2.10 Hydrometer calibrations

2.10.1 Scope

This section outlines the specific technical requirements for a laboratory to be recognized as competent to carry out calibrations of hydrometers.

2.10.2 References

a) ASTM E100-95 (2001): *Standard Specification for ASTM Hydrometers*.

b) ASTM E126-92 (1998): *Standard Test Method for Inspection and Verification of Hydrometers*.

c) Hughes, J. C., "Testing of Hydrometers," *NBS Circular 555* (Washington D. C.: NBS 1954).

d) Burgess, G. K., "Standard Density and Volumetric Tables," *NBS Circular 19, 6th ed.* (Washington DC: NBS 1924).

2.10.3 Assuring the quality of test and calibration results

2.10.3.1 All sources of variability for the hydrometer calibration should be appropriately measured and monitored. Check standards should be used to ensure that the calibrations are carried out under controlled conditions. The laboratory should maintain statistical process control (SPC) commensurate with the level of uncertainty needed for the calibration. The SPC control parameters should be based on measurements of check standards (or closure parameters) and the repeatability of multiple measurements. The frequency and number of process control checks should be appropriate for the level of uncertainty claimed for the calibration.

2.10.3.2 The laboratory should have control hydrometers that adequately span the range of hydrometer materials and sizes normally calibrated by the laboratory. Every measured value of each control should be recorded and compared to its historic value to determine whether or not the process is in control. These values should be plotted on a control chart (may be done on a computer and stored electronically) that has upper and lower control limits.

2.10.4 Accommodation and environmental conditions

2.10.4.1 The environmental conditions (i.e., temperature, atmospheric pressure and relative humidity) in the hydrometer calibration area should have no more than the maximum variations permitted, depending on the materials and the level of uncertainty needed for the calibration. The reference temperature for a particular hydrometer scale may vary from 15.56 °C (15.56 °C is approximately 60 °F, which is the reference temperature for petroleum products in the United States) to 20 °C. The laboratory should have the appropriate instrumentation required to measure the environmental conditions.

2.10.4.2 The density of the water used in hydrometer calibrations should be known to within 0.000005 g/cm^3. Specific gravity is expressed as the ratio of the density of a liquid to the density of water at a specified temperature.

2.10.4.3 Vibration of equipment used in the hydrometer calibrations should be reduced to non-influential levels. If an obvious source of vibration exists, it should not adversely affect the laboratory's claimed uncertainty level.

2.10.4.4 Any laboratory that makes hydrometer comparisons should have an appropriate supply of calibration fluids with suitable surface tensions. Hydrometers should be calibrated in the liquids in which they are to be used.

2.10.4.5 Calibration liquids should be stored in an approved safety cabinet. Laboratories that make hydrometer comparisons should abide by all safety requirements set forth by a regulatory counsel, (e.g., chemical labeling, EPA and OSHA guidelines, etc.).

2.10.5 Equipment

2.10.5.1 The laboratory should have the appropriate equipment required to perform hydrometer calibrations at the uncertainty level for which it is accredited. All equipment should be properly maintained.

2.10.5.2 The laboratory that performs hydrometer comparisons should have master hydrometers for which the calibrations are directly traceable to the appropriate national standards laboratory. The appropriate calibration corrections to these master hydrometers should be applied.

2.10.5.3 The laboratory should have the equipment needed to make auxiliary measurements of hydrometers, (e.g., balances, mass standards, knowledge of water density, etc.).

2.10.5.4 Any laboratory that makes hydrometer comparisons should abide by all safety requirements set forth by a regulatory counsel, (e.g., chemical labelling, EPA and OSHA guidelines, etc.).

2.10.5.5 The laboratory should have temperature measuring capabilities suitable for the calibration procedure. In the case of measuring the specific gravity of a liquid with a master hydrometer, temperature measurement of the liquid accurate to ±0.01 °C is required.

2.10.5.6 A laboratory that makes hydrometer comparisons should have a ventilated chemical hood to exhaust any harmful fumes from the working area.

2.10.6 Test and calibration methods and method validation

2.10.6.1 The wide use of hydrometers for many different purposes has led to various stem scales for unique applications (e.g., specific gravity, percentage alcohol, degrees API, degrees Baume and Brix). The appropriate stem scale should be evaluated so that the appropriate calibration procedure can be selected and performed for the expected use of hydrometer.

2.10.6.2 Ideally, hydrometers under test are compared directly to master hydrometers in the kinds of liquids in which they are to be used. This comparison is performed in a clear, smooth glass cylinder of suitable size. The calibration liquid should be well stirred before each comparison to minimize temperature gradients in the liquid.

2.10.6.3 The laboratory should have a manual detailing the procedures to be followed for each type of hydrometers being calibrated. This manual should contain all pertinent information needed for calibration to the level of uncertainty for which it is accredited.

2.10.7 Handling of test and calibration items

2.10.7.1 Hydrometers should be cleaned and stored in a manner that prevents accidental contact with materials which could damage its surfaces. Since hydrometers are made of glass and can be easily broken, they should be handled only by an experienced operator.

2.10.7.2 Inspection should be made of all hydrometers to be calibrated for bent stems, twisted scales and loose material inside the body of the hydrometer.

2.10.7.3 The hydrometer should be wiped with alcohol and dried to assure a clean surface before it is immersed in the calibration liquid.

2.10.8 Reporting the results

2.10.8.1 As required by ISO/IEC 17025, calibration reports should describe the hydrometer with sufficient detail to avoid any ambiguity.

2.10.8.2 The uncertainty for the hydrometer should be derived from a model of the measurement system that includes, as applicable, the uncertainties due to:

a) master hydrometer,

b) long term reproducibility of the measurement system,

c) thermal expansion, and,

d) other appropriate factors.

2.10.8.3 A historical registry should be kept for all control hydrometers (see 2.10.3.2).

~~2.11 Rockwell hardness~~

~~2.11.1 Scope~~

~~This section of the handbook provides technical criteria needed to assess the competence of a calibration agency that performs Rockwell hardness calibrations. The section is organized beginning with General Technical Criteria that applies to all categories of Rockwell hardness calibration services, followed by Specific Technical Criteria for the specific types of Rockwell hardness calibration services.~~

~~2.11.2 General technical criteria for Rockwell hardness calibration agencies~~

~~2.11.2.1 Introduction~~

~~a) **Requirements and recommendations:** Hardness standards are derived standards based on the method of measurement. The standards used for most other parameters covered by NIST Handbook 150-2, hereafter referred to as the Guide, differ in that they may be traced to measurement artifacts, scientific principles, or defined by physical phenomena. Because Rockwell hardness is more of a procedural standard, the technical guide must be more prescriptive and therefore the requirements that may be only recommendations or indications of good practice in other sections become rigid requirements when~~

considering hardness calibrations. This section therefore uses the terms "shall" and "must" to indicate an absolute requirement in the same way that they are used in NIST Handbook 150. Less stringent requirements are indicated by the use of terms like "should" or "may."

b) Under this accreditation program, laboratories are assessed for competency in performing procedures defined as ***calibrations***. In the case of Rockwell hardness, U.S. national and international test standards use both ***calibration*** and ***verification*** terminology to describe the certification procedures for Rockwell hardness machines, test blocks, and indenters. In the case of test blocks and indenters, these procedures also are referred to as ***standardizations***. The requirements outlined in this section of the Guide include all of these procedures as part of the calibration process, and thus are subject to accreditation. The term ***calibration*** will be used as a general term that will include ***calibration, verification*** and ***standardization*** as they pertain to Rockwell hardness.

c) **Calibration laboratory and calibration agency:** In most cases, the calibrations discussed in the other sections of this Guide are conducted at laboratory facilities that are maintained at permanent sites. This Guide uses ***calibration laboratory*** as the designation for these laboratories. Calibrations of Rockwell hardness machines are often conducted "in the field" at the location where the hardness machine is used. In this case, the term ***calibration laboratory*** is inappropriate as a description of the calibration activity. Consequently, in this section of the Guide, the term ***calibration agency*** is used as the designation for the entity conducting Rockwell hardness calibrations.

d) For purposes of accreditation, the calibration agencies that perform Rockwell hardness calibrations have been divided into four categories which provide services as follows:

1) **Direct verification of Rockwell hardness machines**: A process for verifying that critical components of the hardness machine are within allowable tolerances by directly verifying specified parameters. These parameters may include test force application, the depth measuring system, and machine hysteresis.

2) **Indirect verification of Rockwell hardness machines**: A process for verifying that the measurement performance of the hardness machine is within allowable tolerances by measuring the Rockwell hardness of standardized test blocks.

3) **Standardization of Rockwell hardness test blocks**: A process for verifying the geometrical and physical parameters of the test block, and calibrating the test block hardness with respect to a stated reference standard.

4) **Standardization of Rockwell hardness indenters**: A process for verifying the geometrical and physical parameters of the indenter, and calibrating the indenter performance by comparison measurements with a standardizing indenter.

e) **Rockwell and Rockwell Superficial hardness tests:** The test principles, testing procedures and verification procedures are essentially identical for both the Rockwell and Rockwell Superficial hardness tests. The significant differences between the two tests are that both the preliminary and total applied test forces are smaller for the Rockwell Superficial test than for the Rockwell test. The same type and size indenters may be used for either test, depending on the scale being employed. Although some tolerance values may be different, the basic criteria for assessing calibration agencies are, for the most part, the same for both Rockwell and Rockwell Superficial hardness testing. Therefore, henceforth in this section, the term Rockwell will imply both Rockwell and Superficial Rockwell unless otherwise stated.