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An American National Standard

Standard Practice for Emulsification/Suspension of Multiphase Fluid Waste Materials[1]

This standard is issued under the fixed designation D 5761; 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 generation of a single-phase suspension or emulsion from multiphase samples which are primarily liquid in order to facilitate sample preparation, transfer, and analysis.

1.2 This practice is designed to keep a multiphase fluid sample in an emulsified/suspended state long enough to take a single, composite sample that is representative of the sample as a whole. The sample may reform multiple layers after standing.

1.3 The emulsion/suspension generated by following this practice can be used only for analytical procedures designed for the total sample and procedures not significantly affected by the emulsifier or the presence of an emulsion/suspension.

1.4 This practice assumes that a representative sample of not more than one litre has been obtained.

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:*

D 4057 Practice for Manual Sampling of Petroleum and Petroleum Products[2]

D 4177 Practice for Automatic Sampling of Petroleum and Petroleum Products[2]

2.2 *EPA Standard:*

SW846 Test Methods for Evaluating Solid Waste, Physical/Chemical Methods[3]

3. Terminology

3.1 *Definitions:*

3.1.1 *emulsion*, *n*—a suspension of fine particles or globules, or both, of one or more liquids in another liquid.

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *multiphase fluid waste material*, *n*—a substance or mixture of chemicals that is no longer useful for its original purpose that visibly involves a solid and at least one liquid phase or more than one liquid phase without any solid present.

4. Summary of Practice

4.1 An emulsifier is added and mixed well with a sample of multiphase fluid waste material, to produce a uniform mixture suitable for subsequent aliquoting. A satisfactory homogenization has been attained when the sample appears to remain as a single phase for 30 s or longer.

4.2 A calcium sulfonate emulsifier is used when the original sample is primarily organic in nature; a polyethylene glycol monoalky ester is used when the original sample is primarily aqueous in nature. A blend of the two emulsifiers is used when the original sample contains roughly equal volumes of organic and aqueous material.

5. Significance and Use

5.1 This practice is intended as a solution to the difficulty of obtaining reproducible test results from heterogeneous samples.

5.2 This practice works best with multilayered liquids, but can also be applied to samples with solid particles that are sufficiently small in size to be suspended in an emulsion.

5.3 The emulsified/suspended sample can be used for all bulk property testing such as microwave digestion/inductively coupled argon plasma (ICAP), ion chromatography, heat of combustion, ash content, water, nonvolatile residue, and pH. It may be prudent to retain a portion of the sample in its original, multiphase form for some types of analyses.

6. Interferences

6.1 Not all samples can be emulsified, due to varying chemical reactions with the surfactants. If the emulsion is not stable for at least 30 s after shaking, it may not be suitable for testing as an emulsion.

6.2 Due to their physical composition, some samples are not suitable for splitting and, as a result, cannot be emulsified if a nonemulsified retain is required. For example, excessive amounts of solids and semisolids or tars do not permit splitting.

[1] This practice is under the jurisdiction of ASTM Committee D02 on Petroleum Products and Lubricants and is the direct responsibility of Subcommittee D02.03 on Elemental Analysis.

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[2] *Annual Book of ASTM Standards*, Vol 05.02.

[3] Available from the Environmental Protection Agency, 401 M Street, SW, Washington, DC 20460.

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6.3 In some instances, the amount of sample submitted may not be sufficient for splitting and, as a result, cannot be emulsified if some unemulsified sample must be retained.

6.4 Erroneous results can be obtained if precautions are not taken to avoid the loss of volatile material. Do not open containers unnecessarily. Results for samples from leaky containers must be marked to indicate that the sample integrity was not maintained during shipping/storage.

7. Apparatus

7.1 *Funnel, Splitting*—2-L separatory funnel fitted with twin 0.7-mm inside diameter glass discharge tubes as shown in Fig. 1.[4]

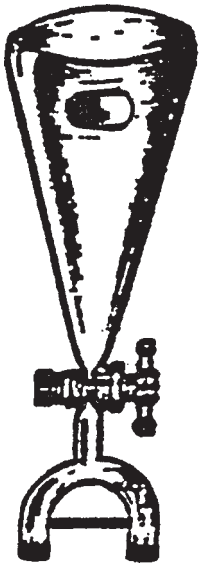

FIG. 1 Sample Splitter Funnel

7.2 *High-Intensity Lamp*.

7.3 *Polyethylene Transfer Pipet*, 3.5-mL draw.

8. Reagents and Materials

8.1 *Aqueous Emulsifier*—Polyethylene glycol monoalky ester, also known as PEG400.[5]

8.2 *Organic Emulsifier*—Calcium sulfonate (60 % weight) in heavy aromatic naphtha.[6]

9. Procedure

9.1 Representative samples are to be provided to the laboratory using standard techniques such as those described in Practice D 4057 or D 4177 and EPA/SW-846. It is assumed for purposes of this practice that the size of the sample in the laboratory is one litre or less. Adjustments in procedure will be necessary for larger volumes.

9.2 Vigorously shake multiphase samples by hand, or by mechanical means, for up to 30 s. If within 1 min after shaking, the sample appears to separate and each of the layers consists of at least 10 % by volume of the total, and if there is a sufficient sample available based on subsequent testing requirements, then the sample is a candidate for the emulsification procedure.

9.3 (Optional.) If some unemulsified sample is to be retained, split the sample into two separate samples using the splitter funnel shown in Fig. 1.

9.4 Tentatively classify any liquid phase, using information known about the sample, as organic or aqueous and determine their volume percentages relative to the whole sample. See Appendix X1 for an example worksheet on which to record the information.

9.4.1 As a guide to classifying a sample in a clear glass jar, measure phase height from the inside bottom of the jar, not from the bench top, using a ruler held up to the outer vertical side of the jar. Phase height shall be measured while looking straight and horizontally at the phase separation.

9.4.2 Use a high-intensity lamp to backlight the sample and provide better definition of phases. Short duration application of the lamp will not heat the sample.

9.5 Select an emulsifier. Select the organic emulsifier for samples which are greater than 60 % organic and the aqueous emulsifier for samples which are greater than 60 % water. For the multiphase samples where the organic layer is between 40 to 60 %, use an equal mixture of the emulsifiers (equal volumetric proportions).

9.6 Estimate the total volume of sample in millilitres. Multiply this number by 0.025 to determine the grams of emulsifier to add. (See the worksheet in Appendix X1.)

NOTE 1—An exact conversion of volume to mass is not necessary for this practice. The amount of surfactant added has been developed assuming this conversion. Record the information.

9.7 Place the sample on a balance reading to at least 0.1 g. Using a plastic transfer pipet or similar device, add the calculated quantity of the selected emulsifier to the sample. Shake as vigorously as possible until a milky homogeneous dispersion is formed.

9.8 If the emulsion does not stay together for at least 60 s, add approximately 1 g of the other surfactant.

9.9 Mix the emulsified sample thoroughly by hand shaking for 15 s immediately before each aliquot is removed.

10. Calculation

10.1 The following is an example of the determination of the amount of surfactant required:

Sample amount in container	400 mL (10.5 cm height)
Organic phase height	3.0 cm
Aqueous phase height	7.5 cm
Percent organic phase	29
Percent aqueous phase	71
Surfactant used	Aqueous (H-31A)

400 mL × 0.025 = 10 g of aqueous surfactant added to the sample.

11. Keywords

11.1 emulsification; heterogeneous sample; multiphase; sample preparation

[4] Available from Universal Instrument Co., 315 W. Colfax, Palatine, IL 60067 (708-991-3933) as Part No. F-4001.

[5] Available from Witco Corp. as Witconol H31.

[6] Available from Witco Corp., 3200 Brookfield, Houston, TX 77045-2817 (1-800-231-1542) as Witconate 605A.

APPENDIX

(Nonmandatory Information)

X1. EMULSIFICATION WORKSHEET

X1.1 Fig. X1.1 is a sample emulsification worksheet.

Sample Number	:			Sample Number	:		
Sample Amount	:		mL	Sample Amount	:		mL
Organic Height	:		cm	Organic Height	:		cm
Aqueous Height	:		cm	Aqueous Height	:		cm
Total Height	:		cm	Total Height	:		cm
Percent Organic	:			Percent Organic	:		
Percent Aqueous	:			Percent Aqueous	:		
Surfactant	:			Surfactant	:		
Surfactant Amount	:		g	Surfactant Amount	:		g
Analyst	:			Analyst	:		
Date	:			Date	:		
Sample Number	:			Sample Number	:		
Sample Amount	:		mL	Sample Amount	:		mL
Organic Height	:		cm	Organic Height	:		cm
Aqueous Height	:		cm	Aqueous Height	:		cm
Total Height	:		cm	Total Height	:		cm
Percent Organic	:			Percent Organic	:		
Percent Aqueous	:			Percent Aqueous	:		
Surfactant	:			Surfactant	:		
Surfactant Amount	:		g	Surfactant Amount	:		g
Analyst	:			Analyst	:		
Date	:			Date	:		
Sample Number	:			Sample Number	:		
Sample Amount	:		mL	Sample Amount	:		mL
Organic Height	:		cm	Organic Height	:		cm
Aqueous Height	:		cm	Aqueous Height	:		cm
Total Height	:		cm	Total Height	:		cm
Percent Organic	:			Percent Organic	:		
Percent Aqueous	:			Percent Aqueous	:		
Surfactant	:			Surfactant	:		
Surfactant Amount	:		g	Surfactant Amount	:		g
Analyst	:			Analyst	:		
Date	:			Date	:		

NOTE 1—Sample Amount (mL) × 0.025 = grams of surfactant to add [(organic height)/(organic + aqueous height)] × 100 = % difference organic [(aqueous height)/(organic + aqueous height)] × 100 = % difference aqueous.

FIG. X1.1 Emulsification Worksheet

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