Region 4 U.S. Environmental Protection Agency Laboratory Services and Applied Science Division Athens, Georgia		
Operating Procedure		
Title: Equipment Certifications	ID: LSASDPROC-1011-R3	
Issuing Authority: Deputy Director, LSASD		
Effective Date: August 16, 2024Next Review Due Date: August 16, 2028		
Method Reference: N/A Responsible Person: Quality Assurance Coordinator		

#### Purpose

This Operating Procedure is specific to the Region 4 Laboratory Services and Applied Science Division to maintain conformance to technical and quality system requirements. Properly functioning support equipment is an essential component of producing accurate test results. As such, a regular schedule for verifying the accuracy of support equipment is necessary. The purpose of this procedure is to ensure that all support equipment (temperature sensors, barometers, balances, weights, pH meters, pipets and diluters) utilized by field investigators and laboratory analysts are properly certified and provide the appropriate level of traceability.

### Scope/Application

The requirements of this procedure apply to all personnel who perform work under the LSASD Quality Management System (QMS). Section 6.5.2 of the ISO/IEC International Standard 17025/2017 requires establishment of traceability for measurement standards and measuring instruments to the International System of Units (SI) by means of calibrations or comparisons linking them to relevant primary standards of the SI units of measurement. All equipment used for obtaining measurements within the LSASD Quality System will be certified on the schedules detailed in this operating procedure. While this SOP may be informative, it is not intended for and may not be directly applicable to operations in other organizations. Mention of trade names or commercial products in this operating procedure does not constitute endorsement or recommendation for use.

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# 1.0 **Procedure**

### **1.1** Certification of Temperature Sensors

Certification of temperature measuring devices is accomplished by comparing temperature readings from working temperature sensors to a reference standard that has an independent certification of accuracy traceable to the National Institute of Standards and Technology (NIST). The Quality Assurance Coordinator (QAC) will maintain certified reference thermometers and thermocouples to be used exclusively for this purpose. The reference thermometers and thermocouples will be sent to an ISO 17025 certified external vendor each year for certification. The vendor selected must provide a certificate indicating the thermometer/thermocouple has been certified or calibrated following ISO 17025 using equipment that is NIST-traceable. Each temperature sensor utilized in LSASD's quality system will minimally be certified annually against a certified reference standard. (Note: IR Thermometers should be verified at least every six *months*). Upon receiving the certified reference standard the provided certificate will be reviewed, and any correction factor will be applied to the reference standard for any subsequent measurements conducted with the certified reference. LSASD generally prescribes an accuracy specification of within  $\pm 1.0$  °C for each of its temperature sensors used in environments with temperatures  $\leq 100^{\circ}$ C; however, the reference standard being utilized for the certification must be capable of achieving the appropriate level of uncertainty for the intended use of the sensor being certified. For example, the reference standard used to verify microbiology laboratory temperature sensors must be accurate to within  $\pm 0.5$  °C. Any other applications requiring a temperature control tighter than  $\pm 1.0$ °C will be identified and information relayed to the OAC by the Section Chiefs. Reference standards must also be certified over the full working range of temperatures expected during use of the LSASD temperature sensor under certification. The equipment will also be labelled with the certification requirements for the temperature measuring devices. It is the responsibility of the staff member performing the certification to verify that the reference standard being utilized for certification meets the accuracy and precision limits required for the device under certification.

Thermometers or thermocouples used in refrigerators, freezers, ovens, water baths, hot blocks, incubators, etc., should be verified at the working temperature or within the working range. Microbiology laboratory incubator temperature loggers must minimally be verified at three different temperatures (i.e., at, below, and above the typical use temperature). The QAC will be responsible for facilitating the certification of all temperature devices within the laboratory areas. The QAC must be notified of new equipment purchases that include temperature measuring devices so that the equipment can be certification records. The QAC will also maintain an inventory. The QAC maintains all certification schedules and make it available for review on the LAN. The following details the certification procedures for each type of temperature sensor in use at LSASD.

- <u>Thermometers and Thermocouples operating in refrigerators, freezers,</u> <u>ovens, water baths, and hot blocks</u> – Place the working and reference temperature sensor side by side within the area the readings will be recorded. Care should be taken to prevent the two sensors from touching. Note: If an ethylene glycol bottle sensor is utilized, both devices should be placed inside the liquid solution. Allow the working and reference devices to stabilize for a minimum of 15 minutes (for devices utilized in freezers, a minimum of one hour should be allowed for proper equilibration). Record the temperature from each device on the LSASD Thermometer Certification Form (most current version). The difference between the two devices should be ≤1.0°C.
- 2. <u>Thermometers and Thermocouples operating below room temperature</u> Prepare an ice bath using crushed ice and distilled water. This bath must have a complete mix of ice/water slurry within the entire container. This mix should be prepared within a 500 mL beaker or larger. Allow the

ice/water mix to stand for 10 minutes and then place the thermometers side by side within the mixture. Allow the working and reference devices to equilibrate for a minimum of 15 minutes. Read the temperature from each device and record it onto the LSASD Thermometer Certification Form (most current version). The difference between the two devices should be  $\leq 1.0^{\circ}$ C.

- 3. <u>High Temperature Thermocouples and Digestion Blocks</u> High temperature applications (>100 °C) such as ovens, muffle furnaces and TKN digestion blocks are verified against a reference thermometer. Fill a beaker or digestion tube with sand and insert the reference standard (thermocouple) in the sand. Place the beaker or digestion tube in the device (muffle furnace, digestion block, etc), ramp the temperature to the maximum temperature prescribed by the method and allow to equilibrate for a minimum of 15 minutes after the set temperature has been achieved. Record the temperature from the thermocouple on the LSASD Thermometer Certification Form (most current version) along with all other required information. The difference between the two devices should be:
  - i.  $\leq 2.0^{\circ}$ C for temperatures >100°C, but <180°C
  - ii.  $\leq 50.0^{\circ}$ C for temperatures >180°C, but <550°C
  - iii.  $\leq 50.0^{\circ}$ C for temperatures  $> 550^{\circ}$ C

If differing criteria are specified in the method, method -specified criteria will be observed.

4. <u>Thermometers and Thermocouples operating at or above room</u> <u>temperature</u> – *Note: Thermometers that are not assigned a specific use or are not clearly labeled as to the specific working range will be certified by this procedure.* Set a constant temperature water bath or drying oven at approximately 75 °C and allow the temperature to stabilize.

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Place the working thermometer next to the reference thermometer in the water bath and let stabilize for approximately 15 minutes. Record the temperature from each device onto the LSASD Thermometer Certification Form (most current version) along with all other required information. The difference between the two devices should be  $\leq 1.0^{\circ}$ C. For field thermometers with both a probe and internal sensor (Indoor and Outdoor displayed values), both sensors will be certified. The probe will be certified as detailed above. The internal sensor will be certified against a second reference thermometer at room temperature.

- <u>Microbiology Laboratory Incubator Temperature Loggers</u> Temperature loggers (sensors) are used in the microbiology laboratory to monitor the incubator's internal temperature over an extended period. These temperature loggers must minimally be verified at three different temperatures (i.e., at, below, and above the typical use temperature).
  - i. First, place the reference thermometer next to the sensor in a clean space within the laboratory. Care should be taken to prevent the two sensors from touching. Log into the sensor data system to determine the time that the logger records to the database (e.g., once every 15 minutes). Allow both devices to equilibrate for a minimum of 15 minutes after room temperature has been achieved. Record the reference thermometer temperature at the same time the thermologger is sending a reading to the database and record it on the LSASD Thermometer Certification Form (most current version). Document the recorded temperature of the working sensor from the data system on the certification form. Repeat this observation three times and calculate the average temperature difference for both the reference standard and sensor under certification. The difference between the two devices should be  $<0.5^{\circ}$ C.
  - ii. Second, place the reference thermometer next to the sensor inside an incubator that has been operating and has achieved temperature stability. Care should be taken to prevent the two sensors from touching. Allow both devices to equilibrate for a minimum of 15 minutes after incubator temperature has been achieved. Record the reference thermometer temperature at the same time the thermologger is sending a reading to the database and record it on the certification form. Document the recorded temperature of the working sensor from the data system on the certification form. Repeat this observation three times and calculate the average temperature difference for both the reference standard and sensor under certification. The difference between the two devices should be ≤0.5°C.

- iii. Third, set a constant temperature water bath or drying oven at approximately 45 °C and allow the temperature to stabilize. Place the working thermometer next to the reference thermometer in the water bath/drying oven and let stabilize for approximately 15 minutes. Care should be taken to prevent the two sensors from touching. Allow both devices to equilibrate for a minimum of 15 minutes after water bath/drving oven temperature has been achieved. Record the reference thermometer temperature at the same time the thermologger is sending a reading to the database and record it on the certification form. Document the recorded temperature of the working sensor from the data system on the certification form. Repeat this observation three times and calculate the average temperature difference for both the reference standard and sensor under certification. The difference between the two devices should be  $\leq 0.5^{\circ}$ C.
- 6. <u>Other Temperature Loggers</u> Temperature loggers (sensors) are used in the custody room coolers or in applications where the temperature of a process needs to be controlled over an extended period. Place the reference thermometer next to the sensor. Log into the sensor data system to determine the time that the logger records to the database (once every fifteen minutes). Record the reference thermometer temperature at the same time the thermologger is sending a reading to the database and record it on the certification form. Document the recorded temperature of the working sensor from the data system on the LSASD Thermometer Certification Form (most current version). Repeat this observation three times and calculate the average temperature difference for both the reference standard and sensor under certification.

The difference between the average of the three readings for the two devices should be  $\leq 1.0^{\circ}$ C.

7. <u>Infrared (IR) Thermometer</u> – IR Thermometers should be verified at least every six months using a reference thermometer over the full range that the IR thermometer will be used. The IR thermometer will be sent to the manufacturer or other ISO accredited calibration laboratory for recertification. Each day of use when logging in drinking water samples, a single check of the IR thermometer should be made by checking the temperature of a bottle of water at the temperature of interest that contains a calibrated thermometer. Agreement between the two should be within 0.5°C, or the device will be recalibrated.

**Note:** Any applications requiring a temperature control tighter than  $\pm 1.0$  °C will be identified and information relayed to the QAC by the Section Chiefs. There

may be some applications where the temperature control is  $> \pm 1.0$  °C as specified in the method; in those instances, the specific requirements should be documented in the technical SOPs and the equipment should be labeled with the specific certification requirements.

### 1.1.1 Certification of the Mettler T90 Temperature Sensor

The Mettler T90 Autotitrator is equipped with a separate temperature sensor which compensates for temperature fluctuations when determining pH. This sensor must be certified annually; however, due to the configuration of the instrument, it can only be certified while the instrument is running. This procedure will require the assistance of an analyst familiar with the operation of the T90 or the Section Chief.

- 1. Turn on the T90 system, log in using the username and password: admin. Rinse the pH probe with DI water and place it in the appropriate slot on the autosampler. Allow the reference thermometer to equilibrate to room temperature.
- 2. Add approximately 100 ml each of pH 4, 7 and 10 buffers into 150 mL beakers. Load these beakers onto the autosampler rack with the pH4 buffer in tray location 1.
- 3. Initiate the calibration procedure on the T90. Once the titrator determines the pH of the buffer, it will display the value and the temperature. Record this value as the working thermometer temperature reading on the LSASD Thermometer Certification Form (most current version).
- 4. Place the reference thermometer probe in the beaker and record this temperature reading as the reference thermometer reading.
- 5. Repeat this process with each of the buffer solutions.
- 6. Average the readings for both the reference and working thermometers. If the difference between the average reference and working values is ≤

 $1.0^{\circ}$ C, the thermometer is determined to be within specification. Affix a label to the autotitrator indicating the certification date, expiration date and the initials of the certifier.

- 7. If the certification is not acceptable, notify the QAC and the Section Chief immediately.
- 8. Rinse the pH probe and place in the storage reservoir. Turn off the autotitrator and dispose of the buffer solutions.

## 1.1.2 Certification of Programmable HotBlock Temperature Sensors

Hotblocks with programmable temperature sensors should be verified annually to ensure the block is capable of achieving and maintaining the desired temperature. The following procedure describes verification of the TKN digestion block which utilizes a programmed temperature gradient to achieve complete digestion of the samples.

- 1. Add sand to 9 digestion tubes to be placed in the digestion block. The level of the sand in the tubes should be flush with the top of the reservoir.
- 2. Space the tubes evenly throughout the digestion block, making sure to place a tube in each of the four corners.
- 3. Create the following temperature program for testing:
  - a. Step to 80°C-Hold for 40 minutes
  - b. Ramp to 160°C-Hold for 40 minutes
  - c. Step to 400°C-Hold for 40 minutes
- 4. Run the first program. After the block has achieved the set temperature, wait 20 minutes. The block may overshoot the set temperature as it was not designed to run without a load. If this occurs, wait for the temperature to fall back down to the set point, and then wait 20 minutes. Now place the reference temperature probe in the first digestion tube, making sure that the probe touches the bottom of the tube. Wait for the temperature to stabilize (this may take 2-3 minutes) and record the temperature. Move the probe to the next digestion tube and repeat.
- 5. Repeat step 4 with the second and third programs created in step 3.
- 6. Attach a diagram of the block indicating the tube placement along with the temperature readings to the LSASD Thermometer Certification Form (most current version).
- 7. The temperature certification is acceptable if the comparison between the set point of the temperature program and the reference standard reading for each tube meets the following criteria:
  - a.  $80^{\circ}C \pm 2.0^{\circ}C$
  - b. 160°C ±5.0°C
  - c.  $400^{\circ}C \pm 10.0^{\circ}C$
- 8. If the certification does not meet the acceptance criteria, notify the Section Chief and the QAC.

### 1.1.3 Certification of the Nippon RA4500 Internal Temperature Sensor

The Nippon RA4500 mercury analyzer is a fully automated preparation and analysis unit. Included in the unit is a Hot Block with an internal temperature sensor. The temperature sensor is required to be certified annually either by the vendor, or the procedure described below. If a newer model of the instrument is purchased the procedure detailed in the user manual should be followed.

- A traceable reference standard capable of achieving 95°C ±5.0°C is required for this procedure. Prepare three sample vessels with 5mL distilled water each. They should be set on the turntable No.1(inner layer), No.47(middle layer) and No.77(outer layer).
- 2. Insert the thermometer into the first vessel.
- 3. Within the main menu of the instrument software, click the Menu

button followed by *Heater Calibration*.

- 4. In the Heater Calibration screen, enter 95 in the Set temp [degC] field.
- 5. Close the front cover on the instrument and click the *Start* button on the *Heater Calibration* screen. A message should appear stating that the unit is heating.
- 6. The heating cycle lasts for 60 minutes, record the temperature of all three vessels prior to the completion of the heating cycle as the unit immediately begins the cooling cycle.
- 7. Enter the temperature readings into the appropriate field in the *Calibration of Heater* menu screen and click on *Calc.* The calibration factor will automatically be calculated and displayed on the menu. The acceptance range for the calculation result is 0.95-1.05. If the value displayed is outside of this range. Notify the QAC, Inorganic Section Chief and Mercury analyst so the appropriate maintenance can be scheduled.

Upon completion of the certifications, all forms should be forwarded to the QAC or designee for review and approval. The sensors will then be affixed with a label indicating the certification date, initials of the certifier, and the expiration date. All certification records will be maintained by the QAC. Any equipment that does not meet the acceptance criteria listed above will be removed from service, tagged as uncertified and returned to the QAC.

## **1.2** Certification of Barometers

Barometers should minimally be calibrated annually, or by manufacturer's specifications, by using the following procedure:

Obtain a currently certified NIST-traceable barometer from the Quality and Support Branch (QSB) Air-Calibration Laboratory to utilize as a reference standard. A copy of the current calibration report for the reference standard should be forwarded to the QAC for inclusion in the certification files.

The working barometer should be placed within approximately 1 ft. horizontally and 3 ft. vertically of the calibration barometer. All barometers used in the calibration should be allowed to equilibrate for a minimum of 30 minutes. After 30 minutes, all electronic barometers, including the calibration barometer, should be turned off and then back on to allow the barometer to restart at the correct pressure reading. Record all information on the LSASD Barometer Certification Form (most current version). If environmental conditions present within the testing areas could impact the certification, document these on the Certification Form.

The working barometer reading must be within  $\pm 2$  mm Hg of the reference standard to be within certification.

Note: In the Laboratory Services Branch Biochemical Oxygen Demand Laboratory, the

YSI Model 5100 Dissolved Oxygen Probe Operations Manual, the TSI Model 5100 has an internal barometer for pressure compensation during AUTO Dissolved Oxygen Calibration. This barometer only needs to be calibrated when it is no longer reading the correct barometric pressure. If the 5100 is kept at a constant ambient temperature (+/-10°C), the barometer calibration should be accurate for approximately 30 days. LSB checks the barometer against a NIST traceable barometer prior to each sample set-up and again before the final DO determination of the samples. If the barometer reading is not within  $\pm 5$  mm Hg, the barometer is calibrated using the NIST traceable device.

#### **1.3** Certification of Laboratory Balances and Balance Checks

Laboratory balances are used throughout the LSASD laboratories. To ensure proper operation of the balances, each balance will be serviced and calibrated annually by an external ISO 17025 accredited calibration laboratory. The vendor selected must provide a certificate indicating the calibration meets ISO 17025 requirements for calibration laboratories. Calibration certificates must include the "as found" and "as left" results from the calibration as well as the measurement uncertainty. A certification sticker indicating the date of the service will be affixed to the balance by the external vendor. The expiration date for the balance will be one year from the date of service. Servicing of the balances and maintenance of the calibration records will be coordinated by the QAC.

In addition to the annual service, each balance must be checked with certified reference weights prior to each use to ensure the balance calibration continues to be valid. Each balance should have a logbook and a set of certified weights associated with it. All balance checks must be performed using certified weights and be recorded in the balance logbooks generated using LSASDFORM-1014, or if the balance is being utilized to weigh sample aliquots, document the checks in the designated sample preparation logbook. Balance checks are performed as follows:

- 1. The testing area should be free of any drafts, direct sunlight, or heat. The balance used should be properly balanced, free from vibration and have sufficient resolution for the certification being performed. If environmental factors are present during the test, note this in the appropriate logbook.
- 2. Weigh the reference weight closest to or equal to the target weight and record the weight displayed on the balance and the actual reference weight in the logbook. Record all weights to as many decimal places as displayed on the balance.
- 3. Weigh an additional reference weight(s) to bracket the target weight and record the face value of the reference weight and the weight displayed in the logbook.
- 4. See Attachments 1 and 2 for acceptance criteria for analytical balance and top loader balance checks.
- 5. If the reference weight is not within specifications, repeat the weighing. If the weight is still not in specification, obtain another reference weight and weigh it on the same balance. If the new reference weight is now in tolerance, the

original weight is suspect and should be removed from service and returned to the QAC. If the new reference weight is still not in tolerance, Notify the QAC of a potential problem that will need further investigation. Perform the balance check on another balance before use.

### **1.4** Certification of Laboratory Weights

All laboratory weights used for balance checks shall be certified annually. The certification will be facilitated by the QAC, who will maintain a set of reference weights (1 mg-100 g) for certifying other laboratory weights. The reference weights will be sent to an external vendor each year for certification. All laboratory weights used for balance checks shall be certified on an analytical balance within 60 days after the yearly balance certification (Section 1.3) is performed. All weights will be tracked by the manufacturer's serial number. Laboratory weights must only be handled with forceps or cotton gloves as provided with the weight set. Laboratory weights will be compared to the reference weights and the % difference will be determined. Certification of weights should take place on the 5-place balance as described below:

- 1. The testing area should be free of any drafts, direct sunlight, or heat. The balance used should be properly balanced, free from vibration and have sufficient resolution for the certification being performed. If environmental factors are present during the test, note this on the certification form.
- 2. Weigh the reference weight and the weight being certified (working weight). Record these weights and the face values on the Weights Certification Form.
- 3. For each measurement, calculate and record the difference between the reference weight and the working weight. The certification form calculates the % difference for each weight check using the formula:

 $\frac{Reference \ Weight - Working \ Weight}{Reference \ Weight} * 100$ 

- 4. The % difference between the reference weight and working weight shall agree within the following tolerance limits:
  - For weights >1 g, the tolerance is 0.05%.
  - For weights from 100 mg to <1 gram, the tolerance is 0.10%.
  - For weights from 10 mg to <100 mg, the tolerance is 0.5%.
  - For weights <10 mg, the tolerance is 1.0%.
- 5. If the weight is within specifications, the weight is considered to be certified, for a period of one year.
- 6. If the reference weight is not within specifications, repeat the weighings and re-check calculations. If the weight is still out-of-specifications, it must be taken out of service. The QAC must be informed of any weights that are out-of-specifications.
- 7. After successful calibration, label the weight(s) with the calibration date, expiration date and the initials of the analyst performing the certification. Ensure the serial number is listed on the outside of the weight's container.
- 8. All certification forms are routed to the QAC, or designee for review and approval.
- 9. The certification dates of the weights are recorded in the inventory by the QAC.

## **1.5** Certification of Laboratory Pipettes

This procedure applies to all non-Class A mechanical volumetric dispensing devices (excluding glass syringes) which are used in support of LSASD analytical activities. Mechanical re-pipetting devices used solely for dispensing non-quantitative reagents are exempt from these requirements. LSASD will send all air-displacement pipettes to an external vendor for servicing and calibration on an annual basis. The vendor selected must provide a certificate indicating the calibration meets ISO 17025 requirements for calibration laboratories. Additionally, the certificate must include, where applicable, "as found" and "as left" results and an assessment of the uncertainty associated with the measurements. LSASD may verify the calibration of the pipets internally throughout the year, as needed. If a pipette exceeds the criteria established in this procedure for pipet verification, it will be returned to the vendor for repairs and recalibration. In house verifications are performed as follows:

- 1. This procedure is based on converting a volume of liquid to a weight based on the density of the liquid. Most published densities are based on a specific temperature, typically 20°C (Note: 20°C=68°F, 25°C=77°F).
- 2. Determine which solvent will be utilized for this verification (based on the intended use of the pipette) and look up the density of the solvent in a reference such as "The Merck Index". Example solvent densities at 20°C:

0.9982

Water

Isooctane	0.6919
Acetone	0.7899
Methanol	0.7914

Example solvent densities at 25°C:

Water	0.9971
Acetone	0.7880
Methanol	0.7866

- 3. Record the room temperature in °C on the Pipette/Syringe Certification Form (most current version). Since density changes based on temperature, the solvent used for all measurements must be allowed to come to room temperature for approximately 1 hour prior to beginning the measurements. The room temperature should be properly measured and recorded on the form. The reference temperature standard (thermometer) ID # and certification date of the standard should also be included on the form.
- 4. The testing area should be free of any drafts, direct sunlight, or heat. The balance used should be properly balanced, free from vibration and have sufficient resolution for the certification being performed. If environmental factors are present during the test, note this on the certification form. *Note: The balance utilized for the verification procedure must meet the resolution and capacity requirements for the pipettes being verified. Prior to performing this procedure, consult the pipette operations manual for balance specifications.*
- 5. Perform a balance check on the analytical balance as detailed in Section 1.4 and document the balance ID, balance certification date, serial number of the weights used for the check and the check results on the Pipette/Syringe Certification Form.
- 6. Multiply the density of the solvent at the recorded room temperature by the volume of liquid to be dispensed by the pipette to obtain a predicted weight of the measured volume in grams. Enter this value into the certification form.
- 7. Prior to checking pipette performance, inspect the pipette. If a seal or O-ring is worn it could cause the pipette to leak and deliver inconsistent volumes. If any parts appear worn or damaged, notify the QAC and the pipet will be sent to an external vendor for servicing and recalibration.
- 8. Place the weighing vessel on the balance and carefully tare the balance.

- 9. Using the manufacturer's recommended pipette tips, warm up the pipette by gently depressing and releasing the plunger 5-10 times. Pre-wet the pipette tip by aspirating and dispensing an aliquot of the solvent 3 times. Pre-wetting the tip influences accuracy by increasing the humidity within the tip, thus minimizing evaporation of the solution. With the plunger depressed to the first stop, immerse a few millimeters of the tip into the solvent and hold there for approximately a half second, then aspirate the liquid into the pipette tip. Deliver the aliquot into the weighing vessel by placing the tip on the side of the container at a 45° angle and slowly depress the plunger past the first stop. Record the weight on the certification form.
- 10. Repeat this process for seven replicates at each range.
- 11. The pipette should be evaluated over the range of its effective use. For pipettes that are not fixed volume, three tests should be performed: one near the lower range, one near the middle range and one near the high range of use.
- 12. If the pipette meets the acceptance criteria, forward the certification form to the QAC, or designee for review and approval. Pipettes that do not meet the acceptance criteria should be removed from service and returned to the QAC for servicing and recalibration by an external vendor. Acceptance criteria for in-house certification of pipets greater than 20  $\mu$ L are:

Bias: % error RPD <1.0% Precision: % RSD <0.5%

Acceptance criteria for in-house certification of pipets  $20 \ \mu$ l or less are wider based on the systematic error associated with the pipet:

Bias: % error RPD <2.0% Precision: % RSD <1.5%

13. Pipettes that have been verified in-house will still be sent to the external vendor for servicing and calibration on the regular schedule. Verification does not replace the requirement for the annual calibration.

### **1.6** Certification of Automated Diluters (i.e., Digiflex®)

LSASD utilizes automated diluters which contain syringes for dispensing a dilution solvent. The syringes will be certified annually over the effective dilution range. Three tests should be performed: one near the low range, one near the middle range and one near the high range of use (i.e., 1:1 dilution, 5:1 dilution, 100:1 dilution). The syringes are certified using the procedure for certifying pipettes detailed in Section 1.5 of this SOP. The acceptance criteria for dispensing measurements are:

Bias: % error RPD <1.0% Precision: % RSD <0.5% The Pipette/Syringe Certification Form (most current version) is utilized to document diluter certifications. Upon successful completion of the procedure, route the completed form to the QAC for review and approval. Label the diluter with the certification date, expiration date, and initials of the certifier. Any diluter that does not meet the acceptance criteria will be removed from service.

## 1.7 Certification of pH Meters

LSASD utilizes pH meters which require calibration prior to each use. Each meter must be calibrated with a minimum of 2 pH buffer solutions bracketing the pH range of the solutions being tested. In addition, a third buffer is recommended to be tested following the calibration to ensure the calibration held and is reproducible. All pH meter calibration results should be documented in a stand-alone pH meter logbook, electronic form or on a preparation or analysis bench sheet. Temperature compensation pH probes are also required to be certified annually for temperature as detailed in Section 1.1 above.

### **1.8 Document Control**

The QAC will maintain an inventory of all LSASD equipment and certification dates. The inventory will be available for all staff to review on the LAN. All certification records will be maintained by the QAC for a minimum of five years and then transferred to the records room, where they will be stored as detailed in the LSASD Operating Procedure for Records Management.

## 2.0 Definitions

- 1. Reference Standard A standard traceable to SI units maintained by the laboratory and used to certify support equipment. Reference standards are certified annually by an external vendor.
- 2. Working Standard- Support equipment in the process of being certified.

### 3.0 <u>References</u>

LSASD Thermometer Certification Form (LSASDFORM-1008), most current version

LSASD Barometer Certification Form (LSASDFORM-1010), most current version

LSASD Weights Certification Form (1011), most current version

LSASD Single-Range Pipette/Syringe Certification Form (SESDFORM-1012), most current version

LSASD Operating Procedure for Control of Records (LSASDPROC-1001), most current version

### 4.0 Revision History

This table shows changes to this controlled document over time. The most recent version is presented in the bottom row of the table. Previous versions of the document are maintained by the appropriate LSASD Document Control Coordinator.

History	Effective Date
SESDPROC-1011-R0, Equipment Certifications, Original Issue	October 1, 2017
SESDPROC-1011-R1.0, Equipment Certifications- Added Section 1.1.4 to address the certification of the internal temperature sensor on the Nippon 4500 Mercury Analyzer.	April 6, 2018
Updated all SESD references to LSASD. Updated all ASB references to LSB or LSASD as appropriate. Added language for certification of field thermometers with dual probes (indoor and outdoor readings) in Section 1.1.4. Modified Attachment 1 to list upper and lower control limits instead of % tolerances for daily balance check criteria. Criteria established at 10% for all weights listed in the Table. Attachment 2 for Top Loader balance check acceptance criteria. Removed Section 1.1.1 regarding Flashpoint Apparatus Certification due to a change in equipment type. The new Flashpoint tester has not been brought on line to date and will be included in the next revision once the unit is fully operational. Added Section on pH meter calibration. Updated Revision number to 2.0.	August 16, 2019

LSASDPROC-1011-R3, <i>Equipment Certifications</i> , replaces LSASDPROC-1011-R2.0	August 16, 2024
General: Abbreviation updates, reference updates, minor editorial changes, and formatting changes throughout.	
Section 1.1: Clarified minimum requirements for verifying thermologgers used for monitoring microbiology laboratory incubator temperatures, to implement SM 9020 QC requirements. Added other clarifying language to support LSASD quality system expectations.	

#### Attachment 1

# Acceptance Limits for Analytical Balance Checks

Laboratory Services and Applied Science Division Acceptance Limits for Daily Balance Checks Analytical Balances			
Weight	Units	LCL (g)	UCL (g)
200	g	199.8000	200.2000
100	g	99.9000	100.100
50	g	49.9500	50.0500
20	g	19.98000	20.02000
10	g	9.99000	10.01000
5	g	4.9950	5.0050
2	g	1.99800	2.00200
1	g	0.99900	1.0010
500	mg	0.49950	0.50050
200	mg	0.19980	0.20020
100	mg	0.09990	0.10010
50	mg	0.0495	0.0505
20	mg	0.01980	0.02020
10	mg	0.0099	0.01001
5	mg	0.0045	0.0055
2	mg	0.0018	0.0022
1	mg	0.00099	0.00101

#### Attachment 2

### Acceptance Limits for Top Loader Balance Checks

### Laboratory Services and Applied Science Division Acceptance Limits for Daily Balance Checks Top Loader Balances

Weight	Units	LCL (g)	UCL (g)
500	g	499.50	500.50
200	g	199.8	200.20
100	g	99.90	100.10
50	g	49.95	50.50
20	g	19.98	20.02
10	g	9.99	10.01
5	g	4.99	5.01
2	g	1.99	2.01
1	g	0.99	1.01