Tex-210-F, Determining Asphalt Content of Bituminous Mixtures by Extraction

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Section 1

Overview

Effective dates: December 2004–March 2008.

Use this test method to determine, by four cold solvent extraction procedures, the percentage of asphalt in a paving mixture based on the weight of an asphalt and aggregate mixture. The aggregate and fines recovered can be used for sieve analysis ("Tex-200-F, Sieve Analysis of Fine and Coarse Aggregates").

Units of Measurement

The values given in parentheses (if provided) are not standard and may not be exact mathematical conversions. Use each system of units separately. Combining values from the two systems may result in nonconformance with the standard.

Sample Size

Use the 'Sample Size' table to determine the minimum sample size.

Notes

Numbered notes cited in the test method are located in the 'Notes' section.

Section 2

Part I, Centrifuge Extraction Method Using Chlorinated Solvent

This part describes the extraction method using chlorinated solvent.

Apparatus

The following apparatus is required:

- extractor, motorized, with explosion-proof features, of the centrifuge type, consisting of a bowl mounted in an assembly so the bowl revolves at a controlled speed. (The apparatus should have a container or outer shell for catching solvent thrown from the bowl and a drain for collecting the solvent.)
- ventilation as required by current specifications for asphalt mix control laboratories. (System requirements for nonchlorinated solvents apply unless tests show that the alternate system lowers the solvent vapor to safe levels throughout the testing process.)
- filter paper to fit the rim of the bowl and having a 20-25 µm retention factor
- scoop, spatula, trowel and small brush
- graduate, 2000 mL capacity, minimum
- filtrate containers
- silica evaporating dish, 200 mL capacity, minimum
- balance, readable to 0.1 g and accurate to 0.5 g
- balance readable to 0.01 g and accurate to 0.01 g
- drying oven, capable of attaining a temperature of 93°C (200°F) or more, or suitable microwave oven. (Vent the drying oven to the outside. The microwave oven must be located in a ventilated area.)
- muffle furnace or burner and hot plate
- large flat pan or microwave dish, solvent pouring container, beakers, etc.
- desiccator (optional)
- sealed magnetic stirring device (optional)
- pipette, 100 mL capacity.

Materials

The following materials are needed:

- extraction solvent: 1,1,1 trichlorethylene or methylene chloride
- ammonium carbonate solution – saturated solution of ACS Grade $(NH_4)_2CO_3$.

Safety Considerations

Follow the safety considerations detailed in 'Safety Considerations (Chlorinated Solvents)' to safely perform this procedure.

Preparing Sample

Follow these steps to properly prepare the sample for this procedure.

Preparing Sample for Centrifuge Extraction Method Using Chlorinated Solvent

Step	Action
1	Remove any base material and soil that might be adhering to the compacted asphaltic concrete. *NOTE:* The bituminous mixtures necessary for the extraction test may be loose material or a small section of compacted pavement from the roadway.
2	Place several thousand grams in a large, flat pan and warm in oven until it becomes workable. If the asphaltic mixture is not workable and cannot be separated and reduced to laboratory test size. ♦ Mix temperature should not exceed 93°C (200°F) due to flammability when used with non-chlorinated solvents. ♦ Use a trowel to separate the particles of the sample using care not to fracture the aggregate particles.
3	♦ Mix, blend, and quarter the mixture continuously until 2 combined opposite quarters weigh a minimum amount corresponding to the 'Sample Size' table. ♦ For mixtures not subject to segregation, thoroughly blend the material and take small portions from several places covering the entire area of the pan.
4	♦ Dry the sample to constant weight at a minimum temperature 93°C (200°F) if the sample consists of hot-mix, cold-laid material, pre-coated aggregate, pavement or any fresh mixture which may contain moisture. ♦ If the sample consists of rock asphalt, dry to constant weight at a minimum temperature of 60°C (140°F) (see NOTE 1 in the 'Notes' section).
5	♦ Select representative portions of the mixture as received and determine the moisture content according to "Tex-212-F, Determining Moisture Content of Bituminous Mixtures," and the hydrocarbon volatile content as outlined in Test Method "Tex-213-F, Determining Hydrocarbon-Volatile Content of Bituminous Mixtures," if the extraction is to be run on material containing moisture and/or hydrocarbon volatiles before drying. ♦ Prepare and weigh the samples for these tests at the same time.'

Procedure

Follow these steps to determine asphalt content by centrifuge extraction method using chlorinated solvent.

Determining Asphalt Content of Bituminous Mixtures by Extraction, Centrifuge Extraction

Step	Action
1	Weigh the laboratory size sample for extraction to the nearest estimated 0.1 g.
2	Transfer the weighed sample of asphaltic concrete into the extractor bowl, taking care not to lose any of the material.
3	♦ Cover the sample with solvent (see Note 2).

Determining Asphalt Content of Bituminous Mixtures by Extraction, Centrifuge Extraction	
Step	**Action**
	♦ Pour all the solvent in the airflow of the ventilation system. If necessary, use a small amount of solvent to wash any material remaining in the pan into the bowl.
4	Weigh a clean, dry, filter paper to the nearest 0.1 g. If necessary, dry the filter paper in an oven at a maximum temperature of 138°C (280°F).
5	Place filter paper and top on extractor bowl and screw on funnel-clamp until tight.
6	Place the bowl containing the sample and solvent into the machine, tighten the lock nut, and secure the extractor lid.
7	Place a container under the drain to catch liquid extracted from the sample and start the machine.
8	Stop the centrifuge and collect the discharged liquid in a 2000 mL graduated cylinder when solvent ceases to flow.
9	Add approximately 300 mL of solvent to the bowl through the funnel, and start the centrifuge.
10	Collect the discharged liquid, stop the centrifuge, pour the discharged liquid back into bowl and restart the centrifuge.
11	♦ Stop the centrifuge and add the discharged liquid to that liquid already in the graduate. ♦ Continue this procedure, centrifuging each addition of solvent several times until the extract is reasonably clear and is approximately the same color as clean solvent. ♦ Stop the motor every time before pouring solvent into the funnel. NOTE: It is impossible to secure a clear extract without disintegrating some of the aggregate or using an excessive quantity of solvent.
12	Collect and measure the total amount of discharged liquid.
13	Carefully remove the top and filter paper from extractor bowl and brush all clinging aggregate particles back into the bowl.
14	Dry the filter paper to constant weight (See Note 1.)
15	Subtract the original weight of filter paper (Step 3) from the weight recorded in Step 14 to obtain the weight of the fine mineral matter (assumed to be particles passing the 75µm [No. 200] sieve) contained in it.
16	♦ Transfer the aggregate in the extraction bowl to a tared pan and dry to constant weight at a minimum temperature of 93°C (200°F) or more. ♦ Weigh and record the net weight of aggregate.
17	Obtain the weight of a 200 mL silica evaporating dish to the nearest 0.01 g.
18	Agitate the liquid collected in the 2000 mL graduate thoroughly and immediately withdraw a 100 mL sample from the middle third of the liquid by means of a 100 mL pipette and suction bulb.
19	Pour the 100 mL into the tared silica dish and evaporate the solvent on a gas or electric hot plate under a hood or well ventilated area. *CAUTION: Depending upon the solvent used, the evaporating fumes may be toxic and flammable.*
20	♦ Ignite the residue over a burner, or, if available, place the dish in a muffle furnace for 45 min. at approximately 760°C (1,400°F) to ash the bituminous material. ♦ Whether burner or muffle furnace is used, continue the process (for an extended period, if necessary) until the ashing is complete.
21	Complete ashing should be obvious from visual observation. Should any doubt arise, completion of ashing can be determined by continuing the process to a constant weight condition.
22	Cool and add just enough ammonium carbonate $(NH_4)_2CO_3$ to wet the ash remaining in the dish.
23	Place the dish on a hot plate and evaporate to dryness at low temperature.
24	♦ Cool the dish and contents to room temperature in a desiccator, if available, and weigh to the nearest 0.01 g. ♦ Record the net weight.

Determining Asphalt Content of Bituminous Mixtures by Extraction, Centrifuge Extraction	
Step	**Action**
25	Determine the weight of the residue. (See Note 7.) NOTE: Omit the use of ammonium carbonate if prior tests do not indicate a need for the re-carbonation of the ash. An Ash Factor may be used in lieu of ash determination.

Retention Factor

Certain types of aggregate retain a small amount of bitumen after the extraction test. If this occurs, consider it when calculating the final percent of bitumen in the mix.

Use the following procedure to determine a factor to make the required adjustment. Where the retention factor is determined to be less than 0.1%, disregard it. Once a retention value is determined for a particular design, use this value until a design change occurs or for any other valid reason. Check it periodically during lengthy projects.

Procedure

When checking retention factors of tested aggregates, follow the procedure outlined in this table.

Establishing the Retention Factor	
Step	**Action**
1	♦ Perform this procedure separately on at least 2 representative specimens of aggregate used in the mix. ♦ Dry the aggregate specimens to a constant weight at a temperature of 93°C (200°F) or more.
2	Mix the design amount of asphalt with the aggregates as outlined in "Tex-205-F, Laboratory Method of Mixing Bituminous Mixtures."
3	♦ Place the mixture in an oven and heat to the anticipated plant mixing temperature. Hold at that temperature for the period of time estimated as that between plant mixing and sampling for extraction. ♦ If the extraction sample is cured in an oven for 2 hr. at 121 ± 3°C (250 ± 5°F) with material for laboratory compaction, it is also necessary to cure the retention factor samples an additional 2 hr. at 121 ± 3°C (250 ± 5°F).
4	Begin the extraction process at the same mixture temperature as that used during job control tests.
5	Calculate the average of the 2 residual bitumen values. *NOTE:* If the values vary by more than 0.2 %, run additional tests (see 'Calculations').

Calculations

The retention factor calculation is:

$$R = J_A - J_E$$

Where:

- R = Retention factor
- J_A = Percent residual bitumen added to each mix
- J_E = Average percent residual bitumen determined from the test specimens.

Ash Factor Alternative

The ash factor is an amount of ash that is to be used repeatedly as part of the total aggregate portion of an extraction test. It takes the place of the ash value usually determined for each extraction.

The ash factor may differ with each individual. Therefore, establish an ash factor for each technician because of personal differences in laboratory technique:

- Run a minimum of 4 extraction tests during 4 different days of production.
- Note the total ash for each extraction.
- If the total ash values of the 4 extractions do not vary by more than 0.1% by weight of the total sample weight, the number of grams of total ash can become the ash factor for each extraction test as long as:
  - there is no change in the aggregate gradation or source
  - the asphalt content does not change
  - the plant production remains uniform.
- Periodic checking of the ash factor by ash determination is essential for justifying its continued use.

Omit ash determination when using extraction for the sole purpose of cleaning aggregate for sieve analysis.

Section 3

Part II, Vacuum Extraction Method Using Chlorinated Solvent

This part describes the vacuum extraction method using chlorinated solvent.

Apparatus

Use the following apparatus:

- vacuum extractor complete with vacuum pump, extractor O-rings, tubing, filter support plate, tank and funnel ring. The department recommends a vane-type vacuum pump. An exhaust hose is required for the vacuum pump to carry fumes to immediately in front of the exhaust fan of the ventilation system.
- ventilation as required by current specifications for asphalt mix control laboratories. (System requirements for nonchlorinated solvents apply unless tests show that the alternate system lowers solvent vapor to safe levels throughout the testing process.)
- ultrasonic cleaner with non-perforated insert tray (optional)
- filter paper, size 331 mm (13 in.) having a 20-25 µm retention factor
- forced draft oven, capable of attaining a temperature of 93°C (200°F) or more, or suitable microwave oven. (Vent the drying oven to the outside. The microwave oven must be located in a ventilated area.)
- balance readable to 0.1 g and accurate to 0.5 g
- small scoop or spoon, spatula, trowel and small brush
- large flat drying pan or microwave container
- plastic wash bottles and glass beaker graduates
- container of adequate size to hold test sample and solvent without spilling when stirred
- comb, hair-pick type, with long stiff tines which are approximately 3.8 mm × 6.4 mm (0.125 in. × 0.25 in.) apart. The comb can be metal or plastic, provided the tine ends are smooth and rounded.

Materials

Use the following materials:

- extraction solvent: 1,1,1 trichlorethylene or methylene chloride
- diatomaceous silica filtering aid (Celite 110 or equivalent)(optional).

Safety Considerations

Use the safety considerations detailed in 'Safety Considerations (Chlorinated Solvents)' to safely perform this procedure.

Test Record Forms

Link to the "Hot Mix Asphalt Properties" worksheet in Excel.

Preparing Sample

Preparing the sample is identical to that described in 'Part I, Centrifuge Extraction Method Using Chlorinated Solvent.'

Procedure

Follow these steps to determine asphalt content by vacuum extraction method using chlorinated solvent.

Determining Asphalt Content of Bituminous Mixtures by Vacuum Extraction Method Using Chlorinated Solvent	
Step	**Action**
1	Weigh and record the laboratory size sample for extraction to the nearest estimated 0.1 g.
2	Transfer the weighed sample into the sample container or ultrasonic cleaner insert tray.
3	♦ Carefully cover the sample with solvent (see Note 2). ♦ Pour all solvents immediately in the airflow of the ventilation system.
4	♦ Gently stir the solvent and mixture sample in the container until visually noticing the asphalt in the solution. ♦ If using an ultrasonic cleaner, fill the tank with water to a level which will closely approximate the solvent level after placing the insert tray in the water bath. *NOTE:* Add a small quantity of wetting agent (liquid detergent) to the water. Water may be stored in the bath for future testing. Occasional replacement is necessary.
5	Place the insert tray and its contents in the ultrasonic cleaner and activate the cleaner. The tray should not touch the side of the water bath in order to obtain the best cleaning action.
6	Stir the mixture occasionally. Hands or fingers should not be in the water bath or solvent when the ultrasonic cleaner is on.
7	Center the filter support over the O-ring of the extractor base.
8	Dry the filter paper in an oven at a maximum temperature of 138°C (280°F).
9	Weigh and record the weight of a clean, dry, filter paper to the nearest 0.1 g.
10	Place the filter paper on the filter support, replace the funnel ring, and tighten the wing nuts finger tight. Inspect the filter paper and filter support for improper alignment and tears. *NOTE:* Reduce clogging of the filter paper by using more than 1 filter or by placing a pair of No. 1.18 mm (No. 16) and 75 µm (No. 200) sieves, nested together, onto the filter paper.
11	Place approximately 30 g of diatomaceous filtering aid, weighed to the nearest estimated 0.1 g, into a beaker and add about 500 mL of solvent. ♦ Mixtures containing rubber or polymer additions may require up to 100 g to adequately assist with filtering. ♦ The filtering aid should be dried and maintained in a moisture-free environment. It should consist of material passing the 75 µm (No. 200) sieve only. *NOTE:* If particles coarser than the 75 µm (No. 200) sieve are found, the technician must sieve all of the material to be used over the 75 µm (No. 200) sieve, discarding the coarser particles.
12	Record the weight of filtering aid used.
13	♦ Vigorously stir the contents of the beaker and immediately pour the solution onto the filter

Determining Asphalt Content of Bituminous Mixtures by Vacuum Extraction Method Using Chlorinated Solvent	
Step	**Action**
	paper. ♦ Rinse any filter aid remaining in the beaker onto the filter paper.
14	♦ Start the vacuum pump and continue to run the pump until the pad formed by the filtering aid is surface dry. ♦ Eliminate use of the diatomaceous filtering aid when the filter paper being used allows efficient extraction of dissolved asphalt without loss of very fine aggregate. *NOTE:* Some diatomaceous filter aid is very fine and washed through the filter paper. This material will be included in the determination of the percent passing of the 75 µm (No. 200) sieve and the gradation may not meet specification. Perform blank tests to determine the amount of filter aid lost through the filter paper if the gradation does not meet specification. Subtract this correction factor from the percent passing the 75 µm (No. 200) sieve.
15	♦ After gently stirring the test sample in the container or has undergone several minutes of ultrasonic cleaning, gently decant the solvent from the container or insert tray into the vacuum extractor. ♦ Decant the solvent onto a trowel placed on the filter paper to avoid undue disturbance of the filtering aid pad. Use a trowel to prevent aggregate from falling from the bowl or insert tray during the decanting process.
16	Again cover the sample in the container or insert tray with solvent and either gently stir or place into and activate the ultrasonic cleaner.
17	♦ Continue the vacuum until all the solution has disappeared from the filter paper. ♦ If the time to filter out the solvent is considerable or a hard crust appears, gently pull a comb across the filter paper while the pump is running, to break the crust, being careful not to tear the filter paper. ♦ Use additional clean solvent to help rinse the excess asphalt through the filter paper (Steps 18-20).
18	Turn the pump off and add approximately 300 mL of clean solvent into the vacuum extractor when the time to filter out the solvent is considerable and after gently combing across the filter paper.
19	Gently stir the solution with the comb for several minutes.
20	Turn the pump on and decant the solvent into the vacuum extractor.
21	Repeat Steps 15-20 until the solvent is approximately the same color as clean solvent and the aggregate is visually clean.
22	After the last washing, pour the entire sample into the extractor.
23	♦ Use a wash bottle with solvent to rinse the aggregate particles from the bowl or insert tray and spoon into the extractor. ♦ Also rinse the inside of the funnel ring to wash down any asphalt or aggregate fines which may have splattered and are adhering to the funnel sides.
24	With a spatula, carefully distribute the aggregate evenly over the filter paper.
25	After vacuuming all the liquid off, stop the vacuum pump and use a spatula to move the sample away from the funnel ring and toward the center of the filter paper. The vacuum pump may run for a period of time to further aid in drying.
26	Weigh and record the weight of a drying pan to the nearest 0.1 g.
27	Remove the funnel ring and brush any clinging aggregate into a tared drying pan.
28	Pick up the filter paper and the aggregate on the filter support plate and transfer the aggregate into the drying pan.
29	Using a spatula and brush, remove the clinging aggregate from the filter paper into the drying pan.

Determining Asphalt Content of Bituminous Mixtures by Vacuum Extraction Method Using Chlorinated Solvent	
Step	**Action**
30	Fold and place the filter paper in the drying pan with the sample.
31	♦ Place the pan containing the extracted aggregates in a vented oven and dry to constant weight at a maximum temperature of 138°C (280°F). ♦ Stagger placement of samples into the oven by at least 1 hr. when extracting multiple samples. ♦ Place the most recently extracted sample closest to the exhaust vent. Do not block the exhaust vent with a sample pan.
31	Dry the extracted aggregate and filter paper to constant weights, then weigh and record the individual weights to the nearest 0.1 g.

Retention Factor

Retention Factor is identical to that described in 'Part I, Centrifuge Extraction Method Using Chlorinated Solvent.'

Calculations

Follow the steps indicated on the 'Test Data' worksheet (Tx2MxProp) to calculate the percentage of asphalt in the mixture.

Section 4

Part III, Centrifuge Extraction Method Using Non-chlorinated Solvent

SPECIAL NOTICE: The solvents used in these procedures are toxic, flammable and or combustible. Follow the safety precautions herein.

Apparatus

Use the following apparatus:

- centrifugal extractor, motorized, with explosion-proof features, consisting of a bowl mounted in an assembly so that the bowl revolves at a controlled speed. (The apparatus should have a container or outer shell for catching the solvent thrown from the bowl and a drain for collecting the solvent.)
- ventilation, as required by current specifications for asphalt mix control laboratories. (Specific requirements contained herein for the arrangement and capacity of the ventilation system may be waived or varied if tests show that the alternate system lowers solvent vapors to safe levels throughout the testing process.)
- filter paper to fit the rim of the bowl having a 20-25 µm retention factor
- scoop, spatula, trowel and small brush
- graduates, 2000 mL capacity, minimum
- filtrate containers
- screw-top container, 4 L (1 gal.), of metal or polyethylene
- balance, readable to 0.1 g and accurate to 0.5 g
- balance, readable to 0.01g and accurate to 0.01g
- forced draft oven, vented to the outside and capable of attaining a temperature of 93°C (200°F) or more
- drying oven, capable of attaining a temperature of 93°C (200°F) or more, or a suitable microwave oven. (Vent the drying oven to the outside. The microwave oven must be located in a ventilated area.)
- large flat pan, solvent pouring container, wash bottles, beakers, etc.
- glass fiber filter paper, Whatman GF/F 12.5 cm (1/2 in.) glass micro fiber filters or equivalent
- Buchner filter funnel. Coors porcelain Buchner funnel, 126 mm (5 in.) internal diameter
- vacuum pump, with an exhaust hose to carry fumes to immediately in front of the exhaust fan of the ventilation system
- vacuum filtering flask, 1000 mL
- desiccator, (optional)

- sealed magnetic stirrer (optional)
- pipette, 100 mL capacity.

Materials

Extraction solvent, a biodegradable extraction solvent, meeting the requirements of the department's specification for nonchlorinated extraction solvent for bituminous mixtures.

Safety Considerations

Follow the safety considerations detailed in 'Safety Considerations (Chlorinated Solvents).'

Preparing Sample

Preparing the sample is identical to that described in 'Part I, Centrifuge Extraction Method Using Chlorinated Solvent.'

Procedure

Follow these steps to determine asphalt content of bituminous mixtures using non-chlorinated solvent.

Determining Asphalt Content of Bituminous Mixtures by Centrifuge Extraction Method Using Non-chlorinated Solvent	
Step	**Action**
1	♦ Weigh the laboratory size sample for extraction. (See Note 1.)
2	Transfer the weighed sample of asphaltic concrete into the extractor bowl, taking care not to lose any of the material.
3	♦ Cover the sample with solvent (see Note 2). ♦ Pour all solvent in the airflow of the ventilation system.
4	If necessary, use a small amount of solvent and wash any material remaining in the pan into the bowl.
5	Stir the mixture in the extractor bowl frequently over a period of approximately 10 min. Stirring every 2 to 4 min. is desirable.
6	Weigh a clean, dry, paper filter ring to the nearest 0.1 g.
7	If necessary, dry the filter ring in an oven at a maximum temperature of 138°C (280°F).
8	Place the filter paper and top on the extractor bowl and screw on the funnel-clamp until tight.
9	Place the bowl containing the sample and solvent into the machine, tighten the lock nut, and secure the extractor lid.
10	Place a container under the drain to catch the liquid extracted from the sample and start the machine.
11	When the solvent ceases to flow, stop the centrifuge and collect the discharged liquid in a 2000 mL graduated cylinder.
12	Add approximately 300 mL of solvent to the bowl through the funnel, and start the centrifuge.
13	Collect the discharged liquid, stop the centrifuge, pour the discharged liquid back into bowl and start the centrifuge again.
14	Stop the centrifuge and add the discharged liquid to that liquid already in the graduate.

Determining Asphalt Content of Bituminous Mixtures by Centrifuge Extraction Method Using Non-chlorinated Solvent	
Step	**Action**
15	♦ Continue this procedure, centrifuging each addition of solvent several times until the extract is reasonably clear and approximately the same color as clean solvent. ♦ Stop the motor every time before pouring solvent into the funnel. NOTE: Due to the nature of the extraction process, it is impossible to secure a perfectly clear extract without disintegrating some of the aggregate or using an excessive quantity of solvent.
16	Collect and measure the total amount of discharged liquid.
17	Add about 300 mL of warm water (preferably above 43°C [110°F]) to the extractor.
18	Start the extractor and collect the filtrate. Discard the filtrate in a manner acceptable to safety and environmental standards.
19	Repeat Steps 17 and 18 until the rinse water is clear.
20	Operate the centrifuge for several minutes to remove as much rinse material from the mix as possible.
21	Carefully remove the top and filter paper from extractor bowl and brush all clinging aggregate particles back into the bowl.
22	Dry the filter paper to constant weight (See Note 1).
23	Subtract the original weight of filter paper (Step 3) from the weight measured in Step 22 to obtain the weight of the fine mineral matter (assumed to be particles passing the 75 µm [No. 200] sieve) contained in it.
24	Transfer the aggregate from the extraction bowl to a tared pan.
25	♦ Place the pan containing the extracted aggregates in a vented oven and dry to a constant weight at a maximum temperature of 138°C (280°F). ♦ If another extraction sample is already in the oven, stagger placement of the samples into the oven by at least one hr. ♦ Place the most recently extracted sample closest to the exhaust vent. Do not block the exhaust vent with a sample pan.
26	Remove from oven and cool.
27	Place a clean, dry, glass fiber filter paper in the Buchner funnel and weigh to the nearest 0.01 g.
28	Dry the fiber filter paper in an oven at a maximum temperature of 138°C (280°F), if necessary.
29	♦ Place the Buchner funnel, containing the glass fiber filter paper, in the vacuum filter flask. ♦ Wet the glass fiber filter paper using clean asphalt extraction solvent from a wash bottle and start the vacuum.
30	Pour the solvent-asphalt mixture (from Step 16) into a 4 L (1 gal.) screw-top container.
31	♦ Agitate thoroughly and immediately pour 100 mL into a 100 mL graduate. An optional method of agitation is to place a magnetic stirring bar in the graduate or beaker containing the solvent and then place this on a sealed magnetic stirrer. ♦ If a sealed magnetic stirrer is used, draw a 100 mL sample from the middle third of the liquid when stirring, by means of a 100 mL pipette and suction bulb.
32	Pour the 100 mL sample into the Buchner funnel and filter out the mineral matter (dust).
33	Repeat Steps 31 and 32 to filter a total of 200 mL of the solvent-asphalt mixture.
34	Rinse the graduated cylinder with clean asphalt extraction solvent. *NOTE:* This insures that all mineral matter washes into the filter.
35	♦ Rinse the sides of the funnel washing all material onto the glass fiber filter paper. ♦ Rinse the glass fiber filter paper with solvent 5 times (just covering the surface of the glass fiber filter paper), allowing the glass fiber filter paper to drain completely between rinses.
36	Continue the vacuum until the glass fiber filter paper and contents appear dry.

Determining Asphalt Content of Bituminous Mixtures by Centrifuge Extraction Method Using Non-chlorinated Solvent	
Step	**Action**
37	Rinse the sides of the funnel with a wash bottle filled with the warm water (preferably above 43°C [110°F]) and cover the glass fiber filter paper with it. It is not necessary to fill the funnel with the water.
38	Allow the glass fiber filter paper to drain dry under the vacuum.
39	♦ Repeat Steps 37 and 38 seven times. ♦ Use only a small amount of rinse material for each rinse. Cover the surface of the glass fiber filter paper each time.
40	Dry the Buchner funnel, glass fiber filter paper, and retained fines in an oven at a maximum temperature of 138°C (280°F) to constant weight.
41	Weigh the Buchner funnel, glass fiber filter paper, and mineral matter to the nearest 0.01 g.

Retention Factor

The retention factor is identical to that described in 'Part I, Centrifuge Extraction Method Using Chlorinated Solvent.'

Ash Factor Alternative

The ash factor is an amount of Buchner fines used repeatedly as part of the total aggregate portion of an extraction test. It takes the place of the Buchner fines value usually determined for each extraction.

The ash factor may differ with each individual. Therefore, establish an ash factor for each technician, because of personal differences in laboratory technique.

- ♦ Run a minimum of 4 extraction tests during 4 different days of production.
- ♦ If the 'Total Buchner fines' values of the 4 extractions do not vary by more than 0.1 % by weight of the 'total sample' weight, the number of grams of 'Total Buchner fines' can become the ash factor as long as:
  - there is no change in the aggregate gradation or source
  - the asphalt content does not change
  - the plant production remains uniform.
- ♦ Periodic checking of the ash factor by Buchner fines determination is essential for justifying its continued use.
- ♦ Omit Buchner fines determination when using extraction for the sole purpose of cleaning aggregate for sieve analysis.

Section 5

Part IV, Vacuum Extraction Method Using Nonchlorinated Solvent

SPECIAL NOTICE: The solvents used in these procedures are flammable and or combustible. Follow the safety precautions herein.

Apparatus

Use the following apparatus:

- vacuum extractor complete with vacuum pump, extractor O-rings, tubing, filter support plate, tank and funnel ring. The department recommends a vane-type vacuum pump. An exhaust hose is required for the vacuum pump to carry fumes to immediately in front of the exhaust fan of the ventilation system.
- ventilation as required by current specifications for asphalt mix control laboratories. (Specific requirements contained herein for the arrangement and capacity of the ventilation system may be waived or varied if tests show that the alternate system lowers solvent vapors to safe levels throughout the testing process.)
- filter paper, size 331 mm (13 in.) having a 20-25 µm retention factor
- forced draft oven, vented to the outside and capable of attaining a temperature of 93°C (200°F) or more
- balance, readable to 0.1 g and accurate to 0.5 g
- small scoop or spoon, spatula, trowel and small brush
- large flat drying pan or microwave container
- plastic wash bottles and glass beaker graduates
- container of adequate size to hold test sample and solvent without spilling during stirring
- comb, hair-pick type, with long stiff tines which are approximately 3.8 to 6.4 mm (0.125 to 0.25 in.) apart. (The comb can be metal or plastic, provided the tine ends are smooth and rounded.)

Materials

Use the following materials:

- biodegradable extraction solvent meeting the requirements of the department's specification for nonchlorinated bituminous mixtures extraction solvent
- diatomaceous silica filtering aid (Celite 110 or equivalent)(optional).

Safety Considerations

Follow the safety considerations detailed in 'Safety Considerations (Chlorinated Solvents).' to safely perform this procedure.

Preparing Sample

Preparing the sample is identical to that described in 'Part I, Centrifuge Extraction Method Using Chlorinated Solvent.'

Procedure

Follow these steps to determine asphalt content of bituminous mixtures by using the vacuum extraction non-chlorinated solvent.

Determining Asphalt Content of Bituminous Mixtures by Vacuum Extraction Method Using Non-chlorinated Solvent	
Step	**Action**
1	Weigh and record the laboratory size sample for extraction to the nearest 0.1 g.
2	♦ Transfer the weighed sample into a sample container. Carefully cover the sample with solvent (see NOTE 2). ♦ Pour all solvents immediately in the airflow of the ventilation system.
3	♦ Gently stir the solvent and mixture sample in the container until visually noticing the asphalt in the solution. ♦ Soak for approximately 10 min.
4	Center the filter support over the O-ring of the extractor base.
5	Dry the clean filter paper in an oven at a maximum temperature of 138°C (280°F).
6	Weigh and record the weight of a clean, dry filter paper to the nearest 0.1 g.
7	♦ Place the filter paper on the filter support, replace the funnel ring, and tighten the wing nuts finger tight. ♦ Inspect the filter paper and filter support for improper alignment and tears. NOTE: Reduce clogging of the filter paper by using more than one filter or by placing a pair of No. 1.18 mm (No. 16) and 75 µm (No. 200) sieves, nested together, onto the filter paper.
8	Place approximately 30 g of diatomaceous filtering aid, weighed to the nearest estimated 0.1 g, into a beaker and add about 500 mL of solvent. ♦ Mixtures containing rubber or polymer additions may require up to 100 g to adequately assist with filtering. ♦ The filtering aid should be dried and maintained in a moisture-free environment. It should consist of material passing the 75 µm (No. 200) sieve only. NOTE: Sieve the filtering aid over the 75 µm (No. 200) sieve and discard the coarser particles coarser than the 75 µm (No. 200) sieve.
9	Record the weight of filtering aid used.
10	♦ Vigorously stir the contents of the beaker and immediately pour the solution onto the filter paper. ♦ Rinse any filter aid remaining in the beaker onto the filter paper.
11	♦ Start the vacuum pump and continue to run the pump until the pad formed by the filtering aid is surface dry.

Determining Asphalt Content of Bituminous Mixtures by Vacuum Extraction Method Using Non-chlorinated Solvent	
Step	**Action**
	♦ Eliminate use of the diatomaceous filtering aid when using filter paper that allows efficient extraction of dissolved asphalt without loss of very fine aggregate. *NOTE:* Some diatomaceous filter aid is very fine and washed through the filter paper. This material will be included in the determination of the percent passing of the 75 µm (No. 200) sieve and the gradation may not meet specification. Perform blank tests to determine the amount of filter aid lost through the filter paper if the gradation does not meet the specification. Subtract this correction factor from the percent passing the 75 µm (No. 200) sieve.
12	Gently stir the test sample in the container, decant the solvent from the container onto the filter paper in the vacuum extractor (or, if used, pair of nested sieves described in Step 7).
13	♦ After gently stirring the test sample in the container or has undergone several minutes of ultrasonic cleaning, gently decant the solvent from the container or insert tray into the vacuum extractor. ♦ Decant the solvent onto a trowel placed on the filter paper to avoid undue disturbance of the filtering aid pad. ♦ Use a trowel to prevent aggregate from falling from the bowl during the decanting process.
14	Again cover the sample in the container tray with solvent and gently stir.
15	♦ Continue the vacuum until all the solution has disappeared from the filter paper. ♦ If the time to filter out the solvent is considerable or a hard crust appears, gently pull a comb across the filter paper while the pump is running, to break the crust, being careful not to tear the filter paper. ♦ Add clean solvent to help rinse the excess asphalt through the filter paper.
16	Turn the pump off and add approximately 300 mL of clean solvent into the vacuum extractor when the time to filter out the solvent is considerable and after gently combing across the filter paper.
17	Gently stir the solution with the comb for several minutes.
18	Turn the pump on and decant the solvent into the extractor.
19	Repeat Steps 12 through 18 until the solvent returns to its original color and the aggregate is visually clean.
20	Add 300 mL of warm water (preferably above 43°C [110°F]) to the mixture in the bowl and stir with a trowel or spoon.
21	After stirring 1 or 2 min., start the vacuum pump and decant this solvent rinse material mixture into the vacuum extractor until there is no standing liquid on the filter paper's surface.
22	Repeat Steps 20 and 21 until the rinse water is clear.
23	♦ After the rinse, pour the entire sample into the extractor. ♦ Use a wash bottle filled with water to rinse all particles from the bowl into the extractor and rinse the inside of the funnel ring to wash down all clinging particles.
24	Using a spatula, distribute the aggregate evenly over the surface of the filter cake.
25	Continue operating the vacuum pump for at least 5 min. and remove most of the rinse fluid from the aggregate.
26	Weigh the drying pan to the nearest 0.1 g.
27	Remove the funnel ring and brush any clinging aggregate into the drying pan.
28	Pick up the filter paper and aggregate on the filter support plate, and transfer the aggregate into the drying pan.
29	Using a spatula, remove most of the clinging aggregate and filter aid from the filter paper into the drying pan, and fold the filter paper in the pan to aid in drying it.
30	♦ Place the pan containing the extracted aggregates in a vented oven and dry to constant weight at a maximum temperature of 138°C (280°F).

Determining Asphalt Content of Bituminous Mixtures by Vacuum Extraction Method Using Non-chlorinated Solvent	
Step	**Action**
	♦ Stagger placement of samples into the oven by at least 1 hr. when extracting multiple samples. ♦ Place the most recently extracted sample closest to the exhaust vent. Do not block the exhaust vent with a sample pan.
31	Weigh the extracted aggregate and filter paper individually to the nearest 0.1 g.

Retention Factor

Retention factor is identical to that described in 'Part I, Centrifuge Extraction Method Using Chlorinated Solvent.'

Calculations

Calculations are identical to those described in 'Part II, Vacuum Extraction Method Using Chlorinated Solvent.'

Section 6

Sample Size

The minimum sample size will be determined by the following table.

Sample Size		
Nominal Maximum Aggregate Size[1]		**Minimum Size of Sample**
mm	**in.**	**g**
4.75	No. 4	500
9.5	3/8	1000
12.5	1/2	1500
19.0	3/4	2000
25.0	1	3000
37.5	1-1/2	4000

[1]The definition of nominal maximum aggregate size is the largest sieve that retains 10% or more of the total aggregate mixture.

Section 7
Safety Considerations

Chlorinated Solvents

Refer to the Material Safety Data Sheet for trichlorethylene and methylene chloride.

Nonchlorinated Solvents

Do not exceed bulk storage inside the laboratory of 19 L (5 gal.) fresh solvent, plus up to 19 L (5 gal.) of used solvent.

Keep stored solvent in closed containers and away from any oven or other heating device.

The storage location must drain away from any known source of ignition.

Have available means to clean solvent spills.

Equip any room where solvents are stored and used with a portable fire extinguisher for burning liquids.

Use a vent fan with at least a 30 m^3/min. (1,000 CFM) rating which adequately removes fumes. The 1,000 CFM rating is the minimum rating and may not be adequate for all laboratories. If needed, use a higher capacity fan to adequately remove all fumes. Pour all solvents even with or below the height of the fan outlet. It is preferable to locate the fans beneath a workbench constructed of expanded metal top and enclosed front and sides. Pull the exhaust through the front opening of the workbench by installing a sliding panel. This design accommodates both centrifuge and vacuum extraction equipment while providing for exhausting fumes from floor spills. The fan must have an explosion proof (sealed) motor or do not draw the solvent vapors where the motor is located. Wire the vent fan and extraction equipment to a single switch so the vent fan will always operate during extraction. Include a means for cutting power to the extraction equipment while the ventilation fan is operating.

Do not place ordinary electrical equipment, including switches, in the immediate hazardous area. Keep this room in negative pressure with respect to the rest of the room by a vent fan. The immediate hazardous area is the space both above and below the centrifuge extractor and the airflow space between the centrifuge and the outside exhaust.

Do not permit sources of open flame (pilot lights, smoking, Bunsen burners, etc.) in the room where solvents are stored or handled.

Section 8

Notes

1. Refer to "Tex-212-F, Determining Moisture Content of Bituminous Mixtures," 'Part II, Procedure' for drying to a constant weight.
2. Exercise caution during the initial addition of solvent to the hot extraction sample since the mix will shortly heat the solvent to temperatures above the solvent flash point and could present a flammability hazard. With stirring, the temperature should cool below the flash point within 2 to 5 min.
3. If the sample is dry when extracted, moisture and hydrocarbon volatiles = 0.
4. Remove solvent from the vacuum tank at least once per day so that solvent attack on the tank is limited.
5. Correction for ash contained in the solvent is generally unnecessary with the vacuum extraction method.
6. When decanting liquids from the container, pour off as much liquid as possible first, then rap the container with a spoon or trowel to settle the contents. More liquid may then be decanted.
7. Add the weight of fine material retained in the filter ring and the weight of ash in the total centrifuged liquid to the weight of material passing the 75 µm (No. 200) sieve for use in calculating the sieve analysis.

Section 9

Archived Versions

The following archived versions of "Tex-210-F, Determining Asphalt Content of Bituminous Mixtures by Extraction" are available:

- 210-0899 for the test procedure effective August 1999 through November 2004.