# *Idaho State Police*
## ***Forensic Services***

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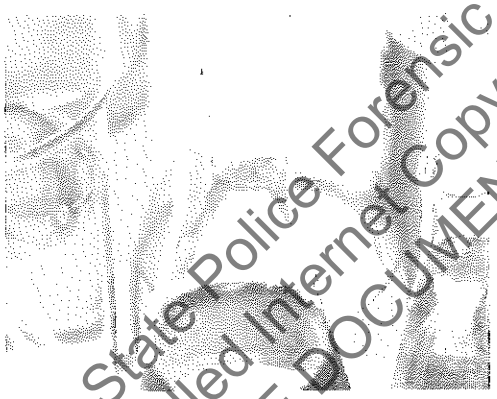



Discipline/Name of Document: Toxicology
5.1.2 Option Two: Gravimetric Intermediate Check

Revision Number: 4

Issue Date: 6/29/2007

APPROVED BY: Carina C Quisley
Quality Manager

6/29/07
Date Signed

# Section Five
# Quality Assurance

## 5.1 Verification of POVA Calibration
## 5.1.2 Option Two: Gravimetric Intermediate Check

### 5.1.2.1 BACKGROUND

The initial calibration of piston or plunger operated volumetric apparatus (POVA) is performed by the manufacturer. Upon receipt of a newly obtained pipette or syringe and thereafter periodically, the calibration must be verified to substantiate that the volume delivered is both accurate and precise. This is accomplished by determining the mass of a volume of liquid of known density that has been delivered into a closed vessel.

### 5.1.2.2 SCOPE

The reliability of the volume delivered by POVA is dependent upon verification of calibration. This method sets forth the requirements for both intermediate checks and calibration. The intermediate check is performed to maintain confidence in calibration. This manual weighing technique is an option to evaluate the performance of each POVA. The procedure is most applicable when larger volumes (≥1mL) are employed. This analytical method applies to air displacement pipettes as well as syringes attached to dilutors and dispensers. An approved external service provider performs actual POVA calibration.



### 5.1.2.3 EQUIPMENT

5.1.2.3.1 Analytical Balance
- Capable of accurately weighing volumes of interest.

5.1.2.3.2 Thermometer
- Long Solid-Stem
- Traceable to NIST Standards
- Subdivisions of ≤0.5 degree
- Capable of reading 20° - 28°C

5.1.2.3.3 Weighing Vessel with Lid
- Nonporous material
- Assorted sizes to accommodate volume under consideration

5.1.2.3.4 Timer
- Capable of accurately monitoring seconds
- Traceable to NIST Standards

5.1.2.3.5 Appropriate disposable pipette tips

**5.1.2.4 REAGENTS**

5.1.2.4.1 Deionized/distilled water

**5.1.2.5 INTERMEDIATE CHECK PROCEDURE**

5.1.2.5.1 General

5.1.2.5.1.1 The requirement for a particular POVA to have periodic intermediate check will be indicated in the applicable analytical method.

5.1.2.5.1.2 Each POVA should be tracked by its serial number and/or other unique identifier.

5.1.2.5.1.3 Intermediate checks of POVAs by an analyst or laboratory technician will be valid for four-months provided no maintenance was necessary during this period.

5.1.2.5.1.4 A POVA not in-use need not have a current intermediate check, however, the POVA must be checked prior to use for an application that requires a calibrated POVA.

5.1.2.5.1.5 An intermediate check must be performed any time a POVA is serviced.

5.1.2.5.2 Initial set-up

5.1.2.5.2.1 The water used for the intermediate check process should be allowed to equilibrate at room temperature for at least two hours prior to the start of this procedure. Verify that the room and water temperature are the same prior to the start of this procedure.

5.1.2.5.2.2 Fill out identifying information on the top portion of POVA intermediate check worksheet.

5.1.2.5.2.3 For adjustable volume POVA, the volume of interest should be recorded.

5.1.2.5.2.4 Determine and record the water temperature

on the logsheet at the beginning and at the end of determinations.

5.1.2.5.2.5 Place a volume of water in the weighing vessel, which completely covers the bottom of the container, and cap.

5.1.2.5.2.6 Place the weighing vessel on the balance and tare.

5.1.2.5.3 First Evaporation Loss (*e*) Check

5.1.2.5.3.1 Record the initial weight of the vessel and the time of recorded on the logsheet.

5.1.2.5.3.2 Remove the lid for approximately 20 seconds. This time should be adjusted to correspond to the approximate time interval between repetitions in 5.1.2.5.4.

5.1.2.5.3.3 Record a post-weight of the vessel and the time noted.

5.1.2.5.4 POVA Determinations

5.1.2.5.4.1 Use designated POVA, to dispense appropriate volume of temperature-equilibrated water into the weighing vessel and cap.

5.1.2.5.4.2 A minimum of ten individual repetitions ($W_i$), along with their corresponding time, should be recorded.

5.1.2.5.4.3 Calculate the Mean Delivered Weight ($\overline{W}$), record on logsheet.

5.1.2.5.5 Second Evaporation Loss (*e*) Check

5.1.2.5.5.1 Record the weight of the vessel and the time of recorded on the logsheet.

5.1.2.5.5.2 Remove the lid for approximately 20 seconds.

5.1.2.5.5.3 Record a post-weight of the vessel and the time recorded.

5.1.2.5.5.4 Calculate the mean evaporation weight,

record on logsheet.

5.1.2.5.6 Mean Delivered Volume

5.1.2.5.6.1 From the table below, note the conversion factor (**Z**) for the mean water temperature. The conversion factor is based upon an air pressure of 1013 hPa.

| *Temperature °C* | *Conversion Factor (Z) (µL/mg)* |
|---|---|
| 20.0 | 1.0029 |
| 20.5 | 1.0030 |
| 21.0 | 1.0031 |
| 21.5 | 1.0032 |
| 22.0 | 1.0033 |
| 22.5 | 1.0034 |
| 23.0 | 1.0035 |
| 23.5 | 1.0036 |
| 24.0 | 1.0038 |
| 24.5 | 1.0039 |
| 25.0 | 1.0040 |
| 25.5 | 1.0041 |
| 26.0 | 1.0043 |
| 26.5 | 1.0044 |
| 27.0 | 1.0045 |
| 27.5 | 1.0047 |
| 28.0 | 1.0048 |

5.1.2.5.6.2 Calculate the Mean Volume Delivered ($V_t$) at the mean recorded temperature.

$$V_t = (\overline{W} + e) \cdot Z$$

5.1.2.5.7 Inaccuracy Calculation

5.1.2.5.7.1 Determine inaccuracy by calculating the percent error ($E_t$) between the expected ($V_o$) and calculated mean ($V_t$) volume.

$$E_t = V_t - V_o / V_o \times 100$$

5.1.2.5.7.2 Record % error on log sheet.

5.1.2.5.8 Imprecision Calculation

5.1.2.5.8.1 Calculate the standard deviation ($s$) for the replicate weights.

$$s = \frac{\sum (W_i - W)^2}{n - 1}$$

$n$ = Total number of repetitions

5.1.2.5.8.2 Record $s$ on worksheet.

5.1.2.5.8.3 Determine the imprecision by calculating the coefficient of variation (CV%). This is also referred to as relative standard deviation (RSD).

$$\mathbf{CV\% = s \cdot 100 / \overline{W} + e}$$

5.1.2.5.8.4 Record CV % on worksheet.

5.1.2.5.8.5 Worksheet must be centrally stored in the laboratory performing the intermediate check.

5.1.2.5.9 Evaluation of Accuracy and Precision
Refer to Analytical Method 5.1.1 for *Acceptance Criteria.*



**5.1.2.6 CALIBRATION CHECKS**

5.1.1.6.1 All pipettes and syringes crucial for the quality of quantitative analysis will be calibrated annually by a qualified external vendor/service provider.

5.1.1.6.2 The requirement that a calibrated pipette/syringe is to be used is indicated in the relevant analytical method.

**5.1.2.7 REFERENCES**

5.1.2.7.1 ASTM Method E-1154-89 (reapproved 2003), Standard Specification for Piston or Plunger Operated Volumetric Apparatus.

5.1.2.7.2 Curtis, R.H., *Performance Verification of Manual Action Pipets: Part I,* Am. Clin. Lab. 12(7):8-9; 1994.

5.1.2.7.3 Curtis, R.H., *Performance Verification of Manual Action Pipets: Part II,* Am. Clin. Lab. 12(9):16-17; 1994.

5.1.2.7.4 Byer, B.J., How to Use and Check Pipetting Equipment, Scientific Newsletters, Inc., 1977.

5.1.2.7.5 ISO 8655-6:2002, Piston-operated volumetric apparatus – Part 6: Gravimetric method for the determination of measurement error.

# *Revision History*

## Section Five
**Quality Assurance**

### 5.1 POVA Intermediate Check
### 5.1.2 Option Two: Gravimetric POVA Intermediate Check

| Revision # | Issue Date | History |
|---|---|---|
| 0 | 10/1997 | Original Issue |
| 1 | 11-27-2001 | Reworked/reformatted |
| 2 | 03-22-2005 | Quality requirements detailed and updated |
| 3 | 05-07-2007 | Updated QA measures and reformatting. |
| 4 | 06-29-2007 | Added yearly outsourcing for calibration requirement. |