Method 1662

Total Extractable Material in Drilling Mud by SDS Extraction and Gravimetry

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1. SCOPE AND APPLICATION

1.2 This method is designed to determine the oil content of drilling mud by Soxhlet/Dean-Stark (SDS) extraction and gravimetric measurement. However, this is a method-defined measurement that does not discriminate oil from other materials capable of being extracted from the mud. EPA Methods 1651, 1654, and 1663 can be used to aid in determining the presence and identity of oil.

1.2 This method is for use in the Environmental Protection Agency's (EPA's) survey and monitoring programs under the Federal Water Pollution Control Act.

1.3 The detection limit of this method is usually dependent on the level of background materials in the drilling mud rather than instrumental limitations. The level in Table I typifies the minimum level that can be detected with no interferences present.

1.4 Any modification of this method beyond those expressly permitted shall be considered a major modification subject to application and approval of alternative test procedures under 40 *CFR* 136.4 and 136.5.

1.5 Each analyst that uses this method must demonstrate the ability to generate acceptable results using the procedure in Section 8.2.

2. SUMMARY OF METHOD

2.1 Approximately 25 g of drilling mud is extracted with toluene in an SDS extractor (Reference 1). The extract is evaporated to dryness using a rotary evaporator and nitrogen blowdown apparatus. The weight of oil is determined using an analytical balance.

2.2 Quality is assured through reproducible calibration and testing of the extraction and gravimetric systems.

3. INTERFERENCES

3.1 Solvents, reagents, glassware, and other sample processing hardware may lead to discrete artifacts and elevated measurements causing misinterpretation of results.

3.2 All materials used in the analysis shall be demonstrated to be free from interferences by running method blanks initially and with each sample hatch (samples started through the extraction process at the same time, to a maximum of ten). Specific selection of reagents and purification of solvents by distillation in all-glass systems may he required.

3.3 Glassware and, where possible, reagents are cleaned by solvent rinse and/or baking at 200°C for a minimum of 1 hour.

4. SAFETY

4.1 The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined; however, each chemical should be treated as a potential health hazard. Exposure to these chemicals must be reduced to the lowest possible level.

4.2 The laboratory is responsible for maintaining a current awareness file of OSHA regulations regarding the safe handling of the chemicals specified in this method. A reference file of material safety data sheets (MSDSs) should be made available to all personnel involved in the chemical analysis. Additional information on laboratory safety can be found in References 2 through 4.

4.3 Methylene chloride has been classified as a known health hazard. All steps in this method which involve exposure to this compound shall be performed in an OSHA-approved fume hood.

5. APPARATUS AND MATERIALS

NOTE: Brand names, suppliers, and pan numbers are for illustrative purposes only. No endorsement is implied. Equivalent performance may be achieved using apparatus and materials other than those specified here, but demonstration of equivalent performance meeting the requirements of this method is the responsibility of the laboratory.

5.1 Sampling equipment for discrete sampling.

- 5.1.1 Sample bottle: Wide-mouth amber glass or opaque cosmetic jars, 100-mL minimum, with screw-cap. If amber bottles are not available, samples shall be protected from light.
- 5.1.2 Bottle caps: Threaded to fit sample bottles. Caps shall be lined with PTFE or aluminum.
- 5.1.3 Cleaning.
  - 5.1.3.1 Bottles: Detergent water wash, tap water rinse, cap with aluminum foil, and bake at 110 to 200°C for a minimum of 1 hour prior to use.
  - 5.1.3.2 Liners: Detergent wash, tap water and solvent rinse, and bake at 110 to 200°C for a minimum of 1 hour prior to use.
- 5.1.4 Bottles and liners must be lot-certified to be free of artifacts by running blanks according to this method. If blanks from bottles and/or liners without cleaning or with fewer cleaning steps than required above show no detectable materials (per Section 8.5), the bottle and liner cleaning steps that do not eliminate these artifacts may be omitted.

5.2 Equipment for glassware cleaning.

- 5.2.1 Laboratory sink with overhead fume hood.
- 5.2.2 Oven: Capable of reaching 200°C within 2 hours and holding 200°C within ± 10°C.

5.3 Equipment for sample preparation.
- 5.3.1 Laboratory fume hood.
- 5.3.2 Balances.
  - 5.3.2.1 Analytical: Capable of weighing 1.0 mg.
  - 5.3.2.2 Top loading: Capable of weighing 100 mg.
- 5.3.3 Beaker: 400- to 500-mL.
- 5.3.4 Spatula: Stainless steel.
- 5.3.5 Desiccator: Cabinet- or jar-type, capable of keeping the Kuderna-Danish concentrator tubes (Section 5.5.1.1) dry during cooling.

5.4 Soxhlet/Dean-Stark (SDS) extractor.
- 5.4.1 Soxhlet: 50 mm i.d., 200-mL capacity with 500-mL flask (Cal-Glass LG-6900, or equivalent, except substitute 500-mL round-bottom flask for 300-mL flat-bottom flask).
- 5.4.2 Thimble: 123 mm long by 43 mm i.d. to fit Soxhlet (Cal-Glass LG-6901-122, or equivalent).
- 5.4.3 Moisture trap: Dean-Stark or Barret with PTFE stopcock to fit Soxhlet (Figure 1).
- 5.4.4 Heating mantle: Hemispherical, to fit 500-mL round-bottom flask (Cal-Glass LG-8801-112, or equivalent).
- 5.4.5 Variable transformer: Powerstat (Cal-Glass LG-8965-100, or equivalent).

5.5 Concentration apparatus.
- 5.5.1 Rotary evaporator or other concentration device capable of evaporating toluene: Any concentration technique may be used provided that the requirements of Section 8.2 and the method detection limit in Table 1 are met.
- 5.5.2 Rotary evaporator with vacuum pump.
  - 5.5.1.1 Rotary evaporator: Operated at approximately 60 rpm with built-in water bath operated at approximately 90°C and condenser with tap water approximately 45°C (Buchi Model Re-121, or equivalent).
  - 5.5.1.2 Vacuum pump: GAST Model 1HAP-25-M100X (or equivalent).
- 5.5.2 Nitrogen blowdown apparatus: Equipped with water bath controlled at 40 to 50°C (N-Evap, Organomation Associates, Inc., or equivalent), installed in a fume hood.
  - 5.5.2.1 Concentrator tube: 10- to 15-mL, graduated (Kontes K-570050-1025, or equivalent) with calibration verified.
- 5.5.3 Pipettes.
  - 5.5.3.1 Disposable, Pasteur, 150 mm long by 5 mm i.d. (Fisher Scientific 13-678-6A, or equivalent).
  - 5.5.3.2 Disposable, serological, 10-mL (6 mm i.d.).

5.6 Calculator or computer: Capable of calculating and maintaining statistics on initial (Section 8.2) and ongoing (Section 11.3.4) performance.

6. REAGENTS

6.1 Reference matrix: Blank drilling mud, playground sand, or similar material in which the compounds of interest and interfering compounds are not detected by this method. May be prepared by pre-extraction with methylene chloride and drying at 110 to 200°C for a minimum of 4 hours.

6.2 Solvent: Toluene and methylene chloride, distilled in glass (Burdick and Jackson, or equivalent).

6.3 White quartz sand, 60/70 mesh: For Soxhlet/Dean-Stark extraction, (Aldrich Chemical Co, Cat No. 27,437-9, or equivalent).

6.4 Standard for diesel oil: Ideally, the oil standard used in this method should be from the oil used on the drilling rig from which the mud sample is to be taken. If this oil is not available, No. 2 diesel oil may be substituted. When not being used, the standard is stored in the dark at -20 to -10°C in a screw-capped vial with PTFE-lined lid. A mark is placed on the vial at the level of the solution so that solvent loss by evaporation can be detected. The vial is brought to room temperature prior to use and solvent is added (if required).

6.5 Stock solution.

6.5.1 Diesel oil in toluene: Weigh 6.25 g of diesel oil to three significant figures in a 100-mL ground-glass stoppered volumetric flask and fill to the mark with toluene. After the oil is completely dissolved, transfer the solution to a clean 150-mL bottle with PTFE-lined cap.

6.5.2 The stock solution should be checked for signs of degradation prior to the preparation of the precision and recovery standard.

6.6 Precision and recovery standard: The stock solution is spiked into the reference matrix (Section 6.1) for the determination of initial precision and recovery (IPR; Section 8.2) and ongoing precision and recovery (OPR; Section 11.2). When 1 mL of this solution is spiked into a 25-g reference matrix sample, a concentration of 0.25% (2.5 g/kg) will be produced.

7. CALIBRATION

7.1 Verify calibration of the balance at 10 mg and 100 mg using class "S" weights.

7.2 Calibration shall be within ±10% (1 mg) at 10 mg and ±2% (2 mg) at 100 mg. If not within these limits, recalibrate the balance and repeat the test.

8. QUALITY ASSURANCE/QUALITY CONTROL

8.1 Each laboratory that uses this method is required to operate a formal quality assurance program (Reference 5). The minimum requirements of this program consist of an initial demonstration of laboratory capability, an ongoing analysis of standards and blanks as a test of continued performance, analyses of spiked samples to assess accuracy, and analysis of duplicates to assess precision. Laboratory performance is compared to established performance criteria to determine if the results of analyses meet the performance characteristics of the method.

8.1.1 The analyst shall make an initial demonstration of the ability to generate acceptable accuracy and precision with this method. This ability is established as described in Section 8.2.

8.1.2 The analyst is permitted to modify this method to improve separations or lower the costs of measurements, provided all performance requirements are met. Such modifications may use alternative extraction or concentration techniques or alternative HPLC columns. Each time a modification is made to the method, the analyst is required to repeat the procedure in Section 8.2 to demonstrate method performance.

8.1.3 Analyses of spiked samples are required to demonstrate method accuracy. The procedure and QC criteria for spiking are described in Section 8.3.

8.1.4 Analyses of duplicate samples are required to demonstrate method precision. The procedure and QC criteria for duplicates are described in Section 8.4.

8.1.5 Analyses of blanks are required to demonstrate freedom from contamination. The procedures and criteria for analysis of a blank are described in Section 8.5.

8.1.6 The laboratory shall, on an ongoing basis, demonstrate through calibration verification and analysis of the OPR sample that the analysis system is in control. These procedures are described in Section 11.

8.1.7 The laboratory shall maintain records to define the quality of data that is generated. Development of accuracy statements is described in Sections 8.3.4 and 11.2.4.

8.2 Initial precision and accuracy (IPR): To establish the ability to generate acceptable precision and accuracy, the analyst shall perform the following operations.

8.2.1 Extract and evaporate four samples of the precision and recovery standard (Section 6.6) according to the procedure beginning in Section 10.

8.2.2 Using results of the set of four analyses, compute the average recovery (X) in g/kg and the standard deviation of the recovery (s) in g/kg for each sample.

8.2.3 For each compound, compare s and X with the corresponding limits for initial precision and accuracy in Table 1. If s and X meet the acceptance criteria, system performance is acceptable and analysis of samples may begin. If, however, s exceeds the precision limit or X falls outside the range for accuracy, system performance is unacceptable. In this event, review this method, correct the problem, and repeat the test.

8.3 Method accuracy: The laboratory shall spike a minimum of 10% (one sample in each set of ten samples) of all drilling mud samples. This sample shall be spiked with the diesel oil that was added to the drilling fluid. If a reference standard of diesel oil that was added to the drilling fluid is not available, No. 2 diesel oil shall be used for this spike. If doubt of the concentration of diesel oil in any of the remaining 90% of the samples exists, that sample shall be spiked to confirm the diesel oil concentration.

8.3.1 The concentration of the spike in the sample shall be determined as follows.

8.3.1.1 If, as in compliance monitoring, the concentration of the oil in the sample is being checked against a regulatory concentration limit, the spike shall he at that limit or at 1 to 5 times higher than the background concentration determined in Section 8.3.2, whichever concentration is higher.

8.3.1.2 If the concentration of the oil in a sample is not being checked against a limit, the spike shall be at the concentration of the precision and recovery standard (Section 6.6) or at 1 to 5 times higher than the background concentration, whichever concentration is higher.

8.3.2 Analyze one sample aliquot to determine the background concentration (B) of oil. If necessary, prepare a standard solution appropriate to produce a level in the sample at the regulatory concentration limit or at 1 to 5 times the background concentration (per Section 8.3.1). Spike a second sample aliquot with the standard solution and analyze it to determine the concentration after spiking (A) of each analyte. Calculate the percent recovery (P) of the oil using Equation 1:

Equation 1

$$P = \frac{100(A - B)}{T}$$

where:
A = Concentration of analyte after spiking
B = Background concentration of oil
T = True value of the spike

8.3.3 Compare the percent recovery for total oil with the corresponding QC acceptance criteria in Table 1. If the results of the spike fail the acceptance criteria, and the recovery of the QC standard in the ongoing precision and recovery test (Sections 10.1.3 and 11.2.4) is within the acceptance criteria in Table 1, an interference may be present. (See Section 3 for identification of interferences). In this case, the result may not be reported for regulatory compliance purposes. If, however, the results of both the spike and the ongoing precision and recovery test fail the acceptance criteria, the analytical system is judged to be out of control, and the problem shall be identified and corrected and the sample batch reanalyzed.

8.3.4 As part of the QA program for the laboratory, method accuracy for samples shall be assessed and records shall be maintained. After the analysis of five spiked samples in which the recovery passes the test in Section 8.3, compute the average percent recovery (P) and the standard deviation of the percent recovery (s_p,). Express the accuracy assessment as a percent recovery interval from P - 2 s_p, to P + 2 s_p,. For example, if P = 90% and s_p, = 10% for five analyses of diesel oil, the accuracy interval is expressed as 70 to 110%. Update the accuracy assessment on a regular basis (e.g., after each five to ten new accuracy measurements).

8.4 The laboratory shall analyze duplicate samples for each drilling-mud type at a minimum of 10% (one sample for each ten sample set). A duplicate sample shall consist of a well-mixed, representative aliquot of the sample.

8.4.1 Analyze one sample in the set in duplicate per the procedure beginning in Section 10.

8.4.2 Compute the relative percent difference (RPD) between the two results per the following equation:

Equation 2

$$RPD = \frac{|D_1 - D_2|}{(D_1 + D_2)/2} * 100$$

where:
D_1 = Concentration of diesel in the sample
D_2 = Concentration of diesel oil in the second (duplicate) sample

8.4.3 The relative percent difference for duplicates shall meet the acceptance criteria in Table 1. If the criteria are not met, the analytical system is judged to be out of control, and the problem must be immediately identified and corrected and the sample set reanalyzed.

8.5 Blanks: Reference matrix blanks (Section 6.1) are analyzed to demonstrate freedom from contamination.

8.5.1 Extract and concentrate a 25-g aliquot of the reference matrix initially and with each sample batch (samples started through the analysis at the same time, to a maximum of ten samples).

8.5.2 If greater than 0.2 g/kg of material is detected in a blank, analysis of samples is halted until the source of contamination is eliminated and a blank shows no evidence of contamination.

8.6 The specifications contained in this method can be met if the apparatus used is calibrated properly, then maintained in a calibrated state. The standards used for initial precision and recovery (IPR, Section 8.2) and ongoing precision and recovery (OPR, Section 11.2) should be identical, so that the most precise results will be obtained.

8.7 Depending on specific program requirements, field replicates and field spikes of diesel oil into samples may be required to assess the precision and accuracy of the sampling and sample transporting techniques.

9. SAMPLE COLLECTION, PRESERVATION, AND HANDLING

9.1 Collect drilling mud samples in wide-mouth glass containers following conventional sampling practices (Reference 6).

9.2 Samples must be representative of the entire bulk drilling mud. In some instances, composite samples may be required.

9.3 Maintain samples in the dark at 0 to 4°C from the time of collection until analysis.

9.4 Analyze samples within 28 days of collection.

10. SAMPLE EXTRACTION AND CONCENTRATION

10.1 Preparation of sample and QC aliquots.

10.1.1 Transfer approximately 25 g of a well-homogenized and representative portion of the drilling mud to a tared 400- to 500-mL beaker. Determine and record the weight.

10.1.2 Add approximately 50 g of quartz sand (Section 6.3) to the beaker and mix the drilling mud and sand thoroughly.

10.1.3 QC standard and blank: Used for tests of initial (Section 8.2) and ongoing (Section 11.2) precision and accuracy. For each of the four initial precision and recovery (IPR) standards, the ongoing precision and recovery (OPR) standard, and the blank (Section 8.5), prepare aliquots as follows.

10.1.3.1 Place approximately 25 g of the reference matrix (Section 6.1) in a clean 400- to 500-mL beaker.

10.1.3.2 Spike 1 mL of the precision and recovery standard (Section 6.6) into the IPR or OPR standard. Do not spike the blank.

10.1.3.3 Add approximately 50 g of quartz sand (Section 6.3) to the beaker and mix thoroughly.

10.2 Soxhlet/Dean-Stark extraction.

10.2.1 Pre-extraction: Used to clean the SDS extractor. Pre-extraction may be eliminated if extractable material is not found in blanks.

10.2.1.1 Charge a clean extraction thimble with 50 g of quartz sand (Section 6.3). Do not disturb the silica layer throughout the extraction process.

10.2.1.2 Place the thimble in a clean extractor. Place 30 to 40 mL of toluene in the receiver and 200 to 250 mL in the flask.

10.2.1.3 Begin the extraction by heating the flask until the toluene is boiling. When properly adjusted, one to two drops of toluene per second will fall from the condenser tip into the receiver. Pre-extract the apparatus for a minimum of 4 hours.

10.2.1.4 After pre-extraction, cool and disassemble the apparatus. Rinse with methylene chloride and allow to air-dry in a hood.

10.2.2 SDS extraction.

10.2.2.1 Load the sample (from Section 10.1.2) and QC aliquot(s) and blank (from Section 10.1.3.3) into pre-cleaned thimbles.

10.2.2.2 Reassemble the apparatus and add a fresh charge of toluene to the receivers and reflux flasks. Rinse the beakers into their respective thimbles using 10 to 20 mL of toluene.

10.2.2.3 Apply power to the heating mantle to begin refluxing. Adjust the reflux rate to match the rate of percolation through the sand bed until sufficient water has been removed so that the flow of toluene is no longer restricted.

10.2.2.4 Drain the water from the receiver at 1 to 2 hours and 8 to 9 hours, or sooner if the receiver fills with water. Record the total volume of water collected. Reflux the sample for a total of 16 to 24 hours. Cool to room temperature.

10.2.2.5 Estimate and record the volume of extract (to the nearest 100 mL).

10.3 Concentration.

10.3.1 Add one or two clean boiling chips to the evaporative flask and attach it to the rotary evaporator.

10.3.1.1 Place the round-bottom flask in the hot-water bath (approximately 90°C) so that approximately one-half of the flask is immersed in hot water.

10.3.1.2 Start the flow of cooling water and turn on the vacuum pump. Rotate the flask slowly at first to control the rate of evaporation of the toluene.

10.3.1.3 When the apparent volume reaches a few milliliters, remove the flask from the hot water bath and allow it to cool for at least 10 minutes. To minimize the loss of the more volatile components of oil, do not take the sample to dryness in the rotary evaporator.

10.3.1.4 Turn off the pump and cooling water.

10.3.1.5 Disassemble the apparatus.

10.3.1.6 Using a squeeze bottle or pipette, rinse the inside surface of the round-bottom flask with a small portion of acetonitrile. Transfer the solution to a calibrated Kuderna-Danish concentrator tube. Repeat the rinsing and transfer two more times to quantitatively transfer the solution to the concentrator tube.

10.4 Extract for other analyses: If a portion of the extract is to be retained for HPLC analysis using Method 1654A or GC/FID analysis using Method 1663, the extract is split as follows.

10.4.1 Adjust the extract volume to 5.0 mL with acetonitrile.

10.4.2 Extracts to be used in Method 1654A and Method 1663: Remove 1.00 mL with a volumetric pipette, place in a clean, calibrated K-D concentrator tube, and exchange to acetonitrile per Section 10.6.

10.4.3 Evaporate the remaining 4 mL of extract per the steps below.

10.5 Evaporation to dryness.

10.5.1 Place the receiver in the water in the nitrogen blowdown apparatus. Adjust the water temperature to 40 to 50°C.

10.5.2 Adjust the height of the blowdown needle to approximately 1 cm above the surface of the solution.

10.5.3 Adjust the nitrogen flow rate so that it is sufficient to create a depression in the surface of the solution but not so great that the solution spatters.

10.5.4 Evaporate the solvent until the volume is constant, but no longer than 30 minutes. Wipe the outside surface of the concentrator tube dry and cool the tube in the desiccator.

10.5.5 Weigh the receiver. If doubt exists that constant weight has been achieved, return the receiver to the blowdown apparatus and evaporate solvent for 15 minutes more. Cool the receiver in the desiccator and weigh the receiver. Constant weight is achieved when the readings differ by less than 5% or 5 mg, whichever is greater.

10.6 Exchange to acetonitrile

10.6.1 Follow steps 10.5.1 through 10.53.

10.6.2 Evaporate to near dryness (final volume approximately 50 µL).
10.6.3 Add 100 µL of methylene chloride and 400 µL of acetonitrile to redissolve the oil,
10.6.4 Remove with a Pasteur pipette and place in a 2- to 3-mL amber vial calibrated to 1.00 mL.
10.6.5 Using a syringe and small portions of acetonitrile, rinse the inside surface of the K-D concentrator tube and quantitatively transfer to the vial.
10.6.6 Seal and store in the dark at -20 to -10°C. Adjust the final volume to 1.00 mL immediately prior to analysis.

11. SYSTEM AND LABORATORY PERFORMANCE

11.1 Calibration verification: Verify calibration of the balance per Section 7 before and after each set of 12 or fewer measurements. (The 12 measurements will normally be ten samples, plus one ongoing precision and recovery standard, plus one blank.) If calibration is not verified after the measurements, recalibrate the balance and reweigh the batch.

11.2 Ongoing precision and recovery.

11.2.1 Weigh the evaporated precision and recovery standard extracted and concentrated with each batch of samples.

1 1.2.2 Calculate the concentration of oil in this standard.

11.2.3 Compare the concentration with the limits for ongoing precision and recovery in Table 1. If the concentration is in the range specified, the extraction and evaporation processes are in control and analysis of blanks and samples may proceed. If, however, the concentration is not in the specified range, these processes are not in control. In this event, correct the problem, re-extract the sample batch, and repeat the ongoing precision and recovery test.

11.2.4 Add results that pass the specification in Section 11.2.3 to initial and previous ongoing data. Update QC charts to form a graphic representation of continued laboratory performance. Develop a statement of laboratory data quality for each analyte by calculating the average percent recovery (R) and the standard deviation of percent recovery (s_r.). Express the accuracy as a recovery interval from R - $2s_r$ to R + $2s_r$. For example, if R = 95% and s_r = 5%, the accuracy is 85 to 105%.

12. QUANTITATIVE DETERMINATION

12.1 Determination of dry weight of sample and of percent solids.

12.1.1 Using the sample weight (Section 10.1.1) and the weight (volume) of water in the moisture trap (Section 10.2.4.4), calculate the dry weight of solids in the sample as follows:

Equation 3

$$W_d = W_s - W_w$$

where:
W_d = *Dry weight of solids, in grams*
W_s = *Weighs of sample, in grams*
W_w = *Weight of water, in grams*

12.1.2 Calculate the percent solids as follows:

Equation 4

$$\% \text{ solids} = 100\frac{W_d}{W_s}$$

where:
W_d = *Dry weight of solids, in grams*
W_s = *Weight of sample, in grams*

12.2 Determination of extractable material concentration.

12.2.1 Calculate the concentration of extractable material in the total (wet) sample using the following equation:

Equation 5

$$Concentration\ (\%) = 0.1\frac{W_r}{W_s}$$

where:
W_r = Weight of extractable material in receiver, in mg
W_s = Weight of sample, in grams

12.2.2 If 1 mL of the 5-mL extract was removed for HPLC or GC analysis, multiply the result by 1.25 to compensate for this loss.

12.3 Report results to two significant figures without correction for recovery.

13. METHOD PERFORMANCE

This method was validated in a single laboratory (Reference 7) using samples of hot-rolled drilling mud (Reference 8).

References

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Table 1. Method Acceptance Criteria for Diesel Oil[1] in Drilling Mud

		Diesel Oil	
Acceptance Criterion	*Section*	*Units*[3]	*Amount*
Method Detection Limit (matrix)[2]		g/kg	1.1
Initial precision and recovery[4]	8.2.3		
Precision (standard deviation)		g/kg	0.85
Recovery (mean; X)		g/kg	1.18 - 3.73
Ongoing precision and recovery[4]	11.2.3	g/kg	1.08 - 3.83
Matrix spike recovery	8.3.3	percent	35 - 159
Precision of duplicates	8.4.3	RPD	34

Notes:

1 CAS Registry number 68534-30-5
2 To convert to weight percent, multiply the amount by 0.1
3 40 *CFR* Part 136, Appendix B; measured in API Mud number 101-1
4 Test concentration: 2.5 g/kg (0.25 percent) diesel oil in mud
5 Precision of duplicate analyses must be <34%

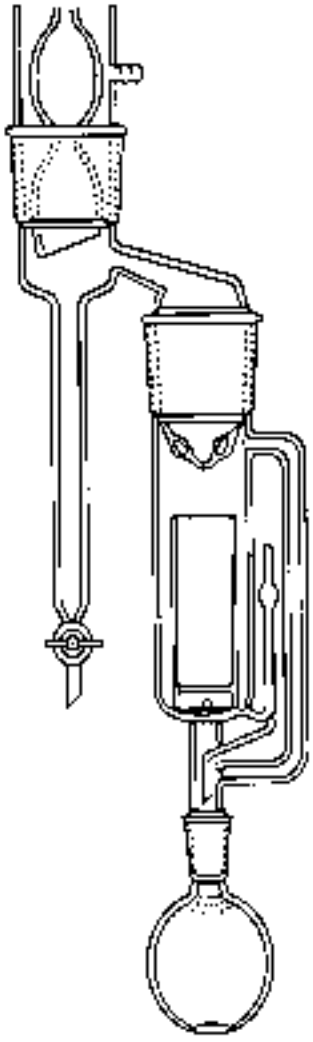

Figure 1. Soxhlet/Dean Stark Extractor