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Standard Guide for the Preparation and Evaluation of Liquid Baths Used for Temperature Calibration by Comparison¹

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INTRODUCTION

Many of the Standards and Test Methods under the jurisdiction of ASTM committee E20 on Temperature Measurement make reference to the use of controlled temperature fluid baths for the calibration of thermometers by the comparison method. In this method the thermometer under test is measured while immersed in an isothermal medium whose temperature is simultaneously determined by a calibrated reference thermometer. The uncertainty of all such comparison calibrations depends upon how well the isothermal conditions can be maintained. The bath temperature must be stable over time and uniform within the working space at the operating temperatures. This guide provides basic information, options and instructions that will enable the user to prepare and evaluate controlled temperature baths for calibrations.

1. Scope

1.1 This guide is intended for use with controlled temperature comparison baths that contain test fluids and operate within the temperature range of -100°C to 550°C .

1.2 This guide describes the essential features of controlled temperature fluid baths used for the purpose of thermometer calibration by the comparison method.

1.3 This guide does not address the details on the design and construction of controlled-temperature fluid baths.

1.4 This guide describes a method to define the working space of a bath and evaluate the temperature variations within this space. Ideally, the working space will be as close as possible to isothermal.

1.5 This guide does not address fixed point baths, ice point baths or vapor baths.

1.6 This guide does not address fluidized powder baths.

1.7 This guide does not address baths that are programmed to change temperature.

1.8 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

1.9 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 ASTM Standards:²

- E1 Specification for ASTM Liquid-in-Glass Thermometers
- E344 Terminology Relating to Thermometry and Hydrometry
- E644 Test Methods for Testing Industrial Resistance Thermometers
- E839 Test Methods for Sheathed Thermocouples and Sheathed Thermocouple Cable

2.2 Other Documents:

- ITS-90 The International Temperature Scale of 1990³
- NIST Monograph 126 Platinum Resistance Thermometry⁴
- NIST Monograph 150 Liquid-in-Glass Thermometry⁴
- NIST SP 250-22 Platinum Resistance Thermometer Calibrations⁴

¹ This guide is under the jurisdiction of ASTM Committee E20 on Temperature Measurement and is the direct responsibility of Subcommittee E20.07 on Fundamentals in Thermometry.

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² For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

³ Preston-Thomas, H., *METROLOGIA*, Vol. 27, 1990, pp 3-10 and 107 (errata). Mangum, B. W., *JOURNAL OF RESEARCH*, National Institute of Standards and Technology, Vol 95, 1990, p. 69.

⁴ Available from National Institute of Standards and Technology (NIST), 100 Bureau Dr., Stop 1070, Gaithersburg, MD 20899-1070, <http://www.nist.gov>.

NIST SP 250-23 Liquid-in-Glass Thermometer Calibration Service⁴

2.3 Military Standards:

MIL-STD-202G Test Methods for Electronic and Electrical Component Parts⁵

3. Terminology

3.1 Standard terms used in this guide are defined in Terminology **E344**.

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *bath gradient error, n*—the error caused by temperature differences within the working space of the bath.

3.2.2 *immersion error, n*—an error caused by heat conduction, radiation or both between the temperature sensing portion of the sensor used in the bath and the environment external to the measurement system. Immersion error is caused by an incorrect immersion length and the resulting incorrect thermal contact of the temperature sensing portion of the sensor with the medium under measurement.

3.2.3 *isothermal, adj*—of, related to, or designating a region of nominally uniform temperature.

3.2.4 *thermal stability, n*—the degree of variability of the temperatures within a specified working space over a specified time interval.

3.2.5 *working space, n*—the region within a controlled temperature bath where the temperature uncertainty is maintained within acceptable limits for the purpose of performing calibrations by the comparison method.

3.2.6 *working temperature range, n*—the minimum to maximum temperature range for which the bath system provides adequate stability and uniformity.

4. Summary of Practice

4.1 This guide is intended to provide basic information that will enable the user to evaluate various controlled temperature bath features and to enable the user to prepare and properly utilize such controlled temperature baths for calibration of thermometers by the comparison method.

5. Significance and Use

5.1 The design of a controlled temperature bath will determine what thermometers can be calibrated and to what extent an isothermal condition is achieved. The lack of thermal stability and uniformity of the bath are sources of error that contribute to the overall calibration uncertainty.

5.2 This guide describes a procedure for determining the effective working space for a controlled temperature fluid bath.

5.3 This guide describes a procedure for determining the thermal stability within a controlled temperature fluid bath. Overall thermal stability is composed of the bath performance as specified by the manufacturer of the bath equipment and as a component of calibration uncertainty.

⁵ Available from Superintendent of Documents, U.S. Government Printing Office, Washington, DC 20401.

5.4 This guide describes a procedure for determining the temperature uniformity of the working space of the controlled temperature fluid bath.

6. Procedure

6.1 *Bath System*—A controlled temperature fluid bath system will incorporate most, if not all, of the following components: a fluid medium; a mechanical design that provides for containment and circulation of the fluid; a monitoring thermometer, a temperature control unit; and, elements that provide for heating, cooling or both. There are many commercially available controlled temperature baths. These baths operate from as low as -100°C to as high as +550°C; although no single bath system is capable of operation over that entire range. The design of each individual bath will create practical limits for the working temperature range. These limits are determined by considering the minimum and maximum temperature ratings for each of the components in the bath system. The user is advised to carefully review the bath manufacturer's literature to be certain that the bath system is suitable for the intended calibration temperature range and the types of thermometers to be tested. **Figs. 1-4** represent various designs of controlled temperature fluid bath systems. **Fig. 4** shows a block diagram of a comparison calibration setup.

6.1.1 *Fluid Medium*—There are many types of fluid media suitable for use in liquid temperature comparison baths. The physical properties of the medium will establish the limits for the safe operating temperature range as well as determine the overall performance of the bath system. **Fig. 5** provides a partial listing of common bath media that have been used successfully for liquid temperature comparison baths. This guide is not intended to restrict the user to only those fluids shown in **Fig. 5**. It is advisable for the user to review carefully the manufacturer's literature on any alternative fluid to be certain that it complies with the safety considerations of **6.1.1.1**.

6.1.1.1 *Safety and Environmental Impact Considerations*—(See **1.8**.) It is strongly recommended that the Material Safety Data Sheet (MSDS) of any material used as a fluid medium be reviewed and understood by the user before the material is handled for the first time. The data sheets of all test fluids should be kept readily available during bath operation in case of accidents or spills. Additionally, some producers of bath fluids provide a Global Warming Potential Index in their specifications that should be considered when choosing a bath fluid.

(1) *Temperature Limits*—**Fig. 5** provides minimum and maximum safe operating temperatures for several common bath media. Flash point temperatures are also given for certain flammable media. Consult the manufacturer's MSDS document for each bath fluid used.

(2) *Flammability*—Fluids are easily ignited above their flash point. Whenever possible, the bath fluid shall be maintained below the specified flash point. Some fluids are flammable at room temperature so the user must exercise caution to prevent the exposure of these fluids to open flames or sparks.

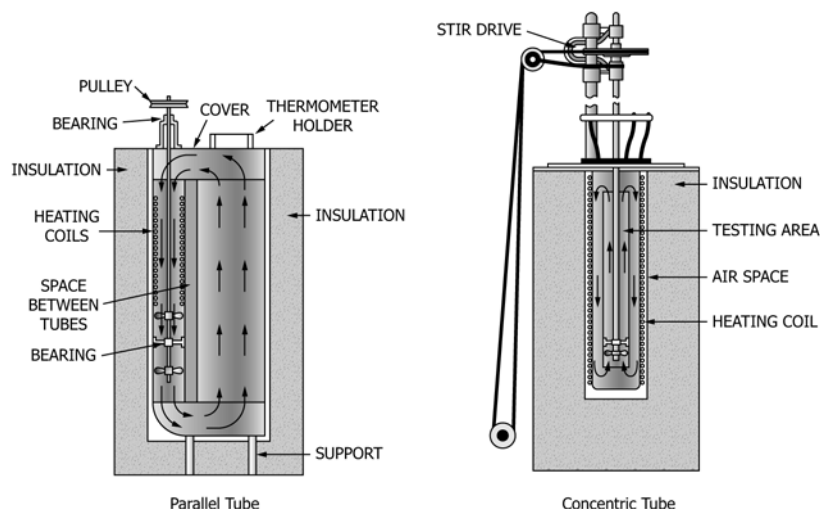


FIG. 1 Alternative Designs of Top Stirred Comparison Baths Without Controllers.

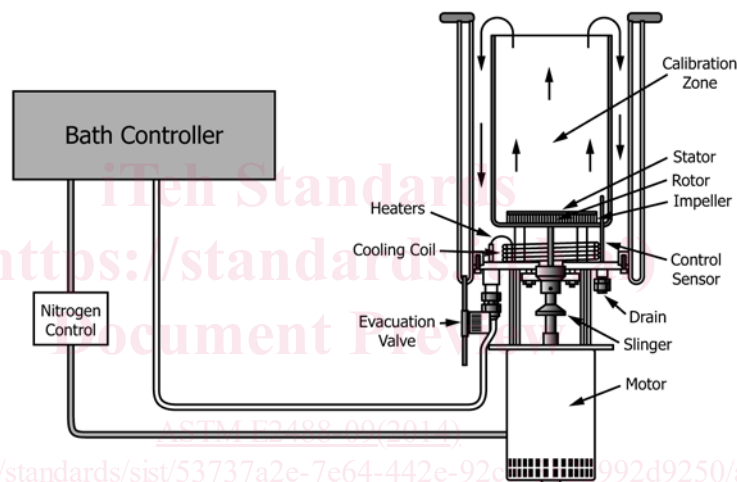


FIG. 2 Sample Design of Bottom-Stirred Comparison Bath with Controller

As a general safety practice, a fire suppression system (for example, extinguisher, blankets, hoods, lids, etc.) should always be readily available when operating a bath with flammable media.

(3) *Ventilation*—Proper ventilation, such as exhaust hoods or vents, is required to remove any fumes or vapors that may be toxic or otherwise harmful to the operators performing the calibration.

(4) *Toxicity*—Protective clothing and shielding shall be required for operators who must handle fluids that are environmentally hazardous or toxic. Proper disposal of excess fluids, spills, residues or materials contaminated by the fluids shall be in accordance with all regulatory policies.

(5) *Chemical Stability*—The bath fluid shall be chemically stable at the operating temperatures and inert to both the container and the components or elements submitted to comparison testing. **Warning**—The salts or molten metals used for calibration at high temperatures (above 260°C) are particularly corrosive to many materials. Special care should be taken to determine the compatibility of the materials used in construc-

tion of the thermometer. **DISCUSSION:** Chemical instability may change the properties of a bath fluid in one or more of the following ways: (1) *Safety*—The flash point of the bath fluid may change over time due to the chemical decomposition, breaking of chemical bonds, caused by repeated use at high temperatures. (2) *Performance*—A bath fluid that is subject to polymerization when exposed to high temperatures for extended periods will become more viscous. The increase in viscosity can degrade performance and will make maintenance and cleanup very difficult.

(6) *Expansion of Fluids*—The bath system must be designed to provide sufficient room for the expansion of fluids when heated so that spills and overflows do not occur. It is also important to consider that fluids will contract when being refrigerated to very low temperatures and then expand when allowed to return to room temperature.

(7) *Cross Contamination Between Baths*—Proper caution must be taken to avoid the mixing of test fluids when thermometers are transferred from one adjacent bath to another during multiple calibrations. Depending upon the fluids and

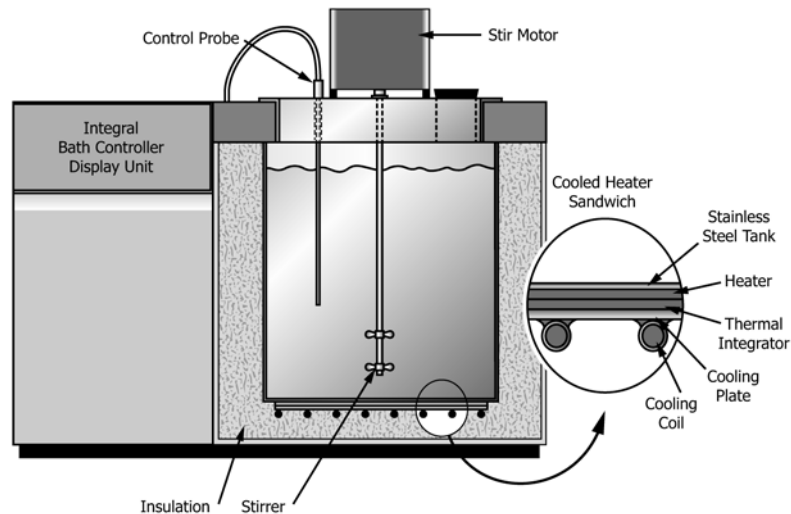


FIG. 3 Sample Design of Comparison Bath with Integral Heating, Cooling and Controller

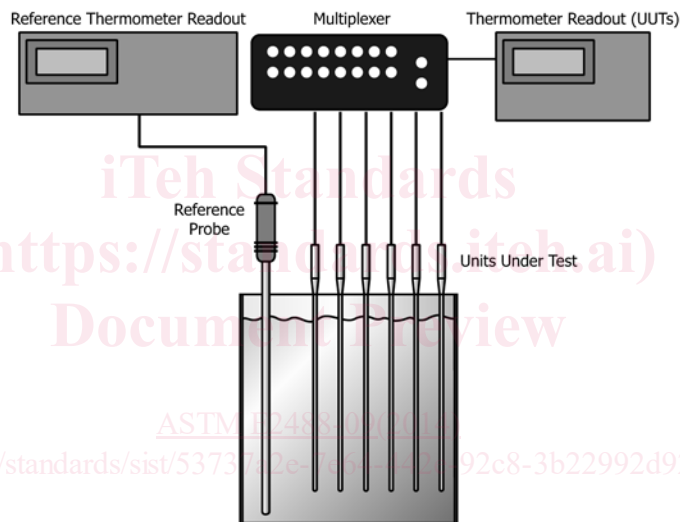


FIG. 4 Block Diagram of Comparison Calibration Setup

temperatures involved, this can be a minor problem compromising bath performance, or it can be a major safety issue. For example, fluids at temperatures above 100°C may react violently if water or a wet object is immersed into them. The introduction of water or organic materials into a molten salt bath can also produce violent reactions.

6.1.1.2 *Performance Considerations*—The physical properties of the fluid media will determine the overall performance of the bath system.

(1) *Fluid Viscosity*—The fluid viscosity can vary greatly over a wide temperature range and this can lead to problems with stirring, agitation, or the establishment of undesirable temperature gradients. In practice, a bath fluid with a viscosity of ten centistokes, or less, usually provides good stirring and mixing action. When the viscosity of the fluid becomes 50 centistokes, or greater, the stirring and mixing becomes less effective and the possibility of temperature gradients is increased. High viscosity also leads to excessive fluid drag-out.

These viscosity numbers are intended only as a general observation. The selection of a test fluid should be based upon a careful consideration of many factors.

(2) *Volatility*—Fluids operated near their boiling points, or that have a high vapor pressure under normal laboratory environments will present a problem in controlling the bath temperature. Evaporation from the surface of the liquid will produce an undesirable temperature gradient because of the increased cooling effect at the surface. In addition, the loss of bath fluid over time due to evaporation will cause the length of immersion of the thermometers to vary unless the bath design is such that it replenishes the lost fluid.

(3) *Moisture Condensation*—Refrigerated baths can cause atmospheric moisture to condense on the surfaces above the fluid. Precipitation of this moisture into the test fluid can seriously degrade the performance of the bath system.

(4) *Dielectric Properties*—The volume resistivity, dielectric strength and dielectric constant of the fluid are important