

ATTACHMENT 4**APPENDIX 3 TO SUBPART A OF PART 435 – PROCEDURE FOR MIXING
BASE FLUIDS WITH SEDIMENTS**

This procedure describes a method for amending uncontaminated and nontoxic (control) sediments with the base fluids that are used to formulate synthetic-based drilling fluids and other non-aqueous drilling fluids. Initially, control sediments shall be press-sieved through a 2000 micron mesh sieve to remove large debris. Then press sieve the sediment through a 500 micron sieve to remove indigenous organisms that may prey on the test species or otherwise confound test results. Homogenize control sediment to limit the effects of settling that may have occurred during storage. Sediments should be homogenized before density determinations and addition of base fluid to control sediment. Because base fluids are strongly hydrophobic and do not readily mix with sediment, care must be taken to ensure base fluids are thoroughly homogenized within the sediment. All concentrations are weight-to-weight (mg of base fluid to kg of dry control sediment). Sediment and base fluid mixing shall be accomplished by using the following method.

1. Determine the wet to dry ratio for the control sediment by weighing approximately 10g subsamples of the screened and homogenized wet sediment into tared aluminum weigh pans. Dry sediment at 105 °C for 18-24 h. Remove sediment and cool in a desiccator until a constant weight is achieved. Reweigh the samples to determine the dry weight. Determine the wet/dry ratio by dividing the net wet weight by the net dry weight: [Wet Sediment Weight (g) / Dry Sediment Weight (g)] = Wet to Dry Ratio [1]
2. Determine the density (g/mL) of the wet control or dilution sediment. This shall be used to determine total volume of wet sediment needed for the various test treatments.

[Mean Wet Sediment Weight (g)]/[Mean Wet Sediment Volume (mL)] = Wet Sediment Density (g/mL) [2]

3. To determine the amount of base fluid needed to obtain a test concentration of 500 mg base fluid per kg dry sediment use the following formulas:

Determine the amount of wet sediment required:

[Wet Sediment Density (g/mL)] x [Volume of Sediment Required per Concentration (mL)] = Weight Sediment Required per Conc. (g) [3]

Determine the amount of dry sediment in kilograms (kg) required for each concentration:

{[Wet Sediment per Concentration (g)]/[Mean Wet to Dry Ratio]} x (1kg/1000g) = Dry Weight Sediment (kg) [4]

Finally determine the amount of base fluid required to spike the control sediment at each concentration:

$$[\text{Conc. Desired (mg/kg)}] \times [\text{Dry Weight Sediment (kg)}] = \text{Base Fluid Required (mg)} \quad [5]$$

For spiking test substances other than pure base fluids (e.g, whole mud formulations), determine the spike amount as follows:

$$[\text{Conc. Desired (mL/kg)}] \times [\text{Dry Weight Sediment (kg)}] \times [\text{Test Substance Density (g/mL)}] = \text{Test Substance Required (g)} \quad [6]$$

4. For primary mixing, place appropriate amounts of weighed base fluid into stainless mixing bowls, tare the vessel weight, then add sediment and mix with a high-shear dispersing impeller for 9 minutes. The concentration of base fluid in sediment from this mix, rather than the nominal concentration, shall be used in calculating LC₅₀ values.
5. Tests for homogeneity of base fluid in sediment are to be performed during the procedure development phase. Because of difficulty of homogeneously mixing base fluid with sediment, it is important to demonstrate that the base fluid is evenly mixed with sediment. The sediment shall be analyzed for total petroleum hydrocarbons (TPH) using EPA Methods 3550A and 8015M, with samples taken both prior to and after distribution to replicate test containers. Base-fluid content is measured as TPH. After mixing the sediment, a minimum of three replicate sediment samples shall be taken prior to distribution into test containers. After the test sediment is distributed to test containers, an additional three sediment samples shall be taken from three test containers to ensure proper distribution of base fluid within test containers. Base-fluid content results shall be reported within 48 hours of mixing. The coefficient of variation (CV) for the replicate samples must be less than 20%. If base-fluid content results are not within the 20% CV limit, the test sediment shall be remixed. Tests shall not begin until the CV is determined to be below the maximum limit of 20%. During the test, a minimum of three replicate containers shall be sampled to determine base-fluid content during each sampling period.
6. Mix enough sediment in this way to allow for its use in the preparation of all test concentrations and as a negative control. When commencing the sediment toxicity test, range-finding tests may be required to determine the concentrations that produce a toxic effect if these data are otherwise unavailable. The definitive test shall bracket the LC₅₀, which is the desired endpoint. The results for the base fluids shall be reported in mg base fluid per kg of dry sediment.

REFERENCES

American Society for Testing and Materials (ASTM). 1996. Standard Guide for Collection, Storage, Characterization, and Manipulation of Sediments for Toxicological

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