GLP 10

Good Laboratory Practice
for the
Purity of Water

Water is used in two ways in the metrology laboratory. It may be used as a cleaning fluid or it may be used as a standard of volume for purposes of calibration. In each case, it must be clean; in the latter case it must be pure, as well.

Cleanliness of water may be achieved by removal of physical contaminating substances, especially by filtration. City water is ordinarily clean but may become dirty from the distribution system and especially from prolonged standing in some kinds of pipes and tubing. Hoses used to transfer water from and into large vessels and tanks may need internal cleaning, as well. Flushing to remove visible contamination is all that is usually required.

Clean water is all that is necessary when making measurements by volumetric transfer since only volumetric comparisons are involved. When a water density value is used in an equation for volume transfer, the density values for the standard and unknown are proportional to each other; therefore, deviations in the density values from pure water are insignificant. (This is *not* true for gravimetric calibrations.) The coefficient of expansion for distilled water and pure water are also essentially the same. It is obvious that dirty water could cause a number of problems, including depositing residues in a volumetric vessel that could cause volumetric errors or could soil its interior, as a minimum. When in doubt of the cleanliness of the water supply, filters should be attached to the supply lines used.

Pure water is needed for gravimetric volume calibrations. For gravimetric calibrations, filtration systems alone are *not* adequate. Pure water requires the removal of chemical contaminants and this may be achieved by distillation, reverse osmosis, ion exchange systems, or combinations of these systems. The purity of the water from any given system and maintenance requirements is often dependent on the quality of the source water. Source water should be tested to determine the best type of system to meet laboratory needs. Density calculations may be accurate even when appreciable levels of dissolved salts remain in the water. However, since it is very difficult to know what type and quantity of salts are tolerable before density is affected, it is prudent to use the best system practical within budgetary constraints.

ASTM Type III or IV Reagent Water[1]* is recommended as adequate for gravimetric calibration purposes. Such water may be produced by distillation or by ion exchange with relatively inexpensive equipment. In fact, most systems designed to meet these specifications actually provide quality better than required. Type III grade of reagent water may be prepared by distillation, ion exchange, continuous electrodeionization reverse osmosis, or a combination thereof, followed by polishing with a 0.45 μm membrane filter. Type IV grade of reagent water may be prepared by distillation, ion exchange, continuous electrodeionization reverse osmosis,

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[1] ASTM D 1193, Standard Specification for REAGENT WATER, ASTM, http://www.astm.org, 1916 Race St., Philadelphia, PA 19103.

electrodialysis, or a combination thereof. Sales literature will usually specify whether the equipment will provide water of the above quality. Additionally, many manufacturers will provide a source water quality test and recommend a system to meet the purity and volume requirements of the laboratory. There are a number of commercial sources for such equipment.

A cartridge-type ion-exchange system is recommended for its simplicity and ease of operation. It can operate intermittently (on demand) and requires little or no maintenance except for change of cartridges, the need for which will be indicated. A relatively small system (2 L/hr to 30 L/hr) is adequate for laboratories calibrating glassware and test measures up to and including 20 L (5 gal) standards. It may be used on demand or to fill a small (20 L to 40 L) storage bottle to assure a continuous supply of calibration water.

There are two broad types of ion-exchange systems. Pressure cartridge systems (PCS) operate directly from line pressure (up to 700 kPa) and need no special operation precautions. The less expensive type operates from the water line through a needle valve to produce a specified flow rate through the cartridge. In this system, the outlet must not become blocked or turned off to prevent the water pressure from building up and bursting the cartridge. It is common practice to plumb directly from the output of this cartridge to a storage tank without using a valve in between. The unit is operated by simply turning the shut-off valve located at the water supply.

Water density may be measured with a five- or six-place oscillation-type density meter calibrated using suitable standard reference materials that are representative of the range of use. Less accurate density meters are not suitable for evaluating the quality of water needed for gravimetric calibrations. These systems typically measure the density at a specific reference temperature (generally 20 °C). Density meters are generally not needed if an appropriate water purification system is used that includes a way to measure conductivity or resistivity.

Conductivity and resistivity measurements do not have a direct correlation to water density, which is the critical attribute of concern for gravimetric volume calibrations. However, conductivity or resistivity measurements are a good indicator of water quality and whether the system is in good operating condition or needs service. Conductivity is simply the reciprocal of resistivity. For water quality specifications and assessment purposes, conductivity is usually measured in microSiemens per centimeter (µS/cm) and resistivity is usually measured in megaohms-centimeter (MΩ·cm), both usually at a reference temperature of 25 °C. Conductivity is greatly influenced by temperature and is not linear. However, this is not a major concern for typical laboratory applications requiring pure water. Either a meter or indicator light should be included in laboratory systems to monitor water quality output. Standard reference materials are available to test conductivity and resistivity units. But, because the measurement values are not used to perform corrections to volume calibrations, traceable calibrations of the units are not essential.

Exposure of pure water in storage to air will likely cause degradation in the conductivity and/or resistivity measurements. However, pure water has been stored for over a year with little degradation in the density quality (provided that storage containers and lines are clean and that there is no bacterial growth, algae, or other contamination).

The ASTM D-1193 specifications for conductivity and resistivity are noted in Table 1.

Table 1. Conductivity and resistivity specifications for water

	Type I	Type II	Type III	Type IV
Electrical conductivity, max, µS/cm at 298 K (25 °C)	0.056	1.0	0.25	5.0
Electrical resistivity, min, MΩ·cm at 298 K (25 °C)	18	1.0	4.0	0.20

Conductivity and resistivity (along with other water quality measurements) are often used to assess the water quality used in cooling towers, boilers, relative humidity systems, micro and nanoelectronic systems and in pharmaceuticals, to ensure water of sufficient purity and to minimize corrosion or build-up within such systems.

Temperature equilibrium is another important factor in density stability of water. This is especially critical for large volumes. Water temperatures must be stable. Temperature accuracy is as important as purity for a correct density determination. If water is coming straight from the tap through the purification system into the prover, the temperature may fluctuate appreciably. Therefore, it is important to store an adequate volume of water to complete a calibration either already purified or ready to go through the system.

Water density tables (see Table 9.8 in NISTIR 6969 or Handbook 145) or calculations are used in most gravimetric calculations. For use in computer programs (most often spreadsheets), the use of a calculation is often preferred to look-up tables. The following equation is recommended for use.[2]

$$\rho(T_w) = \rho_0\left[1-\left(A(T-T_0)+B(T-T_0)^2+C(T-T_0)^3+D(T-T_0)^4+E(T-T_0)^5\right)\right] \qquad \text{Eqn. 1}$$

where:

ρ = density of water

$\rho_0 = 999.973\,58$ kg/m^3

T_w = temperature of water in $^\circ C$

$T_0 = 3.981\,8\ ^\circ C$

$A = 7.013\,4 \text{ x } 10^{-8}\ (^\circ\text{C})^{-1}$

$B = 7.926\,504 \text{ x } 10^{-6}(^\circ\text{C})^{-2}$

$C = -7.575\,677 \text{ x } 10^{-8}(^\circ\text{C})^{-3}$

$D = 7.314\,894 \text{ x } 10^{-10}(^\circ\text{C})^{-4}$

$E = -3.596\,458 \text{ x } 10^{-12}(^\circ\text{C})^{-5}$.

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[2] Patterson, J. B., and Morris, E. C., Measurement of Absolute Water Density, 1 °C to 40 °C, Metrologia, 31, 277-288, (1994).

Equation 1 provides water density in kg/m^3. To convert the result to units of g/cm^3 or g/mL, divide the result by 1000.

Additional References:

ASTM D 1125, Standard Test Method for Electrical Conductivity and Resistivity of Water, ASTM, http://www.astm.org, 1916 Race St., Philadelphia, PA 19103.

ISO 15212-1:1998, Oscillation-type density meters – Part 1: Laboratory instruments, ISO, 1998.