

ENVIRONMENTAL PROTECTION AGENCY

NOTICE OF FINAL NPDES GENERAL PERMIT

Final NPDES General Permit for New and Existing Sources and New Dischargers in the Offshore Subcategory of the Oil and Gas Extraction Category for the Western Portion of the Outer Continental Shelf of the Gulf of Mexico (GMG290000)

SUMMARY: EPA Region 6 reissues the National Pollutant Discharge Elimination System (NPDES) general permit for the Western Portion of the Outer Continental Shelf of the Gulf of Mexico (No. GMG290000) for discharges from new sources, existing sources, and new dischargers in the Offshore Subcategory of the Oil and Gas Extraction Point Source Category (40 CFR Part 435, Subpart A). This reissued general permit will replace the previous permit issued on October 7, 2004 (69 FR 60150). The general permit authorizes discharges from exploration, development, and production facilities located in and discharging to Federal waters of the Gulf of Mexico seaward of the outer boundary of the territorial seas offshore of Louisiana and Texas.

Today's reissuance includes a number of changes and clarifications to the proposed permit. The following changes are included in the final permit. Requirements to comply with new cooling water intake structure regulations were changed to allow expansion of the industry-wide study to include entrainment monitoring. Operators are only required to submit cooling water intake structure design information once per facility. Notification requirements have been added for operators of mobile offshore drilling units required to comply with cooling water intake structure conditions. An end-of-well sample is no longer required for sediment toxicity testing when using non-aqueous based drilling fluids. The toxicity testing frequency for sub-sea fluids has been decreased from once per batch to once per year. Toxicity testing is no longer required for miscellaneous discharges treated using hypochlorite. Minor corrections were made in the produced water whole effluent toxicity testing requirements. Other minor changes in wording were made to clarify EPA's intent regarding the permit's requirements.

FOR FURTHER INFORMATION CONTACT: Ms. Diane Smith, EPA Region 6, 1445 Ross Avenue, Dallas, Texas 75202, Telephone: (214) 665 7191, or via EMAIL to the following address: smith.diane@epa.gov

Authorization to Discharge Under the National Pollutant Discharge Elimination System

In compliance with the Federal Water Pollution Control Act, as amended (33 U.S.C. 1251 et. seq. the "Act"), operators of lease blocks in the Oil and Gas Extraction Point Source Category located in Federal waters of the Western Portion of the Gulf of Mexico (defined as seaward of the outer boundary of the territorial seas off Louisiana and Texas) are authorized to discharge to the Western Portion of the Federal Waters of the Gulf of Mexico in accordance with the effluent limitations, monitoring requirements, and other conditions set forth in Parts I, II, and III hereof. Also, operators of lease blocks located in the territorial seas of Louisiana and Texas are authorized to discharge produced water from wells located in those lease blocks to the Western Portion of the Federal Waters of the Gulf of Mexico in accordance with effluent limitations, monitoring requirements, and other conditions set forth in Parts I, II, and III hereof. Operators of mobile offshore drilling units (MODUs) located in Federal Waters of the Western

Gulf of Mexico are required to comply with the cooling water intake structure provisions of Part I.B.12 of this permit.

Operators of lease blocks located within the general permit area must submit written notification to the Regional Administrator that they intend to be covered (See Part I.A.2). In addition to that notification, operators of facilities which must comply with the cooling water intake requirements in Part I.B.12 of this permit must submit the supplemental notification required under that section. Unless otherwise notified in writing by the Regional Administrator owners or operators requesting coverage are authorized to discharge under this general permit after the postmark date of submission of the notification. Operators of lease blocks within the general permit area who fail to notify the Regional Administrator of intent to be covered by this general permit are not authorized under this general permit to discharge pollutants from those facilities. Operators who have previously submitted a written notification of intent to be covered under this general permit need not submit an additional notification of intent to be covered even in advance of permit expiration. For facilities covered under the previous permit, the monitoring periods shall remain the same unless otherwise notified by EPA. Additionally, the monitoring period for toxicity testing shall correspond with that of other parameters.

This permit will be re-opened and modified or revoked and reissued if necessary based on the results of the ESA consultation with NMFS.

Facilities which adversely affect properties listed or eligible for listing in the National Register of Historic Places are not authorized to discharge under this permit.

This permit shall become effective at Midnight Central Standard Time, October 1, 2007.

This permit and the authorization to discharge shall expire at midnight, Central Standard Time, September 30, 2012.

Signed this day of

Miguel I. Flores
Director, Water Quality Protection Division
EPA Region 6

PART I. REQUIREMENTS FOR NPDES PERMITS

Section A. Permit Applicability and Coverage Conditions

1. Operations Covered

This permit establishes effluent limitations, prohibitions, reporting requirements, and other conditions on discharges from oil and gas facilities, and supporting pipeline facilities, engaged in production, field exploration, developmental drilling, facility installation, well completion, well treatment operations, well workover, and abandonment/decommissioning operations.

The permit coverage area consists of lease blocks located in and discharging to Federal waters in the Gulf of Mexico seaward of the outer boundary of the territorial seas offshore of Louisiana and Texas and shall include lease blocks west of the western boundary of the outer continental shelf lease areas defined as: Mobile, Viosca Knoll (north part), Destin Dome, Desoto Canyon, Lloyd, and Henderson. In Texas, where the state has mineral rights to three leagues, some operators with state lease tracts are required to request coverage under this Federal NPDES general permit. In addition, permit coverage consists of produced water discharges to those Federal waters from lease blocks located in the territorial seas of Texas and Louisiana. This includes produced water from wells located in the area of coverage, which is sent on-shore for treatment and subsequently sent back to the Outer Continental Shelf to be discharged. This permit does not authorize discharges from facilities located in or discharging to the territorial seas of Louisiana or Texas or from facilities defined as "coastal", "onshore", or "stripper" (see 40 CFR Part 435, Subparts C, D, and E).

2. Notification Requirements

Written notification of intent (NOI) to be covered including the legal name and address of the operator, the lease area and block number assigned by the Department of Interior or the state or, if none, the name commonly assigned to the lease area shall be submitted and postmarked prior to the commencement of discharge. Owners/operators of MODUs that are required to meet the conditions of Part I.B.12 shall submit an NOI along with the required supplemental notification information. That information must be submitted prior to commencement of operations. For facilities installed after March 4, 1993, the NOI must also identify that the facility is a New Source and state the date on which the facility's protection from more stringent new source performance standards or technology-based limitations ends. That date is the soonest of: ten years from the date that construction is completed, ten years from the date the source begins to discharge process or non construction related waste water, or the end of the period of depreciation or amortization of the facility for the purposes of section 167 or 169 (or both) of the Internal Revenue code of 1954.

Additionally, if an application for an individual permit for the activity was previously submitted to EPA Region 6, the NOI shall include the application/permit number of that application or the permit number of any individual NPDES permit issued by EPA Region 6 for this activity. The NOI shall be submitted and postmarked prior to commencement of discharge.

The postmark date is evidence of delivery and acceptance for purposes of coverage and shall signify approval to commence discharge.

Permittees located in lease blocks that (a) are neither in nor adjacent to MMS-defined "no activity" areas, or (b) do not require live-bottom surveys are required only to submit an NOI to be covered by this general permit. Permittees who are located in lease blocks that are either in or adjacent to "no activity" areas or require live bottom surveys are required to submit both an NOI to be covered that specifies they are located in such a lease block and are required to submit a notice of commencement of operations.

Permittees located in lease blocks either in or immediately adjacent to MMS-defined "no activity" areas, shall be responsible for determining whether a controlled discharge rate is required. The maximum discharge rate for drilling fluids is determined by the distance from the facility to the "no activity" area boundary and the discharge rate equation provided in Part I.B.1.b. The permittee shall report the distance from the permitted facility to the "no activity" area boundary and the calculated maximum discharge rate to EPA with its notice of commencement of operations.

For permittees located in lease blocks that require live-bottom surveys, the final determination of the presence or absence of live-bottom communities, the distance of the facility from identified live-bottom areas, and the calculated maximum discharge rate shall be reported with the notice of commencement of operations.

See Part I.B.12 for cooling water intake structure notice of intent requirements.

All notifications of intent to be covered, transfer agreements, notices of termination and all subsequent reports under this permit shall be sent to the following address:

Water Enforcement Branch (6EN-WC)
Region 6
U.S. Environmental Protection Agency
1445 Ross Avenue
Dallas, TX 75202

Additional information regarding these reporting requirements may be found at:

<http://www.epa.gov/region6/6en/w/offshore/>

3. Termination of NPDES Coverage

Lease block operators shall submit a notice of termination (NOT) to the Regional Administrator within 60 days of termination of lease ownership for lease blocks assigned to the operator by the Department of Interior. In the case of temporary operations such as hydrostatic testing, the NOT shall be submitted within 60 days of termination of operations. The discharge monitoring report (DMR) for the terminated lease block shall be submitted with the NOT, or

with the annual DMR submittal. The NOT shall be effective upon the date it is received by EPA.

Section B. Effluent Limitations and Monitoring Requirements

1. Drilling Fluids

The discharge of drilling fluids shall be limited and monitored by the permittee as specified in Table 1 of Appendix E and as stated below.

Special Note: The permit prohibitions and limitations that apply to drilling fluids, also apply to fluids that adhere to drill cuttings. Any permit condition that may apply to the drilling fluid discharges, therefore, also applies to cuttings discharges.

[Exception] The discharge rate limit for drilling fluids does not apply to drill cuttings.

a) Prohibitions

Non aqueous Based Drilling Fluids. The discharge of non aqueous based drilling fluid is prohibited, except that which adheres to cuttings and small volume discharges described below in Part 1.B.2.c.2.

Exception: non aqueous base fluids may be used as a carrier fluid (transporter fluid), lubricity additive or pill in water based drilling fluids and discharged with those drilling fluids provided the discharge continues to meet the no free oil and 96-hour LC50 toxicity limits, and a pill is removed prior to discharge.

Oil-Based Drilling Fluids. The discharge of oil based drilling fluids and oil based inverse emulsion drilling fluids are prohibited.

Oil Contaminated Drilling Fluids. The discharge of drilling fluids which contain waste engine oil, cooling oil, gear oil or any lubricants which have been previously used for purposes other than borehole lubrication, is prohibited.

Diesel Oil. Drilling fluids to which any diesel oil has been added as a lubricant may not be discharged.

b) Limitations

Mineral Oil. Mineral oil may be used only as a carrier fluid (transporter fluid), lubricity additive, or pill.

Cadmium and Mercury in Barite. There shall be no discharge of drilling fluids to which barite has been added, if such barite contains mercury in excess of 1.0 mg/kg (dry weight) or cadmium in excess of 3.0 mg/kg (dry weight). The permittee shall analyze a

representative sample of all stock barite used once, prior to drilling each well, and submit the results for total mercury and cadmium in the DMR.

If more than one well is being drilled at a site, new analyses are not required for subsequent wells, provided that no new supplies of barite have been received since the previous analysis. In this case, the results of the previous analysis should be used on the DMR.

Alternatively, the permittee may provide certification, as documented by the supplier(s), that the barite being used on the well will meet the above limits. The concentration of the mercury and cadmium in the barite shall be reported on the DMR as documented by the supplier.

Analyses for mercury shall be conducted using EPA Methods 245.5 or 7471 **A** and the results expressed in mg/kg (dry weight). Analysis for cadmium shall be conducted using EPA methods 200.7, 200.8, or EPA method 3050 B followed by 6010B **or 6020** and the results expressed as mg/kg (dry weight) of barite.

Toxicity. Discharged drilling fluids shall meet both a daily minimum and a monthly average minimum 96-hour LC50 of at least 30,000 ppm in a 9:1 seawater to drilling fluid suspended particulate phase (SPP) volumetric ratio using *Mysidopsis bahia*. Monitoring shall be performed at least once per month for both a daily minimum and the monthly average. In addition, an end-of-well sample is required for a daily minimum when drilling is conducted using aqueous based drilling fluid. The type of sample required is a grab sample, taken from beneath the shale shaker, or if there are no returns across the shale shaker, the sample must be taken from a location that is characteristic of the overall mud system to be discharged. Permittees shall report the results on the DMR using either the full toxicity test or the partial toxicity test as specified at 58 FR 12512, March 4, 1993; however, if the partial toxicity test shows a failure, all testing of future samples from that well shall be conducted using the full toxicity test method to determine the 96-hour LC50.

Free Oil. No free oil shall be discharged. Monitoring shall be performed using the static sheen method once per week when discharging. The number of days a sheen is observed must be recorded.

Discharge Rate. All facilities are subject to a maximum discharge rate of 1,000 barrels per hour.

For those facilities subject to the discharge rate limitation requirement because of their proximity to areas of biological concern, the discharge rate of drilling fluids shall be determined by the following equation:

$$R = 10^{[3 \text{ Log } (d/15) + T_t]}$$

Where:

R = discharge rate (bbl/hr)

d = distance (meters) from the boundary of a controlled discharge rate area

T_i = toxicity-based discharge rate term

$$= [\log (\text{LC50} \times 8 \times 10^{-6})] / 0.3657$$

Drilling fluid discharges (based on a mud toxicity of 30,000 ppm) equal to or less than 544 meters from areas of biological concern shall comply with the discharge rate obtained from the equation above. Drilling fluids discharges which are shunted to the bottom as required by MMS are not subject to this discharge rate control requirement.

All discharged drilling fluids; including those fluids adhering to cuttings must meet the limitations of this section except that discharge rate limitations do not apply before installation of the marine riser.

c) Monitoring Requirements

Drilling Fluids Inventory. The permittee shall maintain a precise chemical inventory of all constituents and their total volume or mass added downhole for each well.

2. Drill Cuttings

The discharge of drill cuttings shall be limited and monitored by the permittee as specified in Appendix E, Table 1 of this permit and as below.

a) Prohibitions which apply to all drill cuttings

Cuttings from Oil Contaminated Drilling Fluids. The discharge of cuttings that are generated using drilling fluids which contain waste engine oil, cooling oil, gear oil or any lubricants which have been previously used for purposes other than borehole lubrication, is prohibited.

Cuttings Generated Using Drilling Fluids which Contain Diesel Oil. The discharge of drill cuttings generated using drilling fluids which contain diesel oil is prohibited.

Cuttings Generated Using Mineral Oil. The discharge of cuttings generated using drilling fluids which contain mineral oil is prohibited except when the mineral oil is used as a carrier fluid (transporter fluid), lubricity additive, or pill.

b) Limitations which apply to all drill cuttings

Cadmium and Mercury in Barite. Drill cuttings generated using drilling fluids to which barite has been added shall not be discharged if the barite contains mercury in excess of 1.0 mg/kg (dry weight) or cadmium in excess of 3.0 mg/kg (dry weight).

Toxicity. Drill cuttings generated using drilling fluids with a daily minimum or a monthly average minimum 96-hour LC₅₀ of less than 30,000 ppm in a 9:1 seawater to drilling fluids suspended particulate phase (SPP) volumetric ratio as measured using the *Mysidopsis bahia* shall not be discharged.

Free Oil. No free oil shall be discharged. Monitoring shall be performed using the static sheen test method once per week when discharging. The number of days a sheen is observed must be recorded.

c) Limitations and Monitoring Requirements Which Apply to Drill Cuttings Generated Using Non aqueous Based Drilling Fluids.

1) Stock Limitations:

The permittee shall analyze a representative sample of the stock base fluids at the frequencies listed below. The test results shall be reported on the Discharge Monitoring Report. Stock limitations are designed to ensure that only stock base fluids meeting BAT criteria are added to existing drilling muds. It is acceptable to mix two or more stock base fluids together as long as they are each compliant with the stock limitation requirements. The stock limitation value reported on the DMR shall be the worst result of any one stock base fluid which is added to the drilling fluid system.

Alternatively, the permittee may provide certification, as documented by the supplier(s), that the stock base fluid being used on the well will meet the limits listed below.

Polynuclear Aromatic Hydrocarbons (PAH). The mass ratio in grams of PAH (as phenanthrene) divided by the mass in grams of base fluids shall not exceed 0.00001. Monitoring shall be performed at least once per year on each base fluid blend. See Part I, Section D.9 of this permit.

Sediment Toxicity. The ratio of the 10-day LC₅₀ of C₁₆ - C₁₈ internal olefin or C₁₂-C₁₄ or C₈ ester reference fluid divided by the 10-day LC₅₀ sediment toxicity test with *Leptocheirus plumulosus* of the base fluid shall not exceed 1.0. Monitoring shall be performed at least once per year on each base fluid blend. See Part I.D.7 **and Part I.D.9** of this permit.

Biodegradation Rate. The ratio of the cumulative gas production (ml) of C₁₆ - C₁₈ internal olefin or C₁₂-C₁₄ or C₈ ester reference fluid divided by the cumulative gas production (ml) of stock base fluid, both at 275 days, shall not exceed 1.0. Monitoring shall be performed at least once per year on each base fluid blend. See Part I.D.8 **and Part I.D.9** of this permit.

Stock limitations are designed to ensure that only base fluids meeting limits established by the Effluent Limitations Guidelines are added to existing drilling

fluids. As long as blends of fluids that are added to a built mud system meet the stock limitations and the original drilling fluid was built using base fluids or blends of fluids that meet the stock limitations, it is acceptable to mix a base fluid with a built whole mud system. It is also acceptable to mix together two built whole mud systems that contain different base fluids so long as they are themselves built with base fluids that are compliant with the stock limitations.

2) **Discharge Limitations:**

Sediment Toxicity. The ratio of the 4-day LC₅₀ of C₁₆ - C₁₈ internal olefin reference drilling fluid divided by the 4-day LC₅₀ of the drilling fluids, removed from cuttings at the solids control equipment, shall not exceed 1.0. Monitoring shall be performed at least once per month on drilling fluids which meet the stock limitations for a C₁₆-C₁₈ internal olefin. For drilling fluids which meet stock limitations for C₁₂-C₁₄ ester or C₈ ester, monitoring shall be performed at least once per well at the end of drilling with non aqueous based drilling fluids. See Appendix A of this permit and sampling protocol in Part I.D.9.

The reference drilling fluid shall be formulated from C₁₆ - C₁₈ internal olefin and meet the criteria listed in Table 1 of 40 CFR Part 435, Subpart A, Appendix 8. A uniform emulsifier package shall be used for all formulations of reference drilling fluids.

Formation Oil. No discharge. Monitoring shall be performed on the drilling fluid as follows:

- a) Once prior to drilling using the gas chromatography/mass spectrometry test method specified in Part I, Section D.10 of this permit. The test results shall be reported on the DMR.

Alternatively, the permittee may provide certification, as documented by the supplier(s), that the drilling fluid being used on the well will meet the no discharge limit for formation oil.

- b) Once per week during drilling using the Reverse Phase Extraction test method specified in Part I, Section D.11 of this permit or the gas chromatography/mass spectrometry method specified in Part I, Section D.11 of this permit.

Base Fluids Retained on Cuttings.

Monitoring shall be performed at least once per day when generating new cuttings, except when meeting the conditions of the Best Management Practices described below. Operators conducting fast drilling (i.e., greater than 500 linear feet advancement of the drill bit per day using non aqueous fluids) shall collect and analyze one set of drill cuttings samples per 500 linear feet drilled, with a maximum of three sets per day. Operators shall collect a single discrete drill cuttings sample for each point of discharge to the ocean. The weighted average of

the results of all discharge points for each sampling interval will be used to determine compliance. See Part I, Section D.12 of this permit.

Drilling Fluids which meet stock limitations for C₁₆-C₁₈ internal olefin:

the end-of-well maximum weighted mass ratio averaged over all well sections drilled using non aqueous fluids shall not exceed 6.9 grams non aqueous base fluids per 100 grams of wet drill cuttings.

Drilling fluids which meet stock limitations for C₁₂-C₁₄ ester or C₈ ester:

the end-of-well maximum weighted mass ratio averaged over all well sections drilled using non aqueous fluids shall not exceed 9.4 grams non aqueous base fluids per 100 grams of wet drill cuttings.

Discharges of Drill Cuttings Made at the Sea Floor. A default value of 14% of base fluids retained on drill cuttings may be used for determining compliance with the base fluids retained on cuttings limits when sea floor discharges are made from dual gradient drilling. In those cases 15% will be used as a default value for the mass fraction of cuttings discharged sub sea. The default values will be averaged with results obtained from daily monitoring to determine compliance with the retention limitations.

Additionally, operators performing dual gradient drilling operations which lead to sub sea discharges of large cuttings for the proper operation of sub sea pumps shall also perform the following tasks:

- (a) Use side scan sonar or shallow seismic to determine the presence of high density chemosynthetic communities as defined by the Minerals Management Service. Chemosynthetic communities are assemblages of tube worms, clams, mussels, and bacterial mats that occur at natural hydrocarbon seeps or vents, generally in water depths of 500 meters or deeper. Discharges of large cuttings for the proper operation of subsea pumps shall not be permitted within 1500 feet of a high density chemosynthetic community.
- (b) Sea floor discharges of large cuttings for the proper operation of sub sea pumps shall be visually monitored and documented by a Remotely Operated Vehicle (ROV) within the tether limit (approximately 300 feet). The visual monitoring shall be conducted prior to each time the discharge point is relocated (cuttings discharge hose) and conducted along the same direction as the discharge hose position. Near-seabed currents shall be measured at the time of the visual monitoring.
- (c) Discharges of large cuttings for the proper operation of sub sea pumps shall be directed within a 150 foot radius of the wellbore.

De Minimis Discharges of Non aqueous Based Drilling Fluids. De minimis discharges of non aqueous based drilling fluids not associated with cuttings shall be contained to the extent practicable to prevent discharge. Allowable de minimis discharges can include wind blown drilling fluids from the pipe rack and minor drips and splatters around mud handling and solids control equipment. Such de minimis discharges are not likely to be measurable and are not considered in the base fluids retained on cuttings limit.

Small Volume Drilling Fluid Discharges. Small volume drilling fluid discharges which are associated with cuttings and for which discharge is authorized are: displaced interfaces, accumulated solids in sand traps, pit clean-out solids, centrifuge discharges made while changing mud weight. To determine the percent of drilling fluids retained on cuttings for those discharges, the operator may either monitor the discharge using the retort test method or use a default value of 25% to determine compliance with the limitation. Required discharge monitoring for small volume discharges consists only of static sheen tests and retention on cuttings (or use of the default retention on cuttings value).

Best Management Practices.

Operators (in conjunction with drilling contractors) may design and implement a Best Management Practices (BMP) Plan in accordance with the following requirements. BMP Plans are an option to help reduce monitoring of base fluids retained on cuttings. Operators are not required to use BMPs if all cuttings discharges generated using non aqueous based drilling fluids are monitored daily as described above. Where BMPs will be used, the BMP plan shall be certified and implemented prior to discharge of drill cuttings produced using non aqueous based drilling fluids.

a) BMP Plan Purpose and Objectives

Operators shall identify in advance of drilling operations each non aqueous base fluid well that will use a BMP Plan. BMP Plans shall be designed to prevent or minimize the discharge of Non Aqueous Fluid (NAF) from the facility to the waters of the United States, through normal operations and ancillary activities. The operator shall establish specific objectives for the control of NAF by conducting the following evaluations.

Each facility component or system controlled through use of BMPs shall be examined for its NAF-waste minimization opportunities and its potential for causing a discharge of NAF to waters of the United States due to equipment failure, improper operation, natural phenomena (e.g., rain, snowfall). When there is a reasonable potential for NAF reaching surface waters, the BMP Plan shall include a prediction of the total quantity of NAF which could be discharged from the facility as a result of each condition or circumstance.

b) BMP Plan Requirements

The BMP Plan may reflect requirements within the pollution prevention requirements required by the Minerals Management Service (see 30 CFR 250.300) or other Federal or State requirements and incorporate any part of such plans into the BMP Plan by reference.

The operator shall certify that its BMP Plan is complete, on-site, and available upon request to EPA. A copy of the certification shall be kept with the BMP Plan.

The BMP Plan shall:

Be documented in narrative form, and shall include any necessary plot plans, drawings or maps, and shall be developed in accordance with good engineering practices. At a minimum, the BMP Plan shall contain the planning, development and implementation, and evaluation/reevaluation components. Examples of these components are contained in “Guidance Document for Developing Best Management Practices (BMP)” (EPA 833-B-93-004, U.S. EPA, 1993).

Address each component or system capable of generating or causing a release of significant amounts of NAF and identify specific preventive or remedial measures to be implemented.

Include the following provisions concerning BMP Plan review:

Be reviewed by operator’s drilling engineer and on-site representative to ensure compliance with the BMP Plan purpose and objectives set forth in paragraph a) of this section.

And

Include a statement that the review has been completed and that the BMP Plan fulfills the BMP Plan purpose and objectives set forth in paragraph a). This statement shall have dated signatures from the operator’s drilling engineer and authorized on-site representative responsible for development and implementation of the BMP Plan.

c) BMP Plan Documentation

The operator shall maintain a copy of the BMP Plan and related documentation (e.g., training certifications, summary of the monitoring results, records of NAF-equipment spills, repairs, and maintenance) at the facility and shall make the BMP Plan and related documentation available to EPA or the NPDES Permit controlling authority upon request.

d) BMP Plan Modification

For those NAF waste streams controlled through BMPs, the operator shall amend the BMP Plan within 14 days whenever there is a change in the facility or in the operation of the facility which materially increases the generation of those NAF-wastes or their release or potential release to the receiving waters.

At a minimum the BMP Plan shall be reviewed once every five years and amended within three months if warranted. Any such changes to the BMP Plan shall be consistent with the objectives and specific requirements listed in this permit. All changes in the BMP Plan shall be reviewed by the operator's drilling engineer and authorized on-site representative.

At any time, if the BMP Plan proves to be ineffective in achieving the general objective of preventing and minimizing the discharge of NAF-wastes the BMP Plan shall be subject to modification. If the BMP requirements in the permit are modified, the BMP Plan must be modified to incorporate the revised BMP requirements within three months.

e) Specific Pollution Prevention Requirements for NAF Discharges Associated with Cuttings

The following specific pollution prevention activities are required in a BMP Plan when operators elect to control NAF discharges associated with cuttings by a set of BMPs.

Establish programs for identifying, documenting, and repairing malfunctioning NAF equipment, tracking NAF equipment repairs, and training personnel to report and evaluate malfunctioning NAF equipment.

Establish operating and maintenance procedures for each component in the solids control system in a manner consistent with the manufacturer's design criteria.

Use the most applicable spacers, flushes, pills, and displacement techniques in order to minimize contamination of drilling fluids when changing from water-based drilling fluids to NAF and vice versa.

A daily retort analysis shall be performed (in accordance with Appendix 7 to Subpart A of 40 CFR Part 435) during the first 0.33 X feet drilled with NAF where X is the anticipated total feet to be drilled with NAF for that particular well. The retort analyses shall be documented in the well retort log. The operators shall use the calculation procedures detailed in Appendix 7 to Subpart A of Part 435 (see Equations 1 through 8) to determine the arithmetic average ($\%BF_{\text{well}}$) of the retort analyses taken during the first 0.33 X feet drilled with NAF.

When the arithmetic average ($\%BF_{\text{well}}$) of the retort analyses taken during the first 0.33 X feet drilled with NAF is less than or equal to the base fluid retained on cuttings limitation or standard (see §§435.13 and 435.15), retort monitoring of cuttings may cease for that particular well. The same BMPs and drilling fluid used during the first 0.33 X feet shall be used for all remaining NAF sections for that particular well.

When the arithmetic average ($\%BF_{\text{well}}$) of the retort analyses taken during the first 0.33 X feet drilled with NAF is greater than the base fluid retained on cuttings limitation or standard (see §§435.13 and 435.15), retort monitoring shall continue for the following (second) 0.33 X feet drilled with NAF where X is the anticipated total feet to be drilled with NAF for that particular well. The retort analyses for the first and second 0.33 X feet shall be documented in the well retort log.

When the arithmetic average ($\%BF_{\text{well}}$) of the retort analyses taken during the first 0.66 X feet (i.e., retort analyses taken from first and second 0.33 X feet) drilled with NAF is less than or equal to the base fluid retained on cuttings limitation or standard (see §§435.13 and 435.15), retort monitoring of cuttings may cease for that particular well. The same BMPs and drilling fluid used during the first 0.66 X feet shall be used for all remaining NAF sections for that particular well.

When the arithmetic average ($\%BF_{\text{well}}$) of the retort analyses taken during the first 0.66 X feet (i.e., retort analyses taken from first and second 0.33 X feet) drilled with NAF is greater than the base fluid retained on cuttings limitation or standard (see §§435.13 and 435.15), retort monitoring shall continue for all remaining NAF sections for that particular well. The retort analyses for all NAF sections shall be documented in the well retort log.

When the arithmetic average ($\%BF_{\text{well}}$) of the retort analyses taken over all NAF sections for the entire well is greater than the base fluid retained on cuttings limitation or standard (see §§435.13 and 435.15), the operator is in violation of the base fluid retained on cuttings limitation or standard and shall submit notification of these monitoring values in accordance with NPDES permit requirements. Additionally, the operator shall, as part of the BMP Plan, initiate a re-evaluation and modification to the BMP Plan in conjunction with equipment vendors and/or industry specialists.

The operator shall maintain retort monitoring data and dates of retort-monitored and non-retort-monitored NAF-cuttings discharges managed by BMPs in their NPDES permit records.

Establishing mud pit and equipment cleaning methods in such a way as to minimize the potential for building-up drill cuttings (including accumulated solids) in the active mud system and solids control equipment system. These cleaning methods shall include but are not limited to the following procedures.

Ensure proper operation and efficiency of mud pit agitation equipment.

Use mud gun lines during mixing operations to provide agitation in dead spaces.

Pump drilling fluids off of drill cuttings (including accumulated solids) for re-use, recycle, or disposal before using wash water to dislodge solids.

3. Deck Drainage

a) Limitations

Free Oil. No free oil shall be discharged, as determined by the visual sheen method on the surface of the receiving water. Monitoring shall be performed once per day when discharging, during conditions when an observation of a visual sheen on the surface of the receiving water is possible in the vicinity of the discharge, and the facility is manned. The number of days a sheen is observed must be recorded.

4. Produced Water

a) Limitations

Oil and Grease. Produced water discharges must meet both a daily maximum of 42 mg/l and a monthly average of 29 mg/l for oil and grease.

Toxicity. The 7-day average minimum and monthly average minimum No Observable Effect Concentration (NOEC) must be equal to or greater than the critical dilution concentration specified in Appendix D, Table 1 of this permit. Critical dilution shall be determined using Table 1 in Appendix D of this permit and is based on the highest monthly average discharge rate for the three months prior to the month in which the test sample is collected, discharge pipe diameter, and water depth between the discharge pipe and the bottom. The monthly average minimum NOEC value is defined as the arithmetic average of all 7-day average NOEC values determined during the month.

Compliance with sub-lethal effects must be achieved within two years after the effective date of this permit.

[Exception] Permittees wishing to increase mixing may use a diffuser, add seawater, or install multiple discharge ports. Alternatively, permittees wishing to reduce the critical dilution of the discharge may make operational changes that reduce the flow rate, such as, shutting-in wells.

Permittees wishing to reduce a produced water discharge rate, and thereby the critical dilution, through operational changes must provide EPA with a description of the specific changes that were made and the resultant flow rate. The permittee must certify

that this flow rate will not be exceeded for the remainder of the DMR period, unless the permittee re-certifies.

Permittees using a diffuser shall install the diffuser designed so that the 7-day average minimum and monthly average minimum No Observable Effect Concentration (NOEC) is equal to or greater than the critical dilution concentration as calculated using CORMIX2 version 3.20. The permittee has the option of using a newer version of CORMIX2, with the following input conditions:

Density Gradient = $0.15 \sigma_t/m$
 Ambient seawater density at diffuser depth = 1017 kg/m^3
 Produced water density = 1070 kg/m^3
 Current speed = 10 cm/sec .

Permittees shall submit a certification that the diffuser has been installed and state the critical dilution corresponding to the diffuser in the certification. The CORMIX2 model runs shall be retained by the permittee as part of its NPDES records.

Permittees discharging produced water at a rate greater than 75,000 bbl/day shall determine the critical dilution using CORMIX version 3.20 (or a newer version of CORMIX) with the input parameters shown above. Permittees shall retain the model run as a part of the NPDES records.

Permittees using vertically aligned multiple discharge ports shall provide vertical separation between ports which is consistent with Appendix D, Table 1-G of this permit. When multiple discharge ports are installed, the depth difference between the discharge port closest to the sea floor and the sea floor shall be the depth difference used to determine the critical dilution from Appendix D, Table 1 of this permit. The critical dilution value shall be based on the port flow rate (total flow rate divided by the number of discharge ports) and based on the diameter of the discharge port (or smallest discharge port if they are of different styles).

When seawater is added to produced water prior to discharge, the total produced water flow, including the added seawater, shall be used in determining the critical dilution from Appendix D, Table 1.

b) Produced Water Monitoring Requirements

- iv) Flow. Once per month an estimate of the flow must be recorded in units of barrels per day (bbl/day).
- v) Samples for oil and grease monitoring shall be collected and analyzed a minimum of once per month. In addition, a produced water sample shall be collected, within two (2) hours of when a sheen is observed in the vicinity of the discharge or within two hours after startup of the system if it is shut down following a sheen discovery,

and analyzed for oil and grease. The sample type for all oil and grease monitoring shall be either grab, or a composite which consists of the arithmetic average of the results of grab samples collected at even intervals during a period of 24-hours or less. If only one sample is taken for any one month, it must meet both the daily maximum and monthly average limits. Samples for oil and grease monitoring shall be collected prior to the addition of any seawater to the produced water waste stream. The analytical method is that specified at 40 CFR Part 136.

- vi) Toxicity. The flow used to determine the frequency of toxicity testing shall be the highest monthly average flow for the three months prior to the month in which the test sample is collected. The required frequency of testing shall be determined as follows:

<u>Discharge Rate</u>	<u>Toxicity Testing Frequency</u>
0 - 4,599 bbl/day	once per annual DMR monitoring period
4,600 bbl/day and above	once per calendar quarter:

The calendar quarters are defined as the following periods: January 1 to March 31, April 1 to June 30, July 1 to September 30, and October 1 to December 31

Toxicity testing requirements for new discharges shall become effective three months after discharge begins and continue on the appropriate calendar quarter or annual DMR monitoring period.

Samples for monitoring produced water toxicity shall be collected after addition of any added substances, including seawater that is added prior to discharge, and before the flow is split for multiple discharge ports. Samples also shall be representative of produced water discharges when scale inhibitors, corrosion inhibitors, biocides, paraffin inhibitors, well completion fluids, workover fluids, and/or well treatment fluids are used in operations.

If the permittee has been subject to quarterly testing and has been compliant with these toxicity limits for one full year (four consecutive quarters), the required testing frequency shall be reduced to once per annual DMR monitoring period. If the permittee has been subject to annual testing and has been compliant for the first year, the required toxicity testing frequency shall remain once per annual DMR monitoring period even if the discharge rate subsequently exceeds 4,599 bbl/day. If the permittee monitored produced water toxicity at the reduced frequency of once per annual DMR monitoring period under the previous Outer Continental Shelf general permit, the required monitoring frequency shall remain once per annual DMR monitoring period. See Part I.D.3.e of this permit, if a test fails the survival or sub-lethal endpoint at the critical dilution in any test.

- vii) Visual Sheen The permittee shall monitor free oil using the visual sheen test method on the surface of the receiving water. Monitoring shall be performed once per day when discharging, during conditions when observation of a sheen on the

surface of the receiving water is possible in the vicinity of the discharge, and when the facility is manned.

5. Produced Sand

There shall be no discharge of produced sand.

6. Well Treatment Fluids, Completion Fluids, and Workover Fluids

a) Limitations

Free Oil. No free oil shall be discharged. Monitoring shall be performed using the static sheen test method once per day when discharging and the facility is manned. The number of days a sheen is observed must be recorded.

Oil and Grease. Well treatment, completion, and workover fluids must meet both a daily maximum of 42 mg/l and a monthly average of 29 mg/l limitation for oil and grease. The sample type for all oil and grease monitoring shall be either grab or a composite which consists of the arithmetic average of the results of grab samples collected at even intervals during a period of 24-hours or less. If only one sample is taken for any one month, it must meet both the daily and monthly limits. The analytical method is that specified at 40 CFR Part 136.

Priority Pollutants. For well treatment fluids, completion fluids, and workover fluids, the discharge of priority pollutants is prohibited except in trace amounts. Information on the specific chemical composition of any additives containing priority pollutants shall be recorded.

[Note] If materials added downhole as well treatment, completion, or workover fluids contain no priority pollutants, the discharge is assumed not to contain priority pollutants except possibly in trace amounts.

b) Monitoring Requirements

This discharge shall be considered produced water for monitoring purposes when commingled with produced water.

7. Sanitary Waste (Facilities Continuously Manned for 30 or more consecutive days by 10 or More Persons)

a) Prohibitions

Solids. No floating solids may be discharged to the receiving waters. An observation must be made once per day for floating solids. Observation must be made during daylight in the vicinity of sanitary waste outfalls following either the morning or midday meal and at a time during maximum estimated discharge. The number of days solids are observed must be recorded.

b) Limitations

Residual Chlorine. Total residual chlorine (TRC) is a surrogate parameter for fecal coliform. Discharge of TRC must meet a minimum of 1 mg/l and shall be maintained as close to this concentration as possible. A grab sample must be taken once per month and the concentration recorded (approved method, Hach CN-66-DPD).

[Exception] Any facility which properly operates and maintains a marine sanitation device (MSD) that complies with pollution control standards and regulations under section 312 of the Act shall be deemed in compliance with permit prohibitions and limitations for sanitary waste. The MSD shall be tested yearly for proper operation and the test results maintained for three years at the facility or at an alternate site if not practicable.

8. Sanitary Waste (Facilities Continuously Manned for thirty or more consecutive days by 9 or Fewer Persons or Intermittently by Any Number)

a) Prohibitions

Solids. No floating solids may be discharged to the receiving waters. An observation must be made once per day for floating solids. Observation must be made during daylight in the vicinity of sanitary waste outfalls following either the morning or midday meal and at a time during maximum estimated discharge. The number of days solids are observed must be recorded.

[Exception] Any facility which properly operates and maintains a marine sanitation device (MSD) that complies with pollution control standards and regulations under section 312 of the Act shall be deemed to be in compliance with permit prohibitions and limitations for sanitary waste. The MSD shall be tested yearly for proper operation and the test results maintained for three years at the facility or at an alternate site if not practicable.

9. Domestic Waste

a) Prohibitions

Solids. No floating solids or foam shall be discharged.

b) Monitoring Requirements

An observation shall be made once per day during daylight in the vicinity of domestic waste outfalls following the morning or midday meal and at a time during maximum estimated discharge. The number of days solids are observed must be recorded.

10. Miscellaneous Discharges

Desalinization Unit Discharge
 Diatomaceous Earth Filter Media
 Blowout Preventer Control Fluid
 Uncontaminated Ballast Water
 Uncontaminated Bilge Water
 Mud, Cuttings, and Cement at the Seafloor
 Uncontaminated Freshwater
 Uncontaminated Seawater
 Boiler Blowdown
 Source Water and Sand
 Excess Cement Slurry
 Sub sea Wellhead Preservation Fluids
 Sub sea Production Control Fluid
 Hydrate Control Fluid
 Umbilical Steel Tube Storage Fluid
 Leak Tracer Fluid
 Riser Tensioner Fluids

a) Limitations

Free Oil. No free oil shall be discharged. Discharge is limited to those times that a visual sheen observation is possible unless the operator uses the static sheen method. Monitoring shall be performed using the visual sheen method on the surface of the receiving water once per week when discharging, or by use of the static sheen method at the operator's option. The number of days a sheen is observed must be recorded.

[Exceptions] Uncontaminated seawater, uncontaminated freshwater, source water and source sand, uncontaminated bilge water, and uncontaminated ballast water may be discharged from platforms that are on automatic purge systems without monitoring for free oil when the facilities are not manned. Additionally, discharges at the sea floor of: muds and cuttings prior to installation of the marine riser, cement, blowout preventer fluid, sub sea wellhead preservation fluids, sub sea production control fluid, umbilical steel tube storage fluid, leak tracer fluid, and riser tensioner fluids may be discharged without monitoring with the static sheen test when conditions make observation of a visual sheen on the surface of the receiving water impossible. Discharges of muds, cuttings, and cement at the seafloor before installation of the marine riser are exempted from the free oil limitation.

Toxicity. Fluids which are used as Sub sea Wellhead Preservation Fluids, Sub sea Production Control Fluids, Umbilical Steel Tube Storage Fluids, Leak Tracer Fluids,

and Riser Tensioning Fluids shall have a 7-day No Observable Effect Concentration (NOEC) of no less than 50 mg/l. The 7-day NOEC shall be measured using Mysidopsis bahia (Mysid shrimp) chronic static renewal 7-day survival and growth test and Menidia beryllina (Inland Silverside minnow) chronic static renewal 7-day larval survival and growth test (Method 1006.0) as described in Section D.3 of this permit. Compliance with this limit shall be measured at least once per year, using the survival and sub-lethal endpoints, on each fluid added to an operation after the effective date of this permit. If the effluent fails the survival or sub-lethal test endpoint in any test, any discharge associated with use of the product will be considered to be in violation of this permit.

11. **Miscellaneous Discharges of Seawater and Freshwater which have been chemically treated.**

Excess seawater which permits the continuous operation of fire control and utility lift pumps

Excess seawater from pressure maintenance and secondary recovery projects

Water released during training of personnel in fire protection

Seawater used to pressure test new and existing piping and pipelines

Ballast water

Once Through Non-contact cooling water

a) Limitations

Treatment Chemicals. The concentration of treatment chemicals in discharged seawater or freshwater shall not exceed the most stringent of the following three constraints:

- 1) the maximum concentrations and any other conditions specified in the EPA product registration labeling if the chemical is an EPA registered product
- 2) the maximum manufacturer's recommended concentration
- 3) 500 mg/l

Free Oil. No free oil shall be discharged. Discharge is limited to those times that a visible sheen observation is possible unless the operator uses the static sheen method. Monitoring shall be performed using the visual sheen method on the surface of the receiving water once per week when discharging, or by use of the static sheen method at the operator's option. The number of days a sheen is observed must be recorded.

Exception: Monitoring for free oil on discharges from existing piping and existing pipelines shall be performed at least three times per discharge as follows: 1) within thirty minutes after commencement of discharge; 2) at the estimated middle of the discharge; and 3) within fifteen minutes before or after the discharge has ceased.

Toxicity. The 48-hour minimum and monthly average minimum NOEC, must be equal to or greater than the critical dilution concentration specified in this permit in Appendix D, Table 2-A for seawater discharges and 2-B for freshwater discharges. Critical dilution shall be determined using Table 2 in Appendix D of this permit and is based on the discharge rate, discharge pipe diameter, and water depth between the discharge pipe and the bottom. The monthly average minimum NOEC value is defined as the arithmetic average of all 48-hour average NOEC values determined during the month. In cases where the discharge point for hydrostatic test water is sub sea, such as the sub sea end of a pipeline, and it is impractical to collect a sample at the discharge point, operators may collect a sample for this monitoring requirement prior to use of the fluid.

b) Monitoring Requirements

Flow. Once per month, an estimate of the flow (MGD) must be recorded.

Toxicity. The required frequency of testing for continuous discharges shall be determined as follows:

<u>Discharge Rate</u>	<u>Toxicity Testing Frequency</u>
0 - 499 bbl/day	once per annual DMR monitoring period
500 - 4,599 bbl/day	once per calendar quarter
4,600 bbl/day and above	once per month

Intermittent or batch discharges shall be monitored once per discharge but are required to be monitored no more frequently than the corresponding frequencies shown above for continuous discharges.

Samples shall be collected after addition of any added substances, including seawater that is added prior to discharge, and before the flow is split for multiple discharge ports. Samples also shall be representative of the discharge. Methods to increase dilution previously described for produced water in Part I.B.4.a. also apply to seawater and freshwater discharges which have been chemically treated.

If the permittee has been compliant with this toxicity limit for one full year (12 consecutive months) for a continuous or routine intermittent discharge of chemically treated seawater or freshwater, the required testing frequency shall be reduced to once per annual DMR monitoring period for that discharge.

Miscellaneous discharges of seawater and freshwater to which chlorine or hypochlorite have been added are excluded from this monitoring provision.

12. Cooling Water Intake Structure Requirements

Applicability: These requirements apply to new facilities for which construction was commenced after July 17, 2006, with a cooling water intake structure

having a design intake capacity of greater than 2 million gallons of water per day, of which at least 25% is used for cooling purposes.

a) **Baseline Study requirements**

These baseline study requirements are effective one year after the effective date of this permit.

As described below, operators of cooling water intake structures subject to Part I.B.12 may either conduct a study at each new facility or they may participate in an industry wide study. Operators may participate after the close of the study.

Operators of new facilities must submit sufficient information to characterize the biological community of commercial, recreational, and forage base fish and shellfish in the vicinity of the intake structure and to characterize the effects of the cooling water intake structure's operation on aquatic life. This biological characterization must include any available existing information along with field studies to obtain localized data. At a minimum, the information must include:

1. A list of the data required by this section that are not available and efforts made to identify sources of the data;
2. A list of species (or relevant taxa) for all life stages and their relative abundance in the vicinity of the cooling water intake structure;
3. Identification of the species and life stages that would be most susceptible to impingement and entrainment. Species evaluated should include the forage base as well as those most important in terms of significance to commercial and recreational fisheries;
4. Identification and evaluation of the primary period of reproduction, larval recruitment, and period of peak abundance for relevant taxa;
5. Data representative of the seasonal and daily activities (e.g., feeding and water column migration) of biological organisms in the vicinity of the cooling water intake structure;
6. Identification of all threatened, endangered, and other protected species that might be susceptible to impingement and entrainment at your cooling water intake structures;
7. If the information above is supplemented with data from field studies, the supplemental data must include a description of all methods and quality assurance procedures for sampling and data analysis including a description of the study area; taxonomic identification of sampled and evaluated biological assemblages (including all life stages of fish and shellfish); and sampling and data analysis

methods. The sampling and/or data analysis methods you use must be appropriate for a quantitative survey and based on consideration of methods used in other biological studies performed within the same source water body. The study area should include, at a minimum, the area of influence of the cooling water intake structure.

Alternatively, operators may comply with these requirements and the entrainment monitoring requirements in section B.12.d of this permit through participation in an EPA approved industry wide study. That study may include a smaller, statistically representative number of facilities. Any industry wide baseline study which is conducted must be commenced within one year after the effective date of this permit and completed within two years after the effective date. Any industry wide study conducted to meet the entrainment monitoring requirements in section B.12 must be commenced within two years after the effective date of this permit or the installation of a new facility subject to the cooling water intake structure requirements of Part I.B.12 whichever is later. The industry wide study must be completed three years after its commencement.

b) **Supplemental Notification Requirements**

Design information must be submitted at least 30 days in advance of a facility commencing operations in the geographical area covered by this permit. Design information required to be submitted for cooling water intake structures is only required to be submitted once for any facility that these requirements are applicable. Design information is not required to be resubmitted for additional leases where the facility subsequently operates or for a subsequent permit. EPA will notify the operator if additional information is required. Owners/operators of MODUs must also submit an NOI, in accordance with Part I.A.2 of this permit. The NOI shall be submitted and postmarked prior to operating.

New non-fixed facilities must submit:

A narrative description and/or maps providing sufficient information on predicted locations during the permit term in sufficient detail for the Director to determine the appropriateness of additional impingement requirements. This information is only required to be submitted once for any facility.

Velocity information, including:

1. A narrative description of the design, structure, equipment, and operation used to meet the requirements of a maximum through screen intake velocity of 0.5 ft/s at each cooling water intake structure; and

2. For surface cooling water intake screens only, design calculations showing that the velocity requirement will be met at the minimum ambient source water surface elevation and maximum head loss across the screens or other device.

Cooling water intake structure data, including:

1. Design and construction technology plans and a description of operational measures which will be implemented to minimize impingement, including:
 - ii. A narrative description of the design, operation of the design, and construction technologies including fish handling and return systems that the facility will utilize to maximize the survival of species expected to be most susceptible to impingement. Provide species specific information that demonstrates the efficacy of the technology; and
 - iii. Design calculations, drawings, and estimates to support the descriptions above.
2. A narrative description of the configuration of each of your cooling water intake structures and where it is located in the water body and in the water column;
3. A narrative description of the operation of each of your cooling water intake structures, including design intake flows, daily hours of operation, number of days of the year in operation, and seasonal changes, if applicable;
4. A flow distribution and water balance diagram that includes all sources of water to the facility, recirculating flows, and discharges; and
5. Engineering drawings of the cooling water intake structure.

New Fixed Facilities must submit:

Source water physical data, including:

1. A narrative description and scaled drawings showing the physical configuration of all source water bodies used by your facility, including aerial dimensions, depths, salinity and temperature regimes, and other documentation that supports your determination of the water body type where each cooling water intake structure is located;
2. Identification and characterization of the source water body's hydrological and geomorphological features, as well as the methods you used to conduct any studies to determine your intake's area of influence within the water body and the results of such studies; and

3. Locational maps.

Cooling water intake structure data, including:

1. Design and construction technology plans and a description of operational measures which will be implemented to minimize impingement, including:
 - i. A narrative description of the design and operation of the design and construction technologies including fish handling and return systems that the facility will utilize to maximize the survival of species expected to be most susceptible to impingement; and
 - ii. For those new fixed facilities that do not employ sea chests as cooling water intake structures, a narrative description of the design, operation, and construction technologies that the facility will utilize to minimize entrainment of those species most susceptible to entrainment.
 - iii. Design calculations, drawings, and estimates to support the design technologies.
2. A narrative description of the configuration of each of your cooling water intake structures and where it is located in the water body and in the water column;
3. Latitude and longitude in degrees, minutes, and seconds for each of your cooling water intake structures;
4. A narrative description of the operation of each of your cooling water intake structures, including design intake flows, daily hours of operation, number of days of the year in operation and seasonal changes, if applicable;
5. A flow distribution and water balance diagram that includes all sources of water to the facility, re-circulating flows, and discharges; and
6. Engineering drawings of the cooling water intake structure.

Velocity information, including:

1. A narrative description of the design, structure, equipment, and operation used to meet the requirement of a maximum through-screen intake velocity of 0.5 ft/s at each cooling water intake structure; and
2. For surface cooling water intake screens only, design calculations showing that the velocity requirement will be met at the minimum ambient source water surface elevation and maximum head loss across the screens or other device.

c) **Cooling Water Intake Structure Requirements**

New non-Fixed Facilities

1. The cooling water intake structure(s) must be designed and constructed so that the maximum through-screen design intake velocity is 0.5 ft/s or less;
2. The operator must minimize impingement mortality of fish and shellfish through use of cooling water intake design and construction technologies or operational measures.

New Fixed Facilities that do not employ sea chests as intake structures

1. The cooling water intake structure must be designed and constructed so that the maximum through-screen design intake velocity is 0.5 ft/s; and
2. The operator must minimize impingement mortality of fish and shellfish and minimize entrainment of entrainable life stages of fish and shellfish through the use of cooling water intake design and construction technologies or operational measures.

New Fixed Facilities that Employ Sea Chests as Intake Structures

1. The cooling water intake structure(s) must be designed and constructed so that the maximum through-screen design intake velocity is 0.5 ft/s or less; and
2. The operator must minimize impingement mortality of fish and shellfish through cooling water intake design and construction technologies or operational measures.

d) **Monitoring Requirements**

New non-Fixed Facilities

1. Beginning two years after the effective date of this permit, the operator must conduct either visual inspections or use remote monitoring devices during the period the cooling water intake structure is in operation. The operator must conduct visual inspections at least weekly, or at a lesser frequency as approved by the director, to ensure that the required design and construction technologies are maintained and operated so they continue to function as designed. Alternatively, the operator must inspect using remote monitoring devices to ensure that the impingement and entrainment technologies are functioning as designed.

However, visual or remote monitoring is not required when conditions such as storms, high seas, evacuation, or other factors make it unduly hazardous to

personnel, the facility, or the equipment utilized. The operator must provide an explanation for any such failure to visually or remotely monitor with the subsequent DMR submittal.

2. For facilities that employ surface intake screens systems, the operator must monitor intake velocity by measuring the head loss across the intake screens and correlating the measured value with the design intake velocity. The operator must measure the head loss at the minimum ambient source water surface elevation using best professional judgment based on available hydrological data. The operator must use the maximum head loss across the screen for each cooling water intake structure to determine compliance with the velocity requirement. For facilities utilizing devices other than surface intake screens, the facility shall monitor intake velocity at the point of entry through the intake device or through a comparable method such as pump curve calculations. The operator shall monitor either head loss or velocity during initial facility startup, and thereafter, at a frequency of no less than once per quarter.

New Fixed Facilities that do not employ sea chests as intake structures

1. Beginning two years after the effective date of this permit, the operator must conduct either visual inspections or use remote monitoring devices during the period the cooling water intake structure is in operation. The operator must conduct visual inspections at least weekly, or at a lesser frequency as approved by the director, to ensure that the required design and construction technologies are maintained and operated so they continue to function as designed. Alternatively, the operator must inspect using remote monitoring devices to ensure that the impingement and entrainment technologies are functioning as designed.

However, visual or remote monitoring is not required when conditions such as storms, high seas, evacuation, or other factors make it unduly hazardous to personnel, the facility, or the equipment utilized. The operator must provide an explanation for any such failure to visually or remotely monitor with the subsequent DMR submittal.

2. Beginning two years after the effective date of the permit or after commencement of operations, whichever is later, the operator must monitor for entrainment. The operator must collect samples to monitor entrainment rates (simple enumeration) for each species over a 24-hour period and no less than biweekly during the primary period of reproduction, larval recruitment, and peak abundance identified during the Source Water Baseline Biological Characterization Study. Representative species may be utilized for this monitoring consistent with their use in the Source Water Baseline Characterization Study. The operator must collect samples only when the cooling water intake structure is in operation. After 24 months of monitoring, the permittee may request from EPA a reduced monitoring frequency for the remainder of the permit.

Alternatively, operators may comply with these requirements through participation in an EPA approved industry-wide study. That study may include a smaller, statistically representative number of facilities. See also section B.12.a of this permit. Any industry wide study conducted to meet the entrainment monitoring requirements in section B.12 must be commenced within two years after the effective date of this permit and must be completed and submitted to EPA Region 6 three years after the effective date.

3. For facilities that employ surface intake screens systems, the operator shall monitor intake velocity by measuring the head loss across the intake screens and correlating the measured value with the design intake velocity. The operator must measure head loss at the minimum ambient source water surface elevation using best professional judgment based on available hydrological data. The operator must use the maximum head loss across the screen for each cooling water intake structure to determine compliance with the velocity requirement. For facilities utilizing devices other than surface intake screens, intake velocity shall be monitored at the point of entry through the intake device or through a comparable method such as pump curve calculations. The operator shall monitor either head loss or velocity during initial facility startup, and thereafter, at a frequency of no less than once per quarter.

New Fixed Facilities that Employ Sea Chests as Intake Structures

1. Beginning two years after the effective date of this permit, the operator must conduct either visual inspections or utilize remote monitoring devices during the period the cooling water intake structure is in operation. The operator must conduct visual inspections at least weekly, or at a lesser frequency as approved by the director, to ensure that the required design and construction technologies are maintained and operated so they continue to function as designed. Alternatively, the operator must inspect using remote monitoring devices to ensure that the impingement and entrainment technologies are functioning as designed.

However, visual or remote monitoring is not required when conditions such as storms, high seas, evacuation, or other factors make it unduly hazardous to personnel, the facility, or the equipment utilized. The operator must provide an explanation for any such failure to visually or remotely monitor with the subsequent DMR submittal.

2. For facilities that employ surface intake screens systems, the operator shall monitor the intake velocity by monitoring the head loss across the intake screens and correlating the measured value with the design intake velocity. The operator must measure head loss at the minimum ambient source water surface elevation using best professional judgment based on available hydrological data. The operator must use the maximum head loss across the screen for each cooling water intake

structure to determine compliance with the velocity requirement. For facilities utilizing devices other than surface intake screens, intake velocity shall be monitored at the point of entry through the intake device or through a comparable method such as pump curve calculations. The operator must monitor either head loss or velocity during initial facility startup, and thereafter, at a frequency of no less than once per quarter

3. No monitoring for entrainment is required.

A status report of the required biological monitoring for each cooling water intake structure must be provided to EPA with the annual DMR for fixed facilities that do not employ sea chests.

The permit may be reopened and modified or revoked and reissued to require additional monitoring or to change the cooling water intake structure requirements if found warranted by the director as a result of either baseline study or entrainment monitoring.

Section C. Other Discharge Limitations

1. Floating Solids or Visible Foam

There shall be no discharge of floating solids or visible foam from any source in other than trace amounts.

[Exception] For new sources, this limitation only applies to miscellaneous discharges and domestic waste discharges.

2. Halogenated Phenolic Compounds

There shall be no discharge of halogenated phenolic compounds as a part of any waste stream authorized in this permit.

3. Dispersants, Surfactants, and Detergents

The facility operator shall minimize the discharge of dispersants, surfactants and detergents except as necessary to comply with the safety requirements of the Occupational Safety and Health Administration and the Minerals Management Service. This restriction applies to tank cleaning and other operations which do not directly involve the safety of workers. The restriction is imposed because detergents disperse and emulsify oil, thereby increasing toxicity and making the detection of a discharge of oil more difficult. Waste water associated with tank and pit cleaning operations shall be classified the same as the former contents of the tank or pit (for example, wash water generated from cleaning drilling fluid pits would be subject to the same discharge limitation as the drilling fluid formerly contained in those pits). The waste water is deemed to have the same compliance status as

the whole fluid that was originally in the tank or pit. No additional sampling/monitoring of the waste water is required.

4. Garbage

The discharge of garbage (See Part II.G.42) is prohibited.

[Exception] Comminuted food waste (able to pass through a screen mesh no larger than 25 mm, approx. 1 inch) may be discharged when 12 nautical miles or more from land.

5. Areas of Biological Concern and Marine Sanctuaries

There shall be no discharge in Areas of Biological Concern and National Marine Sanctuaries. The Flower Garden Banks has been determined to be a National Marine Sanctuary and is within the geographical area covered under this permit.

[Exception]

Facilities located within a National Marine Sanctuary boundary are authorized to discharge in accordance with this permit if **all** of the following conditions are met:

- The platform was installed prior to the designation of the National Marine Sanctuary;
- The platform is located outside of the No Activity Zone defined by the Minerals Management Service;
- All materials are discharged through a shunt pipe that terminates within 10 meters of the sea floor;
- Sanitary waste is treated with an approved marine sanitation device (MSD) that complies with pollution control standards and regulations under section 312 of the Clean Water Act;

and

- The materials discharged are associated with and incidental to oil and gas exploration, development, or production and originate from wells located within the boundaries of the National Marine Sanctuary and outside the No Activity Zone.

6. Wastes Associated with Maintenance Activities such as Surface Preparation and Coating

Maintenance waste, such as removed paint and materials associated with surface preparation and coating applications, must be contained to the maximum extent practicable to prevent discharge. This includes airborne material such as spent or oversprayed abrasives, paint chips, and paint

overspray. Measures such as vacuum abrasive blasting, covering grated areas with plywood, surrounding the area with canvas tarps and similar measures must be employed to capture as much material as practicable. All collected material shall be disposed of at an appropriate shore based facility. Prior to conducting sandblasting or similar maintenance activities, operators shall operate in accordance with the API Recommended Practice (RP91) for Containment of Spent Blast Abrasive and Associated Materials from Surface Preparation and Coating Operations, if approved by EPA and published, or develop and implement a Best Management Practices (BMP) plan for the containment of waste materials. Operators shall supplement RP91 with company or site specific BMPs as needed. Any BMP utilized must include specific containment measures.

Section D. Other Conditions

1. Samples of Wastes

If requested, the permittee shall provide EPA with a sample of any waste in a manner specified by the Agency.

2. Drilling Fluids Toxicity Test

The approved test method for permit compliance is identified as:
Drilling Fluids Toxicity Test at 40 CFR Part 435, Subpart A, Appendix 2.

3. PRODUCED WATER TOXICITY TESTING REQUIREMENTS (7-DAY CHRONIC NOEC MARINE LIMITS)

The approved test methods for permit compliance are identified in 40 CFR Part 136.

- a) The permittee shall utilize the Mysidopsis bahia (Mysid shrimp) chronic static renewal 7-day survival and growth test using Method 1007.0. A minimum of eight (8) replicates with five (5) organisms per replicate must be used in the control and in each effluent dilution of this test.
- b) The permittee shall utilize the Menidia beryllina (Inland Silverside minnow) chronic static renewal 7-day larval survival and growth test (Method 1006.0). A minimum of five (5) replicates with eight (8) organisms per replicate must be used in the control and in each effluent dilution of this test.
- c) The NOEC (No Observed Effect Concentration) is defined as the greatest effluent dilution which does not result in a lethal or sub-lethal effect that is statistically different from the control (0% effluent) at the 95% confidence level. In the case of a test that exhibits a non-monotonic concentration response, determination of the NOEC will rely

on the procedures described in *Method Guidance and Recommendations for Whole Effluent Toxicity (WET) Testing (40 CFR Part 136)*, July 2000, EPA 821-B-00-004.

- d) The effluent dilution series used for the toxicity test shall be based on the critical dilution, using a dilution factor of 0.5. The effluent dilution series must bracket the critical dilution, with two effluent dilutions lower than the critical dilution and two effluent dilutions greater than the critical dilution.
- e) If the effluent fails the survival endpoint (or the sub-lethal endpoint, after two years from the effective date of this permit) at the critical dilution, the permittee shall be considered in violation of this permit limit. Also, when the testing frequency stated above is less than monthly and the effluent fails either endpoint at the critical dilution, the monitoring frequency for the affected species will increase to monthly until such time as compliance with the NOEC effluent limitation is demonstrated for a period of three consecutive months, at that time the permittee may return to the testing frequency in use at the time of the failure. During the period the permittee is out of compliance, test results shall be reported on the DMR for that reporting period.
- f) This permit may be reopened to require chemical specific effluent limits, additional testing, and/or other appropriate actions to address toxicity.
- g) **Test Acceptance**
The permittee shall repeat a test, including the control and all effluent dilutions, if the procedures and quality assurance requirements defined in the test methods or in this permit are not satisfied, including the following additional criteria:
 - i. The toxicity test control (0% effluent) must have survival equal to or greater than 80%.
 - ii. The mean dry weight of surviving Mysid shrimp at the end of the 7 days in the control (0% effluent) must be 0.20 mg per mysid or greater. Should the mean dry weight in the control be less than 0.20 mg per mysid, the toxicity test, including the control and all effluent dilutions shall be repeated.
 - iii. The mean dry weight of surviving unpreserved Inland Silverside minnow larvae at the end of the 7 days in the control (0% effluent) must be 0.50 mg per larva or greater. The mean dry weight of surviving preserved Inland Silverside minnow larvae at the end of the 7 days in the control (0% effluent) must be 0.43 mg per larva or greater.
 - iv. The percent coefficient of variation (%CV) between replicates shall be 40% or less in the control (0% effluent) for: the growth and survival endpoints of the Mysid shrimp test and the Inland Silverside minnow test. The %CV for survival shall be calculated on the arc-sine-square-root transformed data. The %CV for growth shall be calculated on the growth per surviving organism.

- v. The percent coefficient of variation between replicates shall be 40% or less in the critical dilution, unless significant lethal or nonlethal effects are exhibited for the growth and survival endpoints of the Mysid shrimp test and the Inland Silverside minnow test.
- vi. A Percent Minimum Significant Difference (PMSD) range of 11 - 37 for *Mysidopsis bahia* growth shall be applied as described in *Short-term Methods for Estimating the Chronic Toxicity of Effluents and Receiving Waters to Marine and Estuarine Organisms*, Third Edition, October 2002, EPA-821-R-02-014, Section 10.2.8.
- vii. A PMSD range of 11 - 28 for Silverside minnow growth shall be applied as described in *Short-term Methods for Estimating the Chronic Toxicity of Effluents and Receiving Waters to Marine and Estuarine Organisms*, Third Edition, October 2002, EPA-821-R-02-014 or the most recent update thereof.

Test failure may not be construed or reported as invalid due to a coefficient of variation value of greater than 40%. A repeat test shall be conducted within the required reporting period of any test determined to be invalid.

h) Statistical Interpretation

For the Mysid shrimp survival and reproduction test and the Inland Silverside minnow survival and growth test, the statistical analyses used to determine if there is a statistically significant difference between the control and the critical dilution shall be in accordance with the methods for determining the NOEC as described in EPA-821-R-02-012 or the most recent update thereof.

If the conditions of Test Acceptability are met in Item 3.f above and the percent survival of the test organism is equal to or greater than 80% in the critical dilution concentration and all lower dilution concentrations, the survival test shall be considered to be passing, and the permittee shall report a survival NOEC of not less than the critical dilution for the DMR reporting requirements found below.

- i) The permittee shall prepare a full report of the results of all tests conducted pursuant to this section in accordance with the Report Preparation Section of "Short-Term Methods for Estimating the Chronic Toxicity of Effluents and Receiving Waters to Marine and Estuarine Organisms", EPA-821-R-02-014, or the most current publication, for every valid or invalid toxicity test initiated whether carried to completion or not. The permittee shall retain each full report pursuant to the provisions of Part II.C.3 of this permit. The permittee shall submit full reports only upon the specific request of the Agency.
- j) In accordance with Part II.D.4 of this permit, the permittee shall report on the DMR for the reporting period the lowest Whole Effluent Lethality values determined for either

species for the 30-Day Average Minimum and 7-Day Minimum under Parameter No. 22414, and the permittee shall report only the results of the valid toxicity test as follows:

- i. *Menidia beryllina* (Inland Silverside minnow)
 - A) If the NOEC for survival is less than the critical dilution, enter a "1"; otherwise, enter a "0" for Parameter No. TLP6B
 - B) Report the NOEC value for survival, Parameter No. TOP6B
 - C) Report the Lowest Observed Effect Concentration (LOEC) value for survival, Parameter No. TXP6B
 - D) Report the NOEC value for growth, Parameter No. TPP6B
 - E) Report the LOEC value for growth, Parameter No. TYP6B
 - F) If the NOEC for growth is less than the critical dilution, enter a "1"; otherwise, enter a "0" for Parameter No. TGP6B
 - G) Report the highest (critical dilution or control) Coefficient of Variation, Parameter No. TQP6B
- ii. *Mysidopsis bahia* (Mysid shrimp)
 - A) If the NOEC for survival is less than the critical dilution, enter a "1"; otherwise, enter a "0" for Parameter No. TLP3E
 - B) Report the NOEC value for survival, Parameter No. TOP3E
 - C) Report the LOEC value for survival, Parameter No. TXP3E
 - D) Report the NOEC value for growth, Parameter No. TPP3E
 - E) Report the LOEC value for growth, Parameter No. TYP3E
 - F) If the NOEC for growth is less than the critical dilution, enter a "1"; otherwise, enter a "0" for Parameter No. TGP3E
 - G) Report the highest (critical dilution or control) Coefficient of Variation, Parameter No. TQP3E

4. **CHEMICALLY TREATED SEAWATER AND FRESHWATER TOXICITY TESTING REQUIREMENTS (48-HOUR ACUTE NOEC MARINE LIMITS)**

The approved test methods for permit compliance are identified in 40 CFR Part 136.

- a) The permittee shall utilize the Mysidopsis bahia (Mysid shrimp) acute static renewal 48-hour definitive toxicity test using EPA-821-R-02-012. A minimum of five (5) replicates with eight (8) organisms per replicate must be used in the control and in each effluent dilution of this test.
- b) The permittee shall utilize the Menidia beryllina (Inland Silverside minnow) acute static renewal 48-hour definitive toxicity test using EPA-821-R-02-012. A minimum of five (5) replicates with eight (8) organisms per replicate must be used in the control and in each effluent dilution of this test.
- c) The NOEC is defined as the greatest effluent dilution which does not result in lethality that is statistically different from the control (0% effluent) at the 95% confidence level.
- d) If the effluent fails the survival endpoint at the critical dilution, the permittee shall be considered in violation of this permit limit. Also, when the testing frequency stated above is less than monthly and the effluent fails the survival endpoint at the critical dilution, the monitoring frequency for the affected species will increase to monthly until such time as compliance with the Lethal NOEC effluent limitation is demonstrated for a period of three consecutive months. After compliance is demonstrated for three consecutive months, the permittee may return to the testing frequency in use at the time of the initial test failure. During the period the permittee is out of compliance, test results shall be reported on the annual DMR that includes this period.
- e) This permit may be reopened to require chemical specific effluent limits, additional testing, and/or other appropriate actions to address toxicity.
- f) Test Acceptance
The permittee shall repeat a test, including the control and all effluent dilutions, if the procedures and quality assurance requirements defined in the test methods or in this permit are not satisfied, including the following additional criteria:
 - i. Each toxicity test control (0% effluent) must have a survival equal to or greater than 90%.
 - ii. The percent coefficient of variation between replicates shall be 40% or less in the control (0% effluent) for the Mysid shrimp survival test and the Inland Silverside minnow survival test.

- iii. The percent coefficient of variation between replicates shall be 40% or less in the critical dilution, unless significant lethal effects are exhibited for the Mysid shrimp survival test and the Inland Silverside minnow survival test.

Test failure may not be construed or reported as invalid due to a coefficient of variation value of greater than 40%. A repeat test shall be conducted within the required reporting period of any test determined to be invalid.

g) **Statistical Interpretation**

For the Mysid shrimp survival test and the Inland Silverside minnow survival test, the statistical analyses used to determine if there is a statistically significant difference between the control and the critical dilution shall be in accordance with the methods for determining the NOEC as described in EPA-821-R-02-012 or the most recent update thereof.

If the conditions of Test Acceptability are met in Item 4.f above and the percent survival of the test organism is equal to or greater than 90% in the critical dilution concentration and all lower dilution concentrations the test shall be considered to be a passing test, and the permittee shall report an NOEC of not less than the critical dilution for the DMR reporting requirements found in Item i below.

- h) The permittee shall prepare a full report of the results of all tests conducted pursuant to this section in accordance with the Report Preparation Section of "Methods for Measuring the Acute Toxicity of Effluents and Receiving Waters to Freshwater and Marine Organisms," EPA-821-R-02-012, or the latest update thereof, for every valid or invalid toxicity test initiated whether carried to completion or not. The permittee shall retain each full report pursuant to the provisions of Part II.C.3 of this permit. The permittee shall submit full reports only upon the specific request of the Agency.
- i) In accordance with Part II.D.4 of this permit, the permittee shall report on the DMR for the reporting period whether the lowest Whole Effluent Lethality values determined for either species passed the 30-Day Average Minimum and 48-Hour Minimum NOEC.

5. VISUAL SHEEN TEST

The visual sheen test is used to detect free oil by observing the surface of the receiving water for the presence of a sheen while discharging. The operator must conduct a visual sheen test only at times when a sheen could be observed. This restriction eliminates observations when atmospheric or surface conditions prohibit the observer from detecting a sheen (e.g., overcast skies, rough seas, etc.).

The observer must be positioned on the rig or platform, relative to both the discharge point and current flow at the time of discharge, such that the observer can detect a sheen should it surface down current from the discharge. For discharges that have been occurring for a least 15 minutes previously, observations may be made any time thereafter. For discharges of less than

15 minutes duration, observations must be made during both discharge and at 5 minutes after discharge has ceased.

6. STATIC SHEEN TEST

The approved test method for permit compliance is identified as: Static Sheen Test at 40 CFR Part 435, Subpart A, Appendix 1.

7. STOCK BASE FLUID SEDIMENT TOXICITY

The approved test method for permit compliance is identified as: ASTM E1367–99 method: Standard Guide for Conducting Static Sediment Toxicity Tests with Marine and Estuarine Amphipods (Available from the American Society for Testing and Materials, 100 Barr Harbor Drive, West Conshohocken, PA, 19428) with *Leptocheirus plumulosus* as the test organism and sediment preparation procedures specified in Appendix 3 of 40 CFR Part 435, Subpart A and the method found in Appendix A of this permit.

8. BIODEGRADATION RATE

The approved test method for permit compliance is identified as: modified ISO 11734:1995 method: “Water quality - Evaluation of the ‘ultimate’ anaerobic biodegradability of organic compounds in digested sludge - Method by measurement of the biogas production (1995 edition)” (Available from the American National Standards Institute, 11 West 42nd Street, 13th Floor, New York, NY 10036) supplemented with modifications in Appendix 4 of 40 CFR Part 435, Subpart A and detailed in Appendix B of this permit. Compliance with the biodegradation limit will be determined using the following ratio:

$$\frac{\% \text{ Theoretical gas production of reference fluid}}{\% \text{ Theoretical gas production of NAF}} \quad \# \ 1.0$$

Where: NAF = stock base fluid being tested for compliance
 Reference Fluid = C₁₆-C₁₈ internal olefin or C₁₂-C₁₄ or C₈ ester reference fluid

9. SAMPLING PROTOCOL FOR STOCK DRILLING FLUID SEDIMENT TOXICITY TEST, DRILLINGFLUID SEDIMENT TOXICITY TEST AND BIODEGRADATION RATE TEST

Compliance with the 1.0 ratio permit limit shall be based on the ratio of the arithmetic average of up to three test results from two grab samples. The first grab sample must be split into two aliquots (e.g., grab1A and grab1B) and analyzed separately. The second grab sample (grab2) shall be a backup sample, collected within 15 minutes of the first grab sample, and in the case of base fluid testing will be from the same production lot, which shall be retained following proper storage and handling procedures. Permittees shall show compliance based on results from grab1A, or from the ratio of the arithmetic average of grab1A, grab1B, and if necessary grab 2.

All test results obtained shall be submitted with the DMR and all ratios shall be rounded to the nearest tenths.

All test results shall be generated as follows:

- a. The 10-day stock base fluid toxicity test results consist of individual stock base fluid LC50s and individual reference fluid LC50s (paired results). The arithmetic average of the LC50 for the test fluid sample(s) will be compared to determine compliance with the 1.0 ratio permit limit.
- b. The stock base fluid biodegradation test results consist of individual stock base fluid cumulative gas production (ml) and individual reference fluid cumulative gas production (ml) tests (paired results). The arithmetic average of the cumulative gas production (ml) for the test fluid samples(s) will be compared against the arithmetic average of the cumulative gas production (ml) of the reference fluid sample(s) to determine compliance with the 1.0 ratio permit limit.
- c. The 4-day drilling fluid mud toxicity test results consist of the individual field mud LC50s and individual reference mud LC50s (paired results). The arithmetic average of the LC50 for the field mud sample(s) will be compared against the arithmetic average of the LC50 of the reference mud sample(s) to determine compliance with the 1.0 ratio permit limit.

10. **POLYNUCLEAR AROMATIC HYDROCARBONS**

The approved test method for permit compliance is identified as: *Method 1654A*: "PAH Content of Oil by High Performance Liquid Chromatography with a UV Detector," which was published in Methods for the Determination of Diesel, Mineral and Crude Oils in Offshore Oil and Gas Industry Discharges, EPA-821-R-92-008 (incorporated by reference and available from National Technical Information Service at 703/605-6000).

11. **FORMATION OIL CONTAMINATION OF DRILLING FLUIDS**

The approved test method for permit compliance is identified as: Gas chromatography/mass spectrometry (GC/MS) as described below. The GC/MS method reports results for the GC/MS test as percent crude contamination when calibrated for a specific crude oil. In order to define an applicable pass/fail limit to cover a variety of crude oils, the same crude oil used in calibration of the RPE test shall be used to calibrate the GC/MS test results to a standardized ratio of the target aromatic ION Scan 105. Based on the performance of a range of crude oils against standardized ratio, a value will be selected as a pass/fail standard which will represent detection of crude oil.

12. **FORMATION OIL CONTAMINATION OF DISCHARGED DRILLING FLUIDS RETAINED ON CUTTINGS**

The approved test method for permit compliance is identified as: Reverse Phase Extraction (RPE) as described in Appendix 6 of 40 CFR Part 435, Subpart A. If the operator wishes to confirm the results of the RPE method (Appendix 6 of 40 CFR Part 435, Subpart A), the operator may use the GC/MS compliance assurance method (Appendix 5 of 40 CFR Part 435, Subpart A). Results from the GC/MS compliance assurance method shall supercede the results of the RPE method (Appendix 6 of 40 CFR Part 435, Subpart A).

13. **RETENTION OF NON AQUEOUS BASED DRILLING FLUID ON CUTTINGS**

The approved test method for permit compliance is identified as: the Retort Test Method described in Appendix 7 of 40 CFR Part 435, Subpart A. The required sampling, handling, and documentation procedures are listed in Addendum A of 40 CFR Part 435, Subpart A, Appendix 7.

14. **ROUNDING OF RATIOS (TO BE APPLIED IN MEASURING COMPLIANCE WITH THE SEDIMENT TOXICITY AND BIODEGRADATION TESTS)**

All ratios shall be rounded as follows:

The following rounding procedures shall only be applied to the sediment toxicity and biodegradation limitations and standards in this permit:

- a) If the digit 6, 7, 8, or 9 is dropped, increase preceding digit by one unit.
Example: a calculated sediment toxicity or biodegradation ratio of 1.06 should be rounded to 1.1 and reported as a violation of the permit limit.
- b) If the digit 0, 1, 2, 3, or 4 is dropped, do not alter the preceding digit.

Example: a calculated sediment toxicity ratio of 1.04 should be rounded to 1.0 and reported to EPA as compliant with the permit limit.
- c) If the digit 5 is dropped, round off preceding digit to the nearest even number.

Example: a calculated ratio of 1.05 should be rounded to 1.0 and reported to EPA as compliant with the permit limit.

PART II. STANDARD CONDITIONS FOR NPDES PERMITS

Section A. General Conditions

1. Introduction

In accordance with the provisions of 40 CFR Part 122.41, *et. seq.*, this permit incorporates by reference ALL conditions and requirements applicable to NPDES permits set forth in the

Clean Water Act, as amended, (herein-after known as the "Act") as well as ALL applicable regulations.

2. Duty to Comply

The permittee must comply with all conditions of this permit. Any permit noncompliance constitutes a violation of the Act and is grounds for enforcement action or for requiring a permittee to apply and obtain an individual NPDES permit.

3. Toxic Pollutants

- a. Notwithstanding Part II.A.4, if any toxic effluent standard or prohibition (including any schedule of compliance specified in such effluent standard or prohibition) is promulgated under section 307(a) of the Act for a toxic pollutant which is present in the discharge and that standard or prohibition is more stringent than any limitation on the pollutant in this permit, this permit shall be modified or revoked and reissued to conform to the toxic effluent standard or prohibition.
- b. The permittee shall comply with effluent standards or prohibitions established under section 307(a) of the Act for toxic pollutants within the time provided in the regulations that established those standards or prohibitions, even if the permit has not yet been modified to incorporate the requirement.

4. Permit Flexibility

This permit may be modified, revoked and reissued, or terminated for cause in accordance with 40 CFR 122.62-64. The filing of a request for a permit modification, revocation and reissuance, or termination, or a notification of planned changes or anticipated noncompliance, does not stay any permit condition.

5. Property Rights

This permit does not convey any property rights of any sort, or any exclusive privilege.

6. Duty to Provide Information

The permittee shall furnish to the Director, within a reasonable time, any information which the Director may request to determine whether cause exists for modifying, revoking and reissuing, or terminating this permit, or to determine compliance with this permit. The permittee shall also furnish to the Director, upon request, copies of records required to be kept by this permit.

7. Criminal and Civil Liability

Except as provided in permit conditions on "Bypassing" and "Upsets", nothing in this permit shall be construed to relieve the permittee from civil or criminal penalties for noncompliance. Any false or materially misleading representation or concealment of information required to be reported by the provisions of the permit, the Act, or applicable regulations, which avoids or effectively defeats the regulatory purpose of the permit may subject the permittee to criminal enforcement pursuant to 18 U.S.C. section 1001.

8. Oil and Hazardous Substance Liability

Nothing in this permit shall be construed to preclude the institution of any legal action or relieve the permittee from any responsibilities, liabilities, or penalties to which the permittee is or may be subject under section 311 of the Act.

9. State Laws

Nothing in this permit shall be construed to preclude the institution of any legal action or relieve the permittee from any responsibilities, liabilities, or penalties established pursuant to any applicable State Law or regulation under authority preserved by section 510 of the Act.

10. Severability

The provisions of this permit are severable, and if any provision of this permit or the application of any provision of this permit to any circumstance is held invalid, the application of such provision to other circumstances, and the remainder of this permit, shall not be affected thereby.

Section B. Proper Operation and Maintenance

1. Need to Halt or Reduce not a Defense

It shall not be a defense for a permittee in an enforcement action that it would have been necessary to halt or reduce the permitted activity in order to maintain compliance with the conditions of this permit. The permittee is responsible for maintaining adequate safeguards to prevent the discharge of untreated or inadequately treated wastes during electrical power failure either by means of alternate power sources, standby generators or retention of inadequately treated effluent.

2. Duty to Mitigate

The permittee shall take all reasonable steps to minimize or prevent any discharge in violation of this permit which has a reasonable likelihood of adversely affecting human health or the environment.

3. Proper Operation and Maintenance

- a. The permittee shall at all times properly operate and maintain all facilities and systems of treatment and control (and related appurtenances) which are installed or used by permittee as efficiently as possible and in a manner which will minimize upsets and discharges of excessive pollutants and will achieve compliance with the conditions of this permit. Proper operation and maintenance also includes adequate laboratory controls and appropriate quality assurance procedures. This provision requires the operation of backup or auxiliary facilities or similar systems which are installed by a permittee only when the operation is necessary to achieve compliance with the conditions of this permit.
- b. The permittee shall provide an adequate operating staff which is duly qualified to carry out operation, maintenance and testing functions required to insure compliance with the conditions of this permit.

4. Bypass of Treatment Facilities

- a. Bypass not exceeding limitations. The permittee may allow any bypass to occur which does not cause effluent limitations to be exceeded, but only if it also is for essential maintenance to assure efficient operation. These bypasses are not subject to the provisions of Parts II.B.4.b and 4.c. Sanitary Waste discharges which are excepted from discharge limitations due to the proper operation and maintenance of a Coast Guard approved Marine Sanitation Device may allow a bypass during essential maintenance and are not considered to cause effluent limitations to be exceeded.

b. Notice

- (1) Anticipated bypass. If the permittee knows in advance of the need for a bypass, it shall submit prior notice, if possible at least ten days before the date of the bypass.
- (2) Unanticipated bypass. The permittee shall, within 24 hours, submit notice of an unanticipated bypass as required in Part II.D.7.

c. Prohibition of Bypass

- (1) Bypass is prohibited, and the Director may take enforcement action against a permittee for bypass, unless:
 - (a) Bypass was unavoidable to prevent loss of life, personal injury, or severe property damage;
 - (b) There were no feasible alternatives to the bypass, such as the use of auxiliary treatment facilities, retention of untreated wastes, or maintenance during normal periods of equipment downtime. This condition is not satisfied if adequate back-up equipment should have been installed in the exercise of

reasonable engineering judgement to prevent a bypass which occurred during normal periods of equipment downtime or preventive maintenance; and,

(c) The permittee submitted notices as required by Part II.B.4.b.

(2) The Director may allow an anticipated bypass after considering its adverse effects, if the Director determines that it will meet the three conditions listed at Part II.B.4.c(1).

5. Upset Conditions

a. Effect of an upset. An upset constitutes an affirmative defense to an action brought for noncompliance with such technology-based permit effluent limitations if the requirements of Part II.B.5.b. are met. No determination made during administrative review of claims that noncompliance was caused by upset, and before an action for noncompliance, is final administrative action subject to judicial review.

b. Conditions necessary for a demonstration of upset. A permittee who wishes to establish the affirmative defense of upset shall demonstrate, through properly signed, contemporaneous operating logs, or other relevant evidence that:

(1) An upset occurred and that the permittee can identify the cause(s) of the upset;

(2) The permitted facility was at the time being properly operated;

(3) The permittee submitted notice of the upset as required by Part II.D.7; and,

(4) The permittee complied with any remedial measures required by Part II.B.2.

c. Burden of proof. In any enforcement proceeding, the permittee seeking to establish the occurrence of an upset has the burden of proof.

6. Removed Substances

Solids, sewage sludges, filter backwash, or other pollutants removed in the course of treatment or wastewater control shall be disposed of in a manner such as to prevent any pollutant from such materials from entering navigable waters. Any substance specifically listed within this permit may be discharged in accordance with specified conditions, terms, or limitations.

Section C. Monitoring and Records

1. Inspection and Entry

The permittee shall allow the Director, or an authorized representative, upon the presentation of credentials and other documents as may be required by the law to:

- a. Enter upon the permittee's premises where a regulated facility or activity is located or conducted, or where records must be kept under the conditions of this permit;
- b. Have access to and copy, at reasonable times, any records that must be kept under the conditions of this permit;
- c. Inspect at reasonable times any facilities, equipment (including monitoring and control equipment), practices or operations regulated or required under this permit; and
- d. Sample or monitor at reasonable times, for the purpose of assuring permit compliance or as otherwise authorized by the Act, any substances or parameters at any location.

2. Representative Sampling

Samples and measurements taken for the purpose of monitoring shall be representative of the monitored activity.

3. Retention of Records

The permittee shall retain records of all monitoring information, including all calibration and maintenance records and all original strip chart recordings for continuous monitoring instrumentation, copies of all reports required by this permit, and records of all data used to complete the application for this permit, for a period of at least 3 years from the date of the sample, measurement, report, or application. This period may be extended by request of the Director at any time.

The operator shall maintain records at the platform where the discharges occur or another platform in the Field for a period of three years, whenever practicable or at a specific shore-base site whenever not practicable. For example, in the case of unmanned platforms or platforms where records storage is not practicable, records may be maintained at a central field office platform or a specific shore-based site. In either case, the records must be available for review by government inspectors coincident with their inspection. The operator is responsible for maintaining records at exploratory facilities while they are discharging under the operators control and at a specific shore-based site for the remainder of the 3-year retention period.

4. Record Contents

Records of monitoring information shall include:

- a. The date, exact place, and time of sampling or measurements;
- b. The individual(s) who performed the sampling or measurements;
- c. The date(s) and time(s) analyses were performed;
- d. The individual(s) who performed the analyses;
- e. The analytical techniques or methods used;

- f. The results of such analyses; and
- g. A copy of the permit and notice of intent to be covered.

5. Monitoring Procedures

- a. Monitoring must be conducted according to test procedures approved under 40 CFR Part 136, unless other test procedures have been specified in this permit or approved by the Regional Administrator.
- b. The permittee shall calibrate and perform maintenance procedures on all monitoring and analytical instruments at intervals frequent enough to insure accuracy of measurements and shall maintain appropriate records of such activities.
- c. An adequate analytical quality control program, including the analyses of sufficient standards, spikes, and duplicate samples to insure the accuracy of all required analytical results shall be maintained by the permittee or designated commercial laboratory.

6. Flow Measurements

Appropriate flow measurement devices and methods consistent with accepted scientific practices shall be selected and used to ensure the accuracy and reliability of measurements of the volume of monitored discharges. The devices shall be installed, calibrated, and maintained to insure that the accuracy of the measurements is consistent with the accepted capability of that type of device. Devices selected shall be capable of measuring flows with a maximum deviation of less than 10% from true discharge rates throughout the range of expected discharge volumes.

Section D. Reporting Requirements

1. Planned Changes

The permittee shall give notice to the Director as soon as possible of any planned physical alterations or additions to the permitted facility. Notice is required only when:

- (1) The alteration or addition to a permitted facility may meet one of the criteria for determining whether a facility is a new source in 40 CFR Part 122.29(b); or,
- (2) The alteration or addition could significantly change the nature or increase the quantity of pollutants discharged. This notification applies to pollutants which are subject neither to effluent limitations in the permit, nor to notification requirements listed at Part II.D.10.a.

2. Anticipated Noncompliance

The permittee shall give advance notice to the Director of any planned changes in the permitted facility or activity which may result in noncompliance with permit requirements.

3. Transfers

This permit is not transferable to any person except after notice to the Director. The Director may require modification or revocation and reissuance of the permit to change the name of the permittee and to incorporate such requirements as may be necessary under the Act.

4. Discharge Monitoring Reports (DMR) and Other Reports

The operator of each lease block shall be responsible for submitting monitoring results for all facilities within each lease block for which the operator has permit coverage. The monitoring results for the facilities (platform, drilling ship, or semisubmersible) within the particular lease block shall be summarized on the annual DMR for that lease block.

Monitoring results obtained during the previous 12 months shall be summarized and reported on a DMR form (EPA No. 3320-1), or the Offshore Operator Committee's facsimile DMR form, for those operators who requested and received written authorization from the EPA to utilize that DMR form. Operators are to utilize the signed and dated DMR Completion Instructions for the OCS General Permit (GMG290000), as found on EPA's offshore web site at <http://epa.gov/earth1r6/6en/w/offshore>

If any category of waste (discharge) is not applicable for all facilities within the lease block, due to the type of operations (e.g., drilling, production) no reporting is required; however, "no discharge" must be recorded for those categories on the DMR. Operators may list a summary of all lease blocks where there is no activity in lieu of DMRs for those lease blocks. The summary must state each lease block name and outfall number and must include the monitoring period. All pages of the DMR must be signed and certified as required by Part II.D.11 of this permit and submitted to EPA when due (annually). If a summary of no activity lease blocks is submitted, the summary must be signed and certified. All pages of the DMR and / or any summary of lease blocks with no discharge must be signed and certified as required in Part II.D.11 of this permit and submitted to EPA when due.

Additionally, the lease area and block number assigned by the Department of the Interior shall be listed on all DMR.

5. Additional Monitoring by the Permittee

If the permittee monitors any pollutant more frequently than required by this permit, using test procedures approved under 40 CFR Part 136 or as specified in this permit, the results of this monitoring shall be included in the calculation and reporting of the data submitted in the Discharge Monitoring Report (DMR). Such increased monitoring frequency shall also be indicated on the DMR.

6. Averaging of Measurements

Calculations for all limitations which require averaging of measurements shall utilize an arithmetic mean unless otherwise specified.

7. Twenty-Four Hour Reporting

- a. The permittee shall report any noncompliance which may endanger health or the environment. Any information shall be provided orally to 214-665-6593 within 24 hours from the time the permittee becomes aware of the circumstances. Alternatively to oral reporting, the permittee may report by EMAIL at the following address: R6GENPERMIT@epa.gov. A written submission shall be provided within 5 days of the time the permittee becomes aware of the circumstances. The report shall contain the following information:
 - (1) A description of the noncompliance and its cause;
 - (2) The period of noncompliance including exact dates and times, and if the noncompliance has not been corrected, the anticipated time it is expected to continue; and,
 - (3) Steps being taken to reduce, eliminate, and prevent recurrence of the noncomplying discharge.
- b. The following shall be included as information which must be reported within 24 hours:
 - (1) Any unanticipated bypass which exceeds any effluent limitation in the permit;
 - (2) Any upset which exceeds any effluent limitation in the permit; and,
 - (3) Violation of a maximum daily discharge limitation for any of the pollutants listed by the Director in Part II of the permit to be reported within 24 hours.
- c. The Director may waive the written report on a case-by-case basis if the oral report has been received within 24 hours.

8. Other Noncompliance

The permittee shall report all instances of noncompliance not reported under Parts II.D.4 and D.7 at the time monitoring reports are submitted. The reports shall contain the information listed at Part II.D.7.

9. Other Information

Where the permittee becomes aware that he failed to submit any relevant facts in a permit application, or submitted incorrect information in a permit application or in any report to the Director, he shall promptly submit such facts or information.

10. Signatory Requirements

All applications, reports, or information submitted to the Director shall be signed and certified.

- a. All permit applications shall be signed as follows:
 - (1) For a corporation - by a responsible corporate officer. For the purpose of this section, a responsible corporate officer means:
 - (a) A president, secretary, treasurer, or vice-president of the corporation in charge of a principal business function, or any other person who performs similar policy or decision making functions for the corporation; or,
 - (b) the manager of one or more manufacturing, production, or operating facilities, provided: the manager is authorized to make management decisions which govern the operation of the regulated facility including having the explicit or implicit duty of making major capital investment recommendations, and initiating and directing other comprehensive measures to assure long term environmental compliance with environmental laws and regulations; the manager can ensure that the necessary systems are established or actions taken to gather complete and accurate information for permit application requirements; and where authority to sign documents has been assigned or delegated to the manager in accordance with corporate procedures.
 - (2) For a partnership or sole proprietorship - by a general partner or the proprietor, respectively.
 - (3) For a municipality, State, Federal, or other public agency - by either a principal executive officer or ranking elected official. For purposes of this election, a principal executive officer of a Federal agency includes:
 - (a) The chief executive officer of the agency, or
 - (b) A senior executive officer having responsibility for the overall operations of a principal geographic unit of the agency.
- b. All reports required by the permit and other information requested by the Director shall be signed by a person described above or by a duly authorized representative of that person. A person is a duly authorized representative only if:

- (1) The authorization is made in writing by a person described above;
 - (2) The authorization specifies either an individual or a position having responsibility for the overall operation of the regulated facility or activity, such as the position of plant manager, operator of a well or a well field, superintendent, or position of equivalent responsibility, or an individual or position having overall responsibility for environmental matters for the company. A duly authorized representative may thus be either a named individual or an individual occupying a named position; and,
 - (3) The written authorization is submitted to the Director.
- c. Certification. Any person signing a document under this section shall make the following certification:

"I certify under penalty of law that this document and all attachments were prepared under my direction or supervision in accordance with a system designed to assure that qualified personnel properly gather and evaluate the information submitted. Based on my inquiry of the person or persons who manage the system, or those persons directly responsible for gathering the information, the information submitted is, to the best of my knowledge and belief, true, accurate, and complete. I am aware that there are significant penalties for submitting false information, including the possibility of fine and imprisonment for knowing violations."

11. Availability of Reports

Except for applications, effluent data, permits, and other data specified in 40 CFR 122.7, any information submitted pursuant to this permit may be claimed as confidential by the submitter. If no claim is made at the time of submission, information may be made available to the public without further notice.

Section E. Penalties for Violations of Permit Conditions

1. Criminal

a. Negligent Violations

The Act provides that any person who negligently violates permit conditions implementing section 301, 302, 306, 307, 308, 318, or 405 of the Act is subject to a fine of not less \$2,500 nor more than \$25,000 per day of violation, or by imprisonment for not more than 1 year, or both.

b. Knowing Violations

The Act provides that any person who knowingly violates permit conditions implementing sections 301, 302, 306, 307, 308, 318 or 405 of the Act is subject to a fine of not less than \$5,000 nor more than \$50,000 per day of violation, or by imprisonment for not more than 3 years, or both.

c. Knowing Endangerment

The Act provides that any person who knowingly violates permit conditions implementing sections 301, 302, 303, 306, 307, 308, 318, or 405 of the Act and who knows at that time that he is placing another person in imminent danger of death or serious bodily injury is subject to a fine of not more than \$250,000, or by imprisonment for not more than 15 years, or both.

d. False Statements

The Act provides that any person who knowingly makes any false material statement, representation, or certification in any application, record report, plan, or other document filed or required to be maintained under the Act or who knowingly falsifies, tampers with, or renders inaccurate, any monitoring device or method required to be maintained under the Act, shall upon conviction, be punished by a fine of not more than \$10,000, or by imprisonment for not more than 2 years, or by both. If a conviction of a person is for a violation committed after a first conviction of such person under this paragraph, punishment shall be by a fine of not more than \$20,000 per day of violation, or by imprisonment of not more than 4 years, or by both. (See section 309.c.4 of the Clean Water Act)

2. Civil Penalties

The Act provides that any person who violates a permit condition implementing sections 301, 302, 306, 307, 308, 318, or 405 of the Act is subject to a civil penalty not to exceed \$32,500 per day for each violation.

3. Administrative Penalties

The Act provides that any person who violates a permit condition implementing sections 301, 302, 306, 307, 308, 318, or 405 of the Act is subject to an administrative penalty, as follows:

- a. Class I Penalty
Not to exceed \$11,000 per violation nor shall the maximum amount exceed \$32,500.
- b. Class II penalty
Not to exceed \$11,000 per day for each day during which the violation continues nor shall the maximum amount exceed \$157,500.

Section F. Additional General Permit Conditions**1. When the Regional Administrator May Require Application for an Individual NPDES Permit.**

The Regional Administrator may require any person authorized by this permit to apply for and obtain an individual NPDES permit when:

- (a) The discharge(s) is a significant contributor of pollution;
- (b) The discharger is not in compliance with the conditions of this permit;
- (c) A change has occurred in the availability of the demonstrated technology or practices for the control or abatement of pollutants applicable to the point sources;
- (d) Effluent limitations guidelines are promulgated for point sources covered by this permit;
- (e) A Water Quality Management Plan containing requirements applicable to such point source is approved;
- (f) The point source(s) covered by this permit no longer:
 - (1) Involve the same or substantially similar types of operations;
 - (2) Discharge the same types of wastes;
 - (3) Require the same effluent limitations or operating conditions;
 - (4) Require the same or similar monitoring; and
 - (5) In the opinion of the Regional Administrator, are more appropriately controlled under an individual permit than under a general permit.
- (g) The bioaccumulation monitoring results show concentrations of the listed pollutants in excess of levels safe for human consumption.

The Regional Administrator may require any operator authorized by this permit to apply for an individual NPDES permit only if the operator has been notified in writing that a permit application is required.

2. When an Individual NPDES Permit may be Requested

- (a) Any operator authorized by this permit may request to be excluded from the coverage of this general permit by applying for an individual permit.

- (b) When an individual NPDES permit is issued to an operator otherwise subject to this general permit, the applicability of this permit to the owner or operator is automatically terminated on the effective date of that individual permit.
- (c) A source excluded from coverage under this general permit solely because it already has an individual permit may request that its individual permit be revoked, and that it be covered by this general permit. Upon revocation of the individual permit, this general permit shall apply to the source.

3. Permit Reopener Clause

If applicable new or revised effluent limitations guidelines or New Source Performance Standards covering the Offshore Subcategory of the Oil and Gas Extraction Point Source Category (40 CFR 435) are promulgated in accordance with Clean Water Act (CWA) sections 301(b), 304(b)(2), and 307(a)(2), and the new or revised effluent limitations guidelines or New Source Performance Standards are more stringent than any effluent limitations in this permit or control a pollutant not limited in this permit, the permit may, at the Director's discretion, be modified to conform to the new or revised effluent limitations guidelines.

Notwithstanding the above, if an offshore oil and gas extraction point source discharge facility is subject to the ten-year protection period for new source performance standards under the CWA section 306(d), this reopener clause may not be used to modify the permit to conform to more stringent new source performance standards or technology-based standards developed under section 301(b)(2) during the ten-year period specified in 40 CFR Part 122.29(d).

The Director may modify this permit upon meeting the conditions set forth in this reopener clause.

Section G. Definitions

All definitions contained in section 502 of the Act shall apply to this permit and are incorporated herein by references. Unless otherwise specified in this permit, additional definitions of words or phrases used in this permit are as follows:

1. "Act" means the Clean Water Act (33 U.S.C. 1251 et. seq.), as amended.
2. "Administrator" means the Administrator of the U.S. Environmental Protection Agency.
3. "Annual Average" means the average of all discharges sampled and/or measured during a calendar year in which daily discharges are sampled and/or measured, divided by the number of discharges sampled and/or measured during such year.
4. "Applicable effluent standards and limitations" means all state and Federal effluent standards and limitations to which a discharge is subject under the Act, including, but

not limited to, effluent limitations, standards or performance, toxic effluent standards and prohibitions, and pretreatment standards.

5. "Applicable water quality standards" means all water quality standards to which a discharge is subject under the Act.
6. "Areas of Biological Concern" means a portion of the OCS identified by EPA, in consultation with the Department of Interior as containing potentially productive or unique biological communities or as being potentially sensitive to discharges associated with oil and gas activities.
7. "Base Fluid" means the continuous phase or suspending medium of a drilling fluid formulation.
8. "Base Fluid Retained" on cuttings as applied to BAT effluent limitations and NSPS refers to the modified American Petroleum Institute Recommended Practice 13B-2 supplemented with the specifications, sampling methods, and averaging method for retention values provided in Appendix 7 of 40 CFR 435, Subpart A.
9. "Biodegradation Rate" as applied to BAT effluent limitations and NSPS for drilling fluids and drill cuttings refers to the modified ISO 11734:1995 method: "Water quality - Evaluation of the 'ultimate' anaerobic biodegradability of organic compounds in digested sludge - Method by measurement of the biogas production (1995 edition)" (Available from the American National Standards Institute, 11 West 42nd Street, 13th Floor, New York, NY 10036) supplemented with modifications in Appendix 4 of 40 CFR 435, Subpart A, and Appendix B of this permit.
10. "Blow-Out Preventer Control Fluid" means fluid used to actuate the hydraulic equipment on the blow out preventer.
11. "Boiler Blowdown" means discharges from boilers necessary to minimize solids build-up in the boilers, including vents from boilers and other heating systems.
12. "Bulk Discharge" any discharge of a discrete volume or mass of effluent from a pit tank or similar container that occurs on a one-time, infrequent or irregular basis.
13. "Bypass" means the intentional diversion of waste streams from any portion of a treatment facility.
14. "C₁₂-C₁₄ Ester and C₈ Ester" means the fatty acid/2-ethylhexyl esters with carbon chain lengths ranging from 8 to 16 and represented by the Chemical Abstracts Service (CAS) No. 135800-37-2. (Properties available from the Chemical Abstracts Service, 2540 Olentangy River Road, P.O. Box 3012, Columbus, OH, 43210)

15. "C₁₆-C₁₈ Internal Olefin" means a 65/35 blend, proportioned by mass, of hexadecene and octadecene, respectively. Hexadecene is an unsaturated hydrocarbon with a carbon chain length of 16, an internal double carbon bond, and is represented by the Chemical Abstracts Service (CAS) No. 26952-14-7. Octadecene is an unsaturated hydrocarbon with a carbon chain length of 18, an internal double carbon bond, and is represented by the Chemical Abstracts Service (CAS) No. 27070-58-2. (Properties available from the Chemical Abstracts Service, 2540 Olentangy River Road, P.O. Box 3012, Columbus, OH, 43210).
16. "C₁₆-C₁₈ Internal Olefin Drilling Fluid" means a C₁₆-C₁₈ internal olefin drilling fluid formulated as specified in Appendix 8 of 40 CFR 435, Subpart A.
17. "Completion Fluids" means salt solutions, weighted brines, polymers and various additives used to prevent damage to the well bore during operations which prepare the drilled well for hydrocarbon production. These fluids move into the formation and return to the surface as a slug with the produced water. Drilling muds remaining in the wellbore during logging, casing, and cementing operations or during temporary abandonment of the well are not considered completion fluids and are regulated by drilling fluids requirements.
18. "Controlled Discharge Rates Areas" means zones adjacent to areas of biological concern.
19. "Daily Discharge" means the discharge of a pollutant measured during a calendar day or any 24-hour period that reasonably represents the calendar day for purposes of sampling. For pollutants with limitations expressed in terms of mass, the daily discharge is calculated as the total mass of the pollutant discharged over the sampling day. For pollutants with limitations expressed in other units of measurement, the daily discharge is calculated as the average measurement of the pollutant over the sampling day. Daily discharge determination of concentration made using a composite sample shall be the concentration of the composite sample. When grab samples are used, the daily discharge determination of concentration shall be arithmetic average (weighted by flow value) of all samples collected during that sampling day.
20. "Daily Average" (also known as monthly average) discharge limitations means the highest allowable average of daily discharge(s) over a calendar month, calculated as the sum of all daily discharge(s) measured during a calendar month divided by the number of daily discharge(s) measured during that month. When the permit establishes daily average concentration effluent limitations or conditions, the daily average concentration means the arithmetic average (weighted by flow) of all daily discharge(s) of concentration determined during the calendar month where C = daily concentration, F = daily flow, and n = number of daily samples; daily average discharge =

$$C_1F_1 + C_2F_2 + \dots + C_nF_n$$

$$F_1 + F_2 + \dots + F_n.$$

21. "Daily Maximum" discharge limitations means the highest allowable "daily discharge" during the calendar month.
22. "Desalinization Unit Discharge" means wastewater associated with the process of creating freshwater from seawater.
23. "Deck Drainage" means any waste resulting from deck washings, spillage, rainwater, and runoff from gutters and drains including drip pans and work areas within facilities subject to this permit.
24. "Development Drilling" means the drilling of wells required to efficiently produce a hydrocarbon formation or formations.
25. "Development Facility" means any fixed or mobile structure that is engaged in the drilling of productive wells.
26. "Diatomaceous Earth Filter Media" means filter media used to filter seawater or other authorized completion fluids and subsequently washed from the filter.
27. "Diesel Oil" refers to the grade of distillate fuel oil, as specified in the American Society for Testing and Materials Standard Specification for Diesel Fuel Oils D975–91, which is typically used as the continuous phase in conventional oil-based drilling fluids. This incorporation by reference was approved by the Director of the Federal Register in accordance with 5 U.S.C. 552(a) and 1 CFR Part 51. Copies may be obtained from the American Society for Testing and Materials, 1916 Race Street, Philadelphia, PA 19103. Copies may be inspected at the Office of the Federal Register, 800 North Capitol Street, NW., Suite 700, Washington, DC. A copy may also be inspected at EPA's Water Docket, 401 M Street SW., Washington, DC 20460.
28. "Director" means the U.S. Environmental Protection Agency Regional Administrator or an authorized representative.
29. "Domestic Waste" means material discharged from galleys, sinks, showers, safety showers, eye wash stations, hand washing stations, fish cleaning stations, and laundries.
30. "Drill Cuttings" means the particles generated by drilling into subsurface geologic formations including cured cement carried out from the wellbore with the drilling fluid. Examples of drill cuttings include small pieces of rock varying in size and texture from fine silt to gravel. Drill cuttings are generally generated from solids control equipment and settle out and accumulate in quiescent areas in the solids control equipment or other equipment processing drilling fluid (i.e., accumulated solids).

(a) "Wet Drill Cuttings" means the unaltered drill cuttings and adhering drilling fluid and formation oil carried out from the wellbore with the drilling fluid.

(b) "Dry Drill Cuttings" means the residue remaining in the retort vessel after completing the retort procedure specified in Appendix 7 of 40 CFR 435, Subpart A.

31. "Drilling Fluid" means the circulating fluid (mud) used in the rotary drilling of wells to clean and condition the hole and to counterbalance formation pressure. Classes of drilling fluids are:

(a) "Water-Based Drilling Fluid" means the continuous phase and suspending medium for solids is a water-miscible fluid, regardless of the presence of oil.

(b) "Non aqueous Drilling Fluid" means the continuous phase and suspending medium for solids is a water-immiscible fluid, such as oleaginous materials (e.g., mineral oil, enhanced mineral oil, paraffinic oil, C₁₆-C₁₈ internal olefins, and C₈-C₁₆ fatty acid/2-ethylhexyl esters).

(i) "Oil-Based" means the continuous phase of the drilling fluid consists of diesel oil, mineral oil, or some other oil, but contains no synthetic material or enhanced mineral oil.

(ii) "Enhanced Mineral Oil-Based" means the continuous phase of the drilling fluid is enhanced mineral oil.

(iii) "Synthetic-Based" means the continuous phase of the drilling fluid is a synthetic material or a combination of synthetic materials.

32. "Dual Gradient Drilling" means well drilling where a pump is used sub sea to lift drilling fluids and cuttings to the surface. This allows for a dual pressure gradient - one from the hydrostatic weight of water in the riser and one from the mud weight in the well. Dual gradient drilling can include a discharge of the larger size cuttings sub sea.

33. "End of well Sample" means the sample taken after the final log run is completed and prior to bulk discharge.

34. "Enhanced Mineral" oil as applied to enhanced mineral oil-based drilling fluid means a petroleum distillate which has been highly purified and is distinguished from diesel oil and conventional mineral oil in having a lower polycyclic aromatic hydrocarbon (PAH) content. Typically, conventional mineral oils have a PAH content on the order of 0.35 weight percent expressed as phenanthrene, whereas enhanced mineral oils typically have a PAH content of 0.001 or lower weight percent PAH expressed as phenanthrene.

35. "Environmental Protection Agency" (EPA) means the U.S. Environmental Protection Agency.
36. "Excess Cement Slurry" means the excess mixed cement, including additives and wastes from equipment washdown, after a cementing operation.
37. "Exploratory Facility" means any fixed or mobile structure that is engaged in the drilling of wells to determine the nature of potential hydrocarbon reservoirs.
38. "Fecal Coliform Bacteria Sample" consists of one effluent grab portion collected during a 24-hour period at peak loads.
39. "Formation Oil" means the oil from a hydrocarbon bearing formation and other oil which might enter the drilling fluid, which is detected in the drilling fluid, as determined by the GC/MS compliance assurance method specified in Appendix 5 of Subpart A of this part when the drilling fluid is analyzed before being shipped offshore, and as determined by the RPE method specified in Appendix 6 of Subpart A of this part when the drilling fluid is analyzed at the offshore point of discharge. Detection of formation oil by the RPE method may be confirmed by the GC/MS compliance assurance method, and the results of the GC/MS compliance assurance method shall supercede those of the RPE method.
40. "Four (4)-day LC₅₀" as applied to the sediment toxicity BAT effluent limitations and NSPS means the concentration (milliliters/kilogram dry sediment) of the drilling fluid in sediment that is lethal to 50 percent of the *Leptocheirus plumulosus* test organisms exposed to that concentration of the drilling fluids after four days of constant exposure.
41. "Grab sample" means an individual sample collected in less than 15 minutes.
42. "Garbage" means all kinds of food waste, wastes generated in living areas on the facility, and operational waste, excluding fresh fish and parts thereof, generated during the normal operation of the facility and liable to be disposed of continuously or periodically, except dishwater, graywater, and those substances that are defined or listed in other Annexes to MARPOL 73/78
43. "Graywater" means drainage from dishwater, shower, laundry, bath, and washbasin drains and does not include drainage from toilets, urinals, hospitals, and cargo spaces.
44. "Inverse Emulsion Drilling Fluids" means an oil-based drilling fluid which also contains a large amount of water.
45. "Live bottom areas" means those areas which contain biological assemblages consisting of such sessile invertebrates as seas fans, sea whips, hydroids, anemones, ascidians sponges, bryozoans, seagrasses, or corals living upon and attached to naturally occurring hard or rocky formations with fishes and other fauna.

46. "Maintenance waste" means materials collected while maintaining and operating the facility, including, but not limited to, soot, machinery deposits, scraped paint, deck sweepings, wiping wastes, and rags.
47. "Maximum Hourly Rate" means the greatest number of barrels of drilling fluids discharged within one hour, expressed as barrels per hour.
48. "Maximum Weighted Mass Ratio Averaged Over All NAF Well Sections" for BAT effluent limitations and NSPS for base fluid retained on cuttings means the weighted average base fluid retention for all NAF well sections as determined by the modified API Recommended Practice 13B-2, using the methods and averaging calculations presented in Appendix 7 of 40 CFR 435, Subpart A.
49. "Method 1654A" refers to the method "PAH Content of Oil by High Performance Liquid Chromatography with a UV Detector," which was published in Methods for the Determination of Diesel, Mineral and Crude Oils in Offshore Oil and Gas Industry Discharges, EPA-821-R-92-008 (incorporated by reference and available from National Technical Information Service at 703/605-6000).
50. "Minimum" as applied to BAT effluent limitations and NSPS for drilling fluids and drill cuttings means the minimum 96-hour LC₅₀ value allowed as measured in any single sample of the discharged waste stream. *Minimum* as applied to BPT and BCT effluent limitations and NSPS for sanitary wastes means the minimum concentration value allowed as measured in any single sample of the discharged waste stream.
51. "Muds, Cuttings, and Cement at the Seafloor" means discharges that occur at the seafloor prior to installation of the marine riser and during marine riser disconnect, well abandonment and plugging operations. Also included are discharges of drilling fluid and cuttings associated with the operation of a sub sea drilling fluid pump.
52. "National Pollutant Discharge Elimination System" (NPDES) means the national program for issuing, modifying, revoking, and reissuing, terminating, monitoring, and enforcing permits, and imposing and enforcing pretreatment requirements, under section 307, 318, 402, and 405 of the Act.
53. "New Source" means any facility or activity that meets the definition of "new source" under 40 CFR 122.2 and meets the criteria for determination of new sources under 40 CFR 122.29(b) applied consistently with all of the following definitions:
 - (a) The term "water area" as used in the term "site" in 40 CFR 122.29 and 122.2 shall mean the water area and ocean floor beneath any exploratory, development, or production facility where such facility is conducting its exploratory, development, or production activities.

(b) The term "significant site preparation work" as used in 40 CFR 122.29 shall mean the process of surveying, clearing, or preparing an area of the ocean floor for the purpose of constructing or placing a development or production facility on or over the site.

"New Source" does not include facilities covered by an existing NPDES permit immediately prior to the effective date of these guidelines pending EPA issuance of a new source NPDES permit.

54. "Ninety-Six (96)-hour LC₅₀" means the concentration (parts per million) or percent of the suspended particulate phase (SPP) from a sample that is lethal to 50 percent of the test organisms exposed to that concentration of the SPP after 96 hours of constant exposure.
55. "No Activity Zones" means those areas identified by the Minerals Management Service (MMS) where no structures, drilling rigs, or pipelines will be allowed. Those zones are identified in lease stipulations that are applied to MMS oil and gas lease sales. Additional no activity areas may be identified by MMS during the life of this permit.
56. "No Discharge of Free Oil" means that waste streams may not be discharged that contain free oil as evidenced by the monitoring method specified for that particular stream, e.g., deck drainage or miscellaneous discharges cannot be discharged when they would cause a film or sheen upon or discoloration of the surface of the receiving water; drilling fluids or cuttings may not be discharged when they fail the static sheen test defined in Appendix 1 of 40 CFR 435, Subpart A.
57. "Operational waste" means all cargo associated waste, maintenance waste, cargo residues, and ashes and clinkers from incinerators and coal burning boilers.
58. "Packer Fluid" means low solids fluids between the packer, production string and well casing. They are considered to be workover fluids.
59. "PAH (as phenanthrene)" means polynuclear aromatic hydrocarbons reported as phenanthrene.
60. Parameters that are regulated by this permit and listed with approved methods of analysis in Table 1B at 40 CFR 136.3 are defined as follows:
 - (a) Cadmium means total cadmium.
 - (b) Chlorine means total residual chlorine.
 - (c) Mercury means total mercury.
 - (d) Oil and Grease means total recoverable oil and grease.

61. "Priority Pollutants" means those chemicals or elements identified by EPA, pursuant to section 307 of the Clean Water Act and 40 CFR 401.15.
62. "Produced Sand" means slurried particles used in hydraulic fracturing, the accumulated formation sands, and scale particles generated during production. Produced sand also includes desander discharge from produced water waste stream and blowdown of water phase from the produced water treating system.
63. "Produced Water" means the water (brine) brought up from the hydrocarbon-bearing strata during the extraction of oil and gas, and can include formation water, injection water, and any chemicals added downhole or during the oil/water separation process.
64. "Production Facility" means any fixed or mobile structure that is either engaged in well completion or used for active recovery of hydrocarbons from producing formations.
65. "Sanitary Waste" means human body waste discharged from toilets and urinals.
66. "Sediment Toxicity" as applied to BAT effluent limitations and NSPS for drilling fluids and drill cuttings refers to the ASTM E1367-92 method: Standard Guide for Conducting 10-day Static Sediment Toxicity Tests with Marine and Estuarine Amphipods (Available from the American Society for Testing and Materials, 100 Barr Harbor Drive, West Conshohocken, PA, 19428) with *Leptocheirus plumulosus* as the test organism and sediment preparation procedures specified in Appendix 3 of 40 CFR 435, Subpart A, and the method found in Appendix B of this permit.
67. "Severe property damage" means substantial physical damage to property, damage to the treatment facilities which cause them to become inoperable, or substantial and permanent loss of natural resources which can reasonably be expected to occur in the absence of a bypass. Severe property damage does not mean economic loss caused by delays in production.
68. "Sheen" means a silvery or metallic sheen, gloss, or increased reflectivity, visual color or iridescence on the water surface.
69. "Solids Control Equipment" means shale shakers, centrifuges, mud cleaners, and other equipment used to separate drill cuttings and/or stock barite solids from drilling fluid recovered from the wellbore.
70. "Source Water and Sand" means water from non-hydrocarbon bearing formations for the purpose of pressure maintenance or secondary recovery including the entrained solids.
71. "Spotting" means the process of adding a lubricant (spot) downhole to free stuck pipe.

72. "Static Sheen Test" means the standard test procedure that has been developed for this industrial subcategory for the purpose of demonstrating compliance with the requirement of no discharge of free oil. The methodology for performing the static sheen test is presented in Appendix 1 of 40 CFR 435, Subpart A.
73. "Stock Barite" means the barite that was used to formulate a drilling fluid.
74. "Stock Base Fluid" means the base fluid that was used to formulate a drilling fluid.
75. "Suspended Particulate Phase Toxicity" as applied to BAT effluent limitations and NSPS for drilling fluids and drill cuttings refers to the bioassay test procedure presented in Appendix 2 of 40 CFR 435, Subpart A.
76. "Synthetic Drilling Fluid" means a drilling fluid which has synthetic material as its continuous phase with water as the dispersed phase.
77. "Synthetic Material" as applied to synthetic-based drilling fluid means material produced by the reaction of specific purified chemical feedstock, as opposed to the traditional base fluids such as diesel and mineral oil which are derived from crude oil solely through physical separation processes. Physical separation processes include fractionation and distillation and/or minor chemical reactions such as cracking and hydro processing. Since they are synthesized by the reaction of purified compounds, synthetic materials suitable for use in drilling fluids are typically free of PAH's but are sometimes found to contain levels of PAH up to 0.001 weight percent PAH expressed as phenanthrene. Internal olefins and vegetable esters are two examples of synthetic materials suitable for use by the oil and gas extraction industry in formulating drilling fluids. Internal olefins are synthesized from the isomerization of purified straight-chain (linear) hydrocarbons such as C₁₆-C₁₈ linear alpha olefins. C₁₆-C₁₈ linear alpha olefins are unsaturated hydrocarbons with the carbon to carbon double bond in the terminal position. Internal olefins are typically formed from heating linear alpha olefins with a catalyst. The feed material for synthetic linear alpha olefins is typically purified ethylene. Vegetable esters are synthesized from the acid-catalyzed esterification of vegetable fatty acids with various alcohols. EPA listed these two branches of synthetic fluid base materials to provide examples, and EPA does not mean to exclude other synthetic materials that are either in current use or may be used in the future. A synthetic-based drilling fluid may include a combination of synthetic materials.
78. "Ten (10)-day LC₅₀" as applied to the sediment toxicity BAT effluent limitations and NSPS means the concentration (milligrams of drilling fluid/kilogram dry sediment) of the base fluid in sediment that is lethal to 50 percent of the *Leptocheirus plumulosus* test organisms exposed to that concentration of the base fluids after ten days of constant exposure.
79. "Territorial Seas" means the belt of the seas measured from the line of ordinary low water along that portion of the coast which is in direct contact with the open sea and the

line marking the seaward limit of inland waters, and extending seaward a distance of three miles.

80. "Trace Amounts" means that if materials added downhole as well treatment, completion, or workover fluids do not contain priority pollutants then the discharge is assumed not to contain priority pollutants, except possibly in trace amounts.
81. "Treatment Chemicals" means biocides, corrosion inhibitors, or other chemicals which are used to treat seawater or freshwater to prevent corrosion or fouling of piping or equipment. Non-toxic scale inhibitors and dyes are not considered treatment chemicals. However, chlorine generated using an electric current rather than added is considered a treatment chemical.
82. "Uncontaminated Ballast/Bilge Water" means seawater added or removed to maintain proper draft.
83. "Uncontaminated Freshwater" means freshwater which is discharged without the addition of treatment chemicals; included are (1) discharges of excess freshwater that permit the continuous operation of fire control and utility lift pumps, (2) excess freshwater from pressure maintenance and secondary recovery projects, (3) water released during training and testing of personnel in fire protection, and (4) water used to pressure test or flush new piping or pipelines.
84. "Uncontaminated Seawater" means seawater which is returned to the sea without the addition of treatment chemicals. Included are (1) discharges of excess seawater which permit the continuous operation of fire control and utility lift pumps (2) excess seawater from pressure maintenance and secondary recovery projects (3) water released during the training and testing of personnel in fire protection (4) seawater used to pressure test or flush new or existing piping and pipelines, and (5) once through noncontact cooling water which has not been treated with biocides.
85. "Upset" means an exceptional incident in which there is unintentional and temporary noncompliance with technology-based permit effluent limitations because of factors beyond the reasonable control of the permittee. An upset does not include noncompliance to the extent caused by operational error, improperly designed treatment facilities, inadequate treatment facilities, lack of preventive maintenance, or careless or improper operation.
86. "Well Treatment Fluids" mean any fluid used to restore or improve productivity by chemically or physically altering hydrocarbon-bearing strata after a well has been drilled. These fluids move into the formation and return to the surface as a slug with the produced water. Stimulation fluids include substances such as acids, solvents, and propping agents.

87. "Workover Fluids" mean salt solutions, weighted brines, polymers, and other specialty additives used in a producing well to allow safe repair and maintenance or abandonment procedures. High solids drilling fluids used during workover operations are not considered workover fluids by definition and therefore must meet drilling fluid effluent limitations before discharge may occur. Packer fluids, low solids fluids between the packer, production string and well casing, are considered to be workover fluids and must meet only the effluent requirements imposed on workover fluids.
88. The term "MGD" shall mean million gallons per day.
89. The term "mg/l" shall mean milligrams per liter or parts per million (ppm).
90. The term " $\mu\text{g/l}$ " shall mean micrograms per liter or parts per billion (ppb).

Appendix A

METHOD FOR CONDUCTING A SEDIMENT TOXICITY TEST WITH *Leptocheirus plumulosus* AND Non aqueous FLUIDS OR SYNTHETIC BASED DRILLING MUDS

Introduction

This test method describes procedures for obtaining data regarding the effects of non aqueous fluids (NAF) or synthetic based drilling muds (SBMs) on the marine amphipod, *Leptocheirus plumulosus*. The tests are conducted in a similar manner; differences are noted in the text and tables below. USEPA is regulating the sediment toxicity of NAFs and SBMs discharged by oil and gas extraction facilities in coastal and offshore waters as an indication of the toxicity of the drilling muds (USEPA 2000). This test method conforms to the Effluent Limitations Guidelines specified in 40 CFR part 435 (see 66 FR 6849, January 22, 2001). As specified in the Effluent Limitations Guidelines, this test method is consistent with ASTM Standard Guide E 1367-92 (ASTM 1997). Since ASTM E 1367-92 was outdated at the time 40 CFR part 435 (see 66 FR 6849, January 22, 2001) was published in the Federal Register, this test method is also consistent with ASTM E 1367-99 (ASTM 2000), which is the latest version published by ASTM.

Test Species

L. plumulosus is an infaunal amphipod that is indigenous to subtidal regions along the east coast of the U.S. This amphipod constructs U-shaped burrows in the top 5 cm of fine sand to silty clay sediments (ASTM E1367-99). As a result of its broad salinity and particle size tolerances, it is a desirable test species for a variety of toxicity testing programs.

Collection and Handling

In the field, amphipods can be collected using sediment grab samplers such as Peterson and Ponar dredges. This species has been collected in various tributaries of the Chesapeake Bay for various toxicity testing programs (ASTM E 1367-99). The contents of each grab should be sieved through a 500 μ m mesh screen. The sediment and organisms retained on the screen are gently rinsed into plastic buckets containing sediment and water from the collection site. These buckets are quickly transported back to the laboratory and aerated. See ASTM E 1367-99 for more details on collection and handling.

Holding and Acclimation

Amphipods can be placed in aquaria containing a 1-2 cm deep layer of collection site sediment that has been sieved through a 500 μ m mesh screen. Amphipod density should be about 200-300 per 40 L aquarium with vigorous aeration. Two to three days are sufficient for acclimation to test conditions, and during this period a gradual change over from site water to test water is recommended (ASTM E 1367-99).

Environmental Tolerances

L. plumulosus is tolerant of a broad salinity range, from near 0 to 33 g/kg (‰) (ASTM E 1367-99). This species has demonstrated up to 100% survival in >90% silt-clay sediment and an

average of 85% survival in >95% sand/gravel sediment (ASTM E 1367-99). The ASTM data are consistent with data published from other studies indicating that *L. plumulosus* is tolerant of sandy and silty sediments. For example, Schlekot et al. (1992) noted a mean survival of 97.5% when *L. plumulosus* was exposed for 10 days to field collected sediments ranging from 98.1% sand to 96.5% fines. Further, this species was collected in the field in sediments consisting of 99.9% sand and 92.1% fines, indicating that *L. plumulosus* is a generalist and can thrive in a variety of sediment types (Schlekot et al. 1992).

However, the fine fraction of sediments in the Schlekot et al. study did not exceed 55% clay, indicating that the fine fraction was a mixture of silt and clay sized particles. Data from other studies indicated that this species is intolerant of sediments high in clay content. McGee et al. (1999) noted acceptable survival when this species was exposed to Baltimore Harbor sediments containing up to 72% clay. However, Emery et al. (1997) noted significantly reduced amphipod survival when *L. plumulosus* was exposed for 10 days to Magothy River, Maryland sediment (amended with beach sand and kaolinite clay) containing 84%, 90%, and 100% clay.

These data indicated that the tolerance range of this amphipod to clay content is between about 72 to 84%. As such, caution should be used when conducting *L. plumulosus* toxicity tests with sediments with clay content greater than about 70%. This should not have a significant impact on using this species in the NAF and SBM toxicity testing program, since field sediments seldom exceed 70% clay content (Suedel and Rodgers 1991).

Control Sediments

Control sediment must meet certain minimum requirements to be used in the SBM testing program. The primary requirement is that the sediment should be able to support *L. plumulosus* in cultures for extended periods of time. This will ensure that the sediment is chemically nontoxic and that the physical and chemical characteristics of the sediment (e.g., total organic carbon, particle size distribution, and moisture content) are within the tolerance range of the test species. It is expected that separate aliquots of the culture sediment will also be used as a control sediment to be amended by NAFs or SBMs in the NAF/SBM testing program. Any modifications made to the control sediments should be noted in the report.

Characterization

Sediments used in testing should be characterized for total organic carbon (TOC), particle size distribution (sand, silt, and clay), and percent water content. These parameters have been shown to influence the results of NAF/SBM toxicity to *L. plumulosus* in initial experiments. Variations in these sediment characteristics should be quantified so that potential effects of these parameters on test results can be closely monitored.

Collection

Control sediments should be collected from the amphipod collection site or from another area that can provide a consistent source of sediment with characteristics within the tolerance range of *L. plumulosus*. Sediments showing evidence of chemical contamination should not be used in the NAF/SBM testing program. Any site water overlying the sediment should be retained so that fine particles suspended in the water can be re-combined with the sediment before use.

Sediment salinity and temperature should be recorded at the time of collection. Sediment collected for use should be homogenized and a composite sample prepared for analysis for the parameters outlined above.

Sieving

Sediments collected in the field for culturing and testing purposes should be first press-sieved through a 2,000 μ m or similar mesh sieve to remove large debris and then through a 500 μ m mesh sieve to remove any indigenous organisms. Sediments have also been press-sieved through a 250 to 350 μ m mesh sieve prior to testing to aid in the enumeration of amphipods on a 500 μ m mesh sieve at test termination.

Storage

The control sediment should be stored in plastic or glass containers at 4 \pm 3 C until test initiation. The sediment should be stored in the dark and should not be allowed to freeze or dry out during storage (E 1367-92).

Test Water

Water used in the NAF/SBM program should be available in sufficient quantities and be acceptable to *L. plumulosus*. The minimum requirement for acceptable water for use in the NAF program is that healthy test organisms survive in the water, and in the water plus control sediment, for the duration of holding and testing without showing signs of disease or stress (ASTM E 1367-99). Another test for acceptability of the test water would be its successful use in the culturing of *L. plumulosus* (with the control sediment).

Natural seawater or synthetic salt water can be used in the NAF program. Natural salt water should be obtained from an uncontaminated area known to support a healthy, reproducing population of *L. plumulosus* or similar sensitive species. Reconstituted salt water can be prepared by adding commercially available sea salt in specified quantities. Natural seawater should be filtered by passing through a 5 micron filter before use. The reader is referred to ASTM E 1367-92 or E 1367-99 for more information concerning test water.

Mixing NAFs or SBMs with Control Sediment

Appendix 3 to Subpart A of Part 435 – Procedure for Mixing Base Fluids with Sediments (40 CFR parts 9 and 435 pages 6901-6902) describes a method for amending control sediments with synthetic-based drilling fluids. This same method can be used to amend control sediments with NAFs and SBMs. The control sediment should be sieved and homogenized before wet to dry weight ratio and density determinations are made and before NAFs are added to the control sediment. The following steps were given in 40 CFR Appendix 3 for mixing NAFs and SBMs with control sediments (parentheses were added here to provide additional information):

- ! Determine the wet to dry weight ratio for the control sediment (three replicates of 30 g each as been used successfully);
- ! Determine the density (g/ml) of the control sediment (three replicates of >25 ml is suitable for this purpose);
- ! Determine the amount of NAF or SBM needed to obtain a desired test concentration;
- ! Determine the amount of wet sediment required;
- ! Determine the amount of dry sediment in kilograms for each test concentration;
- ! Determine the amount of NAF or SBM required to amend the control sediment at each test concentration;
- ! Mix NAF or SBM with control sediment;
- ! Test for homogeneity of NAF or SBM in sediment, and;
- ! Mix sufficient quantities of NAF or SBM with control sediment for each treatment of amended or spiked sediment.

The six steps given above for base fluids can also be applied to SBMs, except that the third bullet in Step 3 requires a measurement of the density of the SBM. The density of the SBM can then be used to estimate the quantity required for the desired test concentration. Refer to the formulas below for NAF and SBM calculations:

$$NAF \text{ Required (g)} = \frac{[Conc. Desired (mg / kg)]}{1000 \text{ g / kg}} * \frac{[Dry weight Sediment (g)]}{1000 \text{ mg / g}}$$

$$SBM \text{ Required (g)} = [Conc. Desired (ml/kg)] \times [Dry Weight Sediment (kg)] \times [SBM Density (g/ml)]$$

See 40 CFR parts 9 and 435 pages 6901-6902 for more information regarding this procedure.

Mixing Procedure

Mixing the NAF or SBM with the control sediment can be accomplished by following these steps:

- Place appropriate amounts of weighed NAF or SBM into a stainless steel mixing bowl;
- Tare the mixing bowl weight;
- Add appropriate amount of control sediment;
- Mix for 9 to 15 minutes with a hand-held mixer equipped with stainless steel blades (e.g., KitchenAid Model KHM6), and;
- As appropriate, test mixing homogeneity as described below.

The control sediment alone should also be subjected to the mixing procedure to ensure mixing has no effect on sediment toxicity.

Homogeneity of Mixing

As noted above, tests for homogeneity of mixing should be performed, preferably in the procedure development phase (40 CFR part 9 page 6901-6902) by each laboratory performing NAF/SBM toxicity testing. This is to ensure that the NAF or SBM, which can be difficult to

homogenize with control sediments, can be evenly mixed with the control sediment by each testing laboratory. Appendix 3 to Subpart A of Part 435 specifies that the coefficient of variation (CV) for a minimum of three replicate samples of the NAF/control sediment mixture must be less than 20%. Determinations of CV should be based on total petroleum hydrocarbon (TPH) content of the NAF or SBM as measured by EPA Methods 3550A and 8015M. If the initial CV is 20%, then the NAF/SBM-sediment mixture must be re-mixed and reanalyzed until the 20% CV limit is achieved.

Homogeneity measurements should be made on the lowest and highest NAF concentrations for a given test. Laboratories should validate mixing efficiency via TPH measurements (as outlined above) of the low and high NAF concentrations. The homogeneity measurements should be made at least once per year.

Recommended Test Conditions

The recommended test conditions for conducting the 10-day or 96-hr sediment toxicity test with *L. plumulosus* are summarized in Table 1 and are consistent with methods presented in ASTM E 1367-92 and subsequent updates (E 1367-99). Tests should be conducted at 20 °C at 20 ‰ salinity with a 14h light; 10 h dark photoperiod at approximately 500-1,000 lux (or about 46 to 93 footcandles). Test chambers are 1-L glass containers with about a 10 cm inside diameter opening (or similar glass containers) that can contain about 150 ml sediment and 600 ml overlying water to achieve a 4:1 (v/v) water to sediment ratio. There are five (5) test concentrations plus a control for each NAF and SBM test. Five (5) replicates are included for the control sediment (E 1367-99) and for each test concentration.

The control sediment/test material mixture and test water should be added to test chambers the day before amphipods are added. This will allow for suspended particles to settle and allow time for equilibration of temperature and the sediment-water interface. After the overnight equilibration period, amphipods are randomly distributed to each test chamber. Twenty amphipods are added to each replicate and there are five replicates per test treatment. Amphipods caught on the water surface can be pushed under with a glass rod. Individuals that have not burrowed within 5 to 10 minutes can be replaced, unless they are exhibiting an avoidance response. Amphipods are not removed at any time during the course of the toxicity test even if they appear dead. Test water is not renewed (i.e., static) and the amphipods are not fed during the exposure period. The toxicity test is terminated after 96 hours or 10 days for SBMs and NAFs respectively.

Temperature, salinity, pH, and dissolved oxygen (DO) should be monitored daily. Ammonia should also be monitored in overlying water to ensure that the concentrations of this constituent do not exceed the tolerance range of the test species. For *L. plumulosus*, this is about 60 mg/L (as total ammonia) at pH 7.7 in 10-day tests (USEPA 1994). Ammonia has not been a problem in initial *L. plumulosus* 96-hr and 10-day tests with various NAFs.

Biological Data

Mortality is the endpoint for *L. plumulosus* at the end of the exposure period. At test termination, the contents of each test chamber (amphipods plus test sediment) are sieved through

a 500 μ m mesh screen to remove amphipods. Material retained on the screen should be rinsed into a sorting tray with clean salt water. The total numbers of live and dead amphipods should be recorded. Missing animals are presumed to have died and decomposed during the test and disintegrated. Amphipods should be counted alive if there are any signs of movement, such as a neuromuscular pleopod twitch (ASTM E 1367-99). Gentle prodding may be used to elicit movement.

Test Acceptability Requirements

Table 2 provides the acceptability requirements for the 10-day NAF and 96-hr SBM test per ASTM E 1367-92. The primary acceptability requirement for NAF testing is as follows:

A toxicity test is unacceptable if more than a total of 10% of the control organisms die, or if the coefficient of variation (CV) of control survival is equal to or greater than 40%.

If this acceptability requirement is not met, then the data should be discarded and the experiment repeated. If this requirement is met, then the other acceptability requirements in Table 2 should be reviewed and a determination made as to the acceptability of the data.

Reference Tests

A single toxicity test will be used to determine satisfactory laboratory performance and to determine whether an NAF or SBM can be discharged as it adheres to drill cuttings. The reference toxicant for the NAF test will be either a C₁₆-C₁₈ -internal olefin reference standard or a C₁₂-C₁₄ or C₈ ester. The reference toxicant for the SBM testing program will be a C₁₆-C₁₈ internal olefin SBM which has also been specified for determining pass/fail for SBMs. The C₁₆-C₁₈ Internal Olefin (IO) SBM is a 65/35 blend, proportioned by mass, of hexadecene and octadecene, respectively (40 CFR part 9 6849). These reference toxicity tests will be conducted in conjunction with all NAF or SBM tests to discern possible changes in the condition of the *L. plumulosus* population used in testing. The reference toxicant test must be conducted concurrently with each sample or batch of samples and at a minimum should be conducted at least monthly. Control charts of this reference standard should be maintained to perform statistical analyses, help understand the inherent variability in the reference test, and for long-term quality control. Test conditions for the reference test should follow the experimental conditions presented in Table 1.

The reference toxicant test should be performed concurrently-and under the same conditions as the NAF or SBM test. The reference toxicant test should be conducted so that control limits (typically set at 2 standard deviations) can be established (USEPA 1994). If the reference test LC₅₀ falls outside of this range of control limits generated on the most recent test data points, then the sensitivity of *L. plumulosus* and the credibility of the test results are considered suspect. In this case, the test procedure should be examined and the test repeated with a different batch of amphipods. A sediment test should not automatically be judged unacceptable if the reference test LC₅₀ falls outside the expected range or if the control in the reference toxicity test exceeds

10%. The width of the control limits and all performance criteria listed in Table 2 should be considered when determining the acceptability of a given NAF or SBM test.

Interpretation of Result

Procedures presented in this test method are used to calculate point estimates, or LC₅₀ values. The LC₅₀ value and 95% confidence limits of the NAF tests should be calculated on the basis of milligrams of NAF per kg dry control sediment (mg/kg) and amphipod mortality. The LC₅₀ value and 95% confidence limits of the NAF tests should be calculated on the basis of milliliters of NAF per kg dry control sediment (ml/kg) and amphipod mortality. A variety of methods can be used to calculate an LC₅₀ value and its 95% confidence limits, including probit, moving average, trimmed Spearman-Kärber and Litchfield-Wilcoxon methods (ASTM E 1367-99). The method used should take into account the number of partial kills, the number of test chambers per treatment (5), and the number of amphipods per test chamber (20).

The only NAF that will be allowed for use in drilling fluids that are discharges in association with cuttings are those that are as toxic or less toxic, but not more toxic, than the reference NAF (C₁₆-C₁₈ internal olefin or C₁₂-C₁₄ or C₈ ester). This limitation is expressed as follows:

$$\frac{10 - \text{day } LC_{50} \text{ Reference Material}}{10 - \text{day } LC_{50} \text{ NAF}} \leq 1.0$$

The only SBMs that will be allowed for discharge are those that are as toxic or less toxic, but not more toxic, than the C₁₆-C₁₈ internal olefin reference SBM. This limitation is expressed as follows:

$$\frac{96 - \text{hr } LC_{50} \text{ RDF}}{96 - \text{hr } LC_{50} \text{ SBM}} \leq 1.0$$

Where: RDF = Reference Drilling Fluid

The EPA promulgated a sediment toxicity ratio of less than 1.0, indicating that the NAF or SBM can be equally toxic or less toxic, but not more toxic than the reference toxicant test LC₅₀ values for *L. plumulosus*. Hence, the NAF or SBM data should be interpreted by comparing to the reference toxicant test LC₅₀ value and whether it exceeds this value.

Culture Methods

Populations of *L. plumulosus* can be maintained through several generations in the laboratory. The culture conditions specified in ASTM E1367-92 and E1367-99 are provided in Table 3. Besides the conditions specified, there are other conditions that are important in maintaining

healthy *L. plumulosus* cultures, including identifying a source of clean sediment, sieving sediments before use, and the quality of the raw materials used to prepare their food. Preferably,

the sediment and water used to culture the amphipods should be collected from the same area as those used in NAF tests. Fine-grained sediments have been shown to be suitable for this purpose (E1367-92). Sediments collected in the field for culturing purposes should be first sieved through a 2,000 μ m mesh sieve to remove large debris and then through a 500 μ m mesh sieve to remove any indigenous organisms. *L. plumulosus* cultures should be maintained at 20 ± 1 C and 20 ± 1 ‰ salinity. If used, natural seawater should be filtered through a 5 micron filter before adding to cultures. New culture chambers should be aerated and allowed to equilibrate overnight before adding amphipods. Water used to start a new culture chamber should be renewed 24 h after initiation and before amphipods are added to culture chambers; otherwise, culture water should be renewed in conjunction with feeding.

Cultures should be observed daily to ensure sufficient aeration. An abundance of amphipods on the sediment surface during daylight hours may indicate insufficient dissolved oxygen or overcrowding, as amphipods typically remain in their burrows unless they are searching for food or a mate. Culture chambers should be terminated and restarted with fresh sediment about once every 8 weeks to avoid overcrowding. Overcrowding may lead to stress due to food or space limitations, and may also result in reduced female fecundity, thus reducing the relative health of the population of amphipods in a given culture chamber.

Cultures should be routinely inspected for the presence of indigenous worms and copepods, a microbial build-up, or black and sulfurous conditions beneath the sediment surface. Microbial growth appears as a white or gray growth associated with uneaten food, and is indicative of overfeeding. Presence of indigenous species, excess microbial growth, or black and sulfurous conditions may necessitate discarding the affected culture chamber.

Feeding

A mixture of micro-algae, yeast, fish food flakes, alfalfa powder, ground cereal leaves, and shrimp maturation feed has been used to feed cultures (E 1367-92 and E 1367-99). Micro-algae used in culturing include *Pseudoisochrysis paradoxa*, *Phaeodactylum tricorutum*, and *Tetraselmis suecica* mixed in equal parts on a volume basis. These algae provide a source of fatty acids that may otherwise be absent in the diet. In practice, however, it should be noted that *L. plumulosus* has been cultured successfully without the algal mixture and the yeast. The dry food portion of the diet that has been used to successfully culture *L. plumulosus* is shown below.

Dietary Component	Proportion
Fish food flakes (TetraMin®)	48.0%
Alfalfa powder	24%
Ground cereal leaves (dried wheat leaves)	24%
Shrimp maturation feed (Neo-Novum®)	4.0%

This dry food mixture should be homogenized into a fine powder and fed to each culture chamber at a rate of 0.1 to 0.5 g two to three times per week, depending on culture densities. Overfeeding may result in microbial build-up on the sediment surface. The quality of the alfalfa powder and dried wheat leaves may not be consistent among suppliers, thus potentially adversely affecting culture performance. Feeding should occur immediately after culture water changes.

Obtaining Amphipods for Starting a Test

Immature and adult amphipods of mixed sexes and approximately 3 to 5 mm in length (as measured from the base of the first antenna to the end of the third pleon segment along the dorsal surface) are used in toxicity tests, as they are easier to handle and count than younger individuals. Gravid females are not used in testing. The 3 to 5 mm size class individuals are passed through a 1,000 μ m mesh sieve and are retained on a 710 μ m mesh sieve. A 500 μ m mesh sieve has been used previously to retain amphipods of the size needed, but this results in a wider size range of amphipods used for testing. In preliminary NAF experiments, this wide size range may have contributed to variability in mortality observed that was not present when the 710 μ m mesh sieve was used to retain amphipods in later experiments. The amphipods passing through a 1000 μ m mesh sieve but trapped on a 710 μ m mesh sieve provide a more uniform size range of animals that is thought to decrease the previously-observed variability in mortality. Laboratories are encouraged to use this type of approach to reduce the variability in the size of amphipods used in the NAF/SBM testing program.

Table 1. Conditions for conducting 96-hour NAF and 10-day SBM sediment toxicity tests with *L. plumulosus*. Conditions listed are consistent with test conditions specified in ASTM E 1367-92 and subsequent updates (E 1367-99) unless otherwise noted.

Parameter	Conditions
Test type	Static whole sediment toxicity test
Temperature	20 \pm 1 C
Salinity	20 \pm 1‰
Light quality	Wide-spectrum fluorescent lights
Illuminance	500-1,000 lux
Photoperiod	14h light:10h dark*
Test chamber	1-L glass beaker or jar
Sediment volume	150 ml (2 cm depth)
Overlying water volume	600 ml (4:1 [v/v] water to sediment ratio)
Renewal of overlying water	None

	74
Size and life stage of amphipods	3-5 mm; immature and adult
Number of organisms/chamber	20
Number of test concentrations	5
Number of replicate chambers/treatment	5 in both controls and test treatments
Feeding	None
Aeration	Water in each test chamber should be aerated throughout the test.
Overlying Water	Clean natural or synthetic seawater
Overlying water quality	Temperature, salinity, pH, and D.O. daily; ammonia, as needed
Test duration	96 hours
Endpoint	Survival
Test acceptability	Minimum mean control survival of 90% and satisfaction of criteria outlined in Table 2.

***Although ASTM E1367 specifies 16h light:8h dark, the photoperiod was changed to 14h light:10h dark to be consistent with the *Mysidopsis bahia* bioassay for drilling fluids (58 CFR 12453, 1993).**

Table 2. Test acceptability requirements for 10-day NAF and 96-hr SBM tests with *L. plumulosus*. Requirements listed are consistent with those specified in ASTM E 1367-92 and subsequent updates (E 1367-99)*.

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- A 10-day NAF and 96-hr SBM toxicity tests are unacceptable if more than a total of 10% of the control organisms die, or if the coefficient of variation (CV) of control survival is equal to or greater than 40%.

Ten-day NAF and 96-hr SBM toxicity tests should usually be considered unacceptable if one or more of the following occurred:

- All test chambers were not identical.
 - Test organisms were not randomly or impartially distributed to test chambers.
 - Required reference standard was not included in the test.
 - All test animals were not from the same population, were not all of the same species, or were not of acceptable quality.
 - Amphipods from a wild population were maintained in the laboratory for more than two weeks, unless the effects of prolonged maintenance in the laboratory has been shown to have no significant effect on sensitivity.
 - The test organisms were not acclimated at the test temperature and salinity at least 48 hours before they were placed in the test chambers.
 - Temperature and dissolved oxygen concentrations were not measured.
-

***These guidelines are not identical to those listed ASTM E 1367 in part because some acceptability guidelines listed in E1367-92 are not applicable or practical for the NAF/SBM toxicity testing program.**

Table 3. Culture conditions for *L. plumulosus*. Conditions listed are consistent with culture conditions specified in ASTM E 1367-92 and subsequent updates (E 1367-99).

Parameter	Conditions
Temperature	20±1 C
Salinity	20±1‰
Light quality	Wide-spectrum fluorescent or cool white lights
Illuminance	500-1,000 lux
Photoperiod	14h light:10h dark
Culture chamber	Shallow plastic tubs or glass aquaria
Sediment volume	1-2 cm depth at bottom of each culture chamber
Renewal of overlying water	Static renewal (30-50% water volume change 2-4 times per week)
Number of organisms/chamber	Start with about 300 mixed age (mostly immature and young adults) individuals per chamber
Feeding	0.1 to 0.5 g dry mixture 2-3 times per week (see text)
Aeration	Continuous gentle to moderate aeration so as to not suspend sediments
Overlying Water	Clean natural or synthetic seawater
Overlying water quality	Salinity, temperature, and ammonia during culture start-up

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Appendix B

PROTOCOL FOR THE DETERMINATION OF DEGRADATION OF Non aqueous BASE FLUIDS IN A MARINE CLOSED BOTTLE BIODEGRADATION TEST SYSTEM: MODIFIED ISO 11734

Section 1: Summary of Method

This method determines the anaerobic degradation potential of mineral oils, paraffin oils and non aqueous fluids (NAF) in sediments. These substrates are base fluids for formulating offshore drilling fluids. The test evaluates base fluid biodegradation rates by monitoring gas production due to microbial degradation of the test fluid in natural marine sediment.

The test procedure places a mixture of marine/estuarine sediment, test substrate (hydrocarbon or controls) and seawater into clean 120 ml (150 ml actual volume) Wheaton serum bottles. The test is run using four replicate serum bottles containing 2000 mg carbon/kg dry weight concentration of test substrate in sediment. The use of resazurin dye solution (1 ppm) evaluates the anaerobic (redox) condition of the bottles (dye is blue when oxygen is present, reddish in low oxygen conditions and colorless if oxygen free). After capping the bottles, a nitrogen sparge removes air in the headspace before incubation begins. During the incubation period, the sample should be kept at a constant temperature of 29 (+/-1)°C. Gas production and composition is measured approximately every two weeks. The samples need to be brought to ambient temperature before making the measurements. Measure gas production using a pressure gauge. Barometric pressure is measured at the time of testing to make necessary volume adjustments.

ISO 11734 specifies that total gas is the standard measure of biodegradation. While modifying this test for evaluating biodegradation of NAF's, methane was also monitored and found to be an acceptable method of evaluating biodegradation. Appendix 1 contains the procedures used to follow biodegradation by methane production. Measurement of either total gas or methane production is permitted. If methane is followed, determine the composition of the gas by using gas chromatography (GC) analysis at each sampling. At the end of the test when gas production stops, or at around 275 days, an analysis of sediment for substrate content is possible. Common methods which have been successfully used for analyzing NAF's from sediments are listed in Appendix 2.

Section 2: System Requirements

This environmental test system has three phases, spiked sediment, overlying seawater, and a gas headspace. The sediment/test compound mixture is combined with synthetic sea water and transferred into 120 mL serum bottles. The total volume of sediment/sea water mixture in the bottles is 75 mL. The volume of the sediment layer will be approximately 50 mL, but the exact volume of the sediment will depend on sediment characteristics (wet:dry ratio and density). The amount of synthetic sea water will be calculated to bring the total volume in the bottles to 75 mL. The test systems are maintained at a temperature of 29 1°C during incubation. The test systems are brought to ambient temperatures prior to measuring pressure or gas volume.

Section 2.1: Sample Requirements

The concentration of base fluids are at least 2000 mg carbon test material/kg dry sediment. Carbon concentration is determined by theoretical composition based on the chemical formula or by chemical analysis by ASTM D5291-96. Sediments with positive, intermediate and negative control substances as well as a C1618 Internal Olefin type base fluid will be run in conjunction with test materials under the same conditions. The positive control is ethyl oleate (CAS 111-62-6), the intermediate control is 1-hexadecene (CAS 629-73-2), and the negative control is squalane (CAS 111-01-3). Controls must be of analytical grade or the highest grade available. Each test control concentration should be prepared according to the mixing procedure described in Section 3.1.

Product names will be used for examples or clarification in the following text. Any use of trade or product names in this publication is for descriptive use only, and does not constitute endorsement by EPA or the authors

Section 2.2: Seawater Requirements

Synthetic seawater at a salinity of 25 ppt should be used for the test. The synthetic seawater should be prepared by mixing a commercially available artificial seawater mix, into high purity distilled or de-ionized water. The seawater should be aerated and allowed to age for approximately one month prior to use.

Section 2.3: Sediment Requirements

The dilution sediment must be from a natural estuarine or marine environment and be free of the compounds of interest. The collection location, date and time will be documented and reported. The sediment is prepared by press-sieving through a 2000-micron mesh sieve to remove large debris, then press-sieving through a 500-micron sieve to remove indigenous organisms that may confound test results. The water content of the sediment should be less than 60%(w/w) or a wet to dry ratio of 2.5. The sediment should have a minimum organic matter content of 3% (w/w) as determined by ASTM D2974-87 (95) (Method A and D and calculate organic matter as in section 12 of method ASTM D2974-87).

To reduce the osmotic shock to the microorganisms in the sediment the salinity of the sediment's pore water should be between 20-30 ppt. Sediment should be used for testing as soon as possible after field collection. If required, sediment can be stored in the dark at 4°C with 3-6 inches of overlying water in a sealed container for a maximum period of 2 months prior to use.

Section 3: Test Set up

The test is set up by first mixing the test or control substrates into the sediment inoculum, then mixing in seawater to make a pourable slurry. The slurry is then poured into serum bottles, which are then flushed with nitrogen and sealed.

Section 3.1: Mixing Procedure

Because base fluids are strongly hydrophobic and do not readily mix with sediments, care must be taken to ensure base fluids are thoroughly homogenized within the sediment. All concentrations are weight-to-weight comparisons (mg of base fluid to kg of dry control sediment). Sediment and base fluid mixing will be accomplished by using the following method.

- 3.1.1. Determine the wet to dry weight ratio for the control sediment by weighing approximately 10 sub-samples of approximately 1 g each of the screened and homogenized wet sediment into tared aluminum weigh pans. Dry sediment at 105 C for 18-24 h. Remove the dried sediments and cool in a desiccator. Repeat the drying, cooling, and weighing cycle until a constant weight is achieved (within 4% of previous weight). Re-weigh the samples to determine the dry weight. Calculate the mean wet and dry weights of the 10 sub samples and determine the wet/dry ratio by dividing the mean wet weight by the mean dry weight using Formula 1. This is required to determine the weight of wet sediment needed to prepare the test samples.

$$\frac{\text{Mean Wet Sediment Weight (g)}}{\text{Mean Dry Sediment Weight (g)}} = \text{Wet to Dry Ratio} \quad [1]$$

- 3.1.2. Determine the density (g/ml) of the wet sediment. This will be used to determine total volume of wet sediment needed for the various test treatments. One method is to tare a 5 ml graduated cylinder and add about 5 ml of homogenized sediment. Carefully record the volume then weigh this volume of sediment. Repeat this a total of three times. To determine the wet sediment density, divide the weight by volume per the following formula:

$$\frac{\text{Mean Wet Sediment Weight (g)}}{\text{Mean Wet Sediment Volume (ml)}} = \text{Wet Sediment Density (g/ml)} \quad [2]$$

- 3.1.3. Determine the amount of base fluid to be spiked into wet sediment in order to obtain the desired initial base fluid concentration of 2000 mg carbon/kg dry weight. An amount of wet sediment that is the equivalent of 30 g of dry sediment will be added to each bottle. A typical procedure is to prepare enough sediment for 8 serum bottles (3 bottles to be sacrificed at the start of the test, 4 bottles incubated for headspace analysis, and enough extra sediment for 2 extra bottles). Extra sediment is needed because some of the sediment will remain coated onto the mixing bowl and utensils. Experience with this test may indicate that preparing larger volumes of spiked sediment is a useful practice, then the following calculations should be adjusted accordingly.

- 3.1.3.1 Determine the total weight of dry sediment needed to add 30 g dry sediment to 8 bottles. If more bottles are used then the calculations should be modified accordingly. For example:

$$30 \text{ g dry sediment per bottle} \times \frac{81}{8} = 240 \text{ g dry sediment} \quad [3]$$

3.1.3.2 Determine the weight of base fluid, in terms of carbon, needed to obtain a final base fluid concentration of 2000 mg carbon/kg dry weight. For example:

$$\frac{2000 \text{ mg carbon}}{\text{per kg dry sediment}} \times \frac{240 \text{ g}}{1000} = 480 \text{ mg carbon} \quad [4]$$

3.1.3.3 Convert from mg of carbon to mg of base fluid.

This calculation will depend on the % fraction of carbon present in the molecular structure of each base fluid. For the control fluids, ethyl oleate is composed of 77.3% carbon, hexadecene is composed of 85.7% carbon, and squalane is composed of 85.3% carbon. The carbon fraction of each base fluid should be supplied by the manufacturer or determined before use. ASTM D5291-96 or equivalent will be used to determine composition of fluid.

To calculate the amount of base fluid to add to the sediment, divide the amount of carbon (480 mg) by the percent fraction of carbon in the fluid.

For example, the amount of ethyl oleate added to 240 g dry weight sediment can be calculated from the following equation:

$$480 \text{ mg carbon} \left(\frac{77.3}{100} \right) = 621 \text{ mg ethyl oleate} \quad [5]$$

Therefore, add 621 mg of ethyl oleate to 240 g dry weight sediment for a final concentration of 2000 mg carbon/kg sediment dry weight.

3.1.4. Mix the calculated amount of base fluid with the appropriate weight of wet sediment.

3.1.4.1 Use the wet:dry ratio to convert from g sediment dry weight to g sediment wet weight, as follows:

$$240 \text{ g dry sediment} \times \text{wet:dry ratio} = \text{g wet sediment needed} \quad [6]$$

3.1.4.2 Weigh the appropriate amount of base fluid (calculated in section 3.1.3.3) into stainless mixing bowls, tare the vessel weight, then add the wet sediment calculated in equation 5, and mix with a high shear dispersing impeller for 9 minutes.

The sediment is now mixed with synthetic sea water to form a slurry that will be transferred into the bottles.

Section 3.2: Creating Seawater/Sediment Slurry

Given that the total volume of sediment/sea water slurry in each bottle is to be 75 mL, determine the volume of sea water to add to the wet sediment.

- 3.3.1 If each bottle is to contain 30 g dry sediment, calculate the weight, and then the volume, of wet sediment to be added to each bottle

$$30 \text{ g dry sediment} \times \text{wet:dry ratio} = \text{g wet sediment added to each bottle} \quad [7]$$

$$\text{g wet sediment} \div \text{density (g/mL) of wet sediment} = \text{mL wet sediment} \quad [8]$$

- 3.3.2 Calculate volume of sea water to be added to each bottle

$$75 \text{ mL total volume} - \text{mL wet sediment (from eq. 8)} = \text{mL of sea water} \quad [9]$$

- 3.3.3 Determine the ratio of sea water to wet sediment (volume:volume) in each bottle

$$\frac{\text{volume sea water per bottle (eq. 9)}}{\text{volume sediment per bottle (eq. 8)}} = \text{ratio of sea water:wet sediment} \quad [10]$$

- 3.3.4 Convert the wet sediment weight from equation 6 into a volume using the sediment density.

$$\text{g wet sediment (eq. 6)} \div \text{density} = \text{volume (mL) of sediment} \quad [11]$$

- 3.3.5 Determine the amount of sea water to mix with the wet sediment.

$$\begin{aligned} \text{mL wet sediment (eq. 11)} \times \text{sea water:sediment ratio (eq. 10)} \\ = \text{mL sea water to add to wet sediment} \end{aligned} \quad [12]$$

Mix sea water thoroughly with wet sediment to form a sediment/sea water slurry.

Section 3.3: Bottling the Sediment Seawater Slurry

The total volume of sediment/sea water slurry in each bottle is to be 75 mL. Convert the volume (mL) of sediment/sea water slurry into a weight (g) using the density of the sediment and the sea water.

- 3.4.1 Determine the weight of sediment to be added to each bottle
 $\text{mL sediment (eq. 8)} \times \text{density of wet sediment (g/mL)} = \text{g wet sediment} \quad [14]$

- 3.4.2 Determine the weight of sea water to be added to each bottle

$$\text{mL sea water (eq. 9)} \times \text{density of sea water (1.01 g/mL)} = \text{g sea water} \quad [15]$$

3.4.3 Determine weight of sediment/sea water slurry to be added to each bottle

$$\text{g wet sediment (eq. 14)} + \text{g sea water (eq. 15)} = \text{g sediment/sea water slurry [16]}$$

This should provide each bottle with 30 g dry sediment in a total volume of 75 mL.

3.4.4 Putting the sediment:seawater slurry in the serum bottles.

Note: The slurry will need to be constantly stirred to keep the sediment suspended.

Place a tared serum bottle on a balance and add the appropriate amount of slurry to the bottle using a funnel. Once the required slurry is in the bottle remove the funnel, add 2-3 drops (25 μl) of a 1gram/L resazurin dye stock solution. Cap the bottle with a butyl rubber stopper (Bellco Glass, Part #2048- 11800)and crimp with an aluminum seal (Bellco Glass Part #2048-11020).

Using a plastic tube with a (23 gauge, 1 inch long) needle attached to one side and a nitrogen source to the other, puncture the serum cap with the needle. Puncture the serum cap again with a second needle to sparge the bottle's headspace of residual air for two minutes. The nitrogen should be flowing at no more than 100 mL/min to encourage gentle displacement of oxygenated air with nitrogen. Faster nitrogen flow rates would cause mixing and complete oxygen removal would take much longer. Remove the nitrogen needle first to avoid any initial pressure problems. The second (vent) needle should be removed within 30 seconds of removing the nitrogen needle.

Triplicate blank test systems are prepared, with similar quantities of sediment and seawater without any base fluid. Incubate in the dark at a constant temperature of 29 1°C .

Record the test temperature. The test duration is dependent on base fluid performance, but at a maximum should be no more than 275 days. Stop the test after all base fluids have achieved a plateau of gas production. At termination, base fluid concentrations can be verified in the terminated samples by extraction and GC analysis according to Appendix 2.

Section 4: Concentration Verification Chemical Analyses

Because of the difficulty of homogeneously mixing base fluid with sediment, it is important to demonstrate that the base fluid is evenly mixed within the sediment sea water slurry that was added to each bottle. Of the seven serum bottles set up for each test or control condition, three are randomly selected for concentration verification analyses. These should be immediately placed at 4 C and a sample of sediment from each bottle should be analyzed for base fluid content as soon as possible. The coefficient of variation (CV) for the replicate samples must be less than 20%. The results should show recovery of at least 70% of the spiked base fluid. Use an appropriate analytical procedure described in Appendix 2 to perform the extractions and

analyses. If any set of sediments fail the criteria for concentration verification, then the corrective action for that set of sediments is also outlined in Appendix 2.

The nominal concentrations and the measured concentrations from the three bottles selected for concentration verification should be reported for the initial test concentrations. 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 must be stopped and restarted with adequately mixed sediment.

Section 5 Gas monitoring procedures

Biodegradation is measured by total gas as specified in ISO 11734. Methane production can also be tracked and is described in Appendix 1.

Section 5.1 Total Gas monitoring procedures

Bottles should be brought to room temperature before readings are taken. The bottles are observed to confirm that the resazurin has not oxidized to pink or blue. Total gas production in the culture bottles should be measured using a pressure transducer (one source is Biotech International). The pressure readings from test and control cultures are evaluated against a calibration curve created by analyzing the pressure created by known additions of gas to bottles established identically to the culture bottles. Bottles used for the standard curve contain 75 mL of water, and are sealed with the same rubber septa and crimp cap seals used for the bottles containing sediment. After the bottles used in the standard curve have been sealed, a syringe needle inserted through the septa is used to equilibrate the pressure inside the bottles to the outside atmosphere. The syringe needle is removed and known volumes of air are injected into the headspace of the bottles. Pressure readings provide a standard curve relating the volume of gas injected into the bottles and headspace pressure. No less than three points may be used to generate the standard curve. A typical standard curve may use 0, 1, 5, 10, 20 and 40 ml of gas added to the standard curve bottles.

The room temperature and barometric pressure (to two digits) should be recorded at the time of sampling. One option for the barometer is Fisher Part #02-400 or 02-401. Gas production by the sediment is expressed in terms of the volume (mL) of gas at standard temperature (0°C = 273°K) and pressure (1 atm = 30 inches of Hg) using Eqn.17.

$$V_2 = \frac{P_1 * V_1 * T_2}{T_1 * P_2} \quad [17]$$

Where: V_2 = volume of gas production at standard temperature and pressure

P_1 = barometric pressure on day of sampling (inches of Hg)

V_1 = volume of gas measured on day of sampling (mL)

T_2 = standard temperature = 273°K

T_1 = temperature on day of sampling ($^{\circ}\text{C} + 273 = ^{\circ}\text{K}$)

P_2 = standard pressure = 30 inches Hg

A estimation can be made of the total volume of anaerobic gas that will be produced in the bottles. The gas production measured for each base fluid can be expressed as a percent of predicted total anaerobic gas production.

- 5.1.1. Calculate the total amount of carbon in the form of the base fluid present in each bottle

Each bottle is to contain 30 g dry weight sediment. The base fluid concentration is 2000 mg carbon/kg dry weight sediment. Therefore:

$$2000 \text{ mg carbon/kg sediment} \times (30 \text{ g}/1000) = 60 \text{ mg carbon per bottle} \quad [18]$$

- 5.1.2. Theory states that anaerobic microorganisms will convert 1 mole of carbon substrate into 1 mole of total anaerobic gas production

Calculate the number of moles of carbon in each bottle.

The molecular weight of carbon is 12 (i.e. 1 mole of carbon = 12 g). Therefore, the number of moles of carbon in each bottle can be calculated.

$$(60 \text{ mg carbon per bottle}/1000) \quad 12 \text{ g/mole} = 0.005 \text{ moles carbon} \quad [19]$$

- 5.1.3. Calculate the predicted volume of anaerobic gas

One mole of gas equals 22.4 L (at standard temperature and pressure), therefore, $0.005 \text{ moles} \times 22.4 \text{ L} = 0.112\text{L}$ (or 112 mL total gas production). [20]

Section 5.2 Gas Venting

If the pressure in the serum bottle is too great for the pressure transducer or syringe, some of the excess gas must be wasted. The best method to do this is to vent the excess gas right after measurement. To do this, remove the barrel from a 10-mL syringe and fill it 1/3 full with water. This is then inserted into the bottle through the stopper using a small diameter (high gauge) needle. The excess pressure is allowed to vent through the water until the bubbles stop. This allows equalization of the pressure inside the bottle to atmospheric without introducing oxygen. The amount of gas vented (which is equal to the volume determined that day) must be kept track of each time the bottles are vented. A simple way to do this in a spreadsheet format is to have a separate column in which cumulative vented gas is tabulated. Each time the volume of gas in the

cultures is analyzed, the total gas produced is equal to the gas in the culture at that time plus the total of the vented gas.

To keep track of the methane lost in the venting procedure, multiply the amount of gas vented each time by the corrected % methane determined on that day. The answer gives the volume of methane wasted. This must be added into the cumulative totals similarly to the total gas additions.

Section 6: Test Acceptability and Interpretation

Section 6.1 Test acceptability

At day 275 or when gas production has plateaued, whichever is first, the controls are evaluated to confirm that the test has been performed appropriately. In order for this modification of the closed bottle biodegradation test to be considered acceptable, all the controls must meet the biodegradation levels indicated in Table 1. The intermediate control hexadecene must produce at least 30% of the theoretical gas production. This level may be reexamined after two years and more data has been generated.

Table 1: Test Acceptability Criteria

Concentration	Percent Biodegradability as a Function of Gas Measurement		
	Positive control	Squalane negative control	Hexadecene intermediate control
2000 mg carbon/kg	≥ 60% theoretical	≤ 5% theoretical	≥ 30% theoretical

Section 6.2 Interpretation

In order for a fluid to pass the closed bottle test, the biodegradation of the base fluid as indicated by the total amount of total gas (or methane) generated once gas production has plateaued (or at the end of 275 days, which ever is first) must be greater than or equal to the volume of gas (or methane) produced by the reference standard (internal elefin or ester).

The method for evaluating the data to determine whether a fluid has passed the biodegradation test must use the equations:

$$\frac{\% \text{ Theoretical gas production of reference fluid}}{\% \text{ Theoretical gas production of NAF}} \# 1.0$$

Where: NAF = stock base fluid being tested for compliance

Reference Fluid = C₁₆-C₁₈ internal olefin or C₁₂-C₁₄ or C₈ ester reference fluid

Appendix B-1 Methane measurement

Section A1 Methane monitoring procedures

The use of total gas production alone may result in an underestimation of the actual metabolism occurring since CO₂ is slightly soluble in water. An acceptable alternative method is to monitor methane production and total gas production. This is easily done using GC analysis. A direct injection of headspace gases can be made into a GC using almost any packed or capillary column with an FID detector. Unless volatile fuels or solvents are present in the test material or the inocula, the only component of the headspace gas that can be detected using an FID detector is methane. The percent methane in the headspace gas is determined by comparing the response of the sample injections to the response from injections of known percent methane standards. The percent methane is corrected for water vapor saturation using Eqn. 8 and then converted to a volume of dry methane using Eqn. 9.

$$\text{Corrected \% CH}_4 = \frac{\% \text{ CH}_4}{1 - \frac{[D * 22.4 \text{ L/mol}]}{18 \text{ g/mol} * 1000}} \quad [\text{A1}]$$

Where:

D = the density of water vapor at saturation (g/m³, can be found in CRC Handbook of Chemistry and Physics) for the temperature of sampling.

$$V_{\text{CH}_4} (\text{ml}) = (S + V) * \frac{(P - P_w)}{(T + 273)} * \frac{\text{CH}_4}{100} * \frac{273}{760} \quad [\text{A2}]$$

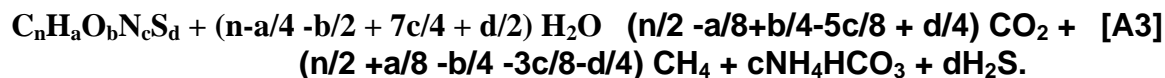
where: V_{CH_4} = the volume of methane in the bottle
 S = volume of excess gas production (measured with a pressure transducer)
 V = volume of the headspace in the culture bottle (total volume - liquid phase)
 P = barometric pressure (mm Hg, measured with barometer)
 T = temperature (C)
 P_w = vapor pressure of water at T (mm Hg, can be found in CRC Handbook of Chemistry and Physics)
 CH₄ = % methane in headspace gas (after correction for water vapor)

The total volume of serum bottles sold as 125 mL bottles (Wheaton) is 154.8 mL.

The volumes of methane produced are then compared to the volumes of methane in the controls to determine if a significant inhibition of methane production or a significant increase of methane production has been observed. Effective statistical analyses are important, as variability in the results is common due to the heterogeneity of the inoculum's source. It is also common to observe that the timing of the initiation of culture activity is not equal in all of the cultures. Expect a great variability over the period when the cultures are active, some replicates will start sooner than others, but all of the replicates should eventually reach similar levels of base fluid degradation and methane production.

Section A2 Expected Methane Production Calculations

The amount of methane expected can be calculated using the equation of Symons and Buswell (Eqn. A3). In the case of complete mineralization, all of the carbon will appear as either CO₂ or CH₄, thus the total moles of gas produced will be equal to the total moles of carbon in the parent molecule. The use of the Buswell equation allows you to calculate the effects the redox potential will have on the distribution of the products in methanogenic cultures. More reduced electron donors will allow the production of more methane, while more oxidized electron donors will cause a production of more carbon dioxide.



An example calculation of the expected methane volume in a culture fed 2000 mg/kg hexadecene is as follows. The application of Symons and Buswell's equation reveals that hexadecene (C₁₆H₃₂) will yield 4 moles of CO₂ and 12 moles of CH₄. Assuming 30 g of dry sediment are added to the bottles with 2,334 mg hexadecene/kg dry sediment (i.e. equivalent to 2000 mg carbon/kg dry sediment) the calculation is as follows.

$$\frac{12 \text{ mole } CH_4}{\text{mole hexadecene}} * \frac{224 \text{ L}}{\text{mole } CH_4} * \frac{1000 \text{ ml}}{\text{L}} * \frac{1 \text{ mole hexadecene}}{2244 \text{ g hexadecene}} * \\ \frac{2.3 \text{ g hexadecene}}{\text{kg dry soil}} * \frac{0.03 \text{ kg}}{\text{culture}} = 84 \text{ ml} \quad [A4]$$

By subtracting the average amount of methane in control bottles from the test bottles and then dividing by the expected volume an evaluation of the completion of the process may be conducted.

Appendix B-2

The Concentration Verification analyses is required at the beginning of the test to ensure homogeneity and confirm that the required amount of fluid was delivered to the sediments at the start of the test

- Three samples per fluid need to be analyzed and achieve $\leq 20\%$ Coefficient of Variability and an average of $\geq 70\%$ to $\leq 120\%$ of fluid delivered to sediment.
- If a third party performs the analysis, then the laboratory should be capable of delivering the homogeneity data within seven days, in order to identify any samples that do not meet the homogeneity requirement as quickly as possible.
- If one sediment/fluid set, out a multiple set batch of samples, fails these criteria, then that one set of samples must be discarded and a fresh set of spiked sediment prepared, started, and analyzed to ensure homogeneity. The same stock sediment is used to prepare the replacement set(s). The remaining sets do not need to be re-mixed or restarted.
- The re-mixed set(s) will need to be run the additional days as appropriate to ensure that the total number of days is the same for all sets of bottles, even though the specific days are not aligned.
- Re-mixing of bottle sets can be performed multiple times as a result of a failure of the analytical criteria, until the holding time for the stock sediment has expired (60 days). If the problem set(s) has not fallen within the acceptable analytical criteria by then, it must not be part of the batch of bottles run. If the problem batch is one of the controls, and those controls were not successfully prepared when the sediment holding time expired, then the entire test must be restarted.

References

The following references identify analytical methods that have historically been successful for achieving the analytical quality criteria

Continental Shelf Associates report 1998. Joint EPA/Industry screening survey to assess the deposition of drill cuttings and associated synthetic based mud on the seabed of the Louisiana continental shelf, Gulf of Mexico. Analysis by Charlie Henry report Number IES/RCAT97-36 GC-FID and GC/MS

EPA Method 3550 for extraction with EPA Method 8015 for GC-FID

Webster, L; Mackie, P.R.; Hird, S.J.; Munro, P.D.; Brown, N.A. and Moffatt, C.F. (1997) Development of Analytical Methods for the Determination of Synthetic Mud Base Fluids in Marine Sediments *Analyst* 122:1485-1490.

Munro, P.D., B Croce, C.F. Moffet, N.A Brown, A.D. McIntosh, S.J.Hird, R.M. Stagg. 1998. Solid-phase test for comparison for degradation rates of synthetic mud base fluids used in the off shore drilling industry. *Environ. Toxicol. Chem.* 17:1951-1959.

PROGRAM QUALITY ASSURANCE AND QUALITY CONTROL:

Calibration

- All equipment / instrumentation will be calibrated in accordance with the test method or the manufacture's instructions and may be scheduled or triggered
- Where possible, standards used in calibration will be traceable to a nationally recognized standard (e.g., certified standard by NIST)
- All calibration activities will be documented and the records retained
- The source, lot, batch number, and expiration date of all reagents used will be documented and retained

Maintenance

- All equipment / instrumentation will be maintained in accordance with the test method or the manufacture's instructions and may be scheduled or triggered
- All maintenance activities will be documented and the records retained

Data Management and Handling

- All primary (raw) data will be correct, complete, without selective reporting, and will be maintained
- Hand-written data will be recorded in lab notebooks or electronically at the time of observation
- All hand-written records will be legible and amenable to reproduction by electrostatic copiers
- All changes to data or other records will be made by:
 - using a single line to mark-through the erroneous entry (maintaining original data legibility)
 - write the revision
 - initial, date, and provide revision code (see attached or laboratory's equivalent)
- All data entry, transcriptions, and calculations will be verified by a qualified person
 - verification will be documented by initials of verifier and date
- Procedures will be in place to address data management procedures used (at minimum):
 - Significant figures
 - Rounding practices
 - Identification of outliers in data series
 - Required statistics

Document Control

- All technical procedures, methods, work instructions, standard operating procedures must be documented and approved by laboratory management prior to the implementation
- All primary data will be maintained by the contractor for a minimum of five (5) years

Personnel and Training

- Only qualified personnel shall perform laboratory activities

- Records of staff training and experience will be available. This will include initial and refresher training (as appropriate)

Test Performance

- All testing will be done in accordance with the specified test methods
- Receipt, arrival condition, storage conditions, dispersal, and accountability of the test article will be documented and maintained
- Receipt or production, arrival or initial condition, storage conditions, dispersal, and accountability of the test matrix (e.g., sediment or artificial seawater) will be documented and maintained
- Source, receipt, arrival condition, storage conditions, dispersal, and accountability of the test organisms (including inoculum) will be documented and maintained
- Actual concentrations administered at each treatment level will be verified by appropriate methodologies
- Any data originating at a different laboratory will be identified and the laboratory fully referenced in the final report.

Appendix C

Determination of Crude Oil Contamination in Non aqueous Drilling Fluids by Gas Chromatography/Mass Spectrometry (GC/MS)

1.0 Scope and Application

- 1.1 This method determines crude (formation) oil contamination, or other petroleum oil contamination, in non aqueous drilling fluids (NAFs) by comparing the gas chromatography/mass spectrometry (GC/MS) fingerprint scan and extracted ion scans of the test sample to that of an uncontaminated sample.
- 1.2 This method can be used for monitoring oil contamination of NAFs or monitoring oil contamination of the base fluid used in the NAF formulations.
- 1.3 Any modification of this method beyond those expressly permitted shall be considered as a major modification subject to application and approval of alternative test procedures.
- 1.4 The gas chromatography/mass spectrometry portions of this method are restricted to use by, or under the supervision of analysts experienced in the use of GC/MS and in the interpretation of gas chromatograms and extracted ion scans. Each laboratory that uses this method must generate acceptable results using the procedures described in Sections 9.2, 10.1, and 13 of this method.

2.0 Summary of Method

- 2.1 Analysis of NAF for crude oil contamination is a step-wise process. Qualitative assessment of the presence or absence of crude oil is performed first. If crude oil is detected in this qualitative assessment, quantitative analysis of the crude oil concentration is performed. When more data are available, the NIST calibration may need to be adjusted.
- 2.2 A sample of NAF is centrifuged, to obtain a solids free supernate.
- 2.3 The sample to be tested is prepared by removing an aliquot of the solids free supernate, spiking it with internal standard, and analyzing it using GC/MS techniques. The components are separated by the gas chromatograph and detected by the mass spectrometer.
- 2.4 Qualitative identification of crude oil contamination is performed by comparing the Total Ion Chromatograph (TIC) scans and Extracted Ion Profile (EIP) scans of test sample to that of uncontaminated base fluids, and examining the profiles for chromatographic signatures diagnostic of oil contamination.
- 2.5 The presence or absence of crude oil contamination observed in the full scan profiles and selected extracted ion profiles determines further sample quantitation and reporting.

- 2.6 If crude oil is detected in the qualitative analysis, quantitative analysis is performed by calibrating the GC/MS using a designated NAF spiked with known concentrations of a designated oil.
- 2.7 Quality is assured through reproducible calibration and testing of GC/MS system and through analysis of quality control samples.

3.0 Definitions

- 3.1 A NAF is one in which the continuous phase is a water immiscible fluid such as an oleaginous material (e.g., mineral oil, enhance mineral oil, paraffinic oil, or synthetic material such as olefins and vegetable esters).
- 3.2 TIC-Total Ion Chromatograph.
- 3.3 EIP-Extracted Ion Profile.
- 3.4 TCB-1,3,5-trichlorobenzene is used as the internal standard in this method.
- 3.5 SPTM-System Performance Test Mix standards are used to establish retention times and monitor detection levels.

4.0 Interferences and Limitations

- 4.1 Solvents, reagents, glassware, and other sample processing hardware may yield artifacts and/or elevated baselines causing misinterpretation of chromatograms.
- 4.2 All Materials used in the analysis shall be demonstrated to be free from interferences by running method blanks. Specific selection of reagents and purification of solvents by distillation in all-glass systems may be required.
- 4.3 Glassware is cleaned by rinsing with solvent and baking at 400 °C for a minimum of 1 hour.
- 4.4 Interferences may vary from source to source, depending on the diversity of the samples being tested.
- 4.5 Variations in and additions of base fluids and/or drilling fluid additives (emulsifiers, dispersants, fluid loss control agents, etc.) might also cause interferences and misinterpretation of chromatograms.
- 4.6 Difference in light crude oils, medium crude oils, and heavy crude oils will result in different responses and thus different interpretation of scans and calculated percentages.

5.0 Safety

- 5.1 The toxicity or carcinogenicity of each reagent used in this method has not been precisely determined; however each chemical should be treated as a potential health hazard. Exposure to these chemicals should be reduced to the lowest possible level.
- 5.2 Unknown samples may contain high concentration of volatile toxic compounds. Sample containers should be opened in a hood and handled with gloves to prevent exposure. In addition, all sample preparation should be conducted in a fume hood to limit the potential exposure to harmful contaminants.
- 5.3 This method does not address all safety issues associated with its use. The laboratory is responsible for maintaining a safe work environment and 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 available to all personnel involved in these analyses. Additional references to laboratory safety can be found in References 16.1 through 16.3.
- 5.4 NAF base fluids may cause skin irritation, protective gloves are recommended while handling these samples.

6.0 Apparatus and Materials

Note: Brand names, suppliers, and part 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.

- 6.1 Equipment for glassware cleaning.
 - 6.1.1 Laboratory sink with overhead fume hood.
 - 6.1.2 Kiln-Capable of reaching 450°C within 2 hours and holding 450°C within $\pm 10^\circ\text{C}$, with temperature controller and safety switch (Cress Manufacturing Co., Santa Fe Springs, CA B31H or X31TS or equivalent).
- 6.2 Equipment for sample preparation.
 - 6.2.1 Laboratory fume hood.
 - 6.2.2 Analytical balance-Capable of weighing 0.1 mg.
 - 6.2.3 Glassware.
 - 6.2.3.1 Disposable pipettes-Pasteur, 150 mm long by 5 mm ID (Fisher Scientific 13-678-6A, or equivalent) baked at 400°C for a minimum of 1 hour.
 - 6.2.3.2 Glass volumetric pipettes or gas tight syringes-1.0-mL $\pm 1\%$ and 0.5-mL $\pm 1\%$.

- 6.2.3.3 Volumetric flasks-Glass, class A, 10-mL, 50-mL and 100-mL.
- 6.2.3.4 Sample vials-Glass, 1- to 3-mL (baked at 400°C for a minimum of 1 hour) with PTFE-lined screw or crimp cap.
- 6.2.3.5 Centrifuge and centrifuge tubes-Centrifuge capable of 10,000 rpm, or better, (International Equipment Co., IEC Centra MP4 or equivalent) and 50-mL centrifuge tubes (Nalgene, Ultratube, Thin Wall 2589 mm, #3410-2539).
- 6.3 Gas Chromatograph/Mass Spectrometer (GC/MS):
- 6.3.1 Gas Chromatograph-An analytical system complete with a temperature-programmable gas chromatograph suitable for split/splitless injection and all required accessories, including syringes, analytical columns, and gases.
- 6.3.1.1 Column-30 m (or 60 m) \times 0.32 mm ID (or 0.25 mm ID) 1mm film thickness (or 0.25mm film thickness) silicone-coated fused-silica capillary column (J&W Scientific DB-5 or equivalent).
- 6.3.2 Mass Spectrometer-Capable of scanning from 35 to 500 amu every 1 sec or less, using 70 volts (nominal) electron energy in the electron impact ionization mode (Hewlett Packard 5970MS or comparable).
- 6.3.3 GC/MS interface-the interface is a capillary-direct interface from the GC to the MS.
- 6.3.4 Data system-A computer system must be interfaced to the mass spectrometer. The system must allow the continuous acquisition and storage on machine-readable media of all mass spectra obtained throughout the duration of the chromatographic program. The computer must have software that can search any GC/MS data file for ions of a specific mass and that can plot such ion abundance versus retention time or scan number. This type of plot is defined as an Extracted Ion Current Profile (EIP). Software must also be available that allows integrating the abundance in any total ion chromatogram (TIC) or EIP between specified retention time or scan-number limits. It is advisable that the most recent version of the EPA/NIST Mass Spectral Library be available.

7.0 Reagents and Standards

- 7.1 Methylene chloride-Pesticide grade or equivalent. Used when necessary for sample dilution.
- 7.2 Standards-Prepare from pure individual standard materials or purchased as certified solutions. If compound purity is 96% or greater, the weight may be used without correction to compute the concentration of the standard.

- 7.2.1 Crude Oil Reference- NIST 1582 Petroleum Crude Oil Standard Reference Material (U.S. Department of commerce national Institute of Standards and Technology, Gaithersburg, MD 20899) . This oil will be used in the calibration procedures.
- 7.2.2 Synthetic Base Fluid-Obtain a sample of clean NAF base fluid (as sent from the supplier- has not been circulated downhole). This NAF base fluid will be used in the calibration procedures.
- 7.2.3 Internal standard-Prepare a 0.01 g/mL solution of 1,3,5-trichlorobenzene (TCB). Dissolve 1.0 g of TCB in methylene chloride and dilute to volume in a 100-mL volumetric flask. Stopper, vortex, and transfer the solution to a 150-mL bottle with PTFE-lined cap. Label appropriately, and store at 5 °C to 20 °C. Mark the level of the meniscus on the bottle to detect solvent loss.
- 7.2.4 GC/MS system performance test mix (SPTM) standards-The SPTM standards used in the development of this method contained octane, decane, dodecane, tetradecane, tetradecene, toluene, ethylbenzene, 1,2,4-trimethylbenzene, 1-methylnaphthalene and 1,3-dimethylnaphthalene. These compounds can be purchased individually, obtained as a mixture, or substituted for by a comparable mixture (i.e. Supelco, Catalog No.4-7300). Prepare a high concentration of the SPTM standard at 62.5 mg/mL (total SPTM mixture) in methylene chloride. Prepare a medium concentration SPTM standard at 1.25 mg/mL by transferring 1.0 mL of the 62.5 mg/mL solution into a 50 mL volumetric flask and diluting to the mark with methylene chloride. Finally, prepare a low concentration SPTM standard at 0.125 mg/mL by transferring 1.0 mL of the 1.25 mg/mL solution into a 10-mL volumetric flask and diluting to the mark with methylene chloride.
- 7.2.5 Crude oil/drilling fluid calibration standards-Prepare a 4-point crude oil/drilling fluid calibration at concentrations of 0% (no spike-clean drilling fluid), 0.5%, 1.0%, and 2.0% by volume according to the procedures outlined below using the Reference Crude Oil:
- 7.2.5.1 Label 4 vials with the following identification: Vial 1-0%Crude in NAF drilling fluid, Vial 2-0.5%Crude in NAF drilling fluid, Vial 3-1%Crude in NAF drilling fluid, and Vial 4-2%Crude in NAF drilling fluid.
- 7.2.5.2 Vial 1 will not be spiked with Reference Oil in order to retain a “0%” oil concentration, add 5 mL of clean NAF base fluid only.
- 7.2.5.3 Weigh 90.5 mg of NIST Crude Oil into Vial 2 and add 5 mL of clean NAF base fluid. This will be the 0.5% Crude equivalent in NAF mud standard.
- 7.2.5.4 Weigh 181 mg of NIST Crude Oil into Vial 3 and add 5 mL of clean NAF base fluid. This will be the 1.0% Crude equivalent in NAF mud standard.

- 7.2.5.5 Weigh 362 mg in NIST Crude Oil in Vial 4 and add 5 mL clean NAF base fluid. This will be the 2.0% Crude Equivalent in NAF mud standard
- 7.2.5.6 Thoroughly mix the contents of each of the 4 vial by shaking vigorously.,
- 7.2.5.7 Weigh 0.5 g of the mixture from Vial 1 directly into a tared and appropriately labeled GC straight vial. Spike the 0.5-g supernate with 500 μ L of the 0.01g/mL 1,3,5-trichlorobenzene internal standard solution (see 7.2.3), dilute with methylene chloride, cap with a Teflon lined crimp cap, and vortex for ca. 10 sec.
- 7.2.5.8 Repeat step 7.2.5.7 except use 0.5 g from Vial 2.
- 7.2.5.9 Repeat step 7.2.5.7 except use 0.5 g from Vial 3.
- 7.2.5.10 Repeat step 7.2.5.7 except use 0.5 g from Vial 4.
- 7.2.5.11 These 4 crude/oil drilling fluid calibration standards are now used for qualitative and quantitative GC/MS analysis.
- 7.2.6 Precision and recovery standard (mid level crude oil/drilling fluid calibration standard)- Prepare a mid point crude oil/drilling fluid calibration using NAF base fluid and Reference Oil at a concentration of 1.0% by volume. Prepare this standard according to the procedures outlined in Section 7.2.5.4. . Remove and spike with internal standard, as many 0.5-g aliquots as needed to complete the GC/MS analysis (see Section 11.6- bracketing authentic samples every 12 hours with precision and recovery standard) and the initial demonstration exercise described in Section 9.2.
- 7.2.7 Stability of standards
- 7.2.7.1 When not used, standards are stored in the dark, at 5 to 20°C in screw-capped vials with PTFE-lined lids. 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.
- 7.2.7.2 Solutions used for quantitative purposes shall be analyzed within 48 hours of preparation and on a monthly basis thereafter for signs of degradation. Standard will remain acceptable if the peak area remains within $\pm 15\%$ of the area obtained in the initial analysis of the standard.

8.0 Sample Collection Preservation and Storage

- 8.1 NAF samples and base fluid samples are collected in 100-to 200-mL glass bottles with PTFE-or aluminum foil lined caps.

- 8.2 Samples collected in the field will be stored refrigerated until time of preparation (not necessary for routine sample).
- 8.3 Sample and extract holding times for this method have not yet been established. However, based on tests experience samples should be analyzed within seven to ten days of collection and extracts analyzed within seven days of preparation.
- 8.4 After completion of GC/MS analysis, extracts should be refrigerated at ca. 4°C until further notification of sample disposal.

9.0 Quality Control

- 9.1 Each laboratory that uses this method is required to operate a formal quality assurance program (Reference 16.4). The minimum requirements of this program consist of an initial demonstration of laboratory capability, and 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.
 - 9.1.1 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 9.2.
 - 9.1.2 The analyst is permitted to modify this method to improve separations or lower the cost of measurements, provided all performance requirements are met. Each time a modification is made to the method, the analyst is required to repeat the calibration (Section 10.4) and to repeat the initial demonstration procedure described in Section 9.2.
 - 9.1.3 Analyses of blanks are required to demonstrate freedom from contamination. The procedures and criteria for analysis of a blank are described in Section 9.3.
 - 9.1.4 An analysis of a matrix spike sample is required to demonstrate method accuracy. The procedure and QC criteria for spiking are described in Section 9.4.
 - 9.1.5 Analysis of a duplicate field sample is required to demonstrate method precision. The procedure and QC criteria for duplicates are described in Section 9.5.
 - 9.1.6 Analysis of a sample of the clean NAF(s) (as sent from the supplier-has not been circulated downhole) used in the drilling operations is required.
 - 9.1.7 The laboratory shall, on an ongoing basis, demonstrate through calibration verification and the analysis of the precision and recovery standard (Section 7.2.6) that the analysis system is in control. These procedures are described in Section 11.6.

- 9.1.8 The laboratory shall maintain records to define the quality of data that is generated.
- 9.2 Initial precision and accuracy-The initial precision and recovery test is performed using the precision and recovery standard (1% by volume Crude Equivalent in NAF drilling fluid). The laboratory shall generate acceptable precision and recovery by performing the following operations.
- 9.2.1 Prepare four separate aliquots of the precision and recovery standard using the procedure outlined in Section 7.2.6. Analyze these aliquots using the procedures outlined in Section 11.
- 9.2.2 Using the results of the set of four analyses, compute the average recovery (X) in weight percent and the standard deviation of the recovery (s) for each sample.
- 9.2.3 If s and X meet the acceptance criteria of 80% to 110%, 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.
- 9.2.4 Accuracy and precision-The average percent recovery (P) and the standard deviation of the percent recovery (Sp) Express the accuracy assessment as a percent recovery interval from $P-2Sp$ to $P+2Sp$. For example, if $P=90\%$ and $Sp=10\%$ for four analyses of crude oil in NAF, the accuracy interval is expressed as 70% to 110%. Update the accuracy assessment on a regular basis.
- 9.3 Blanks-Rinse glassware and centrifuge tubes used in the method with ca. 30 mL of methylene chloride, remove a 0.5-g aliquot of the solvent, spike it with the 500 mL of the internal standard solution (Section 7.2.3) and analyze a 1-mL aliquot of the blank sample using the procedure in Section 11. Compute results per Section 12.
- 9.4 Matrix spike sample-Prepare a matrix spike sample according to procedure outlined in Section 7.2.6. Analyze the sample and calculate the concentration (% oil) in the drilling fluid and % recovery of oil from the spiked drilling fluid using the methods described in Sections 11 and 12.
- 9.5 Duplicates-A duplicate field sample is prepared according to procedures outlined in Section 7.3 and analyzed according to Section 11. The relative percent difference (RPD) of the calculated concentrations should be less than 15%.
- 9.5.1 Analyze each of the duplicates per the procedure in Section 11 and compute the results per Section 12.
- 9.5.2 Calculate the relative percent difference (RPD) between the two results per the following equation:

$$RPD = \frac{100 \times (D1 - D2)}{(D1 + D2) / 2}$$

where:

D1 = Concentration of crude oil in the sample

D2 = Concentration of crude oil in the duplicate sample

- 9.5.3 If the RPD criteria are not met, the analytical system shall be judged to be out of control, and the problem must be immediately identified and corrected and the sample batch re-analyzed.
- 9.6 Preparation of the clean NAF sample is performed according to procedures outlined in Section 7.3 except that the clean NAF (drilling fluid that has not been circulated downhole) is used. Ultimately the oil-equivalent concentration from the TIC or EIP signal measured in the clean NAF sample will be subtracted from the corresponding authentic field samples in order to calculate the true contaminant concentration (% oil) in the field samples (see Section 12).
- 9.7 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 (Section 9.2) and ongoing precision and recovery (Section 11.6) shall be identical, so that the most precise results will be obtained. The GC/MS instrument will provide the most reproducible results if dedicated to the setting and conditions required for the analyses given in this method.
- 9.8 Depending on specific program requirements, field replicates and field spikes of crude oil into samples may be required when this method is used to assess the precision and accuracy of the sampling and sample transporting techniques.

10.0 Calibration

- 10.1 Establish gas chromatographic/mass spectrometer operating conditions given in Table 1 below. Perform the GC/MS system hardware-tune as outlined by the manufacture. The gas chromatograph is calibrated using the internal standard technique. Note: Because each GC is slightly different, it may be necessary to adjust the operating conditions (carrier gas flow rate and column temperature and temperature program) slightly until the retention times in Table 2 are met.

TABLE 1.-GAS CHROMATOGRAPH/MASS SPECTROMETER (GC/MS) OPERATING CONDITIONS

Parameter	Setting
Injection port	280 C.
Transfer line	280 C.
Detector	280 C.

Initial Temperature	50 C.
Initial Time	5 minutes.
Ramp	50 to 300 C @ 5 C per minute.
Final Temperature.....	300 C.
Final Hold	20 minutes or until all peaks have eluted.
Carrier Gas	Helium.
Flow rate	As required for standard operation.
Split ratio	As required to meet performance criteria (~1:100).
Mass range	35 to 600 amu.

TABLE 2.-APPROXIMATE RETENTION TIMES FOR COMPOUNDS

Compound	Approximate Retention Time (minutes)
Toluene	5.6
Octane, n-C8	7.2
Ethylbenzene.....	10.3
1,2,4-Trimethylbenzene	16.0
Decane, n-C10	16.1
TCB (Internal Standard)	21.3
Dodecane, n-C12	22.9
1-Methylnaphthalene	26.7
1-Tetradecene	28.4
Tetradecane, n-C14	28.7
1,3-Dimethylnaphthalene	29.7

- 10.2 Internal standard calibration procedure-1,3,5-trichlorobenzene (TCB) has been shown to be free of interferences from diesel and crude oils and is a suitable internal standard.
- 10.3 The system performance test mix standards prepared in Section 7.2.4 are primarily used to establish retention times and establish qualitative detection limits.
- 10.3.1 Spike a 500- μ L aliquot of the 1.25 mg/mL SPTM standard with 500 μ L of the TCB internal standard solution.
- 10.3.2 Inject 1.0 μ L of this spiked SPTM standard onto the GC/MS in order to demonstrate proper retention times. For the GC/MS used in the development of this method the ten compounds in the mixture had typical retention times shown in Table 2 above. Extracted ion scans for m/z 91 and 105 showed a maximum abundance of 400,000.

- 10.3.3 Spike a 500- μ L aliquot of the 0.125 mg/mL SPTM standard with 500 μ L of the TCB internal standard solution.
- 10.3.4 Inject 1.0 mL of this spiked SPTM standard onto the GC/MS to monitor detectable levels. For the GC/MS used in the development of this test all ten compounds showed a minimum peak height of three times signal to noise. Extracted ion scans for m/z 91 and 105 showed a maximum abundance of 40,000.
- 10.4 GC/MS crude oil/drilling fluid calibration -There are two methods of quantification: Total Area Integration (C8-C13) and EIP Area Integration using m/z's 91 and 105. The EIP Area Integration method should be used as the primary method for quantifying oil in NAFs and enhanced mineral oil (EMO) based drilling fluid. Inject 1.0 μ L of each of the four crude oil/drilling fluid calibration standards prepared in Section 7.2.5 into the GC/MS. The internal standard should elute approximately 21-22 minutes after injection. For the GC/MS used in the development of this method, the internal standard peak was (35 to 40)% of full scale at an abundance of about 3.5×10^7 .
- 10.4.1 Total Area Integration Method-For each of the four calibration standards obtain the following: Using a straight baseline integration technique, obtain the total ion chromatogram (TIC) area from C8 to C13. Obtain the TIC area of the internal standard (TCB). Subtract the TCB area from the C8-C13 area to obtain the true C8-C13 area. Using the C8-C13 and TCB areas, and known internal standard concentration, generate a linear regression calibration using the internal standard method. The r^2 value for the linear regression curve should be ≥ 0.998 . Some synthetic fluids might have peaks that elute in the window and would interfere with the analysis. In this case the integration window can be shifted to other areas of scan where there are no interfering peaks from the synthetic base fluid.
- 10.4.2 EIP Area Integration-For each of the four calibration standards generate Extracted Ion Profiles (EIPs) for m/z 91 and 105. Using straight baseline integration techniques, obtain the following EIP areas:
- 10.4.2.1 For m/z 91 integrate the area under the curve from approximately 10.5 minutes to 25 minutes, including the internal standard. The internal standard area is used in the calculations.
- 10.4.2.2 For m/z 105 integrate the area under the curve from approximately 10.5 minutes to 25 minutes.
- 10.4.2.4 Using the EIP areas for TCB, m/z 91 and m/z105, and the known concentration of internal standard. Calculate the ratio of the total m/z105 area divided by the internal standard area at m/z 91. Generate linear regression calibration curves for the ratios using the internal standard method. The r^2 value for the each of the EIP linear regression curves should be ≥ 0.998 .

10.4.2.5 Some base fluids might produce a background level that would show up on the extracted ion profiles, but there should not be any real peaks (signal to noise ratio of 1:3) from the clean base fluids.

11.0 Procedure

11.1 Sample Preparation-

11.1.1 Mix the authentic field sample (drilling fluid) well. Transfer (weigh) a 30-g aliquot of the sample to a labeled centrifuge tube.

11.1.2 Centrifuge the aliquot for a minimum of 15 min at approximately 15,000 rpm, in order to obtain a solids free supernate.

11.1.3 Weigh 0.5 g of the supernate directly into a tared and appropriately labeled GC straight vial.

11.1.4 Spike the 0.5-g supernate with 500 μL of the 0.01g/mL 1,3,5-trichlorobenzene internal standard solution (see 7.2.3), dilute with methylene chloride, cap with a Teflon lined crimp cap, and vortex for ca. 10 sec.

11.1.5 The sample is ready for GC/MS analysis.

11.2 Gas Chromatography. Table 1 summarizes the recommended operating conditions for the GC/MS. Retention times for the n-alkanes obtained under these conditions are given in Table 2. Other columns, chromatographic conditions, or detectors may be used if initial precision and accuracy requirements (Section 9.2) are met. The system is calibrated according to the procedures outlined in Section 10, and verified every 12 hours according to Section 11.6.

11.2.1 Samples should be prepared (extracted) in a batch of no more than 20 samples. The batch should consist of 20 authentic samples, 1 blank (Section 9.3), 1 matrix spike sample (9.4), and 1 duplicate field sample (9.5), and a prepared sample of the corresponding clean NAF used in the drilling process.

11.2.2 An analytical sequence is run on the GC/MS where the 3 SPTM standards (Section 7.2.4) containing internal standard are analyzed first, followed by analysis of the four GC/MS crude oil/drilling fluid calibration standards (Section 7.2.5), analysis of the blank, matrix spike sample, the duplicate sample, the clean NAF sample, followed by the authentic samples.

11.2.3 Samples requiring dilution due to excessive signal should be diluted using methylene chloride.

11.2.4 Inject 1.0 μL of the test sample or standard into the GC, using the conditions in Table 1.

11.2.5 Begin data collection and the temperature program at the time of injection.

11.2.6 Obtain a TIC and EIP fingerprint scans of the sample (Table 3).

11.2.7 If the area of the C8 to C13 peaks exceeds the calibration range of the system, dilute a fresh aliquot of the test sample weighing $< 0.50\text{-g}$ and reanalyze.

11.2.8 Determine the C8 to C13 TIC area, the TCB internal standard area, and the areas for the m/z 91 and 105 EIPs. These are used in the calculation of oil concentration in the samples (see Section 12).

TABLE 3.-RECOMMENDED ION MASS NUMBERS

Selected Ion Mass Numbers	Corresponding Aromatic Compounds	Typical retention times (in minutes)
91.....	Methylbenzene.....	6.0
	Ethylbenzene.....	10.3
	1,4-Dimethylbenzene.....	10.9
	1,3-Dimethylbenzene.....	10.9
	1,2-Dimethylbenzene.....	10.9
105.....	1,3,5-Trimethylbenzene.....	15.1
	1,2,4-Trimethylbenzene.....	16.0
	1,2,3-Trimethylbenzene.....	17.4
156.....	2,6-Dimethylnaphthalene.....	28.9
	1,2-Dimethylnaphthalene.....	29.4
	1,3-Dimethylnaphthalene.....	29.7

11.2.9 Observe the presence of peaks in the EIPs that would confirm the presence of any target aromatic compounds. Using the EIP areas and EIP linear regression calibrations determine the amount of crude oil contamination equivalent in the sample.

11.3 Qualitative Identification-11.3.1 Qualitative identification is accomplished by comparison of the TIC and EIP area data from an authentic sample to the TIC and EIP area data from the calibration standards (Section 10.4). Crude oil is identified by the presence of C10 to C13 n-alkanes and corresponding target aromatics.

11.3.2 Using the calibration data, establish the identity of the C8 to C13 peaks in the chromatogram of the sample. Using the calibration data, establish the identity of any target aromatics present on the extracted ion scans.

- 11.3.3 Crude oil is not present in a detectable amount in the sample if there are no target aromatics seen on the extracted ion scans. The experience of the analyst shall weigh heavily in the determination of the presence of peaks at a signal-to-noise ratio of 3 or greater.
- 11.3.4 If the chromatogram shows n-alkanes from C8 to C13 and target aromatics to be present, contamination by crude oil or diesel should be suspected and quantitative analysis should be determined. If there are no n-alkanes present that are not seen on the blank, and no target aromatics are seen, the sample can be considered to be free of contamination.
- 11.4 Quantitative Identification-
- 11.4.1 Determine the area of the peaks from C8 to C13 as outlined in the calibration section (10.4.1). If the area of the peaks for the sample is greater than that for the clean NAF (base fluid) use the crude oil/drilling fluid calibration TIC linear regression curve to determine approximate crude oil contamination. (This step will be difficult for NAF samples that have measurable amounts of C8 to C13 peaks in the clean fluid. The EIPs should be used for quantitation of crude oil).
- 11.4.2 Using the EIPs outlined in Section 10.4.2 determine the presence of any target aromatics. Using the integration techniques outlined in Section 10.4.2 to obtain the EIP areas for m/z 91 and 105. Use the crude oil/drilling fluid calibration EIP ratio linear regression curves to determine approximate crude oil contamination.
- 11.5 Complex Samples-
- 11.5.1 The most common interferences in the determination of crude oil can be from mineral oil, diesel oil, and proprietary additives in drilling fluids.
- 11.5.2 Mineral oil can typically be identified by its lower target aromatic content, and narrow range of strong peaks.
- 11.5.3 Diesel oil can typically be identified by low amounts of n-alkanes from C7 to C9, and the absence of n-alkanes greater than C25.
- 11.5.4 Crude oils can usually be distinguished by the presence of high aromatics, increased intensities of C8 to C13 peaks, and/or the presence of higher hydrocarbons of C25 and greater (which may be difficult to see in some synthetic fluids at low contamination levels).
- 11.5.4.1 Oil condensates from gas wells are low in molecular weight and will normally produce strong chromatographic peaks in the C8-C13 range. If a sample of the gas

condensate crude oil from the formation is available, the oil can be distinguished from other potential sources of contamination by using it to prepare a calibration standard.

11.5.4.2 Asphaltene crude oils with API gravity <20 may not produce chromatographic peaks strong enough to show contamination at levels of the calibration. Extracted ion peaks should be easier to see than increased intensities for the C8 to C13 peaks. If a sample of asphaltene crude from the formation is available, a calibration standard should be prepared.

11.6 System and Laboratory Performance-

11.6.1 At the beginning of each 8-hour shift during which analyses are performed, GC crude oil/drilling fluid calibration and system performance test mixes are verified. For these tests, analysis of the medium-level calibration standard (1-% Reference Oil in IO Lab drilling fluid, and 1.25 mg/mL SPTM with internal standard) shall be used to verify all performance criteria. Adjustments and/or re-calibration (per Section 10) shall be performed until all performance criteria are met. Only after all performance criteria are met may samples and blanks be analyzed.

11.6.2 Inject 1.0 mL of the medium-level GC/MS crude oil/drilling fluid calibration standard into the GC instrument according to the procedures in Section 11.2. Verify that the linear regression curves for both TIC area and EIP areas are still valid using this continuing calibration standard.

11.6.3 After this analysis is complete, inject 1.0 mL of the 1.25 mg/mL SPTM (containing internal standard) into the GC instrument and verify the proper retention times are met (see Table 2).

11.6.4 Retention times-Retention time of the internal standard. The absolute retention time of the TCB internal standard should be within the range 21.0 ± 0.5 minutes. Relative retention times of the n-alkanes: The retention times of the n-alkanes relative to the TCB internal standard shall be similar to those given in Table 2.

12.0 Calculations

The concentration of oil in NAFs drilling fluids is computed relative to peak areas between C8 and C13 (using the Total Area Integration method) or peak areas from extracted ion profiles (using the Extracted Ion Profile Method). In either case, there is a measurable amount of peak area, even in clean drilling fluid samples, due to spurious peaks and electrometer “noise” that contributes to the total signal measured using either of the quantitation methods. In this procedure, a correction for this signal is applied, using the blank or clean sample correction technique described in American Society for Testing Materials (ASTM) Method D-3328-90, Comparison of Waterborne Oil by Gas Chromatography. In this method, the “oil equivalents” measured in a blank sample by total area gas chromatography are subtracted from that

determined for a field sample to arrive at the most accurate measure of oil residue in the authentic sample.

12.1 Total Area Integration Method

12.1.1 Using C8 to C13 TIC area, the TCB area in the clean NAF sample and the TIC linear regression curve, compute the oil equivalent concentration of the C8 to C13 retention time range in the clean NAF. Note: The actual TIC area of the C8 to C13 is equal to the C8 to C13 area minus the area of the TCB.

12.1.2 Using the corresponding information for the authentic sample, compute the oil equivalent concentration of the C8 to C13 retention time range in the authentic sample.

12.1.3 Calculate the concentration (% oil) of oil in the sample by subtracting the oil equivalent concentration (% oil) found in the clean NAF from the oil equivalent concentration (% oil) found in the authentic sample. The C8 to C13 TIC area will not work well for clean NAF samples that contain measurable amounts of paraffins in the C8 to C13 range.

12.2 EIP Area Integration Method

12.2.1 Using the ratio of the 105 EIP area to the TCB m/z 91 EIP area in the clean NAF sample, and the appropriate EIP linear regression curve, compute the oil equivalent concentration of the in the clean NAF.

12.2.2 Using the corresponding information for the authentic sample, compute its oil equivalent concentration.

12.2.3). If the ratio of the of the 105 EIP area to the TCB m/z 91 EIP area for the authentic sample is greater than that for the 1% formation oil equivalent calibration standard, the sample is considered contaminated with formation oil.

13.0 Method Performance

13.1 Specification in this method are adopted from EPA Method 1663, Differentiation of Diesel and Crude Oil by GC/FID (Reference 16.4).

13.2 Single laboratory method performance using an Internal Olefin (IO) drilling fluid fortified at 0.5% oil using a 35 API gravity oil was:

Precision and accuracy 94±4%

Accuracy interval-86.3% to 102%

Relative percent difference in duplicate analysis-6.2%

14.0 Pollution Prevention

- 14.1 The solvent used in this method poses little threat to the environment when recycled and managed properly.

15.0 Waste Management

- 15.1 It is the laboratory's responsibility to comply with all federal, state, and local regulations governing waste management, particularly the hazardous waste identification rules and land disposal restriction, and to protect the air, water, and land by minimizing and controlling all releases from fume hoods and bench operations. Compliance with all sewage discharge permits and regulations is also required.
- 15.2 All authentic samples (drilling fluids) failing the RPE (fluorescence) test (indicated by the presence of fluorescence) shall be retained and classified as contaminated samples. Treatment and ultimate fate of these samples is not outlined in this SOP.
- 15.3 For further information on waste management, consult "The Waste Management Manual for Laboratory Personnel", and "Less is Better: Laboratory Chemical Management for Waste Reduction", both available from the American Chemical Society's Department of Government Relations and Science Policy, 1155 16th Street NW, Washington, D.C. 20036.

16.0 References

- 16.1 Carcinogens-"Working With Carcinogens." Department of Health, Education, and Welfare, Public Health Service, Centers for Disease Control [available through National Technical Information Systems, 5285 Port Royal Road, Springfield, VA 22161, document no. PB-277256]: August 1977.
- 16.2 "OSHA Safety and Health Standards, General Industry [29 CFR 1910], Revised." Occupational Safety and Health Administration, OSHA 2206. Washington, DC: January 1976.
- 16.3 "Handbook of Analytical Quality Control in Water and Wastewater Laboratories." USEPA, EMSSL-CI, EPA-600/4-79-019. Cincinnati, OH: March 1979.
- 16.4 "Method 1663, Differentiation of Diesel and Crude Oil by GC/FID, Methods for the Determination of Diesel, Mineral, and Crude Oils in Offshore Oil and Gas Industry Discharges, EPA 821-R-92-008, Office of Water Engineering and Analysis Division, Washington, DC: December 1992.

109
Appendix D

Table 1: Produced Water Critical Dilutions
Table 1-A: Critical Dilution (Percent Effluent) for Discharges with a Depth Difference Between the Discharge Pipe and the Sea Floor of Greater than 0 Meters to 4 Meters

Discharge Rate (bbl/day)	Pipe Diameter (inches)					
	>0" to 5"	>5" to 7"	>7" to 9"	>9" to 11"	>11" to 15"	>15"
0 to 500	0.05	0.05	0.05	0.05	0.05	0.05
501 to 1000	0.12	0.12	0.12	0.12	0.12	0.12
1001 to 2000	0.29	0.29	0.29	0.29	0.29	0.29
2001 to 3000	0.49	0.48	0.48	0.48	0.48	0.49
3001 to 4000	0.66	0.64	0.64	0.64	0.64	0.64
4001 to 5000	0.9	0.87	0.85	0.85	0.86	0.87
5001 to 6000	1.13	1.11	1.07	1.07	1.08	1.09
6001 to 7000	1.36	1.33	1.30	1.28	1.28	1.30
7001 to 8000	1.57	1.55	1.51	1.47	1.48	1.50
8001 to 9000	1.80	1.78	1.74	1.68	1.68	1.70
9001 to 10,000	2.02	2.00	1.95	1.89	1.88	1.90
10,001 to 15,000	3.09	3.17	3.08	3.02	2.92	2.88
15,001 to 20,000	3.90	4.26	4.15	4.07	3.95	3.77
20,001 to 25,000	4.60	5.26	5.25	5.10	5.00	4.60
25,001 to 35,000	5.68	6.92	7.28	7.00	6.86	6.30
35,001 to 50,000	6.83	8.80	9.67	9.80	9.35	8.74
50,001 to 75,000	8.23	11.1	12.8	13.9	14.2	13.1

Table 1-B: Critical Dilution (Percent Effluent) for Discharges with a Depth Difference Between the Discharge Pipe and the Sea Floor of Greater than 4 Meters to 6 Meters

Discharge Rate (bbl/day)	Pipe Diameter (inches)					
	>0" to 5"	>5" to 7"	>7" to 9"	>9" to 11"	>11" to 15"	>15"
0 to 500	0.05	0.05	0.05	0.05	0.05	0.05
501 to 1000	0.104	0.105	0.105	0.105	0.105	0.106
1001 to 2000	0.20	0.20	0.20	0.20	0.20	0.20
2001 to 3000	0.30	0.30	0.30	0.30	0.30	0.30
3001 to 4000	0.40	0.38	0.38	0.38	0.39	0.39
4001 to 5000	0.59	0.58	0.56	0.56	0.57	0.58
5001 to 6000	0.74	0.73	0.71	0.71	0.71	0.72
6001 to 7000	0.88	0.87	0.85	0.84	0.84	0.85
7001 to 8000	1.02	1.00	0.98	0.96	0.96	0.97
8001 to 9000	1.15	1.14	1.12	1.09	1.09	1.10
9001 to 10,000	1.28	1.28	1.25	1.22	1.21	1.22
10,001 to 15,000	1.92	1.97	1.93	1.90	1.84	1.82
15,001 to 20,000	2.46	2.57	2.54	2.51	2.45	2.36
20,001 to 25,000	2.92	3.14	3.14	3.08	3.03	2.85
25,001 to 35,000	3.60	4.15	4.26	4.18	4.13	3.85
35,001 to 50,000	4.32	5.38	5.85	5.83	5.68	5.43
50,001 to 75,000	5.17	6.94	7.88	8.36	8.41	7.94

Table 1-C: Critical Dilution (Percent Effluent) for Discharges with a Depth Difference Between the Discharge Pipe and the Sea Floor of Greater than 6 Meters to 9 Meters

Discharge Rate (bbl/day)	Pipe Diameter (inches)					
	>0" to 5"	>5" to 7"	>7" to 9"	>9" to 11"	>11" to 15"	>15"
0 to 500	0.05	0.05	0.05	0.05	0.05	0.05
501 to 1000	0.104	0.105	0.105	0.105	0.105	0.105
1001 to 2000	0.20	0.20	0.20	0.20	0.20	0.20
2001 to 3000	0.27	0.27	0.27	0.27	0.27	0.27
3001 to 4000	0.32	0.31	0.31	0.31	0.32	0.32
4001 to 5000	0.38	0.37	0.36	0.36	0.37	0.37
5001 to 6000	0.50	0.49	0.48	0.48	0.48	0.49
6001 to 7000	0.60	0.59	0.57	0.57	0.57	0.58
7001 to 8000	0.69	0.68	0.66	0.65	0.65	0.66
8001 to 9000	0.78	0.77	0.76	0.74	0.74	0.75
9001 to 10,000	0.86	0.86	0.85	0.83	0.82	0.83
10,001 to 15,000	1.29	1.32	1.30	1.28	1.25	1.24
15,001 to 20,000	1.64	1.72	1.71	1.69	1.65	1.60
20,001 to 25,000	1.97	2.08	2.09	2.06	2.04	1.93
25,001 to 35,000	2.50	2.74	2.81	2.78	2.75	2.6
35,001 to 50,000	3.05	3.60	3.80	3.82	3.76	3.62
50,001 to 75,000	3.70	4.83	5.40	5.62	5.60	5.40

Table 1-D: Critical Dilution (Percent Effluent) for Discharges with a Depth Difference Between the Discharge Pipe and the Sea Floor of Greater than 9 Meters to 12 Meters

Discharge Rate (bbl/day)	Pipe Diameter (inches)					
	>0" to 5"	>5" to 7"	>7" to 9"	>9" to 11"	>11" to 15"	>15"
0 to 500	0.05	0.05	0.05	0.05	0.05	0.05
501 to 1000	0.104	0.105	0.105	0.105	0.105	0.106
1001 to 2000	0.20	0.20	0.20	0.20	0.20	0.20
2001 to 3000	0.27	0.27	0.27	0.27	0.27	0.27
3001 to 4000	0.32	0.31	0.31	0.31	0.32	0.32
4001 to 5000	0.38	0.37	0.36	0.36	0.37	0.37
5001 to 6000	0.50	0.49	0.36	0.36	0.37	0.37
6001 to 7000	0.55	0.54	0.53	0.52	0.52	0.53
7001 to 8000	0.59	0.59	0.58	0.56	0.57	0.57
8001 to 9000	0.64	0.63	0.62	0.60	0.61	0.61
9001 to 10,000	0.67	0.67	0.66	0.64	0.64	0.65
10,001 to 15,000	0.82	0.84	0.83	0.82	0.80	0.80
15,001 to 20,000	1.00	1.05	1.04	1.03	1.02	1.00
20,001 to 25,000	1.20	1.27	1.28	1.26	1.25	1.20
25,001 to 35,000	1.57	1.67	1.72	1.70	1.69	1.62
35,001 to 50,000	2.00	2.22	2.31	2.33	2.31	2.24
50,001 to 75,000	2.50	3.10	3.37	3.47	3.50	3.43

Table 1-E: Critical Dilution (Percent Effluent) for Lower Volume Discharges with a Depth Difference Between the Discharge Pipe and the Sea Floor of Greater than 12 Meters

Discharge Rate (bbl/day)	Pipe Diameter (inches)					
	>0" to 5"	>5" to 7"	>7" to 9"	>9" to 11"	>11" to 15"	>15"
>0 to 500	0.05	0.05	0.05	0.05	0.05	0.05
501 to 1000	0.104	0.105	0.105	0.105	0.105	0.106
1001 to 2000	0.20	0.20	0.20	0.20	0.20	0.20
2001 to 3000	0.27	0.27	0.27	0.27	0.27	0.27
3001 to 4000	0.32	0.31	0.31	0.31	0.32	0.32
4001 to 5000	0.38	0.37	0.36	0.36	0.37	0.37
5001 to 6000	0.50	0.49	0.48	0.48	0.48	0.49
6001 to 7000	0.55	0.54	0.53	0.52	0.52	0.53
7001 to 8000	0.59	0.59	0.58	0.56	0.57	0.57

Table 1-F: Critical Dilution (Percent Effluent) for Larger Volume Discharges with a Depth Difference Between the Discharge Pipe and the Sea Floor of Greater than 12 Meters

Discharge Rate (bbl/day)	Pipe Diameter (inches)					
	>0" to 5"	>5" to 7"	>7" to 9"	>9" to 11"	>11" to 15"	>15"
Depth Difference Greater than 12 Meters to 14 Meters						
8001 to 9,000	0.64	0.63	0.62	0.60	0.61	0.61
9001 to 10,000	0.67	0.67	0.66	0.64	0.64	0.65
10,001 to 15,000	0.82	0.84	0.83	0.82	0.80	0.80
15,001 to 20,000	0.92	0.96	0.96	0.95	0.93	0.91
20,001 to 25,000	1.00	1.06	1.06	1.05	1.04	1.00
25,001 to 35,000	1.13	1.20	1.23	1.22	1.22	1.17
35,001 to 50,000	1.40	1.51	1.57	1.58	1.57	1.53
50,001 to 75,000	1.83	2.15	2.27	2.34	2.37	2.33

Depth Difference Greater than 14 Meters to 16 Meters						
8001 to 9,000	0.64	0.63	0.62	0.60	0.61	0.61
9001 to 10,000	0.67	0.67	0.66	0.64	0.64	0.65
10,001 to 15,000	0.82	0.84	0.83	0.82	0.80	0.80
15,001 to 20,000	0.92	0.96	0.96	0.95	0.93	0.91
20,001 to 25,000	1.00	1.06	1.06	1.05	1.04	1.00
25,001 to 35,000	1.13	1.20	1.23	1.22	1.22	1.17
35,001 to 50,000	1.28	1.37	1.43	1.44	1.43	1.39
50,001 to 75,000	1.54	1.74	1.82	1.88	1.90	1.88
Depth Difference Greater than 16 Meters to 19 meters						
8001 to 9000	0.64	0.63	0.62	0.60	0.61	0.61
9001 to 10,000	0.67	0.67	0.66	0.64	0.64	0.65
10,001 to 15,000	0.82	0.84	0.83	0.82	0.80	0.80
15,001 to 20,000	0.92	0.96	0.96	0.95	0.93	0.91
20,001 to 25,000	1.00	1.06	1.06	1.05	1.04	1.00
25,001 to 35,000	1.13	1.20	1.23	1.22	1.22	1.17
35,001 to 50,000	1.28	1.37	1.43	1.44	1.43	1.39
50,001 to 75,000	1.30	1.44	1.51	1.56	1.57	1.55
Depth Difference Greater than 19 Meters						
8001 to 9000	0.64	0.63	0.62	0.60	0.61	0.61
9001 to 10,000	0.67	0.67	0.66	0.64	0.64	0.65
10,001 to 15,000	0.82	0.84	0.83	0.82	0.80	0.80
15,001 to 20,000	0.92	0.96	0.96	0.95	0.93	0.91
20,001 to 25,000	1.00	1.06	1.06	1.05	1.04	1.00
25,001 to 35,000	1.13	1.20	1.23	1.22	1.22	1.17
35,001 to 50,000	1.28	1.37	1.43	1.44	1.43	1.39
50,001 to 75,000	1.28	1.42	1.49	1.53	1.54	1.53

Table 1-G: Minimum Vertical Port Separation Distance to Avoid Interference

<u>Port Flow Rate (bbl/day)</u>	<u>Minimum Separation Distance (m)</u>
0 - 500	3.7
501 - 1000	4.5
1001 - 2000	5.4
2001 - 5000	6.4
5001 - 7000	6.6
7001 - 10000	6.6

Table 2-A: Critical Dilutions (Percent Effluent) for Toxicity Limitations for Seawater to which treatment chemicals have been added

Depth Difference (Meters)	Discharge Rate (bbl/day)	Pipe Diameter			
		>0" to 2"	>2" to 4"	>4" to 6"	>6"
All	0 to 1,000	12	24.7	24.5	24.6
	>1,000 to 10,000	11.2	12.4	12.2	14
	> 10,000	9.6	24	23	20

Table 2-B: Critical Dilutions (Percent Effluent) for Toxicity Limitations for freshwater to which treatment Chemicals have been Added

Depth Difference (Meters)	Discharge Rate (bbl/day)	Pipe Diameter			
		>0" to 2"	>2" to 4"	>4" to 6"	>6"
All	0 to 1,000	1.1	1.2	2.9	2.9
	>1,000 to 10,000	19	39	28	24
	>10,000	13	63	41	74

116
Appendix E

Table 1. Effluent Limitations, Prohibitions and Monitoring Requirements

Discharge	Regulated & Monitored Parameter	Discharge Limitation/ Prohibition	Measurement Frequency	Monitoring Requirement	
				Sample Type/Method	Recorded Value(s)
Drilling Fluid ...	Free Oil.....	No free oil.....	Once week(*1).....	Static sheen	Num. Of days sheen observed
	Toxicity(*2)	30,000 ppm daily min	Once/month.....	Grab.....	96-hr LC50
	96-hr LC50	30,000 ppm monthly avg min	Once/end of well(*3)	Grab.....	96-hr LC50
			Once/month.....	Grab.....	96-hr LC50
	Discharge Rate.....	1,000 barrels/hour.....	Once/hour(*1).....	Estimate.....	Max. hourly rate
	Discharge Rate for controlled rate areas	(*4).....	Once/hour(*1).....	Measure.....	Max. hourly rate
	Mercury and cadmium	No discharge. of drilling fluids to which barite has been added, if such barite contains mercury in excess of 1.0 mg/kg or cadmium in excess of 3.0 mg/kg (dry weight)	Once prior to drilling each well (*6)	Absorption Spectro-photometry	mg mercury/kg barite mg cadmium/kg barite
	Oil Based or Inverse Emulsion Drilling Fluids	No discharge			
	Oil Contaminated... Drilling Fluids	No discharge			
	Diesel Oil.....	No discharge of drilling fluids to which diesel oil has been added			
Mineral Oil.....	Mineral oil may be used only as a carrier fluid, lubricity additive, or pill				
Non aqueous Based.. Fluids	No discharge except that which adheres to drill cuttings(*5)				

Table 1. (Continued)

Discharge	Regulated & Monitored Discharge Limitation/ Parameter Prohibition		Measurement Frequency	Monitoring Requirement	
	Sample Type/Method	Recorded Value(s)			
All Drill Cuttings	Free oil.....	No free oil.....	Once/week(*1)....	Static sheen.....	Number of days sheen observed
	Toxicity(*2) 96-hr LC50..	No discharge of cuttings generated using drilling fluids which exhibit a toxicity of less than 30,000 ppm daily min. or 30,000 ppm monthly avg. min.			
	Mercury and cadmium.....	No discharge. if generated using drilling fluid to which barite is added which contains mercury in excess of 1.0 mg/kg or cadmium in excess of 3.0 mg/kg			
	Cuttings generated using Oil Contaminated Drilling Fluids	No discharge			
	Cuttings generated using drilling fluids to which Diesel Oil has been added	No discharge			
	Cuttings generated using drilling fluids to which Mineral Oil has been added	Mineral oil may be used only as a carrier fluid, lubricity additive, or pill			

Table 1. (Continued)

Discharge	Regulated & Monitored Parameter	Discharge Limitation/ Prohibition	Measurement Frequency	Monitoring Requirement	
				Sample Type/Method	Recorded Value(s)
Stock Limits for Drill Cuttings Generated using Non aqueous Based Drilling Fluids	Polynuclear Aromatic Hydrocarbons (PAH)	0.00001 grams PAH per gram of base fluid	Once/year on each base fluid blend	PAH content of Oil by HPLC/UV, EPA Method 1654 (see 40 CFR 435.11(u))	gram PAH / gram stock base fluid
	Sediment Toxicity....	Ratio of 10-day LC50s not to exceed 1.0(*7)	Once/year on each base fluid blend	ASTM method E1367-99 (*8)	Ratio of C16-C18 IO LC50 to stock base fluid LC50
	Biodegradation Rate...	Biodegradation rate ratio... not to exceed 1.0 (*9)	Once/year on each base fluid blend	Modified ISO 11734:1995 (*10)	Ratio of C16-C18 IO biodeg. to stock base fluid biodeg.
Discharge Limits for Cuttings Generated using Non aqueous Based Drilling Fluids	Sediment Toxicity....	Ratio of 4-day LC50s not to exceed 1.0(*11)	Once/month.....	Modified ASTM Method E1367-99 (*12)	Ratio of C16-C18 IO LC50 to stock base fluid LC50
	Formation Oil.....	No Discharge.....	Once prior to drilling	GCMS (*13)	
			Once/week.....	RPE (*14)	
	Base Fluids Retained on Cuttings	6.9% IO (*15) 9.4% ester (*16)	Once/day (*17)...	Retort Test Method (*18)	Percent retained

Table 1. (Continued)

Discharge	Regulated & Monitored Parameter	Discharge Limitation/ Prohibition	Measurement Frequency	Monitoring Requirement	
				Sample Type/Method	Recorded Value(s)
Deck Drainage.....	Free Oil.....	No free oil.....	Once/day(*19).....	Visual sheen...	Number of days sheen observed
Produced Water.....	Oil and grease.....	42 mg/l daily max., 29 mg/l monthly avg.	Once/month.....	Grab(*20).....	Daily max., monthly average
	Toxicity.....	7-day min NOEC(*21) and monthly avg min NOEC(*21)	Rate Dependent(*28)	Grab.....	Lowest NOEC for either species
	Free Oil.....	Monitor.....	Once/day (*19,*29)	Visual sheen....	Number of days sheen observed
	Flow (bbl/day).....	Monitor.....	Once/month.....	Estimate.....	Monthly Average
Produced Sand.....	No Discharge				
Well treatment fluids, completion fluids, and workover fluids (includes packer fluids)(*22)	Free oil.....	No free oil.....	Once/day(*1).....	Static sheen....	Number of days sheen observed
	Oil & Grease.....	42 mg/l daily max., 29 mg/l monthly avg.	Once/month.....	Grab(*20).....	Daily max., monthly average
Sanitary waste(*24) continuously manned for 30 or more days by 10 or more persons	Residual.....	1 mg/l (minimum).....	Once/month.....	Grab.....	Concentration
	Solids.....	No Floating Solids...	Once/day.....	Observation(*27)	Number of days solids observed
Sanitary waste (*24) continuously manned for thirty or more days by 9 or fewer persons or intermittently by any number	Solids.....	No floating solids...	Once/day.....	Observation(*27)	Number of days solids observed
Domestic waste(*26).....	Solids.....	No floating solids or foam	Once/day.....	Observation(*27)	Number of days observed

Table 1. (Continued)

Discharge	Regulated & <u>Monitored</u> Parameter	Discharge Limitation/ Prohibition	Measurement Frequency	Monitoring Requirement	
				Sample Type/Method	Recorded Value(s)
Miscellaneous discharges: Desalinization unit discharge; blowout pre-venter fluid; uncontaminated ballast water; uncontaminated bilge water; uncontaminated freshwater; mud, cuttings and cement at seafloor; uncontaminated seawater; boiler blowdown; source water and sand; diatomaceous earth filter media; excess cement slurry; sub sea wellhead preservation fluids; sub sea production control fluid umbilical steel tube storage; fluid; leak tracer fluid; riser tensioner fluids	Free oil.....	No free oil.....	Once/week(*23)	Visual sheen..	Number of days sheen observed
Miscellaneous discharges of seawater and freshwater to which treatment chemicals have been added: excess seawater which permits the continuous operation of fire control and utility lift pumps, excess seawater from pressure maintenance and secondary recovery projects, water released during training of personnel in fire protection, seawater used to pressure test new and existing piping and pipelines, ballast water, once- through non-contact cooling water	Treatment chemicals	Most stringent of: EPA label registration, maximum manufacturers recommended dose, or 500 mg/l.			
	Discharge Rate.....	Monitor.....	Once/month....	Estimate.....	Monthly average
	Free oil.....	No free oil.....	1/week.....	Visual Sheen.. (*32)	Number of days sheen observed
	Toxicity.....	48-hour average min. NOEC and monthly avg minimum NOEC (*30)	Rate Dependent (*31)	Grab.....	Lowest NOEC observed for either of the two species

Footnotes

- *1 When discharging.
 - *2 Suspended particulate phase (SPP) with *Mysidopsis bahia* following approved test method. The sample shall be taken beneath the shale shaker; or if there are no returns across the shaker then the sample must be taken from a location that is characteristic of the overall mud system to be discharged.
 - *3 Sample shall be taken after the final log run is completed and prior to bulk discharge.
 - *4 See Part I.B.1.b of this permit.
 - *5 See Part I.B.1.a of this permit.
 - *6 Analyses shall be conducted on each new stock of barite used.
 - *7 The ratio of the 10-day LC50 of C16 - C18 internal olefin divided by the 10-day LC50 of the base fluid shall not exceed 1.0. See Part I.B.2.c.1 of this permit.
 - *8 See Part I.D.8.
 - *9 The ratio of the cumulative gas production (ml) of C16 - C18 internal olefin divided by the cumulative gas production (ml) of stock base fluid, both at 275 days, shall not exceed 1.0. See Part I.B.2.c.1 of this permit.
 - *10 See Part I.D.11 of this permit.
 - *11 The ratio of the 4-day LC50 of C16 - C18 internal olefin divided by the 4-day LC50 of the base fluid shall not exceed 1.0. See Part I.B.2.c.2 of this permit.
 - *12 See Appendix A of this permit.
 - *13 See Appendix 5 of 40 CFR Part 435, Subpart A and Part I.D.11 and Appendix C of this permit.
 - *14 See Section I.D.12 of this permit.
 - *15 Drilling fluids which meet the stock base fluid limitations for C16-C18 internal olefins.
 - *16 Drilling fluids which meet the stock limitations for C12-C14 ester or C8 ester.
 - *17 Except when meeting the conditions for the Best Management Practices described in Part I.B.2.c of this permit. Operators conducting fast drilling shall collect and analyze samples once per 500 feet or a maximum of three per day.
 - *18 See Part I.D.13 of this permit.
 - *19 When discharging and facility is manned. Monitoring shall be accomplished during times when observation of a visual sheen on the surface of the receiving water is possible in the vicinity of the discharge._
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- *20 May be based on either a grab sample or a composite which consists of the arithmetic average of the results of grab samples collected at even intervals during a period of 24-hours or less.
 - *21 See Appendix D, Table 1 of this permit.
 - *22 No discharge of priority pollutants except in trace amounts. Information on the specific chemical composition shall be recorded but not reported unless requested by EPA.
 - *23 When discharging for muds, cuttings, and cement at the seafloor, blowout preventer fluid, sub sea wellhead preservation fluids, subsea production control fluid, umbilical steel tube storage fluid, leak tracer fluid, and riser tensioner fluids. All other miscellaneous discharges: when discharging, discharge is authorized only during times when visual sheen observation is possible, unless the static sheen method is used. Uncontaminated seawater uncontaminated freshwater, source water and source sand, uncontaminated bilge water, and uncontaminated ballast water from platforms on automatic purge systems may be discharged without monitoring from platforms which are not manned.
 - *24 Any facility which properly operates and maintains a marine sanitation device (MSD) that complies with pollution control standards and regulations under section 312 of the Act shall be deemed to be in compliance with permit limitations for sanitary waste. The MSD shall be tested yearly for proper operation, and test results maintained at the facility.
 - *25 Hach method CN-66 DPD approved. Minimum of 1 mg/l and maintained as close to this concentration as possible.
 - *26 The discharge of food waste is prohibited within 12 nautical miles from nearest land. Comminuted food waste able to pass through a 25 mm mesh screen (approximately 1 inch) may be discharged more than 12 nautical miles from nearest land.
 - *27 Monitoring shall be accomplished during daylight by visual observation of the surface of the receiving water in the vicinity of sanitary and domestic waste outfalls. Observations shall be made following either the morning or midday meals at a time of maximum estimated discharge.
 - *28 Once per annual DMR monitoring period for discharges from 0 bbl/day to 4599 bbl/day, once/calendar quarter for discharges of 4,600 bbl/day and greater.
 - *29 See Part I.B.4.b. of this permit.
 - *30 See Appendix D, Table 2 of this permit
 - *31 See Part I.B.11.b of this permit.
 - *32 Monitoring for free oil on discharges from existing piping and existing pipelines shall be performed at least three times per discharge as follows: 1) within thirty minutes after commencement of discharge; 2) at the estimated middle of the discharge; and 3) within fifteen minutes before or after the discharge has ceased.
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