Memorandum

U.S. Department
of Transportation

**Federal Highway
Administration**

6300 Georgetown Pike
McLean, Virginia 22101

Subject: **ACTION**: LTPP Directive M-36
Material Test Protocol P-76 (AE11) for Asphalt Binders for Recycled Engine Oil Bottoms (REOB) Using Handheld X-Ray Fluorescence Spectrometers

Date: March 9, 2022

LARRY J WISER Digitally signed by LARRY J WISER Date: 2022.03.09 09:51:38 -05'00'

From: Larry Wiser
Long-Term Infrastructure Performance Team

Reply to
Attn of: HRDI-30

To: Dr. Ramon Bonaquist, PM - LTPP SPS-10 Material Testing Contract

Attached is the Long-Term Pavement Performance (LTPP) Program Directive M-36. This directive updates developing the data set contained in table TST_AE11 for Analysis of Asphalt Binders for Recycled Engine Oil Bottoms (REOB) Using Handheld X-Ray Fluorescence Spectrometers

Please ensure that all personnel involved with Material Test Protocol P-76 are aware of this new directive. Should you have any questions or would like to discuss this directive, please do not hesitate to contact Larry Wiser via email at larry.wiser@dot.gov (202) 493-3079

Attachments (1)

FHWA:HRDI-30 L. Wiser:jeh:202-493-3079:03/09/22
File: M:\LTPP Directives\MATERIALS\M-36
cc:
Gonzalo Rada
Larry Wiser
Gabe Cimini
Directive Binder
LTPP Team
Official file

LONG TERM PAVEMENT PERFORMANCE PROGRAM DIRECTIVE

For the Technical Direction of the LTPP Program

Program Area:	Materials	Directive Number:	M-36
Date:	March 9, 2022	Supersedes:	N/A
Subject:	Materials Test Protocol P-76 (AE11) for Analysis of Asphalt Binders for Recycled Engine Oil Bottoms (REOB) Using Handheld X-Ray Fluorescence Spectrometers		

The following material test protocol documents the method used by the Long-Term Pavement Performance (LTPP) program for developing the data set contained in table TST_AE11 for Analysis of Asphalt Binders for Recycled Engine Oil Bottoms (REOB) Using Handheld X-Ray Fluorescence Spectrometers. This method is based on the information contained in FHWA-HRT-18-043 "Field Analysis of Asphalt Binders for Recycled Engine Oil Bottoms (REOB) Using Handheld XRF Spectrometers."

Prepared by: TSSC

Approved by:

JEAN NEHME Digitally signed by JEAN NEHME Date: 2022.03.09 11:03:03 -05'00'

Jean A. Nehme, Ph.D., P.E.
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PROTOCOL P76
Test Method for Analysis of Asphalt Binders for Recycled Engine Oil Bottoms (REOB) using Handheld X-Ray Fluorescence Spectrometers (AE11)

This protocol describes the analysis procedure used by the Federal Highway Administration (FHWA) to estimate the recycled engine oil bottoms (REOB) content of asphalt binder samples obtained from Long-Term Pavement Performance (LTPP) test sections.

SCOPE

This protocol may involve hazardous materials, operations, and equipment. The protocol does not purport to address all safety issues associated with its use. It is the responsibility of whoever uses this protocol to consult and establish appropriate safety and health practices and to determine the applicability of regulatory limitations prior to use of the protocol.

APPLICABLE DOCUMENTS

"Field Analysis of Asphalt Binders for Recycled Engine Oil Bottoms (REOB) Using Handheld XRF Spectrometers" FHWA-HRT-18-043.

SIGNIFICANCE AND USE

REOB is used as an additive in hot-mix asphalt (HMA) pavement applications, but it may affect the performance of these HMA pavements. Since they are waste products of varying composition, analyzing for them in asphalt binders presents a challenge. X-ray fluorescence (XRF) spectroscopy has been used successfully to determine the approximate amounts of REOB present in asphalt binders.

REOB is an oily black material that is liquid at room temperature. It contains the remains of the additives that were present in the original engine oil. These include polymers, zinc dithiodialkylphosphate, calcium phenate, and molybdenum disulfide. The metals in these additives (calcium, copper, zinc, and molybdenum) can be used as markers for determination of the REOB content in asphalt binders.

CALIBRATION

Calibration is required to prepare the analyses to be used in estimating the percentage of REOB in the binder. Calibration does not require the use of the same asphalt binder used in the mix to be evaluated. LTPP binders were not used in the calibration to determine the REOB content values of the samples stored in the LTPP Pavement Performance Database (PPDB).

It is recommended that 3 different asphalts and 3 different REOB samples be included in the calibration, which provides 9 data sets –e.g., Asphalt 1 blended with REOB 1, Asphalt 1, blended with REOB 2, etc.). (Note: 3 different asphalts and 3 different REOB samples were used to generate the REOB content for the LTPP asphalt binders.)

Samples should be blended to the following REOB levels: 2, 5, 8, 10, and 20 percent REOB for a total of 45 blends – 9 sets of asphalt/REOB combinations times 5 different REOB levels. Using the XRF, analyze the blends for the calcium, copper, zinc, and molybdenum contents in parts per million (ppm).

For the blend of Asphalt 1 with REOB 1, develop a linear regression of the line through the data points as demonstrated in Figure 1.

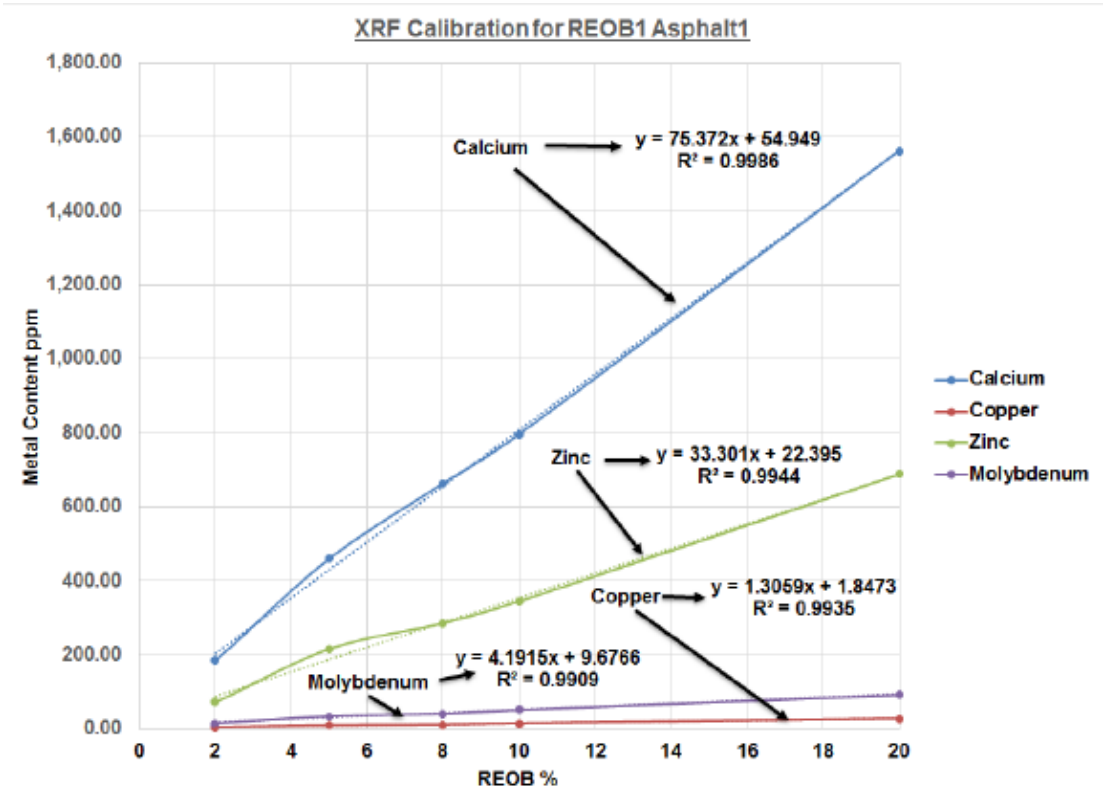

Figure 1. Regression of Metal Content versus REOB content for blends of Asphalt 1 with REOB 1

Develop linear regressions for metal content of the four metals for each of the remaining 8 asphalt-REOB blends.

The calibration will result in a series of 36 equations (9 combinations of REOB and asphalt times 4 metals) that may be used to estimate the REOB content.

These 36 equations may be set up in a spreadsheet to allow for estimating REOB content of a sample such as demonstrated by Figure 2. The 36 equations developed by FHWA for the calibration used to determine the REOB content of the LTPP samples may be found on the FHWA Chemistry Laboratory website at https://www.fhwa.dot.gov/publications/research/infrastructure/pavements/18043/reobtool.xlsx.

Blend	Calcium	Copper	Zinc	Molybdenum	Average
REOB 1 Asphalt 1					
REOB 2 Asphalt 1					
REOB 3 Asphalt 1					
REOB 1 Asphalt 2					
REOB 2 Asphalt 2					
REOB 3 Asphalt 2					
REOB 1 Asphalt 3					
REOB 2 Asphalt 3					
REOB 3 Asphalt 3					
Average					

Figure 2. Spreadsheet For Use in Estimating REOB Content

SAMPLE PREPARATION

A 2-ounce sample of asphalt binder is required for use with the XRF.

Sample is placed in small cup, such as that shown in Figure 3, and allowed to cool to room temperature.

Figure 3. Sample Cup for Use in XRF Analysis

TEST PROCEDURE

Analyze the sample using the XRF for calcium, copper, zinc, and molybdenum content in ppm.

Using the 9 equations (1 equation for each REOB/asphalt combination) associated with the calcium content, use the calcium content result from the XRF to estimate the REOB content of the sample from each equation.

Using the 9 equations (1 equation for each REOB/asphalt combination) associated with the copper content, use the copper content result from the XRF to estimate the REOB content of the sample from each equation.

Using the 9 equations (1 equation for each REOB/asphalt combination) associated with the zinc content, use the zinc content result from the XRF to estimate the REOB content of the sample from each equation.

Using the 9 equations (1 equation for each REOB/asphalt combination) associated with the molybdenum content, use the molybdenum content result from the XRF to estimate the REOB content of the sample from each equation.

The 36 resulting estimates should be entered into a table such as demonstrated by Figure 4.

Blend	Calcium	Copper	Zinc	Molybdenum	Average
REOB 1 Asphalt 1	2.34	3.18	3.74	4.13	**3.35**
REOB 2 Asphalt 1	3.09	4.93	4.87	4.30	**4.30**
REOB 3 Asphalt 1	2.20	3.35	3.78	3.15	**3.12**
REOB 1 Asphalt 2	4.97	6.64	5.30	5.40	**5.58**
REOB 2 Asphalt 2	5.27	6.91	5.68	6.60	**6.12**
REOB 3 Asphalt 2	5.88	9.64	6.49	6.47	**7.12**
REOB 1 Asphalt 3	5.09	5.15	4.72	5.18	**5.04**
REOB 2 Asphalt 3	6.12	7.60	6.49	5.99	**6.55**
REOB 3 Asphalt 3	3.10	4.61	4.57	4.44	**4.18**
Average	**4.23**	**5.78**	**5.07**	**5.07**	
Overall Average					5.04

Figure 4. Estimates of REOB Content From Each of the 36 Equations

Calculate the average by metal and by blend type as shown in the rightmost column and bottom row of Figure 4. The estimated REOB content is the average of the four averages by metal type and the 9 averages by blend (i.e., the numbers shown in bold in Figure 4). For the example shown in Figure 4, the average REOB content is 5.04.

INTERPRETATION OF RESULTS

The results need review to identify inconsistencies. For example, samples containing ground tire rubber have abnormally high levels of zinc. In these cases, the average for the zinc results will be much higher than the other levels of REOB, as demonstrated by Figure 5, which shows a REOB content estimate ranging from 0 percent based on the molybdenum content to over 50 percent based on zinc.

Blend	Calcium	Copper	Zinc	Molybdenum	Average
REOB 1 Asphalt 1	0.0	4.6	42.2	1.2	12.0
REOB 2 Asphalt 1	1.4	6.6	47.2	-1.2	13.5
REOB 3 Asphalt 1	1.6	8.3	61.0	-1.6	17.3
REOB 1 Asphalt 2	3.0	9.3	60.8	-1.5	17.9
REOB 2 Asphalt 2	2.5	7.7	48.1	-0.3	14.5
REOB 3 Asphalt 2	1.6	11.5	68.9	-1.9	20.0
REOB 1 Asphalt 3	1.2	7.4	47.7	-1.2	13.8
REOB 2 Asphalt 3	3.0	10.3	60.8	-1.7	18.1
REOB 3 Asphalt 3	0.8	4.5	40.3	1.2	11.7
Average	1.7	7.8	53.0	-0.8	

Figure 5. Results from a sample containing ground tire rubber