Treatment posing as recycling is called “sham recycling,” and is subject to full regulation and permitting. Regulators use four general criteria when determining whether wastes are being recycled or whether they are treated and disposed instead:<sup>20</sup>

1. Does the end product have value? One indicator of value might be whether the product can be sold on the open market for general use, or is an effective substitute for a product sold on the open market. This criterion may be difficult to apply when the product is new and innovative, or experimental, since a market may not have been previously established.

2. Will it harm human health or the environment? This is as much a question for any treatment and disposal of a hazardous waste as it is for a recycled product. This question is often considered in the approval and regulation process of recycling operations.
3. Is the recycled material effective for the claimed use? Is it analogous to the raw material or product it replaces? Unless the material contributes significantly to the end product, the activity would not be regarded as legitimate recycling.
4. Is the waste material just “along for the ride?” or does it contribute to the end product. Legitimacy of recycling is significantly increased if the waste can be shown to provide a benefit to the recycled product.

The evaluation of pertinent regulatory information is a challenging task and may be confusing when laid out in text format. The flowchart in Figure 2-3 illustrates the process for evaluating any PRR source for its recycling potential.



**Figure 2-3 Regulatory Flowchart to Reach Recycling Status<sup>a</sup>**

<sup>a</sup>Note: This figure assumes that the PRR source is not a listed waste or a characteristic waste based on ignitability and/or reactivity.



## **2.4 *Pavement Characteristics & Structural Criteria***

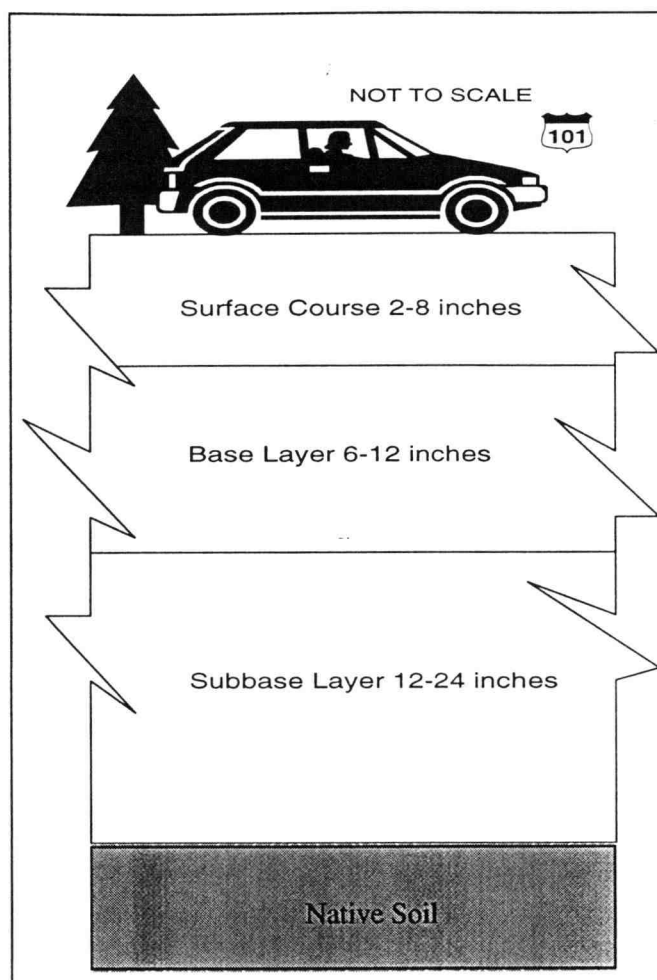
The purpose of a pavement is to provide a smooth and safe ride for a range of vehicles. The facility must be economical and remain in service for long periods of time with minimal maintenance. More than 90 percent of paved roads in the U.S. are surfaced with asphalt pavements.<sup>45</sup>

The structural or thickness design of asphalt pavements may range from a simple seal coat (sprayed asphalt binder and rock chips) to high-quality asphalt concrete. In addition to roads, they are used for airfields, parking lots, industrial sites, and even environmental applications, such as caps over waste or contaminated soils.

The thickness of material layers within a pavement are designed according to the expected traffic, location, environment, and the properties of the materials in each layer. Figure 2-4 illustrates a cross section of a typical pavement structure. Thinner pavements may be adequate for roads with low traffic volume, parking lots, and residential streets. Heavy duty pavements with thicker layers are required for high-volume highways, industrial sites, and commercial airfields.

The thickness of each layer is determined by using design procedures that take into account the expected loads, their frequency, the climate, and available materials. Engineering calculations based on layer theory are used to optimize the various possible combinations and finally select the preferred design. The result is largely based on the nature and characteristics of the materials, with emphasis placed on the surface and base courses. Each layer or course in the pavement

has a role, but the ultimate function is for the combination to transmit moving wheel loads to the native subgrade without deformation (i.e., rutting) or premature failure due to fatigue. Beginning with the lowest quality material as a subbase, each layer is progressively constructed using higher-quality materials to spread the loads. For a material to be considered as “adequate” for any layer, it must demonstrate acceptable strength parameters.



**Figure 2-4 Cross Section of Typical Highway Asphalt Pavement Structure**

### ***2.4.1 Strength Parameters Necessary to make Recycling a Viable Option***

In this study, compaction of specimens was achieved by use of the California Kneading Compactor. Parameters used as structural design criteria include diametral resilient modulus ( $M_r$ ) and Marshall Stability and flow. Table 2-1 offers an acceptable range of these parameters for different pavement layers.<sup>23</sup>

***Table 2-1 Summary of Typical Physical Parameters of Various Pavement Layers***

Strength Parameter	Asphalt Concrete Surface Course or Base	Bituminous Stabilized Mixtures	Granular Base or Subbase	Native Soils
Resilient Modulus (psi)	300,000-600,000	40,000-300,000	10,000-75,000	3,000-15,000
Stability (pounds)	750-1800	100-750	NA	NA
Flow (0.01-in)	8-16	8-16	NA	NA

NA = not applicable

### ***2.4.2 California Kneading Compactor***

The California kneading compactor was developed by the California Department of Transportation in an attempt to duplicate the kneading action that is provided by the equipment now being used for the compaction of asphalt concrete pavement.<sup>24</sup>

This method of compaction consists of a kneading ram with a surface area of approximately  $20.06 \text{ cm}^2$  ( $3.1 \text{ in}^2$ ) which is attached to a mechanical arm that applies two specified stages of loading; 1) 20 blows at 250 psi and 2) 150 blows at 500 psi, covering

approximately 25 percent of the 4 in diameter specimen with each blow. The mold in which the sample is placed is automatically rotated to systematically apply the same pressure over the entire surface area of the sample. This means of compaction was preferred over the Marshall method, due to the induced kneading action and the ability to adjust the compactive effort in the event of soft aggregate or a sensitive mixture.

### ***2.4.3 Diametral Resilient Modulus***

The repeated load diametral test [ASTM D4123-82 (1987)] is routinely used in the U.S. by state highway and other agencies. The publication of the 1986 AASHTO pavement design guide, which recommended use of resilient modulus to characterize pavement materials, has led to accelerated use of this type of test. Kennedy (1977) has reported on extensive development of this type of test at the University of Texas at Austin.<sup>22</sup> This work forms the basis for the ASTM standard for a diametral resilient modulus test. This standard, and the modifications to the method used in this study, will be discussed on further in Chapter 3.

The values of resilient modulus can be used to evaluate the relative quality of materials as well as to generate input for pavement design or pavement evaluation and analysis. The test can be used to study the effects of temperature, loading rate, rest periods, water sensitivity, etc. The procedure is non-destructive, therefore, tests can be repeated on specimens to evaluate the effects of conditioning with temperature and/or moisture.

#### **2.4.4 The Marshall Method**

The Marshall method is a complete mix design procedure, developed by Bruce Marshall of the Mississippi State Highway Department, and subsequently modified by the US Army Corps of Engineers in 1943. This method was originally intended for dense graded hot-mix design. However, research conducted at the University of Illinois developed a modified Marshall method of mix design and moisture durability testing for emulsified asphalt-aggregate cold mixtures.<sup>25</sup> This method and recommended test criteria are applicable to base course mixtures under low traffic volume conditions containing emulsified asphalt and dense-graded aggregates with a maximum top size of 25 mm (1 in.). The design is intended to simulate plant mixes prepared at ambient temperatures.

The primary testing procedures defined under this method include stability and flow of a compacted bituminous mixture. The stability is the maximum load sustained by the specimen when tested at a temperature of 60C (140°F), and the flow is the deformation (0.01 in.) at that load.<sup>26</sup> This procedure was chosen for use in determining the structural suitability and water sensitivity of PRR-modified specimens prepared in the laboratory and cored specimens retrieved from the Oklahoma Demonstration Project.

The objective of any asphalt concrete mix design is to obtain a mixture that when compacted has: 1) sufficient binder to insure a durable pavement; 2) sufficient air voids in the total compacted mix to allow for a slight amount of additional compaction under traffic loading without flushing, bleeding, and loss of stability; 3) sufficient mix stability to satisfy the demands of traffic without distortion or displacement; and 4) sufficient workability to permit efficient placement of the mix without segregation.<sup>27</sup>

### **3 EXPERIMENTAL PROCEDURES**

Sample preparation and testing varies depending on the parameter of consideration. For example, specimens evaluated for structural integrity are compacted and may be lab-cured, whereas specimens tested for environmental suitability are typically shipped in an uncompacted state. However, certain procedures are the same no matter what testing the sample undergoes. This Chapter defines materials used to construct the specimens, differences in sample preparation, and establishes certain standards by which samples are evaluated for structural characteristics and environmental soundness.

#### **3.1 Materials**

The composition of an asphalt concrete typically consists of two basic ingredients: 1) aggregate, constituting greater than 90 percent of the mixture by weight; and 2) asphalt cement. In this study, PRR was a third ingredient incorporated into the asphalt concrete. Two sources of PRR were evaluated in this study, samples from Pennsylvania and Oklahoma. From each location, multiple samples were collected, varying in physical characteristics and appearance. This Chapter will distinguish the differences between each sample of PRR acquired as well as describe the source of other ingredients used, such as, asphalt, emulsion, lime, and cement.

### *3.1.1 Petroleum Refinery Residuals*

#### *3.1.1.1 Description of Pennsylvania PRR*

Physical characteristics of PRR vary, depending on where the sample was retrieved at a particular site (i.e., relative site location and whether the sample was taken at or below the ground surface). One site near Franklin, PA was the source of two slightly dissimilar PRR. For the purpose of this study, these will be referred to as PA PRR1 and PA PRR2.

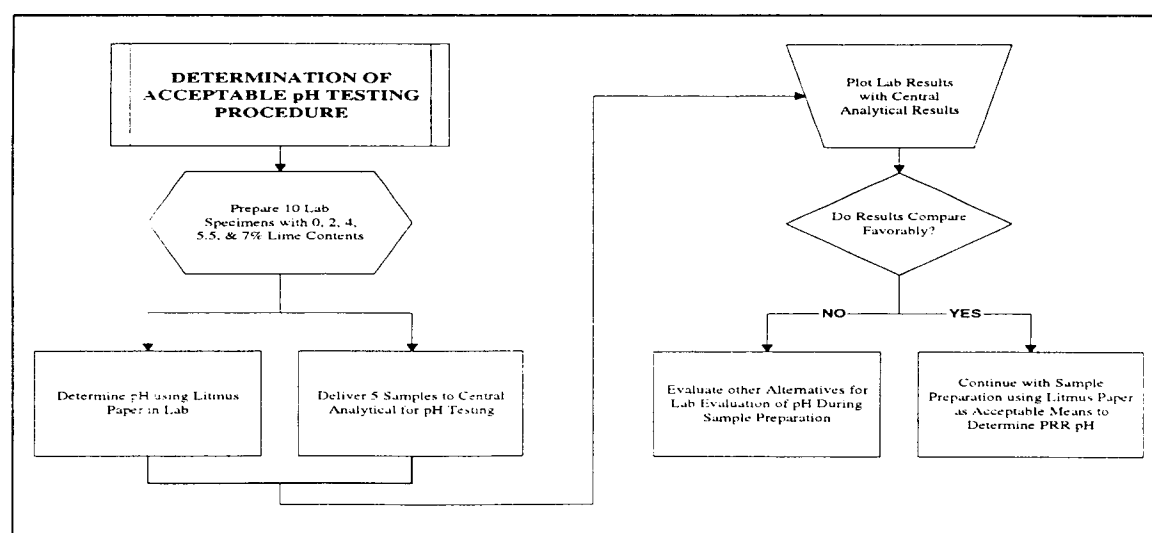
The subsurface sample, PA PRR1, has a relatively low volume of silt and clay, a high water content, and typically a lower pH than samples collected at or near the surface. PA PRR2 has a slightly higher percentage of silt and clay, a relatively low water content, and was gathered from PRR exposed at the surface. Both PRR are acidic with pH values ranging from 1.8 to 4. As discussed in Chapter 2, the acidic nature of the PRR is due to the addition of sulfuric acid, utilized for removal of impurities during the refining process.

#### *3.1.1.2 Neutralization and Stabilization of PA PRR with the Addition of Lime*

Lime was added to increase the pH of the PRR and to control lead leachability. The pH of the material is a concern for workers safety when handling and processing the PRR as well as the potential hazard to the environment. Lead, as discussed in Chapter 4, is present in quantities of approximately one percent by weight of PRR, which is of

concern considering the published regulatory maximum of 5 ppm. Lead leachability will be addressed in Chapter 5.

Determining the pH of a PRR or PRR/lime mixture was therefore necessary, prior to incorporation into a pavement structure. The sludge-like consistency of the PRR made the use of a standard pH meter impractical (pH meters require enough available free water, or solution, to allow submergence of a glass bulb indicator). Litmus paper requires a very small amount of free water to be available and is a practical means of establishing the pH of a mixture in the materials laboratory. However, knowledge pertaining to the pH of the sludge versus that of the small amount of free water available was desired. Therefore, a study was performed to compare the pH results using litmus paper in the laboratory versus the pH of the sludge, conducted by Central Analytical Laboratory, a soil testing facility located on the OSU campus. Figure 3-1 outlines the procedure followed to obtain results (plotted results are presented in Chapter 4).

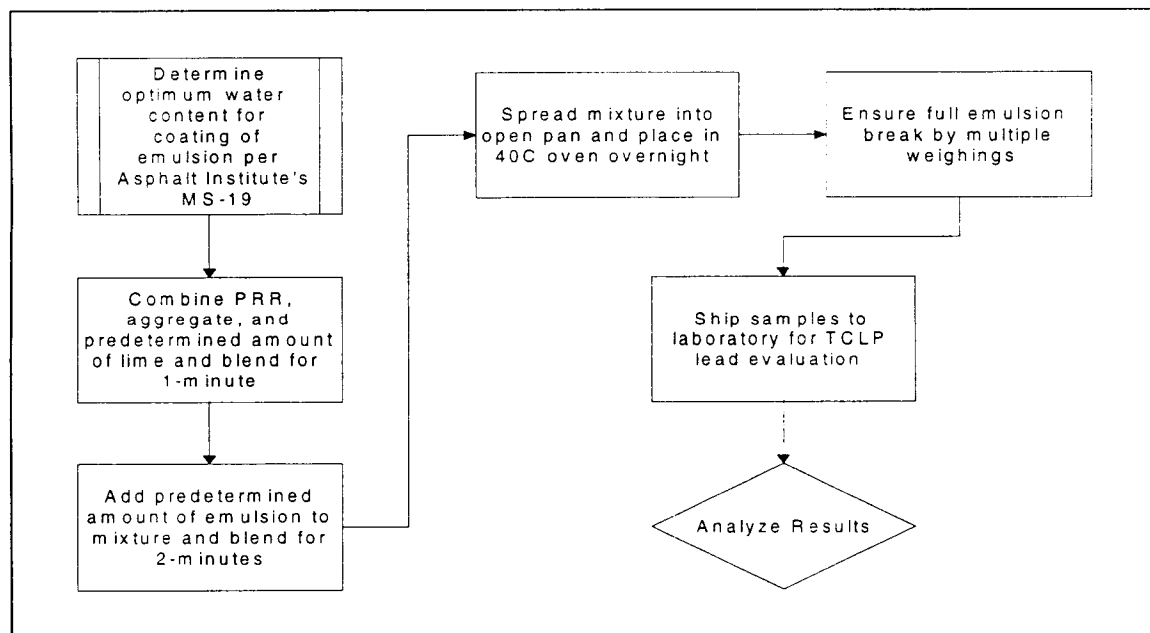


*Figure 3-1 Procedure Followed to Judge the Applicability of Litmus Testing*



It should be noted that a pH value of five was considered the minimum acceptable level in applications where the matrix would be exposed to water and the environment. A pH of five is considered the lowest tolerable limit for most aquatic organisms.<sup>28</sup> Adhering to the minimum pH would minimize the amount of lime, therefore, minimize the cost of treating the PRR.

When stabilization of the lead was the desired result, lime and emulsion were combined in varying proportions to establish the combination that would yield the lowest TCLP lead results (see Section 3.2). Figure 3-2 illustrates the mixing procedure used for evaluating the effect that lime and emulsion combined would have on lead leachability.



*Figure 3-2 Mixing Procedure for Typical Specimens Tested for TCLP Lead*

**3.1.1.3 Neutralization and Stabilization of PA PRR with the Addition of Portland Cement**

Prior to incorporating PA PRR into an asphalt concrete, it was necessary to stabilize the lead to below the regulatory TCLP maximum of 5 ppm. Lime and portland cement both contain caustic pozzolanic constituents that can react to render lead less soluble. Portland cement was mixed with PA PRR in varying quantities, as indicated in Table 3-1, to evaluate its effectiveness in reducing leachable lead.

***Table 3-1 Samples Prepared using Portland Cement***

Sample No.	% Cement by dry weight of PA PRR	% Water by weight of dry PA PRR
1	0	0
2	5	0
3	10	0
4	10	10
5	10	20
6	15	0

Mixing water was added to facilitate hydration of the cement. Samples were prepared then sealed in zip-lock bags for a 7-day curing period prior to being tested for TCLP lead.

#### 3.1.1.4 Oklahoma Refinery Residuals

This source of PRR is acidic with a significant amount of sulfur-based compounds, but contains a minimal amount of lead.<sup>4</sup> This PRR source has been processed and landfilled at a Superfund site near Tulsa, Oklahoma. Processing of the PRR at this facility involved three stages, and thus, three products could be studied for potential use in roadbed structures. The following products are matched with the corresponding process and acronym with which they will be referred throughout this report:

1. Raw refinery residuals (raw OK PRR)—No processing, materials used as excavated;
2. Neutralized refinery residuals (NRR)—Calcium hydroxide added to allow safe handling of raw material (i.e.,  $\text{pH} \cong 5$ ); and
3. Treated refinery residuals (TRR)—NRR that has been further processed by adding quicklime in an enclosed processing unit to stabilize material prior to landfilling.

NRR had been previously treated with calcium hydroxide [ $\text{Ca}(\text{OH})_2$ ] to render them less acidic and more stable (i.e., less likely to flow under their own weight). In addition to hydrated lime, TRR incorporated considerable amounts of quick lime ( $\text{CaO}$ ), for two reasons: 1) to facilitate the “breaking-up” of the NRR globules, yielding a more “soil-like” homogeneous mixture; and 2) to drive off excess moisture. The resulting TRR is a residual-lime mixture with a pH greater than 11. Evaluation of the structural integrity

of Oklahoma residuals, when incorporated into a roadbed structure, predominantly utilized TRR. However, limited testing was also performed on samples incorporating NRR.

### *3.1.2 Other Materials*

#### 3.1.2.1 Lime

Three lime types were used during this study. These included: 1) quick lime ( $\text{CaO}$ ); 2) calcium hydroxide [ $\text{Ca}(\text{OH})_2$ ]; and 3) calcium carbonate ( $\text{CaCO}_3$ ).

Quick lime essentially becomes  $\text{Ca}(\text{OH})_2$  upon hydration with water. One advantage of using quick lime is the heat generated after water is added. For example, if the PRR source has excessive moisture (i.e., more than necessary to hydrate the lime), the heat liberated from the reaction of quick lime with water will help evaporate some of this moisture. However, the disadvantage of using quick lime is its capacity to burn exposed skin. Care must be taken when using this caustic source. Calcium hydroxide [ $\text{Ca}(\text{OH})_2$ ] has one water molecule ( $\text{H}_2\text{O}$ ) attached to each  $\text{CaO}$  molecule, therefore, it does not have nearly the affinity for water as  $\text{CaO}$  and poses less of a health hazard than  $\text{CaO}$ . Though  $\text{Ca}(\text{OH})_2$  will typically have a drying effect on any material it comes into contact, it will not generate as much heat as  $\text{CaO}$ . Calcium carbonate ( $\text{CaCO}_3$ ), otherwise known as agricultural lime, is a more inert form of lime that does not react as readily with water as either  $\text{CaO}$  or  $\text{Ca}(\text{OH})_2$ .

The most commonly used lime source for laboratory purposes is  $\text{Ca}(\text{OH})_2$ , due to its availability, affordability, and relatively low health hazard. The TRR were received in a “ready-to-mix” form (i.e., they had already been fully processed with lime prior to acquisition).

#### 3.1.2.2 Asphalt Binders

Asphalt cement; grade AC-20; was utilized for hot-mixing. The “20” of AC-20 indicates the viscosity in hundreds of poises at 60C (140°F). AC-20 has been adopted by the American Association of State Highway and Transportation Officials (AASHTO) as a standard paving grade asphalt cement.<sup>29</sup>

Two cationic emulsions were utilized for this study. Table 3-2 shows additional information pertaining to the emulsified asphalts used in this study. These are: 1) Chevron CSS-1 containing 62.5% residual asphalt with a penetration of 153 tenths of a millimeter at 25C (77°F); 2) Koch CSS-1hP (polymer modified) containing 62.1% residual asphalt and a penetration of 96 tenths of a millimeter at 25C (77°F).

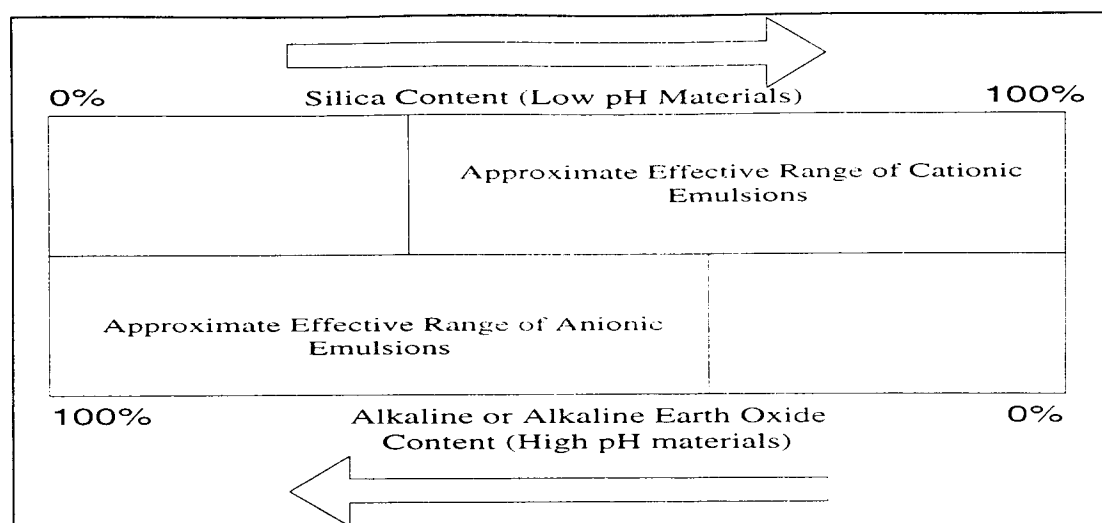
*Table 3-2 Data on Emulsions Used*

Emulsion Type	Chevron CSS-1	Koch CSS-1hP
<b>Property</b>		
Viscosity @ 77°F (saybolt furol)	30.2	28
Residue by Distillation (wt. %)	62.5	62.1
<b>Test on Residue from Distillation @ °F:</b>	N/A	400
Penetration @ 77°F	153	96
R&B Softening Point (°F)	N/A	129
Force Ductility 39.2°F		
Initial Peak	N/A	48.4
Peak Ratio	N/A	0.58
Test Date:	6/21/94	9/26/94

N/A = not available

Cationic emulsions were initially chosen, due to the expected low pH of the PRR to be incorporated into the mixtures. Figure 3-3 illustrates the effective range of cationic and anionic emulsions with positive and negative charges, respectively.<sup>30</sup>

The pH of the TRR is quite high (i.e., pH > 11) because of the presence of lime. Consequently, anionic emulsions may have been more appropriate for this material. The lime treated PRR particles are likely cationic, due to the high lime content, and may not readily undergo coating by a cationic emulsion. This phenomena may introduce inner-particular repulsion forces that influence bonding between the emulsion and PRR.



*Figure 3-3 Conceptual Effective Range of Cationic and Anionic Emulsions*

### 3.1.2.3 Aggregates

Not all specimens contained aggregates (i.e., Oklahoma TRR were mixed with emulsion and compacted without the addition of aggregates). However, for certain mixtures, aggregates were obtained from Pennsylvania or Oklahoma for mix designs pertinent to those particular states. Standard Specifications from both states were referred to for grading limits.<sup>31, 32</sup>

Pennsylvania aggregates were received in four bin classifications: 1"-1/2", 1/2"-3/8", 3/8"-1/4", and 1/4" minus. These aggregates are typical for the region and are essentially a soft, crushed limestone. It was necessary to perform blending calculations to fit the desired gradation (i.e., dense or open graded) after the true gradation was established via ASTM designation C 117-87, Materials Finer than 75-mm (No. 200) Sieve in Mineral Aggregates by Washing.<sup>33</sup> Oklahoma aggregates were received as a

blended bulk sample with 1/2" top size. Again, these aggregates are a crushed limestone. Calculations and subsequent plots will be addressed further in Chapter 4.

### ***3.2 Specimen Preparation and Testing Procedure***

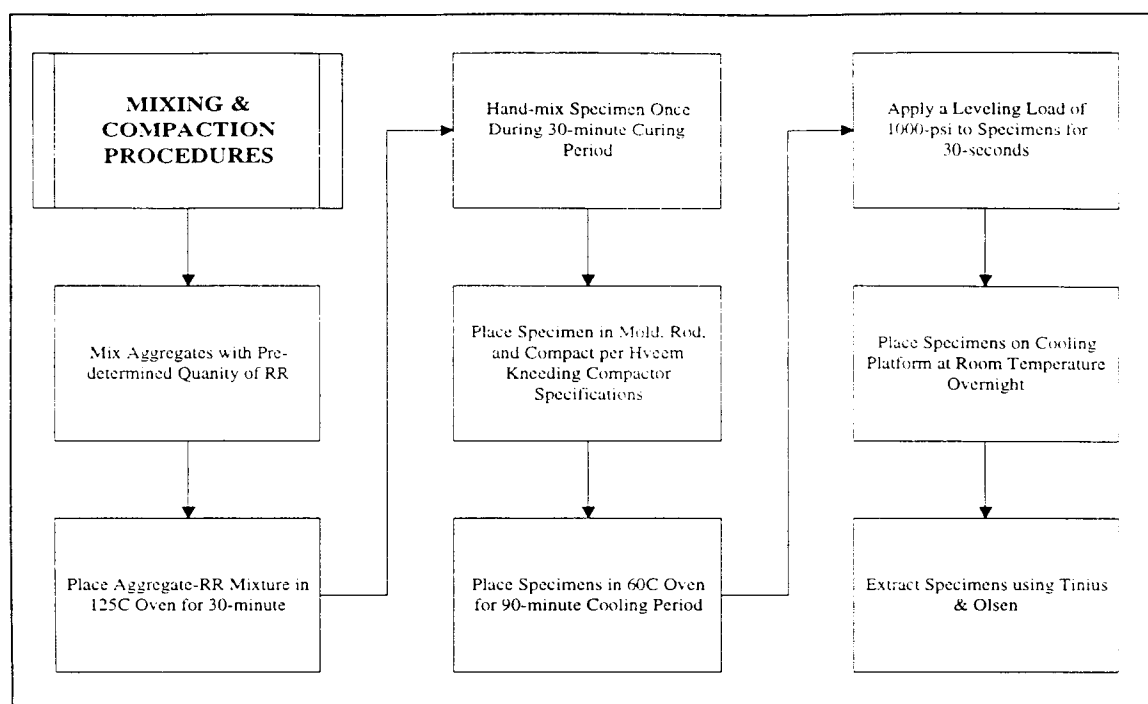
#### ***3.2.1 Structural***

##### ***3.2.1.1 Specimen Preparation for Typical Hot-Mixed Asphalt***

Proportioning of aggregates was based on a sieve analysis and estimated gradation of the PRR. After the desired gradation was achieved, aggregates were batched in appropriate quantities to achieve specimens of approximately 2.5 inches in height and placed in 125C oven overnight.

PA PRR, mixed with predetermined amounts of a hydrated lime  $[\text{Ca}(\text{OH})_2]$ , was placed in an oven at  $105\pm 5^\circ\text{C}$  ( $221\pm 9^\circ\text{F}$ ), until melting occurred. Upon melting, a sufficient quantity of PA PRR was transferred to a counter-top hot-plate for convenience during mixing. The aggregates were then placed in the preheated mixing bowl and weighed prior to the addition of the PA PRR. Predetermined quantities of PA PRR (by weight of dry sample) were added followed by two to three minutes of mechanical mixing. The samples were removed from the mixing bowl, spread evenly into an open pan, and then placed in a  $125\pm 5^\circ\text{C}$  oven for a 30-minute curing period. The samples were hand-mixed midway through the oven curing period. Figure 3-4 outlines this process.





**Figure 3-4 Mixing and Compaction Procedures for Hot-Mixed Specimens**

Upon retrieval of specimens from the oven, compaction per standard California Kneading Compactor Specifications<sup>34</sup> took place. Stage-1 compaction consisted of 20 blows at 150 psi, followed by Stage-2 compaction of 150 blows at 400 psi. Compaction pressures varied from that recommended by the procedure for two reasons: 1) The addition of PRR made the mixtures quite soft under loading imposed by that recommended for stage-1 compaction. For example, the plunger would penetrate into the mixture and result in excessive movement with little compaction occurring. Therefore, stage-1 compaction pressure was reduced from 250 psi to 150 psi; and 2) Under the initial pressure of 500 psi for stage-2 compaction, excessive movement of some mixtures were observed (i.e., considerable displacement and very little compaction took place), as well

as aggregate degradation. As previously mentioned, the PA aggregate was a soft limestone, and therefore, did not withstand the high loading pressure. Consequently, stage-2 compaction was reduced from the recommended 500 psi to 400 psi to diminish movement of the mixture and aggregate. Each specimen was then placed in a 60C (140°F) oven for a 1½ hour cooling period, after which a static load of 1,000 psi was applied by the “double-plunger” method. Following an overnight cooling period, at room temperature [approximately 24C (75°F)], the specimens were extracted from their molds.

#### 3.2.1.2 Specimen Preparation for Cold-Mixed Asphalt Utilizing Aggregates

Two methods were used in preparing cold-mix specimens. This was due to the two distinct types of PRR obtained for this study. As discussed in Sections 3.1.1 and 3.1.2, the NRR, TRR, and PA PRR were all utilized for use in cold mix specimens. This section describes the method used when incorporating aggregates into the mixture. NRR and neutralized PA PRR were the only residuals used in this manner.

The first step involves attaining an ample supply of neutralized PRR (approximately 45 pounds of each, in this study). The NRR received from Oklahoma were already neutralized, as described in section 3.1.2., and therefore, required no further treatment in the laboratory prior to incorporating into a cold-mixed AC.

Generally, following the protocol developed by the research committee<sup>a</sup> (CH2M Hill, Oregon State University, and Terrel Research Inc.) for the use of PRR in cold-mixed asphalt concrete, the following mixing procedure was used: 1) place pre-weighed aggregate in mixing bowl; 2) add appropriate percentage of "coating-water," based on the previously described procedure, and mix two minutes; 3) add predetermined percentage of NRR and mix two minutes; 4) add predetermined percentage of emulsion and mix for two minutes; 5) place mixture in open pan until emulsion breaks; 6) compact specimen; 7) place compacted specimen and mold in 60C oven for a 1 1/2 hour curing period; and 8) extract specimen.

### 3.2.1.3 Specimen Preparation for Cold-Mixed Asphalt Excluding Aggregates

Specimens prepared utilizing TRR without aggregates involved the simplest process. Essentially, this was a five-step process including: 1) a predetermined amount of TRR are place in mixing bowl; 2) the predetermined percentage of emulsion is then added; 3) the mixture is then blended for approximately two minutes; 4) the specimen is compacted; and 5) extracted. The curing period prior to extraction was not necessary when 100% TRR was used, due to the high percentage of lime present and the fine gradation of the TRR, these mixtures had relatively fast emulsion breaks. Hydrated lime accelerates the breaking of the emulsion,<sup>25</sup> and therefore, made it possible to extract the specimens immediately after compaction.

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<sup>a</sup> This protocol was, for the most part, extracted from a combination of existing cold mix procedures defined in Asphalt Institute's Manual Series No. 19.

#### 3.2.1.4 Cold-Mix Curing Procedures

A variety of methods<sup>25</sup> exist for the curing of emulsion-based AC mixtures. Two methods were used for curing cold-mixed specimens in this study. The first is the Chevron Method<sup>35</sup> which involves retaining the specimens in their molds for a 72-hr initial cure after compaction followed by extraction and placement of the samples into a desiccator under an absolute vacuum of 740-750 mm Hg for a period of four days. The second method<sup>36</sup> involved extraction of the specimens after a 72-hr initial cure in the mold, followed by 12 days of bench curing, and then completed with four days in the vacuum desiccator (740-750 mm Hg). The purpose of the vacuum desiccator is to simulate long-term curing. One day of curing in a vacuum desiccator is believed to simulate approximately four days of bench curing.<sup>37</sup> The second method was introduced, due to the fact that the first method was not allowing the specimens to satisfactorily cure. The moisture in the PRR is believed responsible for the increased cure-time of PRR-modified mixtures.

#### 3.2.1.5 Testing Procedures

##### **3.2.1.5.1 Bulk Specific Gravity**

The standard test method used for evaluating the bulk specific gravity was the “Determination of the Bulk Specific Gravity of Compacted Bituminous Mixtures using Paraffin-Coated Specimens” (AASHTO Designation T 275-89).<sup>23</sup> This method should be used on specimens that contain open or interconnected voids and/or absorb more than 2

percent water by volume. The only variation to this standard was the use of parafilm rather than paraffin for encasing of the specimens prior to weighing while submerged in water.

#### **3.2.1.5.2 Theoretical Maximum Specific Gravity**

AASHTO Designation T 209-90, Maximum Specific Gravity (Gmm) of Bituminous Paving Mixtures, was used to determine the theoretical maximum specific gravity (Gmm). One alteration of this Method was made to accommodate the TRR used in this study. In order to accurately establish the Gmm of TRR samples, soaking of the specimen for up to 24 hours prior to testing was necessary. Initially, the TRR material would “float” on the water surface. However, after soaking for a sufficient amount of time, the sample particulates would absorb water, sink, and become suitable for testing via this method. The “water-resistant” characteristic of samples containing 100% TRR is not well understood. However, it is likely associated with the hydrophobic nature of most petroleum-base materials.

#### **3.2.1.5.3 Diametral Resilient Modulus**

The indirect tension test for resilient modulus ( $M_r$ ) of bituminous mixtures (ASTM Designation: D 4123-82) can be used to evaluate the relative quality of materials as well as to generate input for pavement design or evaluation and analysis.<sup>23</sup> This is a

nondestructive test that can be repeated on a specimen to evaluate moisture or temperature conditioning.

The method recommends testing for the  $M_r$  over a range of temperatures. However, for the purpose of this study,  $M_r$  results at room temperature (25°C or 77°F) proved to be sufficient for comparisons among samples prepared.

The test is performed by using a 0.1-s pulse load applied every three seconds across the diameter of the test specimen. The horizontal deflection is measured by a pair of transducers mounted in a yoke that is clamped to the specimen. The  $M_r$  is calculated as follows:

$$M_r = P(v_{RT} + 0.27)/(tDH_T)$$

Where:

$M_r$  = total resilient modulus of elasticity, psi or MPa,

$P$  = dynamic load (lbf),

$v_{RT}$  = total resilient Poisson's ratio (0.35 has been found to be reasonable for asphalt mixtures),

$t$  = thickness of specimen, inches or mm, and

$DH_T$  = total recoverable horizontal deformation, inches or mm.

As noted above, a Poisson's ratio (P-ratio) of 0.35 has become the standard when dealing with asphalt concrete mixtures. This was the ratio used for PRR-modified mixtures as well. However, would the same ratio be applicable to materials incorporating PRR? The definition of the P-ratio is "the ratio of lateral strain to longitudinal strain in a body under tensile or compressive stress."<sup>38</sup> The P-ratio could be calculated for a PRR

source by applying the resilient modulus test to a specimen made from 100% PRR.

Although this procedure is not practical for the material at hand, it is desirable to know where the ratio lies, with respect to asphalt concrete. Table 3-3 offers typical values known for a variety of materials.<sup>39</sup>

**Table 3-3 Poisson Ratios for Different Materials**

Material	Range	Typical Value
Portland cement concrete	0.15-0.20	0.15
Lime-flyash mixtures	0.10-0.15	0.15
Cement-treated granular materials	0.10-0.20	0.15
Lime-stabilized materials	0.10-0.25	0.20
Cement-treated fine-grained soils	0.15-0.35	0.25
Loose sand or silty sand	0.20-0.40	0.30
Hot mix asphalt	0.30-0.40	0.35
Untreated granular materials	0.30-0.40	0.35
Dense sand	0.30-0.45	0.35
Fine-grained soils	0.30-0.50	0.40
Saturated soft clays	0.40-0.50	0.45

Considering the volume of insoluble material in the raw PA PRR source (40.5% as described in Section 2.1) and the consistency of the remaining oil fraction, it is reasonable to predict a P-ratio greater than that of a typical hot mix asphalt concrete (i.e.,  $> 0.35$ —note trends in Table 3-3). However, a lime-stabilized material is shown to have a typical P-ratio of 0.20. This would suggest that as the lime content increases the P-ratio decreases. Therefore, it is reasonable to assume that PRR-mixtures with higher lime

contents will have lower P-ratios than the raw PRR. The P-value of a typical PRR likely lies between 0.4 and 0.5, due to the presence of Fuller's Earth (fine-grained soil) and petroleum constituents similar to that of asphalt cement (P-value = 0.50).<sup>40</sup> The difference in P-ratio between a raw and lime-modified PRR material may significantly influence the design of a pavement structure utilizing PRR. However, the scope of this study did not include evaluation of this parameter.

#### **3.2.1.5.4 Marshall Stability and Flow**

The Marshall stability is said to be the maximum load carried by a specimen while being loaded in a special restraining device. The corresponding flow is the amount of deformation sustained by the specimen at the maximum load, typically reported in 0.01-in.

Some samples were subjected to the specified 60C (140°F) water bath or placed in a zip-locked bag and immersed in a water bath for 30-minutes prior to testing for stability and flow. Other samples were placed in a 60C air bath.<sup>41</sup> This variation in the testing procedure allowed for an evaluation of the water sensitivity of the mixtures, as will be discussed in Chapter 4.

#### **3.2.1.5.5 Water Sensitivity**

The Oregon State Highway Division (OSHD) Standard for Water Sensitivity (Appendix D) is intended to predict the loss of resilient modulus of dense-graded asphalt



concrete when subjected to field conditions of water saturation and freezing. The procedure includes the following steps:

1. Test for "pre-conditioned" resilient modulus.
2. Place in 77°F (ambient) water bath under a partial vacuum (1.2 inches Hg absolute pressure) for not less than 30 minutes,
3. Remove from water bath and immediately rap in parafilm then place in a freezer at  $0\pm 8^{\circ}\text{F}$  for not less than 15 hours.
4. Remove from freezer and immediately place in 140°F water bath for 24 hours,
5. Remove from hot-water bath and place in ambient water bath for 3-6 hours, and
6. Remove from ambient bath and immediately test for "conditioned" modulus.

The ratio of the conditioned modulus to the preconditioned modulus times 100 percent is the index of retained modulus (IRM). An IRM of 70 percent or greater is considered as "passing" the test.

Water sensitivity of a roadbed structure incorporating PRR is of concern. If excessive weakening of the structural matrix results when exposed to water, other layers of the roadbed structure may deform and cause premature failure.

### 3.2.1.5.6 Environmental Conditioning System

The environmental conditioning system (ECS) evaluates the water sensitivity or stripping characteristics of compacted asphalt concrete mixtures under simulated warm and cold climatic conditions. The water sensitivity characteristics of the compacted mixtures are determined based upon measurements of percent stripping, the ECS modulus, and the coefficients of permeability for air and water flow. These characteristics may then be used to determine a mixtures suitability for use as a paving material.<sup>42</sup>

For the purposes of this study, the ECS was intended to primarily function as a water collection device to evaluate the leachate potential of a given mixture via the TCLP. Two specimens were tested: one dense-graded and one gap-graded mixture. Both specimens were produced at optimum PRR and AC contents, as defined in Chapter 4.

The gap-graded specimen experienced a static load of 20-pounds with a repeated load of 200-pounds for three hot cycles (60C) and without repeated loading for one cold cycle (-18C). Each cycle had a duration of four hours with a constant vacuum of 10-inches of mercury (Hg) to draw water through the specimen. However, due to PRR migration and potential other causes (as discussed in Chapter 5) no water was retrieved. Leachate water was therefore obtained for environmental testing via a vacuum-wash method. This method involved breaking apart previously compacted specimens and subjecting them to a vacuum of 29-inches Hg for one-hour. Water obtained using this method was yellowish in color at completion of the vacuum wash.

ECS testing of the dense graded specimen included an initial vacuum cycle. This involved placing the sample in a 20-inch Hg vacuum for 6-hours followed by an 18-hour vacuum at 5-inches Hg prior to beginning the loading cycle. The vacuum cycle was performed in an attempt to draw water through the sample for testing purposes, prior to inducing deformation of the sample through loading. As previously discussed, no water was retrieved. However, standard test procedures commenced regardless, in order to observe structural characteristics of specimens under the ECS simulated environment. It should be mentioned that due to excessive deformation of testing at elevated temperatures and expected applications of the material as a stabilized base (i.e., maintained at moderate temperatures not exceeding 25C), the temperature was held constant at 25C throughout the four loading cycles for the dense graded specimen only.

### *3.2.2 Environmental*

Environmental evaluation of PRR materials involved two primary methods of testing. These are: 1) whole sample analysis and 2) the toxicity characteristic leachate procedure (TCLP).

#### 3.2.2.1 Whole Sample Analysis

A whole sample analysis was performed by CH<sub>2</sub>M Hill's analytical laboratories. The two methods used to evaluate metals and organic semivolatile components were the

inductively coupled plasma (ICP) method and the gas chromatography (GC) method, respectively.

In the ICP method, argon gas is exposed to a strong magnetic field that is produced using a radio-frequency generator.<sup>43</sup> By initiating ionization of the argon gas molecules with a spark and then subjecting the gas stream to the magnetic field, heating results because of the collisions and resistance to movement of the ionized particles. Temperatures from 4000 to 8000 K can be achieved; sufficient to almost completely dissociate molecules so that little interference between them results, and atomic emission becomes highly efficient. Each metal element exhibits a unique wavelength signature that can be identified by a monochromator or polychromator.

Gas chromatography entails the vaporization of a liquid sample followed by the separation of the various gaseous components formed.<sup>44</sup> In this procedure, a small sample is flash-evaporated to convert its components into a gaseous state. Gases travel through the column at different rates so that they emerge from the column at different times. Detection is made by either physical or chemical means. Each constituent is then represented on a recording device, at the appropriate time, characteristic to that compound. These components can then be individually identified and quantitatively measured.

Whole sample analyses were run on representative samples of raw (i.e., nontreated samples collected from the parent source) PRR to establish the quantitative level of hazardous constituents that each source contained.

### 3.2.2.2 Toxicity Characteristic Leachate Procedure

The toxicity characteristic leachate procedure (TCLP) is designed to determine the mobility of both organic and inorganic contaminants present in liquid, solid, and multiphasic wastes. This procedure is intended to simulate a landfill environment where slightly acidic conditions prevail. If a total sample analysis of a waste demonstrates that individual contaminants are not present or below appropriate regulatory thresholds, the TCLP need not be run.

For solid wastes, the procedure involves adding a slightly acidic ( $\text{pH} \cong 5$ ) extraction liquid at 20 times the weight of the solid phase. This solution is then placed and rotated in a zero head-space extraction vessel for 18 hours. The liquid phase is then separated from the solid phase and analyzed for organic constituents by the gas chromatograph/mass spectrometer (GC/MS) method and metals by atomic absorption.<sup>44</sup> It is worth noting that the maximum concentration that the TCLP could render would be one-twentieth of the total concentration of the compound or element tested for. For example, if the whole sample analysis revealed a total lead concentration of 80 ppm and the lead in the mixture was 100% soluble, then the maximum theoretical concentration one might expect to see in the TCLP results would be  $80/20$ , or 4 ppm.

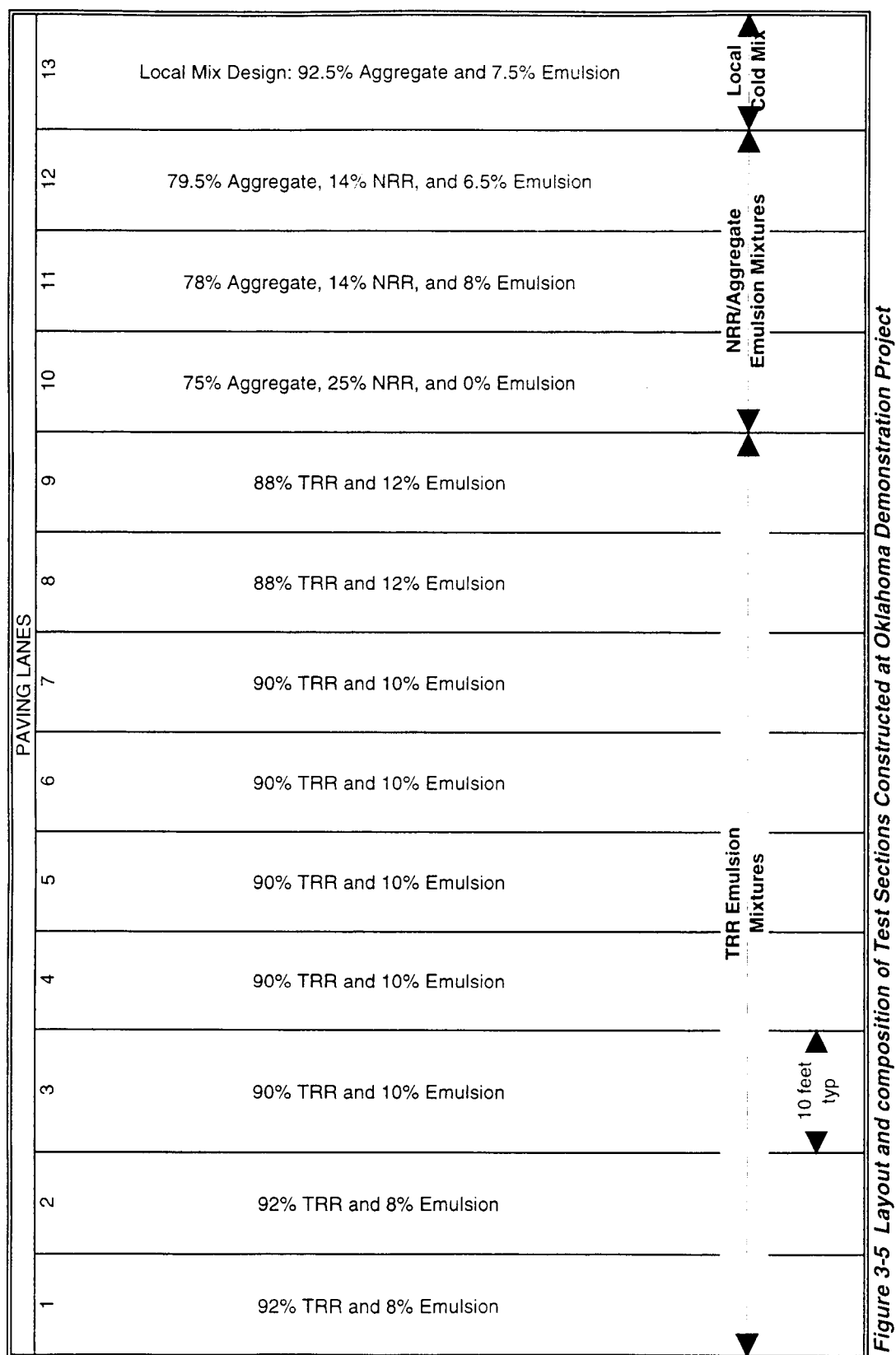
## **3.3 Demonstration Project**

A technology project was undertaken by ARCO Inc. utilizing CH<sub>2</sub>M Hill, Oregon State University, and Terrel Research to develop a recycling application to be

implemented at a site in Sand Springs, Oklahoma. This site was an EPA Superfund site near Tulsa, prior to its closure in June, 1995. The PRR at this site was treated with hydrated lime and a quick-lime slurry in order to stabilize the waste, neutralize the acidity, and reduce  $\text{SO}_2$  gas. After treatment, the waste was placed in a dedicated landfill.

Because of the available treated material, adequate space, existing contractor and equipment availability, this site was selected for a demonstration of the residual recycling technology developed in this study. The objective of this project was to demonstrate the application of reusing heavy refinery residuals to produce a material that can be used in a pavement structure by using conventional construction equipment and procedures.

The purpose of full-scale test sections is to scale up from laboratory testing equipment to typical construction equipment. A total of 13 test strips were placed, each receiving a slightly different mix design and/or treatment. The composition and layout of each lane is illustrated in Figure 3-5. For three months following construction, the pavement was sampled, tested, and evaluated for comparisons with previously determined laboratory results. The demonstration project is intended to help determine the suitability of this type of treated waste for construction and establish guidelines for future full-scale projects.



## **4 RESULTS**

### **4.1 Physical Testing**

Results of the physical and environmental tests will be addressed separately for the two sources of materials acquired from Pennsylvania and Oklahoma. Physical testing entails material gradation and structural parameters (i.e., resilient modulus, Marshall stability and flow, etc.) for hot and cold mix designs. Tests for environmental suitability include lead leachability, whole sample analysis, environmental conditioning system, and water sensitivity. Tables for Pennsylvania and Oklahoma physical and environmental data are presented in Appendices A and B, respectively.

#### **4.1.1 Pennsylvania**

##### **4.1.1.1 PRR Gradation**

The gradation of the PRR was not determined. However, it could have been accomplished by first performing an extraction to separate the organic constituents from the soil followed by drying and sieving of the soil fraction (a method similar to AASHTO Designation: T 170-90). Due to the likely use and subsequent disposal of Attapulgas clay with the PRR, the soil component likely contains predominantly fine silts and clays that would pass a No. 200 (0.074 mm) sieve. It was therefore assumed that any soil contained



in the PRR would pass the No. 200 (0.074 mm) sieve and would replace the finest fraction of any typical asphalt mix gradation.

#### 4.1.1.2 Aggregate Gradation

Wet sieve results of the Pennsylvania aggregates were determined and used to blend to the desired gradations. The fine particulate in the PRR (i.e., silts, clays, and ash) were not included in the blending calculation of the mixtures, due to the varying amounts of PRR to be considered. The scope of this study included evaluating the consequences that varying quantities of PRR would have on the structural integrity of the dense and open gradations (see Appendix A, Tables A1 and A2). To account for the silts and clays incorporated in the PRR would require a different gradation for each PRR content. This would result in a comparison of different mix designs, making inferenced comparisons between mixtures difficult.

Blended mixtures are compared to standard Pennsylvania DOT specifications for dense and open gradations in Figures 4-1 and 4-2, respectively. Figure 4-1 illustrates the blended gradation with the lower and upper limits of a Pennsylvania DOT's dense graded specification. The upper range of the gradation falls below the lower limits. This indicates that the chosen gradation contains a slightly lower percentage of material retained above the #4 sieve than the specifications allow.

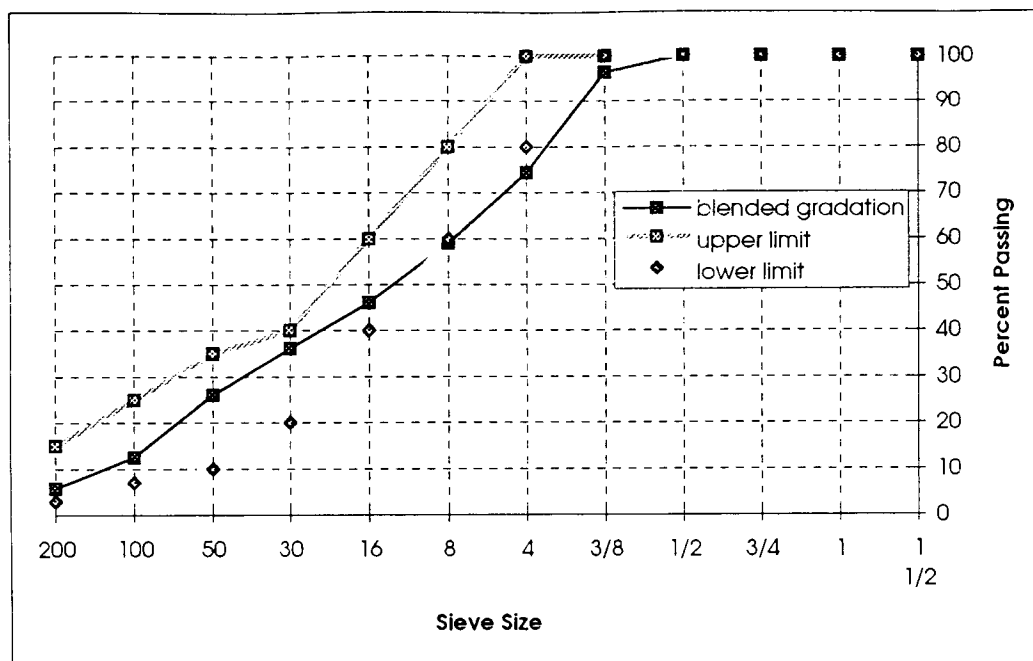


Figure 4-1 Dense-Graded Blend Plotted with PA DOT Specification Limits

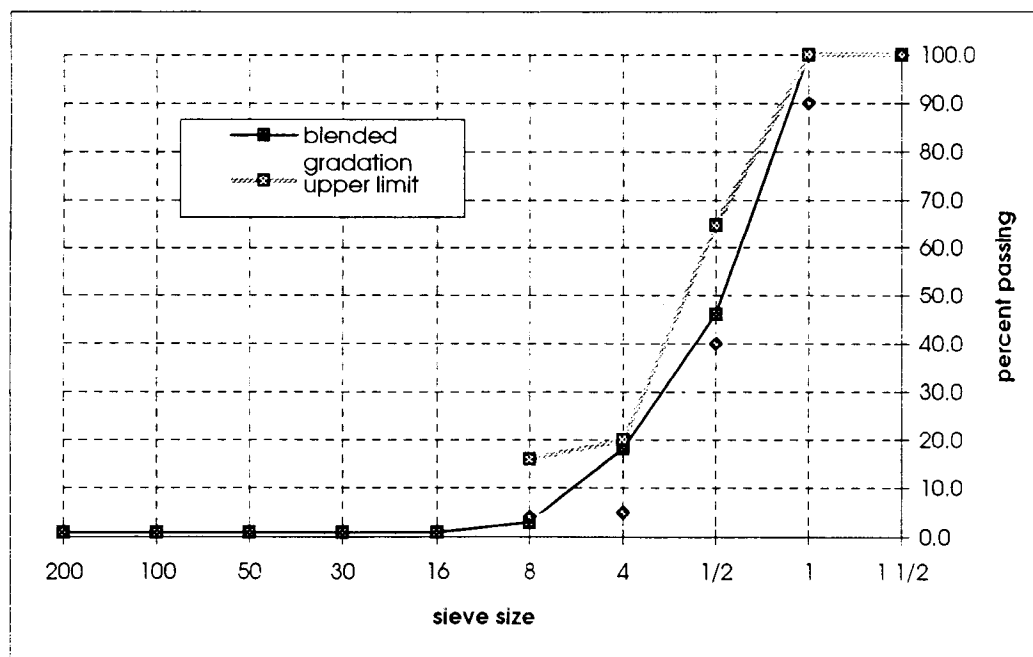


Figure 4-2 Open-Graded Blend Plotted with PA DOT Specification Bands

The open gradation, plotted with upper and lower specifications in Figure 4-2 lacks material smaller than the #4 sieve. Less fine material allows more room, and thus, a higher percentage of PRR to be incorporated into the mix. The fine fraction contained in the PRR is expected to bring the gradation closer to the specified limits.

#### 4.1.1.3 Hot-Mix Results

Observations made while heating of the PRR are as follows:

- PA PRR2 experienced a bulking phase where it behaved as a thick “dough-like” material. Adhesion of the material to itself and the metal mixing bowl, in which it was placed, was minimal during this phase;
- PA PRR1 melted and became more workable sooner than did PA PRR2; and
- Both PA PRR1 and PA PRR2 turned a brownish color during initial heating and became black upon complete melting.
- Heating of the PRR increased odor emissions to a point where a breathing apparatus was necessary.

The procedure to determine optimum PRR and AC contents utilized Marshall testing protocol with the addition of the resilient modulus as an indicator. As described in Chapter 3, the optimum PRR content was determined for both dense and open-graded mixtures. However, optimizing the AC content was performed only on dense-graded mixtures at the optimum PRR content.

Determination of optimum PRR contents for dense and open-graded mixtures is based on the set of plots shown in Figures 4-3 a-e and 4-4 a-e. The optimum AC content for the PRR-optimized dense-graded mixture was determined via Figures 4-5 a-e.

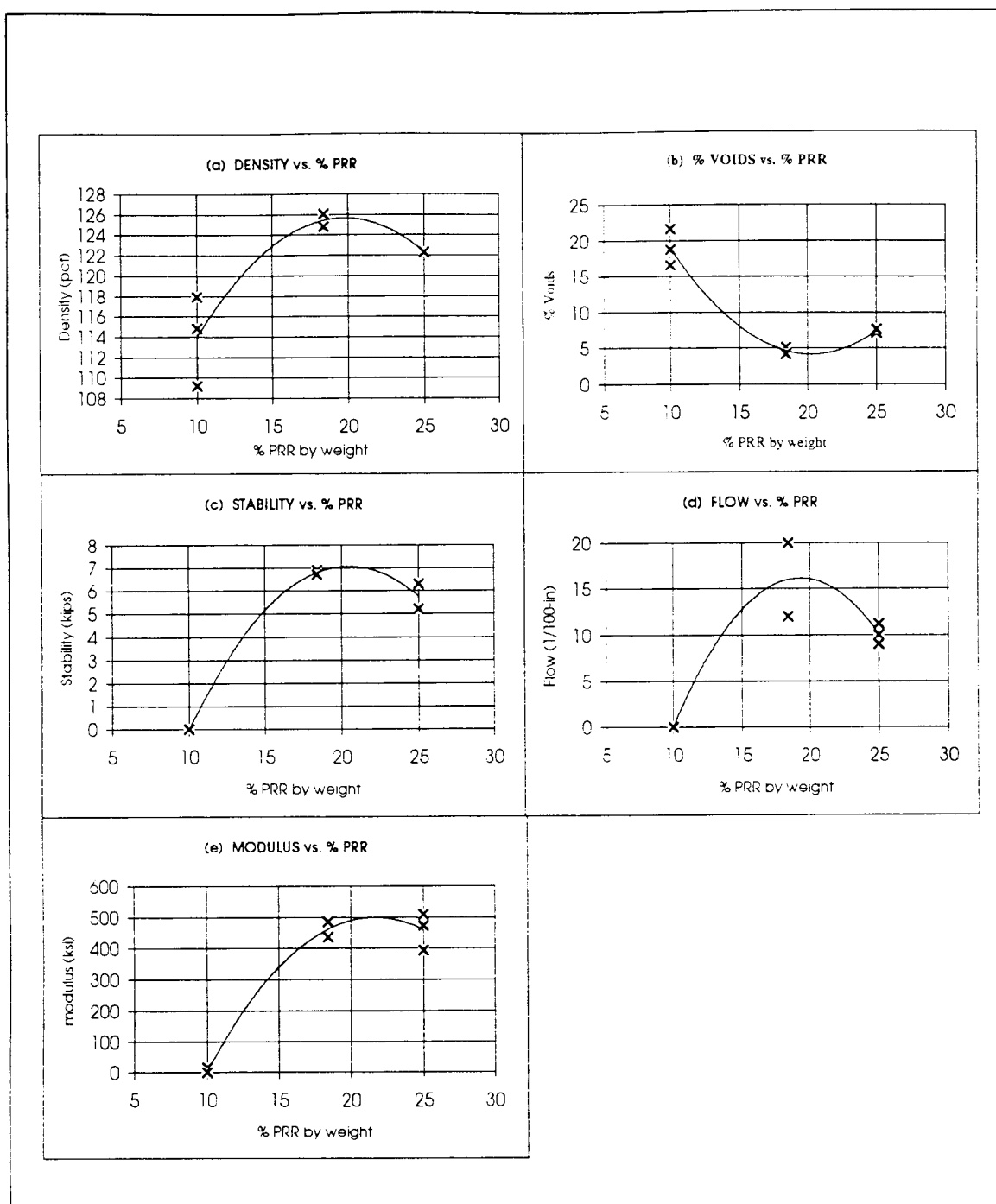


Figure 4-3 Hot-Mixed Dense Graded Marshall Plots for Optimum PRR Content

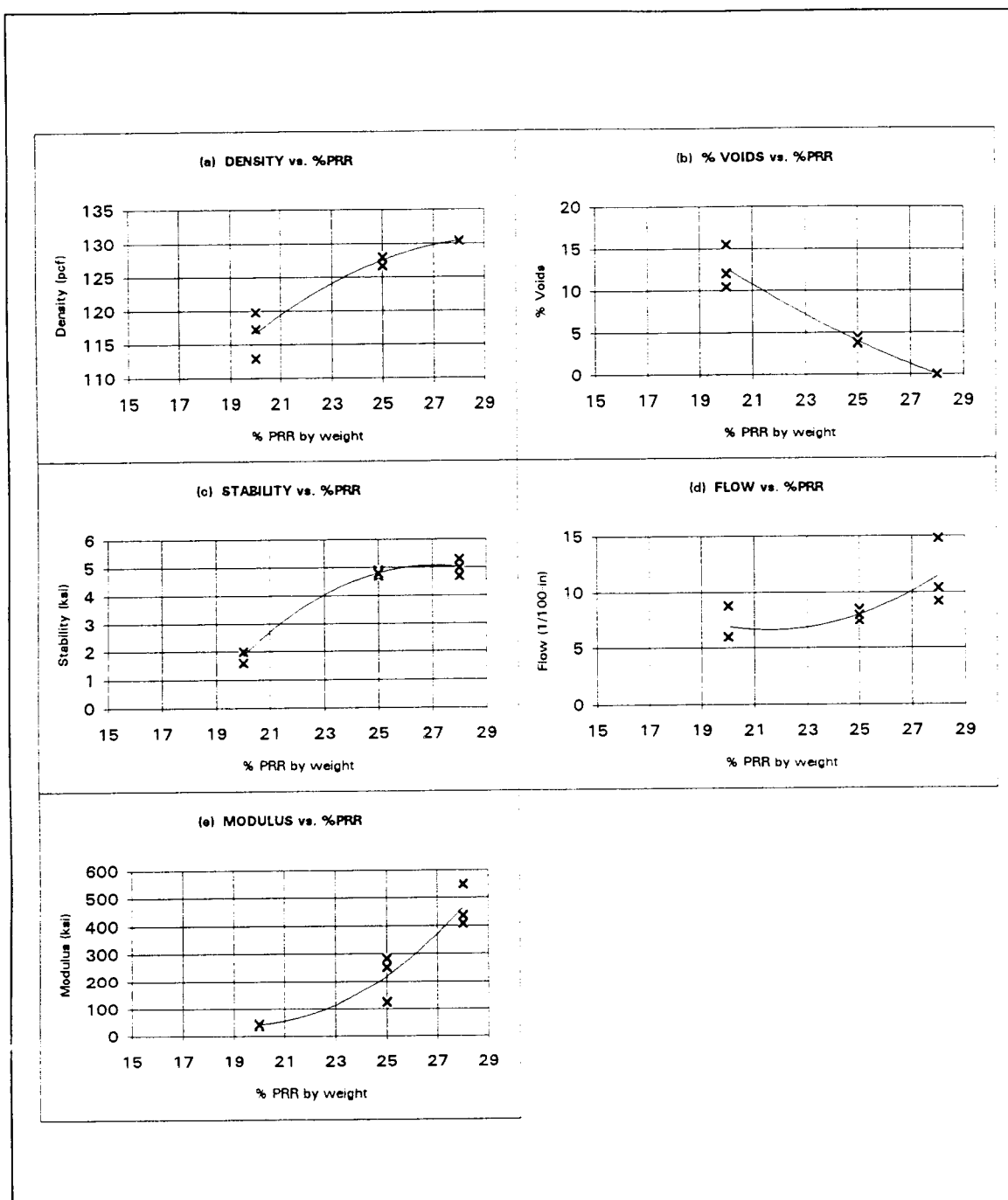


Figure 4-4 Hot-Mixed Gap Graded Marshall Plots for Optimum PRR Content

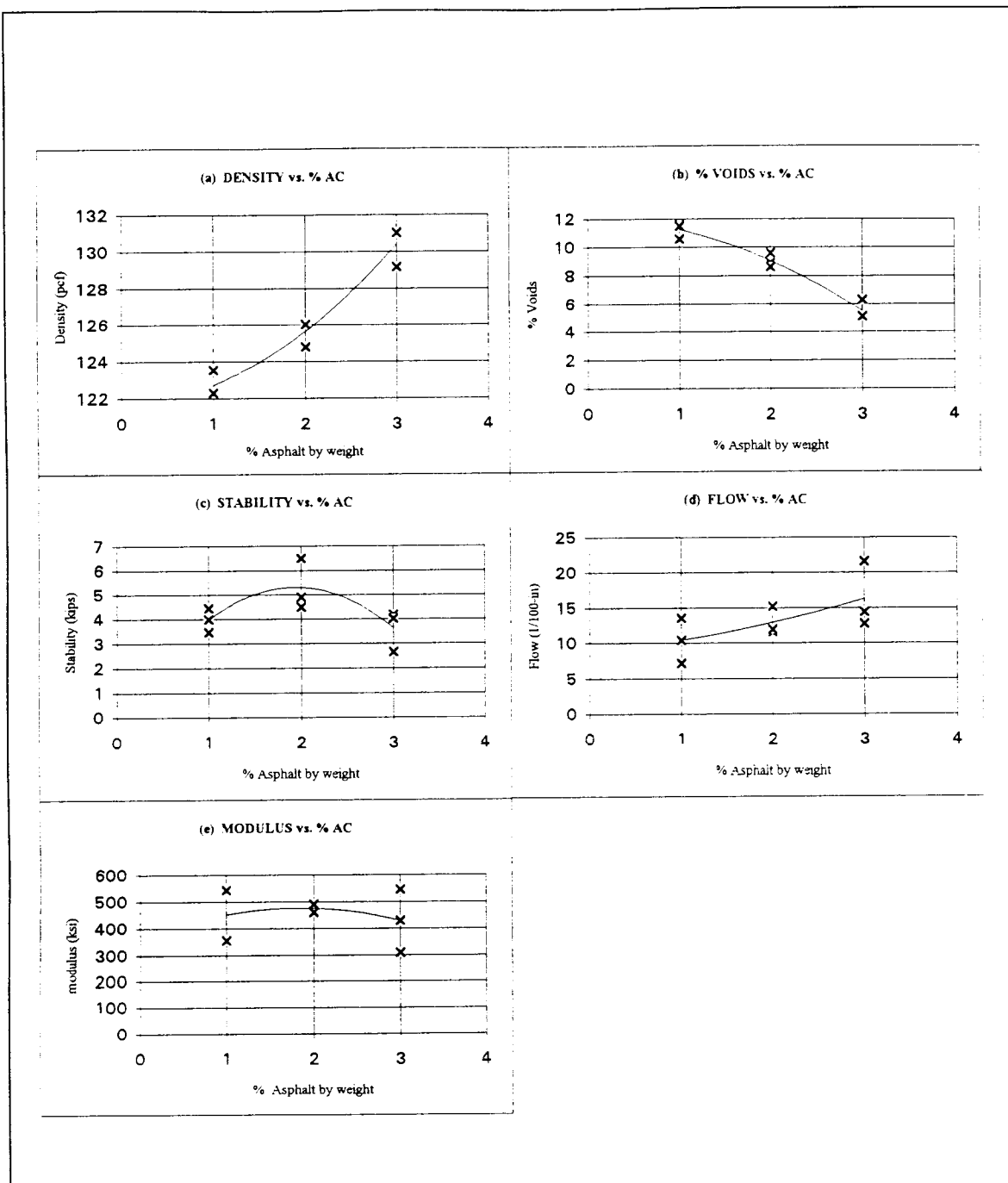


Figure 4-5 Hot-Mixed Dense Graded Marshall Plots at 20% PRR for Optimum AC Content

The following brief descriptions are given for evaluating the optimum of each mixture:

- Figures 4-3 a-e—Maximum density is achieved at 20% PRR; acceptable percent air voids of approximately 5% achieved at 20% PRR; maximum stability achieved at 20% PRR; acceptable flow at 20% PRR; maximum modulus achieved at 22% PRR. Average of optimum parameters is 20% (additional details are provided in Appendix A).
- Figures 4-4 a-e—Maximum density achieved at 28% PRR; acceptable voids at 25% PRR; acceptable stability achieved at 25% PRR; acceptable flow at 25% PRR; acceptable modulus achieved at 28% PRR. Average of optimum parameters is 26.2%, however, 25% PRR was used (additional details are provided in Appendix A).
- Figures 4-5 a-e—Maximum density achieved at 3% AC; acceptable voids at 3% AC; maximum stability achieved at 2% AC; acceptable flow at 2% AC; acceptable retained modulus at 2% AC. Average of optimum parameters is 2.4% (additional details are provided in Appendix A).

#### 4.1.1.4 Cold-Mix Results

Twelve samples were prepared using gap graded aggregate and approximately 17% PRR at three emulsion contents of 3.3, 6.5, and 9.5%, as outlined in Chapter 3 (all percentages given as percent by dry weight of total sample). The Marshall plots shown in Figures 4-6 a-e only have two data points at the two lower emulsion contents, due to slumping of specimens containing 9.5% emulsion while heating in the pre-Marshall oven (see Appendix A, Table A5).



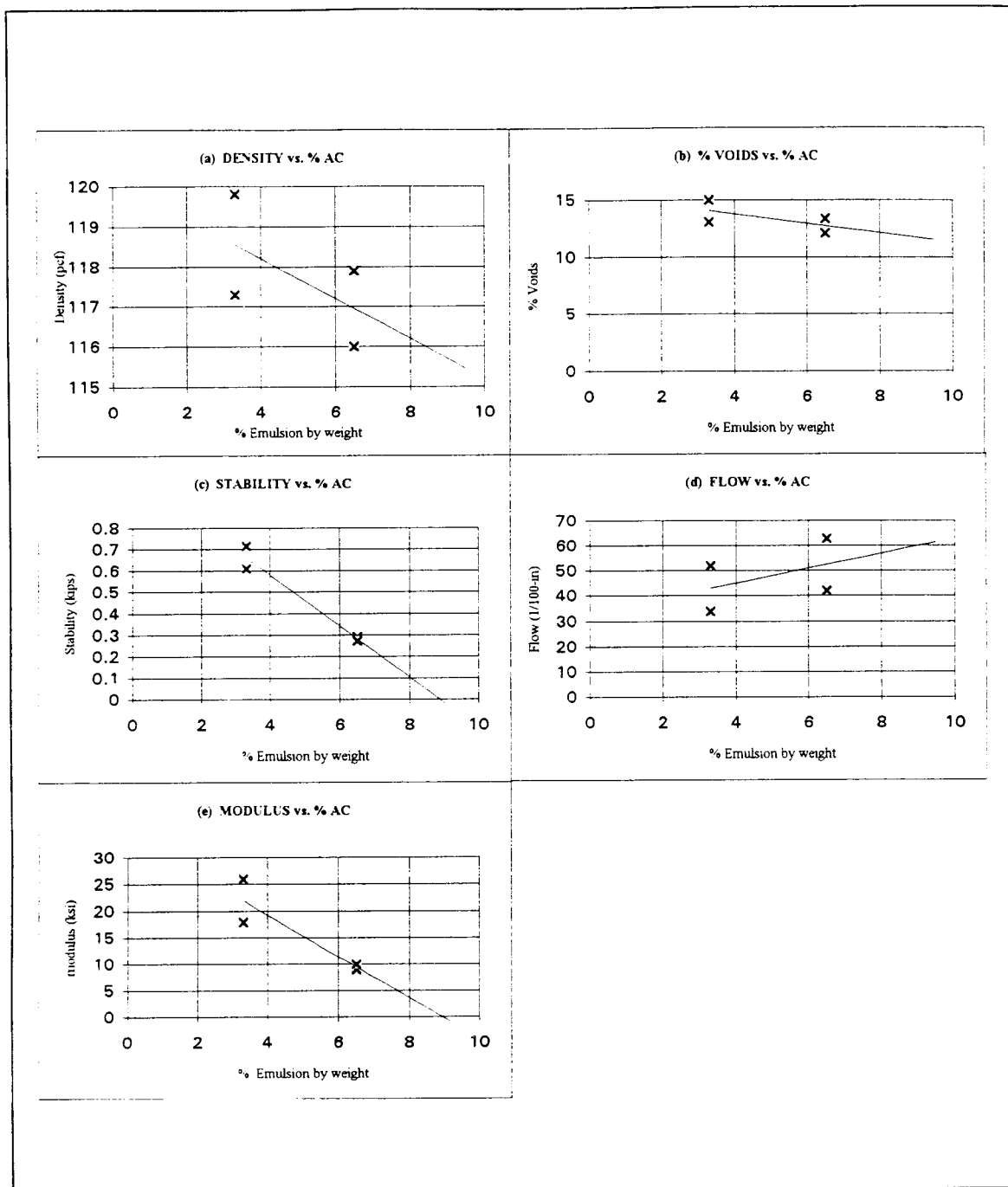


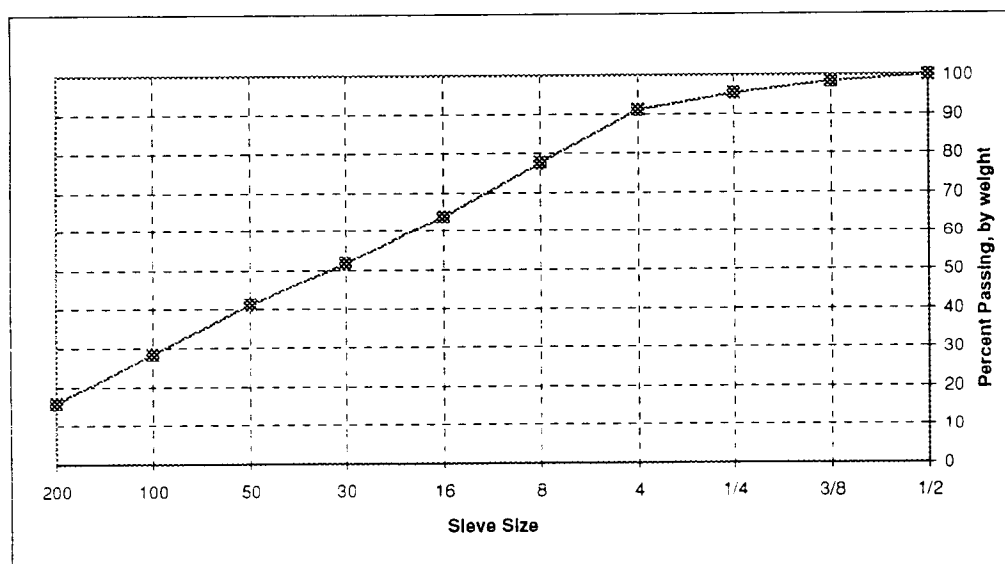
Figure 4-6 Cold-Mixed Gap Graded Marshall Plots with 20% PRR at Different Emulsion Contents

It is not possible to derive an optimum emulsion content from these plots. However, the optimum is likely lower than 3.3%, due to the steep slope of the data points shown. Due to the perceived difficulties of achieving a structurally sound gap-graded PRR mix design, further testing was not performed to determine the optimum of this mixture.

#### 4.1.2 Oklahoma

##### 4.1.2.1 PRR, NRR and TRR Gradations

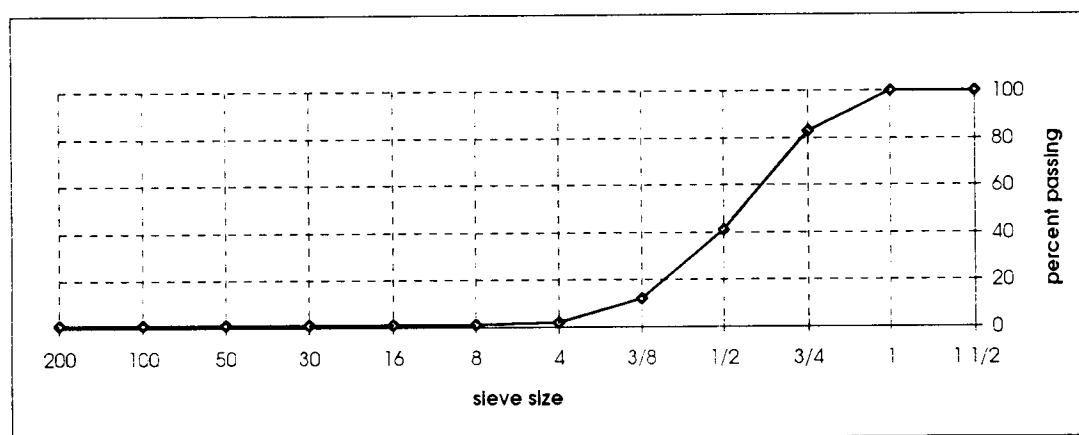
As with Pennsylvania PRR, the gradation of Oklahoma PRR and NRR was not determined. However, the gradation of the treated PRR (TRR) was evaluated by wet sieving as shown in Figure 4-7. The plot reveals a gradation similar to that of a well graded sand.



**Figure 4-7 Wet Sieve Results of Treated PRR**

#### 4.1.2.2 Aggregate Gradation

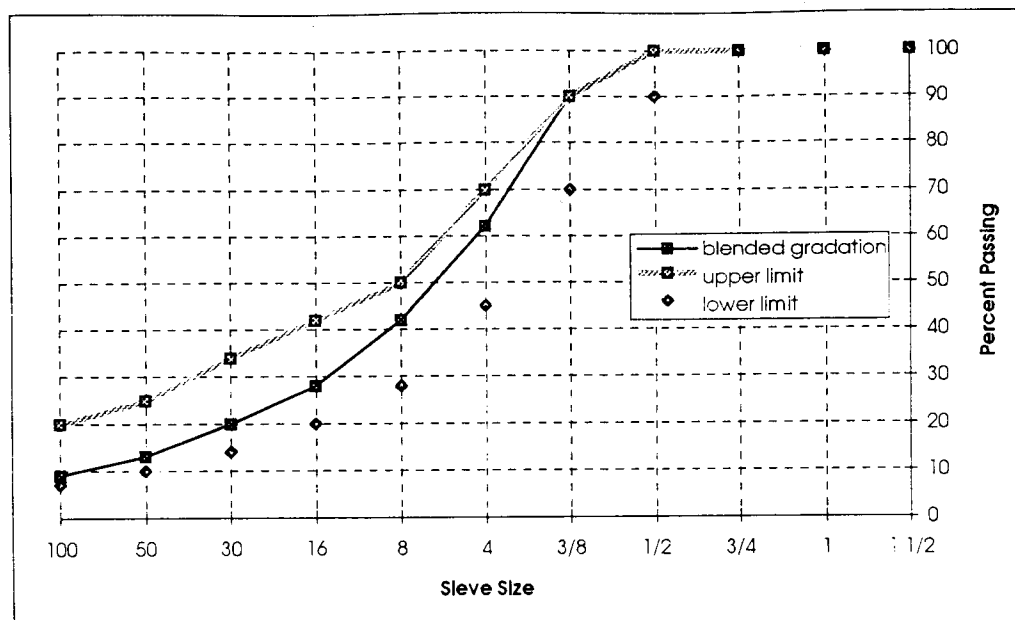
Oklahoma hot-processed dense graded specimens utilized the Pennsylvania aggregate and gradations described in section 4.1.1. Cold-processed gap graded<sup>a</sup> mixtures utilized 100% of the "1"-1/2" bin. This gradation is plotted in Figure 4-8. Theoretically, the voids in the mineral aggregate (VMA) would increase by omitting the mineral filler, thereby, increasing the space available for the NRR.



**Figure 4-8 Open Graded Aggregate Gradation Utilized for Oklahoma Mixtures**

Dense graded Oklahoma aggregates were acquired for mix designs used at the Demonstration site (described later). Figure 4-9 displays the wet sieve results with the lower and upper limits of an Oklahoma DOT's dense graded specification. The gradation of the aggregate fall within the specified limits throughout the range.

<sup>a</sup> Technically, once the PRR is incorporated into the open gradation it becomes a "gap graded" mixture, due to the void of material between the #4 and #200 sieves.



**Figure 4-9 Dense Graded Blend Plotted with Oklahoma DOT Specification Limits**

#### 4.1.2.3 Hot-Mix Results

As previously mentioned, hot mixtures employing Oklahoma NRR utilized Pennsylvania (PA) aggregate and were graded to PA DOT specifications. Using the NRR with the PA aggregate allowed for a relative comparison of the different sources of PRR (PA and OK). The Demonstration Project required a mix design that could only be accomplished using OK aggregates, since that would be the source used in the field. The difference in gradation should be kept in mind when making comparisons between mixtures.

Table A4 gives the results of the hot-mixed dense graded specimens tested. Mixtures were proportioned according to the optimum results of the PA dense graded hot-mix (i.e., one data point using six specimens at 20% NRR and 2% AC) for

comparison with similar PA mixtures. The strength parameters compared favorably with those collected using the PA material. As seen in Table A4, samples prepared using NRR had average  $M_r$ , stability, and flow values of 326 ksi, 4.9 kips, and 8, respectively. Mixtures utilizing PA PRR gave similar results of 500 ksi, 5.3 kips, and 13, with nearly identical mix proportions.

#### 4.1.2.4 Cold-Mix Results

As with PA PRR, cold mixes prepared using OK NRR had significantly reduced modulus and stability values, compared with hot-mixed specimens. Table A6 gives average modulus and stability values of 14,000 psi and 135 lbs, respectively, for gap-graded specimens prepared with 16% NRR and 8% emulsion. Table A8 reveals higher strength parameters for specimens prepared with TRR, relative to specimens prepared using open graded aggregate, PA NRR, and emulsion, described earlier. Figure 4-10 indicates an optimum CSS-1h polymer-modified emulsion content of 10% for TRR mixtures with estimated stability, flow, and modulus values of 1700 lbs, 0.011 in, and 190,000 psi, respectively. Maximum stability and modulus values attained using open graded aggregate, PA NRR, and emulsion were approximately 700 lbs and 25,000 psi, respectively.

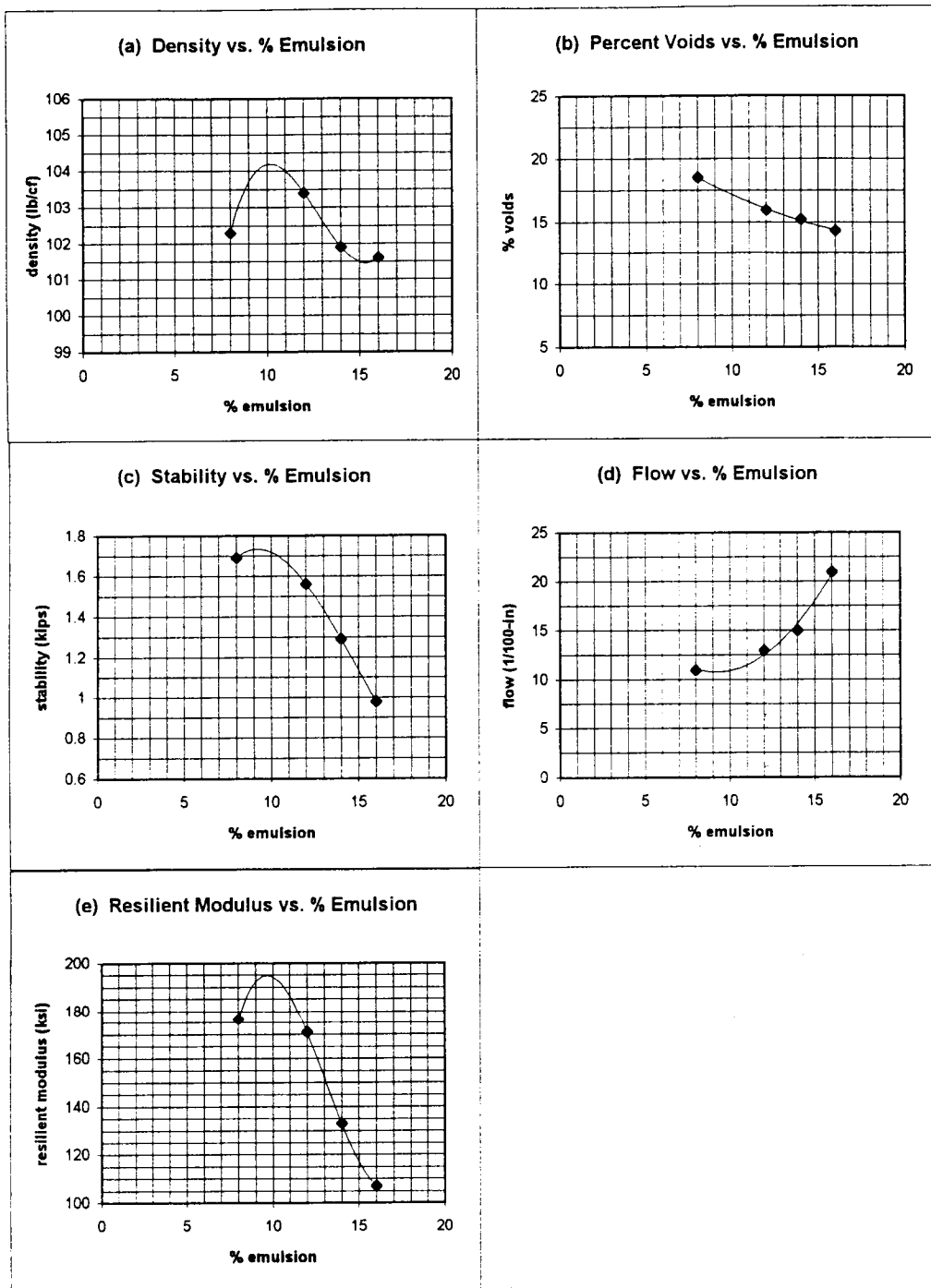


Figure 4-10 Marshall Plots for Cold-Mixed TRR Specimens

Although high by conventional standards, this optimum emulsion content is actually quite accurate according to the Asphalt Institute's method<sup>25</sup> for prediction based on gradation as follows:

$$A \text{ (base mix)} = [(0.06 \times B) + (0.01 \times C)] \times 100 = 5.6\%$$

or

$$A \text{ (surface mix)} = [(0.07 \times B + (0.03 \times C)] \times 100 = 6.6\%$$

$$E = A/(1.00 - A)/R \times 100 = 9.6\% \text{ for base mix and } 11.4\% \text{ for surface mix}$$

Where:      A = Percent residual AC based on dry weight of mix  
                  B = Percent passing the 4.75 mm (No. 4) sieve (91.1% from Figure 4-9)  
                  C = 100 - percent passing the 4.75 (No. 4) sieve (8.9% from Figure 4-9)  
                  R = Percent residual AC by distillation (62% as seen in Table 3-2)  
                  E = Percent emulsion based on dry weight of aggregate

Note: This procedure is for obtaining an estimate only and does not apply to open-graded aggregates.

The high predicted optimum emulsion content is due to the fineness of the TRR gradation (Figure 4-7). Decreasing particle size increases the surface area of a mixture, therefore, requiring more asphalt for coating.

#### 4.1.2.5 Demonstration Project Results

An exceptionally wet spring in and around Tulsa hindered complete curing of mixtures placed. Occurrences of excessive flooding were observed on several occasions

after placement. Infiltration of water into an emulsion mixture, that is not fully cured, could wash away emulsion that has not yet set.

Compaction devices used consisted of a five ton steel roller and a seven ton rubber tire roller used for the first and second compactions, respectively. A ten ton roller would have been preferred, however, one was not available.<sup>45</sup>

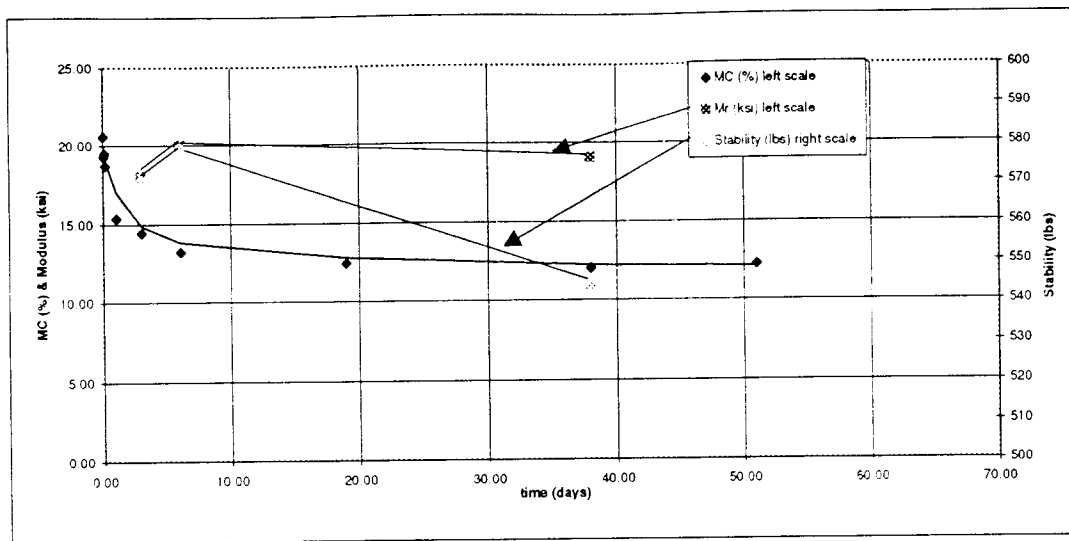
Table A7 summarizes lab testing results of samples collected in the field (refer to Chapter 3 for lane compositions). The following trends were observed:

#### **4.1.2.5.1      Lanes 1-9: Treated Refinery Residuals (TRR) with Emulsion**

Lanes 1-9 were mixtures of TRR and 6% to 14% emulsion.

- Moisture content (MC), resilient modulus ( $M_r$ ), and stability vs. time trends are consistent throughout this section (see Figure 4-11).
- High moisture contents maintain low modulus and stability values.
- Compaction of the mats at an approximate MC of 14 percent made attaining material densities and strength parameters achieved in the laboratory study unlikely.
- Coring difficulties damaged specimens complicating testing.





*Figure 4-11 Lane #1 Moisture Content, Modulus, Stability, vs. Time*

#### 4.1.2.5.2 Lane 10: Neutralized Refinery Residual (NRR)— Excluding Emulsion

Lane 10 contained 25 percent NRR blended into a standard OK DOT dense gradation.

- Difficult sampling (i.e., samples disintegrated upon coring). No consistent trends recorded. However, field observations indicated little surface degradation from rain washing and a high degree of structural integrity throughout the performance period of approximately 90 days (i.e., dump trucks and front-end loaders consistently drove over its surface without deformation).

#### 4.1.2.5.3 Lanes 11 & 12: Aggregate, Neutralized Residuals, and Emulsion

Lane 11 contains 77.7% Oklahoma aggregates, 13.7% NRR, and 8.6% emulsion.

Lane 12 contains 79.5% Oklahoma aggregates, 14.0% NRR, and 6.5% emulsion.

Observations are as follows:

- Figures 4-12 and 4-13 indicate that lane 12 has greater moisture sensitivity than lane 11, due to the decrease in modulus likely caused by exposure to moisture. This may be due to the increased emulsion content of lane 11, thus, limiting moisture penetration.
- A notably steeper initial slope during the first 10 days of curing, compared to TRR-emulsion mixtures in lanes 1-9, suggests that the mixtures containing aggregate have faster curing times.

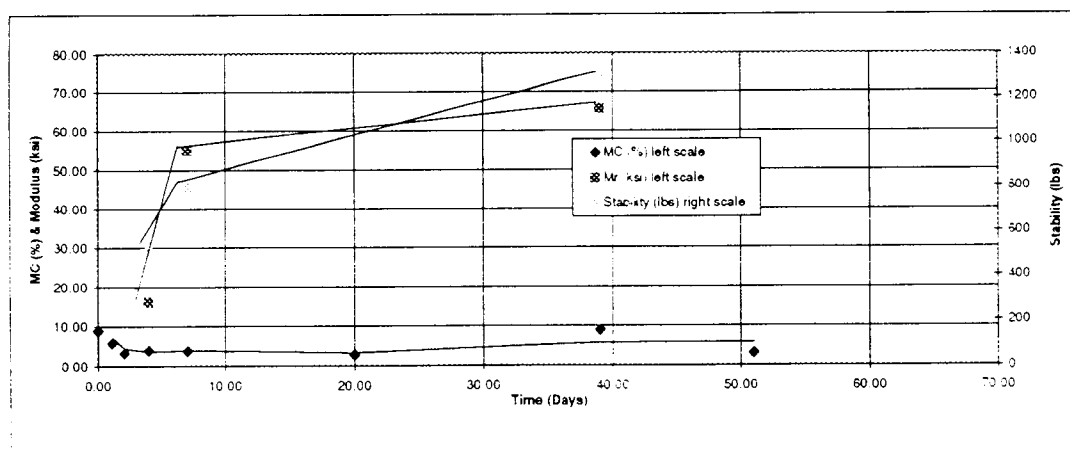


Figure 4-12 Lane #11 Moisture Content, Modulus, and Stability vs. Time

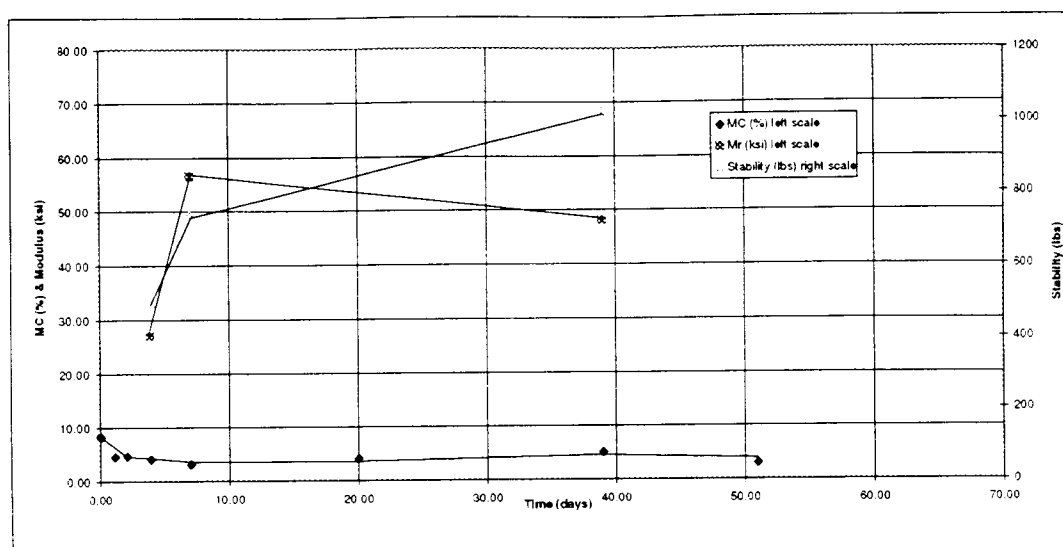


Figure 4-13 Lane #12 Moisture Content, Modulus, and Stability vs. Time of Field-Cure

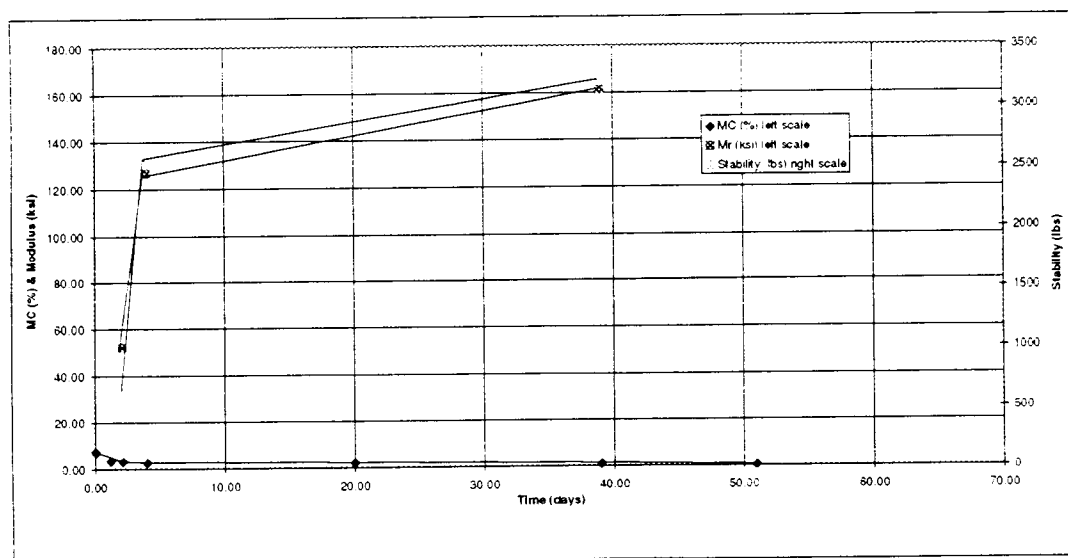


Figure 4-14 Lane #13 Moisture Content, Modulus, and Stability vs. Time of Field-Cure

#### **4.1.2.5.4 Lane 13: Typical Aggregate/Emulsion Mixture—Excluding Residuals**

Lane 13 is a “typical” emulsion/aggregate mixture containing 92.5% aggregates and 7.5% emulsion. Observations were as follows:

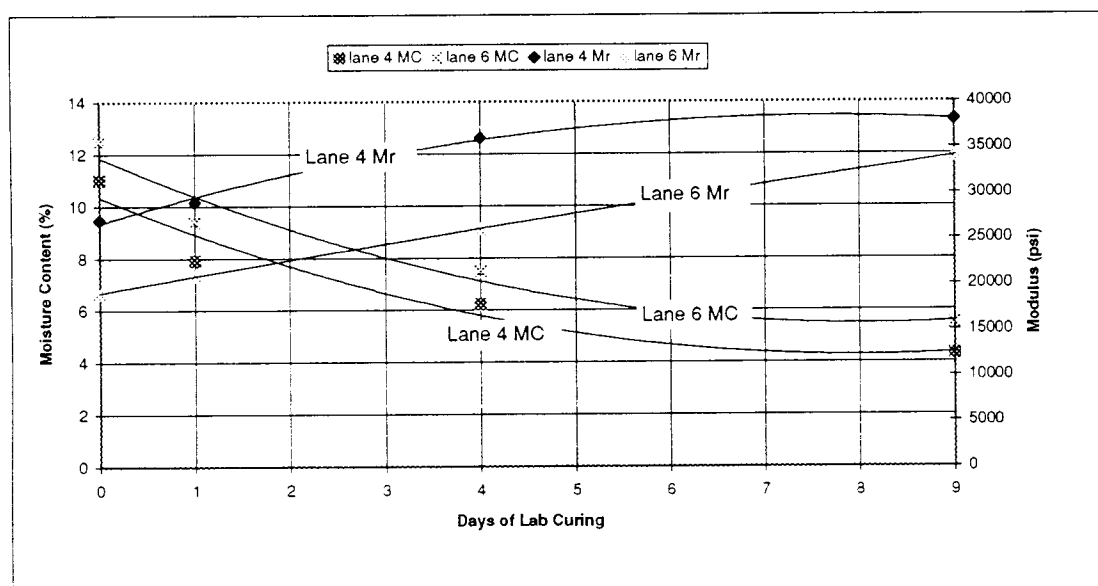
- Steeper slope of strength parameter plots during the first 10 days indicate that the emulsion was able to fully cure (see Figure 4-14).
- Essentially no moisture gain during wet periods (i.e., MC is constant beyond 10 days).
- Continued strength gain noted after rain events (emulsion continued to cure and “tighten” the mixture even when exposed to wet weather).

#### **4.1.2.5.5 Comparison with Laboratory Data**

Strength parameters were significantly lower for TRR/emulsion specimens collected from the field compared with those prepared in the laboratory. High moisture contents at the time of compaction are the likely cause. These mixtures were compacted at 14 percent MC and remained there due to rain—compared with a MC at time of compaction of approximately 5 percent in laboratory specimens (Table A8). Wet weather conditions following field placement maintained high moisture contents, and therefore, inhibited curing and strength gain of mixtures containing TRR.

NRR/emulsion mixtures performed well in the field (lanes 11 & 12). However, no laboratory data was collected on dense-graded NRR/emulsion mixtures, therefore, no comparisons can be made.

Figures 4-15 and 4-16 illustrate curing trends of field compacted samples when bench-cured for extended periods of time in the laboratory. Moisture content is plotted on the left and modulus plotted on the right ordinate with days of curing on the abscissa. Lanes 4 & 6 achieved a maximum  $M_r$  of approximately 20 ksi after field curing for 39



**Figure 4-15 Lanes 4 & 6 Moisture Content and  $M_r$  vs. Days of Lab-Curing**

days. Bench-curing of representative samples in the laboratory for 9 days resulted in  $M_r$  values for lanes 4 & 6 of 38 and 34 ksi, respectively (Figure 4-15). Lane 13 attained 160 ksi after 39 days of curing in the field. As seen in Figure 4-16, the  $M_r$  exceeded 200 ksi after 8 days of bench-curing and achieved near 250 ksi after 33 days.

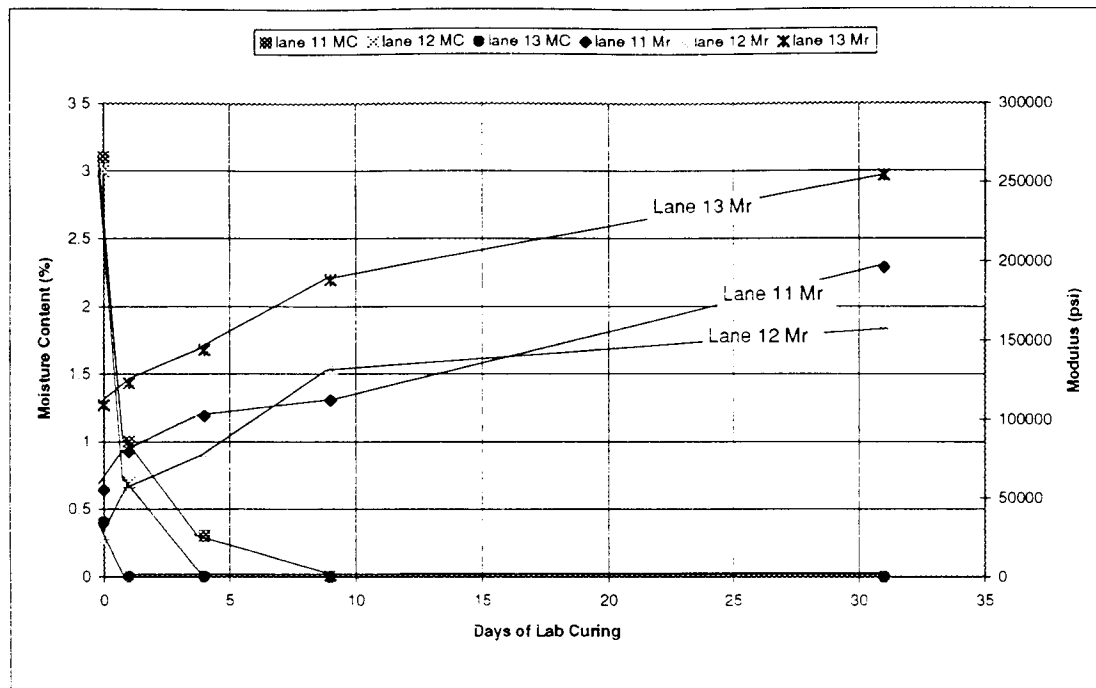


Figure 4-16 Moisture Content and Mr vs. Days of Lab-Curing for Lanes 11, 12, and 13

## 4.2 Testing for Environmental Suitability

### 4.2.1 Pennsylvania

#### 4.2.1.1 pH Testing

A study was performed to evaluate the reliability of using litmus paper (OSU Civil Dept.) vs. a pH meter (Central Analytical Laboratory) and to determine the minimum lime content that would be required to bring the PRR to a pH of approximately five, as described in Chapter 3. Central Analytical Laboratory required the PRR-lime mixture to be ground to 2 mm (#10 sieve) or smaller such that added water would have sufficient surface contact for accurate pH readings. Figure 4-17 shows the OSU and Central Analytical results of the PA PRR1 and indicates a lime content of 2.5% (by dry weight of PRR, assuming a MC of 25%) to be sufficient to raise the pH to five. PA

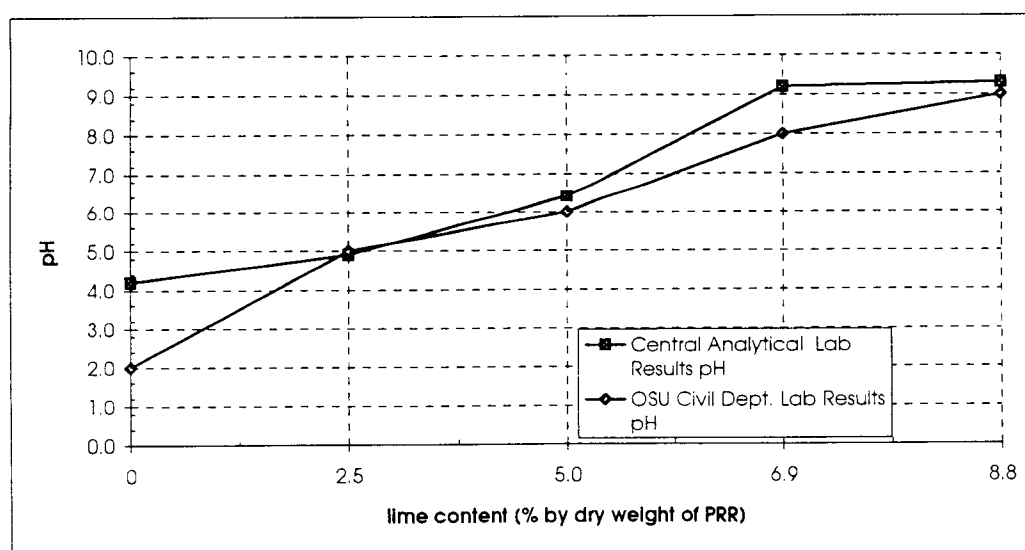


Figure 4-17 pH versus Lime Content---Comparison Between Methods

PRR2 required 3.8% lime to raise the pH to five. Therefore, lime was added at a rate of approximately 3.1% by dry weight to mixtures containing a 50/50 split of PA PRR1 and PRR2.

#### 4.2.1.2 Whole Sample Analysis

Prior to working extensively with PRR in the laboratory or possibly in field applications, quantitative knowledge of existing constituents within the PRR was desired. A whole sample analysis was performed for this purpose.

Tables 4-1 and 4-2 list the results of a whole sample analysis (i.e., metals and organics) on a raw sample of PA PRR. Considerably more constituents were analyzed than those regulated by the EPA for the purpose of gaining general knowledge. Therefore, not all constituents shown in Tables 4-1 and 4-2 have a corresponding regulatory maximum. The sample result given in mg/kg is nearly equivalent to ppm, due to the specific gravity of the PRR being near 1.0.



**Table 4-1 Metals Results of Pennsylvania PRR<sup>ψ</sup>**

Analyte	Reporting Limit	Sample Result	Qualifier	EPA Regulated Maximum Conc. (ppm)
Aluminum (Al)	30	6190		-
Antimony (Sb)	20	64		-
Arsenic (As)	140	206		5.0
Barium (Ba)	0.4	34.7		100.0
Beryllium (Be)	0.2	0.2	U	-
Boron (B)	20	21		-
Cadmium (Cd)	4	4	U	1.0
Calcium (Ca)	18	3090		-
Chromium (Cr)	2	2	U	5.0
Cobalt (Co)	14	14	U	-
Copper (Cu)	2	46		-
Iron (Fe)	12	15000		-
Lead (Pb)	60	8970		5.0
Lithium (Li)	2	3		-
Magnesium (Mg)	2	1800		-
Manganese (Mn)	0.6	391		-
Mercury (Hg)	0.02	0.05		0.2
Molybdenum (Mo)	40	40	U	-
Nickel (Ni)	8	8	U	-
Potassium (K)	40	960		-
Selenium (Se)	100	100	U	1.0
Silicon (Si)	8	8	U	-
Silver (Ag)	6	6	U	5.0
Sodium (Na)	10	398		-
Strontium (Sr)	0.1	21.3		-
Thallium (Tl)	40	40	U	-
Tin (Sn)	60	60	U	-
Titanium (Ti)	30	44		-
Vanadium (V)	2	5		-
Zinc (Zn)	4	909		-
Zirconium (Zr)	0.6	2.6		-

Qualifier Descriptions: U = not detected at specified limits; J = estimated value.

<sup>ψ</sup>Courtesy of CH<sub>2</sub>M Hill Analytical Laboratories, Corvallis, OR.

Table 4-2 Organic Results of Pennsylvania PRR<sup>ψ</sup>

Analyte	GC-FID (mg/kg)	Qualifier	GC-MS (mg/kg)	Qualifier	EPA Regulated Maximum Conc. (ppm)
Phenanthrene	<55		4.1	J	-
Chrysene	118	J	160		-
Fluorene	55		<40		-
Fluoranthene	71		<40		-
Benzo(K)fluoranthene	<55		18	J	-
Benzo(a)pyrene	324	J	75		-
Ideno(1,2,3-Cd)pyrene	40		<40		-
Dibenz(a,h)anthracene	133	J	17	J	-
Benzo(g,h,i)perylene	270	J	74		-
Phenol	---		46		-
Benzoic Acid	---		87	J	-

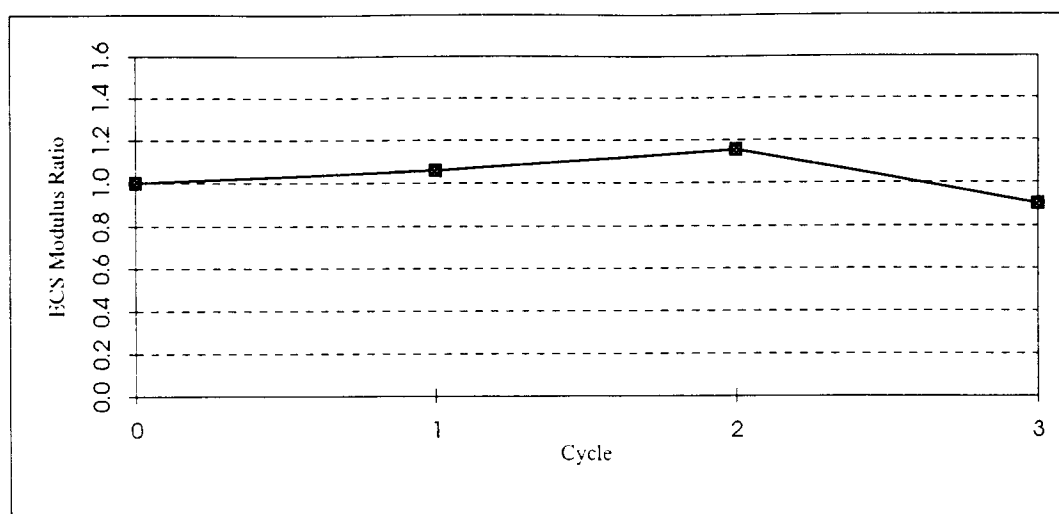
Qualifier Descriptions: U = not detected at specified limits; J = estimated value.

<sup>ψ</sup> Courtesy of CH<sub>2</sub>M Hill Analytical Laboratories, Corvallis, OR.

#### 4.2.1.3 Environmental Conditioning System (ECS) Testing

Two samples, one each of the dense and gap graded mixtures at optimum PA PRR content, were subjected to the Environmental Conditioning System (ECS) for evaluation of stability under loading while saturated with water. Water drawn through the sample was to be collected and shipped to CH<sub>2</sub>M Hill for TCLP testing.

The ECS procedure generates a triaxial modulus ratio (conditioned modulus over non-conditioned modulus). This ratio is then plotted versus the cycle, as shown in Figure 4-18.



**Figure 4-18 ECS Results for Gap Graded Mixture at Optimum PA PRR Content**

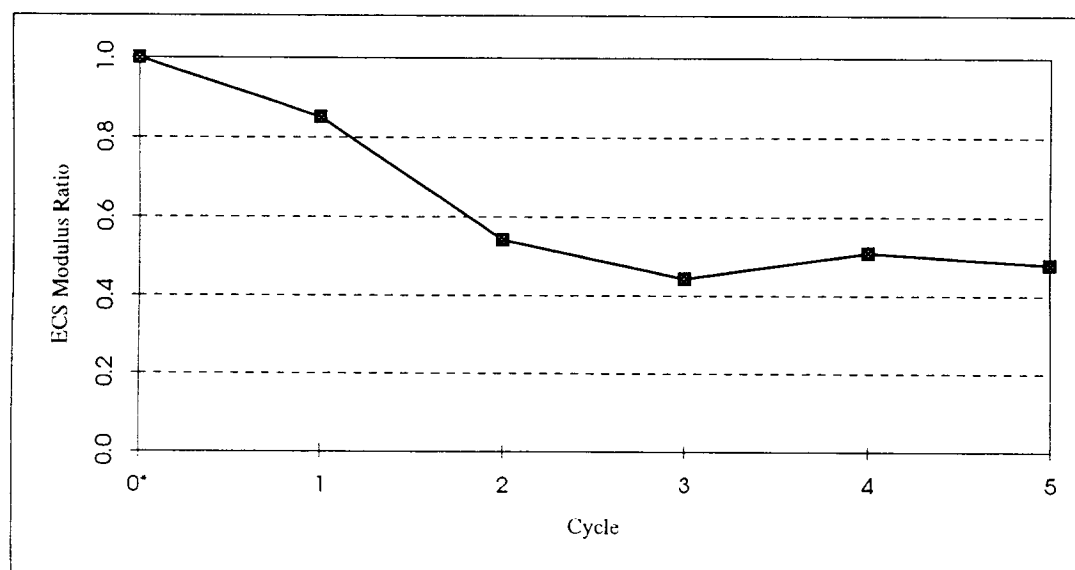
A gap graded specimen was tested using the ECS SHRP protocol. This specimen, subjected to the normal ECS procedures, indicated a high degree of water/temperature sensitivity. As seen in Figure 4-18, the modulus ratio increases over the first two cycles then recedes at the end of the third cycle. This is possibly due to bulking of the specimen in cycle three (the freeze cycle), as will be discussed in Chapter 5.

The gap graded specimen settled and bulged. This deformation caused eccentric loading which may have influenced test results. Relatively little, if any, water passed through the specimen.

The dense graded specimen tested in the ECS had less deformation and little eccentricity compared to that of the gap graded specimen. However, this sample was not subjected to the same temperature variation as the gap graded specimen, but rather was

held at a constant temperature of 25C throughout, to better simulate in situ conditions that would be experienced by a treated base.

Triaxial modulus ratios of the dense graded specimen are plotted versus the cycle in Figure 4-19 which indicates the sample to be very moisture sensitive. SHRP protocol states that a specimen has reached failure when the modulus ratio reaches 0.70, or 70% of the initial modulus. Therefore, this sample failed about halfway through the second cycle. This specimen was exposed to vacuum water pressure for approximately 24-hours prior to testing, in an effort to force water through the specimen for TCLP testing. This procedure in the SHRP protocol. However, it was felt that loading and/or temperature change might hamper water flow through the specimen, therefore, justifying pretesting saturation.



**Figure 4-19 ECS Results for Dense Graded Mixture at Optimum PA PRR Content**

#### 4.2.1.4 TCLP Results and Comparison

The whole sample analysis indicated lead levels to be near 1% (8970 ppm, i.e., the unit weight of the PRR is approximately 1.0 g/ml, therefore, 1 mg/kg @ 1 mg/l = 1 ppm), in the sample tested. TCLP lead levels of untreated specimens were found to be from 6 to 14 ppm when mixed cold. However, hot-mixed specimens had TCLP lead levels exceeding 25 ppm. The regulatory maximum set by the EPA for a characteristically toxic hazardous waste is 5.0 ppm. It was therefore necessary to attempt to facilitate lead fixation in the PRR.

In an attempt to minimize leachable lead, testing of various combinations of PRR, lime, emulsion, and cement/water mixtures were conducted, as outlined and summarized in Tables 4-3 and 4-4 (further details in Appendix B, Table B1). Mixtures made with combinations of lime and emulsified asphalt did not have TCLP lead results exceeding 5 ppm, as seen in Table 4-3. However, Table 4-4 shows TCLP lead results exceeding 5 ppm for certain combinations of cement and mixing water.

**Table 4-3 TCLP Lead Leachate vs. %Lime, %Emulsion, and %PRR of Gap Graded Specimens**

% Lime <sup>1</sup>		2.8	5.5	8.2	11	14.4	17.1	20.2
% Emulsion <sup>2</sup>	PRR Content <sup>3</sup>	Lead Leachate (ppm)						
6.4	8.4% PRR							
	12.6% PRR							
	18.0% PRR	3.4	1.3	0.8	1.2			
9.3	8.4% PRR			1.8	0.4			
	12.6% PRR			0.8	0.5			
	17.7% PRR	2.0	0.9	0.8/1.2	1.3/1.0	1.6		
12.2	8.3% PRR			0.7	0.2			
	12.5% PRR			1.1	1.9			
	17.4% PRR		0.7	2.1	1.4/1.1	0.9/1.1	2.1	
15.0	8.4% PRR							
	12.4% PRR							
	16.9% PRR			2.7	1.2	0.8/1.6	1.0/0.9	0.6

<sup>1</sup>Percent lime based on dry weight of PRR only (PRR MC was approximately 10% at time of mixing).

<sup>2</sup>Percent emulsion is based on total sample dry weight, assuming a PRR MC of 10%.

<sup>3</sup>PRR content is based on dry weight of total sample.

**Table 4-4 TCLP Results of Cement Treated Specimens**

Specimen No.	Cement Content (%)	% Mixing-Water Added <sup>2</sup>	TCLP Lead (ppm)
1	0	0	8.9
2	5	0	6.2
3	10	0	6.7
4	10	10	2.6
5	10	20	2.9
6	15	0.0	2.7

<sup>1</sup>Percent based on dry weight of PRR.

<sup>2</sup>Percent based on dry weight of PRR and cement.

A study conducted by Mr. Anthony Kriech of Heritage Research Group<sup>46</sup> evaluated the leaching potential of a typical asphalt concrete containing AC-20 grade asphalt cement. Tables 4-5, 4-6, and 4-7 compare the TCLP results from a PA PRR-modified mixture, containing 78% aggregate, 20% PRR, 2% AC-20, and 2.5% lime to those found by Kriech, in a typical asphalt concrete mixture. The two middle columns

give the results of the two mixtures tested. The far right column indicates the maximum regulatory concentration (if one exists), set forth by the EPA.<sup>47</sup> Constituents analyzed for include metals, volatile organics, semi-volatile organics, and polynucleated aromatic hydrocarbons. These elements and compounds are of concern in aquatic environments and are important parameters associated with toxicity, as will be further discussed in Chapter 5.

**Table 4-5 Comparison of TCLP Metals for a Typical Asphalt Concrete and a PA PRR-Modified Material**

Metal Constituent	AC-20 Mixture Result (mg/L) <sup>46</sup>	PRR Result (mg/L)	TCLP Maximum Regulatory Concentration (mg/L)
Barium	2U	0.5U	100.0
Cadmium	0.02U	0.08	1.0
Chromium	0.10	0.01U	5.0
Lead	0.2U	2.4	5.0
Silver	0.04U	0.006U	5.0
Arsenic	0.005U	0.7U	5.0
Selenium	0.005U	0.5U	1.0
Mercury	0.005U	0.0009U	0.2

U = Compound analyzed for but not detected (i.e., 0.02U = compound not detected at 0.02 detection limit)

**Table 4-6 TCLP Organics of a Typical Asphalt Concrete vs. PA PRR-Modified Material**

Volatile Organics Constituent	AC-20 Mixture Result (mg/L) <sup>46</sup>	PRR Result (mg/L)	TCLP Maximum Regulatory Concentration (mg/L)
Benzene	5U	25U	70
Carbon Tetrachloride	5U	25U	70
Chlorobenzene	5U	25U	1,400
Chloroform	5U	25U	70
1,2 Dichloroethylene	5U	NA	NR
1,1 Dichloroethylene	5U	NA	700
Methyl Ethyl Ketone	5U	NA	200,000
Tetrachloroethylene	5U	NA	700
Trichloroethylene	5U	NA	500
Vinyl Chloride	5U	50U	50
Semi-Volatile Organic Constituent	AC-20 Mixture Result (mg/L) <sup>46</sup>	PRR Result (mg/L)	TCLP Maximum Regulatory Concentration (mg/L)
1,4-Dichlorobenzene	12U	50U	4300
2,4-Dinitrotoluene	12U	50U	130
Hexachlorobenzene	12U	50U	130
Hexachlorobutadine	12U	50U	720
Hexachloroethane	12U	50U	4300
Nitrobenzene	12U	50U	130
Pyridine	60U	50U	5000
2-Methyl Phenol	30U	50U	10000
4-Methyl Phenol	30U	50U	10000
Pentachlorophenol	60U	250U	3600
2,4,5-Trichlorophenol	30U	250U	5800
2,4,6-Trichlorophenol	30U	50U	300

U = Compound analyzed for but not detected (i.e., 0.02U = compound not detected at 0.02 detection limit)



**Table 4-7 TCLP PAH Results of a Typical Asphalt Concrete and a PA PRR-Modified Material**

Polynuclear Aromatic Hydrocarbon	AC-20 Mixture Result (mg/L) <sup>46</sup>	PRR Result (mg/L)	TCLP Maximum Regulatory Concentration (mg/L)
Naphthalene	0.25(DL=0.096)	50U	NR
Acenaphthylene	0.15U	50U	NR
Acenaphthene	0.194U	50U	NR
Fluorene	0.023U	50U	NR
Phenanthrene	0.033U	50U	NR
Anthracene	0.015U	50U	NR
Fluoranthene	0.037U	50U	NR
Pyrene	0.04U	50U	NR
Benzo(A)Anthracene	0.048U	50U	NR
Chrysene	0.017U	50U	NR
Benzo(B)Fluoranthene	0.02U	50U	NR
Benzo(K)Fluoranthene	0.022U	50U	NR
Benzo(A)Pyrene	0.023U	50U	NR
Dibenzo(A,H)Anthracene	0.018U	50U	NR
Benzo(G,H,I)Perylene	0.036U	50U	NR
Indeno(1,2,3-CD)Pyrene	0.021U	50U	NR

U = Compound analyzed for but not detected (i.e., 0.02U = compound not detected at 0.02 detection limit);  
NR = Not Regulated

## 4.2.2 Oklahoma

### 4.2.2.1 Whole Sample Analysis

A whole sample analysis was performed by CH<sub>2</sub>M Hill on a raw sample of Oklahoma PRR. Table 4-8 and 4-9 list the results of this test. Considerably more constituents were analyzed than those regulated by the EPA for the purpose of gaining general knowledge. Therefore, not all constituents shown in Tables 4-7(a) and (b) have a corresponding regulatory maximum.

**Table 4-8 Metals Analysis Results of Oklahoma PRR<sup>W</sup>**

Analyte	Reporting Limit	Sample Result (mg/kg)	Qualifier	EPA Regulated Maximum Conc. (ppm)
Aluminum (Al)	30	5700		-
Antimony (Sb)	20	<10		-
Arsenic (As)	70	70	U	5.0
Barium (Ba)	0.4	72.7		100.0
Boron (B)	14	14	U	-
Cadmium (Cd)	4	4	U	1.0
Chromium (Cr)	2	7		5.0
Copper (Cu)	2	13		-
Lead (Pb)	60	73		5.0
Magnesium (Mg)	2	1680		-
Manganese (Mn)	0.6	140		-
Mercury (Hg)	0.0009	0.0009	U	0.2
Selenium (Se)	50	50	U	1.0
Silver (Ag)	6	6	U	5.0
Strontium (Sr)	0.1	60		-
Tin (Sn)	0.9	0.9	U	-
Titanium (Ti)	30	80.5		-
Vanadium (V)	2	6		-
Zinc (Zn)	4	39		-
Zirconium (Zr)	5	5	U	-

Qualifier Descriptions: U = not detected at specified limits; J = estimated value.

<sup>W</sup>Courtesy of CH<sub>2</sub>M Hill Analytical Laboratories, Corvallis, OR.

**Table 4-9 Organic Analysis Results of Oklahoma PRR<sup>ψ</sup>**

Analyte	GC-FID (mg/kg)	Qualifier	GC-MS (mg/kg)	Qualifier	EPA Regulated Maximum Conc. (ppm)
Phenanthrene	<55		4.1	J	-
Chrysene	118	J	160		-
Fluorene	55		<40		-
Fluoranthene	71		<40		-
Benzo(K)fluoranthene	<55		18	J	-
Benzo(a)pyrene	324	J	75		-
Ideno(1,2,3-Cd)pyrene	40		<40		-
Dibenz(a,h)anthracene	133	J	17	J	-
Benzo(g,h,i)perylene	270	J	74		-
Phenol	---		46		-
Benzoic Acid	---		87	J	-

Qualifier Descriptions: U = not detected at specified limits; J = estimated value.

<sup>ψ</sup>Courtesy of CH<sub>2</sub>M Hill Analytical Laboratories, Corvallis, OR.

#### 4.2.2.2 OSHD Standard for Water Sensitivity

The following is a summary of water sensitivity test results of specimens obtained from the Sand Springs Oklahoma Demonstration Project. The test compares the moisture sensitivity of a “conventional” asphalt mix (OK aggregates and asphalt emulsion—lane 13) to that of PRR-modified mixtures.

The test uses a change in resilient modulus that results from conditioning to indicate moisture sensitivity. Conditioning consists of water saturation followed by a freeze-thaw cycle and heating to 140°F (see Chapter 3 for procedure). Oregon State Highway Division’s (OSHD) standard test was used (see Appendix D). It should be noted that the procedure and the associated acceptance criteria (IRM = 70 percent) were developed for dense-graded asphalt concrete to be used in surface mixtures, not waste-

modified, subsurface, emulsion mixtures. Table 4-10 contains summary water-sensitivity data.

*Table 4-10 Summary of Modulus, Curing-Time, and IRM Values*

Sample ID	Curing Time (days)	Pre-Conditioning Modulus (psi)	Post-Conditioning Modulus (psi)	Hot or Ambient Conditioning	IRM (%)
11-5-C	0	58000	failed	hot	-
11-5-D	0	32000	failed	hot	-
12-5-C	0	30000	failed	hot	-
12-5-D	0	31000	failed	hot	-
13-5-C	0	136000	45000	hot	33.0
13-5-D	0	93000	34000	hot	36.6
11-5-A	31	197000	39000	ambient	19.8
11-5-B	31	195000	failed	hot	-
12-5-A	31	117000	24500	ambient	20.9
12-5-B	31	200000	failed	hot	-
13-5-A	31	318000	74500	hot	23.4
13-5-B	31	190000	70000	ambient	36.8

#### 4.2.2.3 Detailed Results

Two specimens from each of Lanes 11 through were evaluated for water sensitivity in an "as received" state (i.e., the specimens were not allowed any lab-curing time). The following description of results pertain to these samples:

1. Partial disintegration of specimens from lane 12 (lane 12 contained 6.5% emulsion) occurred during the vacuum-water process. The water in which the specimens were placed was rusty-brown in color by the end of this cycle.
2. No noticeable degradation occurred during the freeze cycle.

3. Within two hours of placing specimens in hot-water bath, specimens from lane 12 completely disintegrated. By the end of the 24-hour cycle, specimens from lane 11 (lane 11 contained 8.6% emulsion) had partially disintegrated (i.e., approximately one-third of the sample had sloughed off).
4. Lane 13 specimens incurred no noticeable degradation throughout conditioning and were the only specimens tested for a "conditioned" modulus.
5. The IRM using the average modulus for the two specimens tested from lane 13 is 34.7%. An IRM of less than 70% is typically considered as failing.

Two additional specimens from each of lanes 11 through 13 were again evaluated for water sensitivity. However, these specimens had been allowed to bench-cure in the lab for approximately 31 days prior to testing. One specimen from each lane was subjected to all the procedures outlined in Chapter 3, while the other specimen, from the same lane, was subjected to an ambient 22C (72°F) water bath for 24 hours, rather than the 60C (140°F) water bath for the same time duration. The following description of results pertain to these samples:

1. No noticeable degradation of any of the specimens occurred during the vacuum-water process. The vacuum-water appeared less murky than the vacuum-water of the previously tested samples.
2. No noticeable degradation of any specimens occurred during the freeze cycle.

3. By the end of the 24 hour hot-bath cycle, specimens from lanes 11 and 12 had partially disintegrated (i.e., approximately one-fourth of the samples had sloughed off).
4. The specimen from lane 13 incurred no noticeable degradation during hot-bath conditioning.
5. No noticeable degradation occurred to any samples conditioned in the ambient water-bath.

#### 4.2.2.4 TCLP Lead

The whole sample analysis indicated there to be 73 ppm of lead in the OK PRR. The resulting TCLP lead results for all combinations of mixtures composed from OK PRR were below the regulatory maximum of 5 ppm. Therefore, no further testing to reduce leachable lead was necessary.

## **5 DISCUSSION OF RESULTS**

Evaluating the physical properties of PRR-modified mixtures included, first and foremost, structural considerations. Lacking adequate strength would eliminate the option of incorporating PRR into a pavement structure. Notably different application procedures, as well as environmental concerns, exist between hot-mix and cold-mix processes. The advantages and disadvantages of hot mixing versus cold mixing will reveal the preferred method and application.

Given adequate time, stresses due to traffic loading, temperature fluctuations, and moisture susceptibility, combine to help break down pavement materials. Environmental implications resulting from such disintegration of a PRR-modified material are of concern.

This Chapter will discuss short-term and simulated long-term testing results pertaining to structural properties and the potential environmental implications of incorporating PRR-modified mixtures into pavement structures.

### **5.1 Physical Parameters**

#### **5.1.1 Structural**

Both laboratory and field applications demonstrate acceptable structural integrity of PRR-modified mixtures for use as base-course materials. Table 5-1 summarizes

physical testing results by comparing laboratory results with a "typical range," published in the 1993 AASHTO Design Guide. This Table will be referred to throughout this section.

**Table 5-1 Summary of Structural Results and Corresponding Typical Range**

	Measured Properties			Typical Range <sup>ψ</sup>	
Trial Pavement Structure	Resilient Modulus (psi)	Stability (lbs)	Flow <sup>ψψ</sup> (1/100-in)	Application and Range of Resilient Modulus (psi)	Application and Stability Range (lbs)
Hot Mixes					
1 PA dense-graded mix with 18% PRR and no AC (Table A1)	480,000	6,000	14	Hot-Mix AC 300,000-600,000	Hot-mix AC, heavy traffic 750-1,800 minimum
2 PA dense graded mix with 19% RR and 2% AC (Table A3)	500,000	5,300	13		
3 OK dense graded mix with 19% PRR and 2% AC (Table A4).	326,000	4,800	8		
Cold Mixes					
4 PA gap-graded mix w/ 17% PRR and 3.3% emulsion (Table A5).	25,000	660	43	Bituminous stabilized base 40,000-300,000	Bituminous stabilized base 100-750
5 OK gap-graded w/ 16% NRR and 8% emulsion (Table A6).	12,000	130	29		
6 OK TRR with 8% emulsion (see Tables A7 & A8).	176,000	NA	NA		
7 OK dense-graded mix with 15% NRR and 8.5% emulsion. Lane 11 (see Table A9).	65,500 (field cure) 196,000 (lab cure)	1,340 (field cure)	40 (field cure)		
8 OK dense-graded mix with 0% NRR and 7.5% emulsion. Lane 13.	161,000 (field cure) 254,000 (lab cure)	3200 (field cure)	29 (field cure)		

<sup>w</sup>AASHTO *Guide for Design of Pavement Structures*. AASHTO, 1993. pp. II24-II27.

<sup>ww</sup>Note: typical flow values for surface and base courses under medium traffic are from 8 to 16.

NA: not analyzed



Trial mixtures No. 4 and 5 (see Table 5-1) are adequate for subbase courses or engineered fill. These materials have reduced physical properties, due to the lack of aggregate-to-aggregate contact within the matrix. Therefore, gap-graded mixtures were not further evaluated. Trial mixture No. 6 is structurally suitable as a bituminous base. This mix design produced stronger physical properties from its field-mixed and compacted counterparts (see Table A9) because the TRR-emulsion mixture was allowed to air dry in the laboratory to a much lower moisture content (MC) than that of the mixtures in the field. For example, compaction MC of laboratory specimens were approximately 5% versus a MC of 14% for field compacted mixtures.

Results from trial mixtures No. 7 and 8 (see Table 5-1) were collected from Lane 11 and 13 of the Oklahoma Demonstration Project. The field-cured samples from Lane 11 (trial mixture No. 7) exhibited lower structural properties than the lab-cured specimens, due to prevailing wet weather conditions at the site during the monitoring time-span. Pavement structure No. 8 (the "typical" cold-mixed emulsified mixture containing no NRR) consisted of field cores collected from Lane 13. Strength parameters for this mixture exceed those of PRR-modified mixtures, both for field cured and laboratory-cured specimens.

Although the modulus and stability values of cold mixtures are significantly lower than for hot mixtures, they are within an acceptable range for the types of applications proposed. When used for lower pavement layers, they can provide good structural

support at reduced cost compared to hot mixes (see Economic Considerations in Section 5.3).

### *5.1.2 Gradation*

The dense gradations utilized in the laboratory and field applications successfully demonstrated their use as a stabilized base-course with PRR contents as high as 20%. However, gap-graded mixtures had significantly reduced strength parameters. Gap-graded mixtures have historically been recipe-specified.<sup>48</sup> For example, a recipe specification defines a mix in terms of the aggregate grading, mix composition and the method by which the mix should be manufactured, placed, and compacted. Recipe mixes are based on experience of known compositions which have performed well in practice. If variations in the components of the mix occur that are outside previous experience, there is no means of assessing what effect these modifications might have on the performance of the mixture. Attempting to combine significant quantities (i.e., greater than 10%) of inherently soft PRR material into a gap-graded matrix is a modification to a recipe that would have unknown implications. The cause of the resulting reduction of the strength parameters after the incorporation of PRR into the mixture is therefore difficult to assess. The inability of the PRR to sufficiently stiffen over time may induce a lubricating effect, and thus, limit interlocking between the aggregate. Therefore, dense-graded aggregate appear to be best suited for the incorporation of PRR.

### ***5.1.3 Evaluation of Hot versus Cold-Mixed PRR***

As previously discussed, a pavement structure can be broken down into a surface-course, followed by a treated base, and then an optional layer of engineered fill (subbase) that rests on the native soil (subgrade). Theoretically, PRR-modified materials could be substituted for any of these layers. However, there are advantages and disadvantages associated with the use of PRR in pavement structures, as identified in Table 5-2. From this summary, it appears the most favorable choice for PRR-modified mixes is cold-mixed base course or cold-mixed engineered fill (subbase). The primary advantages include the reduced potential for air emissions (as compared to a hot mix plant) and confinement to subsurface layers reducing the PRR-modified products exposure to traffic wear and the environment. In addition, this type of construction lends itself well to a wide range of sites, is technically simple, and the equipment and personnel are readily available.

Table 5-2 Advantages and Disadvantages of Hot vs. Cold-Mixing of PRR Products

Pavement Structure	Advantages	Disadvantages
Hot Mix Surface-Course	<ul style="list-style-type: none"> <li>• Extensive contractor and supplier experience</li> <li>• Widely dispersed facilities that manufacture hot mixes</li> </ul>	<ul style="list-style-type: none"> <li>• Acid and sulfur emission problems</li> <li>• Potential high exposure of clay fraction to moisture, and therefore, expansion of the mixture</li> <li>• Moisture-induced damage (i.e., raveling) may occur</li> <li>• Surface runoff susceptible to leaching constituents of residuals</li> <li>• Higher cost</li> </ul>
Cold Mix Surface-Course	<ul style="list-style-type: none"> <li>• Minimize air emissions</li> <li>• Suitable for remote applications</li> <li>• Lower cost</li> </ul>	<ul style="list-style-type: none"> <li>• Potential high exposure of clay fraction to moisture, and therefore, expansion of the mixture</li> <li>• Moisture-induced damage (i.e., raveling) may occur</li> <li>• Surface runoff susceptible to leaching constituents of residuals</li> </ul>
Hot Mix Base-Course	<ul style="list-style-type: none"> <li>• Large number of facilities manufacturing hot mixes</li> <li>• Protected from surface wear and harsh environment</li> <li>• Confinement minimizes material migration</li> </ul>	<ul style="list-style-type: none"> <li>• Acid and sulfur emission problems</li> <li>• Potential exposure of clay fraction to moisture, and therefore, expansion of the layer</li> <li>• Expansion may cause distress in surface-course</li> <li>• Higher cost</li> </ul>
Cold Mix Base-Course	<ul style="list-style-type: none"> <li>• Minimize air emissions</li> <li>• Easily applicable in remote locations</li> <li>• Protected from surface wear and harsh environment</li> <li>• Confinement minimizes material migration</li> <li>• Lower cost</li> </ul>	<ul style="list-style-type: none"> <li>• Expansion due to moisture may cause distress in surface-course</li> <li>• Surface course should be low permeability to limit moisture access to base</li> </ul>
Cold Mix Engineered Fill (subbase)	<ul style="list-style-type: none"> <li>• Minimize air emissions</li> <li>• Easily applicable in remote locations</li> <li>• Protected from surface wear and harsh environment</li> <li>• Confinement minimizes material migration</li> <li>• Layer expansion more tolerable than in upper layers</li> </ul>	<ul style="list-style-type: none"> <li>• Variability of PRR-modified material stiffness in wet and dry seasons may require adjustment of upper layer thicknesses and/or material specifications</li> </ul>

## **5.2 Environmental Considerations**

### **5.2.1 ECS Testing**

As noted in Chapter 4 and Figure 4-18 (gap-graded ECS testing results), the modulus ratios increased over the first two cycles, then decreased at the end of the third cycle. The increase in modulus over the first two cycles is likely due to densification caused by heating and loading of the specimen. The third cycle was a freeze cycle, not a hot cycle, as the SHRP protocol prescribes. This was an error in testing procedures but may help to explain the sudden drop in modulus at the end of the third cycle. A likely scenario would be that the sample expanded during freezing, due to the formation of ice crystals within the porous media, therefore, increasing the void content of the specimen. Additional voids would result in less aggregate-to-aggregate contact and therefore a lower modulus.

Relatively little, if any, water passed through either the dense or gap-graded specimens tested in the ECS. This is likely due to one or more of the following: (1) the dense-graded sample had only 3% air voids, (2) movement of PRR from the specimen caused blockage of the upper disc and platen, and (3) potential swelling of the PRR within the matrix may have inhibited the flow of water through the specimen.

### **5.2.2 OHSD Water Sensitivity**

Test results indicate that mixtures containing neutralized residuals are more sensitive to the final hot water conditioning than the conventional emulsion asphalt

mixes. However, it must be noted that even the conventional mix failed to meet the Oregon criteria of 70% index of retained modulus (IRM). This indicates that the aggregate source used for the Oklahoma Demonstration Project test sections has a moisture sensitivity (stripping) problem that is unrelated to the presence of PRR.

As mentioned in Chapter 3, aggregates from PA and OK were both crushed limestone. The combination of crushed limestone aggregates [consisting predominately of  $\text{Ca}(\text{CO}_3)_3$ ]<sup>49</sup> with lime-treated PRR, results in a PRR-modified mixture very high in alkaline earth. As illustrated in Figure 3-3, anionic emulsions are better suited for mixtures containing excessive amounts of alkaline earth materials. The fact that cationic emulsions were used might help explain the failing results observed in the water sensitivity experiment.

The conventional mixture had an average IRM of 35.5%, indicating a high degree of water sensitivity. The average IRM of the PRR-modified mixes (when tested using an ambient temperature bath) was 20.4%. Based on these results alone, neither of the mixes should be considered for use as a surface course. Either the conventional or the PRR-modified mixes could be considered for use as a base or subbase in a pavement structure, provided they could be isolated from moisture. However, the economics of implementation would make this option impractical. Water sensitivity of PRR-modified mixtures is of concern for the following reasons:

- Water-induced weakening followed by loading may cause degradation of the matrix and eventually result in migration of the residuals, or some component thereof.

- Swelling of the material could result, causing distress within other layers of the pavement structure.
- Swelling may promote loss of adhesion in the mixture.

Further study, to reduce the moisture sensitivity of these mixtures, is warranted. Using a anionic emulsion, in combination with treatment to reduce alkalinity should be considered.

### *5.2.3 Effect of Lime and Asphalt Emulsion on Lead Leachability*

This study evaluated the stabilization of lead in a characteristically toxic PRR. Namely, the PA PRR source, which was toxic due to a high lead content. This section discusses the findings from the stabilization treatability testing of this material. Treatability testing data are summarized in Appendix B.

As previously mentioned, the subject PRR originated from the site of a former refinery in Pennsylvania. The PRR contained almost one percent total lead, a TCLP lead value of 14 mg/L, and the pH measured from 1.8 to 4. Because the PRR did not contain 20 percent free water, this material was not classified as exhibiting the characteristic of corrosivity. The source of lead in the PRR was suspected to be primarily lead oxide, which was added to the crude oil in a sweetening process to remove sulfur odor by forming lead sulfide. The source of the acidity in the PRR was from the sulfuric acid added to polymerize unstable compounds. In spite of the high total lead content (approximately 8,970 ppm), the leachable lead in the TCLP was relatively low, at

approximately 14 ppm in a sample containing 100 percent untreated PRR. The TCLP specifies a 20-fold dilution of the matrix to be tested. Therefore, the maximum concentration that could be achieved (assuming 100% of the lead was soluble) would be near 450 ppm. Based on this observation, it was suspected that most of the lead in the PRR was present in low soluble specie such as lead sulfide or lead sulfate. Due to the 20 fold dilution, it is therefore, theoretically possible for a mixture containing 20 percent untreated PRR to have a TCLP lead result of near 3 ppm (i.e., 14 ppm times 0.2), 2 ppm below the regulatory maximum of 5 ppm. This may be construed as "dilution" by the EPA (i.e., adding only 20 percent PRR to arrive at the desired result of 3 ppm). Samples evaluated for lead leachability, in this study, contained a maximum of 20 percent treated PRR, as described in Chapter 3. An alternate approach may have consisted of minimizing leachable lead in 100 percent PRR samples by varying the lime content. If, at a certain percentage of lime, the leachable lead was found to consistently be below the regulatory maximum of 5 ppm, proceeding with recycling by incorporating varying percentages of PRR into pavement materials would not likely be considered as "dilution."

The majority of the PRR mixtures evaluated consisted of approximately 20 percent PRR with the remainder consisting of aggregate and asphalt. All mixtures prepared for TCLP analysis, other than control samples of 100 percent raw PRR, contained aggregate, emulsion, and PRR with a minimum lime content of 2.8 percent. Lime was added as a percentage of the dry PRR. Initially, hot-mix blends were evaluated using lime and hot asphalt. Of the specimens that were heated, the TCLP leachable lead was almost double that of the raw material that had not been heated (averaging



approximately 25 ppm). It was concluded that the heat adversely affected lead leachability and that hot mix asphalt recycling was not amenable to PRR that contains lead.

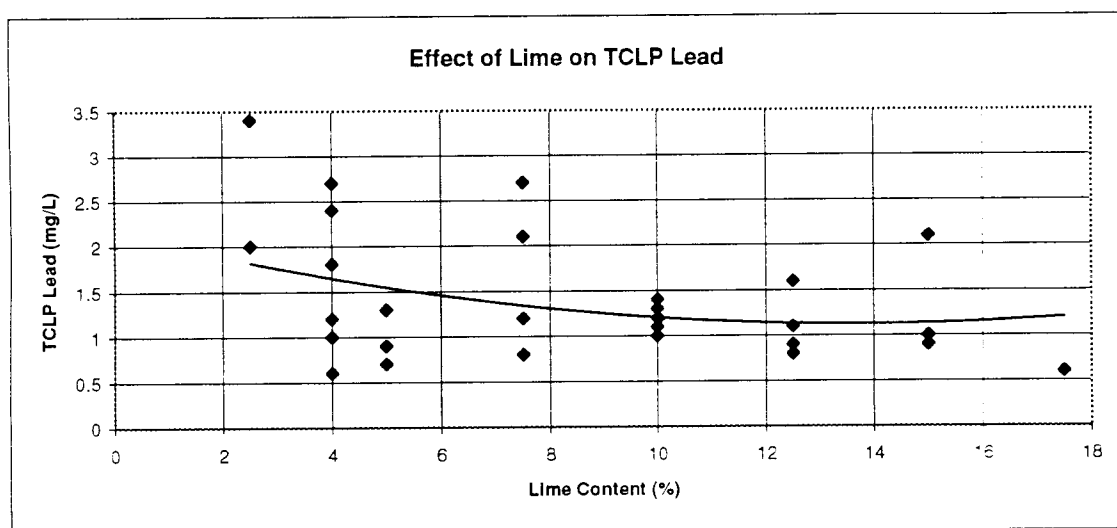
Cold mix asphalt blends were evaluated using asphalt emulsions. These mixtures were composed of 10 to 20 percent PRR (with lime) and 80 to 90 percent aggregate and asphalt. Mixtures were prepared at the lime, emulsion, and PRR percentages outlined in Table 4-3. Adding lime to the PRR was observed to have an effect on the lead leachability of the material, as shown in Figure 5-1.<sup>a</sup> Figure 5.2 indicates that asphalt emulsion had no statistically significant effect on reducing lead leachability. The spread of the data in both Figures 5.1 and 5.2 do not lend themselves well to the standard normal linear regression model. To justify a statement pertaining to a straight-line fit (normal linear regression model), the following features must be present within the data sets:<sup>50</sup>

1. Linearity—The plot of response means against the explanatory variable is a straight line.
2. Constant Variance—The spread of the responses around the straight line is the same at all levels of the explanatory variable.

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<sup>a</sup> It should be noted that these data were not collected exclusively for determining the effect added lime or asphalt would have on lead leachability, but rather, the effect that the combination of lime and asphalt on lead leachability.

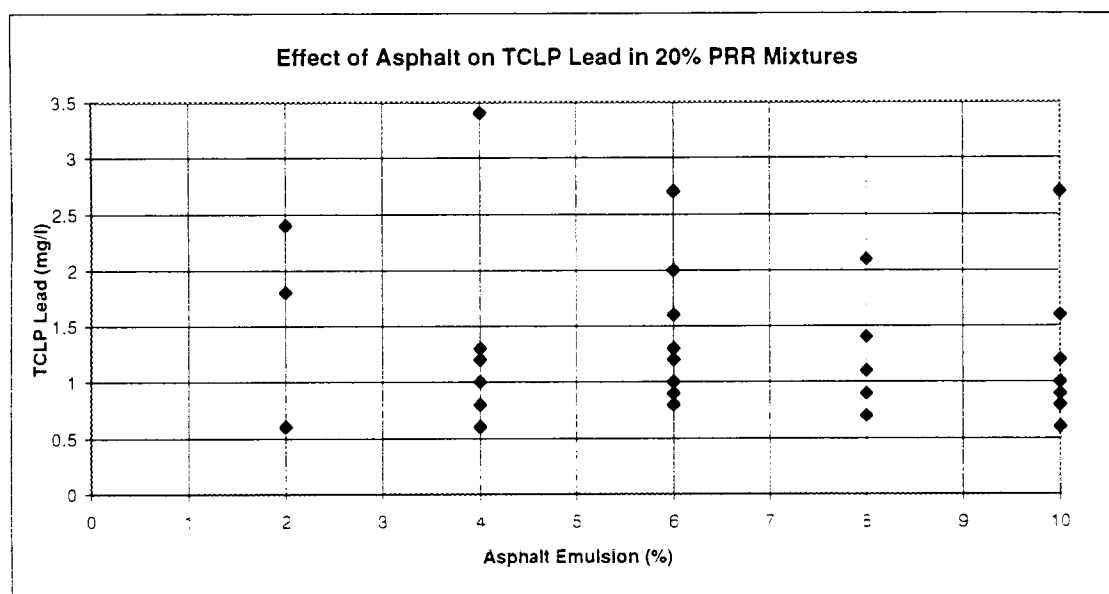
3. Normality—The populations of responses at the different values of the explanatory variable all have normal distributions. In other words, response values should be concentrated around the mean of any given data set (i.e., Figure 5-1 at 10% lime).



**Figure 5-1** *Effect of Lime on Lead Leachability*

It is visually apparent, in Figure 5-1, that if a line were drawn to connect the means of each data set (i.e., the mean at each lime content), a straight line would not result. It may be even more apparent that there is lack of constant variance in the data plotted in Figure 5-1: the spread of the TCLP lead levels are not the same at all lime contents. Lastly, normality appears only pertinent to the data set at 10% lime in Figure 5-1 and not at all relevant to the data in Figure 5-2. For these reasons, it is not possible to conclude that there is a statistically significant relationship among these data (i.e., for a

normal distribution). However, one could hypothesize about the visual significance of this data plot. The data set at 10% lime, in Figure 5-1, shows tightly grouped data compared to data at higher and lower lime contents. The curvilinear plot superimposed in Figure 5-1 is a best fit trend-line performed by Microsoft Excel version 5.0.



**Figure 5-2** *Effect of Asphalt on Lead Leachability*

These results illustrate the complexing behavior common among heavy metals, as discussed in Chapter 2. Different complexes are known to have varying degrees of solubility depending on the pH of the solution, with a minimum solubility occurring at some optimum pH. This may be the phenomena observed in Figure 5-1, however, the abscissa has units of lime content rather than pH.

Things become increasingly more complicated when the matrix considered is not a solution, but a solid, such as PRR. A solid does not lend itself well to stoichiometric calculations. The interaction of metals with soils is very complex. Adsorption and ion exchange by clay minerals, reaction with insolubilizing anions present in the soil, and complexation by humic substances in the organic fraction of the soil all occur.<sup>13</sup>

Complexation may lead either to increased or decreased solubility of the metal. In soils, soluble metal salts are often distributed throughout large, hard pieces of clay and porous rock. This may have occurred over a period of many years, and outward diffusion of the species so that it can react with fixation agents (i.e., lime) can be very slow.

#### *5.2.4 Long-Term Stability of Chemically Fixed Constituents*

Much concern has been expressed by environmentalists and regulators about the long-term stability of fixed species. Certain leaching tests (i.e., TCLP) are said to be equivalent to tens of hundreds of years of natural leaching action in the environment.<sup>51</sup> However, at this time, no actual long-term data are available because the technology has only been practiced for about 25 years. Nevertheless, except for recovery, there is no alternative to chemical fixation and solidification technology for management of hazardous metals.

It should be pointed out that slow leaching of metals at a controlled rate is not detrimental to human health and the environment. The real concern is when a sudden release of contaminants occur, due to the breakdown of the matrix. This could occur, for

example, when the fixation mechanism is pH controlled and the buffering action of the alkali is finally used up in an acidic environment.<sup>13</sup>

The fate of heavy metals, organics, and polynucleated aromatic hydrocarbons (PAH) are of primary concern in aquatic environments. Many constituents within these categories tend to partition to organic matter and resist degradation. Toxicity occurs when a receptor (an organism) is exposed to high concentrations for a sufficient duration that it elicits an adverse response.<sup>13</sup> One might ask "what is considered 'high' concentrations?".

The bioassay technique is often used to determine the toxicity of certain chemicals on organisms, biological systems, or biological processes. A bioassay may be defined as "the laboratory exposure of organisms to field-collected environmental samples for the purpose of identifying actual or potential toxic effects on resident species".<sup>43</sup> Harmful concentrations are typically defined as "the dose that is lethal to 50 percent of the population exposed (or LD<sub>50</sub>)".

Tables 4-5, 4-6, and 4-7 illustrate the TCLP results of two samples tested for metals, organics, and polynuclear aromatic hydrocarbons. The samples tested include: 1) a 'typical' asphalt concrete mixture (extracted from another study);<sup>46</sup> and (2) a PA PRR modified-product from this study. As can be seen in Table 4-5, no metals were reported above the detection limit (DL) for the AC-20 mixture. Only cadmium and lead were reported above the DL, yet remained below the maximum regulatory level, for the PRR-modified mixture. Tables 4-6 and 4-7 show levels below the DL, in most cases, and far below the maximum regulatory concentration (where applicable), in all cases. These

results strongly suggest that the PRR-modified product is no more or less harmful to the environment than a typical asphalt concrete.

### **5.3 Project Summary**

#### **5.3.1 Structural**

Test results clearly show that hot mixes are well within the modulus and stability range expected for conventional paving mixtures. The hot mixtures are considerably stiffer than cold mixtures. This difference is in part caused by the ability of the hot mix process to dry the materials thoroughly while mixing with hot asphalt cement. Upon compaction and cooling, the resulting mixture is a dense and stiff paving material. However, there are disadvantages that tend to preclude the selection of hot mixes for field use. As previously mentioned, possibly harmful emissions are expelled from the PRR upon heating, necessitating the use of a breathing apparatus for any persons in the vicinity of the heated PRR. Also, it appears that heating of the PRR causes increased lead leachability, as discussed in Section 5.2. Furthermore, ECS testing revealed a high degree of water sensitivity, as explained in Chapter 4. It is therefore indicative that the focus remain on cold mixtures.

PRR-modified mixtures were found to have sufficient strength parameters to be considered structurally suitable when used as a stabilized base. However, this study did not show a marked improvement of PRR-modified materials over non-modified mixtures. The Oklahoma Demonstration Project revealed optimum emulsion contents for non-PRR-

modified and PRR-modified mixtures to be approximately 7.5% and 8.6%, respectively (a 1.1% increase). Strength parameters were also higher for non-PRR-modified over PRR-modified materials. This apparent lack of improvement over a typical bituminous-treated base would place the PRR-modified material in the category of “sham recycling,” as described in Chapter 2 (i.e., “is the PRR-modified material an improvement or analogous to the raw material or product it replaces?”). The question remains; if a waste material can be recycled into a useful product, though not identical to that of which it replaces, but adequate for the intended purpose, should it be permitted as an alternate to other disposal options? If PRR-modified products are established as economically and environmentally suitable as an alternative to conventional bituminous stabilized base materials, it would be better that the monetary investment relinquished for treating the PRR contribute to strengthening our infrastructure instead of adding to our ever increasing number of landfills.

Some questions must be answered by the regulatory agencies implementing environmental and/or recycling policies. However, the regulatory agencies adhere to policies established by the Federal Government, which is made up of our State Representatives, whose decisions, in theory, reflect the opinions and interests of the general public.

### *5.3.2 Constructability*

The greatest challenge for successful field implementation of these cold mixtures is proper curing and drying of the PRR, given the high moisture content of the PRR

sources evaluated in this study. In order to compact the mixture, water must be evaporated. This can be accomplished at different stages of the construction process, such as aeration of the PRR prior to mixing and/or after mixing with asphalt emulsion. Compaction tends to seal the surface and inhibits evaporation. However, given sufficient time after compaction (say 2 to 3 weeks of hot, dry weather), the material may attain adequate strength, depending on the predicted loading. Warm, dry weather is an important factor in the cold-mix process that will determine the beginning and duration of the construction season.<sup>45</sup>

### *5.3.3 Economic Considerations*

The use of PRR-modified materials in pavements can result in cost savings compared to the use of conventional road building materials. Initial construction cost savings will depend on equipment availability, the specific combination of materials selected, where in the pavement the PRR-modified material is used, and local construction costs. This section examines several construction scenarios and offers an approach to evaluating economic considerations.

The combinations of PRR and asphalt emulsion, tested in this study, demonstrate that PRR-modified mixes can be used as a replacement for conventional base or subbase. Typical material costs for NRR and TRR-modified products are developed in Tables 5-3 and 5-4.<sup>2</sup> Table 5-5 lists various types of materials with corresponding values of resilient modulus and in-place unit costs of all materials. For example, the NRR-modified base cost of \$1.69 per SY-in includes excavation, neutralization, adding asphalt emulsion and



aggregate, hauling (up to 50 miles), and placement costs. PRR-modified mixtures are structurally superior to aggregate bases and subbases. The following economic analysis compares an aggregate base and a bituminous stabilized base with a PRR-modified product.

**Table 5-3 Detailed Cost Estimate for NRR-Modified Product**

<b>Estimated Cost for Product Containing: 15% NRR, 8% Emulsion, and 77% Aggregate</b>		
<b>Activity</b>	<b>Unit Cost (\$/CY)</b>	<b>Comments</b>
Excavate and neutralize PRR	17.50	Based on OK Project
Aggregate (base rock 1.8 Ton/CY)	13.00	77% aggregate by weight
Emulsion (based on 1.35 Ton/CY)	202.50	Based on KOCK purchase for Demonstration Project
Mixing and handling cost	10.00	With loader, pug mill, oiler, and 3 operators
Transportation cost	7.50	Within 50 mile radius, using a dump truck (\$0.15/CY/mi)
Placement cost	11.00	Dump truck, paver, compactor
<b>Total</b>	<b>\$61/CY or \$32/TN</b>	Based on 1.9 tons/CY

*Table 5-4 Detailed Cost Estimate for TRR-Modified Product*

<b>Estimated Cost for the Product: TRR with 10% Asphalt Emulsion</b>		
<b>Activity</b>	<b>Unit Cost (\$/CY)</b>	<b>Comments</b>
Excavate and treat PRR	44.00	Based on OK Project
Emulsion cost (based on 1.35 ton/CY)	202.50	Based on KOCK purchase for Demonstration Project
Mixing and Handling cost	10.00	With loader, pug mill, oiler, and 3 operators
Transportation cost	7.50	Within 50 mile radius, using a dump truck (\$0.15/CY/mi)
Placement cost	11.00	
<b>Total</b>	<b>\$87/CY or \$67/TN</b>	Based on 1.3 tons/CY

*Table 5-5 Typical Modulus Values and Unit Costs of Various Pavement Materials*

<b>Type of Material</b>	<b>Typical <math>M_r</math> (psi)</b>	<b>Unit Cost (\$/SY-inch in place)</b>
Hot Mix Asphalt (HMA)	1,000,000	1.72
Typical Bituminous Stabilized Base	40,000-300,000 (assume 100,000)	1.44
Typical Base	25,000	0.92
Typical Subbase	10,000	0.75
NRR-modified base 15% NRR, 8% Emulsion, and 77% Aggregate	100,000	1.69
TRR-modified base 90% Treated Residuals and 10% Emulsion	100,000	2.42
Typical Subgrade	6,000	-

Attempting to evaluate the economic advantages of incorporating a PRR-modified bituminous base into a pavement structure is complicated by empirically-based design

tools. The AASHTO Design Guide requires the use of structural coefficients, which are extrapolated from empirically developed plots that correspond to measurable structural parameters, such as resilient modulus and stability. For example, a granular base with a modulus of 30 ksi corresponds to a structural coefficient of 0.14 which corresponds to a bituminous treated base with a 125 ksi modulus. In other words, the AASHTO Guide defines a granular subbase with a 30 ksi modulus to be equivalent to a bituminous treated base with a modulus of 125 ksi. This apparent anomaly is likely due to a problem with the fundamental regression equations on which the aforementioned plots were derived.

The ninth edition of the Asphalt Institutes Manual Series #1 (MS1) is based on mechanistic-empirical methodology. Mechanistic multilayer theory is used in conjunction with empirical failure criteria to determine pavement thicknesses.<sup>52</sup> This method was used to design typical pavement sections for low and moderate traffic loading. The key premise on which these designs rely is that the base materials generated as a result of this technology are characterized as a “Type II—Emulsified asphalt mix made with semi-processed, crusher-run, pit-run, or bank-run aggregates”. Pavement structures and costs are shown in Table 5-6.

**Table 5-6 Typical Pavement Sections Constructed with and without PRR-Modified Materials**

Pavement Structure Composition	Low Traffic Level (50,000 EAL)	Low Traffic Level (50,000 EAL)
Pavement Without PRR-modified materials	4" of asphalt concrete over 6" of aggregate base	2" of asphalt concrete over 4" of bituminous stabilized base
<b>Cost (\$/SY)</b>	<b>12.40</b>	<b>9.20</b>
Pavement With NRR-modified materials	2" of asphalt concrete over 4" of NRR-modified base	2" of asphalt concrete over 4" of NRR-modified base
<b>Cost (\$/SY)</b>	<b>10.20</b>	<b>10.20</b>
Pavement With TRR-modified materials	2" of asphalt concrete over 4" of TRR-modified base	2" of asphalt concrete over 4" of TRR-modified base
<b>Cost (\$/SY)</b>	<b>13.12</b>	<b>13.12</b>
<b>Cost Difference with:</b>		
<b>NRR materials,</b>	<b>2.20 \$/SY</b>	<b>-1.00 \$/SY</b>
<b>TRR materials</b>	<b>-0.72 \$/SY</b>	<b>-3.92 \$/SY</b>

The forgoing economic analysis assumes that the recycled NRR and TRR-modified products will replace an aggregate or bituminous stabilized base. The cost difference realized between each option is shown in the bottom row of Table 5-6. The only scenario that results in cost savings is the use of a NRR-modified base in place of a typical aggregate base (i.e., cost difference of \$2.20/SY). This may not be considered a realistic comparison because it compares an aggregate base with an NRR-modified bituminous stabilized base (i.e., compares apples with oranges). A more accurate representation is made in the third column where the NRR and TRR-modified materials are compared with a "typical" bituminous stabilized base. Increased costs associated

with using NRR and TRR-modified materials over a “typical” bituminous stabilized base are \$1.00/SY and \$3.92/SY, respectively. This assumes that the “typical” bituminous base would have an equivalent modulus as that of the PRR-modified base of 100,000 psi.

It seems apparent that in order for the PRR-modified materials to be used in full-scale applications the additional cost would have to be subsidized. This subsidy would be the expense incurred for remediation by the owner/producer of the PRR-modified product. To evaluate whether this method of remediation would have greater economic incentive over other treatment/disposal methods requires further analysis.

#### *5.3.4 Economic Analysis of Incineration versus Recycling*

Table 5-7 puts forth a relative cost comparison between incineration and the PRR-modified base products developed in this study. The cost of incineration can vary from \$100/ton, for wastes designated as non-hazardous, to \$450/ton. for hazardous wastes.<sup>7</sup>

The NRR-modified product contains 77 percent aggregate, 15 percent NRR, and 8 percent asphalt emulsion. The unit cost of processing the NRR-modified product is high because the end product contains only 15 percent PRR. Therefore, nearly seven tons of the processed product need to be produced to utilize one ton of PRR. The unit cost for processing and the market value of the processed product reflect this multiplicative factor.

NRR and TRR are two PRR materials containing varying quantities of lime, as defined in Chapter 3. The amount of lime contained in the NRR is considered negligible compared with that contained in the TRR. The unit cost for processing the TRR-modified product is based on the following approximate proportions: 60 percent PRR, 30

percent lime, and 10 percent asphalt emulsion. Containing only 60 percent PRR, approximately 1.7 tons of finished product would have to be produced to utilize one ton of PRR. Both the NRR and TRR-modified products could possibly be marketed as a bituminous stabilized base material, valued at approximately \$29 per ton (based on current local market conditions).<sup>53</sup>

*Table 5-7 Economic Analysis of Incineration versus PRR-Modified Products*

<b>Treatment Option</b>	<b>Unit Cost for Processing each Ton of PRR (\$/ton)<sup>†</sup></b>	<b>Market Value of Processed Product (\$ realized per ton of processed PRR)<sup>††</sup></b>	<b>Difference [Processing - Market Value] (\$/ton of PRR) = Remedial Cost Realized</b>
Incineration	100-450	NA	100-450
NRR-Modified Base Product	214	193	21
TRR-Modified Base Product	111	48	63

NA: not applicable

<sup>†</sup>Unit cost per ton of processed product divided by percentage of PRR in mixture.

<sup>††</sup>\$29.00/ton (local market value of bituminous stabilized base) divided by the percentage of PRR in the mixture.

It is apparent that the most economical remedial option is recycling, keeping in mind that this analysis only compares relative costs between incineration and the recycling alternatives defined in this study. Clearly, the remedial action that reveals the optimum economic conditions requires the evaluation of all possible corrective action and is highly site specific.

### 5.3.5 *Environmental*

Environmentally there are two primary factors that critically affect recycling: (1) hazardous classification of the material, and (2) hazardous constituents that require treatment. A PRR classified as toxic (a heavy metal exceeding the TCLP concentration) will be most favorable for recycling. A PRR classified as corrosive may be more difficult to recycle because of the special regulatory approval required, and the need to meet more stringent treatment requirements.

Attempts to stabilize a recycled PRR focused on reducing lead leachability. Using hot mix application significantly increased lead leachability. Lime and cement have proven effective in reducing lead leachability, with lime being more effective and economical. If other hazardous constituents are identified as requiring treatment, then additional treatability evaluations would be necessary to determine effective treatment additives that do not adversely affect the structural properties of the recycled product.

## **6 CONCLUSIONS AND RECOMMENDATIONS**

### **6.1 Conclusions**

This project demonstrates that petroleum refinery residuals can be successfully incorporated into pavement structures, through laboratory evaluation of the two PRR sources acquired and a trial field project.

Considering the early origin of PRR (i.e., containing all the crude but illuminating oils), it is likely that early deposits, in regions where native crudes were known to contain potentially significant amounts of bituminous material, have significant recycling potential. The estimated number of sites where large quantities of PRR are known to exist, justify research into alternative applications of this material. Many PRR sources may prove to be suitable for recycling only after appropriate evaluations are conducted. However, each source of PRR is essentially unique and must be evaluated and judged for its recycling potential on its own merit.

This studies findings and literature review indicate that the chemical nature of certain neutralized PRR are no more of an environmental threat than existing asphalts and emulsions. Combined with Fuller's Earth materials, PRR often exhibits characteristics similar to a nonswelling clay mineral that preferentially bonds with water molecules over hydrocarbons. Therefore, PRR modified pavement materials have a higher degree of water sensitivity than conventional pavement materials.



The OK PRR evaluated in this study would not be classified as either a characteristically toxic or corrosive waste. PA PRR, on the other hand, would be classified a characteristically toxic (D008) hazardous waste based on lead levels exceeding the regulatory maximum of 5 ppm.

The addition of 5 percent lime proved sufficient in neutralizing both PRR sources to acceptable pH levels and reduced lead leachate, in samples that contained 20% and less PRR, to below the regulatory limit of 5 ppm. The addition of cement to the PA PRR did not reduce lead levels to below 5 ppm.

Based on the data collected, a linear relationship could not be established between lime or emulsion content and leachable lead. However, lead leachate versus percent lime reveals a visual curvilinear relationship that closely resembles the complexing characteristics of many heavy metals.

The optimum PA PRR and AC contents for hot dense graded mixtures were determined to be approximately 18% and 2%, respectively. The optimum emulsion content of the mixtures prepared using TRR were established to be approximately 10%. The optimum OK PRR and emulsion contents in a cold-mixed dense graded mixture were found to be approximately 14% and 9%, respectively (lane 11 in OK Demonstration Project). The optimum emulsion content for a non-modified cold mix was determined to be approximately 7.5% (lane 13 in OK Demonstration Project).

Structural parameters for the non-modified dense-graded mixture (lane 13 of Demonstration Project) exceed those of the optimum dense-graded PRR-mixture (lane 11

of Demonstration Project) even though the non-modified mixture contains approximately 1.5% less asphalt emulsion than the PRR mixture. However, strength parameters of PRR-modified mixtures are within acceptable limits for use as a stabilized base or engineered fill and can be constructed using conventional heavy-duty equipment.

If PRR-modified materials were subsidized to make them less expensive than conventional materials, the cost to remediate a site may be less than alternate disposal technologies, such as incineration. Incorporating PRR into pavement structures would serve a dual purpose; 1) recycling offers supplemental materials to a limited supply of raw resources and 2) compared with the expense and ultimately unusable end products of alternative disposal options, recycling PRR into pavement structures contributes to a much needed component of the infrastructure.

## 6.2 ***Recommendations***

The use of this technology requires certain site-specific conditions to be met, including limited variance of the physical state of the PRR (i.e., constituting less than 20 percent free water for “non-corrosive” classification) and quantity of waste present at any particular site. The application of this technology will require regulatory as well as public acceptance of the PRR-modified materials. This is not a simple task to accomplish, and in fact, may prove much more time consuming and costly than refining the technology for potential full-scale implementation. Prior to further consideration of a large-scale project that would utilize a PRR-modified material, the following recommendations are set forth for additional study:

- ◆ The determination of a typical PRR gradation.
- ◆ The effect of lime on the fixation of lead in 100 percent PRR mixtures.
- ◆ Effects of using an anionic versus cationic emulsion.
- ◆ Long-term stability of environmentally questionable constituents.
- ◆ The effects of long-term loading and a harsh environment on the durability of a PRR-modified product.
- ◆ Further evaluation of the water sensitivity of PRR-modified products and the implications of Fuller’s Earth minerals on mixture properties.

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## ***APPENDICES***

***APPENDIX A: Physical Testing Data Tables***



**LEGEND** ID - PRR SOURCE-TYPE OF MIX (dense or gap)-SAMPLE NUMBER\_% PRR\_%AC  
PRR - PETROLEUM REFINERY RESIDUALS

Table A1 Dense Graded Hot Mixtures Utilizing 16% PA PRR and Excluding AC

ID	Agg. Mass (g)	PRR Mass <sup>12</sup> (g)	Time in 125C Pre-comp. Oven (min)	Compaction Effort		Gmb	Gmm	Air Voids (%)	Thickness (in)	Mr (ksi)	Stability (kips)	Correlation Ratio	Corrected Stability (kips)	Flow (1/100-in)	COMMENT(S)
				Stage 1 (#blows/psi)	Stage 2 (#blows/psi)										
TARGET	1080.0	100.0													
PAD1_8.5_0	1072.8	99.8	30	-	-	-	2.24	-	-	-	-	-	-	-	
PAD2_8.5_0	1076.2	100.1	30	20/250	150/400	1.84	-	17.8	2.925	FAILED	-	-	-	-	Failed
TARGET	896.1	83.0													Adjustment for specimen height.
PAD3_8.5_0	891.6	82.9	30	20/250	150/400	1.75	-	21.6	2.210	FAILED	-	-	-	-	Failed
PAD4_8.5_0	892.0	83.0	30	20/250	150/400	1.89	-	16.6	2.404	17	-	1.04	-	-	Disintegrated prior to stability and flow testing.
PAD5_8.5_0	891.4	83.1	30	20/250	150/400	FAILED	-	-	-	-	-	-	-	-	Failed
Average						1.83		18.7	2.513	17					
TARGET	1063.2	200.0													
PAD1_16_0	1060.7	200.4	30	20/250	150/500	2.02	-	4.0	2.860	519	5.7	0.81	4.6	10	Time variability in pre-compaction oven. Heated in 60C oven 2-hrs for Stability and Flow
PAD2_16_0	1060.8	199.8	38	20/250	150/500	2.00	-	5.1	2.917	437	8.9	0.78	6.9	20	" " "
PAD3_16_0	1060.8	200.3	63	20/250	150/500	1.85	-	12.1	3.241	90	4.5	0.76	3.4	6	" " "
PAD4_16_0	1060.8	200.4	45	20/250	150/500	1.87	-	11.3	3.223	-	-	-	-	-	Sample saved
PAD5_16_0	1060.4	200.2	35	20/250	150/500	2.02	-	4.2	2.955	485	8.7	0.78	6.7	12	" " "
PAD6_16_0	1060.8	199.7	30	-	-	-	2.10	-	-	-	-	-	-	-	Maximum theoretical specific gravity sample
Average		200.1				2.58		10.9	3.039	383	7.0		5.4	12	
TARGET	840.0	302.1													
PAD1_26.5_0	837.5	302.1	30												Too much RR---sample destroyed in compaction.
TARGET	840.0	232.7													
PAD1_21.8_0	837.5	232.8	30	20/250	150/400	1.96	-	7.2	2.562	508	6.6	0.96	6.3	10	Specimen tested for Stability & Flow after 2 hours in 60 C oven.
PAD2_21.8_0	836.7	232.3	30	-	-	-	2.11	-	-	-	-			-	
PAD3_21.8_0	836.3	232.7	30	20/250	150/400	1.96	-	7.1	2.591	394	6.9	0.76	5.2	9	Specimen tested for Stability & Flow after 2 hours in 60 C oven.
PAD4_21.8_0	836.6	232.7	30	20/250	150/400	1.95	-	7.7	2.504	472	3.5	1	3.5	11.2	Specimen tested for Stability & Flow after immersion in water bath for 30 min..
Average		232.6				1.96		7.3	2.552	458			5.0	10.1	

<sup>1</sup>PRR dry mass is based on an estimated moisture content at time of mixing of approximately 20%.  
<sup>2</sup>Contains approximately 3% lime by dry weight of PRR (approximate pH of 5).

<b>LEGEND</b>	ID - PRR SOURCE-TYPE OF MIX (dense or gap)-SAMPLE NUMBER_% PRR_%AC
	PRR - PETROLEUM REFINERY RESIDUALS

Table A2 Hot Mixed Gap Graded Specimens with Varying Percentages of PA PRR Excluding AC

ID	Agg. Mass (g)	PRR Mass <sup>1,2</sup> (g)	After Mix Temp. (C)	Time in Pre-Comp. Oven @ 125C (min)	Gmb <sup>3</sup>	Gmm	Air Voids (%)	Thickness (in)	Mr (ksi)	Stability (kips)	Correlation Ratio	Corrected Stability (kips)	Flow (1/100-in)	COMMENT(S)
TARGET	842.1	200.5												
PAG1_19_0	841.3	200.7	88.2	30	-	2.14	-	-	-	-			-	
PAG2_19_0	841.5	200.4	87.0	30	1.81	-	15.5	2.646	44	DISINTEGRATED				Separation & crumbling of compacted surface.
PAG3_19_0	841.3	200.3	81.7	30	1.92	-	10.5	2.486	37	1.6	1.00	1.6	8.8	Separation & crumbling of compacted surface.
PAG4_19_0	841.1	201.0	84.0	30	1.88	-	12.1	2.554	43	2.1	0.96	2.0	6.0	Separation & crumbling of compacted surface.
Average	841.3	200.6	85.2		1.87		12.7	2.562	41			1.8	7.4	
TARGET	805.0	255.5												
PAG1_24_0	804.3	255.3	90.7	30	2.03	-	4.5	2.395	123	2.4	1.07	2.6	12.0	Specimen tested for Stability & Flow after immersion in water bath for 30 min..
PAG2_24_0	803.7	255.7	83.0	30	2.05	-	3.7	2.392	283	4.5	1.09	4.9	7.5	Specimen tested for Stability & Flow after 2 hours in 60 C oven.
PAG3_24_0	804.8	254.8	84.0	30	-	2.13	-	-	-	-	-	-	-	
PAG4_24_0	804.4	255.6	87.9	30	2.05	-	3.8	2.376	252	4.3	1.09	4.7	8.5	Specimen tested for Stability & Flow after 2 hours in 60 C oven.
Average	804.3	255.4	86.4		2.04		4.0	2.388	219			4.1	9.3	
TARGET	841.3	311.6												
PAG1_27_0	843.4	311.5	83.6	30	2.09	-	0.0	2.520	550	5.4	0.98	5.3	9.2	Pumping during compaction.
PAG2_27_0	841.1	312.2	85.7	30	-	1.98	-	-	-	-	-	-	-	
PAG3_27_0	841.3	311.1	83.5	30	2.10	-	0.0	2.459	407	3.4	1.02	3.5	14.8	Excessive pumping during compaction.
PAG4_27_0	841.1	311.5	83.0	30	2.09	-	0.0	2.477	438	4.6	1.02	4.7	10.4	Pumping during compaction.
Average	841.7	311.6	84.0		2.09		0.0	2.485	465			4.5	11.5	

<sup>1</sup>PRR dry mass is based on an estimated moisture content at time of mixing of approximately 5%.

<sup>2</sup>Contains approximately 3% lime by dry weight of PRR (approximate pH of 5).

<sup>3</sup>Sample compaction effort per Hveem procedure: Stage 1 = 20 blows @ 250 psi; Stage 2 = 150 blows @ 400 psi; Stage 3 = leveling load @ approx. 1000 psi.

<b>LEGEND</b>	ID - PRR SOURCE-TYPE OF MIX (dense or gap)-SAMPLE NUMBER_% PRR_%AC
	PRR - PETROLEUM REFINERY RESIDUALS

Table A3 Hot Mixed Dense Graded Specimens Utilizing 17% PA PRR and 1-3% AC

ID	Agg. Mass (g)	PRR Mass <sup>1,2</sup> (g)	AC Mass (g)	Time in Pre-Comp. Oven @ 125C (min)	Compaction Effort		Gmb	Gmm	Air Voids (%)	Thickness (in)	Mr (ksi)	Stability (kips)	Correlation Ratio	Corrected Stability (kips)	Flow (1/100-in)	COMMENT(S)
					Stage 1 (#blows/psi)	Stage 2 (#blows/psi)										
TARGET	900.0	214.3	11.3													
PAD1_19_1	897.5	214.4	11.3	30	20/250	150/400	1.96		11.6	2.665	358	3.91	0.89	3.48	10.4	High initial compactive effort during stage 2. Water heated for stability & flow.
PAD2_19_1	897.6	214.3	11.3	30	20/250	150/400	1.96	-	11.5	2.624	544	4.79	0.93	4.45	7.2	Asphalt foamed in all specimens when placed on RR. Water heated for stability & flow.
PAD3_19_1	897.0	214.3	11.3	-	-	-	-	2.21	-	-	-	-	-	-	-	-
PAD4_19_1	896.4	214.1	11.5	30	20/250	150/400	1.98		10.6	2.630	472	4.29	0.93	3.99	13.6	Water heated for stability & flow.
Average	897.1	214.3	11.4				1.97		11.2	2.640	458			3.97	10.4	
TARGET	860.0	204.8	21.5													
PAD1_19_2	857.7	204.3	21.4	30	20/250	150/400	2.02	-	8.9	2.47	460	4.40	1.02	4.49	11.6	Water heated. Sample was broken up and delivered to CH2M Hill for TCLP testing.
PAD2_19_2	857.3	204.7	21.6	30	-	-	-	2.16	-	-	-	-	-	-	-	Asphalt foamed in all specimens when placed on RR.
PAD3_19_2	857.7	205.0	21.6	30	20/250	150/400	2.02	-	8.6	2.453	493	4.73	1.04	4.92	15.2	Slight pumping during compaction. Water heated for stability & flow.
PAD4_19_2	857.3	204.6	21.7	30	20/250	150/400	2.00	-	9.6	2.501	546	6.49	1.00	6.49	12.0	Dry heated in water for stability. Sample vacuum-washed* for TCLP water sample.
Average	857.5	204.7	21.6		0.0	9.0	2.475		500			5.36	12.9			
* Vacuum of 29-in Hg for 1-hr in distilled water @ 25 C.																
TARGET	860.0	204.8	32.3													
PAD1_19_3	857.6	204.6	32.6	30	-	-	-	2.13	-	-	-	-	-	-	-	Water turned blackish-yellow.
PAD2_19_3	857.7	204.2	32.2	30	20/250	150/350	2.07	-	6.3	2.396	429	3.74	1.12	4.19	21.6	Filter disintegrated during compaction. Pumping during compaction. Water heated.
PAD3_19_3	857.9	204.0	32.3	30	20/250	150/350	2.10	-	5.1	2.372	312	3.57	1.13	4.03	12.8	Dry heated in water for stability & flow.
PAD4_19_3	857.8	204.7	32.6	30	20/250	150/350	2.10	-	5.1	2.269	271	2.31	1.15	2.66	14.4	Excessive pumping during compaction. Water heated for stability & flow.
Average	857.8	204.4	32.4				2.09		5.5	2.346	337			3.63	16.3	

<sup>1</sup>PRR dry mass is based on an estimated PRR moisture content of approximately 5% at time of mixing.

<sup>2</sup>Contains approximately 3% lime by dry weight of PRR (approximate pH of 5).

TABLE A4 Hot Mixed Dense Graded Specimens Utilizing 17% Oklahoma PRR & 2% AC

ID	Agg. Mass (g)	PRR Mass <sup>1,2</sup> (g)	AC Mass (g)	Gmb <sup>3</sup>	Gmm	Air Voids (%)	Avg. Thick. (in)	Avg. Mr (ksi)	Hot Air	Hot Air	Flow (1/100-in)	COMMENT(S)
									Stability (kips)	Corrected Stability (kips)		
TARGET	860.0	205.0	21.5									
OKD1_19_2	853.3	204.4	21.7	2.000	-	11.6	2.490	279	5.02	5.02	8.7	
OKD2_19_2	856.0	206.8	20.8	2.000	-	11.5	2.490	350	4.95	4.95	7.5	
OKD3_19_2	854.2	204.2	21.9	1.360	-	13.4	2.585	320	4.81	4.62	7.5	
OKD4_19_2	856.0	205.6	21.4	1.990	-	11.9	2.532	355	-	-	-	Sample Saved
OKD5_19_2	856.7	204.4	21.8	-	2.260	-	-	-	-	-	-	Rice Specific Gravity
OKD6_19_2	855.4	204.4	21.6	-	-	-	-	-	-	-	-	Shipped to CH2M for TCLP testing.
Average	855.3	205.0	21.7	1.99		12.1	2.524	326	-	4.86	7.9	

<sup>1</sup>PRR dry mass is based on an estimated moisture content at time of mixing of approximately 5%.

<sup>2</sup>Contains approximately 3% lime by dry weight of PRR (approximate pH of 5).

<sup>3</sup>Sample compaction effort per Hveem procedure: Stage 1 = 20 blows @ 250 psi; Stage 2 = 150 blows @ 400 psi; Stage 3 = leveling load @ approx. 1000 psi.

LEGEND ID -PRR SOURCE-TYPE OF MIX (dense or gap) & SAMPLE NUMBER\_% PRR\_%EMULSION  
PRR - PETROLEUM REFINERY RESIDUALS

TABLE A5 Cold Processed Gap Graded Mixtures Utilizing PA PRR and 3.3% to 9.4% Emulsion

ID	Agg. Mass (g)	PRR Mass <sup>1</sup> (g)	Emulsion <sup>2</sup> Mass(g)	Water <sup>2</sup> Mass (g)	Lime Mass (g)	Total Sample Dry Mass (g)	Moisture Loss <sup>3</sup> (g)	Gmb	Gmm	Air Voids (%)	Thickness (in)	Mr (psi)	Hot Air Stability (kips)	Correlation Ratio	Corrected Hot Air Stability (kips)	Flow (1/100-in)	COMMENT(S)
TARGET	823.0	178.0	33.8														
CG1_17_3.3	822.9	177.8	33.8	74.9	8.9	1030.6	13.0	-	-	-	-	-	-	-	-	-	Sample* split and tested for TCLP x 2 (uncured and cured).
CG2_17_3.3	822.2	178.3	33.8	75.2	8.9	1030.4	-	-	2.21	-	-	-	-	-	-	-	
CG3_17_3.3	822.8	177.9	34.3	75.2	8.9	1030.9	-	1.88	-	15.0	2.472	26000	0.703	1.02	0.717	52	Air cured 4-days after vac. desication and before Marshall testing.
CG4_17_3.3	822.7	177.9	34.3	74.8	8.9	1030.8	17.5	1.92	-	13.1	2.492	18000	0.603	1.01	0.609	34	Air cured 4-days after vac. desication and before Marshall testing.
Average	822.7	178.0	34.1	75.0	8.9			1.90		14.1	2.482	22000	-		0.663	43	*Water extruded from sample during Phase I comp. Emuls/RR mixture extruded during phase 2 compaction.
TARGET	823.0	178.0	67.6														
PAG1_16.8_6.4	823.0	177.7	68.1	87.4	8.9	1051.1	-	-	-	-	-	-	-	-	-	-	Sample split and tested for TCLP x 2 (uncured and cured).
PAG2_16.8_6.4	822.8	177.9	67.6	87.3	8.9	1050.8	-	1.86	-	13.4	2.459	9000	0.285	1.02	0.291	63	Air cured 3-days after vacuum desication and before Marshall testing. Sample split during t
PAG3_16.8_6.4	822.4	177.9	67.6	87.4	8.9	1050.4	28.1	1.89	-	12.1	2.519	10000	0.276	0.99	0.273	42	Air cured 4-days after vacuum desication and before Marshall testing.
PAG4_16.8_6.4	822.4	177.7	68.1	87.2	8.9	1050.5	-	-	2.15	-	-	-	-	-	-	-	
Average	822.7	177.8	67.9	87.3	8.9			1.88		12.8	2.489	9500	-		0.188	53	
TARGET	823.0	178.0	101.4														
CG1_16.4_9.4	822.5	177.9	102.2	100.2	8.9	1080.9	-	-	-	ALL SAMPLES SLUMPED IN 60C PRE-MARSHALL OVEN							Pumping during compaction. Sample split and tested for TCLP x 2 (uncured and cured).
CG2_16.4_9.4	822.2	177.9	101.0	100.0	8.9	1079.9	43.3	-	-								Sample slumped in 60C pre-Marshall oven.
CG3_16.4_9.4	822.8	178.0	101.2	100.4	8.9	1080.7	-	-	2.10								Rice Specific Gravity
CG4_16.4_9.4	822.3	178.0	101.8	100.2	8.9	1080.6	44.0	-	-								Sample slumped in 60C pre-Marshall oven.
Average	822.5	178.0	101.6	100.2	8.9												

<sup>1</sup>All PRR contains approximately 5% Ca(OH)<sub>2</sub> based on dry mass of PRR (moisture content of approximately 11%).

\*Water and emuls/PRR solutions extruded during Phase I compaction.

<sup>2</sup>Total mass of water in mixture: including water associated with aggregate, PRR, emulsion, and coating water.

<sup>3</sup>Moisture loss from time of mixing to time of compaction (lab temperature at approximately 25C during mixing and compaction).

<sup>4</sup>All samples prepared using CSS-1 emulsion (penetration of residue = 153).

TABLE A6 Cold Processed Gap Graded Mixtures Utilizing OK NRR and 6.6% to 8% Emulsion

ID	Agg. Mass (g)	PRR Mass <sup>1</sup> (g)	Emulsion <sup>2</sup> Mass(g)	Water <sup>2</sup> Mass (g)	Lime Mass (g)	Total Sample Dry Mass (g)	Gmb	Gmm	Air Voids (%)	Thickness (in)	Mr (psi)	Hot Air Stability (kips)	Correlation Ratio	Corrected Hot Air Stability (kips)	Flow (1/100-in)	COMMENT(S)
TARGET	857.0	169.0	varies													
NRRG1_16_8	856.9	169.0	88.7	96.4	4.2	1084.2	2.1	-	4.1	2.426	18	0.176	1.06	0.187	20.0	
NRRG2_16_8	856.8	169.0	87.9	104.4	4.2	1083.6	2.1	-	4.7	2.462	10	0.110	1.02	0.112	26.0	
NRRG3_16_8	856.9	169.3	87.9	104.6	4.2	1084.0	-	-	-	-	-	-	-	-	-	TCLP
NRRG4_16_8	857.1	169.1	88.5	104.5	4.2	1084.4	-	2.2	-	-	-	-	-	-	-	RICE
NRRG5_16_8	857.5	169.0	87.9	104.4	4.2	1084.4	2.05	-	6.9	2.568	14	0.110	0.96	0.106	40	
Average	857.0	169.1	73.5				1.57		3.9	1.864	10.5	-		0.135	29	

<sup>1</sup>All PRR contains approximately 5% Ca(OH)<sub>2</sub> based on dry mass of PRR (moisture content of approximately 11%).

<sup>2</sup>Total mass of water in mixture: including water associated with aggregate, PRR, emulsion, and coating water.

<sup>3</sup>All samples prepared using CSS-1 emulsion (penetration of residue = 153).

LEGEND ID - PRR SOURCE\_%EMULSION  
TRR - TREATED REFINERY RESIDUALS

Table A7 Cold Processed Mixtures Utilizing OK TRR and Emulsified Asphalt

ID	TRR Mass <sup>1</sup> (g)	Emulsion <sup>2</sup> Mass(g)	Mix Time (min)	Compaction Effort		Thickness (in)	Mr (psi)	COMMENT(S)
				Stage 1 (#blows/psi)	Stage 2 leveling load <sup>3</sup> (kips)			
TARGET	1200.0	varies						
TRR_6.9	1021.8	73.2	3	20/250	20	3.85	62000	Stage 2 compaction immediately followed Stage 1. Sample saved for future observation.
TRR_9.0	1005.0	96	3	20/250	20	3.90	75000	Stage 2 compaction immediately followed Stage 1. Sample saved for future observation.
TRR_11.1	1005.0	120	3	20/250	20	3.60	38000	Stage 2 compaction immediately followed Stage 1. Sample saved for future observation.
Average	1010.6	96.4					58333	

<sup>1</sup>TRR mixture had a moisture content of 19.4% and a pH of approximately 12.5 at time of compaction.

<sup>2</sup>All samples prepared with Chevron CSS-1 (pen of residue = 153).

<sup>3</sup>Leveling load applied for 1-minute.

**LEGEND** ID - PRR SOURCE, SAMPLE NUMBER, TYPE OF EMULSION, \_%EMULSION  
TRR - TREATED REFINERY RESIDUALS

TABLE A8 Cold Processed Mixtures Utilizing Sand Springs RR and Emulsified Asphalt

					Compaction Effort												COMMENT(S)
ID	TRR + Emul mass (g)	TRR Mass (g)	Water Mass (g)	Emulsion Mass(g)	Stage 1 (#blows/psi)	Stage 2 leveling load (kips)	Thickness (in)	Gmb	Gmm	Air Voids (%)	Mr (psi)	Stability (kips)	Correlation Ratio	Corrected Stability (kips)	Flow (1/100-in)		
TARGET	860.0	756.0															
TRR1_1hp_8.2	860.0	756.0	38.3	65.7	20/150	20	2.529	1.63		18.4	171000	1.69	1.00	1.69	11.0		
TRR2_1hp_8.2	860.0	756.0	38.3	65.7	20/150	20	2.530	1.63		18.6	181500	1.690	1.00	1.69	11.0		
TRR3_1hp_8.2	860.0	756.0	38.3	65.7	20/150	20	2.437	1.64		18.4			1.04				
TRR4_1hp_8.2	860.0	756.0	38.3	65.7	20/150	20			2								
Average	860.0	756.0	38.3				2.4987	1.633		18.47	176250	-		1.690	11		

ID	TRR + Emul mass (g)	TRR Mass (g)	Water Mass (g)	Emulsion Mass(g)	Stage 1 (#blows/psi)	Stage 2 leveling load (kips)	Thickness (in)	Gmb	Gmm	Air Voids (%)	Mr (psi)	Stability (kips)	Correlation Ratio	Corrected Stability (kips)	Flow (1/100-in)	COMMENT(S)
TARGET	860.0	723.1														
TRR1_1hp_12.6	860.0	723.1	38.3	98.6	20/150	20	2.489	1.65		16.5	172000	1.54	1.00	1.54	14.0	
TRR2_1hp_12.6	860.0	723.1	38.3	98.6	20/150	20	2.445	1.68		14.8		1.580	1.04	1.64	12.0	
TRR3_1hp_12.6	860.0	723.1	38.3	98.6	20/150	20	2.471	1.65		16.4	171000		1.04			
TRR4_1hp_12.6	860.0	723.1	38.3	98.6	20/150	20			1.97							
Average	860.0	723.1	38.3				2.4683	1.660		15.90	171500	-		1.592	13.00	

ID	TRR + Emul mass (g)	TRR Mass (g)	Water Mass (g)	Emulsion Mass(g)	Stage 1 (#blows/psi)	Stage 2 leveling load (kips)	Thickness (in)	Gmb	Gmm	Air Voids (%)	Mr (psi)	Stability (kips)	Correlation Ratio	Corrected Stability (kips)	Flow (1/100-in)	COMMENT(S)
TARGET	860.0	706.7														
TRR1_1hp_14.8	860.0	706.7	38.3	115.0	20/150	20	2.469	1.65		14.6		1.36	1.04	1.41	16.0	
TRR2_1hp_14.8	860.0	706.7	38.3	115.0	20/150	20	2.493	1.63		15.2	127000	1.230	1.00	1.23	14.0	
TRR3_1hp_14.8	860.0	706.7	38.3	115.0	20/150	20	2.497	1.62		15.7	139500		1.00			
TRR4_1hp_14.8	860.0	706.7	38.3	115.0	20/150	20			1.93							
Average	860.0	706.7	38.3				2.4863	1.633		15.17	133250	-		1.322	15.00	

ID	TRR + Emul mass (g)	TRR Mass (g)	Water Mass (g)	Emulsion Mass(g)	Stage 1 (#blows/psi)	Stage 2 leveling load (kips)	Thickness (in)	Gmb	Gmm	Air Voids (%)	Mr (psi)	Stability (kips)	Correlation Ratio	Corrected Stability (kips)	Flow (1/100-in)	COMMENT(S)
TARGET	860.0	690.2														
TRR1_1hp_17	860.0	690.2	38.3	131.5	20/150	20	2.505	1.62		14.9	98000	0.87	1.00	0.87	22.0	
TRR2_1hp_17	860.0	690.2	38.3	131.5	20/150	20	2.492	1.63		14.0		1.000	1.00	1.00	20.0	
TRR3_1hp_17	860.0	690.2	38.3	131.5	20/150	20	2.491	1.63		13.9	116500		1.00			
TRR4_1hp_17	860.0	690.2	38.3	131.5	20/150	20			1.9							
Average	860.0	690.2	38.3				2.496	1.627		14.27	107250	-		0.935	21.00	

Table A9 Sand Springs Demonstration Project Summary Table

Lane #	Cure Time (hrs)	Samples Taken after First Compaction					Samples Taken after Second Compaction					Samples Taken after 38-39 Days in-place					Samples Taken after 49-50 Days in-place						Remarks
		MC (%)	Unit Wt. D <sub>r</sub> (pcf)	M <sub>v</sub> (psi)	Stability (lbs)	Flow (1/100-in)	MC (%)	Unit Wt. D <sub>r</sub> (pcf)	M <sub>v</sub> (psi)	Stability (lbs)	Flow (1/100-in)	MC (%)	Unit Wt. D <sub>r</sub> (pcf)	M <sub>v</sub> (psi)	Stability (lbs)	Flow (1/100-in)	MC (%)	M <sub>v, Day 1</sub> (psi)	M <sub>v, Day 2</sub> (psi)	M <sub>v, Day 3</sub> (psi)	M <sub>v, Day 4</sub> (psi)	M <sub>v, Day 5</sub> (psi)	
1	4	14.4%	80	18000	572	38	13.2%	81	20000	580	32	12.0%	81.3	19000	544	37.5	12.2%	-	-	-	-	-	WC @ 1st Comp = 19.0% @ 2nd = 14.4%
2	24	13.3%	80	12500	536	32	11.0%	82	23000	647	25	12.5%	80.6	19500	639	37.5	9.6%	-	-	-	-	-	WC @ 1st Comp = 16.7% @ 2nd = 13.3%
3	0	14.5%	80	12500	596	48	N/A	N/A	N/A	N/A	N/A	11.7%	82.6	19500	670	44.0	13.3%	-	-	-	-	-	WC @ 1st Comp = 22.0%
4	4	15.0%	79	13000	521	44	N/A	N/A	N/A	N/A	N/A	13.8%	81.1	17000	651	44.5	11.0%	27000	29000	36000	37500	-	WC @ Comp = 19.4%
5	24	14.5%	79	14500	521	39	N/A	N/A	N/A	N/A	N/A	10.3%	83.0	15000	536	40.0	10.9%	-	-	-	-	-	WC @ Comp = 19.0%
6	4	14.2%	80	14500	516	37	11.9%	84	19000	611	35	14.7%	82.4	18500	705	39.0	12.5%	19000	21000	26000	34000	-	WC @ 1st Comp = 19.6% @ 2nd = 14.2%
7	24	13.8%	79	15000	525	39	12.8%	83	20000	528	40	13.8%	81.1	18500	563	47.0	11.5%	-	-	-	-	-	WC @ 1st Comp = 15.3% @ 2nd = 13.8%
8	24	13.9%	80	10000	450	46	12.7%	82	12000	450	40	11.2%	83.1	12500	568	54.5	11.6%	-	-	-	-	-	WC @ 1st Comp = 16.6% @ 2nd = 13.9%
9	4	15.3%	80	13000	531	45	14.4%	83	15000	600	39	13.9%	84.1	13000	579	47.0	14.2%	-	-	-	-	-	WC @ 1st Comp = 20.4% @ 2nd = 15.0%
10	24	4.4%	90	12000	377	40	DID NOT RETRIEVE SAMPLE					7.5%	111.2	-	122	21.5	4.5%	-	-	-	-	-	WC @ 1st Comp = 7.4% @ 2nd = 4.4%
11	24	4.0%	119	16000	531	50	3.7%	121	55000	800	35	3.8%	122.3	65500	1304	40.0	3.1%	54500	79000	102000	112000	196000	WC @ 1st Comp = 8.8% @ 2nd = 4.0%
12	24	4.2%	121	27000	500	36	3.1%	124	56500	740	16	5.0%	123.6	48000	1021	20.0	3.0%	25000	53500	78000	132000	158000	WC @ 1st Comp = 8.1% @ 2nd = 4.1%
13	24	2.5%	131	52000	683	40	2.5%	132	126500	2600	24	0.8%	133.5	161000	3236	29.0	0.4%	109300	123000	143500	188000	254000	WC @ 1st Comp = 6.8% @ 2nd = 2.5%

***APPENDIX B: Environmental Testing Data Tables***

LEGEND	ID - %PRR_%AC RESIDUAL_%LIME
	PRR - PETROLEUM REFINERY RESIDUALS

TABLE B1 Cold Processed Gap Graded Mixtures Utilizing PA PRR for TCLP Testing Only

ID	Agg. Mass (g)	RR Mass <sup>1</sup> (g)	Sample Mass (g)	% RR	AC Mass (g)	% AC	% Emulsion	Lime Mass (g)	% Lime <sup>4</sup>	Total Water Mass (g)	% Water	TCLP Lead (ppm)	COMMENT(S)
10_6_5	821.6	81.0	964.5	8.4	55.1	5.7	9.4%	7	8.2	70.5	7.3	1.8	
10_6_7.5	822.0	81.0	966.5	8.4	54.7	5.7	9.3%	9	10.9	70.1	7.2	0.4	
10_8_5	821.8	81.2	983.7	8.3	74.0	7.5	12.3%	7	8.2	74.4	7.6	0.7	
10_8_7.5	821.4	81.2	984.6	8.2	73.1	7.4	12.2%	9	10.9	74.0	7.5	0.2	
15_6_5	821.4	128.9	1019.2	12.6	58.4	5.7	9.4%	10	8.1	69.3	6.8	0.8	
15_6_7.5	821.8	128.6	1022.4	12.6	58.0	5.7	9.3%	14	10.8	69.5	6.8	0.5	
15_8_5	822.1	128.7	1039.5	12.4	78.1	7.5	12.3%	11	8.2	82.3	7.9	1.1	
15_8_7.5	822.9	128.8	1043.5	12.3	77.8	7.5	12.2%	14	10.8	81.8	7.8	1.9	
20_4_2.5	822.6	192.2	1062.1	18.1	41.9	3.9	6.5%	5	2.8	74.1	7.0	3.4	
20_4_5	822.8	192.2	1067.5	18.0	42.0	3.9	6.4%	10	5.5	83.4	7.8	1.3	
20_4_7.5 <sup>3</sup>	821.7	190.2	1069.9	17.8	41.3	3.9	6.3%	17	8.8	86.4	8.1	0.8	
20_4_10 <sup>3</sup>	821.8	190.2	1075.6	17.7	41.2	3.8	6.3%	22	11.7	86.8	8.1	1.2	
20_6_2.5	822.6	211.9	1102.8	19.2	62.5	5.7	9.3%	6	2.8	89.0	8.1	2	
20_6_5	822.7	192.2	1088.0	17.7	62.5	5.7	9.4%	11	5.5	95.2	8.7	0.9	
20_6_7.5	822.5	192.2	1092.9	17.6	62.5	5.7	9.4%	16	8.1	95.2	8.7	0.8	
20_6_7.5 <sup>2,3</sup>	821.9	190.1	1090.6	17.4	61.9	5.7	9.3%	17	8.8	99.5	9.1	1.2	
20_6_10 <sup>3</sup>	822.4	190.0	1097.7	17.3	63.0	5.7	9.4%	22	11.7	100.2	9.1	1.3	
20_6_10 <sup>2,3</sup>	822.4	190.0	1094.9	17.3	60.2	5.5	9.0%	22	11.7	99.7	9.1	1	
20_6_12.5 <sup>3</sup>	821.5	189.7	1100.4	17.2	61.7	5.6	9.2%	27	14.4	99.5	9.0	1.6	
20_8_5	823.0	192.2	1109.2	17.3	83.4	7.5	12.3%	11	5.5	100.6	9.1	0.7	
20_8_7.5	823.0	192.2	1114.6	17.2	83.7	7.5	12.3%	16	8.1	101.1	9.1	2.1	
20_8_10	822.7	192.2	1119.1	17.2	83.4	7.5	12.2%	21	10.8	100.2	9.0	1.4	
20_8_10 <sup>2,3</sup>	822.0	190.0	1115.0	17.0	80.4	7.2	11.8%	23	11.8	102.8	9.2	1.1	
20_8_12.5 <sup>3</sup>	822.3	190.0	1122.0	16.9	82.4	7.3	12.0%	27	14.4	104.2	9.3	0.9	
20_8_12.5 <sup>2,3</sup>	822.1	189.9	1120.8	16.9	81.4	7.3	11.9%	27	14.4	103.5	9.2	1.1	
20_8_15 <sup>3</sup>	822.0	190.1	1126.4	16.9	81.7	7.3	11.9%	33	17.1	103.9	9.2	2.1	
20_10_7.5	822.8	192.4	1135.9	16.9	105.2	9.3	15.2%	16	8.1	114.2	10.1	2.7	
20_10_10	822.2	211.7	1156.1	18.3	103.9	9.0	14.7%	22	10.3	104.9	9.1	1.2	
20_10_12.5 <sup>3</sup>	822.3	189.8	1141.7	16.6	102.2	9.0	14.7%	27	14.4	117.2	10.3	0.8	
20_10_12.5 <sup>2,3</sup>	822.2	189.6	1139.8	16.6	100.5	8.8	14.5%	27	14.4	116.0	10.2	1.6	
20_10_15 <sup>3</sup>	822.0	189.5	1147.8	16.5	103.8	9.0	14.8%	32	17.1	118.1	10.3	1	
20_10_15 <sup>2,3</sup>	822.0	190.0	1145.1	16.6	100.5	8.8	14.4%	33	17.2	115.7	10.1	0.9	
20_10_17.5 <sup>3</sup>	821.7	185.1	1147.0	16.1	102.8	9.0	14.7%	37	20.2	116.4	10.1	0.6	

<sup>1</sup>PRR dry mass backcalculated assuming a MC of 10% at time of mixing (certain samples vary, see ID notes).

<sup>2</sup>Samples prepared using CSS-1h emulsion.

<sup>3</sup>PRR dry mass backcalculated assuming a MC of 15.8% at time of mixing.

<sup>4</sup>Percent lime is based on dry mass of PRR only.



***APPENDIX C: RCRA Clarification Documents***

management program approved under part 271 of this chapter;

(iv) Permitted, licensed, or registered by a State to manage municipal or industrial solid waste; or

(v) A facility which:

(A) Beneficially uses or reuses, or legitimately recycles or reclaims its waste; or

(B) Treats its waste prior to beneficial use or reuse, or legitimate recycling or reclamation.

(h) Hazardous waste subject to the reduced requirements of this section may be mixed with non-hazardous waste and remain subject to these reduced requirements even though the resultant mixture exceeds the quantity limitations identified in this section, unless the mixture meets any of the characteristics of hazardous waste identified in subpart C.

(i) If any person mixes a solid waste with a hazardous waste that exceeds a quantity exclusion level of this section, the mixture is subject to full regulation.

(j) If a conditionally exempt small quantity generator's wastes are mixed with used oil, the mixture is subject to part 279 of this chapter if it is destined to be burned for energy recovery. Any material produced from such a mixture by processing, blending, or other treatment is also so regulated if it is destined to be burned for energy recovery.

[§261.5(j) amended at 57 FR 41611, Sept. 10, 1992; 58 FR 26424, May 3, 1993]

#### §261.6 Requirements for recyclable materials.

(a) (1) Hazardous wastes that are recycled are subject to the requirements for generators, transporters, and storage facilities of paragraphs (b) and (c) of this section, except for the materials listed in paragraphs (a)(2) and (a)(3) of this section. Hazardous wastes that are recycled will be known as "recyclable materials."

(2) The following recyclable materials are not subject to the requirements of this section but are regulated under subparts C through H of part 266 of this chapter and all applicable provisions in parts 270 and 124 of this chapter:

[§261.6(a)(2) introductory text amended at 56 FR 32688, July 17, 1991]

(i) Recyclable materials used in a manner constituting disposal (subpart C);

(ii) Hazardous wastes burned for energy recovery in boilers and industrial fur-

naces that are not regulated under subpart O of part 264 or 265 of this chapter (subpart H);

[§261.6(a)(2)(ii) amended at 56 FR 32688, July 17, 1991]

[Former §261.6(a)(2)(iii) removed and (iv) and (v) redesignated as (iii) and (iv) at 57 FR 41611, Sept. 10, 1992]

(iii) Recyclable materials from which precious metals are reclaimed (subpart F);

(iv) Spent lead-acid batteries that are being reclaimed (subpart G).

(3) The following recyclable materials are not subject to regulation under parts 262 through parts 266 or parts 268, 270 or 124 of this chapter, and are not subject to the notification requirements of section 3010 of RCRA:

(i) Industrial ethyl alcohol that is reclaimed except that, unless provided otherwise in an international agreement as specified in §262.58;

(A) A person initiating a shipment for reclamation in a foreign country, and any intermediary arranging for the shipment, must comply with the requirements applicable to a primary exporter in §§262.53, 262.56(a)(1)-(4), (6), and (b), and 262.57, export such materials only upon consent of the receiving country and in conformance with the EPA Acknowledgment of Consent as defined in subpart E of part 262, and provide a copy of the EPA Acknowledgment of Consent to the shipment to the transporter transporting the shipment for export;

(B) Transporters transporting a shipment for export may not accept a shipment if he knows the shipment does not conform to the EPA Acknowledgment of Consent, must ensure that a copy of the EPA Acknowledgment of Consent accompanies the shipment and must ensure that it is delivered to the facility designated by the person initiating the shipment.

(ii) Used batteries (or used battery cells) returned to a battery manufacturer for regeneration;

[Former §261.6(a)(3)(iii) removed and (iv) through (viii) redesignated as (iii) through (vii) at 57 FR 41611, Sept. 10, 1992]

(iii) Scrap metal;

(iv) Fuels produced from the refining of oil-bearing hazardous waste along with normal process streams at a petroleum refining facility if such wastes result from

normal petroleum refining, production, and transportation practices (this exemption does not apply to fuels produced from oil recovered from oil-bearing hazardous waste, where such recovered oil is already excluded under §261.4(a)(12);

[§261.6(a)(3)(iv) revised at 59 FR 38545, July 28, 1994]

(v) (A) Hazardous waste fuel produced from oil-bearing hazardous wastes from petroleum refining, production, or transportation practices, or produced from oil reclaimed from such hazardous wastes, where such hazardous wastes are reintroduced into a process that does not use distillation or does not produce products from crude oil so long as the resulting fuel meets the used oil specification under §266.40(e) of this chapter and so long as no other hazardous wastes are used to produce the hazardous waste fuel;

(B) Hazardous waste fuel produced from oil-bearing hazardous waste from petroleum refining production, and transportation practices, where such hazardous wastes are reintroduced into a refining process after a point at which contaminants are removed, so long as the fuel meets the used oil fuel specification under §266.40(e) of this chapter; and

(C) Oil reclaimed from oil-bearing hazardous wastes from petroleum refining, production, and transportation practices, which reclaimed oil is burned as a fuel without reintroduction to a refining process, so long as the reclaimed oil meets the used oil fuel specification under §266.40(e) of this chapter; and

[Former §261.6(a)(3)(v) removed and former (vi) redesignated as new (v) at 59 FR 38545, July 28, 1994]

(vi) Petroleum coke produced from petroleum refinery hazardous wastes containing oil by the same person who generated the waste, unless the resulting coke product exceeds one or more of the characteristics of hazardous waste in part 261, subpart C.

[Former §261.6(a)(3)(vii) revised and redesignated as new (vi) at 59 FR 38545, July 28, 1994]

(4) Used oil that is recycled and is also a hazardous waste solely because it exhibits a hazardous characteristic is not subject to the requirements of parts 260 through 268 of this chapter, but is regulated under part 279 of this chapter. Used oil that is recycled includes any used oil

[Sec. 261.6(a)(4)]

## HAZARDOUS WASTE CRITERIA

which is reused, following its original use, for any purpose (including the purpose for which the oil was originally used). Such term includes, but is not limited to, oil which is re-refined, reclaimed, burned for energy recovery, or reprocessed.

[§261.6(a)(4) added at 57 FR 41611, Sept. 10, 1992]

(b) Generators and transporters of recyclable materials are subject to the applicable requirements of parts 262 and 263 of this chapter and the notification requirements under section 3010 of RCRA, except as provided in paragraph (a) of this section.

(c) (1) Owners or operators of facilities that store recyclable materials before they are recycled are regulated under all applicable provisions of subparts A through L, AA, and BB of parts 264 and 265, and under parts 124, 266, 268, and 270 of this chapter and the notification requirements under section 3010 of RCRA, except as provided in paragraph (a) of this section. (The recycling process itself is exempt from regulation except as provided in §261.6(d).)

(2) Owners or operators of facilities that recycle recyclable materials without storing them before they are recycled are subject to the following requirements, except as provided in paragraph (a) of this section:

(i) Notification requirements under section 3010 of RCRA:

(ii) Sections 265.71 and 265.72 (dealing with the use of the manifest and manifest discrepancies) of this chapter.

(iii) Section 261.6(d) of this chapter.

(d) Owners or operators of facilities subject to RCRA permitting requirements with hazardous waste management units that recycle hazardous wastes are subject to the requirements of subparts AA and BB of part 264 or 265 of this chapter.

#### §261.7 Residues of hazardous waste in empty containers.

(a) (1) Any hazardous waste remaining in either (i) an empty container or (ii) an inner liner removed from an empty container, as defined in paragraph (b) of this section, is not subject to regulation under parts 261 through 265, or part 268, 270 or 124 of this chapter or to the notification requirements of section 3010 of RCRA.

(2) Any hazardous waste in either (i) a container that is not empty or (ii) an inner liner removed from a container that is not empty, as defined in paragraph (b) of this section, is subject to regulation under parts 261 through 265, and parts 268, 270 and 124 of this chapter and to the notification requirements of section 3010 of RCRA.

(b) (1) A container or an inner liner removed from a container that has held any hazardous waste, except a waste that is a compressed gas or that is identified as an acute hazardous waste listed in §§261.31, 261.4(e) of this chapter is empty if:

(i) All wastes have been removed that can be removed using the practices commonly employed to remove materials from that type of container, *e.g.*, pouring, pumping, and aspirating, *and*

(ii) No more than 2.5 centimeters (one inch) of residue remain on the bottom of the container or inner liner, *or*

(iii) (A) No more than 3 percent by weight of the total capacity of the container remains in the container or inner liner if the container is less than or equal to 110 gallons in size, *or*

(B) No more than 0.3 percent by weight of the total capacity of the container remains in the container or inner liner if the container is greater than 110 gallons in size.

(2) A container that has held a hazardous waste that is a compressed gas is empty when the pressure in the container approaches atmospheric.

(3) A container or an inner liner removed from a container that has held an

[Sec. 261.7(b)(3)]

acute hazardous waste listed in §§261.31, 261.32, or 261.33(e) is empty if:

(i) The container or inner liner has been triple rinsed using a solvent capable of removing the commercial chemical product or manufacturing chemical intermediate;

(ii) The container or inner liner has been cleaned by another method that has been shown in the scientific literature, or by tests conducted by the generator, to achieve equivalent removal; or

(iii) In the case of a container, the inner liner that prevented contact of the commercial chemical product or manufacturing chemical intermediate with the container, has been removed.

#### §261.8 PCB wastes regulated under Toxic Substance Control Act.

The disposal of PCB-containing dielectric fluid and electric equipment containing such fluid authorized for use and regulated under part 761 of this chapter and that are hazardous only because they fail the test for the Toxicity Characteristic (Hazardous Waste Codes D018 through D043 only) are exempt from regulation under parts 261 through 265, and parts 268, 270, and 124 of this chapter, and the notification requirements of section 3010 of RCRA.

#### Subpart B—Criteria for Identifying the Characteristics of Hazardous Waste and for Listing Hazardous Waste

##### §261.10 Criteria for identifying the characteristics of hazardous waste.

(a) The Administrator shall identify and define a characteristic of hazardous waste in subpart C only upon determining that:

(1) A solid waste that exhibits the characteristic may:

(i) Cause, or significantly contribute to, an increase in mortality or an increase in serious irreversible, or incapacitating reversible, illness; or

(ii) Pose a substantial present or potential hazard to human health or the environment when it is improperly treated, stored, transported, disposed of or otherwise managed; and

(2) The characteristic can be:

(i) Measured by an available standardized test method which is reasonably within the capability of generators of solid

waste or private sector laboratories that are available to serve generators of solid waste; or

(ii) Reasonably detected by generators of solid waste through their knowledge of their waste.

##### §261.11 Criteria for listing hazardous waste.

(a) The Administrator shall list a solid waste as a hazardous waste only upon determining that the solid waste meets one of the following criteria:

(1) It exhibits any of the characteristics of hazardous waste identified in subpart C.

(2) It has been found to be fatal to humans in low doses or, in the absence of data on human toxicity, it has been shown in studies to have an oral LD 50 toxicity (rat) of less than 50 milligrams per kilogram, an inhalation LC 50 toxicity (rat) of less than 2 milligrams per liter, or a dermal LD 50 toxicity (rabbit) of less than 200 milligrams per kilogram or is otherwise capable of causing or significantly contributing to an increase in serious irreversible, or incapacitating reversible, illness. (Waste listed in accordance with these criteria will be designated Acute Hazardous Waste.)

(3) It contains any of the toxic constituents listed in appendix VIII and, after considering the following factors, the Administrator concludes that the waste is capable of posing a substantial present or potential hazard to human health or the environment when improperly treated, stored, transported or disposed of, or otherwise managed:

[§261.11(a)(3) introductory text amended at 57 FR 14, Jan. 2, 1992]

(i) The nature of the toxicity presented by the constituent.

(ii) The concentration of the constituent in the waste.

(iii) The potential of the constituent or any toxic degradation product of the constituent to migrate from the waste into the environment under the types of improper management considered in paragraph (a)(3)(vii) of this section.

(iv) The persistence of the constituent or any toxic degradation product of the constituent.

(v) The potential for the constituent or any toxic degradation product of the constituent to degrade into non-harmful constituents and the rate of degradation.

(vi) The degree to which the constituent or any degradation product of the constituent bioaccumulates in ecosystems.

(vii) The plausible types of improper management to which the waste could be subjected.

(viii) The quantities of the waste generated at individual generation sites or on a regional or national basis.

(ix) The nature and severity of the human health and environmental damage that has occurred as a result of the improper management of wastes containing the constituent.

(x) Action taken by other governmental agencies or regulatory programs based on the health or environmental hazard posed by the waste or waste constituent.

(xi) Such other factors as may be appropriate.

Substances will be listed on appendix VIII only if they have been shown in scientific studies to have toxic, carcinogenic, mutagenic or teratogenic effects on humans or other life forms.

(Wastes listed in accordance with these criteria will be designated Toxic wastes.)

(b) The Administrator may list classes or types of solid waste as hazardous waste if he has reason to believe that individual wastes, within the class or type of waste, typically or frequently are hazardous under the definition of hazardous waste found in section 1004(5) of the Act.

(c) The Administrator will use the criteria for listing specified in this section to establish the exclusion limits referred to in §261.5(c).

#### Subpart C—Characteristics of Hazardous Waste

##### §261.20 General.

(a) A solid waste, as defined in §261.2, which is not excluded from regulation as a hazardous waste under §261.4(b), is a hazardous waste if it exhibits any of the characteristics identified in this subpart.

*Comment:* §262.11 of this chapter sets forth the generator's responsibility to determine whether his waste exhibits one or more of the characteristics identified in this subpart.

(b) A hazardous waste which is identified by a characteristic in this subpart is assigned every EPA Hazardous Waste Number that is applicable as set forth in this subpart. This number must be used in complying with the notification requirements of section 3010 of the Act and all

[Sec. 261.20(b)]

applicable recordkeeping and reporting requirements under parts 262 through 265, 268, and 270 of this chapter.

(c) For purposes of this subpart, the Administrator will consider a sample obtained using any of the applicable sampling methods specified in appendix I to be a representative sample within the meaning of part 260 of this chapter.

*Comment:* Since the appendix I sampling methods are not being formally adopted by the Administrator, a person who desires to employ an alternative sampling method is not required to demonstrate the equivalency of his method under the procedures set forth in §§260.20 and 260.21.

#### §261.21 Characteristic of ignitability.

(a) A solid waste exhibits the characteristic of ignitability if a representative sample of the waste has any of the following properties:

(1) It is a liquid, other than an aqueous solution containing less than 24 percent alcohol by volume and has flash point less than 60°C (140°F), as determined by a Pensky-Martens Closed Cup Tester, using the test method specified in ASTM Standard D-93-79 or D-93-80 (incorporated by reference, see §260.11), or a Setaflash Closed Cup Tester, using the test method specified in ASTM Standard D-3278-78 (incorporated by reference, see §260.11), or as determined by an equivalent test method approved by the Administrator under procedures set forth in §§260.20 and 260.21.

(2) It is not a liquid and is capable, under standard temperature and pressure, of causing fire through friction, absorption of moisture or spontaneous chemical changes and, when ignited, burns so vigorously and persistently that it creates a hazard.

(3) It is an ignitable compressed gas as defined in 49 CFR 173.300 and as determined by the test methods described in that regulation or equivalent test methods approved by the Administrator under §§260.20 and 260.21.

(4) It is an oxidizer as defined in 49 CFR 173.151.

(b) A solid waste that exhibits the characteristic of ignitability has the EPA Hazardous Waste Number of D001.

#### §261.22 Characteristic of corrosivity.

(a) A solid waste exhibits the characteristic of corrosivity if a representative sample of the waste has either of the following properties:

(1) It is aqueous and has a pH less than or equal to 2 or greater than or equal to 12.5, as determined by a pH meter using Method 9040 in "Test Methods for Evaluating Solid Waste, Physical/Chemical Methods," EPA Publication SW-846, as incorporated by reference in §260.11 of this chapter.

[§261.22(a)(1) revised at 58 FR 46049, Aug. 31, 1993]

(2) It is a liquid and corrodes steel (SAE 1020) at a rate greater than 6.35 mm (0.250 inch) per year at a test temperature of 55°C (130°F) as determined by the test method specified in NACE (National Association of Corrosion Engineers) Standard TM-01-69 as standardized in "Test Methods for Evaluating Solid Waste, Physical/Chemical Methods," EPA Publication SW-846, as incorporated by reference in §260.11 of this chapter.

[§261.22(a)(2) revised at 58 FR 46049, Aug. 31, 1993]

(b) A solid waste that exhibits the characteristic of corrosivity has the EPA Hazardous Waste Number of D002.

#### §261.23 Characteristic of reactivity.

(a) A solid waste exhibits the characteristic of reactivity if a representative sample of the waste has any of the following properties:

(1) It is normally unstable and readily undergoes violent change without detonating.

(2) It reacts violently with water.

(3) It forms potentially explosive mixtures with water.

(4) When mixed with water, it generates toxic gases, vapors or fumes in a quantity sufficient to present a danger to human health or the environment.

(5) It is a cyanide or sulfide bearing waste which, when exposed to pH conditions between 2 and 12.5, can generate toxic gases, vapors or fumes in a quantity sufficient to present a danger to human health or the environment.

(6) It is capable of detonation or explosive reaction if it is subjected to a strong initiating source or if heated under confinement.

(7) It is readily capable of detonation or explosive decomposition or reaction at standard temperature and pressure.

(8) It is a forbidden explosive as defined in 49 CFR 173.51, or a Class A explosive as defined in 49 CFR 173.53 or

a Class B explosive as defined in 49 CFR 173.88.

(b) A solid waste that exhibits the characteristic of reactivity has the EPA Hazardous Waste Number of D003.

#### §261.24 Toxicity characteristic.

(a) A solid waste exhibits the characteristic of toxicity if, using the Toxicity Characteristic Leaching Procedure, test Method 1311 in "Test Methods for Evaluating Solid Waste, Physical/Chemical Methods," EPA Publication SW-846, as incorporated by reference in §260.11 of this chapter, the extract from a representative sample of the waste contains any of the contaminants listed in table 1 at the concentration equal to or greater than the respective value given in that table. Where the waste contains less than 0.5 percent filterable solids, the waste itself, after filtering using the methodology outlined in Method 1311, is considered to be the extract for the purpose of this section.

[§261.24(a) revised at 58 FR 46049, Aug. 31, 1993]

(b) A solid waste that exhibits the characteristic of toxicity has the EPA Hazardous Waste Number specified in Table 1 which corresponds to the toxic contaminant causing it to be hazardous.

Table 1—Maximum Concentration of Contaminants for the Toxicity Characteristic

EPA HW No. <sup>1</sup>	Contaminant	CAS No. <sup>2</sup>	Regulatory Level (mg/L)
D004	Arsenic.....	7440-38-2	5.0
D005	Barium.....	7440-39-3	100.0
D018	Benzene.....	71-43-2	0.5
D006	Cadmium.....	7440-43-8	1.0
D019	Carbon tetrachloride.....	56-23-5	0.5
D020	Chlordane.....	57-74-9	0.03
D021	Chlorobenzene.....	108-90-7	100.0
D022	Chloroform.....	67-66-3	6.0
D007	Chromium.....	7440-47-3	5.0
D023	o-Cresol.....	95-48-7	200.0
D024	m-Cresol.....	108-39-4	200.0
D025	p-Cresol.....	108-44-5	200.0
D026	Cresol.....		200.0
D016	2,4-D.....	94-75-7	10.0
D027	1,4-Dichlorobenzene.....	106-46-7	7.5
D028	1,2-Dichloroethane.....	107-06-2	0.5
D029	1,1-Dichloroethylene.....	75-35-4	0.7
D030	2,4-Dinitrotoluene.....	121-14-2	0.13
D012	Endrin.....	72-20-8	0.02
D031	Heptachlor (and its epoxide).....	76-44-8	0.008
D032	Hexachlorobenzene.....	118-74-1	0.13
D033	Hexachlorobutadiene.....	87-68-3	0.5
D034	Hexachloroethane.....	87-72-1	3.0
D008	Lead.....	7439-92-1	5.0
D013	Lindane.....	58-89-9	0.4
D009	Mercury.....	7439-97-6	0.2
D014	Methoxychlor.....	72-43-5	10.0
D035	Methyl ethyl ketone.....	78-93-3	200.0

[Sec. 261.24(b)]

APPENDIX XII—NICKEL OR CHROMIUM-BEARING MATERIALS THAT MAY BE PROCESSED IN EXEMPT NICKEL-CHROMIUM RECOVERY FURNACES

Appendix XIII—Mercury Bearing Wastes That May Be Processed in Exempt Mercury Recovery Units

#### Subparts A—B [Reserved]

#### Subpart C—Recyclable Materials Used in a Manner Constituting Disposal

##### §266.20 Applicability.

(a) The regulations of this subpart apply to recyclable materials that are applied to or placed on the land:

(1) Without mixing with any other substance(s); or

(2) After mixing or combination with any other substance(s). These materials will be referred to throughout this subpart as "materials used in a manner that constitutes disposal."

(b) Products produced for the general public's use that are used in a manner that constitutes disposal and that contain recyclable materials are not presently subject to regulation if the recyclable materials have undergone a chemical reaction in the course of producing the products so as to become inseparable by physical means and if such products meet the applicable treatment standards in subpart D of part 268 (or applicable prohibition levels in §268.32 or RCRA section 3004(d), where no treatment standards have been established) for each recyclable material (i.e., hazardous waste) that they contain. Commercial fertilizers that are produced for the general public's use that contain recyclable materials also are not presently subject to regulation provided they meet these same treatment standards or prohibition levels for each recyclable material that they contain. However, zinc-containing fertilizers using hazardous waste K061 that are produced for the general public's use are not presently subject to regulation.

(c) Anti-skid/deicing uses of slags, which are generated from high temperature metals recovery (HTMR) processing of hazardous waste K061, K062, and F006, in a manner constituting disposal are not covered by the exemption in paragraph (b) of this section and remain subject to regulation.

[§266.20(c) added at 59 FR 43499, Aug. 24, 1994]

##### §266.21 Standards applicable to generators and transporters of materials used in a manner that constitutes disposal.

Generators and transporters of materials that are used in a manner that constitutes disposal are subject to the applicable requirements of parts 262 and 263 of this chapter, and the notification requirement under section 3010 of RCRA.

##### §266.22 Standards applicable to storers of materials that are to be used in a manner that constitutes disposal who are not the ultimate users.

Owners or operators of facilities that store recyclable materials that are to be used in a manner that constitutes disposal, but who are not the ultimate users of the materials, are regulated under all applicable provisions of subparts A through L of parts 264 and 265 and parts 270 and 124 of this chapter and the notification requirement under section 3010 of RCRA.

##### §266.23 Standards applicable to users of materials that are used in a manner that constitutes disposal.

(a) Owners or operators of facilities that use recyclable materials in a manner that constitutes disposal are regulated under all applicable provisions of subparts A through N of parts 124, 264, 265, 268, and 270 of this chapter and the notification requirement under section 3010 of RCRA. (These requirements do not apply to products which contain these recyclable materials under the provisions of §266.20(b) of this chapter.)

[§266.23(a) revised at 59 FR 48041, Sept. 19, 1994]

(b) The use of waste or used oil or other material, which is contaminated with dioxin or any other hazardous waste (other than a waste identified solely on the basis of ignitability), for dust suppression or road treatment is prohibited.

#### Subpart D—Hazardous Waste Burned for Energy Recovery [Reserved]

[Subpart D removed and reserved at 56 FR 7206 Feb. 21, 1991]

#### Subpart E—Used Oil Burned for Energy Recovery [Reserved]

[Subpart E removed and reserved at 57 FR 41611, Sept. 10, 1992]

#### Subpart F—Recyclable Materials Utilized for Precious Metal Recovery

##### §266.70 Applicability and requirements.

(a) The regulations of this subpart apply to recyclable materials that are reclaimed to recover economically significant amounts of gold, silver, platinum, palladium, iridium, osmium, rhodium, ruthenium, or any combination of these.

(b) Persons who generate, transport, or store recyclable materials that are regulated under this subpart are subject to the following requirements:

(1) Notification requirements under section 3010 of RCRA;

(2) Subpart B of part 262 (for generators), §§263.20 and 263.21 (for transporters), and §§265.71 and 265.72 (for persons who store) of this chapter;

(c) Persons who store recycled materials that are regulated under this subpart must keep the following records to document that they are not accumulating these materials speculatively (as defined in §261.1(c) of this chapter):

(1) Records showing the volume of these materials stored at the beginning of the calendar year;

(2) The amount of these materials generated or received during the calendar year; and

(3) The amount of materials remaining at the end of the calendar year.

(d) Recyclable materials that are regulated under this subpart that are accumulated speculatively (as defined in §261.1(c) of this chapter) are subject to all applicable provisions of parts 262 through 265, 270 and 124 of this chapter.

#### Subpart G—Spent Lead-Acid Batteries Being Reclaimed

##### §266.80 Applicability and requirements.

(a) The regulations of this subpart apply to persons who reclaim spent lead-acid batteries that are recyclable materials ("spent batteries"). Persons who generate, transport, or collect spent batteries, or who store spent batteries but do not reclaim them are not subject to regulation

*Assigning Treatment Standards  
for Underlying TC Metals*

Note that metallic underlying constituents in D001, D002, and D012-D043 characteristic wastes have to be treated to meet the UTS values in Table 2, not the existing treatment standards associated with the TC metal waste codes (D004-D011). However, this brings up an interesting question. Could a generator declare his waste to be hazardous in order to gain a more lenient treatment standard? For example, if a generator declares his nonwastewater destined for a landfill to be hazardous for lead and assigns waste code D008 to it, he must meet a treatment standard of 5.0 mg/L using the TCLP. If his waste exhibits a non-metal characteristic and he does not add the D008 code, he must meet a lead standard of 0.37 mg/L using the TCLP (reference Table 2).

Our discussions with EPA on this point indicate that the agency is adhering to a policy requiring treatment to the most stringent treatment standards. Simply put, if a waste is hazardous due to a non-TC metal characteristic, any TC metals included in the waste at the point of generation at any concentration levels are considered underlying hazardous constituents and must be treated to meet UTS limits. This requirement is consistent with EPA's policy that requires application of the most stringent treatment standards in a situation where multiple treatment standards apply. This policy is illustrated in the following three examples:

1. A nonwastewater is characteristically hazardous only due to its lead concentration (i.e., it generates >5.0 mg/L lead in the TCLP extract). This material is a D008 waste and must be treated to 5.0 mg/L lead before land disposal.
2. A nonwastewater destined for a landfill is characteristically hazardous only due to its corrosivity but also contains lead that leaches at 4.0 mg/L. This material is a D002 waste and must be treated to deactivate the corrosivity and lower the underlying lead concentration to 0.37 mg/L before land disposal.
3. A nonwastewater destined for a landfill is characteristically hazardous due to both its corrosivity and lead content (i.e., it generates >5.0 mg/L lead in the TCLP extract). This material is both a D002 and D008 waste and must be treated to deactivate the corrosivity and lower the lead concentration to 0.37 mg/L before land disposal.

There is an obvious inconsistency in the treatment requirement for lead between the first and third example above. EPA recognizes that this inconsistency exists and will act to eliminate it, but not until the Phase IV LDR rule, which is scheduled for finalization in mid-1996. At that time, the agency will change the current treatment standards for the TC metals (D004-D011) and/or the UTS limits so that they both reflect the same numerical values.

**Treatment Standards for Newly Listed Wastes**

EPA was required by statute to promulgate LDR standards by May 8, 1990 for all wastes that were either listed or identified as hazardous at the time HSWA was enacted (November 1984). For wastes listed or identified after that date (i.e., newly listed or identified wastes), EPA is required to promulgate treatment standards within six months after a waste is listed or identified. However, the agency failed to meet this latter statutory deadline for a number of wastes, including coke by-product wastes (K141-K145 and K147-K148) and chlorinated toluene production wastes (K149-K151). EPA is now issuing LDR standards for these wastes, as described below and listed in Table 7 (page 17). Unless they meet the specified treatment standards, these wastes can no longer be land disposed.

**Coke By-Product Wastes**

Seven listings for wastes from coke by-product production (K141-K145 and K147-K148) were finalized on August 18, 1992 (57 FR 37284). Since the majority of these wastes are nonwastewaters due to their high organic content, EPA has determined that thermal destruction, such as incineration or fuel substitution, represents BDAT for them. Since the UTS for nonwastewaters are also based on incineration, the agency has promulgated treatment standards for the regulated constituents in these wastes that are numerically equal to the corresponding UTS (see Table 7).

**Chlorinated Toluene Production Wastes**

Three wastes generated during the production of chlorinated toluenes (K149-K151) were listed on October 15, 1992 (57 FR 47377). Similar to the coke by-product wastes noted above, the organics in these wastes may be treated by incineration or fuel substitution as BDAT. Therefore, the treatment standards for the regulated compounds in these wastes (see Table 7) are also equivalent to the UTS.

**New "Combustion" Technology-Based  
Treatment Standard**

When a technology is specified as required LDR treatment for a particular hazardous waste, EPA assigns a five-letter code to indicate the technology-based standard. The agency previously established incineration (which has the five-letter code of INCIN) as the treatment technology required for certain wastes before they could be land disposed. In addition, burning hazardous waste-derived fuel as a substitute for fossil fuels (FSUBS—accomplished in BIFs) had also been promulgated as an alternative to incineration for some, but not all, of the wastes for which incineration had been specified as the required treatment method.

EPA has decided to simplify the treatment standard when both INCIN and FSUBS are specified as alternative treatment technologies. Therefore, in the September 19, 1994 final rule, the agency has consolidated these two distinct

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P.02



UNITED STATES ENVIRONMENTAL PROTECTION AGENCY  
WASHINGTON, D.C. 20460

SEP 23 1993

MEMORANDUMOFFICE OF  
SOLID WASTE AND EMERGENCY RESPONSE

SUBJECT: Interpretation of "Aqueous" as Applied to the  
Corrosivity Characteristic (40 CFR 261.22)

TO: Joseph R. Franzmathes, Director  
Waste Management Division

Redacted for privacy

FROM: David Bussard, Director  
Characterization and Assessment Division

This memorandum responds to your memorandum to Bruce Diamond dated March 11, 1993 requesting clarification of the term "aqueous" as it applies to the corrosivity characteristic. Your memorandum references a September 1992 "Hotline Questions and Answers" publication produced by the RCRA/Superfund Hotline contractors and concurred upon by my Division and by OSW.

The Hotline publication correctly defines "aqueous", for the purposes of the corrosivity characteristic, to mean in a form amenable to pH measurement. This interpretation is consistent with the supporting documentation found in the background document for the corrosivity characteristic final rulemaking (Background Document: Section 261.22 - Characteristic of Corrosivity, May 2, 1980). I have attached the applicable section for your information.

A more specific interpretation of "aqueous" for the purposes of the corrosivity characteristic may be found in the method referenced in the actual regulatory text for the corrosivity characteristic at 40 CFR 261.22(a)(1). The regulation states that "[t]he EPA test method for pH is specified as Method 5.2 in "Test Methods for the Evaluation of Solid Waste, Physical/Chemical Methods" (see attachment). Method 5.2 pH Electrometric Measurement, which was renumbered to Method 9040, specifies under scope and application that the method "is used to measure the pH of aqueous wastes and those wastes where the aqueous phase constitutes at least 20% of the total volume of the waste." Therefore, any waste for which this method is applicable must contain at least 20% free water by volume. This method is also attached for your information.

If you or your staff should have any questions regarding this memorandum, please call me or have your staff call Al Collins, of my staff, at 202-260-4701.

Attachments



#### **8.4 APPENDIX D: OSHD Standard for Water Sensitivity**

**MATERIALS SECTION**  
**OSHD Test Method 315-90**

Method of Test for

**EFFECT OF WATER SATURATION AND FREEZE THAW CYCLE ON DENSE-GRADED  
ASPHALT CONCRETE— INDEX OF RETAINED RESILIENT MODULUS**

**SCOPE**

- 1.1 This method is intended to predict loss of resilient modulus of compacted dense-graded asphalt concrete due to field conditions of moisture and freezing. In addition, current and predicted future modulus values are generated for pavement design purposes.

**SUMMARY**

- 2.1 One or more fabricated asphalt concrete briquets are each tested for unconditioned resilient modulus, then for modulus after one water-saturated freeze-thaw cycle.
- 2.2 Results are Index of Retained Resilient Modulus ( $IRM_R$ ) for the saturated freeze-thaw condition, expressed as a ratio of conditioned to unconditioned modulus, in percent.

**APPARATUS**

- 3.1 Apparatus as described in "Apparatus" in AASHTO T 247-80 (1986) for Preparation of Test Specimens of Bituminous Mixtures; including the described California kneading compactor.
- 3.2 An apparatus for measuring diametral resilient modulus of four-inch by nominal 2-1/2 inch asphalt concrete test specimens. This apparatus and procedures used shall be generally similar to those described in ASTM D4123-82(1987). A load cell with range up to 300 pounds is recommended to accommodate the expected loads.
- 3.3 A temperature-controlled air bath capable of achieving and maintaining specimen temperature of  $25 \pm 1^\circ \text{C}$  ( $77 \pm 1.8^\circ \text{F}$ ).
- 3.4 An air-tight waterproof vacuum chamber capable of holding the desired number of specimens submerged in water while applying a partial vacuum equivalent to an absolute pressure of 3.0 cm Hg (1.2 inches Hg).
- 3.5 A freezer capable of holding sealed specimens at  $-18 \pm 4.4^\circ \text{C}$  ( $0 \pm 8^\circ \text{F}$ ).

- 3.6 A water bath capable of maintaining submerged specimens at  $25 \pm 1^{\circ} \text{ C}$  ( $77 \pm 1.8^{\circ} \text{ F}$ ).
- 3.7 A water bath (which may be the one used in 3.6 if desired) capable of maintaining submerged specimens at  $60 \pm 1^{\circ} \text{ C}$  ( $140 \pm 1.8^{\circ} \text{ F}$ ).

#### PREPARATION OF SPECIMENS

- 4.1 Laboratory-fabricated, four-inch diameter by nominal 2-1/2 inch tall specimens shall be prepared according to normal OSHD procedures for Hveem stabilometer specimens, as described in OSHD Test Method 302-86 (= AASHTO T 247).

#### NUMBER OF SPECIMENS

- 5.1 Normal OSHD procedure for mix design uses one test specimen each at the minimum, the maximum, and the middle asphalt contents tested in the OSHD Hveem-type mix design procedure (total of 3 specimens). This allows a straight-line interpolation to compute the minimum asphalt content required to satisfy OSHD Index of Retained Resilient Modulus ( $\text{IRM}_R$ ) specifications. Each specimen is used non destructively for both phases of the test: unconditioned modulus, and saturated freeze-thaw modulus.

#### PROCEDURE

##### DAY 1

- 6.1 Fabricate 4 inch diameter by nominal 2-1/2 inch high test briquets using California kneading compactor.
- 6.2 Apply "leveling off" load by the application of a static load of 100 psi (6.9 MPa) for 10 seconds.
- 6.3 Cool briquets to room temperature; determine the dry mass (A) of the specimen by OSHD Test Method 302A-86 (AASHTO T 166, Method A).
- 6.4 Place briquets in a temperature-controlled  $77^{\circ} \text{ F}$  air bath for a minimum of 3 hours to stabilize internal temperature. Measure briquet height to nearest 0.05 inch.
- 6.5 Test each briquet for unconditioned resilient modulus at  $77^{\circ} \text{ F}$  on each of 2 perpendicular diametral axes, measuring total horizontal diametral deformation with vertical compressive loading. Use a load pulse of 0.1 second. For each axis, stabilize readings by applying an initial loading sequence of at least 20 load pulses before beginning to record data. Then record 10 sequential load pulse results immediately following the conditioning pulses. The average of the 10 sequential load pulse results is a single resilient modulus result for the axis being measured. The average of the 2 results (one result for each axis) is the resilient modulus of the specimen for the given condition (see Section 7, Calculations).

## CALCULATIONS

- 7.1 The resilient modulus of a briquet for a specific condition is the average of the moduli of the two perpendicular axes tested for that condition. Compute the resilient modulus ( $M_R$ ) for one axis substantially as follows:

$$M_R = \frac{(P)(C)}{(\Delta h)(t)}$$

where:  $P$  = applied load, pounds. If not held constant during the 10 recorded load pulses, this is the average of the 10 applied loads on the given briquet axis.

$C$  = a constant; OSHD uses 618,300 microinches per inch, which is a value supplied by Oregon State University as appropriate for our current modulus apparatus and typical dense graded mixes. A similar value is obtained from several publications by summing recommended Poisson's ratio of 0.35 with derived diametral loading constants of approximately 0.27, with an included conversion for microinches:  
 typical  $C = (0.35 + 0.27)(1 \times 10^6 \text{ microinches/inch})$   
 $= 620,000 \text{ microinches/inch}$

$\Delta h$  = average diametral deformation, in microinches. This is the average of the 10 deformations during the 10 recorded load pulses on one axis.

$t$  = specimen thickness (height normal to briquet ends), in inches, to the nearest 0.05 inch - typically 2.50 inches.

- 7.2 Compute the Index of Retained Resilient Modulus ( $IRM_R$ ) as the ratio of the freeze-thaw modulus of a briquet to the unconditioned modulus of that briquet, expressed in percent:

$$IRM_R = \frac{FrThM_R}{UncondM_R} \times 100\%$$

- 7.3  $IRM_R$  shall be recorded to the nearest percent, for example: 87%.

For unconditioned (dry) briquets, use reasonable speed to achieve modulus testing if an environmental chamber is not available to maintain 77 degrees air temperature. Return the briquet to the 77 degree air bath for at least 10 minutes between tests of axes if testing both axes consecutively would allow the briquet to remain either a) longer than 5 minutes per axes at an ambient temperature outside the range of  $77 \pm 8^{\circ}$  F, or b) longer than 5 minutes total outside the range of  $77 \pm 12^{\circ}$  F.

For conditioned (wet) briquets, use reasonable speed in all cases to avoid possible modulus changes due to water loss or to evaporation temperature effects. Return the briquet to the 77 degree water bath for at least 25 minutes between tests of axes if testing both axes consecutively would allow the briquet to remain for a) longer than 10 minutes exposed to air, or b) longer than 7 minutes outside the range of  $77 \pm 12^{\circ}$  F, or c) longer than 4 minutes outside the range of  $77 \pm 12^{\circ}$  F.

Adjust loading to achieve an average of 96 to 106 microinches (0.000096 to 0.000106 inches) of total diametral deformation during conditioning pulses and during the 10-pulse data recording period. It is preferable to maintain a single load magnitude during the data recording period.

- 6.6 Determine the immersed mass (C) and the saturated surface-dry mass (B) by OSHD Test Method 302A-86 (AASHTO T166, Method A.) Using the dry mass determined in 6.3 above, calculate the bulk specific gravity of the specimen.
- 6.7 Vacuum saturate briquets at a partial vacuum equivalent to an absolute pressure of 3.0 cm Hg (1.2 inches Hg) or less for 30 minutes, while submerged in room-temperature water. Release vacuum and allow briquets to rest submerged for 2 to 5 minutes. After release of vacuum, double wrap each still-wet briquet with waterproof material and seal. A second sealed wrapping containing single-sealed briquets of one mix design is considered double wrapping. Place the sealed briquets in a freezer at  $0 \pm 8^{\circ}$  F for at least 15 hours.

#### DAY 2

- 6.8 Remove specimens from freezer and immediately submerge in  $140^{\circ}$  F water bath with the inner seal intact. After a few minutes, remove the thawed inner sealing material and immediately submerge the briquet in the 140 degree water bath for 24 hours.

#### DAY 3

- 6.9 Move the briquets to a  $77^{\circ}$  F water bath for 3 to 6 hours. If necessary, add cold water to the bath to reduce temperature to very nearly 77 degrees soon after briquet submersion in the 77 degree bath.
- 6.8 Retest modulus as in 6.4, substituting the words "water bath" for "air bath" and the words "freeze-thaw modulus" for "unconditioned modulus" as appropriate.

- 7.4 Using straight-line interpolation between asphalt contents tested, compute the asphalt content at which the computed  $IRM_R$  will meet or exceed the minimum  $IRM_R$  specified by OSHD. Increments of asphalt content shall be computed to one tenth of one percent of total mix weight, for example: 5.7 percent asphalt cement content.

## REPORT

- 8.1 The report shall include all data necessary to fully identify the mix tested and proposed use of mix, including but not limited to:
- Testing agency name ( and if not OSHD, also address, phone number, and contact person); OSHD contract number; Federal Aid identifying number(s) if applicable; asphalt cement used including grade and manufacturer; asphalt additives used (if any) including manufacturer and identifying name, and method of introduction into the mix; aggregate source and gradation used (if not documented elsewhere on the mix design submitted); aggregate additives used (if any) and method of application, for example: "1 percent by total aggregate weight of lime was added as a filler after final aggregate oven drying"; the asphalt contents at which the resilient modulus test was run (by percent of total mix weight); test briquet dimensions and bulk specific gravities.

- 8.2 The report shall also include the individual resilient modulus results for the two conditions for each briquet, and the asphalt content at which the  $IRM_R$  meets OSHD minimum specifications.

If all asphalt contents tested for  $IRM_R$  meet or exceed the required minimum value, report the lowest asphalt content tested as meeting the minimum specifications.