Responsible Use of Re-Refined Engine Oil Bottoms (REOB) in Asphalt Binders
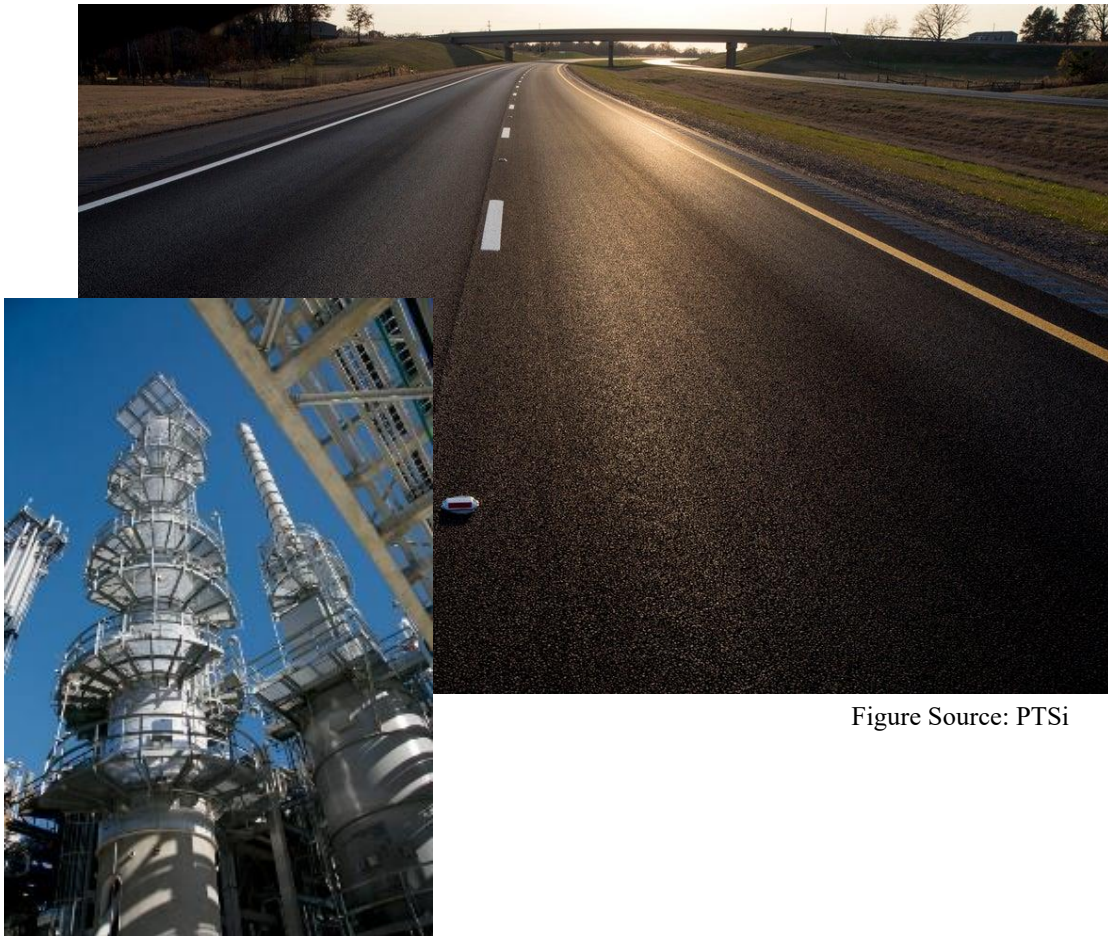
Figure Source: PTSi

U.S. Department of Transportation
Federal Highway Administration

FOREWORD

Re-refined engine oil bottoms (REOB), sometimes referred to as vacuum tower asphalt extender (VTAE), has been used to modify asphalt binders to improve low temperature rheological properties since the 1980s. Since the introduction of the Superpave performance grade (PG) asphalt binders, use of REOB as a blending component or modifier to produce paving asphalt binders has increased.[1] Softer asphalt binder grades in colder climates are in greater demand to reduce low temperature cracking. Additionally, the trend to use higher levels of reclaimed asphalt pavement (RAP) and reclaimed asphalt shingles (RAS) has increased the demand for softer asphalt binder grades. These factors have increased the interest and use of softening or fluxing agents, which include non-asphaltic asphalt binder blending stocks such as REOB. This report discusses use of REOB as an asphalt binder modifier and presents information on detection and quantification of REOB in asphalt binders.

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16. Abstract

Re-refined engine oil bottoms (REOB), also referred to as vacuum tower asphalt extender (VTAE), has been used to modify asphalt binders to improve low temperature rheological properties since the 1980s. Since the introduction of the Superpave performance grade (PG) asphalt binders, use of REOB as a blending component or modifier to produce paving asphalt binders has increased.[1] Softer asphalt binder grades in colder climates are in greater demand to reduce low temperature cracking. Additionally, the trend to use higher levels of reclaimed asphalt pavement (RAP) and reclaimed asphalt shingles (RAS) has increased the demand for softer asphalt binder grades. These factors have increased the interest and use of fluxing agents, which include non-asphaltic asphalt binder blending stocks such as REOB.

This report provides information to supplement existing publications communicating responsible use of re-refined engine oil bottoms (REOB) in asphalt binder formulations. Information is provided on current uses of REOB, available qualitative and quantitative methods to detect presence of calcium, copper, molybdenum, phosphorus, and zinc in asphalt binders, and suggestions as to how these elements might indicate the presence and amount of REOB in asphalt binders.

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SI* (MODERN METRIC) CONVERSION FACTORS

APPROXIMATE CONVERSIONS TO SI UNITS

Symbol	When You Know	Multiply By	To Find	Symbol
LENGTH				
in	inches	25.4	millimeters	mm
ft	feet	0.305	meters	m
yd	yards	0.914	meters	m
mi	miles	1.61	kilometers	km
AREA				
in^2	square inches	645.2	square millimeters	mm^2
ft^2	square feet	0.093	square meters	m^2
yd^2	square yard	0.836	square meters	m^2
ac	acres	0.405	hectares	ha
mi^2	square miles	2.59	square kilometers	km^2
VOLUME				
fl oz	fluid ounces	29.57	milliliters	mL
gal	gallons	3.785	liters	L
ft^3	cubic feet	0.028	cubic meters	m^3
yd^3	cubic yards	0.765	cubic meters	m^3
NOTE: volumes greater than 1000 L shall be shown in m^3				
MASS				
oz	ounces	28.35	grams	g
lb	pounds	0.454	kilograms	kg
T	short tons (2000 lb)	0.907	megagrams (or "metric ton")	Mg (or "t")
TEMPERATURE (exact degrees)				
^oF	Fahrenheit	5 (F-32)/9 or (F-32)/1.8	Celsius	^oC
ILLUMINATION				
fc	foot-candles	10.76	lux	lx
fl	foot-Lamberts	3.426	candela/m^2	cd/m^2
FORCE and PRESSURE or STRESS				
lbf	poundforce	4.45	newtons	N
lbf/in^2	poundforce per square inch	6.89	kilopascals	kPa

APPROXIMATE CONVERSIONS FROM SI UNITS

Symbol	When You Know	Multiply By	To Find	Symbol
LENGTH				
mm	millimeters	0.039	inches	in
m	meters	3.28	feet	ft
m	meters	1.09	yards	yd
km	kilometers	0.621	miles	mi
AREA				
mm^2	square millimeters	0.0016	square inches	in^2
m^2	square meters	10.764	square feet	ft^2
m^2	square meters	1.195	square yards	yd^2
ha	hectares	2.47	acres	ac
km^2	square kilometers	0.386	square miles	mi^2
VOLUME				
mL	milliliters	0.034	fluid ounces	fl oz
L	liters	0.264	gallons	gal
m^3	cubic meters	35.314	cubic feet	ft^3
m^3	cubic meters	1.307	cubic yards	yd^3
MASS				
g	grams	0.035	ounces	oz
kg	kilograms	2.202	pounds	lb
Mg (or "t")	megagrams (or "metric ton")	1.103	short tons (2000 lb)	T
TEMPERATURE (exact degrees)				
^oC	Celsius	1.8C+32	Fahrenheit	^oF
ILLUMINATION				
lx	lux	0.0929	foot-candles	fc
cd/m^2	candela/m^2	0.2919	foot-Lamberts	fl
FORCE and PRESSURE or STRESS				
N	newtons	0.225	poundforce	lbf
kPa	kilopascals	0.145	poundforce per square inch	lbf/in^2

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LIST OF ABBREVIATIONS AND SYMBOLS

Abbreviations

AA	atomic absorption
AASHTO	American Association of State Highway and Transportation Officials
BDA	butane de-asphalter asphalt
cps	counts per second
DOT	Department of Transportation
EDXRF	energy dispersive X-ray fluorescence
EOR	engine oil residue
FHWA	Federal Highway Administration
ICP	inductively coupled plasma spectroscopy
PAV	pressure aging vessel
PDA	propane de-asphalter asphalt
PG	performance grade
PPA	polyphosphoric acid
Ppm	parts per million
PTSi	Paragon Technical Services, Inc.
RAP	reclaimed asphalt pavement
RAS	reclaimed asphalt shingles
REOB	re-refined engine oil bottoms
RHVDB	re-refined heavy vacuum distillation bottoms
RHVDO	re-refined heavy vacuum distillation oil
RVTB	re-refined vacuum tower bottoms
SHRP	Strategic Highway Research Program
U.S.	United States
USPTO	United States Patent and Trademark Office
VTAB	vacuum tower asphalt binder
VTAE	vacuum tower asphalt extender
VTB	vacuum tower bottoms
WDXRF	wavelength dispersive X-ray fluorescence
WEO	waste engine oil
WEOR	waste engine oil residue
WODB	waste oil distillation bottoms
XRF	X-ray fluorescence
ΔT_c	delta T_c

INTRODUCTION

Asphalt, the highest boiling, non-distillable, constituent of petroleum crude oil, is a residual product from refining of petroleum. Refining operations separate low boiling point distillable fractions from crude oil, yielding the highest boiling, high molecular weight, low volatility fraction. Asphalt production is primarily accomplished through vacuum distillation of select crude oil or blends of crude oil and is technically referred to as vacuum residue. Resulting asphalt quality is directly related to the quality of crude oils or crude oil blends selected for refining operations. However, crude oils are typically selected to optimize yields and quality of the lower boiling constituents of greater economic value, rather than desired asphalt quality.[2]

Several refining processes are available to produce asphalt depending on crude source and refinery operation capabilities. Asphalt binders may be produced by direct distillation or by blending refined fractions to achieve desired physical properties necessary to meet specifications. Blending of two or more refined asphalt residua is also a common method for preparation of paving asphalt binders. Blending available residua is generally preferred to facilitate inventory control and reduce tankage demand. Figure 1 exhibits refinery process sources of asphalt ranging from soft to hard.
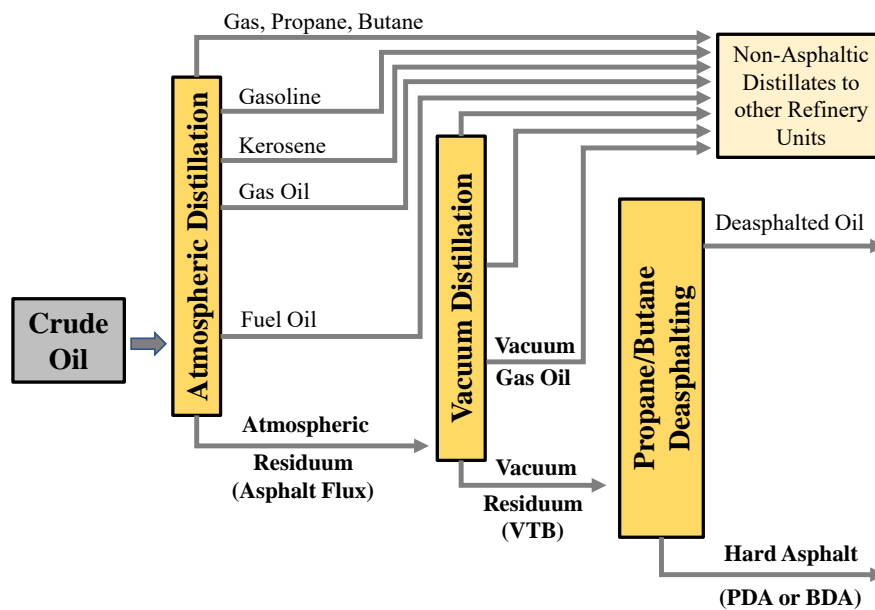
Figure 1. Schematic diagram of refinery processes and resulting asphalt. (Figure source PTSi)

Specification grade asphalt binders may also be blended to produce desired asphalt binder specifications. For example, an asphalt binder supplier may inventory two specification grades of asphalt binders, one soft grade and the other a harder grade. This facilitates preparation of intermediate grade asphalt binders by simple tank or on-demand blending.[3] Blending charts or mathematically modeled blending programs are commonly used for estimating blending proportions to yield desired physical properties.

The process of blending soft oils or "fluxes" with harder asphalt to produce intermediate grades is technically referred to as "fluxing." Fluxing of hard asphalt with lube stock for use as paving asphalt binder dates to as early as the late 1800s.[4] Since at least 1983, residue from re-refining of

1

waste engine oils (WEO), referred to as re-refined engine oil bottoms (REOB) or vacuum tower asphalt extender (VTAE), has been used as a fluxing agent for asphalt binder blending.[5] The terms REOB or VTAE discussed in this document refer specifically to the residue from the vacuum distillation of a WEO in the re-refining process. REOB is not cleaned up (filtered/centrifuged) WEO and it is not the residuum from simple atmospheric distillation. An exemplary asphalt blending chart is shown in Figure 2. Depicted are predicted results of specification performance grade (PG) blending of PG58-28 asphalt binder from REOB and four harder asphalts: PG64-22 asphalt binder, PG70-16 asphalt binder, vacuum tower residuum (VTB), and hard asphalt (propane de-asphalter asphalt (PDA)). Comparatively, predicted results of specification grade blending of PG58-28 asphalt binder from asphalt flux and hard asphalt, and blending of PG58-28 asphalt binder from PG64-22 asphalt binder blended with PG52-34 asphalt binder are presented.
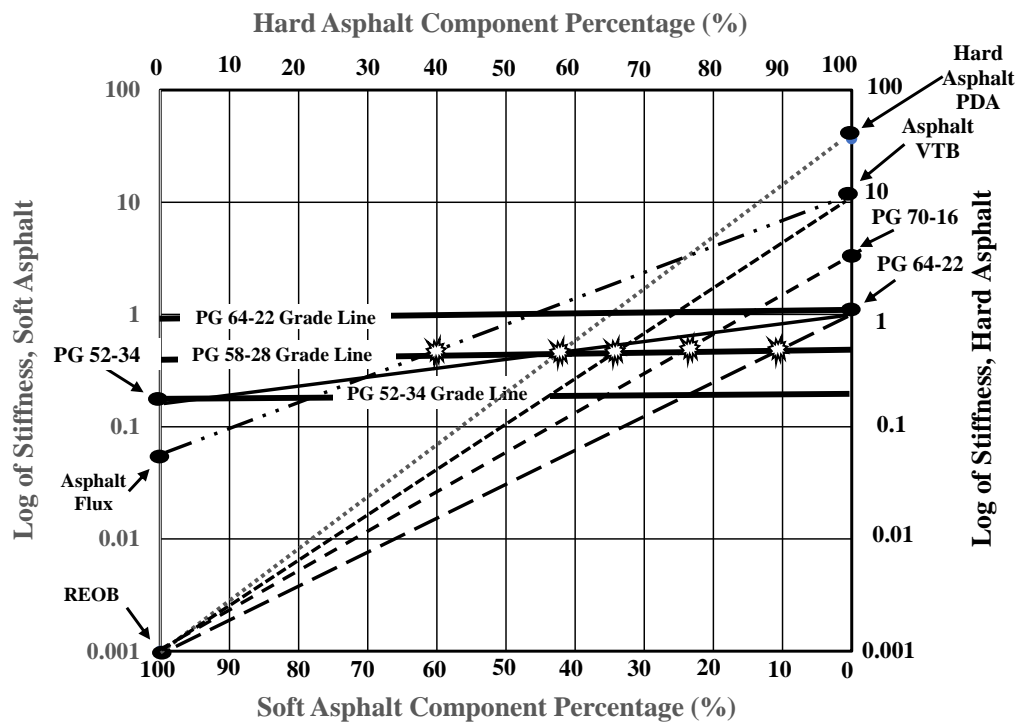
Figure 2. Specification grade asphalt blending chart at 64°C. (Figure source PTSi)

Since the end of the Strategic Highway Research Program (SHRP)[1] in 1993 and implementation of the Superpave PG binder specification in the mid 1990s, use of REOB as a blending component or modifier to produce paving asphalt binders has increased.[1] Softer asphalt binder grades in colder climates are in greater demand to reduce low temperature cracking. Additionally, the trend to use higher levels of reclaimed asphalt pavement (RAP) and reclaimed asphalt shingles (RAS) has increased the demand for softer asphalt binder grades. These factors have increased the interest and use of fluxing agents, which include non-asphaltic asphalt binder blending stocks such as REOB.

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[1]The Strategic Highway Research Program (SHRP) was a 5-year, $150 million applied research program authorized by the Surface Transportation and Uniform Relocation Act of 1987.

Though REOB has been used as a blend component for asphalt binder production for a number of years, some State Departments of Transportation (State DOTs) have expressed concern with pavement durability and may restrict its use. A 2022 review of published State DOT specifications conducted by the authors revealed five State DOTs allowing usage or limited usage of REOB (Figure 3). In these States, there was no common maximum usage limit with one State DOT allowing a maximum of 5.0 percent and a second allowing a maximum of 8.0 percent; the remaining three State DOTs allowing use of REOB had no limits on usage. Thirty-one State DOTs had no stated restrictions, with two requiring prior approval of possible use, and fourteen State DOTs did not allow use of REOB.
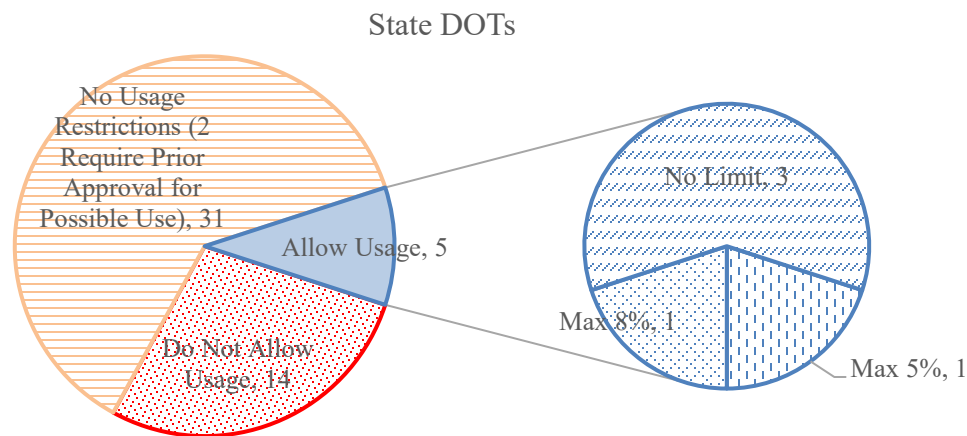

State DOTs

No Usage Restrictions (2 Require Prior Approval for Possible Use), 31

Allow Usage, 5

Do Not Allow Usage, 14

No Limit, 3

Max 8%, 1

Max 5%, 1

Figure 3. Summary of REOB usage by State DOTs based on 2022 review of published specifications. (Figure source PTSi)

BACKGROUND

Petroleum derived lubricating oils are used for a variety of applications to include lubrication and protection of engine parts as well as heat and power transfer. These oils become contaminated or otherwise degraded depending on the application and operating environment and are discarded in the form of WEO. Although the WEO is contaminated and may be extremely dirty, it will never wear out. WEO is recyclable and can be re-refined and made into lubricating oils that meet the same specifications as non-recycled engine lubrication oil. In 2018, domestic consumption of lubricating oil was estimated to be 2.47 billion gallons. More than 1.3 billion gallons of the reported consumption was stated to be WEO available for collection and reuse.[6] Approximately 69 percent of this WEO was reused as home heating oil (18 to 28 percent) or re-refined into new base oils (41 to 51 percent). The remaining 31 percent was either unaccounted for at the source (e.g., burned in space heaters by those generating WEO or, recycled/reused on site) or disposed of improperly (e.g., disposal in landfills or into storm drains by "do-it-yourself" (DIY) oil change service WEO generators.[6] If the approximate 429 million gallons of WEO were reused, it might have produced enough energy to power more than 800 thousand homes; or provide approximately 229 million quarts of high-grade motor oil.[7]

Recycling of WEO has received increased attention of late, mainly due to environmental problems arising with increasing pollution levels and the need to conserve natural resources. The oldest way of treating WEO is treatment with acid and clay to remove pollutants.[8] This process is not typically used due to the low degree of sustainability. Vacuum distillation and clay treatment is

also a process that allows removal of impurities from WEO to transform it into reusable base oil.[8] A process with improved efficiency and sustainability, the re-refining process, uses vacuum distillation and hydrogenation.[1,8] This process is similar to vacuum distillation and clay treatment with clay treatment being replaced by hydrogenation. Figure 4 presents a flow chart of the vacuum distillation and hydrotreating process for re-refining of WEO. In the process, WEO is dehydrated at atmospheric pressure to remove water and light hydrocarbons. Atmospheric distillation separates the heavier fuel contaminants from the reusable lubricating oil. Vacuum distillation is used to separate lighter molecular weight oils yielding crude re-refined base oil and a non-distillable residue as REOB. The crude re-refined base oil is finished via wipe-film evaporation and hydrotreated into re-refined base oil stocks for reuse in production of new lubricating oil.
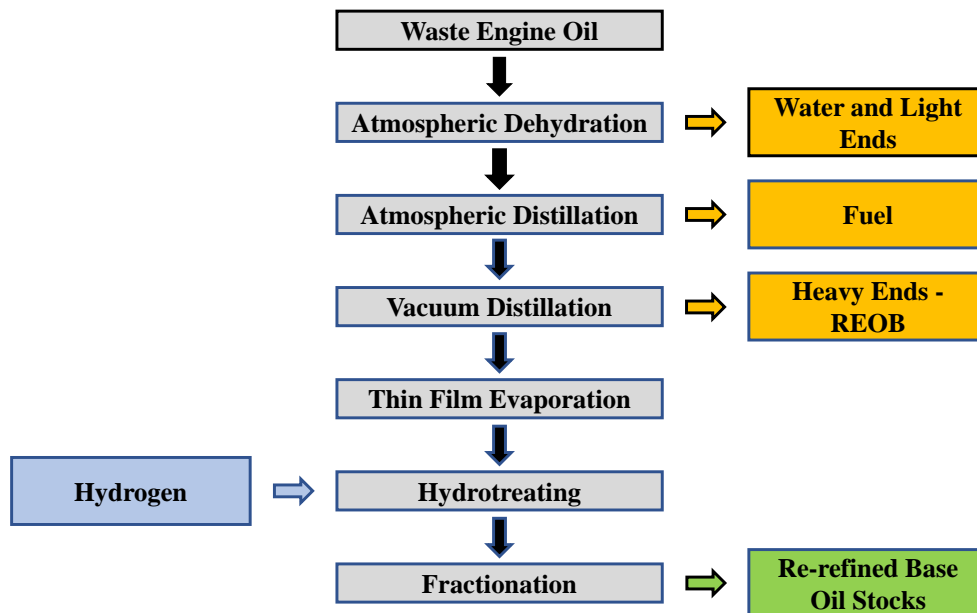

Figure 4. Flow chart of the vacuum distillation and hydrotreating process for re-refining of WEO. (Figure source (8), PTSi)

Automobile engine oil consists of approximately 90 percent base lubrication oil blended with various additives to enhance engine performance as well as provide long-term oil performance and durability.[9] REOB accounts for about 12 percent of the total WEO composition. It is composed of higher boiling components, generally containing paraffinic lubrication oil, various residual additives, and engine wear metals. REOB can contain as much as 60 percent residual lubricating oil, about 4 percent polymer and various soluble metals representing about 5 percent of total REOB composition.[10]

REOBs are viscous liquids at room temperature. Much like chemical and physical properties of asphalt vary depending on crude source, the REOB properties vary depending on WEO source. Comparatively, the full chemical and physical variability of REOB is not known. ASTM International published D8125-18 "Standard Specification for Re-Refined Engine Oil Bottoms (REOB)/Vacuum Tower Asphalt Extender (VTAE)" in 2018; its use is not a Federal requirement.[11] Table 1 presents the specifications for REOB/VTAE used in pavement construction as specified by ASTM D8125-18.

Table 1. ASTM D8125-18 Specifications for REOB/VTAE Used in Pavement Construction. [Table source (11)]

Property	Typical Result
Flash Point, min, degrees Celsius (AASHTO T 48, ASTM D92)	>232
RTFO Weight Loss, max, percent (AASHTO T 240, ASTM D2872)	1.0
Solubility, min, percent (AASHTO T 44, ASTM D7553)	98.0
Viscosity at 60°C, centipoises (cP) (AASHTO T 316, ASTM D4402/D4402M)	200–5,000
Ash Content, max, percent (ASTM D8078 or ASTM E1131)	7.0

Note: AASHTO = American Association of State Highway and Transportation Officials. Use of these AASHTO specifications is not a Federal requirement.

REOB is the name typically used by State DOTs and the Federal Highway Administration (FHWA) while VTAE is used by the manufacturers of the product.[5,11] However, over the past 40 years, the non-distillable residue from re-refining of WEO has been referred to by a variety of names. Some of these names are not consistent with the residuum from vacuum distillation described in Figure 1.[1] Table 2 lists various names that have been associated with REOB.

Used responsibly, REOB can be an effective additive for modifying asphalt binder to improve low temperature PG properties.[1,10,26,42,49] Typical dosage rates, reported by REOB manufacturers, range from 4 to 8 percent to reduce both high- and low- binder PG (AASHTO M 320) as much as one full grade.[1] The degree of both high- and low-binder grade change is dependent on the REOB source, properties (e.g., viscosity), dosage, and the compatibility and interaction with the base asphalt being modified. Those considering use of REOB in asphalt binders and asphalt mixtures are encouraged to investigate specific materials of interest to establish practical limits of usage.

Table 2. Names Associated with REOBs. [Table source (5)]

Acronym	Name
–	Asphalt Flux
–	Asphalt Blowdown
EOR	Engine Oil Residue
REOB	Re-refined Engine Oil Bottoms
RHVDB	Re-refined Heavy Vacuum Distillation Bottoms
RHVDO	Re-refined Heavy Vacuum Distillation Oil
RVTB	Re-refined Vacuum Tower Bottoms
VTAB	Vacuum Tower Asphalt Binder
VTAE	Vacuum Tower Asphalt Extender
VTB	Vacuum Tower Bottoms
WEOR	Waste Engine Oil Residue
WODB	Waste Oil Distillation Bottoms

Note: – = not applicable.

COMMON CONCERNS

Some concerns about REOB usage in asphalt binders are listed below and addressed in the following sections.

- What are potential adverse effects of REOB?
- How does one know if an asphalt binder contains REOB?
- How can one determine how much REOB is used in an asphalt binder?

Effects of REOB

Over the past four decades, REOB modification of asphalt binder for use in asphalt pavements has been an ongoing topic of interest within the asphalt paving industry. A considerable volume of literature has been published regarding various aspects of the use of REOB or WEO as a modifier of asphalt binders. Topical focus of published research can be separated into the following areas:

- use of REOB as a binder extender, modifier, or recycling agent and the effects of use levels on asphalt binder properties and performance,
- laboratory and field performance of asphalt mixtures containing REOB modified asphalt binder,
- impact of laboratory and field aging on performance properties of asphalt binder and mixture, and
- detection and quantification of REOB in asphalt binder using various instrumental methods.

A non-exhaustive list of literature references with respect to the stated topical focuses is presented in Table 3 along with an overall summary of conclusions.

In summary, the published research presented in Table 3 suggests that conclusions of the effects of REOB on asphalt binder performance are conflicting at best. Some research indicates adverse effects of REOB on the performance of asphalt binder with respect to binder aging characteristics and potential cracking of in-service asphalt pavements.[17,18,20,21,22,2324,29,35,39] Other research shows improved or equivalent performance of asphalt mixtures with asphalt binder containing REOB compared to asphalt mixtures with non-REOB containing asphalt binders of the same grade.[25,26,28,31,33,36,40] Despite conflictions of research conclusions, suggestions are that asphalt binder containing low dosages are innocuous while higher dosages may be detrimental to performance.[1,42,43]

Early evaluations of potential use of REOB in production of asphalt binders indicated that paving-grade asphalt binders could be produced by blending vacuum distillation residues from WEO at 2 to 20 percent concentration with refined hard asphalt residue.[12,13,14] The effect of blending REOB with hard asphalt residue was to soften the asphalt residue at all temperatures compared to the base asphalt residue. Over the range of REOB concentrations studied, the observed effects were approximately linear with increasing concentrations of REOB, and the effect of a given concentration on the base asphalt residue were greater, relatively speaking, at lower temperatures. Oxidative hardening properties of blends of REOB and asphalt residue were similar to one another and to standard asphalt binder at moderate temperatures, but at high temperatures these rates typically increased. Wear metals present in REOB were reported to possibly cause increased oxidative hardening due to auto-oxidation reactions catalyzed by metals and their oxides contributing to hydrocarbon degradation.[14] These findings were supported by some researchers [16,22,23] while others found no increase in the rate of aging of blends of REOB and asphalt residue when the pressure-aging vessel (PAV) is used, reporting that REOB modified asphalt mixtures performed as well or better than control asphalt mixtures in laboratory studies.[25,26]

Use of REOB or other fluxing agents for softening and adjusting low temperature parameters of asphalt binders can affect delta $T_{critical}$ or ΔT_c.[1,57,58,59] Research indicates that more negative values of ΔT_c appear to be correlated to non-load related cracking and other destresses related to poor relaxation properties.[42,58,59] While not every source of REOB affects ΔT_c to the same extent, REOB typically affects ΔT_c in REOB modified asphalt binders through more negative values of ΔT_c, as compared to the base asphalt binder being modified. The most significant effect of REOB on ΔT_c is typically directly related to dosage level, with higher REOB contents exhibiting more negative values of ΔT_c.[1,42,49,57,58,59]

Table 3. Authors of Topical Literature in Reference. (Table source PTSi)

Binder Extender/Modifier/ Recycling Agent	Mixture Performance Laboratory/Field	Binder/Mixture Aging	Detection/Quantification of REOB
Herrington, P.R., (1992) [12]	Herrington, P.R., et al. (1993) [13]	Herrington, P.R., et al. (1998) [14]	Hesp, S.A.M., et al. (2009) [17]
Romera, R. et al. (2006) [15]	Hesp, S.A.M., et al. (2009) [17]	Hesp, S.A.M., et al. (2009) [17]	Soleimani, A., et al. (2009) [18]
Villanueva, A., et al. (2008) [16]	Soleimani, A., et al. (2009) [18]	Wright, L., et al. (2011) [21]	Zamhari, K.A., et al. (2009) [0]
Wright, L., et al. (2011) [21]	D'Angelo, J.A., et al. (2013) [26]	Rubab, S., et al. (2011) [22]	Hesp, S.A.M., et al. (2010) [20]
Rubab, S., et al. (2011) [22]	Oliveira, J.R.M., et al. (2013) [27]	Burke, K., et al. (2011) [0]	Hesp, S.A.M., et al. (2012) [24]
Burke, K., et al. (2011) [23]	Golalipour, A. (2013) [28]	D'Angelo, J.A., et al. (2012) [25]	Arnold, T.S., et al. (2015) [37]
D'Angelo, J.A., et al. (2012) [25]	De Dene, C.D., et al. (2014) [30]	Golalipour, A. (2013) [28]	Barbarack, R.C., et al. (2016) [41]
Golalipour, A. (2013) [28]Johnson, K.A.N., et al. (2014) [29]	Mensching, D.J. et al. (2014) [32]	Mensching, D.J. et al. (2014) [32]	Arnold, T.S., (2017) [45]
De Dene, C.D., et al. (2014) [30]	Wielinski, J.C., et al. (2014) [0]	Wielinski, J.C., et al. (2015) [36]	Kaskow, J., et al. (2018) [51]
Golalipour, A., et al. (2014) [31]Mensching, D.J. et al. (2014) [32]	Uzarowski, L., et al. (2015) [39]	Mogawer, W.S., et al. (2015) [38]	Karki, P., et al. (2019) [54]
Jia, J., et al. (2014) [34]	Wielinski, J.C. et al. (2015) [0]	Wielinski, J.C. et al. (2015) [40]	Yan, C., et al. (2020) [55]
Zuammanis, M., et al. (2014) [35]	Bennert, T., et al. (2016) [42]	Bennert, T., et al. (2016) [43]	
Wielinski, J.C., et al. (2015) [36]	Cooper, S.B.Jr., et al. (2017) [46]	Dokandari, P.A., et al. (2017) [47]	
Bennert, T., et al. (2016) [42]	Fernandes, S., et al. (2017) [48]	Mensching, D.J., et al. (2017) [49]	
Bennert, T., et al. (2016) [43]	Mamun, A.A., et al. (2018) [50]	Li, H., et al. (2019) [53]	
Paliukaite, M., et al. (2016) [44]	Li, H., et al. (2019) [53]	Cai, F., et al. (2022) [56]	
Fernandes, S., et al. (2017) [48]			
Mensching, D.J., et al. (2017) [49]			
Wu, s., et al. (2018) [52]			

Note: Some references may relate to multiple topical focuses.

The response of ΔT_c to REOB content has been shown to exhibit more negative values of ΔT_c with increasing REOB content.[1,49,57,58] Producers limiting REOB use levels in asphalt binder formulations may be of better service than State DOT ΔT_c parameter specification limits alone. With respect to softer asphalt binders with lower low-temperatures, extended PAV aging (e.g., 40-hour PAV) may be desirable. Instrumental methods are available to detect presence and estimate content of REOB in asphalt binders.[17,18,0,20,24,37,41,45,51,54,55] Implementation of such methods may also be more functional than limiting REOB use by aging and ΔT_c parameter specifications.

REOB Detection and Quantification

It is not possible to directly determine the presence of or measure the exact content of REOB in asphalt binder as REOB. However, since asphalt binder does not naturally contain some elements present in REOB, the assumption could be made that asphalt binder containing these elements perhaps contains REOB. In this case it would then be possible to calculate the estimated content of REOB used to produce the asphalt binder.

Recall that engine oil consists of approximately 90 percent base lubrication oil blended with various additives to enhance engine performance and provide long-term oil performance and durability.[9,10] Typical additive packages might include heat stabilizers, acid scavengers, boundary lubricants, viscosity modifiers, polymers, and wear reduction additives. Additives may include compounds such as calcium phenate, which contains calcium; molybdenum disulfide, which contains molybdenum and sulfur; and zinc dithiophosphate, which contains zinc, sulfur, and phosphorus. REOB contains all of these non-distillable components from the WEO, as well as engine wear metals such as copper and iron accumulated due to normal engine wear.[9,10] Table 4 presents an example of typical engine oil components and suggested sources of trace metals it may contain. Asphalt binder analysis for trace metals, calcium, copper, molybdenum, phosphorus, and zinc, can provide an indication of the presence of REOB. While these elements can be used to quantify the estimated amount of REOB contained in an asphalt binder it is best to focus on calcium and zinc for quantification. Phosphorus may also be used; however, the presence and level of phosphorus may also indicate possible presence of other additives such as polyphosphoric acid (PPA). Copper and molybdenum are indicators of the presence of REOB but are generally at such low levels that quantification is limited.

Table 4. Engine Oil Components. (Table source PTSi)

Paraffinic Lube Stock (approximately 90 percent)			
Description	**Purpose**	**Chemistry**	**Contains**
Neutral base oil.	Primary lubrication.	Polyalphaolefins, aliphatic hydrocarbons, alkanes, cycloalkanes, and esters.	–

Additives (approximately 10 percent)			
Description	**Purpose**	**Chemistry**	**Contains**
Detergents.	Control chemical breakdown.	Magnesium sulfonates.	Magnesium.
Antioxidants and acid scavengers.	corrosion inhibitor.	Potassium hydroxide, calcium phenate, amines, and phenols.	Potassium calcium.
Metal deactivators.	Stabilizer.	Chelating ligands.	–
Boundary lubricants.	Provides lubricity.	Molybdenum disulfide.	Molybdenum, sulfur.
Wear reducing additives.	Provides lubricity.	Zinc dithiophosphate, calcium sulfonates.	Calcium, phosphorous, sulfur, zinc.
Viscosity modifiers.	Stabilize viscosity.	Polymers.	–

Note: – = not applicable; WEO contains copper and iron as engine wear metals.

Several instrumental analytical approaches are available for determination and quantification of calcium, copper, phosphorus, molybdenum, and zinc in asphalt binders. Such analytical approaches include graphite furnace atomic adsorption (AA) spectroscopy (furnace AA),[60] inductively coupled plasma spectroscopy (ICP),[61] energy dispersive x-ray fluorescence spectroscopy (EDXRF),[62] and wavelength dispersive x-ray fluorescence spectrometry (WDXRF).[62] This discussion will focus on x-ray fluorescence. EDXRF and WDXRF will be collectively referred to simply as x-ray fluorescence (XRF), both approaches can be used for asphalt binder analysis. The WDXRF instruments may offer better signal intensity and resolution; however, this comes at a considerable comparative instrument investment, and longer data acquisition time that creates a risk of overheating samples. EDXRF instruments may be more suitable for asphalt binder analysis at considerable reduced investment, while providing decreased data acquisition time and reduced sample heating. EDXRF instruments are available in both benchtop and handheld versions. Handheld instruments, while unique, may not be the more practical approach for asphalt binder analysis.

All instrumental approaches discussed are capable of identifying the presences of and determining content of metals in asphalt binders. Some may be more suitable than others. For example, sample preparation for AA and ICP requires reducing the asphalt to sufficient viscosity to be sprayed through a nebulizer into a flame. Compared to the AA and ICP methods,the XRF approach requires no sample preparation allowing phosphorus to be directly determined in the asphalt matrix as received. There are XRF methods available for determining the amount of phosphorus in oil matrices,[60, 61, 62,62] however, there are no available standardized XRF methods for determination of phosphorus in an asphalt matrix. In fact, standard test methods for determining phosphorus in asphalt matrices with AA or ICP are non-existent as well. Some suppliers and State DOTs have developed XRF methods for determination of phosphorus in asphalt matrices.[41,54] These XRF methods typically use existing test methods for determination of calcium, copper, molybdenum, phosphorus and zinc in other matrices as the starting point for preparing samples. The remainder of this discussion will focus on EDXRF.

The EDXRF method can be used to analyze many types of matrices including solid, liquid, powder, etc. The elemental range typically includes sodium to uranium on the periodic table. The

concentration range is typically from (sub) parts per million (ppm) levels to 100 percent. The elements with high atomic numbers have better detection limits than the lighter elements with low atomic numbers. In an EDXRF, X-ray produced by the source, an X-ray tube, irradiate the sample. The elements present in the sample will emit fluorescent X-ray radiation with discrete energies that are characteristic for these elements. By measuring the sample's radiation energies, it is possible to determine which elements are present (qualitative analysis); therefore, determination of calcium, copper, molybdenum, phosphorus and zinc presence in asphalt binder is a straightforward procedure. By measuring the intensities of the emitted energies, it is possible to determine how much of each element is present in the sample (quantitative analysis); nonetheless this method is somewhat more involved.

Typically, XRF instruments do not normally require frequent calibration; however, calibration for specific analysis method elements is important upon initial implementation of such methods. Instruments should be calibrated in accordance with the manufacturer's recommendations. Custom prepared calibration standards for elements critical to asphalt analysis (calcium, copper, molybdenum, phosphorous, sulfur, and zinc) are readily available from commercial sources. Calibration is also important when analysis-critical components, such as X-ray source or detector, of the instrument are maintained or replaced.

In analytical chemistry, a calibration curve, also known as a standard curve, is a general method for determining the concentration of a substance in an unknown sample by comparing the unknown to a set of standard samples of known concentration. The calibration or standard curve is a plot of how the instrumental response changes with the concentration of the substance to be measured. This is the analytical method used for determination of the estimated concentration of REOB in asphalt binder samples.

After ensuring instrument operation and calibration, the first step in asphalt binder analysis is preparation of an asphalt binder calibration curve specific to the element of interest. In the current case the elements of interest are calcium, copper, molybdenum, phosphorous, and zinc or more specifically, REOB. In the asphalt binder calibration curve step, REOB modified asphalt binder blends are prepared by addition of REOB to non REOB containing asphalt binder. REOB modified asphalt binders are prepared with increasing quantities of REOB ranging from zero to a desired maximum depending on asphalt binder specification limits or expected use levels. Limiting the total number of calibration samples reduces the number of XRF runs. An eleven-point calibration curve consisting of a sample of neat asphalt binder, or no REOB, and ten samples of this binder modified with equally spaced increasing loading of REOB to a target maximum is suggested. Since some State DOTs limit use levels to less than 8.0 percent, an acceptable range might be 0.0 to 10.0 percent in 1.0 percent increments, as in Table 5. Figure 5 presents a calibration curve of PG64-22 modified with REOB with 11-point loadings from 0.0 to 10.0 percent in 1.0 percent increments.
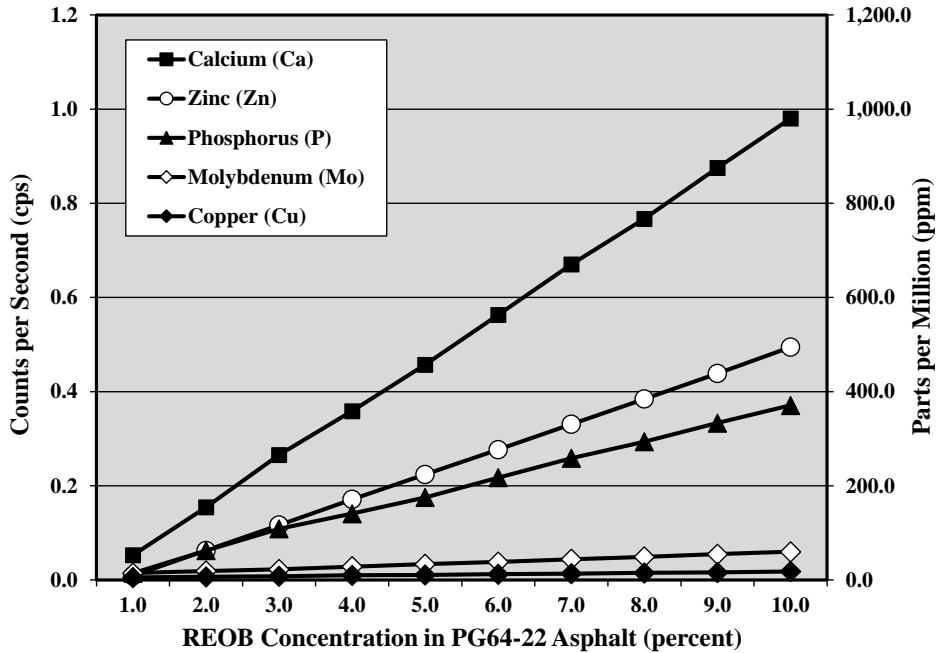
Figure 5. EDXRF calibration curve for PG64-22 asphalt binder containing REOB loadings from 0.0 to 10.0 percent. (Figure source PTSi)

To cover lesser or greater ranges increments could be varied maintaining eleven points of calibration. For example, 0.0 to 5.0 percent or 0.0 to 20.0 percent. Table 5 provides an example of points on an eleven-point calibration curve for a range up to 20.0 percent.

Table 5. Points of Calibration Curve for Each Base Asphalt. (Table source PTSi)

Percent REOB	Weight of Asphalt Binder (g)	Weight of REOB (g)	Total Weight (g)
0.0	100.0	0.0	100
1.0	99.0	1.0	100
2.0	98.0	2.0	100
3.0	97.0	3.0	100
4.0	96.0	4.0	100
6.0	94.0	6.0	100
8.0	92.0	8.0	100
10.0	90.0	10.0	100
14.0	86.0	14.0	100
16.0	84.0	16.0	100
20.0	80.0	20.0	100

Instrumental quantification methods are limited by the accuracy and effectiveness of the calibration generated. With any instrumental method used to quantify elements and estimate REOB content in asphalt binders, it is important to consider the impact of significant changes in binder source or chemistry on the effectiveness of the calibration curve. For example, overlapping of fluorescing energies of phosphorus and sulfur caused by asphalt binders with higher concentrations of sulfur may confound or interfere with accurate determination of the amount of phosphorus in a sample.[64] Spectral overlap of sulfur on phosphorus can be lessened through use of beam filters. To address possible confounding of phosphorus concentration determination, at

least four base asphalts with sulfur content ranging from 2.0 to 6.0 percent should be considered in development of calibration curves. A calibration curve using linear regression is obtained after the analysis of the prepared calibration standards in the XRF spectrometer and plotting intensity readings in terms of counts per second (cps) versus concentration in percent of calcium, copper, molybdenum, phosphorus, and zinc as REOB. This is done for each of the base asphalt binders used in preparation of calibration samples. Accuracy is improved with a correlation coefficient of 0.9950 or better.

Evaluation of unknown samples is straightforward: the samples can be analyzed in the same manner used for analyzing the samples prepared for development of calibration curves. Determination of the percent calcium, copper, molybdenum, phosphorus and zinc representing REOB are made using the prepared calibration curves.

SUMMARY

REOB/VTAE, the residue from re-refining of WEO, has been used as a fluxing agent for asphalt binder blending for more than forty years. The first reported use of REOB as a paving asphalt binder modifier was in 1992. Since the end of the SHRP in 1993 and implementation of the SuperPave PG binder specification in the mid 1990s, use of REOB as a blending component or modifier to produce paving asphalt binders has increased. Softer asphalt binder grades in colder climates are in greater demand to reduce low temperature cracking. Additionally, the trend to use higher levels of RAP and RAS has increased the demand for softer asphalt binder grades. These factors have enhanced interest and use of fluxing agents which include non-asphaltic asphalt binder blending stocks such as REOB.

Though REOB has been used as a blend component for asphalt binder production for a number of years, some State DOTs do not allow use of REOB, and others may restrict its use. While most State DOTs do not indicate restrictions on the use of REOB, there is no common maximum usage limit. State DOTs with specified usage levels have maximum limits from 5.0 to 8.0 percent.

This report has discussed the effects of REOB on asphalt binder properties and performance as well as methods to detect the presence and amount of REOB. Indications are that REOB may improve low temperature PG of asphalt binder; however, this is dependent on REOB source and loading level of REOB in the asphalt binder. Presence of REOB can be indicated via instrumental analysis. Instrumental methods are available to efficiently identify elements present in REOB to aid in quantification of estimated dosage levels of REOB in asphalt binders.

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