DRI STANDARD OPERATING PROCEDURE

Pipette Calibration Verification

DRI SOP #4-005r0
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1.0 GENERAL DISCUSSION

1.1 Purpose of Procedure

The objectives of this standard operating procedure are to:

- Establish the methodology and acceptance criteria for verifying the pipette calibration
- Ensure the integrity of all pipette work

This procedure will be followed by all analysts in the Environmental Analysis Facility, Division of Atmospheric Sciences, at the Desert Research Institute.

1.2 Measurement Principle

The pipette volume (typically ranging from 50 microliters [µL] up to 5 milliliter [mL]) is verified by gravimetric mass measurement of water. A weighing boat is put on a scale with tare mass; then a nominal volume of distilled-deionized water (DDW) is pipetted into the weighing boat, and the DDW weight is recorded. The measured mass is compared to the expected mass calculated from the pipette volume and DDW density (1 g /mL) to verify the accuracy of the pipette.

1.3 Measurement Interferences and Their Minimization

Scales (METTLER AE 200, 0.0001 g – 100g) are calibrated annually by an independent calibration company. Regularly, before each use, various measurements of water are used to check the scale's accuracy. If any residual powders or residue lingers on the scale, the scale is cleaned with Kimwipes and methanol to maintain the integrity of the measurements.

1.4 Ranges and Typical Values of Measurements

5µL – 5 mL

1.5 Typical Lower Quantifiable Limits, Precision, and Accuracy

Quantifiable limits vary by pipette. Precision and accuracy should vary by no more than 0.1% of the listed pipette value. For example, a 1mL pipette would be expected to vary no more than +/- 0.001 g/mL.

1.6 Personnel Responsibilities

All analysts in the laboratory should read and understand this entire standard operating procedure prior to performing the verification of pipette calibration. The analyst is expected to follow this procedure step by step to perform the verification.

The laboratory manager is responsible for ensuring that the verification procedures are properly followed, maintaining the supplies and equipment necessary to ensure uninterrupted verification, ensuring proper pipette verification documentation, and sending pipettes that are out-of-calibration or due for calibration to the manufacture or an independent calibration facility for calibration or service.

The quality assurance (QA) officer of DRI's Division of Atmospheric Sciences has the following responsibilities: 1) to determine the extent and methods of quality assurance applied to each project; 2) to estimate the level of effort involved in the quality assurance; 3) to update this procedure periodically; and 4) to verify that the budgeted tasks are carried out as specified in each contract.

1.7 Definitions

The following term is used in this document:

DDW	Water processed through distillation and deionization by the PureLab Ultra Nanopure Water system with a minimum resistivity of 18.2 megohm-cm.

1.8 Related Procedures

- Extraction of Ionic Species from Filter Samples (DRI SOP #2-109)
- Carbohydrate Analysis of Aqueous Filter Extracts by Ion Chromatography (DRI SOP #2-223)
- DRI Model 2015 Multiwavelength Carbon Analysis (TOR/TOT) of Aerosol Filter Samples - Method IMPROVE_A (DRI SOP #2-226)
- Anion Analysis of Filter Extracts and Precipitation Samples by Ion Chromatography using the DIONEX ICS-5000+ System (DRI SOP #2-232)
- Cation Analysis of Filter Extracts and Precipitation Samples by Ion Chromatography using the DIONEX ICS-5000+ System (DRI SOP #2-233)
- Inductively Coupled Plasma Mass Spectrometry (ICP-MS) Analysis of Elements on PTFE filters (DRI SOP #2-234)

2.0 APPARATUS, INSTRUMENTATION, AND FORMS

2.1 Apparatus and Supplies

2.1.1 Description

- Pipette – Instrument used to transfer known volumes of fluid
- Scale – Instrument used to measure the mass of the object

- Weighing boat – A weighing container that can hold up to 5 mL liquid

2.1.2 Maintenance

If excess liquid is drawn into the pipette, it should be allowed to dry for at least one hour. The volume should be verified gravimetrically immediately after drying to ensure no damage has been done. If the values range more than 0.1% from the specified value, the pipette should be recalibrated.

If a pipette is dropped, it should be gravimetrically checked to ensure no damage or change has occurred due to percussion. If the values range more than 0.1% from the specified value, the pipette should be recalibrated.

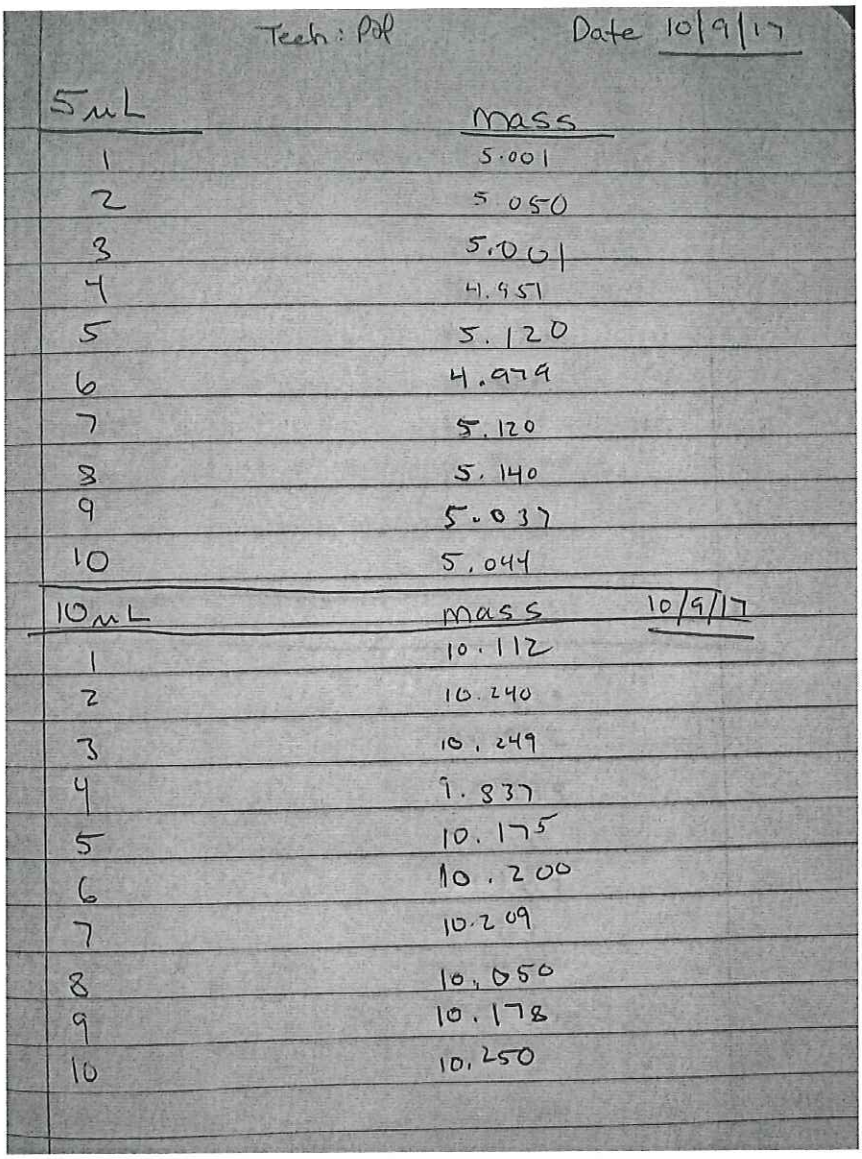
Each pipette should be kept clean. If debris or substance builds up on the handle or components on the pipette exterior, a dilute solution of rubbing alcohol and water or dilute soap can be used to clean. For Ions, do not use soaps, detergents but dilute high purity 2-propanol to ensure no ion contamination can occur.

2.2 Reagents

DDW

2.3 Forms

Pipette Calibration Verification Laboratory Notebook (Figure 2-1).



Tech: PdP Date 10/9/17

5mL	mass
1	5.001
2	5.050
3	5.001
4	4.951
5	5.120
6	4.979
7	5.120
8	5.140
9	5.037
10	5.044

10mL	mass 10/9/17
1	10.112
2	10.240
3	10.249
4	9.837
5	10.175
6	10.200
7	10.209
8	10.050
9	10.178
10	10.250

Figure 2-1. Laboratory notebook sheet for pipette calibration verification.

3.0 CALIBRATION STANDARDS

3.1 Traceability of Standards

An annual calibration of the scales by an independent calibration company makes use of standards traceable to primary weighing standards.

3.2 Use of the Standards

All gravimetric scales used for ion analysis are subject to annual preventative maintenance and calibration performed by an independent calibration/service company annually.

4.0 PROCEDURES

4.1 Verification

Set a weighing boat that can hold at least 1 mL of water onto the gram calibrated scale and tare the mass. Volumetrically aspirate the volume of the pipette with DDW (listed on the pipette as 50µL, 100 µL, 200 µL, 500 µL, 1mL or 5 mL). Expel the DDW onto the weighing boat. The scale should list the appropriate mass listed below:

50 µL – 0.050 gram
100 µL – 0.100 gram
200 µL – 0.200 gram
500 µL – 0.500 gram
1 mL – 1 gram
5 mL – 5 gram

4.2 Correcting the Pipette

Each pipette contains a small pin tool to adjust the internal mechanism that controls the aspiration volume of each pipette. If the mass displayed for the given volume exhibits too large a disparity (greater than 0.5%) it must be recalibrated. Follow the procedure given for the selected pipette brand as they may vary. A typical Calibration is listed below:

- Try a new pipette tip before attempting to calibrate the pipette. If a new tip did not fix the issues, follow steps below
- Unscrew the bottom portion of the Pipette.
- Place the calibration tool into the locked key mechanism.
- Adjust the Pipette to the right or left, depending on whether it was over or under the expected mass.
- Screw the bottom of the pipette back onto the device.

- Weight out the volume listed and readjust following the above until the mass is within 0.5%.

4.3 Pipette Precision Verification

To test the Precision of the pipette, multiple samples must be run. Follow the procedure listed in Section 4.1 six times. Document the average, standard deviation and calculate the relative standard deviation. The average should be within 0.5% of the pipette listed value, the relative standard deviation should be less than 0.1%.

5.0 DATA COMPILATION AND FILE MANAGEMENT

Each calibration verification should be documented in the laboratory notebook. Place the technician initials, the date, and if applicable the project on the page. Calculations can be done in the notebook. Make sure to write down the value for every single test, whether it fails or passes the criteria.

6.0 QUALITY CONTROL

For analysis of ions by ion chromatography for specific projects (e.g., the TCEQ $PM_{2.5}$ speciation), the pipette calibration is verified each time standards are prepared. For all carbon related standard preparation, the pipettes are calibrated annually and logged in the laboratory notebook.

If a pipette is dropped, it should be checked to make sure the internal mechanisms have not been damaged from the percussion. A simple check for volume to mass can be performed using the procedure described in Section 4.1.

7.0 REFERENCES

Standard Operating Procedure for Verification of Digital Pipettes. (03-30-2015). US Environmental Protection Agency Office of Pesticide Programs. Standard Operating Procedure for Verification of Digital Pipettes, 1-15. Office of Pesticide Programs, Environmental Science Center, Ft. Meade, MD.
https://www.epa.gov/sites/production/files/2015-08/documents/qc-19-08.pdf

8.0 DOCUMENT CHANGES

02/07/18: r0 – First version.