

**1852**                    **QUANTITATIVE EXTRACTION OF BITUMINOUS MIXTURES  
(CENTRIFUGE)**  
AASHTO T 164, METHOD A (MN/DOT Modified)

**1852.1**            SCOPE

This method of analysis quantitatively determines the asphalt content of bituminous mixtures using a centrifuge extractor and trichloroethylene or other approved solvents. The use of alternatives like n-propyl bromide and d-limonene, are suitable replacements. These solvents are generally non-carcinogenic and essentially non-toxic. Required alterations to this procedure for using n-propyl bromide and d-limonene are covered in the numbered "Notes" below. This method is Mn/DOT's preferred method.

**NOTE 1:** This procedure differs from AASHTO T 164 as follows: Mn/Dot allows the use of n-propyl bromide and d-limonene. When using d-limonene a number of water rinses are required and then the sample is dried at  $176.7 \pm 5$  °C ( $350 \pm 9$  °F). MN/DOT also requires the use of a diatomaceous earth filtering aid (D.E.) ie. Celite to be added to the mixture. When using D.E., determining the amount of fines in the extract is not required by MN/DOT. (Exception to this requirement is when the mineral matter in the extract is determined.) MN/DOT allows a minimum solvent soak time of 1 hour but the extraction process must be completed within 24hrs.

**1852.2**            APPARATUS

- A.     Extraction apparatus - Centrifuge extractor with 3000 gram sample capacity in which the bowl may be revolved at controlled, variable speeds up to 3,600 RPM. The apparatus should have explosion-proof features.

**NOTE 2:** Several manufacturers claim that their extractors have a capacity of 3000 grams. **THEY DO NOT.** Before ordering new equipment, the bowl size on an existing, satisfactory unit should be carefully measured and those measurements referred to in the specifications for the new unit.

- B.     Filter ring - Paper or felt, to fit the rim of the bowl.
- C.     Balance - Conforming to the requirements of AASHTO M 231 with a readability and sensitivity of 0.1 gram and an accuracy of 0.1 gram or 0.1%. The balance must have sufficient capacity to weigh the bowl and its contents.
- D.     Oven - Thermostatically controlled to  $110 \pm 5$  °C ( $230 \pm 9$  °F).

**NOTE 3:** Use  $176.7 \pm 5$  °C ( $350 \pm 9$  °F) when using d-limonene.

- E.     Chemicals: Trichloroethylene technical grade, n-propyl bromide, d-limonene (or other approved solvent).

**NOTE 4:** D-limonene or "Zecol/Milsolv plus" is a biodegradable solvent with an added surfactant that is primarily derived from citrus processing. Its use requires a

rinsing with water to remove the bio-solvent and a much higher drying temperature for any surfactant residue.

- F. Filter Aid - Diatomaceous Earth (D.E.) ie.Celite. Perform a sieve analysis on each bag for gradation calculations.

**NOTE 5:** It has been found useful to sieve the D.E. on the 75  $\mu\text{m}$  (#200) sieve and discard all that is retained on that sieve. This simplifies the calculation and eliminates any possibility of segregation of the material.

- G. Spoon - Stainless steel, approximately 305mm (12") long. Grind tip of spoon flat for easier manipulation of sample.

- H. Pan - Aluminum or stainless steel approximately, 330 X 230 X 60mm (13 X 9 X 2 1/4").

### 1852.3 LABORATORY CONDITIONS

In addition to good, general lab ventilation the extraction work area should be well ventilated. Humidity control is recommended to prevent excessive water absorption by the dried aggregate.

### 1852.4 DISCUSSION

The centrifuge method of bituminous extraction is an alternate to the Colorado extraction method. Centrifuge extraction uses about 50% less solvent and is faster than Colorado extraction. More fines are lost through the filter in this method than in the Colorado extraction method. Adding 50 grams of D.E. to the sample before extracting will reduce fines loss. In lieu of using D.E., the process of Determining the Amount of Mineral Matter in the Extract is required as per AASHTO T164.

### 1852.5 SAFETY

Diatomaceous Earth contains crystalline silica, which is considered a hazard through inhalation. Goggles or safety glasses and a NIOSH approved respirator for particulates are recommended. Refer to the safety data sheet for proper handling.

N-Propyl bromide is a chemical with a low order of toxicity. If inhaled, it may irritate the nose, throat and lungs. Use in a well ventilated area or with an approved respirator. Chemically resistant gloves are recommended and safety glasses are required. Refer to the Material Safety Data Sheet and your safety officer for proper handling and disposal procedures.

Trichloroethylene is a toxic chemical. It is a clear colorless liquid with a chloroform-like odor. Refer to the literature and your safety officer for proper handling procedures of this and any other solvent used. Do not wash hands with trichloroethylene.

Trichloroethylene is a hazard by ingestion, skin absorption, and vapor inhalation. Repeated or excessive skin contact with its vapor or liquid may cause severe dermatitis. Wash thoroughly after handling. Use of a local exhaust ventilation system is recommended.

Eye protection is mandatory. If eye contact occurs, flush eye with clean water for 10-15 minutes and consult a doctor. Full face shields with top and side shields provide the best protection. Chlorinated solvent resistant rubber gloves and protective apron should be worn at all times while performing extractions.

Passage of trichloroethylene over hot electrical coils or flames, including cigarettes, can cause the formation of phosgene gas and other decomposition products which may be more hazardous than trichloroethylene. Prolonged storage of waste solvent should be avoided because of MPCA regulations. Waste solvent should be periodically sent for recycling. Drums of waste solvent should be checked regularly for pressure build-up. Store in a cool dry ventilated area.

Purchase of new and disposal of used trichloroethylene shall be in accordance with existing laws, contracts and procedures.

**NOTE 6:** D-limonene products may cause skin irritation. Wash skin with soap and plenty of water, the use of protective gloves is recommended. Safety goggles must be used. If eye contact occurs flush eyes with plenty of water. Use with adequate ventilation, d-limonene emits an orange citrus aroma which can be irritating to some. Refer to the literature and your safety officer for proper handling and disposal procedures. D-limonene products may have a flash point below 60 °C (140 °F) and should be handled accordingly.

## 1852.6

### SAMPLE PREPARATION

- A. Heat mixture sample in oven at  $110 \pm 5$  °C ( $230 \pm 9$  °F) until the sample is dry and soft.
- B. Using the appropriate quartering sampling method (See Manual Section 1002) obtain an extraction sample of 2000 – 2100 grams.
- C. The test portion shall be at a “dried to a constant weight “ state (moisture free) before testing.

**NOTE 7:** A minimum drying time of 45 minutes at  $110 \pm 5$  °C ( $230 \pm 9$  °F) has been established for most mixtures that are free of moisture. However, when determining the moisture content the following applies: A constant weight for mixtures shall be defined as the mass at which further drying at  $110 \pm 5$  °C ( $230 \pm 9$  °F)

does not alter the mass by more than 0.05 percent The sample shall be initially dried for 2 hours. Then continue drying for 30 minute intervals until a constant weight is reached. (On a 2000 gram sample this amounts to a difference of 1.0 grams or less.)

- D. RAP aggregate samples shall be dried at  $110 \pm 5$  °C ( $230 \pm 9$  °F) for a minimum of 2 hours and then checked at 30 minute intervals until a constant weight, as defined in NOTE 7 (above), is achieved. Refer to section 1201.4H for RAP preparation.
- E. RAS (Shingles) material should be air dried to a constant weight. Sample size is 500-700 grams. Refer to section 1201.4j for RAS preparation.

### 1852.7 PROCEDURE

- A. Weigh an empty, clean centrifuge bowl and record to the nearest 0.1 gram.
- B. Add sample to bowl and record weight to the nearest .01 gram. Distribute the sample evenly in the bowl
- C. Weigh 50.0 grams of D. E. and add it to the sample by sprinkling it over the mixture in the bowl.

**NOTE 8:** If D.E. is not used, the amount of mineral matter in the extract must be determined. When extracting RAP for aggregate specific gravity testing (Mn/DOT 1204 & 1205) the extraction process is done without the use of D.E.

- D. Fit bowl into centrifuge apparatus and add trichloroethylene or other approved solvent (n-propyl bromide) until the sample is covered.
- E. Weigh and record weight of a dried filter ring to the nearest 0.1 gram.

**NOTE 9:** The filter ring needs to be dried to a constant weight at  $110 \pm 5$  °C ( $230 \pm 9$  °F).

- F. Place the filter ring on the extractor bowl and assemble centrifuge apparatus. Remember to hand tighten the knurled lock nut onto the bowl's cover and to secure the extractor's lid.
- G. Allow the solvent to dissolve the sample for a minimum of 1 hour. The extraction process must be completed within 24 hrs. With some aggregates, like recycled bituminous it is difficult to remove all the asphalt cement. Soaking for a longer period and stirring occasionally may help with this process. Also some solvents will require a longer soak time to dissolve the sample.

- H. Place a suitable container under the extractor's drain to collect the filtrate.
- I. Start the securely anchored centrifuge and slowly increase to the proper speed (2000 – 2500rpm). Solvent will be rapidly expelled from the drainpipe. For automatic models preset the length of time and maximum speed prior to starting.
- J. Run the centrifuge until the solvent flow slows to a dripping rate. Shut the centrifuge down and as the bowl begins to slow down stop the bowl's rotation by applying the manual brake. On automatic models the extractor stops and brakes as the preset time expires.
- K. Add an additional 200 to 400ml of solvent into the top of the extractor. Repeat step I. and run centrifuge until solvent flow slows to a dripping rate. Stop the bowl.
- L. Repeat solvent-washing cycle at least two more times until the solvent flowing from drainpipe is a light straw color when viewed against a white background. Due to the nature of the extraction process it is impossible to obtain a perfectly clear extract without using an excessive amount of solvent.
- M. At the end of the final solvent rinse cycle the bowl should be rotated at approximately 3000 rpm for a minute. The purpose of this is to remove as much of the solution as possible from the aggregates
- N. Remove the container which contains the asphalt/solvent filtrate. Dispose of it in an appropriate manner, unless a determination of mineral matter in the extract is required. For this step refer to AASHTO T-164 Test Method "A", Annex A1.  
  
**NOTE 10:** For d-limonene based solvents an extra water rinse is necessary to remove the biodegradable residue after the solvent rinse cycle. Add about 400ml of hot tap water into the centrifuge, spin it out and repeat in the same way as with the solvent. The exit flow from the initial water rinses will be milky white in appearance. The first one or two water rinse(s) is typically captured into the waste container and disposed into the asphalt/solvent waste barrel. Repeat the rinsing (approximately 4-5 water rinses of 800ml each should suffice) until the exit flow becomes semi-transparent, cloudy or skim milk like in color. Spin the last rinse at 3000 rpm for a minute. Determine the pH of the wash water and adjust to neutral if necessary, then wash down the drain with additional water. Some regulatory agencies may require adjustment of the pH of the rinse water before it is discharged into the sewer system; other areas/agencies may not allow any discharge. - **Check with the local authorities.**
- O. Remove the centrifuge cover and place the filter ring and centrifuge bowl with the aggregate under a hood until the chemical fumes dissipate. Then take the centrifuge bowl with the ring and aggregate and place it into an oven.
- P. Dry the aggregate in the centrifuge bowl along with the filter ring in the  $110 \pm 5$  °C ( $230 \pm 9$  °F) oven to a constant weight. Record weight to the nearest 0.1 gram.

**NOTE 11:** The constant weight for extracted aggregates shall be defined as the mass at which further drying at  $110 \pm 5$  °C ( $230 \pm 9$  °F) does not alter the mass by more than 0.05 percent. (On a 2000 gram sample this amounts to a difference of 1.0 grams or less.) Samples shall be initially dried for 2 hours and then the mass is determined at additional 30 minute intervals until a constant weight is achieved.

**NOTE 12:** Use of d-limonene solvents requires drying at  $176.7 \pm 5$  °C ( $350 \pm 9$  °F). Dry for a minimum of three hours then check for a constant weight as described in NOTE 11 (above) at 30 minute intervals.

Q Carefully brush off any remaining fines from the filter ring into the bowl or pan and record the ring weight #2 to the nearest 0.1 gram. The trapped fines in the ring is considered minus #200 material and needs to be accounted for in the extracted aggregate gradation.

R Record the final weight of the bowl and extracted aggregate to the nearest 0.1 gram.

**NOTE 13:** Since dry aggregate absorbs moisture when exposed to air containing moisture, determine the dry mass of the extracted aggregate within 30 minutes after the constant weight has been determined.

S. Extracted aggregate is then tested for gradation and/or other analysis.

## 1852.8

### CALCULATIONS (See Example Worksheet)

A. Bowl weight.

B. Sample and Bowl weight.

C. Sample Weight = (B-A)

D. Celite weight.

E. Ring<sub>1</sub> = Weight of oven dried filter ring before extraction.

F. Ring<sub>2</sub> = Weight of dried filter ring plus trapped minus #200 fines after extraction.

G. Ring fines = Ring<sub>2</sub> - Ring<sub>1</sub>

H. Final Oven Dried Weight. = Extracted aggregate in bowl with filter ring.

I. Final (Extracted) Aggregate Weight = (H-A-D - E)+K

J. Weight loss = (C-I)

K Mineral Matter in Extract

**Note: 14** When the recovery of the mineral matter in the extract is required, the weight of the mineral matter needs to be added to the final extracted aggregate weight before calculating the % asphalt. Refer to 1852.10

% Asphalt =  $(J / C) \times 100$  (Report the to the nearest 0.01%)

1852.9

EXAMPLE WORKSHEET

SAMPLE NO: \_\_\_\_\_

## CENTRIFUGE WORKSHEET

BOWL NUMBER \_\_\_\_\_  
BOWL WEIGHT \_\_\_\_\_ =A  
SAMPLE & BOWL WEIGHT \_\_\_\_\_ =B  
SAMPLE WEIGHT: (B-A) \_\_\_\_\_ =C  
CELITE WEIGHT \_\_\_\_\_ =D

RING WEIGHT #1 \_\_\_\_\_ =E  
RING WEIGHT #2 \_\_\_\_\_ =F  
FINES IN RING WEIGHT: (F-E) \_\_\_\_\_ =G

OVEN DRY WEIGHT #1 \_\_\_\_\_  
OVEN DRY WEIGHT #2 \_\_\_\_\_  
OVEN DRY WEIGHT #3 \_\_\_\_\_  
FINAL OVEN DRY WEIGHT \_\_\_\_\_ =H

EXTRACTED AGGREGATE WEIGHT:  
(H - A - D - E) + K \_\_\_\_\_ =I  
WEIGHT LOSS: (C-I) \_\_\_\_\_ =J  
MINERAL MATTER IN EXTRACT \_\_\_\_\_ =K  
% ASPHALT:  $(J/C) \times 100$  \_\_\_\_\_

### When Required:

MINERAL MATTER DETERMINATION for line (K)

- Weight of clean centrifuge cup: \_\_\_\_\_
- Weight of centrifuge cup & fines: \_\_\_\_\_
- Weight of Mineral Matter: (b-a) \_\_\_\_\_

GRADATION REQUIRED Yes or No

TESTED BY \_\_\_\_\_ DATE \_\_\_\_\_

## 1852.10 RECOVERING THE AMOUNT OF MINERAL MATTER IN EXTRACT BY USE OF A CONTINUOUS FLOW FILTERLESS CENTRIFUGE

Note: 15 When required Mn/DOT uses a Continuous Flow High Speed Centrifuge to determine the amount of mineral matter in the extract. This device feeds the extract into a cup rotating at approx. 11,000 rpms. The solvent/extract is forced up the walls of the cup and out through an overflow tube. The mineral matter remains in the cup. This procedure requires the use of a balance conforming to the requirements of AASHTO M 231 having an accuracy, sensitivity and readability of 0.01 grams.

- A. Obtain and record the weight of an empty clean centrifuge cup to the nearest 0.01grams.
- B. Place the cup into the centrifuge holder.
- C. Attach the cover and funnel/reservoir. Make sure the feed line valve is closed. Place a container under the overflow spout.
- D. Fill the reservoir with the recovered asphalt/solvent extract from the initial extraction process. Pre-dissolving this extract with additional solvent is permitted.
- E. Start the centrifuge and allow let it to reach a constant speed.
- F. Open the feed line valve. Set feed rate to 100 to 150mL per minute.
- G. Keep the reservoir full and transfer all of the extract into the centrifuge. Rinse the extract container with clean solvent and pour filtrates into the reservoir.
- H. After all the extract has passed through the centrifuge, rinse the reservoir and feeder tube thoroughly with clean solvent. Keep the centrifuge running.
- I. Turn off the centrifuge and allow it to stop. Remove the cover and the cup. Clean the outside of the cup with solvent.
- J. Allow the residual solvent evaporate under an exhaust hood and then place the cup and mineral matter in an oven at  $230 \pm 9F^{\circ}$ . Dry to a constant weight.
- K. Cool the cup for 10-15 minutes then immediately obtain and record the final weight to the nearest 0.01 grams.
- L. The cup's increase in weight is the mass of "Mineral Matter in Extract". Record this weight on the Extraction Worksheet under line "K"



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