Pycnometers, Calibrations, and Insulated Containers: A Guide to Understanding ASTM D 854, Soil Specific Gravity

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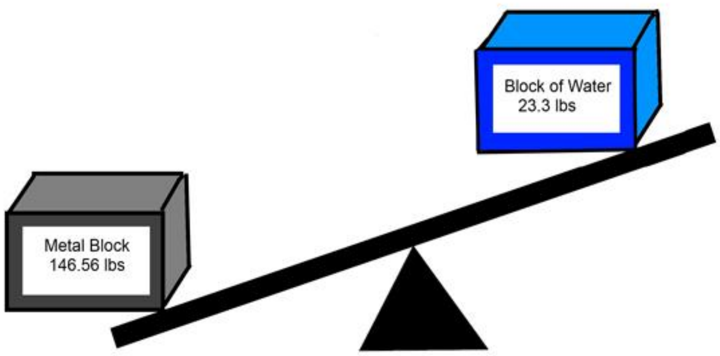
When planning an assessment for a laboratory that will be running ASTM D 854, many thoughts race through my mind. For example, does the technician know that there are significant differences between AASHTO T 100 and ASTM D 854? Have the calibrations been performed correctly? Or, is all of the right equipment available? ASTM D 854 can be a difficult test, and many people are not familiar with the intricacies involved with running it. In this article I will talk through the portions of the test that seem to cause the most confusion, explain reasons behind certain steps, and also offer helpful tips and suggestions.

Importance

Specific gravity is the ratio of the mass of a certain volume of a material to the mass of the same volume of water. This may be difficult to visualize, but think of it this way: Let's say that a block of metal has a specific gravity of 6.29. This means that the metal block weighs 6.29 times the mass of a block of water that is the same volume.



146.56 lbs ÷ 23.3 lbs = 6.29

Soil specific gravity is used to define the phase relationships of soil, define compactibility and density, and in conversions and calculations for other test procedures. With this number influencing others, it is important to be as accurate as possible when determining this soil property.

Apples and Oranges

I have seen more things go wrong than right with this test in my time as a laboratory assessor. Many times the laboratory signs up for both methods (AASHTO and ASTM) assuming that, like most other tests, the two procedures are nearly identical. If you fall into this category, let me be the first to tell you that the differences between AASHTO T 100 and ASTM D 854 are quite significant. The table to the right provides a list of some of the differences between the two methods.

Requirements	T 100 AASHTO	D 854 ASTM
Calibration of Pycnometer	Pycnometer calibrated for the range of temperatures likely to be encountered in the testing environment.	Pycnometer calibrated 5 times and averaged. The Standard deviation of the 5 trials must be less than 0.06 grams. Thermal equilibrium is achieved using insulated the container.
Before Beginning The Test	No verification procedure before running test.	Mass of pycnometer verified that it is within 0.06 grams of the average calibrated mass.
De-airing of Soil Slurry	De-air the soil slurry by either boiling or by vacuuming (agitating periodically) for at least 10 minutes. See Table 8 in AASHTO T 100 for more information on time requirements for different types of soils.	De-air the soil slurry by either boiling for 2 hours or by boiling under vacuum for one hour.
Achieving Final Pycnometer Volume	Once entrapped air is removed, distilled water is added to the calibration line.	Once entrapped air is removed, de-aired water is added to, or close to, the calibration line using small-diameter tubing kept under the surface of the soil slurry during the filling process.
Thermal Equilibrium	Pycnometer and contents are cooled to room temperature after removal of entrapped air.	Pycnometer placed in insulated container to achieve thermal equilibrium after removal of entrapped air.
Pycnometer	Volumetric flask with a minimum capacity of 100 mL or stoppered bottled with a minimum capacity of 50 mL.	Minimum capacity of 250 mL.
Thermometer	Thermometer graduated to 0.5°C (1.0°F).	Thermometer graduated to 0.1°C.

Equipment Requirements

As well as some procedural differences, ASTM D 854 also requires additional equipment that some laboratories do not have readily on hand. For example, the ASTM method requires the following:

- An insulated container that is large enough to fit between 3 and 6 pycnometers (a portable Styrofoam or plastic cooler works well for this purpose)
- Deaired water
- A beaker to store the deaired water
- A funnel with a stem that extends past the calibration mark on the pycnometer
- An eyedropper or pipette
- A desiccator

When running this test, it is important to have all of the equipment listed above available for use. We all know how things have a tendency to grow legs and walk away in a laboratory, usually right before you need them. It is a good idea to store this equipment in the insulated container (except for the desiccator, of course).

Calibration of Pycnometer - Am I Doing This Right?
As with most equipment used during an on-site assessment, the AASHTO re:source assessor will check records to make sure that the calibration have been performed correctly. Calibration is a vital part of the test procedure. If you are using incorrectly calibrated equipment, you could perform the test perfectly and still come out with skewed results. More times than not, I find that calibration of the pycnometer is not performed according to the test method.

Since the calibrations are so time-consuming, the standard allows for up to six pycnometers to be calibrated at the same time. I suggest doing this to maximize efficiency. You will want each pycnometer to have an identifying mark or serial number so that you can match the calibrated volume to the correct pycnometer.

Repeat Measurements: Ensuring Accuracy
The method requires that the mass of the empty pycnometer and the calibrated volume to be determined five times. The average and standard deviation for the dry mass and calibrated volume of the pycnometer are determined, and the standard deviation must be below a certain level in order for the calibration to be considered satisfactory.

I know what you are thinking: "Five times! Are you kidding me?" Well, don't roll your eyes just yet. These steps, while they may seem excessive, are a critical requirement of the test method, and were put there for very good reason. Operator and equipment errors can cause discrepancies in the measurements we take. Repeating these measurements several times helps us to detect and eliminate discrepancies, allowing us to be even more confident in our test results.

One large contributor of error in determining the volume of the pycnometer is adjustment of the meniscus to the calibration line on the neck of the container. The position of the meniscus can appear to be different, based on the line of sight of the operator. Anyone who has run this test before knows that it takes quite a bit of practice and adjustment to get the meniscus to line up exactly with the calibration line each time. Repeat measurements of the calibrated volume ensure that this measurement is done in a consistent manner.

Thermal Equilibrium
Throughout the test procedure, there are many requirements and steps that are required to ensure thermal equilibrium of the pycnometer and its contents. As we know, the density of water varies with temperature, and has to be corrected in order to report accurate specific gravity measurements. It is not enough just to measure the temperature of water and correct the density. ASTM D 854 requires that certain steps be taken to ensure that the temperature of the pycnometer and its contents are homogenous each time a measurement is taken.

The water used for the test is kept in the insulated container to ensure that the temperature remains uniform. The pycnometer is also placed in the insulated container after filling it with water and before determining its mass. The pycnometer is allowed to sit in the insulated container for several hours. These waiting times allow the pycnometer, water, and sample to achieve thermal equilibrium before the mass is determined. Any pipettes, thermometers, or other tools that may be used during testing should also be kept in the insulated container.

Precautions should also be taken when handling the pycnometer to make certain that the temperature is not affected. The pycnometer should be touched only near the rim, and not on the main body of the container. Heat from the hands could change the temperature of the glass and the water inside the pycnometer. While not required, it is also a good idea to use powder-free gloves when handling the pycnometer to prevent oils from the fingers from adhering to the glass.

Finally, the pycnometer should never be placed directly on a laboratory bench top for filling and weighing. The temperature of the countertop could impart a temperature difference on the pycnometer. Instead, use a piece of foam or an insulated block underneath the pycnometer. Alternatively, if your setup allows for it, portions of the test procedure can be performed inside the insulated container.

Deairing, Agitation, Why bother?
The air entrapped in the soil and dissolved in the water can affect mass and volume of the soil-water slurry, resulting in an erroneously low specific gravity measurement. While the method calls for the use of deaired water to be used, air can still be introduced into the system when the soil and water are added to the glass. De-airing can be achieved one of three ways:

1) Boiling
The first option is to bring the contents of the pycnometer to a boil for at least 2 hours while occasionally agitating the pycnometer. The best way to boil the sample is to use a sand bath between the pycnometer and the heat source. The sand bath prevents the pycnometer from overheating, which could crack the pycnometer and redecorate your laboratory with soil-water slurry. Boiling should be kept as low as possible and monitored constantly to ensure that the soil and water do not boil over. It may be necessary to use a pycnometer larger than the 250 mL minimum so that all of the water in the sample is not lost to evaporation.

2) Applying a Vacuum
The pycnometer can be vacuumed for at least two hours along with continual agitation. An added benefit to this method is that since no heat is used, the pycnometer and its contents will achieve thermal equilibrium much faster than with the boiling technique.

3) Vacuuming and Boiling Simultaneously
The final method that may be employed to deair the pycnometer is placing it in a warm water bath not exceeding 40°C and applying a vacuum for one hour. Just keep in mind that the water level in the bath needs to be below the water level in the pycnometer.

Some people wonder, “Why bother agitating?” Well, the agitation during boiling prevents any soil from being dried to the pycnometer walls and forming air bubbles. Continual agitation during vacuuming keeps the slurry in a constant suspension and ensures the removal of all entrapped air. Whether boiling, vacuuming, or both, agitation of the sample during deairing is critical.

To avoid introducing air back into the pycnometer after the deairing process has been completed, a pycnometer filling tube can be used to add water to the calibration mark. The water level in the pycnometer should always be readjusted after thermal equilibrium is reached to account for any volume changes that may occur.

Finished! Not so bad was it?
ASTM D 854 can sometimes seem intimidating, especially during an assessment. However, with the right information and equipment, it is no more difficult than any other test. Hopefully this article has offered some useful tips for determining soil specific gravity in accordance with ASTM D 854. My biggest piece of advice for anyone performing this test method is to review the standard thoroughly, and create data sheets and internal procedures that will help to ensure that each of the steps is being performed correctly.

Editor’s Note: This article was updated in June 2016 to ensure the most accurate information is presented.