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An American National Standard

Standard Practice for Ampulization and Storage of Gasoline and Related Hydrocarbon Materials[1]

This standard is issued under the fixed designation D 6596; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This practice covers a general guide for the ampulization and storage of gasoline and related hydrocarbon mixtures that are to be used as calibration standards or reference materials. This practice addresses materials, solutions, or mixtures, which may contain volatile components. This practice is not intended to address the ampulization of highly viscous liquids, materials that are solid at room temperature, or materials that have high percentages of dissolved gases that cannot be handled under reasonable cooling temperatures and at normal atmospheric pressure without losses of these volatile components.

1.2 This practice is applicable to automated ampule filling and sealing machines as well as to manual ampule filling devices, such as pipettes and hand-operated liquid dispensers.

1.3 *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:*

D 6362 Practice for Certificates of Reference Fuels for Water Analysis[2]

E 826 Practice for Testing Homogeneity of Materials for Development of Reference Materials[3]

2.2 *ISO Standards:*[4]

ISO Guide 30 Terms and Definitions Used in Connection with Reference Materials

ISO Guide 31 Contents of Certificates of Reference Materials

ISO Guide 35 Certification of Reference Materials – General and Statistical Principles

ISO/REMCO N280 Homogeneity Testing Procedure for the Evaluation of Interlaboratory Test Samples

2.3 *Government Standard:*[5]

29 CFR 1910.1200 Hazard Communication

3. Terminology

3.1 *Definitions:*

3.1.1 *accepted reference value (ARV)*—a value that serves as an agreed-upon reference for comparison and that is derived as: *(1)* a theoretical or established value, based on scientific principles; *(2)* an assigned value, based on experimental work of some national or international organization, such as the National Institute of Standards and Technology (NIST); or *(3)* a consensus value, based on collaborative experimental work under the auspices of a scientific or engineering group.

3.1.2 *ampule*—a glass vessel for the storage of liquid materials, possessing a long narrow neck for the purpose of providing a flame-sealed closure.

3.1.3 *headspace*—the unfilled capacity of an ampule that allows for physical expansion due to temperature and pressure changes of the filled material while maintaining the integrity of the package.

3.1.4 *homogeneity*—the uniformity of the characteristics of the packaged material across the entire packaging run determined for the purpose of demonstrating the suitability of the batch for its intended purpose.

3.1.4.1 *Discussion*—There are two homogeneity testing cases; one in which the material is ampulized as a reference material at the time of ampulization, and one in which the material is not.

(*1*) *reference material at time of ampulization*—The material to be ampulized is a reference material that has accepted true or consensus values. Ampulization of a reference material would require homogeneity testing in order to assess the variability caused by the ampulization process on the true or consensus values for the reference material.

(*2*) *not a reference material at time of ampulization*—The material to be ampulized is *not* a reference material at the time of ampulization but is intended to have characterization and assignment of true or consensus values at some future date. Rigid homogeneity testing is not required on such a material at the time of ampulization since the true or consensus values have not yet been determined. However, ampules must be retained at the beginning, middle, and end of the ampulization

[1] This test method is under the jurisdiction of ASTM Committee D02 on Petroleum Products and Lubricants and is the direct responsibility of Subcommittee D02.04 on Hydrocarbon Analysis.

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[2] *Annual Book of ASTM Standards*, Vol 05.04.

[3] *Annual Book of ASTM Standards*, Vol 03.06.

[4] Available from American National Standards Institute, 11 W 42nd Street, 13th Floor, New York, NY 10017.

[5] Available from Superintendent of Documents, U.S. Government Printing Office, Washington, D.C. 20402.

process. It is recommended that qualitative testing be done on at least one sample from each of the beginning, middle, and end of the ampulization process. The remaining ampules should then be retained for future homogeneity testing to determine quantitative or consensus values.

3.1.5 *reference material (RM)*—a material or substance of which one or more properties are sufficiently well established to enable the material to be used for the calibration of an apparatus, the assessment of a method, or the assignment of values to similar materials.

3.1.6 *shelf life*—the period of time, under specified storage conditions, for which the RM will possess the same properties or true values, within established acceptance limits.

3.1.7 *stability testing*—tests required to demonstrate the chemical stability of the ampulized RM for the purpose of determining the shelf life of the RM.

4. Summary of Practice

4.1 The physical and chemical characteristics (for example, volatility, reactivity, flammability, and so forth) of a gasoline or related hydrocarbon mixture is first assessed to determine the appropriate procedures for sample handling, sample transfer, and ampulization. Then a uniform quantity of gasoline or hydrocarbon mixture is dispensed into suitably sized glass ampules (purged with an inert gas), and the ampules are flame-sealed with a torch. A number of ampules from throughout the filling and sealing process are selected and tested by appropriate test methods to determine homogeneity across the lot. Additional ampules are retained for later testing to determine stability and shelf life.

4.2 This practice addresses the common difficulties associated with the ampulization and storage of gasoline and similar liquid hydrocarbon materials, which may contain volatile components. The process of ampulization, whether performed using manual or automated equipment, involves the same fundamental issues, namely, assessment of the characteristics of the material to be ampulized, sources of contamination, sampling of the bulk container, volume dispensing accuracy, inert atmosphere blanketing, flame sealing, sequential ampule labeling, packaging homogeneity sampling, and homogeneity testing. Failure to adequately consider any of the above issues may negatively impact the quality, consistency, and value of the ampulized material as an RM.

4.3 Confidence in the homogeneity of the ampulized product can only be established through homogeneity testing, which involves the sampling, analysis, and statistical treatment of data from randomly selected ampules obtained from the beginning, middle, and end of the ampulized lot. Determination of ampulization homogeneity requires that the order in which the ampules have been filled and sealed be maintained. Homogeneity testing reveals the variability of the product introduced during the ampulization process. Homogeneity results must be within acceptable limits of the ARV or consensus value for the RM.

4.4 Ampulization does not necessarily guarantee sample stability or indefinite shelf life of the RM. Initial homogeneity data establish reference values for future tests of sample stability and determination of shelf life.

5. Significance and Use

5.1 Ampulization is desirable in order to minimize variability and maximize the integrity of calibration standards or RMs, or both, being used in calibration of analytical instruments and in validation of analytical test methods in round-robin or interlaboratory cross-check programs. This practice is intended to be used when the highest degree of confidence in integrity of a material is desired.

5.2 This practice is intended to be used when it is desirable to maintain the long term storage of gasoline and related liquid hydrocarbon RMs, controls, or calibration standards for retain or repository purposes.

5.3 This practice may not be applicable to materials that contain high percentages of dissolved gases, or to highly viscous materials, due to the difficulty involved in transferring such materials without encountering losses of components or ensuring sample homogeneity.

6. Procedure

6.1 *Manual Ampule Filling and Sealing*:

6.1.1 *Apparatus*—Devices used for manual filling of ampules include glass pipettes as well as other types of commercially available hand-operated, mechanical, liquid-dispensing devices.

6.1.2 *Storage of Bulk Material*—Bulk gasoline and similar liquid hydrocarbon materials must be adequately sealed and stored to prevent loss of volatile components prior to ampulization. Refrigerated storage in sealed metal drums, barrels, or amber glass containers is recommended.

6.1.3 *Compatibility of Materials/Sources of Contamination*:

6.1.3.1 Materials that come in contact with the bulk RM and its vapors during dispensing must be compatible with the gasoline or hydrocarbon material. Glass pipettes are recommended. Plastic or rubber materials containing phthalates or other types of plasticizers must be avoided.

6.1.3.2 Any part of the dispensing device that comes in contact with the material, including glass pipettes, hand dispensers, and any necessary connection hardware, must be cleaned prior to packaging a different material. Recommended cleaning procedures involve soaking parts in soapy water, rinsing with clean water, followed by methanol or other suitable solvent, followed by drying under a stream of clean nitrogen.

6.1.4 *Assessment of Material to Be Ampulized*:

6.1.4.1 *Volatility*—Prior to packaging, materials containing highly volatile components must be cooled sufficiently to minimize volatile losses during ampulization. Failure to sufficiently cool the material also may result in difficulty in obtaining effective ampule sealing. The material must not be cooled to temperatures below which the composition of the RM would be affected (for example, producing precipitation or solidification). Gasoline may be cooled to –20°C without incurring compositional changes. The bulk material must be kept cold during the filling process.

6.1.4.2 *Reactivity*—Consideration should be given to the chemical reactivity of the RM being packaged. Gasoline samples containing olefins and diolefins should be packaged under an inert atmosphere blanket of nitrogen, argon, or other