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VERIFICATION/CERTIFICATION OF DEVICES USED IN LIQUID MEASUREMENT and IMPLICATIONS OF HOW MERCURY ISSUES WILL IMPACT THESE PROCESSES

CLASS # 4150

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INTRODUCTION

In the past the standards from the American Petroleum Institute (API) and the American Society for Testing and Measurement (ASTM) provided specifications for instruments and equipment. Simple compliance with these standards was not enough. Therefore, a system of verification and/or certification of equipment used in measurement of liquids was instituted. These requirements were written into the standards as they came up for review. An excellent example of such a standard is Chapter 3.1.A. "Standard Practice for the Manual Gauging of Petroleum and Petroleum Products" of the API's Manual of Petroleum Measurement. 3.1.A. calls for field verification of working tapes against against a National Institute of Standards and Technology traceable master tape when it is new and every year thereafter. This is an example of the requirements to insure the instrument and the equipment meets the specifications of each standard. It is important to understand the definitions of traceability, verification, and certification before discussing the specifications for equipment used in liquid measurement.

TRACEABLILTY, VERIFICATION, AND CERTIFICATION

Many federal regulations and contracts require regulated organizations or contractees to verify that the measurements they make are "traceable" and to support the claim of traceability by auditing records for equipment used in the calibration process. This regulatory requirement implies the ability to relate individual measurement results through an unbroken chain of calibrations to a common source, Usually U.S. national standards are maintained by the NIST, or by intrinsic standards based on fundamental constants of nature with value assigned or accepted by the NIST. Factory certified equipment uses standards that meet these requirements, and although they "remove" the instrument one time from NIST, they are acceptable for traceability and they do meet requirements of the API/ASTM standards.

To adequately establish an audit trail for traceability, a proper calibration result should include: the assigned value, a stated uncertainty, identification of the standards used in the calibration and the specification where correction factors should be applied if the standard or equipment were to be used under different conditions.

Verification on the other hand requires traceability, but it does not require the detailed pedigree as stated above. Rather, it is a confirmation that the equipment checked against a traceable standard is within the tolerances as specified by the API/ASTM standard.

Certification does give a pedigree, which includes all information on the standards and the comparison made to it during the testing procedure. The information is gathered and recorded to produce a certification of the instrument's accuracy. NIST is supportive of the practice of making the user aware of traceability and providing the user details as to how traceability was established; however, at the same time NIST cannot condone the prominent display of its name on proprietary products or in the advertising of them.

NIST does not define nor enforce traceability. Moreover, NIST is not legally required to comply with another party's contract or regulation calling for such traceability. However, NIST can and does

provide technical advice on how to make measurements consistent with national standards. NIST staff serves on the committees that write the standards for the thermometers and hydrometers.

MERCURY-IN-GLASS THERMOMETERS and ALTERNATIVE FLUID THERMOMETERS

Mercury-in-Glass thermometers are considered the "gold standard" for temperature. However, in recent years, there has been a move to ban mercury in many states. This has created a gigantic issue for the petroleum industry, which depends on mercury instruments in the field and for the calibration of devices used in custody transfer of hydrocarbons. More than 13 states have placed curbs on the use of mercury. ASTM has mandated that the following statement be placed in any ASTM standards that contain mercury references:

> WARNING- Mercury has been designated by EPA and many state agencies as a hazardous material that can cause central nervous system, kidney and liver damage. Mercury, or its vapor, may be hazardous to health and corrosive to materials. Caution should be taken when handling mercury and mercury containing products. See the applicable product Material Safety Data Sheet (MSDS) for details and EPA's website http://www.epa.gov/mercury/faq.htm - for additional information. Users should be aware that selling mercury and/or mercury containing products into your state may be prohibited by state law.

In light of these changes, alternative fluids have been developed for the thermometers that have been approved by ASTM 2251 "Standard Specification for Liquid-in-Glass ASTM Thermometers with Low-Hazard Precision Liquids". Tank thermometers, such as the 59 F (0-180 degrees Fahrenheit), can be made with alternative fluids. Tank thermometers are a total immersion instrument and the alternative fluids meet requirements for the accuracy of plus or minus 0.5 degrees Fahrenheit. Partial immersion thermometers on the other hand have not yet been made of the alternatives fluids because the physical properties of the fluid do not allow for stem correction. This is where the key issue comes up. What do you do in order to make sure your temperature devices are calibrated properly—both for verification and certification of equipment? At this juncture, the API, ASTM, EPA, and NIST are all working together on the problem. A task force has been formed to test the alternative fluid instruments and to see what the petroleum industry needs to do in order to comply with state bans on mercury. The government is very interested because it too is a user of mercury instruments. A joint effort is underway by people from industry, manufacturing, and government to review all the issues.

There are many key documents and standards that provide information about thermometers and their uses. E1 "Standard Specification for ASTM Liquid-in-Glass Thermometers" provides the specifications for all thermometers made to ASTM specifications. ASTM E77, "Standard Method for Verification and Calibration of Liquid-in-glass Thermometers" is another key paper for thermometers. The API's Committee on Measurement Quality includes the use of tank thermometers and their verification in Chapter 7 of the API's Manual of Petroleum Measurement , entitled "Temperature Determination". Terminology information can be found in ASTM E344 "Terminology Relating to Thermometry and Hydrometry ". It gives excellent definitions of the terms used in this paper.

The API's Chapter 7 states that before each thermometer's initial use and at least once a year after the initial use, each thermometer shall be compared to a thermometer certified by the National Institute of Standards and Technology (NIST) or an equivalent thermometer of traceable accuracy.

The comparison shall be made at three or more temperatures to ensure that the thermometer is accurate within the limits given in Table 8. Typically, the checkpoints should be at 10%, 50%, and 90% of the temperature range in which the thermometer is expected to be used. At this time, the thermometers used for this purpose are mercury in glass.

The importance of mercury-in-glass thermometers can be understood in a review of the literature about thermometers and their verification and certification. API's Chapter 7 states that all tank thermometers must be accurate to plus or minus 0.5 degrees Fahrenheit Mercury-in-Glass thermometers, other than tank thermometers are referred to the ASTM Verification Temperatures listed in ASTM E-1. If the thermometer is a chemical precision grade thermometer and not an ASTM thermometer, it should be calibrated every 100 divisions. (Thermometers in 1 degree divisions would be calibrated every 100 degrees, thermometers in 0.1 degree divisions would be calibrated every 10 degree, etc.). The ice point should also be calibrated (either an auxiliary ice point or within thermometer scale.) The certificate of calibration shows corrections to $1/10^{th}$ of the smallest scale division. As can be seen by these types of requirements, mercury instruments are important to the testing of thermometers both in the lab and the field. There are thousands of mercury instruments used as field devices and many thousands used to verify or certify those field instruments on a routine basis. Master mercury thermometers are only available from the factory or a third part testing lab. NIST no longer certifies mercury instruments. The announcement was made on the NIST website nist.gov and stated:

Beginning March 1, 2011, the National Institute of Standards and Technology (NIST) will no longer provide calibration services for mercury thermometers. The cessation of the mercury thermometer calibration program marks the end of an era at NIST, which has provided the service since the doors opened in 1901. The closing of the program is part of a larger effort, in collaboration with the U.S. Environmental Protection Agency (EPA) and a number of professional standards organizations and environmental and industry groups, to phase out the use of mercury thermometers altogether.

For further information on the program to phase out mercury thermometers used in industrial and laboratory settings go to www.epa.gov/hg/thermometer.htm.

Mercury instruments can still be certified by the factory or third party labs. It is important to note that after the initial calibration the only re-calibration necessary on a thermometer that has had its initial calibration and includes an ice point is the ice point (0 degrees C or 32 degrees F). I urge you to get Special Publication 1088 by the NIST "Maintenance, Validation, and Recalibration of Liquid-in-Glass Thermometers", by Cross, Miller, Ripple and Strouse. It can be accessed through the NIST website and reproduced for your use.

This paper is important because it indicates how to reaffirm your mercury and alternative fluids instruments. In fact, mercury-in-glass instruments are known to improve with age. After their initial calibration, if they don't break have a separation in the mercury column, they maintain their integrity. This is because any changes in reading occur in the bulb, and therefore will be linear throughout the thermometer. It is unnecessary to do a comparison re-calibration. However, if the thermometer does not have an ice point, the thermometer should be re-calibrated annually at the ASTM verification temperatures or every 100 lines if not an ASTM thermometer.
A thermometer that is used as an in-house calibration standard (a thermometer used to calibrate other thermometers) must have an ice point and the ice point should be read before each use to note any temporary or permanent changes.

Request for certification from any of the above should include the following information: ASTM number, range, number of points to be certified and location of points (see ASTM E-1).

Thermometers will be identified by serial number and be accompanied by papers to attest to their accuracy as stated above.

PORTABLE ELECTRONIC THERMOMETERS

API's Chapter 7 "Temperature Determination" of the Manual of Petroleum Measurement states that portable electronic thermometers or PETs should adhere to the following requirements.

Before initial use, and at least once a year thereafter, each portable electronic thermometer shall be compared at three or more temperature points, near the mid point and ends of the range; with either a National Institute of Technology (NIST) certified reference thermometer or an equivalent thermometer with accuracy traceable to the NIST. The PET shall be calibrated in accordance with the manufacturer's instructions. These standardization checks will ensure that accuracy is maintained with the limits given in Table 3. Again, the use of a mercury-in-glass thermometer has been key to the calibration of PET's in the field and laboratory. Ongoing efforts are being made to develop methods to calibrate these devices in the field and lab, but for now, mercury instruments continue to be the instrument of choice.

The API's Chapter 7, "Temperature Determination" goes into detail about the use and verification of other types of temperature devices used in static and dynamic temperature situations. Please refer to this chapter for your specific needs.

GAUGING TAPES

Gauging tapes can be certified at the NIST or the factory. Fortunately, there are no major changes to the methodologies and issues relating to gauging tapes. At the NIST, the calibration of tapes is carried out in a laboratory that houses two permanent working standards, a laser interferometer, and a 50-meter (200 foot) stainless steel bench. For the most part measurements are performed using a laser system that is referenced against a cub-corner retro reflector attached to a microscope that is used manually for line location. The laboratory is maintained at 20 degree C, but a control system can vary the chamber temperature for special tests. Calibration of tapes will normally be made with the tape under tension and supported on a horizontal-flat surface. Unless otherwise requested, the total length and each 15-meter or 50-foot subinterval will be measured and reported. Each interval calibrated on a tape will have computed lengths for two (single catenary), three, four, and five equidistant points of support. For those wanting the NIST to calibrate per the API standard (every 15 feet), a request must be made for a more intervals to be tested.

The laser standard is capable of calibrating tapes with scribed graduations to an accuracy of 2 parts per million (ppm). Calibrations made with respect to the stainless steel tape bench are normally reported to an accuracy of 10 ppm.

A NIST serial number will be engraved on each tape and bob for identification (the use of NIST certified tapes is limited due to cost and time, but they do serve as the standards by which other tapes are made and certified).

Factory certified tapes follow the same guidelines as the NIST, but use less sophisticated technology. They provide certificates of accuracy for the user. Verified tapes on the other hand

provide a certificate that the tape is within tolerance as stated in chapter 3.1.A. of API's Manual of Petroleum

Measurement (2nd Edition, August, 2005) Requirements for gauging accuracy are:

> The difference between the master tape and the working tape/bob shall not exceed plus or minus 2mm or plus or minus 1/16th inch for any distance from 0 to 30 m (0 to 100 feet). The comparison shall be verified at regular intervals throughout the working length of the tape/bob weigh combination, with such intervals not to exceed 5 m (or 15 feet).

The working tape and bob should be checked for accuracy with the following procedure:

a. New tapes should be inspected prior to use throughout their entire length to determine that numerals and increments between the numerals have been placed on the tape correctly.
b. The tape and bob assembly should be inspected daily or prior to use to ensure that wear in the tape snap catch, bob eye, or bob tip does not introduce error when the tape scale is being read. The tape should also be inspected for kinks at this time. Kinked or spliced tapes shall not be used.
c. The working tape with bob attached should be checked for accuracy when new and at least annually thereafter by comparison with a master tape that has been certified by or traceable to the National Institute of Standard and Technology (NIST). See Appendix A.

Appendix A goes into detail about the methodology to compare tapes with a traceable reference standard. This procedure is tape verification and can be done in-house or by a third party.

Request for factory or NIST certified tapes should contain the following information. Type of tape, length, and the type of test (Standard Tension test and or Tension for Accuracy test). You must be aware that the tape will be checked with bob since the bob is part of the measured section. The bob and tape will be identified by a serial number marked on both pieces and will be accompanied by papers to attest to their accuracy as stated above. In tape verification, the bob and tape will assigned a serial number as well, but the certificate that accompanies verified tape and bobs indicates it is within the required tolerances, rather than giving specific readings.

HYDROMETERS

The NIST has provided standards for reference hydrometers, but these are used as laboratory standards to calibrate other hydrometers in the manufacture of hydrometers. At times they have been available to the other users, but this has been intermittent. Certified hydrometers are available from the manufacturer or laboratory certified to calibrate density instruments. Hydrometers used as reference hydrometers should be made smooth, transparent glass, free of bubbles, or other imperfections. The hydrometer should bear an inscription that indicates the purpose of the instrument. This inscription should denote the reference temperature at which it is to be used. The maker's name or trademark and an identification number should be inscribed on the hydrometer scale. Hydrometers accepted for calibration must comply with the ASTM E 100 specifications and the requirements of NIST Circular 555, "Testing of Hydrometers."

The API's Manual of Petroleum Measurement Chapter 9.3 states:

> Before each use of the combined form hydrometer, thorough examination of the hydrometer should be performed. The ballast should be secure and no lose shot seen in the body. The thermometer scale should be secure and the indicator-pin intact. The gravity scale should be secure as indicated by its slip marker. The instrument should be clean.

Request for calibration should contain the following: ASTM number, range, number of points to be certified on the temperature scale. Hydrometers will be identified by serial number and be accompanied by papers to attest to their accuracy as stated above.

CENTRIFUGE TUBES

The procedure used for testing glass volumetric apparatus is to weigh the amount of distilled water contained or delivered with reference to the graduations marked on the instrument. The volume is then computed from the density of the water. The quality of the markings and the care exercised in reading or setting the liquid level are major factors in test calibration services for glassware. Factory certified glassware is available. The accuracy of centrifuge tube graduation is stated in the API's Chapter 10.4 "Determination of Sediment and Water in Crude Oil by the Centrifuge Method- Field procedure" in the Manual of Petroleum Measurement states:

The accuracy of the tube graduation marks' shall be volumetrically certified before field use of the tube, in accordance with ASTM E 542 using NIST traceable equipment. The verification or certification of the centrifuge tube shall include a calibration check at the 0.05, 0.10, 0.15, 0.25, 0.50, 1.0, 1.5, 2.0, 50.0 and 100.0 milliliter marks. (On a 100 ml tube.) The tube is not to be used if the scale error at any mark exceeds the applicable tolerances as stated in Chapter 10.4. The tolerances range from plus or minus 0.02 ml to 1.50 ml. Verified tubes' certificates state that they are within these tolerances, and certified tubes given an actual value at the stated levels. The tubes will be identified by a serial number and will be accompanied by a report to attest to the accuracy of the marks as stated.

CONCLUSION

There is confusion about verified and certified equipment, but if the standards are reviewed, there is clear information about the requirements. The author urges users to maintain current copies of the appropriate standards for their needs in-house. This supports the standard writing organizations and clarifies the requirements. Verified and Certified equipment is available from third party sources, the manufacture, and the NIST. Each can provide a pedigree of traceability that supports your measurement needs.

Below is a listing of the address of the organizations and their phone numbers for information on ordering standards.

American Petroleum Institute
Order Desk
1220 L. Street, Northwest
Washington, D.C. 20005
Phone: 202-682-8375
FAX: 202-962-4776
Website: www.api.org

ASTM International
Attn: Customer Service Dept.
100 Barr Harbor Drive
West Conshohocken, PA 19428-2959
Phone: 610-832-9585
FAX: 610-832-9555
E-mail: service@astm.org
Website: www.astm.org

National Institute of Standards and Technology
Calibration Program
Building 411, Room A104
Gaithersburg, MD 20899-0001
Phone: 301-975-2002
FAX: 301-869-3548
E-mail:calibrations@enh.nist.gov
Website: www.nist.gov