Designation: D7453 – 09

Standard Practice for Sampling of Petroleum Products for Analysis by Process Stream Analyzers and for Process Stream Analyzer System Validation[1]

This standard is issued under the fixed designation D7453; 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.

INTRODUCTION

The primary focus of sampling petroleum product is the timely presentation of the sample for *(1)* analysis by online analyzers, *(2)* validation of an analyzer system and *(3)* collecting a composite sample for batch physical property determination. Sediment, free water, rust, and other contaminants found in the sample may be removed in the sample conditioning system to protect the hardware and analytical systems. If a sample is being collected for later analysis, the sample receiver must not alter or degrade the physical make up of the sample in any way. If a sample is being feed to an analyzer or sampled for latter determination of water or particulate contamination then filtering is not an option.

1. Scope

1.1 This practice covers the performance requirements for sample systems employed to deliver process stream samples *(1)* to analyzer system for analyses or *(2)* for analyzer validation or *(3)* for composite sample systems. It also outlines the selection and operation of line or batch sampling equipment intended for analyzer flow proportioned average property value system validation. Sample handling, mixing, and conditioning procedures are required to ensure that a representative sample of the liquid petroleum product is collected from the sampling source.

1.2 *Applicable Fluids*—This practice is applicable to single liquid phase petroleum products whose vapor pressure at sampling and sample storage conditions is less than or equal to 110 kPa (16.0 psi), and, with a D86 final boiling point less than or equal to 400°C (752°F).

1.2.1 Specialized sample handling may be necessary to maintain sample integrity of more volatile materials at high temperatures or extended residence time in the receiver. Such handling requirements are not within the scope of this practice. Users should consult the analytical methods to be performed on the sample for special sample storage or conditioning requirements.

1.3 Some or all of the processes outlined in this practice may be applicable to other liquids. Applying this practice to other liquids will require the consideration of additional methods and practices. It is the responsibility of the user of this standard to identify any and all applicable safety and sampling considerations and establish appropriate procedures to handle these additional considerations.

1.4 The values stated in SI units are to be regarded as the standard. The values given in parentheses are for information only.

1.5 *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]

D3764 Practice for Validation of the Performance of Process Stream Analyzer Systems

D6122 Practice for Validation of the Performance of Multivariate Process Infrared Spectrophotometer Based Analyzer Systems

D6624 Practice for Determining a Flow-Proportioned Average Property Value (FPAPV) for a Collected Batch of Process Stream Material Using Stream Analyzer Data

D7278 Guide for Prediction of Analyzer Sample System Lag Times

[1] This practice is under the jurisdiction of ASTM Committee D02 on Petroleum Products and Lubricants and is the direct responsibility of Subcommittee D02.25 on Performance Assessment and Validation of Process Stream Analyzer Systems.

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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. Terminology

3.1 *Definitions:*

3.1.1 *analyzer unit response time*, *n*—time interval between the introduction of a step change in property characteristic at the inlet of the analyzer unit and when the analyzer output indicates a value corresponding to 99.5% of the subsequent change in analyzer results.

3.1.2 *automatic sampler*, *n*—device used to repetitively extract an grab and collect a representative sample of a batch or process stream.

3.1.3 *automatic sampling system*, *n*—system consisting of a sample probe, sample fast cycle loop, sample supply line stream conditioning, an automatic sampler and an associated controller, a flow measuring device, and sample holding, mixing and handling capabilities.

3.1.4 *batch*, *n*—term referring to a volume or parcel being transferred.

3.1.5 *flow proportional sampler*, *n*—sampler designed to automatically adjust the sampling rate to be proportional to the flow rate of the stream.

3.1.6 *grab*, *n*—volume of sample extracted from a batch by a single actuation of the sample extractor.

3.1.7 *lag time*, *n*—time required for material to travel from point A to point B in the total analyzer system (points A and B are user-defined).

3.1.8 *line sample*, *n*—process material that can be safely withdrawn from a sample port and associated facilities located anywhere in the total analyzer system without significantly altering the property of interest.

3.1.9 *primary test method (PTM)*, *n*—ASTM or other established standard test method that produces results accepted as the reference measure of a property.

3.1.10 *sample conditioning unit lag time*, *n*—time required for material to flow from the sample conditioning unit inlet to the analyzer unit inlet.

3.1.11 *sample fast cycle loop*, *n*—a system that continually and rapidly transports a representative sample of process material from the sample probe past the sample supply line and returns the remaining material to the process.

3.1.11.1 *sample fast loop lag time, n*—time required for material to transport from the product takeoff point of the sample loop to the sample conditioning unit inlet.

3.1.12 *total analyzer system response time*, *n*—time interval between the when a step change in property characteristic arrives at the sample loop inlet and when the analyzer output indicates a value corresponding to 99.5% of the subsequent change in analyzer results.

3.1.12.1 *Discussion*—The total analyzer system response time is the sum of the sample fast loop lag time, the sample conditioning unit lag time, and the analyzer unit response time.

3.1.13 *validation*, *n*—statistically quantified judgment that the analyzer system or subsystem being assessed can produce predicted PTM results with acceptable precision and bias performance when compared to actual results from a primary test method measurement system for common materials.

4. Summary of Practice

4.1 Analyzer measurement systems require a process sample that is delivered in a timely manner commensurate with the analyzer and process cycle time at pressure, temperature and flow conditions meeting system requirements, is free of contaminants, and is representative of the process stream.

4.2 Line samples collected from the process or blender stream need to accurately reflect the composition of the analyzer feed stream. This is accomplished by taking into account the total analyzer system response time in order to properly validate online analyzer systems.

4.3 This practice describes functional requirements that need to be addressed in the design and operation of automatic sampling equipment. Automatic sampling equipment is used to obtain a representative batch sample for use in validating an analyzer system or flow proportioned average property value and for manufactured batch quality testing.

5. Significance and Use

5.1 Analyzer systems require representative samples of petroleum products delivered in a timely manner to *(1)* facilitate the control of process or blending units or *(2)* calculate a flow proportioned property value.

5.2 Representative samples of petroleum products are required for the determination of chemical and physical properties. These properties are used to establish the relationship between the analyzer system and the primary test method during initial and ongoing validation of the system.

5.3 Representative samples of petroleum products are tested to determine the chemical and physical properties of a batch offered for tender.

6. Sample Delivery and Conditioning Requirements for Process Stream Analyzers

6.1 The sample will be delivered from the sample stream to the analyzer inlet for measurement in the minimum realistic period of time possible.

6.1.1 When sampling from processes that normally operate in steady state mode, not subject to scheduled operational variable(s) step changes that directly impact the measured variable, the sample fast loop lag time shall be as short as practically possible. It is recommended that where possible, the sample fast loop lag time should be less than the analyzer response time. A minimum realistic time is two minutes.

NOTE 1—Guide D7278 can be used for the prediction of analyzer sample system lag times. Refer to Practice D3764 for analyzer unit response time information.

6.1.2 Sampling from processes that are subject to scheduled operational variable(s) step changes that directly impact the measured variable requires knowledge of the shortest interval between scheduled step changes. The total analyzer system response time shall be less than the shortest interval between scheduled step changes

6.2 The sample system shall deliver the sample to the analyzer without alteration of the properties of interest.

6.2.1 The sample systems shall not cause any unintended phase changes in the sample during its transport to the analyzer.

6.3 *Filtration and Coalescing*—The sample stream should be filtered or suitably treated to remove contaminants such as rust, sediments, and foreign matter. Free and dissolved water not intended to be quantified or analyzed should be removed by coalescing, chilling, or filtration. The porosity of the filter should be selected for effective removal of contaminants that can cause immediate or long term damage to the system hardware including build up and plugging of system solenoids and valves.

6.4 *Temperature and Pressure*—Sample temperature and pressure shall be conditioned to a safe working range as determined by the sample handling requirements and equipment limitations. There shall be no bubbling or frothing during sampling.

6.4.1 Some primary test methods require storing the sample within a prescribed temperature range. These requirements need to be identified and addressed in the sampler design.

7. Line Sample Requirements

7.1 Where possible, the line sample point should in close proximity to the process takeoff point of the sample loop.

NOTE 2—Filters, coalescers, and temperature conditioning units may be required to make the sample stream safe to sample.

7.2 Validate and document the lag time of the line sample point from the sample stream and the lag time from the sample point to the analyzer inlet. This data is required in Practice D3764 or Practice D6122 for analyzer validation. Guide D7278 may also be used.

7.3 Use a sample container and any sample container conditioning procedures defined in the PTM.

7.3.1 The sample point shall be flushed with three times the volume of the sample system from the tie into the analyzer sample supply to the sample outlet. See Fig. 1.

7.3.2 The sample probe shall reach the bottom of the sample container when sampling.

7.3.2.1 The bottom of the sample probe shall have a 45° relief cut.

7.3.3 Flush the sample container to remove contaminants and saturate the vapor phase with hydrocarbons.

NOTE 3—Samples to be analyzed for vapor pressure require special handling, and the appropriate standard test method should be reviewed for requirements.

7.3.4 After a container has been flushed with sample, it should be filled immediately so the vapor space in the container stays fully saturated with hydrocarbon and the bottle temperature is similar to the sample.

7.3.5 Throttle the flow into the sample container at the start of the filling process so the sample is introduced slowly, without turbulence, until there is enough volume so that increasing the flow does not cause bubbling or frothing. If the initial fill rate is too fast, properly dispose of the extracted sample and then refill the sample container to maintain sample integrity.

7.3.6 Seal the sample container and label as required.

NOTE 4—The analyzer reading may need to be documented at the time of filling for validation samples.

8. Automatic Sampling systems

8.1 An automatic composite sample collecting system consists of sample conditioning upstream of the sampling location, a device to physically extract a grab from the sample loop, a flow measurement device for flow proportioning, a means to control the total volume of sample extracted, a sample receiver to collect and store the grabs and, depending on the system, a sample receiver/mixing system. See Fig. 2. Unique properties of the petroleum product being sampled may require the individual components or the entire system be insulated, or heated, or both. Since the sample is collected throughout a full

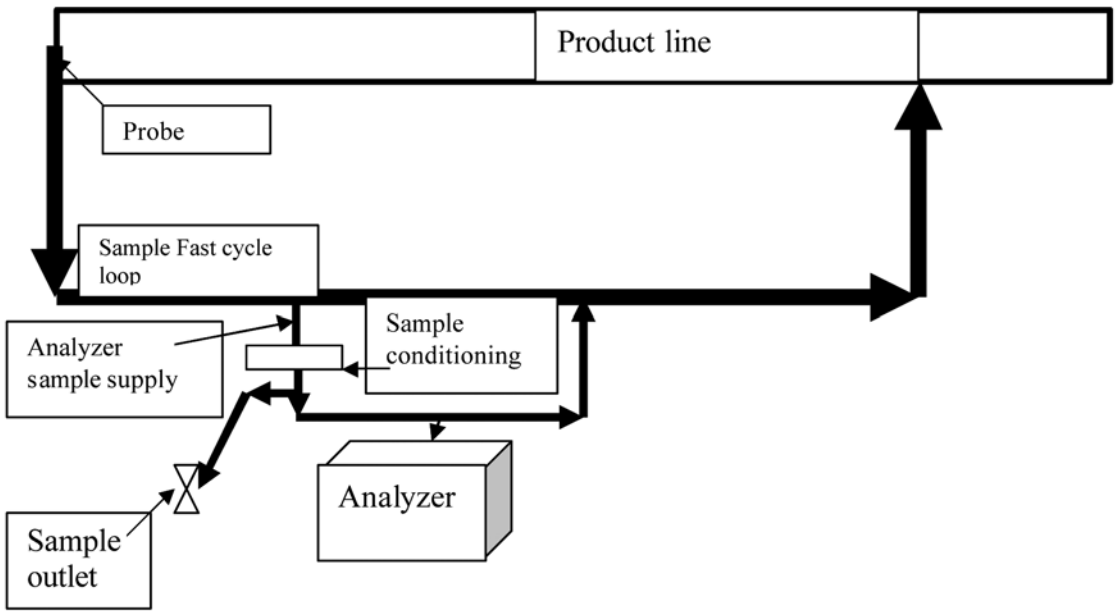

FIG. 1 Diagram Line Sample

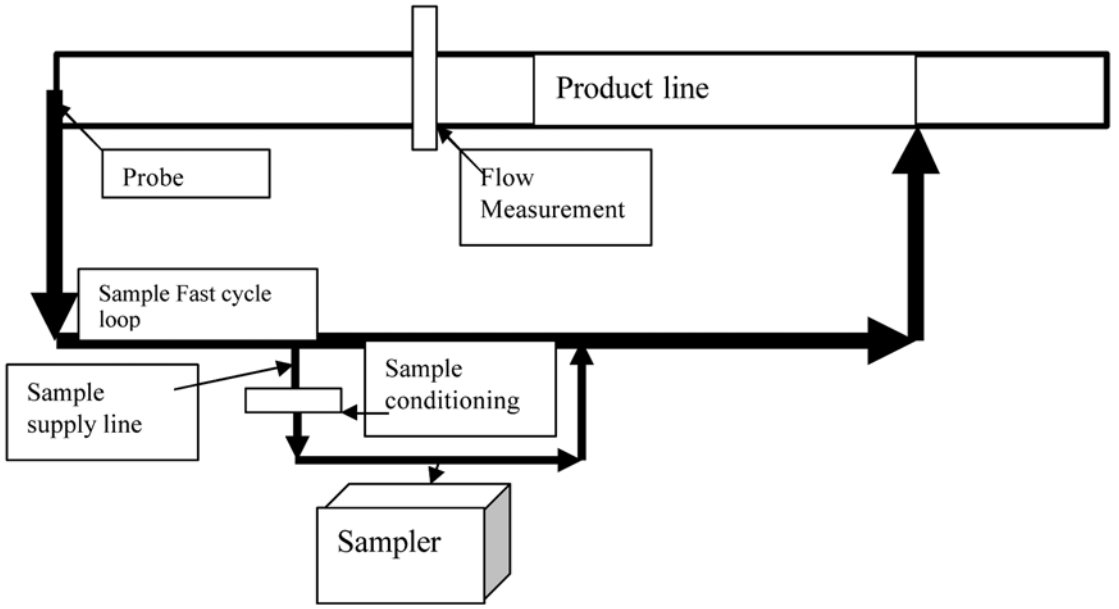

FIG. 2 Diagram Automatic Sampler Layout

batch, the analysis of this sample can be used to validate the analyzer flow proportioned average property value calculated by Practice D6624.

9. Procedure

9.1 *Sampling Frequency*:

9.1.1 The sample shall be extracted in a flow proportioned manner

9.1.2 The grab volume shall be consistent throughout the batch.

9.1.2.1 The variation between the maximum and minimum grab volume will not exceed 10% of the set grab volume.

9.1.3 The batch will be sampled at a minimum of once every 16 cubic metres (100 barrels) of sample stream flow past the sample probe.

9.1.4 Collect the maximum number of grabs from the batch that the equipment's extraction frequency, extraction grab size, and volume limitations will allow. Increasing the number of grabs will make the composite sample more representative of the total batch.

9.1.4.1 The minimum sample receiver volume is determined by the sample container requirements of the PTM used to analyze the sample.

9.1.4.2 The optimum sampling frequency is the maximum number of grabs which may be obtained from any parcel operating within the extraction frequency and grab volume limitations of the equipment.

9.1.5 The maximum sampling frequency will not exceed the capability of the sampling hardware.

9.2 *Sampling Systems*:

9.2.1 Select a sample storage device that will not affect the properties of interest over the period that the sample will be stored in the storage device.

9.2.2 Flush the sample system lines and sample receiver at the start of each batch.

9.2.3 Confirm that the accumulator is empty and clean before the start of sampling.

9.2.4 The following items need to be monitored while the accumulator is filling:

9.2.4.1 The fill rate at a minimum of 10% intervals of the expected sample volume so as to confirm operation and sample representativeness.

9.2.4.2 The sampling frequency has not exceeded the hardware capability.

9.2.4.3 The flow through the sample supply line to the sampler is maintained at the documented flow rates for acceptable lag times.

9.2.4.4 The flow through the sample fast cycle loop is maintained at the documented flow rates for acceptable lag times.

9.2.5 The accumulator shall be thoroughly mixed after the batch has been completed prior to any volume removal.

9.2.5.1 Sample volume removed from the accumulator into sample containers shall be done in compliance with the line sample requirements in 7.3.

10. Probes

10.1 *Probe Location*:

10.1.1 The recommended sampling area is approximately the center one-third of the pipeline cross-section area.

10.1.2 The probe shall be located in a zone where sufficient mixing results in adequate stream conditioning. This zone is generally from 3 to 10 diameters downstream of piping elements and at least 1 diameter upstream of piping elements. When static or power mixers are used, the manufacturer of the device should be consulted for the probe's optimum location.

10.1.3 The preferred installation of a probe is in the horizontal plane.

10.2 *Probe Design*:

10.2.1 The mechanical design of the probe should be compatible with the operating conditions of the pipeline and the fluid being sampled.

10.2.2 Probe designs commonly used are described as follows:

10.2.2.1 A closed end probe equipped with an open orifice.

10.2.2.2 A short-radius elbow or pipe bend.

10.2.2.3 A tube cut at a 45° angle.

10.3 The orientation of the probe opening can be away from the flow to minimize the pickup of particulate contamination.

11. Keywords

11.1 analyzer sampling; composite sampler; line sample