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Manual of Petroleum Measurement Standards (MPMS), Chapter 8.3

Standard Practice for Mixing and Handling of Liquid Samples of Petroleum and Petroleum Products[1]

This standard is issued under the fixed designation D5854; 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 the handling, mixing, and conditioning procedures that are required to ensure that a representative sample of the liquid petroleum or petroleum product is delivered from the primary sample container/receiver into the analytical test apparatus or into intermediate containers.

1.2 Annex A2 covers acceptance test criteria for power mixer and sample container combinations, while Annex A3 and Annex A4 detail acceptance tests for mixing systems. Appendix X1 is a guide for selecting sample containers.

1.3 For sampling procedures, refer to Practices D4057 (API *MPMS* Chapter 8.1) and D4177 (API *MPMS* Chapter 8.2). Practice D5842 (API *MPMS* Chapter 8.4) covers sampling and handling of light fuels for volatility measurement.

1.4 *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]

D4057 Practice for Manual Sampling of Petroleum and Petroleum Products

D4177 Practice for Automatic Sampling of Petroleum and Petroleum Products

D4306 Practice for Aviation Fuel Sample Containers for Tests Affected by Trace Contamination

D4928 Test Method for Water in Crude Oils by Coulometric Karl Fischer Titration

D5842 Practice for Sampling and Handling of Fuels for Volatility Measurement

2.2 *API Documents:*[3]

MPMS Chapter 8.1 Practice for Manual Sampling of Petroleum and Petroleum Products (ASTM Practice D4057)

MPMS Chapter 8.2 Practice for Automatic Sampling of Petroleum and Petroleum Products (ASTM Practice D4177)

MPMS Chapter 8.4 Practice for Sampling and Handling of Fuels for Volatility Measurement (ASTM Practice D5842)

MPMS Chapter 10.9 Test Method for Water in Crude Oils by Coulometric Karl Fischer Titration (ASTM Test Methods D4928)

Recommended Practice 2003, Protection Against Ignitions Arising Out of Static, Lighting, and Stray Currents

Publication 2026, Safe Access/Egress Involving Floating Roofs of Storage Tanks in Petroleum Service

Publication 2217, Guideline for Confined Space Work in the Petroleum Industry

2.3 *Department of Transportation:*[4]

Code of Federal Regulations, Title 49, Section 173

2.4 *Occupational Safety and Health Standards:*[4]

29 Code of Federal Regulations, Subpart Z, "Toxic and Hazardous Substances," Part 1910.1000 and following

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[1] This practice is under the jurisdiction of ASTM Committee D02 on Petroleum Products, Liquid Fuels, and Lubricants and the API Committee on Petroleum Measurement, and is the direct responsibility of Subcommittee D02.02.08 the joint ASTM-API committee on Sampling (API MPMS Chapter 8.0).

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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] Available from American Petroleum Institute (API), 1220 L. St., NW, Washington, DC 20005-4070, http://www.api.org.

[4] Available from the Superintendent of Documents, U.S. Government Printing Office, Washington, DC 20402.

3. Terminology

3.1 *Definitions of Terms Specific to This Standard:*

3.1.1 *intermediate container*—the vessel into which all or part of the sample from a primary container/receiver is transferred for transport, storage, or ease of handling.

3.1.2 *petroleum*—denotes petroleum crudes, as well as petroleum products, normally associated with the petroleum industry.

3.1.3 *primary container/receiver*—the vessel in which a sample is initially collected.

3.1.3.1 *Discussion*—

Examples of primary sample containers include glass and plastic bottles, cans, and fixed and portable sample receivers.

3.1.4 *sampling*—all the steps required to obtain a sample that is representative of the contents of any pipe, tank, or other vessel, and to place that sample in a container from which a representative test specimen can be taken for analysis.

3.1.5 *test specimen*—the representative sample taken from the primary or intermediate sample container for analysis.

4. Significance and Use

4.1 Representative samples of petroleum and petroleum products are required for the determination of chemical and physical properties used to establish standard volumes, prices, and compliance with commercial and regulatory specifications. The treatment of samples from the time of collection until they are analyzed requires care and effort to maintain their compositional integrity.

5. Safety and Health Precautions

5.1 In view of the potential health and safety hazards associated with the handling and mixing of petroleum samples, only qualified personnel should be involved.

5.2 All sample handling and mixing equipment should be approved by the parties involved. All equipment should be installed, operated, and maintained in a manner to minimize potential health and safety hazards.

6. Sample Containers

6.1 No single container type will meet requirements of all petroleum sampling operations. The following are general design and construction considerations for sample containers.

6.2 *Container Configuration:*

6.2.1 Containers should drain continuously toward the outlet to ensure complete liquid withdrawal.

6.2.2 Cylindrical containers are better suited for samples that are to be tested for free water or sediment and water.

6.2.3 Containers should not have internal pockets or dead spots.

6.2.4 Internal surfaces of containers should minimize corrosion, incrustation, water, and sediment clingage.

6.2.5 Container configuration should allow for the transfer of samples from one container to another or to the analytical apparatus while maintaining the integrity of the sample's composition.

6.2.6 Containers should have an inspection cover/closure/cap of sufficient size to facilitate filling, inspection, and cleaning. A means of installing security seals should be provided.

6.2.7 Containers should allow for the preparation of a homogeneous mixture of the sample while preventing the loss of any constituents which affect the representativeness of the sample and the accuracy of the analytical tests.

6.2.8 Containers should be made so as to avoid contamination from external water or other foreign material.

6.2.9 Containers used with closed loop mixers may be equipped with a discharge line inside the container which has multiple outlet ports. Another method of achieving the effect of multiple discharge ports is to split the discharge stream coming from the mixing pump into two or more separate streams with each having its own inlet into the sample container.

6.2.10 Containers used with closed loop mixers should be equipped with a pressure/vacuum relief valve set so as not to exceed the design pressure of the container. A pressure gage should also be provided.

6.2.11 Containers used with closed loop mixers may have multiple suction ports. As a minimum there should be one suction port at the lowest point in the container.

6.3 *Container Size:*

6.3.1 A general rule is that both primary and intermediate containers should be large enough to hold the required sample size within 80 % of the total capacity to facilitate mixing and to provide for thermal expansion.

6.3.2 The size of primary containers is determined from the sampling operation as described in Practices D4057 (API *MPMS* Chapter 8.1) and D4177 (API *MPMS* Chapter 8.2).

6.3.3 The size of intermediate containers should be as large as practical to minimize surface tension effects with due consideration given to storage space requirements, shipping rules and regulations, costs, availability, and other practical considerations.

6.4 *Container Material:*

6.4.1 Sample containers are normally made of glass, metal, or plastic. Exercise care in the selection of container material as it could affect the test results obtained from the sample. Containers acceptable for samples to be tested immediately may not be acceptable for storage of sample.

6.4.2 Glass containers are suitable for many sample test and storage requirements. Clear glass bottles may be examined visually for cleanliness and allow for visual inspection of the sample for free water or solid impurities. Some petroleum samples are affected by exposure to sunlight if clear glass is used. In these cases, brown glass bottles may afford the necessary protection.

6.4.3 Cans coated with tin must have seams that have been soldered on the exterior surfaces with a flux of rosin cleaned in a suitable solvent. Such a flux is easily removed with gasoline, whereas many others are very difficult to remove. Minute traces of flux may contaminate the sample so that results obtained on tests such as dielectric strength, oxidation resistance, and sludge formation may be erroneous. Exercise care also to ensure that samples containing free or entrained water are not corrosive to the metal. Internally epoxy-lined tin cans may have residual contamination and precaution should be taken to ensure its removal.

6.4.4 Cans made of stainless steel with welded seams are suitable for many sampling operations. Other than ensuring the cleanliness, use of these containers presents no unusual concerns.

6.4.5 Plastic bottles must be of a material that is impervious to attack from the sample. This is especially a consideration when using plastic for long term storage of certain petroleum products. Clear plastic bottles are unsuitable for samples sensitive to light.

6.4.6 When sampling aviation fuels, Practice D4306 should be consulted for guidance on container selection. This practice gives information on the types of containers that have been found satisfactory for tests to determine water separation, copper corrosion, electrical conductivity, thermal stability, lubricity, and trace metal content.

6.4.7 Appendix X1 is a guide for selecting the material of which sample containers may be made. It is impossible to cover all petroleum sampling container requirements; therefore, when questions arise as to a container's suitability for a given application, experience and testing should be relied upon.

6.5 *Container Closures:*

6.5.1 For glass bottles, stoppers or screw caps made of a material that will not deteriorate or contaminate the sample may be used. Care must be used when using cork stoppers. Situations where corks should not be used include liquids where loss of light ends may affect the test's results and liquids which are hydroscopic or which have a low water content specification. Rubber stoppers should never be used.

6.5.2 Cans and plastic bottles should be closed with screw caps made of the same material as the container. Caps should provide a vapor tight seal.

6.5.3 Screw caps for cans used to store or transport samples must be protected by a disk faced with a material that will not deteriorate or contaminate the sample. Consideration of closure type is important for samples where vapor loss will affect the test results.

6.6 *Federal Container Requirements* —In addition to the requirements listed above, any sample container that contains hazardous materials or the residue of hazardous material offered for shipment or transportation by air, public roadway, rail, or water, or any combination thereof, must meet the requirements set forth in applicable regulations such as DOT regulations in the Code of Federal Regulations, Title 49, Section 173.

6.7 *Container Cleanliness:*

6.7.1 Sample containers must be clean and free from all substances which might contaminate the material being sampled (such as water, dirt, lint, washing compounds, naphtha and other solvents, soldering fluxes, acids, rust, and oil). Prior to further use, reusable containers such as cans and bottles should be rinsed with a suitable solvent. Use of sludge solvents to remove all traces of sediments and sludge may be necessary. Following the solvent wash, the container should be washed with a strong soap solution, rinsed thoroughly with tap water, and given a final rinse using distilled water. Dry the container either by passing a current of clean warm air through the container or by placing it in a hot dust-free cabinet at 40°C (104°F) or higher. When dry, stopper or cap the container immediately. Normally, it is not necessary to wash new containers.

6.7.2 Depending on service, receivers used in conjunction with automatic samplers may need to be washed with solvent between uses. In most applications, it is not desirable or practical to wash these receivers using soap and water as outlined above for cans and bottles. The cleanliness and integrity of all sample containers/receivers must be verified prior to use.

6.7.3 When sampling aviation fuel, Practice D4306 should be consulted for recommended cleaning procedures for containers that are to be used in tests for determination of water separation, copper corrosion, electrical conductivity, thermal stability, lubricity, and trace metal content.

6.8 *Labels:*

6.8.1 Each sample container is to have a label attached to it which meets the requirements of the parties involved.

6.8.2 Fig. 1 is an example of a label which shows the typical information needed to properly identify the sample. In addition to this basic information, certain governmental agencies such as DOT and OSHA have additional labeling requirements with which personnel involved in the handling and shipping of samples must be familiar.

Sample Identification No.	
Product Name / Grade	
Terminal, Station or Lease	
Sampling Date and Time	
Gauger	

Type of Sample:

☐ All-Level	☐ Running
☐ Bottom	☐ RVP
☐ Clearance	☐ Top
☐ Composite	☐ UML
☐ Line	☐ 1–Foot
☐ Outlet	☐ Other: _____

Type of Sample:

- ☐ Barge Name
- ☐ Pipeline Batch No.
- ☐ Railcar No.
- ☐ Ship Name
- ☐ Tank No.
- ☐ Truck No.
- ☐ Other

Lab / Job Reference	
Date & Time in Lab	
Technician	

FIG. 1 Typical Sample Label

6.9 *Shipping Enclosures*—Many sample containers require special shipping enclosures before they can be transported from the point of collection. Regulations covering the transport of samples should be consulted (see the Code of Federal Regulations, Title 49, Section 173).

6.10 *Storage and Disposal:*

6.10.1 Except when being transferred, samples should be maintained in a closed container in order to prevent loss of light components. Samples should be protected during storage to prevent weathering or degradation from light, heat, or other potential detrimental conditions.

6.10.2 There are many governmental agencies and jurisdictions that have regulations governing the storage and disposal of petroleum samples and containers that can be classified as hazardous materials or hazardous wastes. Those who handle petroleum samples must be familiar with these regulations in addition to their own company policies and procedures.

7. Handling and Mixing Samples

7.1 *General Considerations:*

7.1.1 It is preferable that analytical tests be conducted using test specimens which have been drawn directly from the primary container. However, it is recognized that all sampling methods do not permit this nor do requirements to transport and store samples. The number of transfers using intermediate containers between the initial sampling operation and the analytical test should be minimized. Each use of intermediate containers increases the potential for loss of light hydrocarbons, loss of water due to clingage, or inefficient mixing and contamination of the sample from external sources including weather.

7.1.2 Before a sample is transferred from one container to another, a homogeneous mix must be created and maintained until the transfer is completed.

7.1.3 If the sampling procedure requires that multiple samples be taken from a single tank, or in the case of marine vessels, multiple or single samples from multiple tanks, analytical tests may be performed on each sample or on a composite of the various samples. When analytical tests are performed on individual samples, which is the recommended procedure, the test results are generally averaged. Depending on the particular application, the results may be averaged arithmetically or on a volumetrically proportional basis according to the proportion of the total petroleum which the sample represents.

7.2 *Composite Samples:*

7.2.1 A composite sample may be prepared from individual samples taken from the same tank or, in the case of marine vessels, all tanks that contain the same material. When a composite is required, it must consist of proportional parts from each zone if it is for a single tank. If the composite is for multiple tanks, it must consist of proportional parts from each tank sampled.

7.2.2 Composites normally can be made best in the laboratory. Therefore, samples to be composited should be submitted to the laboratory along with a list of each tank and the volume represented by each sample. The method of compositing should be documented and care taken to preserve the integrity and representativeness of the composite sample.

7.2.3 Making composite samples which will be tested for both density and water or sediment content are especially difficult; the mixing which is necessary prior to compositing for the water or sediment tests can result in loss of light ends which could affect results of the density test.

7.2.4 It is recommended that a portion of each individual sample used in a composite be retained separately (not composited) for retesting if necessary.

7.3 *Other Mixing Protocol*—The guidelines herein are intended to cover most sample handling and mixing requirements and should be used for analytical tests unless determined to be unacceptable for a specific application.

8. Sample Mixing Methods

8.1 Sample mixing methods can be divided into three general categories of power mixing, shaking, and no mixing. These categories vary greatly in severity depending on the type of analytical test to be conducted and the characteristics of the sample. The following is a brief discussion of each category:

8.1.1 *Power Mixers:*

8.1.1.1 Power mixers fall into two general groups of insertion or closed loop. Annex A2 gives the acceptance test criteria for power mixers prior to use. Sample container/mixer systems do not have to be tested individually if they are of the same design and operate within the demonstrated service range (that is, water concentration, viscosity of product, and sample volume).

8.1.1.2 Over-mixing with power mixers may create an oil and water emulsion that will affect the accuracy of certain analytical tests. Power mixers may entrain air into the sample that could affect certain analytical tests. Loss of vapor normally associated with rise in temperature may also occur which could affect tests results for water, RVP, and density.

8.1.1.3 *Insertion Mixers*—These mixers are stand-alone devices that are not an integral part of a given sampling or mixing system. These mixers can be used on a variety of different types and sizes of sample containers. Non-aerating or high-speed shear mixers are examples of insertion mixers. Insertion mixers may also be of a circulating loop design where a suction port is inserted into the sample container and the sample is circulated externally by means of a pump through a static mixer and discharged back into the sample container through a dispersal system. Annex A2 details the acceptance tests for insertion mixers.

8.1.1.4 *Closed Loop Mixers*—These mixers are typically used in conjunction with an automatic pipeline sampling system. The mixer may be an integral part of a stationary sample receiver or a stand-alone unit used for portable sample receivers. Annex A3 gives the acceptance testing for closed loop mixing systems.

8.1.2 *Shaking*—Shaking involves manually or mechanically shaking the sample container to eliminate stratification.

8.1.3 *None (no mixing)*—If a sample is known to be homogeneous, no mixing is required. Samples should not be mixed where the analytical tests to be conducted may be affected by air which could be induced by power mixing or shaking.

9. Selection of Sample Mixing Method

9.1 Table 1 lists the recommended mixing procedure to be used before a sample is transferred from a container. The degree of mixing depends on the type of transfer being made, the analytical test to be conducted and the characteristics of the sample. General guidelines are given in 9.1.1 – 9.1.3.

TABLE 1 Summary of Recommended Mixing Procedures

NOTE 1—Refer to specific analytical test procedure.

NOTE 2—Example: Static sample removed from a storage tank; that is, thief to analytical glassware, at time of sampling.

Test Purpose	Recommended Mixing Procedure		
	Power	Shaking	None
Sample transferred from container			
Density for crude and heavy fuels	X		
Sediment and water	X		
Density for other hydrocarbons		X	
Vapor pressure			X
Cloud point			X
Other tests	Note 1	Note 1	Note 1
Sample transferred from extracting device to analytical device			
All tests (Note 2)			X

9.1.1 Power mixing is required for all crude oil samples to be tested for sediment and water or density. Power mixing is also required when the sample has been transported or stored in either a primary or intermediate container.

9.1.2 No mixing is required if a crude oil sample is transferred from the extracting device to the analytical test device at the time of extraction. However, when such a sample is stored or transported in the extracting device, mixing is required.

9.1.3 Unless the specific procedure prohibits shaking, all other samples should be shaken with the exception of those to be tested for vapor pressure and cloud point.

10. Keywords

10.1 crude petroleum sampling; liquid petroleum sampling; sample containers; sample handling; sample mixing; sample preparation; sampling validation

ANNEXES

(Mandatory Information)

A1. ACCEPTANCE TEST CRITERIA FOR POWER MIXER AND SAMPLE CONTAINER COMBINATIONS

A1.1 Introduction

A1.1.1 Before a sample is transferred from one container to another, a homogeneous mix must be created and maintained until the transfer is completed. Various designs of power mixers can be used for this purpose as outlined in 8.1.1. Before its use, each power mixer design and sample container combination must be tested and proven to be effective. This annex presents the calculation of sample preparation precision, together with a sample calculation. The following annexes outline mixing procedure acceptance testing and present recommended forms for recording the results of such testing.

A1.2 Outline of Testing

A1.2.1 The test for proving the effectiveness of a power mixer and sample container combination begins with placing known amounts of water and oil in a container. Tests are then conducted to see if analytical water test results agree with the known baseline water plus the known water added without affecting density of the total mixture by loss of light ends.

A1.2.2 The acceptance test requires that each mixer/container combination be tested under the following conditions which the system will be operated:

A1.2.2.1 The normal low and high water content.

A1.2.2.2 Liquids that represent the normal or extremes in viscosity. For multi-fluid applications, two fluids should be tested that represent extremes in viscosities.

A1.2.2.3 The normal minimum and maximum expected sample volume.

A1.2.3 The overall testing process is illustrated in the flow chart, Fig. A1.1.

A1.3 Repeatability and Bias Calculations

A1.3.1 During each test run, three test specimens are to be drawn for each time interval being tested. Acceptance criteria for each test run is twofold. First, there must be repeatability between the three test specimens. Second, the system must be shown to be effective or free of bias. Table A1.1 lists the maximum permissible differences between test specimens as well as the maximum permissible differences between the average of all test specimens and total water concentration (bias). The equations on which Table A1.1 is based are as follows.

A1.3.2 The equation for the maximum permissible variation between test specimens (repeatability check) follows:

$$W_r \leq \text{the larger of } 0.05 \text{ or } K\,\sigma sys\,(\%) \tag{A1.1}$$

TABLE A1.1 Maximum Permissible Difference Between Test Specimens and Maximum Permissible Difference Between the Average of All Test Specimens and Total Water Concentration (Based on Three Test Specimens)

NOTE 1—Values in Column A are calculated from the larger of 0.05 % or $2.92 \times 0.064\ (W_k)^{0.5}$. Values in Column B are calculated from the larger of

$$0.05\ \%\ or\ 1.96 \times 0.064\ (W_k)^{0.5/\sqrt{3}}$$

Values of W_k not shown in the table may be obtained by interpolation.

NOTE 2—In developing this practice, the working group found that data available to make reasonable estimates of the expected variability between multiple test specimens during a single test run and the overall efficiency of the system to be limited. Eq A1.1 and Eq A1.2 have been derived from the available data. It is felt that the data is sufficient to provide a reasonable guideline for the industry at this time. It is hoped that by the publication of this practice and industry's use of test report sheets as shown in Attachments A and B that the data base can be expanded for possible refinement of these equations in future revisions.

Total Water Concentration (W_k) (%)	Column A Repeatability Check	Column B Bias Check
	Maximum Permissible Difference Between Test Specimens (%)	Maximum Permissible Difference Between Average of all Test Specimens and Total Water Concentration (%)
0.10	0.06	0.05
0.15	0.07	0.05
0.20	0.08	0.05
0.25	0.09	0.05
0.50	0.13	0.05
1.00	0.19	0.07
1.50	0.23	0.09
2.00	0.27	0.10
2.50	0.29	0.11
3.00	0.32	0.13
3.50	0.35	0.14
4.00	0.37	0.14
4.50	0.40	0.15
5.00	0.42	0.16

where:

W_r = W_t max – W_t min (%),
W_t = weight or volume % of individual test specimens,
k = 2.92 (valid only for three test specimens),
W_k = total water content, baseline + added water (%), and
σsys = $0.064\ (W_k)^{0.5}$.

A1.3.2.1 Another expression of Eq A1.1 is:

$$W_t \max - W_t \min \le \max.05 \text{ or } 2.92 \times 0.064\ (W_k)^{0.5} \tag{A1.2}$$

A1.3.3 To establish when the average of three test specimens is acceptable or the bias is suitably small, use Eq A1.3 and Eq A1.4.

$$W_{avg} \ge W_k - 1.96 \frac{\sigma sys}{\sqrt{n}} \tag{A1.3}$$

and

$$W_{avg} \le W_k + 1.96 \frac{\sigma sys}{\sqrt{n}} \tag{A1.4}$$

where:

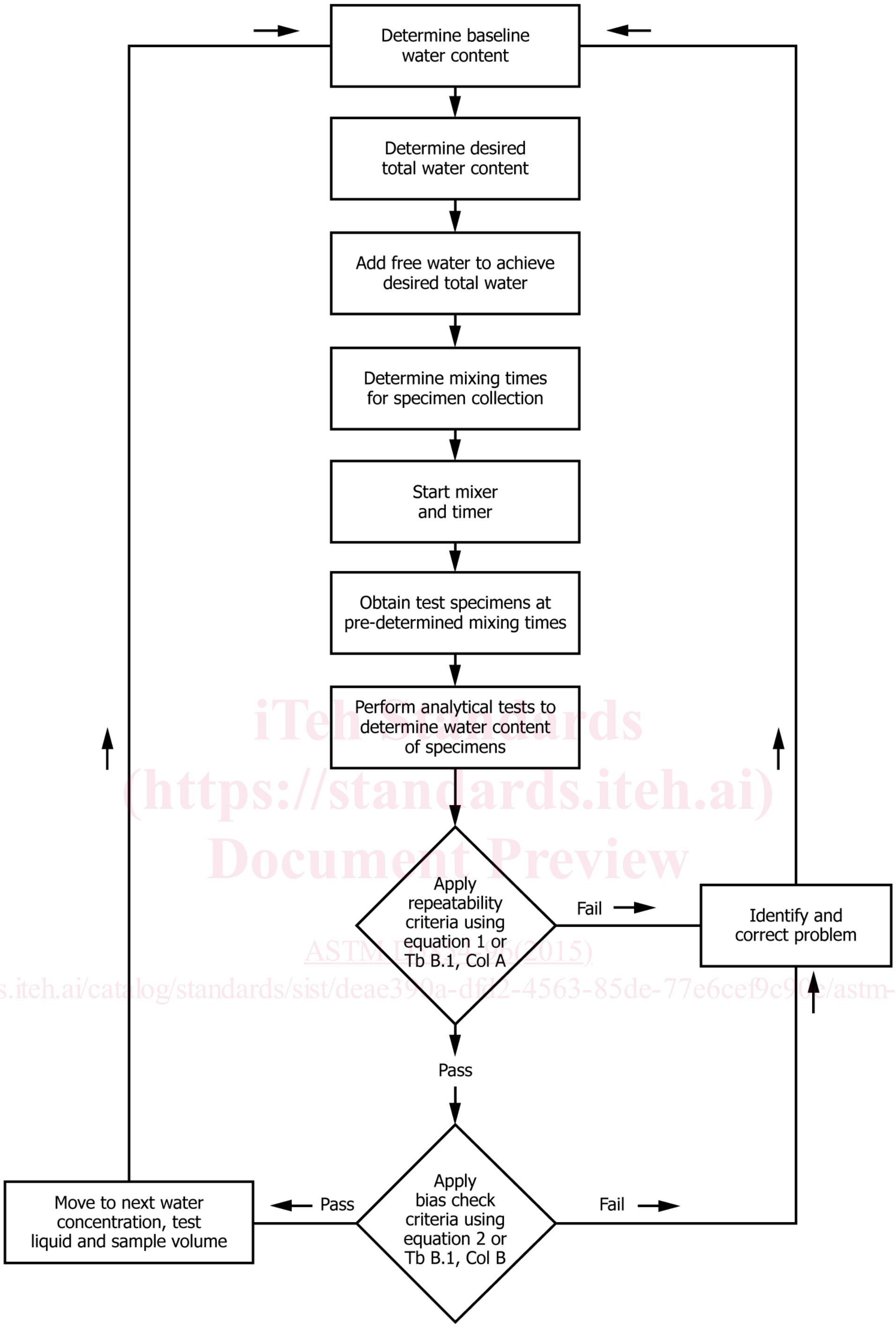

FIG. A1.1 Flow Chart of Power Mixer and Sample Container Acceptance Test

$$W_{avg} = \frac{\sum_{0}^{n} W_t}{n}$$

and n = 3 (number of test specimens).

A1.3.4 The following is a sample calculation for an acceptance test of a power mixer and sample container combination when:
Baseline water concentration = 0.10 %,
Total water concentration, W_k = 1.00 % (0.10 % baseline water plus 0.90 % added water), and
Test three results of W_1 = 0.98 %, W_2 = 1.05 %, W_3 = 1.07 %.

A1.3.4.1 *Step 1*—Determine if repeatability is acceptable for the total water concentration as given in Table A1.1, Column A.
(W_t max − W_t min) ≤ Table A1.1, Column A
$W_3 - W_1 = 1.07 - 0.98 = 0.09$
(*1*) From Table A1.1, Column A and line 1.00 % the maximum allowable difference = 0.19.
(*2*) Because 0.09 ≤ 0.19, the repeatability is acceptable.

A1.3.4.2 *Step 2*—Determine if system bias is acceptable.
(*1*) From Table A1.1, Column B at W_k of 1.00 %, the value of $1.96\,\sigma sys/\sqrt{3}=0.07$ %.
Then:

$$W_k - \frac{1.96\,\sigma sys}{\sqrt{3}} \le W_{avg} \le W_k + \frac{1.96\,\sigma sys}{\sqrt{3}}\,(1.00 - 0.07) \tag{A1.5}$$

$$\le \frac{0.98+1.05+1.07}{3} \le (1.00+0.07) = 0.93$$

(*2*) Because 0.93 ≤ 1.03 ≤ 1.07, the bias is acceptable.

A1.3.4.3 *Step 3*—If repeatability and system bias are acceptable, test next water concentration, another liquid, or sample volume. If repeatability or system bias is not acceptable, identify and correct the problem and then proceed with re-testing.

A2. ACCEPTANCE TEST FOR INSERTION MIXERS

A2.1 Introduction

A2.1.1 The ability of each mixer to create a homogeneous mixture in a given sample container must be evaluated before it is used. In the case of insertion mixers, each mixer must be reevaluated for any change in type of petroleum liquid, volume in the sample container, type of sample container, change in mixing conditions such as mixing speed or mixing time and increase in free water level.

A2.1.2 The following test procedure is based on Test Method D4928 (API *MPMS* Chapter 10.9), the Karl Fischer coulometric mass method for determining water content. Other water test methods are acceptable. The volume of test specimen will therefore need to be adjusted accordingly, if the centrifuge or distillation methods are used. Regardless of the test method used for water in the acceptance test, it is recommended that the acceptance test results be validated using the water test method normally used to determine water content.

A2.1.3 It is recommended that forms such as Fig. A2.1 or Fig. A2.2 be completed and maintained on file for each test conducted.

A2.2 Baseline Water Determination

A2.2.1 Weigh an empty sample container to the nearest ~~0.01 g.~~ 0.01 g. Fill the container to the selected level with petroleum liquid. The petroleum liquid used in the acceptance tests should contain no free water.

A2.2.2 Immerse the mixer head or suction port into the petroleum liquid to a point about ~~1~~1 mm to ~~2 mm~~ 2 mm (1⁄16 ~~in.)~~ in.) above the bottom of the container and mix the petroleum liquid at the speed and for the duration expected to be used in normal operation. Suggested mixing time for variable speed mixers is ~~1~~1 min to ~~5 min~~ 5 min at the manufacturer's suggested speed. The suggested mixing time for constant speed circulation mixers is ~~5 min.~~ 5 min. (For analytical tests using volumetrics, non-aerating shear mixers should be used.)

A2.2.3 Immediately after mixing, determine the water content based on three test specimens. Calculate the average water content to the nearest 0.01 %.

Date _____ Location _____ Technician _____ Test No. _____

Mixer _____ Product _____ Sample Container _____

Speed _____ Mixing Time _____ Ice Bath Required _____

Position of Mixer Head, Suction/Discharge Ports _____

Test Conditions: ❒ Low Water Level ❒ Low Viscosity Liquid ❒ Low Sample Volume

❒ High Water Level ❒ High Viscosity Liquid ❒ High Sample Volume

I. Baseline Water Content of Petroleum Liquid

Test Specimen	Weight of Test Specimen (g)	μg Water	(μg/g) × 10000
1			
2			
3			
		Average Baseline Water, % Mass (a)	

II. Calculated % Water in Sample

Wt. of Sample Container & Petroleum Liquid	(1)	_____ g
Wt. of Empty Container	(2)	_____ g
Wt. of Petroleum Liquid (1) – (2)	(3)	_____ g
Wt. of Water Added	(4)	_____ g
Total Wt. of Sample (3) + (4)	(5)	_____ g
Wt. of Baseline Water [(3) × (a)]/100	(6)	_____ g
Wt. of Water Added (4)	(7)	_____ g
Total Wt. of Water Present (6) + (7)	(8)	_____ g
Calculated % Water = $\frac{(8)}{(5)} \times 100$		_____ % Mass (W_k)

III. Test Runs

Test Specimen (W_t)	Weight of Test Specimen (g)	μg Water	(μg/g) × 10000
1			
2			
3			
		Average Baseline plus Known Water, % Mass (W_{avg})	

FIG. A2.1 Acceptance Test Data Sheet for Insertion Mixers (Gravimetrically Using Coulometric Karl Fischer)

A2.3 Test for Known Water Level

A2.3.1 Weigh the petroleum liquid and container.

A2.3.2 Knowing the weight and baseline water content of the petroleum liquid, add enough water to increase the water content of the *dry* petroleum liquid to the preselected concentration. To add water to sample volumes less than ~~1 L,~~ 1 L, use a syringe. It is preferable to use a needle that will reach to the bottom of the container. The needle should be wiped free of water or petroleum liquid before each weighing. A beaker may be used to add water to sample containers larger than ~~1 qt.~~1 qt.

A2.3.3 Calculate the percent mass of water in the sample container giving consideration to:

A2.3.3.1 Baseline water found in A2.2 and