QAM-I-117

Volumetric Equipment Calibration Verification

Revision 14

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_____ 3-15-21

Laboratory Manager Date

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Concurrence Date

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Texas Institute for Applied Environmental Research

1. Applicability and Purpose

This procedure applies to all adjustable and fixed volume pipetters and any labware used to deliver measured volumes of liquid by laboratory analysts at the Texas Institute for Applied Environmental Research (TIAER), Tarleton State University, Stephenville, Texas. This does not apply to glass pipettes or labware that are certified class A volumetric, nor for labware designed to hold approximate volumes only. The purpose of this procedure is to provide a method for ensuring that pipetters and volumetric labware are checked for calibrated delivery or containment.

2. Definitions

2.1. Pipetter- a hand-held plunger device which draws and delivers a measured volume of liquid into a tip attachment. Measured volumes are defined on the pipetter as an adjustable or fixed reading. Common trade names include Eppendorf, Oxford and MLA.

2.2. Volumetric Equipment- any device used to measure volume for use in an SOP that produces data. Volumetric labware normally refers to graduated cylinders or Class A flasks and not beakers or other labware used for approximate measurements.

2.3. TC (To Contain)/TD (To Deliver): description of the labware function in holding as measured or delivering as measured the required volume.

2.4. Class A- a federally defined specification of quality recognized for reproducibility and accuracy of measurement

3. Equipment, Reagents and Standards

3.1. Analytical balance (calibration checked)- Sartorius MC1 AC210P, Mettler PL-602 or equivalent capable of accurate measurement to 0.0001g for pipetters, or to one significant figure not demarcated on another device (one guessing place beyond the gradation)

3.2. Pipette tips- clean and dry

3.3. Type II ASTM deionized (DI) water

3.4. Disposable weighing boats

3.5. Laboratory thermometer (calibration checked with correction)

3.6. Volumetric equipment to be verified
3.7. Other assorted laboratory labware may be needed

4. **Procedure**
   4.1. For pipetters, draw about 100-150 mL of DI water into a beaker and adjust temperature to between 20.0° and 25.0° C by heating or cooling. Use larger amounts of temperature-stabilized water appropriate to the size of other labware being checked.
   4.2. Record identification numbers of the pipetters or labware in the Volumetric Calibration Check Logbook (or E-log) (Attachment 1, Q-I-117-1) with the time, date and initials of the analyst. Record the observed temperature of the water, thermometer ID, correction factor (CF) and corrected temperature each time prior to use.
   4.3. Ensure the balance has been calibrated in accordance with QAM-I-101, "Operation and Calibration of the Analytical Balance". Choice of balance is made based upon size of the equipment. Items containing approximately more than 50 mL capacity are generally weighed on a top loader balance. Smaller volumes are measured on the analytical balance.
   4.4. For pipetters, draw up a set amount of DI water into the pipetter tip and carefully transfer it to the tared weigh boat. For other TD labware such as graduated cylinders, tare a beaker or other container on the scale, and then fill labware to the desired gradation meniscus mark with the water. Pour the water into the tared beaker and record the weight of the water. If TC, weigh the equipment empty, then full on the scale. Be aware that smaller amounts of water evaporate quickly, so make transfers and weighing quickly but carefully.
   4.5. Record the weight of the water delivered or contained displayed on the scale, as appropriate, the immediate temperature (with CF and corrected temperature) and the measured volume indicated as delivered in the appropriate logbook. Record whether the equipment is TC or TD as labeled. Pipetters are TD and most glassware used is TD. For TD equipment, the weight of the water poured out is recorded. TC equipment is calibration checked by the weight

of water contained, thus it is done by the difference of the container before and after the water is added.

4.6. For adjustable volume pipetters and labware, two separate readings to bracket the range of use are taken in triplicate annually. Take measurement volumes at the lower and upper limits of the equipment. One reading at mid-range is taken on a quarterly basis.

4.7. After the reading(s) have been recorded, calculate the theoretical weight of the measured volume by the following using the corrected temperature:

$$Wt = Vm \times D$$

Where: Wt = theoretical weight
Vm = measured volume
D= density (g/mL) of DI water corresponding to test temperature in Attachment 3.

4.8. Record the theoretical weight in the log.

4.9. Determine the percent variance of the measured to theoretical weights by the following:

$$\% \text{ variance} = \left(1 - \frac{Wt}{Wm}\right) \times 100$$

Where: Wt = theoretical weight
Wm = measured weight

The controlled E-log may provide the calculation automatically.

4.10. If the measured and theoretical weights do not agree within ±5% variance for every volume, repeat steps above for a failed measurement to ensure no mistake was made in weighing, delivery or calculation. Record recheck point.

4.11. If the calibration check fails again, note as such in the logbook and proceed to section 5 to initiate corrective action.

4.12. If the calibration check passes, note as such in the log.

4.13. On a quarterly basis, this check is performed with one reading and recorded in the Volumetric Equipment Calibration Logbook (or E-log) (Attachment 1, Q-I-117-1). On an annual basis, high and low readings are taken. After one

recheck of individual failed points (if needed), the percent variance determines whether the item passes or fails calibration. Both high and low must pass. If passing, the date of the annual check on a waterproof label with the analyst's initials is attached to the equipment. If failing, the check is performed again. Upon failure of a second check, initiate corrective action in accordance with section 5 below and tag the equipment "out of service".

4.14. A one-point, mid-range calibration check is performed quarterly for each pipetter that is in service. Data for this check is recorded in the Volumetric Equipment Calibration Logbook (or E-log) (Attachment 1, Q-I-117-1). Pipetters are easy to damage such that volumes may change. Any time a pipetter is dropped, this check is made again. If major repairs are done, or if a new pipetter is placed into service, the two point check is performed. For other labware, quarterly checks are also performed.

4.15. Record analyst, date and expiration date along with unique equipment ID on the item in such a way that it is not easily removed by washing or use. Replace information if it becomes unreadable.

4.16. Some variation for TC equipment may be seen if the excess water is not shaken out well between readings, especially on smaller items. Water droplets evaporating may display unstable weights. If necessary, oven-dry and cool between readings.

4.17. Perform periodic greasing, cleaning and other minor routine maintenance of the pipetter according to manufacturer's instructions.

5. Quality Control and Safety Aspects

5.1. All aspects of this procedure comply with SOP-Q-101, "Laboratory Quality Control" and SOP-S-101, "Laboratory Safety".

5.2. Volumetric equipment that fails a calibration check is tagged "Out of Service" and is repaired, disposed of, or kept in a separate location from working equipment and only for spare parts.

5.3. Prior to placing any volumetric equipment in service, even if new, the two point check procedure is performed in triplicate. An additional Demonstration of Performance record is **not** required in accordance with QAM-Q-103, “Laboratory Equipment Maintenance”, as this is support equipment.

5.4. No labware is used for volume measure unless it has been volume checked or certified class A. The labware may only be checked one time and prior to use. One of two methods may be used:

5.4.1. Measurement comparison of a class A graduated container is done on the labware to be checked. A 1% deviation from the actual volume on the class A container is acceptable.

5.4.2. Measurement of deionized water weight at a known temperature. The same criterion applies in that if the labware is routinely used for one volume, then a one point check is required. If several volumes are used for the labware measurement, then a two point check (bracketing the range of use) is required.

5.5. Volume checks are also performed on each lot of disposable labware used in volumetric measurements. Examples are dilution and sample bottles for bacteria, disposable pipettes, etc. A one-point check of known temperature water, or comparison to a class A volumetric glassware may also be performed. The volume calibration verification information for disposable labware is written in the Volumetric Equipment Calibration Logbook and on the outside of the boxes that contain the disposable labware. See SOP-C-124 for further sterility determination requirements of each lot of labware used in bacteria determination.

5.6. If any equipment is taken or used out of the main laboratory (i.e. in the mobile lab or another building) and cannot be calibrated or calibration checked under routine maintenance methods, check the equipment immediately upon return to the main laboratory. Write a CAR if maintenance schedules have been exceeded or the equipment fails the check.

5.7. Failed volumetric equipment calibrations are documented on a Corrective Action Report in accordance with SOP-Q-105, “Corrective Actions”.

5.8. Use care to not spill water onto the balance. If a spill occurs, thoroughly clean and dry the balance pan prior to continuing.
5.9. The Laboratory Manager reviews the Volumetric Equipment Calibration Logbook prior to placing devices into service initially, or back into service after repair.
5.10. Keep all volumetric equipment, pipettes and waste used or generated for radiochemistry segregated from those used for stable chemistry.

6. References

6.1. Handbook of Chemistry and Physics, CRC Press, 60th Ed., 1980.
6.2. Standard Methods for the Examination of Water and Wastewater, latest online, approved edition, Ed. by Arnold Greenberg, et al., APHA.
6.3. The NELAC Institute (TNI) Standard, 2016, National Environmental Laboratory Accreditation Conference
6.4. TIAER Lab QAM and SOP manuals.

7. Attachments

7.1. Volumetric Equipment Calibration Logbook (may be electronic)
7.2. Density Table of DI Water at temperatures from 20-25°C.

Volumetric Equipment Calibration Verification

Attachment 1
Volumetric Equipment Calibration Verification Logbook
(example)

Date	Time	Equipment ID (TC/TD)	Balance ID	Therm ID	CF T-72 y= 0.0036x-0.0722	Meas Water Temp	Corr. Water Temp	g/mL @T (Density)	Meas. Vol (mL)	Meas Wt. (mg) Wm	Calc Wt. (mg) Wc	% Variance	Pass/Fail	Analyst	LM review
				T-72	0.02										
				T-72	0.02										
				T-72	0.02										
				T-72	0.02										
				T-72	0.02										
				T-72	0.02										
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				T-72	0.02										
										Review: Date:					

%Variance= (1-Wc/Wm) x 100
Q-I-117-2, rev. 14

Attachment 2
Density Table of DI Water at temperatures from 20-25°C

Temperature (°C)	Density (g/mL)
20.0	0.9982
20.5	0.9981
21.0	0.9980
21.5	0.9979
22.0	0.9978
22.5	0.9976
23.0	0.9975
23.5	0.9974
24.0	0.9973
24.5	0.9972
25.0	0.9971

Alternatively, calculate temperature to nearest 0.1°C.

$$\% \text{ of scale} = \frac{(\text{temperature} - 20°C)}{5}$$

Density= 0.9982 - (% scale x 0.0011)