

Standard Method of Test for

Extraction and Recovery of Asphalt Binder from Asphalt Mixtures by Means of Centrifuge and Rotavapor Apparatus

SCDOT Designation: SC-T-95 (04/2022)

1. SCOPE

- 1.1 This method covers the extraction and recovery of asphalt binder from asphalt mixtures in order to perform various AASHTO asphalt binder tests.

Safety Notice: Not only does this procedure involve temperatures in the range of 100 - 163°C, it also includes handling asphalt binder in this temperature range, as well as toxic solvents.

The use of trichloroethylene to extract the asphalt binder should only be used under a fume hood or with an effective surface exhaust system in a well-ventilated area. This solvent is toxic to some degree as described in 29 CFR 1910.1200. All local, state, and federal regulations must be followed when hauling, using, storing, and discarding the extractant and rinse water. The Safety Data Sheet (SDS) should be followed closely.

This procedure will require the technician to wear appropriate safety protection (gloves rated for the solvent being used and/or the temperature, eye protection, long sleeves and pants, and closed toed shoes). The absence of a warning does not necessarily mean that all material and equipment are safe to handle. The technician should use caution during every step of this procedure.

2. REFERENCED DOCUMENTS

- 2.1 AASHTO T 164, AASHTO T 315
- 2.2 SC-T-62, SC-T-72, SC-T-75, SC-T-79, SC-T-93, SCDOT Form MD419
- 2.3 Fed. Std. No. 29, CFR 1910.1200 OSHA Hazard Communication; see also Permissible Exposure Limits – Annotated Tables, available at <https://www.osha.gov/dsg/annotated-pels/>

3. SUMMARY OF TEST METHOD

- 3.1 The asphalt binder is extracted from the asphalt mix using trichloroethylene and a centrifuge extractor. Then, the process of recovering the extracted asphalt binder begins by distilling the asphalt binder from the trichloroethylene using a rotary evaporator (rotavapor). Once the solution is concentrated to approximately 250 mL, the concentrate is centrifuged to separate out the aggregate fines. The decanted solution is distilled once more to remove all traces of the solvent from the asphalt binder. The recovered asphalt binder is now ready for further physical and chemical testing.

4. SIGNIFICANCE AND USE

- 4.1 This method is used to obtain recovered asphalt binder residue from asphalt mixture samples so further testing can be performed on the asphalt binder.

Note: Asphalt roadway cores can also be tested using this procedure for investigative purposes.

5. STANDARDIZATION

- 5.1 At minimum annually, verify the calibration of the oil bath temperature by using a certified thermometer of suitable range that is accurate to 0.2°C. Record and make note of any correction factor that is required for the oil bath.
- 5.2 At minimum annually, verify the pressure of the rotavapor by using a manometer or other certified pressure measurement device to verify the calibration of the vacuum indicator at the pressures specified in this procedure.

6. APPARATUS

- 6.1 *Centrifuge Extractor* – consisting of a bowl and an apparatus in which the bowl may be revolved at controlled variable speeds up to 3600 rpm. The speed may be controlled manually or with a preset control. The apparatus should be provided with a container for catching the discharged solvent from the bowl and a drain for removing the solvent. The apparatus preferably shall be provided with explosion-proof features and installed in a hood or an effective surface exhaust system to provide ventilation.
- 6.2 *Oven* – capable of maintaining 125 +/- 5°C.
- 6.3 *Filter Rings* – felt or paper, to fit the rim of the centrifuge extraction bowl.
- 6.4 *Rotary Evaporator (Rotavapor)* - complete with a vacuum system capable of reducing the ambient pressure to below 30 mm of Hg (Torr), a digital manometer and vacuum controller that maintains consistent vacuum, a condenser, and an oil bath capable of maintaining temperatures of up to 180°C. The rotavapor apparatus must also include a cooling water source which is to be circulated through the condenser, be capable of rotating the evaporating flask with speeds up to 100 rpm, and have a 2000 mL glass Erlenmeyer flask to store the extracted asphalt binder solution connected to an in-line valve to assist in the slow transfer of solution to the evaporating flask. A round bottom receiving flask must be securely attached to the condenser to catch the solvent being evaporated.
- 6.5 *Centrifuge* – batch unit capable of exerting a minimum relative centrifugal force of 770 times gravity.

Note: Relative centrifugal force (RCF) can be converted to revolutions per minute (RPM) by utilizing the following equation:

$$g = (1.118 \times 10^{-5})RS^2; \text{ where}$$

g = relative centrifugal force,
 R = radius of the rotor in centimeters (cm),
 S = speed of the centrifuge in RPM

- 6.6 Glassware – Other than what is described in 6.4 above, a *50 mL glass beaker* will be used to collect the recovered asphalt binder at the end of each distillation.
- 6.7 *250 mL Wide-Mouth Teflon Bottles* – to centrifuge the aggregate fines from the asphalt binder solution.
- 6.8 *Trichloroethylene, Reagent Grade* – to extract the asphalt binder and allow multiple washes of the glassware and apparatus being used.
- 6.9 *Miscellaneous Equipment - Metal bowls or pans* to hold the HMA after quartering and for transferring the mix from the oven to the centrifuge. *Spatula* to scrape the sides of the extraction bowl once asphalt has been extracted. *Funnel* to transfer the asphalt binder solution from one container to another. *Cloth Rags* for cleaning and preventing oil bath residue from contaminating the asphalt binder being recovered. *Squirt Bottle* full of trichloroethylene for rinsing, cleaning flasks, etc.



Figure 1: Rotavapor



Figure 2: Centrifuge extractor



Figure 3: Inside the centrifuge extractor, depicting the bowl unit with lid and nut

7. TEST SPECIMEN

- 7.1 Obtain the initial bagged sample through the proper sampling techniques in SC-T-62. If overnight silo storage samples are the basis of testing, collect the samples using SC-T-79. Using SC-T-72 or SC-T-93, quarter the hot mix sample down to testing size, as specified in Table 1 below, for the extraction sample.

Test Specimen	Suggested Mass of Sample (g)
Surface Mix	1000
Intermediate Mix	1200

Table 1: Suggested size of asphalt mixture to test for obtaining a suitable amount of recovered asphalt binder to test for stiffness.

- 7.2 Place the quartered sample in a metal pan or bowl and place in a 125 +/- 5°C oven for not more than 1 hour to keep the sample malleable before the asphalt binder extraction begins.

Note: All specimens should be subjected to identical preparation and heating times throughout the entirety of this procedure.

8. PROCEDURE

- 8.1 If asphalt binder content is to be determined, follow SC-T-75. Use the nominal maximum aggregate size from the Job Mix Formula (JMF) to quarter this sample to the appropriate testing size.
- 8.2 Power on the centrifuge extractor, rotavapor, water cooler, and centrifuge. Pre-heat the rotavapor oil bath to 100 +/- 2.5°C.
- 8.3 Ensure all equipment, tubing, funnels, and glassware are clean and there is no residue of any sort that could cause contamination to the asphalt binder being extracted for testing.
- 8.4 Place the warmed, quartered sample into the clean extractor bowl. Ensure the bowl is attached properly to the centrifuge extractor apparatus. Add enough trichloroethylene (TCE) solvent to cover the HMA sample. Using a spatula, break up any pieces of asphalt mix that may be stuck together. Level out the asphalt mixture to prevent the centrifuge from becoming off balance during the spin cycle. Fit a filter ring to the edge of the bowl, and hand tighten to secure the top using the nut. Ensure the bowl and top are aligned and seated properly. Tightly clamp down the outer lid.

Note: If necessary, the TCE covered asphalt mix sample may be allowed to stand for not more than 1 hour in order for the solvent to dissolve the asphalt.

- 8.5 Ensure that a 2000 mL Erlenmeyer flask is in place to catch the extracted asphalt binder-TCE solution coming from the drain port. Ensure the bowl will be allowed to rotate freely without interference of the braking system.

- 8.6 Start the centrifuge extractor, and gradually increase the speed to a maximum of 3600 rpm until the solvent ceases to flow from the drain into the 2000 mL flask. Slowly bring the centrifuge to a stop using the brake. Add 200 mL of TCE to the asphalt mix to the solvent pour port, and repeat the extraction process until the discharged solvent is not darker than a light straw color when viewed against a white background. At minimum, three solvent additions must occur.
- 8.7 Remove both lids, discard the aggregate and filter ring, and rinse all the asphalt binder residue that remains inside the centrifuge extractor and lids into the 2000 mL Erlenmeyer flask containing the extraction liquid. Remove and clean the bowl holding the aggregate. Place a cork in the top of the flask until the extracted solvent is ready for the rotavapor.

Note: If quantitative extraction of the asphalt binder is required to obtain the aggregate for sieve analysis, refer to AASHTO T 164.

- 8.8 Clamp the receiving flask to the condenser and securely attach the evaporating flask onto the rotavapor apparatus. Using the height adjustment handle, lower the rotational evaporating flask until approximately half of the flask is submerged in the hot oil bath. Begin rotation of the evaporating flask at a rate of 30 - 40 rpm.
- 8.9 Begin cold-water (< 25°C) flow through the rotavapor condenser at a rapid flow rate.
- 8.10 Set vacuum on the vacuum controller to 400 mm of Hg. Begin vacuum process, and allow the vacuum to reach set point.
- 8.11 Gradually adjust the fluid transfer valve to begin the addition of the asphalt-TCE solution from the 2000 mL Erlenmeyer flask to the rotational evaporating flask at a rate of 20 - 40 mL/min. Transfer the solution very slowly to ensure that no foaming or back pressure builds inside the flask and condenser. Transfer the solution so that no more than half of the evaporating flask is full. Adjust the fluid transfer valve to stop the flow of solution into the evaporating flask. Continue to add the asphalt-TCE solution to the flask, and distillate until there is no solution remaining in the 2000 mL Erlenmeyer flask. Ensure the 2000 mL Erlenmeyer flask contains no asphalt residue by rinsing and transferring the wash solution into the evaporating flask. After all solution is removed from the flask, close the in-line valve, and continue to distill until approximately 150 - 200 mL of solution remains in the rotational evaporating flask.

Note: If the receiving flask becomes full of solvent and should need to be emptied, release the vacuum pressure, and allow the pressure to reach ambient pressure before attempting to remove the flask. Attach the empty flask, and resume set vacuum point. Save this solvent for subsequent washes, rinsing, and extracting.

- 8.12 Stop the rotation on the rotavapor, and raise the evaporating flask from the oil bath allowing some of the oil to drip back into the bath.
- 8.13 Remove the evaporating flask from the rotavapor, and wipe any excess oil from the outside of the flask. Ensure no oil residue gets into the extracted asphalt binder solution.
- 8.14 Increase the oil bath temperature to 163 +/- 5°C.

- 8.15 Transfer the concentrated asphalt binder solution out of the flask, using a funnel, and into a 250 mL Teflon bottle. Ensure all asphalt solution residue from the flask is rinsed into the Teflon bottle using trichloroethylene. Fill each bottle so their masses are equal. Include the Teflon bottle lids when weighing.
- 8.16 Place the asphalt solution filled Teflon bottles in the centrifuge and counter-balance any empty spaces in the centrifuge with Teflon bottles full of trichloroethylene of equal weight to level the load. Centrifuge the samples at a relative centrifugal force equal to 770 times gravity for 30 minutes.
- 8.17 Once the centrifuge comes to a complete stop, remove the Teflon bottles. Do not disturb the aggregate fines sediment at the bottom of the Teflon bottles. Transfer the extracted asphalt solution from the Teflon bottle to a clean rotational evaporating flask. Ensure no aggregate fines are introduced to the flask during the solution transfer. Clean the Teflon bottle with trichloroethylene, and dispose of the waste wash appropriately.
- 8.18 Place the rotational evaporating flask onto the rotavapor, and lower into the preheated, 163 +/- 5°C, oil bath so approximately half of the flask is submerged. Begin rotation at 60 - 80 rev/min. Ensure cold water flow is maintained through the condenser coils, and set the vacuum to 400 mm of Hg.
- 8.19 Begin vacuum and continue distillation until 1 - 2 drops of TCE per minute are witnessed falling into the receiving flask. The vacuum must then be increased to 200 mm of Hg and distillation continued until 1 - 2 drops of TCE per minute are witnessed falling into the receiving flask. Increase vacuum to 75 mm of Hg and continue the final distillation for 15 minutes.
- 8.20 Discontinue the distillation process by increasing the pressure (decreasing vacuum) to ambient pressure and stopping rotation of the evaporating flask. Lift the evaporating flask from the hot oil. Remove the evaporating flask, and wipe all oil residue from the flask, ensuring no oil residue is introduced to the asphalt binder. Immediately pour the recovered asphalt binder into a 50 mL beaker. Once all samples have been distilled, cut off the water supply and power down all instrumentation.
- 8.21 Use the recovered asphalt binder immediately, or reheat the sample in a 135 - 163°C oven until the sample is flowable. Do not overheat. Stir the sample to a homogenous state prior to preparing recovered binder samples for further testing.
- 8.22 The recovered asphalt binder can be tested using an array of AASHTO and SC Test procedures, including but not limited to AASHTO T 315.

9. CALCULATIONS

- 9.1 None

10. REPORT

- 10.1 Results are reported on various asphalt binder reports. Overnight Storage for Silos are reported on MD419.