TECHBRIEF

Field Analysis of Asphalt Binders for Recycled Engine Oil Bottoms (REOB) using Handheld XRF Spectrometers

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This document is one of the outputs of 6 years of REOB research in the Chemistry Laboratory at the Turner-Fairbank Highway Research Center (TFHRC).

Objective

This TechBrief presents an analytical procedure by which asphalt binders can be analyzed for their content of Recycled Engine Oil Bottoms (REOB) in field locations using handheld battery-operated x-ray fluorescence (XRF) spectrometers.

Introduction

REOB is a residue produced while refining used engine oil. Companies collect used engine oil from garages, railroads, and similar sources. They recover the lubricating oil by vacuum distillation. The residue left at the bottom of the vacuum tower is then marketed under several different names, including recycled engine oil bottoms, re-recycled engine oil bottoms, and vacuum tower asphalt extender (VTAE). In this document, it is referred to simply as REOB.

These materials are used as additives in hot mix asphalt applications. Since they are waste products of varying composition, analyzing for them in asphalt binders presents a challenge. X-ray fluorescence spectroscopy and inductively coupled plasma (ICP) have been used successfully to determine the approximate amounts of these materials present in asphalt.

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.

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The metals in these additives—calcium, copper (a wear metal from the engine), zinc, and molybdenum—can be used as markers for analysis. The amount of each of these metals in a given asphalt sample will be proportional to the amount of REOB present. Figure 1 shows the calcium analysis plotted against REOB content for an asphalt blend. The REOB content is plotted on the x-axis, ranging from 2 to 20 percent. The calcium content is on the y-axis, ranging from 0 to 1,800 ppm.

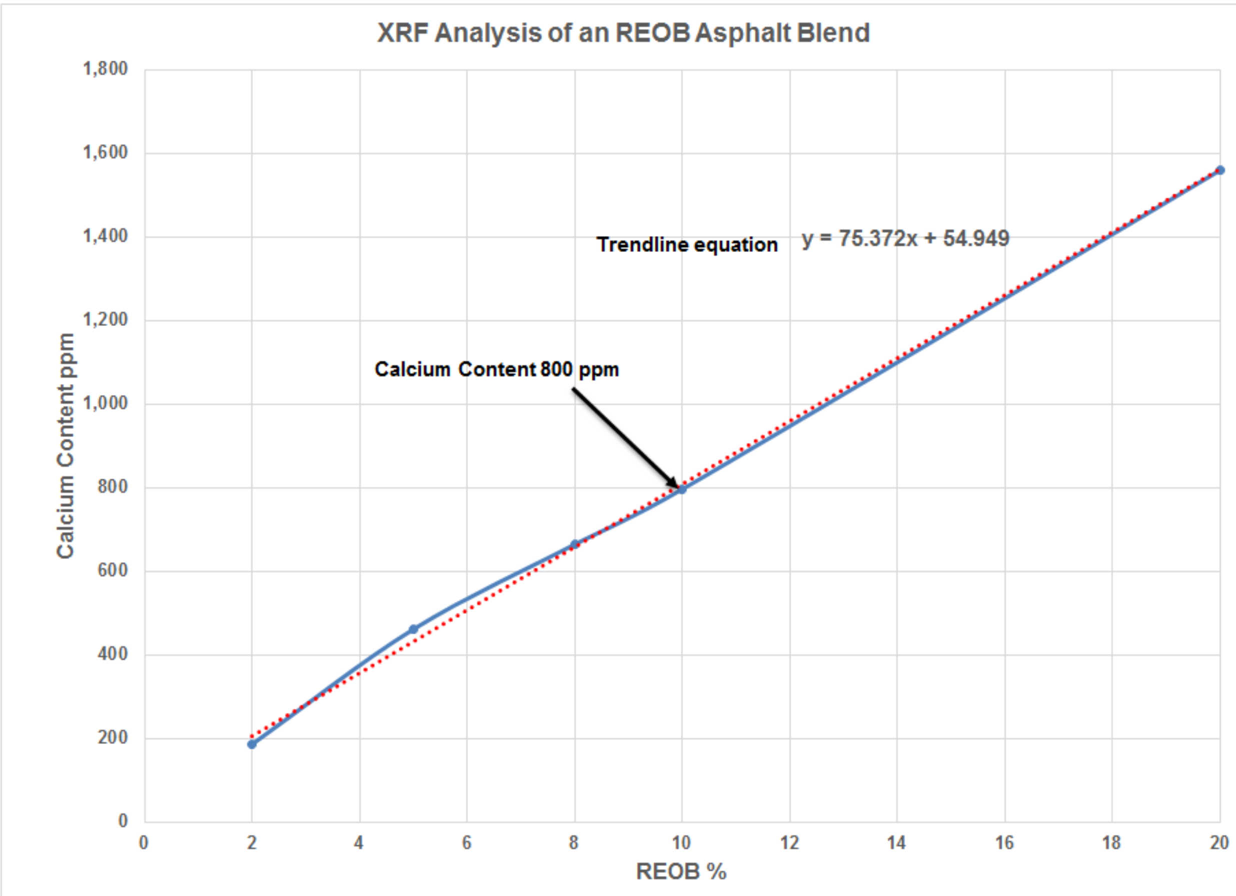

Figure 1. Calcium Analysis of Asphalt Blends Containing REOB.

This graph could be used to calculate the REOB content of an unknown sample. If for instance, we analyzed a sample and it contained 800 ppm of calcium. If we refer to this point on the chart (as indicated by the arrow), then a line drawn down from the point on the curve down to the x-axis, gives us an REOB value of about 10 percent. Alternatively we can use the trendline equation y = 75.372x + 54.949, where y is the calcium analysis in ppm and x is the REOB content. Simply solve for x:

$$y = 75.372x + 54.949$$

$$x = (y - 54.949) \div 75.372$$

or

$$\text{REOB\%} = (800 - 54.949) \div 75.32 = 9.89 \text{ percent}$$

Including the other markers (copper, zinc, and molybdenum) in the analysis confirms that REOB is present and not some other calcium-containing material. Analyses of the other markers would be treated in exactly the same way as the calcium example.

Under normal circumstances, this analysis would be straightforward. The problem here is that the composition of REOBs vary widely even from the same producer at different times. Table 1 shows a list of the elements and the ranges of their concentrations found in samples of REOB and GTR. The analysis may also be affected by the asphalt binder in which the REOB is dispersed. By analyzing a range of different REOBs dispersed in several asphalt binders, an average analysis can be obtained.

Ground Tire Rubber (GTR) is also used in hot mix asphalt. It contains high levels of zinc and calcium. Its presence in a blend can markedly affect the results of an REOB analysis. Some asphalt binders contain calcium (binders recovered from hot mix asphalt always do), some contain zinc compounds that are used as hydrogen sulfide scavengers in refineries, and some contain small amounts of molybdenum.

Table 1. Typical Composition of REOB and GTR

Elements	REOB	GTR
Phosphorus	1.5–1.9%	—
Sulfur	1.5–1.9%	2.6–4.5%
Calcium	7,200–10,900 ppm	530–4,400 ppm
Iron	370–1,800 ppm	220–11,600 ppm
Copper	700–1,500 ppm	40–3,400 ppm
Zinc	4,500–7,200 ppm	12,800–16,400 ppm
Molybdenum	300–700 ppm	—

X-ray Fluorescence Spectroscopy (XRF)

XRF is a well-established analytical technique whereby most of the elements in the periodic table (typically from sodium to uranium) can be analyzed in one shot. In addition to large research spectrometers, small portable handheld units are available; these are battery powered and require no special training to operate.

No sample preparation is needed except to ensure the sample is representative of the bulk. The nose of the handheld spectrometer is normally held against the sample. Since asphalt is sticky, the spectrometer head must be protected from the asphalt. This can be done by simply placing a thin plastic film—ideally those designed for assembling XRF cups—over the asphalt.

Figure 2. Handheld XRF Spectrometer

The test entails measuring the calcium, copper, zinc, and molybdenum content of asphalt samples modified with known amounts of REOB. As the REOB composition varies widely and its analysis is affected by the asphalt in which it is dispersed, a suite of samples using different REOBs dispersed in different asphalt binders should be prepared and analyzed.

The more blends analyzed, the more accurate the results will be. However, the actual REOB and asphalt binder makeup in any unknown sample being analyzed will never be definitively known.

Method for REOB Analysis

Prepare sets of calibration samples by a thorough mechanical blending of the REOB into molten asphalt. For example, blend REOB1 with Asphalt1, then Asphalt2, then Asphalt3. Blend REOB2 with Asphalt1, Asphalt2, Asphalt3, and so on. The addition levels of REOB should be 2, 5, 8, 10, and 20 percent, giving a total of 45 blends.

Analyze the blends for calcium, copper, zinc, and molybdenum and enter the results (in ppm) in a calibration results table (table 2). For each of the nine sets of blends, prepare charts as described in the earlier example (figure 1), plotting the metal analyses for calcium, copper, zinc, and molybdenum, in ppm, against the percentage of REOB in each blend.

Figure 3 contains the data for REOB1 Asphalt1. Table 2 contains real results (in red) should you wish to practice preparing your own charts. For each of the curves, draw the trendline and add the equation. The trendline does not need to pass through the origin. An example is shown in figure 3. Using these regression equations for each of the blends, build a spreadsheet that will calculate the REOB content in unknown samples (figure 4).

Table 2. Analysis of Asphalt Blends Results Table.

REOB %	REOB	Asphalt	Calcium	Copper	Zinc	Molybdenum
2	1	1	184	4	72	15
5	1	1	460	10	217	35
8	1	1	664	12	287	42
10	1	1	797	15	346	52
20	1	1	1561	28	689	93
2	2	1	173	3	60	18
5	2	1	377	5	148	29
8	2	1	588	10	248	47
10	2	1	733	14	314	58
20	2	1	1538	25	665	119
2	3	1	206	3	75	20
5	3	1	439	9	192	38
8	3	1	738	14	317	56
10	3	1	878	19	402	71
20	3	1	1621	31	734	122
2	1	2	51	0	49	9
5	1	2	219	4	133	22
8	1	2	430	8	232	43
10	1	2	539	10	289	54
20	1	2	1144	23	580	109
2	2	2	56	0	45	7
5	2	2	215	4	125	18
8	2	2	373	6	198	28
10	2	2	519	9	283	43
20	2	2	1276	28	698	110
2	3	2	38	0	41	8
5	3	2	186	0	108	19
8	3	2	333	5	179	33
10	3	2	430	6	228	43
20	3	2	1049	16	509	98
2	1	3	35	0	54	8
5	1	3	218	6	152	24
8	1	3	415	12	264	43
10	1	3	577	15	353	61
20	1	3	1302	31	754	124
2	2	3	0	0	33	3
5	2	3	165	4	105	21
8	2	3	322	6	176	37
10	2	3	457	9	243	53
20	2	3	1095	21	529	121
2	3	3	225	5	87	18
5	3	3	213	4	128	24
8	3	3	389	8	212	41
10	3	3	810	15	357	63
20	3	3	1098	23	547	103

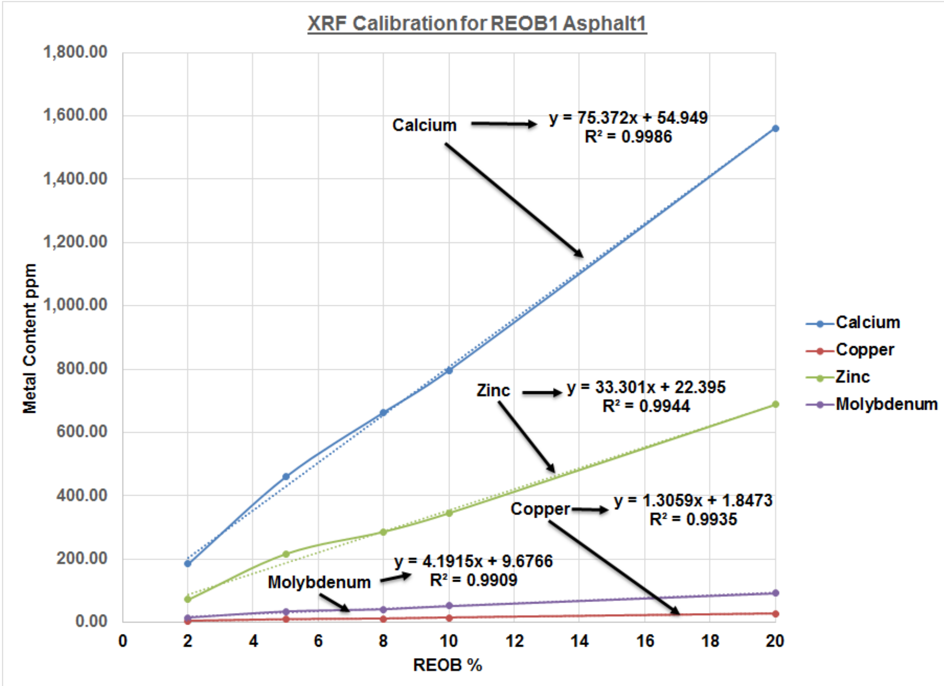

Figure 3. Typical Calibration Curves for REOB Asphalt Blends.

Figure 3 is a chart plotting the REOB content of REOB asphalt blends against the contents of calcium, copper, zinc, and molybdenum. The trendlines and trendline equations are shown.

The calculator is shown in figure 4. It is used to determine the REOB content in an unknown sample. The results (in ppm) for the calcium, copper, zinc and molybdenum analysis from the unknown sample are entered into the yellow cells. The calculator will determine the REOB content in percent.

Handheld XRF Spectrometer Calibration					
Enter metal contents in ppm into the yellow cells					
	Calcium	Copper	Zinc	Molybdenum	
Blend					
REOB1 Asphalt1	-0.73	-1.41	-0.67	-2.31	-1.28
REOB2 Asphalt1	0.07	0.14	0.53	-0.40	0.08
REOB3 Asphalt1	-0.76	-0.60	-0.27	-1.65	-0.82
REOB1 Asphalt2	1.18	1.92	0.34	0.58	1.01
REOB2 Asphalt 2	1.91	3.11	1.71	2.00	2.18
REOB3 Asphalt 2	1.79	3.26	0.88	1.17	1.78
REOB1 Asphalt 3	1.84	1.65	0.99	1.05	1.38
REOB2 Asphalt 3	2.35	2.34	1.21	1.90	1.95
REOB3 Asphalt 3	-1.21	-0.88	-0.91	-1.03	-1.01
	0.72	1.06	0.42	0.15	
Overall Average:	0.59				

Figure 4. REOB Calculator.

The cell outlined in blue at the junction of REOB1 Asphalt1 and calcium is cell C4 in this spreadsheet. It contains the formula =(C4 − 54.949) ÷ 75.372, the calibration from the calcium line in figure 3. The other purple cells contain the calibration equations for the other metals and blends.

The Handheld Calibration Data Excel file containing the calculator used in this TechBrief will be available on the Chemistry Laboratory website at https://www.fhwa.dot.gov/publications/research/infrastructure/pavements/18043/reobtool.xlsx. The Excel file was built from the data in the calibration results table (table 2). All the cells in the sheet are protected except those highlighted in yellow. The calibration results in table 2 could be used to practice building another calculator.

Once constructed, the calculator can be used to determine the REOB content of unknown samples. It is a complex thing to construct, but it only has to be constructed once. It is a good idea to protect all but the yellow cells to avoid overtyping the regression formulae.

To use the calculator, analyze the unknown sample using the same setup and spectrometer conditions as for the REOB/asphalt calibration. This is important. If different spectrometer or running conditions are used, then the results will not be correct. Enter the metal analyses into the yellow cells; the sheet will calculate the REOB content. Figure 5 shows a completed calculator sheet for a real sample.

Sample XRF2-134-1					
Enter metal contents in ppm into the yellow cells					
	Calcium	Copper	Zinc	Molybdenum	
Blend	231.0	6.0	147.0	27.0	
REOB1 Asphalt1	2.34	3.18	3.74	4.13	3.35
REOB2 Asphalt1	3.09	4.93	4.87	4.30	4.30
REOB3 Asphalt1	2.20	3.35	3.78	3.15	3.12
REOB1 Asphalt2	4.97	6.64	5.30	5.40	5.58
REOB2 Asphalt 2	5.27	6.91	5.68	6.60	6.12
REOB3 Asphalt 2	5.88	9.64	6.49	6.47	7.12
REOB1 Asphalt 3	5.09	5.15	4.72	5.18	5.04
REOB2 Asphalt 3	6.12	7.60	6.49	5.99	6.55
REOB3 Asphalt 3	3.10	4.61	4.57	4.44	4.18
	4.23	5.78	5.07	5.07	
Overall Average:	5.04				

Figure 5. Example of an REOB Analysis Calculation.

Deciphering the calculator

- Purple cells give the REOB percentage for each metal in each blend. For example, copper for REOB2 and Asphalt2 shows 6.91 percent REOB.
- The same REOB will have different percentages in different asphalts. For example, zinc for REOB1 in Asphalt1 is 3.74 percent REOB. The same REOB is 5.3 percent in Asphalt2 and 4.72 percent in Asphalt3.
- The green cells are the average of the rows. The average REOB content for REOB3 Asphalt2 calculated from calcium, copper, zinc, and molybdenum analyses is 7.12 percent.
- The red cells are the average of the columns. As shown, the average for calcium for all blends is 4.23 percent.
- The overall average of 5.04 percent is shown in blue at the bottom of the calculator. The actual REOB content was 5.0 percent. Bear in mind that the analyses are not always that close to the actual value.

Building the calculator in this way gives a better idea of the amount of REOB present and shows any anomalies in the results. At TFHRC we have run over 3,000 analyses of asphalt samples from throughout the United States. We have found that these averages, while not exactly correct, are normally in the same ballpark.

For example, samples containing GTR have abnormally high levels of zinc. In these cases, the red average for the zinc column would show an extraordinarily high level of REOB and the overall average is often a ridiculous answer. Similarly, some virgin asphalt binders contain small amounts of molybdenum. These show up as anomalies in the calculator, which they would not do if we just looked at the overall average REOB content (the number in blue).

Some real examples from the round robin test are in table 3; you can try using these in the calculator spreadsheet.

Table 3. Analyses of REOB Asphalt Blends.

Actual REOB (%)	Calcium (ppm)	Copper (ppm)	Zinc (ppm)	Molybdenum (ppm)	Found REOB (%)
3	79	3	58	15	2.62
5	183	3	109	26	3.92
8	440	7	245	48	7.76
5	203	2	111	17	3.40
3	80	2	65	11	2.30
8	377	4	193	34	5.88
10	686	13	363	76	12.09
10	456	10	250	49	8.50

Figure 6 is a calculator example from a table that contained 5 percent REOB. The calculator's overall average is 3.4 percent. The averages of the rows in green and the columns in red, while not the totally accurate, are in the right ballpark. This gives some increased confidence that the sample does contain REOB.

Handheld XRF Spectrometer Calibration					
Enter metal contents in ppm into the yellow cells					
	Calcium	Copper	Zinc	Molybdenum	
Blend	203.0	2.0	111.0	17.0	
REOB1 Asphalt1	1.96	0.12	2.66	1.75	1.62
REOB2 Asphalt1	2.73	1.74	3.81	2.56	2.71
REOB3 Asphalt1	1.84	0.72	2.79	1.37	1.68
REOB1 Asphalt2	4.51	3.49	4.09	3.61	3.93
REOB2 Asphalt 2	4.86	4.38	4.71	4.90	4.71
REOB3 Asphalt 2	5.39	5.39	5.11	4.51	5.10
REOB1 Asphalt 3	4.69	2.82	3.81	3.65	3.74
REOB2 Asphalt 3	5.66	4.09	5.20	4.47	4.86
REOB3 Asphalt 3	2.58	0.95	3.23	2.41	2.29
	3.80	2.63	3.93	3.25	
Overall Average:	3.40				

Figure 6. Calculator for 5-percent REOB Asphalt Blend.

Figure 7 is a calculator example from a real-life sample of asphalt that contains approximately 34 percent REOB. Even at that high level, the averages of the rows and columns are still in the correct range.

Enter metal contents in ppm in the yellow cells					
	Calcium	Zinc	Copper	Molybdenum	Average
	2601	1728	257	158	
REOB 08-1001 Binder AAP	23.2	28.7	24.1	32.1	**27.0**
REOB 12-0002 Binder B6430	36.5	32.2	35.5	24.9	**32.3**
REOB 12-0057 Binder B6430	44.8	41.5	45.7	31.4	**40.9**
REOB 12-0057 Binder B6430	41.8	41.4	41.7	31.4	**39.1**
REOB 12-0002 Binder B6531	34.8	32.8	34.1	25.6	**31.8**
REOB 12-0057 Binder B6430	47.3	46.8	53.8	37.7	**46.4**
REOB 12-0002 Binder B6430	35.5	32.4	34.9	24.8	**31.9**
REOB 12-0002 Binder B6325	45.0	41.3	48.0	33.0	**41.8**
REOB 11-0019 Binder B6462	24.9	27.4	22.4	44.1	**29.7**
REOB 11-0020 Binder B6462	25.4	28.3	35.0	21.8	**27.6**
REOB 12-0002 Binder B6462	23.2	23.7	26.2	24.1	**24.3**
REOB 12-0057 Binder B6462	31.3	32.5	36.9	35.7	**34.1**
Average	**34.5**	**34.1**	**36.5**	**30.6**	
Overall Average	**33.9**				

Figure 7. Calculator for 34-percent REOB Asphalt Blend.

Figure 8 is a calculator example of a real-life sample that contains no REOB, even though the calculator gives an average REOB content of 14.7 percent. The sample actually contains GTR. The high level of zinc in the GTR distorts the analysis. The average REOB content at the bottom of the zinc column shows 50.1 percent REOB. The other columns show almost no REOB present. The negative result for the molybdenum content is an artifact of the calibration curve, which does not pass through the origin. It is effectively zero.

Enter metal contents in ppm in the yellow cells					
	Calcium	Zinc	Copper	Molybdenum	Average
	35.3	2541	42	0	
REOB 08-1001 Binder AAP	0.0	42.2	4.6	1.2	12.0
REOB 12-0002 Binder B6430	1.4	47.2	6.6	-1.2	13.5
REOB 12-0057 Binder B6430	1.6	61.0	8.3	-1.6	17.3
REOB 12-0057 Binder B6430	3.0	60.8	9.3	-1.5	17.9
REOB 12-0002 Binder B6531	2.5	48.1	7.7	-0.3	14.5
REOB 12-0057 Binder B6430	1.6	68.9	11.5	-1.9	20.0
REOB 12-0002 Binder B6430	1.2	47.7	7.4	-1.2	13.8
REOB 12-0002 Binder B6325	3.0	60.8	10.3	-1.7	18.1
REOB 11-0019 Binder B6462	0.8	40.3	4.5	1.2	11.7
REOB 11-0020 Binder B6462	1.1	41.5	6.5	0.9	12.5
REOB 12-0002 Binder B6462	0.8	34.9	5.3	0.7	10.4
REOB 12-0057 Binder B6462	1.0	47.8	7.4	0.9	14.3
Average	1.5	50.1	7.4	-0.4	
Overall Average	14.7				

Figure 8. Calculator for Asphalt Blend Containing GTR.

Figure 9 is a calculator example of a real-life sample that contains both GTR and REOB. While some estimation may be made on how much of each material is present, given the variability in composition of these materials, it is very difficult to tell exactly.

Enter metal contents in ppm in the yellow cells					
	Calcium	Zinc	Copper	Molybdenum	Average
	1737	2452	141	64	
REOB 08-1001 Binder AAP	15.4	40.8	13.6	13.7	20.9
REOB 12-0002 Binder B6430	24.7	45.6	19.9	9.4	24.9
REOB 12-0057 Binder B6430	30.3	58.8	25.5	11.8	31.6
REOB 12-0057 Binder B6430	28.7	58.7	24.2	11.8	30.8
REOB 12-0002 Binder B6531	23.9	46.4	19.8	10.2	25.1
REOB 12-0057 Binder B6430	31.9	66.5	30.9	14.1	35.9
REOB 12-0002 Binder B6430	23.9	46.1	20.1	9.3	24.8
REOB 12-0002 Binder B6325	30.8	58.7	27.6	12.3	32.4
REOB 11-0019 Binder B6462	16.8	38.9	12.7	18.6	21.7
REOB 11-0020 Binder B6462	17.2	40.0	19.6	9.4	21.6
REOB 12-0002 Binder B6462	15.6	33.7	14.9	10.2	18.6
REOB 12-0057 Binder B6462	21.1	46.1	21.0	15.0	25.8
Average	23.4	48.3	20.8	12.2	
Overall Average	26.2				

Figure 9. Calculator for Asphalt Blend containing REOB and GTR.

Does the Method Work?

To determine if this method is practical and if it works, we have been running a round robin test. The participants in the test are the departments of transportation of Massachusetts, Montana, North Carolina, Oklahoma, South Carolina, Vermont, and Wyoming; plus the Ontario Ministry of Transportation, Coco Asphalt Ontario (paving contractor) and two testing labs: PRI in Florida and MTE Services, Inc. in Wisconsin.

TFHRC provided the test method and calibration samples for this research. The more blends that are used as reference standards, the more accurate the analysis is likely to be. We still do not know the exact composition of an unknown sample about to be analyzed. Probably using more reference standards would give diminishing returns.

Table 4. Analysis Results from REOB Round Robin Unknown Samples.

Participant		1	2	3	4	5	6	7	8	9
Method		ICP	EDXRF	EDXRF	XRF	*HHXRF	XRF	WDXRF	EDXRF	*HHXRF
Reference	REOB %	Found REOB %								
17-079	3	2.8	2.81	2.86	1.03	1.27	2.36	2.82	2.5	2.33
17-080	5	4.36	4.3	3.69	1.96	2.06	3.53	4.67	3.7	3.77
17-083	8	7.68	6.84	5.57	5.11	4.98	6.13	7.23	6.1	6.04
17-084	3	3.70	3.64	3.19	0.95	1.99	3.04	4.01	3.0	3.47
17-085	5	5.87	5.43	4.18	2.80	3.42	5.60	6.08	4.8	5.06
17-086	8	9.19	8.57	8.18	6.16	6.76	8.68	9.09	7.9	8.05
17-088	3	2.92	3.39	2.97	0.99	1.62	2.68	3.62	2.8	2.25
17-089	5	4.97	5.44	4.29	2.35	4.59	6.20	5.68	4.4	4.8
17-090	8	8.15	8.41	6.56	6.40	8.25	6.99	8.09	7.7	8.23
17-091	3	2.92	3.13	2.97	1.06	1.74	2.50	3.12	2.8	2.64
17-092	5	3.98	4.26	3.62	1.73	2.25	3.38	4.50	3.6	3.2
17-093	3	2.33	2.49	2.89	0.62	1.11	2.50	2.63	2.35	2.03
17-094	8	7.31	6.61	5.68	4.38	5.45	5.98	7.09	6.0	6.03
17-100	6	7.83	7.93	6.97	6.89	7.47	7.55	8.66	7.5	8.08
17-101	10	10.91	11.89	11.91	11.74	10.59	11.46	12.10	11.0	12.13
17-102	6	5.67	5.73	4.93	4.13	4.95	5.34	6.18	5.05	5.61
17-103	10	8.70	8.68	7.62	7.68	8.49	9.14	8.79	8.3	8.75
17-104	6	6.89	7.11	6.07	5.52	5.61	7.61	7.14	6.65	6.94
17-105	10	10.79	11.57	11.32	10.99	10.65	12.32	11.95	11.0	11.95
17-106	6	5.24	5.23	4.64	3.40	5.03	4.90	5.57	4.7	4.83
17-107	10	8.75	8.56	7.63	6.33	7.55	7.93	9.07	7.9	8.55

*Handheld XRF. The other instruments being used are lab spectrometers.

The participants are using a variety of techniques, including energy dispersive XRF (EDXRF), wavelength dispersive XRF (WDXRF), and inductively coupled plasma (ICP). This round robin test is almost complete. The last stage was to send unknown samples to the participants to see if they could accurately determine the REOB content. This is not yet complete. Table 3 contains the results so far.

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Researchers—This study was performed by Dr. Anant Shastry, SES Group and Associates, and Terry Arnold of FHWA.

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