# FIELD TRIAL OF SOLVENT-FREE EMULSION IN OREGON

#### **Final Report**

#### **STATE PLANNING AND RESEARCH 391**

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This final report summarizes construction, field trial of solvent-free emulsion mix con	laboratory and performance info structed in June 2001. The solv	ormation greater of the second s	sathered by ODOT personnel from a single mulsion mix presented several placement	;
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ac	acres	0.405	hectares	ha	ha	hectares	2.47	acres	ac
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		<b>VOLUME</b>					VOLUMI	<u>.</u>	
fl oz	fluid ounces	29.57	milliliters	ml	ml	milliliters	0.034	fluid ounces	fl oz
gal	gallons	3.785	liters	L	L	liters	0.264	gallons	gal
ft <sup>3</sup>	cubic feet	0.028	meters cubed	m <sup>3</sup>	m <sup>3</sup>	meters cubed	35.315	cubic feet	ft <sup>3</sup>
yd <sup>3</sup>	cubic yards	0.765	meters cubed	m <sup>3</sup>	m <sup>3</sup>	meters cubed	1.308	cubic yards	yd <sup>3</sup>
NO	TE: Volumes greater th	an 1000 L shal	l be shown in $m^3$ .						
		MASS					MASS		
oz	ounces	28.35	grams	g	g	grams	0.035	ounces	OZ
lb	pounds	0.454	kilograms	kg	kg	kilograms	2.205	pounds	lb
Т	short tons (2000 lb)	0.907	megagrams	Mg	Mg	megagrams	1.102	short tons (2000 lb)	Т
TEMPERATURE (exact)				TEMP	ERATURI	E (exact)			
°F	Fahrenheit	(F-32)/1.8	Celsius	°C	°C	Celsius	1.8C+32	Fahrenheit	°F
		PRESSURE	<u>.</u>			]	PRESSUR	E	
psi p	oounds per square inch	6.895	kilopascals	kPa	kPa	kilopascals	0.1450	pounds per square inch	psi
*SI is the	he symbol for the Ir	nternational S	System of Measurer	ment					

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# **1.0 INTRODUCTION**

## 1.1 BACKGROUND

Asphalt emulsions have been used in highway construction and maintenance since the 1920s. Initially, emulsions were used as dust palliatives and in spray applications. Later, emulsions were used in the production of modified base and surface course mixes. In the recent past, the Oregon Department of Transportation (ODOT) has used as much as 500,000 T (450,000 Mg) of cold mix, i.e., emulsified asphalt concrete (EAC) for construction and maintenance activities each year, principally in ODOT Regions 4 and 5. Typical applications consist of the placement of an open-graded EAC followed by the placement of a chip seal.

Emulsions typically consist of asphalt cement, water and an emulsifying agent in the approximate proportions: 67, 30, and 3 percent, respectively. Most commonly used emulsions are mixed with a solvent to facilitate mixing and enhance aggregate coating (e.g., CMS-2S has as much as 12 percent solvent). Volatilization of the solvent represents a potential environmental issue and has led several manufacturers to develop emulsion formulations requiring little or no solvent (herein termed solvent-free).

Laboratory tests of mixes prepared with two solvent-free emulsion formulations were conducted at Oregon State University under the direction of Dr. Rita Leahy. Indirect tensile strength testing was used to monitor the strength of mixes over time. The solvent-free mixes gained strength more rapidly and attained a higher ultimate strength than comparable mixes prepared with conventional emulsions. Detailed results and analyses may be found in the project report (*Leahy, 2000*).

The laboratory results prompted ODOT to plan two field trials to be constructed during the summer of 2001 using Solvent-Free EAC.

# **1.2 OBJECTIVES**

This research documents the construction of a field trial and compares laboratory test results from conventional and solvent-free emulsion mixes. The comparisons will assist ODOT in determining future use of solvent-free emulsion mixes.

## 1.3 SCOPE

Although two field trials were planned, only one trial was constructed and only one type of solvent-free emulsion was used.

# 2.0 FIELD TRIAL

#### 2.1 EXPERIMENT DESIGN

As originally envisioned, two field trials were to be completed during the 2001 construction season. Each field trial would consist of a one-lane wide solvent-free emulsion mix section located along the project. Approximately 2,204 T (2,000 Mg) of solvent-free emulsion mix would be produced according to ODOT Special Provisions (Appendix A). The actual length of the section would be determined by the amount of solvent-free emulsion delivered in a single tank load, the percent emulsion in the mix and the thickness of the lift (nominally 2 in or 50 mm). As noted, only one of the two field trials was placed. Construction scheduling problems precluded the construction of the second field trial.

Conventional and solvent-free emulsion mixes were compared using indirect tensile strength results from cores taken during the first year following construction. The coring pattern is shown in Figure 2.1. Six cores were taken from each mix type at 14, 30, 60, 180 and 360 days following construction in both the wheel path and the center of the lane. Average strength results at each time interval and the overall rate of strength gain were compared for each coring location. In addition to testing field core samples, laboratory batched and mixed samples were prepared and tested. The laboratory test results are reported in Chapter 3.0.

Formal distress surveys were not performed, but some field notes are included in this report.



Figure 2.1: Typical coring plan for each time period (not to scale)

#### Notes:

- 1. Each core was 3.96 in (100.6 mm) in diameter.
- 2. Core spacing was approximately 2.3 ft (0.7 m) on center.
- 3. Six cores were extracted on a line 9 ft (2.75 m) from the centerline and six cores were extracted 6 ft (1.8 m) from the centerline.

# 2.2 MIX COMPOSITION AND MATERIALS

The mix designs for both mixtures were developed using ODOT Test Method 313-95 (*1998*). Mix design specifications are included in the appendices: Appendix B (Solvent-Free Mix Design) and Appendix C (Solvent-Loaded Mix Design). The emulsion designs and aggregate information are described below. Individual laboratory test results on the emulsion binders are reported in Chapter 3.0.

## 2.2.1 Emulsion Design

**CMS-2S** (Solvent Loaded) - The emulsion was manufactured using Canadian base asphalt 120-150 pen grade. The soap solution used was a standard blend of Redicote E-4819 acidified with about 8% by volume heavy naphtha (Initial Boiling Point 160°F (71 °C)) added to the finished emulsion.

**CMS-2** (Solvent-Free) - The emulsion was manufactured using Canadian base asphalt 120-150 pen grade. The soap solution was a specially formulated tall oil, lignin amine hybrid cationic emulsifier (PC-1482) supplied by Mead-Westvaco. It was acidified in the standard format for cationic emulsion. Note that there was no solvent added to this finished emulsion.

#### 2.2.2 Aggregate Source and Gradation

The aggregate used for the project was produced from Davis Pit (ODOT Source # 33-083-4). Davis Pit is a basalt quarry (shot rock, 100% crushed aggregate) with an average specific gravity of 2.717 and water absorption of 1.8%.

Aggregate gradations are plotted in Figure 2.2 for the solvent-free and solvent-loaded mixes, as well as the mix design. Table 2.1 presents the percentage breakdown of each gradation. These results were taken from one test in the case of the solvent-free emulsion and twenty tests of the solvent-loaded mix. The finer gradation and higher dust content of the material used for the solvent-free EAC relative to both the mix design and the solvent-loaded EAC mixture could explain some of the buildup of fine material during placement of the solvent-free mixture as described in Section 2.3.2. If the supplier had known about the higher than expected dust content they may have been able to account for it in their emulsion formulation.



Figure 2.2: Aggregate gradations

Table 2.1: Mix specifications	for solvent-free and	solvent-loaded mixtures
-------------------------------	----------------------	-------------------------

		Mix Design	Solvent-Free EAC	Solvent-Loaded EAC
# of Tests			1	20
	19 mm (0.75 in)	100	100	100
Percentage Passing Sieve	12.5 mm (0.5 in)	73	78	74
	6.3 mm (0.25 in)	23	30	26
	2.00 mm (#10)	2	4	3
	0.075 mm (#200)	0.9	1.6	1.2
% Moisture		1.0	1.5	1.6
Binder Content (%)		5.0	5.6	5.2
Gmm (Rice) at Field Binder Content**			2.567	2.579

\*\*Gmm for the field produced binder content was determined by back-calculation from data provided in the mix designs.

## 2.3 CONSTRUCTION

The field trial was placed just North of Tygh Valley, Oregon near the junction of Oregon Highway 216 and US Highway 197 (see Figure 2.3). The solvent-free emulsion test section was placed on June 1, 2001. Several ODOT personnel were present during the construction including representatives from the Research, Construction, and Materials Units. In addition, the asphalt chemist responsible for formulating the solvent-free emulsion for the emulsion producer was also present.



Figure 2.3: Field trial location

The conventional emulsion used on the project was a CMS-2S with approximately 8% solvent. The job mix formula emulsion content was 5.0 percent by weight of dry aggregate but was field adjusted to 5.2 percent. Ninety to 100 percent coating was achieved with the solvent loaded mix. Modifications to the solvent content and emulsifier content were made by the producer earlier in the project to improve the laydown characteristics of the solvent loaded mixture. All personnel present were satisfied with the initial behavior of the solvent loaded mixture.

#### 2.3.1 Solvent-Free Mixture Production

The plant was modified slightly so the solvent free emulsion could be pumped directly from the delivery trucks. The solvent free emulsion was designed with a higher viscosity than the CMS-2S thus the rate at which the emulsion could be pumped to the pugmill was lower. The

conventional emulsion was pumped at approximately 110 gpm (416 l/min), whereas the solvent-free emulsion was pumped at a maximum rate of about 85 gpm (322 l/min). Other than the emulsion pump rate, there were no other significant problems with producing the solvent-free mixture relative to the solvent-loaded mixture.

The initial load of solvent-free mix looked "dry" and not completely mixed. It had an emulsion content of about 5.2 percent. The second load of mixture also looked dry, and had an emulsion content of about 5.3 percent. The third load of mixture looked a little better, with an emulsion content of about 5.4 percent. Production was stopped at this point to evaluate the behavior and coating of the mixture in place, before making decisions regarding additional adjustments to the emulsion content. It is not unusual to have a significant amount of uncoated aggregate at the pugmill discharge and have the same mixture be almost or fully coated behind the paver. This is due to the additional mixing during the laydown operation.

After evaluation on the grade (see discussion in Section 2.3.2), the emulsion content was raised to 5.6 percent for the remainder of the solvent-free mixture production. Prior to start up for the fourth load, an adjustment was made to the emulsion spray bar in the pugmill to increase the mixing time in the pugmill. The emulsion was sprayed on the aggregate slightly closer to the "intake" end of the pugmill. This adjustment seemed to slightly improve the coating of the mixture at the pugmill discharge. Since there was no silo at the plant the mixture was loaded directly into trucks via a conveyor from the pugmill discharge (see Figure 2.4). A total of approximately 30 truck loads of the solvent-free mix were delivered.



Figure 2.4: Pug Mill

### 2.3.2 Laydown Operations

The paver screed was set at a width of 16 ft (4.88 m) and coated lightly with diesel oil to facilitate flow of the mixture under the screed. This is common practice for cold mixes. A CSS-1 tack coat was shot in front of the paving operation.

The Contractor used belly-dump trucks to deliver the mixture and a pickup machine attached to a Blaw Knox paver to load the mixture into the paver hopper (see Figures 2.5 and 2.6). The haul time from the plant to the grade was less than 5 minutes.



Figure 2.5: Paving operations



Figure 2.6: Behind the paver

The initial load of solvent-free mix had a moderate amount of uncoated aggregate in the windrow and about 10 percent uncoated aggregate behind the screed. The second and third loads had about the same amount of uncoated aggregate. The mixture stayed brown (unbroken) in the windrow except for a small crust at the surface of the windrow. Initially the mixture seemed to flow through the augers and under the screed well. The mixture behind the screed appeared brown, or unbroken. A black crust of mix with broken emulsion developed about 33 ft (10 m) behind the paver.

The coating of asphalt on the aggregate seemed thin relative to the CMS-2S mixture before and after breaking. Given the amount of uncoated aggregate in the first three loads, the emulsion content was increased by 0.2 percent (to 5.6 percent). This change resulted in 90 - 100 percent coating behind the paver. However, the film thickness on the aggregate was still thin relative to the solvent-loaded mixture.

After approximately 6 truck loads, large gouges were noticed in the mat beneath the center auger gearbox of the paver. Gouging was also found on the left and right sides of the mat beneath the main screed and the extensions. The gouges were about 0.4 in (1 cm) deep and about one foot ( $\sim$ 0.3 m) wide. It was suspected that buildup of fine material and asphalt on the screed was occurring at these locations. Repeated attempts were made by the screed operator to spray diesel oil down the front of the screed and to physically loosen the fine material. Neither of these proved successful and the paver was stopped approximately 3300 ft (1000 m) from the beginning.

The screed was lifted and long "stringers" of asphalt and fine material buildup were observed on the leading edge of the screed. These were worse at the aforementioned locations, but were also present more or less across the entire screed width. A propane torch was then used to heat the material so it would run off of the screed. Diesel oil was used in conjunction with a shovel to scrape away the remaining buildup of fine material and asphalt.

After the screed was reset, heat was applied to the screed to attempt to minimize the buildup of fine material and asphalt. After about 2 more truckloads, gouges began to reappear at the same locations, but ultimately spread to the entire left half of the main screed. The angle of attack of the screed was adjusted several times in an unsuccessful attempt to keep the final grade across the travel lane relatively uniform. It appeared that heating the screed with the propane heaters equipped on the screed made the buildup worse. The gouges ultimately became bad enough to stop the paver again. Neither heating nor adjusting the screed were successful.

Construction of the field trial was terminated at this point as there seemed to be no immediate solutions to address the fine material buildup on the screed. Approximately 770 T (700 Mg) of the planned 2204 T (2000 Mg) of solvent-free mixture was placed and served as the field trial section. The overall length of the solvent-free mixture section was approximately 3608 ft (1100 m).

#### 2.3.3 Compaction/Choking Operations

The same compaction equipment and processes were used for the solvent-free mixture as for the solvent-loaded mixture. The solvent-free mixture did seem to be more tender for a longer period

of time compared to the solvent-loaded mixture. Initial breakdown rolling occurred 165 to 245 ft (50 to 75 m) farther behind the paver than for the solvent-loaded mixture. No problems with the choking operation were noted. The pneumatic intermediate roller operator noted that the solvent-free mixture was tearing under the roller in some locations. These tears appeared to be taken out by the steel wheel finish roller as there was no evidence of any tears after the finish roller.

## 2.4 FIELD PERFORMANCE

Approximately 14 months following construction the project site was re-visited. Photos of the solvent-free and solvent-loaded EAC areas are shown in Figures 2.7 and 2.8. There were no discernible differences between the two sections. It should be noted that the entire project received a chip seal following the placement of the emulsion mixes.



Figure 2.7: Photo of solvent-free (right) and solvent-loaded (left) sections after approximately 14 months



Figure 2.8: Photo of the coring locations after 14 months - solvent-free in foreground

# 3.0 RESULTS AND RECOMMENDATIONS

### 3.1 LABORATORY TEST RESULTS

Laboratory tests were conducted on the field cores extracted on site to determine indirect tensile strength (*AASHTO*, 1993). Prior to construction and placement, laboratory compacted samples were prepared and also tested for indirect tensile strength. The ODOT Materials Lab also performed tests on the two emulsion binders used for this project.

#### 3.1.1 Field Laboratory Test Results

Six 3.96 in (100.6 mm) cores were extracted from the wheel path and the center of the lane for both the solvent-loaded and solvent-free emulsion mix sections. Sets of cores were extracted 14, 30, 60, 180 and 360 days following construction of the solvent-free section. The results of the extracted cores are summarized in Figure 3.1. Each data point represents the average indirect tensile strength for all six cores taken at the time indicated. Individual core strength results for each time and location are included in Appendix D.



Figure 3.1: Indirect tensile strength results - Summary of the field cores

Although the solvent-free mixture had the highest average strength (center location), there is no statistical difference among any of the mix types or core locations at any of the times since

construction. Based solely on the indirect strength data, there are no differences between the solvent-free and solvent-loaded mixes.

The indirect tensile strengths for the 14, 30, 60, and 180 day cores are approximately 1 to 3 psi lower than actual. This discrepancy was discovered by re-establishing the calibration coefficient for the proving ring of the testing apparatus used for the first four sets of cores. The 360 day cores were not adjusted because the proving ring used for measuring the strength of the cores was properly calibrated.

The cause of the marked decline in average strength shown for all mix types and core locations for 360 days is not known nor commonly reported in the literature. The reduction in strength from 180 days to 360 days for all data sets could possibly be explained by the following:

The 180 day cores were obtained during the winter (December), when the ambient temperatures are typically very cold in Tygh Valley, Oregon. The mix was probably much stiffer than when the other cores were obtained therefore relating to a higher tensile strength rating. Perhaps the cores were weakened less from the coring operation in the cold weather conditions at 180 days relative to when all of the other core sets were taken, in warmer conditions.

The rates of strength gain shown in the field trials are comparable to those found in the prior laboratory phase of this project (see *Leahy*, 2000). The prior laboratory phase concluded testing at an age of 60 days and indirect strength results ranged from about 20 to 40 psi (137.9 to 275.8 kPa).

The summary of the indirect tensile strengths for the field cores are reported in Table 3.1.

Time since	Solven Cen	t-Free iter	Solven Wheel	t-Free l path	Solvent Cer	-Loaded nter	Solvent- Whee	-Loaded l path
days	Mean psi (kPa)	St. Dev	Mean psi (kPa)	St. Dev	Mean psi (kPa)	St. Dev	Mean psi (kPa)	St. Dev
14	13 (89.63)	4	10 (68.94)	3	9 (62.05)	1	11 (75.84)	2
30	24 (165.47)	10	15 (103.42)	5	12 (82.73)	5	15 (103.42)	6
60	36 (248.21)	5	22 (151.68)	3	27 (186.15)	3	19 (131.00)	3
180	61 (420.58)	6	56 (386.10)	4	48 (330.95)	6	55 (379.21)	4
360	40 (275.79)	6	33 (227.52)	4	38 (262.00)	3	37 (255.10)	6

Table 3.1: Indirect tensile strength results for the field cores - summary

Air voids were estimated for all field specimens using the computed geometric bulk specific gravity and the theoretical maximum specific gravity and are shown in Table 3.2. There are no statistically significant differences in the estimated air voids for any of the mixes.

	Air Voids Computed from Geometric Gmb, percent							
Time since Construction,Solvent-Free Center		Solvent-Free Wheel path		Solvent-Loaded Center		Solvent-Loaded Wheel path		
days	Mean	St. Dev	Mean	St. Dev	Mean	St. Dev	Mean	St. Dev
14	29	2	33	3	32	2	31	1
30	26	4	27	1	27	1	26	1
60	27	3	27	1	29	3	28	1
180	22	1	26	1	26	2	25	1
360	23	1	27	1	26	2	27	4

 Table 3.2: Estimated air voids for the field cores at the time of testing

#### 3.1.2 Laboratory Compacted Test Results

In addition to testing the field-sampled materials, laboratory compacted specimens were also tested to determine their variation in splitting tensile strength as a function of time (Appendix E). Indirect tensile strength results for the laboratory compacted specimens are shown in Figure 3.2. Six specimens for each age and mix type were tested. Test results are summarized in Table 3.3. Paired t-tests were run to determine if the results from the solvent-free and solvent-loaded mixes were statistically similar. A paired t-test volume of 5% or less indicates a statistically significant difference. As can be seen, all of the results were statistically similar except for the seven-day samples.



Figure 3.2: Indirect tensile strength results - Summary of the laboratory specimens

	Solvent-free	e Emulsion Mix	Solvent-load	T-Test	
Age, days	Air voids, %	Indirect Tensile Strength, psi (kPa)	Air voids, %	Indirect Tensile Strength, psi (kPa)	Probability, %
7	22	36 (248.21)	23	27 (186.15)	0
30	24	37 (255.10)	24	34 (234.42)	6
60	24	35 (241.31)	24	35 (241.31)	48
180	24	46 (317.16)	24	44 (303.37)	27
360	23	48 (330.95)	23	46 (317.16)	17

 Table 3.3: Indirect tensile strength results for the laboratory samples

#### 3.1.3 Emulsion Binder Test Results

The ODOT Materials Laboratory conducted tests on each of the two emulsion binders. These results are summarized for the solvent-free and solvent-loaded emulsions in Tables 3.4 and 3.5, respectively. The results are generally similar except that the Saybolt viscosities are significantly higher for the solvent-free emulsion. Lab reports corresponding with the sample numbers are included in Appendix F (solvent-free emulsion binder) and Appendix G (solvent-loaded emulsion binder).

Test Method	Test	Sample 2001-1	Sample 2	Sample 6
T 49	Penetration @ 25°C, mm/10	140 mm/10	111 mm/10	-
T 51	Ductility @ 25°C	>149 cm	>50 cm	-
T 44	Solubility	99.8	-	-
Т 59	Emulsion Distillation @ 260°C	69.5	68.2	-
Т 59	Emulsion Distillation, % Oil	0.4	0.5	-
Т 59	Emulsion Sieve Test, % Retained	Trace	Trace	-
T 59	Saybolt Viscosity @ 50°C, SFS	630	701	538

Table 3.4: Solvent-free emulsion binder test results

 Table 3.5: Solvent-loaded emulsion binder test results

Test Method	Test	Sample 2	Sample EAC-1-1	Sample EAC-1-5	Sample EAC-1-10	Sample EAC-1-15
T 49	Penetration @ 25°C, mm/10	139	116	120	149	130
Т 59	Emulsion Distillation @ 260°C	65.2	63.5	63.2	65.1	63.6
Т 59	Emulsion Distillation, % Oil	7.55	7.5	9.0	8.0	8.0
Т 59	Emulsion Sieve Test, % Retained	No Trace	Trace	No Trace	No Trace	Trace
Т 59	Saybolt Viscosity @ 50°C, SFS	203	137	107	233	166

## 3.2 CONSTRUCTION RECOMMENDATIONS

Plant production and fine tuning of the solvent-free mix was essentially equivalent to the process used for the solvent-loaded mix. The principal differences included the lower pump rates for the higher viscosity solvent-free emulsion and the need to adjust the emulsion input into the pugmill to improve aggregate coating. The buildup of fine material on the screed during placement of the solvent-free mix caused the paving to be halted. Although the exact cause is not known, one plausible explanation follows:

The solvent-free mix developed a thin crust of broken mix on the surface of the windrow. This broken material tended to stick to the front of the screed, particularly in locations where the augers do not do a good job of continuously moving the mixture, such as at the center auger gearbox. The mixture at these locations is static for a longer period of time, so the broken asphalt would have more of an opportunity to buildup on the screed surfaces. Once the buildup starts, the areas grow as more and more fines are caught by the initial buildup. This does not occur with solvent-loaded mixes because the solvent acts as a lubricant making the mixture less sticky and less apt to stick to the metal surfaces, even in a partially broken state.

Several possible solutions to the fine material buildup are listed below:

- Use of a more uniform, lower temperature, less intensely heated screed, such as those heated with hot transfer oil, would be less likely to affect the mix, relative to the propane (open flame) heated screed used on this project;
- Use of an alternate release agent;
- Use of end-dump trucks might be more effective in minimizing the amount of the thin crust of broken material;
- Placement of tarps on the belly dump loads to minimize exposure to the wind during delivery might minimize premature breaking;
- Minimization of the windrow amount deposited in front of the paver;
- Adjustment of the emulsion break mechanism to produce adequate mixes;
- Experimentation with different pre-strike off elevations on the screed to improve the flow of material under the screed;
- Experimentation with different auger elevations and auger positions relative to the screed.

The tenderness of the solvent-free mix evident during construction could be reduced by holding the choking and intermediate compaction operations back about one-half hour to allow a greater depth of the mat to break. This would extend the length of the paving train and the duration of paving.

This field trial demonstrated that a solvent-free emulsion mix could be successfully mixed using conventional equipment and that the strengths would be comparable to solvent-loaded or conventional EAC mixes. However, the solvent-free emulsion mix could not be successfully placed using the available equipment. If additional projects are considered, these paving problems must be addressed.

## 3.3 IMPLEMENTATION

ODOT has no immediate plans to construct additional test sections. The volume of EAC produced in Oregon has declined substantially since 1999 for a number of reasons; making expenditure of resources on continuation of this research less of a priority for ODOT. Should the outlook for EAC show a planned increase in annual volume, then additional work with solvent-free emulsions should be pursued.

Future contracts with solvent-free emulsions should include provisions for an adequate time period for the emulsion supplier to thoroughly analyze and develop emulsion formulations appropriate for the aggregate source, gradation, and anticipated construction conditions prior to producing a mixture in the field. Provisions should also be included for multiple test sections to allow the supplier and contractor opportunities to make field design and equipment adjustments when problems with initial production and placement are encountered.

# 4.0 **REFERENCES**

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