

NIST OWM – NET 22.2 Portable Digital Density Meter proposal submitted by NIST OWM for consideration at the 2022 NCWM Annual Meeting.

NET-22.2 –Section 3.1.1 Test Methods and 3.X. Gravimetric Audit Test Procedure for Viscous and Non-Viscous Liquids by Portable Digital Density Meter.

Preamble under the Item Under Consideration:

Amend NIST Handbook 133, Checking the Net Contents of Packaged Goods, to modify Note 2 in Section 3.1.1. Test Methods and Section 3.X. Gravimetric Test Procedure for Viscous and Non-Viscous Liquids by Portable Digital Density Meter. Add an audit test procedure for 3.X. Gravimetric Test Procedure for Viscous and Non-Viscous Liquids by Portable Density Meter as follows:

3.1. Scope

3.1.1. Test Methods

Notes:

- (2) When checking liquid products using a volumetric or gravimetric procedure for **density determination**, the temperature of the samples must be maintained at the reference temperature $\pm 2\text{ }^{\circ}\text{C}$ ($\pm 5\text{ }^{\circ}\text{F}$), **except when using Section 3.X. Gravimetric Audit Test Procedure for Viscous and Non-Viscous Liquids by Portable Digital Density Meter, where a correction factor is used to correct the density to the reference temperature.**

3.X. Gravimetric Audit Test Procedure for Viscous and Non-Viscous Liquids by Portable Digital Density Meter

Use the following procedure for packages labeled in fluid volume.

Most portable digital density meters are suitable for measuring the density of homogenous liquids free of suspended gas, air, sediment, and suspended matter.

The suitability of a given meter for use with specific product types is determined based upon the specifications of the manufacturer, the intended application, and verification by a recognized laboratory.

Prior to using for audit testing for a specific commodity, the official's metrology laboratory must perform a comparison between the densities obtained between Sections 3.2. Gravimetric Test Procedure for Non-Viscous Liquids or 3.3. Volumetric Test Procedure for Non-Viscous Liquids, and the digital density meter to demonstrate repeatable, reliable, results.

The portable digital density meter shall be verified and approved in accordance with the manufacturer's and other recognized calibration procedures before being put into service. The portable digital density meter must only be used in a manner for which it was designed and calibrated. This device must be routinely recertified according to your agency's measurement assurance policies. Refer to NIST HB 130 Section 11 (h) of Weights and Measures Law and NIST HB 133 Chapter 1, Section 1.7. Good Measurement Practices for additional guidance.

This audit test procedure may be used as an alternative audit test procedure for the following Sections:

- **Section 3.2. Gravimetric Test Procedure for Non-Viscous Liquids.**

NIST OWM – NET 22.2 Portable Digital Density Meter proposal submitted by NIST OWM for consideration at the 2022 NCWM Annual Meeting.

- Section 3.3. Volumetric Test Procedure for Non-Viscous Liquids.
- Section 3.4. Volumetric Test Procedures for Viscous Fluids – Headspace.

This test procedure can also be a timesaver for screening products for proper fill and for quality control purposes.

3.X.1. Test Equipment

- A scale that meets the requirements in Chapter 2, Section 2.2. “Measurement Standards and Test Equipment.”

To verify the scale has adequate resolution, use the following steps.

- Determine the density of the liquid.
- Using the density, convert the labeled volume to weight.
- Based on the labeled volume, determine the MAV using Table 2-6 “Maximum Allowable Variations for Packages Labeled by Liquid and Dry Volume” found in Appendix A.
- Using the density, convert the MAV from volume to weight.
- Next verify that the scale division is no larger than MAV/6 for the package size under test.
- The smallest graduation on the scale must not exceed the weight value for MAV/6.

Example:

Assume the inspector is using a scale with 1 g (0.002 lb) increments to test packages labeled 1 L (33.8 fl oz) that have an MAV of 29 mL (1 fl oz). Also, assume the inspector finds that the weight of 1 L of the liquid is 943 g (2.078 lb).

Density: 1 L = 943 g (2.078 lb)

MAV: 29 mL (1 fl oz)

- Convert the Density into mL and Fl oz:

$$\underline{943 \text{ g} \div 1000 \text{ mL} = 0.943 \text{ g/mL}}$$

$$\underline{(2.078 \text{ lb} \div 33.8 \text{ Fl oz} = 0.0614 \text{ lb/fl oz})}$$

- Convert MAV from Volume (mL/fl oz) to Weight:

$$\underline{29 \text{ mL} \times 0.943 \text{ g/mL} = 27.347 \text{ g}}$$

$$\underline{(1 \text{ Fl oz} \times 0.0614 \text{ lb/fl oz} = 0.0614 \text{ lb})}$$

NIST OWM – NET 22.2 Portable Digital Density Meter proposal submitted by NIST OWM for consideration at the 2022 NCWM Annual Meeting.

$$\text{MAV in Weight/6: } 27.347 \text{ g} \div 6 = 4.557 \text{ g} \quad 0.064 \text{ lb} \div 6 = 0.010 \text{ lb}$$

In this example, the 1 g (0.002 lb) scale division is smaller than the MAV/6 value of 4.557 g (0.010 lb) so the scale is suitable for making a density determination.

- Low pressure air pump (small) – (e.g., an aquarium air pump)
- Syringe (glass or plastic with a Luer fitting 5 mL or larger). The syringe should be free of any lubricating substances)
- Distilled or deionized water
- Cleaning agents (See Table 3.4. Cleaning Agents)
- Waste container
- Barometer for obtaining the prevailing barometric pressure, with an accuracy of ± 3.0 mmHg
- Thermometer for measuring air temperature with a tolerance of ± 1 °C (2 °F)
- Portable digital density meter meeting a minimum requirement of:

<u>Measuring Range</u>	
<u>Density</u>	<u>0 – 3 g/cm³</u>
<u>Temperature</u>	<u>0 – 40 °C (32 – 104 °F)^a</u>
<u>Viscosity</u>	<u>0 – 1000 mPa·s</u>
<u>Accuracy^b</u>	
<u>Density</u>	<u>0.001 g/cm³</u>
<u>Temperature</u>	<u>0.2 °C (0.4 °F)</u>
<u>Repeatability s.d.</u>	
<u>Density</u>	<u>0.0005 g/cm³</u>
<u>Temperature</u>	<u>0.1 °C (0.1 °F)</u>
<u>Resolution</u>	
<u>Density</u>	<u>0.0001 g/cm³</u>
<u>Temperature</u>	<u>0.1 °C (0.1 °F)</u>
<u>Sample Volume</u>	<u>2 mL</u>
<u>Sample Temperature</u>	<u>max. 100 °C (212 °F)</u>
<u>Footnotes</u>	
<u>^a. Filling at higher temperatures possible.</u>	

b. Viscosity < 100 mPa·s, density < g/cm³

OWM recommends that the lower limits on density (example: > 0.5 g/cm³ to 2 g/cm³) be defined. Under the current proposal scenarios can occur where mathematically calculated volume values will not be sufficiently accurate.

OWM also added the density and temperature of the “Resolution” as shown above.

3.X.2. Test Procedure

- 1. Follow Section 2.3.1. “Define the Inspection Lot.” Use a “Category A” sampling plan in the inspection. Select a random sample’**
- 2. Bring the packages and their contents to a temperature, between the reference and ambient temperatures**
- 3. The portable digital density meter must be at ambient temperature or warmer to avoid causing condensation within the unit. If the density meter is warmer than the ambient temperature, condensation is not likely to occur. Condensation must be avoided and could cause digital density meter to malfunction and cause potential damage.**
- 4. Using distilled or deionized water, validate the digital density meter per the manufacturer’s calibration instructions. The portable digital density meter shall be validated and if necessary calibrated prior to each unique commodity. The digital density meter shall be calibrated using a standard sample, within an allowable density range of ± 0.0005 g/cm³.**
- 5. Ensure the portable digital density meter is clean prior to testing. Any residual liquid should be drained, and the unit should be flushed with a small amount of the sample to be tested. Flush and discard the sample two times before taking a measurement.**
- 6. Follow the manufacturer’s instructions to select the correct method, when using a meter with built in correction factors, and measure the density of the sample using the built-in pump or syringe. Fill the sample slowly and gently. If gas or air bubbles are present drain sample and refill. If the correction factor is not known, refer to step 9.**

Note: Use of a syringe may be desirable to allow sample specimen to achieve ambient temperature prior to introduction of specimen into testing cell and for viscous specimens.

- 7. Once the portable digital density meter has stabilized (maintained reading ± 0.2 °C (± 0.5 °F) for 10 seconds) record density and temperature as indicated on instrument.**
- 8. Apply the density coefficient of expansion (Alpha) also known as the density correction factor (DCF), to correct to the reference temperature. See Table X.2. Viscosity Corrections of Common Materials, if the Alpha correction is not known, then the factor can be calculated using the below formula.**

Calculating the Temperature Coefficient Alpha

$$\text{Temperature coefficient Alpha} = \left| \frac{\rho^1 - \rho_2}{T^1 - T_2} \right|$$

ρ_1 density at temperature T_1

ρ_2 density at temperature T_2

T_1 temperature at initial measurement

T_2 temperature at second measurement

Notes:

- If the density correction factor is not known but the volume correction factor (VCF) is known, the DCF can be calculated from the VCF using the following formula.
- Density Temperature Factor Alpha = Absolute Value of Beta × Density.

9. Apply the viscosity correction if viscosity > 85 centipoise at 21 °C (70 °F) by adding the value in Table X.1. Density Measurement to your density measurement. After this correction, this value is the density of the substance in in the vacuum at the prescribed reference temperature.

Note: Some digital density meters may be pre-programmed to automatically apply the viscosity. See Table X.2. Viscosity Corrections of Common Materials

10. Apply the apparent density correction by applying one of the following steps:

- (1) multiplying the density by 0.999; or
- (2) multiplying the density by the Apparent Mass Factor from Table X.3.; or
- (3) calculate apparent density by using the following:

Converting True Density into Apparent Density

The apparent density is defined as:

$$Paap = \frac{P_{true, sample} - P_{air}}{1 - \frac{P_{air}}{8.0 \text{ g/cm}^3}}$$

Where:

$Paap$ = apparent density of the sample

P_{steel} = 8.0 g/cm³

P_{air} = true density of air

$P_{true, sample}$ = true density of the sample

The apparent density is smaller than the true density and can be calculated from the true density considering the buoyancy of the sample in air and the weight and density of a reference weight in steel.

* Pair = true density of air as calculated from equation in Table X.1. Density Measurement.

After application of this factor or calculation, the new value is density of the substance in air.

11. Drain the instrument and repeat Steps 6–10 on a second specimen of the same package for verification of first measurement.

12. Compare the two readings, they must agree within 0.0003 g/cm³. Calculate the average density of the two specimens from the sample. If the difference of two readings is greater than 0.0003 g/cm³, discard results and repeat testing of sample. Air or undissolved gas will cause erroneous measurement errors. The user of the shall always visually inspect for undissolved gas in the measurement tube for a valid test.

13. Repeat testing for the second (or subsequent) package(s) of the lot.

14. Calculate the Average Product Density of sample 1 and 2. The two results must agree within 0.0005 g/cm³. If the difference between the densities of the two packages exceeds 0.0005 g/cm³, use the volumetric procedure in Section 3.3. “Volumetric Test Procedure for Non-Viscous Liquids.”

15. Determine the Average Used Dry Tare Weight of the sample according to provisions of Section 2.3.5. “Procedures for Determining Tare.”

16. Calculate the “nominal gross weight” using the following formula:

$$\text{Nominal Gross Weight} = (\text{Average Product Density [in weight units]} \times (\text{Labeled Volume}) + (\text{Average Used Dry Tare Weight}))$$

17. Weigh the remaining packages in the sample.

18. Subtract the nominal gross weight from the gross weight of each package to obtain package errors in terms of weight. All sample packages are compared to the nominal gross weight.

19. To convert the average error or package error from weight to volume, use the following formula:

$$\text{Package Error in Volume} = \text{Package Error in Weight} \div \text{Average Product Density Per Volume Unit of Measure}$$

20. The digital density meter must be stored clean. After final use of the day or extended period of time, the instrument shall be drained and cleaned following the manufacturer’s recommended cleaning procedures and using two cleaning agents. The first cleaning agent removes sample residue, and the second cleaning agent removes the first cleaning

agent. See Table X.4. Cleaning Agents for examples of cleaning agents recommended by a digital density meter manufacturer.

NOTE: If the unit will be immediately used to measure another sample of similar composition, the unit may be drained and flushed with the new sample three times before the next analysis.

21. Periodically, connect the portable digital density meter to a low-pressure air source after a thorough cleaning, such as an aquarium air pump, to dry the unit’s measurement cell. This step is a better way to ensure no buildup of deposits in the measuring cell and no long-term drift of the instrument calibration. Bypassing the internal pump may be necessary to dry measuring cell. See instrument instruction manual.

3.X.3. Evaluation of Results

Follow the procedures in Chapter 2, Section 2.3.7. “Evaluate for Compliance” to determine lot conformance.

<u>Table X.1. Density Measurement</u>		
<u>Calculate the density of air at the temperature of test using the following equation</u>		
$\rho_{\text{air, g/mL}} = 0.001293[273.15/T][P/760]$		
<u>Where:</u>		
<u>T = temperature, K, and</u>		
<u>P = barometric pressure, torr.</u>		
<u>°C</u>	<u>mmHg</u>	<u>d_{air}, g/mL</u>
<u>15.56</u>	<u>760</u>	<u>0.001223314</u>

<u>Table X.2. Viscosity Corrections of Common Materials</u>		
<u>Material</u>	<u>Viscosity in Centipoise</u>	<u>Correction g/cc</u>
<u>Water</u>	<u>1 cP</u>	
<u>Milk</u>	<u>3 cP</u>	
<u>SAE 10 Motor Oil</u>	<u>85–140 cP</u>	<u>0.0003</u>
<u>SAE 20 Motor Oil</u>	<u>140–420 cP</u>	<u>0.0006</u>
<u>SAE 30 Motor Oil</u>	<u>420–650 cP</u>	<u>0.0007</u>
<u>SAE 40 Motor Oil</u>	<u>650–900 cP</u>	<u>0.0007</u>
<u>Castrol Oil</u>	<u>1,000 cP</u>	<u>0.0008</u>
<u>Karo Syrup</u>	<u>5,000 cP</u>	<u>0.0008</u>

NIST OWM – NET 22.2 Portable Digital Density Meter proposal submitted by NIST OWM for consideration at the 2022 NCWM Annual Meeting.

<u>Honey</u>	<u>10,000 cP</u>	<u>0.00085</u>
--------------	------------------	----------------

<u>Table X.3. Apparent Mass Factor</u>					
<u>Elevation, ft</u>	<u>sea level</u>	<u>1500</u>	<u>3000</u>	<u>4500</u>	<u>6000</u>
<u>Barometer, mmHg</u>	<u>760</u>	<u>720</u>	<u>680</u>	<u>640</u>	<u>600</u>
<u>density, g/cc</u>	<u>Apparent Mass Factor</u>				
<u>0.500</u>	<u>0.9977</u>	<u>0.9979</u>	<u>0.9980</u>	<u>0.9981</u>	<u>0.9982</u>
<u>0.600</u>	<u>0.9981</u>	<u>0.9982</u>	<u>0.9983</u>	<u>0.9984</u>	<u>0.9985</u>
<u>0.700</u>	<u>0.9984</u>	<u>0.9985</u>	<u>0.9986</u>	<u>0.9987</u>	<u>0.9988</u>
<u>0.800</u>	<u>0.9986</u>	<u>0.9987</u>	<u>0.9988</u>	<u>0.9989</u>	<u>0.9989</u>
<u>0.900</u>	<u>0.9988</u>	<u>0.9989</u>	<u>0.9989</u>	<u>0.9990</u>	<u>0.9991</u>
<u>1.000</u>	<u>0.9989</u>	<u>0.9990</u>	<u>0.9991</u>	<u>0.9991</u>	<u>0.9992</u>
<u>1.100</u>	<u>0.9991</u>	<u>0.9991</u>	<u>0.9992</u>	<u>0.9992</u>	<u>0.9993</u>
<u>1.200</u>	<u>0.9991</u>	<u>0.9992</u>	<u>0.9992</u>	<u>0.9993</u>	<u>0.9993</u>
<u>1.300</u>	<u>0.9992</u>	<u>0.9993</u>	<u>0.9993</u>	<u>0.9993</u>	<u>0.9994</u>
<u>1.400</u>	<u>0.9993</u>	<u>0.9993</u>	<u>0.9994</u>	<u>0.9994</u>	<u>0.9994</u>
<u>1.500</u>	<u>0.9993</u>	<u>0.9994</u>	<u>0.9994</u>	<u>0.9994</u>	<u>0.9995</u>
<u>Elevation or prevailing barometric pressure at the location of measurement.</u>					

<u>Table X.4. Cleaning Agents</u>		
<u>(Examples of cleaning agents recommended by digital density meter manufacturers. Verify the proper cleaning agent for the digital density meter used.)</u>		
<u>Commodity</u>	<u>Cleaning Liquid 1</u>	<u>Cleaning Liquid 2</u>
<u>Petroleum products</u>	<u>Toluene, petroleum naphtha, petroleum ether, n-nonane, cyclohexane</u>	<u>Ethanol</u>
<u>Battery acid</u>	<u>Tap water</u>	<u>Ultra-pure (bi-distilled or deionized) water</u>
<u>Liquid soap and detergent, shampoo</u>	<u>Tap water</u>	<u>Ultra-pure (bi-distilled or deionized) water</u>
<u>Salad dressing, mayonnaise</u>	<u>Petroleum naphtha, dish washing agent in water</u>	<u>Ethanol</u>

NIST OWM – NET 22.2 Portable Digital Density Meter proposal submitted by NIST OWM for consideration at the 2022 NCWM Annual Meeting.

<u>Suntan lotion</u>	<u>Tap water</u>	<u>Ethanol</u>
<u>Spirits</u>	<u>Tap water</u>	<u>Ultra-pure (bi-distilled or deionized) water</u>
<u>Grape juice, syrup</u>	<u>Warm tap water</u>	<u>Ultra-pure (bi-distilled or deionized) water</u>
<u>Milk*</u>	<u>Tap water, enzymatic lab cleaner</u>	<u>Ultra-pure (bi-distilled or deionized) water</u>
<u>*NOTE: Do not introduce ethanol or other alcohols into instrument without first flushing all milk products from instruments.</u>		