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Support XRF Determination of Tire Rubber Content in Asphalt Binders

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16. Abstract

This work involved providing support to the Lufkin, Odessa, and Brownwood Districts during their 2024 seal coat season for determining the tire rubber content using portable XRF units. CTR staff travelled to the three districts to conduct training refreshers and assist in analyzing field samples used in district seal coats. Analysis was performed on the roadside, demonstrating the ability to conduct analysis in the field. Additionally, staff conducted a round-robin analysis of samples from across the three districts using AR binder, which also served to develop a calibration for a specific combination of base binder and tire rubber.

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ABSTRACT

This work involved providing support to the Lufkin, Odessa, and Brownwood Districts during their 2024 seal coat season for determining the tire rubber content using portable XRF units. CTR staff travelled to the three districts to conduct training refreshers and assist in analyzing field samples used in district seal coats. Analysis was performed on the roadside, demonstrating the ability to conduct analysis in the field. Staff conducted an analysis of AR Binder samples with the three districts using blended AR binder calibration samples for a specific combination of base binder and tire rubber. Additionally, staff conducted a round-robin analysis of five unknown samples with the three districts to compare accuracy and repeatability.

EXECUTIVE SUMMARY

Tire rubber is required in certain TxDOT asphalt binders; e.g., AC-205TR, used for chip seal construction. These binders are specified and used in several districts across the state. Current Receiving Agency Standard Specification Item 300, Asphalts, Oils, and Emulsions, includes using test procedure Tex 553-C, "Determination of Re-Refined Engine Oil Bottoms, Polyphosphoric Acid, and Tire Rubber Content in Asphalt using X-Ray Fluorescence Spectroscopy."

Previously, this test procedure was only performed at the Receiving Agency Materials and Tests Division (MTD). This test can be implemented in the field with a portable XRF device and test procedure Tex-553-C can be used in conjunction with a calibration chart to evaluate and obtain a quantitative estimate of tire rubber at the district level.

The initial project (completed Dec. 2023) involved making this device available to district personnel (Lufkin, Odessa, and Brownwood) and training them to use the device on a routine basis to analyze tire rubber-modified asphalt binder for tire rubber content. In the initial project, the Performing Agency worked with the Receiving Agency and producers from around the state to develop calibration standards that can be used to determine the tire rubber content using the portable XRF units and conducted a round-robin analysis of samples from across different laboratories. The Performing Agency also revised test procedure Tex-553-C to account for procedures to use in the field.

This project extension allowed the Performing Agency to assist the Lufkin, Odessa, and Brownwood Districts in testing materials used in their 2024 seal coat program. This involved travelling to each district, reviewing the XRF training, and assisting each district during a seal coat project to test binders use on that project, in the field. This field analysis used a sampling and analysis process to sample and test for tire rubber content on the roadway project site.

Additionally, samples were formulated using binder and granulated tire rubber to distribute a set of calibration samples to each of the districts for analysis. This work showed that the XRF can be used to evaluate the tire rubber content of AR binder.

Finally, the Performing Agency conducted a round-robin to evaluate accuracy and repeatability using tire rubber modified binders with "unknown" (unknown to the tester) tire rubber content.

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Chapter 1. Review Training and Provide Technical Support for In-the-Field Sampling and Testing

This Chapter describes the work performed for Tasks 2 and 4, “Review Training” and “Provide technical support to the three (3) districts”. Individual visits were made to the Brownwood, Odessa, and Lufkin Districts to this field support. In those trips, STR staff reviewed the training and operation procedures of the field testing and assisted the district in conducting field testing on the roadside. Below is a summary of these visits.

1.1. Brownwood District Visit

On Thursday, May 9, 2024, CTR Staff travelled to the TxDOT Brownwood District to work with the district personnel for field sampling and testing of AC-20-5TR used in the district seal coat contracts. This project was on US 337 in McCulloch County.

Figure 1 shows the project roadside sampling in progress. The sample is taken directly from the distributor sampling port. The sample could have been taken from a nozzle instead.

Figure 1. Roadside Sampling.

Figure 2 shows the completed roadside sample for testing. This can is labelled for future reference and the remaining sample (only a small amount is needed for XRF testing) can be stored for additional testing if needed or desired.

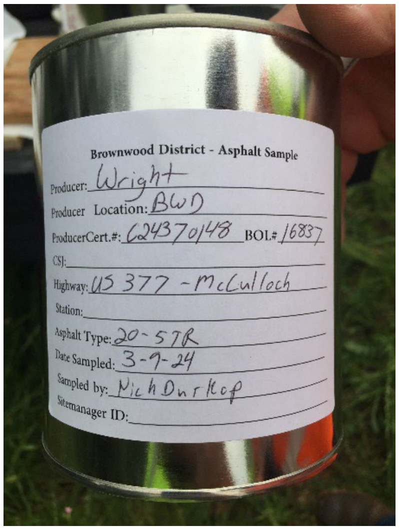

Figure 2. Roadside Sample.

Figures 3 shows the field testing set up in the bed of a pickup truck.

Figure 3. Portable XRF Field Testing Setup.

Figure 4 shows pouring of the field test binder into the sample containers for XRF analysis.

Figure 4. Field Test Sample Preparation.

Figures 5 shows the Zinc ppm for the field sample using both the Brownwood XRF gun and the CTR XRF gun. As shown, they numbers agree well.

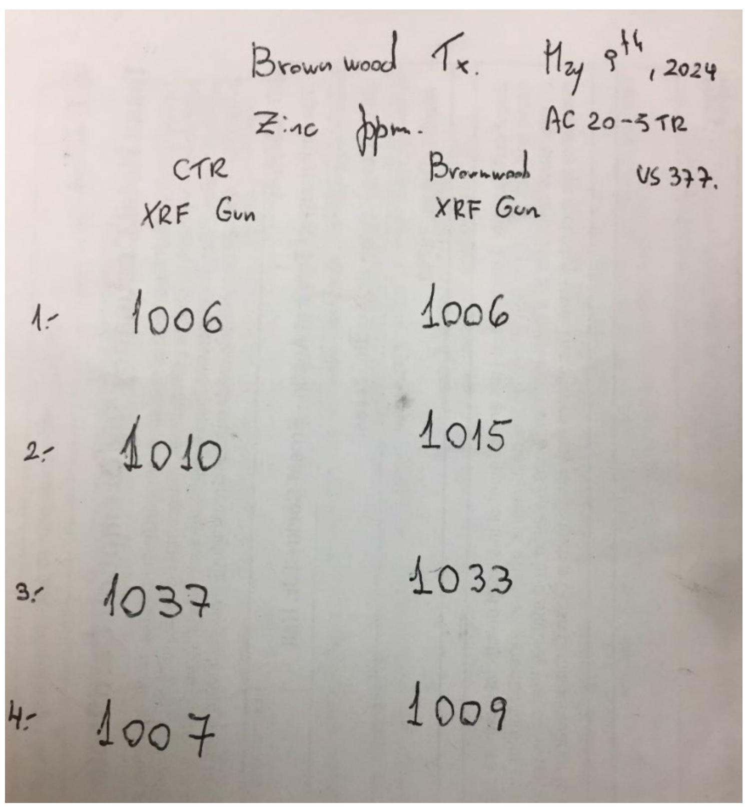

Figure 5. Portable XRF Zinc ppm CTR and Brownwood XRF guns.

Using the calibration curve developed in the initial project, these readings produce the TR percent shown in Table 1.

Table 1. CTR-Brownwood Field Sample Analysis.

Sample	CTR (Zn ppm)	CTR % TR	Brownwood (Zn ppm)	Brownwood % TR
1	1006	4.8	1006	4.8
2	1010	4.8	1015	4.8
3	1037	5	1033	4.9
4	1007	4.8	1009	4.8

The initial report showed accuracy of +/- 0.2 percent, however with a specification requiring 5% minimum TR content, either the binder is on the lower side of meeting the specifications or the calibration curve for this material needs updating to use current base binder and TR.

1.2. Odessa District Visit

On Wednesday, May 8, 2024, CTR Staff travelled to the TxDOT Odessa District to work with the district personnel for field sampling and testing of Type II AR Binder used in the district seal coat contracts. This project was on US 349.

Figure 6 shows sampling of the material. Samples came from the truck.

Figure 6. AR Binder Sampling.

Figure 7 shows the completed roadside sample for testing.

Figure 7. Sample for testing.

Figures 8 shows the field testing set up in the back of a pickup truck. Note there are some heavy items used to weigh down sampling and release paper due to wind.

Figure 8. Portable XRF Field Testing Setup.

Figure 9 shows samples poured for testing on truck tailgate.

Figure 9. Samples Poured for Testing.

Figure 10 shows field testing in progress. Test samples and the XRF gun.

Figure 10. Field Testing.

Figure 11 shows the XRF output screen from a test.

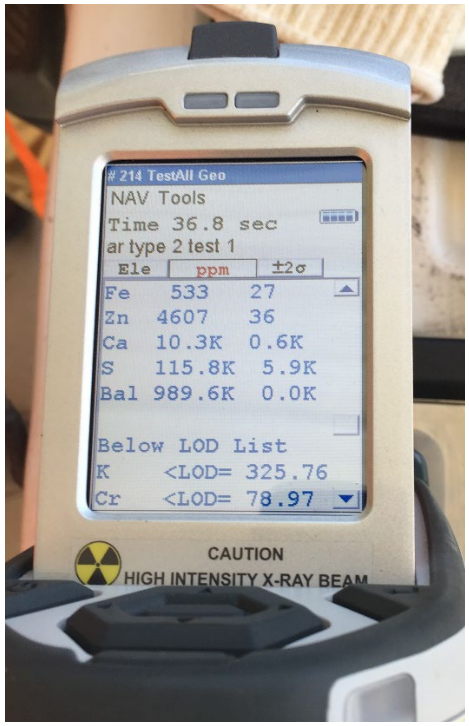

Figure 11. XRF Output Screen.

Figure 12 shows the compilation Zn reading data from the Odessa and CTR XRF guns.

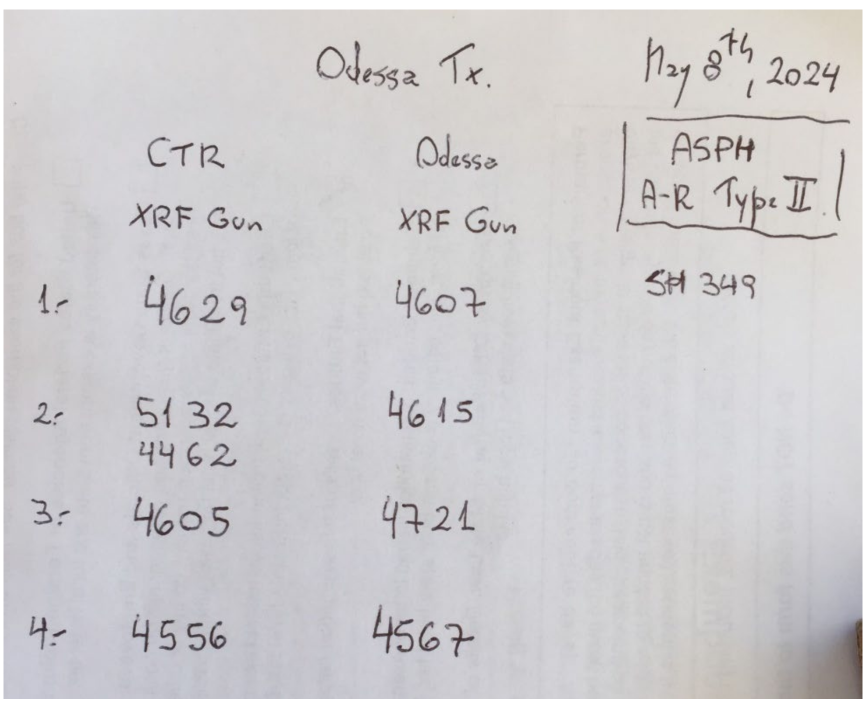

Figure 12. CTR and Odessa Data Compilation.

The use of AR binder on this project did not allow calculation of tire rubber percent, as there was no calibration curve for this set of materials, and no previous work had been done with AR binder for this purpose. This did prove fortunate, as STR staff was able to acquire samples of binder and rubber to blend in the lab to both determine a calibration curve and conduct a round-robin with the other districts.

When calibration curves were developed (shown later in this report), the results are shown in Table 2.

Table 2. Asphalt Rubber Percent.

Sample	CTR (Zn ppm)	CTR % TR	Odessa (Zn ppm)	Odessa % TR
1	4629	21.5	4607	20.6
2	4797	22.3	4615	20.7
3	4605	21.4	4721	21.1
4	4556	21.2	4567	20.5

Type II Asphalt Rubber, in Item 300 indicates that a minimum of 15% tire rubber must be used. Historically, 20% is usually seen in order to meet the physical properties of the AR Binder.

1.3. Lufkin District Visit

On Tuesday, August 20, 2024, CTR staff travelled to the TxDOT Lufkin District to work with the district personnel for field sampling and testing of AC-20-5TR used in the district seal coat contracts. This project was on FM 222 in San Jacinto County.

Figure 13 shows the completed roadside sample for testing. This can is being prepared for pouring the XRF sample for testing.

Figure 13. Roadside Sample.

Figures 14 shows the field testing set up in the bed of a pickup truck.

Figure 14. Portable XRF Field Testing Setup.

Figure 15 shows the field test of binder with the XRF device in progress.

Figure 15. Field Test in Progress.

Figures 16 shows the Zinc ppm for the field sample using both the Lufkin XRF gun and the CTR XRF gun. As shown, they numbers agree well.

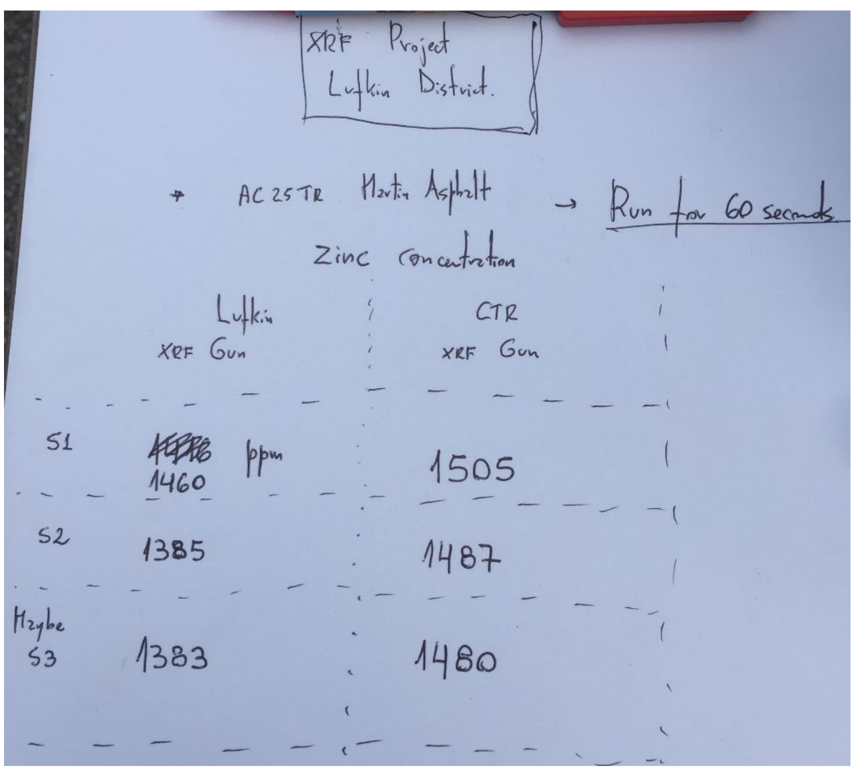

Figure 16. Portable XRF Zinc ppm CTR and Lufkin XRF guns.

Using the calibration curve developed in the initial project, these readings produce the TR percent shown in Table 3.

Table 3. CTR-Lufkin Field Sample Analysis.

Sample	CTR (Zn ppm)	CTR % TR	Lufkin (Zn ppm)	Lufkin % TR
1	1505	3.9	1460	4.0
2	1487	3.9	1385	3.8
3	1480	3.9	1383	3.8

The calibration curves used for the TR content were developed the previous year. It is known that calibrations are specific to the XRF gun, the base asphalt, and the polymer. The Lufkin XRF gun had a malfunction last year and had to be sent for service, this could also add to errors in measurement. However, since the CTR and Lufkin analyses agree quite well, this data may indicate that the XRF calibration curves should be updated using binder and TR for this year's production.

Chapter 2. Investigation of AR Binder

Not every district uses AR binder, but those that do (mainly the Odessa District) should have a way of determining the tire rubber contained in the AR binder.

Type II Asphalt Rubber, in Item 300 indicates that a minimum of 15% tire rubber must be used. Historically, 20% is usually seen in order to meet the physical properties of the AR Binder.

The work in the Odessa District allowed the Performing Agency to develop a calibration curve for AR binder for each XRF gun. The Performing Agency used AR material and base binder to develop calibration standards for AR Binder. These were distributed to each of the three districts and XRF analysis for Zn ppm was performed by each district for each sample. Each tested sample was measured twice. The data is shown in Table 4. This material was composed of one base binder and a corresponding tire rubber used by that manufacturer.

Table 4. Asphalt Rubber Calibration Data.

Tester	CTR	Brownwood	Lufkin	Odessa
XRF Gun	SN 97393	SN 133080	SN 200125	SN 200129
TR Content	Zn (ppm)	Zn (ppm)	Zn (ppm)	Zn (ppm)
0	184	196	180	188
0	186	184	177	193
2.5	579	575	670	329
2.5	506	691	662	315
5	951	1107	1171	888
5	981	1160	1123	692
7.5	1336	1744	1660	1474
7.5	1186	1686	1661	1154
10	2019	2355	2164	2231
10	1986	2327	2106	2008
15	3112	3351	3363	3295
15	3106	3348	3302	3124
25	5658	5570	5339	5762
25	5196	5584	5491	5573
100	30700	n/a	32700	33500
100	29900	n/a	31100	32700

Plotting this data produces Figure 17. One can see that a linear trend lines fit the data on the upper end pretty well, but not so well in the lower end of TR concentration. This is believed to be because the base binder shows a higher Zn level affecting the Zn XRF measurements proportionately more in the lower concentrations. However, as AR binder must have a minimum of 15% TR and usually contains about 20%, a linear trend line in the upper region fits well. This is the trend line fit used to evaluate the Odessa Type II AR Binder in Section 1.2 above.

A calibration for this specific combination of base binder and tire rubber will work to determine TR content in AR Binder.

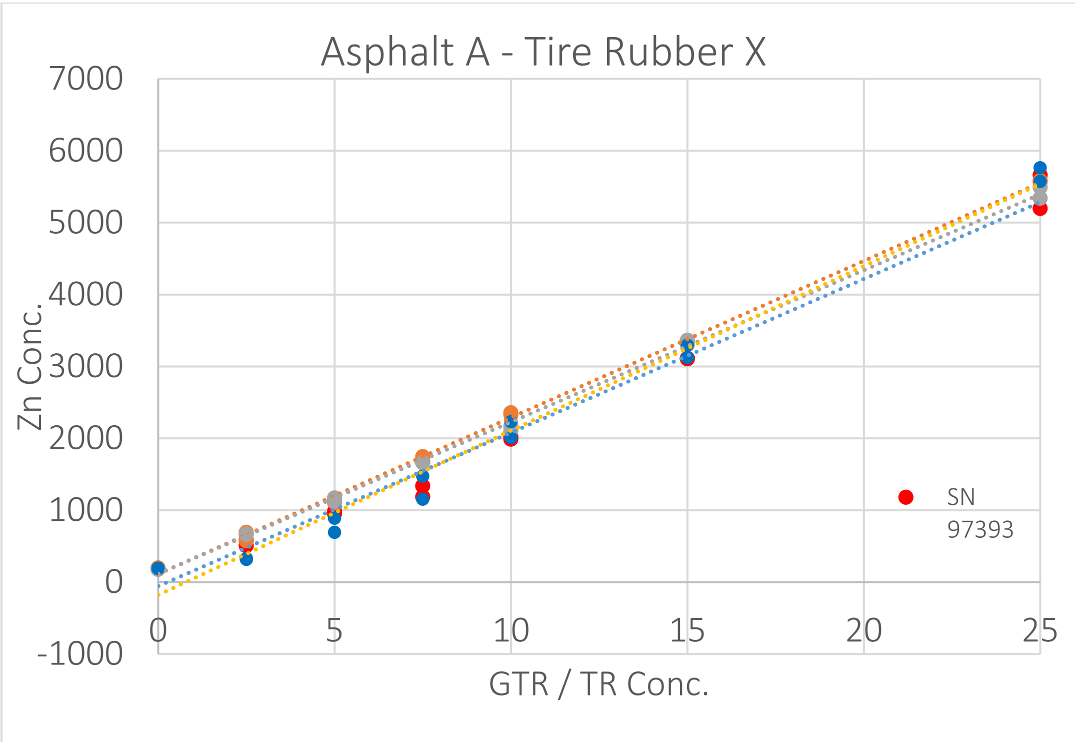

Figure 17. XRF of Type II AR Binder Calibration Samples.

Chapter 3. Conduct Round Robin Program

This Chapter describes the work performed for Task 3, "Conduct Round Robin Program."

Blind samples were created by CTR with the goal of producing samples of the approximate TR content shown in Table 5.

Table 5. Approximate TR content of blind samples.

Sample	Supplier	Approximate TR content (%)
1	Valero Houston	5
2	Jebro Waco	2.5
3	Ergon Lubbock	5
4	Ergon Lubbock	7.5
5	Wright	5

Included are two versions of the round-robin data. Table 6 shows all the data and Table 7 shows the same data with some eliminated due to suspected sample or analysis problems. This is based the significant differences seen in TR content for Ergon Lubbock as analyzed by Lufkin and Odessa.

The complete round-robin data is shown in Table 5.

Table 6. All Blind Round-Robin Analyses

Blind Sample	Percentage of Tire Rubber [%]					Average	StDev
	CTR	Brownwood	Lufkin	Odessa	MTD		
	Lab 1	Lab 2	Lab 3	Lab 4	Lab 5		
1-A	5.4	5.6	5.3	5.5	5.4	5.44	0.10
1-B	5.4	5.5	5.3	5.5	5.5		
2-A	2.6	2.7	2.6	2.7	2.7	2.67	0.05
2-B	2.7	2.7	2.7	2.7	2.6		
3-A	5.8	5.2	6.5	6.6	6.0	5.96	0.55
3-B	5.4	5.5	6.5	6.6	5.5		
4-A	8.1	7.9	9.2	9.5	7.9	8.48	0.76
4-B	8.5	7.7	9.2	9.3	7.5		
5-A	4.8	4.9	4.9	5.1	5.0	4.93	0.11
5-B	4.8	4.9	4.9	5.1	4.9		

Table 7. Modified Blind Round-Robin Analyses

Blind Sample	Percentage of Tire Rubber [%]					Average	StDev
	CTR	Brownwood	Lufkin	Odessa	MTD		
	Lab 1	Lab 2	Lab 3	Lab 4	Lab 5		
1-A	5.4	5.6	5.3	5.5	5.4	5.44	0.10
1-B	5.4	5.5	5.3	5.5	5.5		
2-A	2.6	2.7	2.6	2.7	2.7	2.67	0.05
2-B	2.7	2.7	2.7	2.7	2.6		
3-A	5.8	5.2			6.0	5.57	0.29
3-B	5.4	5.5			5.5		
4-A	8.1	7.9			7.9	7.93	0.34
4-B	8.5	7.7			7.5		
5-A	4.8	4.9	4.9	5.1	5.0	4.93	0.11
5-B	4.8	4.9	4.9	5.1	4.9		

Because of the data analysis problems, only the data in Table 7 will be used for statistical evaluation.

Since each asphalt sample was run twice, each measurement will be treated as a sample we have either 10 samples or 8 samples Using a two-tailed, 95% t-distribution:

For degrees of freedom (df) = number of observations (10) minus 1 = 9. From tabulated t-statistics, t = 2.26. Then the calculated 95% confidence intervals is:

$\overline{X} + \pm 2.26\ (\sigma/\sqrt{n})$

For degrees of freedom (df) = number of observations (8) minus 1 = 7. From tabulated t-statistics, t = 2.36. Then the calculated 95% confidence intervals is:

$\overline{X} + \pm 2.36\ (\sigma/\sqrt{n})$, and

Then applying these to the data, the variation can be estimated as:

Blind 1 = 5.44 +/- 0.07.

Blind 2 = 2.67 +/- 0.04

Blind 3 = 5.57 +/- 0.24

Blind 4 = 7.93 +/- 0.28

Blind 5 = 4.93 +/- 0.08

Some materials produce more repeatable results than others, and is likely dependent on the base asphalt and type of tire rubber used. Any changes would require developing a new calibration curve.

The analysis does show to be accurate in terms of test repeatability.

This analysis, showing higher average lab TR content of blind samples than the estimate of manufactured samples distributed, points to the likelihood that calibration curves require updating at least annually for each supplier.

Chapter 4. Summary/Conclusions

This chapter provides a summary of the work performed and conclusions.

Summary of work performed on this project included:

- CTR provided training and support for the Brownwood, Odessa, and Lufkin districts to conduct tire rubber concentration analysis at the project site.
- Calibration standards were developed and analyzed to determine the viability of using XRF for analysis of Asphalt Rubber Binder.
- A round robin on blind samples from one producer showed that the accuracy of the output to be approximately +/- 0.1 % TR for 3 of the 4 producers and +/- 0.3% TR for one producer.

Conclusions

- Measurement of TR content can be accomplished with a portable XRF analyzer in the field on a project site.
- XRF can be used to determine the tire rubber content in AR Binders, and can be measures in the field on the project site.
- The procedure produces an accurate measure of TR concentration.
- Differences in the formulated blind samples showing higher analyzed TR content than the estimated formulated TR content, show that annual generation of new calibration curves is likely needed. New calibration curves should be generated for any changes to the XRF gun, base binder, and TR (or other polymer) additive. Base asphalt changes are likely over time due to normal production variation.