SEMI C15-95 (Reapproved 1102)
TEST METHOD FOR ppm AND ppb HUMIDITY STANDARDS

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1 Purpose

1.1 This test will determine whether or not the quantity of moisture delivered by a gaseous moisture standard is in agreement with that predicted for the standard on the basis of physical principles. It is expected that this method will be used to validate moisture standards before they are first placed in use, when deviation from expected performance is likely or suspected, and at other times as seems necessary.

1.2 The test method assumes the existence of a reliable moisture analyzer. Although extensive precautions are described to ensure the validity of the analyzer, it is always possible that the test could yield a faulty result through some unforeseen defect in the analytical equipment.

1.3 Given that moisture generation is subject to many pitfalls, it will be required that the predicted moisture concentration delivered by the standard and the measurement of that level by the analyzer be independent, and that they agree. The level of agreement can vary depending on the degree of precision claimed for the moisture standard, but in any case, the measured and predicted results should be within 10% over the entire range of the moisture standard. If not, then the validation should be repeated after verifying the physical measurement upon which the prediction is based and any other parameters deemed suspect.

2 Scope

2.1 This method is intended to be applicable to any type of gaseous standard delivering a quantity of moisture in the ppm range or lower, provided the delivered moisture concentration is predictable on the basis of fundamental principles of physics. Thus, any standards which rely solely on characterization by analysis are specifically excluded.

2.2 As part of this test, a procedure is described for qualifying certain moisture analyzers considered suitable for use in qualifying moisture standards. Moisture analyzers intended for other applications may need to satisfy different criteria.

2.3 As the most accurate and reliable moisture analyzers currently available operate primarily in the ppm range, this test focuses on validation of moisture standards in this range. The need for ppb moisture standards is addressed by specifying procedures for reducing the output of ppm moisture standards to the ppb range.

NOTICE: SEMI Standards and Safety Guidelines do not purport to address all safety issues associated with their use. It is the responsibility of the users of the Documents to establish appropriate safety and health practices, and determine the applicability of regulatory or other limitations prior to use.

3 Limitations

3.1 If no higher level of accuracy is specified by the user, then a moisture standard shown to be valid by this method will generate moisture levels which deviate from the expected values by no more than 20%. The method leaves open to the user the option of using sound statistical methods to specify that a given moisture standard will generate levels showing a closer agreement with expected values.

3.2 Validation based on this test method is good for a limited time. The acceptable interval between validations depends on the nature of the standard and is not addressed by this method. Thus any citation of validation results should be accompanied by the date(s) on which validation was performed.

4 Referenced Documents

4.1 This method is based upon principles outlined in F. Mermoud, M.D. Brandt, and J.J.F. McAndrew, “Low Level Moisture Generation.” Analytical Chemistry, 63 (2), (1991): pp. 198–202.

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5 Device under Test (Test Specimen)

5.1 *Definition of a Moisture Standard* — A moisture 'standard' is defined to be any device capable of delivering a flow of humidified gas at controlled pressure, with no input being provided by the user except nitrogen of UHP grade (moisture <1 ppm) or air of equivalent purity. Any device, such as a permeation tube, which can provide a predictable amount of moisture but does not include a means to deliver gas to and from it, will not be considered a moisture standard by itself. A device which requires gas of higher purity than UHP grade will include a purifier capable of purifying UHP nitrogen to this level.

5.2 *Examples of Moisture Standards* — Some examples of moisture standards and the principles upon which they are based are as follows:

- Permeation and effusion tube standards. These must include purification means and some means of regulating the outlet pressure. The moisture delivery rate can be calculated based on the weight loss of the tube.
- Cylinder standards. The 'standard' will include a regulator specified for use with the cylinder. The moisture concentration delivered can be calculated based on that added to the cylinder in preparation. A minimum use pressure must be specified.
- Methods based on saturation of gas with water vapor at a fixed temperature and pressure. The moisture concentration in the gas may then be calculated from a knowledge of the saturation vapor pressure of water over a plane of the pure phase of ice at the saturation temperature and of the interaction virial coefficients of the gas-vapor mixture. The two-pressure and two-temperature methods are refinements of this approach requiring additional chambers whose temperature and pressure must be known.

5.3 *Dilution of Standards* — For all moisture generation methods, lower concentrations can be generated by dynamic dilution (i.e., by combining a known flow of the standard gas with a known flow of dilution gas). A diluted standard is acceptable provided:

- The standard is validated at high concentration (with the lowest concentration validated being no more than 10,000 times the use concentration),
- The dilution gas has a moisture level below the stated precision of the analysis (to be verified using the same flow path as during the subsequent analysis, and at the lowest flow rate to be used),
- The absolute accuracies of the dilution system components are verified by comparison with a reliable flow standard,
- The linearity of the dilution system can be demonstrated over the entire range of operation.

The last criterion is particularly important whenever some portion of the combined flow is discarded, as mixing problems can easily arise at large dilution factors.

6 Procedure

6.1 Validation will be performed using a hygrometer whose output can be directly related to the moisture concentration on the basis of physical laws or using an analyzer which has recently been calibrated by comparison with such an analyzer.

6.2 *Hygrometer Qualification*

6.2.1 *Primary Hygrometers* — Hygrometers can be certified by the National Institute for Standards and Technology (NIST) in the U.S., and similar certification is available in other countries, although the lowest concentration at which certification is available will vary. Repeated certification at yearly or longer intervals can be used to establish reliability. A hygrometer which has been certified by a national standards laboratory to 1 ppm (or lower) moisture in air or nitrogen, and for which reliability has been established over a period greater than or equal to that which has elapsed since certification occurred, is acceptable for validation of a moisture standard and will be referred to as a 'primary' hygrometer.

Because hygrometers which are suitable for use as primary hygrometers may be less convenient or less readily available than other hygrometers, the use of other hygrometers for validation is also acceptable, provided adequate precautions are taken to ensure that they are operating correctly. These precautions (to be described below) require

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