

2008 CANADIAN ASPHALT EXCHANGE PROGRAM
(Polymer Modified Emulsified Asphalt Portion)
Schedule of Testing and Handling of Test Materials

The package of materials for the testing of an HF-150P emulsified asphalt contains three replicate samples.

Each of the following tests should be performed on each of the three replicate samples:

Tests on Emulsion

1. Saybolt-Furol Viscosity at 50 °C, (seconds)
(Use the same orifice for each test)
2. Residue by Distillation, (% by mass)
(Use the same still apparatus for each test)
3. Oil Portion of Distillate, (% by volume)
4. Demulsibility, 50 ml, 5.55 g/l CaCl₂, (% by mass)
5. Sieve Test

Tests on Residue

1. Penetration at 25 °C, 100 g, 5 seconds, (dmm)
2. Apparent Viscosity at 60 °C, (Pa·s)
(Use the same viscosity tube for each test)
3. Float Test
4. Solubility Test
5. Ash Content Test
6. Elastic Recovery Test

All testing for the specified properties shall be conducted in accordance with the attached HF150P Testing Procedures.

Report the results of a single determination only, not the average of two or more except in cases where an average is called for in the method. Each individual test should be made on the two replicates by the same operator. However, it is not necessary that all tests listed be done by the same operator.

PLEASE PLAN TO CONDUCT YOUR TESTING OF THE POLYMER MODIFIED EMULSION SAMPLES WITHIN FOUR WEEKS OF THE DISPATCH OF THE SAMPLE.

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If the samples have separated during shipping, please order replacement samples by telephoning:

Blaine Morien
Pounder Emulsions
806-50th Street East
Saskatoon, Saskatchewan
S7K 0X6
Phone: (306) 934-1500
Fax: (306) 934-2052

Blaine.MORIEN@huskyenergy.ca

Questions regarding the Exchange Program and completed data collection forms are to be directed/sent to the following:

Magdy Beshara, P.Eng.
Testing Standards Engineer
Saskatchewan Ministry of Highways & Infrastructure
Engineering Standards Branch
1610 Park Street
Regina, Saskatchewan
S4N 2G1
Phone: (306) 787-4922
Fax: (306) 787-4582
mbeshara@highways.gov.sk.ca

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HF-150P TESTING PROCEDURES

Sample Preparation:

- Prior to any testing, the emulsion sample shall be heated and stirred according to ASTM D 244 Par.3.1. Anything unusual should be reported.

Viscosity:

- Follow ASTM D-244 with the following modification:
 - a. Pour about 100 grams of the sample through the 1000 μ m sieve. Heat the sample to 51.4 ± 0.3 °C before pouring into the viscosity tube. Wait 2 minutes, turn off viscometer agitator and run the test.
- Report the results to the nearest 0.1 seconds.

Demulsibility:

- Follow ASTM D-244 and verify the calcium chloride solution concentration according to one of the methods outlined in ASTM D-511.

Residue by Distillation:

- The shear and thermal history of High Float emulsion residues prior to testing can cause variations in their physical properties. Careful distillation and subsequent handling of the residues is important to obtain consistent results.
- Use only the aluminium alloy still. Check the cover and flanges periodically for flatness to ensure perfect seal. Use composition gaskets approximately 1 mm thick between the still and its cover.
- Follow ASTM D-244 with the following modifications:
 - a. Wash the stills after use with petroleum solvent, rinse twice with 1,1,1-trichloroethylene or similar solvent and allow to dry thoroughly before reuse.
 - b. Drop the ring burner to the bottom of the still when temperature reaches 170 °C.
 - c. The rate of distillation will affect the consistency of the final residue. Total distillation time including conditioning at 205 °C, shall be 60 ± 5 minutes. Maintain the final temperature at 205 ± 5 °C accurately for 30 minutes. This 30 minute period shall commence when the residue temperature reaches 200 °C.
 - d. At the termination of the distillation process, weigh the still, place the still on the hot plate while you take off the cover. Stir the contents in the still with a preheated spatula for 10 seconds (about 30 times).
 - e. Pour the residue into the preheated 89 ml (3 oz.) penetration container until it is approximately half full. Pour residue into a preheated 50 ml beaker with a pouring spout and set aside as required for viscosity, float, elastic recovery and solubility/ash content tests. Pour remaining residue into the penetration container. Do not pour residual asphalt through a sieve. (Note: These containers shall be preheated on a hot plate set so that its surface temperature is 205 ± 10 °C. Since Polymer High Float residue is poured at lower temperatures than regular High Float residue. It is

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advisable to pour the Float Thimble as soon as possible to prevent any jelling structure development in the beaker.)

- f. Use a preheated spatula or utility knife to cut off polymer-asphalt strings between pouring.
 - g. Residue in the still should be poured within 2 minutes of the burner being turned off to prevent oil vapour from re-condensing into the residual asphalt.
 - h. All residues shall be conditioned and tested as required immediately after distillation. Cooling and re-heating at a later time for testing shall not be permitted.
- Report the residual content to the nearest 0.1%.

Oil Portion of Distillate:

- The oil portion of the distillate shall be calculated as a percentage of the emulsion as follows:

$$\% \text{ oil} = \frac{\text{Total volume of oil distillate, ml}}{200 \text{ g emulsion}} \times 100$$

- Report this value to the nearest 0.1 % as the "Oil Portion of Distillate, vol./wt%".

Penetration:

- In addition to test procedures given in ASTM D-244, please use the following procedure to prepare the distillation residue for penetration testing;
 - a. Follow ASTM D5 except for the following. As soon as possible after the completion of the distillation test, pour residual asphalt into the penetration container until it is approximately half full. Pour an additional 25 mL of residue into a preheated container with a pouring spout and set this on a hot plate at 205° C for viscosity and float tests. Pour remaining residue into the penetration container to a depth of approximately 60 mm. Do not pour residual asphalt through a sieve.
 - b. Immediately remove the pen can from the hot plate after pouring of the sample. Place the pen can on a 3/4" thick plywood base covered with a paper towel. Note that the piece of plywood should be large enough to ensure full contact with the inverted glass beaker.
 - c. Cover the pen can with a 600 ml low-form glass beaker. Condition the sample 1 1/2 hours at room temperature and 1 1/2 hours in the temperature bath (25° C exactly) before running the penetration test.
 - d. Distilled water should be used in the temperature bath for conditioning sample.

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- e. Report to nearest whole unit the average of three penetrations whose values do not differ by more than the following:

If first reading is between:	Maximum difference between highest and lowest reading:
101 - 150	20
151 - 200	25
201 - 250	30
251 - 300	35

- If, after five penetrations of an individual residue sample three acceptable values are not obtained, then a second residue shall be prepared from the same emulsion sample.
- The intent is to standardise the procedure for residue conditioning in air. The described procedure will reduce the effects of variable heat loss due to air conditioning, drafts and varying types of counter tops.

Sieve Test:

- Follow ASTM D-244 except that a 1000 μm sieve shall be used instead of the 850 μm sieve specified.

Float Test:

- Follow ASTM D-139 with the following modifications:
 - a. Pour the sample immediately after completion of distillation into a suitable container. The container shall be preheated and maintained after filling on a $205 \text{ }^\circ\text{C} \pm 10 \text{ }^\circ\text{C}$ hot plate. The temperature of the hot plate can be confirmed with a suitable bi-metallic surface thermometer. As soon as possible, pour the residue into the float collar. (Note: cooling of residue and re-heating for testing at a later time shall not be permitted.)
 - b. If desired, the pouring temperature can be confirmed by the use of a 30 gauge thermocouple probe and suitable recording device as follows:
 - Position the probe into the top centre of the thimble, from above. Record the maximum temperature measured while the bitumen is being poured into the thimble.
 - c. Allow the residue to cool to room temperature for 30 ± 2 minutes.
 - d. Place the plate and thimble in the ice bath for 5 minutes.
 - e. Trim the material flush to the top of the thimble with a slightly heated spatula.
 - f. Place the charged collar in the ice bath for a period of 30 ± 2 minutes.
 - g. Maintain the test bath at $60 \pm 0.5 \text{ }^\circ\text{C}$

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- **Note:** Upon cooling, soft residues may shrink well below the top of collar. If the collars are "topped" up, the additional residue must be poured at approximately 205 °C. This practice should be reported.

Solubility Test

- Follow ASTM D2042

Ash Content

- Follow ASTM D2415

Apparent Viscosity of Asphalt Residue:

- Follow ASTM D4957 with the following modification:
 - a. All viscosity testing shall be conducted at 60 °C under a 30 cm Hg vacuum. Viscosity determinations with Modified Koppers shall be reported at the shear rate of 0.5 s⁻¹.
 - b. If the residues rise into the B zone of Modified Koppers tubes prior to applying the vacuum, use only the C, D, E and F zone flow times for viscosity and shear rate determinations.
 - c. Flow time in the C zone should be within 50 and 200 seconds.
 - d. Cooling of residues to ambient temperature and re-heating for testing at a later time shall not be permitted.
 - e. Modified Koppers mK150 or mK 200 is the recommended tube size.

Elastic Recovery Test:

- Follow ASTM D113 with the following modification:
- Keep specimen at 10 °C for 85 - 95 minutes. Immediately after conditioning, place the specimen in the ductilometer and proceed to elongate the sample to 20 cm. The rate of pull shall be 5 cm/minute. After the 20 cm elongation has been reached, stop the ductilometer and hold the sample in its elongated position for 5 minutes.
- At this time, clip the sample approximately in half by means of preconditioned scissors or other suitable cutting devices. Let the sample remain in the ductilometer in an undisturbed condition for one (1) hour. At the end of this time period, retract the half sample specimen until the two broken ends touch. Record the elongation in cm.
- Calculation: Calculate percent recovery by the following formula:

$$\% \text{ Recovery} = \frac{20 - x}{20} \times 100$$

Where:

x = observed elongation after rejoining of sample, cm.

- Report the Elastic Recovery to the nearest 0.1%

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A. PARTICIPANT DOCUMENTATION

Agency/Company _____

Address _____

Contact Person _____

Telephone No. _____

Fax No. _____

E-mail Address _____

B. SAMPLE DOCUMENTATION

Date Samples Received _____

Date Testing Commenced _____

Dated Testing Completed _____

C. PARTICIPANT COMMENTS

Please provide any comments which may be of value to this or future exchanges:

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D TEST RESULTS

POLYMER MODIFIED EMULSIFIED ASPHALT GRADE HF-150P			
Replicate Number	<input type="text"/>	<input type="text"/>	<input type="text"/>
TESTS ON EMULSION			Test No.
Saybolt-Furol Viscosity @ 50 °C, (s) <small>(Report to the nearest 0.1 s)</small>	<input type="text"/>	<input type="text"/>	301
Date Saybolt-Furol Viscosity Tested (yy-mm-dd)	<input type="text"/>	<input type="text"/>	<input type="text"/>
Residue by Distillation, (% by mass) <small>(Report to the nearest 0.1 %)</small>	<input type="text"/>	<input type="text"/>	302
Total Distillation Time, (minutes) <small>(Report to the nearest minute)</small>	<input type="text"/>	<input type="text"/>	303
Oil Portion of the Distillate, (ml) <small>(Report to the nearest 0.5 ml)</small>	<input type="text"/>	<input type="text"/>	<input type="text"/>
Oil Portion of the Distillate, (% by Volume of Emulsion) <small>(Report to the nearest 0.1 %)</small>	<input type="text"/>	<input type="text"/>	304
Demulsibility, 50 ml, 5.55 g/l CaCl₂, (% by mass) <small>(Report to the nearest 0.1 %)</small>	<input type="text"/>	<input type="text"/>	305
Sieve Test	<input type="text"/>	<input type="text"/>	310
TESTS ON ASPHALT RESIDUE			Test No.
Penetration at 25°C, 100 g, 5 s, (dmm) <small>(Report to the nearest whole unit)</small>	<input type="text"/>	<input type="text"/>	306
Residue Conditioning Time in Air, (minutes) <small>(Report to the nearest minute)</small>	<input type="text"/>	<input type="text"/>	307
Residue Conditioning Time in Water Bath, (minutes) <small>(Report to the nearest minute)</small>	<input type="text"/>	<input type="text"/>	308
Apparent Viscosity at 60°C, (Pa-s)	<input type="text"/>	<input type="text"/>	309
Float Test (seconds)	<input type="text"/>	<input type="text"/>	311
Solubility	<input type="text"/>	<input type="text"/>	312
Ash Content	<input type="text"/>	<input type="text"/>	313
Elastic Recovery (%)	<input type="text"/>	<input type="text"/>	314
Agency / Company Name:	<input style="width: 100%;" type="text"/>		

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POLYMER MODIFIED EMULSIFIED ASPHALT GRADE HF-150P					
Replicate No.					<input style="width: 50px;" type="text"/>
Vacuum, mm Hg					<input style="width: 50px;" type="text"/>
Date Tested (yy-mm-dd)					<input style="width: 50px;" type="text"/>
Modified Kopper Viscometer Size No.					<input style="width: 50px;" type="text"/>
Zone	B	C	D	E	F
Viscosity Constant					
Flow Time, s					
Tube Shear Constant, K					
Shear Rate, s					
Viscosity, Pa·s at 0.5 s ⁻¹					
Replicate No.					<input style="width: 50px;" type="text"/>
Vacuum, mm Hg					<input style="width: 50px;" type="text"/>
Date Tested (yy-mm-dd)					<input style="width: 50px;" type="text"/>
Modified Kopper Viscometer Size No.					<input style="width: 50px;" type="text"/>
Zone	B	C	D	E	F
Viscosity Constant					
Flow Time, s					
Tube Shear Constant, K					
Shear Rate, s					
Viscosity, Pa·s at 0.5 s ⁻¹					
Replicate No.					<input style="width: 50px;" type="text"/>
Vacuum, mm Hg					<input style="width: 50px;" type="text"/>
Date Tested (yy-mm-dd)					<input style="width: 50px;" type="text"/>
Modified Kopper Viscometer Size No.					<input style="width: 50px;" type="text"/>
Zone	B	C	D	E	F
Viscosity Constant					
Flow Time, s					
Tube Shear Constant, K					
Shear Rate, s					
Viscosity, Pa·s at 0.5 s ⁻¹					
Agency / Company Name:					<input style="width: 250px;" type="text"/>

