Gravimetric Troubleshooting Checklist

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This checklist is intended to help metrologists identify potential problems in their gravimetric volume calibrations. It identifies known errors or difficulties that metrologists have had with the gravimetric volume calibration procedures.

Standards/Test Items/Transfer Vessels

- _ Meniscus Reading (See GMP 3); meniscus readers, cameras, microscopes
- _ Design, Cleanliness
 - If metal: look for corrosion, air entrapment, retention
 - o If glassware: look for clean draining and limited retention
 - If calibrating glassware to contain verify "dry" condition; watch filling on neck wetness
- _ Cubical Coefficient of Expansion
 - Correct for material of use
 - Matching CCE units with temperature units in equations
- _ Static charges on glassware and plastic
- _ Appropriate reference temperature selected
- _ Design of drain tube (Calibron SVP water draw kit)
- _ SVP properly blinded off

Balances

- _ Suitable resolution for desired uncertainty
- If using SOP 13, ASTM E 542 or other direct reading methods, the balance must be calibrated with suitable mass standards prior to test AND the correct density for internal weights must be used with the correct apparent mass correction value for the selected balance
- _ For larger electronic balances (above 5 kg) look for corner loading errors, range of use (minimize low end), relative accuracy/calibration errors, repeatability
- _ Consider air current effects and draft shields (limit movement of people)
- _ Filling on the pan discouraged due to drift and hysteresis problems
- _ Magnetism of leveling pans
- _ Linearity effects (1 point or 2 point reference standards)
- _ Zero balance before each reading
- _ Track & plot your mass standard values (to monitor variability and drift)

Standards

- _ Use TRUE MASS values for mass standards in equations
- _ Magnetism of reference standards
- _ Purity of water (do NOT use TAP Water!!! Some labs will not be able to use RO alone)
- _ Thermometer calibration and resolution (for water temperature) to at least 0.1 °C
- _ Water temperature gradients
- _ T, P, RH measurements within SOP 2 specs for air density calculations
- _ Age of distilled water (check expiration date)

Facility

- Relative Humidity (evaporation, condensation, surface adsorption on transfer vessel or container – esp if direct reading)
- Air and water temperature and stability minimize uncertainty of CCE and weighing convection currents (close to lab specs for both air and water)
- _ Air currents same influence as precision mass calibrations
- _ Proper equipment for moving transfer vessel to & onto balance

Procedure/Calculations

- _ Validated spreadsheets/software
- _ Rounding differences/errors
- Uncertainty of equations air density (0.0012 mg/cm3), water density (0.000010 g/cm3).
 (Use CIPM air density equation and Patterson/Morris water density equation both published in *Metrologia*)
- _ Z values in Z 542; use calculations not tables!!
- E 542 is a "direct reading" procedure and balance calibration errors will be incorporated;
 SOP 14 calibrates the balance in the range of use with reference standards
- _ Leaking valves
- _ Slopped/dripping water (lid or cover)
- _ Plotting Mass values per volume to monitor stability, variability, drift
- _ Water retention in hose between prover and transfer vessel minimize, or stabilize.
- _ Ensure water is air saturated and in equilibrium with atmospheric conditions, (no off-gassing).