Permit No.: AKG-31-5000

United States Environmental Protection Agency
Region 10
1200 Sixth Avenue
Seattle, Washington 98101

AUTHORIZATION TO DISCHARGE UNDER THE NATIONAL POLLUTANT DISCHARGE ELIMINATION SYSTEM (NPDES) FOR OIL AND GAS EXTRACTION FACILITIES IN FEDERAL AND STATE WATERS IN COOK INLET

In compliance with the provisions of the Clean Water Act, 33 U.S.C. §1251 *et seq.*, as amended by the Water Quality Act of 1987, P.L. 100-4, the "Act", the following discharges:

Discharge Number	Discharge Description
001	Drilling Fluids and Drill Cuttings
002	Deck Drainage
003	Sanitary Wastes
004	Domestic Wastes
005	Desalination Unit Wastes
006	Blowout Preventer Fluid
007	Boiler Blowdown
008	Fire Control System Test Water
009	Non-Contact Cooling Water
010	Uncontaminated Ballast Water
011	Bilge Water
012	Excess Cement Slurry
013	Mud, Cuttings, Cement at Seafloor
014	Waterflooding Discharges
015	Produced Water and Produced Sand
016	Completion Fluids
017	Workover Fluids
018	Well Treatment Fluids
019	Test Fluids

are authorized from Offshore and Coastal Subcategories of the Oil and Gas Extraction Point Source Category (40 CFR Part 435, Subparts A and D) to the Cook Inlet in the Area of Coverage as defined in (see Section I.B) and in accordance with the effluent limitations, monitoring requirements, prohibitions and other conditions set forth herein.

This permit applies only to those facilities that have been authorized in accordance with the procedures described in Part I of this permit.

This permit is issued on June 14, 2007

This permit shall become effective on July 2, 2007

This permit and the authorization to discharge shall expire at midnight, on July 2, 2012

The permittee shall reapply for a permit in accordance with Section I.D.3. prior to the expiration date of this permit if the permittee intends to continue operations and discharges at the facility beyond the term of this permit.

Signed this 25th day of MAY, 2007

Michael F. Gearheard
Director
Office of Water & Watersheds, Region 10
U.S. Environmental Protection Agency

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I. APPLICABILITY AND NOTIFICATION REQUIREMENTS

A. Sources. This general permit authorizes discharges from oil and gas extraction facilities engaged in exploration, development and production activities under the Offshore and Coastal Subcategories of the Oil and Gas Extraction Point Source Category (40 CFR Part 435, Subparts A and D). This general permit limits discharges from the following facilities:

1. New sources, as defined in Appendix A of this general permit, are not authorized to discharge produced water, drilling fluids, or drill cuttings under this general permit; and,

2. New exploratory facilities (*i.e.*, exploratory facilities that were not authorized to discharged under the expired general permit) are not authorized to discharge produced water under this general permit.

B. Area of Coverage. This general permit covers Cook Inlet, north of the northern edge of Shuyak Island, with the exception of the prohibited areas of discharge set forth in Section I.C., below. The area of coverage is shown in Figure 1.

C. Prohibited Areas of Discharge. Permit applicants should contact EPA if they are uncertain whether their discharges will be located in a prohibited area. Discharges from operations in Cook Inlet are prohibited in the following areas:

1. <u>10 Meter Isobath</u>. Exploratory facilities are prohibited from discharging shoreward of the 10 meter mean lower low water isobath.

2. <u>5 Meter Isobath</u>. All facilities are prohibited from discharging shoreward of the 5 meter mean lower low isobath which includes intertidal areas.

3. <u>Geographic Restrictions</u>. All facilities are prohibited from discharging in the following areas:

   a. Shoreward of the 5.5 meter isobath adjacent to either (1) the Clam Gulch Critical Habitat Area (Sales 32, 40, 46A, and 49) or (2) from the Crescent River northward to a point one-half mile north of Redoubt Point (Sales 35 and 49).

   b. Within the boundaries or within 4,000 meters of a coastal marsh, river delta, river mouth, designated Area Meriting Special Attention (AMSA), State Game Refuge (SGR), State Game Sanctuary (SGS), Critical Habitat Area (CHA), or National Park.

The seaward edge of a coastal marsh is defined as the seaward edge of emergent wetland vegetation.

The following AMSAs, SGRs, SGSs, CHAs, and National Park are located in the area covered by this permit:

Palmer Hay Flats SGR	Trading Bay SGR	Goose Bay SGR
Kalgin Island CHA	Potter Point SGR	Clam Gulch CHA
Susitna Flats SGR	Kachemak Bay CHA	McNeil River SGS
Anchorage Coastal Wildlife Refuge	Lake Clark National Park	Redoubt Bay CHA
Port Graham/Nanwalek AMSA		

The legal descriptions of state specialty areas are found in Alaska Statute § 16.20 The present boundaries of these state special areas are described in "State of Alaska Game Refuges, Critical Habitat Areas, and Game Sanctuaries," Alaska Department of Fish and Game, Habitat Division, March 1991. Further information can be obtained from the Alaska Department of Natural Resources, Office of Habitat Management and Permitting, 550 West 7th Avenue, Suite 1420, Anchorage, Alaska 99501; phone (907) 269-8690.

c. In Kamishak Bay, west of line from Cape Douglas to Chinitna point.

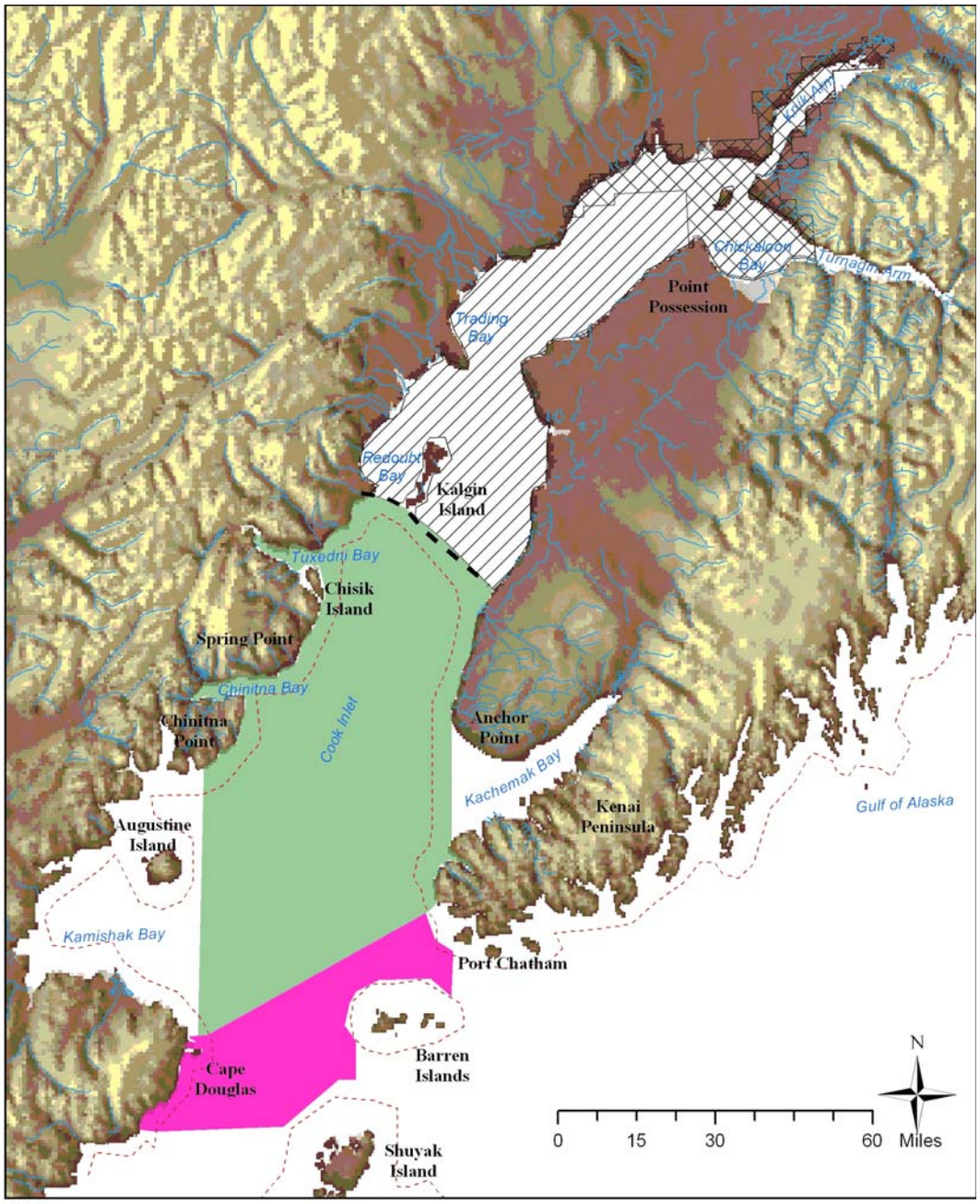
d. In Chinitna Bay, inside of the line between the points on the shoreline at latitude 59°52'45" N, longitude 152°48'18" W on the north and latitude 59°46'12" N, longitude 153°00'24" W on the south (Figure 1).

e. In Tuxedni Bay, inside of the lines on either side of Chisik Island (Figure 1).

   i. From latitude 60°04'06" N, longitude 152°34'12" W on the mainland to the southern tip of Chisik Island (latitude 60°05'45" N, longitude 152°33'30" W).

   ii. From the point on the mainland at latitude 60°13'45" N, longitude 152°32'42" W to the point on the north side of Snug Harbor on Chisik Island (latitude 60°06'36" N, longitude 152°32'54" W).

f. Minerals Management Service (MMS) Lower Kenai Peninsula deferral area and Barren Island Deferral area, including the area between the deferral areas and the shore.



Surface Access Restrictions
Boundary between Coastal Waters and Territorial Seas
Boundary between Territorial Seas and Federal Waters
General permit will authorize discharges from exploration facilities and new source development and production facilities
General permit will authorize discharges from exploration facilities and development and production facilities
Proposed New Area of Coverage for which the general permit will authorize discharges from exploration facilities and new source development and production facilities.

Figure 1: Area of Coverage

g. In Shelikof Strait, south of a line between Cape Douglas on the west (latitude 58°51' N, 153°15' W) and the northenmost tip of Shuyak Island on the east (latitude 58°37' N, 152°22' W).

h. Within 20 nautical miles of Sugarloaf Island as measured from a centerpoint at latitude 58°53' N, longitude 152°02' W.

i. Within tracts identified in the Alaska Department of Natural Resources, Oil and Gas Division's Mitigation Measure Number 33.

D. Authorization to Discharge

1. **New Facilities in Federal Waters and Territorial Seas.** New Facilities in Federal Waters and Territorial Seas who seek coverage under this general permit shall submit a Notice of Intent (NOI) to be covered by this general permit for each facility from which discharges will occur. The completed NOI must be submitted to the Director, at the address provided in Section I.G., below, at least 30 days prior to initiation of the discharges. Submittal of a NPDES permit application constitutes submittal of an NOI.

2. **New Facilities in Coastal Waters.** New Facilities in Coastal Waters who seek coverage under this general permit shall submit an NOI to be covered by this general permit for each facility from which discharges will occur. Applicants may request a 100-meter mixing zone for sanitary wastewater discharges from ADEC by submitting the NOI with a completed mixing zone request to the ADEC Division of Water at the address provided in Section I.G.3.

3. A complete NOI must contain the following information. A sample NOI form is included in Appendix E.

   a. The name, mailing address, contact name, and telephone number for the operator and facility

   b. The name of the lessor (*i.e.*, MMS or Alaska Department of Natural Resources (ADNR));

   c. The lease and block numbers of operations and discharges;

   d. The latitude and longitude and GIS coordinates of the facility;

   e. The range of water depths below mean lower low water (MLLW) in the lease block and the water depth for each discharge;

f. The type of drilling rig used (*i.e.*, jackup, drillship, semisubmersible, etc.);

g. The initial date and expected duration of operations;

h. Copies of any exploration plans, biological surveys, and environmental reports required by MMS for the identification or protection of biological populations or habitats;

i. The types of discharges from the facility;

j. The type of sanitary discharge that will occur, if any (*i.e.*, M10 or M9IM);

k. And a line drawing showing the flow of waste streams from the facility.

The NOI shall be signed in accordance with Section VIII.E.

4. Applicants will be authorized to discharge as of the date of written notification by EPA that the discharge is authorized and a permit number under this general permit has been assigned. Applicants who have requested a sanitary wastewater discharge mixing zone will be authorized to discharge only after receiving a mixing zone authorization from ADEC, which would include the State's mixing zone determination and public notice by ADEC (18 AAC 70.240). For existing dischargers who have previously applied for coverage under this reissued permit, discharge will be authorized beginning on the effective date of this permit. This permit only authorizes the discharge of pollutants from waste streams that have been clearly identified in the NOI.

5. Mobile facilities may operate in an area, rather than at a specific location, only if the applicant requests this type of discharge in their NOI, provides a map and description of the area of coverage, and the latitude and longitude of the initial location of the facility. The permittee must notify EPA, in writing, 7 days prior to moving the facility and must provide the latitude and longitude of the new location.

6. Operators of new exploratory facilities discharging drilling muds and/or cuttings shall submit a plan of study for environmental monitoring to EPA for review with, or prior to, submission of an NOI. (See Section II.B.5)

E. Transfers

1. Authorization under this general permit is not transferable to any person except after notice to the Director.

2. Transfers under this general permit will only be authorized for an existing facility located at the site or area of the original NOI. If a different facility is built at or moved to an existing location authorized by the general permit, or if a currently authorized facility is moved to a location that was not previously authorized by the general permit, then permit authorization cannot be transferred because the facility will be considered a "new facility" and the discharger must submit a new NOI for coverage under this general permit.

F. Termination Notification. The permittee must notify EPA, in writing, within 30 days following cessation of discharges from the discharge site. In the notice of termination, the permittee must certify that it is not subject to any pending enforcement actions under this NPDES permit, AKG-31-5000, including citizen suits brought under State or Federal laws. The notice of termination shall be signed in accordance with the Signatory Requirements of Section VIII.E of this general permit. This will terminate permit coverage at the site or within the mobile area. The notification may be provided in a discharge monitoring report (DMR) or under separate cover. In addition, the permittee is required to submit the final DMRs within thirty days after cessation of discharges.

G. Submission of Information

1. The discharger must submit legible originals of all NOIs and termination notices to EPA at the following address:

   Director, Office of Water & Watersheds
   United States Environmental Protection Agency, Region 10
   1200 Sixth Avenue, OWW-130
   Seattle, Washington 98101

2. The discharger must submit legible originals of all monitoring reports, other reports required by this permit, and notice of noncompliance to EPA at the following address:

   Director, Office of Compliance and Enforcement
   United States Environmental Protection Agency, Region 10
   1200 Sixth Avenue, OCE-133
   Seattle, Washington 98101

3. For discharges to state waters, the discharger must submit a copy of the information in Sections I.G.1 and I.G.2 to the Alaska Department of Environmental Conservation (ADEC) at the following address:

   Alaska Department of Environmental Conservation (ADEC)
   Attn: Division of Water
   555 Cordova Street
   Anchorage, Alaska 99501

H. Requirements for an Individual NPDES Permit

1. The Director may require any permittee to apply for and obtain an individual NPDES permit when any of the following conditions exists:

   a. The discharger is not in compliance with the conditions of this general permit;

   b. A change has occurred in the availability of demonstrated technology or practices for the control or abatement of pollutants applicable to the point source;

   c. Circumstances have changed since the time of the request to be covered so that the discharger is no longer appropriately controlled under this general permit; or

   d. The discharge(s) is a significant contributor of pollutants. In determining whether a discharge is a significant contributor of pollutants, the Directors will consider the factors set forth in 40 CFR § 122.28(b)(3)(i)(G).

2. The Director may require any owner or operator authorized by this general permit to apply for an individual NPDES permit only if the permittee has been notified in writing that an individual NPDES permit application is required.

3. Any permittee authorized by this general permit may request to be excluded from coverage under the general permit by applying for an individual NPDES permit. The permittee shall submit an individual NPDES permit application with reasons supporting the request to the Director no later than 90 days after the publication by EPA of this general permit in the Federal Register.

The permittee's coverage under this general permit will automatically terminate on the effective date of the individual permit.

4. A facility that is excluded from this general permit solely because it is subject to an individual NPDES permit may request the Director to revoke the individual NPDES permit so that it can obtain coverage under this general permit. Upon revocation of the individual NPDES permit, the general permit shall apply to the facility.

II. LIMITATIONS AND MONITORING REQUIREMENTS

A. Requirements for All Discharges

1. During the effective period of this permit, the permittee is authorized to discharge pollutants within the area of coverage set forth in Sections I.B and I.C of this general permit, in accordance with the limits and conditions set forth herein.

2. This permit authorizes the discharge of only those pollutants resulting from facility processes, waste streams, and operations that have been clearly identified in the NOI.

3. The permittee must collect all effluent samples from the effluent stream of each discharge after the last treatment unit prior to discharge into the receiving waters, except as otherwise required by discharge-specific sections of this permit.

4. The permittee must comply with the effluent limits in this general permit at all times unless otherwise indicated, regardless of the frequency of monitoring or reporting required by other provisions of this permit.

5. Unless specifically addressed in this general permit, the permittee shall not discharge floating solids, debris, sludge, deposits, foam, scum, or other residues of any kind.

6. The permittee must minimize the discharge of surfactants, dispersants, and detergents except as necessary to comply with the safety requirements of the Occupational Health and Safety Administration and MMS. This restriction applies to tank cleaning and other operations that do not directly involve the safety of workers. The discharge of dispersants to marine waters in response to oil or other hazardous waste spills is not authorized by this permit. The permittee must report all discharges of

surfactants, dispersants, and detergents in accordance with Section VI.B of this permit.

7. The permittee must separate area drains for washdown and rainfall that may be contaminated with oil and grease from those area drains that would not be contaminated so that the waste streams are not commingled. Deck drainage that is contaminated with oil and grease must be processed through an oil-water separator prior to discharge.

8. The permittee is not required to conduct monitoring for the facility when it is not staffed. The permittee must provide EPA and ADEC written notification that the facility is no longer staffed 30 days prior to terminating monitoring requirements.

9. The permittee shall not discharge diesel oil, halogenated phenol compounds, trisodium nitrilotriacetic acid, sodium chromate, or sodium dichromate.

10. If any discharges are commingled, the most stringent effluent limitations for each individual discharge shall be applied to the resulting discharge. If the individual discharge is not authorized, the commingled discharge is not authorized. Monitoring for compliance with technology based limits, such as the oil and grease concentration of produced water must be accomplished prior to commingling.

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

12. The discharge of maintenance waste such as removed paint and materials associated with surface preparation and coating applications is prohibited. Such materials shall be contained to the maximum extent practicable using vacuum abrasive blasting, covering grated areas with plywood, surrounding the area with canvas tarps and similar measures 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 develop and implement a Best Management Practices (BMP) plan for the containment of waste materials.

B. Requirements for Drilling Fluids and Drill Cuttings (Discharge 001)

1. Effluent Limitations and Monitoring Requirements.

a. The discharge of drilling fluids and drill cuttings is only authorized at exploratory facilities and existing facilities. The discharge of drilling fluids and drill cuttings is not authorized by this general permit at New Source facilities, as defined in Appendix A of this general permit.

b. The discharge of non-aqueous based drilling fluids is prohibited except for situations where such fluids adhere to drill cuttings at facilities located in the Territorial Seas and Federal Waters, as defined in Appendix A of this general permit.

c. The permittee must comply with the following effluent limitations and monitoring requirements:

Table 1. Effluent Limitations and Monitoring Requirements for Drilling Fluids and Drill Cuttings (Discharge 001)

Discharge	Pollutant Parameter	Effluent Limitations	Monitoring Requirements	
		Average Monthly and Maximum Daily Limits	Measurement Frequency	Sample Type
Water-based fluids and cuttings	Suspended Particulate Phase toxicity [note 1]	Minimum 96-hour LC_{50} of 30,000 ppm	Monthly and End-of-Well [note 16]	Grab
	Free oil	No discharge [note 3]	Daily	Grab
	Diesel oil [note 15]	No discharge	Daily	Grab
	Mercury	1 mg/kg [note 4]	Once per well	Grab
	Cadmium	3 mg/kg [note 4]	Once per well	Grab
	Total Volume (mgd) [note 2]	Report	Monthly	Estimate
	Depth Dependent Discharge Rate 0 to 5 meters >5 to 20 meters >20 to 40 meters >40 meters	 No discharge 500 bbl/hr 750 bbl/hr 1,000 bbl/hr	Continuous during discharge	Estimate
Non-aqueous fluids	Drilling fluids	No discharge	Daily	Observation
Non-aqueous stock base fluid (C_{16}-C_{18} internal olefin, C_{12}-C_{14} ester or C_8 ester)	Mercury	1 mg/kg [note 4]	Annual	Grab
	Cadmium	3 mg/kg [note 4]	Annual	Grab
	PAH [note 5]	mass ratio [note 6] $< 1x10^{-5}$	Annual	Grab
	Sediment toxicity	ratio [note 7] < 1.0	Annual	Grab
	Biodegradation rate	ratio [note 8] < 1.0	Annual	Grab
	Total Volume (mgd)	Report	Monthly	Estimate

Table 1. Effluent Limitations and Monitoring Requirements for Drilling Fluids and Drilling Cuttings (Discharge 001)

Discharge	Pollutant Parameter	Effluent Limitation	Monitoring Requirements	
		Maximum Daily	Measurement Frequency	Sample Type
Non-Aqueous Drilling Fluids which adhere to drill cuttings (Offshore Subcategory Only)	Free Oil	No discharge [note 3]	Daily	Grab
	Diesel oil [note 15]	No discharge	Daily	Grab
	SPP toxicity [note 1]	Minimum 96-hour LC_{50} of 30,000 ppm	Monthly	Grab
	Sediment toxicity	Drilling fluid sediment toxicity ratio [note 9] < 1.0	Annual	Grab
	Formation oil	No discharge [note 10]	Daily	Grab
	Base fluid retained on drill cuttings (C_{16}-C_{18} internal olefin stock [note 11])	6.9 g NAF base fluid/100 g wet drill cuttings [note 12]	Daily [note 14]	Grab
	Base fluid retained on drill cuttings [note 13] (C_{12}-C_{14} ester or C_8 ester stock)	9.4 g NAF base Fluid/100 g wet drill cuttings [note 12]	Daily [note 14]	Grab
	Total Volume (mgd)	Report	Monthly	Estimate

Footnotes:

1 As determined by the 96-hour suspended particulate phase (SPP) toxicity test. See 40 CFR Part 435, Subpart A, Appendix 2.

2 Report total volumes for all types of operations (exploratory, production and development). See Section II.B.4.b of this permit for end-of-well requirements..

3 As determined by the Static Sheen Test. See 40 CFR Part 435, Subpart A, Appendix 1.

4 Dry weight in the stock barite. Analysis shall be conducted using EPA Methods 245.5 or 7471. The permittee shall analyze a representative sample of stock barite once prior to drilling each well and submit the results with the DMR for the month in which drilling operations commence for the respective well. If the permittee uses the same supply of stock barite to drill subsequent wells, the permittee may submit the same analysis for those subsequent wells. (See Section II.B.4.f)

5 Polynuclear Aromatic Hydrocarbons.

6 PAH mass ratio = [mass (g) of PAH (as phenanthrene)] ÷ [mass (g) of stock base fluid] as determined by EPA method 1654, Revision A, entitled "PAH Content of Oil by HPLC/UV," December 1992. See Section III.D of this permit.

7 Base fluid sediment toxicity ratio = [10-day LC_{50} of C_{16}-C_{18} internal olefin, C_{12}-C_{14} ester or C_8 ester] ÷ [10-day LC_{50} of stock base fluid] as determined by ASTM E 1367-92 method: "Standard Guide for Conducting 10-day Static Sediment Toxicity Tests with Marine and Estuarine Amphipods,"1992, after preparing the sediment according to the method specified at 40 CFR Part 435, Subpart A, Appendix 3. See Section III.B of his permit. Results of up to 3 tests may be averaged to determine compliance, using 2 samples from the same lot of stock fluids. Equivalent aliquots of one homogenized sample must be split by laboratory (parts 1A and 1B) and tested separately if averaging is used. Permittees may show compliance based on test results from part 1A or from the rounded arithmetic average of the test results from part 1A and 1B. Permittees may also test the second sample for compliance. Where the second sample is analyzed, operators will determine compliance using the arithmetic average of the results from all 3 tests. Permittees shall report the appropriate number on the DMR. With the DMR, the permittee must submit documentation showing how the number was calculated and all applicable test reports.

Table 1. Effluent Limitations and Monitoring Requirements for Drilling Fluids and Drilling Cuttings (Discharge 001)

Footnotes (cont.)

8 Biodegradation rate ratio = [cumulative gas production (ml) of C_{16}-C_{18} internal olefin, C_{12}-C_{14} ester or C_8 ester] ÷ [cumulative gas production (ml) of stock base fluid], both at 275 days as determined by 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)" as modified for the marine environment. See Section III.C of this permit. Results of up to 3 tests may be averaged to determine compliance, using 2 samples from the same lot of stock fluids. Equivalent aliquots of one homogenized sample must be split by laboratory (parts 1A and 1B) and tested separately if averaging is used. Permittees may show compliance based on test results from part 1A or from the rounded arithmetic average of the test results from part 1A and 1B. Permittees may also test the second sample for compliance. Where the second sample is analyzed, operators will determine compliance using the arithmetic average of the results from all 3 tests. Permittees shall report the appropriate number on the DMR. With the DMR, the permittee must submit documentation showing how the number was calculated and all applicable test reports.

9 Drilling fluid sediment toxicity ratio = [4-day LC_{50} of C_{16}-C_{18} internal olefin] ÷ [4-day LC_{50} of drilling fluid removed from drill cuttings at the solids control equipment] as determined by ASTM E 1367-92 method: "Standard Guide for Conducting 10-day Static Sediment Toxicity Tests with Marine and Estuarine Amphipods,"1992, after preparing the sediment according to the method specified in Appendix B of this permit. Results of up to 3 tests may be averaged to determine compliance, using 2 grab samples collected no more than 15 minutes apart. Equivalent aliquots of the first, homogenized sample must be split by the laboratory (parts 1A and 1B) and tested separately if averaging is used. Permittees may show compliance based on test results from part 1A or from the rounded arithmetic average of the test results from parts 1A and 1B. Permittees may also test the second sample for compliance with this limit. Where the second sample is analyzed, operators will determine compliance using the arithmetic average of the results from all 3 tests. Permittees shall report the appropriate number on the DMR. With the DMR, the permittee must submit documentation showing how the number was calculated and all applicable test reports.

10 As determined before drilling fluids are shipped offshore by the GC/MS compliance assurance method (see Section III.E of this permit), and as determined prior to discharge by the Reverse Phase Extraction (RPE) method (see Section III.F of this permit) applied to drilling fluid removed from drill cuttings. If the operator wishes to confirm the results of the RPE method, the operator may use the GC/MS compliance assurance method (Section III.E of this permit). Results from the GC/MS compliance assurance method shall supercede the results of the RPE method.

11 This limitation is applicable only when the NAF base fluid meets the stock limitations defined in this table.

12 As determined by the American Petroleum Institute (API) retort method. See Section III.G of this permit.

13 Averaged over all well sections

14 Monitoring shall be performed at least once per day when generating new cuttings. 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.

15 See Section II.B.4.c

16 At the end-of-well, a sample must be collected for toxicity testing where no mineral oil is used. This sample can also serve as the monthly monitoring sample.

2. The permittee is limited to drilling discharges from no more than five wells at a single drilling site from exploratory facilities. If a step-out or sidetracked well is drilled from a previously drilled well hole, the step-out well is considered a new well. Requests to discharge from more than five wells per site will be considered by the Director on a case-by-case basis. The permittee may only discharge from more than five wells upon approval by EPA. The permittee must submit the following information to EPA for consideration for approval of the discharge from additional wells:

    a. Number of additional wells;

    b. Technical analysis of additional impacts to the receiving waters;

    c. Drilling fluid category and group for each well; and

    d. Well information for each additional well, including well name, number, latitude, longitude, beginning drill date, and hole diameter.

3. Mineral Oil Pills

    a. The discharge of residual amounts of mineral oil pills (mineral oil plus additives) is authorized by this general permit provided that the mineral oil pill and at least a 50 bbl buffer of drilling fluid on either side of the pill are removed from the circulating drilling fluid system and not discharged to waters of the United States. If more than one pill is applied to a single well, the previous pill and buffer must be removed prior to application of a subsequent pill.

    b. Residual mineral oil concentration in the discharged mud must not exceed 2% volume/volume (API Recommended Practice 13-1, 1990). The permittee must report the following information within 60 days of the discharge if drilling mud containing residual mineral oil pill (after pill and buffer removal) is discharged:

        i. dates of pill application, recovery, and discharge;

        ii. results of the Drilling Fluids Toxicity Test on samples of the mud before each pill is added and after removal of each pill and buffer (taken when residual mineral oil pill concentration is expected to be greatest);

iii. name of spotting compound and mineral oil product used;

iv. volumes of spotting compound, mineral oil, water, and barite in the pill;

v. total volume of mud circulating prior to pill application, volume of pill formulated, and volume of pill circulated;

vi. volume of pill recovered, volume of mud buffer recovered, and volume of mud circulating after pill and buffer recovery;

vii. percent recovery of the pill (include calculations);

viii. estimated concentrations of residual spotting compound and mineral oil in the sample of mud discharged, as determined from amounts added and total mud volume circulating prior to pill application;

ix. measured oil content of the mud samples, as determined by the API retort method; and

x. an itemization of other drilling fluid components and specialty additives contained in the discharged mud with concentrations reported in gal/bbl or lbs/bbl.

4. **Monitoring Requirements**

a. Chemical Inventory. For each mud system discharged, the permittee must maintain a precise chemical inventory of all constituents added downhole, including all drilling mud additives used to meet specific drilling requirements. The permittee must maintain these records for each mud system for a period of five years, and must make these records available to EPA upon request.

b. End of Well Reports. The permittee is required to submit an end-of-well report within 90 days of well completion. The permittee shall report the following for each drilling fluid system in the end-of-well report:

i. well name, number, latitude, longitude, beginning drill date, and hole diameter, well completion date;

ii. a precise chemical inventory of all constituents added downhole, including all drilling fluid additives used to meet specific drilling requirements;

iii. the base drilling fluid type;

iv. the name and total amount of each constituent in the discharged drilling fluid;

v. the total volumes of drilling fluid created and added downhole;

vi. the maximum concentration of each constituent in the drilling fluid;

vii. the total volumes of drilling fluid discharged to surface waters; and

viii. the estimated amount of each constituent in the drilling fluid discharged to surface waters.

c. Diesel Oil.

i. Compliance with the limitation on diesel oil must be demonstrated by gas chromatography (GC) analysis of drilling muds collected from the mud used at the greatest well depth ("end-of-well" sample) and of any muds or cuttings which fail the daily Static Sheen Test. In all cases, the determination of the presence or absence of diesel oil must be based on a comparison of the GC spectra of the sample and of the diesel oil in storage at the facility. The method for GC analysis must be described in "Analysis of Diesel Oil in Drilling Fluids and Drill Cuttings" (CENTEC, 1985) available from EPA, Region 10. Gas chromotography/mass spectrometry (GC/MS) may be used if an instance should arise where the operator and EPA determine that greater resolution of the drilling mud "fingerprint" is needed for a particular drilling mud sample.

ii. The results and raw data, including the spectra, from the GC analysis must be provided to the Director by written report (1) within 30 days of a positive result with the Static

Sheen Test when a discharge has occurred, or (2) for the end-of-well analysis, within 90 days of well completion.

d. Static Sheen Test.

i. The permittee must perform the Static Sheen Test on separate samples of drilling muds and cuttings, as required in 40 CFR Part 435, Subpart A, Appendix 1. Samples must be collected on each day of discharge and prior to bulk discharges.

ii. The test must be conducted in accordance with "Approved Methodology: Laboratory Sheen Tests for the Offshore Subcategory, Oil and Gas Extraction Industry," 40 CFR Part 435, Subpart A, Appendix 1. For discharge below ice or during periods of unstable or broken ice, water temperature for the Static Sheen Test must approximate surface water temperatures at ice breakup.

iii. Whenever muds or cuttings fail the Static Sheen Test, and a discharge has occurred in the past 24 hours, the permittee is required to analyze an undiluted sample of the material which failed the test to determine the presence or absence of diesel oil. The determination and reporting results must be performed according to Section II.B.4.c, above.

e. Metals Analysis.

i. The permittee shall analyze each discharged mud system for the following metals: barium, cadmium, chromium, copper, mercury, zinc, and lead. Analyses for total recoverable concentrations shall be conducted and reported for each metal utilizing the methods specified in 40 CFR Part 136. The results shall be reported in "mg/kg of whole mud (dry weight)" and the moisture content (percent by weight) of the original drilling mud sample shall be reported.

ii. Samples shall be collected when the residual mineral concentration is at its maximum value. If no mineral oil is used, the analysis shall be done on a drilling mud sample from the mud system used at the greatest well depth. All samples shall be collected prior to any predilution.

f. Mercury and Cadmium Content in Barite.

i. The permittee must analyze a representative sample of stock barite once prior to drilling each well and submit the results for total mercury and total cadmium in the DMR for the month in which drilling of the well was commenced. Analyses must be conducted by absorption spectrophotometry and results expressed as mg/kg (dry weight) of barite.

ii. If more than one well is drilled at a site, new analyses are not required for subsequent wells if no new supplies of barite have been received since the previous analysis. In this case, the DMR should state that no new barite was received since the last reported analysis. Operators may provide certification, as documented by the supplier(s), that the barite meets the above limits. The concentration of mercury and cadmium in stock barite must be reported on the DMR as documented by the supplier.

5. **Environmental Monitoring Requirements**

a. New Exploratory Facilities. Monitoring of the fate and effects of drilling muds and/or cuttings discharges are required for all new facilities.

b. Environmental Monitoring Study. Operators of new exploratory facilities discharging drilling muds and/or cuttings shall submit a plan of study for environmental monitoring to EPA for review with, or prior to, submission of an NOI.

c. Objectives. The objectives of the environmental monitoring must be to:

i. monitor for discharge-related impacts,

ii. determine statistically significant changes in sediment pollutant concentrations and sediment toxicity with time and distance from the discharge,

iii. monitor for discharge related impacts to the benthic community,

iv. assess whether any impacts warrant an adjustment of the monitoring program, and

v. provide information for permit reissuance.

d. Requirements. The monitoring must include, but not be limited to, relevant hydrographic, sediment hydrocarbon, and heavy metal data from surveys conducted before and during drilling mud disposal and up to a least one year after drilling operations cease. The monitoring plan must address:

i. the monitoring objectives,

ii. appropriate null and alternate test hypotheses,

iii. a statistically valid sampling design,

iv. all monitoring procedures and methods,

v. a quality assurance/quality control program,

vi. a detailed discussion of how data will be used to meet, test and evaluate the monitoring objectives, and

vii. a summary of the results of previous environmental monitoring as they apply to the proposed program plan.

e. Reporting Requirements.

i. The permittee must analyze the data and submit a draft report within 180 days following the completion of sample collection. The report must address the environmental monitoring objectives by using appropriate descriptive and analytical methods to test for and to describe any impacts of the effluent on sediment pollutant concentrations, sediment quality, water quality and/or the benthic community. The report must include all relevant quality assurance/quality control (QA/QC) information, including but not limited to instrumentation, laboratory procedures, detection limits/precision requirements of the applied analyses, and sample collection methodology.

ii. EPA will review the draft report in accordance with the environmental monitoring objectives and evaluate it for compliance with the requirements of the permit. If revisions to the report are required, the permittee must complete them and submit the final report to EPA within two months of the Director's request. The permittee will be required to correct, repeat and/or expand environmental monitoring programs which have not fulfilled the requirements of the permit.

f. Modification of Monitoring Program. The monitoring program may be modified if EPA determines that the modification is appropriate. The modified program may include changes in sampling stations, sampling times, and/or parameters.

g. Exemption. EPA may grant a written exemption to this requirement if the permittee can satisfactorily demonstrate that information on the fate and effects of the discharge is available and/or the discharge will not have significant impacts on the area of biological significance. An exemption to post-drilling monitoring will be granted if no impact was indicated during drilling. An exemption request must be submitted to EPA for review with, or prior to, submission of an NOI.

C. Requirements for Deck Drainage (Discharge 002)

1. **Effluent Limitations and Monitoring Requirements.** In addition to the restrictions set out in Section II.C.2-3, the permittee must comply with the following effluent limitations and monitoring requirements.

Table 2. Effluent Limitations and Monitoring Requirements for Deck Drainage (Discharge 002)

Effluent Parameter	Units	Effluent Limitations		Monitoring Requirements	
		Avg. Monthly Limit	Maximum Daily Limit	Sample Frequency	Sample Type
Free Oil	---	No discharge		Daily [note 2]	Visual [note 1]
WET [note3]	TUc	Report		Once during the first year the permittee is covered by the permit [note 4]	Part III.A
Flow	mgd	---		Monthly	Estimated

Footnotes:

1 If discharge occurs during broken or unstable ice conditions, or during stable ice conditions, the Static Sheen Test must be used (see Appendix 1 to 40 CFR part 435, subpart A).

2 When discharging. The monitoring frequency is reduced to monthly if the permittee has complied with this requirement for three consecutive months.

3 Contaminated deck drainage must be processed through an oil-water separator prior to discharge (See Section II.C.2) and samples for that portion of the deck drainage collected from the separator effluent must be sampled for WET testing.

4 Sample must be collected during a significant rainfall or snow melt. If discharge of deck drainage separate from produced water is initiated after the first year of the permit, sampling must occur during the year following the initiation of separate deck drainage discharge.

2. The permittee must ensure that deck drainage contaminated with oil and grease is processed through an oil-water separator prior to discharge. Once per discharge event, the permittee must sample deck drainage discharges that are processed through the oil-water separator and test for sheen using the Static Sheet Test in 40 CFR part 435, subpart A. For analysis of Total Aromatic Hydrocarbons (TAH) and Total Aqueous Hydrocarbons (TAqH) all analytical requirements cited in the Alaska Standards, 18 ACC 70.020(b) are applicable.

3. **Commingled Waste Streams.** If deck drainage is commingled with produced water, then this discharge shall be considered produced water for monitoring purposes (see Section II.G). However, samples collected for compliance with the produced water oil and grease limits shall be taken prior to commingling the produced water stream with deck drainage or any other waste stream. The estimated deck drainage flow rate must be reported in the comment section of the DMR.

D. Requirements for Sanitary Waste Water (Discharges 003)

1. **Effluent Limitations and Monitoring Requirements**. In addition to the restrictions set out in Sections II.D.2-3, the permittee must comply with the following effluent limitations and monitoring requirements.

2. For any facility located in Federal Waters using a marine sanitation device (MSD), the permittee must conduct annual testing of the MSD to ensure that the unit is operating properly. The permittee must note on the December DMR the results of the test.

Table 3-A: Effluent Limitations and Monitoring Requirements for Sanitary Waste Water (Discharge 003)

Discharge	Effluent Parameter	Effluent Limitations		Monitoring Requirements	
		Avg. Monthly Limit	Max. Daily Limit	Sample Frequency	Sample Type
Sanitary Waste Water, All Discharges note 2	Flow Rate (mgd)	Report		1/Month	Estimate
	Total Residual Chlorine	–	1mg/l Minimum note 5	1/Month	Grab
	Total Residual Chlorine	–	1mg/L note6	1/Month	Grab
	Total Residual Chlorine	–	0.0075 mg/l note6	1/Month	Grab
	Floating Solids	No Discharge		1/Day	Observation note 1
M10 MSD and MSD/ Biological Treatment Units	BOD note 3	30 mg/l	60 mg/l	1/Month	Grab
	TSS note 3	51 mg/l	67 mg/l	1/Month	Grab
M9IM MSD and MSD/Biological Treatment Units	BOD note 3	30 mg/l	60 mg/l	1/Month	Grab
	TSS note 3	51 mg/l	67 mg/l	1/Month	Grab
M10 Biological Treatment Units	BOD note 3	30 mg/l	60 mg/l	1/Month	Grab
	TSS note 3, 4	30 mg/l	60 mg/l	1/Month	Grab
M9IM Biological Treatment Units	BOD note 3	48 mg/l	90 mg/l	1/Month	Grab
	TSS note 3, 4	56 mg/l	108 mg/l	1/Month	Grab

Table 3-B: Platform Specific Limitations and Monitoring Requirements for Sanitary Waste Water (Discharge 003)

Platform	Parameter	Average Monthly Maximum Daily Limits	Sample Frequency	Sample Type
Bruce	Total Residual Chlorine	2.25 mg/l note 7	1/Month	Grab
Dillon	Total Residual Chlorine	0.66 mg/l note 7	1/Month	Grab
Baker	Total Residual Chlorine	2.25 mg/l note 7	1/Month	Grab
Steelhead	Total Residual Chlorine	13.35 mg/l note 7	1/Month	Grab
Granite Point	Total Residual Chlorine	7.68 mg/l note 7	1/Month	Grab
Dolly Varden	Total Residual Chlorine	13.35 mg/l note 7	1/Month	Grab
Tyonek A	Total Residual Chlorine	13.35 mg/l note 7	1/Month	Grab
Platform A	Total Residual Chlorine	13.35 mg/l note 7	1/Month	Grab
Platform C	Total Residual Chlorine	13.35 mg/l note 7	1/Month	Grab

Footnotes:

1 The permittee must monitor by observing the surface of the receiving water in the vicinity of the outfall(s) during daylight at the time of maximum estimated discharge, during conditions when observation on the surface of the receiving water is possible in the vicinity of the discharge. For sanitary waste, observations must follow either the morning or midday meal.

2 In cases where sanitary and domestic wastes are mixed prior to discharge, and sampling of the sanitary waste component stream is infeasible, the discharge may be sampled after mixing. In such cases, the most stringent discharge limitations for both discharges shall apply to the mixed waste stream.

3 The numeric limits for BOD and TSS apply only to discharges to Coastal Waters and Territorial Seas.

4 The TSS limitation for biological treatment units is a net value. The net TSS value is determined by subtracting the TSS value of the intake water from the TSS value of the effluent. Report the TSS value of the intake water on the comment section of the DMR. For those facilities that use filtered water in the biological treatment units, the TSS of the effluent may be reported as the net value. Samples collected to determine the TSS value of the intake water must be taken on the same day, during the same time period that the effluent sample is taken. Intake water samples must be taken at the point where the water enters the facility prior to mixing with other flows. Influent samples must be taken with the same frequency that effluent samples are taken.

5 Immediately after chlorination.

6 Measured immediately prior to discharging from facilities. The 1 mg/L limit applies to facilities located in Territorial Seas and Coastal Waters granted a 100-meter mixing zone by ADEC. The 0.0075 mg/L limit applies to facilities in Coastal Waters not granted a mixing zone by ADEC. The analytical detection limit for this parameter is 0.1 mg/L. These limits do not apply to facilities listed in Table 3-B.

7 Measured immediately prior to discharging.

3. For Facilities Located in Federal Waters

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.

Total residual chlorine is a surrogate parameter for fecal coliform. Discharge of residual chlorine 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).

Any facility which properly operates and maintains a 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.

4. For purposes of calculating monthly averages, zero may be assigned for values less than the method detection limit (MDL), the {numeric value of the MDL} may be assigned for values between the MDL and the ML. If the average value is less than the MDL, the permittee must report "less than {numeric value of the MDL}." If a value is equal to or greater than the ML, the permittee must report and use the actual value. The resulting average value must be compared to the effluent limitation to assess compliance.

5. The effluent limits for total residual chlorine are not quanitifiable using EPA-approved analytical methods. The ML for total residual chlorine is 0.1 mg/L, which is the compliance evaluation level for this parameter.

E. Requirements for Domestic Waste Water (Discharge 004)

1. **Effluent Limitations and Monitoring Requirements**. The permittee must comply with the following effluent limitations and monitoring requirements.

Table 4. Effluent Limitations and Monitoring Requirements for Domestic Waste Water (Discharge 004)

Discharge	Effluent Parameter	Effluent Limitations		Monitoring Requirements	
		Average Monthly Limit	Maximum Daily Limit	Sample Frequency	Sample Type
Domestic Waste Water (004) [note 2]	Flow Rate	Report		1/Month	Estimate
	Floating Solids	No Discharge		1/Day [note 1]	Visual
	Foam	No Discharge		1/Day	Visual

Footnotes:

1 The permittee must monitor by observing the surface of the receiving water in the vicinity of the outfall(s) during daylight at the time of maximum estimated discharge. For domestic waste, observations must follow either the morning or midday meal.

2 In cases where sanitary and domestic wastes are mixed prior to discharge, and sampling of the sanitary waste component stream is infeasible, the discharge may be sampled after mixing. In such cases, the most stringent discharge limitations for both discharges (Discharge 003 and Discharge 004) shall apply to the mixed waste stream.

F. Requirements for Miscellaneous Discharges (Discharges 005-014)

1. **Effluent Limitations and Monitoring Requirements**. In addition to the restrictions set out in Sections II.F.2-4, the discharge of desalination unit wastes (005); blowout preventer fluid (006); boiler blowdown (007): fire control system test water (008); non-contact cooling water (009); uncontaminated ballast water (010); bilge water (011); excess cement slurry (012); mud, cuttings, cement at the seafloor (013); and waterflooding (014) must comply with the following effluent limitations and monitoring requirements.

Table 5: Effluent Limitations and Monitoring Requirements for Miscellaneous Discharges 005 - 014

Parameter	Effluent Limitations		Monitoring Requirements	
	Average Monthly Limit	Maximum Daily Limit	Sample Frequency	Sample Type
Flow (mgd)	Report		Monthly	Estimate
Free Oil	No discharge [note 1]	No discharge [note 1]	Once/Week [note 1]	Visual
Chemical Additives	See Section II.F.3		Monthly	Calculation
WET [note 2]	See Section II.F.4 of this permit	See Section II.F.4 of this permit	Once/Quarter	Grab

Footnotes:

1 Miscellaneous 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 during periods of slack tide 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. For discharges during stable ice, below ice, to unstable ice or broken ice conditions, a water temperature that approximates surface water temperatures after breakup shall be used.

2 Applicable to all discharges to which chemical additives have been added, except Discharges 012 (excess cement slurry) and 013 (mud, cuttings, cement at the seafloor).

2. **Discharge Specific Limitations**

a. **Desalination Unit Waste Water (Discharge 005)**. In addition to the limitations and monitoring requirements in Section II.F.1, the permittee must maintain an annual inventory of the quantities and rates of chemicals and biocides that are added to desalination unit waste water. Each annual inventory must be assembled for the calendar year and submitted to EPA by April 1 for the previous calendar year.

b. **Bilge Water (Discharge 011).** The permittee shall process all bilge water through an oil-water separator prior to discharge.

c. **Commingled Waste Streams**. If excess waterflood water is added to the produced water discharge in order to minimize the possibility of line freezing, then the discharge must be considered produced water for monitoring purposes. The estimated waterflood flow rate must be reported in the comment section of the DMR.

3. **Chemical Additives**. The concentration of treatment chemicals in discharged seawater or freshwater shall not exceed the most stringent of the following three constraints:

a. the maximum concentrations and any other conditions specified in the EPA product registration labeling if the chemical is an EPA registered product

b. the maximum manufacturer's recommended concentration

c. 500 mg/l

Compliance with these limitations shall be calculated based on the amount of treatment chemical added to the volume of water discharged.

4. **Whole Effluent Toxicity Effluent Monitoring Requirements**. Whole Effluent Toxicity (WET) testing shall be accomplished in accordance with the monitoring requirements in Sections II.G.6.a and III.A of this permit and the WET trigger values (expressed as Chronic Toxic Units) shown below in Table 6. This requirement applies to miscellaneous discharges greater than 10,000 gallons per day. WET testing shall commence six months after the effective date of the permit.

New facilities located in Coastal Waters, which are not listed below in Table 6-C, shall conduct WET testing for informational purposes. The testing requirements for these facilities are quarterly for the first year and will be reduced to once every 6 months following the first year of testing.

Table 6-A: WET Trigger Levels for Surface Discharges in the Territorial Seas and Outer Continental Shelf

Discharge Rate (gpd)	Critical Dilution	Trigger Values (TUc)
10,000 to 17,000	0.24%	417
17,001 to 22,000	0.27%	370
22001 to 27,000	0.29%	345
27,001 to 55,000	0.36%	278
55,001 to 150,000	0.46%	217
Greater than 150,000	0.62%	161

Table 6-B: WET Trigger Levels for Submerged Pipe Discharges in the Territorial Seas and Outer Continental Shelf

Discharge Rate (gpd)	Critical Dilution	Trigger Values (TUc)
10,000 to 17,000	0.33%	303
17,001 to 22,000	0.36%	278
22001 to 27,000	0.37%	270
27,001 to 55,000	0.49%	204
55,001 to 150,000	0.62%	161
Greater than 150,000	0.99%	101

Table 6-C: WET Trigger Levels for Discharges in Coastal Waters

Facility	Critical Dilution	Trigger Values (TUc)
Platform Anna Platform	2.4%	42
Dolly Varden	5.5%	18.2
Granite Point Platform	7.1%	14
Platform Grayling	6.1%	16.3
Platform King Salmon	13.6%	7.3
Platform Monopod	5.8%	17.1
Platform Steelhead	0.17%	604

G. Requirements for Produced Water and Produced Sand (Discharge 015)

1. **Effluent Limitations and Monitoring Requirements**. In addition to the restrictions set out in Sections II.G.2-6, the permittee must comply with the following effluent limitations and monitoring requirements. The discharge of produced water from New Sources and new exploratory facilities is not authorized by this general permit.

Table 7-A: Effluent Limitations and Monitoring Requirements for Produced Water and Produced Sand

Parameter	Effluent Limitations		Monitoring Requirements	
	Avg. Monthly	Max. Daily	Sample Frequency	Sample Type
Flow Rate (mgd)	Report	Report	1/Week	Estimate
Produced Sand	No Discharge	No Discharge	–	–
Oil and Grease	29 mg/l	42 mg/l	1/Week	– [note 1]
pH < 1 MGD [note 3]	6.0 to 9.0 S.U.		1/Month	Grab
pH > 1 MGD [note 3]	6.0 to 9.0 S.U.		1/Week	Grab
Free Oil	Report [note 2]		1/Day	Visual [note 2]

Footnotes

1 The sample type shall be either grab, or a 24-hour composite which consists of the arithmetic average of the results of 4 grab samples taken over a 24-hour period. If a sample is unavailable to be analyzed and the permittee has explained the reason in the DMR, averaging of the remaining samples is permitted. If only one sample is taken for any one month, it must meet both the daily and monthly limits. Samples shall be collected prior to the addition of any seawater to the produced water waste stream. See Section II.G.6.b of this permit

2 See Section II.G.6.b of this permit

3 based on the previous month's monthly average discharge rate.

Table 7-B: Facility Specific Incremental Water Quality Based Limits and Monitoring Requirements

Table 7-B1: Granite Point Treatment Facility and Platform

Parameter	Effluent Limitations		Monitoring Requirements	
	Avg. Monthly	Max Daily	Frequency	Sample Type
TAH [note 1]	14 mg/l	20 mg/l	1/Month	Grab
TAqH [note 1]	–	–	1/Month	Grab
Unionized Ammonia	–	–	Quarterly	Grab
Copper[note3]	67 ug/l	130 ug/l	1/Month [note 2]	Grab
Mercury[note3]	3.1 ug/l	7.9 ug/l	1/Month [note 2]	Grab
Manganese[note3]	6.1 mg/l	12.3 mg/l	1/Month [note 2]	Grab
Silver[note3]	37 ug/l	74 ug/l	1/Month [note 2]	Grab
Zinc[note3]	1.5 mg/l	3.1 mg/L	1/Month [note 2]	Grab
WET	1341 TUc	2691 TUc	1/Quarter [note 2]	Grab

Table 7-B2: The East Foreland Facility

Parameter	Effluent Limitations		Monitoring Requirements	
	Avg. Monthly	Max Daily	Frequency	Sample Type
TAH [note 1]	24 mg/l	32 mg/l	1/Month	Grab
TAqH [note 1]	–	–	1/Month	Grab
Unionized Ammonia	–	–	Quarterly	Grab
Copper[note3]	60 ug/l	90 ug/l	1/Month [note 2]	Grab
Mercury[note3]	0.5 ug/l	0.8 ug/l	1/Month [note 2]	Grab
Manganese[note3]	7.9 mg/l	15.8 mg/l	1/Month [note 2]	Grab
Silver[note3]	46 ug/l	149 ug/l	1/Month [note 2]	Grab
Zinc[note3]	3.1 mg/l	6.1 mg/L	1/Month [note 2]	Grab
WET	1209 TUc	2425 TUc	1/Quarter [note 2]	Grab

Table 7-B3: Platform Anna

Parameter	Effluent Limitations		Monitoring Requirements	
	Avg. Monthly	Max Daily	Frequency	Sample Type
TAH [note 1]	109 mg/l	183 mg/l	1/Month	Grab
TAqH [note 1]	–	–	1/Month	Grab
Unionized Ammonia	–	–	Quarterly	Grab
Copper[note3]	53 ug/l	79 ug/l	1/Month [note 2]	Grab
Mercury[note3]	3.8 ug/l	9.5 ug/l	1/Month [note 2]	Grab
Manganese[note3]	7.4 mg/l	14.8 mg/l	1/Month [note 2]	Grab
Silver[note3]	687 ug/l	1378 ug/l	1/Month [note 2]	Grab
Zinc[note3]	22 mg/l	57 mg/L	1/Month [note 2]	Grab
WET	574 TUc	1152 TUc	1/Quarter [note 2]	Grab

Table 7-B4: Platform Bruce

Parameter	Effluent Limitations		Monitoring Requirements	
	Avg. Monthly	Max Daily	Frequency	Sample Type
TAH note 1	78 mg/l	143 mg/l	1/Month	Grab
TAqH note 1	–	–	1/Month	Grab
Unionized Ammonia	–	–	Quarterly	Grab
Copper note3	1429 ug/l	2867 ug/l	1/Month note 2	Grab
Mercury note3	3.7 ug/l	9.2 ug/l	1/Month note 2	Grab
Manganese note3	7.2 mg/l	14.4 mg/l	1/Month note 2	Grab
Silver note3	7.3 ug/l	11.0 ug/l	1/Month note 2	Grab
Zinc note3	28 mg/l	47 mg/L	1/Month note 2	Grab
WET	2149 TUc	4312 TUc	1/Quarter note 2	Grab

Table 7-B5: Platform Baker

Parameter	Effluent Limitations		Monitoring Requirements	
	Avg. Monthly	Max Daily	Frequency	Sample Type
TAH note 1	128 mg/l	257 mg/l	1/Month	Grab
TAqH note 1	–	–	1/Month	Grab
Unionized Ammonia	–	–	Quarterly	Grab
Copper note3	435 ug/l	873 ug/l	1/Month note 2	Grab
Mercury note3	0.3 ug/l	0.4 ug/l	1/Month note 2	Grab
Manganese note3	7.1 mg/l	14.2 mg/l	1/Month note 2	Grab
Silver note3	173 ug/l	347 ug/l	1/Month note 2	Grab
Zinc note3	6.7 mg/l	14.3 mg/L	1/Month note 2	Grab
WET	172 TUc	345 TUc	1/Quarter note 2	Grab

Table 7-B6: Platform Dillon

Parameter	Effluent Limitations		Monitoring Requirements	
	Avg. Monthly	Max Daily	Frequency	Sample Type
TAH [note 1]	31 mg/l	42 mg/l	1/Month	Grab
TAqH [note 1]	–	–	1/Month	Grab
Unionized Ammonia	–	–	Quarterly	Grab
Copper[note3]	9.3 ug/l	14.0 ug/l	1/Month [note 2]	Grab
Mercury[note3]	1.2 ug/l	2.5 ug/l	1/Month [note 2]	Grab
Manganese[note3]	2.3 mg/l	4.6 mg/l	1/Month [note 2]	Grab
Silver[note3]	28 ug/l	55 ug/l	1/Month [note 2]	Grab
Zinc[note3]	1.2 mg/l	2.3 mg/L	1/Month [note 2]	Grab
WET	293 TUc	588 TUc	1/Quarter [note 2]	Grab

Table 7-B7: Trading Bay Production Facility

Parameter	Effluent Limitations		Monitoring Requirements	
	Avg. Monthly	Max Daily	Frequency	Sample Type
TAH [note 1]	18 mg/l	27 mg/l	1/Month	Grab
TAqH [note 1]	–	–	1/Month	Grab
Unionized Ammonia	–	–	Quarterly	Grab
Copper[note3]	47 ug/l	117 ug/l	1/Month [note 2]	Grab
Mercury[note3]	0.6 ug/l	1.0 ug/l	1/Month [note 2]	Grab
Manganese[note3]	25 mg/l	50 mg/l	1/Month [note 2]	Grab
Silver[note3]	23 ug/l	47 ug/l	1/Month [note 2]	Grab
Zinc[note3]	0.9 mg/l	1.9 mg/L	1/Month [note 2]	Grab
WET	283 TUc	568 TUc	1/Quarter [note 2]	Grab

Table 7-B8: Tyonek A

Parameter	Effluent Limitations		Monitoring Requirements	
	Avg. Monthly	Max Daily	Frequency	Sample Type
TAH [note 1]	0.09 mg/l	0.14 mg/l	1/Month	Grab
TAqH [note 1]	–	–	1/Month	Grab
Unionized Ammonia	–	–	Quarterly	Grab
Copper[note3]	328 ug/l	1033 ug/l	1/Month [note 2]	Grab
Mercury[note3]	0.05 ug/l	0.10 ug/l	1/Month [note 2]	Grab
Manganese[note3]	0.1 mg/l	0.2 mg/l	1/Month [note 2]	Grab
Silver[note3]	205 ug/l	411 ug/l	1/Month [note 2]	Grab
Zinc[note3]	8.4 mg/l	17.0 mg/L	1/Month [note 2]	Grab
WET	268 TUc	537 TUc	1/Quarter [note 2]	Grab

Footnotes:

1 For analysis of TAH and TAqH, all analytical requirements cited in the Alaska Standards, 18 AAC 70.020(b) are applicable.

2 See Section II.G.6.a of this permit

3 All metals limits are in total recoverable form, except mercury which is total.

2. The operator of the Trading Bay Production Facility shall install a diffuser within two years of the effective date of the permit.

3. **Rerouting Platform Discharge to a Shore-Based Facility.** In situations where the platforms are not able to treat produced water and a bypass (as defined in Section VII.G) may occur, the Anna, Bruce, and Granite Point platforms may route their produced water discharge to the Granite Point Tank Farm/Treatment Facility for treatment and discharge. Platforms A, C, Baker, and Dillon may route their produced water discharge to the East Forelands Production Facility. The permittee must provide a written submission with the next DMR that describes why rerouting was necessary, and the anticipated time that rerouting is expected to continue. The permittee must cease rerouting as soon as possible.

4. **Trading Bay Production Facility Groundwater**. Trading Bay is authorized to discharge treated ground water extracted pursuant to State Compliance Order #91-23-01-053-02 as part of the produced water waste stream. The produced water limitations and monitoring requirements apply to the combined waste stream of treated ground water and produced water.

5. **Spill Clean-Up**. Water that is collected as a result of spill clean-up can be treated as produced water and discharged with the produced water waste stream. The permittee must report the treatment and discharge of spill clean-up water by telephone or facsimile to EPA within 24 hours of initiating such treatment, and must provide a written submission within five days of initiating treatment that describes the spill, the anticipated volume of spill clean-up water, and the anticipated time that treatment and discharge of spill clean-up water is expected to continue.

6. **Monitoring Requirements**.

   a. **Monitoring Frequencies**. This section is applicable to the monitoring requirements for TAH, TAqH, Total Recoverable Copper, Manganese, Silver, and Zinc, Total Mercury, and WET. This Section also applies to WET monitoring for Discharges 005-014.

      1. Monitoring Reductions. The required minimum monitoring frequency for TAH, TAqH, Total Recoverable Copper, Manganese, Silver, and Zinc, and Total Mercury is reduced to once per quarter if the permittee has complied with the limits for those parameters for a period of 12 consecutive months. The required monitoring frequency for WET is reduced to once per six months if the permittee has complied with the limits or not exceeded the triggers for a period of one year (4 consecutive quarters). If the permittee subsequently violates the limit or exceeds the triggers for any parameter in this section, the increased monitoring provisions of Section II.G.6.a.2 shall apply. Permittees meeting the requirements for reduced monitoring shall report the monitoring frequency on the DMR.

      2. Increased Monitoring. If a permittee is not in compliance with a limit or exceeds the trigger, the monitoring frequency for that parameter shall increase until compliance has been demonstrated for a period of three consecutive months. After compliance has been established for a period of three consecutive months, the required monitoring frequency shall return to that shown in Table 7. The increased monitoring frequence is once per week for TAH, TAqH, Total Recoverable Copper, Manganese, Silver, and Zinc, and Total Mercury. The

increased minimum monitoring frequency for WET is explained in Section III.A.7. Permittees shall report the required monitoring frequency on the DMR.

b. **Visual Sheen and Oil and Supplemental Grease Monitoring**
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. The visual sheen monitoring requirement does not apply to shore-based facilities.

A produced water sample shall be collected and analyzed for oil and grease when a sheen is observed in the vicinity of the produced water discharge. At a minimum, a sample shall be collected and analyzed once per month. If there is a permit violation or a spill, the permittee shall report the incident pursuant to Sections VI.G and VI.H of the permit.

H. Requirements for Well Treatment, Completion, Workover, and Test Fluids (Discharges 016-019)

1. **Effluent Limitations and Monitoring Requirements**. In addition to the restrictions set out in Sections II.H.2-3, the permittee must comply with the following effluent limitations and monitoring requirements.

Table 8: Effluent Limitations and Monitoring Requirements for Well Treatment, Completion, Workover, and Test Fluids

Parameter	Effluent Limitation		Monitoring Requirements	
	Avg. Monthly	Max. Daily	Frequency	Sample Type
Discharge frequency	Report		1/Discharge [note 1]	Occurrences
Volume discharged (mgd)	Report		Once/Day	Estimate
Oil and grease [note 2]	29 mg/l	42 mg/l	1/Discharge [note 1]	Grab [note 4]
Oil-based fluids	No discharge		–	–
Free oil [note 2]	No free oil		1/Discharge [note 1]	Grab [note 3]
pH	6.5 - 8.5		1/Discharge [note 1]	Grab

Footnotes:

[1] The type of discharge (*i.e.*, completion, workover, treatment, test fluid, or any combination) must be reported. Discharge of individual waste streams must be reported separately from the discharge of commingled waste streams.

[2] Limits for free oil and oil and grease apply to each discharge, whether these waste streams are discharged individually or are commingled. All fluids must be processed through an oil-water separator prior to discharge. Samples must be collected after the final step of treatment.

[3] Monitoring for compliance with the free oil prohibition shall be accomplished using the Static Sheen Test method. The number of days a sheen is observed must be reported.

[4] The sample type may be either grab, or a 24-hour composite consisting of the arithmetic average of the results of 4 grab samples taken within the 24-hour period. 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.

2. **Commingled Waste Streams.** If well treatment, completion, workover or test fluids are mixed with produced water, then this discharge must be considered produced water for monitoring purposes (See Section II.G.). The estimated flow rate of treatment, completion, workover, or test fluids must be reported in the comment section of the DMR.

3. **Chemical Inventory**. The permittee must maintain an inventory of the type and quantity of chemicals (other than fresh or seawater) added to completion, workover, well treatment, and test fluids. The inventory(ies) must be submitted annually. The inventories must be assembled for the calendar year, and must be submitted to EPA by April 1 for the previous calendar year.

III. SPECIFIC TEST REQUIREMENTS

A. WET Testing Requirements (Outfalls 002 and 005 - 015 only, except 012 and 013). For Outfall 001 drilling fluids toxicity tests see Section III.B, below and 40 CFR Part 435, Subpart A.)

1. The permittee must conduct tests on grab effluent samples with one vertebrate and two invertebrate species, as follows.

   a. Vertebrate (survival and growth): Topsmelt, *Atherinops affinis*. In the event that topsmelt is not available, inland silverside (*Menidia beryllina*) may be used as a substitute. The permittee shall document the substitute species in the next DMR.

   b. Invertebrate: the permittee must conduct tests with a bivalve species, Pacific Oyster, *Crassostrea gigas*, or mussel, *Mytilus sp.* (larval development test), and an echinoderm, purple sea urchin, *Strongylocentrotus purpuratus*, or sand dollar, *Dendraster excentricus* (fertilization test). Due to seasonal variability, testing may be performed during reliable spawning periods (*e.g.*

December through February for mussels; June through August for oysters).

2. Each year, the permittee must rescreen with the three species listed above, and continue to monitor with the most sensitive species. Rescreening must consist of one test conducted at a different time of year from the previous year's test. After screening is completed, monitoring shall be continued at the frequency required in Part II of this permit.

3. The presence of chronic toxicity must be estimated as specified in *USEPA Short-Term Methods for Estimating the Chronic Toxicity of Effluents and Receiving Waters to Marine and Estuarine Organisms, the Third Edition* (EPA-821-R-02-014). For the bivalve species, chronic toxicity must be estimated as specified in *Short-Term Methods for Estimating the Chronic Toxicity of Effluents and Receiving Water to West Coast Marine and Estuarine Organisms* (EPA/600/R-95/136).

4. Results must be reported in TUc, where TUc = $100/IC_{25}$. The reported IC_{25} must be the lowest IC_{25} calculated for the applicable survival, growth or fertilization endpoints.

5. A series of at least five dilutions and a control must be tested. The series must include the critical dilution and two dilutions both above and two below the critical dilution.

6. In addition to those quality assurance measures specified in the methodology, the following quality assurance procedures must be followed:

   a. If organisms are not cultured by the testing laboratory, concurrent testing with reference toxicants must be conducted, unless the test organism supplier provides control chart data from at least the last 5 months of reference toxicant testing. Where organisms are cultured by the testing laboratory, monthly reference toxicant testing is sufficient.

   b. If either of the reference toxicant tests or the effluent tests do not meet all test acceptability criteria as specified in the test methods manual, then the permittee must re-sample and re-test as soon as possible.

   c. Control and dilution water should be receiving water, or salinity adjusted lab water. If the dilution water used is different from the

culture water, a second control, using culture water must also be used.

7. Accelerated Testing

a. If chronic toxicity is detected above the permit limits or trigger values set forth in Sections II.F.4 or II.G.1, collection and analysis of one additional sample is required within two weeks of receipt of the test results.

b. If chronic toxicity is not detected in the sample required by Sections III.A.7.a, the permittee must notify EPA and ADEC in writing of the results within fifteen (15) days of receipt of the results, and must discuss the cause of the exceedance, and the corrective actions that were taken.

c. If chronic toxicity is detected in the sample required by Sections III.A.7.a, then the permittee must conduct four bi-weekly tests over an eight week period. Accelerated testing must be initiated within fifteen (15) days of receiving the sample results required by Sections III.A.7.a.

8. Toxicity Reduction Evaluation (TRE) and Toxicity Identification Evaluation (TIE):

a. If chronic toxicity limits or triggers are exceeded during accelerated testing, the permittee must initiate a toxicity reduction evaluation (TRE) in accordance with *Generalized Methodology for Conducting Industrial Toxicity Reduction Evaluations* (EPA/600/2-88/070) within two weeks of the receipt of the test results showing an exceedence. At a minimum, the TRE must include:

i) Further actions to investigate and identify the cause of toxicity;

ii) Actions the permittee will take to mitigate the impact of the discharge and to prevent the recurrence of toxicity; and

iii) A schedule for these actions.

b. If a TRE is initiated prior to completion of the accelerated testing, the accelerated testing schedule may be terminated, or used as necessary in performing the TRE.

c. The permittee may initiate a Toxicity Identification Evaluation (TIE) as part of the TRE process. Any TIE must be performed in accordance with EPA guidance manuals, *Toxicity Identification Evaluation; Characterization of Chronically Toxic Effluents, Phase I* (EPA/600/6-91/005F), *Methods for Aquatic Toxicity Identification Evaluations, Phase II: Toxicity Identification Procedures for Samples Exhibiting Acute and Chronic Toxicity* (EPA/600/R-92/080), and *Methods for Aquatic Toxicity Identification Evaluations, Phase III: Toxicity Confirmation Procedures for Samples Exhibiting Acute and Chronic Toxicity* (EPA-600/R-92/081).

9. Reporting.

a. Results of toxicity testing shall be reported on the DMR for the month in which the tests are conducted.

b. The full report shall be submitted by the end of the month following the month in which the DMR is submitted.

c. The full report shall consist of:

i the toxicity test results;

ii the dates of sample collection and initiation of each toxicity test

iii the flow rate at the time of sample collection

iv the results of the effluent sampling for chemical parameters required for the outfalls.

d. Test results for chronic tests shall be reported according to the procedures described in EPA's *Short-term Methods for Estimating the Chronic Toxicity of Effluents and Receiving Waters to Marine and Estuarine Organisms*, most recent edition.

B. **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 B of this permit.

C. **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 C of this permit. Compliance with the biodegradation limit will be determined using the following ratio:

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

Where:

NAF = stock base fluid being tested for compliance
Reference Fluid = C_{16}-C_{18} internal olefin or C_{12}-C_{14} or C_8 ester reference fluid

D. **Polynuclear Aromatic Hydrocarbons.** For analysis of TAH and TAqH all analytical requirements cited in the Alaska Standards, 18 ACC 70.020(b) are applicable.

E. **Formation Oil Contamination of Drilling Fluids.** The approved test method for permit compliance is identified as: 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.

F. **Formation Oil Contamination of Discharged Drilling Fluids Retained on Drill 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).

G. Retention of Non-Aqueous Based Drilling Fluids 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.

IV. BEST MANAGEMENT PRACTICES (BMP) PLAN REQUIREMENTS

A. Development. The permittee must develop a BMP Plan which achieves the objectives and the specific requirements listed below.

The permittee must certify that its BMP Plan is complete, on-site, and available upon request by EPA. This certification must identify the NPDES permit number and be signed by an authorized representative of the permittee. For new exploration facilities, the certification must be submitted with the NOI. For existing facilities, the certification must be submitted within one month of the effective date of the permit.

B. Purpose. The BMP Plan must be designed to prevent or minimize the generation and the potential release of pollutants from the facility to the waters of the United States through normal operations and ancillary activities.

C. Objectives. The permittee shall develop and amend the BMP Plan consistent with the following objectives for the control of pollutants:

1. The number and quantity of pollutants and the toxicity of effluent generated, discharged or potentially discharged at the facility shall be minimized by the permittee to the extent feasible by managing each waste stream in the most appropriate manner.

2. The permittee shall establish specific objectives for the control of pollutants by conducting the following evaluations.

    a. Each facility component or system shall be examined for its waste minimization opportunities and its potential for causing a release

of significant amounts of pollutants to waters of the United States due to equipment failure, improper operation, and natural phenomena such as rain or snowfall, etc. The examination shall include all normal operations and ancillary activities including loading or unloading operations or spillage or leaks.

b. Where experience indicates a reasonable potential for equipment failure, natural condition (*e.g.*, precipitation), or other circumstances to result in significant amounts of pollutants reaching surface waters, the program should include a prediction of the direction, rate of flow and total quantity of pollutants which could be discharged from the facility as a result of each condition or circumstance.

D. Requirements. The BMP Plan shall be consistent with the objectives listed above and the general guidance contained in the publication entitled *Guidance Manual for Developing Best Management Practices (BMPs)* (USEPA, 1993) or any subsequent revisions to the guidance document. The BMP Plan shall:

1. Be documented in narrative form, 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 must contain the planning, development and implementation, and evaluation/reevaluation components discussed in *Guidance Manual for Developing Best Management Practices (BMPs)* (USEPA, 1993) or any subsequent revisions to the guidance document.

2. Include the following provisions concerning BMP Plan review:

   a. Be reviewed by facility engineering staff and the facility manager.

   b. Be reviewed and endorsed by the permittee's BMP Committee.

   c. Include a statement that the above reviews have been completed and that the BMP Plan fulfills the requirements set forth in this permit. The statement shall be certified by the dated signature of each BMP Committee member.

3. Establish specific BMPs to meet the objectives identified above, addressing each component or system capable of generating or causing a release of significant amounts of pollutants, and identifying specific preventative or remedial measures to be implemented.

E. **Documentation.** The permittee must maintain a copy of the BMP Plan at the facility and must make the plan available to EPA and ADEC upon request.

F. **BMP Plan Modification.** The permittee shall amend the BMP Plan whenever there is a change in the facility or in the operation of the facility that materially increases the generation of pollutants or their release or potential release to the receiving waters. The permittee shall also amend the Plan, as appropriate, when facility operations covered by the BMP Plan change. Any such changes to the BMP Plan shall be consistent with the objectives and specific requirement listed above. All changes in the BMP Plan must be reviewed by the plant engineering staff and plant manager.

G. **Modification for Ineffectiveness.** At any time, if the BMP Plan proves to be ineffective in achieving the general objective of preventing and minimizing the generation of pollutants and their release and potential release to the receiving waters and/or the specific requirements above, the permit and/or the BMP Plan shall be subject to modification to incorporate revised BMP requirements.

V. PRODUCED WATER DISCHARGE STUDY REQUIREMENTS

A. **Produced Water Discharge Study.** Operators discharging greater than 100,000 gallons per day of produced water shall plan and conduct a single study that addresses the fate and transport of pollutants in the water column and sediments.

B. **Objectives.** The overall objective of the study is to evaluate contaminant fate and transport from large volume produced water dischargers. This can be accomplished by statistically comparing contaminant concentrations at the discharge point with concentrations at distances from the discharge point (transport) and evaluating the accumulation of contaminants in Cook Inlet's water column and/or sediments (fate).

C. **Schedule.** Within six months of the effective date of this permit, permittees shall submit a study plan to EPA Region 10 for approval. The final report shall be submitted to EPA within three years after the effective date of the permit.

D. **Requirements.** The plan must address a monitoring approach that:

1. Can statistically evaluate the potential accumulation of discharge contaminants in Cook Inlet through a combination of total concentration analysis and fingerprinting;

2. Includes dissolved and total recoverable metal and hydrocarbon concentration analyses that can statistically compare discharge concentrations with receiving water concentrations with distance from the discharge point;

3. Evaluates and provides justification for including or excluding contaminants measured in the dissolved and/or total recoverable phase; in the water column and/or benthic sediments; and,

4. May include a phased study design, with detailed analyses of archived samples following initial screening-level analyses for some or all parameters.

VI. RECORDING AND REPORTING REQUIREMENTS

A. Representative Sampling. The permittee must ensure that samples and measurements taken for the purpose of monitoring are representative of the monitored activity.

B. Reporting of Monitoring Results. The permittee must summarize monitoring results each month on the DMR form (EPA No. 3320-1) or equivalent. The permittee must submit reports monthly, postmarked by the 20th day of the following month. WET testing results must be reported with the DMR the month following the completion of the WET test. Annual sampling results must be reported on the January DMR unless otherwise indicated by this permit. The permittee must sign and certify all DMRs, and all other reports, in accordance with the requirements of Section VIII.E ("Signatory Requirements") of this permit. The permittee must submit legible originals of these documents to the Director, Office of Water & Watersheds, with copies to ADEC, at the addresses in Section I.G ("Submission of Information").

C. Monitoring Procedures. The permittee must conduct monitoring according to test procedures approved under 40 CFR Part 136, unless other test procedures have been specified in this permit.

D. Additional Monitoring by Permittee.

1. 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 permittee must include the results of this monitoring in the calculation and reporting of the data submitted in the DMR.

2. Upon request by the Director, the permittee must submit results of any other sampling, regardless of the test method used.

E. Records Contents. The permittee must ensure that records of monitoring information include:

3. the date, exact place, and time of sampling or measurements;

4. the name(s) of the individual(s) who performed the sampling or measurements;

5. the date(s) analyses were performed;

6. the names of the individual(s) who performed the analyses;

7. the analytical techniques or methods used; and

8. the results of such analyses.

F. Retention of Records. The permittee must 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, copies of DMRs; a copy of this NPDES permit, and records of all data used to complete the application for this permit, for a period of at least five years from the date of the sample, measurement, report or application. This period may be extended by request of the Director or ADEC at any time.

G. Twenty-four Hour Notice of Noncompliance Reporting.

9. The permittee must report the following occurrences of noncompliance by telephone or facsimile within 24 hours from the time the permittee becomes aware of the following circumstances:

a. any noncompliance that may endanger health or the environment;

b. any unanticipated bypass that exceeds any effluent limitation in the permit (See Section VII.G, “Bypass of Treatment Facilities”);

c. any upset that exceeds any effluent limitation in the permit (See Section VII.H, “Upset Conditions”); or

d. any violation of a maximum daily discharge limitation for any of the pollutants in Part II of the permit requiring 24-hour reporting.

10. The permittee must also provide a written submission within five business days of the time that the permittee becomes aware of any event required to be reported under paragraph VI.G.1. The written submission must contain:

a. a description of the noncompliance and its cause;

b. the period of noncompliance, including exact dates and times;

c. the estimated time noncompliance is expected to continue if it has not been corrected; and

d. steps taken or planned to reduce, eliminate, and prevent recurrence of the noncompliance.

11. The Director may waive the written report on a case-by-case basis if the oral report has been received within 24 hours by the NPDES Compliance Hotline in Seattle, Washington, by telephone, (206) 553-1846.

12. The permittee must submit reports to the addresses in Section VI.B ("Reporting of Monitoring Results").

H. Other Noncompliance Reporting. The permittee must report all instances of noncompliance not required to be reported within 24 hours, at the time that monitoring reports for Section VI.B ("Reporting of Monitoring Results") are submitted. The reports must also contain the information listed in Section VI.G ("Twenty-four Hour Notice of Noncompliance Reporting") of this permit.

I. Changes in Discharge of Toxic Substances. The permittee must notify the Director and ADEC as soon as it knows, or has reason to believe:

13. That any activity has occurred or will occur that would result in the discharge, on a routine or frequent basis, of any toxic pollutant that is not limited in the permit, if that discharge will exceed the highest of the following "notification levels":

a. One hundred micrograms per liter (100 µg/l);

b. Two hundred micrograms per liter (200 µg/l) for acrolein and acrylonitrile; five hundred micrograms per liter (500 µg/l) for 2,4-

dinitrophenol and for 2-methyl-4,6-dinitrophenol; and one milligram per liter (1 mg/L) for antimony;

c. Five (5) times the maximum concentration value reported for that pollutant in the permit application in accordance with 40 CFR § 122.21(g)(7); or

d. The level established by the Director in accordance with 40 CFR § 122.44(f).

14. That any activity has occurred or will occur that would result in any discharge, on a non-routine or infrequent basis, of any toxic pollutant that is not limited in the permit, if that discharge may reasonably be expected to exceed the highest of the following "notification level":

a. Five hundred micrograms per liter (500 μg/l);

b. One milligram per liter (1 mg/L); for antimony;

c. Ten (10) times the maximum concentration value reported for that pollutant in the permit application in accordance with 40 CFR § 122.21(g)(7); or

d. The level established by the Director in accordance with 40 CFR § 122.44(f).

VII. COMPLIANCE RESPONSIBILITIES

A. 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; for permit termination, revocation and reissuance, or modification; or for denial of a permit renewal application.

B. Penalties for Violations of Permit Conditions.

1. Civil Penalties. Pursuant to 40 CFR Part 19 and the Clean Water Act, any person who violates CWA Section 301, 302, 306, 307, 308, 318, or 405, or any permit condition or limitation implementing any such sections in a permit issued under CWA Section 402, or any requirement imposed in a pretreatment program approved under CWA Sections 402(a)(3) or 402(b)(8) is subject to a civil penalty not to exceed the maximum amounts authorized by CWA Section 309(d) and the Federal Civil Penalties

Inflation Adjustment Act (28 U.S.C. § 2461 note) as amended by the Debt Collection Improvement Act (31 U.S.C. § 3701 note).

2. Administrative Penalties. Any person may be assessed an administrative penalty by the Administrator for violating CWA Section 301, 302, 306, 307, 308, 318, or 405, or any permit condition or limitation implementing any of such sections in a permit issued under CWA Section 402. Pursuant to 40 CFR Part 19 and the Clean Water Act, administrative penalties for Class I violations are not to exceed the maximum amounts authorized by CWA Section 309(g)(2)(A) and the Federal Civil Penalties Inflation Adjustment Act (28 U.S.C. § 2461 note) as amended by the Debt Collection Improvement Act (31 U.S.C. § 3701 note). Pursuant to 40 CFR Part 19 and the Clean Water Act, penalties for Class II violations are not to exceed the maximum amounts authorized by CWA Section 309(g)(2)(B) and the Federal Civil Penalties Inflation Adjustment Act (28 U.S.C. § 2461 note) as amended by the Debt Collection Improvement Act (31 U.S.C. § 3701 note).

3. Criminal Penalties.

   a. Negligent Violations. Any person who negligently violates a permit condition implementing CWA Sections 301, 302, 306, 307, 308, 318, or 405 is subject to a fine and/or imprisonment as specified in CWA Section 309(c)(1).

   b. Knowing Violations. Any person who knowingly violates a permit condition implementing CWA Sections 301, 302, 306, 307, 308, 318, or 405 is subject to a fine and/or imprisonment as specified in CWA Section 309(c)(2).

   c. Knowing Endangerment. Any person who knowingly violates a permit condition implementing CWA Sections 301, 302, 303, 306, 307, 308, 318, or 405, and who knows at that time that he thereby places another person in imminent danger of death or serious bodily injury, is subject to a fine and/or imprisonment as specified in CWA Section 309(c)(3).

   d. False Statements. 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 Clean Water Act or who knowingly falsifies, tampers with, or renders inaccurate any monitoring device or method required to be maintained under the

Clean Water Act, is subject to a fine and/or imprisonment as specified in CWA Section 309(c)(4).

C. **Need to Halt or Reduce Activity not a Defense.** It shall not be a defense for the permittee in an enforcement action that it would have been necessary to halt or reduce the permitted activity in order to maintain compliance with this permit.

D. **Duty to Mitigate**. The permittee must take all reasonable steps to minimize or prevent any discharge in violation of this permit that has a reasonable likelihood of adversely affecting human health or the environment.

E. **Proper Operation and Maintenance.** The permittee must at all times properly operate and maintain all facilities and systems of treatment and control (and related appurtenances) which are installed or used by the permittee to 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 back-up or auxiliary facilities or similar systems which are installed by the permittee only when the operation is necessary to achieve compliance with the conditions of the permit.

F. **Removed Substances.** Solids, sludges, filter backwash, or other pollutants removed in the course of treatment or control of water and wastewaters must be disposed of in a manner such as to prevent any pollutant from such materials from entering navigable waters.

G. **Bypass of Treatment Facilities.**

1. Bypass not exceeding limitations. The permittee may allow any bypass to occur that 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 Sections VII.G.2 and VII.G.3.

2. Notice.

   a. Anticipated bypass. If the permittee knows in advance of the need for a bypass, it must submit prior notice, if possible at least 10 days before the date of the bypass.

   b. Unanticipated bypass. The permittee must submit notice of an unanticipated bypass as required under Section VI.G ("Twenty-four Hour Notice of Noncompliance Reporting").

3. Prohibition of bypass.

   a. Bypass is prohibited, and the Director or ADEC may take enforcement action against the permittee for a bypass, unless:

      i. The bypass was unavoidable to prevent loss of life, personal injury, or severe property damage;

      ii. 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 judgment to prevent a bypass that occurred during normal periods of equipment downtime or preventative maintenance; and

      iii. The permittee submitted notices as required under paragraph G.2 of this Section.

   b. The Director and ADEC may approve an anticipated bypass, after considering its adverse effects, if the Director and ADEC determine that it will meet the three conditions listed above in paragraph G.3.a of this Section.

H. Upset Conditions.

1. 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 permittee meets the requirements of paragraph H.2 of this Section. 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.

2. Conditions necessary for a demonstration of upset. To establish the affirmative defense of upset, the permittee must demonstrate, through properly signed, contemporaneous operating logs, or other relevant evidence that:

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

b. The permitted facility was at the time being properly operated;

c. The permittee submitted notice of the upset as required under Part VI.G ("Twenty-four Hour Notice of Noncompliance Reporting"); and

d. The permittee complied with any remedial measures required under Section VII.D ("Duty to Mitigate").

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

I. Toxic Pollutants. The permittee must 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 establish those standards or prohibitions, even if the permit has not yet been modified to incorporate the requirement.

J. Planned Changes. The permittee must give notice to the Director and ADEC as soon as possible of any planned physical alterations or additions to the permitted facility whenever:

1. The alteration or addition to a permitted facility may meet one of the criteria for determining whether a facility is a new source as determined in 40 CFR § 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 that are subject neither to effluent limitations in the permit, nor to notification requirements under Section VI.I ("Changes in Discharge of Toxic Substances").

K. Anticipated Noncompliance. The permittee must give advance notice to the Director and ADEC of any planned changes in the permitted facility or activity that may result in noncompliance with this permit.

VIII. GENERAL PROVISIONS

A. Permit Actions. This permit may be modified, revoked and reissued, or terminated for cause as specified in 40 CFR §§ 122.62, 122.64, or 124.5. The filing of a request by the permittee for a permit modification, revocation and

reissuance, termination, or a notification of planned changes or anticipated noncompliance, does not stay any permit condition.

B. **Duty to Reapply.** If the permittee intends to continue an activity regulated by this general permit after the expiration date of this permit, the permittee must either apply for and obtain an individual permit or submit an NOI to be covered under a new general permit. In accordance with 40 CFR § 122.21(d), and unless permission for the application to be submitted at a later date has been granted by the Regional Administrator, the permittee must submit an application for an individual permit or submit a new NOI at least 180 days before the expiration date of this permit.

C. **Duty to Provide Information.** The permittee must furnish to the Director and ADEC, within a reasonable time specified in the request, any information that the Director or ADEC 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 must also furnish to the Director or ADEC, upon request, copies of records required to be kept by this permit.

D. **Other Information.** When the permittee becomes aware that it failed to submit any relevant facts in a permit application, or that it submitted incorrect information in a permit application or in any report to the Director or ADEC, it must promptly submit such facts or information.

E. **Signatory Requirements.** All applications, reports or information submitted to the Director and ADEC must be signed and certified as follows:

1. All permit applications must be signed as follows:

    a. For a corporation: by a responsible corporate officer.

    b. For a partnership or sole proprietorship: by a general partner or the proprietor, respectively.

    c. For a municipality, state, federal, or other public agency: by either a principal executive officer or ranking elected official.

2. All reports required by the permit and other information requested by the Director or ADEC must 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:

    a. The authorization is made in writing by a person described above;

b. 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, position of equivalent responsibility, or an individual or position having overall responsibility for environmental matters for the company; and

c. The written authorization is submitted to the Director and ADEC.

3. Changes to authorization. If an authorization under Section VIII.E.2 is no longer accurate because a different individual or position has responsibility for the overall operation of the facility, a new authorization satisfying the requirements of Section VIII.E.2 must be submitted to the Director and ADEC prior to or together with any reports, information, or applications to be signed by an authorized representative.

4. Certification. Any person signing a document under this Section must 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."

F. Availability of Reports. In accordance with 40 CFR Part 2, information submitted to EPA pursuant to this permit may be claimed as confidential by the permittee. In accordance with the Act, permit applications, permits and effluent data are not considered confidential. Any confidentiality claim must be asserted at the time of submission by stamping the words "confidential business information" on each page containing such information. If no claim is made at the time of submission, EPA may make the information available to the public without further notice to the permittee. If a claim is asserted, the information will be treated in accordance with the procedures in 40 CFR 2, Subpart B (Public Information) and 41 Fed. Reg. 36924 (September 1, 1976), as amended.

G. Inspection and Entry. The permittee must allow the Director, ADEC, or an authorized representative (including an authorized contractor acting as a

representative of the Administrator), upon the presentation of credentials and other documents as may be required by law, to:

1. 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;

2. Have access to and copy, at reasonable times, any records that must be kept under the conditions of this permit;

3. Inspect at reasonable times any facilities, equipment (including monitoring and control equipment), practices, or operations regulated or required under this permit; and

4. 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.

H. **Property Rights.** The issuance of this permit does not convey any property rights of any sort, or any exclusive privileges, nor does it authorize any injury to persons or property or invasion of other private rights, nor any infringement of state or local laws or regulations.

I. **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.

J. **Oil and Hazardous Substance Liability.** Nothing in this permit shall be constructed to preclude the institution of any legal action or relieve the co-permittees from any responsibilities, liabilities, or penalties to which the co-permittees is or may be subject under Section 311 of the CWA or Section 106 of the Comprehensive Environmental Response, Compensation and Liability Act of 1980 (CERCLA).

K. **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 the circumstances, and the remainder of this permit shall not be affected thereby.

APPENDIX A
DEFINITIONS

24-hour composite sample means a flow-proportioned mixture of not less than 4 discrete aliquots. Each aliquot must be a grab sample of not less than 100 ml and must be collected and stored in accordance with procedures prescribed in the most recent edition of *Standard Methods for the Examination of Water and Wastewater*.

Act means the Clean Water Act.

ADEC means Alaska Department of Environmental Conservation.

Administrator means the Administrator of the EPA, or an authorized representative.

Average Monthly Limit (AML) means the highest allowable average of "daily discharges" over a calendar month, calculated as the sum of all "daily discharges" measured during a calendar month divided by the number of "daily discharges" measured during that month.

Ballast water means harbor or seawater added or removed to maintain the proper ballast floater level and ship draft.

Best Management Practices (BMPs) means activities, prohibitions or practices, maintenance procedures, and other management practices to prevent or reduce the pollution of waters of the United States. BMPs also include treatment requirements, operating procedures, and practices to control plant site runoff, spillage or leaks, sludge or waste disposal, or drainage from raw material storage areas.

Bilge water means water which collects in the lower internal parts of the drilling vessel hull.

Biocide means any chemical agent used for controlling the growth of or destroying nuisance organisms (e.g., bacteria, algae, and fungi).

Blowout preventer fluid means fluid used to actuate hydraulic equipment on the blowout preventer.

BOD means biochemical oxygen demand.

Boiler blowdown means the discharge of water and minerals drained from boiler drums.

Bypass means the intentional diversion of waste streams from any portion of a treatment facility.

Chronic toxic unit (TU_c) is a measure of chronic toxicity. The number of chronic toxic units in the effluent is calculated as $100/IC_{25}$, where the IC_{25} is measured in percent effluent.

Coastal means any location in or on a water of the United States landward of the inner boundary of the territorial seas (40 CFR 435.40).

Cooling water means once-through non-contact cooling water.

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 units of mass, the "daily discharge" is calculated as the total mass of the pollutant discharged over the 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 day.

Deck drainage means any waste resulting from platform washings, deck washings, spillage, rainwater, and runoff from curbs, gutters, and drains including drip pans and work areas within facilities subject to this permit.

Desalination unit wastes means wastewater associated with the process of creating fresh water from seawater.

Development facilities are those operations that are engaged in the drilling and completion of production wells. These operations may occur prior to or simultaneously with production operations.

Diesel oil means the grade of distillate fuel, as specified in the American Society for Testing and Materials (ASTM) Standard Specifications for Diesel Fuel Oils D975-81, that is typically used as the continuous phase in conventional oil-based drilling fluids, which contains a number of toxic pollutants. For the purpose of this permit, "diesel oil" includes the fuel oil present at the facility.

Director means the Director of the Office of Water and Watersheds, EPA, or an authorized representative.

DMR means discharge monitoring report.

Domestic waste means materials discharged from sinks, showers, laundries, safety showers, eye-wash stations, hand-wash stations, fish cleaning stations, and galleys.

Drill cuttings means particles generated by drilling into subsurface geological formations and carried out from the wellbore with the drilling fluid. Examples of drill cuttings include small pieces of rock varying is 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.

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. The classes of drilling fluids are water-based fluid and non-aqueous drilling fluid.

Enhanced mineral oil, for the purposes of this permit, 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 phenenthrene.

Enhanced mineral oil-based drilling fluid means "drilling fluid" that has an enhanced mineral oil as its continuous phase with water as the dispersed phase.

EPA means the United States Environmental Protection Agency.

Excess cement slurry means the excess cement and wastes from equipment washdown after a cementing operation.

Exploratory facility, for the purposes of this permit, means any fixed or mobile structure that is engaged in the drilling of wells to determine the nature of potential hydrocarbon reservoirs. Any exploratory facility that begins discharging after the expiration of the previous general permit is a new exploratory facility.

Federal Waters means the contiguous zone or ocean.

Filter Backwash means wastewater generated when filters are cleaned and maintained.

Fire control system test water means the water released during the training of personnel in fire protection and the testing and maintenance of fire protection equipment.

Garbage means all kinds of victual, domestic, and operational waste, excluding fresh fish and part thereof, generated during the normal operation 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.

Grab sample is an individual sample collected over a period of time not exceeding 15 minutes.

Hydrotest water is water that is used to hydrotest the integrity of pipelines, tanks, or equipment.

IC_{25} means a point estimate of the toxicant concentration that causes a 25 percent reduction (p) in a non-quantal biological measurement (e.g., reproduction or growth) calculated from a continuous model (the EPA Interpolation Method).

LC_{50} means the concentration of effluent that is acutely toxic to 50 percent of the test organisms exposed.

M9IM means those offshore facilities continuously manned by nine (9) or fewer persons or only intermittently manned by any number of persons.

M10 means those offshore facilities continuously manned by ten (10) or more persons.

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.

Maximum daily limit (MDL) means the highest allowable "daily discharge."

Mineral oil means a class of low volatility petroleum product, generally of lower aromatic hydrocarbon content and lower toxicity than diesel oil.

Mineral oil pills (also called mineral oil spots) are formulated and circulated in the drilling fluid system as a slug in attempt to free stuck pipe. Pills generally consist of two parts; a spotting compound and mineral oil.

Marine sanitation device (MSD) means a sanitary wastewater treatment system specifically designed to meet U.S. Coast Guard requirements.

Muds, cuttings, cement at sea floor means the materials discharged at the surface of the ocean floor in the early phases of drilling operations, before the well casing is set, and during well abandonment and plugging.

New Source, for the purposes of this permit, means any facility or activity that initiates the process of surveying, clearing or preparing an area of the water body floor for the purpose of constructing or placing a development or production facility on or over the site after New Source Performance Standards have been promulgated. For Offshore Subcategory facilities, New Source Performance Standards were promulgated on March 4, 1993 (see 58 FR 12454). For Coastal Subcategory facilities that date was December 16, 1996 (see 61 FR 66125).

Non-aqueous drilling fluid (NAF) means "drilling fluid" that has water-immiscible fluid as its continuous phase and the suspending medium for solids, such as oleaginous materials (e.g., mineral oil, enhanced mineral oil, paraffinic oil, C_{16}-C_{18} internal olefins, and C_8-C_{16} fatty acid/2-ethylhexyl esters). Types of non-aqueous drilling fluids include oil-based fluid, enhanced mineral oil-based fluid, and synthetic-based fluid.

Oil-based drilling fluid means "drilling fluid" that has diesel oil, mineral oil, or some other oil, but neither a synthetic material nor enhanced mineral oil, as its continuous phase with water as the dispersed phase.

PAHs means polynuclear aromatic hydrocarbons.

Per day means through a 24-hour period.

Produced water means fluid extracted from a hydrocarbon reserve during development or production, and hydrotest water. The fluids is generally a mixture of oil, water and natural gas. This may include formation water, injection water, and any chemicals added downhole or during the oil/water separation process.

Production facilities are those operations involving active recovery of hydrocarbons from production formations. These operations may occur simultaneously with or following development operations.

QA/QC means quality assurance/quality control.

Regional Administrator means the Regional Administrator of Region 10 of the EPA, or the authorized representative of the Regional Administrator.

Sanitary wastes means human body waste discharged from toilets and urinals.

Severe property damage means substantial physical damage to property, damage to the treatment facilities that causes 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.

Sidetracked well means a new hole drilled from a main well to a different bottom-hole location.

Site means the single, specific geographical location where a mobile drilling facility (jackup rig, semisubmersible, or arctic mobile rig) conducts its activity, including the area beneath the facility, or to a location of a single gravel island.

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.

Stable ice means ice that is stable enough t support discharged muds and cuttings.

Static sheen test means the standard test procedures in appendix 1 to subpart A of 40 CFR part 435 that have been developed for this industrial subcategory for the purpose of demonstrating compliance with the requirement of no discharge of free oil.

Step-out well means a new hole drilled from a main well to a different bottom-hole location.

Stock barite means the barite that was used to formulate a drilling fluid.

Stock base fluid means the base fluid that was used to formulate a drilling fluid.

Synthetic-based drilling fluid means "drilling fluid" that has a synthetic material or a combination of synthetic materials as its continuous phase with water as the dispersed phase.

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.

Territorial Seas means the first three miles measured from the coastline or boundary between coastal and offshore waters.

Test fluid means the discharge that would occur should hydrocarbons be located during exploratory drilling and tested for formation pressure and content. This would consist of fluids sent downhole during testing along with water from the formation.

Unstable or broken ice conditions means greater than 25 percent ice coverage within a one (1) mile radius of the discharge site after spring breakup or after the start of ice formation in the fall, but not stable ice.

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.

Victual waste means any spoiled or unspoiled food waste.

Water-based drilling fluid means "drilling fluid" that has water as its continuous phase and the suspending medium for solids, whether or not oil is present.

Water depth means the depth of the water between the surface and the seafloor as measured at mean lower low water.

Waterflooding discharges means discharges associated with the treatment of seawater prior to its injection into a hydrocarbon-bearing formation to improve the flow of hydrocarbons from production wells, and prior to its use in operating physical/chemical treatment units for sanitary waste. These discharges include strainer and filter backwash water.

Well completion fluids are 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.

Workover fluids are salt solutions, weighted brines, polymers, or other specialty additives used in a producing well to allow for maintenance, repair, or abandonment procedures. Drilling fluids used during workover operations are not considered workover fluids by definition. Packer fluids (low solid fluids between the packer, production string, and well casing) are considered to be workover fluids.

4-day LC_{50} as applied to the sediment toxicity means the concentration (milligrams/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.

10-day LC_{50} as applied to the sediment toxicity means the concentration (milligrams/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 ten days of constant exposure.

96-hour LC_{50} 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.

C_{12}-C_{14} *ester* and C_8 *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.

C_{16}-C_{18} *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 15, and internal double carbon bond, and is represented by the Chemical Abstracts Service (CAS) No. 27070-58-2.

C_{16}-C_{18} *internal olefin drilling fluid* means a C_{16}-C_{18} internal olefin drilling fluid formulated as specified in Appendix 8 of subpart A of 40 CFR Part 435 (See Attachment 9 of this permit).

APPENDIX B

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 textand 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 μm 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 μm 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, Schlekat 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 (Schlekat et al. 1992).

However, the fine fraction of sediments in the Schlekat 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 μm or similar mesh sieve to remove large debris and then through a 500 μm mesh sieve to remove any indigenous organisms. Sediments have also been press-sieved through a 250 to 350 μm mesh sieve prior to testing to aid in the enumeration of amphipods on a 500 μm mesh sieve at test termination.

Storage

The control sediment should be stored in plastic or glass containers at 4±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 NAFsand 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):

1. Determine the wet to dry weight ratio for the control sediment (three replicates of 30 g each as been used successfully);
2. Determine the density (g/ml) of the control sediment (three replicates of >25 ml is suitable for this purpose);
3. 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;

4. Mix NAF or SBM with control sediment;
5. Test for homogeneity of NAF or SBM in sediment, and;
6. 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:

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

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

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 to15 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±1˚C at 20±1‰ 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 μm 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 toxicants for the NAF test will be either a C_{16}-C_{18} -internal olefin reference

standard or a C_{12}-C_{14} or C_8 ester. The reference toxicant for the SBM testing program will be a C_{16}-C_{18} internal olefin SBM which has also been specified for determining pass/fail for SBMs. The C_{16}-C_{18} 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 LC_{50} 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 LC_{50} 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 Results

Procedures presented in this test method are used to calculate point estimates, or LC_{50} values. The LC_{50} 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 LC_{50} 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 LC_{50} value and its 95% confidence limits, including probit, moving average, trimmed Spearman-Karber 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 (C_{16}-C_{18} internal olefin or C_{12}-C_{14} or C_8 ester). This limitation is expressed as follows:

The only SBMs that will be allowed for discharge are those that are as toxic or less toxic, but not more toxic, than the C_{16}-C_{18} internal olefin reference SBM. This limitation is expressed as follows:

$$\frac{96-hr\ LC_{50}\ RDF}{96-hr\ LC_{50}\ SBM} \leq 1.00 \qquad \frac{10-day\ LC_{50}\ \text{Re}\,ference\ Material}{10-day\ LC_{50}\ NAF} \leq 1.00$$

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 LC_{50} values for *L. plumulosus*. Hence, the NAF or SBM data should be interpreted by comparing to the reference toxicant test LC_{50} 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 μm mesh sieve to remove large debris and then through a 500 μm 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 tricornutum*, 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 µm mesh sieve and are retained on a 710 µm mesh sieve. A 500 µm 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 µm mesh sieve was used to retain amphipods in later experiments. The amphipods passing through a 1000 µm mesh sieve but trapped on a 710 µm 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±1°C
Salinity	20±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
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)*.

- 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

Parameter	Conditions
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

References

American Society for Testing and Materials (ASTM). 1997. Standard Guide for Conducting 10-day Static Sediment Toxicity Tests with Marine and Estuarine Amphipods. E 1367-92. Annual Book of ASTM Standards, Vol. 11.05.

American Society for Testing and Materials (ASTM). 2000. Standard Guide for Conducting 10-day Static Sediment Toxicity Tests with Marine and Estuarine Amphipods. E 1367-99. Annual Book of ASTM Standards, Vol. 11.05.

Emery, V.L., D.W. Moore, B.R. Gray, B.M. Duke, A.B. Gibson, R.B. Wright and J.D. Farrar. 1997. Development of a chronic sublethal sediment bioassay using the estuarine amphipod *Leptocheirus plumulosus* (Shoemaker). Environ. Toxicol. Chem. 16:1912-1920.

McGee, B.L. D.J. Fisher, L.T. Yonkos, G.P. Ziegler and S. Turley. 1999. Assessment of sediment contamination, acute toxicity, and population viability of the estuarine amphipod *Leptocheirus plumulosus* in Baltimore Harbor, Maryland, USA. Environ. Toxicol. Chem. 18:2151-2160.

Schlekat, C.E., B.L. McGee and E. Reinharz. 1992. Testing sediment toxicity in Chesapeake Bay with the amphipod *Leptocheirus plumulosus*: an evaluation. Environ. Toxicol. Chem. 11:225-236.

Suedel, B.C. and J.H. Rodgers, Jr. 1991. Variability of bottom sediment characteristics of the continental United States. Water Res. Bull. 27:101-109.

USEPA. 1994. Methods for Assessing the Toxicity of Sediment-associated Contaminants with Estuarine and Marine Amphipods. EPA/600/R-94/025. USEPA Office of Research and Development, June 1994.

USEPA. 2000. Final Effluent Limitations Guidelines and Standards for Synthetic-Based Drilling Fluids Fact Sheet. EPA-821-F-00-014. USEPA Office of Water, December 2000. (www.epa.gov/ost/guide/sbf/final/finalfact.html).

APPENDIX C

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)ºC. 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±1ºC 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 dos not constitute endorsement by EPA or the authors

Section 2.2: Seawater Requirements

Synthetic seawater at a salinity of 25 ±1 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 4ºC 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.

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

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:

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

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 g dry sediment per bottle x 8 = 240 g dry sediment [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:

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

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 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:

$$480 \text{ mg carbon} \div (77.3/100) = 621 \text{ mg ethyl oleate} \qquad [5]$$

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 g dry sediment x wet:dry ratio = g wet sediment needed [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 g dry sediment x wet:dry ratio = g wet sediment added to each bottle [7]

g wet sediment ÷ density (g/mL) of wet sediment = mL wet sediment [8]

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

75 mL total volume - mL wet sediment (from eq. 8) = mL of sea water [9]

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

volume sea water per bottle (eq. 9)
---------- = ratio of sea water:wet sediment [10]
volume sediment per bottle (eq. 8)

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

g wet sediment (eq. 6) ÷ density = volume (mL) of sediment [11]

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

mL wet sediment(eq. 11) x sea water:sediment ratio (eq. 10)
= mL sea water to add to wet sediment [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

mL sediment (eq. 8) x density of wet sediment (g/mL) = g wet sediment [14]

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

mL sea water (eq. 9) x density of sea water (1.01 g/mL) = g sea water [15]

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

g wet sediment (eq. 14) + g sea water (eq. 15) = 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 ($0^oC = 273^oK$) and pressure (1 atm = 30 inches of Hg) using Eqn.17.

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

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^oK
T_1 = temperature on day of sampling ($^oC + 273 = {}^oK$)
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 mg carbon/kg sediment x (30 g/1000) = 60 mg carbon per bottle [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 mg carbon per bottle/1000) ÷ 12 g/mole = 0.005 moles carbon [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 moles x 22.4 L = 0.112L (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	$\geq$ 60% theoretical	$\leq$ 5% theoretical	$\geq$ 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:

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

Where: NAF = stock base fluid being tested for compliance

Reference Fluid = C_{16}-C_{18} internal olefin or C_{12}-C_{14} or C_8 ester reference fluid

Appendix C-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 CO_2 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.

$$\text{Corrected \% } CH_4 = \frac{\%CH_4}{1 - \frac{[D * 22.4\ L/mol}{18\ g/mol * 1000}} \qquad [A1]$$

Where:

D = the density of water vapor at saturation (g/m^3, can be found in CRC Handbook of Chemistry and Physics) for the temperature of sampling.

$$V_{CH_4}(ml) = (S + V) * \frac{(P - P_w)}{(T + 273)} * \frac{CH_4}{100} * \frac{273}{760} \qquad [A2]$$

where: V_{CH4} = 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)
CH_4 = % 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 wither CO_2 or CH_4, 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.

$$\mathbf{C_nH_aO_bN_cS_d + (n\text{-}a/4 - b/2 + 7c/4 + d/2)\ H_2O \rightarrow (n/2 - a/8 + b/4 - 5c/8 + d/4)\ CO_2 + (n/2 + a/8 - b/4 - 3c/8 - d/4)\ CH_4 + cNH_4HCO_3 + dH_2S.} \qquad \textbf{[A3]}$$

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 ($C_{16}H_{32}$) will yield 4 moles of CO_2 and 12 moles of CH_4. 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.

$$\frac{12\ \text{mole } CH_4}{\text{mole hexadecene}} * \frac{22.4\ \text{L}}{\text{mole } CH_4} * \frac{1000\ \text{ml}}{\text{L}} * \frac{1\ \text{mole hexadecene}}{224.4\ \text{g hexadecene}} *$$

$$\frac{2.3\ \text{g hexadecene}}{\text{kg dry soil}} * \frac{0.03\ \text{kg}}{\text{culture}} = 84\ \text{ml} \qquad [A4]$$

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 C-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 ≤20% Coefficient of Variability and an average of ≥70% to ≤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.

Appendix C-3

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 with 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 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 D
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 contaminates.

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 ±10˚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 ± 1% and 0.5-mL ± 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 25´89 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) ´ 39 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 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.

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 ±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:

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

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-µL aliquot of the 1.25 mg/mL SPTM standard with 500 µL of the TCB internal standard solution.

10.3.2 Inject 1.0 µL 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-µL aliquot of the 0.125 mg/mL SPTM standard with 500 µL 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 µL 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 3.5e+07.

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 ³ 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 ³ 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).

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

11.2.7 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-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).

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 it 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 s ample, 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 form 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.

APPENDIX E

NOTICE OF INTENT (NOI) INFORMATION SHEET
NPDES GENERAL PERMIT AKG-31-5000
OIL AND GAS EXPLORATION FACILITIES
ON THE OUTER CONTINENTAL SHELF AND CONTIGUOUS STATE WATERS

APPLICANT (*Owner/Operator*)			
Owner Name:		Operator Mailing Address:	
Telephone Number:			
Operator Name:			
Telephone Number:			

FACILITY				
Facility Name:		Facility Mailing Address:		
Contact Name:				
Telephone Number:				
Beginning Date of Operation:		Stationary Facilities	Latitude:	
Expected Duration of Operation:			Longitude:	
Facility Type (*check applicable type*)	☐ Jackup	Mobile Facilities	Initial Latitude:	
	☐ Drill Ship			
	☐ Semisubmersible		Initial Longitude:	
	☐ Other (*specify*):			

Submit a site map showing the exact location of facility and discharges associated with the project. Mobile facilities may designate an area where they may be operating and must include a map showing those areas and a description of operations within those areas. If the operation is within 4000 meters of a prohibited area of discharge indicated by the permit, those areas and their distance from the operation must be shown on the map.

RECEIVING WATER – Cook Inlet			
☐	Coastal Waters	☐	Territorial Seas
☐	Offshore Waters		

Initial date and expected duration of operations:

LOCATION OF DISCHARGE					
MMS	Lease Number		ADNR	Lease Number	
	Block Number			Block Number	
Range of water depths below mean lower low water (MLLW) in the lease block:	From:		To:		

NOTICE OF INTENT (NOI) INFORMATION SHEET
NPDES GENERAL PERMIT AKG-31-5000
OIL AND GAS EXPLORATION FACILITIES
ON THE OUTER CONTINENTAL SHELF AND CONTIGUOUS STATE WATERS

Discharges (*check all that apply*)			
☐	001 Drilling Mud and Cuttings	Water Depth:	
☐	002 Deck Drainage	Water Depth:	
☐	003 Sanitary Waste	Water Depth:	
☐	004 Domestic Waste	Water Depth:	
☐	005 Desalination Unit Waste	Water Depth:	
☐	006 Blowout Preventer Fluid	Water Depth:	
☐	007 Boiler Blowdown	Water Depth:	
☐	008 Fire Control System Test Water	Water Depth:	
☐	009 Non-Contact Cooling Water	Water Depth:	
☐	010 Uncontaminated Ballast Water	Water Depth:	
☐	011 Bilge Water	Water Depth:	
☐	012 Excess Cement Slurry	Water Depth:	
☐	013 Mud, Cuttings, Cement at Seafloor	Water Depth:	
☐	014 Waterflooding Discharges	Water Depth:	
☐	015 Produced Water and Produced Sand	Water Depth:	
☐	016 Completion Fluids	Water Depth:	
☐	017 Workover Fluids	Water Depth:	
☐	018 Well Treatment Fluids	Water Depth:	
☐	019 Test Fluids	Water Depth:	

Type of Sanitary Discharge:
☐ M10 ☐ M91M
☐ Other (specify):

Provide a brief description of the treatment process(es) and disposal practices (e.g., backhauled, reinjected, discharged, etc.) at the facility.

Provide a line drawing that shows flows of discharged waste streams through the facility. Indicate intake sources, operations contributing to the effluent, and treatment units labeled to correspond to the discharges (001 - 019). Construct a flow balance on the line drawing by showing average flows between intakes, operations, treatment units, and outfalls. If a flow balance cannot be determined, provide a pictorial description of the nature and amount of any sources, and any collection or treatment measures.

Drilling Fluid

Category (*check all that apply*)			Group (*check all that apply*)		
	☐	Water-based		☐	Lignosulfonate
	☐	Oil-based		☐	Lime
	☐	Synthetic-based		☐	Gyp
	☐	Other (*specify*):		☐	Sea-water
				☐	Saltwater
Estimated Total Discharge Volume:				☐	Saturated Saltwater
				☐	Nondispersed (Viscosifier/Polymer)

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100-Meter Mixing Zone Request
(*applicable to sanitary discharges within State of Alaska/Coastal Waters*)

Are you requesting a mixing zone from ADEC?	☐	Yes (*continue filling out this section*)	☐	No (*skip this section and proceed to Special Conditions, below*)

THE FOLLOWING INFORMATION MUST BE PROVIDED IF REQUESTING A MIXING ZONE. The burden of proof for justifying a mixing zone through demonstrating compliance with the requirements of 18 AAC 70.240 through 18 AAC 70.270 rests with the applicant.

Distance from shoreline of discharge point or first port of diffuser (measured at M.L.L.W.):		Length of diffuser:	
Depth of discharge port or diffuser (measured at M.L.L.W.):		Diameter of port(s):	
Orientation of diffuser to shoreline (e.g., perpendicular, 45°, parallel):		Number of ports:	
Maximum current:		Port spacing:	

USES OF RECEIVING WATER AT DISTANCE FROM DIFFUSER i.e. Supply for drinking water, Supply for agriculture including irrigation & stock water, Supply for aquaculture, Supply for industrial use, Contact recreation, Secondary recreation, Fish spawning, Harvesting and consumption of raw fish, or other aquatic life (Not needed if not requesting a mixing zone from ADEC):

If possible, provide salinity and temperature data from the receiving water surface to the depth of the discharge port or diffuser.

Special Conditions (*provide justification for all that are not required, completed or provided*)

Special Monitoring	☐	Required	☐	Not Required	Justification:
Exploration Plans	☐	Attached	☐	Not Provided	Justification:
Biological Survey(s)	☐	Attached	☐	Not Provided	Justification:
Environmental Report(s)	☐	Attached	☐	Not Provided	Justification:
Drilling Fluid Plan	☐	Complete	☐	Not Complete	Justification:
Environmental Monitoring Study Plan (II.B.5.)	☐	Attached	Date of Submittal: _____		

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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.			
Signature:		Date:	
Printed Name:		Title:	

Mail Completed NOI to EPA and ADEC at the following addresses:	
US EPA 1200 6th Avenue, M/S OWW-130 Seattle, WA 98101	ADEC, Water Division 555 Cordova Street Anchorage, Alaska 99501